API RP 550

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MANUAL

ON

INSTALLATION OF REFINERY INSTRUMENTS AND CONTROL SYSTEMS

PART II—PROCESS STREAM ANALYZERS

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THIRD EDITION
1977

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FOREWORD

This manual is based on the accumulated knowledge and experience of engineers in the petroleum industry. Its purpose is to aid in the installation of the more generally used measuring and control instruments and related accessories in order to achieve safe, continuous, accurate, and efficient operation with minimum maintenance. Although the information contained herein has been prepared primarily for petroleum refineries, much of it is applicable without change in chemical plants, gasoline plants, and similar installations.

This third edition of Part II of the manual, represents the latest suggested or generally used practices in the installation of all the devices covered in the second edition plus additional information based on revisions suggested by many individuals and several organizations. (The first edition of the manual was issued in 1960, the second edition in 1965.)

Users of this manual are reminded that in the rapidly advancing field of instrumentation no publication of this type can be complete, nor can any written document be substituted for qualified engineering analysis.

Certain instruments are not covered herein because of their very specialized nature and limited use. When one of these devices (or classes of devices) gains general usage and installation reaches a fair degree of standardization, this manual will be revised to incorporate such additional information.

Suggested revisions are invited and should be submitted to the director of the Refining Department, American Petroleum Institute, 2101 L Street, N.W., Washington, D.C. 20037.

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INTRODUCTION

Accepted methods for the permanent or semipermanent installation of analyzers in refining units are presented in this part of the manual. Analyzers which are used to monitor the characteristics of process streams either continuously or on a fixed-time cycle are described. The methods outlined herein are for use in processing units rather than in laboratories and are for permanent rather than temporary or portable setups. Selection, application, and economic justification of stream analyzers are outside the scope of this manual. General practices common to analyzer installations are discussed. More detailed recommendations are made for gas chromatographs; viscometers; distillation, flash, and vapor pressure instruments; moisture analyzers; oxygen analyzers; pH instruments; and densitometers.

Although the purpose of this manual is to cover widely used types of equipment, the practices described, also, will assist in the installation of less frequently used instruments. Part II primarily deals with sampling systems and the analytical portion of analyzers and other basic parts. For information pertaining to the installation of control, signal transmitting, or recording equipment, refer to Part I of the manual; see, especially, Sections 5, 7, and 12.

Before the preparation of this edition of Part II, careful consideration was given to the extent of the revisions necessary to update the manual. It was decided to approach the revision in two phases. The first phase would maintain the format of the 1965 edition and would update it to reflect the ideas and techniques of the past 10 years. This would provide the reader with an interim document to serve his needs until the second phase of the Part II rewrite was finished. The second phase, now underway, will consider publishing each section of Part II individually as has been done with Part I. In addition, certain sections will be reorganized and new sections added.

It is believed that this edition of Part II should adequately serve the user until the second phase rewrite is finished and published.

PART II—PROCESS STREAM ANALYZERS

SECTION 14—ANALYZERS

14.1 Scope

This section covers the application and installation of process stream analyzers. The intent of this section is to facilitate economical, trouble-free, and satisfactory installations. However, the recommended practices discussed cannot be considered as substitutes for expert skill and broad knowledge of the field.

14.2 General

14.2.1 SIGNIFICANCE OF ANALYZER INSTALLATION PRACTICES

Companies install process stream analyzers in order to obtain current information on stream composition or characteristics so that materials being processed can be used more advantageously in meeting product commitments. Properly applied and installed analyzers usually result in economical and trouble-free operation. Improperly applied and installed analyzers generally create maintenance and operating problems extending over long periods of time. Analyzer installations may be costly because considerable skill, experience, and painstaking attention to detail are required for successful operation.

14.2.2 STREAM ANALYZERS USED

Types of stream analyzers used in plant operations include those measuring—

1. Physical properties (normally used for product specifications):
    - Boiling points (including initial and end points)
    - Cloud point
    - Color, turbidity or opacity
    - Conductivity
    - Density or specific gravity
    - Flash point
    - Freeze point
    - Octane
    - Pour point
    - Vapor pressure
    - Viscosity
2. Chemical properties:
    - Acid strength
    - Catalytic or chemical activity
    - Combustibility
    - Dissolved oxygen
    - Electrolytic effects
    - Hydrogen ion concentration (pH)
    - Moisture content
    - Oxidation-reduction potential
    - Salt in crude
    - Specific ion
    - Total organic carbon
    - Total oxygen demand
3. Stream compositions by:
    - Chromatography
    - Dielectric constant
    - Magnetic effects
    - Chemiluminescence
    - Flame ionization
    - Mass spectrometry
    - Radiation (beta or gamma)
    - Refractive index
    - Spectroscopy (infrared, ultraviolet, x-ray)
    - Thermal conductivity

These groupings are arbitrary, nonexclusive, and in some cases overlapping. Most analyzers measure gas or liquid streams. Some measure properties of solids, such as catalyst adsorption, or the carbon or moisture content of catalysts.

14.2.3 INSTALLATION DESIGN CONSIDERATIONS

Many factors are involved in providing a complete analyzer system. The installation must—

1. Meet all safety requirements;
2. Make certain that the objective—an accurate, reliable analysis—can be attained;
3. Provide facilities for necessary testing, calibration, and isolation (valves, switches, and so forth) and for economical maintenance; and
4. Be designed for lowest cost commensurate with required performance.

14.2.4 PURPOSE OF THE INSTALLATION

It is important to state precisely why the analyzer is being installed so that all personnel (engineering, laboratory, installation, operating, and maintenance) are informed of the purpose and final objective of the installation. For example, essential information should include the following:

1. The stream(s) to be sampled (with the name and location of each stream clearly specified) and the data to be obtained;

2. The objective of the installation:
   a Operator information only
   b. Operator information and his corrective action
   c. Direct process control
   d. Study information
   e. Management information

14.2.5 PREASSEMBLED SYSTEMS

Preassembled analyzer systems are generally convenient and economically attractive. They may include one or more analyzers with sample conditioning and utility connections mounted on a frame or housed in a shelter, or both. There are several advantages in this type of construction:

1. Systems designed and factory constructed by a specialist supplier often may be superior to those normally produced on site.
2. Factory construction is independent of weather and labor conditions at site.
3 Systems can be fully tested under simulated operating conditions and major design, equipment, and construction faults corrected before delivery to site.

14.3 Safety Considerations

Analyzer installation safety problems may require considerable engineering study. Some of the electrical, ignition, and safety factors are discussed in 14.3.1 and 14.3.2.

14.3.1 ELECTRICAL AND IGNITION POSSIBILITIES

Petroleum hydrocarbon sample streams may be analyzed in enclosures which also contain analyzer electrical equipment and arcing devices. Failure or maloperation of the electrical equipment can provide a source of ignition. The *National Electrical Code* [1]* does not cover these situations thoroughly. Thus, adequate groundwork must be laid, before actual equipment installation is made, to assure that there will be no difficulty in meeting inspection requirements. From a practical viewpoint there are several ways to handle this problem The best selection depends upon many factors which include the following:

1. Whether the atmosphere at the analyzer location is likely to contain flammable material in explosive or ignitable portions with air, and whether this condition exists normally or only as the result of an abnormal condition (for example, equipment failure)
2. Whether or not it will be necessary to open the enclosures with the units energized
3. Whether any portion of the electrical or analytical system is normally, or abnormally, apt to be at a temperature above 80 percent of the ignition temperature (in degrees Celsius) of flammable materials expected or likely to be present; or whether electrical sparks or other ignition sources may be present during operation of the analyzer
4. Whether it is practical to confine the ignition sources in explosion-resistant enclosures, hermetically seal them (as in hermetically sealed relays), or otherwise isolate them from potentially explosive or ignitable materials or atmospheres.

The installation designer should be familiar with the requirements of national and local codes applicable to the proposed location of the analyzer. Helpful guidance may be obtained from API RP 500A, *Classification of Areas for Electrical Installations in Petroleum Refineries* [2], ISA RP 12.1, *Electrical Instruments in Hazardous Atmospheres* [3], and ISA RP 12.2, *Intrinsically Safe and Non-Incendive Electrical Instruments* [4]. Suggestions for the use of electrical instruments in potentially hazardous atmospheres may be found in ISA S12.4, *Instrument Purging for Reduction of Hazardous Area Classification* [5]. References 6 and 7 contain much of the background developed by the Instrument Society of America upon which these practices are based; see also the appendix of Reference 8. A few electrical instruments, current- or energy-limited, may be intrinsically safe, that is, the portion of the electrically energized equipment and wiring which is in a potentially hazardous location is incapable of releasing sufficient energy under normal or abnormal conditions to cause ignition of the specific hazardous atmospheric mixture. Many thermocouple circuits and some capacitance analyzers are examples.

Some analyzers are fully enclosed in properly sealed explosionproof housings. Others have their electrical equipment isolated and so enclosed. Still others may be installed in metal housings located in a potentially hazardous area and protected from the external atmosphere by purging, sealing, or submergence. The installation designer must ensure against loss of purge medium and against overheating of the external case (such as could occur when a loose wire shorts to a ground).

Preventing or confining internal explosions may still be a problem. Inasmuch as the sample itself may produce a combustible mixture in the analyzer, it may be necessary to house the analyzer in an explosionproof case even though it is located in a nonhazardous area.

Another approach is to continuously purge the analyzer case with enough nonhazardous gas or "safe" air to sweep out flammable materials before they can build to unsafe concentrations Air should not be used unless it can be determined that there is no danger of producing an explosive mixture. Purge requirements vary ac-

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* See Reference list at end of section.

cording to the purge classifications for the installations. Purge classifications are defined in ISA S 12.4, from which excerpts from Section 3 are presented herein as an aid, as well as a reminder, for the installation designer.

> 3.3.1 Type Z Purging. Covers purging requirements adequate to reduce the classification of the area within an enclosure from Division 2 (normally non-hazardous) to non-hazardous.
>
> 3.3.2 Type Y Purging. Covers purging requirements adequate to reduce the classification of the area within an enclosure from Division 1 (hazardous) to Division 2 (normally non-hazardous).
>
> 3.3.3 Type X Purging. Covers purging requirements adequate to reduce the classification of the area within an enclosure from Division 1 (hazardous) to non-hazardous.[1]

Type X involves the greatest risk. The external atmosphere may be hazardous at any time and a source of ignition may simultaneously exist within the case. If the purging system fails, the equipment must immediately be de-energized to avoid a possible explosion.

When an analyzer can be a continuous source of atmospheric ignition, thought must be given to providing for its maintenance and protection against mechanical damage, case corrosion, inadvertent opening of cases, and so forth. Adequate interlocks should be provided along with warning signs designating safety precautions to be followed. For example, when planning the installation of a chromatograph employing peak-holding capacitors, provision should be made for discharging these capacitors before instrument enclosures are opened. Alternatively, these capacitors are often in the analyzer control chassis which is located in a general purpose area.

Sample lines leading to, as well as inside, the explosionproof case should be assembled with the highest quality material and fittings. Frequent checks should be made for leaks.

The electrical system should be installed with the highest grade components. Twisted and taped joints should be used only where it is impossible to use superior types of splices. Signal wires should not be spliced.

Many analyzer systems contain spark-generating devices such as relays. When it is not necessary to locate these units at or near the sample source, they may be mounted in hydrocarbon-free areas.

14.3.2 SAMPLE DISPOSAL

Safe disposal of sampled process material, which cannot be economically returned to the process system, is a prime consideration. Piping should be provided so that vapors can be vented to a safe location or liquids can be drained in a clean and orderly fashion to a safe point. Small amounts of light hydrocarbon vapors when properly discharged into the atmosphere will diffuse rapidly to less than the minimum explosive concentrations. Such venting systems may be preferable to discharging vapors into closed systems where back pressure may occur sporadically. Toxic vapors should not be vented directly to the atmosphere. All venting systems should conform to the limits established by regulatory groups.

14.4 Analyzer Location

Sampling problems may be minimized by locating the analyzer as close as practicable to the sample point. In some cases, it is essential to locate the analyzer right at the sample point with the minimum practicable sample line length. When monitoring a sulfur plant, for example, off-gas, sulfur dioxide (SO_2), and hydrogen sulfide (H_2S) may continue to react forming sulfur in the sample line, possibly causing erroneous readings or plugging of the line.

The analyzer components must be protected from (a) hot equipment, (b) severe ambient temperature changes, or other vagaries of the weather, (c) shock, (d) vibration, and (e) mechanical damage. Because many analyzers are sensitive to or damaged by vibration, such conditions should be avoided when selecting the analyzer location. This is not difficult in an existing plant. In a new plant, proximity to heavy reciprocating equipment and locations near highways, railroads, and processes where intermittent movement of solids produces earth tremors should be avoided. Shock mountings should be used where vibration is unavoidable.

Many analyzers require weekly maintenance, and in some instances daily maintenance is required. Good installation practices can save manhours and improve reliability.

The analysis cell location should be conveniently accessible, whether at grade or at some elevated point. Accessibility may be provided by allowing space for portable platforms or by installing permanent platforms. Avoid installations where a ladder would be necessary.

Even in the most benevolent climate, a roof over the instrument is justified to protect service personnel and equipment from thundershowers, process and water spills, falling objects, and sunlight. In some cases, shielding against radiant heat from surrounding equipment must be provided. Normally, housing requirements increase with climatic severity. In some installations, lighted, heated, and ventilated housings, preferably of the walk-in type, will be desirable. See RP 550, *Manual on Installation of Refinery Instruments and Control*

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[1] ISA S12.4, *Instrument Purging for Reduction of Hazardous Area Classification*, Instr. Soc. Am., Pittsburgh, Pa., p. 4, 1970.

Systems, Part I, Section 8—Seals, Purges, and Winterizing for information on weather protection The term "housing" as used in this section is in the same context as in Section 8.

Housings should be located to avoid explosion hazards. Due to their construction, many analyzers must be considered small explosive areas that are rendered nonhazardous by their enclosures. Frequently, troubleshooting and repair can be expedited by removing the bells or covers from the enclosures and observing operation of the internals with electric power turned on. Location in nonhazardous or Division 2 areas permits this procedure It is advisable, however, that all such houses, regardless of location, be equipped with prominent signs warning that safety checks should be made with a portable explosimeter both before and after opening the enclosure or that a combustible gas alarm be installed permanently within the house. In Division 1 areas this procedure is prohibited Therefore, the instrument must be moved to a safe location before repairs can be made. To transport it with necessary caution, the unit should be mounted on a rack equipped with feet, which can be adjusted for mobility. Walk-in analyzer buildings (housings) should be provided with exits so located as to permit safe evacuation in the event of an emergency within the process area.

Material selected for the construction of the house should be of a type that will not cause operational difficulties nor create a safety hazard For example, a house constructed of sheet aluminum can absorb enough heat on a hot sunny day to raise the temperature of the analyzer enclosure higher than that of the thermostatic control point, thus causing false readings and malfunctions. A house constructed of cement-asbestos material can be a source of shrapnel if it is exposed to fire or explosion.

