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Recommended Practices for Core Analysis

RECOMMENDED PRACTICE 40
SECOND EDITION, FEBRUARY 1998

Strategies for Today's
Environmental Partnership

American
Petroleum
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STEP

Strategies for Today's
Environmental Partnership

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Recommended Practices for Core Analysis

Exploration and Production Department

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FOREWORD

Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

SCOPE

These recommended practices for core analysis replace API RP 40, *Recommended Practice for Core Analysis Procedure*, 1960, and API RP 27, *Recommended Practice for Determining Permeability of Porous Media*, 1952, (reissued 1956). In the first section of the new recommended practices, Planning a Coring Program, key factors to be taken into consideration in obtaining core samples are explained and advantages of different coring procedures are given. The second section, Wellsite Core Handling Procedures and Preservation, addresses documentation of coring conditions and how cores should be handled once they reach the surface, including marking and preservation. The third section, Core Screening and Core Preparation, describes how the condition and nature of core samples can be documented through core gamma logs and various imaging techniques, and how samples should be selected and prepared for basic testing. Also covered in the third section are methods of preserving samples prior to testing, and procedures for cleaning and drying samples. The fourth section, Fluid Saturation, explains how fluid saturations can be determined on different types of samples and the limitations of the various techniques. The fifth section, Porosity Determination, defines different types of porosity and explains the measurements. The sixth section, Permeability Determination, explains the theory and methods for measurement of permeability of porous media to a single phase. Relative permeability measurements to two or three phases are not covered in this document. The seventh section, Supplementary Tests, covers determination of grain size, brine salinity, oil gravity, and acid solubility. The eighth and final section, Reporting, supplies forms to assist in recording the details of core handling and testing methodology that could be critical in interpreting basic core analysis data.

Core analysis, like other technical areas, is continually evolving so that both methodology and costs are changing. The recommended practices provided here represent a snap shot in time of the consensus advice of a large international committee.

Geologic materials come in a vast range of chemical compositions and physical states. For unusual samples or extraordinarily accurate data, it may be necessary to develop special procedures.

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Recommended Practices for Core Analysis

1 Planning a Coring Program

1.1 GENERAL

1.1.1 Scope

This section addresses the complexities of planning a coring program, the decisions to be made, and the factors that influence the choices.

1.1.2 Principle

A coring program is similar to many engineering projects. It begins with the premise that an investment will reap a reward. It progresses through a phase of exploring alternate sources of information; well tests, logs, previous cores, and cuttings or sidewall cores.

Planning begins by listing the objectives of the coring program. This is best done by a team of petrophysical, reservoir, geological, drilling, and production personnel. When discussing objectives, every expenditure must ultimately lead to producing more oil or gas at a lower unit cost. Constraints in budget, location, and timing will be placed on the program. Hole size, hole angle, temperature, pressure, and rock type will influence the selection of the coring tools. Planning becomes an interactive process where consensus is built and a detailed program formulated.

The keys to a successful coring operation are planning and communication.

1.1.3 Objective

The objective of every coring operation is to gather information that leads to more efficient oil or gas production. Some specific tasks might include the:

a. Geologic objectives:
   1. Lithologic information:
      (a) Rock type.
      (b) Depositional environment.
      (c) Pore type.
      (d) Mineralogy/geochemistry.
   2. Geologic maps.
   3. Fracture orientation.

b. Petrophysical and reservoir engineering:
   1. Permeability information:
      (a) Permeability/porosity correlation.
      (b) Relative permeability.
   2. Capillary pressure data.
   3. Data for refining log calculations:
      (a) Electrical properties.
      (b) Grain density.
      (c) Core gamma log.
      (d) Mineralogy and cation exchange capacity.
   4. Enhanced oil recovery studies.
   5. Reserves estimate:
      (a) Porosity.
      (b) Fluid saturations.

c. Drilling and completions:
   1. Fluid/formation compatibility studies.
   2. Grain size data for gravel pack design.
   3. Rock mechanics data.

1.1.4 Coring Fluids

1.1.4.1 The selection of a coring fluid should be based on four points:

a. Safety.
b. The primary objective of the coring program.
c. Environmental concerns.
d. Cost.

1.1.4.2 Safety takes precedence over all other factors. The drilling fluid must be designed to hold the expected formation pressures as well as clean, lubricate, and stabilize the borehole. The objectives of the coring program should influence the selection of the coring/drilling fluid. All coring fluids should be designed to have low static API filter loss and very low dynamic spurt loss to minimize core flushing.

1.1.4.3 Environmental concerns should also be considered and budgeted for. This may mean using a more expensive drilling fluid system to meet environmental objectives, or providing additional drilling fluid handling equipment to ensure containment.

1.1.4.4 Cost is important; still, it is a good practice to review the cost of the entire core analysis program and the expected benefits from it while pricing drilling fluid systems. Savings on drilling fluids may increase the cost of the core analyses, and put the accuracy of the core studies at risk.

1.1.4.5 The question of which drilling fluid is best for coring cannot be answered directly. Water-based, oil-based, foam, and air/mist drilling fluids have all been used to successfully cut cores. The best recommendation is to follow the criteria given above. Evaluating the needs of the drilling and core analysis program will lead to an appropriate selection.

1.2 CORING EQUIPMENT

1.2.1 Scope

This section presents an overview of coring tools, including guidelines for selecting coring tools for specific applications. Details of particular coring systems, and job specific coring recommendations should be obtained from appropriate service companies.

1.2.2 Principle

Coring equipment is designed to retrieve rock samples from deep in the earth for geologic and engineering studies. The tools do an excellent job of recovering core material, and specialized equipment has been developed to trap reservoir fluids and even seal in bottom-hole pressure.

1.2.3 Apparatus

With several notable exceptions coring systems consist of an inner core barrel suspended by a swivel assembly within an outer core barrel that is attached to the drill string. A coring bit is attached to the bottom of the outer barrel and a core catcher is fitted to the bottom of the inner core barrel. Drilling fluid is pumped down the drill string, through the swivel assembly, through the annulus between the inner and outer core barrels, and out the core bit.

1.3 CONVENTIONAL CORING SYSTEMS

1.3.1 Conventional Core Barrel

Conventional coring tools are available to cut cores with outer diameters from 1.75 to 5.25 inches (44.5 to 133.4 millimeters). Core length can run from 1.5 feet (.46 meter) for short radius horizontal well applications to over 400 feet (121.9 meters) for thick, uniform, consolidated formations. Hole size, hole angle, rock strength, and lithology will control the diameter and length of core that may be cut in one trip. The final selection of a particular system will depend upon the formation, location, and objectives of the coring program. Table 1-1 summarizes the conventional coring options available.

1.3.2 Heavy-Duty Conventional Core Barrels

Special heavy-duty coring tools have been developed to core harder than normal formations, and cut extended length cores. Heavy duty threads allow more torque to be applied to the bit, and improve the margin of safety against tool failure. Designed to cut cores up to 5.25 inches (133.4 millimeters) in diameter, these tools are especially attractive in situations where rig time is the largest coring expense. Heavy-duty coring systems are used to best advantage when coring longer lengths of homogeneous formations or when anticipating higher than normal torque loads.

The marine core barrel was the precursor to today's generation of heavy-duty core barrels. Developed to be stronger than existing coring systems, the tool was developed for use in offshore applications. The marine core barrel does increase the margin of safety against tool failure, but is restricted to cutting a 3-inch (76.2-millimeter) diameter core.

1.3.3 Core Barrel Liners

The use of a core barrel liner in a steel inner core barrel has two primary functions: to improve core quality by physically supporting the core material during handling and to serve as a core preservation system. PVC and ABS plastic, fiberglass, and aluminum have all been used as inner core barrel liners. The liners slip inside a conventional inner core barrel and are held in place by the core-catcher assembly and friction. Liners are typically 30 feet (9.14 meters) long. They may be cut shorter for special applications, but their maximum length is rarely more than 30 feet (9.14 meters) due to manufacturing and material handling limitations.

Liners are most often specified when coring unconsolidated or fractured formations. They are also appropriate when cutting hard rock in remote and offshore locations when immediate core preservation is required. Plastic liners are suitable up to temperatures of 180°F (82.2°C). Fiberglass liners may be used up to 250°F (121°C); 350°F (176.7°C) if special high temperature resin is used. Aluminum is generally recommended when temperatures in excess of 250°F (121°C) are expected. The disadvantage of core barrel liners is that

Table 1-1—Conventional Coring Systems

Inner Barrel	Core Length	Special Features
Mild steel	30 to 120 ft. (9.14 to 36.58 m)	Ready-made core preservation system. High temperature applications.
Mild steel	1.5 ft. (.46 m)	Designed for short-radius coring.
High strength steel	120 to >400 ft. (36.38 to >121.9 m)	Stronger barrel, includes additional inner and outer core barrel stabilization.
Fiberglass	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. Used for consolidated and unconsolidated formations. Maximum operating temperatures: normal resin 250°F (121°C), high temperature resin 350°F (176.7°C).
Aluminum	30 to 90 ft. (9.14 to 27.43 m)	Ready-made core preservation system. High temperature applications, maximum 350°F (176.7°C).
Steel with a plastic liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 180°F (82.2°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with a fiberglass liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 250°F (121°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).
Steel with an aluminum liner	30 ft. (9.14 m)	Ready-made core preservation system. Maximum temperature of 350°F (176.7°C). Reduces core diameter by $^1/_2$ in. (12.7 mm).

they reduce the effective diameter of the inner core barrel by approximately 0.5 inch (12.7 millimeters).

1.3.4 Disposable Inner Core Barrels

Disposable inner core barrels serve the same general purposes as core barrel liners. They improve core quality by physically supporting the core material during handling and serve as a core preservation system. In addition, the outside diameter of the core is not reduced, as it would be with an inner barrel liner. Disposable inner core barrels are available in aluminum, fiberglass, and mild steel, and are manufactured in a variety of sizes to fit most conventional coring systems. In addition, the fiberglass inner core barrel has a low coefficient of friction that allows the core to slide more easily into the core barrel, thereby reducing the risk of core jamming.

1.3.5 Coring High Angle or Horizontal Well

Medium radius [290 to 700 feet (88.4 to 213.4 meters) radius] and extended length wells can be cored with conventional core barrels powered from the rotary table or by a downhole motor. Most cores will be cut without using a downhole motor, but cases will arise where the use of a mud motor is justified. Using a downhole motor enables coring to proceed without rotating the drill string. Typically a 30-foot (9.14-meter) long conventional core barrel would be placed ahead of the downhole mud motor. Mud motors produce high torque at low rotating speed for optimum coring power. Core barrel length and core diameter may be varied to accommodate drilling constraints. The inner core barrel is stabilized by fitting it with special roller bearing or bushing assemblies to centralize the inner core barrel. A special drop ball sub may be placed between the motor and core barrel to allow drilling fluid to flow through the inner core barrel, cleaning it of debris before coring. Activating the sub diverts the drilling fluid flow between the inner and outer core barrel for coring.

In some instances during coring it may be necessary to keep very tight control on the angle of the well. Coring without the downhole motor may improve well-angle control.

1.4 SPECIAL CORING SYSTEMS

1.4.1 General

Special coring systems have evolved to fill specific coring needs. Pressure-retained and sponge core barrels arose from a need for better oil saturation data. The rubber-sleeve and full-closure coring systems were developed specifically to improve the quality of cores cut from unconsolidated formations. Other special coring systems have equally unique capabilities, making them all useful to the engineers and geologists employing them. Table 1-2 summarizes some of the available special coring options.

1.4.2 Pressure-Retained Coring

Pressure-retaining core barrels are designed to retrieve cores maintained at reservoir pressure conditions. Accepted as the best method for obtaining core-based oil saturation data, pressure-retained cores also capture reservoir gases. The tool is especially useful when studying the feasibility of enhanced recovery projects and estimating the methane content of coal.

Pressure-retained core barrels are available in two sizes: 6-inch (152.4-millimeter) and 8-inch (203.2-millimeter) outside diameter that cut cores 2.50- and 3.75-inch (63.5- and 95.3-millimeter) outside diameter, respectively. The 6-inch (152.4-millimeter) outside diameter barrel cuts up to 20 feet (6.1 meters) of 2.5-inch (63.5-millimeter) diameter core while holding a maximum of 10,000 psi (69 MPa) pressure. The 8-inch (203.2-millimeter) outside diameter barrel cuts 10 feet (3.05 meters) of 3.75-inch (95.3-millimeter) diameter core while retaining a maximum of 5,000 psi (34.5 MPa) internal pressure. The maximum recommended operating temperature is 180°F (82°C).

Pressure core barrels are sophisticated tools requiring an on-site facility to service the barrel and handle the pressurized cores. Core handling procedures may be found in 2.2.5.

Table 1-2—Special Coring Systems

Coring System	Maximum Core Dimensions	Special Applications
Pressure-retained	3.75 in. x 10 ft. (5000 psi) [95.3 mm x 3.05 m (34.5 MPa)] 2.5 in. x 20 ft. (10000 psi) [63.5 mm x 6.1 m (69 MPa)]	Pressure-retained analyses, fluid saturations, gas volume and composition.
Sponge-lined	3.5 in. x 30 ft. (88.9 mm x 9.1 m)	Fluid saturations.
Full-closure	4.0 in. x 60 ft. (101.6 mm x 18.3 m)	Recovering unconsolidated formations.
Rubber-sleeve	3.0 in. x 20 ft. (76.2 mm x 6.1 m)	Recovering unconsolidated, fractured, or conglomeritic formations.
Wireline retrievable	2.75 in. x 30 ft. (69.9 mm x 9.1 m)	Coring is possible without tripping pipe.
Wireline percussion sidewall	1 in. x 1.75 in. (25.4 mm x 44.5 mm)	Samples obtained after drilling and logging.
Wireline drilled sidewall	.94 in. x 1.75 in. (23.9 mm x 44.5 mm)	Samples obtained after drilling and logging.
Sidewall corer	2.5 in. x 10 ft. (63.5 mm x 3.05 m)	Core obtained after drilling and logging.

1.4.3 Sponge-Lined Coring System

The sponge-lined coring system was developed to improve the accuracy of core-based oil saturation data. A sponge coring system does not trap reservoir gases, instead it traps oil expelled as the core is brought to the surface. The saturation information is very useful when evaluating enhanced oil recovery projects.

A sponge coring system has the advantage of being less expensive to operate than a pressure-retained coring system, while providing an opportunity to improve the accuracy of the core based oil saturation data. The sponge is stable to a temperature of 350°F (176.7°C). The sponge coring system is limited to cutting a maximum of 30 feet (9.14 meters) of 3.5-inch (88.9-millimeter) diameter core per trip.

1.4.4 Full-Closure Coring Systems

Full-closure coring systems were developed to improve the recovery of unconsolidated formations. These systems use core barrel liners or disposable inner core barrels, and a special core catching system to retrieve the troublesome rocks.

Full-closure coring technology allows the inner core barrel to slip gently over soft core with a minimum of disturbance, and then seal the core within the core barrel. This is done by using a full-closure core catcher assembly that allows unobstructed entry of the core into the inner core barrel, and then after coring seals off the bottom of the inner barrel. Full-closure coring systems are currently limited to cutting either 3.5-inch (88.9-millimeter) or 4-inch (101.6-millimeter) diameter cores. The recommended core length is 30 feet (9.14 meters). The smooth bore and the absence of an exposed core catcher may result in lost core if the tool is lifted off bottom before activating the full-closure core catcher.

1.4.5 Rubber-Sleeve Core Barrel

The rubber-sleeve coring system was the first system developed to improve the chances for recovering unconsolidated sands, conglomerates, and hard fractured formations. The rubber-sleeve barrel is unique in that the top of the inner barrel does not move relative to the core during coring. The outer barrel is drilled down around a column of rock that is progressively encased in a rubber sleeve. The rubber sleeve is smaller than the diameter of the core; it stretches tightly around the core, wrapping it securely and protecting it from the scouring action of the drilling fluid. The core is supported by the rubber sleeve thus, aiding in the recovery of soft formations that would not support their own weight.

There is only one size of rubber-sleeve core barrel, that cuts 20 feet (6.1 meters) of 3-inch (76.2-millimeter) diameter core per trip. The rubber sleeve itself is limited to temperatures no higher than 200°F (93°C). The tool is not recommended for use in holes with more than 45 degrees of inclination. In addition, coring must be stopped approximately every two feet to allow the tool to be reset; this might lead to core jamming in fractured formations. The system works best from fixed drilling structures, yet it can be operated from floating rigs if rig movement is minimal.

1.4.6 Wireline-Retrievable Core Barrel

Wireline-retrievable coring tools are operationally similar to conventional coring systems except they are designed for the inner core barrel to be pulled to the surface by a wireline. This speeds the coring operation by eliminating the need to trip the entire drill string for each core. A new section of inner core barrel is pumped down the drill string and latched into place for additional coring, or a drill plug is pumped down to facilitate drilling ahead.

Wireline-retrievable coring tools are usually smaller and lighter than conventional coring systems. This is an asset when they must be transported to remote locations or by helicopter. Unfortunately, the core diameters are limited since the entire inner core barrel assembly must pass through the drill string. Also, care must be taken to prevent “swabbing” oil or gas into the wellbore as the inner barrel is recovered.

1.5 WIRELINE SIDEWALL CORING

1.5.1 General

Wireline sidewall coring systems were developed to obtain core samples from a wellbore after it has been drilled and logged, and before casing is run. These tools may be positioned in zones of interest using data from gamma or spontaneous potential logs as guides. The samples provide small pieces of formation material, suitable for geologic and engineering studies.

1.5.2 Percussion Sidewall Coring

Most wireline sidewall cores are obtained by percussion sidewall coring systems. These tools shoot hollow, retrievable, cylindrical bullets into the wall of an uncased hole. The tool (gun) is lowered to the desired depth on a wireline, and then fired by electrical impulses controlled from the surface. The bullets remain connected to the gun by wires, and movement of the gun pulls the bullets, containing the samples, from the hole wall. Up to 66 samples, 1 inch (25.4 millimeters) in diameter by $1^3/_4$ inches (44.5 millimeters) in length, may be taken during one downhole trip. Different bullet “core barrel” designs are available for unconsolidated, soft, and medium-to-hard formations. It is wise to have more than one type of core barrel on location until acceptable core recovery can be shown.

The advantages of percussion sidewall coring are speed, low cost, and the ability to sample zones of interest after open hole logs have been run. The disadvantage is that the bullet usually alters the formation, shattering harder rock or compressing softer sediments. This reduces the quantitative value of the sidewall core analysis data. Percussion sidewall core recovery tends to be low in very hard or fractured rock, and in very permeable unconsolidated sand.

1.5.3 Drilled Sidewall Coring

The rotary or drilled sidewall coring tool was developed to recover wireline sidewall core samples without the shattering impact of the percussion system. Suitable for hard-to-friable rock, the rotary sidewall coring tool uses a diamond-tipped drill to cut individual samples. Leverage applied to the drill snaps the sample from the sidewall. The drill and sample are retracted into the body of the tool where the sample is deposited. The tool is moved to a new location after depositing each sample. A maximum of 30 samples, $^{15}/_{16}$-inch (23.9 millimeter) diameter by $1^{3}/_{4}$-inch (44.5-millimeter) length, may be taken during one trip.

An advantage of the rotary sidewall coring system is that it produces samples of hard rock suitable for quantitative core analysis. Disadvantages are that it is more expensive than percussion sidewall coring in terms of rig time costs, and sample recovery tends to be low in unconsolidated formations.

1.5.4 Sidewall Coring Systems

Some new sidewall coring systems are coming on the market, and they merit discussion for two reasons. First, they are designed to acquire a larger, more-continuous core sample from a drilled and logged wellbore than is possible with existing sidewall coring tools. Secondly, the emergence of new tools confirms there is still room for improvement in the area of acquiring high quality, low cost core samples.

The first system is similar to a conventional core barrel. The sidewall coring system is designed to cut up to 10 feet (3.05 meters) of $2^{1}/_{2}$-inch (63.5-millimeter) diameter core. The tool is attached to a conventional drill string and lowered to the zone of interest. There an integral arm pushes the core barrel against one side of the wellbore. From then on the tool is operated as a conventional core barrel. The second system uses a removable whipstock to direct a conventional core barrel out into the formation. Both systems address the need to acquire quality core samples after logging.

1.6 ORIENTED CORING

1.6.1 General

Oriented cores are used to orient fractures, stress fields, and permeability trends. Exploration, production, and drilling operations use the information to explore for fractured reservoirs, design waterfloods, and plan horizontal wells.

Oriented cores are typically cut using a conventional core barrel fitted with a special scribe shoe, and a device for recording the orientation of the primary scribe knife relative to magnetic north. Laboratory methods used to orient cores are correlation of the core with borehole imaging logs and the paleomagnetic method. Table 1-3 lists methods commonly used to orient cores.

Table 1-3—Core Orientation Methods

Method	Location	Comments
Multishot survey	Well	Must stop drilling to take reading.
Electronic survey	Well	Records orientation versus time.
Paleomagnetic method	Laboratory	Orients one continuous interval.
Log correlations	Laboratory	Requires correlatable features in core and wellbore.

1.7 CORING BITS

1.7.1 General

Coring bits are a basic part of the coring system. Unfortunately for the experts and novices alike, coring bits come in a bewildering array of styles. Fortunately, general bit/formation guidelines are available from manufacturers to aid in selection of the proper bit. With a little background information, it is possible to make informed decisions on cutter types, bit profile, and hydraulic considerations for the range of anticipated coring conditions. Final bit selection should be guided by the goals of the coring program, coupled with a confirmation that the bit has proven itself in the field for similar applications.

The hardness (compressive strength), abrasivity, and variability of the rocks to be cored will have the greatest influence on cutter selection. General guidelines suggest use of smaller, more impact-resistant cutters as the formations get harder.

Low-invasion, face-discharge core bits designed for unconsolidated-to-medium strength formations can be used in harder or more abrasive rocks, but bit life may be drastically reduced.

The information presented in Table 1-4 provides an overview of the types of coring bits that are available. Specific details on coring bits and recommendations for particular applications should be obtained from service companies.

1.7.2 Natural Diamond Bits

Natural diamond core bits are used when the formation is too hard (high compressive strength) and/or abrasive for other type cutting elements. Large natural diamonds can be surface-set in a tungsten carbide matrix, or fine diamond chips can be dispersed in matrix to form what is called an impregnated diamond bit. Impregnated natural diamond bits are for ultra-hard formation applications.

1.7.3 Polycrystalline Diamond Compact—PDC

Polycrystalline diamond compact (PDC) cutters are man-made diamond materials that consist of a layer of micron sized diamond grit sintered together and bonded to tungsten carbide studs. The thickness of the polycrystalline diamond layer is only 0.020 to 0.060 inches (.51 to 1.52 millimeters). PDC bits are used to efficiently core formations ranging from

Table 1-4—General Coring Bit Guide

Rock Properties	Rock Type	Core Bit
Ultra-hard, abrasive rock	Quartzite, Igneous Rocks	Impregnated natural diamond.
Hard, abrasive rock	Sandstone, Shale, Siltstone	Natural diamonds surface set or TSP cutters.
Hard, non-abrasive rock	Limestone, Dolomite, Anhydrite	TSP cutters.
Medium to hard rock with abrasive layers	Sandstone, Limestone, Shale	TSP or surface set natural diamonds.
Soft to medium strength rock	Sandstone, Chalk, Shale	PDC cutters, low fluid invasion design.
Soft rocks, no sticky layers	Salt, Anhydrite, Shale	PDC or roller cone cutters.
Soft, sticky rock	Gumbo Clay	PDC cutters, face discharge.

very soft to medium hard. The bits are designed to cut by shearing resulting in a rapid rate of penetration. Due to the geometry of the PDC cutter, they are susceptible to impact damage, and therefore, are not recommended for very hard, highly fractured, or cherty formations.

1.7.4 Thermally Stable Diamond—TSP

Thermally stable (diamond) product, TSP, is similar to PDC in that it is also a man-made diamond material. The main difference in the TSP material is that it has a higher range of thermal stability due to the leaching of the metal catalyst used in the sintering process of manufacture. These cutters are suitable for formations generally considered too hard and/or abrasive for PDC cutters. They are not recommended for soft formations.

1.7.5 Roller Cone

The roller cone core bit uses four rotating cones set with tungsten carbide inserts or hard-faced milled-tooth cutters for coring purposes. The cutters in the cones roll and impact the hole bottom and fail the formation in compression by a chipping action. Due to the slow cutting action (chipping-compressive failure) and the number of moving parts, roller cone core bits are not commonly used.

1.8 FLUID DISCHARGE CHARACTERISTIC OF CORE BITS

1.8.1 Throat Discharge

Throat discharge core bits are designed to have 100 percent of the fluid pass between the core shoe and the inside diameter of the core bit (the “throat”). Throat discharge bits are designed to clean the inside diameter of the core bit, removing cuttings from this area to ensure a very smooth entry of the core into the core barrel. The cleaning action reduces the tendency of hard and/or brittle formations to jam.

1.8.2 Face Discharge

Face discharge core bits are designed to divert some fluid that would normally pass through the throat of the bit to the face of the bit. This cleans the face of the bit and reduces the amount of fluid scouring the core as it enters the core barrel. Face discharge bits are recommended for use in soft and friable formations.

1.8.3 Low-Invasion Profile

The low-invasion profile coring bits are designed to maximize penetration rate, and minimize drilling fluid filtrate invasion into the core. The design incorporates face discharge ports, a reduced number of cutters, and a diminished clearance between the inner core barrel and the bit face. Use of low-invasion profile core bits is recommended for soft to medium strength formations. Harder formations would slow the rate of penetration and possibly damage the cutters.

1.9 CORE CATCHERS

1.9.1 General

The single most critical part of every coring system is the core catcher that holds the core in the barrel as it is brought to the surface. Table 1-5 lists the core catchers available and suggests those most appropriate for specific rock types.

Many situations call for combining two or more catchers to ensure success. Sequences of friable sand interbedded with shale might require both slip and flapper type catchers. Full-closure catchers, run primarily to ensure success when coring unconsolidated sand, also incorporate split-ring or slip-type catchers to improve core recovery in case coring ends in hard rock.

Table 1-5—Core Catchers

Type	Recommended Usage
Split-ring, spring	Consolidated formations.
Collet	Where formation characteristics are unknown.
Slip	Consolidated formations, normally run with flapper catcher or with orientation knives.
Dog or flapper	Consolidated, fractured, and unconsolidated formations where geology is unknown.
Basket	Unconsolidated formations, normally run with another core catcher type.
Full closure	Friable to unconsolidated formations to provide positive full closure.

SECTION 2—WELLSITE CORE HANDLING PROCEDURES AND PRESERVATION

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2 Wellsite Core Handling Procedures and Preservation

2.1 GENERAL

2.1.1 The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2.1.2 Wellsite core handling procedures and preservation should follow the best possible practices because the value of all core analysis is limited by this initial operation. The objectives of a core handling program are as follows:

a. Obtain rock material that is representative of the formation.
b. Minimize physical alteration of the rock material during core handling and storage.

The major problems confronting those handling and preserving reservoir rocks for core analysis are as follows:

a. Selection of a nonreactive preservation material and a method to prevent fluid loss or the adsorption of contaminants.
b. Application of appropriate core handling and preservation methods based upon rock type, degree of consolidation, and fluid type.

Different rock types may require additional precautions in obtaining representative core data (see 2.4). All core material should be preserved at the wellsite as soon as possible after retrieval to minimize exposure to atmospheric conditions.

2.1.3 The terminology that has evolved to describe the state of core preservation is important historically, but may be confusing because it is sometimes not used consistently. For example, the term “native state” has been often used to designate core drilled with oil-based mud or lease crude for the purpose of making accurate water saturation measurements. Similarly, “fresh state” has often been used to imply that core was drilled with bland, water-based drilling fluid and preserved at the wellsite to limit evaporative losses. This term has also been used to include cores cut with oil-based mud. In the interests of consistency, the following terminology is recommended:

2.1.3.1 fresh core: Any newly recovered core material preserved as quickly as possible at the wellsite to prevent evaporative losses and exposure to oxygen. The fluid type used for coring should be noted, e.g., fresh state (oil-based drilling fluid), fresh state (water-based drilling fluid).

2.1.3.2 preserved core: Similar to fresh core, but some period of storage is implied. Preserved core is protected from alteration by any of a number of techniques (see 2.5).

2.1.3.3 cleaned core: Core from which the fluids have been removed by solvents. The cleaning process (sequence of solvents, temperature, etc.) should be specified.

2.1.3.4 restored-state core: Core that has been cleaned, then reexposed to reservoir fluids with the intention of reestablishing the reservoir wettability condition. This is often the only alternative available, but there is no guarantee that reservoir wettability is restored. The conditions of exposure to the crude oil, especially initial water saturation, temperature and time, can all affect the ultimate wettability.

2.1.3.5 pressure-retained core: material that has been kept, so far as possible, at the pressure of the reservoir in order to avoid change in the fluid saturations during the recovery process.

None of these terms alone adequately describes the state of the core; a full description of drilling mud, handling, preservation, and subsequent treatment is required.

2.1.4 For testing, the core must be sampled. In order to obtain a representative core analysis from the formation(s) of interest, it is recommended that the entire core be sampled. The entire core section should be retained. Wellsite core sampling can be important for a variety of reasons (see 2.3.1). If sampling of the core is required, it should be performed with an awareness that the sampling procedure may impact future core analysis efforts and results. Wellsite sampling must be minimal to maintain the integrity of the core. Samples for lithology description, for example, can be taken from small broken pieces of core without damaging any of the intact rock. If intact, measurable lengths of core are removed, a note or block should be left in their place describing the sample length, lithology, the reason for removal, and any other pertinent information. If samples are required from within an intact core segment, a non-percussive sampling method should be employed. The objective of a standard core-sampling procedure is to obtain samples under a uniform procedure so that the results will be independent of human bias. The selection of samples is fairly simple for uniform formations. However, where a formation contains widely varying lithology and heterogeneous porosity types (such as conglomerates, cherts, vugular or fractured reservoirs, and interlaminated shales and sands), the proper selection of representative samples requires greater care. A qualified person (engineer, geologist, etc.) should follow an established sampling procedure to minimize statistical bias.

2.1.5 The prescribed core handling and preservation procedures are applicable to all conventionally cored rock material. Many of the same practices apply to sidewall cores and drill cuttings. These recommended procedures have been selected as those which will yield core materials for the most reliable and representative core analysis. The success of any given technique is directly related to the rock properties of the core. Handling procedures should also be based on the technology used to retrieve the rock material and the objectives of the coring program. A review of core preservation materials is also presented. Each coring job and reservoir should be carefully examined prior to the design of a wellsite handling and preservation program.

2.2 CORE HANDLING PROCEDURES

2.2.1 General

Several methods are available for core acquisition. Conventional, full-diameter, continuous coring techniques can be divided into two groups; those that employ a standard reusable steel inner barrel, and those that utilize disposable inner barrels or liners. Other coring methods such as the sidewall devices and wireline-retrieved coring apparatus obtain rock material using special equipment. Special coring processes, including pressure-retained and sponge methods, are available to obtain core and fluid analysis results more representative of in situ conditions.

Consolidated core material obtained with a standard reusable inner barrel should be removed from the barrel as soon as possible after reaching the surface to minimize drilling fluid imbibition. Among the possible undesirable effects of fluid imbibition are as follows:

a. Changes in fluid saturations, geochemical and gas solution equilibrium.

b. Changes in wettability.

c. Mobilization of interstitial clays and fine-grained minerals.

d. Clay swelling and associated degradation of mechanical properties.

Any delay in removal of the core from the barrel should be reported. Various rock types and coring methods require varying levels of attention and can be broken into two major categories:

a. Basic handling—This category requires minimum training and/or experience and includes:

   1. Standard reusable steel inner barrel used to obtain core on fairly homogeneous consolidated rock.
   2. Wireline sidewall core acquisition from percussion or rotary coring.

b. Special handling—This category requires extensive training and/or equipment and includes:

   1. Disposable inner barrels and oriented core barrels used to obtain core from fractured or unconsolidated rock that may require mechanical stabilization (Skopec, et al., 1992).
   2. Pressure-retained core barrel to maintain core at reservoir pressure to minimize fluid expansion from pressure reduction and fluid expulsion as the core is brought to the surface (Sattler, et al., 1988).
   3. Aluminum core barrel with sponge liner inside a standard steel inner barrel to trap fluids during expansion from pressure reduction as the core is brought to the surface (Park, 1983).

The use of any core inner barrel liner reduces the diameter of the resultant core.

2.2.2 Removal of the Core From a Standard Reusable Steel Inner Barrel

The core should be removed from the inner core barrel in a horizontal position whenever possible. Care should be exercised to minimize the mechanical shock during extraction. The core should be allowed to slide out of the core barrel by slightly elevating the top end of the core barrel. If the core will not slide a rod may be used to push the core from the barrel. It may be necessary to gently tap the core barrel with a hammer to initiate movement of the core. However, do not hammer the core barrel in a manner that imparts severe mechanical shock to the core. In all physical manipulations, attempt to expose the core to the minimum mechanical stress. If the core cannot be removed by the foregoing method, it should be pumped out of the barrel with a fluid. If this is necessary, a suitable piston arrangement should be used that will prevent fluids from directly contacting and contaminating the core. The coring fluid should be used if pumping directly with fluids is necessary. The use of fresh water or other fluids foreign to the core should be avoided. If water is forced past the piston and contacts the core, erroneously high water saturation values may be obtained in subsequent core analysis as any excessive pressuring of the barrel may cause fluid to penetrate the core. Any difficulty or irregularity encountered while removing the core from the barrel should be noted, e.g., pressure used if pumped out with fluid, loss of core material, etc.

2.2.3 Labeling and Logging of the Core

The core should be laid out and boxed on the rig floor if space is available. Alternatively, the pipe rack can be used for this purpose. The labeling and logging of the core should not interfere with the drilling and/or coring operation. If the decision is made to handle the core on the rig floor, place appropriately marked trays, boxes, or troughs near the core barrel. If the core is to be laid out on the catwalk, prepare a cleared area and place the core between two lengths of drill pipe.

Care must be taken to maintain the orientation, and to preserve the correct sequence of the core pieces. The key point is that the core must be labeled and marked in such a way that the entire cored interval can be reassembled at some future time. The core should be protected from temperature extremes, moisture, and dehydration, i.e., direct sunlight, hot engines, rain, strong wind, and low relative humidity. Core preservation materials and equipment should be close to the core handling area to facilitate a rapid operation. Accurate measurements of recovery must be made and recorded. Any recovery in excess of the core cut should be reported, as well as nonrecovery. Assign nonrecovery and overrecovery to the bottom of each core, unless some special observation indicates that an exception should be made. All such exceptions should be noted. The following data and observations may be helpful in determining the origin of overrecovery and nonrecovery:

a. Drilling parameters—drilling time, torque, rate of penetration, pump pressure, etc.
b. General conditions of the core—continuity, broken sections, induced fractures, etc.
c. Condition of the bottomhole coring assembly.

Label the core depths from top to bottom and assign overrecovery and underrecovery to the bottom. The top of the next core should be given the drilled depth. This means that in the case of overrecovery there will be the same depth in two cores. However, these cores will be distinguishable from each other because of their core numbers. Core depths must be adjusted to log depths before correlations can be made between log properties and core properties and between cored and uncored wells. Core-to-log adjustment can be made using detailed core descriptions or core scans.

It is recommended that all core barrels be laid down on the catwalk or rig floor before removal of core. The following guidelines are appropriate for laying out and marking the core:

a. The bottom of the core comes out of the barrel first and the first piece of core should be placed at the bottom of tray, box, or trough, with each succeeding piece being placed closer to the top.

b. Caution must be exercised in maintaining the proper sequence and core orientation to ensure that individual core segments are not out of place or turned upside down. Any portion of the core that is badly broken should be put in thick plastic bags and placed in its proper position.

c. Fit the core together so that the irregular ends match, then measure total recovery.

d. Do not wash the core (see 2.4, 3.5, and 3.6). If excess drilling fluid is on the core surface, it may be wiped off with a clean drilling fluid saturated cloth and wrung out as often as needed.

e. With red and black indelible markers, taped together, stripe the core from top to bottom with parallel lines (see Figure 2-1). The red line should be on the right as the individual performing the marking is facing from the bottom of the core towards the top. Arrows pointing toward the top of the core should be used to avoid confusion.

f. With an indelible marker or paint stick, starting from the top, draw a line across the core at each foot and label each line with appropriate depth.

g. To obtain reliable core analysis, speed is essential in removing, laying out, labeling, and preserving the core to minimize any alteration due to exposure (see 2.5).

h. The core should be preserved (see 2.5) and placed in numbered containers for transportation to the laboratory. It is recommended that the entire core interval be preserved at the wellsite, with sampling being reserved for the controlled conditions at the laboratory.

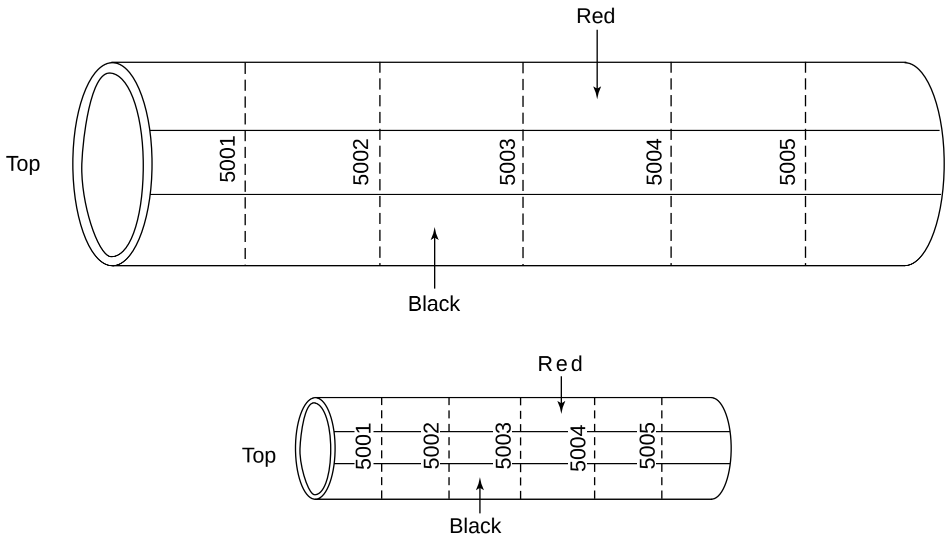

Figure 2-1—Core Marking

Even a few minutes exposure, depending on atmospheric conditions, can cause a significant loss of both water and light hydrocarbon fractions from cores. If the core is accidentally washed with water, allowed to remain in the core barrel, or let stand before preservation, then this information should be noted.

2.2.4 Handling Liners and Disposable Inner Barrels

Inner core barrel liners and disposable inner barrels improve the recovery of poorly consolidated or fractured formations. They are made of plastic, fiberglass, or aluminum and are rated to various temperatures. When coring unconsolidated or poorly consolidated formations, select the liner or disposable barrel to withstand the circulating temperature. Hard beds such as shale are best cored using either fiberglass or aluminum to prevent jamming and consequently poor core recovery. Certain coring fluid additives such as caustic react with aluminum barrels causing release of aluminum ions, that may react with the core to alter its surface properties.

When coring a poorly consolidated formation, to avoid rock compaction it is advisable to cut short lengths, 30 feet or less depending upon the rock strength. In long lengths, the lower section of core may be over compacted and damaged by the weight of the overlying material. Damaged core is of limited use for core analysis. When coring fractured formations, short core lengths may also be beneficial to decrease the risk of jamming.

The core barrel should be brought to the surface smoothly. During the last five hundred feet the core should be surfaced slowly to minimize gas expansion that can severely damage unconsolidated core if the pressure is reduced too quickly. Where gas expansion damage is expected, a perforated liner or a perforated disposable inner barrel can be used to provide a means for gas escape. All perforations must be sealed if the liner or disposable inner barrel is used as a core preservation container. Alternatively, the entire perforated section can be placed in plastic bags to prevent fluid loss.

A core-filled liner can be lowered to the catwalk, within the metal inner core barrel, using a system of pulleys anchored to the end of the catwalk. The barrel should not be banged on the rig and should be lowered gently onto the catwalk. Core-filled inner barrels flex, particularly those made of fiberglass or plastic, and should be supported by a splint. The splint should be attached to the inner barrel while it is hanging in the derrick.

a. Wedge the core barrel to prevent rotation and remove the core catcher. Transfer the core catcher material to a suitable length of liner or disposable inner barrel. The core catcher material is generally too disturbed to be used for quantitative core analysis.

b. A catwalk extension can be used to remove the entire core-filled liner from the inner barrel without flexing.

1. If the entire liner is removed or if handling a disposable inner barrel, feel inside and locate the top of the core. Cut the liner at this point. Label the core with orientation lines (red on the right and black on the left), depths, and other identification (see 2.2.3). Label depths every foot starting from the top.
2. (Preferred Method) If there is no room to remove the entire liner, pull out a little over 3 feet (0.91 meter) at a time using adequate support to prevent it from flexing. Label each 3-foot (0.91-meter) length with orientation lines and with a number to represent its position in the sequence of cut lengths. Label depths on the 3-foot (0.91-meter) lengths once the entire recovered interval is processed and the top of the core is located.

c. Cut the liners and core into 3-foot (0.91-meter) lengths using an air or electric powered circular saw. Fiberglass and aluminum liners should be cut using a mounted circular saw. Be careful to avoid vibration and rotation of the core. Clamps on the core barrel should be used with caution to avoid damaging the rock. Alternatively, the entire 30-foot (9.1-meter) length of core can be capped and shipped with a splint attached to the liner or inner barrel to prevent it from flexing. The core can then be cut in the laboratory to any specified length. This minimizes the handling of the core at the wellsite; however, preservation, sampling, and shipping procedures become more difficult.

d. Physically stabilize the 3-foot (0.91-meter) lengths using a nonreactive casting material (e.g., epoxy) to fill the annular space between the core and liner. Alternatively, the annulus can be filled with a nonreactive fluid in order to prevent evaporation. As a minimum precaution, the 3-foot (0.91-meter) lengths should be sealed with standard end caps.

e. Transfer the lengths to labeled boxes and cushion them for the trip to the laboratory. Use screws to attach wood lids to wood core boxes to avoid core damage caused by hammering.

2.2.5 Pressure-Retained Core

Pressure retaining core barrels are designed to obtain the best possible in situ fluid saturation. This method of coring offers an alternative to the conventional core barrel that loses pressure upon its retrieval to the surface. To enable the fluid saturations to be measured in the laboratory, the core must go through extensive handling. The core barrel assembly is placed in a special core service unit and the drilling fluid is flushed from the annulus between the inner and outer barrel using a nonreactive fluid while maintaining proper back pressure on the entire system. The entire core barrel assembly is then placed in a freezer box filled with dry ice (see 2.5.2.2).

To this point in the handling of a pressure-retained core, the work should be performed by trained service company personnel to their specifications.

The following procedures performed on frozen cores should be supervised by the operating company:

a. Remove the pressure-retained inner barrel from the ice, place in a safety shroud, and cut to the desired lengths.
b. Place each section back on ice, as it is cut. Take care to ensure that sections are laid down in such a way that top and bottom and position in the core section can be clearly identified. (see 2.2.3).
c. Raise one end of the core section at a time and place core cap with label on each end, securing with a hose clamp (see 2.2.4).
d. The labels should be marked with the company's name, recovered pressure, legal location, depth range of core, and processed core depth.
e. Place processed core sections in insulated shipping boxes and pack with dry ice. Mark the box with core numbers and depths along with company, location, and shipping information. If insulated shipping boxes are in transit for more than 24 hours, additional dry ice may be required.

2.2.6 Sponge Core

The sponge coring assembly is designed to improve the measurement of reservoir fluid saturations. As the core is brought to the surface, fluid that would otherwise be lost by expulsion because of pressure reduction is trapped by an absorbent polyurethane sponge surrounding the core. The coring assembly consists of 6 pre-cut 5-foot (1.52-meter) lengths of aluminum liner run within a standard steel inner barrel. The sponge core is, for the most part, then handled according to procedures established in 2.2.4. In most cases, the liner must be pumped from the coring assembly. The precut liner is stored and preserved in PVC shipping tubes filled with a suitable nonreactive fluid. The PVC shipping tube is sealed with one rigid and one rubber gas expansion end cap. For orientation purposes, each sponge liner section is beveled on one end.

Once the sponge core arrives at the laboratory, it is milled open and both the core and sponge are extracted of all reservoir fluids (see 4.3.4).

2.2.7 Wireline Sidewall Coring

Wireline sidewall cores are extracted from the formation by a variety of means. Percussion sidewall coring involves the use of an explosive charge that propels a hollow projectile into the formation. Because of the forces produced by the entry of the percussion core bullet into the formation, compaction, fracturing, and rearrangement of rock grains occur; great care must be exercised when handling this core material.

Alternatively, wireline sidewall cores may be mechanically drilled from the formation with a rotary bit. Damage is minimized with this technique; however, this method is not feasible in all types of rock. If the sample breaks during removal from the coring tool, it should be pieced together and any damage should be noted. Other sidewall sampling devices involving the use of pressure-actuated coring receptacle samplers are also available.

When using sidewall coring techniques, fragile samples should be placed in plastic or glass jars with metal lids. Paper or other materials capable of absorbing liquids should not be placed in jars to act as sample cushioning material. Heat-sealable plastic laminates are an acceptable preservation technique for the drilled sidewall samples. All samples should be stabilized and cushioned during transport to the laboratory (see 2.5.2.1) and be accurately labeled (see 2.2.3).

2.2.8 Wireline-Retrieved Continuous Coring

In wireline-retrieved continuous (WRC) coring operations, the core barrel is recovered while the drill string remains in the borehole. Trip time is reduced and consequently the method can be less expensive than conventional coring. Typically, long vertical intervals are cored continuously, and certain wells may be cored from surface to total depth.

2.2.8.1 Depth Marking of WRC Cores

Good communication between the wellsite core retrieval personnel and the driller is necessary for accurate depth marking of WRC core. In conventional coring, the driller provides the top and bottom depths of the cored interval. This may cause some confusion in assigning core depths, because conventional cores are referenced from the top of the cored interval. Because WRC coring is continuous, it is best to use the bottom of the previous coring run as the top of the subsequent run. Monitoring for possible fillup between runs is suggested to minimize errors from this cause. Maintaining sound core accounting procedures in the form of a realtime spreadsheet should minimize the potential for errors in determining the top of the next interval. The spreadsheet information should include core run number, top of cored interval, bottom of cored interval, percent recovery, and a column noting the point from which core marking started.

All WRC core should be marked and labeled in accordance with 2.2.3, except assignment of depth should be modified in accordance with the following procedures. The conventional procedures for assigning core depths are not always appropriate for WRC core. In WRC coring, core dropped in one coring run may be retrieved in the next coring run, so the true depth of the current core may be in the previously cored interval. The retrieval of "dropped core" also means that the volume of overrecovered core is larger than is commonly encountered in conventional coring. Given the larger volumes of core involved, it is more important to use a core marking procedure that avoids assigning the same depth to more than one piece of core. If overrecovery occurs, the core depth marking can start with the bottom of the core, with the depth of that piece of the core being assigned the bottom reported by the driller. Core marking should then move from bottom to top. If 100 percent or greater recovery was also obtained in the previous coring run, depth assignments should follow conventional procedures, with the depth marking beginning at the top.

For underrecovery, the following equation should be used to account for missing core:

$$\begin{aligned} \text{Missing core} &= \text{Depth Drilled} - \text{Core Length} \\ &= [CDD - PDD] - [CBL + PC - VOID] \qquad (1) \end{aligned}$$

Where:

MC = missing core length, feet (meters).
CDD = current driller's depth, feet (meters).
PDD = previous driller's depth, feet (meters).
CBL = core barrel length, feet (meters).
PC = protruding core, feet (meters).
$VOID$ = void in core barrel, feet (meters).

In underrecovery, the core depths should be labeled from top to bottom, assigning underrecovery to the bottom of the cored interval.

2.2.8.2 Alternate Method for Depth Marking of WRC Cores

An alternate to the method discussed in 2.2.8.1 is to assign depths in the same manner as conventional cores (see 2.2.3). As for conventional cores, there may be underrecovery or overrecovery. Starting at the top, mark the core with depth marks every foot through to the end of the core. No attempt should be made to resolve underrecovery or overrecovery intervals before core-to-log adjustment. If depths are consistently marked from the top of each core run and each core run has a unique number, there may be the same "depth" in two consecutive cores, but they will be distinguished by their core run number. If an attempt is made to adjust for overrecovery at the wellsite, this can result in tremendous confusion with cores relabeled multiple times.

2.2.8.3 Washing of WRC Cores

As for conventional coring, care should be exercised to avoid damaging the rock; e.g., by washing it with inappropriate fluid(s). If there is any ambiguity about damaging effects, err on the side of caution, for example, by avoiding washing the core.

2.2.8.4 Analysis of WRC Cores

The extent to which the WRC core is analyzed varies from operator to operator. The core analysis procedures for WRC cores differ significantly from those used for conventional cores. In conventional coring operations, the core is returned to a laboratory for analysis and, at some future time, the core analysis results are adjusted to downhole log depths. With WRC core, much of the analysis is performed in the field, and in some cases, the core may never be archived. Typically, a wellsite geologist should describe the section with sufficient lithological detail to enable core-to-log adjustment and correlation. The geologist should note the hydrocarbon shows, porous intervals, and facies changes. Some operators use mobile laboratories through which they perform fairly sophisticated analyses of the WRC core, including core gamma scanning, ultraviolet photography, gas chromatography, and measurements of porosity, density, magnetic susceptibility, mineralogy, and acoustic properties.

2.2.9 Oriented Core Barrel

Orientation of the core barrel is accomplished using electronic, multi-shot instruments and specialized core scribing equipment. Alternatively, paleomagnetic rock signature can be used for core orientation purposes. Strict handling procedures must be followed to ensure that orientation data are positively depth correlated and matched to the proper piece of core material. This is particularly critical in fractured rock units where disposable inner barrels and liners are commonly used.

2.3 FIELD SAMPLING AND ANALYSIS

2.3.1 General

In general, sampling of recovered core material at the wellsite is not recommended. If it is necessary to sample immediately, precautions must be taken to minimize the exposure time of the core. Sampling should be quick, efficient, and performed in accordance with sound safety practices. Always obtain samples using the least damaging or contaminating method available. The entire core should be saved in all cases.

Specific reasons for wellsite sampling include, but are not limited to, a) chip sampling for lithologic description and/or mineralogical determination, b) measurement of basic rock properties, c) fluid compatibility-completion testing, d) wettability studies, e) observation of oil fluorescence/cut, f) anelastic strain recovery measurements, and g) methane desorption studies for the analysis of coal. All sections removed from the cored interval should be recorded on the field data sheet (see 2.3.3) and physically represented in continuous core sections using rigid spacers. The removed sample should be preserved, labeled, and packaged in a manner consistent with the test desired. Any additional pertinent data should accompany the samples to the laboratory or be available for wellsite analysis. Other special wellsite core analysis techniques are feasible if adequate mobile facilities are available to run tests under controlled conditions.

Hammering can damage the core and may preclude core analysis. If possible, chip samples should be taken at naturally occurring breaks in the core or with a precision trim saw. The sample size should be kept to the minimum necessary to perform the desired analysis. Place samples in individual bags and preserve the fluid saturation with a suitable preservation technique (see 2.5).

If a detailed core examination is to be made at the wellsite, sampling and core handling should be done quickly, and only if the examination is critical to the success of the well objective.

2.3.2 Transportation and Logistics

The transport method should be expedient and provide protection against damage from environmental changes, mechanical vibration, and mishandling. Other important factors to consider when choosing the mode of transport include, a) distance and remoteness from the wellsite to the laboratory, b) onshore or offshore conditions and terrain, c) core material competence, d) weather conditions, e) type of preservation or packaging, and f) cost.

In all cases, precautions must be taken to securely stabilize the core material. In air transport, the storage cabin may not be pressurized, and this can be a factor in the core preservation. Do not stack core in such a manner as to damage the core material. Commercial carriers unaccustomed to transporting fragile materials should be used with caution. For safety reasons, cores packed in dry ice may have to be treated as a "chemical" for transportation purposes.

In all methods of transportation, a transmittal letter or contents documentation form with pertinent shipping information should accompany the shipment. A separate copy of this letter should be sent to the recipient via surface mail or fax. All applicable U. S. Department of Transportation regulations should be followed in the shipment of core materials. When standard core boxes are used they can be palletized, banded, and shipped as is. Cores, particularly those of unconsolidated materials, may be frozen or refrigerated at the wellsite for preservation and stabilization during transportation and storage. If freezing is used, the core must be entirely frozen before shipment to avoid mechanical damage. Frozen core is usually crated in insulated containers and packed with dry ice. Refrigerated core is usually shipped in self-contained refrigerator units. A temperature monitoring and recording device should accompany the core to ensure the desired conditions are maintained during transportation (see 2.5.2.2).

2.3.3 Data Sheet

A suitable data sheet should be provided for and completed by the wellsite engineer or geologist, to supply as complete a record as possible of the conditions of coring. This information will be valuable in qualifying the interpretation of the core analysis data. Further, this record may suggest either that certain additional tests be run to supplement the basic tests, or that other tests would not yield significant data. This will result in the most useful analysis for the least time and cost. Figures 2-2 and 2-3 are example forms, and the use of these or similar forms is recommended.

It is important to have as much pertinent data as possible accompany the core material. The following is a list of desirable information:

a. Well identification, API well number, elevation, vendor names and contacts, as well as telephone/telefax numbers and addresses.
b. Drilling fluid type, contents, and measured data.
c. Core type and equipment used.
d. The formation(s) cored, with the top and bottom driller's depth.
e. Designation of critical coring information and any pertinent coring notes, i.e., total coring/trip time, difficulties, and recovery.
f. Formation water salinity and production fluid data.
g. Preservation guidelines. Exposure time.
h. Analysis requested.
i. Coring log and drilling records.
j. A core description.
k. Well logs and mud logs (if available).

2.4 ROCK TYPES AND SPECIAL CONSIDERATIONS IN HANDLING

2.4.1 General

The term "rock type" is used to describe the major distinguishing feature(s) of core material. This can refer to the degree of consolidation, presence of fractures or vugs, composition (shale), or physical properties (e.g., low permeability) of the rock. Geological descriptions of rock are more complex and classification schemes have been devised to categorize specific rock types with respect to texture, type of cementation, grain size, etc. Many special considerations must be taken into account when designing a wellsite core handling program. Paragraphs 2.4.2 through 2.4.13 include general guidelines for various rock types.

2.4.2 Consolidated Rock

Consolidated rocks are hardened as a result of cementation. They need no special treatment at the wellsite. Cementation in rocks is defined as the process of precipitation of cementing materials around the solid grain surfaces. Rocks can be described as consolidated, poorly consolidated (friable), or unconsolidated, depending on the degree of compaction and cementation. Common consolidated rocks include limestone, dolomite, sandstone, and chert.

2.4.3 Unconsolidated Rock

Unconsolidated rocks have little or no cement and are essentially compacted sediments. Poorly consolidated rocks have minor cement but not enough to make them hard. These rocks are best cored using an inner core barrel liner or a disposable inner barrel (see 2.2.4). Care must be taken to prevent

Prepared By: ____________________ *Date:* M M D D Y Y Core Number ____________________

Operator ____________________

Well Identification ____________________

API Number ____________________

County/State/Country ____________________

Persons Requesting Work ____________________

Report Results To ____________________

Alternate ____________________

Rig Contract ____________________

Coring Company ____________________

Contact/Phone ____________________

Field/Reservoir ____________________

☐ Vertical Well ☐ DeviatedWell ☐ Sidetrack

Legal Location/Block ____________________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Core Analysis Company ____________________

Contact/Phone ____________________

Address ____________________

____

Elevations: _______ Ground/Mean Sea Level ______ Kelly Bushing ____

Address ____________________

Address ____________________

Address ____________________

Address ____________________

Coring/Drilling Fluid
Type/Contents ____________ Weight _______ ppg Funnel Viscosity _______ sec Water Loss ________ cm^3/30 min Chlorides ________ ppm pH ________ Tracer ____________

Type of Core
☐ Conventional ____________________
☐ Sponge
☐ Pressure Retained
Inner Barrel Type ____________________
☐ Oriented Barrel
☐ Other ____________________
Sidewall: ☐ *Percussion or* ☐ *Mechanically Drilled* *Number Attempted* _____ *Number Recovered* _____

Analysis Planned
☐ Plug Size or ☐ Full Diameter
Method
☐ Fluid Saturation ____________________
☐ Porosity ____________________
☐ Grain Density ____________________
☐ Permeability ____________________
☐ Surface Gamma Log ____________________
☐ Special Instructions ____________________

Transportation
☐ By Service Company ____________________
☐ Ground ____________________
☐ Air ____________________
☐ Other ____________________
Carrier ____________________
Date shipped ____________________

Preservation
Method ☐ Plastic Laminate/Type ______________
☐ Freeze ☐ Dry Ice or ☐ Liquid Nitrogen
☐ Refrigerate _____ °F _____°C
☐ Core Inner Barrel
☐ Core Wrap and/or ☐ Dip-Type ☐ Resination
☐ Other

Exposure Time/Climate ____________________
Notes ____________________
____
____
____

Laboratory/Long Term Preservation ____________________
Special Instructions ____________________
Post Core Analysis Instructions/Core Material Distribution ____________________

Formation(s)
____
Expected Core Point ______________
____
Expected Core Point ______________
____
Expected Core Point ______________

Depths (Drillers)
Interval Cut ____________________
Interval Recovered ____________________
Interval Cut ____________________
Interval Recovered ____________________
Interval Cut ____________________
Interval Recovered ____________________

Well Inclination ____________________
Total Core Recovered ____________________
Length Cored ______________ Units ________
Coring Time ____________________

Core Diameter ______________ Units __________
Bit Type ____________________
Percent Recovery ____________________
Trip Time ____________________

Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _______ @ _______ ☐ °F ☐ °C

Estimated Production ☐ Oil
☐ Condensate
☐ Dry Gas

Notes ____________________
____
____
____
____

Attach Coring log And Core Description

Figure 2-2—Core Analysis Wellsite Data

General Information
Analysis Requested Date M M D D Y Y
API # ____________
File # ____________

Requested By: ____________

☐ Full Diameter or ☐ Plug : Size ________ Units ________

Method	*Special Instructions*
☐ Saturation	____________
☐ Porosity	____________
☐ Permeability	____________
☐ Other	____________

Objective of the tests as agreed by parties involved ____________

Transportation and Inventory

Carrier ________ Date Shipped ________ Core Arrival Date ________

Core condition on arrival:

☐ Preserved ☐ Frozen ☐ Unpreserved ☐ Cleaned ☐ Other ________

Correlation Depths: Driller's ______ Log ______ TVD ______ Units ______

Allocation of the missing cored interval: ☐ Bottom ☐ Middle ☐ Top ________

Screening

☐ CT ☐ X-ray ☐ NMR ☐ Fluroscopy ☐ Other ________

Sample Handling

Core Gramma (yes/no, type) ____________

Lab Preservation (prior to analyses) ____________

Sampling Method

Sample Sleeve ☐ Yes ☐ No, Type ____________

Nominal Sample Diameter/Length____________ Units ______

Plugging/Trimming Fluid____________

Treatment prior to testing (flushing, evaluation, resaluration, etc.) ____________

Core Disposition (removed samples, storage location, preservation, etc.)____________

Lab Analyst ____________

Other Service

☐ Slab ☐ Resination ☐ Photo: ☐ BW ☐ Color ☐ U.V. ☐ Other ________

Supplementary Tests ____________

Methods and Conditions

Cleaning:

Methods	Conditions
☐ No Cleaning	Solvents ____________
☐ Dean-Stark	Temperature ____________Units ______
☐ Soxhlet	Pressure ____________Units ______
☐ CO2/Solvent	Time ____________
☐ Flow Through	Volume and Rate____________ Units ______
☐ Others	Others ____________

Drying:

Methods	Conditions
☐ Convection oven	Temperature ____________ Units ______
☐ Vacuum oven	Time ____________
☐ Humidity oven	Relative Humidity % ____________
☐ Others	

Porosity:

Methods

Pore Volume	Grain Volume	Bulk Volume
☐ Boyle's Law	☐ Boyle's Law	☐ Caliper
☐ Saturation	☐ Archimedes	☐ Archimedes
☐ Summation-Of-Fluids	☐ BV-GV	☐ Mercury Displacement
☐ BV-GV	☐ Other	☐ GV+PV
☐ Other		☐ Other

Conditions/Fluids

Confining Stress (Magnitude and Type)____________Units ______

Gas ____________ Liquid____________

Pressure ____________

Permeability:

Methods	Conditions
☐ Steady State	Fluid Type ____________
☐ Unsteady State	Confining Stress ____________Units ______
☐ Probe	Sleeve Durometer ____________
☐ Empirical	Pore Pressure ____________Units ______
☐ Not Measured	Klinkenberg: ☐ Measured ☐ Empirical ☐ No Correction
☐ Others	Inertial Factor: ☐ Measured ☐ Empirical ☐ No Correction

Saturation:

Methods	Conditions
☐ Distillation Extraction (DS)	Temperature ________Units ______
☐ High Temperature Retort	Fluids ________
☐ Others	Water Density______ Units ______
	Oil Density ______ Units ______

Correction for Salt: ☐ Corrected for Water Volume
☐ Corrected for Oil Weight ☐ Not Corrected

Quality Assurance: (Page number in report where the information is provided) ____________

Comments/Remarks: ____________

Data Anomalies: ____________

Figure 2-3—Basic Core Analysis Laboratory Data the core from disintegrating. This includes making sure the core is brought to the surface, laid down smoothly, and preserved in such a way that it will survive transportation (see 2.5.2).

2.4.4 Unconsolidated Rock—Light Oil and Gas

It is critical to preserve unconsolidated cores containing light oil in an efficient and expedient manner. Any unnecessary movement of the core should be avoided. The two methods commonly used to preserve this rock type involve environmental methods, such as freezing or chilling and mechanical stabilization with epoxy, foam resin, etc. Unconsolidated core containing light oil is susceptible to significant fluid loss during handling at the surface. As with most rock types, by the time the core has been brought to the surface, it has undergone mechanical stress relief due to the removal of overburden pressure and has had varying amounts of gas expansion when internal pore pressure is lost. The degree to which these effects will alter the core depends on depth, reservoir pressure, oil gravity, fluid properties, sediment type, and coring procedures. Care must be taken to ensure that pressure does not build-up within the core barrel during handling. The inner core barrel liner or disposable inner barrel can be predrilled with holes [$^1/_8$ inch (3.18 millimeter) diameter] to avoid pressure buildup. Cored intervals should be limited in length to prevent possible rock damage under its own weight. As the core is raised through the upper 500 feet (152 meters) of the well, the core barrel should be retrieved at a slow rate to minimize chances of disaggregating the rock and causing damage to the core. If freezing is used to stabilize unconsolidated materials, the core should not be transported before it is fully frozen, as partial freezing can cause structural damage to the core (see 2.5.2.2).

Filling the annulus between the disposable inner barrel or liner and core with coring fluid is recommended for stabilization when epoxy or a permanent setting material is not used; however, this procedure can alter rock fluid saturation and wetting characteristics. When epoxy, resin, or foam injection are used, the drilling fluid must be fully displaced or drained from the annulus. The casting material must completely conform to and encase the core surface.

2.4.5 Unconsolidated Rock—Heavy Oil

The greatest difficulty in handling unconsolidated rocks that contain viscous heavy oil is prevention or minimization of delayed core expansion. The expansion is the result of slow gas evolution from the heavy oil, with no possibility of short-term drainage because of the low mobility to gas. Swelling of the rock may easily continue until the gas phase becomes continuous, and this may require volumetric expansion in excess of 6 percent to 8 percent. In liners, unconsolidated heavy oil sandstones will expand radially to fill empty annular space. Once the swollen core is tight in the liner, further gas evolution may cause a piston action, causing core to extrude from the ends of the liner sections leading to overrecoveries as high as 5 percent. It is tempting to simply cut off the extruded core and discard it, but this must be avoided. Extruded material is kept at the liner end from which it came, the plastic end caps may aid in keeping it slightly retained.

Improving core quality in unconsolidated heavy oil sandstones requires the following considerations:

a. Provide mechanical restraint to expansion.
b. Provide a means to allow gas drainage.
c. Provide mechanical strength to the core.

Item a involves use of liners that have an internal diameter only slightly larger [$^1/_8$ inch (3.18 millimeters)] than the core bit, to reduce radial expansion. During handling and preservation, flexing of liners, heating of core, and prolonged exposure of liner ends must be avoided. Axial restraint will help reduce the tendency to extrude the core. This restraint may be provided in a number of ways, including:

a. Rigid liner caps rather than rubber end caps, with the end caps stapled to the liner at a number of points, and secured with screw clamps.
b. Cutting liner segments to precise lengths and placing in a strong core box so that the ends of the box provide axial restraint. Alternatively, cores can be wedged into the core boxes with pieces of wood planks cut to lengths.
c. Special handling techniques devised by various operators may also be used, including special storage cylinders or axial retention methods.

Gas evolves slowly from heavy oil, and will continue for months. It is recommended that pre-perforated liners be used in all areas to shorten the gas flow path and eliminate piston effects. The pre-drilled holes [$^1/_8$ inch (3.18 millimeter) diameter] should be spaced no further apart than the radius of the liner. Post-drilling of liners is not recommended as an acceptable alternative because it prolongs core handling and may lead to damage. If required, liner and core segments may be placed in cylinders and repressured with an inert gas (N_2) to slow or stop gas evolution, and to avoid oxidation.

Freezing of unconsolidated heavy-oil core may be necessary, although in general, freezing is not well understood. Freezing has the following effects: a) it reduces gas evolution speed and volume, b) it enhances oil viscosity that restricts expansion, and c) it freezes interstitial water that gives the core some mechanical strength to restrict expansion and fracturing. Because the pore water is usually saline, the temperature will have to be reduced below –40°F (–40°C) to ensure full mechanical benefits (see 2.5.2.2).

Core handling during transportation and storage for unconsolidated materials containing heavy oil must maintain the mechanical restraint and low temperature. When preparing core sections for analysis, the temperature should be allowed to rise slowly, so that evolved gas can dissipate; mechanical restraint should be fully maintained until the core is equilibrated, a process that may take weeks because of the high oil viscosity and low relative permeability.

2.4.6 Vuggy Carbonates

Large vugs can weaken the core material and cause difficulties with recovery. In many cases, core recovery is reduced in friable vuggy intervals. Standard consolidated core preservation methods should be used on this rock type (see 2.5).

2.4.7 Evaporites

Salt rocks are generally quite competent, and, except for their solubility, may be considered as consolidated rocks. Core containing salts in continuous sequences or as vug and fracture fillings should not be washed with fresh water under any circumstances. Because the physical properties of salt rocks can be altered by small changes in moisture content, cores containing salts must be immediately wiped to a surface dry state and preserved. Transportation and storage of cores containing salts must always be undertaken keeping the soluble nature of the material in mind. Cores of evaporite, anhydrite, gypsum, or calcite present no special core handling problems.

2.4.8 Fractured Rock

Many reservoir rocks are naturally fractured. Disposable inner barrels or liners made of aluminum or fiberglass are recommended for coring fractured rock (see 1.6 and 2.2.4). An oriented core can be useful in determining fracture strike and in situ stress direction (see 1.6 and 2.2.4).

2.4.9 Rocks Rich in Clay Minerals

Clay minerals may be present in small quantities in rocks, yet have a major impact on rock properties. Some of the major concerns in rocks containing clay minerals include:

a. The presence of smectite (a swelling clay mineral), even in very small quantities (1 percent), is of importance in core handling because of swelling potential, high cation exchange capacity, and osmotic suction potential.

b. Interstitial clay minerals can be physically mobilized by changes in fluid content, chemistry, or mechanical disturbance, leading to pore throat blocking or changes in surface wetness characteristics of pores or other physical changes.

c. Clay minerals in contact with their natural pore fluids are in thermodynamic equilibrium, and exposure to other fluids will alter this leading to changes in clay mineral activity, exchangeable cations, and consequent changes in mechanical and flow behavior.

d. Smectitic shales and sandstones may swell when confining stress is removed if free water is available, even if the free water has properties identical to the interstitial fluids.

Any excess fluid or mud cake should be immediately wiped from cores of smectitic materials and clay mineral rich materials, followed by immediate preservation (see 2.5.2).

2.4.10 Shale

In addition to the recommendations for clay mineral containing rocks (see 2.4.9), there are special issues relating to handling highly fissile shale. These materials have fissility planes of low strength that may split spontaneously, even if core is handled with great care. Once a fissile shale core has split, it may be impossible to obtain specimens large enough for core analysis.

It is recommended that fissile shale cores be handled in the following manner:

a. Avoid excessive handling or movement of the core.

b. Remove any excess water.

c. Preserve immediately to stop desiccation.

d. Masking tape or fiberglass packaging tape may be wrapped around core segments perpendicular to the fissility planes to reduce further splitting. Alternatively, heat-shrinkable plastics can be used.

Shales have low permeability and slow internal transfer of moisture will inevitably take place between beds of different mineralogy and fabric during long-term storage. If the core is totally unconstrained, this may result in delayed splitting even without desiccation or handling. Fissile shales are exceptionally sensitive to temperature changes, and should be maintained at a constant temperature during transportation and storage. Freezing of shales must not be allowed, since this leads to massive microfissuring and internal moisture movement.

Oil shales with organic chemical volumes in excess of 20 percent are sensitive to temperature and oxidation, and must be preserved with particular speed if detailed analysis is required. The much stronger matrix-supported oil shales are typically more quartzose and with organic chemical volumes less than 20 percent and in general do not require special handling.

2.4.11 Low Permeability Rock

Evaporation of fluids, a problem with all core materials, is a particular difficulty in low permeability and low porosity core where the percentage change in saturation may be much greater for the same volume of fluid evaporated. The time period before core is protected from evaporation is critical for these samples. The presence of clay minerals may make damage by evaporation irreversible in some samples (see 2.4.9).

2.4.12 Coal

In situ gas content, gas sorption behavior, permeability, relative permeability, cleat and fracture analysis, core composition, and mechanical behavior are the major interests in coal analysis for coalbed methane production. Gas desorption studies may be performed at the wellsite with desorption canisters. Procedures for handling of coal core should include instructions for these special studies. Wireline retrieved core barrels, core barrels with disposable inner barrels or liners, and pressure-retained core barrels have been used to cut coal cores.

Gas content and gas desorption rate are commonly measured from coal by canister desorption methods using conventional cores, mechanically-drilled sidewall cores, wireline-retrieved continuous cores, or drill cuttings. Coal segments are sealed in a canister and isothermally maintained while the volume of gas evolved from the sample is measured. Gas content measurement by canister desorption requires an estimate of gas volume lost while bringing the core to the surface and before sealing the samples in the canister. Gas content must be normalized to a mineral free matter basis. Since the gas evolved will not be 100 percent methane, the composition of the gas must be analyzed.

Pressure-retained core technology, that does not require an estimate of lost gas, offers a more accurate means to determine the total in situ gas content of a coalbed. The volume of gas evolved from the coal core in the pressure barrel is measured as a function of time, temperature, and pressure. If pressure-retained methods for coring coal are used, a special internal temperature sensor is incorporated, and no coring fluid flushing is carried out (see 2.2.5). In one procedure, the entire pressure-retained barrel is returned to bottomhole temperature and allowed to equilibrate for up to several days before the pressure is allowed to dissipate and the device is disassembled. During the temperature and pressure dissipation phase, changes should be gradual to minimize both the magnitude of the pressure gradient and the temperature gradient. The former may cause the coal to fracture internally, whereas rapid cooling may cause tensile cracks to open in the outer part of the coal core. These may affect both permeability and mechanical behavior. Alternatively, the pressure-retained coal core can be cut into one foot sections and placed in desorption canisters for measurement of the gas content and gas desorption rate.

Coal core must be handled with care at the wellsite because of its heterogeneous nature, and because the methane found in small pores within the core is under pressure. Careless treatment, liner flexing, or a sharp blow to the core barrel may cause the core to fragment, rendering the core of little use for core analysis. The internal gas pressure may aid this tendency to deteriorate, and time is required to permit gas dissipation. Coring fluids in contact with coal intended for fluid flow or mechanical tests should not contain materials capable of altering the coal structure.

Fresh coal oxidizes when exposed to air, potentially changing its surface properties and sorption characteristics. Exposure time to air must be minimized, and special handling may include canisters to receive coal core segments, followed by flushing of the cylinders with inert gas or methane.

White or yellow pens or paint crayons should be used to mark the surface of the core lengths. As with any core, preservation of moisture and minimization of exposure time is advised. Freezing of coal core is not recommended, nor is it considered necessary.

2.4.13 Diatomite

Diatomites are generally high-porosity, low-permeability rocks composed of opaline-quartz phases with varying amounts of detrital material. Diatomites are cored with disposable inner barrels or liners (see 2.2.4).

Diatomite can be preserved by environmental means, wrapping, etc. Freezing of diatomite is not recommended. Temperature should be controlled to maintain a constant temperature [35 to 40°F (1.67 to 4.44°C)] during wellsite and transportation operations.

2.5 PRESERVATION OF CORES FOR ANALYSIS

2.5.1 General

The preservation of a core is an attempt to maintain it, prior to analysis, in the same condition as existed upon its removal from the core barrel. In the process of cutting a core, recovering it, and bringing it to the surface, the fluid content of the rock is altered by unavoidable changes in pressure, temperature, etc. Pressure-retained core methods attempt to minimize these effects (see 2.2.5). Careless or incorrect practices in handling and preservation cause further alteration of the core and its fluids, thereby making the core even less representative of the formation.

Preservation and packaging of cores may vary depending upon the test(s) required, the length of time before testing, and the potential of performing wellsite tests. If the core samples are to be used to determine fluid saturation or for special core analysis, it is necessary that they be preserved for transportation to the laboratory. Evaporation and migration of fluids as well as oxidation within the sample must be avoided to obtain reliable core analysis. An additional objective of the preservation program is to prevent breakage of the cores during shipment and storage. Consolidated core may be durable enough not to require special handling procedures. However, special care should be taken with unconsolidated or fractured rock, etc. (see 2.4).

The use of unprotected glass jars, easily deformable plastics (if not properly stabilized), paper cartons, non-rigid containers, and air-tight cans are not recommended for core preservation purposes.

2.5.2 Methods of Preserving Cores

There is no one best preservation method. Experience can help determine the most satisfactory method for the rock type in question. The choice of method will depend on the composition, degree of consolidation, and distinguishing features of the rock. Therefore, general use of one specific method of preservation will not apply to all rock types. The techniques required to preserve cores for testing may depend upon the length of time for transportation, storage, and the nature of the test to be performed. Some variation in the method of preservation may depend upon whether the cores will be analyzed locally or whether they must be prepared for long-distance shipping. Preferred methods to preserve cores for laboratory analysis include one or more of the following:

a. Mechanical stabilization.
b. Environmentally controlled preservation using chilling, regulated humidity, or freezing, if necessary (see 2.5.2.2).
c. Heat-sealable plastic laminates.
d. Plastic bags.
e. Dips and coatings.
f. Sealing in disposable inner barrels, liners, and tubes.
g. Anaerobic jars.

2.5.2.1 Mechanical Stabilization

All rock types should be mechanically stabilized prior to shipment to the laboratory. This is particularly true for unconsolidated rock (see 2.4.4 and 2.4.5). Core that has been cut using plastic, fiberglass, or aluminum liners/disposable inner barrels can be cast using resin, wax, or foam to fill the annular space between the core and the sleeve. Resin has low viscosity and will fill fine fractures. However, it is only poured into the annulus and is not under enough pressure to displace pore fluids in the rock and therefore does not impregnate the core.

Mechanical stabilization for well consolidated cores may also be as simple as wrapping the core in bubble wrap or other suitable cushioning materials. All core material should be considered fragile and handled carefully (see 2.3.2). Care should be taken to avoid disturbing poorly consolidated or fractured core prior to mechanical stabilization.

2.5.2.2 Environmental Preservation

Controlling environmental conditions to which the core is subjected by chilling or maintaining a humid environment can help to preserve the core (refer to core preparation information in Section 3). Core chilling is used primarily to minimize fluid evaporation and provide mechanical stabilization. This technique is useful in preventing the core from drying; however, its effectiveness is subject to the coring fluid type and the reservoir rock and fluid properties. When chilling core, it is still necessary to mechanically stabilize the rock for transport to the laboratory (see 2.5.2.1).

Cores that are preserved by freezing should be frozen by application of dry ice, liquid nitrogen, or placement in an electrically-operated freezer unit. Freezing may result in the migration or diffusion of fluids within the core structure or breakage of the core. Freezing can cause significant evaporative losses through sublimation. Unconsolidated cores that have been frozen can be packed with a $^1/_4$ inch (6.35 mm) thick layer of frozen brine (surface ice cake) to reduce the sublimation process. This measure is critical if freezing is used for long-term core storage. Structural damage to the core may occur if it dehydrates while frozen.

The practice of freezing core is most common for unconsolidated rocks (see 2.4.4 and 2.4.5). The full effect of freezing on a core's petrophysical properties is unknown. The freezing of consolidated core with interstitial water is not well understood. Expansion of ice crystals may cause irreversible structural damage to core. Freezing may affect the properties of rock flushed with fresh water more than those flushed with saline drilling fluid filtrate. These effects will decrease with decreasing water saturation. If it is necessary to allow the core to warm to ambient temperature before testing, the condensation of moisture from the atmosphere onto the core surface must be prevented. Thawing of the core may cause some redistribution of the fluids within the core matrix.

The fluid saturation and reservoir (mineral) properties can also be preserved by controlling the relative humidity of the core environment with specially designed ovens. This technique has wide applicability and is most effective with rocks containing moisture sensitive clay minerals and/or chemically bound water contained in minerals (see 2.4.9).

2.5.2.3 Heat-Sealable Plastic Laminates

Several heat-sealable plastic laminates are available. Aluminum foil or mylar may be used to add rigidity to the laminate. The laminated core preservation package should act as an impermeable barrier to water vapor and gases, and be resistant to chemical alteration and degradation by fluids. Laminates are easy to use and the preservation process can be performed quickly. Care should be taken to prevent tears or punctures in the laminate. A clean, flat surface is required for smoothing the laminate prior to sealing. All core should be prewrapped and taped with durable plastic or other material to cover the core ends and sharp edges. The packaged core segment should be labeled with well and depth information.

The heat sealing process is critical to the success of using this preservation method. The heat sealer must be set to the proper temperature in accordance with manufacturer's specifications to obtain an effective seal. Any discontinuity in the seal will negate the barrier properties of the material. Some laminates are available in tubular form that requires the sealing of only two ends rather than four. The head space in the preservation package should be minimized; however, enough material should be used to prevent weakening if the package will be opened and resealed more than once. In some cases, it may be advisable to evacuate the gas space where the loss of light-end hydrocarbons is not an issue. An inert gas such as nitrogen can also be blanketed over the rock to minimize oxidation. When core material degasses, the laminate package will inflate; this will pose no problem if the package is sealed properly. If necessary, the evolved gas may be sampled directly with a standard gas syringe and the package resealed. The preservation package must be labeled and mechanically stabilized for shipping (see 2.5.2.1). At no time should the preservation package be subjected to extremes of temperature.

2.5.2.4 Plastic Bags

Plastic bags are only recommended for short-term preservation. Core samples should have a minimum of air space between the core and bag wall. Any excess bag can be folded against the core wall and taped to assure a tight fit. As always, clear labeling and proper stabilization procedures should be followed.

2.5.2.5 Dips and Coatings

Dips and coatings are used when cores are not to be tested within a few hours or days and when the material is to be transported over long distances. Dip coatings can also be used with plastic laminates to add mechanical integrity.

CAUTION: Cores should never be dipped directly into any molten wax or plastic material.

All core should be prewrapped with a heat-sealable laminate or plastic film and aluminum foil prior to dipping. All core segments should be labeled with well and depth information. The purpose of the plastic film wrap is to prevent contact of the core and pore fluids with the aluminum foil outerwrap. Such contact can cause oxidation of the foil and loss of its moisture and oxygen barrier properties. The following procedures should be used with the wrap and dip method:

a. Prepare a heating vat for dip-coating several hours prior to preserving the core. Observe all safety precautions. Follow the dip manufacturer's recommendations for handling. Overheating the dip can cause the coating to be ineffective.

b. Wrap the core tightly in plastic film that will conform to the surface of the rock, crimping the free ends together. Several layers of high quality plastic film are desirable to prevent puncturing.

c. Wrap the core with several layers of aluminum foil, crimping the free ends together. Avoid puncturing the aluminum wrap.

d. Tie a wire around the core to make a handle.

e. Dip the foil-wrapped core sample in the molten coating material. A liberal amount of dip coating should encase the core; a $^1/_8$- to $^1/_4$-inch (3.18- to 6.35-millimeter) thick coating is recommended. This is accomplished through the use of multiple dips, allowing each dip coating to harden prior to the application of additional dip material. It is recommended that the dip coating be hardened by suspending the core in air by means of the wire handle.

f. The wire handle should be cut flush with the dip coating. Additional dip should be applied to the wire end to eliminate a pathway for evaporation or oxidation.

Coating material must have certain properties, as follows:

a. It must be dimensionally stable over long periods of time.

b. It must not react with oil or water and not contain acids, oils, solvents or any other liquid that may be exuded when set.

c. Permeability to gases, oils, and water must be low when set.

d. It should have a low melting point, preferably below 200°F (93.3°C) maximum and have a fairly low viscosity when melted. Higher melting points are acceptable if exposure time to the dip is minimized.

e. When removed from heat and exposed to ambient conditions, it should be dry and set tack-free within 5 to 15 seconds.

f. When set, it should be tough but pliable, slightly elastic but with good tensile strength, and not melt at temperatures below 180°F (82.2°C).

As with all core preservation methods currently used, the long term effectiveness of dips and coatings remains uncertain.

2.5.2.6 Disposable Inner Barrels, Liners, and Rigid Tubes

A convenient means of core preservation is possible when disposable inner barrels or liners made of plastic, aluminum, or fiberglass are used (see 2.2.4). The core may be preserved as-is by sealing the ends of the cut inner barrel or liner. This is not recommended as a long term preservation method, but will allow the core to be processed quickly and without special equipment. Holes in the inner barrel should be sealed prior to shipping. The core can be rapidly shipped to the laboratory for sampling and testing, and, if required, additional preservation of the sealed containers is possible (see 2.5.2.2). When a conventional core barrel is used, steel, aluminum, or plastic tubes with suitable couplings, caps, or O-ring seals may be used to preserve the core. To be used effectively, the tube and end pieces must be nonreactive with the core and its fluids. Segments may be stabilized according to guidelines established in 2.5.2.1 to minimize damage in shipment.

2.5.2.7 Anaerobic Jar

Immersion of the core in liquid within an anaerobic jar can be used to prevent oxidation, evaporation, and drying during the handling of core. The anaerobic vessel is an elongated jar with a sealable lid, into which a liquid can be introduced and any free oxygen removed. The immersion liquid must be compatible with the core and pore fluids, and be able to maintain the current wettability of the sample (see 2.6). Typically, the following fluids are used for immersion:

a. Deoxygenated formation brine or synthetic formation brine with biocide.
b. Crude oil.
c. Depolarized refined mineral oil.

As always, follow all safety precautions when using anaerobic jars for preserving reservoir samples.

2.6 RECOMMENDATIONS FOR CORE HANDLING TO PRESERVE WETTABILITY

2.6.1 General

Wettability alteration can take place during coring, core treatment at the wellsite, or during the period of storage before measurements are made in the laboratory. Only core treatment at the wellsite is discussed here, although the measures recommended can only be successful in preserving wettability if adequate precautions are taken at each step of the core recovery and analysis process.

The validity of many laboratory core tests depends on maintenance or reestablishment of reservoir wetting conditions; however, there is rarely an initial reference point to define in situ wettability. The practices recommended here are intended to provide measures of wettability as early as possible after removal of core from the formation so that a reference point for wettability is established. Cores should not be exposed to air for any longer than is necessary.

Wettability tests should be made at the wellsite. A simple observation of imbibition of water and oil droplets placed on the core surface should be recorded routinely. In many cases, this will be the extent of wellsite wettability testing, but much more comprehensive tests can be initiated at the wellsite as well. Some special facilities would be required, including:

a. The capability to cut core plugs from newly recovered whole core. Any delay in cutting core plugs allows capillarity and diffusion to distribute surface active drilling fluid constituents to the uninvaded portions of the core. Synthetic reservoir brine or refined laboratory oil should be used as the cutting fluid.
b. Core holders and core flooding equipment to allow flushing of plugs are needed. It may be possible to avoid damage done by an unstable oil phase (one from which asphaltenes or paraffins are deposited because of changes in pressure and temperature) if the oil phase is quickly replaced by a stable, non-damaging oil.
c. Imbibition test facilities are also needed since tests of imbibition rate and extent are the best indicators of wettability in porous media.

Requirements for maintaining wettability will vary from one reservoir to another and to some extent will have to be experimentally determined. Suitable core preservation methods to control fluid loss are described in 2.5.2.

2.7 PRECAUTIONS

2.7.1 General

The fundamental objective of core analysis is to obtain data representative of in situ reservoir rock properties. Coring, handling, and preservation should be conducted in such a manner as to prevent both loss of the interstitial fluids and contamination with foreign fluids. The core should never be washed with water or oils prior to preservation. Suitable coring and handling procedures must be adopted to obtain laboratory data that are meaningful. Alteration of the rock can occur during coring, handling, preservation, sampling, and preparation prior to or during analysis. For a reliable determination of the fluid content of cores, a uniform procedure must be designed for handling and preservation. It should be emphasized that a properly designed program will benefit not only the near term user, but also future users of the core material. Some precautions in the handling and packaging of samples are listed:

a. All cores should be preserved as soon as possible after removal from the core barrel. Even after preservation, the samples should not be exposed to extreme conditions. The long term effectiveness of core preservation materials currently in use is unknown. A tested and trusted material should be used if the core is to be stored for extended periods of time.
b. Always minimize the gas head space in core preservation containers to prevent evaporation losses during core storage. This procedure will also minimize condensation losses on the inside surface of the container and help prevent breakage of the more loosely consolidated samples during shipment.
c. To minimize fluid loss, do not contact the core with cloth, paper, or any other dry material with fine capillaries.
d. Do not dip or coat the core directly with any fluid.
e. Follow stringent handling instructions for the processing and preservation of unconsolidated core (see 2.2.4 and 2.5).
f. Do not preserve an unconsolidated rock or other rock type in the same container as a rock of a vastly different lithology. This will minimize the potential of mechanical damage to weak samples.
g. Should any core be exposed to harsh handling conditions or washing, this should be noted. All pertinent data must be supplied with the core (see Figures 2-2 and 2-3).
h. Label each preservation container properly. In cases where confidentiality is an issue, this data should be coded with numbers and referenced to a master list.
i. It is recommended to have a company representative familiar with wellsite handling and preservation present during the coring operation. If this is not possible, explicit written instructions should be given to the service company

representative responsible for this activity. Upon arrival at the laboratory, cores should remain in a preserved state until ready for analysis.

j. ALL APPLICABLE SAFETY REGULATIONS MUST BE FOLLOWED WHEN HANDLING CORING EQUIPMENT AND CORE MATERIAL. The wellsite team should be protected, as necessary, against exposure to hazardous material using overalls, gloves, protective eyewear, etc. A hard hat, steel-toed shoes, and hearing protection are also recommended. Where toxic gases such as hydrogen sulfide are present, appropriate personal protective breathing apparatus must be available. Safety training is required for all wellsite workers prior to handling coring equipment, processing equipment, machinery, and core material.

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SECTION 3—CORE SCREENING AND CORE PREPARATION

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3 Core Screening and Core Preparation

3.1 GENERAL

The recommendations included in this document may involve the use of hazardous materials, operations, and equipment. This document does not address all of the relevant safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials and equipment.

3.2 CORE DESCRIPTION

3.2.1 Principle

The purpose of core examination and description is the recognition of lithological, depositional, structural, and diagenetic features of whole or slabbed core. Qualitative and quantitative core descriptions provide the basis for routine core analysis sampling, facies analysis, and further reservoir studies such as reservoir quality and supplementary core analyses. A description provides a permanent, accessible record of the core.

3.2.2 Apparatus and Supplies

The following equipment is recommended for use in standard descriptions of the core:

a. Logging form for systematic data collection.
b. Microscope or hand lens.
c. Length measurement scale.
d. Grain size scale.
e. Appropriate chemicals such as:
    1. Water or brine to improve visibility of geological structures.
    2. Dilute HCl acid to identify carbonate minerals.
    3. Alizarin red to differentiate calcite and dolomite.
    4. Hydrocarbon solvents to facilitate the detection of oil fluorescence under ultraviolet light.
f. Coring log, drilling report, mud logs, well site information on lost core.
g. Core gamma log.
h. Ultraviolet light.

3.2.3 Precautions

The following precautions should be observed:

a. If the samples being described have been preserved for special testing, exposure of the samples to air and chemicals should be avoided until tests on the preserved core have been completed.
b. Choose an appropriate logging format. The objective of a logging form should be to accurately represent the core. When describing core, it is important to collect and record data in a systematic manner.

3.2.4 Procedures

The following procedures should be used:

a. Lay out the core to be analyzed on a viewing table.
b. Check the amount of core with the coring report and ensure that no core material has been lost during transportation. Note any damage or alteration to the core during well-site handling and transportation.
c. Check the numbering and order of boxes or containers against cumulative depth.
d. Check core continuity and orientation with respect to the top of the core. If the core has been marked in accordance with 2.2.3, the top of the core is up when the red scribe mark is on the right.
e. Check the order of core segments in the boxes; look for matching “breaks” or scribe marks from one piece to the next and from one box to the next.
f. Measure and mark footage on each box. Mark core to the nearest $^1/_2$ inch (1 centimeter).
g. If downhole gamma-ray logs are available, a comparison should be made with the core gamma-ray logs (see 3.3) to verify that the cored interval depths are consistent with well log depths.
h. Assess the entire cored sequence before commencing. Seek out distinguishing features such as units, contacts, unique markers (e.g., bentonite, coal).
i. Record major features such as the following using standardized nomenclature and abbreviations (see Section 8):
    1. Core lithology (shale, sandstone, limestone, etc.).
    2. Color.
    3. Bedding (thickness, bed contacts, erosional markers).
    4. Obvious sedimentary structures.
    5. Texture (grain size, angularity/roundness and sorting).
    6. Composition (grains, cement, fossils).
    7. Porosity types.
    8. Diagenetic and tectonic features.
    9. Note any oil stain and related fluorescence.
j. Minimize centimeter-by-centimeter descriptions. Some fine scale features may be important and should be recorded.
k. Record nonreservoir rocks, e.g., mudrocks, in addition to potential pay sequences. Subtle changes in lithology may assist correlation between core and well logs and definition of relevant zonation.

l. Record fracture information such as width, extent, density, fracture orientation if core is oriented, and presence of cementation or mud. Where possible, differentiate between natural and coring induced fractures.

3.3 CORE GAMMA LOGS AND CORE SPECTRAL GAMMA LOGS

3.3.1 Principle

Naturally occurring gamma-ray emitters (radiogenic daughters of uranium and thorium along with potassium-40) give a measurable gamma-ray response that can be recorded with depth. If such a record measured at the surface is compared with the gamma-ray readings taken from a downhole gamma-ray log, the results can frequently be used to adjust the depth of the core to coincide with the open-hole logging depths and to identify zones where core has been lost.

3.3.2 Apparatus

The recommended apparatus consists of a conveyer for moving core, lead shielding to reduce background gamma radiation, and suitable gamma-ray detectors. A typical detector system consists of a shielded scintillation crystal coupled with a photomultiplier. The scintillation crystal is normally sodium iodide doped with thallium [NaI(Tl)]. Other scintillation crystals include cesium iodide (CsI) and bismuth germanate (BiGeO).

Signals from the detectors are processed and the gamma-ray events are energy-sorted and counted. Standard gamma-ray units should provide total gamma counts in APIU units. With the spectral gamma units these counts are converted into potassium, uranium, and thorium concentrations, and standard gamma-ray log API units. Signals are displayed on a computer graphics screen or plotted in downhole log formats and scale for direct comparison with downhole logs.

3.3.3 Procedures

The following procedures should be used:

a. Set the recorder readout to correspond to the scale used with the downhole gamma-ray log.
b. Log the core gamma-ray response from bottom to top. This conforms with the sequence used in downhole logs.
c. Core gamma-ray logging should be carried out smoothly, without interruption, in the correct order and spacing with matching core breaks fitted together.
d. Record a repeat section near the bottom of the core to establish the reproducibility of the measurement.

3.3.4 Advantages

Core gamma-ray logging is widely available and is used in general practice to correlate core depth with log depth. The spectral gamma-ray apparatus will differentiate uranium, thorium, and potassium concentrations and can be used to better identify and differentiate shale, particularly in sandstone cores with large amounts of potassium feldspar and mica.

3.3.5 Limitations

This technique is unable to detect low gamma-ray activity and can suffer from significant background interference. Analysis of samples for spectral gamma-ray response requires a slower rate of logging than that used to determine total gamma-ray response.