14.5 Sampling Systems

A major problem in most analyzer installations concerns supplying the analyzer with a sample which is continuously representative of the process stream and compatible with the analyzer used.

A representative sample is one that has been correctly taken from the process stream and conditioned without altering the variable factor to be measured. A sample which is suitable for measurement will not alter or adversely affect the analyzer's accuracy. Such a sample may have been treated, for example, to remove a material which would corrode or plug the analyzer but is still representative with regard to the property to be measured.

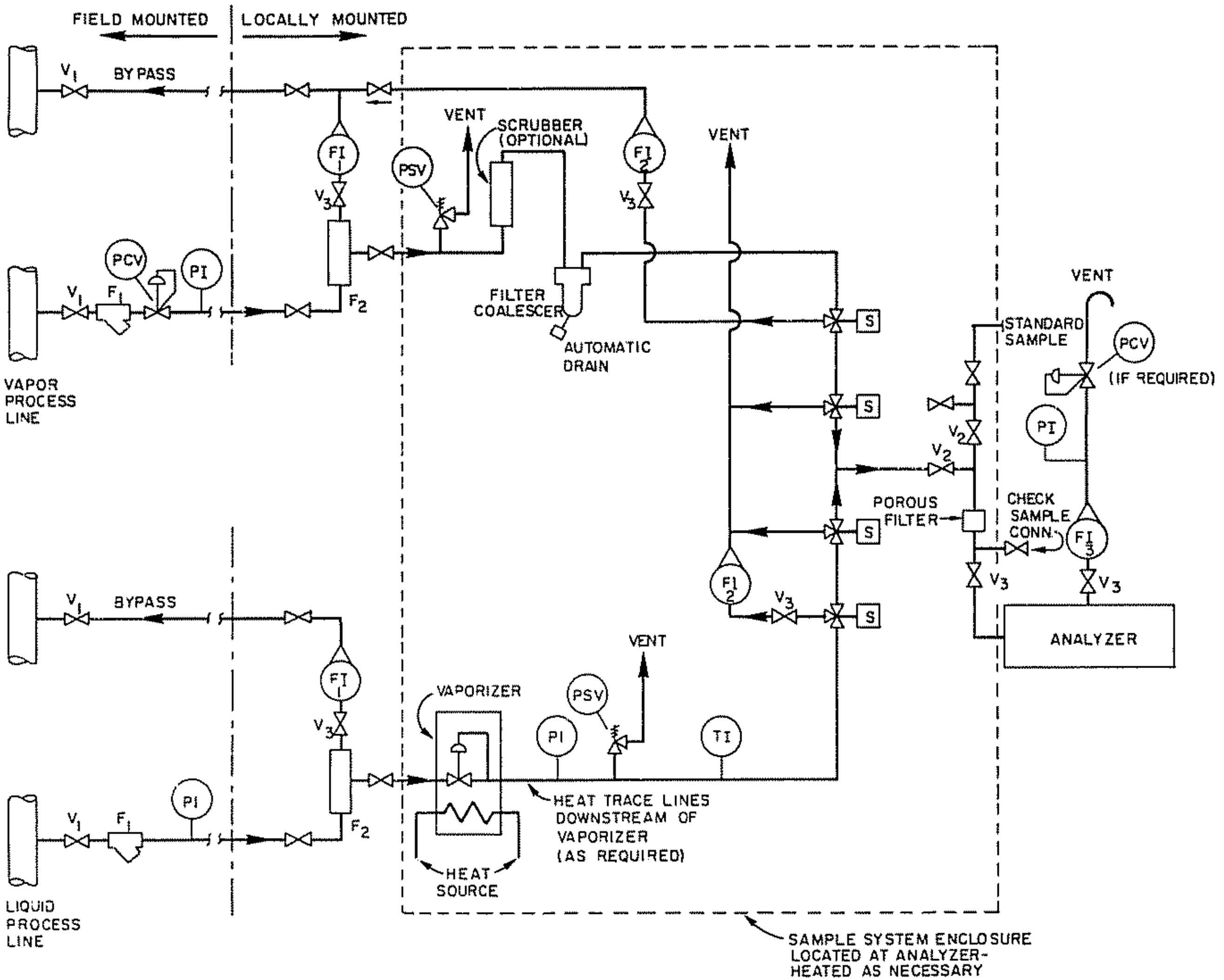
Analyzer streams may be drawn continuously or intermittently. Frequently, one of more fast moving bypass streams are circulated past the analyzer location (see Figure 14-1) so that a small sample may be directed into the analyzer continuously or periodically. Such use of a bypass flow loop past the analyzer allows time lag reduction without a need for excessive velocity in the sample line to the analyzer. Some analyzers measure continously, others handle samples periodically. Either type may be connected, in turn, to one of several streams to provide for the analysis of the several streams using one analyzer. This practice necessitates careful design engineering based on experience and the manufacturer's recommendations. Depending on the requirement of the analyzer application (on closed loop control or for alarm or shutdown, or both) and the overall timing and difficulty of analysis, it may be preferable to avoid multistream analysis using one analyzer. Experience has indicated the necessity of considering stream flow rate, pressure, temperature, corrosivity, cleanliness, consistency, phase, and dewpoint. These factors must be taken into account even though the installation is no more complex than inserting a capacitance probe into a pipe or a vessel.

There is no single simple solution to the sampling problem and no universal system suitable for most applications. Each installation, or type of installation (see 14.5.1 through 14.5.7), has its own problems and requires individual study before accurate analysis can be expected. Experience has shown that the study will prove valuable in providing reliable data and in reducing maintenance.

14.5.1 SAMPLE-POINT LOCATION

Although process characteristics govern the stream to be analyzed, there is usually a choice as to the physical location of the sample point. Preferably, sample points should be located to—

1. Provide a clean, representative, and measurable sample of the process stream, as influenced by the corrective action (action taken by the operator or by controlling valves and so forth to regulate the stream property measured) (Avoid sampling of any multiphase process system.)
2. Reduce the process time lag between the point in the process where corrective action is applied (for example, control valve location) and the sample point (Sample lines should be kept short to reduce the time lag between the sample point and the analyzer.)
3. Provide a clean, dry sample (except where moisture is a required factor in the analysis) to minimize the need for filters, dryers, knockout traps, and steam tracing
4. Obtain the sample at moderate pressure and temperature conditions to reduce requirements for pressure reducing valves, sample coolers, or heaters



Legend:

V_1—Block valve
V_2—Shutoff valve
V_3—Precision needle valve
F_1—First filter
F_2—In-line filter
PCV—Pressure control valve
TI—Temperature gage

PI—Pressure gage
FI_1—Flow indicator, main bypass stream
FI_2—Flow indicator, secondary bypass stream
FI_3—Flow indicator, sample stream
PSV—Pressure relief valve
S—Solenoid valve

NOTE: All vents and drains terminate outside of enclosure. Also see text for safe disposal.

Figure 14-1—Typical Multiple Stream Sampling Systems

5. Allow convenient access to the sample location.
6. Provide means for obtaining laboratory check samples from the sample line immediately upstream of the analyzer.

A poorly located sample point usually provides a non-representative sample and can cause entrainment of foreign matter. Also, such a location may cause excessive time lags or plugging or may make a calibration check difficult or impracticable.

A sample point close to the junction of two streams may provide a sample which is not thoroughly mixed. Sampling downstream from a pressure reducing valve may give mixed-phase problems. Entrainment may be minimized by properly locating the sample tap. The tap should be located on the side of the line to minimize liquid or dirt entrainment for vapor samples and to reduce dirt and bubbles in liquid samples. Sometimes a probe (see Figures 14-2 through 14-4) is used to withdraw the sample from the center of the line to avoid contamination from material carried on the inner surface of the pipe. A probe inserted through a packing gland and block valve, if permitted by piping specification, can be of value when removal of the probe is required for cleaning or for performing a sampling traverse. Designs using probes must consider maximum allowable velocity to prevent vibrational failure, provide blow out protection, and allow probe inlet orientation.

Sample lines should be routed as directly as possible and, in the case of gas sampling, in such a manner as to avoid trapping of liquid or contaminating particles.

When monitoring a fractionator, there is usually a place in the column that will show greater or faster composition changes than those that occur in the product stream. A sample from this location will often provide more responsive control. Perhaps the requirements of the application will make analysis desirable at two points —one to monitor product purity, the second to provide faster control. The sample location for overhead product control is between the feed tray and the tower top; for bottom product, it is between the feed tray and the tower bottom. Sometimes a computer is used to calculate fractionating conditions for guidance in locating the sample point. However, this choice may be incorrect if conditions or end use of the data change.

14.5.2 SAMPLE CONDITIONING

It is safe to assume that process stream samples will contain contaminants. Some samples must be chemically treated to remove or inactivate contaminants, however, this may cause other problems from the reactants. Often liquid samples must be vaporized, and vapor samples must be heated to prevent condensation or possible deposition of dissolved solids (see RP 550, Part I, Section 8). The line between a vaporizer and the analyzer must be heated to maintain a temperature higher than the dewpoint. Some materials may polymerize or react chemically when heated. When gas pressure is sharply reduced some samples may freeze, deposit ice, or form hydrates due to refrigeration effect. Frequently, both the sample and analyzer temperature and pressure require close regulation. Usually the sample flow rate, or sampling quantity and frequency, must be closely controlled. Neglect by the installation designer of any one of these requirements may result in an erroneous analysis or no analysis at all.

Most refinery streams are water saturated, therefore, unless the moisture content is to be measured or the component to be measured is soluble in water, moisture removal usually must be provided. Entrainment separators or coalescers will remove water droplets. However, special precautions may be needed when water in solution or water vapor is to be removed. For example, water accumulation in an initial boiling point analyzer would cause the readout to be meaningless.

Where saturated vapor samples are transported through an area where ambient temperature is lower than the temperature at the sample inlet point, the sample line should be heat traced or jacketed to avoid formation of condensate.

14.5.3 STANDARD SAMPLES

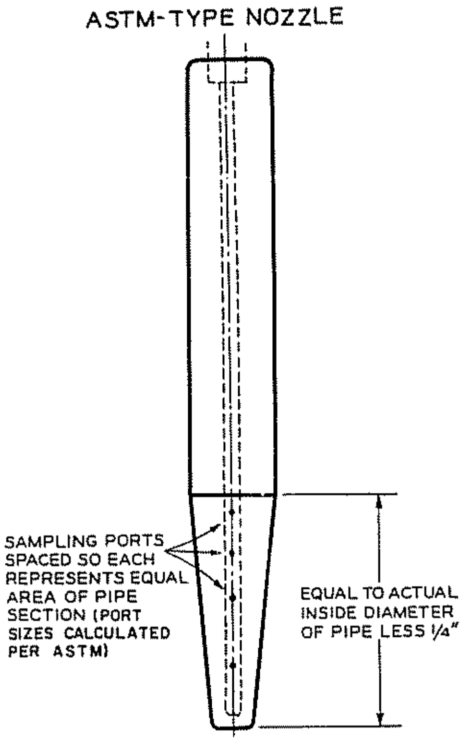
Standard samples are usually laboratory or commercially prepared volumes of gases or liquids which contain known quantities of the component or components which the analyzer was designed to detect. Such samples must be uniform, representative, and stable. The sample should be kept under conditions which will not cause changes in the analyzed properties. These values can change because of evaporation, polymerization, stratification, contamination, and so forth. Storage conditions should be selected for each particular type of sample.

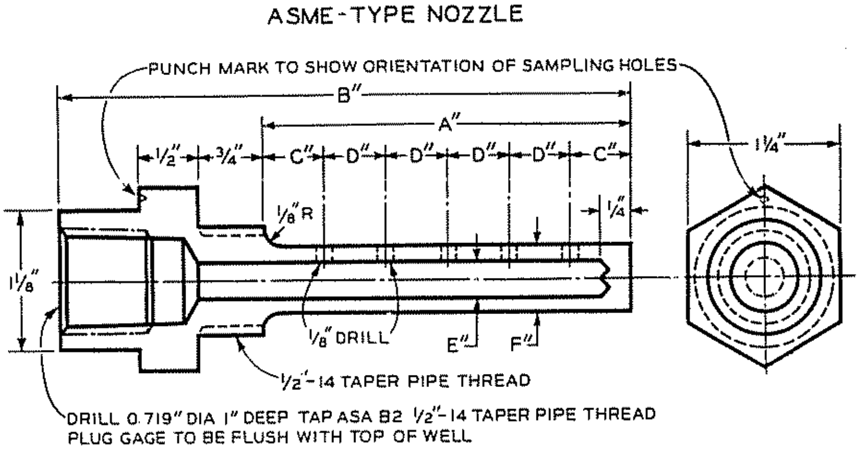
Standard samples must be obtained from a reliable source, and it is advisable to have the control laboratory run initial and periodic analyses of the standard sample. This often helps point out direct correlations between laboratory readings and process readings and is a quick method of troubleshooting. Determination of absolute values for standard sample components can be a serious problem because of the difficulty in obtaining repeatable laboratory results. Therefore, the average value from a number of laboratory analyses may be required to provide a reasonably accurate value for the standard sample.

Checking or calibration of analyzers can be accomplished more easily if, in the original installation, provisions are made to supply a standard sample to the analyzer (see Figure 14-1). This may permit the process unit operator to make sure the analyzer is recording accurately.

14.5.4 USER-SUPPLIER RELATIONSHIPS

It is essential to carefully review sample requirements with the analyzer supplier. This helps avoid trouble which may develop from the user's failure to fully understand the equipment or from the supplier's failure to fully understand the application. With the analyzer, the supplier may also furnish all the equipment needed to properly condition and regulate the sample. It is necessary to carefully define all conditions of operation, all components of the process stream sampled, and all variations of sample composition. Furthermore, it is necessary to obtain realistic assurance that the supplier com-





NOTE: Some piping specifications will require a flanged or Van-Stone type configuration for the sample nozzle.