3.3.6 Calibration

The apparatus should be calibrated before evaluating any core material. Calibrations are sensitive to the size of core and the gamma-ray energy range. Calibration should be done by measuring the gamma-ray response to calibration tubes containing pure samples of the elements of interest. The total gamma-ray apparatus requires a calibration tube containing potassium (K-40), uranium (U-238), and thorium (T-232) of known activities. The calibration procedure for the spectral gamma unit requires the same elements but in individual tubes. A blank calibration tube should be measured to ensure a minimal amount of background gamma-rays are interfering with the apparatus. The speed of the conveyor should be slow enough, to provide an acceptable ratio of signal to background noise. A ratio of three times the square root of the count rate to background count rate greater than one is acceptable, i.e.:

$$\frac{3 + \sqrt{\text{count rate}}}{\text{background count rate}} > 1$$

3.3.7 Accuracy

The precision of the measurement varies with the square root of the count rate.

3.4 CORE IMAGING

A recorded image of the core is essential. This record will provide information that can be used if viewing of the core is not possible. The record may include visual images of the surface features of the core using photographic techniques, visual representations of the internal structures of the core such as x-radiographs, computed x-ray tomographs, magnetic resonance images, or acoustic images. Any record should include information on core depth along with a scale or legend indicating intensity range of the recorded image.

In addition to photographic representations, modern imaging techniques provide quantifiable information for the core that can be used as the basis for routine core analysis sampling, facies analysis, reservoir quality studies, supplementary core analyses, and downhole log interpretations.

3.4.1 Photography

3.4.1.1 Principle

Core is normally photographed under both natural (5,500 K) and ultraviolet light (254-365 nm) along with a standard color scale. The natural light photographs show lithology and sedimentary structures and allow specific features recorded in the core description to be examined. Ultraviolet light photography can highlight hydrocarbon-bearing zones by causing most oils to fluoresce in shades ranging from orange-brown for heavy oils to bright yellow for high gravity oils; condensates can appear as a very light white to blue-white color. Non-hydrocarbon bearing zones will appear as purple regions, although some minerals such as chalky-limestones also appear purple.

3.4.1.2 Advantages

Core photography provides a visual record of the core that can be used to reconstruct damaged core pieces, minimize core handling, and identify the location of samples if photographed following sampling.

3.4.1.3 Limitations

Photographic colors may not represent true colors of the core. Highlighting of features may require wetting the core surface for photography. A scale and color bar are required if adjustments are necessary.

3.4.2 X-Ray Techniques

3.4.2.1 Principle

X-ray techniques can be used to noninvasively examine the internal character of a core. An x-ray beam is directed through the core and variations in attenuation of the incident x-ray beam are measured. In order of increasing definition, these techniques are: fluoroscopy, x-radiography, and computed tomography (CT scanning).

The utility of these methods relies on their sensitivity to density contrasts within the core. Consequently, non-porous, dense areas will be contrasted against porous, low-density areas. High definition of density variations within the core requires multi-directional measurements or spatial separation of the contrasted areas.

3.4.2.2 Fluoroscopy

In this technique, the core is moved across an x-ray source. The beam, attenuated by the core, impinges on a fluorescent screen, is intensified, and is recorded by a video camera. The captured image can be viewed on a monitor, recorded on a video cassette, and converted to digital format for later processing and viewing. The output from this technique is a continuous image along the length of the core.

Because the core is moving, the resolution of the resulting image may be diminished. It does, however, enable identification of density variations, some geological structures, and areas of lost core within plastic or fiberglass sleeves. It is also useful in identifying fractured and badly broken zones.

3.4.2.3 X-Radiography

A core sample is positioned between an x-ray source and an x-ray sensitive film. Attenuation of the x-ray beam by the core is captured on film. The developed film can be viewed on a light table, or used to make a black and white print.

It is possible to obtain multiple images of the same piece of core and display them side-by-side. The core should be first x-rayed to obtain an initial image and is then rotated 90 degrees in order to obtain a second image. This is useful in orienting sleeved core prior to slabbing because it can establish the bedding direction.

During x-radiography, the core sample is stationary and provides a higher resolution image than the fluoroscopy method. X-radiography enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density.

3.4.2.4 Computed Tomography (CT)

In CT, the core is scanned by a highly collimated x-ray beam. Detectors on the opposite side of the core measure the intensity of the transmitted beam. The x-ray source and/or detectors rotate or translate around the sample. A series of x-ray attenuation measurements are made and numerically reconstructed to give the spatial distribution of x-ray attenuation coefficients within the sample.

The resolution of the image depends on the thickness of the x-ray beam, the number of detectors in the scanner, and the size of the pixel array used to reconstruct the image. X-ray beam thickness varies from about 2 millimeters to 10 millimeters. CT enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density. Combination of sequential x-y scans along the core axis can provide slab images or three-dimensional images of the core.

3.4.2.5 Advantages

X-ray techniques provide quantified, objective representations of the core. These representations can provide similar advantages as described for core photographs in 3.4.1.2 without the need for exposing a surface of the core.

3.4.2.6 Limitations

Resolution of the images is less than that provided by photographs. X-ray attenuation may vary with mineralogy, depending on the energy of the x-ray beam. Some applications are still in the development stage. Further development of the relationships between rocks and x-ray attenuation may extend these methods to other areas of core analysis.

3.4.3 Nuclear Magnetic Resonance (NMR)

3.4.3.1 Principle

NMR imaging is used to provide a reconstructed image of fluids within a core sample. NMR measurements are based on the observation that when the proper radio frequency excitation energy is applied to an ensemble of nuclei, a portion of nuclei at a lower energy level can be promoted to a higher energy level. When the excitation is removed, the rate at which excited nuclei return to the lower level can be detected and measured. The excitation energy is supplied by an oscillating magnetic field in resonance with the nuclei. The nuclei response is measured through a tuned receiver coil. The location of the excited nuclei can be determined by the application of magnetic field gradients during the excitation and detection processes.

The primary nucleus used in core analysis applications is ^{1}H. Other nuclei that can be imaged include, but are not limited to, ^{2}H, ^{31}P, ^{23}Na, ^{13}C.

3.4.3.2 Advantages

Magnetic resonance images are noninvasive and provide an image showing fluid locations within a sample. The advantages described in 3.4.2.5 apply provided the sample is contained within a suitable non-metallic holder.

3.4.3.3 Limitations

This technique is not descriptive of the core but of the liquids within the core. The technique requires a high density of resonant nuclei for an adequate signal. Hence, low porosity cores will give weak signals. Parmagnetic or ferromagnetic minerals may inhibit or degrade the measured signal and can distort the image.

3.5 CORE SAMPLING AND CORE PREPARATION (BASIC CORE ANALYSIS)

3.5.1 Principle

The sampling procedure for basic core analysis is determined by the type of information required. Sampling will generally take into account one or more of the following:

a. Lithological distribution.

b. Porosity and permeability variations within the lithological units.

c. Distribution of hydrocarbons.

3.5.1.1 Plug Samples

Referred to as plugs, should be removed from sections of whole core oriented either vertically or horizontally with respect to the whole core axis or with respect to the normal of the bedding planes. These plugs will provide data on matrix properties. See 3.5.2.4 for procedures for removal of core plugs.

3.5.1.2 Full Diameter Samples

Full diameter samples (sections of whole core), in addition to plug samples, should be taken in the following types of zones or where there are significant large scale heterogeneities that are different from the matrix properties. These can occur in, but are not limited to:

a. Vuggy carbonates—Vugs can bias the measurement of porosity within a sample. Samples should be taken to obtain a representation of the relative proportion of vugs-to-rock in the reservoir.
b. Fractured reservoirs—Fractured zones may be difficult to sample due to the fragile nature of the rock. However, natural fractures can have a significant positive impact on reservoir permeability and should be sampled where possible or appropriate.
c. Conglomerates—Within a conglomerate, if the pebble size is large with respect to the sample size, sample size can greatly bias rock property measurements. To obtain a representative sample of a unit of conglomerate it is necessary that sample size be sufficient to include all pebble sizes.

3.5.2 Sample Cutting, Trimming, and Mounting

3.5.2.1 Principle

The core should be cut and trimmed to provide regularly shaped samples, most commonly right cylinders. Unconsolidated, disintegrating, and very friable samples may have to be mounted prior to testing. Regardless of the degree of lithification, sample handling should be kept to a minimum.

3.5.2.2 Apparatus and Supplies

The following equipment and supplies are commonly used in core sample cutting and trimming operations:

a. Large slab saw with a diamond blade.
b. Trim saw with a diamond blade.
c. Drill press with diamond-edged core drills, capable of drilling cylindrical samples.
d. Dressing wheel or saw for squaring plug ends.
e. Fluid pumps for delivering various coolants (brine, oil, air, water, liquid N_2) to the cutting surfaces.
f. Indelible marking medium such as India ink.
g. Sleeves of lead, aluminum, or heat shrinkable plastic for mounting soft, unconsolidated, or very fragile samples.

The following information should be available prior to cutting, trimming, or mounting operations:

a. The total number of samples required.

b. Size and orientation needed.

c. Exact depth locations for samples and how they are to be labeled.

d. Fluids to use for cutting samples (cores containing clays or shale laminations sensitive to fresh water will deteriorate unless proper drilling fluids are used).

e. Available core images.

f. If required, materials for the preferred method of preservation for both plugs and core from which the plugs are cut.

3.5.2.3 Precautions

The following precautions should be enforced during sample cutting, trimming, and mounting operations:

a. Saw and drill press operators should wear protective earplugs, goggles, gloves, and other necessary safety equipment.

b. Lubricant/coolant flow should be sufficient to cool the drill bit or saw blade and remove the cuttings without eroding the sample.

3.5.2.4 Procedures

The following procedures for preparing various type core samples should be followed:

a. Plug samples:

   1. Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform.

   2. Trim plugs to the required length making sure that the ends are parallel. Keep and label trimmed ends.

   3. Label, preserve, and/or store samples, as required.

b. Full diameter samples:

   1. Cut sections of the core selected for analysis slightly longer than required to allow for finish grinding operations.

   2. Remove barbs and smooth chipped edges by dressing the ends. Cylinder ends should be as close to parallel as possible.

   3. Clearly mark the samples and preserve or store as required.

c. Unconsolidated samples—There are two distinct methods of preparing unconsolidated cores. The procedures depend on whether the core is stabilized by freezing, epoxy impregnation, or both or is unstabilized. Stabilized cores are generally drilled while unstabilized cores are plunge cut.

   1. Stabilized cores—Frozen cores are obtained using the practices listed for plug samples, with appropriate modifications to avoid thawing of the core:

      (a) Pre-cool the core with dry ice and drill with liquid N_2 coolant.

      (b) Drill plugs at specified points using the appropriate size bit. Care should be taken to drill straight plugs. If too much pressure is applied during the drilling operation, the bit will flex causing the plug to deform. Ensure that there is adequate liquid nitrogen flow so that the sample is kept frozen and the cuttings are being removed.

      (c) Trim plugs to the required length using a liquid N_2 cooled saw blade, making sure that the ends are parallel. Keep and label trimmed ends.

      (d) Mount plugs in a suitable pre-weighed sleeve with pre-weighed and properly sized end screens.

      (e) Label, preserve, and/or store samples as required.

   Epoxy-stabilized cores can be frozen and drilled using the procedures described in a-e above or by the procedures described for plug samples (see 3.5.2.4.a).

   2. Unstabilized cores—Unfrozen cores can be cut using a plunge cut technique. The cutter should be slowly pushed in the core and removed. The cut plug should be gently removed from the cutter and inserted directly into a pre-weighed sleeve. The plug ends are then trimmed.

3.6 CORE CLEANING

3.6.1 Introduction

Prior to most laboratory measurements of porosity and permeability, the original fluids must be completely removed from the core sample. This is generally accomplished through flushing, flowing, or contacting with various solvents to extract hydrocarbons, water, and brine.

3.6.2 Apparatus and Supplies

Various techniques and apparatus for cleaning are described in the procedures below and in Section 4.

Some solvents used for hydrocarbon extraction purposes are listed in Table 3-1. Listed solvents are those most frequently used for extracting samples for routine analysis. Some are preferred for specific applications; e.g., chloroform has been found to be excellent for many North American crudes and toluene has been found useful for asphaltic crudes. Prior to cleaning samples with unknown oil properties, a subsample should be tested with various solvents for cleaning efficiency.

Since residual salt crystals affect the measured porosity and permeability, core samples containing a formation water with high salinity may require additional extraction to remove salt. Salt can be removed with methyl alcohol or other solvents in which salt is soluble.

The various solvents used for extracting core samples can be reclaimed by well-known physical and chemical methods. Such recovery techniques can make practical the use of an expensive solvent.

Table 3-1—Selected Solvents and Their Use

Solvent	Boiling Point, °C	Solubility
Acetone	56.5	oil, water, salt
Chloroform/methanol azeotrope (65/35)	53.5	oil, water, salt
Cyclohexane	81.4	oil
Ethylene Chloride	83.5	oil, limited water
Hexane	49.7–68.7	oil
Methanol	64.7	water, salt
Methylene chloride	40.1	oil, limited water
Naphtha	160.0	oil
Tetrachloroethylene	121.0	oil
Tetrahydrofuran	65.0	oil, water, salt
Toluene	110.6	oil
Trichloroethylene	87.0	oil, limited water
Xylene	138.0 –144.4	oil

3.6.3 Precautions

The following precautions should be enforced during core sample cleaning operations:

a. When using solvents, it is the responsibility of the user to establish appropriate safety and health practices prior to use and to comply with all applicable regulatory requirements regarding use and disposal of materials.

b. The solvent selected should not attack, alter, or destroy the structure of the sample.

c. Chloroform may hydrolyze during extraction, forming hydrochloric acid as a product.

d. Not all solvents are compatible with all extraction equipment configurations. Possible solvent/equipment reactions should be considered in selecting equipment and solvent for use in core cleaning operations.

e. Closed-type electrical heaters should be used whenever flammable solvents are used. Safety precautions such as adequate ventilation of the laboratory and ready accessibility of fire extinguisher equipment and safety showers should be observed.

f. Extraction should be conducted under ventilating hoods equipped with forced-draft ventilation.

g. Temperature effects on the samples should be considered. See 3.7 for recommendations regarding drying of core samples.

3.6.4 Procedures

3.6.4.1 Solvent Flushing by Direct Pressure

Extraction of hydrocarbons and salt from reservoir rocks can be achieved by injecting one or more solvents into the core sample under pressure and at room temperature. The pressure used should be dependent on the sample permeability and may range from 10 to 1,000 psi. The core samples may be held in a rubber sleeve under overburden pressure or in a suitable core-holding device that will permit the flow of solvent through the matrix of the sample. The volume of solvent required to completely remove hydrocarbons in the core sample is dependent on the hydrocarbons present in the sample and the solvent used. The core is considered clean when the effluent is clean. In some instances, more than one solvent may be required to remove heavy, asphaltic-type crude oils.

3.6.4.2 Flushing by Centrifuging

A centrifuge with a specially designed head is used to spray warm, clean solvent (from a still) against the core samples. The centrifugal force causes solvent to flow through samples displacing and extracting the oil (and water). The speed of rotation should be varied from a few hundred to several thousand revolutions per minute (rpm), depending on the permeability and degree of consolidation of the core. Most common solvents can be used. The apparatus and procedure are described in Conley and Burrows.[1]

3.6.4.3 Gas-Driven Solvent Extraction (U.S. Patent 2,617,719)

In this procedure, a core is subjected to repeated cycles of internal-dissolved or solution-gas drive until the core is cleaned of hydrocarbons. The remaining solvent and water are removed by means of a drying oven.

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[1] Conley, F. R., and Burrows, D. B., “A Centrifuge Core Cleaner,” *Journal of Petroleum Technology*, VIII, 61, October 1956.

When a core from an oil-bearing formation is brought to the surface and depressurized, the gas dissolved in the oil comes out of solution and displaces some of the oil and water out of the core. This results in some gas-filled pore space at atmospheric pressure. The gas-filled space in the core can be almost completely filled with solvent by surrounding the core with a suitable solvent containing a dissolved gas and applying sufficient hydraulic pressure. Under this condition, the solvent mixes with oil in the core and subsequent depressuring to atmospheric pressure removes some of the residual oil.

Carbon dioxide gas is excellent for this purpose because of low fire or explosion hazards and high solubility in most solvents. Some of the solvents that can be used are naphtha, toluene, or mixtures of solvents. With certain types of crude oil, cleaning time may be reduced if the core chamber is heated by a water bath, steam bath, or by electric heaters. One successful application of this method for routine cleaning of cores uses carbon dioxide and toluene at 200 psig, with a hydraulic pressure of 1,000 psig. Cycles of approximately 30 minutes are used. The apparatus and procedure are described by Stewart.[2]

3.6.4.4 Distillation Extraction Method

A Soxhlet extractor and suitable solvent or solvents should be used to dissolve and extract oil and brine. Extraction can be arranged in a manifold so that the oil-and water-laden solvent siphons from each extractor into a common still from which fresh solvent is continuously distilled, condensed, and again distributed to all the extractors.

The cleanliness of the sample is best determined from the color of the solvent that siphons periodically from the extractor. Extraction should be continued until the extract remains clear. Non-luminescence of extract under fluorescent light is a good criterion for determining complete extraction of the oil for a given solvent. It should be noted that the complete extraction of certain oils from core samples may require more than one solvent, and the fact that one solvent is clear after contact with the sample does not necessarily mean that oil has been completely removed from the sample. Details of one apparatus and procedure may be found in 4.3.

3.6.4.5 Liquefied Gas Extraction

Liquefied gas extraction uses a pressurized Soxhlet extractor and a condensed, low-boiling-point, polar solvent. The process is a distillation extraction procedure that uses pressurized solvent to clean the core. Solvent is regenerated through low temperature distillation. Because the extraction is conducted at or below room temperature, it can be performed on heat-sensitive core such as those containing gypsum.

3.6.5 Advantages

Cleaning a core removes original fluids from the core, preparing it for further tests that do not require those fluids.

3.6.6 Limitations

Individual test conditions may require a particular technique for optimal results. Some techniques may be more applicable to particular rock types or cleaning requirements.

3.7 DRYING

Conventional core samples can be dried by the methods listed in Table 3-2:

Table 3-2—Core Sample Drying Methods

Rock Type	Method	Temperature, °C
Sandstone (low clay content)	Conventional oven Vacuum oven	116 90
Sandstone (high clay content)	Humidity oven, 40% relative humidity	63
Carbonate	Conventional oven Vacuum oven	116 90
Gypsum-bearing	Humidity oven, 40% relative humidity	60
Shale or other high clay rock	Humidity oven, 40% relative humidity Conventional vacuum	60

Each core sample should be dried until the weight becomes constant. Drying times may vary substantially, but are generally in excess of four hours.

3.7.1 Precautions

Some precautions that should be observed in drying samples for routine core measurements are:

a. Samples containing clays must not be dehydrated during preparation. Care must be exercised in drying these samples. In some cases, temperatures lower than those indicated in Table 3-2 should be used to prevent the dehydration of clays.
b. Samples containing gypsum will require specific procedures during preparation. Extreme care should be exercised to avoid both loss of water and change of the crystalline structure.
c. Samples must be protected from erosion by the drip of clean solvent when utilizing the distillation extraction technique.
d. The extraction technique should not physically damage the core.
e. The usual criterion for sample cleanliness is a clean extract. However, many solvents are not complete solvents for all types of oils and a clean extract may reflect oil solubility and not complete extraction.

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[2] Stewart, Charles R., U. S. Patent Number 2,617,719, “Cleaning Porous Media,” November 11, 1952.

f. Samples containing heavy asphaltic oils may require the cycling of more than one solvent.

g. More effective core cleaning can often be achieved by a combination of solvents.

h. Allow solvent laden samples to vent in a fume hood before placing in a closed drying oven.

3.8 SAMPLE PRESERVATION

Preservation of samples in the laboratory will depend on the length of time between tests and the type of tests to be done. Any storage or preservation technique should ensure that structural integrity is maintained and that unwanted drying, evaporation, and oxidation are avoided.

Laboratory samples can be preserved using the techniques described in 2.5. Freezing of samples in the laboratory should follow the precautions outlined in 2.4.4, 2.4.5, and 2.5.2.2.

SECTION 4—FLUID SATURATION METHODS

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Recommended Practices for Core Analysis

4 Fluid Saturation Methods

4.1 INTRODUCTION

This section documents the specialized techniques developed for analysis of core samples with different sample physical characteristics and sizes, obtained by various coring methods. Table 4-1 lists the principal techniques for determining core fluid saturations.

Paragraphs 4.2 through 4.5 describe the seven principal saturation analysis methods listed in Table 4-1 for full-diameter cores and plugs/sidewall cores. Plugs are defined as samples drilled or shaped from cores recovered with any of several full-diameter coring devices. Sidewall cores are defined as cores that are obtained after the wellbore has been created by the drill bit or some type of full-hole coring device.

Paragraphs 4.6 through 4.8 address fluid saturation analysis methods for coal, oil shale, and gypsum-bearing samples. Oil shale and coal samples may be obtained with full-diameter coring devices. However, oil shale and coal may also be quarried from surface or shallow formations, and require non-routine sampling procedures different from those typically used for reservoir core. The reporting format of fluid saturations for these latter materials differs from typical reservoir rocks in that the saturations may be expressed as weight percent or gallons per ton instead of percent of pore volume.

Scanning methods that are capable of measuring fluid saturations by non-destructive techniques are included in 4.5. Additionally, a historical section (see 4.9) is included in which some previously acceptable methods for fluid saturation determination are described, but are not recommended.

A difficult, if not impossible, formation evaluation task is recovering a core having the same saturation and distribution of fluids as occurred in the formation prior to coring. Changes in fluid content and distribution frequently take place during the coring, retrieval, preservation, and transportation phases. Laboratory handling, sampling, and testing processes can bring about further alterations. These issues are not addressed in detail in this section, but the reader should be aware of the many complications that influence the final fluid saturation values furnished in a laboratory core analysis report. Fluid saturation analysis may alter the wetting of the core and thus affect further analyses on the core material.

4.2 RETORT METHOD AT ATMOSPHERIC PRESSURE

4.2.1 Basic Procedure

4.2.1.1 Principles of Analysis

Table 4-1—Rock Type and Recommended Fluid Saturation Testing Method(s)

Rock Type	Recommended Testing Methods
Consolidated clastics, carbonates	a,b,c,d,e,f
Unconsolidated (light oil)	c,d,e
Unconsolidated (heavy oil)	c, c(*),e
Vuggy carbonates	b,d,e,f
Fractured	a,b,d
Clay bearing	a,c(*),e
Evaporites	g,e
Low permeability	a,b,c,d,e,f
Coal	h
Shales	a,b,c
Oil shale	a(*)
Diatomite	c,e

Legend:
a = retort at atmospheric pressure.
b = distillation extraction (full diameter).
c = distillation extraction (plug).
d = pressure-retained core method.
e = solvent flushing/Karl Fischer.
f = sponge core barrel method.
g = gypsum-bearing method.
h = coal method.
(*) = modified procedure.

Oil and water fluid saturations are obtained by a high-temperature retorting process in which the oil and water contained in a fresh sample of crushed core material are vaporized, condensed, and collected in calibrated glassware. The gas saturation is determined on an adjacent, lithologically similar sample by placing it in a mercury pump and measuring the amount of mercury injected with the water and/or oil in place.

4.2.1.2 Apparatus

The following items describe suitable apparatus used in the basic retort method:

a. Stainless steel retort(s)—See Figure 4-1. Such retorts are designed to hold 100 to 175 grams of crushed core material. Each retort is equipped with a threaded cap, that in turn is equipped with a gasket to prevent leakage of condensable gases. A long, stainless steel condensing tube is connected to the opposite end of the retort. A coarse mesh screen is placed in the bottom of the retort to prevent crushed core material from entering the condensing tube.

b. Oven—See Figure 4-1. Such ovens are capable of holding multiple retorts. Strip-type electrical heating elements are preferred. A temperature controller, thermocouple, and temperature readout device are also required.

c. Water bath—See Figure 4-1. A water bath should be located so that the condensing tubes pass through it, thereby enhancing condensation of the evolved gases.

d. Calibrated glass receiving tube—A common 15-ml centrifuge tube is generally acceptable, although a tube with a larger capacity may be required. The required tube size depends on the amount of retorted material and the total fluids contained within that material. The receiving tube is affixed to the end of the condensing tube with a rubber stopper arrangement.

e. Rock hammer and crusher—A geologist's pick or a masonry hammer may be used. Typical crushers are of the "chipmunk" variety.

f. Diamond saw—A variety of commercial core saws, equipped with diamond saw blades, may be used.

g. Mercury pump—Typical mercury pumps are equipped with a cell, lid, and valve arrangement. Typical cells can accommodate a sample with a bulk volume of 10 to 15 cm^3. The mercury pump is equipped with a vernier caliper graduated in 0.1 cm^3 increments. A pressure gauge capable of indicating pressures to 1,000 psi (6.895 MPa) is connected to the system.

h. Centrifuge—A hand-crank centrifuge capable of holding four of the 15-ml centrifuge tubes is suitable. Other centrifuges commensurate with the tube size and centrifugal forces required may be used.

4.2.1.3 Procedures/Precautions

The basic procedure is normally conducted on samples taken from full-diameter cores. Typical analyses are done on a foot-by-foot basis, except some lithologies such as shale, anhydrite, gypsum, and heavy clays are usually described and skipped in this type of analysis. A representative sample approximately two inches in length is broken from each foot using a geologist's or similar type hammer. This sample is broken lengthwise into two halves. One half is designated for the fluid saturation sample and the other half is used to obtain a plug for the determination of permeability and/or Boyle's Law porosity (see 5.3.2.1.1 and 5.3.2.2.1). Alternatively, a 1 to 2 inch (25.4 to 50.8 millimeter) piece may be broken adjacent to the piece selected for fluid saturation testing and the plug drilled from it. The advantage of this latter sampling method is that it can afford a larger and longer plug, thereby reducing the potential for drilling fluid contamination.

The sample (10 to 15 grams) for the gas space measurement should be chipped or shaped to a convenient size, i.e., dimensions that allow it to be placed in the cell of a mercury pump. In addition, the sample should be well rounded to permit mercury conformance. The weight of the sample is recorded and the bulk volume of this sample is determined by mercury displacement for later use in the calculation of a summation-of-fluids porosity (see 5.3.2.2.2 and 4.2.1.4). The cell valve is closed and the pressure increased to 750 to 1,000 psi (5.171 to 6.895 MPa) depending upon the permeability and/or porosity of the sample. The amount of mercury injected (properly corrected by pump calibration procedures, see 4.2.1.8) is recorded.

The sample for the water and oil measurement should be crushed into approximately $^1/_4$-inch (6.4-centimeter) pieces. The crushed core material is screened using a 3 or 4 Tyler mesh screen [approximately 0.25 inch (6.4 millimeter)] to remove any fines created in the crushing process. An amount of this material (usually 100 grams to 175 grams) is weighed to the nearest 0.01 gram and poured into the retort. The retort lid is secured and the retort, along with other similarly prepared retorts, is placed in the oven. The oven temperature is initially maintained at 350°F (177°C) (Hensel, 1982[1]) until all samples cease to give up water. The water volumes are recorded and the oven temperature is then raised to a temperature between 1,000 and 1,200°F (538 and 649°C). The fluid collection tubes are monitored and when all samples cease to give off fluids the process is considered complete. The retorting time usually ranges from 20 to 45 minutes. The total water and oil volumes are recorded.

The initial oven setting of 350°F (177°C) is selected to remove pore water, absorbed water, interlayer clay water (e.g., smectite), and waters of hydration (e.g., $CaSO_4$), but not hydroxyl clay water. However, this method is not appropriate for samples containing gypsum or large amounts of montmorillonite. Gypsum bearing core analysis procedures are described in 4.8 as a special core analysis procedure. Although samples containing montmorillonite can pose problems in determining accurate fluid saturation, heating to 350°F (177°C) is documented (Brown, 1961[2]; Mackenzie, 1970[3]) as the lowest temperature at which hydroxyl waters are desorbed. Although the clay hydroxyl water weight loss at this temperature may be as small as 5 percent, it may impact the final water and oil saturation values. This method is superior to other retorting techniques, e.g., the plateau method (Hensel, 1982[1]). The plateau method suggests that the plotting of water collected versus time data, while continuous heat is applied, will yield plateaus whereby a proper time for reading only pore water can be determined for each formation being tested.

The final retorting temperature of 1,000 to 1,200°F (538 to 649°C) may yield incorrect oil volumes. Some core material may contain solid hydrocarbons, e.g., kerogen, gilsonite, etc. that break down at these temperatures. The analyst should take mineral composition into consideration and may elect to make arbitrary and judicious corrections for the oil collected. In extreme cases, the retort procedure should not be used and other methods, e.g., distillation extraction (Dean-Stark) (see 4.3) procedures should be employed.

Other sources of error in the measurement of oil volumes stem from: (a) mechanical hold-up in condensing tubes, (b) leaking retort seals, and (c) coking and cracking of certain

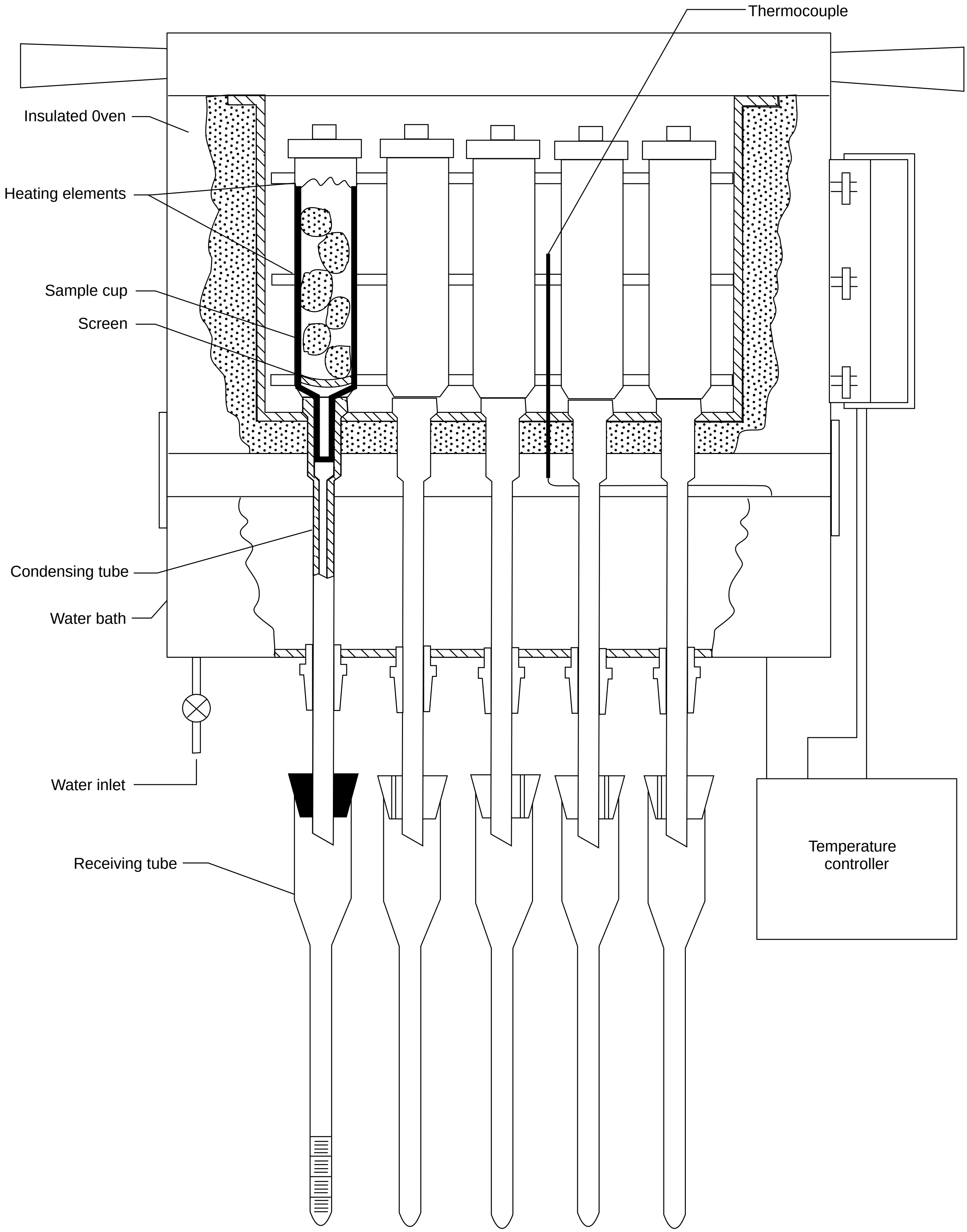

Figure 4-1—Stainless Steel Oven Retort and Water Bath—Atmospheric Pressure

oils. Oil volume correction curves (See Figure 4-2) must be employed routinely.

The "total" water collected after the complete process may be of value in determining if "pore" water may have been mechanically retained in the condensing tube. The analyst may note a calculated grain density for a sample that is anomalous to the general trend. A comparison between the initial (pore) water volume and the final (total) water volume indicates a difference greater than expected based on similar water volume differences from adjacent samples. Laboratory personnel may then elect to make judicious corrections to the initial water volumes so that reasonable data may be obtained. The reporting of obviously anomalous data is not in the best interest of the end user. Such corrected data should be flagged and a footnote inserted.

Crushed core material is vulnerable to fluid loss due to evaporation, particularly of water, due to the high surface area of the crushed core. Exposure time for the crushed core material should be minimized.

Note: Since handling mercury is required for this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.1.4 Calculations

The corrected oil (See Figure 4-2) and pore water collected for each sample along with the gas volume may be expressed as a percentage of bulk volume as follows:

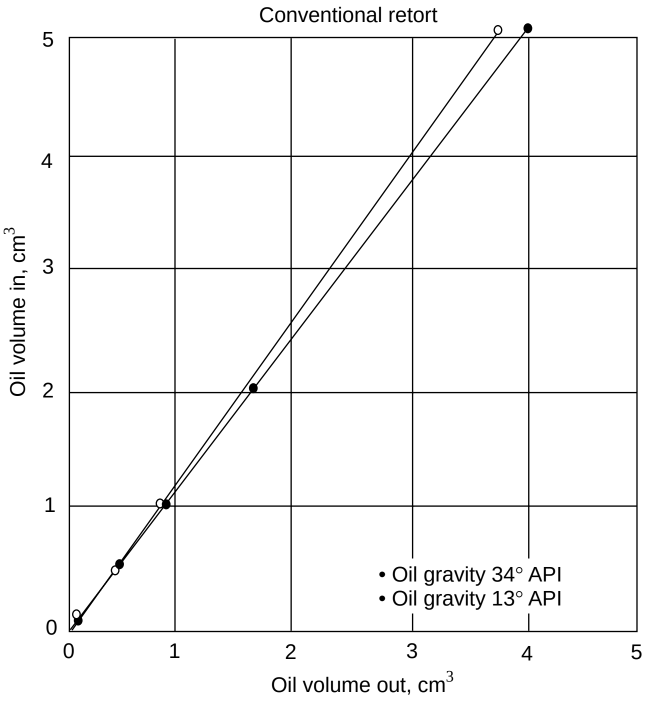

Figure 4-2—Retort Oil Correction Curve

$$NDHg = WtHg/BvHg$$

$$BvRet = WtRet/NDHg$$

$$GB = (HgInj \times 100)/BvHg$$

$$OB = (OilVol \times 100)/BvRet$$

$$WB = (WatVol \times 100)/BvRet$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

NDHg = natural density of mercury pump sample, g/cm^3.
WtHg = initial weight of mercury pump sample, g.
BvHg = bulk volume of the mercury pump sample, cm^3.
WtRet = weight of the retort sample, g.
HgInj = volume of injected mercury corrected by pump calibration factors, cm^3.
OilVol = volume of recovered oil corrected with oil calibration curves, cm^3.
WatVol = volume of initial water recovered, cm^3.
BvRet = bulk volume of the crushed retort sample, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing procedure (Hensel, 1982[1]). These grain density data are of primary value as a means of quality control to ensure that erroneous readings have not been taken, that all fluids have been determined properly, and no calculation errors have been made. This is not the most accurate means of determining the true grain density of the rock material; however, the values obtained should be reasonably representative of the lithology being tested. Thus, the grain density calculation may be considered as a qualitative data evaluation tool.

4.2.1.5 Advantages

Advantages of the basic retort method include:

a. Fluids are collected from relatively large samples ensuring better representation of the lithology and increasing the potential precision of all measurements.
b. The analytical process is rapid and provides required saturation data within hours. Many samples can be evaluated in a short time and in a cost-effective manner, if sufficient retorts and ovens are available.
c. Fluid volumes are measured directly as opposed to other analytical techniques that rely on combinations of fluid volumes and total weight loss.

d. Grain loss, commonly associated with friable sandstones and some carbonates, does not affect the fluid saturation data.

4.2.1.6 Limitations

Limitations of the basic retort method include:

a. Water saturation (and porosity) may be too high if samples contain large amounts of montmorillonite or gypsum that break down at high temperature. This in turn will result in oil saturations that are too low since the volume of oil would be expressed as a percentage of a pore volume that is too high.

b. Oil saturation (and porosity) may be too high if the sample (e.g., some shales) contains solid hydrocarbons that break down at high temperature. This in turn will result in water saturations that are too low since the pore water is expressed as a percentage of a pore volume that is too high.

c. Oil volume correction curves are required and it may not be feasible to obtain the corrections for the oil in question. It may be necessary to use generalized correction curves, based on reported or assumed API gravities.

d. A second piece of core material is required to determine bulk volume, natural density, and gas-filled pore volume. This sample must be lithologically similar to the sample that is crushed for the oil and water data.

e. The distilled liquids may form emulsions.

4.2.1.7 Accuracy/Precision

Accuracy/precision of the basic retort method is:

a. By using an oil volume correction curve, the accuracy of the oil value obtained is within ±5%, and reproducibility is within ±2.5% of measured volumes.

b. The accuracy of the water volumes obtained by this method is ±2.5% of measured volumes.

4.2.1.8 Calibration

Calibration issues for the basic retort method include:

a. Graduated glassware should be checked for accuracy by using a microburette with water. The cleanliness of the glassware could impact readings due to a lack of complete wetting and indistinct meniscus.

b. The mercury pump used for bulk volumes should be calibrated with steel blanks of known volumes. Additionally a pump/system factor should be determined at the pressure used to determine the gas-filled pore volume. Correction curves and/or equations should be used in both instances.

c. All balances should undergo routine maintenance.

d. Retorts should be regularly subjected to vacuum tests to ensure the seals are performing properly.

4.2.2 Percussion Sidewall Cores

4.2.2.1 Principles of Analysis

The method is a modification of the basic retort procedure (see 4.2.1). The main difference is that gas, oil, and water volume measurements are made on the same sample as opposed to the basic procedure where adjacent core pieces are required. Smaller samples are involved in the sidewall procedure and therefore the equipment is scaled down to provide comparable accuracy. This method may be applied to rotary sidewall cores, but damages the samples for further testing.

4.2.2.2 Apparatus

Suitable apparatus for retorting of sidewall cores include:

a. Stainless steel retorts—See Figure 4-3. The overall design is similar to the retorts used in the basic procedure, i.e., the retort is composed of a cup section that holds the sample, a gasket/lid arrangement, and a long condensing tube. The cup section is greatly reduced in size. A bottle cap may be substituted for a screw-on lid, such cap being affixed with a standard capping device. The condensing tube is shorter and its outside and inside diameters smaller than the tubes used for the large retorts.

b. Oven—The sidewall oven is composed of the same components as the oven used in the basic procedure (see 4.2.1.2, b). The sidewall oven is tailored to accommodate multiple sidewall retorts.

c. Calibrated glass receiving tubes—The 15-ml centrifuge tube is not recommended, rather the receiving tube should be designed to accommodate very small fluid volumes (e.g., a maximum of 5 ml). The graduations should be such that readings/interpolations to the nearest 0.01 ml are possible.

d. Mercury pump—The same mercury pump used for the Basic Procedure (see 4.2.1.2, g) may be used, however a small sample chamber will enhance the bulk volume and gas space measurements.

e. An apparatus capable of piercing the jar lid is optional (see 4.2.2.3).

f. A hot wire gas detector capable of a qualitative measurement of combustible gases is optional (see 4.2.2.3).

g. A gas chromatograph is optional (see 4.2.2.3).

4.2.2.3 Procedures/Precautions

4.2.2.3.1 Procedures

Samples should be received in the laboratory sealed in glass jars with screw-top lids. The samples should be arranged in descending depth order. Prior to opening the jar many laboratories employ a procedure that involves puncturing the jar lid and making a measurement of the contained gases. This procedure will not be discussed in detail in this section, however this optional procedure measures combustible gases with a hot wire detection device and some laboratories determine the head space gas composition by chromatography.

The sample recovery (sample length), while it is still in the jar, is noted by calibration marks on the side of the jar. Each sample is then removed from the jar and is cleaned of drilling fluid solids by using a single edge razor blade or utility knife. A brief description of the sample is recorded including rock type, grain or crystalline size, siltiness, shaliness, fossils, color, intensity, and distribution of fluorescence, and other megascopic lithological features. The sample is weighed to the nearest 0.01 gram.

Some laboratories may divide the prepared sample so as to provide a portion for a permeability and/or other measurements, e.g., particle size distribution, x-ray diffraction, etc. Obviously this is limited to initial samples of large size, e.g., 2 inches (50.8 millimeters) in length. Even so the fluid saturation data are compromised by testing a smaller sample. Most laboratories employ an empirical approach to sidewall permeability data. Neither of these practices will be discussed in this section.

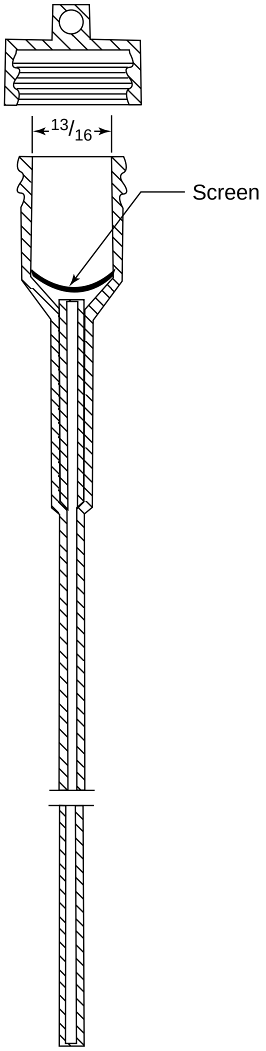

Figure 4-3—Retort Cup—Sidewall Sample

The sample is placed in the cell of a precalibrated mercury pump, and bulk volume is determined by mercury displacement (see 5.2.1). With the sample immersed in mercury, a valve is closed and the pressure raised to 750 psi (5.171 MPa). In tight (low permeability) samples, the pressure is raised to 1,000 psi (6.895 MPa). This procedure measures the gas-filled pore volume assuming that the compressibility of the water and/or oil present in the other pore spaces is minimal.

The sample is removed from the cell and broken into smaller pieces to observe the mercury penetration pattern. The penetration pattern is helpful in predicting probable production.

The retorting procedure described in 4.2.1.3 is followed, including the recording of initial (pore) and total water collected and the use of oil volume correction curves (see Figure 4-2).

4.2.2.3.2 Precautions

Sidewall core material is subject to fluid loss due to evaporation so exposure time to room conditions should be minimized.

Note: Since handling mercury is required with this fluid saturation method all existing safety and health standards should be followed. Consult relevant environmental regulations and local laws.

4.2.2.4 Calculations

The oil, pore water, and gas-filled pore volume may be expressed as percentage of bulk volume as follows:

$$GB = (HgInj \times 100)/BV$$

$$OB = (OilVol \times 100)/BV$$

$$WB = (WatVol \times 100)/BV$$

$$GS = (GB \times 100)/(GB + OB + WB)$$

$$OS = (OB \times 100)/(GB + OB + WB)$$

$$WS = (WB \times 100)/(GB + OB + WB)$$

Where:

BV = bulk volume of the sample, cm^3.
$HgInj$ = volume of injected mercury corrected by pump calibration factors, cm^3.
$OilVol$ = volume of recovered oil corrected with oil calibration curves, cm^3.
$WatVol$ = volume of initial recovered water, cm^3.
GB = gas saturation as a percentage of bulk volume.
OB = oil saturation as a percentage of bulk volume.
WB = water saturation as a percentage of bulk volume.
GS = gas saturation as a percentage of pore volume.
OS = oil saturation as a percentage of pore volume.
WS = water saturation as a percentage of pore volume.

Grain density may be calculated from the data obtained during the testing by slight modifications to the method proposed by Hensel, 1982.[1]

The bulk density, sometimes used in various laboratories, may be calculated by dividing the initial (whole) sample weight by the bulk volume.

4.2.2.5 Advantages

Advantages of the sidewall retort procedure include:

a. Gas, oil, and water measurements are made using one sample.
b. The method is relatively rapid.
c. This is a "direct" measurement method where gas, oil, and water measurements are made independently.

4.2.2.6 Limitations

Limitations of the sidewall retort procedure include:

a. The distilled liquids may form emulsions.
b. Oil and water calibration is required.
c. Volumes of oil <0.1 ml are difficult to determine accurately.
d. For very friable or unconsolidated sands the measured gas-filled pore volume may be high due to sample expansion when overburden pressure is released.
e. Samples must be handled carefully to minimize loss of liquids, especially water. In a low humidity atmosphere, water loss may be substantial.
f. Porosity and fluid saturation values can be in error if gypsum or hydratable clays (e.g., montmorillonite) are present and precautions are not taken in recording only "pore" water.
g. Calibration of all equipment is critical because samples are typically small and may contain equally small amounts of oil and water. Minute differences in recorded fluid volumes may result in significant ranges of saturation and porosity values.

4.2.2.7 Accuracy/Precision

Accuracy/precision for the sidewall retort procedure is:

a. The method's accuracy is strongly dependent on sample size, especially when determining oil volumes.
b. Analytical discretion should be used on testing samples with an initial weight of less than 3 grams.
c. By using oil volume correction curves, the precision of the oil value obtained is within ±5 percent of the measured volumes.
d. The precision of the water volumes is within ±3 percent of the measured volumes.

4.2.2.8 Calibration

Refer to 4.2.1.8 of the basic retort procedure.

4.3 DISTILLATION EXTRACTION METHOD (DEAN STARK)

4.3.1 Plug Samples

4.3.1.1 Principles of Analysis

This procedure is appropriate for plug samples and for rotary sidewall cores. The distillation extraction (Dean-Stark) method of determining fluid saturation depends upon the distillation of the water fraction, and the solvent extraction of the oil fraction from the sample. The sample is weighed and the water fraction is vaporized by boiling solvent. The water is condensed and collected in a calibrated receiver. Vaporized solvent also condenses, soaks the sample, and extracts the oil. The sample is oven dried and weighed. The oil content is determined by gravimetric difference.

4.3.1.2 Apparatus and Reagents

The following apparatus is suitable for this method and is illustrated in Figure 4-4. The apparatus should be set up in a fume hood or room suitable for exhausting solvent vapors.

a. General—The apparatus consists of an electric mantle or heating device with thermostatic controls. The distillation/extraction unit for one sample consists of a boiling flask, thimble, trap or calibrated sidearm, and condenser.
b. Flask—The flask is wide-mouthed, long-necked, and may contain indentations at the base of the neck to support the extraction thimble.
c. Trap—The trap or sidearm has a graduated section marked in 0.1 ml divisions. The graduated trap can be made smaller or larger to accommodate samples with very small or very large water volumes. Attached to the graduated section is a glass tube or arm that is bent at a right angle with a glass joint on the end. The glass joint has a drip tip molded on it and the opening in the tip should be designed so that the dripping solvent is directed in the center of the flask to ensure soaking of the core plug below. A modification to the trap (Figure 4-5) enables the water to be determined gravimetrically.
d. Condenser—The condenser is a water-cooled, reflux, glass-tube type, with a condenser jacket approximately 11.8 inches (300 millimeters) long and an inner tube (see Figure 4-4). The bottom is fitted with a drip tip and the inside tube should be vertical to reduce the difficulty of removing water from the surfaces of the condenser and trap.
e. Desiccant holder—A glass tube desiccant holder is fitted to the top of the condenser when samples are being extracted and a rubber stopper is used when the apparatus is idle.
f. Extraction thimbles—A glass thimble with a fritted glass bottom is recommended for holding a plug sample. Alternatively, a cellulose thimble may be used. However, types of thimble materials other than glass may cause errors in weighing because of absorption of atmospheric water. Also, thimbles made of glass allow viewing of the plug to ensure

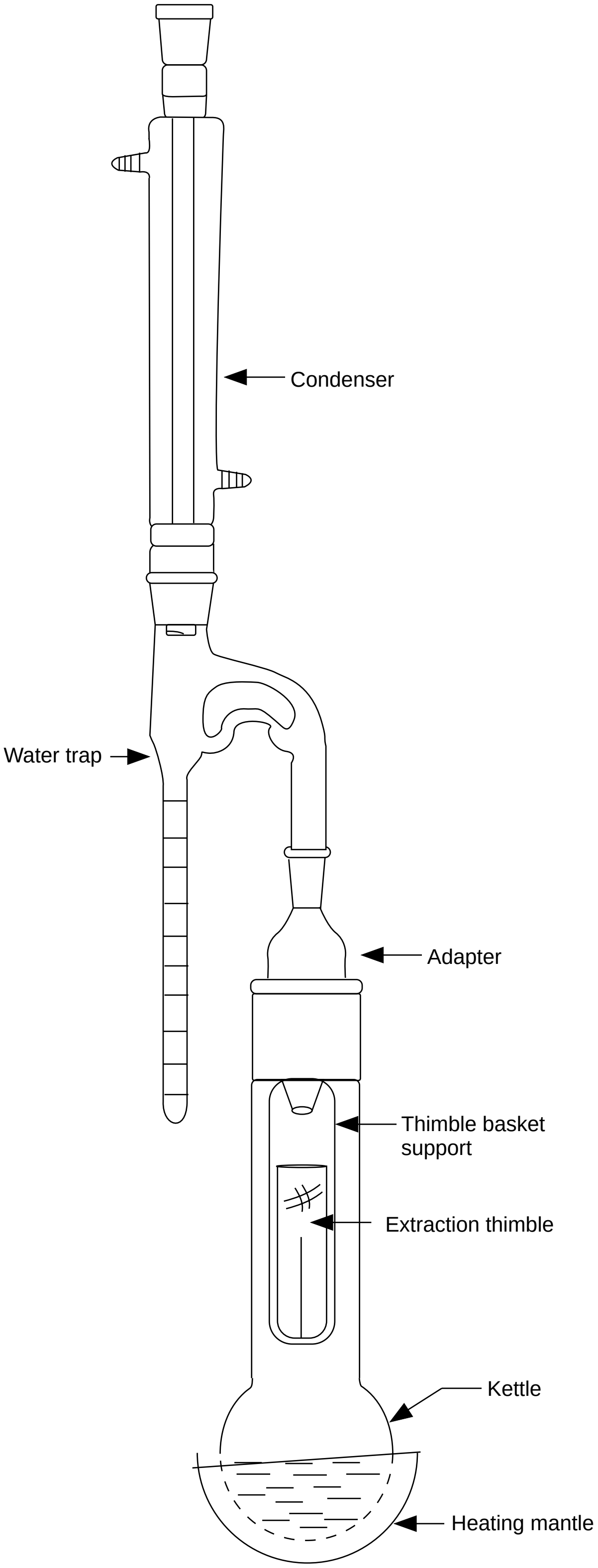

Figure 4-4—Dean-Stark Apparatus for Volumetric Determination of Water

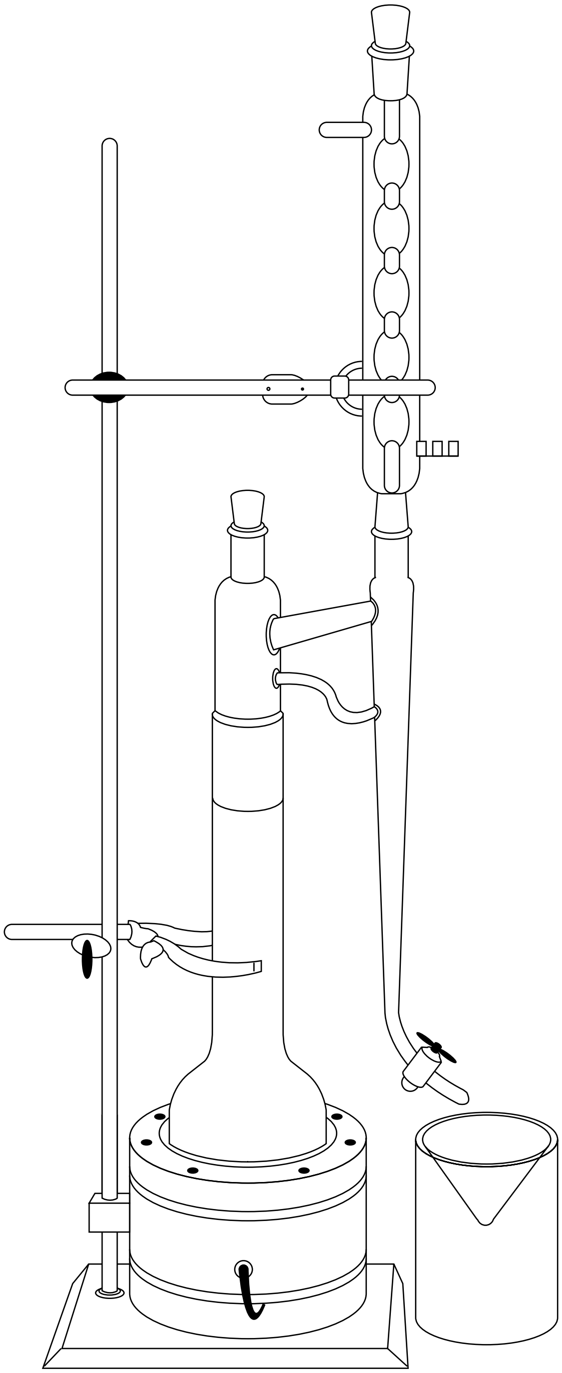

Figure 4-5—Dean-Stark Extraction Apparatus for Gravimetric Determination of Water

that the solvent is dripping and soaking the sample for thorough extraction. A small piece of glass wool may be used to cover the core plug to prevent erosion by the solvent reflux from the condenser.

g. Extraction cup—Cups fitted with siphons can be used to hold the thimbles and allow alternating immersion/drainage cycles. This can increase the efficiency of the extraction process.

h. Drying oven—A convection or vacuum oven may be used having a temperature control of ±2°C. An explosion-proof model is preferred, but non-explosion proof models can be used. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate the expected hydration state of the clays in the reservoir. (Humidity levels during drying procedures are important in maintaining proper waters of hydration.)

i. Boiling chips—Glass beads or small chips of alundum material may be placed in the bottom of the boiling flask. These aid in reducing the tendency of the solvent to "bump" as it becomes more saturated with oil. If bumping or super heating occurs it creates an undesirable boiling process, contaminating the sample and extending the time required for the initial phase of the distillation extraction process.

j. Solvent—Toluene (reagent grade) or other suitable solvent.

4.3.1.3 Procedures/Precautions

4.3.1.3.1 Procedures

The samples are prepared using a fluid that is compatible with the fluid used to cut the whole core or drilled sidewall cores in the well. This ensures the preservation of residual oil or water saturation depending on the use of oil or water-based fluid. In the case of frozen oil sands samples, liquid nitrogen should be used. The excess fluid remaining on the sample after the trimming of the ends should be wiped off using a material that will remove the surface fluid but will not pull fluid from the interior in a sponge-like manner. This process is best described as a squeegee action. The sample's surface should appear dry (with a dull luster) and not shiny with excess fluid. The thimble/plug sample should be weighed to the nearest milligram (0.001 gram) on an analytical balance. The process should be accomplished quickly to minimize evaporation of the fluids in the sample. After final weighing, the samples and/or thimbles should be placed in the apparatus immediately, or should be stored in a container to prevent evaporation until they are placed in the apparatus. This storage period should be kept to a minimum. The necessity for weighing each component is to ensure complete accountability for weight loss. When extraction thimbles are used they should be at room temperature and thoroughly dry.

The samples are extracted with a solvent that does not donate to or absorb any of the collected water. Solvent conditioning can be provided by adding at least 1 percent water by volume to the solvent and pre-boiling the solvent until the water concentration is stable. Some laboratories add 15 percent water by volume to the solvent. Water should be in excess of the amount required to bring common solvents into equilibrium when the water is boiled away.

The desiccant trap ensures that atmospheric moisture (humidity) does not affect the water collected.

Water can adhere to the condenser and to the side of the trap. This fluid may cause appreciable error, particularly for a plug sample of low porosity. The adhered water is dislodged with a stiff solvent stream from a laboratory squirt bottle or a small gauge wire with a very small loop at the end. The wire is used to physically dislodge water particles that are not removed by the stream of solvent. Detergent is sometimes used to flatten the solvent/water interface for improved accuracy; but the use of detergent is not generally recommended because the effects of three phase fluid interaction are unpredictable and the wettability of the sample will be altered.

The distillation/extraction process continues for a minimum of 48 hours. Water levels should be monitored daily and the process stopped only when there is no change in volume of water recovered in 24 hours. Longer times may be required depending on the size of the plug sample and its permeability. This is to ensure that the solvent used has extracted all the oil possible from the plug material. Where heavy oil (low-gravity, high-asphaltene) is involved, another solvent type (see 3.1) is required to thoroughly clean the plug. A complete record of the volumes of water collected should be noted. The rate of extraction and efficiency may be increased by using extraction cups equipped with siphons. Alternative methods that speed extraction involve using the described apparatus until the amount of water collected is at a stable end point, and then doing one of the following with the samples and the thimbles:

a. Place the samples in a vapor phase unit to complete the oil extraction.

b. Place the samples in a CO_2-toluene pressure core cleaner to complete the oil extraction. This should be restricted to competent, low-permeability samples that will not be physically altered by this process.

c. Place the sample in a Soxhlet extractor for alternating immersion and drainage type oil extraction.

d. Place the sample in a flow-through oil extraction apparatus.

e. Alternate between solvent types (e.g., toluene and methanol).

The extraction efficiency is evaluated by treating the sample with chlorothene under an ultraviolet light source to determine if there is any remaining oil, that fluoresces, or by measuring the grain density of the sample. If the grain density is lower than anticipated for the rock type, the sample may need further extraction.

The sample/thimble should then be dried to a stable weight. Samples with high saturations of flammable solvents should be dried in an explosion-proof convection or vacuum oven, unless the excess solvent is allowed to evaporate before being put into the oven. This avoids a potential explosion or fire situation. When the sample/thimble is dried, it should be allowed to cool to room temperature in a sealed container such as a desiccator and the weighed. An oven that adds moisture to the sample should not be used for this step. However, when porosity and permeability will be measured, samples containing large amounts of montmorillonite and other clays may need to be dried in a humidity oven to preserve the hydrated state found in the reservoir. (The humidity level in the oven is important in maintaining proper waters of hydration for measuring permeability and porosity.)

Errors occur by not accounting for a chipped thimble, the core losing small particles, and/or the loss on the sample surface of precipitated salt from concentrated brines. In the case where heavy salt precipitation occurs, the salt is removed from the sample by extraction with methanol or similar solvent. The sample is then dried and weighed. The weight of water collected in the trap is subtracted from the total liquid weight loss to determine the oil weight extracted from the plug sample.

Proper water flow in the condenser will maintain a condenser temperature sufficiently cool so that the vapors condense in the bottom third of the condenser column.

4.3.1.3.2 Precautions

Local regulations regarding the safe use of reagents should be followed. In general, however, the following safety considerations should be noted:

a. Oil may contain compounds exhibiting carcinogenic properties. It may be flammable.

b. Toluene is moderately toxic by skin absorption and inhalation. It possesses irritant and anesthetic properties and is highly flammable.

c. Many other solvents, while effective for the oil removal process, may be dangerous and toxic. Consult relevant environmental regulations and local laws governing the use of these solvents.

d. Solvent vapors should condense in the lower third of the water-cooled heat exchanger on top of the water trap.

e. The analyst should be aware of the change in solvent boiling point with altitude or the change in the water boiling point due to salt in solution. The solvent's boiling point should be checked to ensure an adequate temperature for water distillation. When KCl drilling fluids are used, the filtrate water will have salt concentration on the order of 300,000 ppm, and, consequently, boil at a much higher temperature than fresher water. The use of orthoxylene is suggested as a substitute for toluene in these cases.

4.3.1.4 Calculations

The following calculations are appropriate for this method:

$$\text{Weight \% Water (Gravimetric)} = \frac{(\text{Weight of Water}) \times 100}{(\text{Initial Sample Weight})} \tag{1}$$

or

$$\text{Weight \% Water (Volumetric)} = \frac{(\text{Volume of Water}) \times (\text{Density of Water}) \times 100)}{\text{Initial Sample Weight}}$$

$$\text{Weight \% Solids (Gravimetric)} = \frac{\text{Dry Weight of Sample} \times 100}{\text{Initial Sample Weight}} \tag{2}$$

$$\text{Weight \% Oil (Gravimetric)} = \frac{(\text{Initial Weight} - \text{Dry Weight} - \text{Weight of Water}) \times 100}{\text{Initial Weight of Sample}} \tag{3}$$

The saturations are normally expressed as percentages of the sample pore space. Therefore, the sample porosity, water density, and oil density are required. If the connate water is a highly concentrated salt solution, the water density must be corrected for the salt in solution.

The following calculations apply:

$$\text{\% Water} = \frac{\text{Volume of Water} \times 100}{\text{Pore Volume}} \tag{4}$$

$$\text{\% Oil} = \frac{(\text{Weight of Oil})/(\text{Density of Oil}) \times 100}{\text{Pore Volume}} \tag{5}$$

The liquid content of the sample is reported to the nearest 0.1 percent of the pore space; e.g., 22.1 percent oil and 43.7 percent water.

Knowing the brine salinity and density, one can calculate volume of brine that was in the core from the volume of distilled water recovered as follows:

$$Vbr = [(Vw)(\rho_w)/\rho_b]\,[1{,}000{,}000/(1{,}000{,}000 - Cs)] \tag{6}$$

Where:

Vbr = volume of brine corresponding to the volume of distilled water collected from the plug, cm^3.

Vw = volume of distilled water collected from the plug (e.g., Dean-Stark), cm^3.

ρ_w = density of distilled water, g/cm^3.

ρ_b = density of lease brine having a concentration C_s of salt, g/cm^3.

C_s = concentration of dissolved salts in lease brine, =

$$\frac{(1{,}000{,}000)(\text{Weight of Salt})}{(\text{Weight of Brine})}\text{ppm}$$

4.3.1.5 Advantages

Advantages of this method include:

a. Water volume determinations are generally very accurate.

b. Typically, the sample is not damaged and can be used for further testing. However, the wettability may be altered and certain clays (e.g., montmorillonite) or gypsum may also be subject to change.

c. Relatively low temperatures [212°F (100°C)] are used; hence, little if any of the clay hydroxyl waters are removed.

d. The procedure is simple and requires little attention during distillation.

4.3.1.6 Limitations

Limitations of this method include:

a. Inaccuracies arise in the water determination due to the following:

1. Atmospheric water condenses in the condenser when atmospheric humidity is high. Desiccant tubes can be used to avoid the problem.
2. Water evaporates from the sample at room temperature when it is not immediately set up in the extractor with the condenser water circulating.
3. Water droplets stick to unclean glass in the sidearm or condenser.
4. Salt may precipitate inside the sample from connate brines (saline interstitial water). This can result in significant changes in porosity and/or permeability. Salt can be removed from the sample with methanol extraction.
5. Correction for the higher density of salt water is required when the total solids concentration exceeds 20,000 ppm (see 4.3.1.4, Equation 5).
6. Incomplete drying of solvents.
7. Loss of water due to the joints of the extraction flask not being vapor tight, or from too high an extraction temperature, or from insufficient water flow in the condenser.
8. The density of air must be considered for buoyancy only when the sample is weighed to the nearest 0.1 mg.
9. Extraction time may be insufficient.
10. Water saturation may be too high if samples contain large amounts of gypsum (see 4.8) or montmorillonite clays (water of hydration). Permeability and porosity values can also be altered if waters of hydration that are present in the reservoir are removed during extraction and drying procedures (humidity oven drying may be preferred).
11. If the true oil density is not known, error is introduced into the calculation of oil saturation because an oil density value must be assumed.

b. Oil volumes are not found directly and may be inaccurate due to the following:

1. Additional water collected or lost from the sample as mentioned above.
2. Loss of solids.
3. Incomplete cleaning of oil.
4. Drying at a higher temperature than the extraction temperature may remove additional water of hydration and overstate the oil volume.

c. Rock wettability may be altered.

d. The clay fabric may be altered, that may result in inaccurate permeability measurements.

e. There is no check on the analysis accuracy.

4.3.1.7 Accuracy/Precision

There are no documented standards and the accuracy of the methods cannot be assessed. However, with the calibration procedures noted in 4.3.1.8, water volume reproducibility can be evaluated. Similar procedures assess oil volume accuracy. On relatively small samples or samples containing high gas saturation with residual volumes of oil and water, the percent error for liquid saturations may be ±50 percent of values measured. Percent error will be significantly less as liquid volumes increase.

4.3.1.8 Calibration

The water measurement accuracy should be checked regularly to be sure no bias affects the results.

a. For gravimetric determination—Known weights of water added to the extractors are plotted against the weights of water recovered by extraction under conditions identical to those used in extracting an oil sand sample. The water correction factors may change due to the efficiency of the condensers used in the apparatus. Typical values are listed below:

$$\text{Corrected Weight Water} = (\text{Weight Water} \times a) + b$$

Where: a and b, respectively, are the slope and intercept of the calibration equation, that is, $a = 1.003, \pm 0.001$; $b = 0.090, \pm 0.009$.

b. For volumetric determination—A calibrated buret is used to deliver known volumes of water into the water trap. The traps should have a maximum scale error of 0.02 ml. Volume correction factors can be calculated and applied, if necessary.

4.3.2 Full Diameter Cores

4.3.2.1 Principles of Analysis

The distillation extraction method of determining fluid saturations for full diameter samples is governed by the same principles and procedures as those for plug samples (see 4.3.1.1). Differences in the procedure and apparatus are discussed below.

4.3.2.2 Apparatus

The following apparatus are appropriate for this method:

a. Flask—The flask used to accommodate the full diameter core sample must be larger than the plug analysis flask with a larger solvent volume for oil removal.

b. Trap—The trap or sidearm has to be large enough to accommodate the volumes of water from large samples. The graduated section of the trap should be marked in 0.1 ml divisions.

c. Extraction core protector—Extraction thimbles are not generally used for full diameter samples. Instead a stocking material made from unbleached cotton can be used to prevent the loss of fines from the full diameter core. Thimbles normally used in plug core analysis are not manufactured in sizes that fit the full diameter core.

4.3.2.3 Procedures

The procedures are the same as those for plug samples with the exception that sample weights should be made to the nearest 0.1 gram. Also, the cleaning time of 48 hours may have to be extended in order to fully clean the larger volume samples. Longer times and supplemental cleaning are frequently required.

4.3.2.4 Calculations

See equations in 4.3.1.4.

4.3.2.5 Advantages

Refer to 4.3.1.5. There are additional advantages when performing this test on full diameter cores, as follows:

a. A high precision analytical balance is not required as the weights need only to be determined to the nearest 0.1 gram.

b. Grain loss during the handling and testing procedures is not as critical as with plugs, but still should be minimized.

c. Collected water volumes are quite large as compared to those associated with plug analyses, therefore the water readings need to be recorded only to the nearest 0.1 ml.

d. As the oil volumes are determined by gravimetric difference the final values are not impacted to the same degree by small measurement errors as those determined on plugs, except at very low oil saturations.

4.3.2.6 Limitations

There are additional limitations when analyzing full diameter cores as follows:

a. Larger, more costly equipment is required, e.g., glassware, ovens, cut-off saws.

b. Laboratory space requirements are greater.

c. Large volumes of solvents are used thereby requiring a higher investment in same. Storage and/or disposal logistics are more complex.

d. Longer distillation and core cleaning times are required thereby decreasing data reporting timeliness.

e. Health and safety standards should be carefully monitored and observed due to the large volumes of fumes and solvents that can be involved.

4.3.2.7 Accuracy/Precision

Refer to 4.3.1.7.

4.3.2.8 Calibration

Refer to 4.3.1.8.

4.3.3 Pressure-Retained Core Analysis

4.3.3.1 Introduction

The objective of pressure-retained core analysis is to provide fluid saturation data on cores for which fluid expulsion has been minimized during core barrel retrieval by preventing pressure depletion from bottom hole to surface conditions. A ball valve seals the core barrel and prevents the natural pressure drop as the barrel is brought to the surface. Additionally, the barrel is designed to allow preset pressure maintenance during retrieval with a pressure supply that compensates for pressure loss due to a lower surface temperature.

Flushing of the core with drilling fluid filtrate before the core enters the core barrel is minimized with special, low-water-loss drilling fluid, a high coring penetration rate, a low drilling fluid overbalance, and a special core bit design. In addition, prior to coring, the inner barrel is generally filled with a low-invasion gel material. This material minimizes further drilling fluid filtrate invasion via imbibition by displacing drilling fluid from the core surface as the core enters the barrel.

Fluid saturations are the amounts of oil and water present in the core at laboratory conditions. Alteration of original fluid saturations by flushing can only be qualitatively defined. Any drilling fluid filtrate invasion alters saturations from in situ values unless the in situ saturations are at waterflood residuals. In under-pressured reservoirs, filtrate invasion is minimized by using foam as the drilling fluid. With the foregoing precautions, saturations under these conditions can reflect saturations near in situ values.

Rig site handling procedures are addressed in 2.2.5.

4.3.3.2 Principles of Analysis

The cores are kept frozen with dry ice until the laboratory analysis is initiated. This reduces pore pressure, freezes water, immobilizes oil, and traps gas that does not freeze or liquefy at dry ice temperature.

As the core is allowed to thaw in the gas collection cells, gas comes out of solution and expels both oil and water that are captured in a receiving tube at the bottom the cell. Gas is collected in the void space within the cell. This fluid volume does not necessarily reflect the amount of fluid produced by pressure depletion of a conventional core since both different pressure and different temperature conditions exist in the reservoir.

Since oil volumes are determined from the distillation extraction and gas driven solvent extraction steps of the analysis, grain loss must be kept to a minimum for maximum accuracy.

If no free gas is present in the cored zone and the core is captured above the bubble point of the oil, the total liquid saturation should be equal to the pore space after a formation volume factor is applied to the oil. Saturations should not vary more than ±5 percent.

Filtrate invasion analysis allows assessment of the fluid saturation alteration from in situ values. If available, relative permeability fractional flow curves will aid in this assessment. Detailed discussion of invasion principles is provided in 4.3.7.

4.3.3.3 Apparatus

The following equipment is necessary for the analysis of pressure-retained core. These items are considered exclusive to the analysis of pressure-retained cores. Standard core analysis items used in the analysis are not included.

a. Core storage boxes—Boxes used for storage of the frozen core until analysis is initiated. The boxes should be of sufficient size to hold 5-foot (1.52-meter) sections of core in its inner barrel and enough dry ice to maintain the core in a frozen state. These boxes should be insulated to minimize dry ice sublimation.

b. Dry ice—Used to maintain the core in a frozen state until analysis is initiated.

c. Liquid nitrogen—Used for milling of core barrels and during sample preparation such as core cleaning, drilling plugs, and full diameter sample facing.

d. Milling machine—Used to cut diametrically opposed grooves down the length of the inner barrel to facilitate frozen core removal.

e. Two-liter dewar flask—Used for periodic dipping of samples into liquid nitrogen during the cleaning process to aid in cleaning and to keep the core frozen.

f. Drilling fluid or low invasion gel removal tools—Various tools including carpet knife, insulated gloves, small hammer, etc.

g. Gas collection cells—Cell equipped with a pressure gauge to determine gas volumes bled from the core and core barrel, and to allow for chromatographic gas analysis.

h. Gas collection cylinders (250 cm^3)—Used for sampling of cell gas for chromatographic analysis.

i. Centrifuge—Used for centrifuging gas collection cell receiving tubes to obtain good oil/water/solid separation.

j. Heat lamp—Used for gas mixing in the gas collection cell prior to sampling.

k. Portable vacuum pump—Used for pulling a vacuum on the gas collection cell and 250 cm^3 cylinders prior to gas collection.

l. Stainless steel tubes [3-inch (76.2-millimeter) diameter with screen bottoms]—The core samples are placed in these tubes, thereby preventing grain loss from samples during the gas collection and distillation extraction steps of analysis.

m. Surgical stocking material—Used to encapsulate core samples during CO^2 extraction process to minimize grain loss.

4.3.3.4 Procedures

4.3.3.4.1 Core Preparation Procedures

Core preparation procedures include:

a. Core samples, encased in steel tubing and frozen in chests of dry ice, are brought from the wellsite to the laboratory.

b. Each length of tubing-encased frozen core is placed into a dry ice filled trough attached to a milling machine. Two diametrically opposed grooves are milled down the length of the steel tubing to a depth slightly less than the wall thickness of the tubing. Liquid nitrogen is directed at the point of milling to ensure a tubing and core temperature at or below the dry ice temperature.

c. The tubing is separated into two halves and the frozen core is removed.

d. The low invasion gel that is frozen on the core is removed by scraping and brushing. The core sections are periodically dipped in liquid nitrogen to ensure that the core remains frozen.

e. Cores are visually examined for lithological characteristics and samples are selected for analysis.

f. The ends of each selected core segment are faced with a diamond saw using liquid nitrogen as a coolant. The faced frozen core segments are wrapped in plastic wrap and aluminum foil and stored under dry ice to await laboratory tests. The plastic wrap and foil protect the core from sublimation damage.

g. Care while milling open the core barrel ensures safe core handling. Placing a heavy mesh net over the core barrel during the milling process prevents analyst injury should the core barrel separate due to excessive internal pressure caused by drilling fluid expansion during freezing. Natural gas pockets where core is missing in the barrel may ignite in air. The carbon dioxide and nitrogen environment surrounding the core barrel during milling should prevent gas ignition.

4.3.3.4.2 Gas Collection Procedures

Gas collections procedures include:

a. The faced frozen core sample is placed in a thin-walled, metal cylinder with a fine mesh screen in the bottom, weighed quickly, and placed in the gas collection system (see Figure 4-6).

b. The system is immediately assembled and evacuated for 45 seconds to remove air without pulling gas from the sample. The frozen core is then allowed to thaw to room temperature.

c. Water and oil expelled by the evolving gas are collected in a graduated receiving tube attached to the gas collection cell.

d. The evolved gas is collected in the gas collection cell. The system is equipped with a pressure gauge to allow pressure monitoring inside the cell. If the gas collection cell pressure exceeds 0 psig, an attached and previously evacuated secondary cell is then opened to allow collection of additional evolved gas.

e. Barometric pressure, room temperature, system pressure, and produced liquid volumes are recorded periodically. Thawing of the core is considered to be completed when consecutive readings indicate system equilibrium.

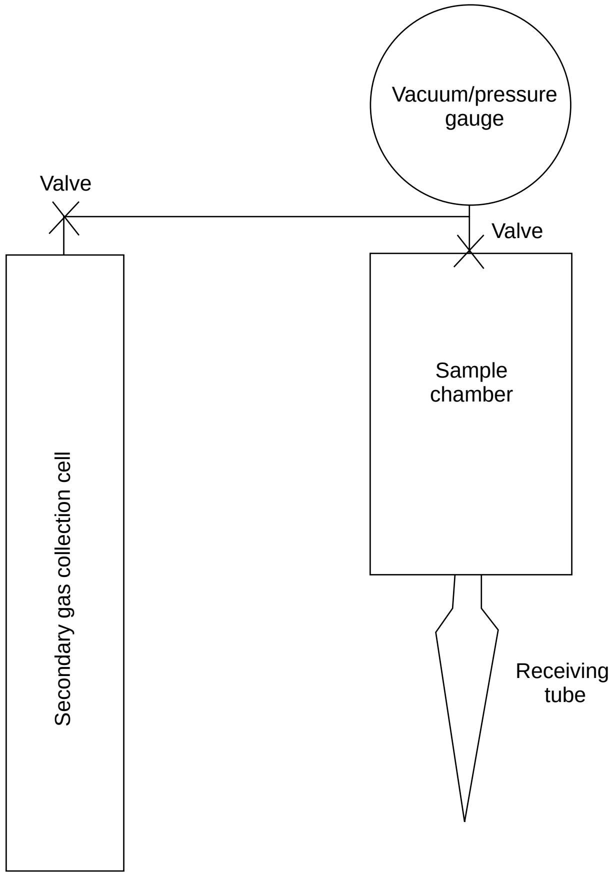

Figure 4-6—Gas Collection/Pressure Depletion System

f. Evolved gas samples may be collected in more than one cell. Gas samples are collected separately from the primary collection cell and, if used, the secondary gas collection cell. The gas samples are analyzed to determine gas gravity and component mole percent. Volumes of oil and water collected are measured. The chloride and bromide content of the water and the produced oil gravity are determined.

g. The sample and its cylinder are removed from the cell, weighed, and placed in a previously prepared distillation extraction apparatus.

4.3.3.4.3 Distillation Extraction (Dean-Stark) Procedures

Distillation extraction procedures include:

a. The sample is placed in the distillation extraction apparatus (see 4.3.1.2), the system is assembled, and heat is applied to distill the remaining water in the core and to extract the remaining oil.

b. When the distillation is complete, as determined by consecutive water readings, the water volume is recorded. The sample and thimble are then removed and placed in a vacuum oven at 240°F (116°C) to remove the extraction solvent. When drying is completed, the sample and thimble are removed from the oven, allowed to cool in the presence of a desiccant, and then reweighed.

c. The volume of additional oil extracted is determined gravimetrically, using an oil density determined from the oil produced in the gas collection step. The volume of water distilled is corrected to reflect the equivalent volume of water having the same salinity as the water recovered during the gas collection phase. If the water expelled during the gas collection is contaminated with drilling fluid filtrate, the water recovered during the gas collection phase may not have the same salinity as less contaminated formation water at the center of the core.

d. The core sample with all loose grains is then encased in a surgical stocking material to minimize grain loss prior to additional extraction procedures.

4.3.3.4.4 Gas-Driven Solvent Extraction and Saturation

Gas-driven solvent extraction and saturation procedures include:

a. The cores are weighed in the stocking material and placed in the gas-driven solvent extractor where they are subjected to further cleaning using carbon dioxide-charged toluene heated to 180°F (82°C). This phase is designed to remove any oil remaining. The weight loss resulting from this extraction process is the weight of additional oil removed that is converted into an oil volume using the previously calculated oil density. In actual practice, the total weight loss from distillation extraction and gas-driven solvent extraction minus the weight of water recovered is used to determine oil volume.

b. The samples are then placed into a convection oven (or vacuum oven) and dried at 240°F (116°C) until weights stabilize. For samples containing large amounts of montmorillonite and other clays, a humidity oven can be used to help preserve a single or double water layer to better approximate expected clay/water interactions in the reservoir. (Humidity levels during drying are important in maintaining proper waters of hydration.)

c. Full diameter porosities along with horizontal air permeabilities are measured on each core segment. In the cases where more than one sample is combined in a foot of analysis, these values are weighted by representative portions of a foot and averaged to arrive at a mean value.

d. Liquid saturations at stock tank conditions are calculated using the measured total pore volume of all of the core segments comprising the sample and the total oil and water contents recovered from these segments. The volume of gas collected from the samples is then determined from the known volume of the gas collection system, corrected for the grain and tare volumes as well as the total liquid and salt contents. This gas volume is then further corrected to standard conditions.

4.3.3.5 Calculations

The following calculations are used for this method:

a. Gas volume at standard temperature and pressure and gas weight from gas collection:

$$\text{Gas Volume} = \frac{\text{Gas Collection Cell Volume} \times \text{Cell Pressure} \times 520 \times (1 - \text{Air Fraction})}{14.7 \times (460 + \text{Temperature})}$$

$$\text{Gas Weight} = \text{Gas Volume} \times 0.0012232 \times \text{Gas Gravity}$$

Where:

Gas collection cell volume is in ml.
Cell pressure is in psia.
Temperature is in degrees F.
Gas weight is in g.
Gas volume is in ml at STP.
Gas gravity = (density gas, g/ml)/(density of air, g/ml).
Density of air at STP = 0.0012232g/ml.
STP is standard temperature (0°C) and pressure (1 atmosphere).

The (1 – air fraction) term is a correction factor. Evacuation of the gas collection cells removes all but about 5 percent of the air in the system and small leaks may develop at the seal lid allowing air to be pulled into the system. Analysis of a gas sample drawn from the collection cell determines the amount of air present.

The gas weight is used in checking the material balance of the total weight loss as a result of extracting gas, oil, and water from the sample with the calculated weight loss.

b. Saturations:

$$\text{Oil Saturation} = \frac{(\text{gcc Oil Vol} + \text{Dist Ext Oil Vol} + \text{CO2 Ext Oil Vol}) \times 100}{\text{Pore Volume}}$$

$$\text{Water Saturation} = \frac{(\text{gcc Water Vol} + \text{Dist Ext Water Vol}) \times 100}{\text{Pore Volume}}$$

Where:

gcc Water Vol = gas collection water volume, cm^3.
gcc Oil Vol = gas collection oil volume, cm^3.
Oil saturation is given as percent pore volume.
Water saturation is given as percent pore volume.
All oil and water volumes are in ml.

c. Water volume from distillation extraction—See 4.3.2.

d. Pore volume and grain density correction—When salts are not leached from the core samples, corrections in pore volume and grain density are made based on water salinity determined from the water produced during gas collection and pressure depletion. Alternatively, methanol can be used to leach salt from the core.

$$\text{Pore Volume (corrected)} = \text{Pore Volume} + \frac{\text{Water Volume} \times \text{Salinity NaCl}}{C}$$

Where:

Pore volume is in ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in grams per ml of distilled water.
C = 2.165 g/ml (density of NaCl).

$$\text{Grain Density (corrected)} = \frac{\text{Sample Weight} - (\text{Water Volume} \times \text{Salinity NaCl})}{\text{Bulk Volume} - \text{Pore Volume (corrected)}}$$

Where:

Grain density is in g/ml.
Water volume from distillation extraction is in ml.
Salinity NaCl is in g/ml.
Bulk volume is in ml.
Pore volume is in ml.

4.3.3.6 Advantages/Limitations

Advantages/limitations of this method are:

a. Pressure-retained coring is an expensive, meticulous, and time-consuming operation. It requires a straight and clean hole containing a specially formulated drilling fluid or foam.

b. A laboratory including the necessary special equipment and experienced supervisors should be located as close as possible to the well location since it may be difficult to safely transport a frozen core over great distances.

c. Pressure-retained core analysis provides fluid saturations more indicative of in situ saturation values than those values obtained by conventional core analysis. The ability to prevent fluid expulsion during core retrieval (pressure depletion) prevents saturation alteration during core retrieval. Refer to advantages/limitations for distillation extraction cleaning (see 4.3.1.5 and 4.3.1.6) to understand the impact on saturation determination.

d. Minimizing pore space flushing during coring is another factor that must be considered in order to obtain near in situ saturations. Pressure-retained coring procedures are designed to prevent alteration from depletion, but not from flushing. As a result, core saturations, from cores normally recovered in water base drilling fluids, are indicative of in situ oil saturation values only if filtrate flushing is minimal (see 4.3.7). If a large amount of flushing occurs, then the measured saturations are more indicative of residual values. This is important for the evaluation of tertiary recovery projects.

e. Flushing is minimized by minimizing drilling fluid overbalance during coring, through use of low fluid loss drilling fluid, and by penetrating the formation as rapidly as possible while maintaining good core recovery. In certain situations, i.e., depleted bottom-hole pressures, a stabilized-foam drilling fluid minimizes flushing. However, if the foam breaks down during coring, the core is subjected to a high surfactant fluid loss and appreciable flushing occurs.

f. The ability to capture oil and water in the core provides a distinct advantage over conventional and sponge core analysis. By capturing all water that existed in the core at the completion of coring, it is possible to determine the filtrate volume that displaced gas, oil, or water during coring operations. By knowing the measured saturations and the filtrate amount, saturation alteration during coring is qualitatively assessed by using the formation relative permeability fractional flow characteristics.

In comparison, conventional and sponge cores allow determination of only the residual core filtrate after coring because fluids are expelled from the core during retrieval. However, since most filtrate is located in the core's periphery, the measurement of filtrate is erroneously low. In a conventional core, the oil saturation is also reduced during pressure depletion, although this oil may be retained by the sponge in a sponge core and analyzed. If oil saturations are near residual, the k_g/k_o is such that usually very little oil is expelled by gas drive.

g. Since the gas is retained in the pressure-retained core, it is possible to measure gas volume as well as gas composition from a pressure-retained core. This is important when evaluating sweep efficiencies from gas injection. Residual saturations in highly-swept zones allow assessment of various petrophysical rock properties at residual saturations after gas injection.

h. Gas-oil ratios, when averaged over an interval, have agreed favorably with production measurements.

i. Oil gravity can be determined on a foot-by-foot basis throughout the cored interval to determine if oil gravity changes with depth. Gravity changes must be appreciable to be observed by this method.

j. The diameter of pressure cores is presently 2.5 to 3.75 inches (64.5 to 95.3 millimeters). The smaller diameter core increases the surface area to volume ratio of the sample. The effect of drilling fluid filtrate invasion and saturation alteration is greater than in larger-diameter core. As in any core analysis measurement, the degree of accuracy is decreased as sample size is reduced; therefore small measurement errors usually will have less impact on pressure-retained core analyses as the core diameter is increased.

4.3.3.7 Accuracy/Precision

Accuracy/precision of this method is:

a. Fluid saturations—Accuracy limits of fluid determination from each phase of analysis are as follows:

1. Gas collection: fluid volumes are ±0.5 ml; water volumes (distillation extraction) are ±0.5 ml.
2. Gas driven solvent extraction: oil volume is ±0.1 g or ±0.1 ml.

b. Gas volume—Gas volume is determined by Boyle's Law. Resulting gas volume is ±2% of actual volume. The variables in calculation of gas volumes have an accuracy as follows:

Cell volume = ±5 ml.
Cell temperature = ±0.5°F.
Cell pressure = ±0.2 psia.
Air fraction = ±0.5 mol %.

4.3.3.8 Calibration

Calibration issues for this method include:

a. Receiving tubes on gas collection cells and distillation extraction apparatus should be calibrated using deionized water on a scale reading to 0.01 grams. Weights at each interval are converted to volumes using the water density at the appropriate temperature. Volumes should be determined for each 10 ml reading on the receiving tubes. Interpolation is used to determine true volumes between the calibrated volumes.

b. Gas collection cell volumes should be calibrated using an accurate gasometer or meter. Each cell is evacuated and the volume determined. Repeatability of measured volumes shall be within 5 ml.

c. Balances should be checked at regular intervals for accuracy.

4.3.4 Sponge Core Analysis

4.3.4.1 Principles of Analysis

In sponge coring, the core enters a half-inch thick polyurethane or cellulose/polyurethane sponge sleeve inside an aluminum liner. As the core is brought to the surface, expanding gases displace crude oil that is captured by an oil-wet sponge sleeve (or water that is captured by a water-wet sponge sleeve).

The oil-wet sponge sleeve consists of polyurethane sponge with about 70 percent porosity that is easily compressed to almost no porosity. The sponge is saturated with brine prior to going into the well. If the formation brine properties are known, the brine used to saturate the sponge should match the salinity and density of the water in the formation to be cored. Any oil that comes out of the core will displace water in the sponge and will spread out in a thin layer over the walls of the sponge pores.

The water-wet sponge consists of a cellulose fiber mixed in with the polyurethane. The sponge is saturated with dry mineral oil prior to going into a well cored with an oil-based drilling fluid. The objective is to capture any water escaping the core as it is brought to the surface.

4.3.4.2 Apparatus

4.3.4.2.1 Equipment

High speed table saw with at least a 10-inch (25.4-centimeter) diameter carbide blade and 1.5 horsepower motor to open the aluminum core barrel.

4.3.4.2.1.1 Oil-Wet Sponge Analysis Equipment

The following additional equipment is required for oil-wet sponge analysis.

a. Container to hold sponge (large Soxhlet, metal can, etc.).
b. Spectrometer for determining amount of crude in solution, e.g.:
   1. Visible spectrometer (detects intensity of crude color).
   2. Ultraviolet-fluorescence spectrometer (detects aromatics).
   3. Near-infrared (NIR) spectrometer (detects C-H bonds, asphaltenes).
   4. Nuclear magnetic resonance (NMR) spectrometer (detects the C-H shift).
c. Analytical balance for preparing crude extract standards used in calibrating spectroscopic response.
d. Gas chromatograph (optional).

4.3.4.2.1.2 Water-Wet Sponge Analysis

In addition to 4.3.4.2.l, water-wet sponge analysis requires a standard distillation extraction apparatus (e.g., Dean-Stark) for determining water volume recovered (see 4.3.2).

4.3.4.2.2 Reagents

4.3.4.2.2.1 Oil-Wet Sponge Analysis

Any good crude-oil solvent that has:

a. No visible color, if using visible spectrometer.
b. No aromatics, if using UV-fluorescence spectrometer.
c. Preferably no C-H bonds, if using NIR or NMR (e.g., hydrogen-free chlorofluorocarbons, or chlorocarbons).

4.3.4.2.2.2 Water-Wet Sponge Analysis

Any standard distillation extraction solvent, e.g., toluene.

4.3.4.3 Procedure/Precautions

4.3.4.3.1 Rig Site Handling

At the rig site, care should be taken not to jar the ends of the sponge core liner, as this can cause the rock core to shift in the barrel resulting in a mismatch of the sponge and rock core. Although the sponge core should be kept cool, freezing it with the rock core still inside carries some risk of inducing fractures in the rock core.

For certain formations, such as fractured or vuggy carbonates, core can break and wedge into the sponge causing it to bunch together. The larger-diameter sponge core is less susceptible to this problem.

Immediately after retrieval, the cores may be cut into sections (typically about 5 feet) and stored in transport containers filled with the same fluid used to saturate the sponge.

4.3.4.3.2 Oil-Wet Sponge Analysis

Because the sponge is presaturated with water and is highly compressible, any analysis of the amount of oil in the sponge should measure oil directly, and not infer oil volume from a measurement of the water in the sponge and the porosity (as is done in the distillation extraction method for measuring oil saturations of rocks).

Because the oil in the sponge is spread out in a thin layer over the oil-wet sponge, mechanical compression of the sponge is not a good method for trying to recover the captured oil.

A large-bore Nuclear Magnetic Resonance instrument, such as an NMR imager, can be used to directly determine the amount of oil in the sponge, provided the sponge sleeve is removed from the electrically-conducting aluminum liner. The direct NMR method will become more attractive as the measurement cost declines and NMR availability increases.

As of 1993, the least expensive, most widely applicable method, and the method recommended here, is solvent dissolution of the captured crude followed by a spectroscopic determination of the amount of crude in solution. Since sponge may be subject to attack by solvents during the solvent dissolution step, one must either select solvents that are gentle on sponge or be sure that the subsequent analysis is unaffected by dissolved sponge components mixed in with the dissolved crude. For spectroscopic determination, a calibration curve should be developed by injecting known weights or volumes of lease crude into pieces of sponge, then solvent-extracting the sponge and determining the spectroscopic response of the extracts. Sponge samples should be taken from the actual lot of sponge used in the sponge coring operation. If different lots of sponge are used during coring, a new calibration curve should be developed for each new sponge lot.

4.3.4.3.3 Water-Wet Sponge Analysis

The water-wet sponge is hygroscopic, so care must be taken to prevent absorption of moisture from the atmosphere because this could impact the final analysis for the water saturation. Standard distillation extraction analysis can be performed on water-wet sponge to determine water content.

4.3.4.3.4 Separation of Sponge Sleeve and Aluminum Liner From Rock Core

With a table saw, make two cuts 180 degrees apart down the length (long axis) of the aluminum liner that jackets the sponge sleeve. Set the depth of cut to 0.25 inches (6.35 millimeters) so that the saw blade cuts only through the aluminum liner and not the sponge sleeve. Also, make the cut between a pair of aluminum centralizer vanes (the vanes that hold the rock core centered within the aluminum liner) instead of on top of a vane.

Next, cut through the sponge sleeve with a knife and remove each section of whole core from the liner for trimming. Note any evidence that the core has shifted relative to the liner. Mark the liner so it can be cut to the same length as the corresponding section of trimmed core and label the matching pieces of whole core and liner.

Complete the process of cutting and sealing the rock and sponge samples within 30 minutes to minimize exposure to the atmosphere. This minimizes evaporation of core fluids, oxidation of liquid crude, and the resulting errors in quantifying the amount of crude. If the sponge is not immediately analyzed, carefully preserve it until it is analyzed (see 2.2.6 for preservation methods).

The corrected whole-core oil (or water) saturation is obtained by adding the volume of oil (or water) in each piece of trimmed core to the volume of oil (or water) in the matching piece of sponge sleeve and dividing by the total whole-core pore volume.

4.3.4.3.5 Solvent Extraction and Spectroscopic Analysis (Oil-Wet Sponge)

Analysis of oil-wet sponge is as follows:

a. Prepare sponge samples with known amounts of lease crude and extract sponge with chosen solvent. Calibrate the spectroscopic response using these extracts.

b. Place sponge core crude into solution by any of the following:

1. Soxhlet extraction.
2. Submerged soak and vigorous shaking.
3. Submerged soak and disaggregation of sponge in blender.

For options (2) and (3) it will be necessary to separate the sponge sleeve from the aluminum liner.

4.3.4.4 Calculations

4.3.4.4.1 Oil-Wet Sponge Analysis

Calculations for oil-wet sponge analysis are as follows:

a. Determine the spectroscopic response of an aliquot of the solvent extract.
b. Knowing the total amount of solvent extract and using a calibration performed with prepared standards, determine the total amount of crude that was originally imbibed by sponge.

$$V_{tc} = W_{tc} / \rho_c = (R_a / R_{pwf}) \, (W_{ts} / \rho_c)$$

Where:

R_{pwf} = Response of spectrometer per weight fraction of sponge-imbibed lease crude in the final solvent extract. This response calibration is done using prepared standards consisting of solvent extracts of sponge samples (of the current lot) that have been injected with known amounts of lease crude. (For example, absorbance or fluorescence of solvent extract per weight fraction of sponge-imbibed crude when using a given sample cell at a given wavelength of light.) Units of R_{pwf} must be the same as the units of R_a.

R_a = Response of spectrometer for an aliquot of the solvent extract recovered from a piece of sponge. Response is obtained under the same conditions (e.g., using same sample cell, wavelength, etc.) that were used when calibrating the spectrometer response with prepared standards. Units of R_a must be the same as the units of R_{pwf}.

W_{ts} = Weight of the total crude solution recovered from a piece of sponge core. Weight is measured before the small aliquot is removed, g.

W_{tc} = Total weight of crude that was in a piece of sponge core, g.

V_{tc} = Total volume of crude that was in a piece of sponge core, cm^3.

ρ_c = Density of lease crude, g/cm^3.

4.3.4.4.2 Water-Wet Sponge Analysis

Knowing the brine salinity and density, the volume of brine that was in the sponge can be calculated from the volume of distilled water recovered from the sponge as follows:

$$V_{br} = [(V_w)(\rho_w)/\rho_b]\,[1{,}000{,}000/(1{,}000{,}000 - C_s)]$$

Where:

V_{br} = Volume of brine corresponding to the volume of distilled water collected from a piece of sponge, cm^3.

V_w = Volume of distilled water collected from a piece of sponge (e.g., Dean-Stark), cm^3.

ρ_w = Density of distilled water, g/cm^3.

ρ_b = Density of lease brine having a concentration, C_s, of salt, g/cm^3.

C_s = Concentration of dissolved salts in lease brine = (1,000,000) (weight of salt)/(weight of brine), ppm.

4.3.4.5 Advantages/Limitations

Sponge core is a less expensive alternative to pressure-retained coring (as described in 4.3.3) and is operationally simpler. The amount of oil (or water) captured in the sponge is added to the amount of oil (or water) remaining in the core to obtain more accurate values of oil (or water) saturation.

Unlike pressure core, any hydrocarbon gases that are released are not retained, and so are not available for analysis. Also, with sponge core, a choice must be made between determining corrected values of the core's oil saturation or determining corrected values of the core's water saturation. Both corrected saturations cannot be determined using the same sponge core.

4.3.4.6 Accuracy/Precision

For extraction and spectroscopic analysis combined, the oil volume from oil-wet sponge core analysis is within 5 percent of the actual volume of oil or 0.2 ml of oil, whichever is larger. The water volume from water-wet sponge core analysis is within 0.1 ml of the actual volume of water from which the corresponding volume of brine can be calculated if the brine salinity and density are known. The water volume is determined more accurately than the oil volume because the water is determined by a direct measurement of volume. In contrast, the oil sample is highly diluted in solvent and only an aliquot of the dilute solution is analyzed spectroscopically.

4.3.4.7 Calibration

Refer to 4.3.1.8. Standard calibration procedures should be followed for all commercial equipment.

4.3.5 Productive Shale

4.3.5.1 Principles of Analysis

This paragraph covers the special considerations for measuring fluid saturations of the pore space in "oil productive shale"—rocks composed of clay-to-silt-sized particles and potentially oil productive. Analysis of samples from this type formation may need special handling due to their low permeability, possible presence of tightly bound or structural water, and potential for solid organics. A retort process is not recommended for samples containing solid organics. Oil will be generated from the solid organics at temperatures used in this process yielding high oil volumes. A distillation process should be used to determine fluid saturation data.

The principle is the same as in 4.3. This method should be used if solid organics present are not to be included as oil volume. If shorter analysis times are needed, removal of hydrocarbons can be expedited by crushing the sample. The crushed sample will also expedite equilibrium time for grain volume measurements.

4.3.5.2 Apparatus

The apparatus for distillation extraction is the same as in 4.3.1.2. For sample-crushing, a mortar and pestle are needed.

4.3.5.3 Procedures/Precautions

The procedures are the same as in 4.3.1.3. If samples are to be crushed to expedite hydrocarbon removal or grain volume determinations, then bulk volumes should be measured and samples weighed prior to crushing. Crush sample using mortar and pestle and reweigh. If there is weight loss during crushing, a correction to either rock or fluid volumes must be made. If weight loss is high, the sample should be discarded and another sample tested. Careful control of sample loss throughout the procedure is critical to accurate oil volume measurement.

4.3.5.4 Calculations

Calculations should be performed as stipulated in 4.3.1.4.

4.3.5.5 Advantages

Advantages of this method include:

a. All measurements can be made on the same sample.
b. Solid organics are not represented in oil volumes.
c. Accurate grain densities can be measured.

4.3.5.6 Limitations

Limitations of this method include:

a. Oil volumes are sensitive to drying techniques.
b. Long extraction times are needed due to the low permeability of this type of rock material.
c. Sample loss is critical in determination of oil content.

4.3.5.7 Accuracy/Precision

Proper distillation and drying techniques should permit oil and water content determination to within 2 percent of the values measured. Special care is necessary when drying samples because of the rocks' ability to rehydrate quickly, which will cause erroneously low oil volumes.

4.3.5.8 Calibration

Calibration techniques should be performed as outlined in 4.3.1.8.

4.3.6 Tar (Oil) Sand Analysis

4.3.6.1 Principles of Analysis

The distillation extraction method can be used to determine the fluid saturations of unconsolidated tar (oil) sands. However, certain modifications to the procedure and data presentation are required. The required changes are described in the following paragraphs.

4.3.6.2 Apparatus

The same basic equipment as described in 4.3.1.2 may be used in this procedure.

4.3.6.3 Procedures/Precautions

The unconsolidated core may be frozen prior to obtaining a sample. The plug should be drilled using liquid nitrogen as a lubricant. The frozen plug is then sleeved, usually with heat shrinkable polytetraflouroethylene (PTFE), in order to keep the plug intact when the bitumen is removed. The sample is then placed in the distillation extraction apparatus (see Figures 4-4 and 4-5) with toluene as the solvent. In some instances, the core may not be frozen, or if the sample is obtained during the quarry process of a tar sand deposit, the test sample may be analyzed on a volume/weight basis. In such instances, the test specimen is transferred to an appropriate extraction thimble such as those discussed in 4.3.1.2.(f).

Avoid violent boiling by supplying a minimum heat rate to boil the solvent. Change the toluene, as required, to prevent tar build up and subsequent “bumping” or super heating. This latter condition, should it occur, can slow down the distillation/extraction process or possibly negate the complete test.

If the bitumen has an initial boiling point greater than 392°F (200°C), the oil content can be determined directly. For direct determination, the toluene-bitumen bottoms are transferred into a volumetric flask and toluene is added until the total volume equals the volumetric flask’s volume. An aliquot is taken from the flask and placed on glass fiber filter paper. The toluene is evaporated and the bitumen is weighed. The total bitumen weight is then calculated. This method is known as the Modified Dean-Stark (distillation extraction) Method or AOSI-3573.[4]

4.3.6.4 Calculations

The liquid contents of the sample are usually reported as weight percent as opposed to percent pore volume.

$$\text{Water Content} = [(V_w \times \rho_w) / \text{Sample Weight}] \times 100$$

$$\text{Oil Content} = \frac{[(\text{Sample Weight}) - (V_w \times \rho_w) - \text{Dry Weight}] \times 100}{\text{Sample Weight}}$$

Where:

Water Content = weight percent of the sample weight.
Oil Content = weight percent of the sample weight.
V_w = volume of water recovered, ml.
ρ_w = density of water, g/cc.
Sample Weight = original (net) sample weight, gm; i.e., excludes sleeve weight.
Dry Weight of cleaned and dried sample, g.

Note: For practical purposes the density of the water may be assumed to be 1.00 g/cc.

(For ACOSA Method calculations, see 4.10, Reference 4.)

4.3.6.5 Advantages/Limitations

Advantages/limitations for this method include:

a. The liquid content values are determined on one sample.
b. The method is fairly rapid.
c. The calculations are not complex.
d. If the sample has a high tar (oil) content, it may be necessary to stop the distillation process so that the dirty toluene in the boiling flask can be replaced with clean toluene.

4.3.6.6 Accuracy/Precision

The weight percent values should be reported to the nearest 0.1 percent. Although there are no industry standards, the weight percent values should fall in the range of ±0.5 percent of the calculated value. This level of accuracy should be acceptable for most applications.

4.3.6.7 Calibration

Refer to 4.3.1.8.

4.3.7 Filtrate Invasion Analysis (Pressure Cores)

4.3.7.1 Principles of Analysis

The objective of filtrate invasion analysis is to quantify the amount of drilling fluid filtrate that invades the core during the coring operation. This is accomplished by adding a tracer(s) to the drilling fluid system in known quantities and measuring this tracer(s) content in the core. The degree of this tracer(s) concentration/activity/dilution allows determination of the drilling fluid filtrate amount contained in the core. Knowledge of the drilling fluid filtrate amount in the core allows a qualitative assessment of the saturation alteration that occurs by flushing during coring and calculation of the salinity of the formation water (see 7.7).

Filtrate invasion studies may be planned for other than pressure cores using a variety of water-based and oil-based coring fluids. Procedures for such studies will not be addressed in this publication. The wide range of core types along with a similar broad spectrum of coring fluids and potential tracer materials precludes the recommendation of standard procedures for these other techniques.

Use of relative permeability fractional flow characteristics of the formation enhances the assessment of the fluid saturation alteration. The use of low-invasion profile coring bits minimizes drilling fluid filtrate invasion and saturation changes due to flushing.

The drilling fluid system is tagged with a tracer material. For water-base drilling fluid, tritiated water is recommended and is the basis of the following procedural descriptions. For oil base drilling fluid, tritiated hexadecane has been used.

The drilling fluid system is circulated sufficiently to allow even mixing of the tracer. A "rule of thumb" is to circulate the drilling fluid "bottoms up" three times. Periodically during coring, a drilling fluid sample is taken for use as a base reference for tracer concentration/activity in the drilling fluid for filtrate concentration calculations in the core.

A vertical plug is drilled through selected intervals resulting in "plug" and "donut" samples (see Figure 4-7). Water is extracted from these samples and a determination of the tritium activity is made by liquid scintillation analyses. The decrease in tracer concentration/activity in comparison with the drilling fluid filtrate is equal to the dilution of the drilling fluid filtrate by the formation water. The amount of drilling fluid filtrate invasion can be determined for both the exterior of the core and the interior of the core as well as the total drilling fluid filtrate invasion.

To minimize the extent to which drilling fluid filtrate migrates into the interior of the core by diffusion or imbibition, the samples should be cut at the well site as soon as possible after core retrieval. If this is not feasible, then the inner core barrel should be promptly frozen and preserved. Gidman and Conner, 1992[5] have reported that core freezing causes some fluid migration that may lead to incorrect assessment of the amount of drilling fluid filtrate invasion.

4.3.7.2 Apparatus

The following apparatus are used:

a. Drill press capable of cutting samples using liquid nitrogen as the bit lubricant.
b. Distillation extraction apparatus in 4.3.1.2 for plug analysis.
c. Drilling fluid filter press for extraction of the coring fluid filtrate.

4.3.7.3 Procedures/Precautions

Procedures and precautions include:

a. Use established safety procedures.
b. Designate selected intervals for the filtrate invasion study. Generally a sample is taken approximately every 4 feet (1.22 meters) throughout the cored section.
c. Care should be taken to avoid evaporation of water during handling of the core, plugs, and donuts.
d. The selected full diameter core intervals should be faced into right cylinders approximately 2 to 3 inches (50.8 to 76.2 millimeters) long. A vertical plug is drilled from the center of each sample to provide plug and donut portions of the sample (see Figure 4-7).

There are other methods to determine the spatial extent of drilling fluid filtrate invasion, such as cutting a plug perpendicular to the axis of the whole core. This plug is then sliced into many segments and each segment is analyzed for drilling fluid tracer.

e. The plug and donut samples are weighed and placed into separate Dean-Stark distillation extraction apparatuses to distill the water from the core and to extract the oil. Thimbles should be used to minimize grain loss. The water removed from each sample is measured, bottled, and marked. Since tritiated water has nearly the same chemical and physical properties as water, it is not separated from the ordinary water during the distillation process.
f. The samples are dried in a convection oven at 240°F (117°C) until weights are stable. The dry weight of each sample is recorded.

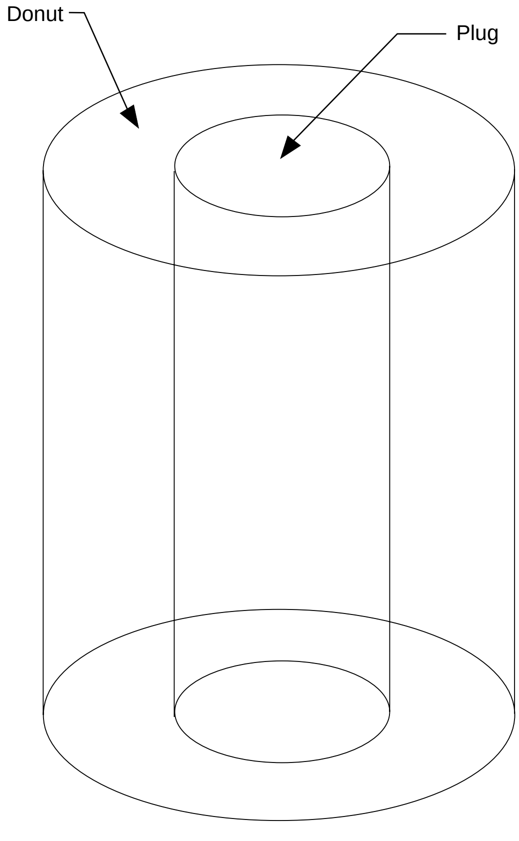

Figure 4-7—Plug and Donut Sample For Filtrate Invasion Study

g. Porosity is determined from measurements on the plug sample. The donut is assumed to have the same porosity as the corresponding plug. The pore volume of the companion donut is determined by the ratio of weight of the plug to the weight of the donut.

h. Water saturation is calculated for the plug and donut samples.

i. The amount of drilling fluid filtrate in the water extracted from each plug and donut sample pair is determined. The ratio of drilling fluid filtrate to total water in each sample is calculated and expressed as a percent of each sample's pore volume.

j. The analyst should observe appropriate safety measures when using tritiated water.

4.3.7.4 Calculations

The following calculations are appropriate for this method:

a. Water saturation in the plug and donut:

$$S_w \text{ (plug)} = [S_{wt} \text{ (plug)}/P_v \text{ (plug)}] \text{ x } 100$$

$$S_w\text{(donut)} = [S_{wt}\text{(donut)}/(P_v\text{(plug)} \text{ x } \text{(donut wt/plug wt)}] \text{ x } 100$$

Where:

S_w (plug) = water saturation of the plug given as a percent of pore volume.
S_w (donut) = water saturation of the donut given as a percent of pore volume.
S_{wt} (plug) = total water volume of the plug, ml.
S_{wt} (donut) = total water volume of the donut, ml.
P_v (plug) = pore volume of the plug, ml.

The plug and donut weights are given in g.

b. Filtrate invasion in the plug and donut:

$$\text{Filtrate (drilling fluid)} = \frac{\text{Tritium Activity (sample)} \times 100}{\text{Tritium Activity (drilling fluid)}}$$

$$\text{Filtrate } (PV) = \text{Filtrate (drilling fluid) x } S_w/100$$

Where:

Filtrate (drilling fluid) = drilling fluid filtrate as a percent of total water.
Filtrate (PV) = pore volume, percent.
S_w = water saturation, percent pore volume.

Tritium activity is in pCi/ml.

4.3.7.5 Advantages/Limitations

Advantages and limitation of this method include:

a. The advantage of determining coring fluid filtrate invasion on pressure-retained core as opposed to either conventional or sponge core is that all the water phase is retained within the core during retrieval since the core barrel pressure is maintained at original coring pressure. Thus, the quantity of drilling fluid filtrate contained in the core segments at the completion of the coring operations prior to surfacing can be quantitatively determined.

Fluids are expelled from conventional or sponge cores as the core barrel is retrieved from downhole to the surface. Since conventional cores contain a larger percent of drilling fluid filtrate in the periphery of the core, this filtrate is the first aqueous fluid to be expelled as the pressure drops during core barrel retrieval. The volume of drilling fluid filtrate is reduced by this fluid expulsion, and the amount of drilling fluid filtrate measured from these types of cores will be only the residual value.

Either a water-retaining or an oil-retaining sponge liner can be selected. The water expelled by gas evolution is recovered with a water-wet sponge and can be analyzed for drilling fluid filtrate tracer. The core fluids can also be analyzed for tracers by Dean-Stark extraction and analysis of the recovered waters.

b. In situ saturation may not be qualitatively determined even with knowledge of the amount of drilling fluid filtrate contained in the core segments. The drilling fluid filtrate displaces both oil and water. The amount of each phase displaced depends on the in situ saturations, fluid properties, and formation properties. If in situ, the formation is at irreducible water saturation, then the filtrate displaces mostly oil or gas. Conversely, if in situ, the formation is near residual oil saturation then mostly water or gas is displaced. Between these two extremes, oil and water displacement depend on the respective relative permeabilities. Relative permeability fractional flow curves should be used in assessment of the fluid saturation alteration during coring operations.

c. Tritiated water is the recommended drilling fluid filtrate tracer because it is can be recovered by Dean-Stark extraction of water from the core material. If ionic tracers are used instead of, or in addition to tritiated water, a selection criterion is that they should not be chemically or biologically altered by the reservoir rock. Anionic tracers used with varying degrees of success in water-based drilling fluid include bromide, iodide, and nitrate salts. Many reservoirs waters contain bromide and iodide in significant amounts, so accurate analysis of the reservoir water composition is important in selecting the appropriate tracer. The nitrate ion tracer requires a drilling fluid bactericide to prevent microbiological reduction from nitrate to nitrite.

Ionic tracers must be recovered from the cores by a method of water extraction, that requires considerably more time and effort than extraction of tritiated water. The procedures are provided in 7.7. Ionic tracers provide semi-quantitative water invasion results.

d. Since the plug and donut samples are placed into the Dean-Stark distillation extraction apparatus in a frozen state, gas is prevented from escaping from the samples. Weight loss from gas release during extraction is included in the weight loss attributed to oil extraction. Oil is determined gravimetrically. If sufficient gas is present, a significant error may be introduced into the oil saturation determination. Thus, only water saturation is reported from filtrate invasion studies.

4.3.7.6 Accuracy/Precision

The precision of the measurements of the extent of drilling fluid filtrate invasion into the core is within about 5 to 20 percent. The simultaneous use of more than one drilling fluid tracer adds confidence in the reported filtrate invasion. There is no information on the accuracy of the method, since there is no accepted reference value for the extent of drilling fluid filtrate invasion downhole.

4.3.7.7 Calibration

Calibration of the liquid scintillation equipment, anionic tracer detection equipment, and ion chromatograph is essential for consistent results.

4.4 SOLVENT FLUSHING

4.4.1 Karl Fischer Titration

4.4.1.1 Principles of Analysis

Core plug samples are cleaned by dynamic miscible displacement with an appropriate sequence of solvents and the water content of the produced effluents analyzed by Karl Fischer titration (refer to ASTM D1364-90[6] and ASTM D4377-88[7]).

4.4.1.2 Apparatus

Apparatus appropriate for this method includes:

a. General—The apparatus includes a core flow system for the solvent injections and a Karl Fischer titrimeter for the effluent analysis. The core flow system is composed of a core holder assembly, solvent delivery device, and related hardware. The Karl Fischer analysis requires a titrimeter, appropriate Karl Fischer reagents, analytical balance, and syringes.

b. Core holder assembly—Hassler or hydrostatic core holder, sleeves, end-pieces, and a source of pressure appropriate for the expected confining and flow pressures.

c. Solvent delivery device—A constant rate high pressure liquid chromatographic (HPLC) pump or a gas-driven transfer system may be utilized. The pump may be a syringe or a continuous-flow type system. The constant pressure method requires a high pressure gas bottle, regulator, and a vessel such as a floating piston accumulator.

d. Related hardware—Consists of corrosion-resistant tubing, valves, and fittings to connect the core holder and solvent delivery system.

e. Karl Fischer titrimeter—Volumetric or coulomatic titrimeter.

f. Analytical balances—Covering range from 0.1 milligram to 1000 g.

g. Syringes—0.1–10 ml capacity, plastic or glass.

h. Materials and reagents:

    1. Karl Fischer reagents: Utilize reagents appropriate for choice of titrimeter.
    2. Toluene, reagent grade.
    3. Methanol, Karl Fischer grade.
    4. Glass vials, 25 ml capacity with caps lined with polytetrafluoroethylene (PTFE).
    5. Glassware, stoppered vessels of approximately 1000 ml capacity, 10 ml receiving tubes.
    6. Silver nitrate, 0.1 Normal.
    7. Check solution, 0.1 weight percent water in methanol standard.

4.4.1.3 Procedures/Precautions

4.4.1.3.1 Procedures

This method is suitable for determining water saturation in whole core and plug-sized samples and can be used as an alternative to the distillation extraction (Dean-Stark) method.

a. The sample is weighed to the nearest 0.1 gram (assuming a plug-size sample), and loaded into the core holder. The desired confining stress is applied and any produced fluids are collected in a receiving tube and the volumes are recorded.

b. An alternating methanol-toluene solvent injection sequence is performed, beginning with the solvent that is miscible with the mobile phase, i.e., methanol if the brine is mobile or toluene if the oil is mobile.

c. All storage vials, syringes, or collection vessels should be weighed or tared prior to use.

d. A glass vial is filled with the initial injectant reagent and sealed. The solvent is then injected into the core sample at rates or pressures suitable for the core material, and the effluent is collected in a stoppered vessel. Care should be taken to minimize exposure of the solvent to atmospheric humidity.

    1. For methanol injection, the effluent is sampled periodically and tested for the presence of salts using silver nitrate. Injection is complete when a drop of silver nitrate fails to form precipitates in the effluent aliquot.
    2. For toluene, injection is continued until (a) the effluent appears clear, or (b) refractive index of the effluent equals the injectant.

3. The Karl Fischer titration is performed as soon as possible to minimize changes in water content due to absorption of moisture. (refer to Steps g–i).

e. Step d is repeated using the alternating solvent.

f. Step d is repeated using the initial solvent.

g. The storage vials and effluent vessels are reweighed and the weights of the solvents calculated.

h. Using a syringe, a portion of the injectant solution is analyzed for water content by Karl Fischer titration according to the manufacturers' recommended procedures. The syringe is reweighed to ascertain the exact weight of solvent analyzed. Making sure the effluent sample is uniform, water content is determined as described above. Results are reported in weight percent or ppm equivalent.

i. The sample is unloaded, dried using appropriate techniques, weighed, and the pore volume determined by helium injection.

4.4.1.3.2 Precautions

Precautions for Karl Fischer titration include:

a. Material safety data sheets should be consulted for proper handling procedures for all required chemicals, including the Karl Fischer reagents.

b. Methanol is highly susceptible to water absorption from atmospheric humidity as well as from untreated glassware. Since it is extremely difficult to quantify these variables, exposure to air should be minimized, and the Karl Fischer titrations performed as quickly as possible.

c. The Karl Fischer titration requires only a small amount of solvent; therefore, the analyzed sample must be representative of the entire solution to yield accurate results.

4.4.1.4 Calculations

The following calculations should be performed:

a. Water Saturation:

$$S_{wes} = W_{es} \times (S_{we} - S_{wi})$$

$$S_{ws} = \text{Sum } S_{wes}$$

$$S_{wb1} = S_{ws} / [(1 - A) \times \rho_b]$$

$$S_{wb2} = (S_{wb1} \times 100)/PV$$

Where:

ρ_b = density of brine, g/ml.
S_{wes} = water content of effluent solvent, g.
W_{es} = weight of effluent solvent, g.
S_{we} = effluent water content, g/g.
S_{wi} = injectant water content, g/g.
S_{ws} = sample water content, g.
Sum S_{wes} = summation of the water contents of the effluent solvents, g.
S_{wb1} = sample brine content, ml.
S_{wb2} = sample brine saturation, percent of the pore volume.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

b. Oil Saturation:

If the sample was fully saturated, oil saturation may be determined by volumetric difference:

$$S_o = 100 - S_{wb2}$$

If the sample contained an initial gas saturation, the oil saturation must be determined by gravimetric difference:

$$W_o = W_i - W_d - [S_{ws} / (1 - A)]$$

$$V_o = W_o / \rho_o$$

$$S_o = \frac{V_o \times 100}{PV}$$

Where:

S_o = oil saturation as a percent of the pore volume (*PV*).
S_{wb2} = sample brine saturation, percent of the pore volume.
W_o = weight of oil, g.
W_i = initial weight of the sample, g.
W_d = weight of the dry sample, g.
S_{ws} = sample water content, g.
V_o = volume of oil, ml.
ρ_o = density of oil, g/ml.
A = salt content of the brine, grams of salt per gram of brine.
PV = pore volume of the sample, ml.

c. Data reporting—Fluid saturations are reported to the nearest 0.1 percent pore space.

4.4.1.5 Advantages

Advantages of this method include:

a. All saturation levels can be determined.

b. The Karl Fischer titration is very precise.

c. Damage to sensitive minerals is minimized.

d. The procedure removes salts from the sample.

e. Saturations can be determined in special core tests in which stress hysteresis effects prohibit unloading the sample.

4.4.1.6 Limitations

Limitations of this method include:

a. Methanol readily absorbs moisture from the environment.

b. Accuracy of the method is dependent on solvent handling and storage techniques.

c. The analyzed sample must be representative of the bulk effluent.

d. The technique is more complex and expensive than the distillation extraction method.

e. Oil saturations are determined indirectly. Oil saturation by gravimetric difference assumes no grain loss of the sample.

f. This technique is not appropriate for samples that contain halite, sulfur, or other minerals that are soluble in methanol.

4.4.1.7 Accuracy/Precision

Precision of automated Karl Fischer titration units is ±0.5 percent of the measured value. However, the accuracy of the technique is limited to the ability to handle and store the solvents without altering the water content.

4.4.1.8 Calibration

Instrument operation is checked by analyzing standard solutions of known water content. Spiked samples should yield percent recoveries between 90 and 110 percent. Duplicate analyses are performed on 10 percent of the total samples analyzed and should have a relative percent difference less than 2 percent.

4.5 SCANNING METHODS

4.5.1 Introduction

Several laboratory techniques for scanning measurements of water, oil, and gas saturations in core samples have been reported. These techniques include: (a) linear x-ray absorption, (b) microwave absorption, (c) computer assisted (axial) tomography (CT), (d) linear gamma-ray absorption, and (e) nuclear magnetic resonance (NMR). The linear x-ray, gamma-ray, microwave, and CT methods may be considered as emerging technologies for saturation measurement. Other scanning techniques that have been used for saturation determinations are radio wave resonance and neutron attenuation radiography. The neutron radiography has a good potential for determination of fluid saturation because neutrons are attenuated much more by fluids (proton) than by rocks. Thus, neutron tomography may not require any addition of tagging agents to the resident fluids for saturation determination. These techniques are not widely used for core analysis.

4.5.2 Principles

The x-ray, CT, and gamma-ray techniques measure the absorption of high energy electromagnetic radiation by fluids that have been tagged with high absorption agents (usually high atomic weight elements) to determine fluid saturations. The microwave absorption technique is based on the absorption of microwave energy by water molecules. NMR is based on the detection of the proton (H1), carbon (C13), sodium (Na23), phosphorous (P31), and fluorine (F19) containing fluids by magnetic fields alternating at radio frequencies while the sample is located in a large constant magnetic field.

4.5.3 Apparatus

Apparatus for these techniques are generally sophisticated and expensive. X-ray and CT equipment manufactured for medical use are often adequate for saturation measurements. Medical NMR equipment is not often suitable for saturation measurements. Microwave and gamma-ray absorption apparatus have usually been specially built.

4.5.4 Procedures/Precautions

Several parameters must be considered for accurate saturation determination when scanning methods are used, such as the power level setting, tagging material and its concentration, tagging fluid-rock and fluid-fluid interaction (refer to the Bibliography for the many procedures and precautions for the individual techniques).

4.5.5 Calculations

Because all techniques (except NMR) generate signals from both the solid matrix and the fluids, measurements at a minimum of two known saturations (normally 0 and 100 percent) are required to establish a calibration line. The unknown saturations for the oil-brine system are then calculated based on the following equation:

$$S_w = (U_{sat} - U_o)/(U_w - U_o)$$

Where:

S_w = unknown brine saturation, as a fraction.
U_{sat} = attenuation of core at the unknown saturation (S_w).
U_w = attenuation of core at 100% brine saturation.
U_o = attenuation of core at 100% oil saturation.
(The units of U_{sat}, U_w, and U_o must all be the same.)

4.5.6 Advantages

An important advantage of these methods is their ability to provide information on the spatial distribution of the fluid saturation. Another advantage of these techniques is that the measurements are made noninvasively and nondestructively. Extraction of fluids is not required prior to flow tests on preserved cores (that can be difficult in full-sized or tight cores). Saturations can be monitored without terminating a laboratory experiment.

An advantage of CT and NMR imaging techniques is that they provide the saturation distribution of fluids in all three dimensions.

An advantage of the microwave and NMR techniques is their capability to determine water saturation in the core without addition of any tagging agents. The microwave and NMR techniques can be used for saturation determination in standard core analyses because they do not require any tagging agents and calibration curves are established after core cleaning and resaturation processes.

4.5.7 Limitations

A limitation of the x-ray, CT, and gamma-ray techniques is that they can determine saturations in cores only if they are saturated with tagged fluids. Therefore, they may not be suitable for the basic core analyses.

A limitation of the NMR technique is the inability to handle cores containing significant ferromagnetic materials, clays, or gas.

4.5.8 Accuracy/Precision

The accuracy and capability of some of the scanning techniques for saturation measurement are listed in Table 4-2. The accuracies listed in this table represent values achievable only under optimum calibration and measurement methodology.

4.5.9 Calibration

The absorption techniques require measurements of signal intensity at a minimum of two saturation levels to establish a calibration curve to relate instrument response to saturations. Calibration is instrument dependent.

4.6 COAL ANALYSIS

4.6.1 Principles of Analysis

Basic core analysis techniques, such as retort analysis and solvent distillation extraction, are not suitable for the determination of fluid saturations of coal samples. Alternative methods have been developed to measure the fluid saturation of coal. With coal, the fluid of primary concern is water. Water saturation, or more appropriately, moisture content is a fundamental property of coal that must be accurately determined to properly evaluate a coal seam. The method described here for determining the moisture content of coal involves drying water from a crushed sample in a convection oven and taking successive weight measurements until equilibrium is achieved. The weight loss is equivalent to the amount of water driven off, and moisture content is reported as the weight percentage of moisture with respect to the weight of the wet coal sample.

4.6.2 Apparatus

The following apparatus are appropriate for this method:

a. Crusher—Use a jaw, cone, or rotary crusher to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve.

b. Drying pans—The pans used should be of sufficient size to accommodate crushed samples spread to a thickness of less than 2.5 centimeters. The sides of the pans should not be greater than 3.8 centimeters in height. The pans should be made of a material that is stable and will not corrode at the temperature used.

c. Drying oven—A forced-draft oven that is capable of maintaining a constant temperature of 225°F, ±5°F (107°C, ±3°C) should be used. The velocity of forced air in the oven should be sufficiently low so as not to disturb the coal sample. An oven with a gas port capable of accommodating a hose from a nitrogen tank is desirable, because flowing nitrogen, rather than air, through the oven can help minimize oxidation of the coal sample.

d. Balance—The balance used to weigh the samples should be sensitive to 0.1 gram and have a capacity large enough to accommodate both the sample and the pan.

e. No. 8 Sieve (2.33 millimeter)—The sieve should be large enough to hold the entire prepared sample while sifting out the larger particles.

f. Glass desiccator—The desiccator should be large enough to hold the prepared sample and sample pan. It is recommended that either no desiccant or fresh color-coded desiccant be used because some desiccants actually give off moisture if not fresh.

4.6.3 Procedures/Precautions

4.6.3.1 Sampling

4.6.3.1.1 Crushing

Use a jaw crusher or other suitable type of crusher or instrument to reduce the coal to the size retained on a No. 8 (2.38 millimeter) sieve. At least 95 percent of the crushed sample should pass through a No. 8 sieve.

Table 4-2—Measurement Capabilities of Scanning Saturation Techniques

Technique	Dimensions	Tagging requirements	Accuracy, saturation units	Spatial resolution, cm	Maximum sample size, in.	Measured fluids
Linear x-ray	2	yes	1	1.0	12	oil, water, gas
Computer Assisted Tomography	3	yes	1	0.2	12	oil, water, gas
Gamma-ray	2	yes	3	0.1	12	oil, water, gas
Microwave	2	no	1	2.0	2	water
Nuclear Magnetic Resonance	3	no	1	0.1	4	oil, water

4.6.3.1.2 Sample Size

The sample used for moisture content determination should have a minimum weight of about 500 grams.

4.6.3.2 Weighing

4.6.3.2.1 Drying Pan

Immediately after sample preparation is complete measure and record the weight of the clean and dry drying pan. Pour the crushed, sifted coal into the drying pan and spread to a thickness of not more than 2.5 cm.

4.6.3.2.2 Sample Initial Weight

Measure and record the weight of the wet coal sample and the drying pan. Place the pan in the oven at a temperature of 225°F, ±5°F (107°C, ±3°C).

4.6.3.2.3 Dry Weight Measurements

After the sample has dried for a period of 1 hour, remove it from the oven and place it in a glass desiccator to cool. The use of desiccant is not recommended. Weigh the sample immediately after cooling and record the weight. Return the sample to the oven. Repeat this weighing procedure at half-hour intervals until the weight loss between successive measurements is less than 0.05 percent of the sample weight. The final measurement taken is considered the dry weight of the sample plus the weight of the drying pan.

4.6.4 Precaution

Do not over-dry the coal sample. Oxidation of the coal occurs if it dries too long in air.

4.6.5 Calculations

Calculate moisture content using the following equation:

$$M = [(W_2 - W_3)/(W_2 - W_1)] \times 100$$

Where:

M = moisture content of coal sample,%.
W_1 = weight of empty pan, g.
W_2 = weight of wet coal plus pan, g.
W_3 = weight of dry coal plus pan, g.

4.6.6 Advantages

The principle advantage of this method is its simplicity. The procedure does not require a large capital investment in specialized equipment. Neither does it require specialized training to perform the measurements. More sophisticated techniques are available to determine the moisture content of coal (refer to 4.10, References 8, 9, and 10). However, in the context of basic core analysis, the technique described here is adequate.

4.6.7 Limitations

Some types of coals have a tendency to oxidize using the technique described here. Moisture content results will be too low if oxidation occurs; oxidation adds weight and will cause the dry weight to be too high. It is recommended that drying times be kept within the guidelines outlined here to minimize the possibility of oxidation. Forcing nitrogen, rather than air, through the oven may also help to reduce the potential for oxidation.

4.6.8 Accuracy

The accuracy of this technique is very good because it depends only upon the accuracy of the balance. Thus, the accuracy for this technique is ± 0.05 percent.

4.6.9 Precision

Results of duplicate moisture content determinations performed on the same sample by the same technician using the same equipment should not differ by more than ±0.3 percent. Results of moisture content measurements performed by different laboratories on adjacent samples should not differ by more than ±0.5 percent.

4.6.10 Calibration

Use of a calibration weight set is recommended to ensure that the balance works properly. Periodic calibration of the balance at regular intervals should also be performed by a trained technician according to factory specifications to ensure measurement accuracy.

4.7 OIL SHALES

4.7.1 Principles of Analysis

Oil shale is defined as rock composed of clay to silt size particles containing various quantities of solid organic material (e.g., kerogen). The solid organics present in these samples are typically liquefied to generate oil. Analysis of this type of formation, with low permeability and potential for solid organics, requires special procedures.

Oil and water saturations are obtained by a high temperature retorting process. No attempt is made to measure gas-filled pore volume. The fluid saturations are reported in gallons per ton.

4.7.2 Apparatus

The apparatus is the same as used for the Retort Method at Atmospheric Pressure (see 4.2.1.2). The fluids are collected in 15 cm^3 centrifuge tubes. Typically, gas is evolved during the retorting process. Should it be desirable to collect/measure these gases, modified glassware may be used consisting of a calibrated liquid receiving section with the upper portion of the glassware fitted with a sidearm to allow produced gases to be channeled to collection cells or measuring equipment.

4.7.3 Procedures/Precautions

The procedure is similar to the procedures described in 4.2.1.3, with the following exceptions:

a. The representative sample, approximately two inches in length, is not broken in half. Permeability is a not a consideration in oil shale analysis.

b. No attempt is made to screen the crushed sample to exclude fines created during the crushing process.

c. An adjacent sample is not prepared for the mercury pump tests described in 4.2.1.3, because neither the gas-filled pore volume nor the porosity are required.

d. Oil coking and cracking losses are ignored. If significant, a correction for mechanical oil hold-up should be made.

e. The oven is allowed to heat at a rate consistent with the heating elements and number of retorts in place, i.e., no attempt is made to measure pore water at an initial oven temperature setting. The oven temperature is set initially at 1000°F (538°C). When the oven has stabilized at this temperature and all collection tube volumes remain constant, the retorting process is considered complete.

f. Under certain conditions (e.g., samples containing different varieties of kerogen) the volume of oil recovered may vary with the rate of heating.

g. Only the total water collected is used for the final calculation of water content.

h. Retorting operations should be carried out with adequate ventilation (e.g., under fume hoods), as typical oils recovered from kerogens are high in sulfur content and give off noxious vapors.

4.7.4 Calculations

The oil and water contents are calculated as follows:

$$\text{Oil Content} = \frac{\text{Volume of Oil Recovered} \times 239.7}{\text{Sample Weight}}$$

$$\text{Water Content} = \frac{\text{Volume of Water Recovered} \times 239.7}{\text{Sample Weight}}$$

Where:

Oil volume is in milliliters.

Water volume is in milliliters.

Sample weight is in grams.

Oil content is in gallons/ton.

Water content is in gallons/ton.

4.7.5 Advantages

Advantages of this method include:

a. Large volumes of sample can be used.
b. The method is fairly rapid.
c. All measurements are made directly and independently of others.
d. Standard retorting equipment and procedures can be used.
e. Care is not required to differentiate pore water from total water.
f. Oil volume correction curves are not required.
g. Duplicate samples are more easily obtained since a large volume of crushed material may be prepared, and samples obtained, by the “cone and quarter” technique.

4.7.6 Limitations

Limitations of this method include:

a. Oil yields may be dependent on rate of heating.
b. Due to emulsions, there may be difficulty in reading the meniscus in the collection glassware.

4.7.7 Accuracy/Precision

Accuracy/precision of this method is:

a. The accuracy of the oil and water volumes is ±2.5 percent of the measured volumes.
b. The reproducibility should fall in the ±0.5 gal/ton range for low yield samples (e.g., 3 to 10 gal/ton) and ±1.0 gal/ton in high yield samples (e.g., 20 to 40 gal/ton).

4.7.8 Calibration

For glassware calibration procedures, refer to 4.2.1.8.

4.8 GYPSUM-BEARING CORES

4.8.1 Principles of Analysis

Cores containing gypsum ($CaSO_4.2H_2O$) often are from carbonate formations. Such formations are frequently heterogeneous with vugs and/or fractures so that full-diameter core analysis procedures are preferred. However, determination of fluid saturations of gypsum-bearing cores by the distillation extraction method (Dean-Stark) for full diameter cores (see 4.3.2) using toluene as the solvent is not recommended. At the boiling point of toluene, gypsum dehydrates. If the core contains a significant amount of gypsum, erroneously large water volumes and incorrect pore volumes will be measured. The pore volume used to determine saturation will also be in error because of the increase in pore volume due to the dehydration of the gypsum. The volume of oil extracted from the sample is not affected by the presence of gypsum.

There are emerging technologies, e.g., NMR that show promise for the quantification of gypsum and in the future may be offered as basic services by commercial laboratories. The Karl Fischer titration method (see 4.4.1) can be used to analyze gypsum-bearing cores by determining water content of flushed solvents during cool solvent extraction. However, this method is generally not available at service companies for analysis of a large number of core samples. The qualitative method described in the following paragraph can be performed by commercial laboratories.

The qualitative method presented here is based on adjustment of retort saturations for the dehydration of the gypsum by comparing retort and low temperature porosities and weights on adjacent samples. A plug sample is cleaned by pressure cycling solvents at low temperature so that oil and water are extracted from the sample without removing the chemically-bound water held within the gypsum. Boyle's law porosity is then measured on the cleaned sample. After the Boyle's law porosity is determined, the plug sample is retorted to determine the amount of bound water in the sample. The difference in porosity between the low and high temperature extractions is used to calculate the bulk volume of gypsum contained in the core sample. The volume of water given off in the dehydration of gypsum is approximately 1.27 times the increase in pore volume that results from shrinkage of gypsum (grain density = 2.32 g/cm^3) as it converts to anhydrite (2.89 to 2.98 g/cm^3). Retort saturation measurements are obtained on trimmed ends of the plug sample or on material adjacent to where the plug was drilled. The retort method at atmospheric pressure (see 4.2) removes the fluids in the core, and water of crystallization, from the gypsum. The summation-of-fluids porosity is determined from the retort data. Water saturations determined by the retort method are then adjusted. The adjustment will result in a decrease in water saturation. This adjustment is qualitative because carbonates are noted for being heterogeneous; so the companion samples may not contain the same amount of gypsum.

4.8.2 Apparatus

Appropriate apparatus for this method include:

a. High temperature retorting equipment (see 4.2.1.2).

b. Low temperature cleaning apparatus, e.g., pressure saturator large enough to pressure cycle a solvent through several samples at one time.

c. Boyle's law porosimeter.

4.8.3 Procedures

The core to be analyzed should be laid out on a table and fitted together as in standard practice (see 3.2.4). From each foot of core to be analyzed, drill a plug sample. If 1-inch diameter plugs are used, the samples should be at least one inch long. After the plugs are trimmed to length, they should be weighed. Check fluorescence of the plug and record. The initial visual estimate of hydrocarbon saturation will allow the analyst to better evaluate the sample during the pressure-cleaning phase. The plug should be cleaned by a low temperature method that minimizes gypsum alteration.

The fluid saturation of the trimmed plug ends and/or the remaining material from the segment of core from which the above mentioned plug has been drilled, is determined by the retort method at atmospheric pressure (see 4.2.1.3). Use the maximum amount of core material possible for the best precision. This will yield high temperature fluid saturation data and the summation-of-fluids porosity may be calculated (see 5.3.2.2.2 and 4.2.1).

The drilled plug samples should be cleaned in a pressure vessel using an azeotrope mixture of $^1/_3$ acetone, $^1/_3$ cyclohexane, and $^1/_3$ methanol. The samples are placed in the pressure vessel and totally immersed in the azeotropic mixture. Air or nitrogen pressure is applied to the top of the mixture forcing the azeotrope into the pore spaces of the plug samples. The pressure is cycled to force the mixture in and out of the plugs. After approximately 24 hours, the fluorescence of the samples should be checked to see if extraction is complete. Plugs with oil fluorescence should be returned to the pressure vessel for another cleaning cycle. If there is no visible oil fluorescence, the samples are deemed clean. The samples are placed in a vacuum oven at 120°F (49°C) until weight stabilizes. Samples should stay in the vacuum oven for at least 24 hours to remove the azeotrope mixture from the samples. Low temperatures are used during the cleaning and drying phases of this process to minimize alteration of gypsum in the plug samples. After the plug samples have been cleaned and dried, Boyle's law porosity is determined.

After the Boyle's law porosity is measured on the plug sample, the plug is retorted to remove the water bound in the gypsum. After retorting, the plug is reweighed and the porosity of the plug sample is remeasured by Boyle's law. Comparison of the retort porosity with the low temperature porosity identifies zones with gypsum. When the high temperature porosity is much greater than the low temperature porosity, the gypsum content is very high. When the two porosities agree, the gypsum content is low.

The oil volume extracted by the retort method is not affected by the presence of gypsum. However, the pore volume used to calculate the oil saturation must be corrected for the presence of gypsum. The oil saturation can be calculated from the corrected pore volume and the oil volume.

4.8.4 Precautions

Care should be exercised drying samples taken from the pressure saturator to prevent dehydration of any gypsum present. Monitor vacuum oven heat with a temperature probe.

4.8.5 Calculations

Refer to Retort Method at Atmospheric Pressure (see 4.2) and Summation-of-Fluids Porosity Calculations (see

5.3.2.2.2). Refer to 5.3.2.1.1, "Boyle's Law Porosity Calculations".

$$S_{oc} = V_o / (V_{br} \times \phi_{lt})$$

Where:

V_o = volume of oil obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

V_{br} = bulk volume of the retorted material, cm^3.

ϕ_{lt} = porosity of the plug after low temperature extraction, fraction.

S_{oc} = adjusted oil saturation, fraction.

If the gas saturation is assumed to be zero, then the water saturation, S_{wc}, is:

$$S_{wc} = 1 - S_{oc}$$

If the gas saturation is non-zero, the water saturation can be calculated by assuming a grain density for anhydrite:

$$\Delta V_w = (\rho_a / \rho_g) \times (V_{br} \times \Delta\phi)$$

$$\Delta_{wcorr} = V_w - \Delta V_w$$

$$S_{wc} = V_{wcorr} / (V_{br} \times \phi_{lt})$$

Where:

Swc = water saturation adjusted for the gypsum dehydration, fraction.

ΔV_w = volume of water from the dehydration of the gypsum, cm^3.

ρ_a = grain density of anhydrite, g/cm^3.

ρ_g = grain density of gypsum, g/cm^3.

$\Delta\phi$ = difference in porosity between the high and low temperature extractions of the plug sample, fraction.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

V_w = Volume of water obtained from the retorting of the trimmed ends or material adjacent to the plug sample, cm^3.

Alternatively, the saturations can be expressed in terms of sample weights:

$$f_{wat} = W_{wat} / (W_{wat} + W_{oil})$$

$$W_{ewat} = (W_{po} - W_{pr}) \times f_{wat}$$

$$W_{eoil} = (W_{po} - W_{pr}) \times (1 - f_{wat})$$

$$W_{gyp} = W_{pl} - W_{pr}$$

$$V_{wcorr} = (W_{ewat} / \rho_{br}) - (W_{gyp} / \rho_{gw})$$

$$S_{oc} = (W_{eoil} / \rho_{oil}) / V_{plt}$$

$$S_{wc} = V_{wcorr} / V_{plt}$$

Where:

W_{po} = weight of the plug after drilling, g.

W_{pl} = weight of the plug following low temperature extraction, g.

W_{pr} = weight of the plug following retorting, g.

W_{wat} = weight of water determined by the retort extraction of the end pieces or adjacent material, g.

W_{oil} = weight of the oil determined by the retort extraction of the end pieces or adjacent material, g.

f_{wat} = weight fraction of water removed by retort extraction, fraction.

W_{ewat} = estimated water content of the plug sample including the bound gypsum water, g.

W_{eoil} = estimated oil content of the plug sample, g.

W_{gyp} = gypsum water of hydration in the plug sample, g.

ρ_{br} = density of the formation brine, g/cm^3.

ρ_{gw} = density of the gypsum bound water, g/cm^3.

V_{wcorr} = volume of water adjusted for the gypsum dehydration, cm^3.

ρ_{oil} = density of the formation oil, g/cm^3.

V_{plt} = pore volume of the plug sample following low temperature cleaning, cm^3.

S_{oc} = oil saturation calculated for the plug sample accounting for the change in pore volume due to the dehydration of the gypsum, fraction.

S_{wc} = water saturation calculated for the plug accounting for the bound gypsum water and the change in pore volume due to the dehydration of the gypsum, fraction.

4.8.6 Advantages

Advantages of this method include:

a. Damage to gypsum is minimized when the sample is cleaned by pressure cycling. Pore volume measurements used to calculate saturations are more representative if the gypsum is not damaged.

b. Gypsum content can be estimated by porosity comparisons.

4.8.7 Limitations

Limitations of this method include:

a. The low temperature cleaning process can be time consuming depending on the oil gravity and the permeability of the core material.

b. The retort oil and water saturation values are not as precisely determined as for samples that contain no gypsum.

4.8.8 Accuracy/Precision

Accuracy/precision of this method is:

a. If the water of crystallization is preserved during pressure cycling, pore volume determination will have the standard accuracy.

b. The precision of oil and water saturation calculations determined by retort method will be reduced due to the estimation of gypsum content by porosity comparisons.

c. Porosities obtained by the Boyle's Law helium method are more reliable than those obtained by the summation-of-fluids method. When porosity values obtained by Boyle's Law and summation-of-fluids methods are in good agreement, the saturations are assumed to be more reliable than in intervals in which there are large differences between the two porosities.

4.8.9 Calibration

The equipment used to measure porosity by both methods must conform with standards for the appropriate porosity method.

4.9 HISTORICAL

4.9.1 Vacuum Retort Method

The vacuum retort method for obtaining core fluid saturations is a full-diameter technique. This method is used extensively in "hard rock" regions, e.g., West Texas, where production is predominately from carbonate formations. Although several core analysis companies still maintain equipment for this procedure it is not a recommended procedure, having been replaced by the distillation extraction method (see 4.3).

The procedure consists of distilling off the pore fluids at a maximum temperature of 450°F (232°C) while maintaining a partial vacuum on the system. The fluid collection glassware should be immersed in an alcohol/dry ice bath maintained at approximately –75°F (–59°C) to prevent the vapors from being lost through the vacuum pump.

The procedure lends itself to the determination of the pore volume by the summation-of-fluids method. This is possible because the first step consists of saturating the unoccupied or gas-filled pore spaces with fresh water. The unoccupied pore volume is determined by gravimetric difference. An oil volume correction factor is employed and the pore water is determined by difference. The three values comprise the pore volume and the collected fluids can be expressed accordingly. A porosity can be calculated with a bulk volume determined on the same sample used for the distillation process.

One of the prime disadvantages of this method is the condition of the cores after the distillation process. It is not uncommon for the cores to be black from coked oil and no cleaning process will restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures, e.g., a Boyle's Law porosity, capillary pressure test, relative permeability, etc. For this and other reasons, the authoring Subcommittee decided not to record this as a recommended procedure.

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SECTION 5—POROSITY DETERMINATION

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5 Porosity Determination

5.1 GENERAL

5.1.1 Introductory Comments/Equations

5.1.1.1 Porosity, which is defined as a ratio of the void-space volume to the bulk volume of the material, is an intrinsic property of all reservoir rocks. The amount of void space which can be occupied by hydrocarbons or water in a reservoir must be known for an intelligent estimate of the initial oil/gas in place. The precision with which porosity can be determined is largely a function of the methods used in these measurements. Several logging tools using either electrical, nuclear, density, or sonic methods are used for continuous porosity determination in the wellbore. The measurements obtained from these well logging tools should be calibrated against core porosities determined under simulated subsurface conditions, bearing in mind the volume of rock investigated by logging tools. Effects of core sample bias, sample frequency and sample volume must be taken into consideration. With one exception, all discussions refer to porosity determination at zero to <400 psi (2760 kPa) confining stress. The method to determine porosity at simulated overburden pressure is discussed in 5.3.2.2.1.2.

5.1.1.2 Unless otherwise stated, the analysis methods described are applicable to cylindrical core plugs drilled from consolidated and relatively homogeneous rock. Porosity from core analysis may commonly be arrived at by several different methods (see Table 5-1). Measurement of bulk volume (*BV*) and grain volume (*GV*) yields pore volume (*PV*) by difference, and porosity (ϕ) by:

$$PV = (BV - GV) \tag{1}$$

$$\phi = (BV - GV) / BV$$

Direct measurement of pore volume (*PV*) and grain volume (*GV*) yields:

$$\phi = PV / (PV + GV) \tag{2}$$

Direct measurement of pore volume (*PV*) and bulk volume (*BV*) yields:

$$\phi = PV / BV \tag{3}$$

5.1.1.3 In the laboratory, usually one of two types of porosity are measured; viz., effective porosity or total porosity. There is a difference between the historical definitions of these porosities and the current definition used by log analysts. This is further complicated by core analysis techniques designed to leave several molecular layers of clay- or mineral-bound water within the pore space (see Figure 5-1).

5.1.1.4 Historically, effective porosity has been defined as a measure of the connected voids. It is derived from either the difference between bulk volume and apparent grain volume determinations, or by a direct measurement of the connected void space. The measured volume of the connected void space may vary with sample preparation and the analytical method used. Total porosity is a measure of the total void space, both connected and isolated, in the rock sample. This can be determined by bulk volume, dry weight, and grain volume measurement on a disaggregated sample.

5.1.1.5 In the field of log interpretation, effective porosity has been defined as interconnected pore volume occupied by free fluids. In this definition, effective porosity does not include the volume of clay-bound or mineral-bound water, and in some analytical approaches, does not include the additional water held within the shale by capillary forces. Also in the field of log interpretation, total porosity is that volume occupied by all fluid (connected and isolated pore space) in the rock, including the volume occupied by the clay-bound water (see Figure 5-1).

5.1.1.6 Core analysis experience indicates that for most reservoir rocks there are few isolated pores, and hence there is very little or no measurable difference in historically defined effective and total porosity (see Figure 5-1). Thus, with these definitions, effective porosity determined from core analyses on a totally dried sample at 210° to 240°F (99° to 116°C) more closely corresponds to the total porosity as defined by the log analysts. In certain circumstances, such as volcanic rocks, there can be a measurable difference between the effective (connected) and total (connected and isolated) porosity from core analysis. In addition, core humidity drying techniques can be tailored to leave some amount of bound water on mineral surfaces.

5.1.1.7 There is no universal agreement between log analysts on the definition of effective porosity (see Figure 5-1). The lowest value for this parameter is obtained if all shale-associated water (adsorbed/anion free water plus capillary-held water within the shale) is excluded as noneffective pore space. A higher value is obtained if only the adsorbed/anion-free water (defined as a function of both salinity and cation exchange capacity or the concentration of clay counterions per unit pore volume, meq/cm^3) is excluded. Core porosities based upon humidity-controlled, dried samples are normally between this higher value and the total porosity. At this time, it is difficult to select laboratory conditions that yield effective porosity on core samples as defined by log analysts.

5.1.1.8 Another facet of porosity measurement, which in fact applies to the entire field of core analysis, is the selection of conventional (small sample) analysis versus full-diameter analysis. The decision as to which type should be used would ideally be based entirely on the homogeneity of the formation being analyzed. Many sandstones are sufficiently homogeneous so that a small sample can be considered representative of the analysis increment. On the other hand, when the formation is heterogeneous as to pore structure or lithology, such as in vugular or fractured carbonates, full-diameter core techniques are more appropriate. Therefore, the size of the sample required to adequately represent the pore structure and lithology of the core material, as well as the sampling strategy should control the type of analysis used.

5.1.2 Poorly Consolidated/Unconsolidated Core Samples

5.1.2.1 Poorly consolidated/unconsolidated samples present unique requirements. The term "poorly consolidated" encompasses a wide range of core materials, from friable samples to completely unconsolidated samples with no apparent cementation between grains. For the purposes of this discussion, "poorly consolidated" refers to any plug sample that requires mounting in an appropriate jacketing material to withstand the cleaning, preparation, and measurement process.

5.1.2.2 Plug samples are commonly jacketed using a cylinder of metal or polymer about the circumference of the sample. Table 5-2 lists some common jacketing material along with their advantages and disadvantages.

Table 5-1—Bulk, Grain, and Pore Volume Directory

Method Number	Title	Purpose
5.2.1	Archimedes (Buoyancy) Mercury Immersion	Bulk volume measurement
5.2.2	Mercury Displacement	Bulk volume measurement
5.2.3	Caliper	Bulk volume measurement
5.2.4	Archimedes (Buoyancy) with Fluids Other than Mercury	Bulk volume measurement
5.2.5	Bulk Volume by Summation of Grain and Pore Volume	Bulk volume determination
5.3.1	Total Pore Volume from Disaggregated Sample Grain Density	Grain volume measurement
5.3.2.1.1	Boyle's Law Double-Cell Method	Grain volume measurement
5.3.2.2.1	Boyle's Law Single-Cell Method	Direct pore volume measurement
5.3.2.2.2	Summation of Fluids Porosity	Summary bulk and pore volume measurements
5.3.2.2.3	Liquid Saturation Method	Direct pore volume measurement
5.4	Historical Procedures	Bulk, grain and pore volume measurements
5.5	Organic Rich Rocks	Pore volume and adsorbed gas

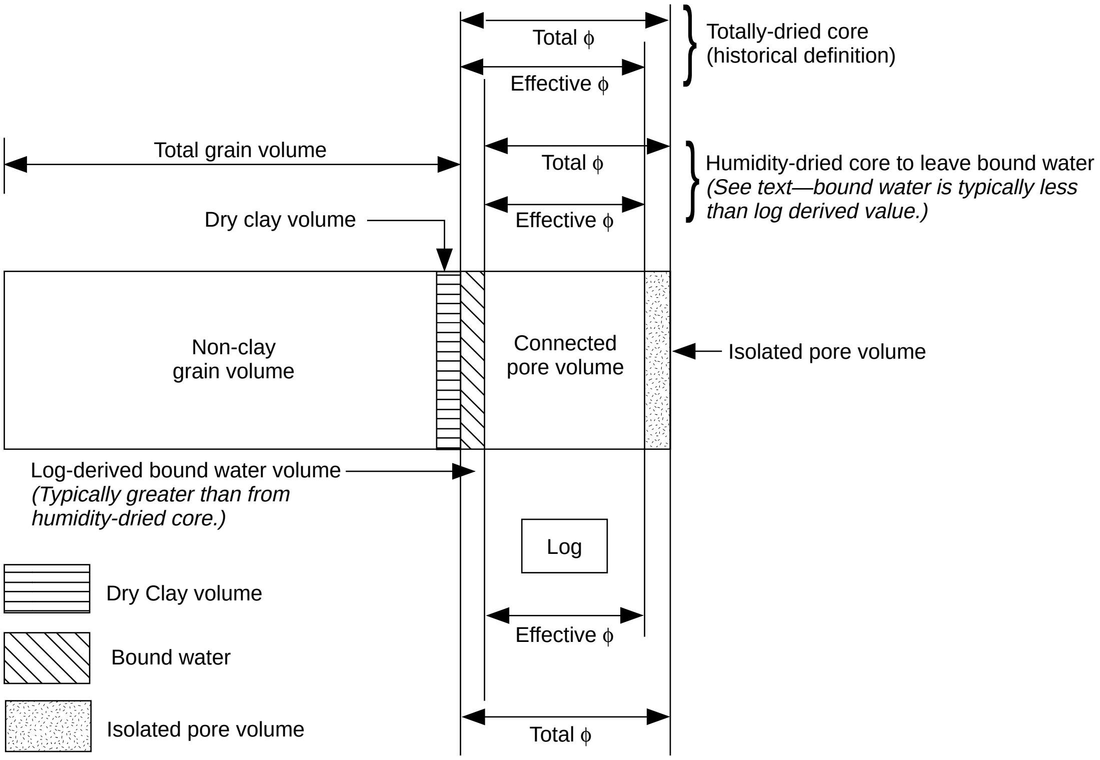


Figure 5-1—Total and Effective Pore Volumes as Defined by Core and Log Analysts

Table 5-2—Jacketing Material for Poorly Consolidated/ Unconsolidated Core Samples

Jacketing Material	Advantages	Disadvantages
Lead Sleeves	Malleable, conforms well to sample	Interacts with mercury and some brines
Teflon® tape	Inert	Difficult to apply, porous and permeable
Heat shrink Teflon tubing	Inert, easy to apply	May not conform well to sample at low confining stress, possible sample alteration from heat and radial stress applied as tubing shrinks
Aluminum sleeve	Malleable, conforms reasonably well to samples	Interacts with some brines and may not conform well at low confining stress
Paint-on epoxy or other coating	Cheap, easy to apply	Interacts with solvents, poor mechanical strength, can imbibe into sample
Tin	Malleable, conforms well to sample	Interacts with mercury and some brines

5.1.2.3 To allow flow, the plug faces are covered with screens or other porous material. The mesh size of the screens must be small enough to prevent grain loss, but large enough to avoid plugging by mobile fines. Screens with a mesh size of 200 or 120 are typically used. Samples with some consolidation require only a single screen. Less consolidated samples may require more sophisticated end treatments. For instance, two screens may be used. One screen is of finer mesh to prevent grain loss, and a second less flexible, coarser screen is added for mechanical strength. The material chosen for the screens must be inert to the solvents and brines used for cleaning and testing. A high quality stainless steel such as 316 or Monel is recommended.

5.1.2.4 The apparent pore and grain volume of the jacketing material and end screens must be determined and accounted for in the porosity measurement. The volume of jacketing material on an individual sample can be determined by applying the Boyle's Law Double-Cell Method for grain volume (see 5.3.2.1.1) or the Archimedes method for bulk volume using either mercury (where suitable, see 5.2.1) or other solvents (see 5.2.4). In practice, it is easier to determine the density of the jacketing material and then determine the weight of jacketing material applied to each sample. Jacket volume can then be computed. This method assumes that the density of the material is constant from piece to piece.

5.1.2.5 The "grain" and "pore" volume of the screens must be determined as well. The grain volume can be determined by taking a number of identical screens and measuring their solid volume in a Boyle's Law Double-Cell apparatus. The value determined is then divided by the number of screens tested and this average value is applied to each sample. The pore volume is more sensitive to the test method applied. For the Boyle's Law method, the pore or void volume in the screens can be determined by mounting a solid non-porous plug in the same manner as the real samples are prepared. The Boyle's Law Single-Cell method will then yield the screens' void volume.

5.1.2.6 Once a sample is packaged with a jacketing material and screens, it can be tested using many of the same techniques applied to consolidated samples. In the following sections, comments about the applicability of each measurement method for jacketed poorly consolidated samples will be made in 5.2.1.3.

5.1.2.7 Since the physical properties of poorly consolidated samples are more sensitive to confining stress than consolidated samples, measurements performed at stresses below in-situ effective reservoir stress will not be as representative of the reservoir rock. Therefore, the recommended method for determining the porosity of poorly consolidated samples is to use the Boyle's Law Single Cell method for direct pore volume measurement at elevated confining stress (see 5.3.2.2.1.2), and the Boyle's Law Double-Cell method for grain volume (see 5.3.2.1.1).

5.2 BULK VOLUME MEASUREMENT (PLUG SIZE)

The bulk volume of a rock sample is required to determine the porosity of that sample. Bulk volume of a core plug can be determined by several methods. Measurement techniques include Archimedes immersion, mercury displacement, and calipering.

Bulk volumes can also be calculated by summing direct measurements of grain volume and pore volume.

The bulk volume of a sample selected for porosity measurement should preferably be at least 10 cm^3. Normally, samples are a right cylinder with diameters from 2.54 cm to 3.81 cm and lengths of at least 2.54 cm and 3.81 cm, respectively. Irregular shapes may be used with proper precautions if samples of regular dimensions cannot be obtained.

5.2.1 Archimedes (Buoyancy) Mercury Immersion

5.2.1.1 Principle

A core plug is immersed in mercury and the volume of mercury displaced by the sample is determined gravimetrically (Archimedes' principle).

5.2.1.2 Advantages

Advantages of this method include:

a. Samples can be used for subsequent tests if no mercury penetration occurs.
b. The method is very accurate if careful technique is used and precise measurements are made.

5.2.1.3 Limitations

Limitations of this method include:

a. Trapping air around the samples will create errors and yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing. Samples with a vugular surface or containing open fractures are not recommended for bulk volume analysis by mercury immersion. However, if it is necessary to analyze such samples using this technique, the sample surface must be sealed by coating or filling the vugs to prevent mercury penetration. This may not be an adequate treatment.

1. Materials such as heat-shrink Teflon tubing can be used for coating the cylindrical surfaces of a sample to cover the vugs. The volume of any coating must be subtracted from the total bulk volume measured, and air gaps on nonvugular surfaces must not exist between the tubing and the rock.
2. The surface vugs can be filled (e.g., with modeling clay, paraffin, or an epoxy) which will allow a direct measurement of the bulk volume when the sample is immersed under the mercury. Usually, filling the vugs is a destructive test and should be the last step in the analysis.
3. Calipering sample length and diameter, and computing bulk volume (see 5.2.3), is a method utilized for vuggy samples. Experience has shown that computed bulk volumes are often too high. Calibration techniques presented in 5.2.3.6 improve data accuracy.

c. The method is unsuitable for poorly consolidated/unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.1.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. Bulk volume measurements can be repeated to within ±0.01 cm^3.

5.2.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Electronic, single-pan, analytical balance accurate to ±0.01 gm.

b. Mercury vessel large enough that plug positioned with the large dimension horizontal can be fully immersed without touching the sides of the container.

c. Mercury to fill the vessel so that the plugs can be fully immersed.

d. Adjustable, pronged fork with reference mark.

e. Thermometer.

5.2.1.6 Procedure

Bulk volume (BV) is measured by the Archimedes Mercury Immersion method with the apparatus shown in Figure 5-2. A cup of mercury is placed on a single-pan electronic balance, and a pronged fork is immersed to the fork reference mark. The mark is located such that the top of the core plug will be submersed 3 to 7 millimeters under the mercury. The balance is then tared. The fork is withdrawn from the mercury, and the plug to be measured is floated lengthwise on the mercury surface. The plug is then forced into the mercury with the fork to the same reference mark. The sample must not touch the side of the mercury vessel. The resulting weight represents the mass of the mercury displaced. Eighteen-gauge

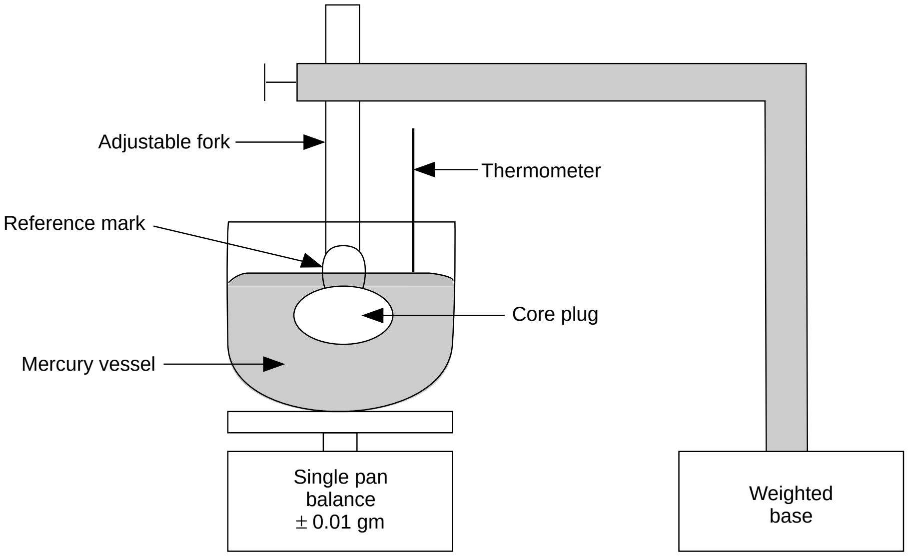


Figure 5-2—Archimedes Mercury Immersion Apparatus

(1.0-millimeter diameter) stainless steel wire is a suitable material for the fork. Heavier gauge wire has been observed to result in unstable weight measurements (changing weights with time) and long balance equilibrium times.

5.2.1.7 Calculations

The bulk volume is calculated using the following equation:

$$BV = \frac{\text{Mass of mercury displaced}}{\begin{array}{c}\text{Density of mercury at}\\ \text{measurement temperature}\end{array}} \qquad (5)$$

5.2.1.8 Precautions

A heavy weighted base for supporting the fork assembly is essential to hold the sample firmly in place to ensure accurate weights. It is important to use the correct mercury density for the mercury temperature. A temperature variation of 5°C will induce a systematic error of 0.02 percent in the bulk volume (see Table 5-3).

Table 5-3—Density of Mercury Versus Temperature[a]

°C	Hg Density gm/cm^3	°C	Hg Density gm/cm^3
18.0	13.5512	27.0	13.5291
19.0	13.5487	28.0	13.5266
20.0	13.5462	29.0	13.5242
21.0	13.5438	30.0	13.5217
22.0	13.5413	31.0	13.5193
23.0	13.5389	32.0	13.5168
24.0	13.5364	33.0	13.5144
25.0	13.5340	34.0	13.5119
26.0	13.5315		

[a] From *Handbook of Chemistry and Physics*, 71st Edition, 1990-1991, Editor-in-Chief: David R. Lide, Ph.D., CRC Press, Boca Raton, Florida.

In order to obtain stable weights, the fork should be of such design as to yield point contact on the sample. If the fork volume is too large relative to the plug volume, erroneous weights will be measured. Unstable weights may indicate an unsuitable fork or mercury invasion. Mercury contamination can be confirmed by comparing sample weights before and after mercury immersion.

5.2.2 Mercury Displacement (Volumetric Displacement Pump)

5.2.2.1 Principle

Sample bulk volume is measured by mercury displacement utilizing a volumetric displacement pump to which a stainless-steel sample chamber is attached.

5.2.2.2 Advantages

Advantages of this method include:

a. This procedure allows very rapid measurements.

b. The technique is employed as part of the Summation of Fluids porosity measurement.

c. Samples are suitable for subsequent tests if no mercury penetration or adsorption occurs.

5.2.2.3 Limitations

Limitations of this method include:

a. Air trapped around the sample will yield erroneously high bulk volumes.

b. Vuggy samples or extremely high permeability samples will be penetrated by the mercury resulting in low bulk volume values and rendering the samples unsuitable for further testing (see 5.2.3).

c. In most mercury displacement pumps, the sample is immersed approximately 50 millimeters beneath the mercury. This mercury height induces a pressure of about 1 psi (6.9 kPa) to the top of the plug. This may make the bulk volumes systematically low because of 1) conformance to microscopic surface roughness or 2) penetration into large pores. The depth of immersion is minimized (3 to 7 millimeters) in the Archimedes Mercury Immersion method (see 5.2.1).

d. The method is unsuitable for unconsolidated samples that are mounted in lead sleeves because of lead-mercury amalgamation. Mercury may also be trapped between jacketing materials and the sample; consequently, this is not a recommended procedure for jacketed samples.

5.2.2.4 Accuracy

The measurement can be reproduced to 0.01 cm^3 if the pump has been calibrated and is zeroed for each sample.

5.2.2.5 Apparatus

Figure 5-3 illustrates a high pressure, volumetric mercury displacement pump. The sample chamber will hold samples up to approximately 25 cm^3 in volume.

5.2.2.6 Procedure/Calculations

The bulk volume of a regularly or irregularly shaped core sample is obtained by mercury displacement. The chamber, with no sample in place, is filled with mercury to the reference level. The instrument volume read-out is zeroed. The mercury level is then lowered, the sample is inserted and the chamber is again filled to the reference level. The instrument volume read-out is obtained. The difference between this reading and the instrument zero is the bulk volume of the sample.

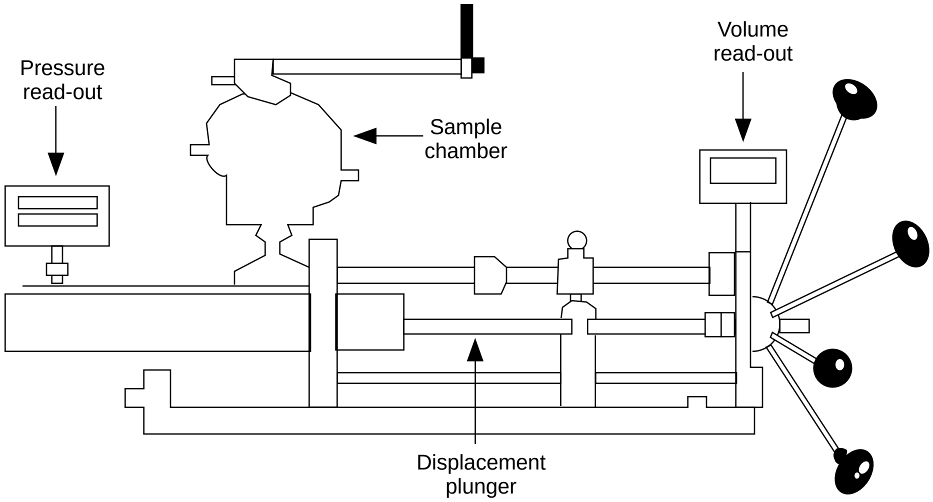


Figure 5-3—Volumetric Mercury Displacement Pump

5.2.2.7 Precautions

The mercury and chamber should be clean and free from surface films, solids, and oils.

5.2.2.8 Calibration

The mercury pump is calibrated with steel billets of known volume.

5.2.3 Caliper

5.2.3.1 Principle

Samples which are right cylinders or other regular shapes may be calipered to obtain bulk volume. A micrometer or vernier caliper, which can be read to the nearest 0.002 cm, can be used to measure length and diameter. A minimum of five measurements is recommended.

Note: Samples with surface vugs and/or open fractures may be analyzed using this approach if bulk volume data are calibrated on samples using the technique discussed in 5.2.3.6.

5.2.3.2 Advantages

Advantages of this method include:

a. The sample may be used for other tests.
b. The procedure is rapid.

5.2.3.3 Limitations

Limitations of this method include:

a. Samples with uneven shapes cannot be measured by this method.
b. Irregularities in sample surfaces may yield erroneously high bulk volumes.
c. Calipered bulk volumes are not generally recommended for porosity methods where $PV = (BV - GV)$. The accuracy of the bulk volume is not as critical where $f = PV / BV$ and the pore volume is determined by direct measurement (see 5.3.2.2). Where rock type (vuggy or fractured) dictates the use of the caliper technique, data should be calibrated as discussed in 5.2.3.6.
d. This method is not preferred for jacketed, poorly consolidated, or unconsolidated samples. If employed, corrections for screen and jacket thickness must be applied to both calipered length and diameter.

5.2.3.4 Accuracy

Length and diameter measurements of actual samples have been shown to be reproducible with a 99 percent confidence level (within 3 standard deviations) to within 0.15 millimeters for length and 0.04 millimeters for diameter. Bulk volumes can be repeated to 0.15 cm^3. See 5.2.3.6 for accuracy improvement.

5.2.3.5 Apparatus

Vernier or digital calipers may be utilized.

5.2.3.6 Procedure

The length and diameter of a cylinder or dimensions of a regularly shaped sample are measured at a minimum of five different positions to define any irregularities in the shape. Small deviations in the shape may be averaged out. Several samples without surface vugs and/or open fractures, whose gross dimensions cover the range of samples to be analyzed, should have their bulk volume determined by an appropriate immersion technique. Their bulk volume should then be determined using the exact caliper technique that will be used for measurement of subsequent samples. Correction factors may then be determined to calibrate calipered bulk volumes to those measured by immersion. These factors are then applied to subsequent calipered bulk volume measurements to correct them to an equivalent immersion bulk volume.

Less time is required and more readings are taken if digital calipers are used. Compared to vernier calipers, the measurements are not as susceptible to human error since the readings are displayed as digital output. Digital calipers can be interfaced with a computer so the readings will be automatically recorded. About 10 length or diameter readings can be taken in less than 60 seconds.

5.2.3.7 Calculations

The cross-sectional area of the cylindrical sample is calculated from the average diameter and multiplied by the average length to obtain bulk volume. Appropriate formula can be used for noncylindrical samples.

5.2.3.8 Precautions

Precautions for this method include:

a. Diameter measurements should be taken at a minimum of five positions evenly spaced along sample length.
b. Length measurements should be obtained at a minimum of five positions around the sample's periphery.
c. Ensure that calipers are zeroed between successive measurements.
d. When measuring diameters and lengths of samples with surface irregularities, ensure that calipers do not intrude into surface vugs or fractures yielding measurements less than the true dimensions.

5.2.4 Archimedes (Buoyancy) With Fluids Other Than Mercury (e.g., Brine, Refined Oil, or Toluene)

5.2.4.1 Principle

A body placed in a liquid is buoyed up by a force equal to the weight of the displaced liquid.

5.2.4.2 Advantages

Advantages of this method include:

a. Accurate values can be attained if proper technique is used.
b. The sample can be totally saturated with liquid for other tests which may be desired.
c. If the sample is 100-percent saturated with a single fluid prior to the measurement of bulk volume, the pore volume, grain volume, and grain density can be calculated from recorded weights.

5.2.4.3 Limitations

Limitations of this method include:

a. The liquid may be unsuitable for subsequent tests and have to be removed.
b. Cores containing vugs should not be measured by this method.
c. Liquids which may leach the sample or cause swelling of the matrix cannot be used.
d. Vugs or large pores in extremely high permeability samples will be filled while submerged under the liquid, resulting in low bulk volume values (see 5.2.2.3, b.). Filling the vugs prior to measurement will allow a direct measurement of the bulk volume when the submerged weight is taken.
e. This is not a preferred technique for jacketed, poorly consolidated/unconsolidated samples because of the possibility of trapping extraneous fluid volume between the plug surface and the jacket.

5.2.4.4 Accuracy

The measurement of weight can be repeated within ±0.015 grams if air is not trapped when the sample is submerged and the temperature remains constant. The accuracy of bulk volume measurements will vary depending upon the density and volatility of the liquid used in the measurements. Good technique should yield repeatable bulk volume within 0.01 cm^3.

5.2.4.5 Apparatus

An analytical balance accurate to one milligram, fine wire cradle, liquid container, and thermometer are required. Figure 5-4 shows two possible experimental configurations.

5.2.4.6 Procedure

5.2.4.6.1 A sample is saturated with a liquid of known density, such as a nondamaging brine, light refined oil or a high boiling point solvent. The sample can be essentially 100-percent saturated with liquid by evacuating the pore space, introducing the saturant, and following with pressure saturation (see 5.3.2.2.3.6).

5.2.4.6.2 Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure that fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are acceptable methods for removing excess liquid.

5.2.4.6.3 A beaker is filled with the saturating liquid. A fine wire (maximum diameter of 1.0 millimeter) cradle attached to the stirrup of a balance is first submerged under the liquid to a reference mark, and the balance is tared. The sample is then placed on the cradle, submerged to the reference mark, and a submerged weight of the sample is obtained (see Figure 5-4A).

5.2.4.6.4 Figure 5-4B illustrates an alternate procedure. A beaker is filled with the saturating liquid and placed on a balance. A fine wire cradle is then lowered into the liquid to the reference mark, and the balance is tared. The saturated sample is then placed on the cradle, submerged to the reference mark, and the immersed weight of the sample is obtained.

5.2.4.6.5 A check on the pore volume can be made by taking (a) the difference in the weight of the 100-percent saturated sample in air and the dry weight, and (b) dividing by the saturating liquid density.

Note: It is not essential for the sample to be 100-percent saturated with the liquid for the determination of the bulk volume. The technique provides an accurate bulk volume as long as the sample is not imbibing liquid when the weight is taken while suspended under the liquid. However, if the sample is not 100-percent saturated, the pore volume, grain volume, and grain density calculated from the weights will be incorrect.

5.2.4.7 Calculations

Bulk volume from the apparatus shown in Figure 5-4A equals the initial weight of the saturated sample (or partially saturated sample with stabilized weight) in air minus the weight when submerged, divided by the density of the immersion liquid. Bulk volume from the apparatus shown in Figure 5-4B equals the immersed weight of the saturated sample divided by the density of the immersion fluid.

A B

$$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Fluid}} \qquad BV = \frac{\text{Immersed Weight}}{\text{Density of Immersion Fluid}} \tag{6}$$

5.2.4.8 Precautions

Precautions for this method include:

a. Care must be taken in the design of the apparatus. The arrangement should be such that both the samples and the wire cradle are totally immersed during measurement, are not supported in any way by the immersion container, and only a length of straight wire (maximum diameter of 1.0 millimeter) penetrates the surface of the liquid. The wire and fluid should be clean.

b. It is essential to use the correct fluid density for the temperature of the liquid at the time of the submerged weight measurement. The density of the fluid should be known to at least the nearest 0.005 gm/cm^3. An error of 0.005 gm/cm^3 results in a porosity bias of 0.5 porosity points (porosity expressed as percent) and a grain density bias of 0.01 gm/cm^3. The density of the fluid can be measured with a pycnometer, an electronic digital density meter, various specific gravity balances, or it can be calculated using a silicon standard available from the National Institute of Standards and Technology.

$$V_{SS} = \frac{W_{SS}}{\rho_{SS}} \tag{7}$$

Where:

V_{SS} = silicon standard volume.
W_{SS} = air weight of silicon standard.
ρ_{SS} = silicon standard density from National Institute of Standards and Technology.

$$\rho_F = \frac{W_{SS} - W_{ISS}}{\rho_{SS}} \tag{8}$$

Where:

ρ_F = density of fluid in which silicon is immersed.
W_{ISS} = immersed weight of silicon standard.

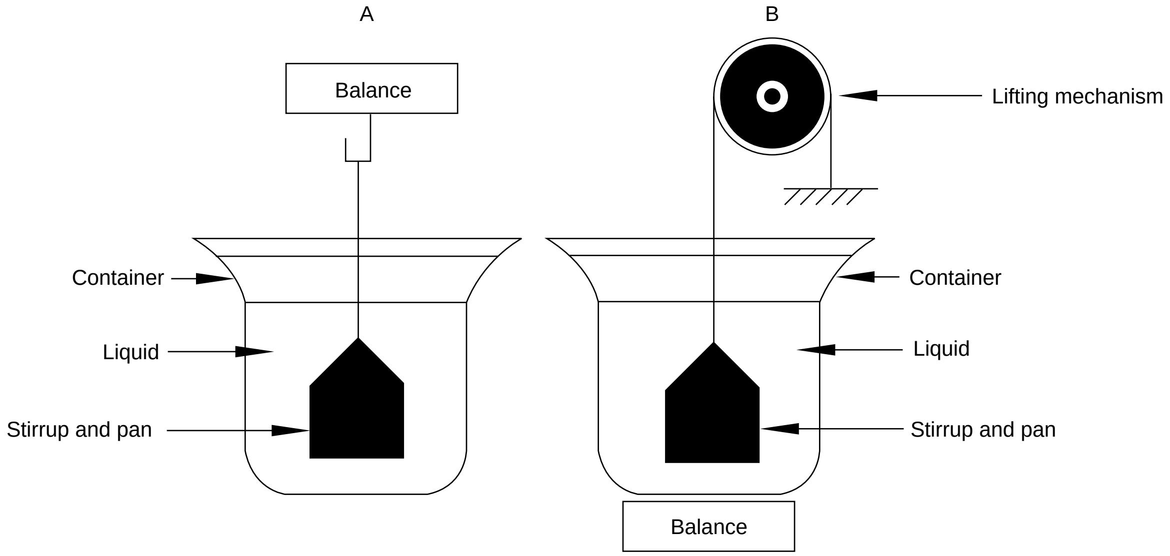


Figure 5-4—Archimedes (Buoyancy) With Fluids Other Than Mercury—Apparatus

c. It is probable that unstable decreasing weights indicate that fluid is imbibing into the sample, which will result in an erroneously low bulk volume.
d. Temperature fluctuations should be minimized as immersion liquid density will vary.
e. Care should be used in wiping the surfaces of the saturated sample so as to avoid grain loss or excess saturant removal. See 5.2.4.6.2 for suitable techniques.

5.2.5 Bulk Volume Calculated By Summing Direct Measurements of Grain Volume (5.3.2.1.1) and Pore Volume (5.3.2.2.1)

5.2.5.1 Calculations

The basic calculation is:

$$BV = GV + PV \tag{9}$$

5.3 PORE VOLUME MEASUREMENTS

Total pore volume is the total void space, both connected and isolated, in a rock sample.

5.3.1 Total Pore Volume From Disaggregated Sample Grain Density

The total pore volume equals the difference between the sample bulk volume and the disaggregated grain volume. Disaggregation is done to expose any isolated pore volume.

5.3.1.1 Dry Method for Grain Density/Grain Volume

5.3.1.1.1 Principle

The bulk volume of a cleaned sample is first determined (see 5.2). The sample is then dried, weighed, and disaggregated. After disaggregation, a weighed portion is placed into a Boyle's Law porosimeter to determine the grain volume (see 5.3.2.1.1). The grain volume of the total sample is calculated using the ratio of the dry weight of the consolidated sample to the dry weight of the disaggregated sample placed in the porosimeter.

5.3.1.1.2 Advantages

The advantages of this method are:

a. Fast.
b. Sample can be used for supplemental measurements requiring disaggregated sample. See Section 7, Supplementary Tests.

5.3.1.1.3 Limitations

The limitations of this method are:

a. Drying required.
b. Not viable for gypsum-bearing rock.
c. Not viable for rocks containing minerals such as halite that are soluble in the cleaning fluid.
d. Technique assumes any isolated pore volume remains isolated during the time the sample is dried.
e. If water fills the isolated pore space and is not removed while drying the sample, the calculated grain volume (GV) will be erroneously high.

5.3.1.1.4 Accuracy

Technique is capable of supplying grain density within ±0.01 g/cm^3. No comparative porosity data are available, but technique is expected to supply porosity within ±0.4 porosity units, or better.

5.3.1.1.5 Apparatus

The following apparatus are appropriate for this method:

a. Pulverizer with adjustable tolerance between the grinding plates.
b. U.S. Bureau of Standards 60-mesh sieve.
c. Analytical balance, with accuracy to 0.1 milligram.
d. Boyle's Law porosimeter modified to handle powdered samples. (1) Sample Cup cover (to keep the powdered sample in the cup).

5.3.1.1.6 Procedure

The procedure for this method is as follows:

a. Sample Preparation.
    1. Clean.
    2. Dry at 225°F (107°C).
    3. Cool in Desiccator.
    4. Weigh the sample and record weight (W).
    5. Repeat the above (steps 2 through 4) until weight (W) has stabilized.
    6. Determine Bulk Volume (BV) (see 5.2).
b. Crush the sample and pass the crushed sample through a 60-mesh sieve.
c. Dry a portion of the sample for Grain Volume measurement at 210° to 240°F (99° to 116°C) to a constant weight (W_d).
d. Determine the Grain Volume (GV_d) of the disaggregated sample portion by the Boyle's Law Double-Cell method (see 5.3.2.1.1).
    1. Calibrate the porosimeter with the sample cup cover in place.
    2. Place a known weight (W_d) of crushed and dried sample into the sample cup.
    3. Measure the Grain Volume of the disaggregated and dried sample portion with the cover in place.

5.3.1.1.7 Calculations

The calculations for this method are as follows:

$$PV = BV - GV \tag{10}$$

Where:

PV = total pore volume.
BV = bulk volume of original consolidated sample.
GV = grain volume of sample calculated by equation b.

$$GV = GV_d \times W/W_d \tag{11}$$

Where:

GV_d = measured grain volume of disaggregated sample portion placed in porosimeter.
W = original sample weight.
W_d = weight of dry, disaggregated sample portion placed in porosimeter cup.

$$GD = W_d/GV_d \tag{12}$$

Where:

GD = grain density of sample portion.

5.3.1.1.8 Precautions

Precautions for this method include:

a. The portion of rock selected for grain density measurement must be representative of the total sample.
b. See 5.3.2.1.1.8, Precautions for Boyle's Law Double-Cell (matrix cup) method for grain volume, items a through f.

5.3.1.1.9 Calibration

See 5.3.2.1.1.9, Calibration for Boyle's Law Double Cell (matrix cup) method for grain volume.

5.3.2 Effective Pore Volume of Aggregated Samples

Effective pore volume can be computed by subtracting the measured grain volume from the sample bulk volume, or by direct measurement of sample void volume.

5.3.2.1 Grain Volume Measurement

5.3.2.1.1 Boyle's Law Double-Cell (Matrix Cup) Method for Grain Volume

5.3.2.1.1.1 Principle

Boyle's Law: When the temperature remains constant, the volume of a given mass of ideal gas varies inversely with its absolute pressure.

$$\frac{V_1}{V_2} = \frac{P_2}{P_1} \quad \text{or} \quad P_1V_1 = P_2V_2 \tag{13}$$

Extension of the equation to account for temperature variation and nonideal gas behavior is required for accurate grain volume determination.

$$\frac{P_1V_1}{z_1T_1} = \frac{P_1V_1}{z_2T_2} \tag{14}$$

This latter equation should be utilized with a double-cell device when determining sample grain volume (GV). Gas is admitted into a reference cell of known volume (V_r) at a predetermined reference pressure (100 to 200 psig). The reference cell gas is then vented into a connected chamber of known volume containing a core sample. This results in a lower equilibrium pressure, from which GV is calculated. The GV is subsequently subtracted from bulk volume to yield pore volume and thence porosity.

5.3.2.1.1.2 Advantages

The advantages of this method include:

a. The test sample is not damaged in any way and can be used for other measurements.
b. The operation is quick, simple, and has excellent repeatability.
c. Irregular-shaped samples and vuggy cores can be tested.

5.3.2.1.1.3 Limitations

The limitations of this method include:

a. For good accuracy, extremely careful and frequent calibration is required.
b. Changes in either temperature or barometric pressure must be accounted for in the calculations.
c. The resultant porosity value will be higher than the true porosity value if gas adsorbs on the core surfaces. The use of helium gas minimizes this possibility.
d. This method is suitable for poorly consolidated/unconsolidated jacketed cores if proper techniques are employed. The volume of the jacketing material and end screens must be accurately determined and subtracted from the measured apparent grain volume. The jacketing material and screen solid volume can be determined by 1) direct measurement in a Boyle's Law Double-Cell matrix cup prior to use, or 2) jacketing material volume may be computed from a measured weight, and a previously-determined jacketing sleeve material density.

5.3.2.1.1.4 Accuracy

A well calibrated system will supply grain volume within ±0.2 percent of the true value. This corresponds to approximately ±0.03 cm^3 in one-inch diameter samples approximately 1 inch long and ±0.1 cm^3 in $1^1/_2$-inch diameter samples approximately 2 inches long.

5.3.2.1.1.5 Apparatus

The grain volume is measured in an apparatus consisting of two connected chambers of known volumes. An example of such an apparatus is shown in Figure 5-5.

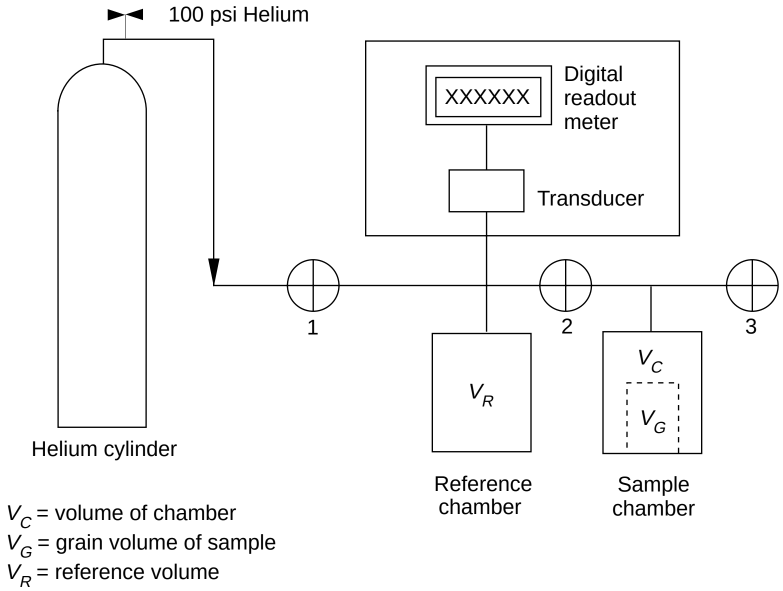


Figure 5-5—Double-Cell Boyle's Law Porosimeter

Note: For accurate measurements, it is important to (a) incorporate a valve having a zero displacement volume (e.g., a ball valve) between the reference volume and the sample chamber, or (b) the valve displacement volume must be known. The ball valve must always be vented to atmospheric pressure before it is turned to the closed position. Used in this manner, the valve bore volume is incorporated into the calibrated chamber volume and measurements are accurate. Otherwise, the valve displacement volume must be measured and accounted for in calculations for accurate measurement of grain volume.