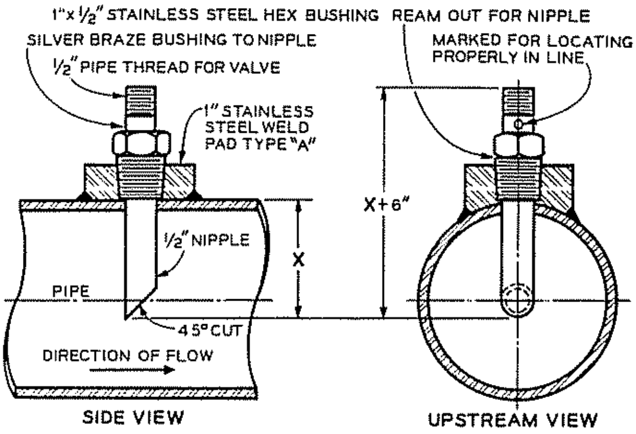
Number of ports for ASTM-type nozzles are as follows:

Inside Diameter of Pipe (inches)	Number of Ports
2 to 6	4
Over 6 to 12	6
Over 12	8

STANDARD ASME TYPES OF NOZZLES

Steam Line		Sampling Nozzle						
				Length		Sampling Holes		
Nominal Size (inches)	Internal Diameter (inches)	Outside Diameter F (inches)	Bore E (inches)	A (inches)	B (inches)	Quantity	C (inches)	D (inches)
2	1.939	0.540	0.300	1¾	3⅝	3	⅝	½
2½	2.323	0.540	0.300	2	3⅞	3	½	½
3	2.9	0.540	0.300	2¾	4⅝	5	⅜	½
3½	3.364	0.540	0.300	3	4⅞	5	½	½
4	3.826	0.540 or 0.675	0.300 or 0.437	3⅝	5½	6	9/16	½
4½	4.290	0.540 or 0.675	0.300 or 0.437	4	5⅞	6	7/16	⅝
5	4.813	0.540 or 0.675	0.300 or 0.437	4⅝	6½	6	7/16	¾
6	5.761	0.540 or 0.675	0.300 or 0.437	5½	7⅜	6	⅞	¾
8	7.625	0.540 or 0.675	0.300 or 0.437	7½	9⅜	6	⅝	1¼
10	9.75	0.675	0.437	9½	11⅜	6	⅜	1¾
12	11.75	0.675	0.437	11½	13⅜	7	½	1¾
14	13.0	0.675	0.437	12½	14⅜	7	⅝	1⅞
16	14.75	0.675	0.437	14¼	16⅛	8	9/16	1⅞
18	16.5	0.675	0.437	16	17⅞	8	1 7/16	1⅞
20	18.5	0.675	0.437	18	19⅞	9	1	2

Figure 14-2—Examples of ASTM and ASME Types of Nozzles



Stainless Steel Gas Samples

Line Size (inches)	"X" Diameter (inches)	Mark No.
3	2¼	A
4	2¼	B
6	3$\frac{13}{16}$	C
8	4$\frac{13}{16}$	D
10	5⅞	E
12	6⅞	F

NOTE: All nipples are to be seamless, extra heavy stainless steel

Figure 14-3—Typical Gas Sample Probe

pletely understands the problem and will furnish equipment fully suitable for the application.

It is very advantageous to the purchaser of an analyzer to send unconditioned samples, in sufficient quantity, to the supplier so that he can operate the analyzer in his laboratory on the actual process stream material. This will enable him to determine what equipment is necessary for sample conditioning and to check the ability of the analyzer to handle the raw or conditioned sample material. Equally important are the records the supplier produces. These records should be given to the people responsible for analyzer operation and maintenance. They are a most valuable reference for startup procedures and for solving future operational problems. Chromatograms and boiling-point-analyzer records can be especially helpful to the user of this type of equipment.

Transferring sample system design responsibility to the supplier may cause trouble. Usually a middle course is best—combining the design abilities of the user, installer, and supplier—so that the instrument receives a correctly conditioned sample to ensure satisfactory performance. Care should be taken to fully define the system responsibilities.

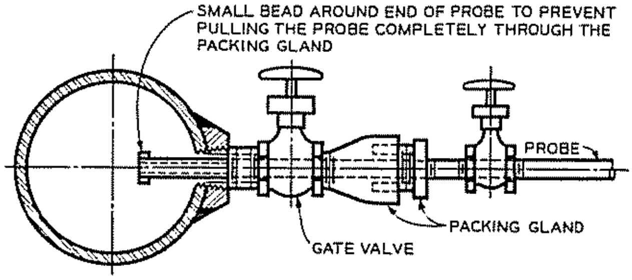


Figure 14-4—Special Gas Sample Probe

14.5.5 SAMPLING SYSTEM COMPONENTS

Equipment used in sampling systems may include pumps, ejectors, regulating valves, solenoid valves, flowmeters, sample coolers or heaters, filters, coalescers, dryers, knockout traps and trap legs, manifolds for switching samples, and timing equipment. Those responsible for the installation design must consider not only the materials of construction, but also the sizing and the location of these components. A sampling system must be assembled with the minimum number of parts compatible with proper transportation and conditioning of the sample. Extra components should not be supplied on the theory that they may be needed later because they increase the volume, lag, and servicing problems of the system. Particular care must be exercised to avoid introduction of excessive lag time in applications where the analyzer is used for control.

Each sample loop of a multiple-stream system must be considered as if it were the only sample to the analyzer. A careful study of all loop components will show which units can be used in common with all streams and which units must be isolated when the switching valve is actuated.

14.5.6 SAMPLING SYSTEM MATERIALS

By properly selecting materials for construction of the sampling system, a designer can avoid the following:

1. Reaction of materials with the sample
2. Absorption of components from the sample
3. Entrance of contaminants through osmosis or diffusion
4. Leaching material from the sampling system components
5. A fire hazard or other unsafe condition

When monitoring for trace constituents, some of these effects seriously reduce and even destroy the reliability of the analyzer's output. The following precautions should help to minimize such problems:

1. Use stainless steel tubing where practicable. (Be certain that the tubing is clean and grease-free before use.)
2. Protect sample tubing from mechanical abuse.

3. Design the sampling system to be pressure tight as determined by requirements.

FOR EXAMPLE: Molecular size leaks have been known to allow oxygen to diffuse into a sample stream when the partial pressure of oxygen in the sample was below that of air. This has been reported at pressure levels as high as several thousand pounds. Moisture can diffuse into a pressurized system in a similar manner. [9]

4. Examine each component in the system—such as filters, pressure regulators, valves, and flow controllers—for materials which may react with or absorb the sample.

FOR EXAMPLE: In one installed sampling system, a sample containing not more than 500 parts per million of hydrogen sulfide required from 3 to 7 days to reach equilibrium because the hydrogen sulfide reacted with components of the sampling system.

When pipe thread compounds are used, care should be taken that surplus quantities are not forced into the system. Sealing compounds either in liquid or paste form, or even in tape form, should be applied only to male pipe threads. Use of socket or seal-weld connections is frequently desirable. However, the designer should know that such connections leave small internal cracks or pockets in the pipe wall in which contaminants can accumulate.

Many pressure regulators, flow controllers, and the like contain absorbent diaphragms, or small cork or felt filters. When measuring low-moisture content or highly absorbent material, such as alcohol and solvents, these elements should be removed or replaced, otherwise the sampling system may require a long period of time to reach equilibrium. To prevent the possible loss of sample by absorption, metallic or ceramic filters should be used in preference to fiber or paper filters. It is recognized, however, that on special applications the fiber or paper filters are the superior choice.

14.5.7 SIZING COMPONENTS

The volume of the sampling system must be kept to a minimum and the flow velocity in the sampling system must be as high as is practicable consistent with required flow and permissible pressure drop. This is necessary so that the analytical system will have adequate speed of response to fluctuations of the measured variable in the process stream. Large sample lines introduce long time lags. Low velocities permit entrained liquid or foreign material to settle in the line which may eventually cause plugging. Good design velocity for flow through sample tubing has been found to range between 5 and 10 feet per second. Sample line tubing as small as ⅛-inch OD is frequently used on clean samples.

Dryers and filters should be as small as practicable and still perform their function. Large sizes increase lag time, cause back-mixing from diffusion, and delay the instrument's response to an actual change in sample composition. When a liquid sample is to be vaporized, it is especially important to minimize the dead ended liquid volume upstream of the vaporizing regulator.

With gas systems where long sample lines cannot be avoided, a pressure reduction to a level lower than that of the process at the sample point may be advantageous. A reduction in the pressure increases flow velocity and reduces the time required for gas to reach the analyzer. [10]

Sampling systems have been specially designed so that flow rates in the system (as distinguished from flowing quantities) were high. In order to reduce the time needed for transporting a sample from the process equipment to the analyzer, the volumes of knockout traps, filters, tubing, and valves were made as small as reasonable. Designers have gone even further in the case of chromatographs. By specially designing analyzer components, they have greatly reduced the size of the sample required. These designs have decreased the response time by a factor of ten or more over previously installed units.

14.6 Filtration [11]

As a general rule, all sampling systems should contain suitable filters. Self-cleaning bypass type filters are often used.

It is the responsibility of the installer to provide protection for the sampling system. Most sampling systems will have one or more pressure regulators, flow controllers, orifices, or other devices with flow restrictions needing the protection of a filter to remove solid matter and prevent plugging.

The selection of a filter that will prevent the plugging of a sampling system, which includes the filter itself, requires a knowledge of the physical and chemical conditions of the sample. For example, the porosity of the filter surface must be small enough to stop any solid particles that could cause plugging but not so small that the filter will readily foul. The length of service expected from the filter depends upon the percentage of solid particles in the sample stream, their size, the sample flow rate, and the surface area of the filter.

Cellulose cartridge types of filters may be satisfactory, provided the cellulose does not absorb some component of interest. In general, these filters will remove particles as small as 3 microns at pressure differences up to 100 pounds per square inch gage. Ceramic or porous-metallic types of filters also have been used satisfactorily in sampling service. Ceramic and metallic-plate types of filters are available to trap particles from approximately 13 microns in size and larger. Sintered-metallic filters are available to trap particles as fine as 2 microns in size. Some models of either type are constructed so that only a portion of the total stream passes through the filter element. The remainder of the stream acts as a flushing medium to keep the internal filter surface clean.

When anticipating a high content of solids in a sample stream, installation of filters in parallel—with means provided for the isolation of each filter—is recommended. Motorized self-cleaning filters or automatic blowback systems may be considered. Important considerations are the accessibility and interchangeability of the filter elements.

14.7 Entrainment Removal [11]

Entrainment may be minimized through proper sample tap location (see 14.5.1). However, entrainment may occur even though the best tap location available has been selected. Entrainment can be in the form of liquid droplets in gaseous sample streams or in the form of gas bubbles in liquid sample streams. Water droplets can be entrained in most streams. Very few analyzers, if any, can tolerate entrainment. Therefore, entrainment removal should be of primary concern.

14.7.1 ENTRAINED LIQUID

Many gas samples contain droplets which are not a representative part of the sample. Sometimes these droplets are well dispersed and must be removed. Numerous methods for the removal of entrained liquid are available, including cold traps, baffles, sintered disk filters, or centrifuges. The entrainment separator shown in Figure 14-5 is perhaps the simplest and most widely used.

In this system, the gas enters the separator near the top and flows down through the packing to within 3 or 4 inches of the bottom. There the gas flow reverses direction, passing up through the tubing connected to the sampling system. As the gas passes through the packing, the small liquid droplets collect on the fibers. The droplets continue to collect and agglomerate until they are large enough to break away from the fiber. The larger drops of liquid fall to the bottom where they are accumulated and disposed of. Disposal is usually accomplished by means of an automatic float valve.

14.7.2 ENTRAINED GAS

A similar system, shown schematically in Figure 14-6, can be used to remove entrained gas bubbles from a liquid stream. For this service, the liquid enters near the bottom of the separator and flows with the entrained gas bubbles up through the packing. The gas bubbles collect and agglomerate on the packing, forming large bubbles which break away and continue on to the top, where the gas is separated from the liquid and vented from the sampling system.

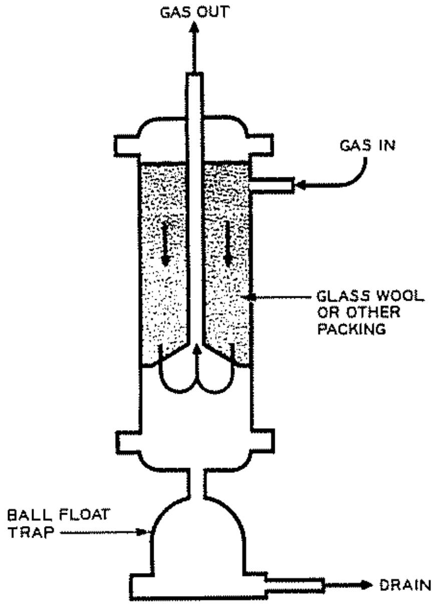


Figure 14-5—Entrained Liquid Separator

14.8 Scrubbers and Chemical Treatment [11]

Sample streams may contain corrosive gases and condensable vapors which can cause inaccuracies of measurement and may even damage the instrument. These and other sample contaminants can be removed by passing the sample stream through a liquid solution. Scrubbers, as these devices are called, take many forms. Some contain only a liquid solution; others use packings, such as beads, shot, and rings in addition to the liquid solution; still others use such mechanical devices as mixers or stirrers to provide more intimate contact of the sample streams and the liquid solution. Beds of solids (for example, caustic pellets) also have been used to prevent entrainment. Varied combinations of these methods are currently in use. The complexity of the scrubber system is dependent upon the needs of the application.

14.8.1 SPARGERS

The scrubber shown in Figure 14-7 is the sparger type in which the gas is made to enter the scrubbing medium through a sintered disk. Because of the small holes in the sintered disk, the gas breaks up, or separates, into small bubbles. The smaller the bubble, the greater the liquid-to-gas contact per unit volume of gas, which, in turn, produces greater scrubbing efficiency. The small bubble size tends to cause foaming, and there may be a carryover of liquid droplets. The gas velocity should be kept low to avoid such lift effects. However, the use of an entrainment separator following the scrubber will remove the liquid droplets.

14.8.2 PACKED TOWERS

The packed-tower type of scrubber, shown in Figure 14-8, is widely used. The tower contains both a liquid solution and a nonreactive packing (for example, beads) Gas enters through a pipe connection near the bottom and bubbles up through the solution The packing helps keep the gas stream in bubble form and provides a wetted surface to improve gas/liquid contact as the sample flows in a random and circuitous path through the packing to the top of the scrubber. Liquid carryover, which results from foaming in the scrubber, can be removed by use of downstream baffles or entrainment separators.

14.8.3 GAS LIFT SCRUBBERS

The scrubber depicted in Figure 14-9 is the gas-lift type. The gas sample enters the gas lift and breaks up into bubbles, each lifting a little liquid ahead of it, traveling up the tower. At the top of the tower the gas and liquid separate with the liquid returning to a reservoir to be used again. This scrubber is particularly suited to those applications where considerable precipitates are formed in the scrubber. The gas lift carries the precipitates over into a filter where the solid content is separated from the liquid.