5.3.2.1.1.6 Procedure

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and the sample chamber volume (V_c). A core plug is then placed in the sample chamber. Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1380 kPa). About 30 seconds should be allowed for pressure equilibrium and then p_1 (pressure indicated by the transducer digital readout) should be recorded. The gas is then allowed to expand into the sample chamber. The resulting lower pressure (p_2) is measured after the system has reached equilibrium (see 5.3.2.1.1.8, e). The grain volume of the sample is calculated using the gas law equations discussed in 5.3.2.1.1.7.

5.3.2.1.1.7 Calculations

The grain volume of the sample is calculated from the initial reference chamber pressure and the final system pressure by means of the gas law equation. The pore volume is the difference between the bulk volume and the grain volume.

The following grain volume equation is derived by a mass balance of gas within the reference and sample chamber:

$$\frac{P_1V_r}{z_1T_{1r}} + \frac{P_a(V_c - V_g)}{z_aT_{1c}} = \frac{P_2V_r}{z_2T_{2r}} + \frac{P_2(V_c - V_g + V_v)}{z_2T_{2c}} \qquad (15)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample chamber.
z_1 = z-factor of gas at P_1 and T_1.
z_2 = z-factor of gas at P_2 and T_2.
z_a = z-factor of gas at T_1 and atmospheric pressure.
T_{1r} = absolute temperature of reference volume at P_1.
T_{1c} = absolute temperature of sample chamber at P_1.
T_{2r} = absolute temperature of reference volume after P_2. is stabilized.
T_{2c} = absolute temperature of sample chamber after P_2 is stabilized.
V_g = grain volume.
V_c = sample chamber volume.
V_r = reference chamber volume.
V_v = valve displacement volume (from closed to open position).
p_1 = initial reference volume gauge pressure.
p_2 = final system gauge pressure.

If isothermal conditions exist (T_1 = T_2) and z values are assumed to equal 1.0, the equation reduces to:

$$V_g = V_c - V_r\left(\frac{P_1 - P_2}{P_2 - P_a}\right) + V_v\left(\frac{P_2}{P_2 - P_a}\right) \qquad (16)$$

If absolute pressures P_1 and P_2 are expressed as equivalent gauge pressures (i.e., $P_1 = (p_1 + P_a)$), and are substituted in the above equation, the following results:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) + V_v\left(1 + \frac{p_a}{p_2}\right) \qquad (17)$$

If a zero displacement volume ball valve is used and the valve is always vented to the atmosphere before closing, V_v is included in the sample chamber volume, V_v = 0.0 and the equation further simplifies to:

$$V_g = V_c - V_r\left(\frac{p_1}{p_2} - 1\right) \qquad (18)$$

Note: Calculations ignoring either z factors, or valve volume as small as 0.1 cm^3, can result in porosity errors of approximately 0.5 porosity points.

5.3.2.1.1.8 Precautions

Precautions for this method include:

a. The matrix cup must be designed to ensure that it will have the same internal volume each time it is reassembled.

b. The system must be recalibrated if there are changes in temperature or barometric pressure.

c. The core plug must be thoroughly and properly dried. Some samples require special drying techniques.

d. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).

e. Equilibrium pressure is essential for accurate grain volumes. Equilibrium is usually attained in 1 to 2 minutes, but low permeability and porosity cores require longer stabilization times (30 minutes to several hours). Longer times yield greater chance for temperature and barometric pressure changes.

f. Extraneous or dead volume (volume not occupied by the sample in the sample chamber) must be kept to a minimum or erroneous grain volumes will be determined. Solid stainless steel cylinders of known volume can be added to fill the sample chamber void volume left by shorter samples. When added, these steel volumes must be subtracted from the calculated grain volume to yield sample grain volume.

Note: If steel cylinders are present in the matrix cup during the calibration to yield V_c and V_r, their volumes should not be subtracted.

g. The pore volume of the sample to be measured should approximate the reference cell volume for best analytical results. Accuracy of the method on small, irregular test samples can be checked by determination of grain density on larger, and then smaller, similarly sized samples of a known grain density (i.e., Ottawa Sand).

5.3.2.1.1.9 Calibration

Porosimeter calibration varies with the instrument. In principle, two or more measurements are made; one is with the sample chamber filled with solid stainless steel cylinders of known volume. Subsequent measurements are made after removing one or more cylinders representing approximately 80, 60, 40, and 20 percent of the sample volume chamber. For best accuracy, sufficient cylinders should be removed to reduce p_1 by one-half.

Helium gas is admitted into the reference chamber at a predetermined pressure, typically 100 to 200 psig (690 to 1,380 kPa). About 30 seconds should be allowed for pressure equilibrium, and then p_1 (reference cell pressure indicated on the transducer digital read-out) is recorded. The gas is then expanded into the sample chamber.

The resulting lower pressure (p_2) is measured after the system has reached equilibrium (approximately 30 seconds). With valve volume (V_v) zero, temperature constant, and V_g known from volume of the steel cylinder in the chamber, V_r and V_c can be calculated from simultaneous solution of the appropriate equation presented in 5.3.2.1.1.7.

When V_v is not equal to zero or temperature varies, the solution becomes more complex, but the principle remains the same.

Note 1: In some systems equations are rearranged, and data are fit by linear regression over the cylinder volume range to yield V_c and V_r.

Note 2: Pressure transducers should be calibrated over 5 psi intervals with a dead weight tester or 0.02 percent full scale (or better) secondary standard pressure transducer. Transducer output can be fit to “true pressure” with a fourth degree polynomial using regression techniques. Check of the calibration can be made by measuring the volume of each steel cylinder and comparing it to the known volume. Agreement should be equal to or less than 0.03 cm^3 to assure porosity within 0.5 porosity points over the range of grain volumes measured.

5.3.2.2 Void Volume Measurement

5.3.2.2.1 Boyle’s Law Single Cell Method for Direct Pore Volume

5.3.2.2.1.1 Low Confining Stress

5.3.2.2.1.1.1 Principle

Pore volume is determined in an apparatus consisting of a gas charged reference cell of known volume and initial pressure, which is then vented into a sample’s pore volume. The sample is held in a core holder which utilizes an elastomer sleeve and end plugs. These conform closely to the sample when confining pressure is exerted on their external surfaces. The sleeve and end stems in turn exert compressive stress on the core sample. Pore volume is therefore determined directly using Boyle’s Law. This is in contrast to the double cell method whereby grain volume is determined and pore volume is calculated by subtracting grain volume from bulk volume.

The core holder may be a Hassler, isostatic, biaxial, or triaxial load cell. Low confining stresses, which are generally 400 psig or less, are employed. In consolidated, hard rocks, the type core holder is of little significance as minimum pore space reduction occurs at low stress. Although these are typically referred to as “ambient measurements,” it is important to note that some finite confining stress is necessary, and stress can have significant effects on unconsolidated or friable rocks.

Helium gas is admitted into the reference cell of known volume (V_r), at a predetermined reference pressure (100 to 200 psi). The reference cell gas is then vented into the sample pore volume. This results in a lower equilibrium pressure, from which the pore volume is calculated.

5.3.2.2.1.1.2 Advantages

Advantages of this method include:

a. The sample, if clean and dry at test initiation, is clean at test conclusion and ready for subsequent measurements.
b. Porosity and permeability can be run in sequence with a single loading of the sample.
c. Operation is quick and simple.
d. Harmful reactions between the rock and saturating fluid are eliminated by use of non-reactive gas.
e. Direct measurement of the pore volume eliminates the sensitivity of the pore volume to errors in the measurement of either *BV* or *GV*, where both *BV* and *GV* are large numbers relative to the pore volume, and $PV = BV - GV$.

5.3.2.2.1.1.3 Limitations

Limitations of this method include:

a. The system must be carefully calibrated for dead volume.
b. Changes in temperature and barometric pressure must be accounted for in the calculations.
c. The sample must be a good quality right cylinder with no surface vugs or chipped corners. (Vugs will yield erroneously low pore volume and non-parallel ends will yield high pore volume.) A porosity error exceeding 1.5 porosity units can result from a single, non-parallel sample face, which results in a reduction of sample length of 1.0 millimeter on one side of an otherwise one-inch diameter by one-inch long right cylindrical plug.
d. The sample must be clean of hydrocarbons and dry, or erroneously low pore volume will be determined.
e. The gas used must not adsorb to active mineral sites such as clays or carbonaceous materials. Adsorption results in erroneously high pore volume, and can be minimized by the use of helium gas.
f. Some low permeability rock (<0.01 md) may require a long time (thirty minutes to several hours) to reach pressure equilibrium. Longer times yield greater chances for temperature and barometric pressure changes.
g. When pore water salinities (either from formation water or mud filtrate) are greater than 100,000 mg/l, samples should be leached with methanol to remove salts. Ignoring the leaching step when pore water salinities exceed 100,000 mg/l can bias porosities low by 0.4 porosity points (typical 20 percent porosity and a water saturation of 50 percent pore space).
h. This method is suitable for jacketed, poorly consolidated/unconsolidated samples if proper precautions are observed. The void volume of the screens or porous plates attached to the plug faces must be determined. This volume must be subtracted from the jacketed sample’s measured void volume to yield sample pore volume. A solid non-porous jacketed plug equal to sample diameter may be prepared and tested as a sample. This will yield the screen or porous plate void volume, as well as any volume resulting from overlap of the jacketing material on the sample ends.
i. Porosity is determined at low confining stress, which results in a porosity higher than present in the reservoir.

5.3.2.2.1.1.4 Apparatus

The basic apparatus shown in Figure 5-6 is the same as that illustrated for the double-cell Boyle’s Law porosimeter shown in Figure 5-5 (see 5.3.2.1.1.5). The primary difference is the design of the sample chamber, which eliminates volume around the periphery of the sample.

5.3.2.2.1.1.5 Procedures

The porosimeter is first calibrated, yielding the reference chamber volume (V_r) and system dead volume (V_d). A clean, dry core plug is then inserted into an elastomer sleeve. An end stem with diameter equal to that of the plug is placed in contact with each end of the sample. A confining stress of 400 psi or less is applied to the external surface of the elastomer. If the sample is confined in an isostatic core holder, an equal

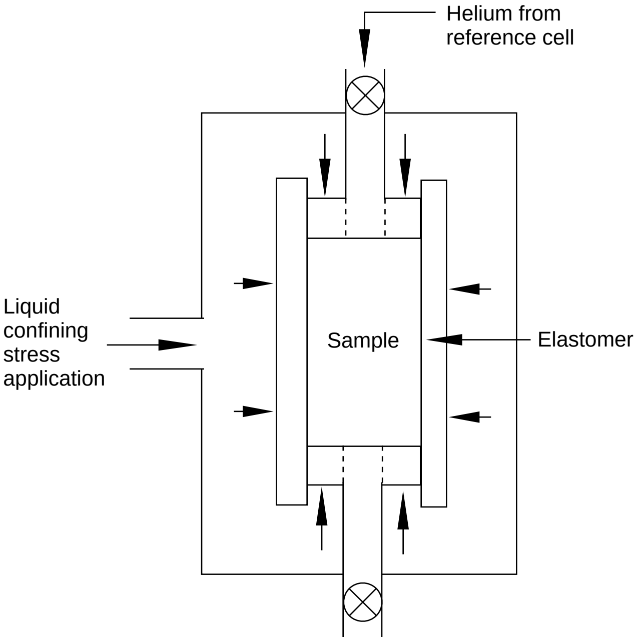


Figure 5-6—Schematic of Isostatic Load Cell for Direct Pore Volume Determination

confining stress is also applied to the outside surface of the end stems (see Figure 5-6).

Helium gas is admitted to the porosimeter reference chamber (V_r) at a predetermined pressure (p_1), typically equal to 100 to 200 psig (690 to 1380 kPa). Pressure is recorded, and the reference chamber is vented into the void volume of the sample. The resulting lower equilibrium pressure (p_2) is recorded. Pore volume of the sample is computed utilizing equations in 5.3.2.2.1.1.6.

5.3.2.2.1.1.6 Calculations

Sample volume is determined by expanding helium from a reference cell at known initial pressure directly into the porous rock. The following pore volume equation is derived by mass balance of gas within the reference cell, system dead volume, valve volume and sample pore volume.

$$\frac{P_1 V_r}{Z_1 T_{1r}} + \frac{P_a(V_p + V_d)}{Z_a T_1} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2 T_2} \qquad (19)$$

Where:

P_1 = absolute initial reference volume pressure.
P_2 = absolute expanded pressure.
P_a = absolute atmospheric pressure initially in sample.
Z_1 = gas deviation factor at P_1 and T_1.
Z_2 = gas deviation factor at P_2 and T_2.
Z_a = gas deviation factor at P_a and T_1.
T_{1r} = absolute temperature of reference volume at P_1.
T_1 = absolute temperature of sample pore volume at P_a.
T_2 = absolute temperature of reference volume and sample after P_2 is stabilized.
V_r = reference chamber volume.
V_p = sample pore volume.
V_v = valve displacement volume (from closed to open position).
V_d = system dead volume.

If isothermal conditions exist, ($T_1 = T_2 = T_{1r} = T_1$) and:

$$\frac{P_1 V_r}{Z_1} + \frac{P_a(V_p + V_d)}{Z_a} = \frac{P_2(V_r + V_p + V_d + V_v)}{Z_2} \qquad (20)$$

grouping terms yields:

$$V_p = \frac{V_r\left(\frac{P_1 Z_2}{P_2 Z_1} - 1\right) - V_v}{1 - \frac{P_a Z_2}{P_2 Z_a}} - V_d \qquad (21)$$

Note: This equation is valid if no change in pore volume occurs as the sample pore pressure increases from P_a to P_2, i.e., (a) when the net effective confining stress remains constant, or (b) when it is not constant, but pore volume change with net confining stress change is negligible.

5.3.2.2.1.1.7 Precautions

Precautions for this method include:

a. Confining pressure on the external surface of the elastomer sleeve should be applied by liquid and not gas. Gas may diffuse into and through the sleeve with extended testing times.

b. Durometer of the elastomer sleeve must be low enough (suggest 50 durometer maximum) so that the sleeve seals on the metal end stems and properly conforms to the sample surface at the low confining stress utilized.

c. Samples with ends that deviate slightly from parallel can sometimes be successfully tested by utilization of two deformable, rubber discs. Each disc should be soft, equal to end butt diameter, and contain a drilled center hole. A single disc should be inserted between each metal end stem and the sample face. The disc will compress and conform to fill the void space between sample and end stem.

Note: This is recommended only when sample ends cannot be squared. The procedure is not a substitute for good quality samples.

5.3.2.2.1.1.8 Calibration

Equipment calibration utilizes (a) billets of known volume which are added or removed from a special chamber introduced only for calibration, or (b) a series of steel plugs precision bored to yield a variety of void volumes that cover the range of pore volumes to be measured. The reference volume is charged to initial pressure and vented into the special chamber or into the known void volume of the confined calibration plug. The measurement is repeated with multiple calibration billets or plugs. The pore volume equations can be solved simultaneously to yield the reference cell volume V_r and the dead volume. These values then become a constant for subsequent measurements of pore volume.

Dead volume (V_d) of the system can also be determined by utilizing a solid steel billet as a sample within the core holder or by placing the end stems adjacent in the elastomer sleeve and applying the same low confining stress to be used for sample measurement. The charged reference cell (V_r) is vented to the steel billet or to the adjacent end stems, after which the pore volume equation is used to calculate V_d. This value also remains constant as long as no changes are made to the system. Steel check plugs of known void volume can be measured as a final check of system operation and proper V_r and V_d values. These check plugs should also be utilized as daily confirmation of proper equipment performance.

5.3.2.2.1.1.9 Accuracy/Precision

A properly calibrated system will yield pore volumes within ±0.03 cm^3 in perfect cylinders. Measurements on actual core samples indicate scatter of approximately 0.1 cm^3 on samples of 50 cm^3 bulk volume, yielding porosity deviation of ±0.2 porosity units from the true value.

5.3.2.2.1.2 Elevated Confining Stress

5.3.2.2.1.2.1 Principle

The principle is similar to that discussed under low confining stress; however, in this measurement, porosity is determined at elevated stress. These laboratory data in turn are used to estimate reservoir porosity at in-situ reservoir stress conditions. Unlike low confining stress measurements, knowledge of the (a) elevated confining stress magnitude, and (b) method of application (isostatic stress, triaxial stress, biaxial stress, or uniaxial strain) must be known to scale results to reservoir conditions (see Figures 5-7 and 5-8). Historically, most routine pore volume measurements at elevated stress have been made with an isostatic (equal in all directions) confining stress, because this is an easily applied, relatively inexpensive method. Data are subsequently presented as porosity versus effective (confining pressure minus pore pressure) isostatic stress. In some reservoirs, a more complex and time consuming pore volume compressibility should be measured to more accurately determine porosity under reservoir conditions. These compressibility measurements typically require that liquid be present in the pore space to model liquid-rock interaction effects, if any,[1] and are beyond the scope of this document (see references in 5.6).

5.3.2.2.1.2.2 Advantages

Advantages of this method include:

a. Advantages are similar to those for low confining stress (see 5.3.2.2.1.1.2).

b. Measurements at elevated confining stress more closely represent original reservoir porosity than measurements at zero or low confining stress.

5.3.2.2.1.2.3 Limitations

Limitations of this method include:

a. Limitations are similar to items a through h for low confining stress (see 5.3.2.2.1.1.3).

b. Penetration of the elastomer sleeve into the depressions between sand grains or surface vugs on the sample surface as the confining stress increases may indicate pore volume reduction, even if no sample compression actually occurs. This penetration seldom exceeds 0.2 porosity units for non-vuggy sandstones (e.g., 10.0 percent to 9.8 percent), and while present, normally has no significant impact on porosity values. For vugular samples, this error can be significant. To reduce this error, a mylar or metal inner sleeve may be used. Surface effects can be minimized by increasing sample radius (*r*), as the ratio of surface area to bulk volume equals $2/r$ and decreases as sample radius increases.

c. The initial confining stress must be high enough to seal the elastomer sleeve to the sample and sample holder end butts, and to press the sample holder end butts tightly against the sample face. Sealing of the elastomer to the sample surface is dependent on the elastomer stiffness and sample roughness. Sealing stress must also be high enough to prevent bypassing of gas if permeability is to be measured in conjunction with porosity. Consequently, two opposing effects on porosity can occur. Poor conformance of the sample holder end butts with the sample, which yields porosity values that are too high, may be offset by sample pore volume compression. Use of good quality, right cylindrical samples with parallel ends minimizes conformance difficulties.

d. The resultant porosity at any elevated stress may be dependent on the rate of stress application, on the stabilization time and on the stress history.[2]

e. Reservoir effective stress must be known or estimated in order to properly utilize the laboratory-determined elevated-stress porosity data. Actual reservoir stresses causing pore volume reduction are usually not isostatic. The maximum principal stress in most reservoirs is vertical. This vertical effective stress equals the weight per unit area of overburden rock minus the reservoir pressure. Overburden stress can be computed from the depth and a downhole density log, or estimated assuming rock density equals 1.0 psi/ft.[3] Reservoir pressure can be measured, or in normally pressured areas, is assumed to be depth times the water gradient, which is approximately 0.45 to 0.5 psi/ft.

Note: Offshore reservoirs have a component of overburden stress resulting from water depth and density, as well as overburden rock.

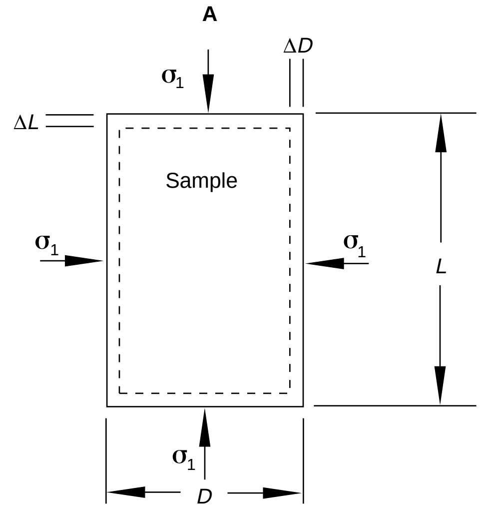
Typically, horizontal stresses are some fraction of the vertical stress, and can be estimated from hydraulic fracture treatments or formation integrity (leakoff) tests.[4,5] The mean reservoir effective stress is an average of vertical and horizontal effective stresses. Consequently, the mean reservoir effective stress is typically less than an isostatic stress equal to the vertical effective stress.

f. The presence of water within the pore space has been shown to weaken some rocks, thereby increasing pore volume reduction as confining stress is applied.[1] Since routine measurements, as discussed here, are on cleaned and dried rock samples, while the reservoir contains interstitial water, some uncertainty is introduced. While this effect is minimal in hard, well-cemented rocks, the importance of this effect can be quantified by conducting specialized tests on samples that contain water.

g. Friable or poorly-consolidated cores can exhibit significant compression at low confinement stresses.

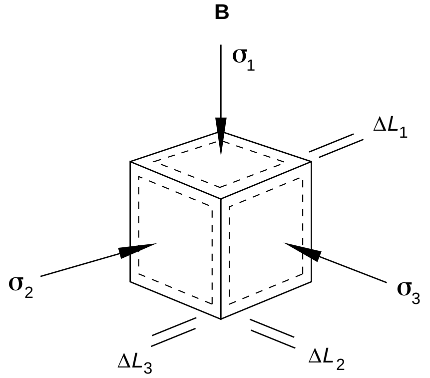
5.3.2.2.1.2.4 Apparatus

The apparatus is similar to that for low confining stress. However, high confining stress measurements require a sleeve material made of a tough elastomer that can withstand forces imposed during confining stress application, and yet will conform to the sample at low stress.



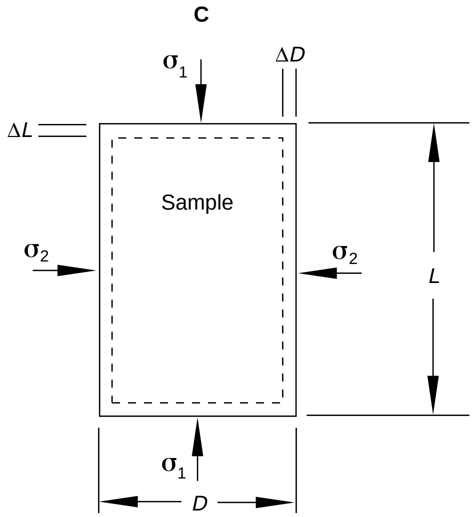
Isostatic Stress

Under isostatic stress loading, equal stress is applied to the sample in all directions, and sample strain can occur on all axes. Excessive porosity reduction typically occurs when the imposed isostatic stress is equal to the vertical reservoir stress (i.e., the overburden stress).



Triaxial Stress

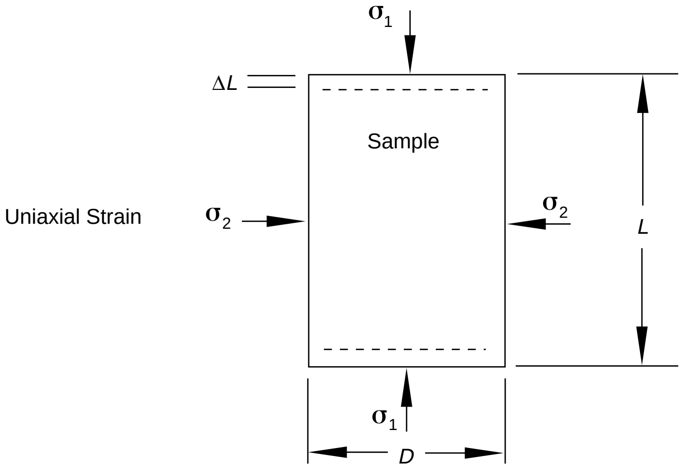
Under true triaxial stress conditions, unequal stress is applied to the three major axes of the sample. In the general case, strains will be different on each axis. Typically, a cube or rectangular prism-shaped sample will be used.



Biaxial Stress

Biaxial stress loading conditions are a special case of triaxial stress loading. In the biaxial stress loading of a cylinder, the stress parallel to the cylinder axis is different than the stress applied around the sample's circumference. Strains can occur parallel to both the axis and diameter of the cylinder.

Figure 5-7—Laboratory Loading Schematic



Uniaxial strain compression is a special case of biaxial stress loading; the stress applied to the circumference is just sufficient to maintain the diameter constant as the stress parallel to the cylinder axis is increased. Strain occurs only parallel to the axis of the cylinder.

Figure 5-8—Laboratory and Reservoir Loading Schematic

5.3.2.2.1.2.5 Procedures

Procedures for elevated confining stress measurements are similar to those for low confining stress discussed in 5.3.2.2.1.1.5.

Typically, sample pore volume is determined at selected levels of increasing confining stress. Equilibrium pressure within the sample pore space at each confining stress is normally vented prior to subsequent pore volume measurement. Equations shown in 5.3.2.2.1.1.6 are used to compute pore volumes.

5.3.2.2.1.2.6 Calculations

The basic equations for pore volume (*PV*) calculation are the same as for low confining stress measurements, but calculation of porosity must also account for reduction in sample bulk volume (*BV*) as confining stress increases. Approaches that can be used include the following:

$$\text{Porosity} = PV/(\text{Non-stressed } BV - \Delta PV) \qquad (22)$$

Where ΔPV = reduction in pore volume between initial PV and PV at any stress. This assumes the bulk volume reduction at any stress equals the pore volume reduction at the same stress.

$$\text{Porosity} = PV/(GV + PV) \qquad (23)$$

Where PV = pore volume at any stress. This assumes grain volume (*GV*) as determined at zero stress is constant at all applied confining stress levels.

$$\text{Porosity} = PV/\text{calculated } BV \qquad (24)$$

Measure pore volume (*PV*), sample diameter, and length at each stress imposed and calculate bulk volume. This is not routinely done.

5.3.2.2.1.2.7 Calibration

Calibration of equipment utilizes the same principles discussed in low confining stress (see 5.3.2.2.1.1.8). Validity of the system operation at elevated confining stress can be confirmed by measurement of steel check plug void volumes at the confining stresses selected for sample pore volume determination.

5.3.2.2.1.2.8 Accuracy/Precision

A properly calibrated system will yield pore volume accuracy of the same magnitude as observed at low confining stress (see 5.3.2.2.1.1.9), yielding porosity scatter of ±0.2 to 0.3 porosity units.

5.3.2.2.2 Summation of Fluids Porosity Summary

The “Summation of Fluids” technique of porosity determination is discussed in detail in the Fluid Saturation Methods under “Retort Method at Atmospheric Pressure” (see 4.2). It furnishes porosity data quickly as samples do not require cleaning or drying. The pore volume is computed by measuring and summing oil, gas, and water volumes present in a freshly recovered core sample. Porosity is determined by dividing pore volume by bulk volume of the rock sample.

When proper procedures are followed, and rocks are reasonably uniform and contain no hydratable minerals to complicate determination of water volumes, Boyle’s Law porosities on adjacent core will typically agree within ±0.5 porosity units.[6] Without special precautions and techniques, the summation of fluids porosity can be erroneously high due to excess water recovery from hydratable minerals. This technique is not valid on weathered core from which liquids have evaporated. Measurements on weathered rock yield an erroneously low gas volume, and hence porosity, because the injected mercury at the pressure utilized does not fill the smaller pores from which water has evaporated.

Grain density can be calculated from the measured volumes of rock and fluids. This can be compared to an estimated grain density based on lithology. The magnitude of the difference between calculated and estimated values yields a quality control check that will expose erroneous measurements or data calculation. This is not valid when extraneous minerals (heavy or light) are present in the rock. These conditions make the grain density variable and difficult to accurately estimate.

Retorting of the rock destroys the sample, so adjacent core must be used for permeability measurements. These permeability samples can be tested for porosity utilizing the Boyle's Law procedure, and compared with the summation of fluids values as a quality control check. Perfect sample-to-sample agreement is not expected, but the data should exhibit no bias.

5.3.2.2.3 Liquid Saturation Method

5.3.2.2.3.1 Principle

The measurement of porosity (connected pore space) by the liquid saturation method involves the gravimetric determination of pore volume by obtaining: (a) the weight of the core sample clean and dry, (b) the weight of the sample saturated with a liquid of known density, and (c) the weight of the saturated sample submerged in the same liquid.

5.3.2.2.3.2 Advantages

Advantages of this method include:

a. Many samples can be handled at one time.
b. Provides a direct measurement of pore volume.
c. Refer to 5.2.4.2, a and b.

5.3.2.2.3.3 Limitations

Limitations of this method include:

a. The procedure is slow in regard to total elapsed time of measurement.

b. The procedure is basically accurate, but limited to samples which can be 100-percent saturated and the saturated weight successfully determined.

c. Cores containing surface vugs are not recommended for porosity determination by this method because of the potential loss of liquid from the vugs during the weighing process. If liquid is lost, this volume would not be included in the pore volume, and erroneously low porosity will be calculated. However, grain volume can be accurately determined on vuggy cores by this method.

d. Refer to 5.2.4.3, a and c.

5.3.2.2.3.4 Accuracy

The method should determine porosity (connected pore space) within 0.5 porosity units and grain volume to ±0.2 percent of true value if proper technique is used.

5.3.2.2.3.5 Apparatus

Appropriate apparatus for this method include:

a. Analytical balance accurate to 1 milligram.
b. Suitable containers which can be used to hold deaerated liquid under vacuum.
c. Vacuum desiccator and pressure saturator.
d. A suitable vacuum source capable of maintaining less than 0.1 millimeter of mercury pressure.
e. A filtered, low-viscosity, low vapor-pressure liquid of known density for the purpose of saturating the core samples. Some liquids which have been used are: 1) brines, 2) refined laboratory oils, 3) decane, and 4) toluene.
f. Suitable containers which can be used to hold saturated cores under deaerated liquid.

5.3.2.2.3.6 Procedure

Obtain the weight of the dry, clean sample. The sample should be desiccated over a suitable dehydrating material, such as $CaCl_2$ or silica gel, prior to the determination of the dry weight.

Place the weighed, dry sample in a chamber (vacuum desiccator and pressure saturator) and apply a high vacuum for about 8 hours. The evacuation period may be followed by injection of CO_2 to remove the absorbed air on the rock. This replaces adsorbed gases such as O_2 or N_2 with CO_2, usually a more soluble gas in liquids typically used. Several cycles of evacuation and CO_2 injection may be required for low permeability samples. Longer CO_2 contact time and longer vacuum cycles are required to displace N_2 in the tighter rock. For very low-permeability samples, the evacuation period should be as long as 12 to 18 hours (overnight).

The liquid used to saturate the cleaned core sample should be free from dissolved air; therefore, the liquid should be deaerated before introduction into the core. Extreme care should be taken to avoid air contact with the liquid after its deaeration. Hydrocarbons are stronger wetting fluids than brines and are preferred if only porosity measurements are desired and samples saturated with brine are not required for further testing.

The deaerated liquid is drawn into the evacuated vessel containing the core sample. The core is allowed to saturate. After the sample has been completely submerged in the liquid, the vacuum is continued for an additional 30 minutes to 1 hour.

The preferred method is to then pressure the liquid surrounding the sample to 2,000 to 3,000 psi (13,800 to 20,700 kPa) for at least four hours to assure complete saturation. Pressure saturation should result in the liquid entering additional voids which are not well connected or where spontaneous imbibition has not occurred. The sample is then removed from the saturating vessel and weighed submerged in the saturating liquid.

Excess liquid is carefully removed (avoiding grain loss) from the sample and the saturated sample is weighed in air. When removing excess liquid from the sample surface, precautions must be taken to ensure fluids are not removed from pores exposed at the surface. Materials (such as dry towels) which remove liquid from surface pores due to capillary action should be avoided, as should any mechanical method such as violent shaking. Rolling the sample over a lint free cloth or paper towel dampened with the saturating liquid, or carefully wiping the sample with the dampened cloth or the fingers are suitable techniques for removing excess liquid.

After weighing each batch of samples, the density of the deaerated saturant used in the saturation chamber is determined. This must be done with accuracy, since the saturant density is probably the greatest source of error unless Equation 5.3.2.2.3.8e is used. Refer to procedures described in 5.2.4.6 for the determination of bulk volume. When coupled with the original dry weight and fluid density, grain volume is computed directly (see Equation 5.3.2.2.3.8c).

For storage until use, the core plugs are usually transferred to screw-capped jars or other suitable containers that are filled with deaerated liquid in order to minimize the presence of air in the container.

5.3.2.2.3.7 Precautions

Precautions for this method include:

a. Special precautions are necessary to ensure complete saturation. Low permeability cores may not be fully saturated by this method.

b. For acceptable accuracy, it is essential to use the correct fluid density at the time the saturated weight of the sample is determined.

c. Error in the pore volume can result from improper wiping of the sample prior to the determination of the saturated weight.

5.3.2.2.3.8 Calculations

The calculations for this method are as follows:

a. $$PV = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}$$

b. $$BV = \frac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}} \qquad \text{(see 5.2.4)}$$

c. $$GV = \frac{\text{Dry Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}$$

d. Pore Volume check: $PV = BV - GV$ (PV by difference should agree with PV calculated in a.)

e. $$\text{Porosity} = \frac{PV}{BV} = \frac{\dfrac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Density of Saturant}}}{\dfrac{\text{Saturated Weight} - \text{Immersed Weight}}{\text{Density of Immersion Liquid}}}$$

If temperature is constant, and density of saturant and immersion liquid are equal, fluid density cancels and is not needed in the above equation to calculate porosity.

$$\text{Porosity} = \frac{\text{Saturated Weight} - \text{Dry Weight}}{\text{Saturated Weight} - \text{Immersed Weight}}$$

f. Porosity check: $\text{Porosity} = \dfrac{BV - GV}{BV}$

(Porosity should agree with that calculated in e.)

g. $$\text{Grain Density} = \frac{\text{Dry Weight}}{GV}$$

Where:

PV = pore volume.
GV = grain volume.
BV = bulk volume.

5.4 HISTORICAL PROCEDURES

This section lists and summarizes techniques that have been used historically to measure porosity, bulk volume, grain volume, and grain density. These have generally been replaced by newer techniques. Details about these procedures can be found in the First Edition of API RP 40, *API Recommended Practice for Core-Analysis Procedure,* August 1960.

5.4.1 Mercury Displacement

This technique consists of immersing a dried core sample in mercury inside a calibrated pycnometer. The volume of mercury displaced by the sample is weighed to determine the bulk volume of the core sample. (API RP 40, 1960, Sec. 3.311, p. 15; Sec. 3.56, p. 26.)

5.4.2 Bulk-Volume Meter

This procedure also involves submerging a core sample under mercury. The volume of mercury displaced by the sample further displaces a second liquid into an inclined, graduated glass tube. The bulk volume of the core is read directly off the graduated glass tube. (API RP 40, 1960, Sec. 3.312, p. 15; Sec. 3.57, p. 26.)

5.4.3 Boyle's Law Single-Cell Method Using the Kobe Porosimeter

The Kobe Porosimeter consists of a mercury pump, pressure gauge, gas inlet and outlet valves, and a sample chamber. Determination of the bulk volume of the sample requires filling the sample chamber containing the core sample with mercury. This leaves the core sample immersed in mercury. Bulk volume is determined by subtracting the volume of mercury needed to fill the sample chamber without a sample from the volume of mercury needed to fill the chamber with a sample.

Grain volume is calculated by backing the mercury pump out to a "preset" volume. During this portion of the test the sample is not immersed in mercury. The pressure in the cell is vented to atmospheric pressure (P_1). Mercury is pumped into the sample cell (gas outlet valve shut) and the new pressure (P_2) is recorded. Knowing the volumes of mercury in the cell, the empty preset volume of the cell, and the pressures, Boyle's Law can be used to calculate grain volume ($P_1 \times V_1 = P_2 \times V_2$). Porosity is calculated from the bulk volume and the grain volume. Grain density is calculated from sample weight and grain volume. (API RP 40, 1960, Sec. 3.32211, p. 17; Sec. 3.5.10, p. 28.)

5.4.4 Washburn-Bunting Method

The Washburn-Bunting porosimeter measures the pore volume of the sample. The apparatus consists of a sample chamber with a graduated capillary tube on top with a stopcock for opening and closing the system to the atmosphere. A tube is attached to the bottom of the sample chamber which connects to a glass bulb filled with mercury. A sample is placed in the sample chamber. The procedure is begun by raising the mercury bulb to force mercury into the sample chamber, thereby immersing the sample, and up into the calibrated capillary. When the mercury is above the stopcock, the stopcock is closed. The mercury bulb is now lowered until the core sample is floating on the mercury inside the sample chamber. The sample is now under vacuum, and air from the pores of the sample fill the sample chamber and calibrated capillary. After a few minutes, atmospheric pressure is restored to the escaped air by raising the mercury bulb until the mercury level is equal to that in the capillary. The air volume in the calibrated capillary is equal to the pore volume of the rock sample. Improved accuracy is obtained by subtracting the air volume measured by using a solid billet in place of the core sample to account for adsorbed air on the sample surface. (API RP 40, 1960, Sec. 3.3221, p. 17; Sec. 3.5.12, p. 30.)

5.4.5 Dry Method for Grain Density and Pore Volume

In this procedure, the rock sample is extracted, dried, crushed, and sieved. A portion of the crushed rock that passes a 60-mesh screen and is caught by a 100-mesh screen is weighed and added into the sample cup of the apparatus. A mercury pump is then used to pump mercury into the sample cup. When the system pressure reaches the preset calibration value, the volume of injected mercury is recorded. By calculating the volume of the empty cup, the bulk volume of the sample in the cup can be determined. Using the sample weight, grain density can be computed. Using a companion rock sample, grain volume is computed by dividing the companion sample dry weight by the calculated grain density from the crushed sample. Pore volume can be determined by subtracting the grain volume from measured bulk volume of the sample. (API RP 40, 1960, Sec. 3.3211, p. 16; Sec. 3.58, p. 26.)

5.4.6 Wet Method for Grain Density and Pore Volume

The bulk volume is measured and the dry sample is weighed and crushed. The crushed sample is added to a calibrated volumetric flask containing a known volume of a suitable wetting fluid (e.g., toluene, water, etc.). The volume increase is equal to the grain volume of the crushed sample. The grain density can be calculated by dividing the weight of the crushed sample by its measured volume. The pore volume can be calculated by subtracting grain volume from bulk volume. (API RP 40, 1960, Sec. 3.3212, p. 16; Sec. 3.59, p. 27.)

5.4.7 Full Diameter Summation of Fluids Utilizing the Vacuum-Retort Method

This method was used extensively in "hard rock" regions (e.g. West Texas) where production is predominately from carbonate formations. Each full diameter test sample was weighed and then the gas filled pore space was pressure saturated with water. The sample was again weighed, with the weight increase equal to the gas volume. Fluids within the pore space were distilled from the core under partial vacuum, at a maximum temperature of 450°F (232°C). Fluids were collected in glassware immersed in an alcohol/dry ice bath maintained at –75°F (–59°C). This was to condense vapors and prevent loss through the vacuum system. Condensed volumes were read, and an oil correction factor was employed. The sample pore water was computed by subtracting the gas volume from total water collected in the condensing tube. Pore volume was computed by summing the gas, corrected oil, and pore water volumes. A bulk volume determined on the test sample was used for calculation of porosity (API RP 40, 1960, Sec. 4.21, p. 39; and 4.52, p. 42).

One of the prime disadvantages of this method was the condition of the cores after the distillation process. It was not uncommon for the cores to be black from coked oil and no cleaning process could restore the samples to a condition whereby they could be deemed suitable for use in further testing procedures (e.g., a Boyle's Law Porosity, Capillary Pressure Tests, Relative Permeability). For this and other reasons, the Subcommittee charged with the rewriting of Recommended Practice 40 elected not to record this as a recommended procedure.

5.5 ORGANIC-RICH ROCKS

In recent years, there has been considerable interest in the development of coalbed methane and gas shale reservoirs. Coals and gas shales are typically highly organic fractured formations in which much of the gas-in-place is adsorbed to the organic material. Because adsorbed gas comprises a significant portion of the gas-in-place in these reservoirs, both the adsorbed gas content and how the gas is released (desorption/adsorption isotherms) are necessary for reserve estimates and production forecasts. Oil shales contain solid organic material, typically liquefied by application of heat to generate oil. Techniques to evaluate these type of deposits are discussed in 4.7. Porosity, as discussed herein, is not normally required.

5.5.1 Coal

There are no generally accepted methods in the industry for the laboratory measurements of porosity, permeability, or relative permeability of coal samples, nor are there published laboratory measurements that are accepted standards. Standard laboratory techniques for measurement of porosity in oil and gas bearing reservoir rock must, therefore, be modified when applied to coal used in coalbed methane studies. Following are comments and references reflecting current practices.

In production of methane from coal seams, "porosity" may be defined in terms of three distinct parameters. For calculation of coalbed methane gas-in-place, these are (a) the "in-situ gas content" of the coal, which is equivalent to the gas volume contained in the gas-filled porosity in a conventional gas reservoir. In addition, coal is a dual porosity system consisting of (b) matrix (micropore) porosity wherein the methane is adsorbed on the coal surface, and (c) cleat porosity (naturally occurring microfracture porosity). The cleat network provides the path and permeability for fluid flow.

5.5.1.1 In Situ Gas Content

The only method by which total in situ gas content of coalbeds can be measured directly is with the use of pressure core technology.[7] The volume of gas evolved from the coal core in a pressure core barrel is measured as a function of time, temperature, and pressure.

Gas content and desorption rate are often determined by canister desorption methods.[8] Coal samples utilized include cores obtained using conventional coring, drilled sidewall coring, wireline retrieval, and coal drill cuttings. The coal cores (or cuttings) are sealed in the canister and the volume of gas evolved from the sample is measured as above.[7] Gas content determination by canister desorption requires an estimation of the volume of gas lost while retrieving the samples to the surface and before sealing the cores in the canister.[9,10]

Gas content from conventional and sidewall core samples by the canister desorption method is much less expensive than pressure coring, and is the most commonly used technique. Even pressure cores are often placed in desorption canisters to complete the desorption process. Gas content from different methods or samples can be compared after normalizing the coal to an ash-free basis.[7] Since the gas evolved is not always 100 percent methane,[11] the composition of the gas must be analyzed to determine methane content.

5.5.1.2 Matrix Porosity

The matrix porosity of coal is generally larger than the cleat porosity. However, since the methane is adsorbed in the matrix rather than existing as a free gas, conventional oil or gas field matrix porosity measurements are seldom performed for coalbed methane applications. If conventional pore volume is required, equilibrium moisture[12] and helium Boyle's Law porosity[13] techniques can be used to measure matrix porosity.

Note: Any gas that adsorbs will yield erroneous matrix porosity values with the Boyle's Law technique. Helium, which is not appreciably adsorbed, is often used.

5.5.1.3 Cleat Porosity

Although the bulk of coal porosity is the matrix porosity, the cleat network provides the path and permeability for fluid flow. The measurement of cleat porosity in coal is, therefore, important in determining pore volume compressibility, as well as the saturations necessary for gas-water relative permeability or capillary pressure measurements.

Cleat porosity is a function of net confining pressure. Cleat porosity should be measured with the coal core in a uniform confining pressure. Jacketing materials commonly used for unconsolidated cores[16] are often used on coal cores.

Measurements of coal porosity have been made on coal cores dried in vacuum ovens.[14] Studies on coal weathering indicate such drying alters the coal structure,[15] yielding erroneous results. A suitable procedure for cleat porosity measurement on non-dried core is referred to as the miscible drive technique. In this procedure, the fluid saturating the core is displaced from the core by a second fluid with different physical properties, but which is miscible with the saturating fluid.[16]

The miscible drive technique may employ tracer concentration in water. A tracer that is adsorbed will yield erroneous results.[17] Within experimental error, the immiscible displacement with water-vapor-saturated helium of water from a water-saturated coal core yields the same results as the miscible drive tracer technique.[17]

Note: Coal cores often have residual methane in the matrix, which can affect porosity measurements. Saturation of coal core with helium (saturated with water vapor), followed by evacuation, has been used to remove the residual methane.[17]

5.5.2 Gas Producing Shale

In gas shales, such as the Devonian, gas may be stored as free gas in the matrix or fractures, and also exist as adsorbed gas on the organic and clay surfaces. Adsorbed gas may account for as much as 80 percent of the gas-in-place, emphasizing the need for adsorbed gas isotherms. Volumes of adsorbed gas in shale are $^1/_5$ to $^1/_{50}$ of that for coal, and measurement apparatus and procedures must be tailored to supply accurate data. As is currently the case for coal, no industry standard procedures currently exist for conducting shale measurements.

Reported results of adsorbed gas isotherm measurements on shale samples indicate that a wide disparity in test conditions currently exists between testing laboratories.[18] These disparities result in varying adsorption results, and emphasize the need for persons using the data to clearly understand what is being measured and what is reported.

Simulators utilized in reservoir modeling often utilize as input adsorbed methane content versus pressure data. Data are typically reported in units of scf/ton. Consequently, it is important to know if the isotherm measured and reported is for adsorbed gas alone, or if it represents a total sorption isotherm, which includes adsorbed gas as well as gas stored as free gas in the matrix and/or fractures.

The presence of water reduces gas volumes adsorbed. Adsorption tests on dry rock, therefore, will over estimate gas reserves, and tests on rock samples containing an equilibrium moisture condition are preferred.

Note: Techniques to establish appropriate equilibrium moisture conditions representative of the reservoir are not yet defined. Consequently, many of the adsorption isotherms measured have been completed on shale "as received," or on dry rock.

Although the technology for measurement is still evolving, certain important elements affecting measurement results and reported data have been identified.[18] Engineers utilizing these data should discuss with the measurement laboratory the following key elements. Procedures should be established that are most likely to yield data representative of the reservoir and that are required in the mathematical simulator utilized.

5.5.2.1 Key Elements Affecting Measurement

Factors affecting the measurement include:

a. Core recovery techniques (including coring fluid).
b. Sample preservation technique.
c. Sample size and shape (crushed? sieved to what size?).
d. Sample drying conditions:
    1. "As received."
    2. Dried (at what temperature and how long?).
    3. At equilibrium conditions (what temperature and how long?).
e. Standard temperature and pressure used for reporting measured gas volumes as scf/ton.
f. Metric ton or U.S. ton of "as received," dried or equilibrium weight rock used as base for scf/ton.
g. Total sorbed (including free porosity) gas or adsorbed gas only isotherm.
h. Cell calibration technique.
i. Temperature measurement and control of cell.
j. Sample density reported (bulk or grain; wet or dry).
k. Was equilibrium reached at each adsorption level, or was test step terminated at selected adsorption time?
l. Sample volume tested.
m. Sample evacuated prior to testing.
n. Gas used in adsorption measurement (usually methane).
o. Gas used in "free gas volume" porosity measurement (usually helium).

5.5.2.2 Apparatus

An apparatus designed to determine volumetric adsorption is illustrated in Figure 5-9.[19] It resembles a commonly used double-cell porosimeter.

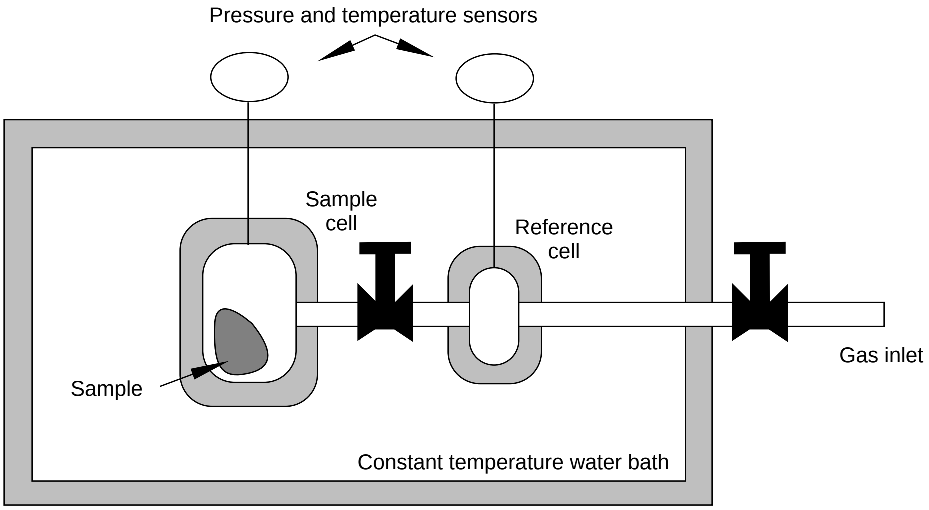


Figure 5-9—Volumetric Adsorption Measurement Method

5.5.2.2.1 Free (Fracture and Matrix) Porosity

Fracture and matrix void volume can be determined utilizing Boyle's Law double-cell porosimeter (see Figure 5-5) with helium as the gas phase.

5.5.2.2.2 Adsorption Isotherms

An isotherm measurement with helium gas is first completed. This yields free gas space with minimum adsorption. Methane gas is than charged into the reference cell and the gas is expanded into the sample chamber. The reference chamber is again isolated from the sample cell and is charged with gas at higher pressure. The procedure is repeated, and the gas adsorbed at the higher pressure level is calculated. This process is repeated until desired pressure levels are reached. The difference between the helium and methane isotherms represent adsorbed gas. Equations for calculation of isotherms, as well as an error analysis identifying factors that affect the accuracy of the measurement have been presented.[19]

5.5.3 Oil Shale

Evaluation of oil shales do not typically require measurement of rock porosity, as the oil recovered is generated from solid organic materials subjected to heat. See 4.7 for oil content determination.

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6 Permeability Determination

6.1 INTRODUCTION

Permeability is a property of a porous medium and is a measure of its ability to transmit fluids. The measurement of permeability of a porous rock, or stratum, is a measurement of the fluid conductivity of the particular material. Permeability is the fluid-flow analog of electrical or thermal conductivity. The reciprocal of permeability represents the viscous resistivity that the porous medium offers to fluid flow when low flow rates prevail. This condition of flow is customarily called "viscous flow," or formally, "Stokes flow". Measurement of the flow of fluid through a sample in a particular direction yields permeability of the sample in that direction. The permeability of a homogeneous, isotropic medium is the same everywhere, and in all directions. However, real rocks are neither perfectly homogeneous nor isotropic.

6.1.1 Definitions

6.1.1.1 Darcy Flow

The mid-nineteenth century experiments and studies of Darcy[1] led to an empirical expression of the relationships among the variables involved in the flow of fluids through porous media, known now as Darcy's Law. Briefly, Darcy's Law states that the volumetric rate of flow per unit cross-sectional area of permeable medium (the volumetric flux) is directly proportional to the potential gradient, and inversely proportional to the viscosity of the fluid. The coefficient of proportionality is permeability. Thus, the potential gradient required for Stokes flow is equal to the product of the fluid's viscosity and volumetric flux, divided by the permeability of the rock. The potential gradient for horizontal liquid flow, or in any direction for practical measurements with low density gas, is equal to the pressure gradient.

6.1.1.2 Forchheimer Inertial Resistance

The work of Forchheimer[2] at the beginning of the twentieth century showed that Darcy's Law is a limiting case, restricted to low volumetric fluxes. At higher fluxes, Forchheimer observed that the potential gradient required for a given volumetric flux is greater than that predicted by Darcy's Law by an amount proportional to the product of the fluid's density and the square of its volumetric flux. The coefficient of proportionality, β, is the inertial resistivity of the porous medium. Inertial energy dissipation is due to innumerable accelerations (i.e., time-rates of change in the direction and magnitude of velocity) that a fluid undergoes as it travels tortuous paths through a porous medium. These accelerations cause secondary flow patterns, in which, part of the flow energy is converted to heat through viscous shear.

In a petroleum reservoir, especially away from a producing or injection well, volumetric fluxes are generally so low that Darcy's Law is applicable. However, high near-wellbore fluxes associated with high pressure gradients can cause non-negligible inertial effects known as "rate-sensitive skins." These are particularly prevalent near gas-producing wellbores, and in perforations, where low gas viscosity permits very high fluxes.

In laboratory measurements of high permeability samples using gas (where inertial effects are most often observed), low volumetric fluxes require low pressure gradients, which can be difficult to measure accurately. Higher fluxes will cause the calculation of permeability to be low if Darcy's Law is employed. A procedure to estimate the maximum allowable pressure gradient as a function of permeability, to avoid significant permeability errors due to inertial resistance, is presented in 6.2.1.3. An alternative is to make multiple flow rate measurements and employ Forchheimer's equation to obtain both permeability and inertial resistivity of each sample. As a practical matter, this is most quickly accomplished with the pressure-transient technique presented in 6.4.1.1.

6.1.1.3 Klinkenberg Gas Slippage

Even when inertial effects are properly accounted for, the permeability of a porous medium to gas is dependent upon the mean free path of the flowing gas, hence among other things, upon its absolute pressure. This is due to a phenomenon known as slip, a fact first pointed out to the oil industry by Klinkenberg.[3] Gas slippage had been overlooked or ignored by prior investigators of permeability despite theory and experimental data for slip in the flow of gases through small capillary tubes presented in scientific literature as early as 1875.[4] When gas slippage is ignored, permeability calculated from the Forchheimer equation, or from Darcy's Law (provided that inertial resistance is negligible), is higher than that obtained using a non-reactive liquid. When expressed as a percentage, this difference is small for high permeability samples, but becomes progressively larger with decreasing permeability. It is minimized by using high mean pore pressures in gas permeability measurements.

To avoid the problem of obtaining pore-pressure-dependent gas permeabilities, Klinkenberg presented a method in which gas permeability measurements made at several different mean pore pressures can be extrapolated to infinite pore pressure. He showed that this extrapolated gas permeability (now called "Klinkenberg permeability", k_∞) is equal to the permeability obtained using a non-reactive liquid, such as a clean, refined hydrocarbon.

Permeability of rock samples, especially those that contain certain clays, can be altered when they are cleaned and dried in preparation for gas permeability measurements. Often, k_∞ is higher in these samples than permeability measured using aqueous solutions. Special cleaning and/or drying techniques may be required.

6.1.1.4 Liquids

The use of liquids for permeability measurements eliminates the problem of gas slippage, and at reasonable and usual flow rates, inertial resistance is generally negligible. Thus, Darcy's Law can be used directly to calculate permeability from a single flow rate measurement. However, potential permeability alteration from interaction of rock constituents and liquids (especially aqueous solutions), fines movement, and microbial plugging requires special attention. Also, the liquid remaining in a sample may have to be removed before other measurements can be performed. Because of these problems, most routine permeability measurements have been made using gases. However, for some samples, such as those sensitive to drying techniques, liquid permeability measurements are considered to be the only acceptable alternative.

6.1.1.5 Confining Stress Dependence

Permeability of a porous medium is sensitive to the magnitude of net compressive stresses to which the medium is subjected, and to its stress history. As confining stresses are increased in a sample holder, the rock's permeability decreases. The decrease observed, when confining stresses are increased from 200 or 300 psi to several thousand psi, varies considerably—from just a few percent for reasonably permeable, well-cemented samples, to an order of magnitude or more for low permeability samples that contain micro fractures. In general, it is desirable to approximate reservoir stresses in laboratory permeability measurements to obtain values closer to expected in situ reservoir permeabilities. Additional, smaller permeability decreases may also be observed as a function of time after confining stresses are raised—due to creep. Creep is most prevalent at compressive stresses just lower than those that cause sample failure.

6.1.2 Unit of Permeability

6.1.2.1 Traditional Unit

In order to make practical use of the concept that porous media possess the measurable property of permeability, an arbitrary standard unit, the "darcy," has been defined. A porous medium has a permeability of one darcy when a single-phase fluid of one centipoise viscosity that completely fills the voids of the medium will flow through it under "conditions of Stokes flow" at a rate of 1 cm^3/s per square centimeter of cross-sectional area under a pressure or equivalent hydraulic gradient of 1 atm/cm. "Condition of Stokes flow" shall mean simply that "the rate of flow be sufficiently low to be directly proportional to the pressure or hydraulic gradient." The unit of permeability (the darcy), as a coefficient of proportionality between physical quantities, possesses dimensions of length squared. Permeability represents a property of the medium alone and is independent of the fluid, except as hereinafter qualified. For convenience the millidarcy sub-unit (equal to 0.001 darcy) may be used. The spelling of the plural forms of the unit have been standardized and established in the literature as "darcys" and "millidarcys." Although other sub-units are not recommended, the microdarcy (equal to 0.001 millidarcy or 10^{-6} darcy) is often used in conjunction with low-permeability gas sands. Also, the nanodarcy (equal to 0.001 microdarcy or 10^{-9} darcy) is often referred to for very tight rocks, such as micro fractured granite.

6.1.2.2 SI Unit

The fundamental SI unit of permeability, m^2, is defined as follows: a permeability of one meter squared will permit a flow of 1 m^3/s of fluid of 1 Pa·s viscosity through an area of 1 m^2 under a pressure gradient of 1 Pa/m. One darcy equals 0.986923×10^{-12} m^2. Because of the small exponent, this is an awkward unit for common usage. The Society of Petroleum Engineers of AIME preferred permeability unit is the micrometer squared (μm^2). One darcy (the traditional unit) equals 0.986923 μm^2.

6.1.2.3 Typical Units Used in Hydrology

Most environmental engineers have a background in hydrology, in which the terms intrinsic permeability, hydraulic conductivity, and transmissivity are used. The physics of fluid flow that environmental engineers, core analysts, or petroleum engineers deal with is the same, but the language used by one group is often foreign to another group. This arises from different applications. Hydrologists most often deal with the flow of ground water in aquifers, in which water viscosity, density, and compressibility exhibit rather small variations. In these aquifers, it is often convenient to determine flow potential differences by measuring differences in liquid heads—the elevations to which water rises in small non-flowing test wells drilled into the aquifer. In the laboratory, an analogous measurement technique would be to attach a series of water manometers to a core sample through which water is flowing. The height of water in the manometer nearest the upstream end of the core would be greatest, and water-level elevations (from a horizontal reference) would be progressively lower further downstream.

In petroleum reservoirs, fluid properties vary widely, more than one fluid phase is usually present, and pressures are often too great to make manometric head measurements. Pressures are measured directly with pressure gauges or transducers. Thus, equations for these applications deal with pressures, not heads. Flow potentials must be calculated from pressures, flow rates, and fluid and rock properties through appropriate governing partial differential equations. Solution often requires numerical finite difference approximations of these equations.

6.1.2.3.1 Flow Potential Differences vs. Pressure Differences

It is important to understand that flow potential difference, not pressure difference, is the driving force for fluid flow, to know what flow potential is, and to ascertain when pressure differences can legitimately be used in flow equations. Relationships between flow potential and pressure are illustrated by Figure 6-1. Two tanks, each open to atmospheric pressure at its top and fitted with manometer tubes at several depths, are connected together by a small pipe. Each is partially filled with water to the same depth. Pressure in each tank at the air-water interface is atmospheric, and increases with depth.

We observe that the water level in every manometer tube is the same, and except for a very small capillary rise in each tube, it is the same as the water level inside the tanks. This water level in the manometer tubes is a measure of the flow potential inside the tanks, that is the same at every depth, even though pressure increases with depth. If pressure, alone, were the driving force for flow, higher pressure water from the bottom of a tank would flow upward, toward lower pressure. This is not observed. If we assume that the density of water everywhere in each tank is constant (a very close approximation), then flow potential, φ, everywhere is:

$$\varphi = p - \rho g z / C_4 \tag{1}$$

Where:

p = pressure at depth z (below the water surface; z increases downward).

ρ = density of the water.

g = local acceleration of gravity.

C_4 = conversion factor (found in Table 6-1) to make units consistent.

However, pressure at any depth in the tank is:

$$p = p_1 + \rho g z / C_4 \tag{2}$$

Where p_1 is the pressure at the air-water interface, atmospheric pressure in this case. Thus, combining Equations 1 and 2, we see that $\varphi = p_1$, and flow potential is the same at any depth. It is also the same in both tanks; so no flow occurs within or between tanks.

Condition 1:
- a. Both tanks are filled with water to same depth, and are open to atmospheric pressure.
- b. Water pressure increases in both tanks with increasing depth.
- c. However, flow potential (indicated by water levels in manometer tubes) is the same for all depths, and is the same in both tanks.
- d. Therefore, no flow occurs within or between tanks.

Condition 2:
- a. Air pressure above water in left tank is increased. Then vent is capped.
- b. Flow potential in left tank is increased as indicated by increased water levels in manometer tubes there.
- c. Water flows from left tank to right tank until water levels in manometer tubes are the same for both tanks.

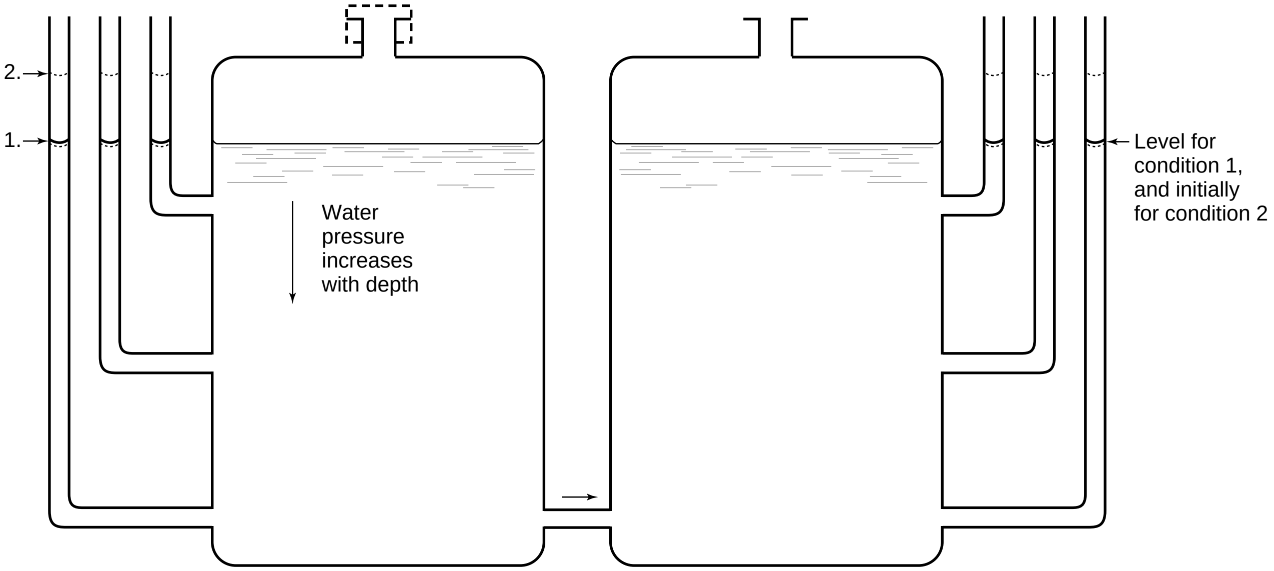

Figure 6-1—Illustration of Flow Potential vs. Pressure Differences

Table 6-1—Constants in Forchheimer or Darcy Equation with Various Sets of Units

Variable or Constant	Units or Values of Constants			
	SI	Preferred SPE	Traditional	Common Usage
A, area perpendicular to flow	m^2	m^2	cm^2	cm^2
b, Klinkenberg slip factor	Pa	Pa	atm	psi
D, diameter of sample	m	m	cm	cm
g, gravitational acceleration	m/s^2	m/s^2	cm/s^2	cm/s^2
k, permeability	m^2		darcy	millidarcy
L, length of sample	m	m	cm	cm
M, gas molecular weight	kg/kg-mole	kg/kg-mole	g/g-mole	g/g-mole
P, absolute pressure	Pa	Pa	atm	psia
p, gauge pressure	Pa	Pa	atm	psig
Δp, differential pressure	Pa	Pa	atm	psi
q, volumetric flow rate				
r, radius of sample or seal	m	m	cm	cm
S, compressive storage				
s, flow-direction distance	m	m	cm	cm
T, absolute temperature	K	K	K	K
V_p, pore volume				
V_T, total reservoir volume				
v_s, volumetric flux	m/s	m/s	cm/s	cm/s
β, inertial resistivity	m^{-1}	m^{-1}	cm^{-1}	ft.$^{-1}$
ρ, gas or liquid density	kg/m^3	kg/m^3	g/cm^3	g/cm^3
μ, gas or liquid viscosity	Pa·s	Pa·s	cp	cp
R, gas-law constant	8314	8314	82.05	1205.8
C_1, in Darcy or Forch. eq.	1.0	1.0	1.0	6.8046E-2
C_2, in Darcy or Forch. eq.	1.0	1.0E+12	1.0	1000.
C_3, constant in Forch. eq.	1.0	1.0	9.8692E-7	3.2379E-8
C_4, constant in eqs. with g	1.0	1.0	1.0133E+6	68 950.

Now, suppose air is pumped into the left tank through its vent so that air pressure above the water is greater than atmospheric pressure. Then the vent is capped. We would observe that water levels in all manometers attached to the left tank have risen equally. Except for the amount of water required to increase levels in the tubes, the water level inside the tank is unchanged. Initially, conditions in the right tank are unchanged. From the higher manometer levels in the left tank (indicated by dashed lines in Figure 6-1), relative to those in the right tank, we conclude that flow potential is higher in the former, even though water levels inside the two tanks are the same. Indeed, water flows from left to right until manometer levels are the same for both tanks. When flow ceases, the water level inside the left tank will be lower than in the right tank.

In laboratory single-phase gas permeability measurements, “gas head” differences are generally smaller than the accuracy of pressure measurements, and can be ignored. Therefore pressure differences or pressure gradients are very close approximations of flow potential differences or gradients in any flow orientation (horizontal or vertical). Conditions for single-phase liquid measurements are discussed in detail in 6.2.2. Pressure differences or gradients can be substituted for flow potential differences or gradients only in special, restricted situations.

6.1.2.3.2 Intrinsic Permeability

Intrinsic Permeability, k, as used by hydrologists is identical to permeability defined in 6.1.2.2, even though it is defined in terms of heads and kinematic viscosity, v, rather than dynamic viscosity, μ, used in the Darcy equation as defined in 6.1.2.1. Kinematic viscosity is equal to dynamic viscosity divided by density of the fluid. A porous medium has an intrinsic permeability of one unit of length squared if it will transmit in unit time a unit volume of fluid of unit kinematic viscosity through a cross section of unit area measured at right angles to the flow direction under a unit potential gradient. If SI units are used, intrinsic permeability has the unit of m^2.

6.1.2.3.3 Hydraulic Conductivity

Hydraulic conductivity, K, is not solely a property of a porous medium. It depends also upon the kinematic viscosity of the flowing fluid, and is appropriate only for ground water aquifers. Its units are those of velocity, usually feet per day or meters per day. A medium has a hydraulic conductivity of unit length per unit time if it will transmit in unit time a unit volume of ground water at the prevailing viscosity through a cross section of unit area, measured at right angles to the direction of flow, under a hydraulic gradient of unit change in head through unit length of flow. Hydraulic conductivity is related to permeability (or intrinsic permeability) by:

$$K = \frac{k\rho g}{\mu} \tag{3}$$

Where k is permeability, and the other symbols are defined above. Any consistent set of units may be used in Equation 3. With SI units, K will have the unit of meters per second.

6.1.2.3.4 Transmissivity

The transmissivity, T, of a ground water aquifer includes the thickness of the aquifer, b, and the kinematic viscosity of the confined water. Transmissivity is the rate at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient. It has the unit of length squared per unit time, often ft^2/day, or m^2/day. Transmissivity is equal to Kb. It can be calculated from permeability by:

$$T = \frac{k\rho g b}{\mu} \tag{4}$$

Definitions for intrinsic permeability, hydraulic conductivity, and transmissivity are taken from Lohman.[42] Conversion factors for several sets of units can be found in *Ground Water* (1993).[43]

6.1.3 Terminology

6.1.3.1 Specific Permeability

The definition of the standard unit of permeability requires that the porous medium contain only one homogeneous, single-phase fluid. The property of the medium so determined is properly termed specific permeability; but it shall simply be called permeability, and denoted by the symbol k. By this definition, the term permeability becomes a property solely of the porous medium; and the numerical value of permeability is constant and independent of the fluid used in the measurement. Whenever certain minerals and fluids are brought in contact and interaction results, or material is deposited from the fluid, this phenomenon, in effect, produces a new medium. The permeability of the new medium may differ from that of the unaltered medium. Whenever this occurs, it should be indicated clearly by appropriate references to both medium and fluid, e.g., “the permeability of Woodbine sand to salt water (fresh water, crude oil, heptane, etc., as the case may be).”

Uncorrected gas permeabilities are precluded from the above definition due to gas slippage effects. Gas permeabilities, which shall be called k_g (or k_{air}, k_{N2}, k_{He}, etc.) depend upon the particular gas used in the permeability determination, and upon its mean pore pressure. Both must be specified to define the permeability of the medium. Only the Klinkenberg, slip-corrected gas permeability, when unaffected by high velocity (Forchheimer) effects, is constant and independent of the fluid used in the measurement.

Because the stress condition under which a measurement is made can have a significant effect upon permeability, it also should be specified. Examples are: k_{air} with a mean pore pressure of 32.3 psia and a radial confining stress of 400 psig; or k_∞ at a net hydrostatic stress of 5,000 psi. The first specification implies that a gas permeability was obtained using air at an average absolute pore pressure of 32.3 psia, and that a pressure of 400 psig was applied to the rubber boot of the sample holder. The net radial stress is therefore 400 + 14.7 – 32.3, or about 382 psi. The net axial stress is unknown. In the second example, a Klinkenberg (slip-corrected) permeability was obtained that is independent of the gas used or its mean pore pressure (except as pore pressure affects net stress). Equal magnitude radial and axial (“hydrostatic”) net stresses of 5,000 psi were applied during the measurement.

6.1.3.2 Effective and Relative Permeability

This manual of routine recommended practices deals only with single-phase permeability measurements. Therefore, effective permeability and relative permeability will only be defined. The effective permeability of a porous medium is a measure of its fluid conductivity to a particular phase of a multi-phase fluid system residing within the medium, where the saturation of each phase (the fraction of its total effective pore volume that is filled by each phase) is specified. Relative permeability is the ratio of the effective permeability of a particular fluid phase, at definite saturations of all phases, to some arbitrary reference permeability. The reference permeability can be the sample’s specific permeability, its Klinkenberg permeability, the effective permeability of one of the fluid phases at a specified saturation condition, etc. The particular reference permeability used must be specified.

Book three of the five volume, *History of Petroleum Engineering* (API, 1961),[39] is a rich reference source for early work on laboratory permeability measurements, including the pioneering efforts of Fancher, Lewis, and Barnes,[40] and of Wyckoff, Botset, Muskat, and Reed.[41]

6.2 THEORY

Single-phase permeability measurements can be separated into four major categories: those utilizing flowing gas or liquid under steady-state or unsteady-state (transient) conditions. Equations for steady-state flow of gases and liquids are presented in this section. Several practical variations, each with particular advantages and limitations, will be outlined in 6.3, and the theory and application of transient pressure gas and liquid measurements are presented in 6.4. These are all considered to be direct measurements.

The permeability of a porous medium can also be inferred from empirical correlations using indirect measurements, such as those of inter granular porosity and grain size (Kozeny[5] and Carman[6]); porosity and irreducible water saturation (Wyllie and Rose[7]); pore-throat and pore-size distribution by mercury injection or capillary pressure measurements (Swanson[8], Katz and Thompson[9], etc.); effective electrical conductivity (Archie[10], Ondracek[11]); transmissivity of acoustic waves (Biot[12]); electrokinetic emf (Moran[13]); etc. Because these can be less accurate and often more time consuming than making the direct measurements, they will not be considered here.

All measurements are assumed to be conducted under isothermal conditions, which may require considerable technique and effort to achieve under certain circumstances. Also, even though temperature can affect permeability, most routine permeability measurements are made at, or near, room temperature.

Tables 6-2 and 6-3 are quick selection and reference guides for gas and liquid permeability determinations, respectively, using both steady-state and transient techniques. They list major advantages and disadvantages of each technique, its approximate permeability range, and particular application. They are intended to be a starting point only, and the reader is cautioned to be aware of details given in sections referenced by these tables.

Table 6-2—Quick Selection and Reference Guide for Permeability Measurements Using Gases*

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.1.1 6.3.1.1.1.1	Axial flow, steady state in core plugs	0.1-10,000	Low pressure apparatus with manometers, orifice flow meters	Low capital cost; simple manual system; workhorse for decades; large data base for comparison	Labor intensive; high operating cost; low-stress perms; no slip correction; must check for inertial resistance
6.3.1.1 6.3.1.1.1.2	Axial flow, steady state in core plugs	0.1-10,000	Apparatus with electronic sensors, high pressure. core holder	Can be automated; reservoir stresses can be approximated; better precision and accuracy than with manual system	Must make multiple measurements for gas slippage correction; must check to ensure negligible inertial resistance
6.4.1.1 B.6.8.2	Axial flow, pressure falloff in core plugs	0.001-30,000	Wide range; med. to high stress measurements with corrections for b and β	Well adapted for automation; no flow meters required; can yield reservoir-condition perms (k_∞), and k_g	Higher capital cost for automated system with high accuracy pressure transducers and data acquisition system
6.4.1.3 D.6.8.4	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; porosity can be determined in same apparatus	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—higher capital cost
6.3.1.2	Probe perm., s.s., on whole core	1-10,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); relatively fast; can be automated or made portable	Zero stress, non slip corrected perms are high at low end of range; prone to high inertial resistance at high end
6.4.1.2 C.6.8.3	Probe perm., pressure falloff on whole core	0.001-30,000	Zero stress, high density, localized measurements for heterogeneous cores	No plug preparation required (core slabbing recommended); very fast; automated; corrected for b, β	Zero stress perms are high, especially at low end of range; higher capital cost for automated system
6.3.1.3	Transverse, s.s. perm. in whole core	0.02-500	Directional perm. in whole core (or plug) for k_{max} and k_{90*}	Can measure “horizontal” perm. in various directions; averaging obtained using whole-core sample.	Cleaning and preparation of whole core sample more expensive; only k_g obtained without multiple measurements
6.3.1.4	Radial, s. s. perm. in whole core	0.01-250	Average permeability in all radial directions in whole core samples	Measures average “horizontal” permeability in large sample	Difficult to prepare samples; no radial stress; perm. critically dependent on condition of central “wellbore”

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*Major advantages of using gas rather than liquid:

a. Easy to use—does not require special saturation techniques.
b. Non-reactive with rock; non-corrosive to equipment.
c. No post-measurement cleanup required.
d. Less prone than liquid to mobilizing fines in rock sample.
e. Does not support microbial growth, nor require special filtration.

Major disadvantages:

a. Requires correction for gas slippage—especially with lower perms.
b. Prone to significant high-velocity inertial resistance in high perm. rock.
c. Necessary leak-tightness harder to achieve than with liquids.
d. In some cases, may be less representative of permeability in reservoir.

Table 6-3—Quick Selection and Reference Guide for Permeability Measurements Using Liquids

Reference Section Numbers	Type of Measurement	Approx. Perm. Range, md	Apparatus or Application	Major Advantages	Major Limitations
6.3.2.2	Axial, s.s. flow in plugs using liquid heads	1000-40,000	Gravity flow of liquid through high perm., cylindrical core plugs	Simple, low cost equipment (but does require electronic balance for flow rate determination)	Low back pressure—difficult to ensure that no gas remains in core plug.
6.3.2.1	Axial flow, steady state in core plugs	0.1-20,000	Apparatus with electronic sensors, high pressure. core holder, imposed	Can be automated; reservoir stresses can be approximated; may be most representative measurement of perm. in reservior	Corrosion resistant, moderate to high pressure pumping and control equipment is expensive
6.4.2.1 E.6.8.5	Axial flow, pulse-decay in core plugs	.00001-0.1	High stress apparatus for very low perms.	Only method for ultra-low perms; well adapted for automation; no flow meters required: rate is calc. from Δp & t	Requires high pressure, leak-tight system with high quality transducers and data acquisition system—high capital cost
6.3.2.1 6.3.1.3.1	Transverse, s.s. flow in whole cores	0.005-500	Directional perm. in whole core (or plug) for k_{max} and k_{90^*}	Can measure "horizontal" perm. in various directions; averaging obtained using whole-core sample.	Same as for s.s. axial flow (above); cleaning, preparation and handling of whole core sample more expensive
6.3.1.4 6.3.1.4.2	Radial, steady state flow in whole cores	0.002-250	Average permeability in all radial directions in whole core samples	Measures average "horizontal" permeability in large sample	Same as above; difficult to prepare samples; no radial stress; perm. critically dependent on condition of central "wellbore"

6.2.1 Steady-State Permeability Equations For Gases

The general equation for steady-state gas permeability measurements will be presented first. It includes provision for gas slippage and inertial resistance effects, which become somewhat complicated and require more measurements than is usually practicable using steady-state measurements. However it serves to unify the theory of gas measurements and provides principles and guidelines for use of the Darcy equation, which is the basis for the practical steady-state cases that follow.

Unlike liquids, gases are highly compressible. Also, under normal laboratory conditions, the density of gas is sufficiently low that gravity effects may be ignored in making permeability measurements. The viscosity of air, nitrogen, or helium (see Tables 6-4, 6-5, and 6-6, respectively) is less than $^1/_{50}$ that of water at room temperature. Therefore, volumetric fluxes of gases are correspondingly higher for a given pressure gradient, and can deviate from conditions of Stokes flow, demanded by Darcy's Law. Finally, the phenomenon of gas slippage, or the Klinkenberg effect, requires special treatment. Because of these differences, the point of departure for developing equations pertaining to the measurement of permeability using gases is the differential form of the Forchheimer equation, into which the Klinkenberg relationship will be inserted:

$$\frac{-dP}{ds} = \frac{C_2 \mu q_s}{C_1 A k_g} + \frac{C_3 \beta \rho_s^2}{C_1 A^2} \qquad (5)$$

Where:

s = distance along the direction of flow.
q_s = volumetric rate of gas flow through area A per unit time.
A = area perpendicular to the flow of gas.
$\frac{dP}{ds}$ = pressure gradient along s to which q_s / A refers.
μ = dynamic gas viscosity.
k_g = apparent permeability of the medium to a particular gas.
β = coefficient of inertial resistivity.
ρ = gas density.

The constants C_1, C_2, and C_3 allow for various sets of dimensions to be used in the gas-flow equations that follow. Table 6-1 shows values of these constants for SI, preferred-SPE, traditional, or common-usage dimensions. Note that an upper case P appears in the equation above. This non-standard symbol denotes that the pressure is *absolute*. A lower-case p is used to denote a *gauge* or *differential* pressure in any equation below. These conventions have been adopted in this document because of the confusion between the two pressures, and the frequent error made when a measured gauge-pressure is not converted to an absolute pressure when required. Thus, in keeping with this convention:

$$P = p + P_a \qquad (6)$$

Where P_a is the true ambient barometric pressure, converted to the proper units (not the barometric pressure from the local airport, which has been converted to sea-level pressure).

The density of a real gas is:

$$\rho = \frac{MP}{zRT} \qquad (7)$$

Table 6-4—Viscosity of Air (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	176.8	179.6	182.3	185.0	187.7	190.4	171.8	176.8	181.8	186.6	191.4	196.2
1	177.1	179.9	182.6	185.3	188.0	190.6	172.3	177.3	182.3	187.1	191.9	196.6
2	177.4	180.1	182.9	185.6	188.2	190.9	172.8	177.8	182.8	187.6	192.4	197.1
3	177.7	180.4	183.1	185.8	188.5	191.2	173.3	178.3	183.2	188.1	192.9	197.6
4	177.9	180.7	183.4	186.1	188.8	191.4	173.8	178.8	183.7	188.6	193.3	198.0
5	178.2	181.0	183.7	186.4	189.0	191.7	174.3	179.3	184.2	189.0	193.8	198.5
6	178.5	181.2	184.0	186.6	189.3	192.0	174.8	179.8	184.7	189.5	194.3	198.9
7	178.8	181.5	184.2	186.9	189.6	192.2	175.3	180.3	185.2	190.0	194.7	199.4
8	179.0	181.8	184.5	187.2	189.8	192.5	175.8	180.8	185.7	190.5	195.2	199.9
9	179.3	182.1	184.8	187.4	190.1	192.8	176.3	181.3	186.2	191.0	195.7	200.3

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*Calculated from Sutherland's equation:

$$\mu_{air} = \frac{14.969T^{1.5}}{T + 120}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{air} is the viscosity of air at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on work of Montgomery[35] and Birge[36].

Table 6-5—Viscosity of Nitrogen (for Degrees F and C) at One Atmosphere*

Viscosity of air at one atmosphere, micropoises												
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	171.3	173.8	176.4	178.9	181.4	183.8	166.6	171.3	175.9	180.4	184.8	189.2
1	171.5	174.1	176.6	179.1	181.6	184.1	167.1	171.7	176.3	180.8	185.2	189.6
2	171.8	174.3	176.9	179.4	181.9	184.3	167.5	172.2	176.8	181.3	185.7	190.0
3	172.0	174.6	177.1	179.6	182.1	184.6	168.0	172.7	177.2	181.7	186.1	190.5
4	172.3	174.8	177.4	179.9	182.3	184.8	168.5	173.1	177.7	182.2	186.6	190.9
5	172.5	175.1	177.6	180.1	182.6	185.1	168.9	173.6	178.1	182.6	187.0	191.3
6	172.8	175.3	177.9	180.4	182.8	185.3	169.4	174.0	178.6	183.0	187.4	191.8
7	173.1	175.6	178.1	180.6	183.1	185.5	169.9	174.5	179.0	183.5	187.9	192.2
8	173.3	175.9	178.4	180.9	183.3	185.8	170.3	174.9	179.5	183.9	188.3	192.6
9	173.6	176.1	178.6	181.1	183.6	186.0	170.8	175.4	179.9	184.4	188.7	193.0

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*Calculated from Sutherland's equation:

$$\mu_{N_2} = \frac{13.85T^{1.5}}{T + 102}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{N_2} is the viscosity of nitrogen at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are based on the work of Licht and Stechert[37].
The viscosity of nitrogen at temperatures reasonably close to 25°C and pressures to 253 atmospheres can be calculated from:

$$\mu_{N_2}[T, P] =$$
$$\mu_{N_2}[T, 1] - 0.12474 + 0.123688P + 1.05452\text{E-}03P^2 - 1.5052\text{E-}06P^3$$

Where: $\mu_{N_2}[T, 1]$ is the viscosity of nitrogen at one atmosphere pressure (from the tables above), micropoises, and P is pressure, atmospheres. The pressure dependence is fitted to the data of Gracki, *et al.*[34] to within a maximum deviation of 0.1%. Pressure dependence at 0°C and 50°C is nearly the same as at 25°C.

Table 6-6—Viscosity of Helium (for Degrees F and C) at One Atmosphere*

	Viscosity of air at one atmosphere, micropoises											
	Temperature, °F						Temperature, °C					
plus	50°F	60°F	70°F	80°F	90°F	100°F	0°C	10°C	20°C	30°C	40°C	50°C
0	191.7	194.2	196.8	199.3	201.8	204.4	187.0	191.7	196.3	200.8	205.4	209.8
1	191.9	194.5	197.0	199.6	202.1	204.6	187.5	192.1	196.7	201.3	205.8	210.3
2	192.2	194.7	197.3	199.8	202.3	204.9	187.9	192.6	197.2	201.7	206.3	210.7
3	192.4	195.0	197.5	200.1	202.6	205.1	188.4	193.1	197.6	202.2	206.7	211.2
4	192.7	195.3	197.8	200.3	202.9	205.4	188.9	193.5	198.1	202.7	207.1	211.6
5	193.0	195.5	198.1	200.6	203.1	205.6	189.3	194.0	198.6	203.1	207.6	212.0
6	193.2	195.8	198.3	200.8	203.4	205.9	189.8	194.4	199.0	203.6	208.0	212.5
7	193.5	196.0	198.6	201.1	203.6	206.1	190.3	194.9	199.5	204.0	208.5	212.9
8	193.7	196.3	198.8	201.3	203.9	206.4	190.7	195.4	199.9	204.5	208.9	213.4
9	194.0	196.5	199.1	201.6	204.1	206.6	191.2	195.8	200.4	204.9	209.4	213.8

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*Calculated from:

$$\mu_{He} = 187.0\left(\frac{T}{273.1}\right)^{0.685}$$

Where: T is absolute temperature, degrees Kelvin (degrees Celsius + 273), and μ_{He} is the viscosity of helium at one atmosphere pressure, micropoises. Multiply by 1.0 E-04 to convert to centipoises, or by 1.0 E-07 to convert to Pa·s. Constants are from Chapman and Cowling[38].
According to the data of Gracki, *et al.*[34] the viscosity of helium at 25°C passes through a very shallow minimum with pressure. At 37 atm. it is 0.43 percent lower than at 1 atm., and at 158 atm., its viscosity is 0.17 percent higher than at 1 atm.

Where z is the gas deviation factor, which is equal to 1.0 for an ideal gas. Values of z as functions of temperature and pressure are given in Tables 6-7, 6-8, and 6-9 for air, nitrogen, and helium, respectively. They are calculated from the Beattie-Bridgeman equation of state[14]. See Table 6-1 for values of the universal gas-law constant, R, for the various sets of units. M is the gas molecular weight. Temperature in Equation 7 is absolute, in degrees Kelvin:

$$\begin{aligned} T &= {}^\circ\mathrm{C} + 273.15 \\ &= ({}^\circ\mathrm{F} - 32)/1.8 + 273.15 \end{aligned} \tag{8}$$

6.2.1.1 Treatment of the Gas Slippage Factor

The Klinkenberg relationship, written as a point function (i.e., pertaining to a particular point in a sample, not an average value) is:

$$k_g = k_\infty\left(1 + \frac{b}{P}\right) \tag{9}$$

Note that k_g depends upon pressure. Therefore it cannot be treated as a constant when the Forchheimer (or Darcy) equation is integrated. In Equation 9, b must have the same units as those of the pressure used.

Unfortunately, the gas slippage factor, b, is partly a rock property and partly a gas property, which has caused confusion and some problems. For example, a b measured using air, but not indicated as such, might be misused in calculations involving a different gas with significantly different properties. It is a fairly simple procedure to separate the gas properties from the slip factor, so that only the rock-dependent property remains. According to Klinkenberg[3], b is related to the mean free path of gas molecules, $\bar{\lambda}$, by the relationship:

$$\bar{\lambda} = \frac{br}{4cP} \tag{10}$$

But, from the kinetic gas theory[15], the mean free path is given by:

$$\bar{\lambda} = \frac{1.881\mu}{P}\sqrt{\frac{RT}{M}} \tag{11}$$

In Equations 10 and 11, any consistent units may be used. From these two equations, and adjusting for the units given below, we obtain:

$$b = \left[99.5\mu\sqrt{\frac{T}{M}}\right]\left(\frac{c}{r}\right) \tag{12}$$

Where:
P = absolute gas pressure.
R = universal gas law constant.
b = Klinkenberg gas slippage factor, psi. (The constant 99.5 becomes 6.77 if b has the unit of atm).
μ = gas viscosity, cp.

Table 6-7—Gas Law Deviation Factors for Air, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:							
psia	55	60	65	70	75	80	85	90
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99968	0.99970	0.99972	0.99973	0.99975	0.99977	0.99979	0.99980
20	0.99935	0.99939	0.99943	0.99947	0.99951	0.99954	0.99957	0.99961
30	0.99904	0.99910	0.99915	0.99921	0.99926	0.99931	0.99936	0.99941
40	0.99872	0.99880	0.99888	0.99895	0.99902	0.99909	0.99916	0.99922
50	0.99840	0.99850	0.99860	0.99869	0.99878	0.99887	0.99895	0.99903
60	0.99809	0.99821	0.99833	0.99844	0.99854	0.99865	0.99875	0.99884
70	0.99778	0.99792	0.99806	0.99818	0.99831	0.99843	0.99854	0.99866
80	0.99747	0.99763	0.99779	0.99793	0.99808	0.99821	0.99834	0.99847
90	0.99717	0.99735	0.99752	0.99769	0.99784	0.99800	0.99815	0.99829
100	0.99687	0.99706	0.99726	0.99744	0.99762	0.99779	0.99795	0.99811
110	0.99657	0.99678	0.99699	0.99720	0.99739	0.99758	0.99776	0.99793
120	0.99627	0.99651	0.99673	0.99695	0.99717	0.99737	0.99757	0.99776
130	0.99597	0.99623	0.99648	0.99671	0.99694	0.99716	0.99738	0.99758
140	0.99568	0.99596	0.99622	0.99648	0.99672	0.99696	0.99719	0.99741
150	0.99539	0.99568	0.99597	0.99624	0.99651	0.99676	0.99701	0.99724
160	0.99510	0.99541	0.99572	0.99572	0.99601	0.99601	0.99629	0.99656
170	0.99481	0.99515	0.99547	0.99578	0.99608	0.99637	0.99664	0.99691
180	0.99453	0.99488	0.99522	0.99555	0.99587	0.99617	0.99646	0.99675
190	0.99425	0.99462	0.99498	0.99533	0.99566	0.99598	0.99629	0.99659
200	0.99397	0.99436	0.99474	0.99510	0.99545	0.99579	0.99611	0.99643
210	0.99369	0.99410	0.99450	0.99488	0.99525	0.99560	0.99594	0.99627
220	0.99342	0.99385	0.99426	0.99466	0.99505	0.99542	0.99577	0.99612
230	0.99315	0.99360	0.99403	0.99445	0.99485	0.99523	0.99560	0.99596
240	0.99288	0.99335	0.99380	0.99423	0.99465	0.99505	0.99544	0.99581
250	0.99261	0.99310	0.99357	0.99402	0.99445	0.99487	0.99528	0.99566
260	0.99235	0.99285	0.99334	0.99381	0.99426	0.99470	0.99511	0.99552
300	0.99132	0.99190	0.99246	0.99299	0.99351	0.99401	0.99449	0.99496
400	0.98891	0.98968	0.99041	0.99112	0.99180	0.99246	0.99.09	0.99370
500	0.98676	0.98770	0.98861	0.98948	0.99032	0.99113	0.99190	0.99265
600	0.98486	0.98598	0.98705	0.98808	0.98907	0.99002	0.99094	0.99183
700	0.98322	0.98450	0.98573	0.98692	0.98805	0.98915	0.99020	0.99122
800	0.98185	0.98329	0.98467	0.98600	0.98728	0.98850	0.98968	0.99082
900	0.98074	0.98233	0.98386	0.98532	0.98673	0.98809	0.98939	0.99065
1000	0.97989	0.98163	0.98329	0.98489	0.98642	0.98790	0.98932	0.99069
1100	0.97931	0.98118	0.98297	0.98470	0.98635	0.98794	0.98947	0.99094
1200	0.97900	0.98099	0.98290	0.98474	0.98651	0.98821	0.98984	0.99141
1300	0.97894	0.98105	0.98308	0.98503	0.98690	0.98869	0.99042	0.99208
1400	0.97915	0.98137	0.98350	0.98555	0.98751	0.98940	0.99122	0.99296
1500	0.97961	0.98193	0.98416	0.98630	0.98835	0.99033	0.99223	0.99405
1600	0.98032	0.98273	0.98505	0.98728	0.98941	0.99147	0.99344	0.99534
1700	0.98128	0.98378	0.98618	0.98848	0.99069	0.99281	0.99485	0.99682
1800	0.98248	0.98505	0.98752	0.98990	0.99218	0.99436	0.99647	0.99849
1900	0.98391	0.98656	0.98909	0.99153	0.99387	0.99611	0.98827	1.00034
2000	0.98558	0.98828	0.99088	0.99337	0.99576	0.99805	1.00026	1.00238
2100	0.98747	0.99022	0.99287	0.99540	0.99784	1.00018	1.00243	1.00460
2200	0.98957	0.99237	0.99506	0.99764	1.00011	1.00249	1.00478	1.00698
2300	0.99189	0.99472	0.99744	1.00006	1.00257	1.00498	1.00730	1.00953
2400	0.99440	0.99727	1.00002	1.00266	1.00520	1.00763	1.00998	1.01223
2500	0.99711	1.00000	1.00277	1.00543	1.00799	1.01045	1.01282	1.01509
2600	1.00000	1.00291	1.00570	1.00838	1.01095	1.01343	1.01581	1.01810
2700	1.00307	1.00599	1.00879	1.01148	1.01407	1.01655	1.01894	1.02124
2800	1.00631	1.00923	1.01204	1.01474	1.01733	1.01983	1.02222	1.02794
2900	1.00971	1.01264	1.01545	1.01815	1.02074	1.02323	1.02563	1.02794
3000	1.01327	1.01619	1.01900	1.02169	1.02428	1.02678	1.02917	1.03147

P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99957	0.99962	0.99966	0.99969	0.99973	0.99976	0.99979	0.99982
20	0.99915	0.99923	0.99931	0.99939	0.99946	0.99952	0.99958	0.99964
30	0.99873	0.99886	0.99897	0.99908	0.99919	0.99928	0.99937	0.99946
40	0.99831	0.99848	0.99864	0.99878	0.99892	0.99905	0.99917	0.99928
50	0.99790	0.99811	0.99830	0.99848	0.99866	0.99882	0.99897	0.99911
60	0.99748	0.99773	0.99797	0.99819	0.99839	0.99859	0.99876	0.99893
70	0.99707	0.99736	0.99764	0.99789	0.99813	0.99836	0.99857	0.99876
80	0.99666	0.99700	0.99731	0.99760	0.99788	0.99813	0.99837	0.99859
90	0.99626	0.99663	0.99698	0.99731	0.99762	0.99791	0.99818	0.99843
100	0.99585	0.99627	0.99666	0.99703	0.99737	0.99769	0.99798	0.99826
110	0.99545	0.99591	0.99634	0.99674	0.99712	0.99747	0.99779	0.99810
120	0.99505	0.99555	0.99602	0.99646	0.99687	0.99725	0.99761	0.99794
130	0.99466	0.99520	0.99571	0.99618	0.99662	0.99703	0.99742	0.99778
140	0.99426	0.99485	0.99539	0.99590	0.99638	0.99682	0.99724	0.99762
150	0.99387	0.99450	0.99508	0.99563	0.99613	0.99661	0.99705	0.99747
160	0.99349	0.99415	0.99477	0.99535	0.99589	0.99640	0.99687	0.99732
170	0.99310	0.99381	0.99447	0.99508	0.99566	0.99619	0.99670	0.99717
180	0.99272	0.99347	0.99416	0.99481	0.99542	0.99599	0.99652	0.99702
190	0.99234	0.99313	0.99386	0.99455	0.99519	0.99579	0.99635	0.99687
200	0.99196	0.99279	0.99356	0.99428	0.99496	0.99559	0.99618	0.99673
210	0.99159	0.99246	0.99327	0.99402	0.99473	0.99539	0.99601	0.99659
220	0.99121	0.99212	0.99297	0399377	0.99450	0.99520	0.99584	0.99645
230	0.99084	0.99180	0.99268	0.99351	0.99428	0.99500	0.99568	0.99631
240	0.99048	0.99147	0.99239	0.99326	0.99406	0.99481	0.99552	0.99617
250	0.99011	0.99115	0.99211	0.99300	0.99384	0.99462	0.99535	0.99604
260	0.98975	0.99082	0.99182	0.99275	0.99362	0.99444	0.99520	0.99591
300	0.98833	0.98957	0.99071	0.99178	0.99278	0.99371	0.99459	0.99540
400	0.98498	0.98660	0.98812	0.98952	0.99084	0.99207	0.99321	0.99428
500	0.98190	0.98391	0.98578	0.98751	0.98913	0.99064	0.99206	0.99337
600	0.97911	0.98149	0.98370	0.98576	0.98767	0.98945	0.99112	0.99268
700	0.97662	0.97935	0.98189	0.98425	0.98645	0.98850	0.99041	0.99219
800	0.97442	0.97750	0.98035	0.98301	0.98547	0.98777	0.98992	0.99192
900	0.97253	0.97593	0.97908	0.98202	0.98474	0.98728	0.98965	0.99185
1000	0.97093	0.97465	0.97809	0.98129	0.98426	0.98702	0.98960	0.99200
1100	0.96965	0.97366	0.97737	0.98081	0.98401	0.98699	0.98977	0.99235
1200	0.96867	0.97295	0.97692	0.98060	0.98402	0.98719	0.99016	0.99291
1300	0.96800	0.97254	0.97674	0.98064	0.98426	0.98762	0.99076	0.99368
1400	0.96763	0.97241	0.97683	0.98093	0.98474	0.98828	0.99157	0.99464
1500	0.96756	0.97256	0.97718	0.98147	0.98545	0.98915	0.99260	0.99580
1600	0.96779	0.97299	0.97780	0.98226	0.98640	0.99024	0.99382	0.99716
1700	0.96832	0.97369	0.97867	0.98329	0.98757	0.99155	0.99525	0.99870
1800	0.96913	0.97466	0.97979	0.98455	0.98896	0.99306	0.99688	1.00043
1900	0.97021	0.97590	0.98116	0.98604	0.99057	0.99478	0.99869	1.00234
2000	0.97157	0.97738	0.98276	0.98775	0.99238	0.99669	1.00069	1.00442
2100	0.97320	0.97912	0.98460	0.98968	0.99440	0.99879	1.00287	1.00668
2200	0.97508	0.98109	0.98666	0.99182	0.99662	1.00108	1.00523	1.00909
2300	0.97720	0.98329	0.98893	0.99416	0.99902	1.00354	1.00775	1.01167
2400	0.97957	0.98571	0.99141	0.99670	1.00161	1.00618	1.01044	1.01440
2500	0.98216	0.98835	0.99410	0.99943	1.00438	1.00899	1.01328	1.01728
2600	0.98497	0.99120	0.99697	1.00233	1.00732	1.01196	1.01627	1.02030
2700	0.98799	0.99423	1.00003	1.00541	1.01042	1.01507	1.01941	1.02345
2800	0.99120	0.99746	1.00327	1.00866	1.01367	1.01834	1.02269	1.02674
2900	0.99461	1.00086	1.00667	1.01206	1.01708	1.02175	1.02610	1.03016
3000	0.99820	1.00444	1.01023	1.01562	1.02063	1.02529	1.02964	1.03369

Table 6-8—Gas Law Deviation Factors for Nitrogen, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:								P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	55	60	65	70	75	80	85	90	psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	0.99975	0.99977	0.99979	0.99981	0.99983	0.99984	0.99986	0.99987	10	0.99965	0.99969	0.99973	0.99977	0.99980	0.99983	0.99986	0.99989
20	0.99950	0.99954	0.99958	0.99962	0.99965	0.99969	0.99972	0.99975	20	0.99930	0.99939	0.99946	0.99954	0.99960	0.99967	0.99973	0.99978
30	0.99926	0.99932	0.99938	0.99943	0.99948	0.99953	0.99958	0.99963	30	0.99896	0.99908	0.99920	0.99931	0.99941	0.99950	0.99959	0.99968
40	0.99902	0.99910	0.99917	0.99925	0.99932	0.99938	0.99945	0.99951	40	0.99862	0.99878	0.99894	0.99908	0.99922	0.99934	0.99946	0.99957
50	0.99878	0.99888	0.99897	0.99907	0.99915	0.99924	0.99932	0.99940	50	0.99828	0.99849	0.99868	0.99886	0.99903	0.99919	0.99933	0.99947
60	0.99855	0.99867	0.99878	0.99889	0.99899	0.99909	0.99919	0.99928	60	0.99795	0.99819	0.99843	0.99864	0.99884	0.99903	0.99921	0.99937
70	0.99832	0.99845	0.99858	0.99871	0.99883	0.99895	0.99906	0.99917	70	0.99761	0.99790	0.99817	0.99843	0.99866	0.99888	0.99909	0.99928
80	0.99809	0.99824	0.99839	0.99854	0.99868	0.99881	0.99894	0.99906	80	0.99729	0.99762	0.99792	0.99821	0.99848	0.99873	0.99896	0.99918
90	0.99786	0.99804	0.99820	0.99837	0.99852	0.99867	0.99882	0.99896	90	0.99696	0.99733	0.99768	0.99800	0.99830	0.99858	0.99885	0.99909
100	0.99764	0.99783	0.99802	0.99820	0.99837	0.99854	0.99870	0.99885	100	0.99664	0.99705	0.99744	0.99779	0.99813	0.99844	0.99873	0.99900
110	0.99742	0.99763	0.99784	0.99803	0.99822	0.99841	0.99858	0.99875	110	0.99632	0.99677	0.99719	0.99759	0.99795	0.99830	0.99862	0.99892
120	0.99720	0.99743	0.99766	0.99787	0.99808	0.99828	0.99847	0.99866	120	0.99601	0.99650	0.99696	0.99739	0.99779	0.99816	0.99851	0.99883
130	0.99698	0.99724	0.99748	0.99771	0.99793	0.99815	0.99836	0.99856	130	0.99569	0.99623	0.99672	0.99719	0.99762	0.99802	0.99840	0.99875
140	0.99677	0.99704	0.99730	0.99755	0.99779	0.99803	0.99825	0.99847	140	0.99538	0.99596	0.99649	0.99699	0.99745	0.99789	0.99829	0.99867
150	0.99656	0.99685	0.99713	0.99740	0.99766	0.99790	0.99814	0.99837	150	0.99508	0.99569	0.99626	0.99680	0.99729	0.99776	0.99819	0.99860
160	0.99636	0.99667	0.99696	0.99725	0.99752	0.99779	0.99804	0.99829	160	0.99478	0.99543	0.99604	0.99660	0.99713	0.99763	0.99809	0.99852
170	0.99615	0.99648	0.99680	0.99710	0.99739	0.99767	0.99794	0.99820	170	0.99448	0.99517	0.99582	0.99642	0.99698	0.99750	0.99799	0.99845
180	0.99595	0.99630	0.99663	0.99695	0.99726	0.99756	0.99784	0.99812	180	0.99418	0.99491	0.99560	0.99623	0.99683	0.99738	0.99790	0.99838
190	0.99576	0.99612	0.99647	0.99681	0.99713	0.99744	0.99775	0.99804	190	0.99389	0.99466	0.99538	0.99605	0.99667	0.99726	0.99780	0.99831
200	0.99556	0.99595	0.99631	0.99667	0.99701	0.99734	0.99765	0.99796	200	0.99360	0.99441	0.99517	0.99587	0.99653	0.99714	0.99771	0.99825
210	0.99537	0.99577	0.99616	0.99653	0.99689	0.99723	0.99756	0.99788	210	0.99331	0.99416	0.99496	0.99569	0.99638	0.99703	0.99763	0.99819
220	0.99518	0.99560	0.99601	0.99639	0.99677	0.99713	0.99747	0.99781	220	0.99303	0.99392	0.99475	0.99552	0.99624	0.99691	0.99754	0.99813
230	0.99500	0.99544	0.99586	0.99626	0.99665	0.99703	0.99739	0.99774	230	0.99275	0.99368	0.99454	0.99535	0.99610	0.99680	0.99746	0.99807
240	0.99482	0.99527	0.99571	0.99613	0.99654	0.99693	0.99730	0.99767	240	0.99247	0.99344	0.99434	0.99518	0.99596	0.99669	0.99738	0.99802
250	0.99464	0.99511	0.99557	0.99600	0.99643	0.99683	0.99722	0.99760	250	0.99220	0.99321	0.99414	0.99502	0.99583	0.99659	0.99730	0.99796
260	0.99446	0.99495	0.99542	0.99588	0.99632	0.99674	0.99715	0.99754	260	0.99193	0.99298	0.99395	0.99485	0.99570	0.99649	0.99722	0.99791
300	0.99378	0.99435	0.99489	0.99541	0.99591	0.99639	0.99686	0.99731	300	0.99089	0.99208	0.99320	0.99424	0.99520	0.99611	0.99695	0.99774
400	0.99230	0.99304	0.99375	0.99443	0.99508	0.99571	0.99632	0.99691	400	0.98851	0.99008	0.99154	0.99289	0.99416	0.99534	0.99644	0.99747
500	0.99112	0.99202	0.99289	0.99372	0.99453	0.99530	0.99604	0.99675	500	0.98647	0.98839	0.99018	0.99185	0.99339	0.99484	0.99618	0.99744
600	0.99024	0.99130	0.99232	0.99330	0.99424	0.99514	0.99601	0.99685	600	0.98478	0.98704	0.98914	0.99109	0.99291	0.99460	0.99618	0.99766
700	0.98967	0.99088	0.99204	0.99315	0.99422	0.99525	0.99624	0.99720	700	0.98343	0.98602	0.98841	0.99064	0.99271	0.99464	0.99644	0.99811
800	0.98940	0.99074	0.99204	0.99328	0.99447	0.99562	0.99672	0.99779	800	0.98244	0.98532	0.98800	0.99048	0.99279	0.99494	0.99694	0.99881
900	0.98943	0.99091	0.99232	0.99368	0.99499	0.99625	0.99746	0.99862	900	0.98180	0.98496	0.98789	0.99061	0.99315	0.99550	0.99769	0.99974
1000	0.98976	0.99136	0.99289	0.99436	0.99577	0.99713	0.99844	0.99969	1000	0.98151	0.98493	0.98810	0.99104	0.99378	0.99632	0.99869	1.00090
1100	0.99039	0.99209	0.99373	0.99530	0.99681	0.99826	0.99966	1.00100	1100	0.98156	0.98522	0.98861	0.99176	0.99468	0.99740	0.99993	1.00229
1200	0.99131	0.99311	0.99484	0.99651	0.99811	0.99964	1.00112	1.00254	1200	0.98196	0.98583	0.98942	0.99276	0.99585	0.99873	1.00141	1.00390
1300	0.99251	0.99441	0.99623	0.99797	0.99965	1.00126	1.00281	1.00430	1300	0.98270	0.98676	0.99053	0.99403	0.99728	1.00030	1.00312	1.00574
1400	0.99399	0.99597	0.99787	0.99969	1.00144	1.00312	1.00474	1.00629	1400	0.98376	0.98800	0.99193	0.99558	0.99897	1.00212	1.00505	1.00778
1500	0.99575	0.99780	0.99976	1.00165	1.00346	1.00521	1.00688	1.00849	1500	0.98515	0.98955	0.99362	0.99740	1.00091	1.00417	1.00721	1.01004
1600	0.99777	0.99988	1.00191	1.00385	1.00572	1.00751	1.00924	1.01090	1600	0.98686	0.99138	0.99558	0.99947	1.00308	1.00645	1.00958	1.01249
1700	1.00005	1.00221	1.00429	1.00628	1.00820	1.01004	1.01181	1.01351	1700	0.98886	0.99350	0.99780	1.00179	1.00550	1.00894	1.01215	1.01514
1800	1.00258	1.00479	1.00690	1.00894	1.01089	1.01277	1.01458	1.01631	1800	0.99117	0.99589	1.00028	1.00435	1.00813	1.01165	1.01493	1.01798
1900	1.00535	1.00759	1.00974	1.01181	1.01379	1.01570	1.01754	1.01930	1900	0.99375	0.99855	1.00301	1.00715	1.01099	1.01457	1.01790	1.02100
2000	1.00834	1.01061	1.01279	1.01489	1.01690	1.01883	1.02069	1.02248	2000	0.99661	1.00147	1.00598	1.01017	1.01406	1.01768	1.02105	1.02419
2100	1.01156	1.01385	1.01605	1.01816	1.02019	1.02214	1.02402	1.02582	2100	0.99973	1.00463	1.00918	1.01340	1.01733	1.02098	1.02439	1.02756
2200	1.01499	1.01729	1.01950	1.02163	1.02367	1.02563	1.02752	1.02934	2200	1.00310	1.00802	1.01259	1.01684	1.02079	1.02447	1.02789	1.03108
2300	1.01862	1.02093	1.02315	1.02528	1.02733	1.02930	1.03119	1.03301	2300	1.00670	1.01163	1.01622	1.02047	1.02444	1.02812	1.03156	1.03476
2400	1.02244	1.02475	1.02697	1.02910	1.03115	1.03312	1.03501	1.03684	2400	1.01053	1.01546	1.02004	1.02430	1.02826	1.03195	1.03538	1.03859
2500	1.02645	1.02875	1.03096	1.03309	1.03513	1.03710	1.03899	1.04081	2500	1.01458	1.01949	1.02405	1.02830	1.03225	1.03593	1.03936	1.04256
2600	1.03063	1.03292	1.03512	1.03724	1.03927	1.04123	1.04311	1.04492	2600	1.01882	1.02371	1.02824	1.03247	1.03640	1.04006	1.04348	1.04666
2700	1.03497	1.03725	1.03943	1.04153	1.04355	1.04550	1.04736	1.04916	2700	1.02326	1.02371	1.00824	1.03247	1.03640	1.04006	1.04348	1.04666
2800	1.03947	1.04173	1.04389	1.04597	1.04797	1.04990	1.05175	1.05353	2800	1.02788	1.03267	1.03713	1.04128	1.04515	1.04875	1.05211	1.05524
2900	1.04412	1.04635	1.07849	1.05054	1.05252	1.05443	1.05626	1.05802	2900	1.03267	1.03740	1.04181	1.04591	1.04973	1.05329	1.05661	1.05971
3000	1.04891	1.05110	1.05321	1.05524	1.05720	1.05907	1.06088	1.06262	3000	1.03762	104228	1.04663	1.05067	1.05444	1.05796	1.06123	1.06429

Table 6-9—Gas Law Deviation Factors for Helium, Calculated from Beattie-Bridgemann Equation of State[14]

P,	Z-Factors for Air at Temperatures (Degrees F) of:								P,	Z-Factors for Air at Temperatures (Degrees C) of:							
psia	55	60	65	70	75	80	85	90	psia	0	5	10	15	20	25	30	35
0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	0	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000	1.00000
10	1.00038	1.00038	1.00037	1.00037	1.00037	1.00036	1.00036	1.00036	10	1.00040	1.00039	1.00038	1.00038	1.00037	1.00036	1.00036	1.00035
20	1.00076	1.00075	1.00074	1.00074	1.00073	1.00073	1.00072	1.00071	20	1.00079	1.00078	1.00077	1.00075	1.00074	1.00073	1.00072	1.00071
30	1.00114	1.00113	1.00112	1.00111	1.00110	1.00109	1.00108	1.00107	30	1.00119	1.00117	1.00115	1.00113	1.00111	1.00109	1.00108	1.00106
40	1.00152	1.00150	1.00149	1.00148	1.00146	1.00145	1.00144	1.00143	40	1.00158	1.00155	1.00153	1.00150	1.00148	1.00146	1.00144	1.00141
50	1.00189	1.00188	1.00186	1.00184	1.00183	1.00181	1.00180	1.00178	50	1.00198	1.00194	1.00191	1.00188	1.00185	1.00182	1.00179	1.00177
60	1.00227	1.00225	1.00223	1.00221	1.00219	1.00217	1.00216	1.00214	60	1.00237	1.00233	1.00229	1.00226	1.00222	1.00219	1.00215	1.00212
70	1.00265	1.00263	1.00260	1.00258	1.00256	1.00254	1.00251	1.00249	70	1.00276	1.00272	1.00267	1.00263	1.00259	1.00255	1.00251	1.00247
80	1.00303	1.00303	1.00297	1.00295	1.00292	1.00290	1.00287	1.00285	80	1.00316	1.00311	1.00306	1.00301	1.00296	1.00291	1.00287	1.00282
90	1.00341	1.00338	1.00335	1.00332	1.00329	1.00326	1.00323	1.00320	90	1.00355	1.00349	1.00344	1.00338	1.00333	1.00328	1.00323	1.00318
100	1.00378	1.00375	1.00372	1.00368	1.00365	1.00362	1.00359	1.00356	100	1.00395	1.00388	1.00382	1.00376	1.00370	1.00364	1.00358	1.00353
110	1.00416	1.00412	1.00409	1.00405	1.00402	1.00398	1.00.95	1.00391	110	1.00434	1.00427	1.00420	1.00413	1.00406	1.00400	1.00394	1.00388
120	1.00454	1.00450	1.00446	1.00442	1.00438	1.00434	1.00430	1.00427	120	1.00473	1.00465	1.00458	1.00450	1.00443	1.00436	1.00430	1.00423
130	1.00491	1.00487	1.00483	1.00478	1.00474	1.00470	1.00466	1.00462	130	1.00513	1.00504	1.00496	1.00488	1.00480	1.00473	1.00465	1.00458
140	1.00529	1.00524	1.00520	1.00515	1.00511	1.00506	1.00502	1.00498	140	1.00552	1.00543	1.00534	1.00525	1.00517	1.00509	1.00501	1.00493
150	1.00567	1.00562	1.00557	1.00552	1.00547	1.00542	1.00538	1.00533	150	1.00591	1.00581	1.00572	1.00563	1.00554	1.00545	1.00537	1.00529
160	1.00604	1.00599	1.00594	1.00588	1.00583	1.00578	1.00573	1.00568	160	1.00630	1.00620	1.00610	1.00600	1.00591	1.00581	1.00572	1.00564
170	1.00642	1.00636	1.00631	1.00625	1.00620	1.00614	1.00609	1.00604	170	1.00670	1.00659	1.00648	1.00637	1.00627	1.00617	1.00608	1.00599
180	1.00680	1.00673	1.00668	1.00662	1.00656	1.00650	1.00645	1.00639	180	1.00709	1.00697	1.00686	1.00675	1.00664	1.00654	1.00644	1.00634
190	1.00717	1.00711	1.00704	1.00698	1.00692	1.00686	1.00680	1.00675	190	1.00748	1.00736	1.00724	1.00712	1.00701	1.00690	1.00679	1.00669
200	1.00755	1.00748	1.00741	1.00735	1.00728	1.00722	1.00716	1.00710	200	1.00787	1.00774	1.00762	1.00749	1.00737	1.00726	1.00715	1.00704
210	1.00792	1.00785	1.00778	1.00771	1.00765	1.00758	1.00752	1.00745	210	1.00826	1.00813	1.00799	1.00787	1.00774	1.00762	1.00750	1.00739
220	1.00830	1.00822	1.00815	1.00808	1.00801	1.00794	1.00787	1.00781	220	1.00866	1.00851	1.00837	1.00824	1.00811	1.00798	1.00786	1.00774
230	1.00867	1.00859	1.00852	1.00844	1.00837	1.00830	1.00823	1.00816	230	1.00905	1.00890	1.00875	1.00861	1.00847	1.00834	1.00821	1.00809
240	1.00905	1.00897	1.00889	1.00881	1.00873	1.00866	1.00858	1.00851	240	1.00944	1.00928	1.00913	1.00898	1.00884	1.00870	1.00857	1.00844
250	1.00942	1.00934	1.00925	1.00917	1.00909	1.00902	1.00894	1.00886	250	1.00983	1.00967	1.00951	1.00935	1.00921	1.00906	1.00892	1.00879
260	1.00980	1.00971	1.00962	1.00954	1.00946	1.00937	1.00929	1.00922	260	1.01022	1.01005	1.00988	1.00973	1.00957	1.00942	1.00928	1.00914
300	1.01129	1.01119	1.01109	1.01099	1.01090	1.01081	1.01071	1.01062	300	1.01178	1.01158	1.01139	1.01121	1.01103	1.01086	1.01069	1.01053
400	1.01502	1.01488	1.01475	1.01462	1.01450	1.01437	1.01425	1.01413	400	1.01567	1.01541	1.01515	1.01491	1.01468	1.01445	1.01423	1.01401
500	1.01873	1.01856	1.01840	1.01824	1.01808	1.01792	1.01777	1.01762	500	1.01953	1.01921	1.01890	1.01859	1.01830	1.01801	1.01774	1.01747
600	1.02242	1.02222	1.02202	1.02183	1.02164	1.02145	1.02127	1.02109	600	1.02338	1.02229	1.02262	1.02226	1.02191	1.02157	1.02124	1.02092
700	1.02609	1.02586	1.02563	1.02541	1.02519	1.02497	1.02476	1.02455	700	1.02721	1.02676	1.02633	1.02590	1.02550	1.02510	1.02472	1.02434
800	1.02974	1.02948	1.02922	1.02897	1.02872	1.02847	1.02823	1.02799	800	1.03102	1.03051	1.03001	1.02953	1.02907	1.02862	1.02818	1.02776
900	1.03338	1.03309	1.03280	1.03251	1.03223	1.03195	1.03168	1.03142	900	1.03482	1.03424	1.03368	1.03315	1.03262	1.03212	1.03163	1.03115
1000	1.03700	1.03668	1.03635	1.03604	1.03573	1.03542	1.03512	1.03483	1000	1.03859	1.03795	1.03734	1.03674	1.03616	1.03560	1.03506	1.03454
1100	1.04061	1.04025	1.03990	1.03955	1.03921	1.03887	1.03854	1.03822	1100	1.04235	1.04165	1.04097	1.04032	1.03969	1.03907	1.03848	1.03790
1200	1.04420	1.04381	1.04342	1.04305	1.04267	1.04231	1.04195	1.04160	1200	1.04609	1.04533	1.04460	1.04388	1.04320	1.04253	1.04188	1.04126
1300	1.04777	1.04735	1.04693	1.04653	1.04613	1.04573	1.04535	1.04497	1300	1.04981	1.04899	1.04820	1.04743	1.04669	1.04597	1.04527	1.04459
1400	1.05133	1.05087	1.05043	1.04999	1.04956	1.04914	1.04872	1.04832	1400	1.05352	1.05264	1.05179	1.05096	1.05016	1.04939	1.04864	1.04792
1500	1.05487	1.05438	1.05391	1.05344	1.05298	1.05253	1.05209	1.05165	1500	1.05721	1.05627	1.05536	1.05448	1.05363	1.05280	1.05200	1.05122
1600	1.05839	1.05788	1.05737	1.05688	1.05639	1.05591	1.05544	1.05498	1600	1.06089	1.05988	1.05892	1.05798	1.05707	1.05620	1.05534	1.05452
1700	1.06190	1.06136	1.06082	1.06030	1.05978	1.05927	1.05877	1.05828	1700	1.06454	1.06348	1.06246	1.06147	1.06051	1.05958	1.05867	1.05780
1800	1.06540	1.06482	1.06426	1.06370	1.06316	1.06262	1.06210	1.06158	1800	1.06819	1.06707	1.06599	1.06494	1.06392	1.06294	1.06199	1.06107
1900	1.06888	1.06827	1.06768	1.06710	1.06652	1.06596	1.06540	1.06486	1900	1.07181	1.07064	1.06950	1.06839	1.06733	1.06629	1.06529	1.06432
2000	1.07235	1.07171	1.07109	1.07047	1.06987	1.06928	1.06870	1.06813	2000	1.07543	1.07419	1.07299	1.07184	1.07072	1.06963	1.06858	1.06756
2100	1.07580	1.07513	1.07448	1.07384	1.07321	1.07259	1.07198	1.07138	2100	1.07902	1.07773	1.07648	1.07527	1.07409	1.07296	1.07186	1.07079
2200	1.07924	1.07854	1.07786	1.07719	1.07653	1.07588	1.07525	1.07462	2200	1.08260	1.08125	1.07994	1.07868	1.07746	1.07627	1.07512	1.07401
2300	1.08266	1.08194	1.08123	1.08053	1.07984	1.07917	1.07850	1.07785	2300	1.08617	1.08476	1.08340	1.08208	1.08080	1.07957	1.07837	1.07721
2400	1.08607	1.08532	1.08458	1.08385	1.08314	1.08243	1.08174	1.08107	2400	1.08972	1.08826	1.08684	1.08547	1.08414	1.08285	1.08161	1.08040
2500	1.08947	1.08869	1.08792	1.08716	1.08642	1.08569	1.08497	1.08427	2500	1.09326	1.09174	1.09027	1.08884	1.08746	1.08613	1.08483	1.08358
2600	1.09285	1.09204	1.09124	1.09046	1.08969	1.08893	1.08819	1.08746	2600	1.09679	1.09521	1.09368	1.09220	1.09077	1.08939	1.08804	1.08674
2700	1.09622	1.09538	1.09456	1.09375	1.09295	1.09217	1.09140	1.09064	2700	1.10030	1.09866	1.09708	1.09555	1.09407	1.09263	1.09124	1.08990
2800	1.09958	1.09871	1.09786	1.09702	1.09619	1.09538	1.09459	1.09381	2800	1.10379	1.10210	1.10047	1.09888	1.09735	1.09587	1.09443	1.09304
2900	1.10293	1.10203	1.10114	1.10028	1.09943	1.09859	1.09777	1.09696	2900	1.10728	1.10553	1.10384	1.10221	1.10062	1.09909	1.09761	1.09617
3000	1.10626	1.10533	1.10442	1.10353	1.10265	1.10179	1.10094	1.10010	3000	1.11075	1.10894	1.10720	1.10551	1.10388	1.10230	1.10077	1.09929

T = absolute temperature of the gas during the measurement of b, degrees K.

M = gas molecular weight.

c = dimensionless "acceptance coefficient," which is slightly less than 1.0, according to Klinkenberg.[3]

r = radius of randomly distributed (spatially) capillaries that approximate a porous medium in Klinkenberg's model, μm.

In Equation 12, the factor in brackets depends upon gas properties only, and the factor in parentheses is rock dependent. If we define the rock-dependent factor, r/c, as the rock's "effective gas slippage radius," r_b, and solve for it by rearranging Equation 12:

$$r_b = \frac{99.5\mu}{b}\sqrt{\frac{T}{M}}, \qquad (13)$$

then this is the recommended gas slippage factor to be reported, which is independent of the gas used and temperature. It should be reported in addition to b, or instead of it.

Klinkenberg's randomly distributed capillary analogy relates r_b to permeability and porosity by:

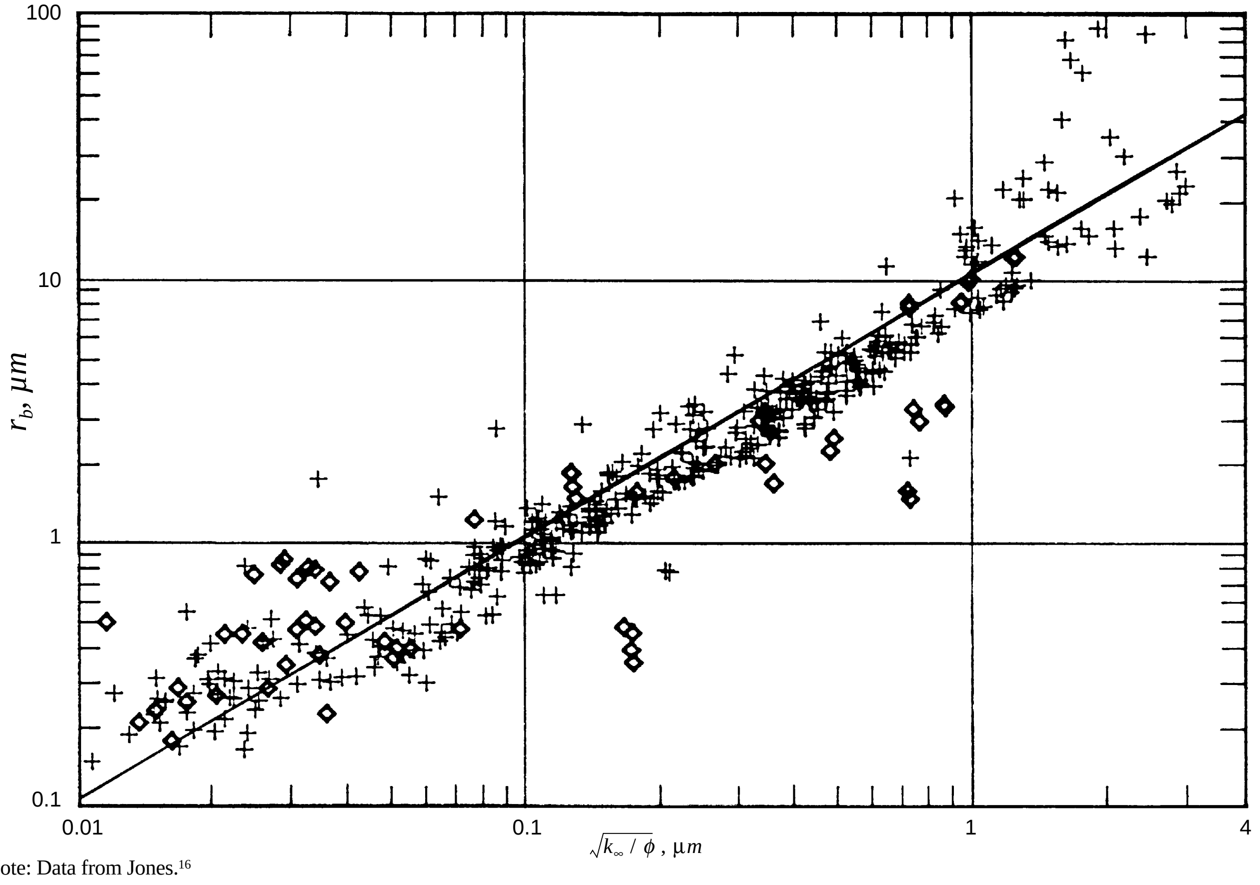
$$r_b = C_b\sqrt{\frac{k_\infty}{\phi}} \qquad (14)$$

where k_∞ is Klinkenberg permeability, (μm^2), and ϕ is porosity (fraction). In Klinkenberg's model, C_b is equal to $\sqrt{24/c}$, or approximately 5. If we relax this "too simple" model, C_b can be found empirically. In Figure 6-2, r_b is plotted vs. $\sqrt{k_\infty/\phi}$. The slope of the line drawn in this log-log plot is unity. From its position, C_b is calculated to be approximately 11. The data, obtained using helium in several core plugs, show considerable scatter. Except for value of the constant, they qualitatively support Klinkenberg's model. The gas slippage parameters, b or r_b, are anticipated to be functions of pore structure, which may account for some of the scatter.

6.2.1.2 Steady-State Flow of Gas—The Forchheimer Equation

Steady state is achieved when the upstream and downstream pressures and flow rate all become invariant with time. At steady state, mass flow rate is constant throughout the sample and does not change with time. Therefore:

$$\rho q_s = \rho_r q_r \qquad (15)$$



Note: Data from Jones.[16]

Figure 6-2—Rock-Dependent Gas Slippage Radius, r_b, as a Function of Permeability and Porosity

The subscript r refers to reference conditions of temperature and pressure under which the mass flow rate is measured. Thus at steady-state, isothermal conditions, where the reference temperature is the same as the flowing gas temperature:

$$\frac{Pq_s}{z} = \frac{P_r q_r}{z_r} \tag{16}$$

Substituting Equations 7, 9, and 16 into Equation 5 yields the differential form of the Forchheimer equation for isothermal, steady-state flow of gases, corrected for gas slippage:

$$C_1\left(\frac{-dP}{ds}\right) = \frac{C_2 \mu P_r q_r z}{A k_\infty z_t (P+b)} + \frac{C_3 \beta M P_r{}^2 q_r{}^2 z}{A^2 R T z_r{}^2 P} \tag{17}$$

The integrated form of this equation can be used to obtain k_∞, b, and β. This requires a minimum of three, and preferably at least six measurements in which the upstream pressure (and/or optionally the downstream pressure) is varied widely to cause substantial variations in the mean pore pressure and flow velocity. For routine measurements, this is not practicable (cost-wise) under steady-state conditions. However, for most cases, adequate information for the calculation of these parameters can be obtained from a single pressure-falloff run (see 6.4.1.1 and 6.4.1.2).

6.2.1.3 The Slip-Corrected Darcy Equation

Whenever its last term becomes negligibly small, Equation 17 reduces to the differential form of Darcy's Law, corrected for gas slippage, and is the basis for most practical, routine measurements. Small pressure gradients lead to small flow velocities, that are squared in the last term, and are therefore small. The term's magnitude also depends on the values of β and gas density. Density is a function of the temperature, pressure, and molecular weight of the gas used.

Figure 6-3 shows typical values of β as a function of permeability for sandstone (open circles) and carbonate rocks (squares) (data from Jones[16]), and for homogeneous unconsolidated sands (solid circles, data from Geertsma[17]). The considerable scatter is believed to be related to heterogeneity in the rock.[16] Samples with fractures or thin layers of higher permeability material parallel to the flow direction have higher β's than homogeneous rock, due to locally high velocities in these high permeability "streaks." Figure 6-3 contains two dashed curves that encompass most of the points, and a central "best fit" curve.

Figure 6-4, which is based on the data in Figure 6-3, presents maximum allowable overall pressure gradients, $\Delta p/L$, that are sufficiently small to permit use of the slip-corrected Darcy equation for the calculation of permeability without incurring significant error due to inertial resistance. The curves shown are for air at 72°F, and for downstream pressures of 14.7, 50, 100, and 200 psia. Because of the scatter in β, these curves are approximate, and are to be interpreted as follows: If the actual β for a particular sample happens to lie on the upper dashed line in Figure 6-3, and the maximum $\Delta p/L$ as shown in Figure 6-4, for the conditions used, were applied for the permeability measurement, then the calculated permeability would be 5 percent low if the final term in Equation 17 were ignored. If the actual β were twice the value shown on the upper dashed line of Figure 6-3, and the same $\Delta p/L$ were applied, then the calculated permeability would be 10 percent too low. On the other hand, if the actual β were a factor of 10 smaller than the one indicated by the upper dashed line (thus, is closer to the high density of points), the permeability error would be only 0.5 percent, etc. Use of the maximum $\Delta p/L$ values shown in Figure 6-4 will result in errors (due to inertial resistance) of less than 2 percent for most samples when the slip-corrected Darcy equation is used for the calculation of permeability.

Starting with the highest permeabilities for the 14.7 psia downstream pressure in Figure 6-4, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{\max}/L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage.

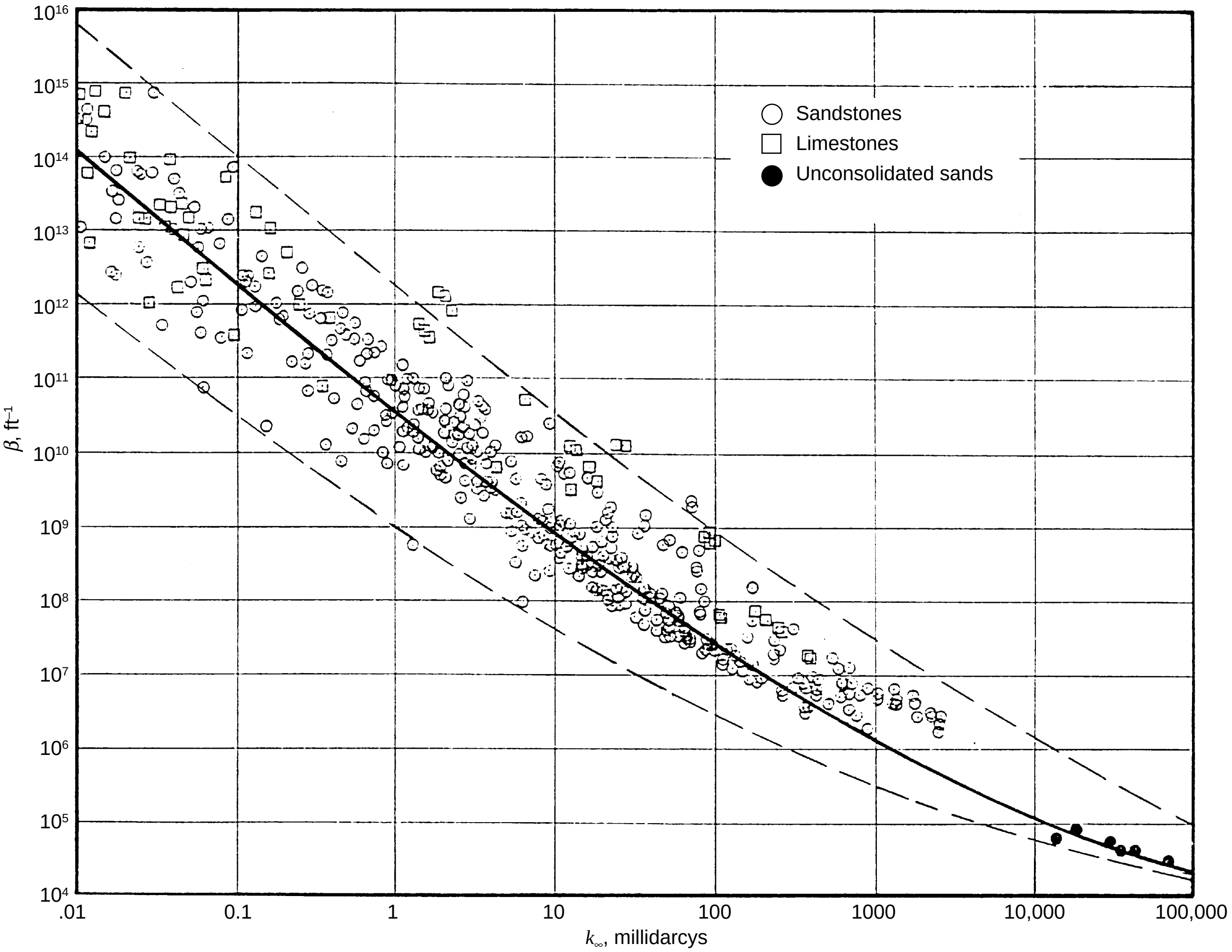
Except for very low permeabilities, the maximum allowable overall gradient is reduced as downstream pressure is increased. Increased back pressure increases gas density, thereby increasing the significance of the final term in Equation 17. This result should be acknowledged when making multiple measurements with increasingly high pore pressures for the Klinkenberg gas slippage correction. Apparent gas permeabilities measured at high pore pressures can be too low due to the inadvertent introduction of non-negligible inertial resistance.

Maximum $\Delta p/L$ values are gas specific. Values for nitrogen and air are similar, but those for helium are higher. Helium's viscosity is higher, and its molecular weight is less than that of air. Equations for calculating $\Delta p/L$ for other gases and liquids are developed in A.6.8.1.

The condition of Stokes flow is met when the last term of Equation 17 is negligible. The remaining slip-corrected Darcy equation can be integrated and used for calculation of Klinkenberg permeabilities for several frequently used flow configurations:

$$k_\infty = \frac{2C_2 \mu P_r q_r z_m}{C_1 z_r G_f (P_1 - P_2)(P_1 + P_2 + 2b)} \tag{18}$$

Where C_1 and C_2 are conversion constants from Table 6-1 to allow for various sets of units for the variables. P_1 and P_2 are the injection and outflow absolute gas pressures, respectively.



Note: Data from Jones[16], and Geertsma[17].

Figure 6-3—Inertial Resistivity, β, as a Function of Permeability

To perform this integration, it was assumed that z in Equation 17 is replaceable by its mean value, z_m. Gas deviation factors z_m and z_r are calculated at the mean pore pressure, P_m (see Equation 20, below), and the absolute reference pressure, P_r, at which q_r is measured, respectively, and at the temperature of the gas. G_f is a geometric factor, which has the dimension of length. The particular factor for each of several flow configurations is given in sections following 6.3.1. The Klinkenberg gas slippage factor, b, must be determined from multiple measurements, or from a correlation. If it is obtained from a correlation, this fact shall be clearly stated.

To obtain b, from which r_b is calculated using Equation 13, the apparent gas permeability, k_g, is measured at a minimum of three (and preferably at least four) different mean pore pressures. Each permeability is calculated from:

$$k_g = \frac{2C_2\mu P_r q_r z_m}{C_1 z_r G_f(P_1 - P_2)(P_1 + P_2)} \qquad (19)$$

and is plotted against the reciprocal of the mean pore pressure as illustrated by Figure 6-5. Mean pore pressure is defined as:

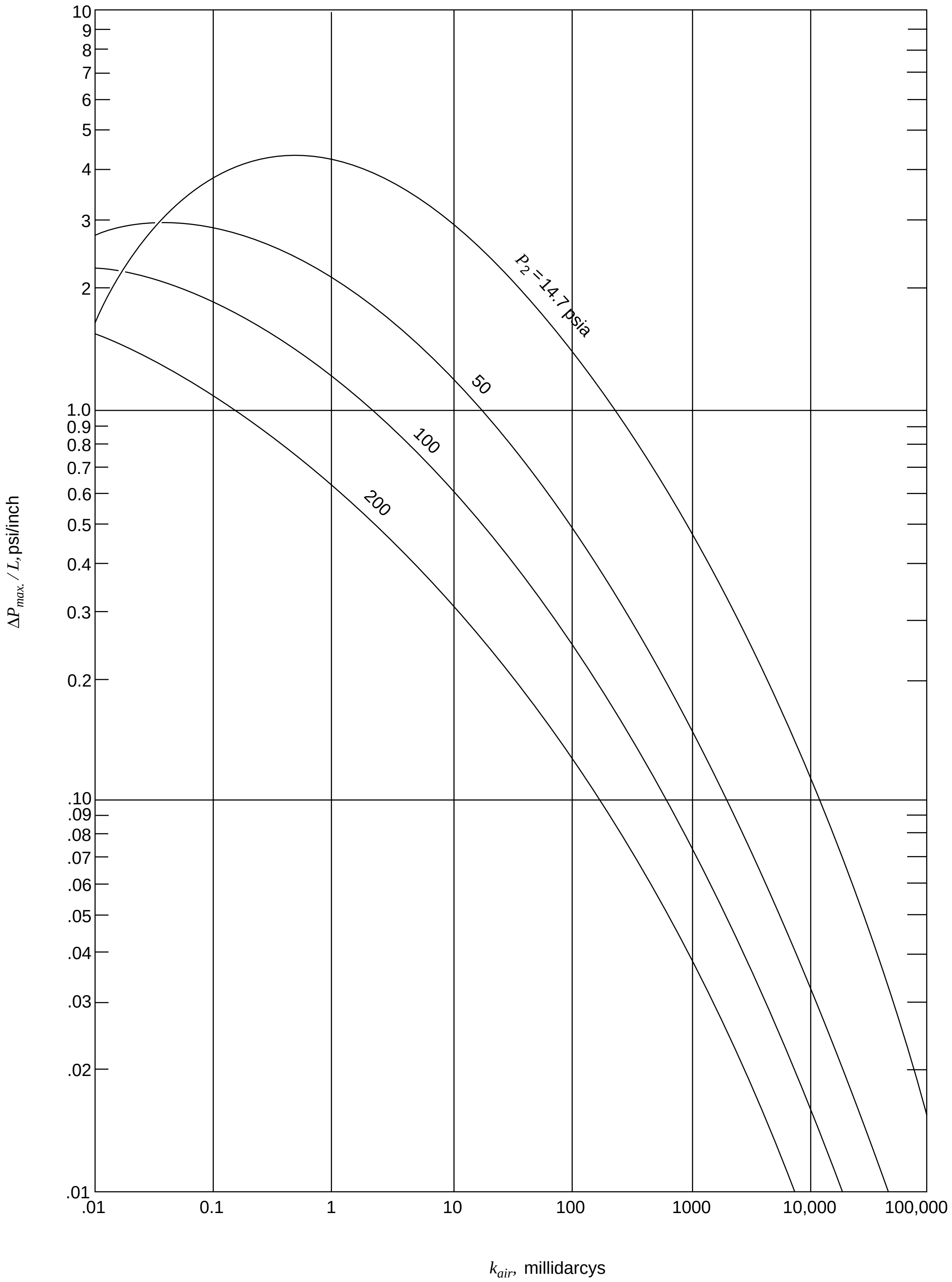
$$P_m = \tfrac{1}{2}(P_1 + P_2) \qquad (20)$$

The intercept of such a plot is k_∞, and its slope is equal to bk_∞, in accordance with the Klinkenberg relationship:

$$k_g = k_\infty\left(1 + \frac{b}{P_m}\right) \qquad (21)$$

The slope and intercept can be found by linear regression. It is essential to ensure that inertial resistance is negligible for each measurement. Otherwise, k_g will be low, especially at higher P_m values. Departure from linearity provides a self check on consistency.

Often, for routine permeability measurements, only gas permeabilities (uncorrected for gas slippage), as calculated from Equation 19, are reported. For these measurements, it is



Note: For air at 72°F.

Figure 6-4—Maximum Allowable Overall Pressure Gradients for Use of Slip-Corrected Darcy Equation

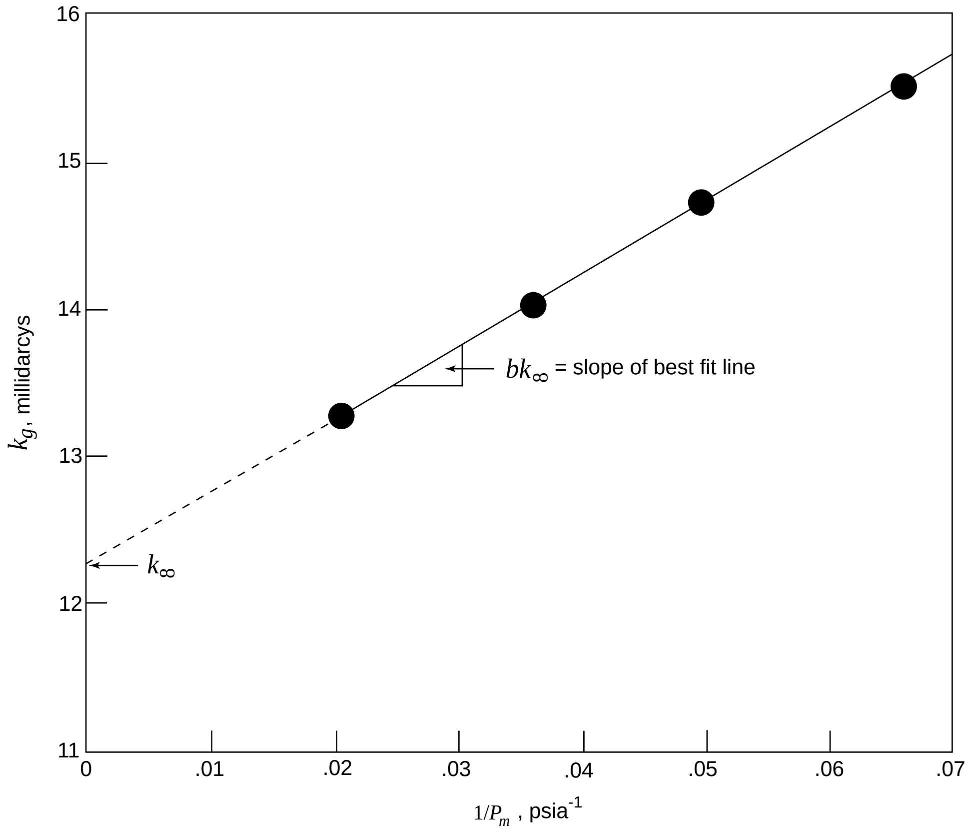


Figure 6-5—Type of Plot Used To Obtain Klinkenberg Gas Slippage Factor, *b*

essential to report the gas used and its mean pore pressure. Otherwise, the reported permeabilities are not fully specified.

6.2.2 Steady-State Permeability Equations For Liquids

Liquids have lower compressibilities, higher densities, and higher viscosities than gases. For steady-state measurements involving small pressure differences, liquids can be treated as incompressible fluids. Due to the higher liquid densities, gravity effects normally cannot be ignored in permeability measurements, except for horizontal flow. Departure from Darcy's Law is rarely a problem with liquids. Their higher viscosities result in significantly lower volumetric fluxes than observed for gases at the same pressure gradients. But their higher densities somewhat offset the viscosity effect with regard to inertial resistance. See A.6.8.1 for the calculation of maximum allowable $\Delta p/L$ values to be used for liquid permeability measurements when Darcy's Law is used.

The mean free path between flowing liquid molecules is so small that slippage is not observed. However, liquids—especially aqueous solutions—can react with constituents of a porous rock, altering its permeability. Also, care must be taken to avoid mobilizing fines in a rock at high liquid fluxes.

Darcy's Law, including gravity effects, for Stokes flow of liquids through permeable media may be stated in general terms as:

$$v_s = \frac{q}{A} = \frac{-C_1 k}{C_2 \mu}\left(\frac{dp}{ds} - \frac{pg}{C_4}\frac{dz}{ds}\right) \qquad (22)$$

Where:

s = distance along the direction of flow.
v_s = volumetric flux (volume of flow through a unit area of the porous medium per unit time).
z = vertical coordinate (increasing downward).
ρ = density of the liquid.
$\frac{dp}{ds}$ = pressure gradient along s at the point to which refers.
μ = viscosity of the liquid.
k = permeability of the medium.
q = volume rate of flow.
A = cross-sectional area perpendicular to lines of flow.

C_1, C_2, and C_4 are constants, found in Table 6-1, that make the units consistent.

6.2.2.1 Special Cases for Steady-State Flow of Incompressible Liquids

Equation 22 may be reduced to simpler forms under special conditions of flow. Some of the more important of these are as follows.

6.2.2.1.1 Horizontal Flow of Liquids

Because there is no vertical component of flow, $dz/ds = 0$, and integration of Equation 22 results in:

$$k = \frac{C_2 q\mu}{C_1 G_f (p_1 - p_2)} \quad (23)$$

Where G_f is a geometrical factor, which has the dimension of length. Factors for axial, transverse, or radial flow are given by Equations 27, 29, or 30, respectively, following 6.3.1.

6.2.2.1.2 Vertical Flow of Liquids

Two sub-cases arise, depending whether the flow is downward or upward, and dz/ds is equal to +1 or -1, respectively:

a. Downward flow with a driving head h (as defined by Figure 6-6):

$$k = \frac{C_2 C_4 q\mu L}{C_1 A\rho g(h + L)} \quad (24)$$

where L is the length of the sample and h is the height of the driving head. If the driving head, h, is zero, we have free downward flow, in which flow rate is independent of sample length.

b. Upward flow with driving head, h (as defined by Figure 6-7):

$$k = \frac{C_2 C_4 q\mu L}{C_1 A\rho g h} \quad (25)$$

If upward steady-state flow is achieved with an imposed pressure difference, Δp, which is measured with a differential pressure transducer with both ports at the same elevation (as shown in Figure 6-8) and both transducer lines completely filled with the same liquid as the flowing liquid, then:

$$k = \frac{C_2 q\mu L}{C_1 A\Delta p} \quad (26)$$

After steady-state is achieved (i.e., the Δp and flow rate are invariant with time), the inlet and outlet valves should be closed simultaneously, and the differential pressure transducer allowed to reach equilibrium. The equilibrium reading should be zero. If it is not zero, this reading should be subtracted from the steady-state reading.

6.2.3 Permeability Decrease At Elevated Net Stresses

The permeability of reservoir rock decreases with increasing net stress. Net stress for permeability is usually defined as the difference between confining stress and mean pore pressure. Figure 6-9 illustrates typical permeability reduction with increasing net hydrostatic stress. Percentage reductions are usually greater with lower permeability plugs. For example, zero-stress permeabilities of plugs 6-2, 8-2, and 11-2 in Figure 6-9 are about 700, 40, and 10 millidarcys, respectively. The permeability of a tight gas sand (e.g., 0.01 millidarcy or less at zero stress) can be reduced to 10 percent or less of its zero-stress permeability by high net compressive stresses.

6.3 PRACTICAL APPLICATIONS FOR STEADY-STATE PERMEABILITY MEASUREMENTS

Several practical configurations for routine, steady-state measurement of permeability are described, first for gases, then for liquids. Each may have particular advantages and limitations (e.g., speed; cost; accuracy; permeability range that can be accommodated; stress levels achievable; directional measurements; susceptibility to error; etc.) that are discussed. Certain precautions to avoid major pitfalls are presented.

Even though we ultimately calculate the permeability of a sample, which is a measure of its fluid conductivity, it is the conductance of the sample that dictates its fluid flow behavior. Conductance depends upon path length and area available for flow, as well as the permeability of the sample. Therefore, the measurable permeability range can be extended in some flow configurations by judicious choice of sample dimensions. For example, to maintain reasonable flow rates with axial flow when permeabilities are very high, long, small-diameter samples can be prepared. Conversely, for low permeability samples, short, larger-diameter plugs are desirable. Other conditions being equal, the maximum permeability that can be measured accurately with a three-inch-long plug is three times as large as can be measured on an otherwise identical one-inch-long plug.

Samples submitted for measurement often range in permeability from less than 0.001 to more than 30,000 millidarcys—a ratio of more than 3×10^7. To accommodate this variation, multiple ranges of pressure and flow rate measuring and/or controlling devices must either be insertable into a permeameter, or selectable from multiple devices that are built in. For simplicity, only a single set of devices is illustrated for each configuration listed below.

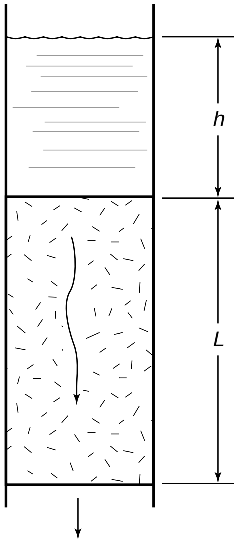


Figure 6-6—Downward Flow of Liquid with Driving Head, *h*

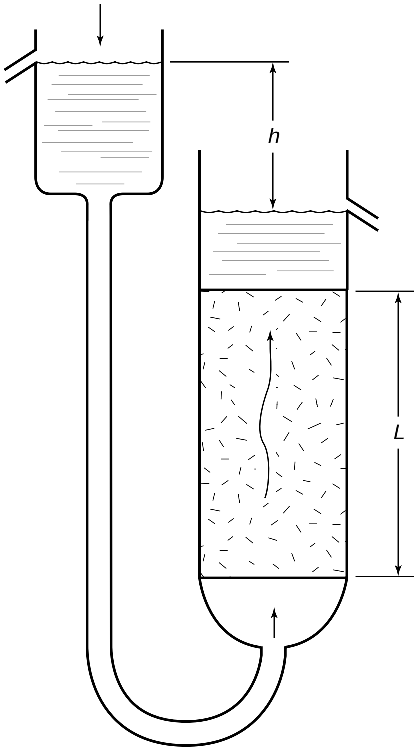


Figure 6-7—Upward Flow of Liquid with Driving Head, *h*

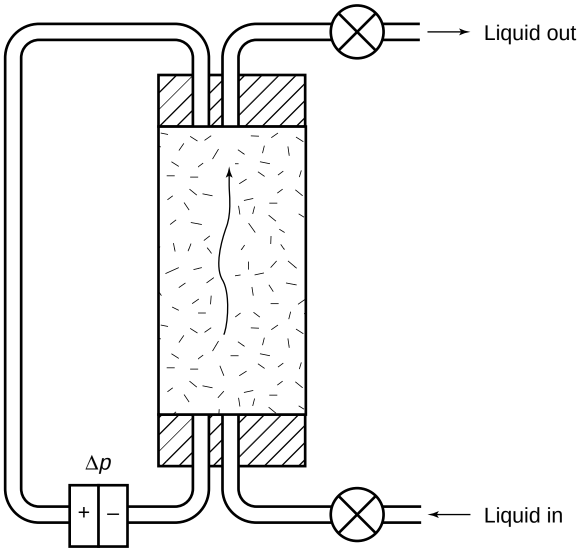
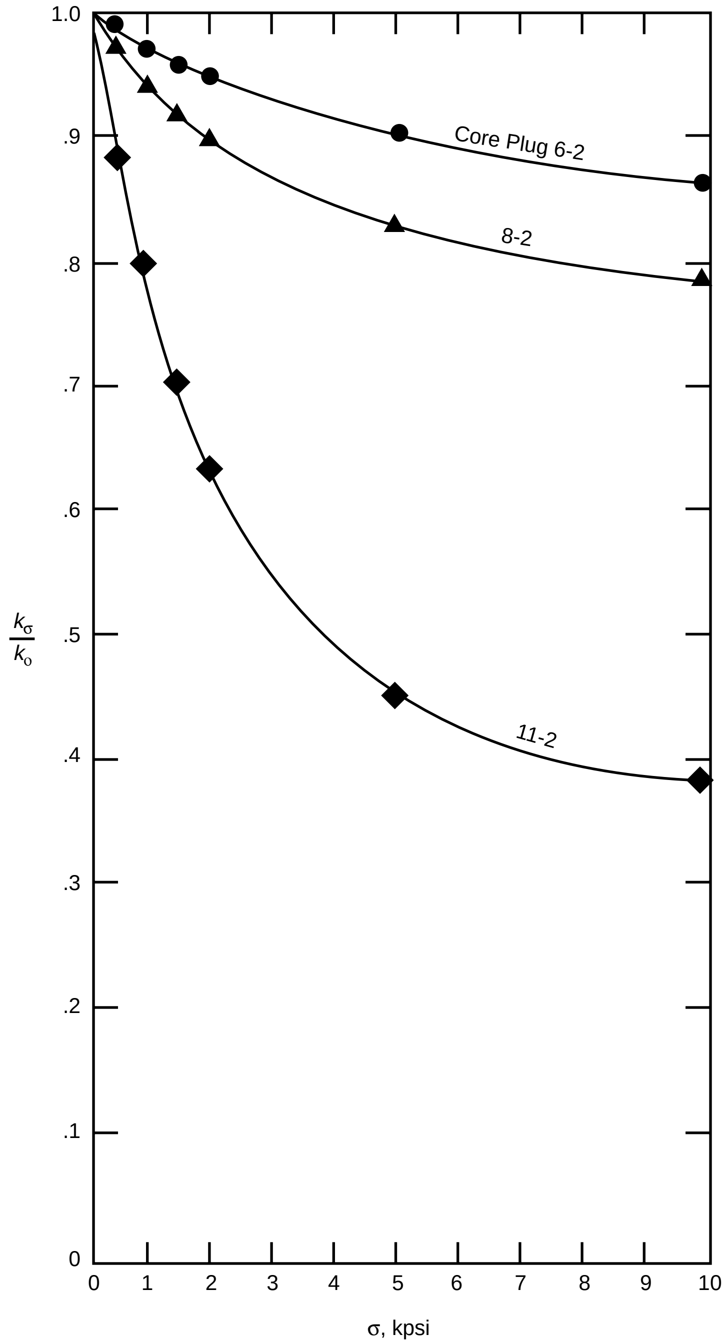


Figure 6-8—Upward Flow of Liquid with Imposed Pressure Difference, Δp



Note: Data from Jones[18].

Figure 6-9—Permeability Reduction with Increasing Net Hydrostatic Stress

6.3.1 Gases

6.3.1.1 Axial, Steady-State Flow of Gases

A schematic arrangement for axial flow of gases is shown in Figure 6-10. A cleaned and dried cylindrical core plug or whole core sample of length *L* and diameter *D* is mounted in a sample holder. The holder contains a flexible sleeve for purposes of making a gas-tight seal on the cylindrical walls of the sample, and for applying radial confining stresses. Axial stress can be transmitted to the sample by applying force to one or both end plugs, by mechanical, pneumatic, or hydraulic means. If the magnitudes of the radial and axial stresses are equal, the sample is said to be *isostatically* or *hydrostatically* stressed. If the magnitudes are unequal, the sample is biaxialy stressed. When gases at relatively low pressures (up to a few hundred psig) are used, gravity effects are negligible, and the sample holder may be oriented either horizontally or vertically.

The two end plugs are provided with an axial port for transporting gas to and from the sample. Each should have radial and circular grooves or other means for distributing gas to its entire injection face, and for collecting gas from all parts of its outflow face. Each end plug, preferably, also contains a second port for measuring upstream and downstream pressures, P_1 and P_2, respectively; the upstream pressure and differential pressure, Δp, as illustrated in Figure 6-10; or the differential and downstream pressures. However, lines for connecting the pressure-measuring devices can be teed into the flow lines near the axial ports, provided that the latter are sufficiently large not to cause significant pressure drops between each tee and the corresponding end face of the sample. It is essential that the branch of each tee (not the run) is connected to the pressure transducer. Otherwise, dynamic pressure effects may influence the pressure measurements.

Note that Δp, as used throughout this document is always a positive number, therefore is equal to $P_1 – P_2$ or to $p_1 – p_2$. As indicated previously, an upper case *P* denotes an absolute pressure, and a lower case *p*, a gauge or differential pressure.

The outflow line can be vented directly to atmospheric pressure (when the flow meter is upstream of the sample), connected to a flow rate measuring device, or to a back pressure regulator for purposes of creating elevated mean pore pressures. In the latter case, the flow meter can be either upstream of the sample or downstream of the back pressure regulator.

The volumetric flow rate, q_r, can be measured at the upstream or downstream pressure, or some other pressure, which in all cases is denoted as P_r, an absolute pressure. The temperature at which the flow rate is measured is assumed to be the same as the flowing gas temperature. Alternatively, the group $(q_r\ P_r)/(z_r\ T_r)$, which is proportional to mass flow rate, can be determined by a mass flow meter. See 6.6.2 for calibration of mass flow meters and application of results to the calculation of permeability.

6.3.1.1.1 Apparatus

6.3.1.1.1.1 Low Confining Stress

Figure 6-11 shows a simplified flow diagram for low pressure measurements. Figure 6-12 shows a low pressure Hassler-type core holder, in which a 400 psi radial confining stress and axial stress of unknown magnitude is typically applied to

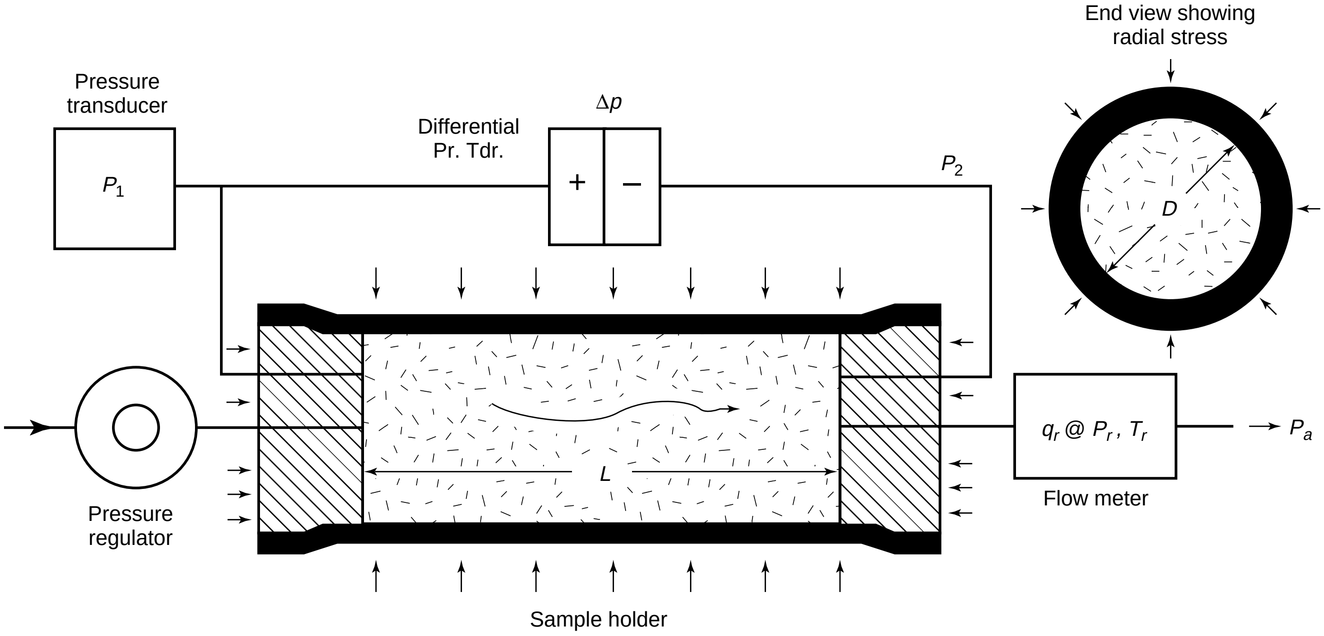


Figure 6-10—Schematic of Permeability Apparatus for Axial Flow of Gas

either a 1.0- or 1.5-inch (25- or 38-millimeter) diameter, 0.75- to 3-inch (19- to 76-millimeter) long core plug. These dimensions and stresses have become virtually standard in conventional, low stress core analysis. Radial stress is transmitted to the plug when pneumatic or hydraulic pressure is applied to the annular space between the rubber sleeve and core holder. Axial stress is typically transmitted from a screw that constrains the lower end plug. Normally, this stress is not measured, and is somewhat operator dependent.

Fancher-type core holders (see page 35 of API RP 40, 1960) are not recommended due to operator-dependent and non-reproducible sealing and confining stresses produced.

6.3.1.1.1.2 Elevated Confining Stress

A core holder for applying hydrostatic stresses, typically to a maximum of 10,000 psi, is illustrated in Figure 6-13. For safety, minimization of gas diffusion through the sleeve, and prevention of corrosion, hydraulic oil, or a heavy mineral oil is strongly recommended for stress generation. Axial stress in this core holder is reduced by the cross-sectional area of the tube connecting the internal end plug to the outer pressure vessel. For $^1/_8$-inch O.D. tubing, the reduction is not serious, but with larger diameters, the axial stress can be significantly less than the radial stress generated. For room temperature operation, 40 to 70 durometer (Shore A hardness), nitrile (Buna-N) rubber is recommended in sleeve thicknesses of 0.125 to 0.25 inch. The lower durometer and thickness can be used for lower stresses. Cold flow occurs with increasing stress and temperature. At stresses above about 6,500 psi, durometers higher than 70 are desirable for extended sleeve life.

Figure 6-14 illustrates a high pressure core holder that can be used to apply either hydrostatic or biaxial loading to core plugs. Axial stress is generated by a stress intensifier. Either pneumatic or hydraulic pressure bears on a piston of larger diameter than that of the end plug. The resulting force is transmitted by the lower end plug to the sample. The pressure required is inversely proportional to the ratio of areas. For example, if the larger piston has an area 10 times that of the end plug, a pressure of 600 psig applied to the large piston would transmit a stress of 6,000 psi to the core plug. If the axial stress is equal to the hydraulic pressure applied to the rubber sleeve, then the core plug is hydrostatically stressed. Otherwise, it is biaxialy stressed. In addition to its capability for providing unequal stresses, the main advantage of this type of core holder is that no disassembly is required for changing samples. Confining pressure is relieved (and often a vacuum is applied to expand the sleeve), the lower end plug is withdrawn, the previous sample is removed, and the new sample is inserted.

Strict safety precautions must be observed in both the design and operation of high pressure equipment. All parts that contain high pressures must suffer no permanent deformation at pressures at least equal to 1.5 times the maximum working pressure, or otherwise, must conform to local, national, or international codes that apply for high pressure vessels.

6.3.1.1.2 Calculations

Gas permeability, k_g, is calculated from Equation 19 (see 6.2.1.3). If multiple measurements are made at different mean pore pressures, k_∞ and b are obtained from a Klinkenberg plot

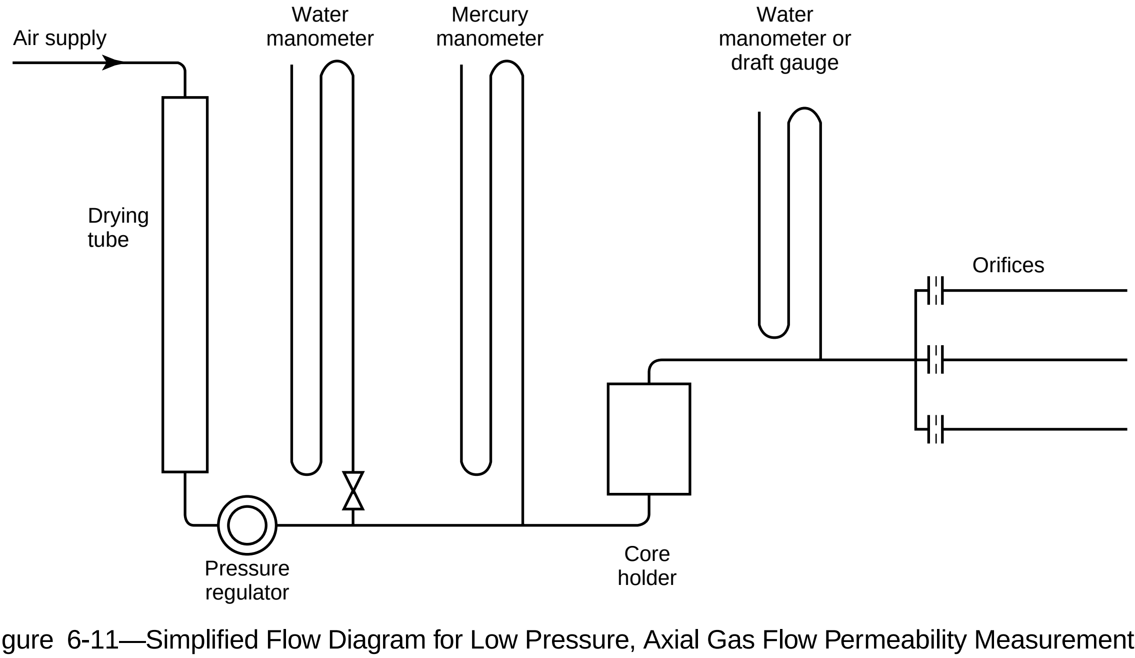


Figure 6-11—Simplified Flow Diagram for Low Pressure, Axial Gas Flow Permeability Measurements

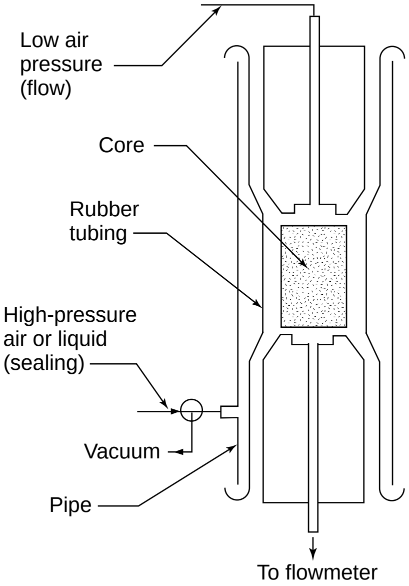


Figure 6-12—Low-Pressure Hassler-Type Core Holder

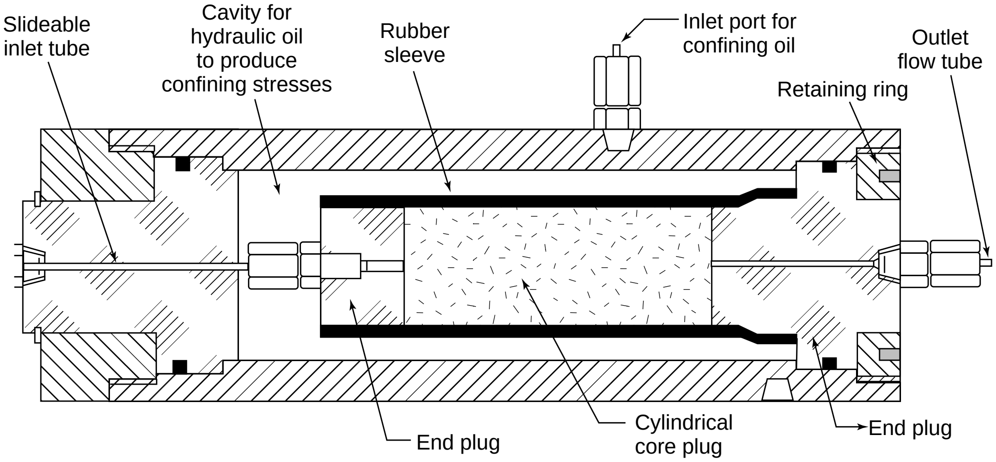


Figure 6-13—High-Pressure Core Holder for Hydrostatic Stresses

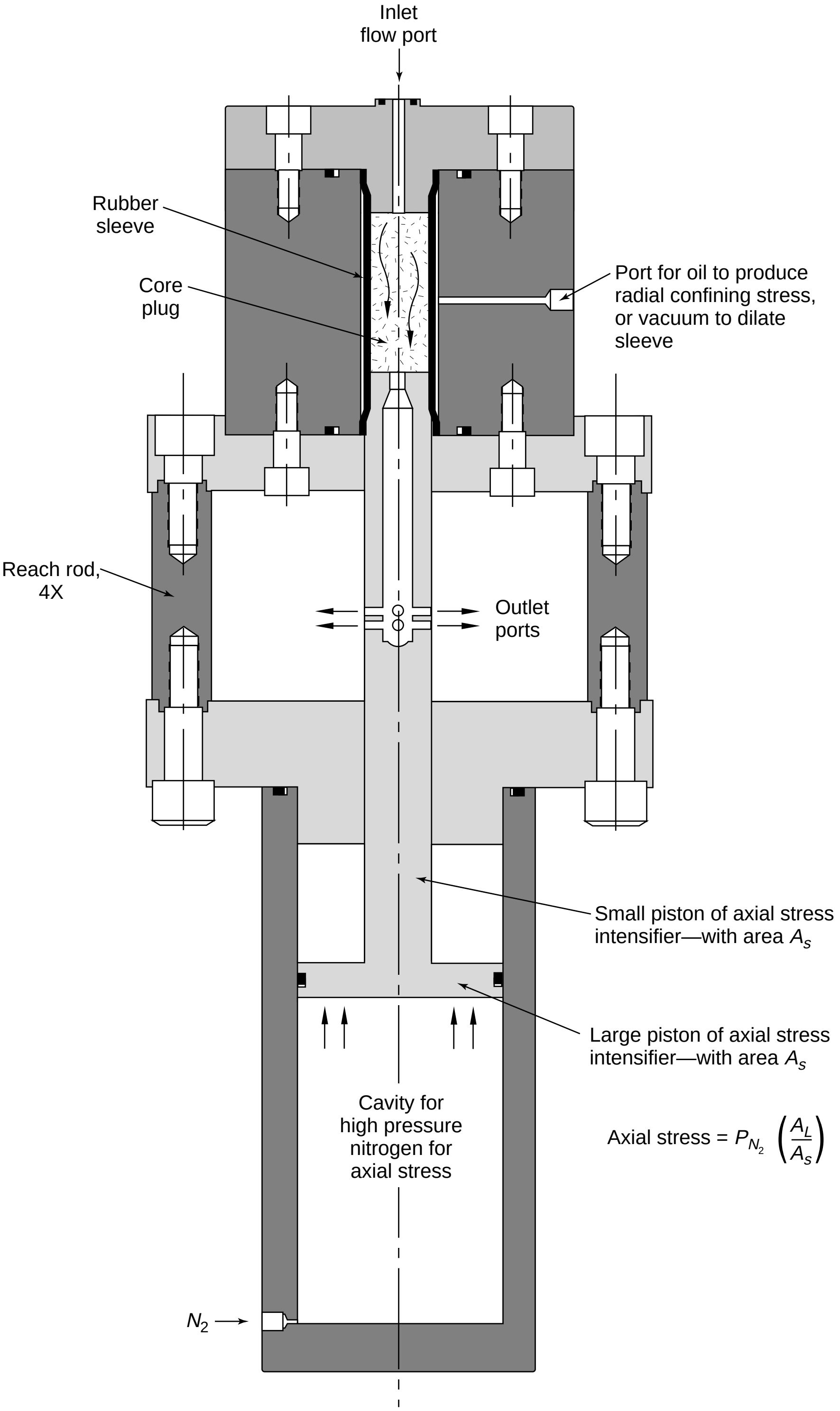


Figure 6-14—High-Pressure Core Holder For Hydrostatic or Biaxial Loading

(see Figure 6-5), or linear regression as described earlier. If b is found from a correlation, then k_∞ is calculated from Equation 18. These statements also apply to all other gas flow configurations listed below. The geometric factor for axial flow in Equations 18 and 19 is:

$$G_f = \frac{\pi D^2}{4L} \tag{27}$$

Traditionally, when a plug or whole core is cut so that its axis is parallel to bedding planes or permeability strike, the measured permeability is called its "horizontal" permeability, k_H. Similarly, a "vertical" permeability, k_V, is obtained when the sample's axis is perpendicular to bedding planes.

6.3.1.1.3 Advantages

Advantages of this method include:

a. The experimental method is simple and has been an industry standard for many years. Consequently, there is a large historic data base for direct comparison.

b. Gas does not react with rock, and samples are clean at the conclusion of measurements.

c. The technique lends itself to a wide range of equipment. With a low-pressure Hassler holder, manometers, orifice-type volumetric flow devices, and manual data acquisition, capital costs are low. However, an upgraded apparatus that includes a high pressure hydrostatic or biaxial core holder and automated control and data acquisition systems is strongly recommended.

d. Flow meters and pressure transducers can easily be exchanged to extend permeability range.

6.3.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. The lower practical permeability limit with this technique is about 0.1 millidarcy. In low permeability rocks the time required to achieve steady state becomes long, and flow measurements can become rather inaccurate.

b. The primary disadvantage of non-slip-corrected, low stress measurements (typically, 400 psi radial confining stress, and unknown axial stress) is that they over estimate in situ reservoir permeability, especially in tight samples. The Klinkenberg slip correction can be made from measurements at several mean pore pressures, but routine measurements are typically made at only one pressure, and this pressure is seldom reported. If this pressure is not reported, the permeability is difficult to normalize, and is incompletely specified. The gas used and the mean pore pressure must be reported. If a gas slippage correction is made from a correlation, this fact must be reported.

c. Attainment of Stokes flow, especially with high permeability samples, can be difficult to ascertain with single point measurements. Measurements should be checked per section 6.2.1.3 to make sure that the maximum allowable $\Delta p/L$ has not been exceeded.

d. It is critical to cut perfectly square ends on core plugs, especially with high confining stresses. A high point will receive a disproportionately high stress, which may crush this portion of the plug. This, of course, applies to all techniques involving cylindrical core plugs.

6.3.1.2 Steady-State Probe Permeability Measurements With Gases

When gas flows from the end of a small-diameter tube (or "probe") that is sealed against the surface of a slabbed or unslabbed whole-core sample, or a permeable rock outcrop, the flow pattern is somewhat similar to that of hemispherical flow (see Figure 6-15). This configuration is widely used in making inexpensive, non-destructive, zero-stress gas permeability measurements within small radii of influence. If the cores are not cleaned and dried, a measurement yields an effective permeability at some unknown oil and water saturation. Such measurements can be made at close spacing to determine permeability variation in a heterogeneous formation. Instruments for making these measurements are generically called "probe permeameters," or "mini-permeameters."

True hemispherical flow geometry would require: (a) a large, homogeneous, isotropic sample with a plane upper surface, (b) a hemispherical cavity under this surface, immediately under the probe, with a radius, r_i, equal to the inside radius of the probe seal, (c) a very large (infinite) extent of the sample in all directions under the plane of contact, and (d) a seal everywhere on this plane except over the hemispherical cavity. Because these conditions are totally impractical, Goggin, et al.[19] performed numerical calculations to produce dimensionless geometrical flow factors that account for the lack of a hemispherical cavity on the surface of a small, finite sample, and a probe seal of small, finite interior and exterior radii.

These dimensionless factors are displayed in Figure 6-16 as a function of the ratio of the outer radius, r_o, of the probe seal to its inner radius, r_i. For true, semi-infinite, hemispherical flow, G_0 would equal 2π. The relationship shown in Figure 6-16 is intended for samples with a plane upper surface and sufficient lateral extent and depth that exterior boundaries have no influence on permeability measurements. For practical purposes, the factors shown can be used with little error on samples that have a depth at least equal to four times the interior radius of the probe seal, and where the nearest lateral boundary is at least $4r_i$ from the axis of the probe. Also, little error is introduced if the probe is placed on a cylindrical surface of a sample, provided that a positive, gas-tight seal is made, the radius of the sample is at least $12r_i$, and the above sample boundary restrictions are observed. See Goggin, et al.[19] for dimensionless geometric factors that relate to nearer boundaries.

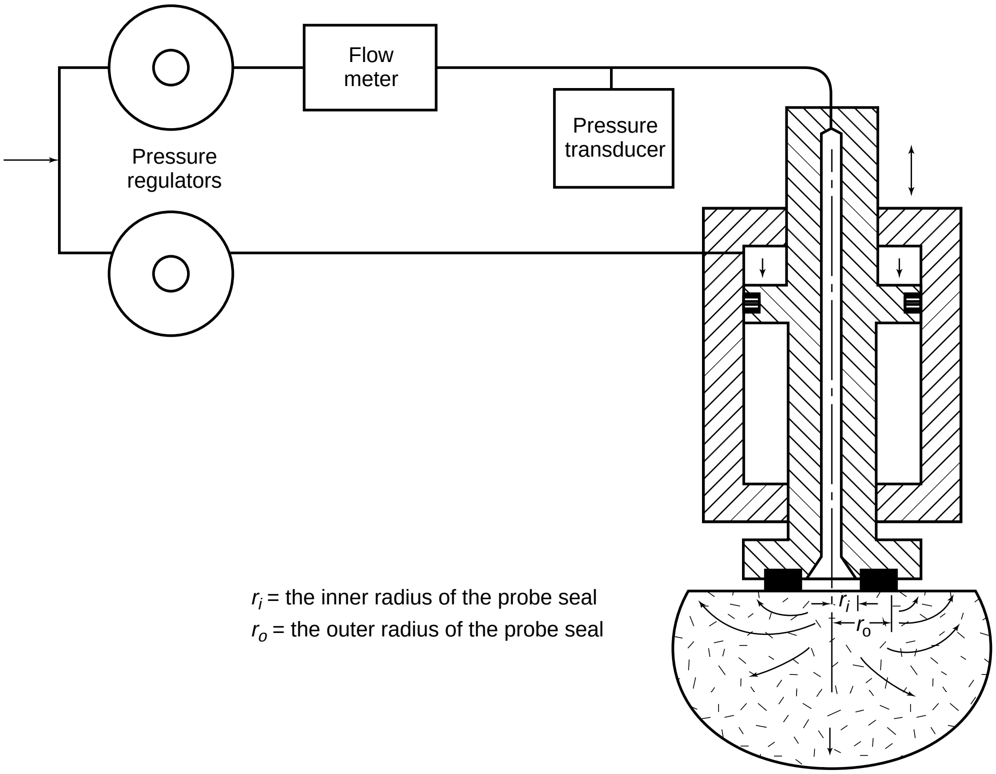
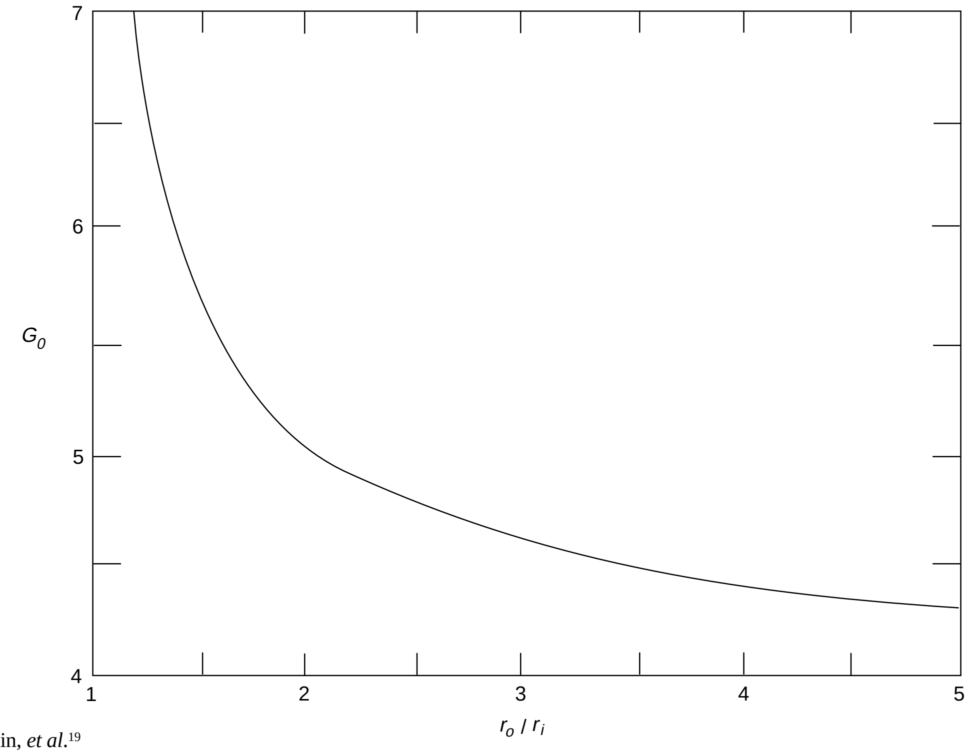


Figure 6-15—Schematic of Steady-State Probe Permeameter



Note: From Goggin, *et al.*[19]

Figure 6-16—Dimensionless Geometric Factor for Measurements with Probe Permeameter

6.3.1.2.1 Apparatus

For gas permeability measurements (see Figure 6-15), the probe must be provided with a soft rubber seal that has dimensionally stable (or reproducible) r_i and r_o when pressed against the sample, and means for delivering a constant, reproducible sealing force to it. This sealing force must be adequate to prevent gas from escaping anywhere on the contact surface between the seal and the rock. The inner radius of the seal is typically 2 to 3 millimeters, and the ratio of its outer to inner radii is typically 1.5 to 2.5. The smaller radii and ratios yield more resolution with regard to small scale heterogeneity, and larger values are used to obtain more permeability averaging. These dimensions must also be scaled to the coarseness of the rock texture.

The probe is positioned above a desired point on a sample, which must be related to depth and position relative to the core's axis or some other reference point. The probe is lowered to the rock's surface, and sealed against it. Gas at pressure P_1 is delivered from the probe to the sample. It exits from the sample at atmospheric pressure. Provision must be made for regulating pressure in the probe, and for measuring it and the volumetric gas flow rate at this pressure. Flow meter range is selected on the basis of the permeability being investigated. Pressure and flow rate are recorded when both become constant.

6.3.1.2.2 Calculations

From these measurements, plus the seal dimensions, atmospheric pressure, and gas viscosity, k_g can be calculated from Equation 19, wherein, if p_1 is the measured upstream gauge pressure:

$$P_1 = p_1 + P_a \quad ,$$

$$P_2 = P_a \quad ,$$

$$P_r = P_1 \quad ,$$

and

$$G_f = G_0 r_i \tag{28}$$

The permeability obtained is most heavily weighted (in non-homogeneous samples) within a radius equal to the inside seal radius, and progressively less with increasing radii. Because of the small volume of rock investigated, permeability on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.2.3 Advantages

Advantages of this method include:

a. The method is nondestructive in the sense that there is no need to cut core plugs. It is rapid and low cost, thereby permitting high-density sampling.

b. The probe investigates only a small volume of rock. The measurement is well suited for investigation of spatial permeability variation in cores containing thin laminations and small scale heterogeneity. Directional permeability variation around the circumference of a whole core can be measured.

c. The device can be engineered for portable well site service, and for use on outcrops, provided that an adequate seal can be achieved.

d. With multiple flow meters a permeability range of about 1 to 10,000 millidarcys can be measured.

6.3.1.2.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Minimum flow path lengths in a sample (from r_i to r_o) range from 0.2 to 0.4 cm for typical probe tip seals. Most of the total pressure drop occurs within this distance, which is an order of magnitude shorter than the path length for axial flow through most core plugs. The injection pressure, p_1, must be reduced correspondingly to avoid significant inertial flow resistance. Even though Goggin, et al.[19] proposed a "high velocity flow correction" using a correlation to obtain β, this correction can only be approximated with single-rate measurements, because β varies by plus or minus at least an order of magnitude at a given permeability for most permeability ranges. However, this correction is smaller the lower the injection pressure.

b. Nearly zero stress is applied to the rock during a measurement. Therefore, permeabilities tend to be very optimistic, especially in tight samples. If Klinkenberg corrections are not made (from a correlation), permeability is further overestimated, especially in low permeability rock.

c. Unless the tip seal is laterally confined, and essentially flat on the bottom (in the unstressed condition) its geometric factor can be very sensitive to the sealing stress applied. This stress, therefore, must be highly reproducible.

d. Although the technique can be used on whole core segments, slabbing is strongly recommended to remove mud solids invasion and other surface contamination, and to provide a flat, smooth surface for probe tip sealing, which otherwise is hard to guarantee.

e. If the surface of the core is not dry, measured permeability may be erroneously low due to severe relative permeability effects. Permeability may increase with time, then stabilize as mobile liquids are evaporated or blown away from the probe tip.

f. Probe tip seals should be inspected regularly for abrasion, deterioration, and imbedded sand grains, and replaced as needed. Periodic leak-testing on a smooth, flat impermeable surface is useful for detection of flaws or inclusions.

g. Due to the small volume of rock investigated by this technique, permeabilities on a macro scale must be obtained by appropriate averaging techniques.

6.3.1.3 Transverse, Steady-State Flow of Gases

This technique is used primarily to measure directional, "horizontal" permeability in (but not limited to) whole core samples. Gas flows from an inlet screen, covering a subtended angle θ and the entire length, L, of a whole-core sample (or core plug), across the sample, and into a similar outlet screen placed diametrically across from the inlet screen (see Figure 6-17). The flow pattern is complex, and the area normal to the streamlines for gas flow is variable throughout the flow path. Collins[20], using a conformal mapping transformation, computed a dimensionless geometrical factor, G_θ. It is shown in Figure 6-18, and is a function of the angle subtended by the screens. Permeability is normally measured in two directions—one giving the maximum value (usually along the direction of principal fracturing or permeability strike), and the other at 90 degrees to the maximum.

6.3.1.3.1 Apparatus and Calculations

The flow system and measurements required for calculation of transverse flow permeabilities are similar to those for steady-state axial flow. Figure 6-19 shows a Hassler-type core holder for transverse permeability measurements. Gas is allowed to enter and leave the sample through diametrically opposed openings through the sleeve. These connect to two mesh screens that cover the entire length of the sample and a known subtended angle on its circumference. If the subtended angle of each screen is 90 degrees, then the dimensionless geometrical factor, G_θ, is equal to 1.0. The seal materials must not penetrate the mesh and restrict flow. Both ends of the sample are sealed with soft rubber disks.