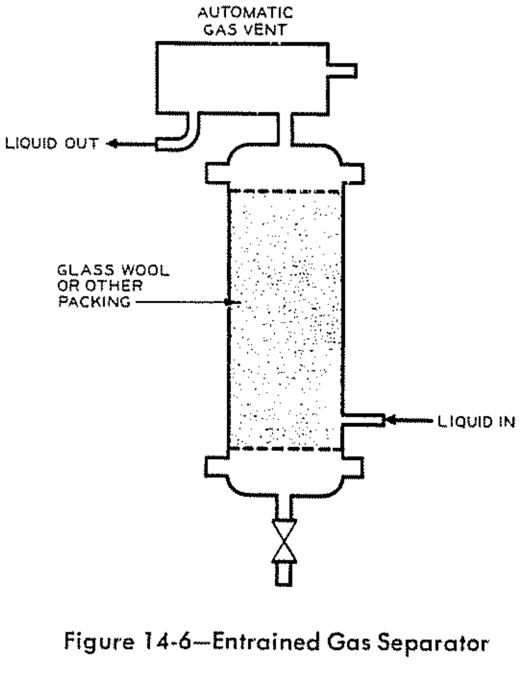


Figure 14-6—Entrained Gas Separator

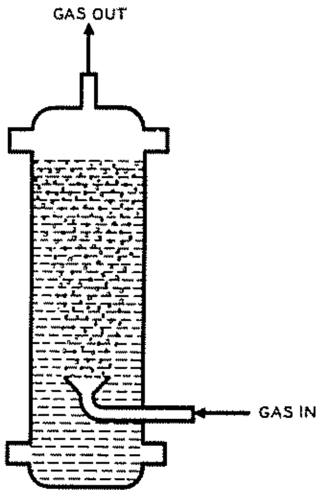


Figure 14-7—Sparger

14.8.4 TREATING SOLUTIONS

Hydrogen sulfide is frequently removed from a gas stream by bubbling it through a caustic soda solution. Moisture can be removed by bubbling the gas stream through concentrated sulfuric acid, provided the acid does not react with other components, such as unsaturates or aromatics, in the sample It is desirable for scrubbers to be sized to hold from one week's to a month's supply of reagent so chemical changing may be done in a routine but infrequent manner. Specialized solutions may be used where normal treatment does not meet the requirements.

14.8.5 CLEANUP OF TREATED SAMPLES

Entrainment of chemicals may cause false indications or even damage the analyzer. Reaction or scrubber pots for gas streams should be designed using at least one

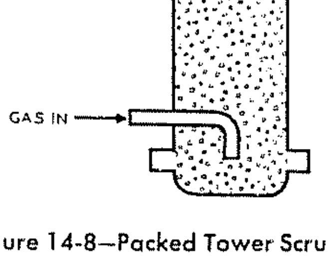


Figure 14-8—Packed Tower Scrubber

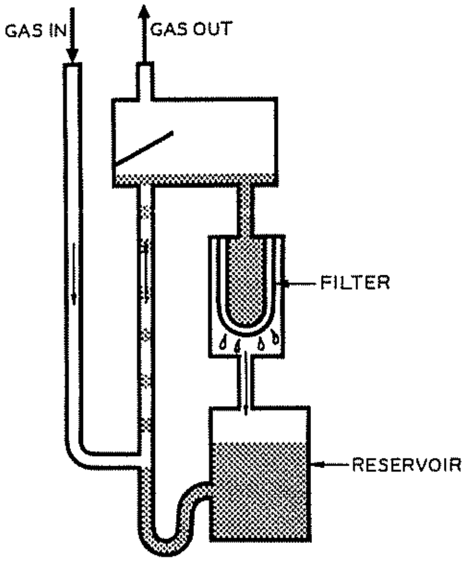


Figure 14-9—Gas Lift Scrubber

and one-half times the cross-sectional area calculated by conventional published design methods. Usually knockout pots should be slightly larger in area to be certain entrainment is eliminated. Knockout pots may have tangential inlets. In sizing pots, the internal volume above the liquid must be considered. Prescribed levels should be clearly indicated for the benefit of maintenance personnel because an overfilled pot may create carryover problems, and a half-empty pot may increase time lag. If an appreciable liquid carryover is expected, both scrubbers and knockout pots may require gage glasses to determine the liquid level.

14.9 Valving

Proper operation and tight seating of valves is important in a sampling system. Small leaks between sample streams or leakage of calibration gases into the sampling system causes erroneous results. Special attention must be given to valve selection for sampling systems. There are several requirements common to all types of valves. Construction materials must resist corrosion by sample components and must not absorb or contaminate these components. There should be as few moving parts as possible, and they should be designed for minimum wear and dependable service without lubrication. There should be no dead space to hold up a portion of sample material and then later release that quantity to contaminate the sample stream or carrier. Leakage must be avoided particularly where the valve is mounted inside housings or in the analyzer enclosure.

14.9.1 BLOCK VALVES [11]

In order to allow the system to be shut down for maintenance, a block valve should be installed immediately downstream of the sample takeoff point, at the inlet and outlet of the analyzer, and in the sample return line. Where reverse flow is possible, provide a check valve in the return line. Block valves should be either gate valves or cocks of the proper material and with suitable packing. The valves at the sample takeoff point and in the sample return line must meet or exceed process line specification. Care must be taken to install the valves in the correct flow direction. This is especially true on systems that run at pressure levels below atmospheric.

14.9.2 SWITCHING

Automatic switching may be accomplished through solenoid valves. These should be installed on the assumption that they will leak and means should be provided to eliminate mixing of materials between two or more lines. Double-block and bleeder valves, properly synchronized, can eliminate this mixing. If several streams are involved, a simpler system uses three-way solenoid valves on each stream with the material being diverted to a vent or drain when not passing to the analyzer.

In some services the use of solenoid-operated valves may not be satisfactory. In these cases the use of piston-, diaphragm-, or motor-operated valves should be considered.

14.9.3 SPECIAL VALVES

For such analyzers as chromatographs, special valves have been developed to deliver small discrete gas or liquid stream samples whose volume or mass[2] is held constant. By minimizing the sample volume, the analysis time is greatly reduced.

These special valves are commercially available from several manufacturers. Their use on clean streams speeds up analysis and reduces analyzer maintenance since less material passes through the instrument. Where a representative liquid sample can be obtained, liquid sampling valves may be used. These valves may reduce vaporization difficulties. However, the installer should be certain that vaporization will not occur in the sample valve when transferring liquid "slugs." Frequently, it is essential to maintain a back pressure on the sample valve discharge line to keep the pressure of the liquid sample above its bubble point. Special valves associated with particular types of analyzers are included in the discussions of the analyzers covered throughout the text.

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[2] This use of the word *mass* connotes a fixed volume of material in a single phase at fixed pressure and temperature conditions and assumes that successive samples are nearly the same composition.

14.10 Volume-Mass Relationships [11]

Some quantitative measurements are functions of the number of molecules of gas in the analysis cell. In such cases the temperature and pressure of the sample are highly significant to accuracy.

14.10.1 LIQUIDS

For liquids, only the temperature effect need be considered. Most manufacturers of the liquid type of analytical instruments build in temperature compensation where necessary.

With uncompensated instruments, if the temperature coefficient is small (that is less than 1 percent of full scale per degree Celsius) temperature compensation or control is necessary only if the instrument experiences large temperature variations at the analyzer cell. If the temperature coefficient is less than 5 percent per degree Celsius, temperature compensation of the analyzer is usually sufficient. Coefficients larger than this may require temperature compensation and control for both the instrument and sample stream. However, temperature control of the sample can introduce errors in the measurement. For example, heating can cause bubble formation, polymerization, decomposition, or hydrolysis. Therefore, caution must be exercised in such instances.

14.10.2 GASES AND VAPORS

For gases and vapors, the number of molecules per unit volume at constant pressure is inversely proportional to the absolute temperature. For gas analyzers, the measurement may be exponential for changes in the number of molecules in the measuring cell. Two variables, therefore, may affect the measurement when the temperature of the gas in the measuring cell varies. Whether or not the error introduced requires temperature control of the gas in the measuring cell depends upon the required measurement accuracy, the temperature coefficient of the measuring cell, and the temperature variations without control of the gas sample and measuring cell.

The temperature coefficients usually range from approximately 0.2 percent of full scale to several percent per degree Celsius. Gas sample temperatures may fluctuate as much as 50 to 60 degrees. Therefore, in most cases, the gas sample temperature must be controlled in order for an analyzer to be accurate within 1 percent of full scale.

14.10.3 VAPORIZING

Frequently, liquid samples must be analyzed in instruments designed to handle vapor only. In this case, a vaporizer is included as a part of the sample system. Vaporizing can be a difficult problem, thus vaporizers should be carefully selected. The most popular methods for vaporization are the vaporizing pressure regulator or the capillary-tube vaporizer. Although satisfactory for most clean hydrocarbon streams, both methods are unsatisfactory for other streams because of three phenomena. The first factor is sample fractionation, or separation of sample components in a manner that cannot be predicted or controlled, thus giving a sample which is not representative. The second factor is a change in one or more of the components themselves, an example being polymerization, which makes the sample nonrepresentative and sometimes plugs the apparatus. The third factor is deposition of dissolved solids such as salts, which, although present only in minute quantities, can build up rapidly in continuous operation. These factors must be considered for any liquid sample before the vaporization principle is used. The systems should be designed so that the temperature of the vaporized sample is always maintained above its dewpoint.

14.11 Flow Rate and Control [11]

Sample flow is usually a compromise between several requirements. The maximum flow rate may be determined by the amount of sample which can be drawn from the process by pressure drop available for the design of the high-speed sample loop, by the maximum pressure drop across a vent line where pressurization of the sample in the cell occurs, or by the temperature effects caused by heat or lack of heat. The advantage of a high flow rate is that it minimizes the time lag in the sampling system. However, the design engineer must also keep in mind the capacity of the various sampling system components—filters, scrubbers, separators—that are being provided to prevent or alleviate certain undesirable conditions. At high flow rates, filters may need more frequent cleaning or replacement, and scrubbers will need to be regenerated more often. High flow rates tend to increase the possibility of entrainment whether it be gas bubbles in a liquid stream or liquid droplets in a gas stream. High flow rate sampling systems require higher capacity system components than do low flow rate systems.

Where high flow rates are necessary to minimize time lag and yet a clean sample is required at the analyzer, a bypass loop (see Figure 14-1) may be provided up to the analyzing unit of the instrument at which point only a small portion of the sample need be removed and conditioned for analysis. The bypass flow being high in comparison to the quantity passing through the instrument permits a high sampling rate. However, it is usually unnecessary to condition bypassed material beyond

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1. *Needle valve or orifice:* The pressure upstream of a simple needle valve or orifice is regulated and the sample vented to the atmosphere. This method is applicable where precise control is unnecessary. The flow rate may drift over periods of time.
2. *Differential pressure flow controller:* A differential pressure flow controller may be used (many models of which are marketed as purge units). As shown in Figure 14-10, an indicating rotameter is usually provided, but it is not always an integral part of the controller unit. The flow rate to the analyzer is indicated by the rotameter and can be varied by adjusting the needle valve. The relay maintains a constant differential pressure across the needle valve, thus reducing the probability of flow rate drift. This method requires either a constant upstream or constant downstream pressure.
3. *Additional requirements:* Ordinarily, the function of the sample flow control system is to regulate flow rate. However, there are measurement and analysis systems that require additional regulation, such as automatic correction for variations in barometric pressure, gas specific gravity, and so forth.

The sample stream flow control methods discussed heretofore have been primarily for streams in gaseous state; however, most of these methods can be satisfactory for streams in liquid state. Normally, for the analyzers that process the sample stream in liquid state, the sample stream flow rate control method need not be complex. The simple flow control unit shown in Figure 14-11 can be used where there is little change in sample viscosity or density.

A positive displacement pump, driven at fixed speed, is frequently used for controlling flow. The metering pump minimizes the effect of viscosity and density changes experienced in other types of flow controllers, but maintenance requirements often limit its use. A rotary pump delivers a more uniform flow than a piston or diaphragm type. However, the rotary pump must have close clearances to hold delivery constant.

The listing of sample flow control methods could be lengthy. Therefore, each application should be studied to determine the minimum acceptable method required to obtain results with the degree of precision desired.

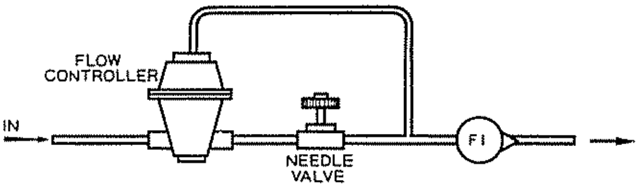

Figure 14-10—Differential Pressure Flow Controller

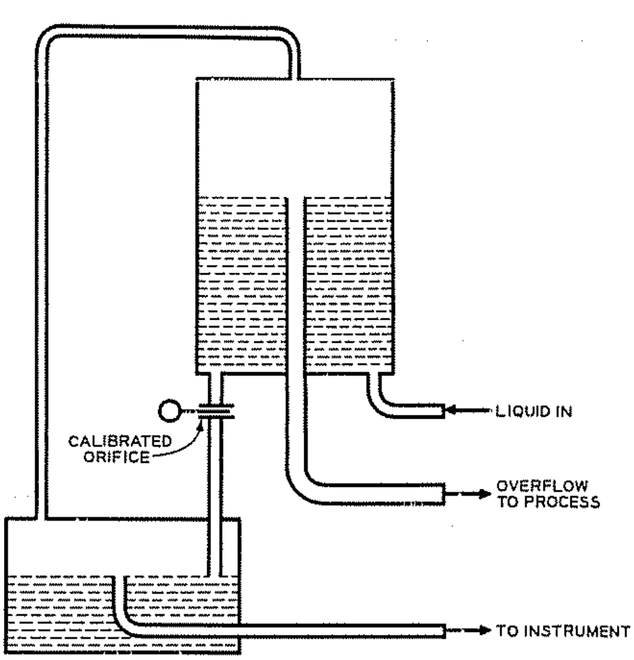

Figure 14-11—Liquid Flow Rate Controller

Rotameters are generally used for flow indication. They should be the armored type where required by process conditions.

14.12 Venting [11]

If a separate vent for the analyzer is used, the location of that vent should be in an area of minimum air turbulence. Often it is inadvisable to vent analyzers into a common vent or blowdown system. Frequently, the back pressure in a common system will fluctuate by several inches of mercury. Pressure variations of that order in a sample cell can cause excessive errors in measurement. If it is necessary to vent the gas from the analyzer into a common vent, the sample cell should be operated at an elevated pressure, and a back-pressure regulator should be used. When changes in barometric pressure may cause relatively large errors in measurement, the use of an absolute-pressure regulator may be necessary. Several types of regulators are described in manufacturers' literature. A good back-pressure regulator can hold the upstream pressure constant regardless of variations in barometric pressures. Some are capable of maintaining the set pressure within ± 0.5 inches of water.

Whenever a chance of diffusion from the surroundings into the analysis stream may exist (such as a fire hazard or other unsafe condition), the possibility of back-diffusion from the venting system should not be overlooked. Maintaining good flowing velocity in the vent system may be helpful in such cases. Use of a properly located back-pressure regulator will assist in this regard.

Consideration should be given to the vent piping design to prevent condensate from accumulating in low points and obstructing a free vent flow. Also, the open end of the vent riser should be designed to prevent the entrance of rain or snow and to minimize the effects of wind impacting on the open end of the vent. A tee with a side port connected to the vent line is an effective discharge arrangement.

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SECTION 15—GAS CHROMATOGRAPHS

15.1 Scope

This section outlines methods for the installation of process analyzers involving the techniques of gas-solid and gas-liquid partition chromatography. Instruments for laboratory use are not covered nor are instruments for high-speed liquid chromatography.

15.2 General

The gas chromatograph is a device which is used for making qualitative and quantitative analyses of selected stream components and for promptly supplying information equivalent to that obtained from a refinery control laboratory The purpose of the gas chromatograph is to determine quantities of individual components, or combinations of components, to the extent necessary for intelligent operation of a process. It can be used for the following:

1 Single key component analysis
2 Multicomponent analysis (single stream)
3 Complete analysis (single stream)
4 Multistream analysis, either partial or complete.