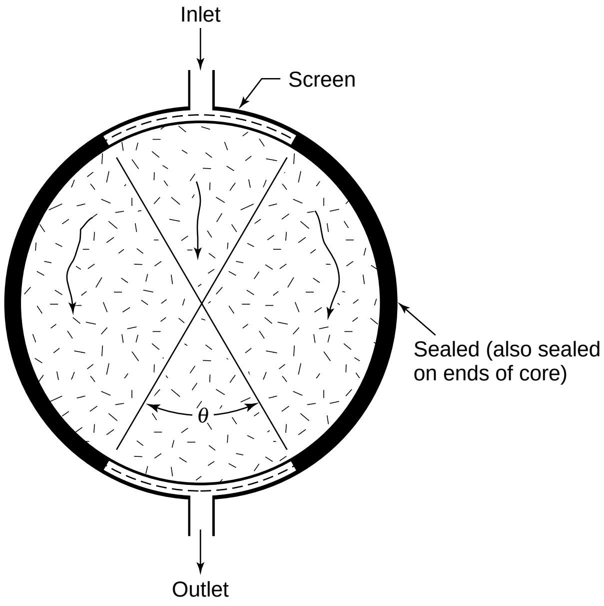
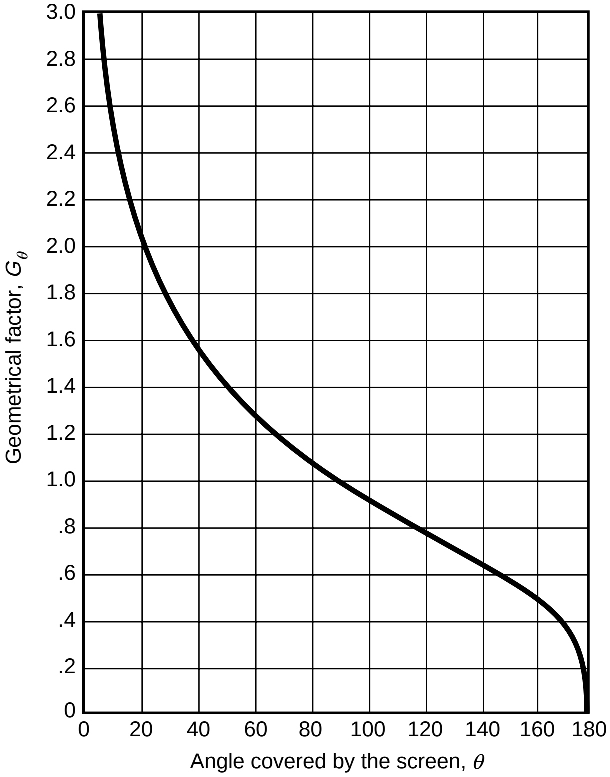


Figure 6-17—Transverse Flow Configuration for Gases or Liquids



Note: From Collins[20].

Figure 6-18—Dimensionless Geometric Factor for Transverse Flow

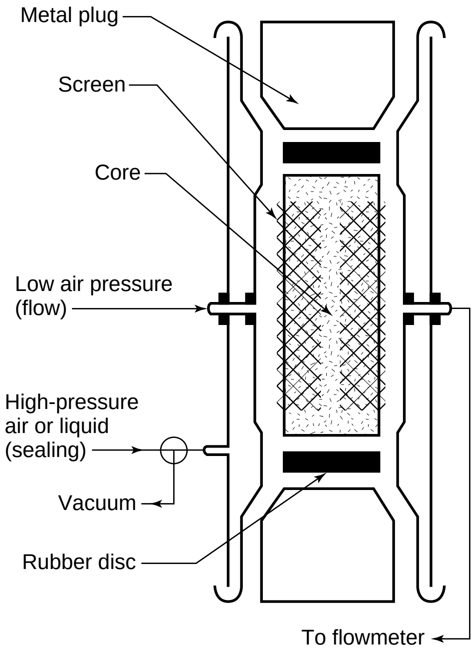


Figure 6-19—Hassler-Type Core Holder for Transverse Permeability Measurements

The dimensional geometric factor to be used in Equations 18 or 19 for transverse flow is:

$$G_f = \frac{L}{G_\theta} \tag{29}$$

A whole core sample can be placed in a large core holder for longitudinal "vertical" permeability measurements, before or after the transverse measurements. Calculations are those for axial flow (see 6.3.1.1.2).

6.3.1.3.2 Advantages

Advantages of this method include:

a. Measurements on whole core samples involve large volumes of rock, thereby permitting considerable permeability averaging. This minimizes the effect of small scale heterogeneity.

b. On oriented whole core samples, orthogonal measurements can be made to determine directional permeability.

6.3.1.3.3 Precautions and Limitations

Precautions and limitations of this method include:

a. A whole core sample is seldom cut precisely normal or parallel to the rock's bedding planes. Therefore conventionally defined "vertical" and "horizontal" permeabilities may not be obtained exactly with longitudinal and transverse measurements, respectively, on these samples.

b. Larger samples take considerably longer to clean, dry, and reach steady state. Experiments therefore take longer and are more expensive than those on core plugs.

c. The same precautions relating to gas slippage and inertial resistance, given in 6.3.1.1.4 for axial flow, should be observed.

6.3.1.4 Radial, Steady-State Flow of Gases

Figure 6-20 shows the flow configuration for steady-state radial flow. The upper and lower faces of a clean, dry cylindrical sample of length L and exterior radius r_e, into which an axial bore of radius r_w is cut, are sealed everywhere except over the central bore. Gas at pressure P_1 flows from the unsealed cylindrical surface radially to the central bore, and exits at pressure P_2. Otherwise, the flow system and measurements are the same as for axial flow. In principle, the flow direction can be reversed, so that gas is injected into the central bore at pressure P_1, and exits through the sample's exterior cylindrical surface at pressure P_2. However, injection into the well bore is generally not recommended because its smaller flow area is more prone to damage from trace amounts of particulates in the injected fluids.

6.3.1.4.1 Apparatus and Procedure

The full diameter radial permeameter, shown in Figure 21, consists of three parts: the cell, which is sufficiently large to maintain a uniform inlet pressure; a piston to apply the sealing force; and the floating plate assembly that consists of a lower fixed plate, a pivot ball, three springs 120 degrees apart, and the upper floating plate.

The core is placed on a 1-inch thick solid rubber gasket that is attached to the lower floating plate. The core is then raised against the closed lid, the center hole of the core matching that of the upper gasket. As the piston pressure increases, the lower floating plate automatically adjusts if the ends are not parallel. To check for an air leak between the ends of the core and the rubber gaskets, the piston pressure is increased. A decrease in the flow rate indicates that a leak had existed. This test is repeated until no change in flow rate is noted.

6.3.1.4.2 Calculations

Flow rates and pressure drops are measured and used in the same manner as for axial flow measurements. Steady state is reached when both become invariant with time. Permeability, k_g, is calculated from Equation 19, wherein the geometrical factor for radial flow is:

$$G_f = \frac{2\pi L}{\ln[r_e/r_w]} \tag{30}$$

Average permeability of non-homogeneous samples is most heavily weighted by the permeability at the inner wellbore radius, r_w (where the pressure gradient is greatest), and progressively less at larger radii.

6.3.1.4.3 Advantages

The measured permeability is averaged throughout the length of the sample and in all radial directions.

6.3.1.4.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Permeability is critically dependent upon the internal wellbore radius. Small errors in its value, especially with small diameter holes, will cause large permeability errors. Similarly, induced fractures or surface damage near this hole will greatly affect apparent permeability.

b. Sample preparation for this measurement is not easy. Therefore the test tends to be somewhat more costly than alternative configurations.

c. Axial stresses are not balanced by radial stresses. The method is not easily adapted to application of elevated stresses for measurement of realistic in situ reservoir permeability.

d. A large piece of core material is altered by the central bore hole required for this technique, and the sample cannot usually be used for other tests.

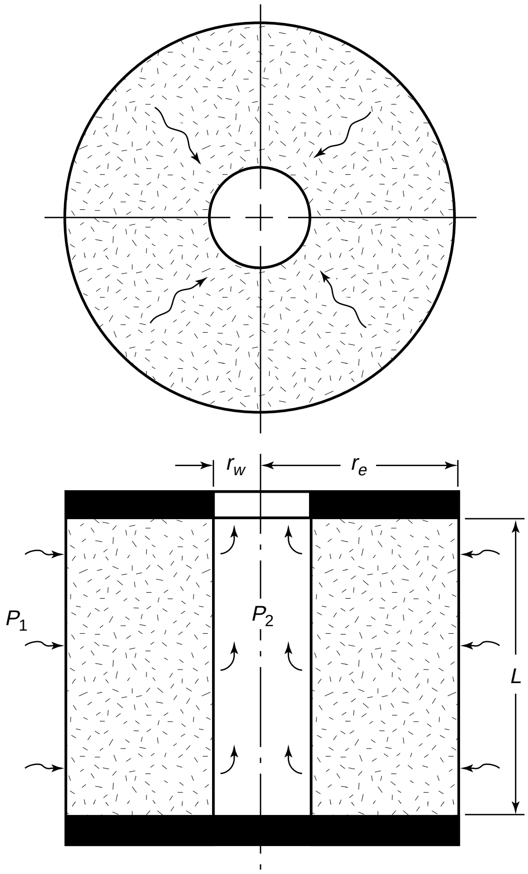


Figure 6-20—Schematic for Inward Radial Flow

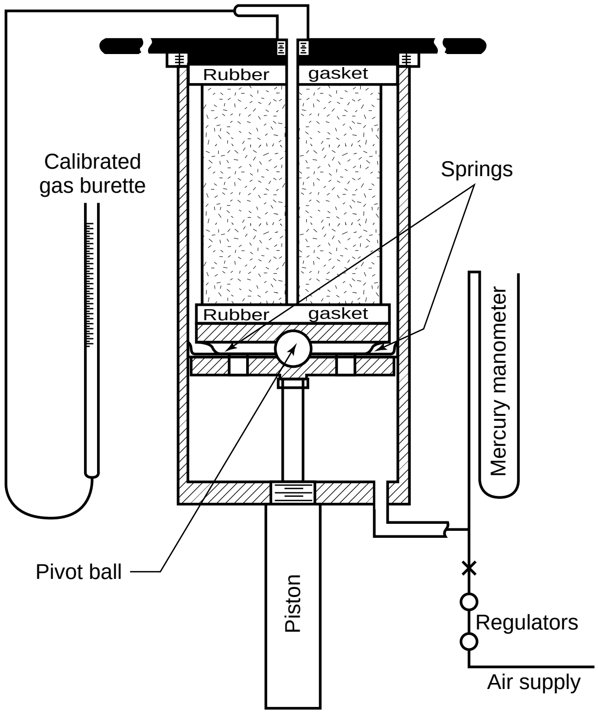


Figure 6-21—Full-Diameter Radial Flow Permeameter

6.3.2 Liquids

Steady-state liquid permeability apparatus have many features in common with those for gas. Differences between liquids and gases that may require equipment modifications are: liquid viscosity and density are considerably greater and compressibility is much smaller than the corresponding gas properties. Furthermore, liquids (especially aqueous solutions containing salts) can be corrosive, support microbial activity, and react with clay and mineral constituents of the rock.

Permeability to liquids is often observed to be lower than slip-corrected gas permeability. This can be due in part to liquid-clay interaction (especially with aqueous solutions), fines migration with high liquid flow rates, or incomplete liquid saturation.

6.3.2.1 Steady-State Liquid Permeability Measurements with an Imposed Δp

6.3.2.1.1 Apparatus

This type of permeability apparatus does not depend upon liquid heads for delivery of fluid. It comprises three separate units: sample holder, pressure measurement system, and fluid delivery system. Except for corrosion resistance and compatibility of the rubber sleeve with a particular liquid, the sample holder, delivery lines, and pressure measuring equipment for liquid permeability measurements are not different from systems for gases. The higher liquid density requires that attention be paid to liquid heads. These normally cancel out with horizontal flow, provided that both ports of the differential pressure transducer used are at the same elevation, and that all pressure-measuring lines are completely filled with the same fluid as the flowing fluid, and are at the same temperature. In this case, when flow through the sample is terminated, the Δp reading should go to zero. Under these circumstances Equation 23 (see 6.2.2.1.1), which applies to horizontal flow, also becomes valid for upward or downward vertical flow (see comments following Equation 26 in 6.2.2.1.2).

Because of the 50-fold (or more) higher viscosity of most liquids compared to gases, liquid flow rates are correspondingly lower at the same pressure gradient. The fluid delivery system is normally either a primary constant flow rate source or a constant pressure source that can be regulated to result in a constant flow rate. Single-stroke, positive displacement pumps offer the most accurate means of delivering a liquid at a controlled constant rate. The use of such pumps precludes the need to collect produced liquids from a core to determine flow rate. However, the volume contained in a single-stroke pump may be insufficient to reach steady state. To completely eliminate pressure upsets and flow rate discontinuities, two such pumps and a sophisticated control system are recommended for a "once-through" flow system, and three are desirable for a recirculating flow system.

Slow attainment of steady state, provided that the sample's permeability is not changing due to progressive plugging, clay or mineral interaction, etc., is due to high compressive storage in the system. Compressive storage is the product of the volume and effective compressibility of each component in the system. Effective compressibility is the sum of fluid and vessel compressibilities. Fluid compressibility can be reduced markedly (if any gas is present) by applying a high back pressure to the system, thereby dissolving gas bubbles. Care should be taken to remove trapped gas from the system, and to de-gas liquids by applying a vacuum. Vessel compressibility is reduced by making vessels more rigid. System volumes should be minimized wherever possible. Use of transfer vessels (where, for example, oil from a pump displaces brine that is injected into a core plug) at least doubles the volume, compared to displacing brine directly from the pump. However, corrosion considerations may preclude direct pumping. Whenever compressive storage is nearly zero and permeability remains constant, achievement of steady state is virtually instantaneous.

When using a chromatography-type pump, the operator should ensure that its delivery is tested over the entire range to be employed. To calculate permeability, flow rates should be measured independently. This can be accomplished by collecting and weighing produced liquid in a weighing vessel on an electronic balance at frequent, timed intervals. Volumetric flow rate is calculated by dividing the rate of mass accumulation by the liquid's density.

Constant pressure systems utilize either a gas or liquid source to displace de-gassed liquid from a vessel and through the sample. The liquid to be displaced is contained in a piston accumulator or collapsible rubber bladder. The displacing fluid passes through a pressure regulator. As for the case of chromatography type pumps, flow rate must be determined independently.

6.3.2.1.2 Procedure and Calculations

The liquid to be used should be well-filtered through a fine filter shortly before use. A 0.2 micron filter will remove bacteria as well as solid particles. A fully saturated test sample should be carefully loaded into an appropriate sample holder, ensuring that no air is trapped in the process. Confining stresses are then applied. In the absence of any other permeability data, a low Δp or flow rate should be applied initially to allow the operator to make an initial approximation of permeability, and thereby set conditions of flow rate and pressure drop, and select appropriate equipment for these measurements. See A.6.8.1 to determine the maximum $\Delta p/L$ to avoid significant inertial resistance. However, this is generally not a serious problem with liquids. Care should be taken to avoid the mobilization of rock fines. The effluent should be collected and observed for contamination. Confining stresses should be adjusted to obtain the desired net stress.

Close temperature control must be maintained to ensure constant viscosity, and avoid volumetric flow rate changes with liquid expansion or contraction. This is particularly critical when large vessels are involved, accompanied by low displacement rates from a positive displacement pump, with low permeability plugs. Huge flow rate variations can occur in this situation with small changes in temperature over short time intervals. The critical factor is the thermally induced volumetric expansion rate, Δq:

$$\Delta q = \beta_T V\left(\frac{dT}{dt}\right)$$

relative to the pump's displacement rate.

Where:

β_T = coefficient of volumetric thermal expansion of liquid, °C^{-1} or °F^{-1}.

V = volume of liquid being displaced.

$\frac{dT}{dt}$ = time rate of temperature change.

To illustrate, suppose that a positive displacement pump contains 1,000 cm^3 of decane that is being injected into a low permeability plug at a nominal pump displacement rate of 1.000 cm^3/hr., and that the temperature of the decane is increasing at a rate of 0.02°F/minute, due to changes in the ambient temperature. The coefficient of thermal expansion of decane is about 5.5 x 10^{-4} volumes per volume per degree F. Therefore the rate of thermal expansion of decane is 5.5 x 10^{-4} x 1000 x 0.02 = 0.011 cm^3/min. With the pump's displacement rate of 0.0166 cm^3/min, this causes an error in flow rate of 66 percent of the nominal rate! If the displacement rate were increased by a factor of 10, the error would be reduced to 6.6 percent. Additionally, if the pump's volume were reduced to 10 cm^3, the error would be only 0.066 percent. Another way to reduce volumetric expansion is to increase the thermal mass of the pump, and improve its insulation.

With a constant-pressure delivery system (in which flow rate must be determined independently), thermal expansion of liquid in its displacement vessel is not a problem, provided that the upstream pressure regulator is self-relieving. In this case pressure will remain constant in spite of thermal expansion.

After both flow rate and differential pressure have stabilized they should be read and recorded, then the flow terminated. The transducer lines must be completely filled with the test liquid, and both ports must be at the same elevation. After the differential pressure stabilizes with zero flow, it is read. If it is not zero, the reading should be subtracted from the stabilized, flowing Δp. Liquid permeability is calculated from Equation 23, using the appropriate dimensional geometric factor, G_f. These factors for axial flow, transverse flow, and radial flow are given by Equations 27, 29, and 30, respectively. With the above transducer line restrictions, Equation 23 applies to vertical, upward or downward axial flow as well as to horizontal flow for all three geometric configurations.

After the measurement has been completed and the sample is to be removed for further analysis, care should be taken to ensure that, as the sample dilates while confining stress is reduced, liquid is imbibed and the ingress of air is precluded.

6.3.2.1.3 Advantages

Advantages of this method include:

a. Pressure differentials for Stokes flow with liquids are higher and easier to measure than those with gases, especially for high permeability samples.
b. No gas slippage correction is required.
c. Carefully made liquid permeability measurements may be more representative of reservoir permeabilities.
d. Sample drying, a potentially damaging operation, is not required.
e. Liquid permeability measurements may be advantageous if additional analyses require the plug to be saturated with the same liquid.

6.3.2.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. Saturation and preparation of the rock sample, and preparation and handling of liquids is generally more difficult and time consuming than preparations for gas measurements.
b. Corrosion resistant, high pressure pumping and control equipment is expensive.
c. Care must be taken to avoid fluids that interact with rock constituents.
d. Steady state liquid measurements with low permeability plugs may be difficult to make accurately, and require long periods to reach steady state. Small volumes, short plug lengths, low compressive storages, and very tight temperature control are strongly recommended.

6.3.2.2 Steady-State Liquid Permeability Measurements Using Liquid Heads

Figures 6-6 and 6-7 (Section 6.2.2.1.2) show two configurations for axial flow measurements in which the liquid is delivered at a constant rate and pressure by means of constant liquid heads. The configuration of Figure 6-7 is particularly useful for accurate, low cost measurements with moderate to high permeability core plugs. No pump or pressure transducer is required. The adjustable-height inlet reservoir and fixed outlet reservoir are both provided with overflow drains. Liquid is allowed to flow into the inlet reservoir from another, higher-elevation supply vessel through a metering valve at a slightly higher rate than the rock sample will accept. Excess liquid flows out the drain vent and is collected for future recycle to the supply vessel. Liquid overflowing from the outlet reservoir drops into a weighing vessel on an electronic balance. Volumetric flow rate, q, is calculated by dividing the rate of mass accumulation (from weight and elapsed time measurements) by the density of the liquid, which must be determined. Permeability is calculated from Equation 25, where ρ is the density of the liquid at the measurement temperature, g is the local value of gravitational acceleration, and h is the elevation difference between the free surfaces of the two liquid heads. Because very little back pressure is exerted, extreme care must be taken to exclude air from all portions of the system, including the core plug.

For very high permeability samples (10 to 40 darcys), the configuration of Figure 6-7 is useful in that the overflow vent in the downstream vessel minimizes variation in the liquid head due to capillary retentive forces. These forces can be significant in the tip of small diameter exit nozzles or ports, creating a large relative variation in the Δp.

6.4 THEORY AND APPLICATION OF UNSTEADY-STATE PERMEABILITY DETERMINATIONS

The advent of high speed data-acquisition systems, accurate pressure transducers, and digital computers has made it not only feasible, but convenient to measure permeabilities under transient, or unsteady-state flow conditions. Transient measurements employ fixed-volume reservoirs for gas or liquid. These may be located either upstream of the sample—from which the gas or liquid flows into the sample being measured, or downstream—into which it flows from the sample, or in both places. When fluid flows from an upstream reservoir, its pressure in that reservoir declines with time. Similarly, when fluid flows into a downstream reservoir, pressure there builds with time. Instantaneous flow rates can be calculated from the volume of the reservoir and the instantaneous rate of pressure change, obviating the need for a flow-rate measuring device.

When fluid expands, it performs flow work at the expense of loss of internal energy. This is observed as a decrease in its temperature. Similarly, when fluid is compressed, work is done on it, and its temperature increases. Because instantaneous flow rates are calculated from rates of pressure change, it is mandatory to maintain isothermal conditions, or to measure instantaneous temperatures and use appropriate mathematical formulations. Equations presented herein assume isothermal conditions.

6.4.1 Transient Pressure Techniques For Gases

Fortunately, gas has low heat capacity, and work-related temperature changes can be virtually eliminated by fabricating reservoirs from material of high thermal conductivity and packing them with copper tubes parallel to the flow axis. Also, where high flow rates are anticipated (with high permeability samples), helium is the gas of choice. Its thermal diffusivity is much higher than that of air or nitrogen.

Other potential thermal problems can arise from changes in ambient temperature in long-duration measurements of low permeability samples, and from Joule-Thomson expansion as gas flows through a sample. The first is minimized with excellent thermal insulation, improved ambient temperature control, and by reducing measurement time. Joule-Thomson expansion is best handled by using helium, which increases in temperature (at ambient conditions) upon expansion, unlike all other gases except hydrogen. This warming tends to counteract work-related cooling.

Two main categories of transient pressure techniques have emerged for measuring permeability in the laboratory. One is called the "pulse-decay" method. It is characterized by using both upstream and downstream reservoirs, one or both of which are relatively small in volume. These and the sample are filled with gas to a fairly high pressure, 1,000 to 2,000 psig, which reduces gas slippage and compressibility. After pressure equilibrium is achieved throughout the system, pressure in the upstream reservoir is increased, typically by 2 to 3 percent of the initial pressure, causing a pressure pulse to flow through the sample. This technique is well suited for low permeability samples, 0.1 millidarcy to about 0.01 microdarcy. Small differential pressures and low permeabilities virtually eliminate inertial flow resistance. Only "late time" techniques will be discussed in this document. They yield overall permeabilities that are comparable to steady-state values. "Early time" transients provide information regarding heterogeneity in samples, which is beyond the intended scope of this document.

The other technique is called the "pressure falloff" method. It is characterized by upstream reservoir(s) only. The downstream end of the sample is vented to atmospheric pressure. The maximum upstream pressure used is fairly low, 10 to 250 psig (varying inversely with the permeability to be measured). A single transient pressure falloff produces data for 6 to 30 separate permeability calculations, each at a different flow velocity and mean pore pressure. Adequate variation of flow conditions during a single transient test makes possible the calculation of slip-corrected (Klinkenberg) permeability (k_∞), Klinkenberg slip factor (b), and inertial resistivity (β) of the porous medium. This technique, which has a useful permeability range of 0.001 to 30,000 millidarcys (through the use of multiple upstream gas reservoirs and pressure transducers), complements the pulse-decay method. For high permeability samples (>1,000 md), where b is small, it is difficult to determine b accurately when a sample is vented to atmospheric pressure. It is more reliably approximated from a correlation.

In addition to physical differences between flow systems, approaches to the derivation of flow equations are quite different for the pulse-decay and pressure-falloff techniques. In the former, Darcy's equation and the continuity equation (which is a statement of the conservation of mass) are solved simultaneously. In the pressure-falloff method, the steady-state solution of the slip-corrected Forchheimer equation is used as a starting point. This solution, which does not properly account for the increase in mass flux with distance along a sample's length at a particular instant (with non-steady-state flow), is differentiated. It is then inserted into the continuity equation, which is integrated to provide a correction (and improvement) to the final flow equation. This process is iteratively repeated until both the Forchheimer and continuity equations are satisfied.

The deviation between steady-state and pressure-falloff solutions thus obtained depends upon the ratio of the sample's pore volume to the volume of the upstream gas reservoir. When this ratio is small, the steady-state solution is nearly exact. As the ratio increases (larger PV or smaller reservoir volume), the correction that must be applied to the steady-state solution progressively increases.

6.4.1.1 Pressure-Falloff, Axial Gas Flow

6.4.1.1.1 Apparatus and Procedure

The pressure falloff apparatus (see Figure 6-22) employs an upstream gas manifold that is attached to a sample holder capable of applying hydrostatic stresses to a cylindrical sample of diameter D and length L. An upstream gas reservoir of calibrated volume can be connected to, or isolated from, the calibrated manifold volume by means of a valve. (Multiple reservoir volumes are used to accommodate a wide range of permeabilities.) The outlet port from the sample holder is vented to the atmosphere. This port is provided with a valve, which can be closed for start-up purposes. An accurate pressure transducer that measures gauge pressure is connected to the manifold immediately upstream of the sample holder.

The reservoir, manifold, and sample—to the outlet valve—are filled with gas. After a few seconds for thermal equilibrium, the outlet valve is opened to initiate the pressure transient. When the upstream pressure has decayed to about 85 percent of the fill pressure, during which time a smooth pressure profile is established throughout the length of the sample, data collection is started. Pressures at selected intervals and corresponding elapsed times are read and recorded.

6.4.1.1.2 Calculations

Procedures for calculating k_∞, b, and β from axial pressure falloff data are given in B.6.8.2.

6.4.1.1.3 Advantages

Advantages of this method include:

a. Slip-corrected permeability, k_∞, Klinkenberg gas slippage factor, *b* (up to about 1,000 md), and Forchheimer coefficient of inertial resistivity, β, can all be determined from a single pressure falloff test.

b. Non-slip-corrected permeability, k_g, can be calculated (see 6.4.1.1.5) from these results for any desired gas at any mean pore pressure for purposes of comparison with conventional measurements.

c. No flow meters are required for the method. Flow rates are calculated from pressure-time measurements.

d. For a core plug of a given permeability, the time required for pressure to fall from one pressure to another pressure is directly proportional to the volume of the upstream gas reservoir. Therefore, measurement times can be controlled for a wide range of permeabilities by providing several reservoir volumes—small for low permeabilities and larger for higher permeabilities.

e. The practical permeability range is from 0.001 to 30,000 millidarcys if multiple reservoir volumes and transducer ranges are used.

f. Departure from Stokes flow is not a concern. This is accounted for in the data analysis scheme.

g. Slip-corrected permeability measurements at high confining stresses more accurately reflect reservoir conditions, especially for low permeability samples.

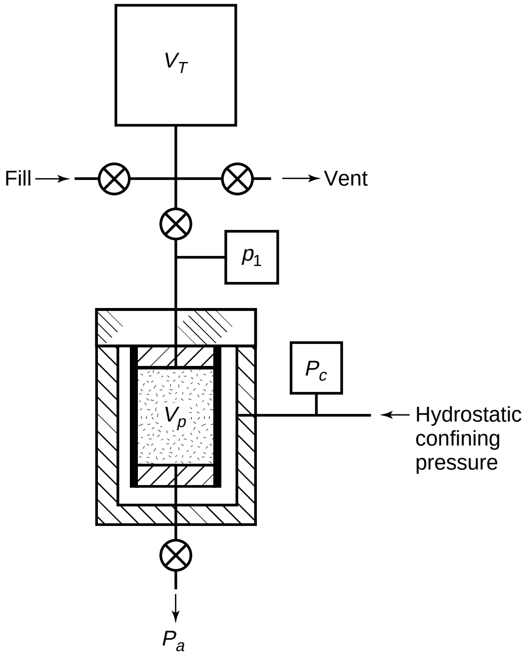


Figure 6-22—Schematic of Pressure-Falloff Gas Permeameter

6.4.1.1.4 Precautions and Limitations

Precautions and limitations of this method include:

a. To minimize thermal effects with high permeability plugs, small fill pressures (<25 psig), small diameter (1 inch), and long lengths (≥2 inches) should be used.

b. To minimize measurement time with low permeability samples, plug diameter should be large (1.5 inches) and its length short (≤1.5 inches).

c. The technique requires high quality pressure transducers, fast data acquisition equipment, and a high computational demand. As a practical matter, therefore, it requires a digital computer.

d. Gas reservoirs must be provided with considerable thermal mass and have high heat transfer capability to avoid work-related temperature changes during gas expansion, especially with high permeability samples. Temperature changes are further minimized by using helium, and by reducing the starting pressure for these falloff tests. High permeability samples also demand large diameter ports in the gas delivery system to minimize internal pressure differentials.

e. Low permeability samples require a leak-tight system (for this or any other technique), and no bypass in the sample holder (past the rubber sleeve). Adequate thermal insulation must be provided to minimize temperature changes induced from ambient changes.

f. Low permeability samples often exhibit extreme stress sensitivity. As mean pore pressure decreases, net stress increases (with a constant confining stress), and permeability decreases. In calculations, permeability is assumed to remain constant during the entire pressure falloff. This assumption can result in the calculation of low or negative β for very stress-sensitive samples. Because the largest changes in permeability occur at low net stresses, stress sensitivity is minimized by making measurements at higher net stresses (>2000 psi), and by reducing the variation in pore pressure during a test.

6.4.1.1.5 Calculation of Gas Permeability From Klinkenberg Permeability

Due to the historical prevalence of steady-state axial gas permeability measurements made without the Klinkenberg correction, huge data bases have been developed from these measurements. It is often desired to compare new data with previous measurements on the same reservoir. To do this, k_∞ must be "uncorrected" for gas slippage. The non-slip-corrected permeability, k_g, for any gas at any desired temperature and mean pore pressure can be calculated from a measured k_∞ and *b* as follows:

a. Calculate the sample's "gas slippage radius," r_b, (from Equation 13 in 6.2.1.1) using appropriate values of molecular weight, viscosity, and the b obtained for the gas used in the measurement at the absolute temperature of the measurement.

b. From this r_b, find b from Equation 12 for the new gas, using its molecular weight and viscosity at the desired temperature.

c. From the new b, the measured k_∞, and the desired mean pore pressure, calculate k_g for the new gas from Equation 21.

These steps can be combined into a single equation:

$$k_g = k_\infty \left(1 + \frac{\frac{b_m \mu_c}{\mu_m} \sqrt{\frac{T_c M_m}{T_m M_c}}}{P_{m_c}} \right) \tag{31}$$

Where the subscript m refers to the measurement gas or condition, and c refers to the desired gas or condition for which k_g is to be calculated. To illustrate the use of Equation 31, suppose that for a particular rock, the measured k_∞ and b are 10.62 millidarcys and 6.57 psi, respectively, using helium at 72°F. For purposes of comparing this measurement with old data taken on the same formation, it is desired to calculate k_{air} at a mean pore pressure of 18.0 psia and temperature of 75°F. The molecular weight of helium is 4.0026, and its viscosity at 72°F is 0.01967 cp. The average molecular weight of air is 29.0, and its viscosity at 75°F is 0.01837 cp. Therefore, from Equation 31, k_{air} at 75°F and 18.0 psia is:

$$k_{air} = 10.62 \left(1 + \frac{\frac{6.57 \times 0.01837}{0.01967} \sqrt{\frac{(75 + 459.7)4.0026}{(72 + 459.7)29.0}}}{18.0} \right) = 11.97 \text{md}$$

A note of warning: many of the earlier measurements were made at low stress levels, typically at 250 or 400 psi. Even with the above correction, the old k_{air} values on comparable rock may be higher if the new measurements were made at significantly higher stresses. Furthermore, old databases may not include the mean pressure and temperature required to perform these calculations.

6.4.1.2 Pressure Falloff, Probe Permeability Measurements with Gases

The pressure falloff technique is easily adapted for probe-permeameter measurements to provide rapid, non-destructive gas permeabilities that are corrected for both gas slippage and inertial resistance. As with steady-state devices, measurements are typically made at high sampling densities and nearly zero stress on slabbed or unslabbed whole core samples.

6.4.1.2.1 Apparatus and Procedure

Figure 6-23 schematically shows the instrument described by Jones.[22] To operate, the upstream volumes are filled with gas (usually dry nitrogen) to a pressure of about 10 psig. The probe is positioned above a sample, and then lowered and sealed against its surface by a fixed pneumatic pressure on the probe operator piston.

The pressure falloff is initiated by opening the internal valve nearest the probe. The appropriate gas reservoir volume for the particular permeability measurement is selected via internal valves in the manifold. This selection is made on the basis of the rate of pressure decline when all internal volumes are connected to the probe. If the rate is slower than a predetermined value, appropriate valve(s) to the larger tank(s) are closed.

Pressures at preselected intervals and corresponding elapsed times are read and recorded for the calculation of k_∞.

6.4.1.2.2 Calculations

Calculation procedures are discussed in C.6.8.3.

6.4.1.2.3 Advantages

Advantages of this method include:

a. The practical permeability range of this technique is from 0.001 to 30,000 millidarcys.

b. Measurement times in this range vary from 35 to 2 seconds.

c. No flow meters are required. Only time-pressure measurements are made.

d. Both slip-corrected (k_∞) and nonslip-corrected (k_g) permeabilities are obtained from a single pressure falloff test.

e. Inertial flow resistance effects, which can be severe even with small pressure differences and low permeability samples, are eliminated from the calculation of permeability with this technique.

f. The method is nondestructive in the sense that there is no need to cut core plugs. It permits high density sampling in sections that exhibit frequent and severe permeability variation.

g. The volume of rock sample investigated by a single test can be varied somewhat by varying the size of the probe tip seal dimensions. However, in general this volume is quite small (the permeability measured is localized). This statement also applies to steady-state measurements (see 6.3.1.2.3).

6.4.1.2.4 Precautions and Limitations

Except for the problems of inertial resistance and gas slippage, which are handled by the calculation procedures, the precautions and limitations that apply to steady-state probe permeability measurements (see 6.3.1.2.4) also apply to the pressure-falloff measurements.

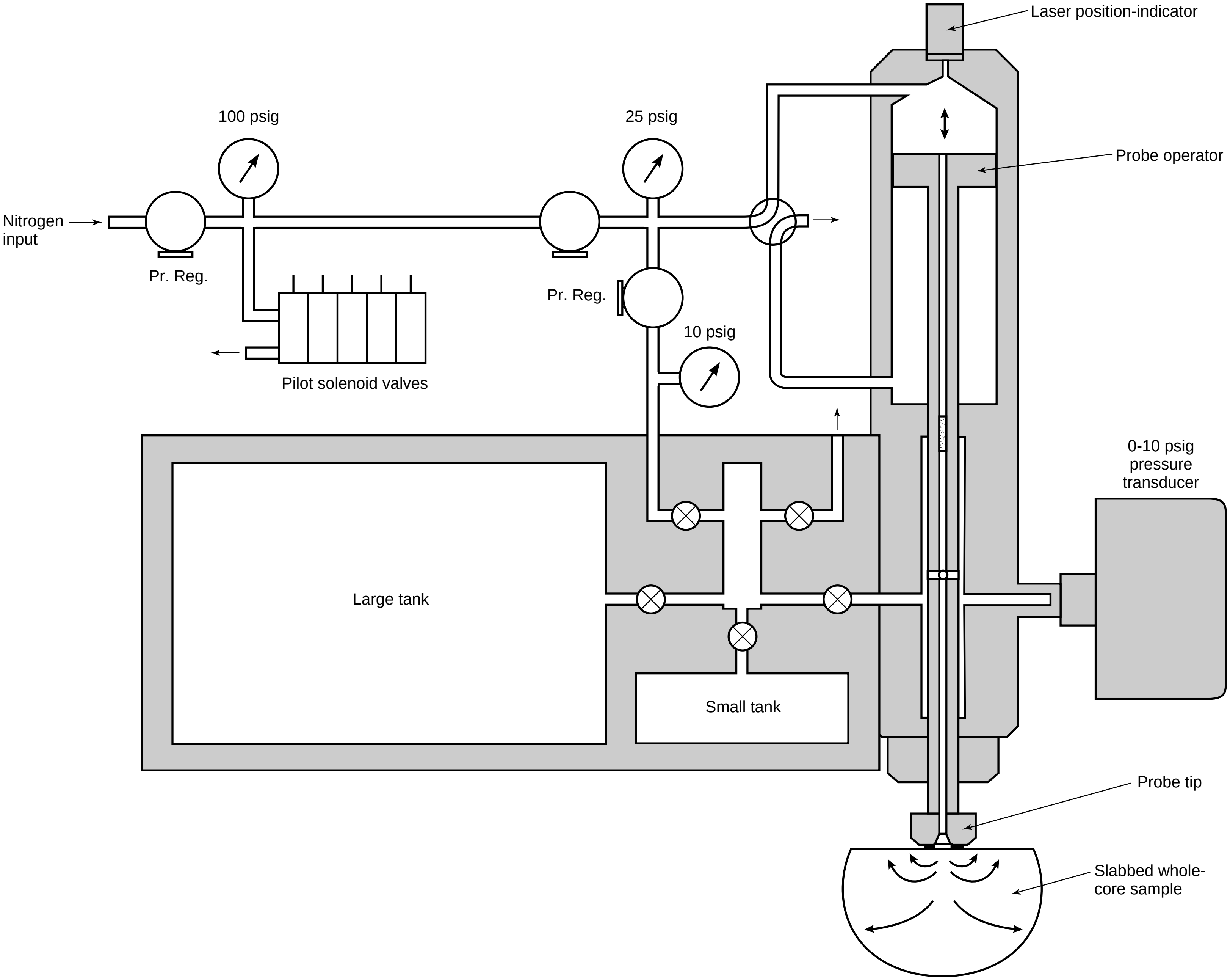


Figure 6-23—Schematic of Pressure-Falloff Probe Permeameter (U.S. Patent 5,237,854)

6.4.1.3 Pulse-Decay, Axial Gas Flow

6.4.1.3.1 Apparatus

The pulse decay apparatus, shown in Figure 6-24, consists of an upstream gas reservoir of volume V_1, a sample holder capable of applying high confining stresses (usually isostatic), which contains the sample of nominal pore volume V_p, and a downstream reservoir of volume V_2. A differential pressure transducer measures the pressure difference between the reservoirs, and a second transducer measures the absolute pressure in the downstream reservoir.

6.4.1.3.2 Procedure

With valves 1 and 2 open, both reservoirs and the sample are filled with gas (usually dry nitrogen) to a pressure typically between 1,000 and 2,000 psig. The fill period must allow adequate time for the gas to diffuse into the (typically) low-permeability sample. After the fill period, the fill valve is closed and the pressure is monitored until no further change is observed, indicating thermal and pressure equilibrium. This stabilized pressure is $P_2[0]$. All valves should be of the type that have no change in internal volume when opened or closed.

After equilibrium, valves 1 and 2 are closed, and the pressure in the upstream reservoir is increased by Δp_1, which is 2 to 3 percent of $P_2[0]$. After the pressure in V_1 becomes stable, i.e., when Δp_1 becomes constant, valve 1 is opened, initiating the pressure-transient portion of the measurement.

6.4.1.3.3 Calculations

See D.6.8.4 for details.

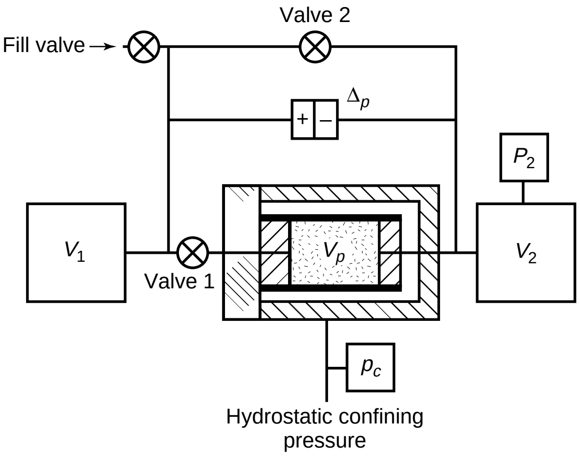


Figure 6-24—Pulse Decay Apparatus for Axial Flow of Gases

6.4.1.3.4 Advantages

Advantages of this method include:

a. The pulse decay technique is applicable to the measurement of very low permeabilities, 0.1 millidarcy to about 0.01 microdarcy, and possibly can be extended in either direction by careful selection of gas reservoir volumes and pressure transducer ranges.

b. No flow meters are required. Only time-pressure measurements are made.

c. These measurements are well adapted for application of reservoir-condition stresses to provide representative permeabilities.

d. Porosity can be measured simultaneously or separately in the same apparatus.

6.4.1.3.5 Precautions and Limitations

Precautions and limitations of this method include:

a. Due to the very low permeabilities that can be measured by this technique, leak-tightness of the apparatus is of ultimate importance. Control of ambient temperature variations is also critical.

b. Even though high back pressures are employed, permeabilities obtained are not corrected for gas slippage and may be somewhat high. For example if b for nitrogen were 100 psi for a particular plug, and the mean pore pressure were 1,000 psia, the gas permeability obtained would be 10 percent higher than k_∞.

6.4.2 Transient Pressure Techniques for Liquids

6.4.2.1 Pulse Decay, Axial Liquid Flow

Unless large pressure gradients are imposed (100 to 1,000 psi/cm or more), liquid permeabilities of tight rocks (k <0.1 millidarcy) are difficult to measure, or are at least time-consuming with steady-state methods. However, permeabilities down to about 0.01 microdarcy can be measured using pulse decay techniques.

Very low flow rates are calculated from rates of pressure change in liquid-filled reservoirs of known compressive storage—as liquid expands from an upstream reservoir or is compressed into one downstream of the sample. Thus, important parameters in these measurements are effective compressive storages of the upstream and downstream reservoirs and of the rock sample. The effective compressive storage of each reservoir is its internal volume multiplied by the sum of the liquid compressibility and the compressibility of the vessel. The sample's compressive storage is the product of its pore volume and the sum of liquid and PV compressibilities. The liquid pulse decay apparatus should have provision for measuring compressive storages of the upstream and downstream volumes (see E.6.8.5.1).

6.4.2.1.1 Procedure

For a pulse decay liquid permeability measurement (see Figure 6-25), a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The high stress minimizes permeability variation from changes in net stress due to varying pore pressure. The system is filled with liquid, as described in E.6.8.5, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse, Δp_1, is generated by screwing in the needle valve. All valves, except for the needle valve, should be of the type that have no change in internal volume when opened or closed.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample,$\Delta p[0]$, will be slightly less than Δp_1, which was generated by the needle valve with valve 1 closed, thereby excluding the upstream dead volume, V_d, from the total upstream volume, V_1. Therefore $\Delta p[0]$ must be calculated, as shown in E.6.8.5.

6.4.2.1.2 Indication of Fairly Homogeneous Sample

The upstream pressure decreases while the pressure pulse traverses the length of the sample, but the downstream pressure remains constant until the pulse "emerges." Thereafter, the upstream pressure continues to decrease, and the downstream pressure rises. If the sample is reasonably homogeneous and upstream and downstream compressive storages are equal, then shortly after the pulse-propagation period, the upstream pressure decrease is offset by an equal downstream increase, and the mean pore pressure will remain constant as the $\Delta p[t]$ continues to decrease linearly—in a semi-logarithmic decay.

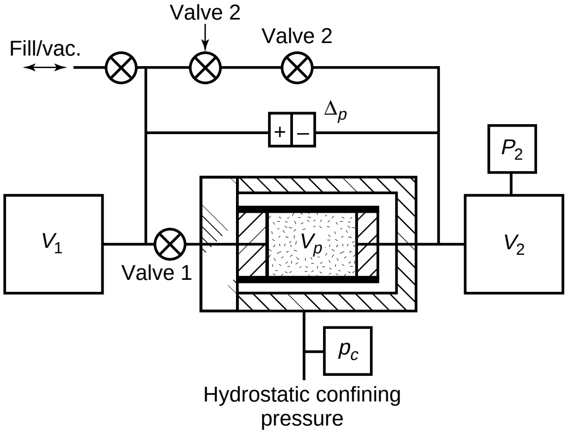


Figure 6-25—Schematic of Liquid Pulse-Decay Permeameter

6.4.2.1.3 Heterogeneous Sample Indication

However, if the sample is not homogeneous, but is characterized by a fracture system of relatively high fluid conductivity and a matrix porosity of low conductivity, and if the compressive storage of the sample is nearly as large, or larger, than those of the upstream and downstream reservoirs (small reservoirs), the mean pore pressure will continue to decrease even after $\Delta p[t]$ has reached zero, and the linearity of the semi-logarithmic pressure decay may have been distorted. In this case $\Delta p[t]$ is fairly quickly dissipated by the fracture system, but liquid continues to diffuse slowly into the tight matrix. If the reservoir storages are large compared to that of the matrix, the mean-pore-pressure decrease, due to liquid movement into the matrix, is barely observable. For a more complete discussion of the measurement of heterogeneity in core samples, see Kamath, et al.[29]

6.4.2.1.4 Calculations

See E.6.8.5.3 for details of calculations for axial liquid pulse decay measurements.

6.4.2.1.5 Advantages

Advantages of this method include:

a. No corrections for slippage are required for liquid measurements.
b. High stress liquid permeability measurements may be more representative of reservoir conditions than gas measurements.
c. This technique is useful for measurement of low permeability rocks (from about 0.01 microdarcy to 0.1 millidarcy).
d. The work of expansion or compression is small in liquid-filled systems. Hence work-related temperature changes are correspondingly small.

6.4.2.1.6 Precautions and Limitations

Precautions and limitations of this method include:

a. Maintenance of isothermal conditions is of utmost importance. Small ambient temperature changes can cause large pressure changes in high-pressure liquid-filled systems.
b. High quality pressure transducers and data acquisition system are required.
c. A leak tight system is essential for high pressure measurements of low permeability plugs.
d. The usual precautions of corrosion resistance, fluid compatibility, careful filtration of liquids, and exclusion of gas from the system must be observed.

6.5 ACCURACY AND PRECISION

6.5.1 Part 1

6.5.1.1 Introduction

Permeability data represent quantitative measurements, and correct values are important. All analytical methods are subject to random scatter and to operator, equipment, and technique-related errors, which, taken together, comprise experimental uncertainty. To distinguish between a real trend in a data set and variation due to experimental uncertainty, it is recommended that core analysis data be reported together with a statement of the uncertainty with which these data were recorded.

6.5.1.2 Definitions

6.5.1.2.1 precision (or reproducibility) of a measurement: An expression of the limits within which the measurement, with a stated probability, can be reproduced provided that the measurement is subject only to stochastic variation. Precision is determined from repeated measurements on a single sample, and is normally given as a standard deviation or relative standard deviation, also known as the coefficient of variation.

6.5.1.2.2 accuracy of a measurement: An expression of the closeness of agreement between the experimentally obtained value and the true value. The difference between the measured and the true value is called the error. Results falling outside the maximum allowable error or tolerance may be caused by operating errors or systematic errors.

6.5.1.2.3 bias (or systematic) error: A deviation that persists during a measurement cycle and affects all measurements in the same way. Bias is normally due to instrumental drift, or faulty calibration, or operating procedures.

6.5.1.2.4 applicability: The concept of applicability, in addition to precision and accuracy, is appropriate here. Permeability measurements may have been performed with high precision and accuracy, producing results close to true values of permeability for the reported conditions under which they were measured. Yet, these precise, accurate values might be quite different from those that would be obtained for conditions that exist in the formation from which the samples were taken. Reasons for differences include different stress conditions, rock-fluid interaction, differences in pore pressure, or gas composition if gas slippage is ignored, etc.

6.5.1.3 Error Statements in Permeability Measurements

The concept of accuracy requires that the true value of a sample be known. This is usually not the case. Instead certified standard reference materials for scientific or industrial use are obtained from recognized standard institutions. These materials are analyzed at regular intervals together with routine samples. Accuracy is then obtained by comparing the mean value, calculated from repeated analyses on the standard material, with the certified value for that standard.

In permeability measurement, suitable standard materials could be natural rocks or permeable synthetic materials. However, synthetic samples cannot yet be produced in a way that allows an accurate pre-determination of permeability. Therefore, each standard must pass an extensive testing program before it is certified, and afterwards there is risk that the standard will change during regular handling in the laboratory. So far, no certified permeability standards have been available from recognized standard institutions. Therefore, a different approach for determining accuracy in permeability measurement is recommended.

6.5.1.4 Internal Standards

The concept of internal permeability standards is introduced for determining accuracy, and as a facility in testing equipment for bias or systematic errors. Internal standards can be synthetic and/or natural samples covering a large range of permeability values, which have been analyzed repeatedly by different operators on different (carefully calibrated) apparatus using the required fluid. When many measurements have been collected, a mean value and standard deviation is calculated for the sample. If the variation about the mean is small, i.e., the standard deviation is low, the sample is accepted as an internal standard, and can be used routinely to check accuracy of equipment and correct for bias in the results.

Synthetic porous and permeable media are available in different materials, e.g., as porous ceramics and glasses, and as sintered plastic, glass, or metal beads. These materials are often supplied from the manufacturer with a specified permeability value, which, however, should only be regarded as approximate.

6.5.1.5 Methodological Errors

All equations for calculating permeability presented herein assume fluid flow through homogeneous, isotropic samples, under isothermal conditions. Thus they define an average or effective permeability in the overall direction of flow dictated by the experiment (i.e., axial, radial, transverse, etc.). Unless the internal, macroscopic structure of each sample is determined by independent means, it is virtually impossible to model the flow otherwise. Because real rocks are almost *never* isotropic or homogeneous, the assumption that they are will invariably distort actual flow patterns, and consequently, the calculated permeability, and also inertial resistivity and gas slippage factor, whenever they apply.

Equations for unsteady-state analyses require correction for nonconstant mass flow (with distance from the injection point) at any instant during the pressure transient. Therefore, a sample's pore volume must be determined, either independently or in conjunction with the permeability measurement itself. Inaccuracies in the estimation of pore volume will lead to permeability errors, especially when *PV* is nearly as large, or larger than the gas reservoir volume. Small *PV* errors are inconsequential when reservoir volumes are large on a relative basis.

Furthermore, the transient techniques assume constant permeability throughout the duration of a test. Changes in pore pressure change the net stress to which the sample is subjected, thereby slightly changing its permeability during a test. In a pressure falloff measurement, this can distort the calculation of inertial resistivity (β). These errors are minimized by limiting the magnitude of the pressure falloff, and by increasing the confining stress (where changes in net stress have less effect on permeability—per Figure 6-9).

Serious errors can result when operating conditions are such that Darcy's law does not apply because of significant inertial resistance. These are most prevalent with steady-state, axial flow gas measurements on high permeability plugs (several darcys), or steady-state probe permeability measurements on samples of about 10 millidarcys or more. These errors are minimized by using very small values.

Other errors can be introduced by improper sample preparation techniques, such as inadequate cleaning, or nonremoval of salt crystals from extracted samples that previously contained nearly saturated brine. Improper drying of samples with high clay content can either enhance or reduce permeability. Embedded fines from cutting or grinding the ends of samples can reduce permeability. High points on nonsquare ends of a core plug can be crushed at high axial stresses, altering permeability. Incompatible or dirty liquids cause plugging. High velocity fluids (especially liquids) can cause fines migration and plugging.

Gas permeabilities (uncorrected for gas slippage), measured at low confining stress, if properly reported (including the gas used, mean pore pressure, and net stress), are properly specified and do not per se constitute an error. However, if they are expected to predict reservoir performance accurately without correction, they may not be particularly applicable, especially for lower permeability samples, where the corrections are substantial.

6.5.1.6 Equipment Component Errors

Main error sources associated with test equipment are leaks, sample bypass, pressure transducer error, flow meter error, and ambient temperature variation. Additionally, nonisothermal fluid expansion, inaccurate calibration of reservoir volumes, and inadequate data acquisition resolution and speed can cause errors in unsteady-state measurements. With probe permeameters, probe positioning accuracy and sealing force repeatability are also important.

6.5.1.6.1 Leaks and Sample Bypass

All types of permeameters are subject to leaks in tubing, fittings, valves, transducers, flow meters, and core holder. Bypass of fluid between a core plug and rubber sleeve of the sample holder, or between a sample and the probe seal of a probe permeameter, also constitutes a leak. The magnitude of error caused by a leak generally increases as permeability decreases. For example, a small leak in the measurement of a 3 darcy plug would barely affect its calculated permeability, whereas the same leak rate with a 0.001 millidarcy plug would be disastrous.

Large leaks can be located in gas permeameters with the aid of a soap solution that forms bubbles when applied to a leak. Small leaks are most easily located by means of pressure measurements. Various portions of the flow system are pressurized with gas, then monitored by the system's pressure transducer. The leak is located through a process of elimination. It may be necessary to load an impermeable steel plug in the sample holder to isolate the upstream and downstream portions of the apparatus. Some systems incorporate automated leak-detection routines.

Sample bypass usually can be eliminated by increasing confining stress and/or reducing the hardness (durometer) of the rubber.

6.5.1.6.2 Pressure Transducer Error

Accuracy of a pressure transducer is usually specified as the root sum of squares (RSS) of deviations of its output from actual pressures throughout its range as a percent of its full scale (FS) reading. RSS accuracies of 0.5 percent FS or better are generally available. Most transducers are provided with at least two adjustments—zero and span. The zero adjustment is an offset to permit zero electrical output at zero applied pressure. The span adjustment changes the transducer's gain or multiplier that converts its electrical output to a pressure. Some transducers also have one or more internal linearity adjustments. Pressure transducers are also affected by temperature changes, and are provided with temperature compensation. Price is often nearly proportional to the quality of the temperature compensation.

The user should keep in mind consequences of the "full scale accuracy" specification. For example, a 0.5 percent transducer, used at 10 percent of full scale, can have a 5 percent error in the value of its pressure reading, and be within specification. In view of the RSS definition, the particular error at a given point could be greater than the given RSS percent of the full scale reading.

With proper calibration techniques that allow for frequent mathematical rezeroing capability and nonlinear curve fitting, as outlined in 6.61, a transducer's repeatability is among its most important characteristics. High quality transducers with 0.01 or 0.02 percent FS repeatability are available. To minimize hysteresis effects, a transducer should always be calibrated in the direction of its anticipated use. For example, for pressure falloff measurements, a transducer should be calibrated to its full scale pressure first, followed by a sequence of descending pressures.

Unsteady-state techniques place extra demands on pressure measurements. Pressure-time derivatives are calculated. Therefore noise, or small voltage fluctuations must be minimized, and resolution of the reading must not be truncated by an analog-to-digital voltage converter (ADC) with too few bits. Noise is minimized by starting with a good quality transducer with low noise output and shielded cables. An analog, low-pass filter can be used in conjunction with digital filtering. With a reasonably fast ADC (1,000 conversions per second) multiple readings can be taken, then averaged. A slow, integrating ADC (60 conversions per second) performs averaging automatically. A 15 bit ADC (0.0031 percent of full scale resolution) minimum is recommended for unsteady-state measurements to minimize truncation error.

High quality timer cards are available, with accuracies in the part per million range. It is imperative to query the timer card immediately either before or after reading the ADC, with no intervening commands in the operating program, so that pressure intervals correspond exactly to time intervals for the calculation of pressure-time derivatives. Furthermore, pressure intervals must be sufficiently large that noise in the measurements does not destroy accuracy of the derivative calculation. Optimum intervals may have to be determined by trial and error for a particular system.

A non-smooth plot of pressure-time derivatives vs. geometric mean pressures from pressure falloff data indicates either excessive noise or truncation of time or pressure readings. The problem is usually eliminated by increasing the pressure and/or time intervals, increasing the resolution (number of bits) of the ADC, or by improving the quality of the transducer. The sampling routine should be written in assembly language for maximum speed.

6.5.1.6.3 Flow Meter Error

Steady-state techniques depend on accurate flow rate determination. Direct reading volumetric devices in combination with time readings can yield accuracies of better than 1 percent. Indirect devices (calibrated orifices, mass flow meters and controllers) can normally be adjusted to yield a precision better than 1 percent, but accuracy is totally dependent upon calibration. Several flow meters are required to cover the entire permeability range. Each one should be used only within its linear working range. See 6.6 for mass flow meter calibration.

6.5.1.6.4 Ambient Temperature Variation

Changes in temperature affect both volumetrics and fluid viscosity. Temperature should be kept constant during a permeability measurement. Never place the apparatus near a heating or cooling vent or near a window where the sun's radiation can cause large temperature changes. In unsteady-state equipment, the rate of temperature change within fluid reservoirs must be such that temperature-induced pressure changes are small relative to pressure decreases that are caused by flow of fluid from a reservoir.

In field applications of probe permeameter measurements, temperature changes affect fluid viscosity, flow rate and pressure measurements.

6.5.2 Part 2

6.5.2.1 Error Statements in Reporting

Presentation of measured data with associated error limits in a core analysis report can be based on two different approaches: (a) measured values are used to calculate and report a mean value with associated error limits as calculated below, and (b) measured values are given together with a statement of accuracy, which the laboratory can base on repeated measurements of internal standards.

6.5.2.1.1 Calculated Error Limits

Suppose that a large number of repeated measurements (normally more than 100) are performed on a single sample. If the measurements are only subject to stochastic variation, it is generally believed that data have a normal or gaussian distribution with a mean value, $\bar{X}$:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_1$$

and standard deviation, *s*:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(X_i - \bar{X})^2}{n-1}}$$

Where $(X_i - \bar{X})$ is the deviation of a single measurement from the mean and n is the total number of measurements. The result is reported as $\bar{X} \pm s$. The standard deviation is a measure of the spread or variation of data about the mean. A normal distribution has a probability that 68.3 percent of the observations will fall within the limits of plus or minus one standard deviation from the mean. The coefficient of variation, *CV*, is the relative standard deviation expressed as a percentage:

$$CV = 100(s/\bar{X})$$

Often, only a limited number of measurements are recorded. This means that we are dealing with small sample statistics and the *t*-distribution should be applied. Often it is more relevant to define confidence intervals about the mean than to quote the deviation. The confidence interval is an estimate of the limits within which the true value lies with a stated probability. When reporting core analysis results it is therefore recommended to quote results as:

$$\overline{X} \pm t(s/\sqrt{n-1})$$

which is the confidence interval. Values of t are found in most statistical textbooks or in statistical tables, e.g., Fisher and Yates[31]. These values are given for various "degrees of freedom," n–1, and for different "confidence levels" or probabilities. A 95 percent confidence level is often used (corresponding to standard deviations, adjusted for the small number of repeat measurements).

The following example illustrates these concepts. Three repeat measurements were made on a sample. Calculated permeabilities are 3.35, 3.18, and 3.42 millidarcys, from which the mean and standard deviation are calculated:

$$\bar{X} = 3.32 \text{ md}$$

$$s = 0.12 \text{ md}$$

The value of t for 2 degrees of freedom (= n–1, where n = 3 measurements) and a 95 percent confidence level is 4.303. Therefore the confidence interval is:

$$3.32 \pm 4.303(0.12/\sqrt{3-1}) = 3.32 \pm 0.37 \text{ md}$$

In other words, these calculations imply that with a probability of 95 percent, the true permeability lies within the interval from 2.95 to 3.69 millidarcys, provided that the measurements were not biased.

Klinkenberg permeability is calculated from gas permeability measurements by performing linear regression on permeability vs. reciprocal mean pore pressure values. The goodness of fit of the regression line can be assessed from the coefficient of correlation, r, which should be reported with the permeability value. If we still want to have a confidence level of 95 percent, r must be greater than or equal to 0.95 for a 4 point regression line, and at least 0.997 for a 3 point line. Samples giving values of r lower than these should be inspected for fractures, vugs, or other irregularities, and if present this should be reported in the data listing. Also the measurements should be checked for the possibility of non-negligible inertial resistance at the higher mean pore pressures.

6.5.2.1.2 Laboratory Reported Error Limits

In routine core analysis, a large number of determinations on internal standard plugs will be accumulated over a period of time. It is valid that a laboratory prepare a table showing the accuracy and precision obtained for different permeability ranges based on standard measurements.

An impression of the accuracy that can be obtained in permeability measurements was reported by Thomas and Pugh[32]. Using conventional steady-state measurements performed by many laboratories all over the world on sets of standard plugs, they quoted the statistically derived confidence intervals with a probability (confidence level) of 99 percent from which the other confidence intervals were calculated in the table below.

k_g range, millidarcys	Confidence interval, %, with probability of: 68.3% (= mean CV)	95%	99%
0.01-0.1	±8%	±16%	±21%
0.1-1.0	8	16	21
1-50	5	10	13
50-1,000	3	6	8

The numbers shown in this table can be interpreted as follows. Suppose a certain core plug has a true permeability of 10.0 millidarcys, which lies in the 1-50 range above. Thus, if a total of one thousand measurements were made on this plug by laboratories throughout the world, approximately 683 of the reported permeabilities (68.3 percent of the total) would be expected to lie within the range from 9.5 to 10.5 millidarcys (±5 percent of the true value). Similarly 950 of them would be expected to lie within 9.0 to 11.0 millidarcys, and 990 within the range from 8.7 to 11.3 millidarcys (with 10 measurements lying outside this range).

6.5.2.1.3 Number of Significant Figures

The number of figures given in a report should respect the error associated with the measured value. For example, 4.2357, ±0.0327 md should be reported as 4.24, ±0.03 md; i.e., the error is understood to occur on the last decimal in the rounded number. In view of the table above, probably only two significant figures are justified (i.e., 4.2 md), unless a particular laboratory produces significantly better results than those upon which the table is based.

6.6 CALIBRATION OF INSTRUMENTS

6.6.1 Pressure Transducer Calibration

This discussion applies to a gauge pressure transducer, which should read zero when it is exposed to ambient atmospheric pressure. It also applies to an absolute transducer, provided that a high vacuum is applied for "zero (absolute) pressure" readings. Any pressure transducer must be calibrated periodically. Even though a transducer is normally calibrated at the factory within stated tolerances, it should be recalibrated in its flow system configuration, with the system's analog-to-digital converter (ADC). This is accomplished with a high quality primary standard, such as a dead weight tester or precision manometer and cathetometer, or with a secondary standard pressure transducer (preferably accurate to 0.01 to 0.02 percent full scale), traceable to a primary standard. A known pressure is applied, and the resulting voltage or ADC output is read. This is repeated at several, nearly equally spaced intervals throughout the range to be calibrated, including zero pressure. A 21 point calibration (intervals of 5 percent of full scale), minimum, is desirable.

6.6.1.1 Calculations

These data (if reasonably linear—to within 0.1 to 0.5 percent of full scale) are fit with a polynomial, using least squares techniques. A good rule of thumb for choosing the polynomial order is: the number of data points taken in the calibration must be at least double the number of coefficients calculated from the polynomial fit. For 21 points or more, a fourth-order polynomial is a good choice.

In general, it is nearly impossible to set the transducer's zero-adjust potentiometer so that the transducer outputs exactly zero voltage at zero pressure at the time of calibration. Furthermore, some ADCs read only positive voltages (negative voltages are output as zero). For these ADCs, the transducer's zero offset must be sufficiently positive that zero drift will never cause a negative zero-pressure voltage. A fourth-order polynomial, with which calibration data are fit, has the form:

$$p = a_0 + a_1 v + a_2 v^2 + a_3 v^3 + a_4 v^4 \tag{37}$$

Where p is the actual pressure (determined from the standard), and v is the voltage read from the transducer for each data point. If the zero-pressure voltage is positive, then a_0, from the least-squares fit, will be negative.

It is critical to have calibrations that pass through zero at zero pressure. To illustrate, suppose that a zero-pressure voltage shift of 0.5 percent of the full scale voltage occurred—with time and temperature changes—from the zero-pressure voltage at the time of calibration. This magnitude of zero shift is not uncommon, and does not appear to be too bad. However, even with a perfect span adjustment, this represents a 5 percent error for a pressure reading at 10 percent of full scale, or a 25 percent error for a pressure reading at 2 percent of full scale.

To avoid this problem, operating software should have provision to "mathematically zero" all transducers. Unless a_0, from the calibration, is identically equal to zero in Equation 37, zero drift cannot be corrected by simply subtracting the current zero-pressure voltage from all subsequent voltage readings, then using these voltage differences in Equation 37. Instead, Equation 37 must first be mathematically transformed to:

$$p = A_0 + A_1(v - v_0) + A_2(v - v_0)^2 + A_3(v - v_0)^3 + A_4(v - v_0)^4 \tag{38}$$

To accomplish this transformation, the "best fit" zero-pressure voltage, v_0, at the time of calibration, must be found. This voltage is calculated from all points in the calibration, not just the value read when zero pressure was applied. A first estimate is calculated from:

$$v_0 \approx -a_0 / a_1 \tag{39}$$

This value of v is inserted into Equation 37 to calculate the "best fit" pressure (p_0) that corresponds to this voltage. If the value of v_0 were correct, then p_0 would be exactly equal to zero. The estimate is refined using:

$$v_0[new] = v_0[old] - p_0 / a_1 \tag{40}$$

This new value of the zero-pressure voltage is inserted into Equation 37 to calculate a refined value of p_0. Then these values of v_0 and p_0 are used in Equation 40 to produce a further refinement of v_0. This process is continued until $p_0 \leq 1 \times 10^{-10}$ psig.

Next, the coefficients $A_0 \ldots A_4$ are determined by a least-squares fit of the original calibration data using Equation 38 with the final value of v_0 as determined above. Equations 37 and 38 yield exactly the same calibration, except Equation 38 (after its coefficients have been determined) permits elimination of zero drift by inserting the current value of v_0.

The coefficients $A_0 \ldots A_4$, for a particular transducer, must be installed in an appropriate file in that transducer's operating program. The current value of v_0 must be periodically determined by the operating program of a particular instrument. It is found by pressurizing the transducer to its maximum pressure normally used in the instrument. It is then vented to atmospheric pressure and allowed to stabilize (usually for 30 seconds), after which v_0 is read and stored for subsequent computations with Equation 38.

The fit by a fourth-order polynomial reduces the average absolute deviation by a factor of 4 to 20, compared to that of a *linear* least-squares fit. Equation 38 also eliminates zero drift. The overall improvement (especially at low pressures) is significant compared to a calibration that has not been corrected for zero drift. The coefficient A_0 in Equation 38 should theoretically be equal to zero. It is usually less than 1×10^{-8}, and is calculated and reported only to assure that the transformation of Equation 37 to Equation 38 was successful. For purposes of calculating pressure from an ADC-output, A_0 should be deleted from Equation 38.

6.6.2 Calibration of Mass Flow Meters

Gas mass flow meters respond to mass flow rate, not volumetric flow rate. The volumetric flowrate at some reference condition of temperature and pressure (usually the current ambient temperature and atmospheric pressure), of gas flowing through the mass flow meter, is measured by some independent device (such as a soap bubble meter for low flow rates, which is connected in series with the mass flow meter).

Alternatively, a floating piston with mercury seals in a precision bore volumetric displacement device may be used. Voltage (or some scaled reading) from the mass flow meter is often calibrated against this volumetric flow rate, thereby providing an equation from which volumetric flow rate can be calculated from a voltage acquired. This is not the correct procedure, and unnecessarily limits the calibration! This volumetric flow rate calibration is valid only if the mass flow meter is used at exactly the calibration temperature and pressure. Changes in either pressure or temperature cause errors in the calibration, which could be avoided by simple procedural changes.

Because the mass flow meter's output is proportional to the mass flow rate of a gas, the volumetric flow rates through the bubble meter during a calibration must be multiplied by the absolute pressure in the standard meter (not in the mass flow meter). This is normally the current atmospheric pressure, which can be read from a good quality mercury barometer, with appropriate corrections. This pressure must be converted to the same units that are used in the calculation of permeability (e.g., psia, Pa, or atm). In addition, the volumetric flow rates must be divided by the absolute temperature, (459.67+°F), or (273.15+°C), in the standard meter, and by z, the gas deviation factor, calculated at the temperature and pressure within the standard meter. We will define the result of this manipulation as the "mass flow factor," y_{mf}:

$$y_{mf} = \frac{qP_{cal.}}{z_{cal.}T_{cal.}} \tag{41}$$

The subscripts, *cal.*, above refer to the absolute pressure and temperature that correspond to the volumetric flow rates measured during the calibration.

The advantage of this procedure is that the calibration is not restricted to exactly the same run-time pressure and temperature. Except for small instrumental shifts, the mass flow meter could be operated at any pressure and temperature within its specifications. This often, for example, permits the flow meter to be used at upstream conditions. Upstream operation has advantages: (a) no sand or grit from the core plug enters the flow meter, degrading its accuracy, (b) there is virtually no flow resistance between the mass flow meter and the core plug, allowing faster attainment of steady-state conditions.

Because of instrumental shifts, the mass flow meter should be calibrated at several different pressures and temperatures for most accurate results. Remember that, even though calibrations are reported for conditions in the mass flow meter, the pressure and temperature in the bubble meter (or other volumetric device) are used to calculate the mass flow factors. Normally, effects of temperature and pressure changes are small, and can be interpolated easily.

6.6.2.1 Calculations

For purposes of fitting calibration data, the same procedure should be used as described for pressure transducers, to produce a calibration equation that can be corrected for zero drift. In Equations 37, 38, and 39, the p and p_0 are replaced by y_{mf} and y_{mfo}, respectively. If only 6 to 8 flow rates are calibrated at each pressure, then the fourth-order polynomial should be replaced by a second order polynomial. A minimum of 11 equally spaced points should be obtained for the fourth-order polynomial. Whatever polynomial is used, it should be carefully tested by calculating 50 to 100 points over the entire range to make sure that the final equation does not produce unexpected results between calibration points.

Voltage (or the instrument's reading) at zero mass flow rate is determined at run time by closing valves on both the upstream and downstream ends of the mass flow meter (at its current operating pressure) and ensuring that there is absolutely no flow through it. After 30 seconds for stabilization, output from the ADC, v_0, should be recorded and subtracted from the flowing voltage output, in the flow-equivalent of Equation 38.

Mass flow factors are used in the calculation of gas permeability as follows:

$$k_g = \frac{29392\mu z_m y_{mf} T}{G_f(P_1 - P_2)(P_1 + P_2)} \tag{42}$$

Where:

k_g = non-slip-corrected gas permeability, millidarcys.
μ = gas viscosity, cp.
z_m = gas law deviation factor, calculated at the mean pore pressure and current temperature.
y_{mf} = mass flow factor, (cm^3/s)(psia/°R), or (cm^3/s)(psia/°K).
T = absolute temperature at run time; same units as $T_{cal.}$, °R, or °K.
G_f = dimensional geometric factor for a particular flow configuration, cm.
P_1 = absolute upstream pressure, psia.
P_2 = absolute downstream pressure, psia.

In other words, $y_{mf}T$ is used in place of q_rP_r / z_r in the calculation of permeability. If other units than the above are used in Equation 42, the 29392 is replaced by $2C_2 / C_1$ (see Table 6-1).

6.6.3 Reference Volume Calibration

Unsteady-state gas permeameters contain gas reservoir volumes and dead volumes that must be accurately calibrated. One convenient method is to use gas expansion in conjunction with Boyle's Law, using seven or eight cylindrical steel plugs, each of which contains an accurately-measured axial hole that extends through its entire length. The length and diameter of each hole should be measured to the nearest 0.0001 inch. These plugs must fit inside the sample holder, and must be tightly sealed by the holder's rubber sleeve. The holes should range in volume from nearly zero to about the volume of the reservoir to be calibrated. The hole volume changes, from one plug to the next, should all be approximately equal. The pressure transducer must be carefully calibrated prior to the volumetric calibration, and it must read zero at zero psig. Ambient temperature changes during the volumetric calibration should be minimized.

6.6.3.1 Calculations

Because the configuration of each system may vary slightly, only the principles of a Boyle's Law calibration will be illustrated. Boyle's Law states that the total of the masses of gas in each part of the system before an expansion is equal to the total mass after the expansion; i.e., no gas enters or leaves the system. Under isothermal conditions, the group (VP/z) is proportional to the mass of gas in volume V, where P is the absolute pressure, and z is the gas deviation factor at the pressure and temperature of the gas contained in volume V.

Referring to Figure 6-26, suppose that one of the several steel plugs has been loaded into the sample holder and is sealed by its rubber sleeve. Then the gas reservoir of volume V_0, that includes the bore of the isolation ball valve; the upper dead volume V_1, which includes the volumes inside the transducer and upper end plug; the known hole volume in the steel plug, V_H; and the lower dead volume, V_2, are filled with helium or nitrogen to pressure p_1 (a gauge pressure). For the most accurate work, this gas should be vented from the system to remove air. The fill-vent cycle should be repeated, and the system filled a third time. Then the isolation valve is closed, and V_0 is vented to atmospheric pressure, after which the vent valve is closed. After pressure equilibration, the final pressure, p_1, is read and recorded.

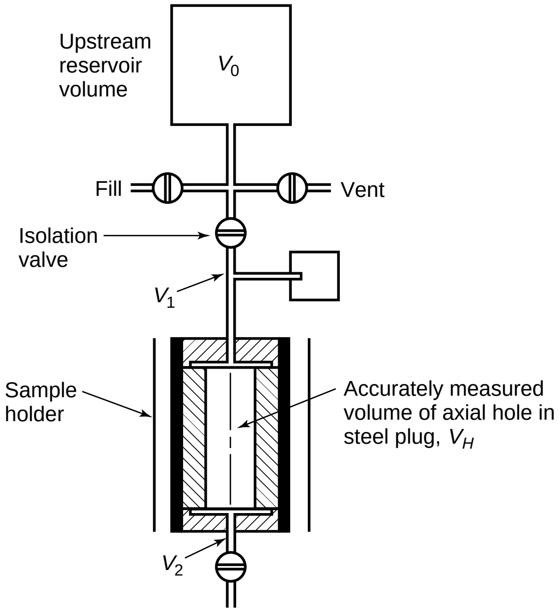


Figure 6-26—Schematic for Calibration of Volumes in Unsteady-State Permeameters

The isolation valve is now opened, expanding gas from the lower portions of the system into V_0. After pressure equilibrium is attained, the final pressure in all parts of the system, p_2, is read and recorded. Each of the remaining steel plugs is loaded into the sample holder and the procedure repeated. Atmospheric pressure, p_a, must be read from an accurate mercury barometer with appropriate corrections.

Now the mass of gas in each part of the system before an expansion is entered on the left hand side of the equation below, and the masses after the expansion are on the right hand side:

$$(V_1+V_2+V_{H_i})\left(\frac{p_1+P_a}{z_1}\right)+(V_0)\left(\frac{P_a}{z_a}\right) = (V_1+V_2+V_{H_i}+V_0)\left(\frac{p_2+P_a}{z_2}\right) \tag{43}$$

A similar equation is obtained for each of the steel plugs. These equations are rearranged to:

$$V_{H_i} = V_0\left[\frac{\dfrac{p_2+P_a}{z_2}-\dfrac{P_a}{z_a}}{\dfrac{p_1+P_a}{z_1}-\dfrac{p_2+P_a}{z_2}}\right]_i-(V_1+V_2) \tag{44}$$

The volumes V_0 and $-(V_1+V_2)$ are now found as the slope and intercept, respectively, from linear regression, where V_H is the y-variable, and the quantity inside the brackets is the x-variable for each plug.

The upper dead volume, V_1, is found by inserting a solid steel plug into the core holder. Gas is expanded from this volume into the now known volume, V_0. An equation similar to Equation 43 is written for the new configuration, and solved for V_1. The reservoir volume, V_T, for a transient permeability measurement is equal to V_1 when the isolation valve is closed, or to (V_0+V_1) when it is open.

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6.8 APPENDICES

A.6.8.1 Derivation of Equations to Estimate the Maximum Δ*p* for Darcy’s Law to Apply

The “condition of Stokes flow,” which is a requirement for the legitimate use of Darcy’s equation, is met when inertial flow resistance is negligible relative to viscous flow resistance. The purpose of this appendix is to present equations for estimating the maximum allowable Δp to be used in axial flow permeability measurements. If this maximum is not exceeded, Darcy’s equation can be safely used in most instances. Equations will first be derived for gases, followed by an equation for the flow of liquids.

A.6.8.1.1 Gases

When Equation 17 (see 6.2.1.2), the differential Forchheimer equation for steady-state flow of gases, corrected for gas slippage, is integrated with respect to length it becomes:

$$\frac{C_1 \Delta p}{L} = \frac{C_2 \mu P_r q_r z_m}{A k_\infty z_r (P_m + f_{Fo} b)} + \frac{C_3 \beta M P_r^2 q_r^2 z_m (P_m + b)}{A^2 R T z_r^2 P_m (P_m + f_{Fo} b)} \quad \text{(A-1)}$$

When the second term on the right hand side is small relative to the first term, f_{Fo} is virtually equal to 1.0. If we assume this value, set all gas deviation factors to 1.0 (which is a good assumption for low pressures), and choose the arithmetic mean pressure, P_m, to be the reference pressure, P_r, for which q_r is calculated, then Equation A-1 simplifies to:

$$\frac{\Delta p}{L} = \frac{C_2 \mu\, v_m}{C_1 k_\infty \left(1 + \dfrac{b}{P_m}\right)} + \frac{C_3 \beta M P_m v_m^2}{C_1 R T} \qquad \text{(A-2)}$$

where v_m, the Darcy velocity, is equal to q_m / A. This can alternatively be written:

$$\frac{\Delta p}{L} = \frac{C_2 \mu v_m}{C_1 k_\infty \left(1 + \dfrac{b}{P_m}\right)} \left[1 + \frac{C_3 k_g \beta M P_m v_m}{C_2 \mu R T}\right] \qquad \text{(A-3)}$$

In the numerator of the second term inside the brackets, k_g replaces the group $k_\infty(1 + b / P_m)$, per Equation 21. This entire second term is dimensionless, and can be thought of as a “Reynolds number for flow through porous media.” Ruth and Ma[33] suggest that this number be called the “Forchheimer number:”

$$N^*_{Fo} = \left(\frac{C_3}{C_2}\right)(k_g \beta \phi)\left(\frac{M P_m}{R T}\right)\left(\frac{v_m}{\phi \mu}\right) \qquad \text{(A-4)}$$

The factors on the right hand side of Equation A-4 are grouped into the various components of the Forchheimer number, which is the ratio of inertial to viscous resistance. The first factor contains constants (see Table 6-1) to make the dimensions consistent. The next factor is the characteristic length, analogous to pipe diameter in a Reynolds number for fluid flow through pipes. The next factor can be recognized as gas density at the mean pore pressure. The final factor contains fluid velocity divided by its viscosity. The velocity is obtained by dividing Darcy velocity by rock porosity. Thus it is based upon the average actual area available for flow, not the superficial area. The product of this velocity and gas density is the mass flux of gas through the plug. The porosity that was included in the characteristic length cancels porosity in the final factor.

Unlike pipe flow, where there is a sudden transition from laminar to turbulent flow over a narrow range in the Reynolds number, inertial resistance associated with flow through porous media increases gradually and smoothly as the Forchheimer number increases. This difference can be explained easily. In laminar pipe flow, fluids are in parallel, streamline flow with virtually no accelerations. Suddenly, at some critical velocity the flow becomes unstable and suffers numerous changes in direction, thus undergoes many accelerations. By contrast, flow through a porous medium is tortuous at any velocity, and involves many accelerations, which increase in magnitude smoothly as mass flux increases. Also, there is a whole spectrum of equivalent pipe sizes in a porous medium. Therefore, sudden transitions from laminar to turbulent flow, with a large increase in the pressure gradient, are not observed.

When the Forchheimer number is zero, all flow resistance is due to viscous shear, and Darcy's law is completely applicable. When it is equal to one, half of the total flow resistance is due to viscous shear. If the Forchheimer number is nine, only 10 percent of the total flow resistance is due to viscous resistance, and the other 90 percent to inertial resistance.

An asterisk is used in Equation A-4 to differentiate the Forchheimer number from the one defined by Equation B-5, which contains k_∞ instead of k_g. It is correct as used in Equations B-6 and B-7, but N^*_{Fo} is needed for the following calculations.

If a Δp value could be found for a core plug such that N^*_{Fo} were equal to 0.005 (i.e., the value inside the brackets in Equation A-3 equals 1.005), then Darcy's equation (or equivalently Equation A-3 with a value of 1.000 inside the brackets) would underestimate k_∞ by only 0.5 percent. We will find this Δp by first setting N^*_{Fo} in Equation A-3 to 0.005, then solving for v_m:

$$v_m = \frac{C_1 k_\infty \left(1 + \frac{b}{P_m}\right) \Delta p}{C_2 \mu L (1.005)} \qquad \text{(A-5)}$$

This and the chosen value of N^*_{Fo} are substituted into Equation A-4, which is solved for Δp, keeping in mind that $P_m = P_2 + \frac{1}{2}\Delta p$:

$$\Delta p_{max} = (P_2 + b)\left(\sqrt{1 + \frac{2C_2^2 \, RT\mu^2 L(0.005)(1.005)}{C_1 C_3 M k_\infty k_g \beta (P_2 + b)^2}} - 1\right) \qquad \text{(A-6)}$$

Now the task remains to find appropriate values of β (in view of the large amount of scatter in a β vs. k_∞ plot). A slightly conservative approach was taken: values of β lying on the upper dashed curve in Figure 6-3 were divided by 10. This corresponds to a curve that lies above the central solid curve by about a factor of 3 for low values of k_∞, and gradually falls below the central curve at the highest permeabilities. Thus actual β's for the majority of core plugs would be expected to be somewhat less than the values chosen, and the error incurred from calculating permeability using the Darcy equation (provided that Δp_{max} as calculated from Equation A-6 is not exceeded during the permeability measurement) would be less than 0.5 percent. On the other hand, if a plug's actual β happened to lie on the upper dashed line, use of the same Δp_{max} would result in a calculated permeability that is 5 percent low. If the plug's actual β were twice the value shown by the upper dashed curve, the error would be 10 percent, etc.

The calculation of Δp_{max} is not completely straightforward. Equation A-6 contains b, k_g, and k_∞. For purposes of preparing Figure 6-4, b was found from a correlation proposed by Jones[16] for data on a large number of core plugs using helium:

$$b_{He} = 16.4 k_\infty^{-.0382} \qquad \text{(A-7)}$$

where b_{He} is the Klinkenberg gas slippage factor for helium, psi, and k_∞ is the slip-corrected permeability, millidarcys. To calculate b_{air}, the constant 16.4 in Equation A-7 becomes 5.71, using the technique illustrated by Equation 31. Values for C_1, C_2, C_3, and R for desired units are found from Table 6-1.

The required k_{air} is calculated from:

$$k_{air} = k_\infty \left(1 + \frac{b_{air}}{P_2 + \frac{1}{2}\Delta p_{max}}\right) \qquad \text{(A-8)}$$

Because Δp_{max} is unknown, use k_∞ as the first guess for k_g in Equation A-6, and solve for Δp_{max}. Insert this value into Equation A-8 to solve for k_{air}. Now use this value in Equation A-6 for k_g, and iteratively continue to refine the estimates until there is no further change in Δp_{max}. Convergence is usually achieved in 4 to 10 iterations.

Results of these calculations are shown in Figure 6-4 for downstream pressures of 14.7, 50, 100, and 200 psia for air at room temperature. The $\Delta p / L$ values are reported as "psi per inch of core plug length." Although these ratios are slightly dependent upon length, and were calculated for two-inch long plugs, results are close enough for the intended purpose for plugs ranging in length from 1 to 3 inches.

Starting with the highest permeabilities for the 14.7 psia downstream pressure, the maximum allowable pressure drop increases with decreasing permeability, as expected. However, for permeabilities below about 0.3 millidarcys, $\Delta p_{max} / L$ decreases with decreasing permeability. This unexpected result is a consequence of gas slippage. Slippage is greatest at the lowest permeabilities, causing higher gas flow rates than would occur without slippage. It is decreased when the mean pore pressure is increased at the higher back pressures.

At the higher permeabilities, $\Delta p_{max} / L$ values are reduced with increasing back pressure. This is mainly a consequence of increased gas density, which increases mass flux, and consequently inertial resistance, for a given velocity.

A.6.8.1.2 Liquids

Liquids are nearly incompressible and are not subject to slippage. Therefore the derivation for the maximum pressure drop to maintain the condition of Stokes flow is simpler than that for gases. Again, choosing a value of 0.005 for the Forchheimer number and the same beta vs. permeability relationship as for gases, the equation for liquids is:

$$\frac{\Delta p_{max}\rho_L}{L\mu_L^2} = \frac{5.764\text{E}12}{\beta k_L^2} \qquad \text{(A-9)}$$

Where:

Δp_{max} = maximum pressure drop for Stokes flow, psi.
L = length of core plug, inches.
μ_L = viscosity of liquid, cp.
ρ_L = density of liquid, g/cm^3.
β = coefficient of inertial resistivity, ft^{-1}.
k_L = permeability of plug to liquid, millidarcys.

For a given β and k_L the left hand side of Equation A-9 applies for a core of any length. Results are plotted in Figure A.6-1. Use of this figure is illustrated by an example: Suppose we determined permeability of a 2.50 inch long core plug with a pressure drop of 35.8 psi and a liquid with viscosity of 1.53 cp. and density of 0.816 g/cm^3. A permeability of 99.8 millidarcys was calculated using Darcy's law. Is it likely that Darcy's law was valid to use?

From Figure A.6-1, a permeability of 99.8 yields a value of about 6.36, which is equal to the left hand side of Equation A-9:

$$\frac{\Delta p_{max}\rho_L}{L\mu^2} = 6.36$$

therefore:

$$\Delta p_{max} = \frac{6.36 \times 2.50 \times 1.53^2}{0.816} = 45.6 \text{ psi}$$

Because the Δp actually used for the measurement, 35.8 psi, was less than the maximum allowable value, we conclude that Darcy's equation probably gave valid results.

B.6.8.2 Calculation of Permeability From Pressure-Falloff, Axial Gas Flow Measurements

The equation for axial flow, used with the pressure falloff method proposed by Jones,[21] starts with the steady-state Forchheimer equation for isothermal axial flow, corrected for gas slippage. It is obtained by integration of Equation 17 (see 6.2.1.2), which yields:

$$\frac{(P_1 - P_2)(P_m + f_{Fo}b)z_r}{P_r q_r z_m} = A_1 + A_2\left(\frac{P_r q_r (P_m + b)}{z_r P_m}\right) \qquad \text{(B-1)}$$

Where:

$$A_1 = \frac{C_2 \mu L}{C_1 A k_\infty} \qquad \text{(B-2)}$$

$$A_2 = \frac{C_3 \beta M L}{C_1 A^2 R T} \qquad \text{(B-3)}$$

$$A = \frac{\pi D^2}{4} \qquad \text{(B-4)}$$

$$N_{Fo} = \frac{q_r P_r A_2}{z_r A_1} \qquad \text{(B-5)}$$

$$E = \frac{b N_{Fo}}{1 + N_{Fo}} \qquad \text{(B-6)}$$

and

$$f_{Fo} = \frac{\left(1 - \frac{E}{P_1 - P_2}\ln\left[\frac{P_1 + E}{P_2 + E}\right]\right)\left(\frac{P_1 + P_2 + 2E}{P_1 + P_2}\right) + N_{Fo}}{(1 + N_{Fo})} \qquad \text{(B-7)}$$

The Forchheimer number, N_{Fo} (Equation B-5), which is the dimensionless ratio of inertial to viscous resistivities, is a "Reynolds number for flow through media." It is discussed more fully in Appendix A. The "Forchheimer interaction factor," f_{Fo} (Equation B-7), is a dimensionless number that has a maximum value of 1.0 (whenever b or N_{Fo} goes to zero). Its minimum value for any practical case is about 0.95.

The instantaneous gas flow rate entering the sample is determined from the total connected reservoir and manifold volume upstream of the inlet core face, V_T, and the pressure-time derivative:

$$q_{1,t} = \frac{V_T f_{z,t}}{P_{1,t}}\left(\frac{-dP_{1,t}}{dt}\right) = V_T f_{z,t}\left(\frac{-d\ln[P_{1,t}]}{dt}\right) \qquad \text{(B-8)}$$

Where:

$$f_{z,t} = 1 - \frac{P_{1,t}}{z_{1,t}}\left(\frac{dz_{1,t}}{dP_{1,t}}\right) \qquad \text{(B-9)}$$

is a correction for non-ideal gases. The subscript 1 in these equations refers to conditions just upstream of the inlet face of the sample. Because all flow rates, pressures (except atmospheric pressure), and gas deviation factors refer to this location, this subscript will be dropped in equations below. Subscripts n will refer to time, not position.

In general, it is more useful to measure a gauge pressure than the absolute pressure required by Equation B-8. Because the sample is vented directly to atmospheric pressure, the gauge pressure is equal to the Δp across the sample at any instant. If we multiply and divide Equation B-8 by p and realize that $dp = dP$, we obtain:

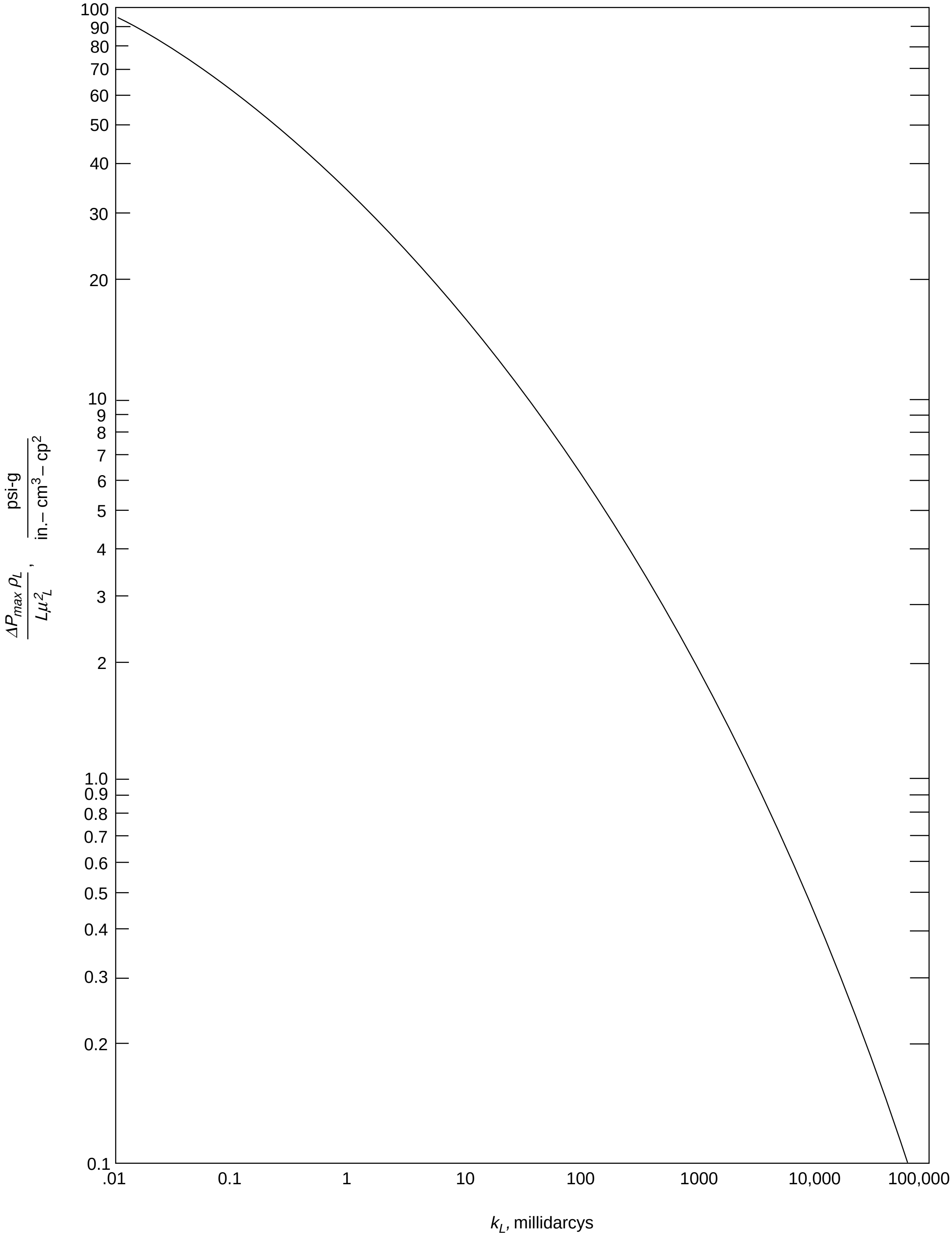

Figure A.6-1—Plot for Determining Maximum Allowable Δp in Axial Flow Liquid Permeability Measurements

$$q = \frac{-pV_T f_z}{pP}\left(\frac{dp}{dt}\right) = \frac{-pV_T f_z}{P}\left(\frac{d\ln p}{dt}\right) \tag{B-10}$$

We can now define an instantaneous flow rate function, y:

$$y \equiv -V_T\left(\frac{d\ln p}{dt}\right) = \frac{qP}{f_z p} \tag{B-11}$$

The derivative in Equation B-11 is accurately approximated from any pair of adjacent pressure-time points:

$$y_n = \frac{V_t \ln\left[\frac{p_{n-1}}{p_n}\right]}{t_n - t_{n-1}} \tag{B-12}$$

This approximation most closely equals the true slope (derivative) of the ln[p] vs. time curve (see Figure B.6-1) at the pressure-midpoint of the interval, which is the geometric mean of the two pressures:

$$p_{g_n} = \sqrt{p_{n-1}p_n} \tag{B-13}$$

Thus y_n is calculated for the instant when the upstream pressure is p_{g_n}. The mean pore pressure in the sample at this time is:

$$P_{m_n} = \tfrac{1}{2}p_{g_n} + P_a \tag{B-14}$$

Now, except for one complication, any of the several sets of y_n, p_{g_n}, P_{m_n}, and f_{z_n} values obtained from the pressure falloff could be used to calculate the flow variables in Equation B-1. With steady-state flow, where the pressure at any point in the sample is invariant with time, the mass flow rate is constant throughout the length of the sample. The complication is that this is not true for unsteady-state flow.