The gas chromatograph is made up of separate elements,[1] each of which has its own operating principles and characteristics and, consequently, its own installation requirements The combination of elements employed in a particular chromatograph establishes its installation requirements. Because possible combinations are numerous, this section of the manual describes only the more common variations of each element, and enumerates the characteristics and limitations that affect installation of each variation. Total installation requirements for a specific instrument can be determined by adding up the requirements for each of its elements.

A fairly thorough knowledge of gas chromatography is desirable for proper application and installation The reader is referred to general textbooks for detailed information on gas chromatographic theory and practice and is advised to study the publications of equipment vendors and to consult the indexes of periodicals and technical abstracts for the latest information available.

Although design, procurement, and installation procedures for analyzers are generally the same as those used for conventional instruments, increasing use is being made of separate analyzer contractors to handle the analyzer portion of larger projects. This technique has the advantage of utilizing an analyzer specialist for this unique requirement.

15.3 Functions

A gas chromatograph employs elements for separating the sample into components and for identifying and measuring those components.

Reduced to simplest terms, the gas chromatograph is a tube (column) packed or coated with a material that exerts different retarding effects on the components of a gas or vapor sample passing through it Hence, the components of a mixture introduced as a batch at one end of the column and transported through the column by a moving carrier gas will be separated. The components will emerge from the downstream end of the column at different times, but all the molecules of a given component will emerge together within a narrow time span. A sensitive detector is employed at the column outlet in order to reveal the presence and concentration of emerging components. The detector signals may be displayed on a strip chart recorder in the form of deflections (peaks).

Changes in the column packing material and in the temperature at which the column is maintained provide a means for changing the type of separation achieved. Other factors also influence the separation. The gas chromatographic instrument is designed to hold all factors which influence the separation constant, so that repetitive analyses on a given sample will yield identical patterns of peaks (chromatograms). Therefore, with a properly operating process gas chromatograph, any changes in peak sizes or number of peaks observed on successive process samples will reflect changes in the process.

15.4 Chromatograph Requirements

A gas chromatograph may be divided into seven elements each of which performs specific functions

1. A sampling apparatus for injecting a controlled volume of the sample
2. A temperature-controlled chamber housing at least the column and usually the detector

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[1] The more common term *components* is avoided in describing the instrument to prevent confusion with sample components and sample system components

3. A chromatographic column or columns for making the component separation
4. A carrier gas system for transporting the components through the column
5. A detector for measuring the eluted components
6. A readout device for recording the component peaks that constitute the chromatogram
7. A programmer for automatically operating certain of these elements at prescribed times during the operating cycle.

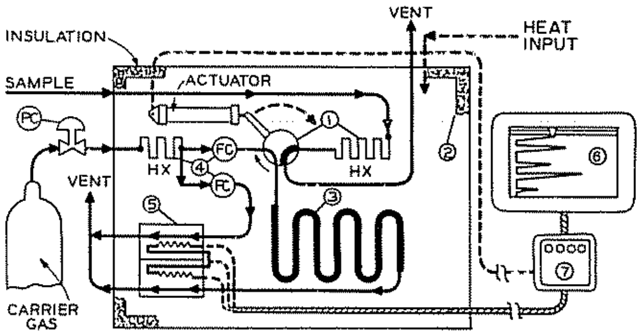
Several versions of each element are available and for a particular application compatible versions of all seven basic elements are combined in an instrument best suited to the analysis. This has resulted in a wide variety of instruments which, although they may appear similar, have quite different installation requirements.

15.5 Automatic Gas Chromatograph

An automatic chromatograph is shown schematically in Figure 15-1. The elements (numbered 1 through 7 for reference) are discussed in 15.5.1 through 15.5.7. Since each element has individual functions and requirements that affect installation, a few variations of each element are discussed with emphasis on precautions to be observed in installation.

15.5.1 SAMPLING APPARATUS

A sampling apparatus in an automatic chromatograph, identified as Element 1 in Figure 15-1, is usually a special valve which performs the function of introducing a constant volume of sample into the upstream end of the chromatographic column. A clean, representative sample, conditioned as in Section 14, enters the temperature-controlled chamber and flows through the heat exchanger (HX), which raises it to the correct temperature through the measured volume section of the sampling valve. A coil of tubing in the chamber is often used as a heat exchanger. The sampling valve is arranged so that, in normal position, carrier gas flows continuously to the column while the sample flows continuously out the vent to provide a current sample. All sample valves also incorporate some type of trap section for accurately measuring sample volume. On command from the programmer, this trapped volume is injected into the carrier gas path by automatically changing the slider, piston, or rotary valves to a new position. After the sample has been purged out of the measuring trap section, the valve returns to its normal position.



Elements:
1. Sampling apparatus
2. Temperature controlled chamber
3. Chromatographic column
4. Carrier gas system
5. Detector
6. Readout device
7. Programmer

Figure 15-1—Schematic Diagram of an Automatic Chromatograph

15.5.1.1 Requirements and Developments

A sampling valve should accomplish two purposes. First, on command, the sampling valve should inject a fixed mass of sample. Because most sample valves measure volume, the installation must be designed for maintaining a constant mass-volume relationship (see Section 14). Second, to reduce transportation lag, the valve should continuously vent the sample stream. Rate of venting depends upon three factors. One factor is the speed at which the analyzer must operate. The operating speed of the analyzer is determined by the speed of the process and by whether the instrument is used for information or for automatic control. The second factor is the volume of the sampling system preceding the valve. Even when a circulating sample is used, the system may contain conditioning elements that increase venting requirements; however, the venting rate must not exceed the capacity of other parts of the apparatus, such as the heat exchanger which regulates sample temperature. The third factor is the disposal of vented material (see Section 14). Safety is the primary consideration during disposal, but there are other factors such as recovery of a valuable sample or pressure variations at the vent.

Sampling valves are available for gases, vapors, and liquids. Operational systems for their respective use are described in 15.9.1. For multiple-column services, additional valves are available for switching carrier gas and column contents as shown in Figures 15-2 through 15-4. These systems and the precautions for each type of service are individually discussed in 15.5.1.2 through 15.5.1.4. For requirements which are common to all types of valves see Section 14.

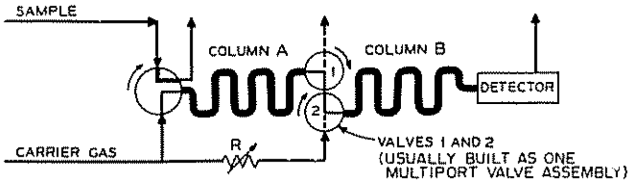


Figure 15-2—Partial Analysis with Forward Flush

15.5.1.2 Gas Service

Sample valves for gas service are simple, reliable, and require fewer precautions than other types. Sample size will usually be between 0.1 milliliters and 2.0 milliliters. Normally, the sample is measured at constant pressure and constant temperature; thus, the mass of successive samples of the same material seldom changes. From the analytical standpoint, pressure variations at the vent are most objectionable, and temperature variations should be minimized. From the safety standpoint, the possibility of hydrocarbon leaks into the enclosure is a major consideration; consequently, pipe thread connections should be minimized.

15.5.1.3 Vapor Service

Sample valves for vapors are identical in construction to those used for gases. So that the gas valve can be used for analyzing vaporized liquids, the vendor supplies a vaporizer which is installed ahead of the valve in the sample stream. The vaporizer, therefore, is an integral part and a critical part of the sample conditioning system. In most vapor analyses, sample volume is similar to that for gas analysis (between 0.1 milliliters and 2.0 milliliters), but in order to maintain sample mass constant, both temperature and pressure levels must be carefully controlled. It is imperative that the purge stream through a vapor sample valve be vented to a constant pressure point. Temperature of the vaporizer system and valve must be high enough to convert all sample components to vapor and maintain them in that state until they are injected into the column.

Two commonly used types of vaporizers are the vaporizing pressure regulator and the capillary vaporizer. The vaporizing pressure regulator is a small diaphragm-operated, pressure-reducing valve equipped with integral electrical or steam heating facilities for vaporizing the sample. It is designed for minimum volume on the vapor side to reduce the possibility of fractionation. A bypass stream (with valve and flow indicator) usually is installed downstream to increase velocity, decrease fractionation, and reduce transportation lag.

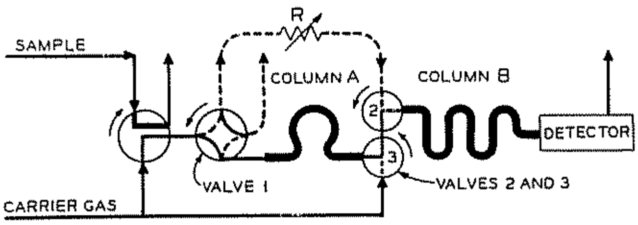


Figure 15-3—Partial Analysis with Back Flush

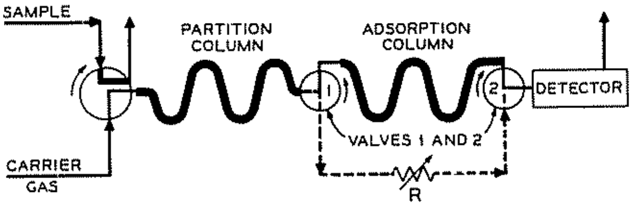


Figure 15-4—Total Analysis for Hydrocarbons and Fixed Gases

The capillary vaporizer is a section of capillary tubing heated to vaporization temperature through which the sample flows continuously. It has the advantage of acting as a flow restriction. Normally, steam is used for heating by means of a tube-in-tube exchanger, where the sample capillary is enclosed in a larger tube with the annulus filled with steam. Saturated-steam temperature must be high enough to vaporize and superheat all sample components, or electrical heating must be used. Electrical heating apparatus are too varied and complicated for discussion here; however, the user must study the method employed to assure that it is adequate from safety and operational standpoints.

For operational and protective purposes, as many of the following devices as are required are installed between the vaporizer and the sampling valve: a pressure gage, a relief valve, a filter, and a flow indicator or controller. These devices must add as little volume as possible to the system and must be arranged to avoid contamination of successive samples. Since all this equipment must be kept hot to prevent condensation, careful attention to heat tracing and jacketing is required. To prevent steam leaks, only the highest quality tubing fittings should be used. The number of fittings should be minimized. Where electrical heating is used, precautions to be observed will be determined by the principle employed, industry codes, statutory requirements, and plant practices. Often the most practical solution is to mount the various sample system components in a heated box.

Where the sample is taken directly as a vapor, the vaporization step is eliminated; however, most of the equipment and all of the precautions noted herein are generally required. Pressure reduction, usually by means of a valve, is normally required. The sample temperature must always be maintained above its condensation point. Deposition of dissolved solids is seldom a problem, but fractionation and chemical reactions can occur.

15.5.1.4 Liquid Service

Because of stringent operating conditions, sample valves for liquids require careful design and precise workmanship. The volume of the sample must be very small (usually between 0.2 and 20 microliters) and must not vary more than ± 1 percent for successive samples. Because some hydrocarbon samples contain (a) high-boiling point materials which vaporize only at excessive temperatures and (b) volatile materials that flash with a slight reduction in pressure, operating pressure in some applications may be 500 pounds per square inch gage or higher. Because sample mass must be kept constant, the valve must be operated at sufficient pressure to prevent flashing. High pressure increases the possibility of leakage; thus, a valve used in high pressure service should not be housed with other parts of the analyzer unless leakage is vented to prevent contaminating the enclosure.

Certain construction features are incorporated in liquid sample valves. The trap section for volume measurement is a straight-through port with no dead volume or crevices. The connection to the column is as short as possible. Because the volume of the trap section is so small, its diameter may restrict venting through the valve and increase lag. Depending upon the volume of the system, an auxiliary parallel vent may be necessary with the upstream connection located as close to sample valve entry as possible.

15.5.1.5 Switching Valves

Many analyses cannot be achieved with a single column. Furthermore, where only partial analysis is necessary, analysis time can be minimized by the use of multiple columns. This technique permits isolating and measuring the desired portion of the sample while the remainder is purged out of the precut or stripping columns shown in Figures 15-2 and 15-3 Occasionally, simple operations of this type can be accomplished by modifying the sample valve to perform the necessary switching. In most cases, however, additional switching valves (sometimes ganged so that more than one can be operated by a single actuator energized by the programmer) must be provided. Such valves are integral parts of the instrument, and the vendor is responsible for their efficiency. However, the user should examine all features to assure their adequacy from safety, maintenance, and operational standpoints. From the analytical point of view, one possible source of error is contamination of successive samples. Contamination can result from dead volume in the valves and connections or condensation in back-flushing operations.

15.5.2 TEMPERATURE-CONTROLLER CHAMBER

The enclosure depicted in Figure 15-1, Element 2, houses all elements for making the component separation and the detector which transmits the electrical signal. The temperature level must be automatically controlled to a fraction of a degree because temperature markedly affects the separating properties of the chromatographic column.

15.5.2.1 Requirements and Developments

Chromatographs may employ single temperature controlled chambers, as depicted in Figure 15-1, or two or more temperature-controlled zones. Single-zone operation is greatly preferred and should be used where possible.

15.5.2.2 Methods For Column Heating

Commercial chromatographs generally employ electrical heating. The most common method depends upon the circulation of heated air inside an insulated enclosure. By means of fans, jets, and similar devices, air is forced across electrical heating elements and circulated within the housing. The temperature sensing element is suspended in the air space near the chromatographic column and the detector.

Another method is the use of a heat sink in the form of a metal mandrel about which the column is coiled in good contact. This assembly is mounted in an insulated enclosure, and the temperature sensing element is located in a well in the mandrel. The electrical heating elements are also embedded in the metal at points appropriate to good heat distribution and uniform temperature.

15.5.2.3 Column Temperature Ranges and Tolerances

The temperature at which a column is operated will be determined by its duty and physical characteristics. Columns for process chromatographs usually operate between 60 degrees Celsius and 200 degrees Celsius. Temperature is usually fixed at some optimum value from which it should not deviate more than ± 1 degree Celsius. A fixed temperature level is not always used. In order to achieve analysis in some cases, column temperature is changed in accordance with a program based on other factors. Where a temperature program is used, the controller must follow it faithfully on both cooling and heating cycles. This requires apparatus of high quality. Temperature-programmed gas chromatographs are seldom used because of their complexity and the extra operating time required to cool the system to the initial temperature.

15.5.2.4 Safety Precautions for Temperature Control Apparatus

From the safety standpoint, the user must be sure that all electrical apparatus is housed in enclosures suitable for the operating conditions. These may vary from one instrument to another; for instance, the use of hydrogen as a carrier gas demands Class 1, Group B[2] design The user should not feel secure simply because all analytical elements are housed in an explosionproof enclosure A small hydrocarbon leak in the enclosure can generate a mixture readily ignited by arcing contacts, heater elements, or other parts of the temperature control system The fact that an explosion is confined does not prevent damage to the instrument. The user is advised to review such possibilities with the vendor.