Whenever pressure at any point in the sample decreases with time, gas density there decreases proportionally, with a resultant mass-depletion of gas. Because the upstream pressure decreases continually during a pressure falloff, the pressure throughout the length of the sample also decreases, except at the outlet end, which always remains at atmospheric pressure. The rate of mass depletion of resident gas in the sample is accompanied by an equal increase in the mass rate of flowing gas. Therefore, at any given time during a transient pressure decline, the mass flow rate of gas leaving a sample is

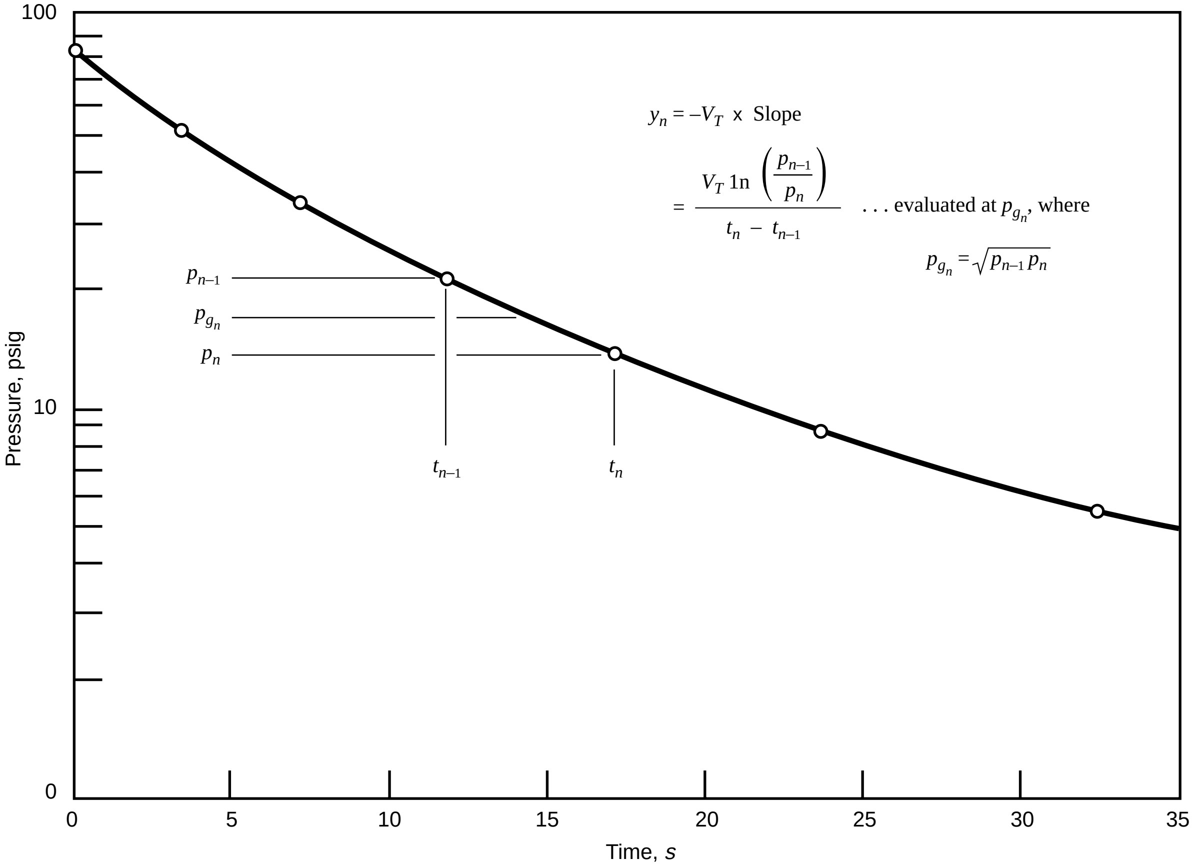

Figure B.6-1—Construction of Flow Variables y_n and p_{gn}

greater than the mass rate entering the sample. If the gas reservoir volume, V_T is large compared to the pore volume of the sample, V_p, then the relative increase is small. If these volumes are comparable, the increase is significant. In the limit, an infinite reservoir volume yields steady-state flow—no increase. At the opposite extreme, when the reservoir volume is zero, all the gas exiting from the sample originates from within the sample itself.

Because of the non-constant mass flux throughout the length of a sample at any instant during the pressure transient, we cannot use the steady-state solution directly. The correct solution can be achieved, however, by an iterative technique in which the continuity equation is decoupled from the flow equation, but where both equations are ultimately satisfied within an acceptable tolerance: the partial derivative of gas density with respect to time at any length, taken from the steady-state solution of the flow equation, is inserted into the continuity equation from which the approximate mass flux variation with length is derived for several conditions of pressure, slip-factor magnitude, and ratios of pore volume to reservoir volume. The corrected, non-constant mass flux is inserted into the differential form of the flow equation, which is then re-integrated with respect to length. The process is repeated until any further change is negligible. Four iterations were adequate for all practical cases. The corrected flow rate that can be inserted into the integrated flow equation to allow for the weighted average increase in mass flow with length at any instant is:

$$q_{c_n} = q_n(1 + \gamma G_m[c_n, \gamma]) \tag{B-15}$$

Where:

$$\gamma = \frac{2V_p}{3V_T} \tag{B-16}$$

and

$$c_n = \frac{(P_a + b)^2}{2p_{g_n}(P_{m_n} + b)} \tag{B-17}$$

The correction factors for non-constant gas mass flow, $G_m[c_n, \gamma]$, are shown as a function of c for several values of γ in Figure B6-2. We can now define a "corrected instantaneous flow rate function," γ_{c_n}:

$$y_{c_n} \equiv \frac{y_n f_{z_n}}{z_n}(1 + \gamma G_m[c_n, \gamma]) = \left(\frac{q_r P_r}{z_r p_g}\right) \tag{B-18}$$

where f_{z_n} (Equation B-9) is evaluated at pressure P_n, where $P_n = p_{g_n} + P_a$. This function can be inserted into Equation B-1 to produce the integrated form of the slip-corrected Forchheimer equation for transient flow:

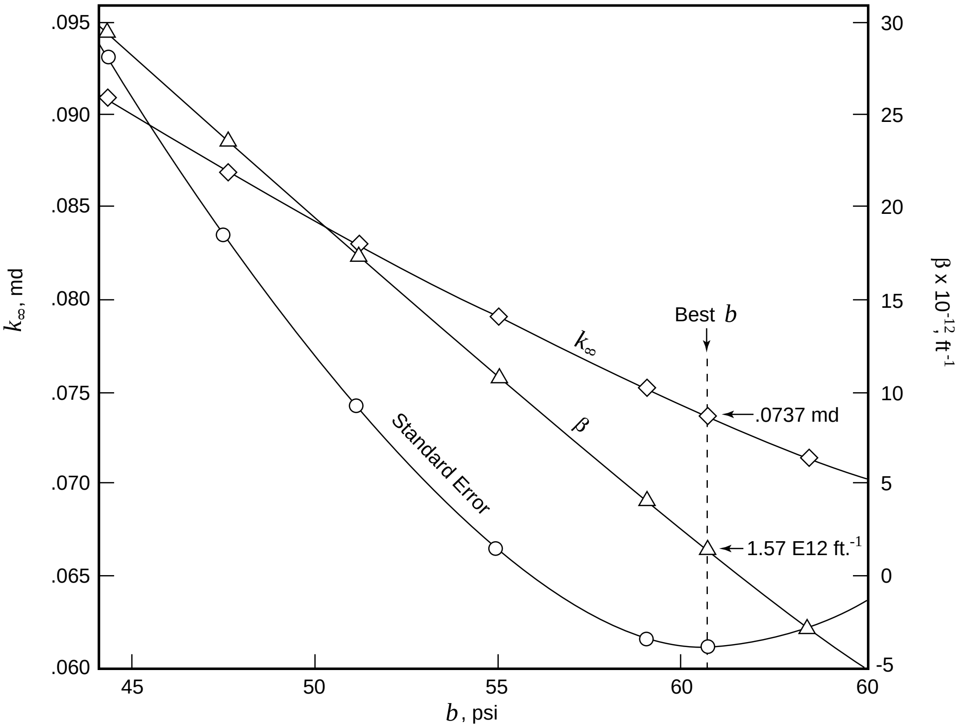

Figure B.6-2—Dimensionless Correction Factors for Non-Constant Mass Flux in Pressure-Falloff Permeability Measurements

$$\left(\frac{P_m + f_{Fo}b}{y_c z_m}\right)_n = A_1 + A_2\left(\frac{y_c p_g (P_m + b)}{P_m}\right)_n \qquad \text{(B-19)}$$

The Forchheimer number (Equation B-5) must be calculated for each f_{Fo_n} in the equation above. In terms of the corrected flow function, this is:

$$N_{Fo_n} = \frac{y_{c_n} p_{g_n} A_2}{A_1} \qquad \text{(B-20)}$$

E_n (Equation B-6) must also be calculated for each data point obtained in order to calculate f_{Fo_n} (Equation B-7).

The coefficients A_1 and A_2 in Equation B-19, which yield k_∞ and β, respectively, are determined from the set of equations corresponding to each data point, n, using linear regression as follows:

1. Calculate an initial guess for the slip factor, *b*. For helium, this is:

$$b[\text{first guess}] = 0.803\left(\frac{D^2}{\mu_{He} L y_{c_m}}\right)^{0.467} \qquad \text{(B-21)}$$

where y_{c_m} is the flow function that corresponds to the lowest p_g value obtained.

2. Set f_{Fo} for all *n*.
3. Calculate c_n (Equation B-17) and y_{c_n} (Equations B-12 and B-18) for each *n*.
4. Calculate the left-hand and right-hand terms in Equation B-19 for each *n*.
5. Perform a linear regression to obtain A_1 and A_2.
6. From these values, calculate N_{Fo} (Equation B-20), *E* (Equation B-6), and f_{Fo} (Equation B-7) for each *n*.
7. Repeat steps 4 through 6 until the change in any f_{Fo} does not exceed 0.001 from its value in the previous iteration. Three iterations are usually adequate.
8. Calculate a Standard Error, SE, from the last linear regression.
9. Choose a new *b* that is 10 percent higher than the first-guessed *b*.
10. Using the most recently calculated set of values for f_{Fo} and the new *b*, repeat steps 3 through 8.
11. If the new SE is lower than the previous one, increase *b* by 10 percent. Otherwise, reduce the original *b* by 10 percent. Repeat step 10.
12. Continue to increase or decrease *b*, repeating step 10, until the minimum SE is found. This corresponds to the best values of *b*, A_1 and A_2, in a least-squares sense. Calculate k_∞ and β from A_1 and A_2 (Equations B-2 and B-3), respectively.

Typical changes in k_∞, β, and SE with *b* are illustrated by Figure B.6-3. (The scale for the SE has been omitted for clarity.) As *b* increases, calculated values of both k_∞ and β decrease. The correct *b* for this example, which occurs at the minimum SE, is 60.6 psi. The calculated value of β becomes

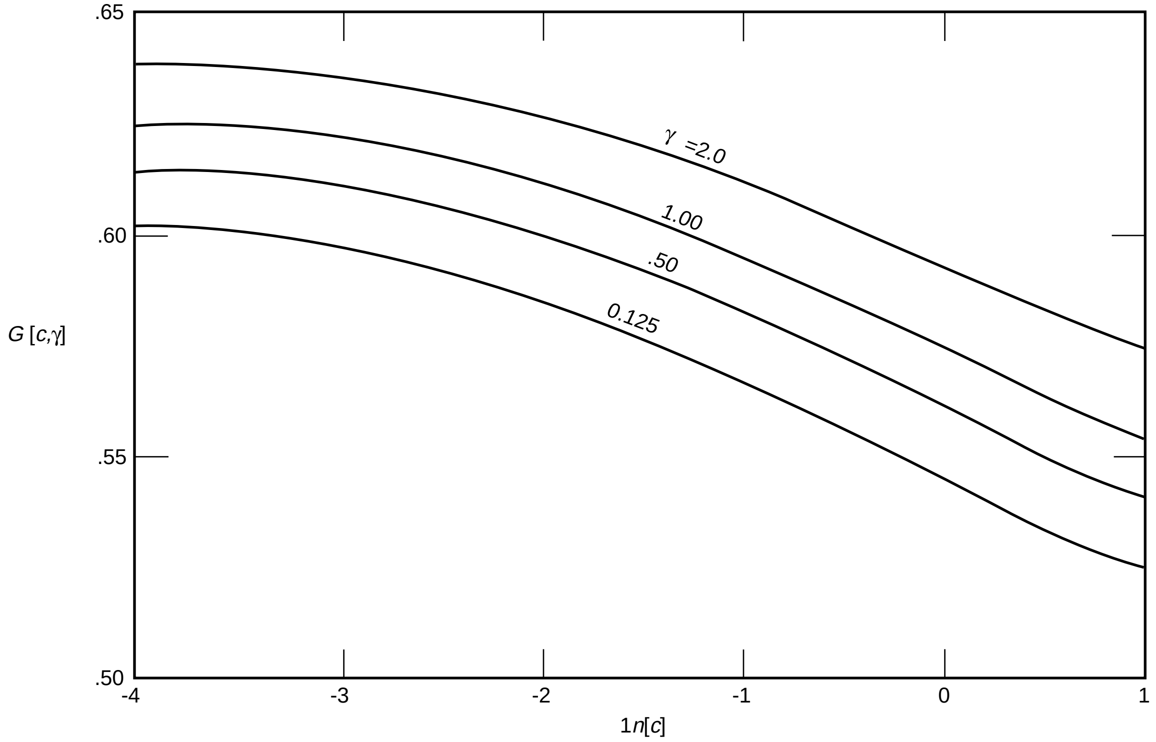

Figure B.6-3—Variation of k_∞, ß, and Standard Error with *b* for Pressure-Falloff Data zero at a slip factor of 61.5 psi, and is negative for larger *b*s. A negative calculated β usually indicates that *b* is too high. However, extreme sensitivity of permeability to small changes in net stress can also cause negative βs. Changes in k_∞ and β with *b*, as shown in Figure B.6-3, are general, in the sense that they always decrease with increases in *b*.

The computational effort for the pressure falloff technique is considerably greater than for steady-state calculations. However, the entire procedure outlined above, with 25 *y* and p_g values each, is accomplished nearly instantaneously with a small computer.

Low-permeability rocks at small pressure differentials often exhibit little inertial resistance. In these cases, the term in Equation B-19 that is multiplied by A_2 is small, f_{Fo} is nearly equal to 1, and Equation B-19 can be approximated by:

$$y_{c_n} z_{m_n} = \frac{1}{A_1}(P_{m_n} + b) \tag{B-22}$$

For the approximation to be valid, a plot of $y_{c_n} z_{m_n}$ vs. P_{m_n} must be linear. Equation B-22 is the Darcy equation, corrected for gas slippage, for pressure-falloff measurements. Its slope and intercept are equal to $1 / A_1$ and b / A_1, respectively. Therefore k_∞ and *b* are calculated from:

$$k_\infty = \frac{C_2 \mu L(\text{slope})}{C_1 A} \tag{B-23}$$

and

$$b = \frac{(\text{intercept})}{(\text{slope})} \tag{B-24}$$

Departure from linearity, especially at the higher P_{m_n} values, indicates that inertial resistance is not negligible at these conditions.

C.6.8.3 Calculation of Permeability From Pressure-Falloff, Probe Gas Flow Measurements

Instantaneous flow rate functions, y_n, and geometric mean pressures, p_{g_n}, are calculated from Equations B-12 and B-13, respectively. These are inserted into:

$$\frac{\tfrac{1}{2}p_{g_n} + P_a + b^*}{y_n} = A_3 + A_4\left(\frac{y_n p_{g_n}(\tfrac{1}{2}p_{g_n} + P_a + b^*)}{\tfrac{1}{2}p_{g_n} + P_a}\right) \tag{C-1}$$

Where:

$$A_3 = \frac{C_2 \mu}{C_1 (G_0 r_i) k_\infty} \tag{C-2}$$

and

$$A_4 = \frac{C_3 \beta M}{C_1 G_{f_{fo}} RT} \tag{C-3}$$

The dimensional geometric factor for the Forchheimer term above, $G_{f_{Fo}}$, must be determined by numerical modeling. If this geometric factor can be determined, then β could be calculated from Equation C-3. Regardless, use of Equation C-1 corrects for inertial resistance, whether or not β can be calculated.

Note that except for the different geometrical factors contained in A_3 and A_4, Equation C-1 is virtually identical to Equation B-19 (for axial flow), except for certain simplifications in the probe-permeameter equations. These simplifications are: (a) gas deviation (*z*) and Forchheimer interaction (f_{Fo}) factors are omitted. Pressures are so low that these factors are nearly unity, and (b) y_n values are not corrected for non-constant (spatial) mass flow. With commonly used probe-seal dimensions, the rock pore volume that influences permeability measurement is so small, relative to the volume of even the smallest gas reservoir, that the steady-state assumption of constant mass flow through any isopotential surface at any instant does not introduce any appreciable error in the calculation of permeability. For example, the error with an interior probe seal radius of 3.8 millimeters, and reservoir volume of 5.2 cm^3 is less than 0.1 percent.

The *b** that appears in Equation C-1 is a gas slippage factor taken from a correlation. Although *b* could theoretically be determined directly from the pressure falloff data, the range of mean pore pressures measured (with a fill pressure of 10 psig or less) is too small to determine *b* reliably. The correlation *b** is obtained using an iterative technique. Its first approximation (for nitrogen, in psi) is calculated from:

$$b^*_{N_2} = 0.22\sqrt{\frac{(G_0 r_i)}{\mu_{N_2} y_m}} \tag{C-4}$$

where y_m corresponds to the lowest value of p_g obtained from the pressure-falloff data. This first-guess value of *b** is used in Equation C-1 to calculate its left and right hand terms for each y_n and p_{g_n} value obtained. Next, the coefficients A_3 and A_4 are found from Equation C-1 using linear regression. The first estimate of the Klinkenberg permeability is obtained from rearrangement of Equation C-2:

$$k_\infty = \frac{C_2 \mu}{C_1 (G_0 r_i) A_3} \tag{C-5}$$

Using this k_∞, an improved estimate of $b^*_{N_2}$ is calculated from the correlation:

$$b^*_{N_2} = 6.9 k_\infty^{-0.382} \tag{C-6}$$

This new value of *b** is reinserted into Equation C-1, and the process is repeated until the change in *b**, from one iteration to the next, is less than 0.1 psi. Convergence should be obtained within 2 to 4 iterations.

Inertial resistance virtually disappears for samples with permeabilities of less than 0.1 millidarcy for starting pressures of less than 10 psig. For these samples, the A_4 term in Equation C-1 can be ignored, and the Forchheimer equation reduces to the slip-corrected Darcy equation:

$$k_\infty = \frac{C_2 \mu y^*}{C_1 (G_0 r_i)(\tfrac{1}{2} p_g^* + P_a + b^*)} \tag{C-7}$$

This equation can be used when as few as one to three $y - p_g$ points are obtained for low permeability samples. The y^* and p_g^* refer to any one of these points. The calculation is again iterative. The initial estimate of b^* is calculated from Equation C-4. The estimate is refined by calculating k_∞ (Equation C-7), which is then used in Equation C-6. The process is continued until the change in b^*, from one iteration to the next, is less than 0.1 psi. Convergence is usually achieved in 2 to 4 iterations.

Gas permeability, k_g, for any desired gas at any mean pore pressure can be calculated from k_∞ and b^* as outlined in 6.4.1.1.5 for the axial flow case.

D.6.8.4 Calculation of Permeability From Pulse Decay, Axial Gas Flow Measurements

The initial pressure difference across the sample, $\Delta p[0]$, which is slightly less than Δp_1 (see 6.4.1.3.2), must be calculated from a gas mass balance:

$$\Delta p[0] = \Delta p_1 \left(\frac{V_1 - V_d}{V_1} \right) \tag{D-1}$$

V_1, the total upstream volume, includes: internal volumes of the upstream reservoir; the upstream chamber of the differential pressure transducer; connecting lines to the transducer and valves 1, 2, and the fill valve; and the upstream dead volume, V_d, (the volume inside valve 1); the upstream end plug, and the line connecting them. The total downstream volume, V_2, includes the volumes in the downstream reservoir, transducer, and end plug; the downstream chamber of the differential transducer; and the lines connecting them, including the line to valve 2. The volumes V_1, V_2, and V_d must all be found from careful calibration using Boyle's Law techniques (see 6.6.3.1).

The differential pressure and downstream pressure are monitored as functions of time. Data are analyzed from the solution of the diffusivity equation, that is derived by combining the differential form of Darcy's Law with the continuity equation. The sample is presumed to be initially at a uniform pore pressure throughout. Then at time $t = 0$, a pulse of slightly higher pressure, $\Delta p[0]$, is applied to its upstream end from the upstream reservoir. As gas flows from V_1 into the sample, the pressure in V_1 declines. The pressure in V_2 remains constant for a short period of time until the pressure pulse has traversed the length of the sample. Then the pressure in V_2 rises. Because $P_1[t]$ declines and $P_2[t]$ rises, $\Delta p[t]$ continues to diminish and gradually approaches zero as the upstream and downstream pressures become equal. The rate of pressure decay depends on permeability: the lower the permeability the slower the decay. The general solution for the pressure difference as a function of time as presented by Dicker and Smits,[23] following the original work of Brace et al.,[24] the error-function solution of Bourbie and Walls,[25] the general analytical solution of Hsieh et al.,[26] Chen and Stagg,[27] Haskett et al.,[28] and others is:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = 2 \sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b \sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2 (\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m^2 t_D] \tag{D-2}$$

Where θ_m are roots of the equation:

$$\tan\theta = \frac{(a+b)\theta}{\theta^2 - ab} \tag{D-3}$$

a, the ratio of the compressive storage (the product of volume and compressibility) of the sample's pore volume to that of the upstream reservoir, is:

$$a = \frac{V_P (c_g + c_{pv})}{V_1 (c_g + c_{v_1})} \tag{D-4}$$

b, the ratio of the compressive storage of the sample's pore volume to that of the downstream reservoir, is:

$$b = \frac{V_P (c_g + c_{pv})}{V_2 (c_g + c_{v_2})} \tag{D-5}$$

and t_D, dimensionless time, is:

$$t_D = \frac{C_1 k_g t}{C_2 \mu \phi (c_g + c_{pv}) L^2} \tag{D-6}$$

where C_1 and C_2 are conversion constants, given in Table 6-1, required to make t_D dimensionless. The c_g, c_{pv}, c_{v_1}, and c_{v_2} are compressibilities of gas, the sample's pore volume, and upstream and downstream reservoirs, respectively. Gas compressibility is:

$$c_g = \frac{1}{P}\left(1 - \frac{d\ln[z]}{d\ln[P]}\right) \equiv \frac{f_z}{P} \tag{D-7}$$

In Equation D-7, we have defined f_z as the value of the group within parentheses that accounts for deviation from ideal gas behavior. At pressures of interest, this deviation is not negligible. Values of f_z for nitrogen at 72°F are given by Jones.[44] If the system is built rigidly, i.e., with thick-walled vessels, and if displacement volumes of the transducers are small relative to V_1 and V_2, then c_{V_1} and c_{V_2} are small compared to c_g, and can be ignored. Pore-volume compressibility varies considerably among samples of different lithologies. However, it is minimized by using high net confining stresses, and is usually less than 2 x 10^{-5} psi^{-1} for net stresses of 5,000 psi or more. A c_{pv} of 1 x 10^{-5} psi^{-1} is equal to 1 percent of the gas compressibility at a gas pressure of 1000 psia, or 2 percent at 2,000 psia.

The squared pressure differences (that apply to gases only) that appear on the left hand side of Equation D-2 are:

$$\frac{\Delta(P[t])^2}{\Delta(P[0])^2} \equiv \frac{P_1[t]^2 - P_2[t]^2}{P_1[0]^2 - P_2[0]^2} = \frac{\Delta p[t](P_2[t] + \frac{1}{2}\Delta p[t])}{\Delta p[0](P_2[0] + \frac{1}{2}\Delta p[0])} \tag{D-8}$$

Equation D-2 applies for all values of a, b, and t_D, provided that all roots of Equation D-3 are used, and all compressibilities are constant. Haskett et al.[28] minimize the effect of the restriction by incorporating time-varying compressibility into an "adjusted pseudo time." However, with the proper experimental conditions, neither of the restrictions poses a serious problem, as will be discussed below. The use of the squared pressure differences in Equation D-2 is nearly equivalent to using pseudo-pressures as suggested by Haskett et al. Because the change in the mean pore pressure is so small in the technique outlined here, changes in the z-factor and gas viscosity are small. Their ratios between the numerator and denominator of Equation D-9 are virtually unity. For experimental conditions other than outlined below, where variations in the mean pore pressure are greater, the use of pseudo-pressures is recommended.

The choice of reservoir sizes is not limited directly by Equation D-2. Some investigators have used large upstream vessels ($a \cong 0$) or large downstream vessels ($b \cong 0$). Others have advocated reservoir volumes that are similar in magnitude to the pore volume of the sample (a or $b \cong 0.2$ to 5). In general, to keep measurement times reasonably short with low-permeability samples, at least one of the reservoirs must be small.

Dicker and Smits[23] present compelling reasons to make the upstream and downstream volumes identical and fairly small. The equal volumes create a desirable situation of symmetry. Following the period when the pressure pulse traverses the length of the sample, the pressure decrease in the upstream vessel is offset by an equal downstream pressure increase, thereby keeping the mean pore pressure constant and reducing the overall pore volume compressional changes nearly to zero. Because the average pressure in the upstream half (of a homogeneous sample) decreases with time, while the average pressure in the downstream half increases by the same amount, the mass flow rate of gas entering the sample is equal to that leaving the sample at the same instant. The maximum rate at any instant occurs about midway along the length of a reasonably homogeneous sample.

Another consequence of symmetry (where $a = b$) is that all even roots of Equation D-3 cancel out in Equation D-2. The first root of Equation D-3, θ_1, is slightly less than the square root of $(a + b)$ for small values of a and b (large reservoirs). If we define f_1 as:

$$f_1 \equiv \frac{\theta_1^{\,2}}{a + b} \tag{D-9}$$

then $f_1 = 1.000$ when $a + b = 0$, and gradually decreases as $a + b$ becomes larger, as illustrated in Figure D6-1. In this figure, V_s is the smaller of V_1 or V_2, and V_L is the larger of the two reservoir volumes.

The square of the first root of Equation D-3 is found from:

$$\theta_1^{\,2} = f_1(a + b) \tag{D-10}$$

The higher roots of Equation D-3 are equal to:

$$\theta_m = (m - 1)\pi, \text{ for } (a + b) = 0 \tag{D-11}$$

and increase gradually as $(a + b)$ increases.

Except for early times when t_D is small, only the first term in the summation (Equation D-2) is significant. The second term is zero when a equals b. At a t_D of 0.1, all higher terms add only 0.16 percent to the contribution of the first term when $a = b = 1$, and less for smaller values of a and b. Therefore, if early times are not used, Equation D-2 reduces to a single exponential, which can be written as:

$$\ln\frac{\Delta(P[t])^2}{\Delta(P[0])^2} = \ln f_0 - \left(\frac{\theta_1^{\,2} k_g}{(c_g - c_{pv})\mu\phi L^2}\right)t \tag{D-12}$$

Where:

$$f_0 = \frac{2[a(b^2 + \theta_1^{\,2}) + b\sqrt{(a^2 + \theta_1^{\,2})(b^2 + \theta_1^{\,2})}]}{\theta_1^{\,2}(\theta_1^{\,2} + a + a^2 + b + b^2) + ab(a + b + ab)} \tag{D-13}$$

Thus, if experimentally obtained values of $\ln[\Delta(P[t])^2 / \Delta(P[0])^2]$ were plotted vs. time, a straight line would result (except for early times), which has an intercept of $\ln[f_0]$, and a negative slope of absolute value:

$$|slope| = \frac{C_1\theta_1^{\,2}k_g}{C_2(c_g + c_{pv})\mu\phi L^2} = \frac{C_1 f_1(a + b)k_g}{C_2(c_g + c_{pv})\mu\phi L^2} \tag{D-14}$$

where C_1 and C_2 are constants from Table 6-1, used to make the units consistent. If c_{v_1} and c_{v_2} are negligible compared to c_g, then:

$$|slope| = \frac{C_1 f_1 A k_g P_m}{C_2 \mu L f_z}\left(\frac{1}{V_1} + \frac{1}{V_2}\right) \tag{D-15}$$

The mean pore pressure in Equation D-15, P_m, is an absolute pressure. When $a = b$, and the rock sample is homogeneous (or if a and b are small, e.g., less than 0.3), P_m should not change during the linear portion of the semi-log decay. It is calculated from:

$$P_m[t] = P_2[t] + \tfrac{1}{2}\Delta p[t] \qquad \text{(D-16)}$$

Calculation of the slope can be performed during the measurement, using the latest two time-pressure readings:

$$|slope| = \frac{\ln\left(\dfrac{\Delta p[t_{n-1}](P_2[t_{n-1}] + \frac{1}{2}\Delta p[t_{n-1}])}{\Delta p[t_n](P_2[t_n] + \frac{1}{2}\Delta p[t_n])}\right)}{(t_n - t_{n-1})} \qquad \text{(D-17)}$$

The absolute value of this slope will be high initially (if $a \cong b$), and will decrease fairly quickly, becoming constant with increasing time. After the slope has become constant for several readings, the intercept and slope of the linear portion of the decay can be obtained by linear regression of:

$$\ln\left(\frac{\Delta p[t_n]P_m[t_n]}{\Delta p[0]P_m[0]}\right) = A_0 + A_1 t_n \qquad \text{(D-18)}$$

where A_0 is the intercept and A_1 is the slope. Only data points yielding the constant slope (from Equation D-17) are to be used in the regression. From Equation D-12, we see that f_o can be obtained from the intercept:

$$f_0 = \exp[A_0] \qquad \text{(D-19)}$$

Equation D-13 shows that f_0 is a function of a and b, as is θ_1. Remarkably, when $a = b$, f_1 is only slightly greater than f_0: 0.0055 percent for $a = 0.1$; 0.12 percent for $a = 0.5$; and 0.44 percent for $a = 1.0$. Values of θ_1^2, f_1, and f_0 are listed in Table D.6-1 for various values of a, when $a = b$. These can be calculated from Equations D-3, D-10, and D-13 when the reservoirs are not of equal volume (more precisely, of equal compressive storage).

Figure D.6-2 shows f_1 as a function of f_0 for different ratios of V_2 / V_1. Because V_1 and V_2 are both determined by calibration, f_1 can be determined uniquely from f_0, and permeability calculated from:

$$k_g = \frac{-A_2 C_2 \mu L f_z}{C_1 f_1 A P_m\left(\dfrac{1}{V_1} + \dfrac{1}{V_2}\right)} \qquad \text{(D-20)}$$

Using the value of f_0 obtained, the pore volume of the sample can be estimated from:

$$V_p = \frac{aV_1}{1 + P_m c_{pv} / f_z} \qquad \text{(D-21)}$$

where a is found from Table 6-10, or from Figure D.6-1. If c_{pv} is estimated to equal 8 x 10^{-6} psi^{-1}, which may be too high or too low by a factor of 2 to 4 compared to the true value at a high net effective stress (4,000 to 5,000 psi), the maximum error from the assumption is about 1.6 percent for $P_m = 1000$ psia, or 3.2 percent for $P_m = 2{,}000$ psia. At lower net stresses, c_{pv} can be expected to be higher and more variable.

The differential pressure transducer should be electrically or mathematically zeroed just before valve 2 is closed (when the true differential pressure is zero at $P_2[0]$). If the transducer maintains its linearity, even though its gain or sensitivity may shift slightly in going from a low to a higher reference pressure (i.e., when P_2 is raised from atmospheric pressure to 1,000-2,000 psia), no error is incurred (except in the calculation of P_m) because all the Δp values are relative; they occur as ratios.

Table D.6-1—Pulse Decay Constants When $a = b$ ($V_1 = V_2$, or $S_1 = S_2$)

$a = b =$	θ_1^2	$f_1 = \theta_1^2 / (\dot{a} + b)$	f_0 = exp[intercept]	f_1 / f_0
0.0	.000000	1.000000	1.000000	1.000000
0.1	.196711	.983553	.983500	1.000055
0.2	.387016	.967539	.967334	1.000212
0.3	.571166	.951944	.951500	1.000466
0.4	.749404	.936755	.935999	1.000808
0.5	.921963	.921963	.920826	1.001235
0.6	1.089064	.907553	.905980	1.001737
0.7	1.250923	.893516	.891458	1.002309
0.8	1.407744	.879840	.877255	1.002947
0.9	1.559725	.866514	.863366	1.003646
1.0	1.707053	.853527	.849789	1.004399
1.1	1.849910	.840868	.836516	1.005202
1.2	1.988469	.828529	.823544	1.006053
1.3	2.122896	.816499	.810866	1.006947
1.4	2.253350	.804768	.798477	1.007878

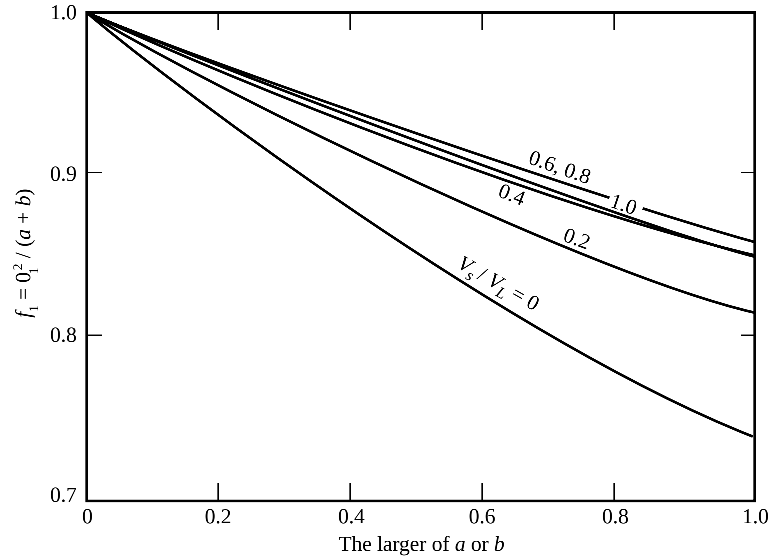

Figure D.6-1—First Root Relationship for Pulse-Decay Equation

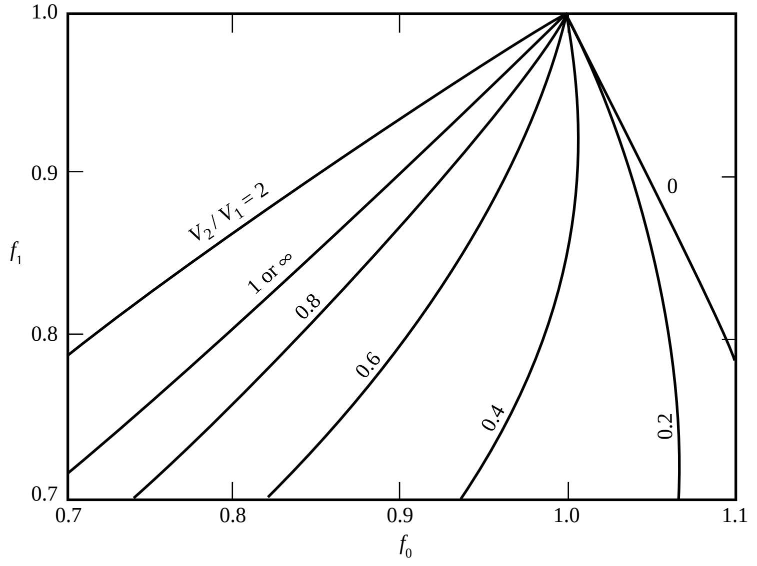

Figure D.6-2—Pulse-Decay Plot for Finding f_1, from f_0 with Known Ratios of V_2 / V_1 (for Gases) or S_2 / S_1 (for Liquids)

The practical range of permeability for gas pulse-decay measurements is about 0.1 millidarcy to 0.01 microdarcy (1 E-4 to 1 E-8 μm^2). The pulse-decay time is nearly proportional to sample length and inversely proportional to permeability and mean pore pressure. The time also depends strongly on the how much of the total pulse decay is measured.

As a general guideline, the reservoir volumes, V_1 and V_2, should be at least as large as the largest pore volume anticipated, and thereby 2-10 times the pore volume of the majority of samples to be run. The onset of the linear semi-log portion of the pulse decay occurs at about the same elapsed time regardless of the *PV*-reservoir volume ratio. The work of Kamath et al.[29] shows that the sensitivity to permeability heterogeneity is also reduced as *a* and *b* become smaller (larger reservoirs).

The upper permeability limit can be extended by increasing the reservoir volumes. However the sensitivity (thus the accuracy) of the pore volume determination (Equation D-21) is reduced with larger volumes.

Perhaps the most time-consuming part of this technique is allowing the system to reach true pressure equilibrium prior to generation of the pressure pulse. As pressures become uniform everywhere in the system, driving forces approach zero. If the pore volume of a sample is determined independently, the pressure equilibration step can be eliminated as suggested by Jones.[44]

E.6.8.5 Calculation of Permeability From Pulse Decay, Axial Liquid Flow Measurements

E.6.8.5.1 Apparatus and Measurement of Compressive Storage

One convenient method of measuring compressive storages in apparatus for liquid pulse decay measurements is to include a needle valve with a micrometer screw (as illustrated in Figure 6-25), which has a total calibrated displacement volume (cross-sectional area of the plunger at the stem seal multiplied by the length of its available stroke) of about 0.02 to 0.05 cm^3. Valves 1 and 2 should be of the type that have no internal volume change when opened or closed. To measure the storages, load a solid steel cylinder into the sample holder and apply confining stress. With valves 1 and 2 open, apply a vacuum to the fill/vacuum valve to remove all air from the apparatus. Then fill the entire system with degassed liquid to about 80 percent of the full scale pressure of the downstream pressure transducer. After the pressure has stabilized, close valves 1 and 2. The needle valve, valve 3, should be near fully-open. The differential pressure transducer should read zero, and the p_2 reading should not change when valves 1 and 2 are closed. Record the micrometer reading. Then screw in the micrometer, decreasing the upstream system volume until the differential pressure transducer reads full scale, *e.g.*, 100 psi. This pressure rise from the initial zero reading is Δp_1, and the volume decrease, calculated from the micrometer readings, is ΔV_1.

Because the volume between valve 1 and the upstream end of the steel plug in the sample holder (the "upstream dead volume") was excluded from the compression measurement, the "upstream compressive storage," S_1, less the "dead volume compressive storage," S_d, is calculated from:

$$S_1 - S_d = \frac{\Delta V_1}{\Delta p_1} \tag{E-1}$$

To determine S_d, open valve 1. After the differential pressure reading stabilizes to Δp_2, calculate the storage for the upstream dead volume from:

$$S_d = \Delta V_1\left(\frac{1}{\Delta p_2} - \frac{1}{\Delta p_1}\right) \tag{E-2}$$

and the upstream compressive storage from:

$$S_1 = \frac{\Delta V_1}{\Delta p_2} \tag{E-3}$$

To determine the downstream storage, first unscrew the needle valve just past—then back to—the initial reading. As a check, the differential transducer should again read zero, and the downstream transducer should display its initial reading. Open valve 2. Read the downstream transducer pressure, $p_2[1]$. Screw in the needle valve until the stabilized downstream pressure, $p_2[2]$, is nearly equal to the full-scale reading of this transducer. Calculate the volume decrease, ΔV_2, from the micrometer readings, and the downstream compressive storage from:

$$S_2 = \frac{\Delta V_2}{p_2[2] - p_2[1]} - \frac{\Delta V_1}{\Delta p_1} - S_d \tag{E-4}$$

These calibrations of compressive storage must be performed for each different liquid used.

E.6.8.5.2 Procedure for Permeability Measurement

For a pulse decay liquid permeability measurement, a completely liquid-saturated rock sample is loaded into the sample holder, which is then pressurized, preferably to a fairly high (usually hydrostatic) confining stress. The system is filled with liquid, as described above, and pressurized to the initial pore pressure. The fill valve is left open long enough to allow the high pressure liquid to diffuse into the sample. With valves 1 and 2 still open, the fill valve is closed, and the downstream transducer is monitored until no further change in pressure is observed. Then valves 1 and 2 are closed and the pressure pulse is generated by screwing in the needle valve. After all pressures have stabilized, $S_1 - S_d$ is calculated from Equation E-1. This should have the same value as that obtained from the calibration.

The pulse decay is initiated by opening valve 1. The initial pressure difference across the sample, $\Delta p[0]$, will be slightly less than $\Delta p[1]$, which was generated with the needle valve with valve 1 closed, thereby excluding the upstream dead volume from the upstream volume. Therefore, $\Delta p[0]$ must be calculated, using the $\Delta p[1]$ and ΔV_1 that correspond to the run-time pressure-pulse generation, and the S_d that was determined from the calibration:

$$\Delta p[0] = \frac{1}{\dfrac{S_d}{\Delta V_1} - \dfrac{1}{\Delta p_1}} \qquad \text{(E-5)}$$

E.6.8.5.3 Pulse Decay Calculations

Equations D-2 and D-3 apply to liquid, as well as gas, pulse decay measurements. However, the squared pressure differences on the left hand side of Equation D-2 must be replaced by first-power pressure differences for liquids:

$$\frac{\Delta p[t]}{\Delta p[0]} = 2\sum_{m=1}^{\infty} \frac{a(b^2 + \theta_m^2) - (-1)^m b\sqrt{(a^2 + \theta_m^2)(b^2 + \theta_m^2)}}{\theta_m^2(\theta_m^2 + a + a^2 + b + b^2) + ab(a + b + ab)} \cdot \exp[-\theta_m{}^2 t_d] \qquad \text{(E-6)}$$

The a and b now refer to the ratios of liquid compressive storages:

$$a = \frac{V_p(c_L + c_{pv})}{V_1(c_L + c_{v_1})} \equiv \frac{S_{pv}}{S_1} \qquad \text{(E-7)}$$

and

$$b = \frac{V_p(c_L + c_{pv})}{V_2(c_L + c_{v_2})} \equiv \frac{S_{pv}}{S_2} \qquad \text{(E-8)}$$

and k_g and c_g in Equation D-6 are replaced by k_L and c_L, respectively.

After the early-time portion of the pressure decay, a plot of the natural logarithm of $\Delta p[t]/\Delta p[0]$ vs. time results in a straight line:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = \ln[f_0] - \left[\frac{C_1 f_1 kA}{C_2 \mu L}\left(\frac{1}{S_1} + \frac{1}{S_2}\right)\right] t_n \qquad \text{(E-9)}$$

or, equivalently:

$$\ln\left[\frac{\Delta p[t_n]}{\Delta p[0]}\right] = A_0 + A_1 t_n \qquad \text{(E-10)}$$

A_0 and A_1, which are the intercept and slope of the straight line, are found from Equation E-10 using linear regression on the linear portion only of the pressure decay data. The value of f_0, defined by Equation D-14, is calculated from the intercept:

$$f_0 = \exp[A_0] \qquad \text{(E-11)}$$

The parameter f_1, which is needed for the calculation of permeability, is found from f_0 and the ratio of the calibrated downstream and upstream storages, S_2 / S_1, with the aid of Figure 6.D-2. Permeability is calculated from:

$$k_L = \frac{-A_1 C_2 \mu L}{C_1 f_1 A\left(\dfrac{1}{S_1} + \dfrac{1}{S_2}\right)} \qquad \text{(E-12)}$$

Amaefule, et al.[30] used pore pressures ranging from 500 to 4,500 psi. Kamath, et al.[29] recommend pore pressures of nearly 1000 psi and pulse sizes of about 20 percent of the pore pressure, or more preferably, about 10 percent of the net confining stress. Volumes of the upstream and downstream reservoirs should be equal if possible, for reasons given in the gas pulse-decay section. They should be slightly smaller or about the same volume as the pore volume of the rock to be measured if heterogeneity is to be investigated, or 5 to 10 times larger, or more, if average permeabilities, comparable to steady-state values, are desired. See Kamath, et al.,[29] regarding “early time solutions” for investigation of heterogeneity, which is beyond the intended scope of this document.

SECTION 7—SUPPLEMENTARY TESTS

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7 Supplementary Tests

7.1 GENERAL

Acid solubility or calcimetry, oil gravity, core water salinity, grain size distribution, basic oil characterization, and petrographic characterization are supplementary data frequently obtained in conjunction with core analyses. Accordingly, these tests have been included because the data are useful in understanding and interpreting the core analysis results.

7.2 PETROGRAPHIC CHARACTERIZATION

Petrographic characterization is an important part of core analysis. Together, petrographic information and basic core analysis measurements provide an integrated framework for reservoir evaluations, including log interpretation. With geologic data, the results of core analyses can be extended to parts of a reservoir where there are no core measurements. Petrographic analysis also provides a basis for interpreting core analysis results, especially those that are unexpected or anomalous. Unexpected results may be valid and caused by variations in rock texture and/or mineralogy.

7.2.1 Petrographic characterization is best performed on the core plug used for routine core analysis so that the data are directly applicable. In those cases in which the plug cannot be consumed, the end sections obtained in preparing the core test samples or the core material from the same bedding plane as the test samples can be used. A minimum of one cubic inch (approximately 40 grams) of core should be saved for petrographic characterization.

7.2.2 Description of core analysis samples provides vital information on sample quality and character. Descriptions should be performed on extracted samples with the aid of a good binocular microscope. Visual porosity and pore type (e.g. intergranular, vuggy, moldic), drilling—or coring—induced features (e.g., drilling fluid invasion or induced fractures), and natural heterogeneity (clay laminae, lithologic variation normal to bedding, mineralized fractures, etc.) should be recorded. Petrographic characterization should include textural information and major mineralogical components. Textural data should include information on grain size, sorting, rounding, and grain shape. Mineralogical observations should include information on grain types and abundance. Approximate amounts of cement and matrix components can also be estimated with a binocular microscope in many cases.

7.2.3 Quantitative or semi-quantitative petrographic information can be obtained from thin sections, x-ray diffraction (XRD), bulk rock chemical analysis (usually by x-ray fluorescence, XRF), and Fourier transform infrared spectroscopy (FTIR). Valuable information regarding the spatial distribution of clay minerals within a sample can be obtained from scanning electron microscopy (SEM).

7.3 GRAIN SIZE DISTRIBUTION

Grain size distribution data have: (a) engineering application in well completion programs in friable and unconsolidated sediments, (b) geological application in assessing sand heterogeneity and depositional environment interpretation in both consolidated and unconsolidated clastic sediments, and (c) petrophysical application in various phases of formation evaluation to effect an understanding of log responses. Grain size distribution data are commonly utilized in sidewall core analysis to derive permeability. Methods commonly employed include wet and dry sieving, settling tube particle size analysis, thin section grain size analysis, and laser diffraction particle size analysis.

7.3.1 Sieve Analysis (Mechanical Shaker)

7.3.1.1 Principle

A known mass of extracted, disaggregated sample should be mechanically vibrated through stacked screens with progressively smaller openings. The partial sample weights retained on individual screens are used to produce a sieve analysis report of screen opening size versus percent sample retained.

7.3.1.2 Apparatus

The following equipment is recommended:

a. Mechanical sieving device.
b. U.S. or Tyler sieves (the sieves must conform to ASTM Specification E-11, *Specification for Wire Cloth Sieves for Testing Purposes*).[1]
c. Balance (0.01 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Drying oven.

7.3.1.3 Procedure

The screens should be cleaned, dried, weighed, and nested from the largest to the smallest screen size. A pan is placed at the bottom of the screen assembly. Table 7-1 illustrates the commonly available screens and the size equivalents.

A representative core sample is extracted to remove hydrocarbons and salt. The sample is then dried to weight equilibrium to remove solvent and water. The sample is gently disaggregated, weighed to the nearest 0.01 gram (sample size typically 100 grams if eight-inch diameter screens are used),

Table 7-1—Commonly Available Screen Sizes

	U.S. Standard Sieve Mesh	Size in Millimeters
Gravel	5	4.00
Gravel	6	3.36
Gravel	7	2.83
Gravel	8	2.38
Gravel	10	2.00
Very coarse sand	12	1.68
Very coarse sand	14	1.41
Very coarse sand	16	1.19
Very coarse sand	18	1.00
Coarse sand	20	0.84
Coarse sand	25	0.71
Coarse sand	30	0.59
Coarse sand	35	0.50
Medium sand	40	0.42
Medium sand	45	0.35
Medium sand	50	0.30
Medium sand	60	0.25
Fine sand	70	0.21
Fine sand	80	0.18
Fine sand	100	0.15
Fine sand	120	0.13
Very fine sand	140	0.11
Very fine sand	170	0.09
Very fine sand	200	0.07
Very fine sand	230	0.06
Silt	270	0.05
Silt	325	0.04
Silt	430	0.03

and placed into the top screen. The nest of screens is vibrated with a mechanical sieve shaker or manually. The grains travel downward through the nest of screens until retained on a screen having a mesh size smaller than the minimum grain dimensions. Each loaded screen is weighed. The empty screen weights are deducted from the loaded screen weights to obtain the weight of the material retained on individual screens. The data are generally reported as weight percents retained on individual screens and cumulative weight percents to and including each screen. The data are typically reported in both tabular and graphical form (see Table 7-2 and Figure 7-1).

7.3.1.4 Precautions

Precautions of this method include:

a. The sample must be thoroughly extracted to remove residual hydrocarbons. In some cases, salt must be extracted. Care must be taken throughout the extraction process to avoid fines loss.

b. Sample disaggregation must be sufficient to reduce the sample to individual grains without crushing grains or otherwise artificially creating fines.

Table 7-2—Example of Sieve Analysis Data

U.S. Standard Sieve Mesh	Size in Microns	Percent Retained	Cumulative Percent
10	>2000	3.72	3.72
18	>1000	12.69	16.41
35	>500	34.74	51.15
60	>250	21.74	72.89
120	>125	7.30	80.19
170	>88	2.36	82.55
230	>63	3.08	85.63
325	>44	1.55	87.18
430	>30	2.90	90.08
pan	<30	9.92	100.00

c. The screens must be microscopically inspected to ensure that they are not damaged. Puncture-type holes and tears at the screen to retainer contact are particularly troublesome in the finer mesh screens.

d. Sufficient time must be allowed in vibrating the screens to ensure that each size fraction has completely settled. The test duration will vary relative to the quantity and type of sample introduced for a given type of mechanical shaker.

e. The balance must be properly calibrated.

f. In shaly sands, static electricity can prevent charged <.044-millimeter fines from settling to the pan (see 7.3.3 for wet sieve alternates).

7.3.1.5 Calculations

Calculations for this method are as follows:

$$\%\text{Retained Each Screen} = \frac{(\text{loaded screen weight} - \text{empty screen weight})}{\text{total sample weight}} \times 100 \qquad (1)$$

$$\%\text{Retained cumulative} = \text{sum of percent retained on each screen (to and including each mesh size from largest screen to pan)} \qquad (2)$$

7.3.1.6 Advantages

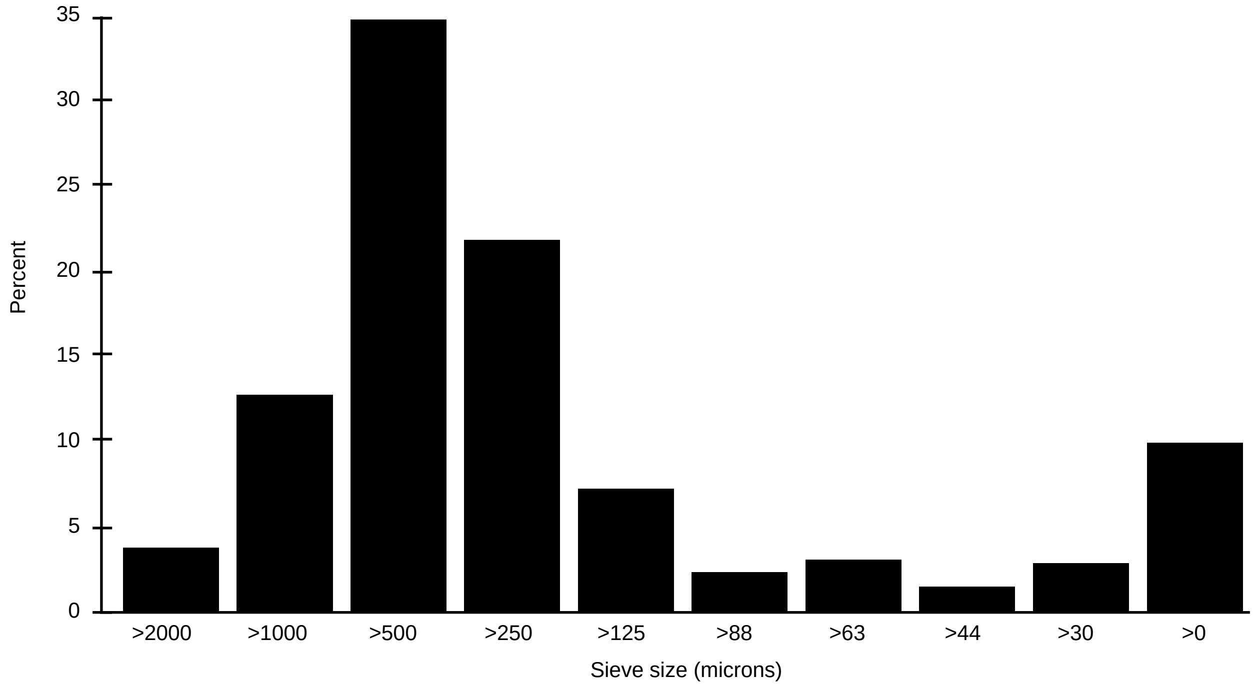
Advantages of this method include:

a. This method is acceptable for classifying grain sizes over the >.044 millimeter size range for minimally cemented sands. The method is a standard method and is accurate.

b. The sample can be recombined for reruns to ensure repeatability.

c. The sample can be recombined or maintained in separate size fractions for additional testing.

d. The test is simple and can be conducted with a minimum of equipment.



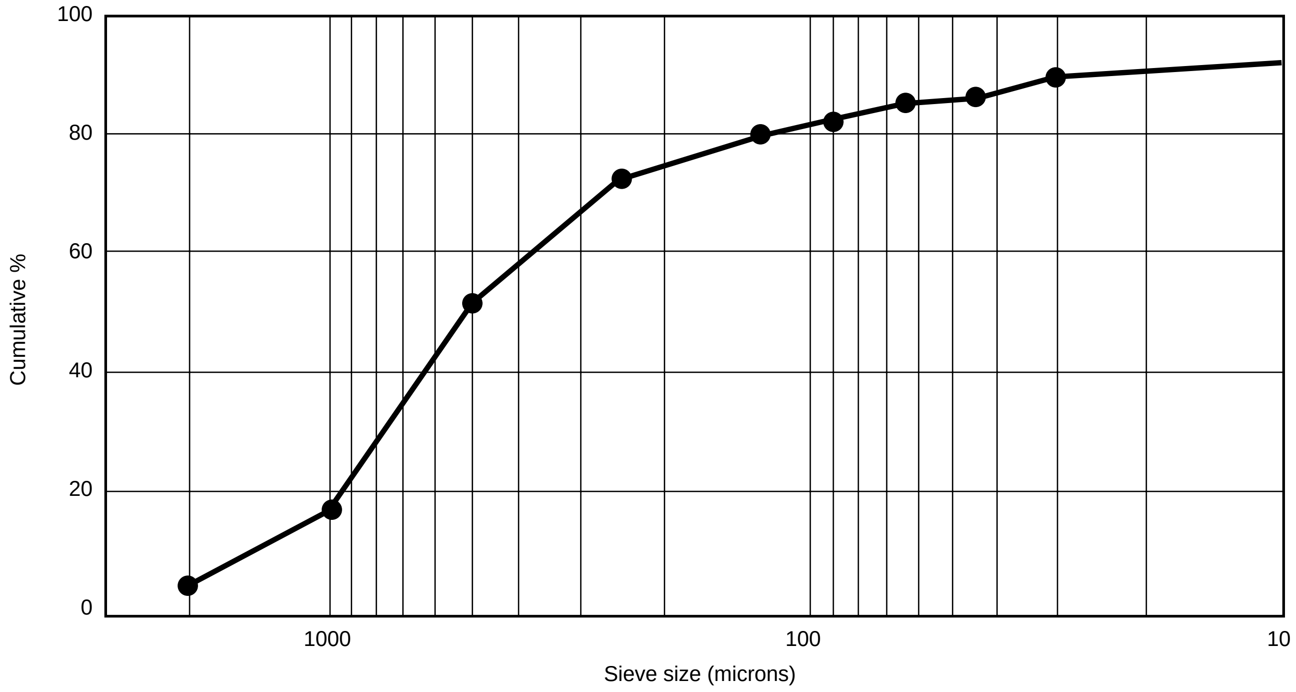


Figure 7-1—Examples of Graphical Forms for Reporting Sieve Analysis Data

7.3.1.7 Limitations

The method is relatively time consuming (one hour per sample for eight-inch diameter screens), requires a relatively large sample, and can yield inaccurate data in shaly sands and consolidated sands.

7.3.1.8 Accuracy and Precision

Provided the test is properly conducted and the precautionary measures above are observed, the expected deviation is less than 3 percent for the total sample and generally less than 0.5 percent for each screen.

7.3.1.9 Calibration

Referenced documents are ASTM C-136, *Standard Method for Sieve Analysis of Fine and Coarse Aggregates,*[2] and ASTM C-702, *Practice for Reducing Field Samples of Aggregate to Testing Size.*[3]

7.3.2 Sieve Analysis (Sonic Shaker)

7.3.2.1 Principle

Refer to 7.3.1, except that properly prepared samples are vibrated through the sieve screens with acoustic energy.

7.3.2.2 Apparatus

The following equipment is recommended:

a. Sonic sieving apparatus.
b. U.S. or Tyler sieves (see 7.3.1.2b).
c. Balance (0.001 gram).
d. Mortar and soft-tipped pestle.
e. Soft bristle brush.
f. Sample splitter.
g. Drying oven.

7.3.2.3 Procedure

Sample preparation and measurement procedures follow the material provided in 7.3.1. Five to fifteen grams of properly prepared sample are placed in the top screen. Sift and pulse amplitudes are adjusted to effectively agitate grains. Sifting time for effective sorting varies from ten to twenty minutes and is dependent upon sample size and degree of shaliness.

7.3.2.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a through f.
b. More accurate weight determinations are required than in mechanical sieve analysis because the sample sizes are smaller.

7.3.2.5 Calculations

Refer to 7.3.1.5.

7.3.2.6 Advantages

Advantages of this method include:

a. The method is acceptable for classifying grain sizes over the >0.044 millimeter size range.
b. The test is simple and can be conducted with a minimum of equipment.
c. The test can be conducted more rapidly that the mechanical shaker sieve analysis method (approximately 15 minutes per sample).
d. Less sample is required for the sonic method (5-15 grams) than for the mechanical method.
e. The sample can be recombined for reruns, retained in separate size fractions, or recombined for additional testing.

7.3.2.7 Limitations

Limitations of this method include:

a. Care must be taken to obtain a representative sample due to small sample size.
b. Charged fines retention can be particularly troublesome using this method because the screen frames are plastic. As a result, the sieving time must be increased in shaly sands. In many cases, a significant increase in sieving time may not be adequate to move the <0.044 millimeter fines to the pan.

7.3.2.8 Accuracy

Refer to 7.3.1.8.

7.3.2.9 Calibration

Refer to 7.3.1.9.

7.3.3 Sieve Analysis (Wet and Wet/Dry Combination)

7.3.3.1 Wet sieve analysis refers to the technique of washing a representative, extracted sample through individual sieve screens as opposed to the dry sieving technique, whereby the sample is mechanically agitated or sonified through a nest of sieve screens. The technique requires significantly more time than dry sieving techniques but is preferred in shaly sands.

7.3.3.2 Wet/dry combination sieve analysis, as the name implies, is a combination of both wet and dry techniques. This technique is a very good alternative in shaly sands. The sample is weighed and then wet sieved through the 325 mesh screen to remove the <0.044 millimeter fraction. The material retained on the 325 mesh screen is dried, weighed, and dry sieved in the manner prescribed in the sieve analysis (mechanical or sonic shaker) sections (see 7.3.1 and 7.3.2).

7.3.4 Particle Size Analysis (Laser Diffraction)

7.3.4.1 Principle

An extracted, disaggregated sample is placed into a suitable carrier fluid (carrier fluid may be oil-based or water-based, depending upon test criteria). The carrier fluid is typically stirred or recirculated to ensure particle dispersion. A laser beam is projected through the dispersed sample and the diffracted light patterns are analyzed by microcomputer. The principle upon which grain size distribution is computed is that of Fraunhofer diffraction theory. The angles through which the incident light is scattered are inversely proportional to particle sizes, and the intensity of light scattered by the particles is directly proportional to particle quantities.

7.3.4.2 Apparatus and Supplies

Recommended equipment for this method include:

a. Laser optics device/appropriate computer hardware.
b. Sample splitter.
c. Balance (0.0001 gram), not required for all devices.
d. Mortar and soft-tipped pestle.
e. Sonifier.
f. Deflocculant solution.
g. Drying oven.

7.3.4.3 Procedure

A representative sample is extracted to remove hydrocarbons and dried to remove solvent. The sample is sonified prior to measurement to disperse the particles. The carrier fluid reservoir is charged with carrier fluid and a background measurement is taken. The sample is introduced and surfactant (typically sodium hexametaphosphate) is added to reduce surface tension and to promote particle deflocculation. The carrier fluid/sample mixture is recirculated or stirred sufficiently to ensure uniform particle dispersion. During the measurement phase, a laser beam of known and fixed wave length is projected through the carrier fluid/sample mixture. The forward-diffracted light impacts a photodetector array and Fraunhofer diffraction theory is invoked to compute particle size distribution from the diffracted light flux. The data may be reported in sieve analysis format in tabular and graphical form to the sub-micron particle size range (see Table 7-2 and Figure 7-1).

7.3.4.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a and b.
b. The instrumentation must be properly calibrated (including laser alignment, photodetector array calibration, etc.).
c. The carrier fluid must be bubble-free and particle-free throughout the entire measurement range.
d. The sample must be uniformly dispersed in the carrier fluid and must be deflocculated during the measurement phase.
e. Care must be taken to obtain a representative sample.

7.3.4.5 Calculations

The algorithms to calculate grain size distribution from composite diffraction patterns are complex, manufacturer-dependent, and proprietary. However, the following equations can be utilized to illustrate the relationships between the angular extent of the diffraction pattern, particle size, scattered light intensity, and particle quantity.

The angular extent of the diffraction pattern is given by:

$$\sin\theta = \frac{1.22\lambda}{d} \tag{3}$$

Where:

θ = half-angle to the first minimum of the pattern, degrees.
λ = incident light wave length, microns.
d = particle diameter, microns.

The total intensity of the scattered light is given by:

$$I = Knd^2 \tag{4}$$

Where:

I = total diffracted light, watts.
K = instrument calibration constant, watts/micron^2.
n = number of particles.
d = particle diameter, microns.

7.3.4.6 Advantages

Advantages of this method include:

a. The grain size measurement range provides classification of particles to and including the sub-micron range (very fine silt and clay sized particles).
b. The data are highly reproducible.
c. The technique requires very little sample (typically <1.0 gram).
d. The measurement is rapid (30 seconds to several minutes).

7.3.4.7 Limitations

Limitations of this method include:

a. A size limit exists above which measurements cannot be made. This limit typically falls within the coarse sand fraction. If particle sizes exceed this limit, pre-sieving is required and the data sets must be combined.
b. The instrumentation and associated hardware is expensive.

7.3.4.8 Calibration

Calibration requirements are instrument-manufacturer dependent but generally involve accurate laser beam alignment, photodetector array calibration, and diffraction pattern calibration using a precision standard.

7.3.4.9 Accuracy

Insufficient data exist at present to establish accuracy.

7.3.5 Thin Section Measurement of Grain Size (Visual Technique)

7.3.5.1 Principle

Grain size measurement using thin sections can be undertaken on samples that range from unconsolidated sand to well-consolidated rock. It is the method of choice for consolidated rock, because such rocks cannot be disaggregated successfully without either breaking grains or failing to separate adjacent, well-cemented grains. The technique requires preparation of a thin section and measurement of the size of individual grains visually or with an image analysis system, using a petrographic microscope.

7.3.5.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Petrographic microscope equipped with an eyepiece micrometer.

b. Thin section preparation equipment.

c. Glass slides.

d. Colored epoxy resin.

7.3.5.3 Procedure

Extracted samples are usually impregnated with colored epoxy resin, mounted on a glass slide, and ground to 30 microns thickness. This thickness allows light to be transmitted through most sand grains. Grain size is measured utilizing the longest axis of individual grain cross sections in the two-dimensional thin section. In many cases, unconsolidated samples need only be dispersed in oil on a glass slide. It is customary to restrict size measurement to a single specific detrital mineral species (usually quartz in sands), although other species may be included (such as feldspar). Typically, 200 to 300 grains are measured. The actual number is dependent upon sorting; poorly-sorted sands require more measurements than well-sorted sands. Grains are selected randomly using the point count technique described by Chayes (1956).[4] Measurements are typically made in millimeters and units are subsequently converted to units of preference (inches, phi units, etc.).

7.3.5.4 Precautions

Precautions for this method include:

a. Thin sections allow for grain size measurement in only two dimensions. Because grains are irregularly shaped and non-uniform in size, and because almost all sediments composed of non-spherical particles show anisotropic dimensional fabrics (i.e., preferred grain orientation), without gross simplifying assumptions there is no unique solution for inferring true grain size distributions determined from thin section measurements (Blatt et al., 1980).[5]

b. Apparatus must be calibrated.

c. Thin sections should be representative of the reservoir interval.

d. Care must be taken to obtain a representative split of the sample because relatively few grains are measured.

e. Sufficient grains must be available for measurement in the thin section to ensure statistical reliability.

f. For laminated sands with contrasting grain size between laminae, unbiased grain size selection can be difficult.

7.3.5.5 Calculations

Grain size is measured using non-dimensional units visible through the microscope eyepiece. These units are scaled to millimeters using a conversion factor dependent upon the magnification used. Size distribution is calculated on a volume frequency basis.

7.3.5.6 Advantages

Advantages of this method include:

a. The measurement range is typically from U.S. Sieve mesh 10 to 400.

b. Thin section measurement of grain size distribution is well suited to most reservoir rocks (sandstones and coarser grained siltstones).

c. The technique allows for standardized measurement of grain size in reservoirs characterized by a range of rock consolidation.

7.3.5.7 Limitations

Limitations of this method include:

a. The technique is tedious, relatively time consuming, and limited to a two-dimensional view.

b. Equipment and instrumentation is expensive.

c. Derivation of mean grain size and the size distribution from the thin section is not straightforward and can be biased. The data are adequate in a relative sense (Kellerhals et al.).[6]

d. Image resolution (magnification and thin section thickness dependent) precludes measurement of very small particles (finer silt and clay size fractions).

7.3.5.8 Accuracy and Precision

The data are slightly biased towards smaller sizes. Precision is a function of the number of grains measured.

7.3.5.9 Calibration

Calibration of size measured in thin section with size measured by sieving and other techniques is possible (Rosenfeld et al. 1953).[7]

7.3.6 Thin Section Measurement of Grain Size (Image Analysis)

7.3.6.1 Principle

Grain size estimates from unconsolidated and consolidated sands can be obtained using automated or semi-automated image analysis procedures. The information provided in 7.3.6 primarily refers to grain size estimates in unconsolidated sands.

7.3.6.2 Apparatus and Supplies

Recommended equipment for this method includes:

a. Image analyzer.

b. Petrographic microscope with light source.

c. Thin section preparation equipment.

d. Glass slides.

e. Colored epoxy resin.

7.3.6.3 Procedure (Unconsolidated Sands)

Sand grains can be dispersed on a glass slide such that few are touching. The projection area or diameter of each grain can be obtained using a simple image analysis apparatus comprised of a videocamera attached to a microscope (Mazullo and Kennedy, 1985).[8] Grains are separated from background using the difference in intensity of the background (brighter) and the grains (darker) as a criterion for thresholding. Typically 200 to 500 grains are required which require 5 to 15 minutes to process.

7.3.6.4 Procedure (Consolidated Sands)

Grain-size distribution using image analysis techniques outlined in 7.3.6 are complicated in consolidated sands by virtue of grain contact. However, techniques exist that utilize the center-to-center distance of intergranular pores to estimate grain size.

7.3.6.5 Precautions

Precautions for this method include:

a. Refer to 7.3.5.4.
b. Inaccurate measurements can occur if the grains to be measured are in contact with one another. This can result in diameters greater than actual.

7.3.6.6 Advantages

Advantages of this method include:

a. Acquisition of raw data is reasonably fast and data are stored in a digital format.
b. Very small samples can be analyzed.

7.3.6.7 Limitations

Limitations of this method include:

a. Care must be taken such that the few hundred grains are representative. This may require careful sample splitting and a careful grain selection procedure on the grain mount.
b. The image acquisition system with software and videocamera is more expensive than the visual technique.

7.3.6.8 Calculations

Data must be converted to volume frequency. This is done by: (a) defining a number of class intervals based on grain diameter, (b) cubing the radii of all grains in each class interval and adding volumes within each interval, and (c) summing all of the class interval volumes to obtain the total volume. The volume fraction in each class interval is calculated by dividing by this total.

7.3.6.9 Accuracy and Precision

The data have been demonstrated to be accurate and deviate only slightly from sieve data (the difference is due to the fact that sieves are shape-selective as well as size-selective).

7.3.6.10 Calibration

Pixel size for a given magnification must be defined when an image analysis system is utilized.

7.3.7 Stoke's Law Particle Size Analysis

7.3.7.1 Principle

A representative, extracted sample is introduced into a liquid column, and the settling velocities of the particles are determined by monitoring the weight increase of a pan suspended in the column. The sedimentation diameter of the particles (the diameter of a sphere of equal density having an equal settling velocity) is calculated using Stoke's Law, a modified form of Stoke's Law, or Gibbs' formula (Gibbs et al., 1971[9]). The data are typically reported in the same manner as standard sieve data.

7.3.7.2 Apparatus

Recommended equipment for this method includes:

a. Mortar and soft-tipped pestle.
b. Soft bristle brush.
c. Bottom loading balance (0.001 gram) or strain transducer.
d. Settling column and associated computer hardware.
e. Drying oven.

7.3.7.3 Procedure

Sample preparation should follow the protocol outlined in 7.3.1. The sample is precisely weighed (0.001 gram) and introduced into the settling column. The particles fall through the liquid at rates related to size, shape, and density. The particles accumulate on a pan suspended from a precision balance or strain transducer a known distance from the top of the settling column. Weight and time data are acquired by a microcomputer. These data are utilized to compute settling velocities. The test duration varies from several to twenty minutes relative to the type of equipment/instrumentation and the particle size measurement range desired. A modified form of Stoke's Law or Gibbs' formula is typically utilized to calculate sedimentation diameters. If all of the sample is not recovered at the test end point, the weight difference is classified as being finer than the equivalent sedimentation diameter of the last measurement.

7.3.7.4 Precautions

Precautions for this method include:

a. Refer to 7.3.1.4, items a, b, and c.
b. The settling column must be clean (the fluid density must be constant, and suspended particle fallout on a subsequent measurement must be negligible).
c. The particles must be evenly dispersed upon introduction into the column and must be deflocculated.
d. Very careful weighing is required as a result of the small sample quantity used.
e. Convection currents must be eliminated.

7.3.7.5 Calculations

Stoke's Law, a modified form of Stoke's Law or Gibbs' formula are typically used to calculate grain radii from the settling velocity data.

Stoke's Law:

$$V = \frac{2gr^2(\rho_g - \rho_f)}{9\mu} \tag{5}$$

Gibbs' Formula:

$$V = \frac{-3\mu + \sqrt{9\mu^2 + gr^2\rho_f(\rho_g - \rho_f)(0.015476 + 0.19841r)}}{\rho_f(0.011607 + 0.14881r)} \tag{6}$$

Where:
V = settling velocity, cm/sec.
g = acceleration of gravity, cm/sec^2.
r = grain radius, cm.
ρ_g = grain density, g/cc.
ρ_f = fluid density, g/cc.
μ = fluid viscosity, poise.

7.3.7.6 Advantages

Advantages of this method include:

a. The test is rapid (several to twenty minutes).
b. The equipment is relatively inexpensive.
c. The measurement range is relatively broad (through U.S. Sieve 500 mesh).

7.3.7.7 Accuracy and Precision

Information not available.

7.3.7.8 Calibration

The calibration procedure required is described by Gibbs *et al* (1971).[9]

7.4 OIL GRAVITY

The specific gravity of the oil must be known in order to convert oil weight to oil volume in the distillation-extraction method for determining fluid saturations (see 4.3). The oil gravity may be determined by an oil drop method, a refractive index method, a gravimetric method, a digital densitometer method, or a nuclear magnetic resonance method.

7.4.1 Oil Drop Method

7.4.1.1 Principle

The oil drop method consists of suspending a drop of the oil in a liquid medium, the gravity of which can be measured with a hydrometer or specific-gravity balance.

7.4.1.2 Apparatus

Apparatus for this method includes:

a. Glass or clear plastic cylinder.
b. API hydrometer or specific-gravity balance.

7.4.1.3 Procedure

A drop of the oil recovered from the core during the fluid saturation test should be placed in a glass or plastic cylinder containing a solution of methyl alcohol and water. The gravity of this solution is subsequently adjusted by the addition of alcohol or water until the oil drop remains suspended; i.e., after agitation, the drop neither rises nor falls. The gravity of the solution is then measured with an API hydrometer or a specific-gravity balance. The correct procedure for measuring gravity with a hydrometer is found in ASTM D287-55, *Method of Test for API Gravity of Petroleum.*[10] The observed API gravity at the test temperature is corrected to the gravity at 60°F and converted to specific gravity by the use of Tables 5 and 3, respectively, of the ASTM-IP *Petroleum Measurement Tables* (American Edition).[11] A specific-gravity balance permits a direct reading of gravity. A simple chart for converting units of liquid gravity and density may be useful.

7.4.1.4 Precautions

Precautions for this method include:

a. Any air bubbles adhering to the surface of the oil drop must be removed before adjusting the gravity of the alcohol-water solution.

b. The solution must be agitated after each addition of alcohol or water to ensure a uniform solution and gravity.

7.4.1.5 Advantages

Elaborate equipment is not required.

7.4.1.6 Limitations

Adjustment of the alcohol-water ratio is time consuming.

7.4.1.7 Accuracy and Precision

Information not available.

7.4.2 Refractive Index Method

7.4.2.1 Principle

The refractive index method for determining API gravity is most commonly used to determine the gravity of oil recovered from core samples during the summation of fluids saturation test. The technique is also used to determine the API gravity of stock tank oil, formation test oil, drill stem test oil, etc., in those cases in which very small quantities of oil are available.

A suite of stock tank oil samples of known API gravities for the normally encountered gravity range is required. The refractive indices of the oils, before and after the summation of fluids procedure, are measured. Refractive index versus oil gravity plots are made. Oil gravity can then be determined graphically or calculated using a simple linear regression.

7.4.2.2 Apparatus

Apparatus for this method includes:

a. API hydrometers.
b. Assorted glassware.
c. Glass pipette.
d. Refractometer (refractive index range 1.30-1.90).

7.4.2.3 Procedure

The API gravity of a stock tank oil sample is determined with an API hydrometer using ASTM D287-55: *Method of Test for API Gravity of Petroleum,*[10] or with a specific-gravity balance. A representative aliquot of oil is recovered from the stock tank sample with a glass pipette and the refractometer is charged with the sample. The refractive index of the sample is determined. The stock tank oil sample is then subjected to the retorting phase of the summation-of-fluids analysis (see 4.2). (Retort method for fluid saturation determinations, both sidewall and conventional core analysis.) The refractive index of the recovered oil is determined. Plots of refractive index before and after retorting versus API gravity should be made. API gravities can then be determined graphically or calculated.

7.4.2.4 Precautions

Precautions for this method include:

a. Operation of the refractometer must be verified by measuring the refractive index of a known liquid.

b. The refractometer prism and the glass plate of the refractometer eyepiece must be thoroughly clean.

c. The refractometer prism must be securely placed against the refractometer eyepiece when making measurements.

d. Contaminants such as water and solids must be removed from the oil prior to measurement.

e. The crude must be light-colored and a thin-film refractometer must be utilized.

7.4.2.5 Calculations

A simple linear regression of the stock tank oil API gravities and refractive indices can be made. The refractive index, slope, and intercept can be utilized to calculate the API gravity.

7.4.2.6 Advantages

Advantages of this method include:

a. The technique is simple and rapid, and the data are reasonably accurate.
b. Elaborate equipment is not required.
c. The test requires very little sample (0.03 cc).

7.4.2.7 Accuracy and Precision

API gravity determined using this method should be ±1 degree API of the gravity determined by ASTM D287-55[10], if the calibration is properly made and if the precautionary measures are taken.

7.4.3 Gravimetric Method (Micro-Pycnometer Method)

7.4.3.1 Principle

The density of an oil sample is determined using a small pycnometer and the API gravity is computed.

7.4.3.2 Apparatus

Equipment for this method includes:

a. Balance (0.001 gram).

b. Calibrated pycnometers—The pycnometers can be made of thin-walled glass capillary tubing formed in a U-shape. Various sizes can be prepared by varying the length and/or the inside diameter of the tubing. The volumes should be calibrated with a liquid of known density, with the pycnometer completely full or filled to reference lines (gas-free distilled water may be used).

7.4.3.3 Procedure

A clean, dry pycnometer is weighed. A portion of the oil to be analyzed is drawn into the pycnometer. It is preferable that the largest practical amount of sample be utilized for the gravity determination. The pycnometer is re-weighed. The oil weight is calculated and divided by the capillary volume to determine the oil density. The specific gravity is calculated by dividing the oil density by the water density at the test temperature. The API gravity is calculated.

7.4.3.4 Precautions

The pycnometer weight should be minimized relative to the oil weight. This will reduce error as the weight difference becomes more accurate.

7.4.3.5 Calculations

The following are the calculations for this method:

$$\text{Oil Density (g/cc)} = \frac{\text{charged pycnometer wt (g)} - \text{initial pycnometer wt (g)}}{\text{pycnometer volume (cc)}} \quad (7)$$

$$\text{Specific Gravity} = \frac{\text{oil density (g/cc)}}{\text{water density (g/cc)}} \quad (8)$$

$$\text{API Gravity (deg)} = \frac{141.5}{\text{specific gravity}} - 131.5 \quad (9)$$

7.4.3.6 Advantages

Advantages of this method include:

a. Accurate data can be obtained if the pycnometer has been properly calibrated.

b. The determination is rapid.

7.4.4 Digital Densitometer Method

7.4.4.1 Principle

The measurement principle of the digital densitometer is based on the change of the natural frequency of a hollow oscillator when fluid-filled. The oscillator frequency change is related to the mass change of the oscillator subsequent to sample introduction, and as a result, to the sample density.

7.4.4.2 Apparatus

A digital densitometer is required.

7.4.4.3 Procedure

Instrument calibration constants are determined by measurement of the air-filled and distilled water-filled oscillator frequencies. The sample is injected into the oscillator and the oscillator frequency is determined. The API gravity is calculated as indicated in 7.4.3.5.

7.4.4.4 Advantages

The technique is rapid and accurate.

7.4.4.5 Limitations

Relatively large amounts of oil are required (1 to 2 cc).

7.4.5 Nuclear Magnetic Resonance (NMR) Method

7.4.5.1 Principle

The NMR method for determining the viscosity of crude oils is based on Stoke's relationship between viscosity and molecular translational and rotational diffusion that cause NMR spin-lattice relaxation. Either ^{1}H or ^{13}C NMR can be used. A major advantage is that the method can be applied to bulk crude oil and/or oil/water emulsions, crude oil inside core plugs or whole cores, or crude oil dissolved in solvent extracts.

7.4.5.2 Apparatus

Fourier Transform NMR Spectrometer with ^{1}H chemical shift or ^{13}C capabilities. For NMR diffusion measurements, a pulsed gradient NMR probe is required with field gradients above 100 gauss/cm.

7.4.5.3 Procedure

The ^1H or ^{13}C spin lattice relaxation time (T1) is measured with an inversion recovery or saturation recovery pulse sequence. Viscosity and API gravity are then determined from published correlations. ^{13}C NMR is preferred for analysis of oils inside cores because chemical shift separation between oil and water phases in the core is not required.

The application of a large, steady-pulsed magnetic field gradient enables direct ^1H NMR measurements of translational diffusion in liquids. The pulse sequence is normally a 90°-T-180°-spin echo with magnetic field gradient pulses applied after the 90° and 180° RF pulses. Diffusion in an applied field gradient causes an irreversible loss in the spin echo amplitude because only the nuclei that do not diffuse to a region with a different magnetic field will be refocused. The interpulse time (T) is kept very short (approximately 1 millisecond) to prevent restricted diffusion at the pore walls, and the water and oil signals are separated either by chemical shift spectroscopy or by doping the water with Mn-EDTA to eliminate the water signal. The oil diffusion coefficient can be related to viscosity via Stoke's Law and API gravity can be estimated from published correlations between viscosity and API gravity.

7.4.5.4 Advantages

Advantages of the NMR method include:

a. The technique is rapid, noninvasive, and can be automated.
b. The technique is applicable to bulk oil, oil in plug cores, or whole core, and liquid extracts.
c. Minimal sample preparation is required.
d. Measurements can be made on oil/water emulsions.
e. Very small amounts of oil are required for ^1H NMR (<0.1 cc).

7.4.5.5 Limitations

Limitations of the NMR method include:

a. The NMR spectrometer is expensive.
b. The T1 method is applicable for oils only up to approximately 1,000 cp (approximately 15 degrees API) because of the flattening of the T1 versus viscosity correlation. This limitation does not apply to NMR diffusion measurements, which are only limited by the strength of the applied field gradient.

7.5 OIL CHARACTERIZATION

7.5.1 Oil Characterization (Chromatographic Method/Hydrocarbon Vaporization)

Residual hydrocarbons entrained in drill cuttings and core samples can be analyzed to quantify hydrocarbon volume and to evaluate the compositional characteristics of the oil. These data can be interpreted to provide information in the following areas:

a. Alteration processes—The crude oil quality is dictated by one or more of the alteration processes that affect the oil during, or subsequent to, its accumulation in a reservoir. The changes resulting from alteration processes are reflected in the composition of the residual hydrocarbon found in the rock pore spaces.
b. Type of hydrocarbon production—The composition of the residual hydrocarbon in the rock matrix at the surface reflects the original reservoir composite less components that have escaped through pressure depletion. The distribution of intermediate and heavier components can be used to predict the type of hydrocarbon production (non-productive, gas, condensate, or oil).
c. Changes in rock characteristics (permeability)—The escape of components during pressure depletion is limited by molecular size and hydrocarbon volatility, but is also greatly influenced by permeability. Compositional contrast can often be related to permeability and permeability variation.
d. Contamination (drilling fluids and/or additives)—Drilling fluids and/or additives can be analyzed by the same procedure as the rock samples. Compositional contrast and hydrocarbon ratio techniques can be utilized to distinguish indigenous hydrocarbons from drilling contaminants.
e. Correlation of produced oil to drill cuttings or core sample oil.

7.5.1.1 Principle

This method involves vaporization of residual hydrocarbons in drill cuttings or core samples directly onto a chromatographic column. The components separated by the column are detected by a flame ionization detector (FID). Peak heights and areas are obtained from the recorded output.

7.5.1.2 Apparatus

Apparatus recommended for this method includes:

a. Chromatograph—The chromatograph should contain a large bore capillary column (0.5 millimeter ID) containing a stationary phase that is suitable for separation and a flame ionization detector.
b. Vaporization cell (low dead volume, accurate temperature control, and even temperature distribution).
c. Sample boat (quartz tube).
d. Integrator.
e. Balance (0.001 gram).

7.5.1.3 Procedure

A representative sample (0.01 to 1.0 gram) should be selected and placed in a tared quartz sample boat (drill cuttings must be rinsed with water to remove drilling fluid and air dried for two to four hours prior to charging the boat). The quantity of sample used is dependent on the oil saturation, the detector capacity, and the column limitations. The charged sample boat is weighed and placed in the vaporization cell. Human skin oil is a possible contaminant and care should be taken to avoid directly handling the sample.

The hydrocarbons are vaporized at a temperature that will not induce cracking. Temperatures above 320°C will cause cracking of larger molecules. The gas evolved is passed directly onto the column without splitting. Resolution can be enhanced by increasing the column temperature at a programmed rate. Output of the flame ionization detector can be converted to millivolts (height) and millivolts x seconds (area). Specific peaks are identified by their retention time.

7.5.1.4 Precautions

Precautions for this method include:

a. The sample must be representative.

b. Large samples may increase reproducibility but will restrict peak resolution.

c. High bound-water saturation may cause a suppression of detector response, particularly in the lighter midrange components.

d. Accurate weights are required (±0.001 gram).

e. Care must be taken to avoid heating during sample drying to prevent lighter component losses.

7.5.1.5 Calculations

7.5.1.5.1 Quantification of residual hydrocarbon volume:

$$\text{Normalized total area } (A_{nt}) = \frac{\text{total area } (A_t)}{\text{sample weight } (g)} \qquad (10)$$

7.5.1.5.2 Alteration processes—Indices can be tabulated to distinguish changes in composition.

Following are several indices that are typically used to determine type, origin, and alteration processes:

a. $$n\text{-C15+ } (\%) = \frac{> n\text{-C15 area}}{A_t} \times 100 \qquad (11)$$

b. $$n\text{-alkane } (\%) = \frac{n\text{-c components area}}{A_t} \times 100 \qquad (12)$$

c. ratio n-C17 + n-C18 to pristane+phytane =

$$\frac{(\text{height n-C17} + \text{height n-C18})}{(\text{height pristane} + \text{height phytane})} \qquad (13)$$

d. $$\text{pristane/phytane ratio} = \frac{\text{height pristane}}{\text{height phytane}} \qquad (14)$$

e. Carbon preference index =

$$\left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C16-C30}}\right| + \left|\frac{\Sigma\text{odd heights n-C17-C31}}{\Sigma\text{even heights n-C18-C32}}\right| \qquad (15)$$

7.5.1.5.3 Type of hydrocarbon production:

The area percent within each carbon group (light, intermediate and heavy) is calculated as follows:

a. $$\%\text{ light components} = \frac{< \text{C8 area}}{A_t} \times 100 \qquad (16)$$

b. $$\%\text{ intermediate components} = \frac{< \text{C13 area}}{A_t} \times 100 \qquad (17)$$

c. $$\%\text{ heavy components} = \frac{> \text{C18 area}}{A_t} \times 100 \qquad (18)$$

Prediction of hydrocarbon production can be based on the following general guidelines:

a. Non-hydrocarbon productive—relatively high percentage of lighter components, relatively low total area.

b. Gas productive—relatively high percentage of lighter components with lower percentages of intermediate components, relatively high total area.

c. Oil productive—relatively high percentage of intermediate and heavy components, high total area.

7.5.1.6 Advantages

Advantages of this method include:

a. Extremely small samples (0.01-1.0 gram) can be utilized.

b. Solvent extraction and sample splitting are not required.

c. Oil composition and residual hydrocarbon content can be monitored simultaneously.

7.5.1.7 Limitations

Bound water may suppress detector response, particularly in the lighter midrange components.

7.5.1.8 Accuracy

Accurate sample weights and careful control of operating conditions should yield oil volumes reproducible to within 3 percent. Reproducibility of component distribution is dependent on maintenance of operating conditions and column condition and can be verified by repeat runs.

7.5.1.9 Calibration

Equipment calibration is essential for consistent results. The following areas are critical:

a. Component identification—Identification of individual peaks is based in part on retention time. Retention time is dependent on carrier gas flow rate, column temperature, and the condition of the column stationary phase. Carrier gas flow rate should be calibrated and the system should be leak tested routinely. Retention times for specific peaks can be measured. A sample can be spiked with specific components, such as the normal alkanes for calibration purposes.

b. Calibration of detector response—A standard should be used to check peak resolution and system noise. Relative areas should be consistent. Relative standard deviation of 2 to 3 percent is typical.

c. Reproducibility—Reproducibility can be evaluated by making repeated runs of the same sample. Specific indices used should be checked along with total area normalized by weight.

7.6 ACID SOLUBILITY

7.6.1 Principle

Acid solubility tests on drill cuttings or core samples are typically used to assess HCl and/or mud acid (HCl/HF mixture) reactivity with reservoir rock. These data are used to evaluate the potential success of acid stimulation, near wellbore damage removal, to assess formation damage sensitivity, to determine calcite/dolomite ratios in carbonates, and to determine quartz content in carbonates. Test procedures can be adapted to determine acid volumes required for field treatment and to determine acid response coefficients.

The acid solubility test is a gravimetric test performed to determine formation sensitivity to acid. A known mass of extracted and dried sample is placed in excess acid for a specific residence period. Subsequently, the sample is dried and reweighed. The weight difference is used to calculate the percent acid-soluble material.

7.6.2 Apparatus and Supplies

Equipment for acid solubility determination includes:

a. Balance (0.001 gram).

b. Assorted glassware for HCl solubility (beakers, funnels, graduated cylinders, etc.).

c. Assorted polyethylene or polypropylene beakers, funnels, graduated cylinders, etc. for mud acid solubility.

d. Mortar and pestle.

e. U.S. Sieve, 80 mesh screen.

f. Drying oven.

g. Acid-resistant filter paper.

h. HCl, 15 weight percent.

i. Mud acid (HCl, 12 weight percent—HF, 3 weight percent).

j. Appropriate safety equipment (face shield, apron, etc.).

7.6.3 Procedure (HCl Solubility, Calcite Content)

A representative sample is extracted to remove hydrocarbons and dried to constant weight to ensure removal of solvent and water. The sample is disaggregated sufficiently to produce several grams of material finer than U.S. Sieve 80 mesh. Approximately 1 gram of sample is weighed and placed in 150 cc of 15 percent HCl for a minimum of 60 minutes and a maximum of 65 minutes without stirring. The filtration apparatus is prepared and the filter paper is weighed. The acid/sample mixture is filtered and rinsed with deionized water. The remaining sample is dried and weighed. The weight difference is used to compute the percent acid soluble material.

7.6.4 Procedure (HCl Solubility, Calcite Plus Dolomite Content)

Dolomite is also soluble in HCl at 80°C. Applying the procedure in 7.6.3 at this temperature yields a value referred to as the total carbonate content (calcite plus dolomite).

7.6.5 Procedure (Mud Acid Solubility, Total Acid-soluble Content)

Carbonates and siliceous materials such as feldspars and clays dissolve in a mixture of HCl and HF. This mixture is generally called mud acid. Although various concentrations are in use, a mixture of 12 weight percent HCl and 3 weight percent HF is most commonly applied. Applying the procedure above, using HF tolerant apparatus, with mud acid results in the total acid solubility (i.e., carbonates, feldspars, iron oxides, clays, etc.).

7.6.6 Precautions

The procedure is gravimetric and requires strict adherence to procedures and good laboratory technique to provide reproducibility.

CAUTION: In all procedures outlined in 7.6.3, 7.6.4, and 7.6.5, appropriate safety precautions for handling acids must be employed.

7.6.7 Calculations

The following are the calculations for this method:

Calcite Content (HCl, room temperature):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (19)$$

Total Carbonate Content (HCl, 80°C.):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (20)$$

$$\% \text{ Dolomite} = \% \text{ total carbonate content} - \% \text{ calcite content} \quad (21)$$

Total Acid Soluble Content (Mud acid):

$$\% \text{ Acid soluble} = \frac{\text{initial sample weight} - \text{final sample weight}}{\text{initial sample weight}} \times 100 \quad (22)$$

7.6.8 Advantages

The technique is uncomplicated and requires simple equipment.

7.6.9 Limitations

Limitations of this method include:

a. The procedure is indicative of maximum rock reactivity, as the sample is disaggregated and the test is conducted in excess acid.

b. Siderite is soluble in HCl at room temperature and, as a result, can distort the calculated calcite content.

7.6.10 Accuracy and Precision

Information not available.

7.6.11 Calibration

The balance must be properly calibrated.

7.7 CORE WATER SALINITY DETERMINATION

A salinity determination on the water present in the core can be desirable because it could aid in core analysis data interpretation and in electric log evaluation.[12,13] Salinity or total dissolved solids (TDS) is defined as the sum of all dissolved materials in terms of milligrams of salt per kilogram of solution (mg/kg) which is equivalent to parts per million (ppm). Salinity is often calculated from the amount of chloride ion present in the core water and is expressed as the equivalent amount of sodium chloride. However, if the salinity is calculated from a resistivity measurement, it represents an NaCl equivalent of the contribution from all soluble electrolyte ions. Measurements of the core water salinity are based on the assumption that all of the chemical ions and gases in the core formation water at reservoir conditions are still contained in the core water at ambient conditions. This may not be the case for brines with ion concentrations near the solubility limits, i.e., soluble at reservoir temperature and pressure but insoluble at ambient conditions.

During the coring operation, the coring fluid (mud) filtrate may invade the core and dilute the formation water. There are three factors to minimize mud filtrate diluting the formation water. The core material should be obtained using a low invasion coring bit, the coring fluid should have a low API fluid loss, core samples for analysis should be taken at well site right after the core surfaces.[14,15,16] The core plugs for core water salinity should be taken from the center of the core, farthest from the perimeter of the core where filtrate invasion will be the greatest (see 4.3.7).[15] To avoid evaporation of water from the core/plug material, the core/plug should be sealed immediately after retrieval (see 2.5). Freezing the whole core after removal from the core barrel to prevent tracer migration and counter-current imbibition is not a recommended alternative to taking a plug sample at well site from the middle of the core material. A salinity front develops as the core freezes which concentrates salts ahead of the freeze line.[17]

If the salinity of the formation water and the mud filtrate are known, the degree of flushing by the coring fluid can be estimated by the core water salinity (but may be affected by nonuniform distributions). Even if the salinity of formation water is not known, the degree of flushing by the water based coring fluid may be estimated if the coring fluid contains a tracer chemical; such as tritiated water, bromide, or iodide, that can be assumed to be at a low concentration in the formation water (see 4.3.7).[18]

If the coring fluid contains a high concentration of potassium, because potassium chloride is the main source of salinity in the coring fluid, then the potassium ion can be used as the tracer. In formation waters, the ratio of sodium to potassium is much greater than one. A cation analysis (sodium and potassium) of the core water provides a method to determine if the core is relatively free of coring fluid. The use of potassium ion as a tracer can only provide a qualitative indication of the extent of coring fluid filtrate invasion compared with the more quantitative assessment using coring fluid tracer chemicals; such as tritiated water, bromide, or iodide. These anions are not chemically attenuated by the rock matrix as is possible for cations like potassium.

7.7.1 Core Water Recovery

To measure salinity of the core water, it must first be separated from the core. This can be achieved by mechanically expelling the core water from the core using an immiscible fluid flush or by centrifugation. When the core water is near irreducible saturation, mechanical expulsion may be unsuccessful. When no brine is expelled by mechanical means, then the salts can be water-extracted from a dried core. The salinity can then be estimated from the amount of salt extracted and the amount of water initially present in the core.

7.7.1.1 Centrifugation

7.7.1.1.1 Principle

Core fluids, including the formation water, may be expelled by centrifuging the sample.

7.7.1.1.2 Apparatus

Recommended apparatus for this method includes:

a. Centrifuge and sample holders.
b. Assorted glassware.

7.7.1.1.3 Procedure

The centrifuge should be of such a size and so driven that a force equal to at least 1,000 times the force of gravity can be exerted on the center of gravity of the core sample. The centrifuge sample holder should be an airtight unit having a perforated bottom with a receiving chamber. Unconfined core samples are placed in weight-matched centrifuge sample holders. The samples are centrifuged for at least one hour, preferably overnight if the sample permeability is less than 100 md. A sample of the produced brine is separated from the fluid mixture centrifuged from the core, with the use of a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.1.4 Precautions

Precautions for this method include:

a. The temperature of the centrifuge should be controlled to reduce the potential for water loss from the sample holders.
b. Care should be taken to avoid evaporation of water during all steps, starting with handling the core material to transferring of the brine sample to the sample container for salinity determination.

7.7.1.1.5 Advantages

Advantages of this method include:

a. The core sample does not have to have a cylindrical shape or be consolidated.
b. The presence of reservoir crude oil in the core sample does not interfere with this procedure or the validity of the results.

7.7.1.1.6 Limitations

Limitations of this method include:

a. The time required to centrifuge out the brine may be long for low permeability samples.
b. The procedure only works if the core contains a high enough brine saturation such that a portion can be removed at the highest available centrifuge speed.
c. The centrifugal force on the unconfined sample may irreversibly alter the matrix of the rock, changing porosity and permeability, such that the core may not be suitable for subsequent core analysis tests.
d. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.1.7 Accuracy/Precision

The accuracy/precision of the separation technique is limited by the ability to preserve core from well site to the laboratory and the subsequent handling of the core samples and the extracted brine sample without altering the salinity of the core water. Evaporation effects for relatively small samples or samples containing very low brine saturations, *Sw*=<5%, may increase salinity. Percent error will be significantly reduced as the amount of core water volume increases. Careful core handling practices may result in errors in core salinities due to core and brine handling being less than 2% of total salinity.[13]

7.7.1.2 Immiscible Fluid Flush

7.7.1.2.1 Principle

The formation water may be expelled by an immiscible displacement flush.

7.7.1.2.2 Apparatus

Apparatus for this method includes:

a. Hassler or triaxial core holder.
b. Solvent pump.
c. Assorted glassware.

7.7.1.2.3 Procedure

A core plug is mounted in a rubber sleeve Hassler or triaxial core holder cell that will permit fluid to flow through the matrix of the sample. Details of this procedure are given in 3.6.4.1. The water-immiscible fluid could be a refined mineral oil or an organic solvent like toluene. The higher the viscosity contrast between the water-immiscible fluid and water, the more likely that some formation water will be produced during the flush. A sample of the produced brine is separated from the fluid mixture flushed from the core using a separator funnel or by pipetting a sample from the brine layer. This sample should be saved for salinity determination.

7.7.1.2.4 Precautions

Precautions for this method include:

a. The fluid used to expel the formation water should have a low solubility for water so as not to change the salinity of the extracted water.
b. Care should be taken to avoid evaporation of water during handling of the core, flushing, and transferring of the brine to the sample container for salinity determination.

7.7.1.2.5 Advantages

The presence of reservoir crude oil in the core sample does not interfere with this procedure or affect the validity of the results.

7.7.1.2.6 Limitations

Limitations of this method include:

a. For low permeability samples, the time required to flush out the brine may be long.
b. This procedure only succeeds if the core contains a brine saturation which can be reduced by the available applied pressure drop, e.g., core is from water or oil-water transition zone.
c. If the net hydrostatic pressure used to confine the sample while flushing exceeds the net reservoir overburden pressure, the sample porosity may be irreversibly compacted and the sample may not be suitable for subsequent core analysis tests.
d. The core sample must have cylindrical shape.
e. The salinity of the core water expelled may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.1.2.7 Accuracy/Precision

See section 7.7.1.1.7.

7.7.2 Water Extraction of Core Salt

Core water salts are extracted from a dried core sample.

7.7.2.1 Apparatus

Apparatus for this method includes:

a. Mortar and pestle.
b. Balance.
c. Desiccator.
d. Assorted glassware.

7.7.2.2 Procedure

Approximately 50 grams of sample are selected. The water saturation must be known. The preferred method is to use a portion of the sample used in a previous saturation determination test. Less preferably, the sample may be selected from core material at a point as close as possible to the sample used in the saturation measurement test.

The sample must be free of hydrocarbons. Core material from a Dean-Stark toluene extraction is suitable. The sample is ground in a mortar to approximately 16-mesh size and dried in an oven to constant weight. After being cooled in a desiccator, the sample is weighed and transferred to a flask. 100 ml of distilled water is added and the mixture is stirred vigorously for several minutes. Agitation is continued intermittently for a minimum of one hour. The resulting salt solution is filtered or decanted and saved for salinity determination.

7.7.2.3 Precautions

Precautions for this method include:

a. If the core sample is suspected of containing clay material, a constant humidity oven should be used at a temperature of about 140°F (60°C) and 45 percent humidity during the drying step for determining core water content. This is done to avoid removing chemically- and physically-bound water (water that does not contribute to the salinity),[19] and thus to avoid overstating the amount of core water, which results in underestimating the core water salinity. Since constant humidity drying under these conditions does not dehydrate clays, no detectable fraction of the 100 ml of water added to the sample will be used to rehydrate the clays.
b. Care should be taken to avoid evaporation of water during all steps, starting with core preservation to transferring of the brine sample to sample container for salinity determination.

7.7.2.4 Advantages

This procedure is simple and can be conducted with very little equipment in a short time.

7.7.2.5 Limitations

Limitations of this method include:

a. The presence of hydrocarbons in the core sample interferes with the determination of core water content. The more volatile components of the hydrocarbons will be lost during the drying step, and thus the amount of water present will be overstated.
b. If the core material contains any sulfide minerals, such as pyrite that has oxidized during core handling, storage, or during extraction, the amount of salts extracted will be overstated. Oxidation of pyrite will produce water soluble iron sulfate salts.
c. If the in situ core rock matrix includes solid phase soluble salts (that are in solubility equilibrium with the formation brine), i.e., anhydrite or sodium chloride, then during drying and extraction these salts may be extracted because of the

high water to rock ratio during extraction, and the salinity of the core water will be overstated.

d. The sample is destroyed.

e. The calculated salinity of the core water may not be representative of the core water salinity at reservoir pressure and temperature conditions.

7.7.3 Determination of Core Water Salinity

The salinity of the water is determined, using either: (a) chemical determination of the chloride ion, (b) refractive index, or (c) resistivity measurement. The salinity is expressed as parts of sodium chloride per million parts of core water (although other salts are usually present), which is equivalent to milligrams of sodium chloride per kilogram of solution (sodium chloride and water).

There are several other analytical techniques for measuring the salt content of the core water. These include ion chromatography, atomic absorption, and inductively coupled plasma spectroscopy. A combination of these methods can accurately determine the cationic and anionic composition of core water, but the equipment is much more expensive than that required by procedures described in 7.7.3.1, 7.7.3.2, and 7.7.3.3.

7.7.3.1 Chemical Determination of Chloride Ion

The chloride concentration is measured in the undiluted brine solution expelled from the core or the filtrate obtained by separation of the crushed sample and the water used in the leaching process. The salinity of the water is expressed as parts per million sodium chloride in the core water (mg/kg).

7.7.3.1.1 Apparatus and Procedure

The apparatus, reagents, and procedure for chloride determination are described in detail in API RP 45,[20] *Recommended Practices for Analysis of Oilfield Waters*, and also in ASTM D512-89,[21] *Standard Test Methods for Chloride Ion in Water.* Of the four ASTM methods listed, Method A (Mercurimetric Titration), Method B (Silver Nitrate Titration), Method C (Colorimetric), and Method D (Ion-Selective Electrode), Method B is preferred. The calculation described in this reference will give the salinity in terms of chloride ion instead of sodium chloride. The calculation as sodium chloride is described in 7.7.3.1.3.

7.7.3.1.2 Precautions

Precautions are as follows:

a. Brine sample should be diluted to fall within optimum range of the titration solutions and equipment.

b. Make blank chloride determinations to correct for any chloride present in the glassware and water used in the tests.

7.7.3.1.3 Calculations

The following calculations are used:

a. The salinity of the core water expelled from the core sample can be calculated directly. The normality of salt solution measured by a chloride titration can be converted to sodium chloride concentration, mg/kg, as follows:

$$\text{Conc. of NaCl (mg/kg)} = 1{,}000 \times N_1 \times MW_{NaCl}/D_w \qquad (23)$$

Where:

$N_1 = V_2 \times N_2/V_1$ (24)

N_1 = normality of salt solution titrated, meq/ml.

V_1 = volume of salt solution titrated, ml.

N_2 = normality of chloride reagent (i.e., silver nitrate), meq/ml.

V_2 = volume of chloride reagent at titration endpoint, ml.

D_w = density of core water, gm/ml.

MW_{NaCl} = equivalent weight of NaCl = 58.5 gm/equ.

b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally-present core water must be back-calculated. The salinity from the chemical determination method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride.

The milligrams of sodium chloride leached from the core sample determined from the chloride titration are calculated as follows:

$$\text{Weight of NaCl (mg)} = N_1 \times 58.5 \times V_w \qquad (25)$$

Where:

$N_1 = V_2 \times N_2/V_1$ (26)

N_1 = normality of salt solution titrated.

V_1 = volume of salt solution titrated, ml.

N_2 = normality of chloride reagent (i.e., silver nitrate).

V_2 = volume of chloride reagent at titration endpoint, ml.

V_w = volume of water used to leach or extract the sample, ml.

The weight of the core water can be obtained directly by difference between the weight of the sample before and after drying, if no oil is present. The drying should follow the directions as given in 5.3.2.2.3.6.

The salinity (mg/kg) of the core water is obtained by:

$$\text{mg NaCl/kg of core water} = \frac{\text{mg NaCl}}{(\text{gm core water} + \text{gm NaCl})} \times 1000.0 \qquad (27)$$

If the sample contains oil, the following calculations can provide an approximate salinity value. The fluid saturations and the porosity must be determined either on this sample or an adjacent core. The amount of water contained in the sample can be calculated as follows:

$$\text{Weight of core water, gm} = V_b \times Ø \times S_w \times D_w \quad (28)$$

Where:

V_b = $[(W/D_g)/1 - Ø)]$ (29)
W = dry weight of sample, gm.
D_g = grain density, gm/cc.
$Ø$ = porosity, as fraction.
V_b = bulk volume of sample, cc.
S_w = water saturation, as fraction of pore volume.
D_w = density of core water, gm/ml.

If the grain density has not been determined, use a representative value for the formation of interest. The density of the core water is assumed to be 1.00 as an initial estimate. After the salinity is calculated with Equation 27, the weight of the core water, Equation 28, is recalculated using a new density based on the previously-calculated salinity; and a new salinity is recalculated using Equation 27.

7.7.3.1.4 Advantages

Advantages of this method are as follows:

a. Method is rapid and convenient.
b. Chloride determination is accurate.

7.7.3.1.5 Limitations

Limitations of this method include:

a. Result is expressed as sodium chloride. Anions other than chloride, such as sulfate and bicarbonate, present in the solution are not detected.
b. Other halides, such as bromide and iodide, are determined as chloride.

7.7.3.1.6 Accuracy/Precision

When the core water can be recovered from the core by centrifugation or an immiscible flush, the accuracy in the determination of salinity (mg/kg) is controlled by the salinity determination technique, chloride titration, on the core water sample which is about ±1 to 2% of the total salinity. API Recommended Practice 45 reports a precision of about 1% and an accuracy of about 2% of the amount present. When the core water salts have to be extracted from a dried core, the accuracy in the determination of core water salinity (mg/kg) is limited by the determination of the water content.[13] McCoy, et al.[13] report field data where chloride salinity determinations on oil-based-mud (OBM) cores intersecting the oil-water contact agreed within 1% on the average with produced water salinity. Chloride concentration measurements were obtained on the water extracts of about 2,000 OBM dried core samples (using procedures similar to 7.7.2 and 7.7.3).

7.7.3.2 Refractive Index Measurement

The total ionic constituents of water may be estimated by measuring the refractive index. The refractive index varies in a direct manner with the ionic concentration of sodium chloride and other salts. Standard graphs, which show the refractive index value for various salinities and temperatures of sodium chloride solutions, can be prepared from data in the literature (see Figure 7-2).[22,23] If the refractive index is known, the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured refractive index value.

7.7.3.2.1 Apparatus

Apparatus for this method includes:

a. Refractometer.
b. Assorted glassware.

7.7.3.2.2 Procedure

The instrumentation should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-2. Details on apparatus and procedures may be found in ASTM D542-85, *Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractomer.* The refractive index is measured on brine sample. With the use of a standard graph (see Figure 7-2) and suitable calculations, the refractive index value is converted to a salinity value for the core water.

7.7.3.2.3 Precautions

Refractive index value must be corrected to a standard temperature.

7.7.3.2.4 Advantages

Advantages of this method include:

a. Rapid determination.
b. The total equivalent sodium chloride concentration is determined.

7.7.3.2.5 Limitations

All ions present in the water are calculated as sodium chloride.

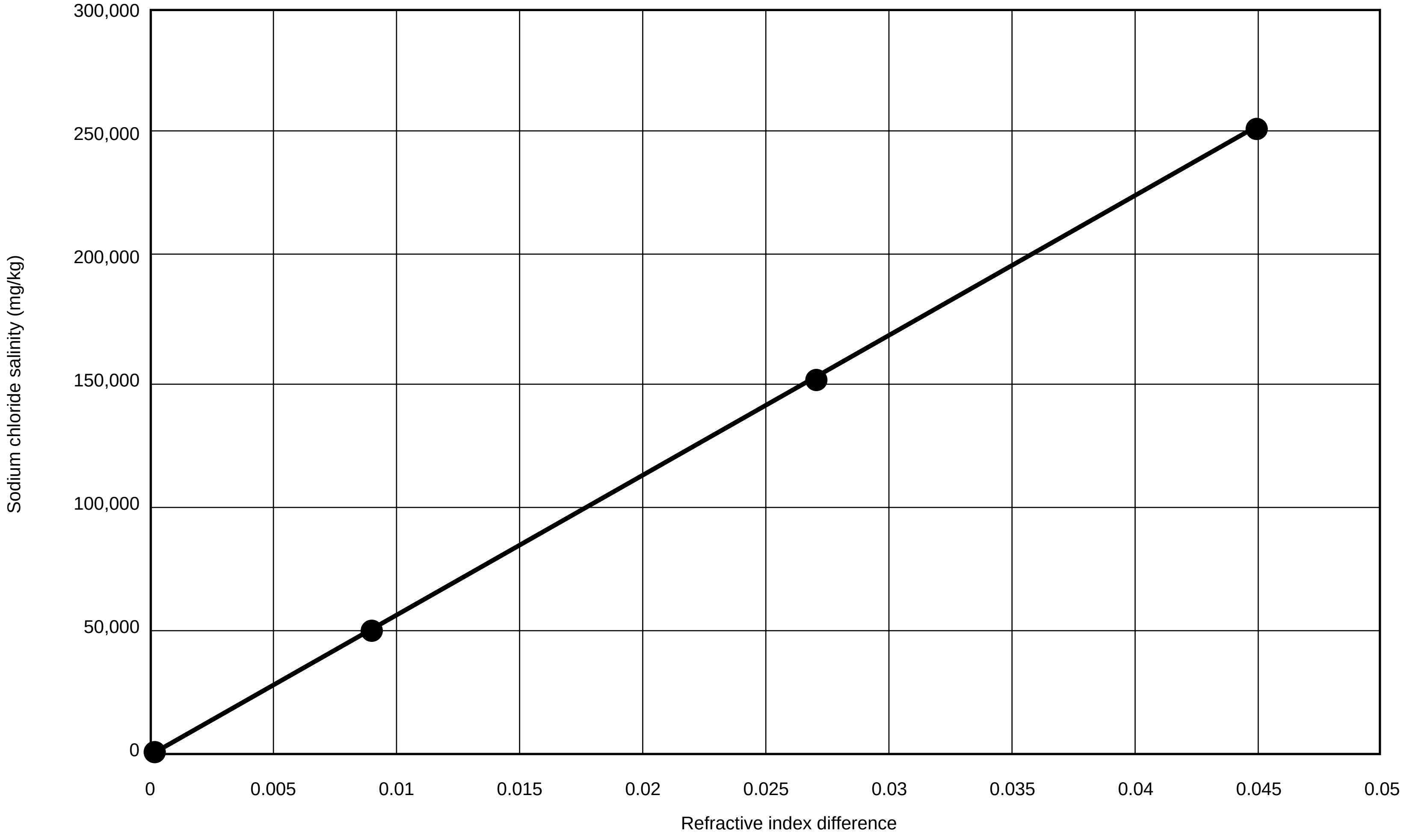


Figure 7-2—Refractive Index (RI) Difference Versus Salinity (RI of Solution—RI of Distilled Water)

7.7.3.2.6 Accuracy/Precision

See 7.7.3.1.6 for general comments. According to ASTM D4542, salinity determination using the refractometer should be accurate within ±300 mg/kg salinity.

7.7.3.3 Resistivity Measurement

The total ionic constituents of water may be estimated by measuring the resistivity. The resistivity varies in an inverse manner with the ionic concentration of sodium chloride and other salts over a wide range of concentrations. Standard graphs, illustrated by Figure 7-3, show the resistivity value for various salinities and temperatures of sodium chloride solutions. If the resistivity is known, then the salinity of the core water, expressed as parts per million of sodium chloride (mg/kg), can be determined from such a graph using a measured resistivity value.

7.7.3.3.1 Apparatus

Apparatus for this method includes:

a. Resistivity cell.
b. Resistivity meter.
c. Assorted glassware.

7.7.3.3.2 Procedure

The resistivity cell should be calibrated with known concentrations of sodium chloride solutions, resulting in a graph similar to that shown in Figure 7-3. A portion of the solution obtained by leaching the core is placed in the resistivity cell. The resistivity is measured on a suitable meter and calculated in ohmmeters. With the use of a standard graph (Figure 7-3) and suitable calculations, the resistivity value is converted to a salinity value for the core water. Additional details on apparatus and procedures may be found in ASTM D1125-82, *Method of Test for Electrical Conductivity for Water.*[24,25]

7.7.3.3.3 Precautions

Precautions for this method include:

a. Resistivity meter and cell must be calibrated.
b. Resistivity value must be corrected to a standard temperature.
c. Be certain that electrodes of the resistivity cell are clean before making a measurement.
d. Calibrate electrode with known standard salt solution over the entire range of use to determine/check cell constant as described by Worthington, et al.[25]
e. Hydrogen sulfide may cause some electrodes to change calibration.

7.7.3.3.4 Calculations

Calculations for this method are as follows:

a. The salinity of the core water expelled from the core sample can be calculated directly. The resistivity of the salt solution can be converted directly to concentration milligrams of salt per kg of solution, using a conversion chart of the type illustrated in Figure 7-3.[20]

b. When core water cannot be expelled and the salts are water extracted, the salinity of the originally present core water must be back calculated. The salinity from the resistivity measurement method is converted to total milligrams of sodium chloride leached from the sample. This number represents the total salt from the core water of the sample. The total salt in milligrams and the weight of the volume of core water obtained by saturation tests are used to calculate the concentration of salt in the core water, and this value is expressed in parts per million of sodium chloride. The salinity (mg/kg) of the core water is obtained by following the calculation outlined in 7.7.3.1.3b.

7.7.3.3.5 Advantages

Advantages of this method include:

a. Rapid determination.
b. The resistivity data relate directly to electric log measurements.
c. The total equivalent sodium chloride concentration is determined.

7.7.3.3.6 Limitations

All ions present in the water are calculated as sodium chloride.

7.7.3.3.7 Accuracy/Precision

See 7.7.3.1.6 for general comments and to ASTM D1125[24] for resistivity specific information.

7.7.3.4 Determination of Cations

The direct analysis for sodium, potassium, calcium, and magnesium allow for the estimation of core water salinity and may provide a method to determine if the core water is free of coring fluid. Other cations are present in formation waters but typically account for less than 5 percent of the salinity. Core water salinity can also be estimated from the concentrations of these four cations.

The concentration of these cations can be measured rapidly, simultaneously, and accurately on an inductively coupled plasma atomic emission spectrometer (ICPES). These cations can also by determined individually with an atomic absorption spectrometer (AAS).

Some modern drilling/coring fluids contain high concentrations of potassium. In most formations waters, the ratio of sodium to potassium is much greater than one. Thus for

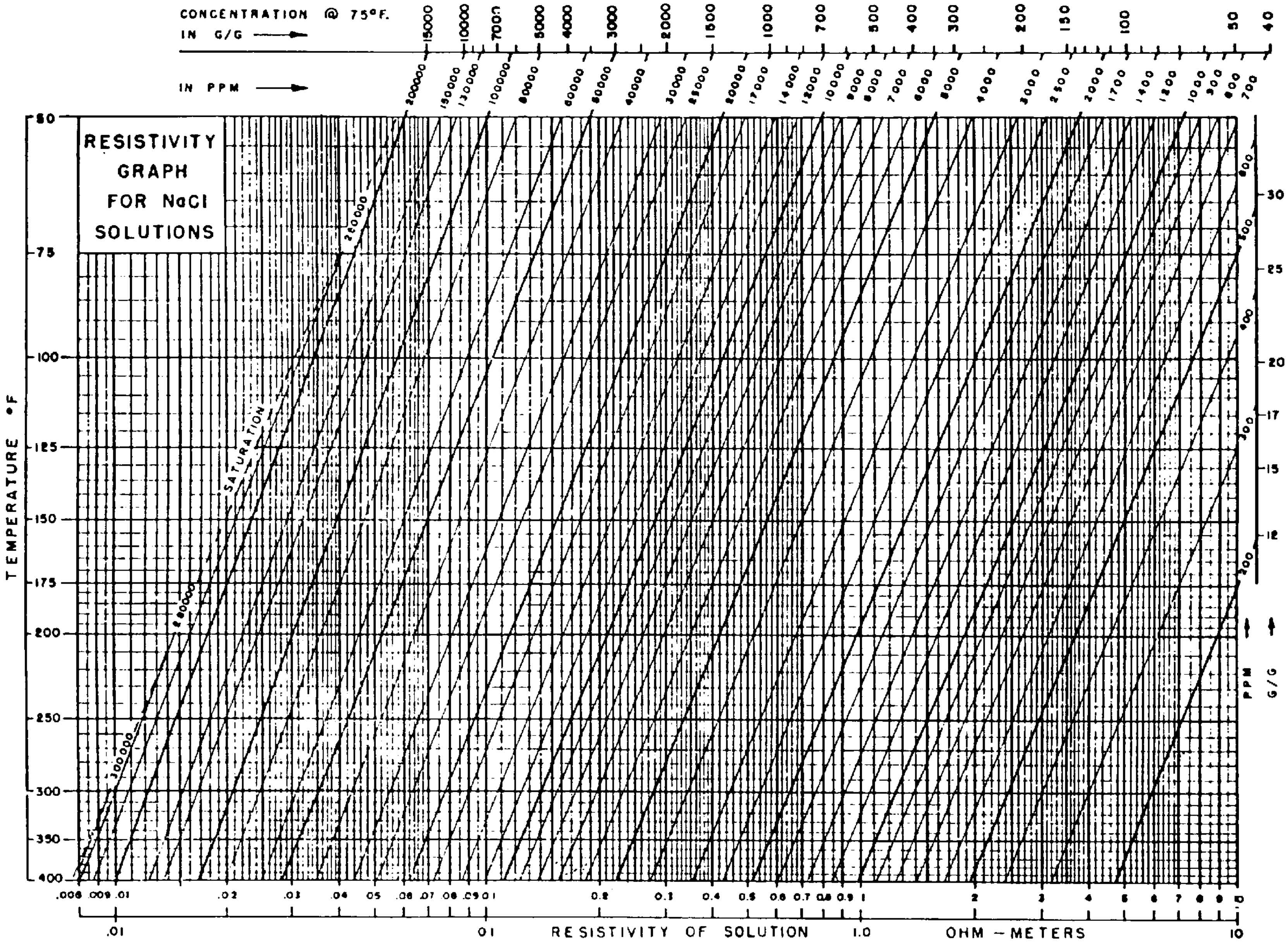


Figure 7-3—Conversion Chart for Salinity Determination Resistivity Versus Temperature and Salinity

potassium-based coring fluids, invasion can be estimated based on the ratio of potassium to sodium in the core water.

7.7.3.4.1 Apparatus and Procedure

The apparatus and procedure for cation determination using AAS are described in detail in ASTM D4191[26] for sodium, ASTM D4192[27] for potassium, and ASTM E508[28] for calcium. The apparatus and procedure for cation determination using ICPES are described in detail in ASTM D1976.[29]

7.7.3.4.2 Precautions

Precautions for this method include:

a. For salinity calculations, the anion is assumed to be chloride even though there are commonly significant sulfate and bicarbonate ions and smaller amounts of other ions.
b. Coring fluid invasion estimation using Na/K ratio requires a knowledge of the interaction of potassium with the sodium bentonite clay in the coring fluid and with the clays in the formation rock.
c. An understanding of local evaporites and their effects on sample preparation is required.
d. The analysis should be performed by personnel trained in these techniques and in a laboratory with the equipment permanently installed.

7.7.3.4.3 Calculations

The methods described in 7.7.3.1.3 for calculating sodium chloride concentration in the undiluted brine and the core leachate can be used to calculate the concentration of cations by replacing the molecular weight of sodium chloride with the atomic weight of the cation.

7.7.3.4.4 Advantages

Advantages of this method include:

a. Both ICPES and AAS are accurate analytical methods.
b. Coring fluid invasion estimates can be made with these data.
c. Cation analysis can provide a more complete picture of the brine salinity instead of assuming that sodium is the only cation present when total salinity is based only on chloride analysis.

7.7.3.4.5 Limitations

The lack of availability and high cost of ICPES and AAS analyses are a limitation.

7.7.3.4.6 Accuracy/Precision

Refer to 7.7.3.1 for general comments and to ASTM D4191, D4192, E508, and D1976 for more specifics.[26-29] In general, AAS and ICPES should be able to determine the concentration of these cations with an accuracy and precision of 1% to 2%.

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b. Brown, A.A., Berry IV, W.R., Coates, G.R., Fertl, W.H., Hoyer, W.A., Patchett, J.G., and Ransom, R.C.; W.H. Lang, Jr., chairman, "SPWLA Reprint Volume Shaly Sand," Society of Professional Well Log Analysts, July 1982, Section V, *Cation Exchange Models*.

c. Hill, H.J., Shirley, O.J., and Klein, G.E., edited by Waxman, M.H., and Thomas, E.C., "Bound Water in Shaly Sands—Its Relation to Qv and Other Formation Properties," *The Log Analyst*, Volume XX, No. 3, 1979.

d. API RP 45, *Recommended Practice for Analysis of Oil-Field Waters*, American Petroleum Institute, July 1981.

SECTION 8—REPORTING

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8 Reporting

8.1 INTRODUCTION

An essential part of a core analysis program is the data documentation phase. Typical data reporting formats are tabular, graphical, and/or digital. These constitute the permanent record(s) of the testing program. Such reports are furnished to the user and are filed by the testing laboratory in accordance with sound archiving procedures.

Tables 8-1 and 8-2 are developed for data documentation at the wellsite and in the laboratory. Commentary which may serve to aid in the understanding and/or the interpretation of the data should be furnished by the way of attachments and/or footnotes. Deviations from accepted standard practices must be noted. Such deviations may include: (a) unusual coring conditions, (b) alterations to the planned coring fluid design, (c) necessary changes in planned on-site or laboratory core handling/preservation methods, and (d) unusual laboratory procedures, etc. The report should contain sufficient wording so as to leave no question or doubt as to the testing methods that were employed.

8.2 TABULAR REPORT

A tabular report should include all of the data, positively identified and tabulated, in some convenient form. Identification should include such items as listed in Tables 8-1 and 8-2. Porosity values should be reported in percent to 0.1 whereas the grain density values should be reported to 0.01 g/cm^3 accuracy. The permeability values should be reported with no less than three significant figures. The specific presentation of data may be determined between the user and the analysts. The suggested numbers of significant figures to be reported are as follows:

Porosity values to	3	(e.g. 0.251 fraction or 25.1%)
Grain density values to	3	(e.g. 2.65 g/cm^3)
Saturation values to	3	(e.g. 50.5% pore volume)
Pore volume values to	4	(e.g. 21.85 cm^3)
Permeability values to	3	(e.g. 2.55 x 10^{+3} md)

8.3 GRAPHICAL REPORT

Graphical presentations are often included to provide the user with a pictorial overview of various core data. Through the continued advances in computer graphic software a large number of pictorial formats are readily available including crossplots, histograms, and core data profiles (logs). The selection of any specific format is left to the discretion of each individual core analyst; however, two graphs have been widely accepted and are recommended for inclusion in every basic core analysis report: a *Permeability vs. Porosity* plot and a *Core Data vs. Depth (sub-surface or sub-sea)* plot.

8.3.1 Permeability vs. Porosity Crossplots

A plot of permeability vs. porosity, with porosity plotted on a linear x-axis and permeability on a logarithmic y-axis, has been included in most conventional core analysis (percussion sidewall data reports rarely contain these crossplots) reports (see Figure 8-1). Also, some best-fit lines are "estimated" while others are derived by curve-fitting procedures. The latter is not always the better approach, depending on the amount of data and the presence of out-liers or fliers that may or may not impact a mathematical approach. A standard labeling procedure should include the essential data identification as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot. While the selection of axis scaling is data dependent and may vary according to the intent of the analyst, a general recommendation is made to select an appropriate scale which will allow the data to fill the entire plot while maintaining easily interpolated axis divisions such as unit divisions of 2, 5, or 10.

8.3.2 Core Data Profiles

The core profile log is a multi-plot figure sometimes referred to as a "core analysis log" (see Figure 8-2). This multi-plot provides a pictorial overview of a variety of core data as a function of sample depth. Generally included on this plot is a gamma ray log, a log of permeability, porosity, reservoir fluids saturation, and possibly a log of bulk or grain density. The recommended standard for vertical scale is 5 inches per 100 feet of depth. The horizontal scale will vary with a log and should be individually selected to allow the data to span the entire plot. A standard labeling procedure should include the essential data identification, as adopted through the rest of the core report, as well as any assumptions, notations, or scaling unit parameters unique to each specific plot.

8.4 DIGITAL REPORT

Current and emerging technologies provide means for core analysis data to be recorded automatically in a digital format. Initial (raw) and calculated (final) data storage, as a result, is being done increasingly on computer systems. Current technology offers a wide choice of data gathering and storage methods. A standardized method of data reporting within the petroleum industry could reduce the cost and effort required to translate data between the various computer platforms.

The Digital Core Analyses Interchange Standard (DCIS) defines the logical data organization on any type of physical

Table 8-1—Core Analysis Wellsite Data

Prepared By: ______________________ Date: ☐M ☐M ☐D ☐D ☐Y ☐Y Core Number ______________________

Operator ______________________

Well Identification ______________________

API Number ______________________

County/State/Country ______________________

Persons Requesting Work ______________________

Report Results To ______________________

Alternate ______________________

Rig Contract ______________________

Coring Company______________________

Contract/Phone ______________________

Field Reservoir ______________________

☐ Vertical Well ☐ DeviatedWell ☐ Sidetrack

Legal Location/Block______________________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Phone ____________ Fax ____________

Core Analysis Company______________________

Contact/Phone ______________________

Address ______________________

____

Elevations: _______ Ground/Mean Sea Level/ Derrick Floor _______ Kelly Bushing ____

Address ______________________

Address ______________________

Address ______________________

Address ______________________

Coring/Drilling Fluid
Type/Contents ____________ Weight _______ ppg Funnel Viscosity _______ sec Water Loss ________ cm^3/30 min Chlorides ________ ppm pH ________ Tracer ____________

Type of Core
☐ Conventional ______________________
☐ Sponge
☐ Pressure Retained

Inner Barrel Type ______________________
☐ Oriented Barrel
☐ Other ______________________

Sidewall: ☐ Percussion or ☐ Mechanically Drilled Number Attempted _____ Number Recovered _____

Analysis Planned
☐ Plug Size or ☐ Full Diameter
Method
☐ Fluid Saturation ______________________
☐ Porosity ______________________
☐ Grain Density ______________________
☐ Permeability ______________________
☐ Surface Gamma Log ______________________
☐ Special Instructions ______________________

Transportation
☐ By Service Company ______________________
☐ Ground ______________________
☐ Air ______________________
☐ Other ______________________
Carrier ______________________
Date Shipped ______________________

Preservation
Method ☐ Plastic Laminate/Type ______________
☐ Freeze ☐ Dry Ice or ☐ Liquid Nitrogen
☐ Refrigerate _____ °F _____°C
☐ Core Inner Barrel
☐ Core Wrap and/or ☐ Dip-Type ☐ Resination
☐ Other

Exposure Time/Climate ______________________
Notes ______________________
____
____
____

Laboratory/Long Term Preservation ______________________

Special Instructions ______________________

Post Core Analysis Instructions/Core Material Distribution ______________________

Formation(s)/ Zones
____
Expected Core Point ______________
____
Expected Core Point ______________
____
Expected Core Point ______________

Depths (Drillers)
Interval Cut ______________________
Interval Recovered ______________________
Interval Cut ______________________
Interval Recovered ______________________
Interval Cut ______________________
Interval Recovered ______________________

Well Inclination ______________________
Total Core Recovered______________________
Length Cored ______________ ☐ ft ☐ m
Coring Time ______________________

Core Diameter ______________ ☐ In ☐ cm
Bit Type ______________________
Percent Recovery ______________________
Trip Time ______________________

Estimated Connate Water Salinity _____ ppm Chlorides or Estimated R_W _______ @ _______ ☐ °F ☐ °C

Estimated Production ☐ Oil ________ °API
☐ Condensate
☐ Dry Gas

Notes ______________________
____
____
____
____

Attach Coring Log and Core Description

Table 8-2—Basic Core Analysis Laboratory Data

General Information
Analysis Requested Date M M D D Y Y
API # ____________
File # ____________

Requested By: ____________

☐ Full Diameter or ☐ Plug : Size ________ ☐ In ☐ cm

Measurement	Method/Special Instructions
☐ Saturations (O,W,G)	____________
☐ Porosity	____________
☐ Permeability	____________
☐ Grain Density	____________
☐ Other	____________

Objective of the tests as agreed by parties involved ____________
____

Transportation and Inventory
Carrier ________ Date Shipped ________ Core Arrival Date ________
Core condition on arrival:
☐ Preserved ☐ Frozen ☐ Unpreserved ☐ Cleaned ☐ Other ____________
Correlation Depths: Driller's ______ Log ______ TVD ______ ☐ ft ☐ m
Allocation of the missing core dinterval: ☐ Bottom ☐ Middle ☐ Top ____________

Screening
☐ CT ☐ X-ray ☐ NMR ☐ Fluroscopy ☐ Other ____________

Sample Handling
Core Gramma (yes/no, type) ____________
Lab Preservation (prior to analyses) ____________
Sampling Method
Sample Sleeve ☐ Yes ☐ No, Type ____________
Nominal Sample Diameter/Length____________ ☐ In ☐ cm
Plugging/Trimming Fluid____________
Treatment prior to testing (flushing, evaluation, resaluration, etc.) ____________
____
Core Disposition (removed samples, storage location, preservation, etc.)____________
____
Lab Analyst ____________

Other Service
☐ Slab ☐ Resination ☐ Photo: ☐ BW ☐ Color ☐ U.V. ☐ Other ____________
Supplementary Tests ____________
____

Methods and Conditions

Cleaning:	Methods	Conditions
	☐ No Cleaning	Solvents ____________
	☐ Dean-Stark	Temperature ____________ ☐ °F ☐ °C
	☐ Soxhlet	Pressure ____________ ☐ psi ☐ kPa
	☐ CO2/Solvent	Time ____________
	☐ Flow Through	Volume and Rate________ ☐ cc ☐ cc/sec
	☐ Others	Others ____________
Drying:	Methods	Conditions
	☐ Convection oven	Temperature ____________ ☐ °F ☐ °C
	☐ Vacuum oven	Time ____________
	☐ Humidity oven	Relative Humidity % ____________
	☐ Others	

Porosity: Methods

Pore Volume	Grain Volume	Bulk Volume
☐ Boyle's Law	☐ Boyle's Law	☐ Caliper
☐ Saturation	☐ Archimedes	☐ Archimedes
☐ Summation-Of-Fluids	☐ BV-GV	☐ Mercury Displacement
☐ BV-GV	☐ Other	☐ GV+PV
☐ Other		☐ Other

Conditions/Fluids
Confining Stress (Magnitude and Type)____________ ☐ psi ☐ kPa
Gas ________ Liquid________
Pressure ________

Permeability:	Methods	Conditions
	☐ Steady State	Fluid Type ____________
	☐ Unsteady State	Confining Stress ____________ ☐ psi ☐ kPa
	☐ Probe	Sleeve Durometer ____________
	☐ Empirical	Pore Pressure ________Units ______
	☐ Not Measured	Klinkenberg: ☐ Measured ☐ Empirical ☐ No Correction
	☐ Others	Inertial Factor: ☐ Measured ☐ Empirical ☐ No Correction
Saturation:	Methods	Conditions
	☐ Distillation Extraction (DS)	Temperature ________ ☐ °F ☐ °C
	☐ High Temperature Retort	Fluids ________
	☐ Others	Water Density______ ☐ g/cm^3 ☐ kg/m^3
		Oil Density ______ ☐ g/cm^3 ☐ kg/m^3
	Correction for Salt:	☐ Corrected for Water Volume
		☐ Corrected for Oil Weight ☐ Not Corrected

Quality Assurance: (Page number in report where the information is provided) ________
____
Comments/Remarks: ____________
Data Anomalies: ____________

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

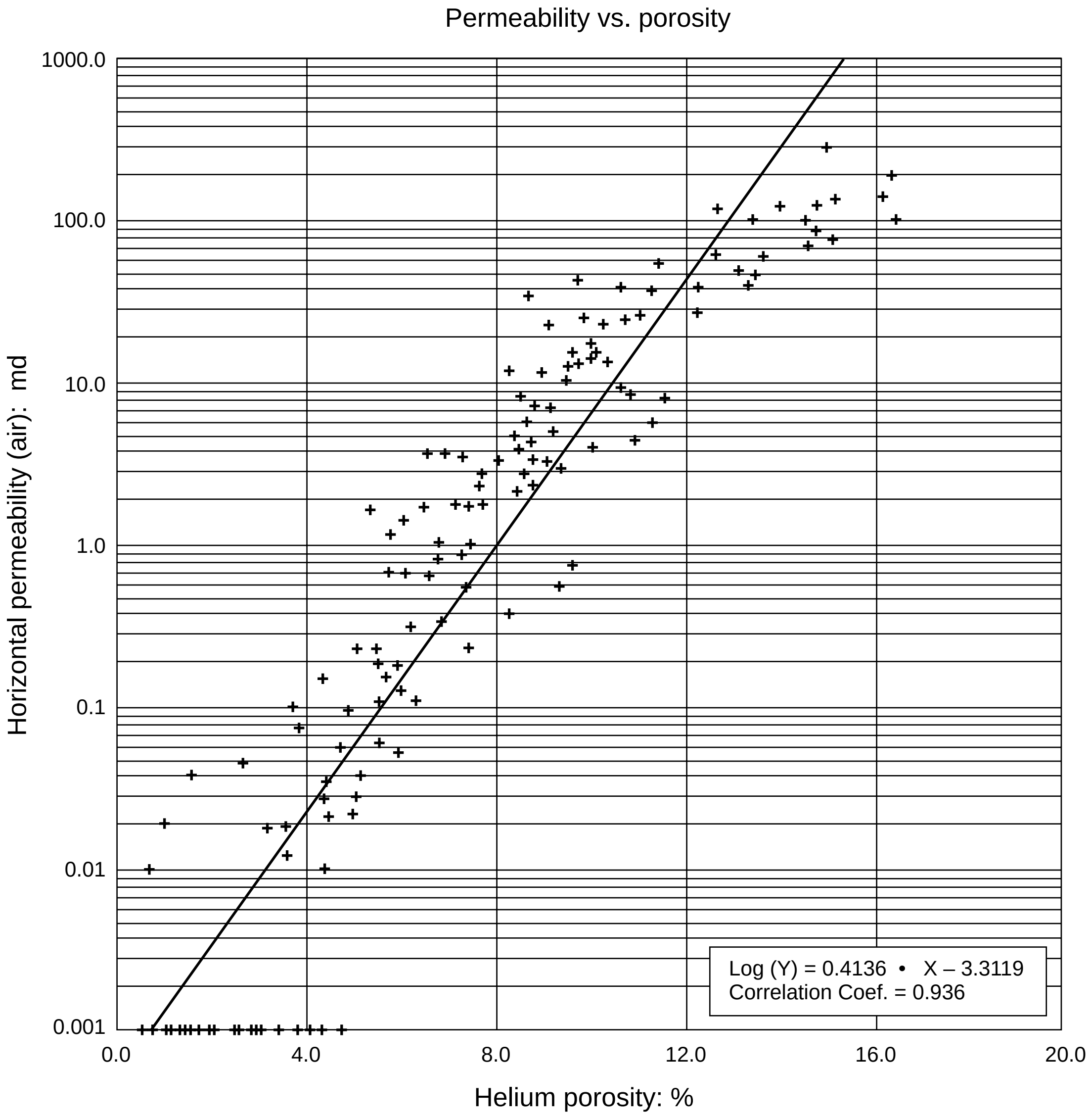

Figure 8-1—Permeability vs. Porosity

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block:		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

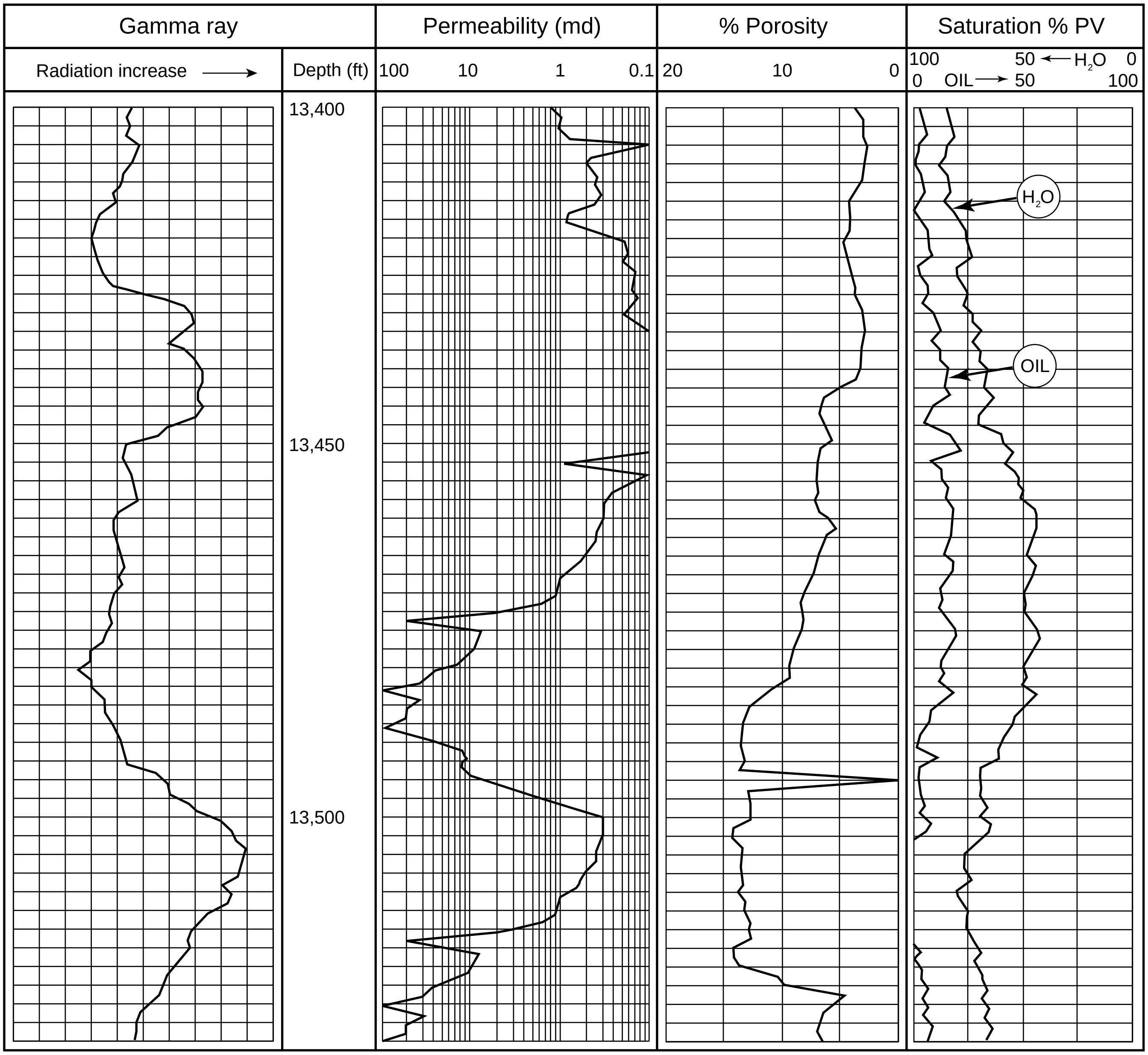

Figure 8-2—Core Data Profile

storage media (i.e., floppy disk, magnetic tape, optical disk, etc.). The physical format is the way in which stored data is located and organized on a physical medium. The specific way in which the logical and physical formats are bound depends on the medium and access mechanism and therefore are beyond the scope of this standard.

8.4.1 Digital Format

The DCIS format consists of the following elements:

a. Logical Record—A group of 8-bit bytes or data characters.
b. Logical File—A group of related Logical Records.

At the byte level, DCIS data are an ordered stream of 8-bit bytes, in which byte k precedes byte k+1. Data encryption is not allowed. No high-order bytes are allowed; all data should be represented in printable ASCII characters (ASCII 32 through ASCII 126). The only exception is ASCII 13 (carriage return) and ASCII 10 (line feed) at the end of each logical record. A combination carriage return and line feed signifies the end of the Logical Record. All records start at the byte location immediately following the carriage return-line feed combination of the previous record.

The layout of the logical file should be as follows (see example in Figure 8-3).

8.4.1.1 Logical File Header

This segment provides general information about the rest of the Logical File. Each record in this segment, as in other segments, is terminated by a carriage return, followed by a line feed character. Starting at logical location of byte 1 at record number 1, the following information should be present:

a. Record 1—Record Count: A positive integer, represented as ASCII characters, right justified, with no embedded commas, and with preceding blanks, if needed, to define the number of logical records including the record count in this logical file. This number includes records in the Logical File Header.
b. Record 2—Maximum Record Length: A non-negative integer represented as 3 ASCII characters, right justified, and with preceding blanks, if needed. A value of 0 indicates that the maximum length of a record is unknown. A positive value indicates that no record in the Logical File will exceed Maximum Record Length in size. A positive value need not correspond to the actual maximum record length; it needs only be an upper bound.

8.4.1.2 Logical File Body Header

The digital presentation of Basic Core Analysis reports should closely follow the format and layout of the hard copies. A maximum of 15 records is allowed in this section. Each record in this section is represented as ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the "Number of Records In Logical Body Header," which is represented by a two character, right justified, positive integer. The Logical File Body Header should consist of the following, as a minimum, in this order:

a. Number Of Records In Logical File Body Header (this is included in the count).
b. Company.
c. Well.
d. Field.
e. Formation.
f. State.
g. County.
h. Core Type.
i. Mud Type.
j. Elevation (KB).
k. Date Of Report.
l. Analyzed by.

The first characters in each record in this segment must define the record (e.g. Company, Well, Field, etc.). A colon, :, is used as a separator between the definition and the value.

8.4.1.3 Logical File Body

This segment contains the main body of the core analyses data. One record is devoted to each sample reported in the hard copy report. This segment consists of:

a. Column Header—This is a segment giving the layout of the information to follow in the next segment. Information in this segment will provide the definitions for each column of data. A maximum of 25 records is allowed in this section. Each record in this section is represented by ASCII characters, left justified, with trailing blanks (designated by the ASCII space character) allowed. The only exception to this is the first record in this segment, the "Number Of Records In Logical File Body Column Header," which is represented by a two character, right justified, positive integer. It should be remembered that the count of records, identified as the "Number of Records in Logical File Body Header" found within the Logical File Body Header includes the line listing of the column header record count. This segment should contain, as a minimum, the following:
   1. Number Of Records In Logical File Body Column Header (including this one in the count).
   2. Sample Number.
   3. Depth.
   4. Sample type (plug, full diameter, etc.).

Additional records in this segment must specify the layout of additional data columns (e.g. permeability, oil saturation, etc.). Units of measurement must be included (%PV, gram, cm^3, etc.).

b. Body—This segment contains the main body of core analysis data, the layout of which is defined by the preceding segment. Various data columns for each sample analyzed must be separated by at least two ASCII space characters. The layout should be the same as that for the hardcopy of the report.

8.5 QUALITY ASSURANCE

Quality assurance/quality control (QA/QC) is an integral component of the core analysis process. This component is the foundation of all laboratory techniques and management. QA/QC impacts all technical decisions, training of personnel, selection of personnel and analytical equipment, use of instruments and equipment, and adherence to defined standards and quality control methodologies. In addition, operational procedures, i.e., regular procedural audits, documentation control, and quality control verifications are also impacted. QA/QC should be considered in the planning phase, before actually cutting the core. Quality assurance is the examination of the overall process and procedures used to obtain the data. Cost restraints and the purpose or use of the data may govern the testing methods and thoroughness used in obtaining data. How well the data represent the in situ formation may be quite uncertain and its assessment should be left to the evaluating engineer and/or the end user.

Quality control is the process of evaluating a particular testing method or instrument. It is a procedure which results in definable error limits as determined from the current study, or in a statement of precision which the laboratory has established based on comparable measurements of standard samples.

Quality control data should be supplied with every direct or indirect measurement along with the measurement procedures to provide the beginning of quality assurance. Quality control is the responsibility of the laboratory; and quality assurance is the responsibility of both the laboratory and the end user.

Proprietary procedures need not be divulged in detail in the report.

A high standard of QA/QC depends upon commitment by analysts, supervisor, laboratory director, and QA manager to quality production in strict accordance to established protocols. The International Organization for Standardization and National Measurement Accreditation Service are two organizations that are dedicated to setting quality assurance standards.

29	Record Count							Logical File Header
225	Maximum Record Length							
16	No. of Records in Logical File Body Header							Logical File Body Header
Company:	Gusher Oil Company							
Well:	Gusher #1							
API #:	3507123474							
Section:	18							
Township:	T-42-S							
Range:	R-18-E							
Field:	Gusher							
Formation:	Huge							
State:	California							
County:	Los Angeles							
Core Type:	Conventional With plastic Liner							
Mud Type:	Water base							
Elevation (KB):	20 ft. (6.1 meters)							
Date of Report:	June 9, 1992							
Analyzed by:	M. M. H.							
9	No. of Records in Logical File Body Column Header							Logical File Column Header
Sample Number								
Depth (ft.)								
Sample type								
Permeability (md)								
Porosity (% BV)								
Oil Saturation (% PV)								
Water Saturation (% PV)								
Lithology								
1	1234	“Whole Core”,	12.3	35.5	30.0	42.0	SS: 1t brn, slt-med gr, p cmt	Body
2	1235	or “Plug”, or	10.3	30.2	29.0	50.8	SS: 1t brn, slt-med gr, p cmt	
3	1236	“Sidewall, etc.	0.3	8.5	21.3	62.3	SS: 1t brn, slt-med gr, p cmt	

Figure 8-3—Example of DCIS Logical File

8.6 QA/QC PROTOCOL

8.6.1 Step 1

a. Ensure that the Standard Operating Procedure (SOP) is compatible with sample type and supervisor's instructions.

b. Assume custody of all or portions of the samples.

c. Set up instrument/apparatus consistent with specified SOP.

d. Perform analysis (including calibration) according to specified SOP.

e. Derive data from analyses and ensure that data is within control limits.

f. Maintain all results, instrument control, calibration checks, and documentation in accordance with SOP documentation requirements.

g. Maintain all QA/QC data control charts.

8.6.2 Step 2

a. Review procedural requirement for sample type and ensure that correct SOP was followed.

b. Review all data generated and check calculations.

c. Review QA/QC criteria and ascertain that the data are within limits.

d. Evaluate results for reanalysis if data are not within QA/QC limits and note in report when acceptance limits have been exceeded.

e. Prepare a final report and submit it to the Laboratory Director.

8.6.3 Step 3

a. Review final report data and compare each new set of data with previously completed data for consistency.

b. Verify that the data are within acceptable QA/QC criteria limits.

c. Order reanalysis of sample when necessary and if possible.

d. Review final report for analytical quality, custodial documentation, and completeness. Confirm that all laboratory procedures and documentation satisfy in-house and client requirements.

8.6.4 Step 4

a. Perform completeness audits of randomly and non-randomly selected final reports. This audit includes document verification, data reduction, and report narrative evaluations.

b. Review final reports for analytical and documentary completeness, client or contractual requirements, and timeliness.

c. Report to senior management.

d. Perform biannual system audits with blind check samples and periodic performance evaluation studies.

Tabulated data in written reports could follow the format presented in Tables 8-3 through 8-8. Standard abbreviations for lithologic descriptions could follow the nomenclature presented in Tables 8-9 and 8-10. Table 8-11 presents some of the SI units, conversion factors, and equations that are often used by core analysts. Table 8-12 presents common nomenclature.

Table 8-3—Basic Core Plug Analysis Report*

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Interval represented, ft. or m	Gas Permeability[b], md k_h $k_{h\infty}$[c] k_v $k_{v\infty}$[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
______								
______								
______								

*This form can also be used for reporting core analysis of rotary sidewall plugs.

[a] The indicated depth is driller's depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).

[b] The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.

[c] Whether the correction for gas slippage or inertia is made (extrapolation or correlation) and the type of gas used for flow should be specified.

[d] Optional, intended for data QA/QC.

[e] See the standard abbreviation table.

Table 8-4—Basic Full Diameter Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm^3	Porosity[b] % BV	Grain Density g/cm^3	Saturation, % PV Oil Water Gas	Lithology[e]
______							
______							
______							

[a] In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).

[b] The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.

[c] Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.

[d] Optional, intended for data QA/QC.

[e] See the standard abbreviation table.

Table 8-5—Basic Sidewall Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Recovered length in. or cm	Depth[a] ft. or m	Permeability, md (empirical)	Porosity % BV	Pore Volume[b] cm^3	Saturation, % PV (retort) Oil Water	Saturation[c] % PV Oil Gas	Probable[d] Production	Critical Water[e] Saturation, % PV (empirical)	Gas[f] Detector Units	Lithology[g]
______										
______										
______										

[a] The indicated depth is the wireline depth to tenth of the foot or hundredth of meter (0.1 ft. or 0.01 m).

[b] Optional, intended for QA/QC.

[c] Optional.

[d] Optional, expected fluid to be produced.

[e] Optional, estimated reservoir water saturation above which one should expect water production (empirical value obtained from correlation).

[f] Presence of hydrocarbon gas measured by conductivity of hot wire element inserted into each sidewall jar or suitable container used to transport each core.

[g] See the standard abbreviation table.

Table 8-6—Basic Oil-Wet Sponge Core Analysis Report

Client Company:		Date:
API Number:		Other Information: Oil density used in calculations, ________ ☐g/cm³ ☐kg/m³
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[b] cm^3	Porosity % BV	Grain Density g/cm^3	Sponge Oil Saturation %PV[e]	Core Oil Saturation %PV	Total Oil Saturation %PV (Core + Sponge)	Water Saturation %PV	Lithology [f]
______										
______										
______										

[a] In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).

[b] The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.

[c] Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum horizontal permeability value, whereas k_{90} is the horizontal value 90° to k_{max}.

[d] Optional, intended for data QA/QC.

[e] Standard condition should be specified.

[f] See the standard abbreviation table.

Table 8-7—Basic Full Diameter Pressure-Retained Core Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_{max}[c] k_{90}[c] k_v[c]	Pore Volume[d] cm^3	Porosity[b] % PV	Grain Density g/cm^3	Saturation, % PV Pressure Depletion Oil Water	Saturation, % PV Total @ Stand. Cond.[e] Oil Water	Lithology[f]
______								
______								
______								

[a] In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of a meter (0.1 ft. or 0.01 m).

[b] The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.

[c] Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified (See Table 8-2). k_{max} is not the maximum permeability but the greater of two measured horizontal permeability values, whereas k_{90} is the horizontal value 90° to k_{max}.

[d] Optional, intended for data QA/QC.

[e] Standard condition should be specified.

[f] See the standard abbreviation table.

Table 8-8—Basic Core Fracture Analysis Report

Client Company:		Date:
API Number:		Other Information:
Well Identification:		
Legal Location/Block		
Formation:		
Interval Analyzed:		
County/State/Country:		
Field/Reservoir:		

Sample Number	Depth[a] ft. or m	Gas Permeability[b], md k_h, N-S[c]k_h, E-W[c] k_h, NW-SE[c] k_h, NE-SW[c]	Gas Permeability[b], md k_v[c]	Pore Volume[d] cm³	Porosity[b] % BV	Grain Density g/cm³	Saturation, % PV Oil Water	Lithology[e]
______								
______								
______								

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[a] In full diameter core analysis, the depth associated with the actual top and bottom of the sample should be specified. The indicated depth should be drillers depth to tenth of a foot or hundredth of meter (0.1 ft. or 0.01 m).

[b] The confining stress should be specified. See Table 8-2 for amount and type of stress used during measurements.

[c] Whether the correction for gas slippage or inertia is made and the type of gas used for flow should be specified.

[d] Optional, intended for QA/QC.

[e] See the standard abbreviation table.

Table 8-9—Standard Abbreviations for Lithologic Descriptions (Sorted alphabetically by term.)

about	abt
above	abv
absent	abs
abundant	abd
acicular	acic
after	aft
agglomerate	aglm
aggregate	agg
algae algal	alg
allochem	allo
alter (ed ing)	alt
altered altering	alt
amber	amb
ammonite	amm
amount	amt
and	&
angular	ang
anhedral	ahd
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
appears	aprs
approximate (ly)	aprox
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
as above	a.a
asphalt (ic)	asph
assemblage	assem
associated	assoc
at	@
authigenic	authg
average	av
band (ed)	bnd
Barite (ic)	bar
basalt (ic)	bas
basement	bm
become (ing)	bcm
bed (ded ing)	bdg
bentonite (ic)	bent
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
birdseye	bdeye
birefringence	bifg
bitumen (inous)	bit
black (ish)	blk blksh
blade (ed)	bld
block (y)	blky
blue (ish)	bl blsh
bored (ing)	bor
botryoid (al)	btry
bottom	btm
boudinage	boudg
boulder	bldr
boundstone	bdst
brachiopod	brac
brackish	brak
branching	brhg
break	brk
breccia (ted)	brec
bright	brt
brittle	brit
brown	brn
bryozoa	Bry
bubble	bubl
buff	bu
Bulbous	bulb
burrow (ed)	bur
calcarenite	clcar
calcareous	calc
calcilutite	clclt
calcirudite	clcrd
calcisiltite	clslt
calcisphere	clcsp
calcite filled fractures	ccf
calcite (ic)	CaCO3
caliche	cche
carbonate	CO3
carbonized	cb
cavern (ous)	cav
caving	cvg
cement (ed ing)	cmt
center centered	cntr
cephalopod	ceph
chalcedony (ic)	chal
chalk (y)	chk chky
charophyte	char
chert (y)	cht chty
chitin (ous)	chit
chitinozoa	chtz
chlorite (ic)	chlor
chocolate	choc
circulate (ion)	circ
clastic	clas
clay (ey)	cly
claystone	clyst
clean	cln
clear	clr
cleavage	clvg
cluster	clus
coal	coal

(continues)

Table 8-9—(Continued)

coarse (ly ness)	crs
coated grains	ctd gn
coated (ing)	ctd
cobble	cbl
color (ed)	col
common	com
compact	cpct
compare	cf
concentric	cncn
conchoidal	conch
concretion (ary)	conc
conglomerate (ic)	cgl
conodont	cono
considerable	cons
consolidated	consol
conspicuous	conspic
contact	ctc
contamination (ed)	contm
content	cont
contorted	cntrt
coquina (oid)	coq
coral coralline	cor corin
core	c
covered	cov
cream	crm
crenulated	cren
crevice	crev
crinkled	crnk
crinoid (al)	crin crinal
cross	x
crossbedded (ing)	xbd xbdg
crosslaminated	xlam
crossstratified	xstrat
crumpled	crpld
cryptocrystalline	crpxl
cryptograined	crpgr
crystal (line)	xl xln
cube (ic)	cub
cuttings	ctgs
dark (er)	dk dkr
dead	dd
debris	deb
decrease (ing)	decr
dendritic	dend
dense (er)	dns
depauperate	depau
description	descr
desiccation	dess
determine	dtrm
detrital (us)	dtrl
devitrified	devit
diabase	db
diagenesis (etic)	diagn
diameter	dia
difference	dif
disseminated	dism
distillate	dist
ditto	“
dolocast (tic)	dolc
dolomite (ic)	dol
dolomold (ic)	dolmd
dolostone	dolst
dominant (ly)	dom
drill stem test	DST
drilling	drlg
druse	dru
drusy	drsy
earthy	ea
east	E
echinoid	ech
elevation	elev
elliptical	elip
elongate	elg
embedded	embd
enlarged	enl
equant	eqnt
equivalent	equiv
euhedral	euhd
euxinic	eux
evaporite (itic)	evap
excellent	ex
expose (ed ure)	exp
extraclast (ic)	exclas
extremely	extr
extrusive rock, extrusive (ion)	extr
facet (ed)	fac
faint (ly)	fnt
fair	fr
fault (ed)	flt
fauna	fau
feet	ft
feldspar (athic)	fspr
fenestra (al)	fen
Ferromagnesian	Femag
ferruginous	Fe
fibrous	fibr
figured	fig
fine (ly)	f fnly
fissile	fis
fissures	fiss
flaggy	flg
flake (s y)	flk flks flky
flat	fl
flesh	fls
floating	fltg
flora	flo
fluorescence (ent)	flor
foliated	fol
foot	ft
foraminifera (al)	foram

(continues)

Table 8-9—(Continued)

formation	fm
fossil (iferous)	fos
fracture (d)	frac
fragment (al)	frag
framework	frmwk
frequent	freq
fresh	frs
friable	fri
fringe (ing)	frg
frosted quartz grains	F.Q.G.
frosted	fros
fucoid (al)	fuc
fusulinid	fus
gabbro	gab
gas	G
gastropod	gast
generally	gen
geopetal	gept
gilsonite	gil
glass (y)	glas
glauconite (itic)	glau
Globigerina (inal)	glob
gloss (y)	glos
gneiss (ic)	gns
good	gd
grade (s ing d)	grd
grain (s, ed)	gr
grainstone	grst
granite wash	grnt.w
granite (ic)	grnt
granule (ar)	grnl
grapestone	grapst
graptolite	grap
gravel	grv
gray, grey (ish)	gry
grysh, graywacke	gwke
greasy	gsy
green (ish)	gn gnsh
grit (ty)	gt
gypsum (iferous)	gyp
hackly	hky
halite (iferous)	hal
hard	hd
heavy	hvy
hematite (ic)	hem
heterogeneous	hetr
heterostegina	het
hexagonal	hex
high (ly)	hi
homogeneous	hom
horizontal	hztl
hornblend	hornbl
hydrocarbon	hydc
igneous rock, igneous	ig
impression	imp
in part	I.P.
inch	in
inclusion (ded)	incl
increasing	incr
indistinct	indst
indurated	ind
inoceramus	inoc
insoluble	insl
interbedded	intbd
intercalated	intercal
intercrystalline	intxl
interfingered	intfr
interfragmental	intfrag
intergranular	intgran
intergrown	intgn
interlaminated	intlam
interparticle	intpar
interpretation	intpt
intersticies (iitial)	intst
interval	intvl
intraclast (ic)	intclas
intraformational	intfm
intraparticle	intrapar
intrusive rock, intrusive	intr
invertebrate	invtb
iridescent	irid
iron	Fe
ironstone	Fest
irregular (ly)	irr
irridescent	irid
isopachous	iso
jasper (oid)	jasp
joint (s, ed, ing)	jt jts jtd
kaolin (itic)	kao
lacustrine	lac
lamina (tions, ated)	lam
large larger	lge
laterite (itic)	lat
lavender	lav
layer	lyr
leached	lchd
ledge	ldg
lens, lenticular	len lent
light (er)	lt
lignite (itic)	lig
limestone	ls
limonite (itic)	lim
limy	lmy
lithic	lit
lithographic	lithgr
lithology (ic)	lith
little	ltl
littoral	litt
local	loc
long	lg

(continues)

Table 8-9—(Continued)

loose	lse
lower	low
lumpy	lmpy
lustre	lustr
lutite	lut
macrofossil	macrofos
magnetite, magnetic	mag
manganese, manganiferous	mn
marble	mbl
marine	marn
marl (y)	mrl mrly
marlstone	mrlst
maroon	mar
massive	mass
material matter	mat
matrix	mtrx
maximum	max
median	mdn
medium	med
member	mbr
meniscus	men
metamorphic (osed)	meta metaph
metasomatic	msm
mica (ceous)	mic
micrite (ic)	micr
microcrystalline	micxl
microfossil (iferous)	microfos
micrograined	micgr
micromicaceous	micmica
microoolite	microol
micropore (osity)	micropor
microspar	microspr
microstylolite	microstyl
middle	mid
miliolid	milid
milky	mky
mineral (ized)	mnrl
minimum	min
minor	mnr
minute	mnut
moderate	mod
mold (ic)	mol
mollusc (a)	moll
mosaic	mos
mottled (ing)	mot
mud (dy)	md mdy
mudstone	mdst
muscovite	musc
nacreous	nac
no show	n/s
no	n
no sample	n.s.
no visible porosity	n.v.p.
nodule (s, ar)	nod
north	N
novaculite	novac
numerous	num
object	obj
occasional	occ
ochre	och
odor	od
oil source rock	OSR
oil	O
olive	olv
Olivine	olvn
oncolite (oidal)	onc
ooid (al)	oo
oolicast (ic)	ooc
oolite (itic)	ool
oomold (ic)	oomol
opaque	op
orange (ish)	orng
orbitolina	orbit
organic	org
orthoclase	orth
orthoquartzite	otz
ostracod	ostr
overgrowth	ovgth
oxidized	ox
oyster	oyst
packstone	pkst
paper (y)	pap
part (ly)	pt
particle	par
parting	ptg
parts per million	ppm
patch (y, es)	ptch
pearl (y)	prly
pebble (ly, s)	pbl
pelecypod	plcy
pellet (al, oids)	pel
pelletoid (al)	peld
pendular (ous)	pend
permeability (able)	perm K k
petroleum petroliferous	pet
Phenocrysts	phen
phlogopite	phlog
phosphate (atic)	phos
phreatic	phr
phyllite, phyllitic	phly
pink	pk
pinkish	pkish
pinpoint porosity	p.p.
pisoid (al)	piso
pisolite pisolitic	pisol
pitted	pit
plagioclase	plag
plant	plt
plastic	plas
platy	plty

(continues)

Table 8-9—(Continued)

polish polished	pol
pollen	poln
polygonal	poly
poor (ly)	p
porahyry	prphy
porcelaneous	porcel
porosity porous	por ϕ
possible (ly)	pos
predominant (ly)	pred
preserved (ation)	pres
pressure deformation	p d
primary	prim
prism (atic)	pris
probable (ly)	prob
production	prod
prominent (ly)	prom
pseudo	psdo
pseudo oolite (ic)	psool
pumicestone	pst
purple	purp
pyrite (itized itic)	pyr
pyrobitumen	pybit
pyroclastic	pyrcl
pyroxene	pyrxn
quartz (ose, ic)	qtz qtzs qtzt
radial (ate, ating)	rad
radiaxial	radax
range (ing)	rng
rare	rr
recemented	recem
recovery (ered)	rec
recrystal (lize, ed, ation)	rexl rexlzd rexlt
red (ish)	rd rdsh
reef (oid)	rf
regular	reg
remains, remnant	rmn
replaced (ment, ing)	rep
residue (ual)	res
resinous	rsns
rhomb (ic)	rhb
ripple	rpl
rock	rk
round (ed)	rnd
rounded frosted pitted	r.f.p.
rubble (bly)	rbl rbly
rudist	rud
rugose, ruga	rug
rugose coral rugosa	rugc
saccharoidal	sacc
salt and pepper	s & p
salt water	s.w.
salt (y)	sa
saltcast (ic)	sac
same as above	a.a.
sample	spl
sand (y)	sd sdy
sandstone	ss
saturation (ated)	sat
scales	sc
scaphopod	scaph
scarce	scs
scatter (ed)	scat
schist (ose)	sch
scolecodont	scol
secondary	sec
sediment (ary)	sed
selenite	sel
septate	sept
shadow	shad
shale (ly)	sh shy
shell	shl
shelter porosity	shlt por
show	shw
siderite (itic)	sid
sidewall core	S.W.C.
silica (iceous)	sil
silky	slky
silt (y)	slt slty
siltstone	sltst
similar	sim
size	sz
skeletal	skel
slabby	slb
slate (y)	sl
slickenside (d)	sks
slight (ly)	sli
small	sml
smooth	sm
soft	sft
solitary	sol
solution soluble	sln
somewhat	smwt
sort (ing, ed)	srt srtg srtd
south	S
spar (ry)	spr
sparry calcite	spc
sparse (ly)	sps spsly
speck (led)	spk spkld
sphalerite	sphal
spherule (itic, s)	sph
spicule (ar)	spic
splintery	splty
sponge	spg
spore	spo
spot (ted, y)	sp sptd spty
stain (ed, ing)	stn
stalactitic	stal
stippled	stip
strata (ified, tion)	strat
streak (ed)	strk

(continues)

Table 8-9—(Continued)

streaming	stmg
striae (ted)	stri
stringer	strg
stromatolite (itic)	stromlt
stromatoporoid	strom
structure	struc
stylolite (itic)	styl
sub	sb
subangular	sbang
subhedral	sbhed
sublithic	sblit
subrounded	sbrd
sucrosic	suc
sugary	sug
sulphur (ous)	S su
superficial oolite (ic)	spfool
surface	surf
syntaxial	syn
tabular (ate)	tab
tan	tn
tension	tns
terriginous	ter
texture (d)	tex
thick	thk
thin section	T.S.
thin	thn
thinbedded	t.b.
throughout	thru
tight (ly)	ti
top	tp
tough	tgh
trace	tr
translucent	trnsl
transparent	trnsp
trilobite	tril
tripoli (itic)	trip
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
unconformity	unconf
unconsolidated	uncons
underclay	uc
underlying	undly
unidentifiable	unident
uniform	uni
upper	u
vadose	vad
variation (able ed)	var
varicolored	vcol
variegated	vgt
varved	vrvd
vein (ing ed)	vn
veinlet	vnlet
vermillion	verm
vertebrate	vrtb
vertical	vert
very poor sample	vps
very	v
vesicular	ves
violet	vi
visible	vis
vitreous (ified)	vit
volatile	volat
volcanic rock volcanic	volc
vug (gy)	vug
wackestone	wkst
washed residue	W.R.
water	wtr
wavy	wvy
waxy	wxy
weak	wk
weather (ed)	wthr
wthrd well	wl
west	W
white	wh
with	w/
without	w/o
wood	wd
yellow (ish)	yel yelsh
zeolite	zeo
zircon	Zr
zone	z

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981.

Table 8-10—Standard Abbreviations for Lithologic Descriptions
(Sorted alphabetically by abbreviations.)

ditto	“
and	&
at	@
same as above	a.a.
as above	a.a.
abundant	abd
absent	abs
about	abt
above	abv
acicular	acic
after	aft
aggregate	agg
agglomerate	aglm
anhedral	ahd
algae, algal	alg
allochem	allo
alter (ed ing)	alt
altered, altering	alt
amber	amb
ammonite	amm
amount	amt
angular	ang
anhydrite (ic)	anhy
aphanitic	aph
apparent	apr
approximate (ly)	aprox
appears	aprs
aragonite	arag
arenaceous	aren
argillaceous	arg
argillite	argl
arkose (ic)	ark
asphalt (ic)	asph
assemblage	assem
associated	assoc
authigenic	authg
average	av
Barite (ic)	bar
basalt (ic)	bas
become (ing)	bcm
birdseye	bdeye
bed (ded ing)	bdg
boundstone	bdst
bentonite (ic)	bent
birefringence	bifg
bioclastic	biocl
bioherm (al)	bioh
biomicrite	biomi
biosparite	biosp
biostrom (al)	biost
biotite	biot
bioturbated	biotb
bitumen (inous)	bit
blue (ish)	bl blsh
blade (ed)	bld
boulder	bldr
black (ish)	blk blksh
block (y)	blky
basement	bm
band (ed)	bnd
bored (ing)	bor
boudinage	boudg
brachiopod	brac
brackish	brak
breccia (ted)	brec
branching	brhg
brittle	brit
break	brk
brown	brn
bright	brt
bryozoa	Bry
bottom	btm
botryoid (al)	btry
buff	bu
bubble	bubl
Bulbous	bulb
burrow (ed)	bur
core	c
calcite (ic)	$CaCO_3$
calcareous	calc
cavern (ous)	cav
carbonized	cb
cobble	cbl
calcite filled fractures	ccf
caliche	cche
cephalopod	ceph
compare	cf
conglomerate (ic)	cgl
chalcedony (ic)	chal
charophyte	char
chitin (ous)	chit
chalk (y)	chk chky
chlorite (ic)	chlor
chocolate	choc
chert (y)	cht chty
chitinozoa	chtz
circulate (ion)	circ
clastic	clas
calcarenite	clcar
calcilutite	clclt
calcirudite	clcrd
calcisphere	clcsp
clean	cln
clear	clr
calcisiltite	clslt
cluster	clus
cleavage	clvg
clay (ey)	cly

(continues)

Table 8-10—(Continued)

claystone	clyst
cement (ed ing)	cmt
concentric	cncn
center centered	cntr
contorted	cntrt
carbonate	CO3
coal	coal
color (ed)	col
common	com
concretion (ary)	conc
conchoidal	conch
conodont	cono
considerable	cons
consolidated	consol
conspicuous	conspic
content	cont
contamination (ed)	contm
coquina (oid)	coq
coral coralline	cor corin
covered	cov
compact	cpct
crenulated	cren
crevice	crev
crinoid (al)	crin
crinal cream	crm
crinkled	crnk
cryptograined	crpgr
crumpled	crpld
cryptocrystalline	crpxl
coarse (ly ness)	crs
contact	ctc
coated (ing)	ctd
coated grains	ctd gn
cuttings	ctgs
cube (ic)	cub
caving	cvg
diabase	db
dead	dd
debris	deb
decrease (ing)	decr
dendritic	dend
depauperate	depau
description	descr
desiccation	dess
devitrified	devit
diameter	dia
diagenesis (etic)	diagn
difference	dif
disseminated	dism
distillate	dist
dark (er)	dk dkr
dense (er)	dns
dolomite (ic)	dol
dolocast (tic)	dolc
dolomold (ic)	dolmd

dolostone	dolst
dominant (ly)	dom
drilling	drig
drusy	drsy
druse	dru
drill stem test	DST
detrital (us)	dtrl
determine	dtrm
east	E
earthy	ea
echinoid	ech
elevation	elev
elongate	elg
elliptical	elip
embedded	embd
enlarged	enl
equant	eqnt
equivalent	equiv
euhedral	euhd
euxinic	eux
evaporite (itic)	evap
excellent	ex
extraclast (ic)	exclas
expose (ed ure)	exp
extrusive rock extrusive (ion)	extr
extremely	extr
fine (ly)	f fnly
frosted quartz grains	F.Q.G.
facet (ed)	fac
fauna	fau
ferruginous	Fe
iron	Fe
Ferromagnesian	Femag
fenestra (al)	fen
ironstone	Fest
fibrous	fibr
figured	fig
fissile	fis
fissures	fiss
flat	fl
flaggy	flg
flake (s y)	flk flks flky
flora	flo
fluorescence (ent)	flor
flesh	fls
fault (ed)	flt
floating	fltg
formation	fm
faint (ly)	fnt
foliated	fol
foraminifera (al)	foram
fossil (iferous)	fos
fair	fr
fracture (d)	frac
fragment (al)	frag

(continues)

Table 8-10—(Continued)

frequent	freq
fringe (ing)	frg
friable	fri
framework	frmwk
frosted	fros
fresh	frs
feldspar (athic)	fspr
feet	ft
foot	ft
fucoid (al)	fuc
fusulinid	fus
gas	G
gabbro	gab
gastropod	gast
good	gd
generally	gen
geopetal	gept
gilsonite	gil
glass (y)	glas
glauconite (itic)	glau
Globigerina (inal)	glob
gloss (y)	glos
green (ish)	gn gnsh
gneiss (ic)	gns
grain (s, ed)	gr
graptolite	grap
grapestone	grapst
grade (s ing d)	grd
granule (ar)	grnl
granite (ic)	grnt
granite wash	grnt.w
grainstone	grst
gravel	grv
gray, grey (ish)	gry grysh
greasy	gsy
grit (ty)	gt
graywacke	gwke
gypsum (iferous)	gyp
halite (iferous)	hal
hard	hd
hematite (ic)	hem
heterostegina	het
heterogeneous	hetr
hexagonal	hex
high (ly)	hi
hackly	hky
homogeneous	hom
hornblend	hornbl
heavy	hvy
hydrocarbon	hydc
horizontal	hztl
in part	I.P.
igneous rock, igneous	ig
impression	imp
inch	in
inclusion (ded)	incl
increasing	incr
indurated	ind
indistinct	indst
inoceramus	inoc
insoluble	insl
interbedded	intbd
intraclast (ic)	intclas
intercalated	intercal
intraformational	intfm
interfingered	intfr
interfragmental	intfrag
intergrown	intgn
intergranular	intgran
interlaminated	intlam
interparticle	intpar
interpretation	intpt
intrusive rock intrusive	intr
intraparticle	intrapar
intersticies (iitial)	intst
interval	intvl
intercrystalline	intxl
invertebrate	invtb
iridescent	irid
irridescent	irid
irregular (ly)	irr
isopachous	iso
jasper (oid)	jasp
joint (s, ed, ing)	jt jts jtd
kaolin (itic)	kao
lacustrine	lac
lamina (tions, ated)	lam
laterite (itic)	lat
lavender	lav
leached	lchd
ledge	ldg
lens, lenticular	len lent
long	lg
large larger	lge
lignite (itic)	lig
limonite (itic)	lim
lithic	lit
lithology (ic)	lith
lithographic	lithgr
littoral	litt
lumpy	lmpy
limy	lmy
local	loc
lower	low
limestone	ls
loose	lse
light (er)	lt
little	ltl
lustre	lustr
lutite	lut

(continues)

Table 8-10—(Continued)

layer	lyr
macrofossil	macrofos
magnetite, magnetic	mag
maroon	mar
marine	marn
massive	mass
material matter	mat
maximum	max
marble	mbl
member	mbr
mud (dy)	md mdy
median	mdn
mudstone	mdst
medium	med
meniscus	men
metamorphic (osed)	meta metaph
mica (ceous)	mic
micrograined	micgr
micromicaceous	micmica
micrite (ic)	micr
microfossil (iferous)	microfos
microoolite	microol
micropore (osity)	micropor
microspar	microspr
microstylolite	microstyl
microcrystalline	micxl
middle	mid
miliolid	milid
minimum	min
milky	mky
manganese, manganiferous	mn
minor	mnr
mineral (ized)	mnrl
minute	mnut
moderate	mod
mold (ic)	mol
mollusc (a)	moll
mosaic	mos
mottled (ing)	mot
marl (y)	mrl mrly
marlstone	mrlst
metasomatic	msm
matrix	mtrx
muscovite	musc
no show	n/s
no	n
north	N
no sample	n.s.
no visible porosity	n.v.p.
nacreous	nac
nodule (s, ar)	nod
novaculite	novac
numerous	num
oil	O
object	obj

occasional	occ
ochre	och
odor	od
olive	olv
Olivine	olvn
oncolite (oidal)	onc
ooid (al)	oo
oolicast (ic)	ooc
oolite (itic)	ool
oomold (ic)	oomol
opaque	op
orbitolina	orbit
organic	org
orange (ish)	orng
orthoclase	orth
oil source rock	OSR
ostracod	ostr
orthoquartzite	otz
overgrowth	ovgth
oxidized	ox
oyster	oyst
poor (ly)	p
pressure deformation	p d
pinpoint porosity	p.p.
paper (y)	pap
particle	par
pebble (ly, s)	pbl
pellet (al, oids)	pel
pelletoid (al)	peld
pendular (ous)	pend
permeability (able)	perm K k
petroleum, petroliferous	pet
Phenocrysts	phen
phlogopite	phlog
phyllite, phyllitic	phly
phosphate (atic)	phos
phreatic	phr
pisoid (al)	piso
pisolite, pisolitic	pisol
pitted	pit
pink	pk
pinkish	pkish
packstone	pkst
plagioclase	plag
plastic	plas
pelecypod	plcy
plant	plt
platy	plty
polish, polished	pol
pollen	poln
polygonal	poly
porosity, porous	por ϕ
porcelaneous	porcel
possible (ly)	pos
parts per million	ppm

(continues)

Table 8-10—(Continued)

predominant (ly)	pred
preserved (ation)	pres
primary	prim
prism (atic)	pris
pearl (y)	prly
probable (ly)	prob
production	prod
prominent (ly)	prom
porahyry	prphy
pseudo	psdo
pseudo oolite (ic)	psool
pumicestone	pst
part (ly)	pt
patch (y, es)	ptch
parting	ptg
purple	purp
pyrobitumen	pybit
pyrite (itized itic)	pyr
pyroclastic	pyrcl
pyroxene	pyrxn
quartz (ose, ic)	qtz qtzs qtzt
rounded frosted pitted	r.f.p.
radial (ate, ating)	rad
radiaxial	radax
rubble (bly)	rbl rbly
red (ish)	rd rdsh
recovery (ered)	rec
recemented	recem
regular	reg
replaced (ment, ing)	rep
residue (ual)	res
recrystal (lize, ed, ation)	rexl rexlzd rexlt
reef (oid)	rf
rhomb (ic)	rhb
rock	rk
remains remnant	rmn
round (ed)	rnd
range (ing)	rng
ripple	rpl
rare	rr
resinous	rsns
rudist	rud
rugose, ruga	rug
rugose coral, rugosa	rugc
salt and pepper	s & p
south	S
sulphur (ous)	S su
salt water	s.w.
sidewall core	S.W.C.
salt (y)	sa
saltcast (ic)	sac
saccharoidal	sacc
saturation (ated)	sat
sub	sb
subangular	sbang
subhedral	sbhed
sublithic	sblit
subrounded	sbrd
scales	sc
scaphopod	scaph
scatter (ed)	scat
schist (ose)	sch
scolecodont	scol
scarce	scs
sand (y)	sd sdy
secondary	sec
sediment (ary)	sed
selenite	sel
septate	sept
soft	sft
shale (ly)	sh shy
shadow	shad
shell	shl
shelter porosity	shlt por
show	shw
siderite (itic)	sid
silica (iceous)	sil
similar	sim
skeletal	skel
slickenside (d)	sks
slate (y)	sl
slabby	slb
slight (ly)	sli
silky	slky
solution soluble	sln
silt (y)	slt slty
siltstone	sltst
smooth	sm
small	sml
somewhat	smwt
solitary	sol
spot (ted, y)	sp sptd spty
sparry calcite	spc
superficial oolite (ic)	spfool
sponge	spg
spherule (itic, s)	sph
sphalerite	sphal
spicule (ar)	spic
speck (led)	spk spkld
sample	spl
splintery	splty
spore	spo
spar (ry)	spr
sparse (ly)	sps spsly
sort (ing, ed)	srt srtg srtd
sandstone	ss
stalactitic	stal
stippled	stip
streaming	stmg
stain (ed, ing)	stn

(continues)

Table 8-10—(Continued)

strata (ified, tion)	strat
stringer	strg
striae (ted)	strl
streak (ed)	strk
stromatoporoid	strom
stromatolite (itic)	stromlt
structure	struc
stylolite (itic)	styl
sucrosic	suc
sugary	sug
surface	surf
syntaxial	syn
size	sz
thinbedded	t.b.
thin section	T.S.
tabular (ate)	tab
terriginous	ter
texture (d)	tex
tough	tgh
thick	thk
thin	thn
throughout	thru
tight (ly)	ti
tan	tn
tension	tns
top	tp
trace	tr
trilobite	tril
tripoli (itic)	trip
translucent	trnsl
transparent	trnsp
tube (ular)	tub
tuff (aceous)	tuf
type (ical)	typ
upper	u
underclay	uc
unconformity	unconf
unconsolidated	uncons
underlying	undly
uniform	uni
unidentifiable	unident
very	v
vadose	vad
variation (able ed)	var
varicolored	vcol
vermillion	verm
vertical	vert
vesicular	ves
variegated	vgt
violet	vi
visible	vis
vitreous (ified)	vit
vein (ing ed)	vn
veinlet	vnlet
volatile	volat
volcanic rock volcanic	volc
very poor sample	vps
vertebrate	vrtb
varved	vrvd
vug (gy)	vug
with	w/
without	w/o
west	W
washed residue	W.R.
wood	wd
white	wh
weak	wk
wackestone	wkst
well	wl
weather (ed)	wthr wthrd
water	wtr
wavy	wvy
waxy	wxy
cross	x
crossbedded (ing)	xbd xbdg
crystal (line)	xl xln
crosslaminated	xlam
crossstratified	xstrat
yellow (ish)	yel yelsh
zone	z
zeolite	zeo
zircon	Zr

Note: Most of the terms and abbreviations are extracted from *Sample Examination Manual*, R. G. Swanson, AAPG, 1981
Units and Conversions.

Table 8-11—Units and Conversions

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
t = time	d	s	8.6400	E+04
	s	d	1.1574	E-05
L = length	ft	m	3.048	E-01
	in	mm	2.54	E+01
A = area	sq ft	m^2	9.290	E-02
	sq in	mm^2	6.4516	E=02
V = volume, capacity	liter	dm^3	1.0	
	ft^3	m^3	2.831685	E-02
m = mass	lbm	kg	4.535942	E-01
	kg	g	1.0	E+03
	g	kg	1.0	E-03
T = temperature	˚F	˚C	(˚F - 32)/1.8	
	˚C	˚C	1.0	
	˚R	K	5/9	
	K	K	1.0	
P = pressure	atm (760 mm Hg at 0˚C or	MPa	1.01325	E-01
	14.696 lbf/in^2)	kPa	1.01325	E+02
	bar	MPa	1.0	E-01
	bar	kPa	1.0	E+02
	lbf/in^2 (psi)	MPa	6.894757	E-03
	lbf/in^2 mm Hg (0˚C)	kPa	6.894757	
	torr	kPa	1.333224	E-01
	dyne/cm^2	Pa	1.0	E-01
q = flow rate	ft^3/D	m^3/d	2.831685	E-02
	U.S. gal/min (liquids)	dm^3/s	6.309020	E-02
	ft^3/D	cm^3/s	3.277413	E-01
u = volumetric velocity (flux, or superficial)	ft/D	m/d	3.048	E-01
	ft/D	cm/d	3.048	E+01
	ft/D	mm/d	3.48	E+02
	ft/s	m/s	3.048	E-01
ρgas = Density (gases)	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/m^3	1.601846	E+04
ρw, ρo = Density	lbm/U.S. gal (liquids)	kg/m^3	1.198264	E+02
	lbm/U.S. gal (liquids)	g/cm^3	1.198264	E-01
	lbm/ft^3	kg/m^3	1.601846	E+01
	lbm/ft^3	g/cm^3	1.601846	E-02
	g/cm^3	kg/m^3	1.0	E+03
	g/cm^3	kg/dm^3	1.0	
	˚API	specific gravity	141.5/(131.5+˚API)	
ρma = density (solids)	lbm/ft^3	kg/m^3	1.601846	E+01
ν = viscosity (Kinematic)	cm^2/s	mm^2/s	1.0	E+02
	ft^2/hr	mm^2/s	2.58064	E+01
	cST mm^2/s	1.0		
μ = viscosity (dynamic)	dyne-s/cm^2	Pas	1.0	E-01
	cP	Pas	1.0	E-03
	lbm/(ft-hr)	Pas	4.133789	E-04

Table 8-11—Units and Conversions (Continued)

Quantity	Customary unit	Metric unit SPE preferred	Conversion factor multiply customary unit by factor to get metric unit	
	cP	(Ns)/m^{2}	1.0	E-03
σs = surface tension	dyne/cm	m(milli)N/m	1.0	
γg = interfacial tension (IFT)	dyne/cm	m(milli)N/m	1.0	
k = absolute/specific permeability	darcy (d)	µm^{2}	9.869233	E-01
	millidarcy (md)	µm^{2}	9.869233	E-04
	microdarcy (µmd)	µm^{2}	9.869233	E-07
ω = angular velocity	rpm	radian/s	1.047198	E-01

Table 8-12—Nomenclature*

k_a = air permeability, md

k_h = horizontal permeability, md

k_v = vertical permeability, md

k_∞ = equivalent liquid permeability (Klinkenberg corrected), md

k_{90} = permeability 90 degrees from horizontal permeability (k_{max}), md

k_{max} = higher of the k_h measured values in two directions 90 degrees apart

°C = degree Centigrade

°F = degree Fahrenheit

°R = degree Rankine

°K = degree Kelvin

cm^3 = cubic centimeter

dm^3 = cubic decimeter

ft = feet

ft^2 = square feet

in = inch

m = meter

m^2 = square meter

mm^2 = square micrometer = square micron

μm^2 = square micrometer

Pa = pascal

kPa = kilo pascal

kg = kilogram

g = gram

lbm = pound mass

lbf = pound force

lbm/ft^3 = pound/ft^3

kg/m^3 = kilogram/cubic meter

g/m^3 = gram/cubic meter

g/cm^3 = g/ml = gram/cubic centimeter = gram/milliliter

lbm/US = pound/US gallon

cST = centistokes

cP = centipoise

d = day

s = second

S_g = gas saturation, percent of pore volume

S_o = oil saturation, percent of pore volume

S_w = water saturation, percent of pore volume

ϕ = porosity, percent bulk volume

ϕ_e = effective porosity, percent of bulk volume

ϕ_t = total porosity, percent of bulk volume

PV = pore volume

BV = bulk volume

GV = grain volume

V_g = gas volume

V_o = oil volume

V_w = water volume

ρg = grain density

ρb = bulk density

*The SI Metric System of Units and SPE Metric Standard, SPE, Dallas, 1984.

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