15.5.3 CHROMATOGRAPHIC COLUMN

The chromatographic column (see Figure 15-1, Element 3) consists of a suitable length of small-bore tubing packed with the materials required for the particular component separation.

15.5.3.1 Requirements and Developments

Column technology has developed into a science, a detailed discussion of which is beyond the scope of this section The major requirements are that the column perform the desired separation and that it be as stable as possible. Stability is important because analysis is time-based. The tubing material, packing solids, and liquid (where used) must be inert to all sample components and the carrier gas. Packing materials must have a high surface area, must pack uniformly, must resist attrition, and must offer low resistance to flow. There are two basic types of chromatographic columns —adsorption columns and partition columns.

15.5.3.2 Adsorption Columns

Adsorption columns are used in gas-solid chromatography for the separation of fixed gases, such as nitrogen, oxygen, carbon dioxide, hydrogen sulfide, and the rare gases. These columns can also be used to separate volatile hydrocarbons in the C_1 to C_3 range. Components are separated by their differences in adsorption, which can be defined as their tendency to adhere to the adsorbent. Packing materials are surface-active solids, such as activated alumina, charcoal, molecular sieves, silica gel, and synthetic zeolites. These materials are operated dry, that is, they are not coated with a liquid. For petroleum service, adsorption columns have a limited range of application except where they are used in combination with other columns.

15.5.3.3 Partition Columns

Partition columns are used in gas-liquid chromatography for separating complex hydrocarbon samples. The separating medium (the column packing) is a granular solid impregnated with a liquid substrate that is essentially nonvolatile under the conditions of use. The packing appears to be dry but in reality exposes a large liquid surface to the vaporized sample components as they are borne through the column by the carrier gas. The sample components are partitioned between the gas and liquid phases. Those components least soluble in the liquid pass rapidly through the columns and emerge early; those components most soluble in the liquid are retarded and emerge later. The most volatile components generally emerge earliest. The versatility of gas-liquid chromatography is a result of the large variety of liquids (stationary liquid, partitioning liquid) that can be used to obtain different separations.

The granular solid (solid support) used to support the liquid substrate may be crushed firebrick, celite, or other solids of moderate surface area (1 to 4 square meters per gram). It may be treated to reduce residual adsorptive effects.

The stationary liquid substrate must have a very low vapor pressure at the operating temperature so that the column may have a long service life.

15.5.3.4 Polymer Packings

Some granular polymeric materials are used as column packings These yield separations much like gas-liquid chromatography but do not require additional solid support

15.5.3.5 Special Columns

Special columns for specific services are variations of the two basic types. The capillary column actually is a partition column in which the wall of hollow tubing, of very small diameter, serves as the solid support and is coated with the substrate Columns are also built in which an active, rather than inert, solid is used for support, and only part of it is coated with the substrate. Despite the fact that it is contained in a single tube, this is actually a column combination.

15.5.3.6 Column Combinations and Arrangements

In general, only relatively simple analyses can be made with a single column. More complicated samples require two or more columns Multiple columns are also used for other reasons. Column contamination can be minimized by multicolumn techniques. Where only partial analysis is needed, analysis time can be materially

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[2] Classified locations as defined in the *National Electrical Code*, Article 500.

reduced with multiple columns. No attempt is made to discuss all possible column arrangements because their number is limited only by the complexity of analysis and the ingenuity of the designer. Diagrams of dual-column arrangements for three possible services are shown in Figures 15-2 through 15-4. Because they are examples, switching valves and other equipment are schematic for convenience; the methods depicted are not intended to be the utmost in simplicity.

Figure 15-2 depicts a system sometimes used to reduce analysis time for the light components in a complex sample (for example, components through the C_4's in a gasoline stream). Operation is in the following sequence. After sample injection, the sample valve returns to the position shown on the illustration. Carrier gas and sample then flow along the solid lines until the light ends have passed through Valves 1 and 2 and into Column B. On a time basis, the programmer then rotates switching Valves 1 and 2 in the direction shown to establish the paths depicted by the dotted lines. Carrier gas continues to flow into Column A and flushes the heavier components remaining therein out through the vent in Valve 1. Simultaneously, an auxiliary carrier stream enters Column B through the dotted path in Valve 2 and elutes the lighter components to be analyzed. The Resistance R in the auxiliary carrier line approximates the pressure drop of Column A but is not always required.

The system shown in Figure 15-3 reduces analysis time as described in the preceding example, but it also prevents heavy contaminants from damaging the analysis column. Operation is similar to the system shown in Figure 15-2 until the components to be separated are in Column B. The programmer operates Valves 1, 2, and 3 as shown, and the streams flow along the dotted paths. The carrier gas continues to flow through the sample valve, the upper dotted path in Valve 1, Resistance R, the dotted path in Valve 2, and elutes the components in Column B through the detector. Simultaneously, an auxiliary carrier stream enters Valve 3 on the dotted path and flows backward through Column A to flush the heavy components out through the lower dotted path in Valve 1 and the vent. Rate of flow of the backflush carrier stream is established so that Column A is completely flushed of all components by the time the material in Column B is analyzed.

In Figure 15-4, the carrier gas and the sample flow along the solid lines until the fixed gases, which are not separated by the partition column, flow into the adsorption column. The programmer rotates Valves 1 and 2 in the directions shown and traps the fixed gases in this column. Carrier gas continues to flow and elutes the hydrocarbons in the partition column through the dotted path in Valve 1, Resistance R, dotted path in Valve 2, and the detector. When hydrocarbon analysis is complete, the programmer rotates Valves 1 and 2 back to their original positions, and carrier gas flows along the solid lines to elute the fixed gases previously trapped in the adsorption column to complete the analysis.

15.5.4 CARRIER GAS SYSTEM

The carrier gas system depicted in Figure 15-1, Element 4, is schematic and represents one of several arrangements which may be used. This system consists of a bottled supply of suitable carrier gas with instrumentation for close control of gas flow.

15.5.4.1 Requirements and Developments

The carrier gas and its control system constitute a very important element in a process chromatograph because they not only affect resolution and detection, but also establish the elution times on which analysis is based.

Theoretically, any gas can be used as a carrier if it is pure and inert to the column and the sample. In fact, however, a carrier is selected for its effect on resolution and detection sensitivity. The latter is the more important because resolution usually can be achieved by other means. To achieve sensitivity, the carrier gas and detector must be chosen so that eluted components generate large signals. For this reason, gases with high thermal conductivities, such as helium and hydrogen, are used with the thermal-conductivity detector; argon is used with the beta-ray detector; air is used with the catalytic-combustion detector; and so forth. Any one of several carrier gases may be used with the hydrogen flame ionization detector, which also requires bottled, oil-free air and hydrogen for operation. No carrier-detector combination is completely satisfactory for all analyses. The popular helium thermal conductivity combination presents problems if the sample contains varying amounts of hydrogen and it is a component requiring analysis. Under certain circumstances, special carrier gases must be used for other reasons. For instance, when helium is used as carrier in the analysis of combustion flue gases, argon appears with oxygen as a single peak. Since oxygen is a component of interest, argon (requiring a more sensitive detector) can be used as the carrier. As a result, argon interference on the oxygen peak is eliminated and a true measurement is obtained.

Selection of the carrier is the vendor's responsibility, but the user must assume responsibility for purity of the gases employed as carriers. Normally bottled gases are used and purity is relatively high; however, particularly for trace analysis, extra care should be taken to avoid contaminants that might affect measurement. Process gases should never be used as carriers unless they are continuously analyzed for purity. Where air is used, it should be dry and free of oil and other contaminants.

15.5.4.2 Carrier Control System

Rate of flow of carrier gas has two effects on analysis which must be considered. The first is its effect on signal amplitude; the manufacturer will normally select a flow rate for optimum signal. The other is its effect on elution time; once a rate has been chosen, it must be maintained constant. Because carrier flow rate is critical to proper operation, it is essential that means be provided for measuring and adjusting flows without opening the oven door and upsetting the temperature control system. It is recommended that instruments be selected which provide for these procedures without opening the insulated case.

The first item in the control system is a pressure-reducing valve at, or near, the storage bottles to reduce pressure to that required for the analysis. A high-quality, multistage regulator is preferable, and it should be the nonbleed type to prevent wasting carrier gas.

To control the rate of flow of carrier gas, the analyzer may employ one of two methods It may utilize a precision pressure regulator, which maintains constant pressure upstream of restrictions (usually variable, such as small needle valves), supplying carrier gas to the column and the reference side of the detector. Alternatively, constant flow may be accomplished by a small but high-quality differential pressure regulator which maintains a constant pressure drop across a (usually variable) restriction (see the unit shown in Section 14). By these means, carrier flow is maintained constant as long as the instrument outlet pressure remains constant

A miniature rotameter is sometimes included as a flow indicator, but it should not be regarded as an accurate flow measuring device. Absolute flow should be measured at the vent by means of a soap bubble flowmeter, the water displacement method, a calibrated flowmeter, or one of the other methods described in the literature.

15.5.4.3 Precautions for Carrier Gas System

From the analytical standpoint, the primary consideration for the carrier gas system is to assure that it is accurate and flexible enough for the service involved. Threaded connections should be minimized, and where used, they must not be lubricated or doped with materials that can introduce contaminants into the system. All gas lines and associated equipment from the bottle supply to the chromatograph must be thoroughly cleaned and dried before use. Special precautions must be taken to insure clean lines since the chromatograph is equipped with highly sensitive detectors. From the safety standpoint, hydrogen is the only commonly used carrier gas that requires special attention. Fittings used with hydrogen must meet Class 1, Group B requirements.

15.5.5 DETECTOR

The detector is the element located at the outlet of the chromatographic column which reveals the presence and concentration of the separated components in the carrier gas. It is an extremely sensitive device since it must be able to detect traces of components in the carrier gas. A wide variety of detectors is available, but only a few types are in common use.

The thermal conductivity cell is the most widely used detector in process chromatographs. The filament type shown in Figure 15-1, Element 5, is typical of these devices. The cell is arranged and operates in the following sequence. Two separate gas passages, one for reference and one for measurement, are each equipped with filaments which are sensitive, electrical resistance elements. The filaments are electrically matched, as nearly as possible, and are connected to form the arms of a Wheatstone bridge which is energized by a precise power supply. Cells may have two or four filaments, depending upon the sensitivity required.

The filament temperature will rise to some value determined by the flowing current, thermal conductivity of the surrounding gas, and the temperature difference between the cell walls and the filament. Thus, heat removal from each side of the cell is a function of the thermal conductivity of the gas in that side. When connected to the recorder, the detector behaves according to the following description. With pure carrier gas flowing through both sides of the detector, there is no difference in conductivity in the cells and the recorder pen follows the baseline on the chart. As the first component is eluted, it starts passing through the sample side, changing thermal conductivity and thus unbalancing the bridge; the recorder pen starts upward from the baseline on the chart. As component concentration increases, the pen proceeds farther out on the chart to some value for maximum concentration. Then as concentration starts to decrease, the pen moves downward. When all of the component has passed through the cell, the pen arrives at the baseline where it remains until the second component starts passing through, and the peaking process is repeated. If during this operation, the chart is running at some predetermined speed, the pen traces a more or less triangular-shaped peak which represents a plot of component concentration in the carrier versus time.

15.5.5.1 Requirements and Developments

The detector is the element in the chromatograph most affected by environmental changes. A good detector should respond rapidly. It should respond repeatably with regard to component composition and concentrations and linearly to changes in component concentration. It also should operate stably without baseline drift.

A detector for a process chromatograph should be simple in construction and rugged enough to withstand vibration and other process conditions. It should be matched to the carrier gas for high sensitivity and should not respond appreciably to small changes in carrier flow rate. It should be operable over a wide temperature range and should be reasonably insensitive to small temperature changes. Finally, its output signal should facilitate attenuation or amplification for the readout device. These requirements limit detectors for commercially available instruments to the two fundamental types which are discussed in 15.5.5.2 and 15.5.5.3, with emphasis on the precautions to be observed. Supplying a suitable detector is the vendor's responsibility, but because they affect installation requirements, the user should know their limitations.

15.5.5.2 Thermal Conductivity Detectors

The thermal conductivity cell described in 15.5.5 most nearly conforms to the ideal detector, but it has limitations. When ruggedness is sacrificed for sensitivity, the detector, particularly after embrittlement from operation, will not withstand shock and is vulnerable to vibration. The cell is sensitive to temperature changes even when the arms of the Wheatstone bridge are closely matched. Replacement filaments should be installed in matched pairs to maintain a balanced bridge. This type of circuit requires accurate temperature control for the detector block. Usually, an automatic feature is also included which balances the bridge after each analysis; however, it cannot compensate for a poor temperature controller. Special precautions must be taken so that random conditions which cause variations in thermal conductivity in the two sides of the cell are avoided. For instance, pressure in the reference and sample sides of the cell should always be equal; thus, they should be vented to the same pressure point. Another fundamental characteristic of the thermal conductivity cell is that because the conductivity characteristics of all components in a hydrocarbon sample are almost never identical, response to different components is not identical. Calibration normally overcomes this limitation.

Two available types of thermal conductivity detectors are the filament type and the thermistor type. The filament type operates at relatively high current flows (up to 0.5 amp with helium and hydrogen), and sensitivity increases exponentially with increased current flow. The filament type can be used at relatively high temperatures because sensitivity is affected only slightly by increased operating temperature. However, it has low impedance and requires careful attention to wiring and connections. Its principal disadvantage is reduced sensitivity which occurs at lower temperatures and with carrier gases having lower thermal conductivity than helium.

The thermistor type has a somewhat higher sensitivity than filaments at relatively low temperatures (for example, 60 degrees Celsius) because the temperature coefficient of resistance is much greater in that range. However, sensitivity decreases rapidly as operating temperature increases. Furthermore, current flow in thermistor bridges is limited to low values (approximately 0.02 amp with impedance levels of a few hundred ohms). Unlike the filament type, sensitivity of the thermistor type decreases with increased current flow beyond optimum. Combination of these factors causes thermistor detectors to be more sensitive than the filament type only up to about 90 to 110 degrees Celsius. The gas density balance is a special variant of the thermistor/thermal conductivity detector which, in some cases, offers even more sensitivity plus considerable resistance to element fouling. Similar readout devices are used with both types of detectors.

15.5.5.3 Flame Ionization Detectors

This detector utilizes a small hydrogen flame to burn and ionize specific components of the sample. An electrode in close proximity to the flame collects the ions and produces an electrical signal. Amplification is required to convert this weak signal to a level suitable for most readout devices. Several variations of this detector can be used.

In one type of detector, hydrogen is used as the carrier gas and subsequently is burned at a capillary tip to supply the ionizing flame. In another type, a separate hydrogen flame is continuously maintained and carrier gas and elutants pass through the flame. Almost any type of carrier gas can be used in this system, and this is one of its advantages. The principal advantages of the flame ionization detector, however, are its very high sensitivity and its extremely fast speed of response. These factors permit precise analysis of very small samples (50 to 100 microliters of gas); they also enable the analysis of larger samples for a trace component in the parts-per-billion range.

One disadvantage of this detector is the added bottled gas requirement. Even when hydrogen is used as the carrier, either bottled oxygen or bottled hydrogen-free air is required for the hydrogen flame. An automatic igniter or hydrogen shutoff, or both, is required in the event of flame failure, and this complicates the apparatus. The presence of the flame in the instrument is a disadvantage from the safety standpoint, and a thorough study is necessary to assure that safety requirements are met. The fact that an expensive amplifier is required with this detector is a disadvantage from the cost standpoint. Finally, with this detector, not all combustible components can be determined.

15.5.5.4 Special Detectors for Specific Services

Although the two types of detectors described are most widely used on commercially available instruments, special detectors can sometimes be obtained for specific services.

In most cases they are more expensive, but often the added expense is offset by the simplicity of the other elements in the system. An example is the use of an ultraviolet detector for measuring the aromatics in a complex stream. Because it is specifically designed for aromatics, only the simplest sampling apparatus, carrier gas system, and chromatographic column are required. Savings in installation and maintenance costs are often a factor. Another example is the catalytic combustion detector for use with hydrocarbons and other catalytically combustible components. Detectors are available which give much greater response to some types of compounds than to others. Examples are the flame photometric detector, which responds to sulfur or phosphorus compounds, and the electron capture detector, which gives a strong response to chlorine compounds. Such selective detectors are finding increased use in air pollution monitoring and in trace contaminant analysis. The user may profit by discussing special detector possibilities with the vendor.

15.5.6 READOUT DEVICE

A strip-chart recorder (shown in Figure 15-1, Element 6) is the basic readout device used for process chromatographs for two reasons.

1. The electrical signal from the detector can be amplified or attenuated before recording, and this supplies flexibility difficult to achieve with other apparatus.
2. The chromatogram made on this recorder is the basic measurement to which all other forms of readout or display must be referred not only for accuracy, but also to detect evidence of inadequate analysis, such as tailing of a component.

Attenuation of the detector signals is usually necessary so that a component present in a high concentration can be measured with the same recorder used for a trace component. For example, one component (such as, propylene in a sample) may be present in only a small percentage, but because it is a component of major interest, changes of only a fraction of a percent must be detected. To supply a propylene peak large enough to reveal small changes, a sensitive recorder (for example, 1 millivolt full scale) may be required. Such a recorder is too sensitive for signals (50 millivolts) generated by major components, and the peaks of major components will go off-scale unless their signals are reduced to values within recorder range. The attenuator performs this function. Although other types of circuits can be used, the most popular is a voltage-divider circuit made up of resistance elements which divide the signal. In process instruments, suitable resistance elements are provided for each recorded component so that its response can be calibrated in appropriate units. As a component is eluted, its attenuating elements are switched in automatically on a time basis by the programmer wherein the resistances are mounted.

A process stream chromatograph should never be installed unless it is equipped for making a complete chromatogram, regardless of whether peak height or integrated peak area is used for measurement. Both of these methods of measurement are discussed from the standpoint of operational advantages and limitations. The reader is referred to the literature for a discussion of relative accuracy.

15.5.6.1 Peak-Height Readout Devices

The simplest form of readout is a chromatogram on which all component peak heights of interest have been calibrated quantitatively. By selecting an appropriate chart speed, total analysis can be made and interpreted for a stream consisting of only a few components. However, if a complete chromatogram is too complex for interpretation, it is possible to modify the recorder to produce a bar graph of selected components. This is accomplished by a stepping mechanism which advances the chart one increment (approximately ⅛ inch), after which it remains stationary. During the time the chart is stopped, the pen moves to maximum peak height for the selected component and returns to the baseline where it remains until the next chart step. Chart stepping and component selection are performed by the automatic programmer on a time basis. Bar-graph display is best suited to partial analysis of a complex stream where the number of components must be minimized for chart readability. It is advisable to periodically compare the peak heights on the bar graph with values derived from a chromatogram.

It is also possible to utilize a multiple-point recording potentiometer for similar analysis. Specific recorder numbers are assigned to selected components with signals switched by the programmer on a time basis. The recorder is equipped with a print-on-balance mechanism, and a record of 12 or more components can be obtained. Total analysis of simple streams is possible with this method. However, if the recorder numbers are grouped near each other on the chart, or are roped back and forth across each other, it is difficult to follow the trend of a particular component. Another disadvantage of this type of readout is that a complete chromatogram cannot be run on the same potentiometer except with specially equipped recorders.

In automatic control and single-component analysis, another type of peak-height measurement is sometimes employed. This type consists of a peak-picking device which transmits the peak value of a component to a secondary instrument where it is held until the next analysis. In trend recording, this results in a continuous record of intermittent analyses. This readout device can be obtained with either pneumatic or electrical output. Detailed discussion of the various types of peak-pickers is beyond the scope of this section. Regardless of type, the operating principle is usually similar in that as analysis proceeds on a time basis, the programmer energizes the peak-picker when a peak of interest is about to be eluted. When the maximum value of the peak is reached, its signal is stored or transmitted, or both, to a secondary instrument. The device is then readied for the next component of interest. With proper storage facilities, the same peak-picker can be used for several components in the same analysis; however, all peaks are time-based, and a chromatogram displaying these peaks is necessary for checking accuracy.

Another frequent use of peak-pickers is to incorporate standard miniature panel recorders which continuously display trends of only those components of interest. One recording strip-chart potentiometer may be utilized with several chromatographs plugged in as needed to provide full chromatograms for operating or maintenance needs.

15.5.6.2 Area Management Readout Devices

Area integrating devices are sometimes incorporated in process chromatographs which are used for several streams or to which a variety of bomb samples can be attached manually. The many different peak shapes encountered in such service sometimes prevent accurate measurement by peak height. In such cases, a simple mechanical integrator attached directly to the recording potentiometer is often satisfactory. Like the integrator used on laboratory recorders, it measures the area under each selected peak, and displays results either by pipped markings on the chart, or by digits on some form of counter. For an accuracy check, a chromatogram usually can be made on the same recorder.

Sometimes an application will require an integrator on a chromatograph for repetitive analyses of similar samples. The integrator may be required by unusual peak geometry that prohibits peak-height measurement; but more often it is needed for some special requirement, such as summing or totalizing a group of component peaks (for example, totalizing all aromatics in a complex sample). In such an operation, the programmer energizes the integrator when the first aromatic peak is about to be eluted. Integration proceeds until detector output decreases to baseline value. At this point the programmer de-energizes the integrator. The total value of accumulated increments is stored until elution of the second aromatic begins. Again the programmer "gates" in the integrator for duration of the peak, and it adds increments of area to the total already in storage. This process is repeated for each aromatic peak, and the final stored value is the sum of all aromatic peak areas. Both pneumatic and electronic integrators are available for such services, and output can be either analog or digital.

All-electronic integrators with coded digital output can also be obtained for process chromatographs. As an example, one unit consists of a very accurate voltage-to-digital transducer that electronically converts detector signals to pulses which drive a coded electronic counter. Digital output can be displayed, printed out, or used to feed other equipment such as a computer. Usually a separate recorder is included for making a chromatogram.

15.5.7 PROGRAMMER

The programmer commands that certain intermittent operations be performed at preset times during the analysis cycle. The programmer,[3] shown in Figure 15-1, Element 7, merely initiates the sample injection at the proper time and attenuates detector signals. Thus, it is a relatively simple type when compared with programmers for complex applications.

15.5.7.1 Requirements and Developments

The programmer for a process chromatograph should provide for both automatic and manual operation. With fully automatic operation, the programmer performs as many of the following functions as are required: sample injection, signal attenuation, bridge-balance standardization, column switching, stream selection, peak picking or integration, and data presentation. In all cases, the option should be included for making a chromatogram or using the other form of readout provided.

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[3] The term *control unit* is avoided to prevent confusion with the controlling apparatus of a chromatograph used for automatic process control

In manual operation, all functions are initiated by a human operator. Manual operation is necessary for calibration and maintenance, and all adjustments required for running a complete chromatogram should be made available to the operator. This availability is particularly necessary where purging is required, because opening the case inactivates the purge. Continuously adjustable attenuation should be provided for all recorded components so that each can be made a direct reading. An indicator should be included to show the progress of the analysis cycle. Provisions for measuring detector current and voltage and for adjusting bridge balance should be supplied to facilitate troubleshooting and calibration.

Chromatograph manufacturers use different approaches in supplying standard, mass-produced programmers to meet requirements for applications of varying complexity One may design a basic programmer for simple analysis (see Figure 15-2) to which modules can be added for as many of the functions as are required for the application. Another manufacturer may offer several models, each with increasing capability, that cover all applications from simple single-stream analysis to multistream, multicomponent, multicolumn service Because a combination of these approaches is usually adopted for flexibility, a programmer for any application can usually be supplied from stock. Before purchase, the user should study the proposed programmer to assure that it meets all analytical and operational requirements and incorporates accessible facilities for maintenance and calibration adjustments.

The user is also advised to study construction of the proposed programmer Usually, the prorgammer is a complex device compactly filled with motors, cams, gears, switches, attenuating potentiometers, and other apparatus subject to wear. For reliability, construction materials must be of high quality, and moving parts must be arranged to minimize friction Electrical apparatus must be critically examined for both reliability and safety. The user must determine electrical classification of the intended location and be sure that the proposed programmer meets all requirements. Purging (see Section 14) normally must be used for safety in Class I, Group D, Division 1 areas and the precautions for purging must be adhered to rigidly For Division 2 areas, programmers are commercially available that avoid arcing contacts by use of sealed relays and switches. Use of sealed contacts is often advisable for reliability regardless of area classification To provide a clean atmosphere, purging is often used even in safe areas.

The new generation of chromatograph programmers uses logic circuitry to perform switching functions electronically. The majority are intrinsically safe but may require purging to keep printed circuit board contacts clean.

Programming of gas chromatographs can also be achieved by digital computers. The use of dedicated computers for this function most likely would occur in extremely complicated analyses or in installations involving a number of gas chromatographs. A computer, shared for other functions, could be utilized if its characteristics are suitable for gas chromatograph programming.

15.6 Packaging the Chromatograph

To make the elements of the chromatograph suitable for the process environment, they are usually packaged in two sections—the analyzer section (see Figure 15-1, Elements 1 through 5) and the readout device and programmer section (see Figure 15-1, Elements 6 and 7). As noted in Section 14, the analyzer section should be located at, or as near as feasible to, the sample point. For this reason it is mounted in a weatherproof enclosure suitable for outdoor installation. In addition, all elements involving electrical apparatus are mounted in explosionproof housings even when inside the weatherproof enclosure (which can be a small hazardous area itself). Some vendors prefer to mount all elements comprising the analyzer section in a large, standard explosionproof housing. Where they are so constructed, instruments should be selected which provide for measurement and adjustment of critical operating variables, such as carrier flow rate and temperature controller set points, without opening the explosionproof analyzer housing.

To be convenient to the operator, the display unit and the programmer are usually located at the control center on or near the control panel. Both of these elements incorporate electrical apparatus, but it is neither feasible nor desirable to enclose them in explosionproof housings. Purging should be provided if required.

15.7 Operating Environment

Suitable operating environments must be provided for the two major portions of the chromatograph. The limitations and precautions to be observed for the various elements have been outlined in 15 6. The user must study all elements of a proposed instrument and determine the features requiring special installation procedures.

15.7.1 ANALYZER SECTION

Although it is sometimes necessary to locate the analyzer at the sample point, the user is allowed a certain amount of latitude in location by using the circulating sample referred to in Section 14. In either case, it is necessary to protect the instrument from the conditions and sources of damage as outlined in Section 14. Many users locate one or more analyzers in walk-in shelters which are designed to provide an improved environment both for the analyzers and for operating and maintenance personnel.

Before an analyzer location is selected, safety precautions, sample procurement, processing, and disposal should be thoroughly reviewed. A review of this material will serve as a valuable guide for installation procedures; however, no standard or recommended practice can substitute for engineering skill and experience coupled with studious attention to detail.

15.7.2 READOUT AND PROGRAMMER SECTION

The readout device and programmer should be considered as a unit and should be mounted in a location convenient for plant operations and maintenance. Particularly when a reference chromatogram is made, these elements are analogous to a recorder-controller, because results of manipulating the programmer (controller) can be observed on the display section (recorder). If the chromatograph is used for trend recording or automatic control, the secondary instrument will obviously be installed at the control center.

Precautions to be observed with the basic potentiometer and programmer are outlined in 15.5.6 and 15.5.7. General safety precautions are presented in Section 14. In addition, the following description is offered as an example of the details necessary for installation.

The programmer is usually connected by a closed conduit to a number of devices at the analyzer location. In the case of multiple stream sampling systems, the stream selector (normally a part of the programmer) is sometimes connected to solenoid valves as described in 15.10.1. A leak may develop in the barrier between the armature and solenoid coil of such a valve permitting process fluid to enter the coil housing. The *National Electrical Code* (NEC) requires a seal between such a coil housing and the conduit and also at the conduit entry to the instrument or control center. The electrical seal, however, is designed only to prevent flame propagation from an explosion and not to hold process pressure. Cases are on record where an NEC seal leaked process fluid into the conduit and to the programmer where it created an explosive atmosphere. Similarly, damage can also result from a nonflammable sample if it contains components that will attack parts of the instrument. For instance, hydrogen sulfide in a sample has been known to destroy copper wiring and bare switch contacts in a programmer. To prevent such occurrences, some users require double seals wherein the standard electrical compound is reinforced by a nonsetting plastic material that seals against leakage. Merely designing such a system is not sufficient; a thorough inspection to assure good workmanship and adequate safety is required after construction.

15.8 Influence of Operating Duty on Installation

The duty a chromatograph must perform is the most important factor to be considered in its installation. The complexity of the instrument itself is determined by whether it is used for automatic control or only for analysis. If it is used for analysis only, the numbers and character of the streams to be analyzed by it will also be factors. These factors determine the degree to which a sample must be treated or conditioned before analysis, and this, in turn, determines the details of the sampling system.

15.9 Single-Stream Analysis

A single stream can be analyzed for a single component, a group of components, or for all of its components. Single-component analysis is normally used to measure a trace contaminant or a key component that determines process efficiency. Rapid analysis may or may not be required. The elements of the chromatograph are usually selected for accuracy in measurement of the key component even if total resolution or other features must be sacrificed. The sampling system is designed for maintaining the representative status of the key component.

Multiple-component analysis requires good resolution for all components to be recorded and may require a precut column (as in Figures 15-2 and 15-3) for protection of the analysis column. The sampling system must be designed to remove all contaminants that cannot be eliminated by such a protective column.

Complete analysis normally can be justified only for simple streams. Because of resolution requirements, multiple columns (as in Figure 15-4) may be required and, in some cases, dual detectors may be needed. The sampling system requires careful design and installation to assure removal of solids, water, and similar substances without changing sample composition.

15.9.1 SAMPLING SYSTEMS FOR SINGLE-STREAM ANALYSIS

Proper design and construction of the sampling system are the most important features in the installation of a chromatograph; they should not be attempted without prior study and cooperation. Repeated, thorough analysis of the sample under flowing conditions is necessary to assure that no component or contaminant is overlooked, and this calls for the cooperation of laboratory analysts. Technologists, process design engineers, and the plant operator also have an interest in the analyzer, and each can make a contribution. The best sampling systems are the result of the combined efforts of such people. The instrument manufacturer should also be consulted in the design stage. Most vendors can supply components (see Section 14) specifically designed for chromatograph sampling systems. Some vendors combine operations, such as filtering and coalescing, in one component. Prior experience will also make the vendor a valuable member of the design team.

The designer will benefit from a thorough study of the literature on sampling systems (see reference list, Section 14). However, he should not expect to find the exact answer to a specific problem. A remark in Section 14 is reiterated here for emphasis: there is no standard sampling system. The designer should know the condition and contaminants of the sample from exploratory analyses, and he must supply all the system components dictated thereby. Section 14 discusses line velocities, component volumes, and other features concerned with lags and should be reviewed. Construction materials and other pertinent details are also discussed.

Examples of sampling systems for single-stream analysis are shown in Figures 15-5 through 15-7. Similar arrangements have been used for the analysis described under each illustration; however, they may not be satisfactory for other analyses. Furthermore, presentation is not identical to actual arrangement. For clarity, only single components with relatively long connections are shown, whereas in an actual system combination components coupled as closely as possible are used.

15.9.1.1 Sampling Systems for Single-Stream Gas Analysis

The system shown in Figure 15-5 was designed for a medium pressure stream of gas that contained solids and required further conditioning. Because the higher pressure helped in conditioning, the conditioning component was located ahead of the pressure-reducing valve. Depending upon the application, it may be desirable to locate the conditioner (which can be any one of the items noted in Section 14) behind the reducing valve. Connections for the pressure gage and relief valve are taken from the bypass or vent line to prevent contaminating the main stream from their dead volumes. Careful attention to such small details often determines the success of a sampling system. The standard sample is introduced via a three-way valve which could be remotely actuated by a separate switch at the programmer. Actuation positively shuts off the process sample and allows the standard sample to flow through the instrument. A three-way valve with no dead volume should be used to prevent diffusion and contamination. Where a circulating sample is used (as depicted in Figure 15-5), all of the sampling system components can usually be mounted on a rack at the analyzer. In some instances, however, it is preferable to mount the pressure-reducing valve at the sample point to increase velocity and reduce lag. A study should be made for each application.

15.9.1.2 Sampling Systems for Single-Stream Vaporized Liquid Analysis

The system shown in Figure 15-6 is satisfactory for a cool, rather volatile liquid carrying free water and

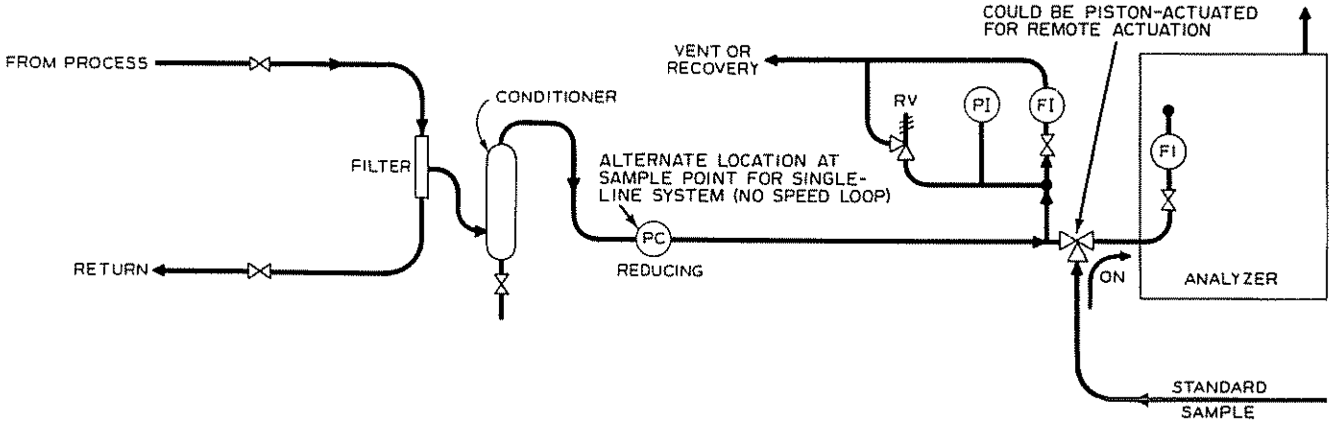

Figure 15-5—Sampling System for Single-Stream Analysis

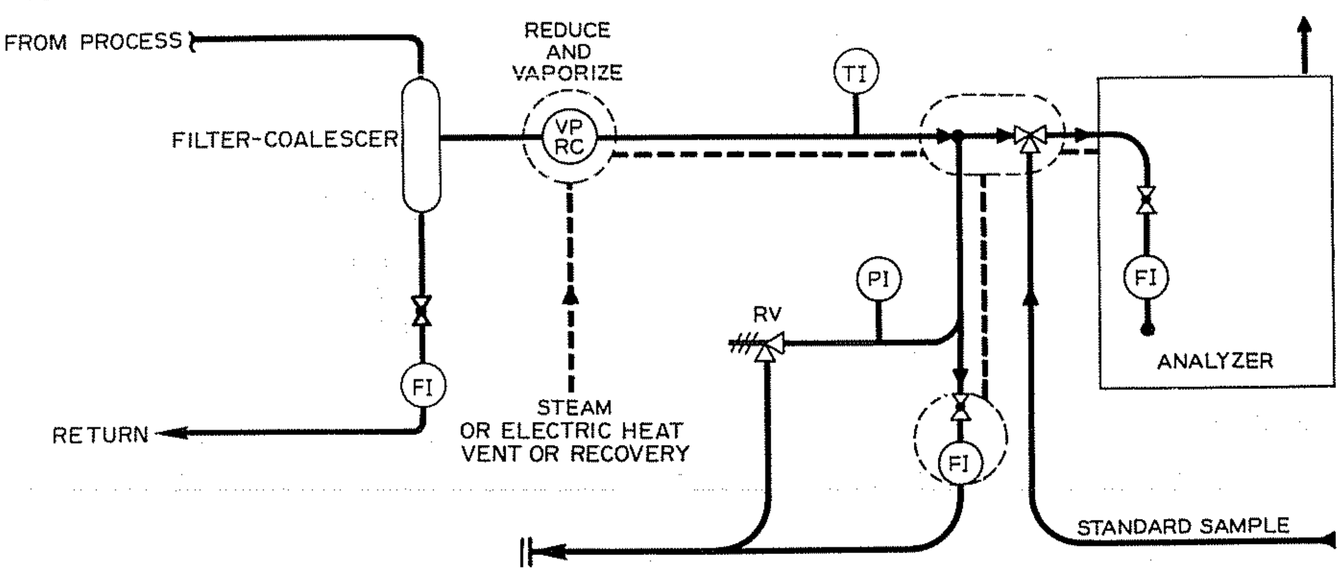

Figure 15-6—Sampling System for Single-Stream Vaporized Liquid Analysis

other materials which can be removed by simple treatment. The filter-coalescer is in the circulating stream to take advantage of velocity which can be set to an optimum by the hand valve and flow indicator. The water-free stream from the side outlet of the filter-coalescer goes to the heated, vaporizing, pressure-reducing valve. Once vaporized, the sample is maintained above condensing temperature by heat tracing, jacketing, or if temperature level permits, by mounting all items in a heated cabinet adjacent to the analyzer. References and notes on the other items are similar to those in the preceding paragraph except for one point. In addition to manifolding the relief valve and pressure gage to the vent line, the vent line is run downward from the sample line to prevent condensed material from running backward down the vent and contaminating the sample.

15.9.1.3 Sampling Systems for Single-Stream Liquid Analysis

The system shown in Figure 15-7 has been used for a high-pressure liquid sample from one of the bottom trays of a rectified absorber column. As such, it is a hot stream of heavy absorption oil carrying considerable free water and saturated with light materials that vaporize on pressure reduction. After the stream has been cooled, the water is coalesced and removed through the hand valve and flow indicator at the bottom of the coalescer Cooler and coalescer may be mounted at any convenient location with the circulating, water-free stream run to a rack or framework at the analyzer. If an element with very fine porosity is used, the in-line filter can serve as the final filter. Again, all auxiliary connections are taken from the vented stream to prevent contamination. Velocity of this stream is set by the hand valve and flow indicator. A back-pressure regulator and flow indicator are used to prevent flashing and to regulate vent rate through the sample injection valve. This means that the sample valve must operate at essentially process pressure and must be so rated; thus, precautions noted in 15.5.1.4 should be observed. With cooling, stream composition may permit some pressure reduction without flashing. If this is possible, a reducing regulator should be mounted in the circulating stream immediately ahead of the in-line filter in place of the back-pressure regulator shown downstream of the sample injection valve.

15.10 Multiple-Stream Analysis

Partial analysis of multiple streams with a single instrument is quite common, but usually this type of analysis requires a sacrifice in accuracy of one or more components to achieve the desired accuracy in others. An example is a chromatograph which measures tops and bottoms from a fractionating column. Usually this is a key component analysis because normally there is one component in the tops and another in the bottoms which is indicative of operating efficiency. For example, it is desirable to minimize nC_4 in the tops of a deisobutanizer column and at the same time minimize iC_4 in the bottoms. A chromatograph with a minimum number of attenuators can be used for both analyses by setting the iC_4 and nC_4 attenuators to high sensitivities so that each will supply a readable peak at low concentration. The nC_4 peak will go off the chart when the bottoms are analyzed, as will the iC_4 peak when the tops are ana-

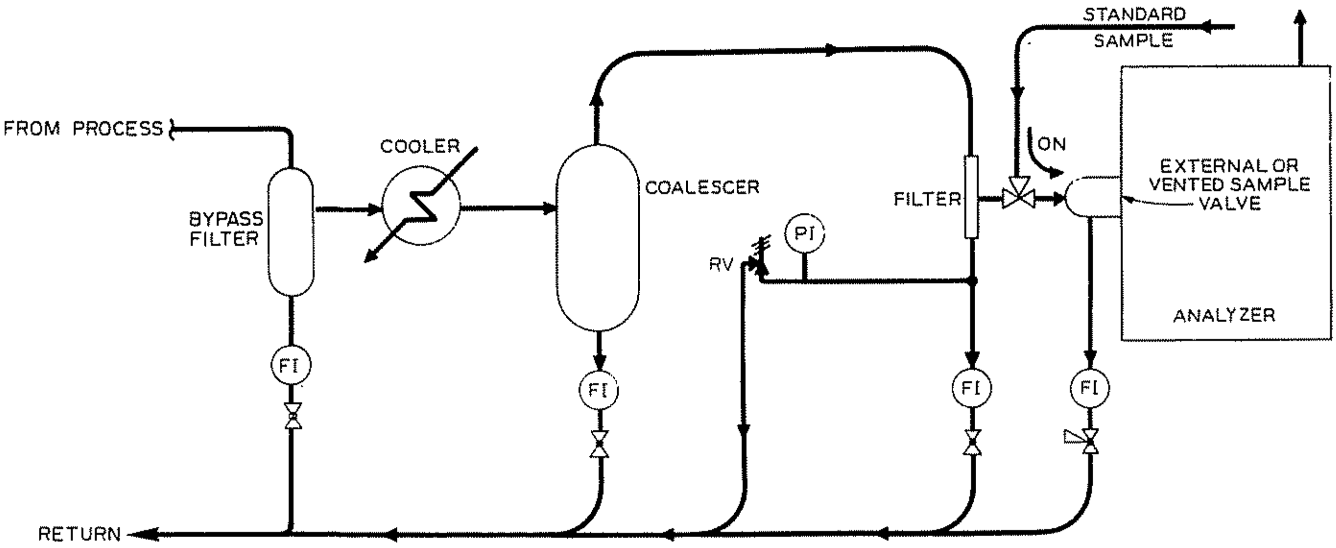

Figure 15-7—Sampling System for Single-Stream Liquid Analysis

lyzed, but this is of little importance. Likewise, an attenuator setting to give a significant reading of the C_3's in the tops will be of value despite the fact that it will register zero on bottoms analysis. Usually, a similar compromise will be necessary for any multistream chromatograph in order to avoid an expensive instrument and expensive installation.

Because of the resolution required, complete analysis of multiple streams should not be attempted with a single chromatograph unless all of the streams are simple and similar in composition. Attenuation of peaks is also a problem unless additions are incorporated in the programmer. The additional instrument and sampling cost, plus reduced analysis frequency, is often sufficient to justify separate instruments for the streams particularly when reliability and maintenance are considered. The user is advised to study this possibility even when streams are relatively similar.

15.10.1 SAMPLING SYSTEMS FOR MULTIPLE-STREAM ANALYSIS

Many technical articles have been published on the precautions to be observed in designing sampling systems for multistream analyzers. These precautions are particularly important when installing process chromatographs; however, except for detail, they can all be covered if two fundamental principles are observed. The first principle requires that each stream should be treated as if it were the only sample to the analyzer. Each stream should flow continuously, through its own filter, coalescer, regulator, and other conditioning devices. In short, all the remarks concerning single-stream sampling systems (see 15.9.1) also apply to the individual streams of a multistream sampling system.

The second principle states that the streams thus conditioned must be manifolded to a common line to the analyzer in such a way that one does not contaminate another by valve leakage, diffusion from dead volumes, or any other source while the sample is being injected for analysis. Reliable valves of proper construction must be provided for each stream and, to prevent diffusion, they must be arranged so that dead volume is minimized. A purge system must be provided so that, when one stream is cut out of the manifold and another cut in, the old stream will continue to flow through a bypass. The new sample will completely scavenge the system of the previous sample and be fail-safe (it will leak to the bypass or vent). Only in this way will the individual samples be representative when analyzed. However, this procedure usually requires purging considerable material. Its value must always be considered during the design phase, and a recovery system provided where justified. Some users prefer double block and vent valves to prevent cross-contamination in the event that a valve leaks.

Many varied diagrams appear in vendor's catalogs, technical journals, and other publications which will suggest ingenious ideas to the designer. Also, such publications may express decided preferences for individual system components. The author of a technical paper may crusade for pneumatic valves for sample switching despite the fact that they must be triggered by air solenoids actuated by the stream selector. Among the arguments he may enumerate are greater thrust, positive seating, and freedom from process fluid leakage (see 15.7.2). The designer must be sufficiently familiar with available equipment to evaluate such arguments when designing an installation.

15.11 Closed Loop Control

In some instances, gas chromatographs have been used for automatic, direct control of valves. In other situations, they have been used through cascade with a process controller, and even through mathematical elements that compute ratios of components. However, successful application is difficult and a thorough study should be made before closed loop control is attempted. A frequency response study of the process is helpful. It can determine whether or not closed loop control is possible and the speed at which such analyses must be made. This, in turn, determines the caliber of the instrument that must be supplied and its installation requirements. Extreme speed is not always required. An instrument with a 1-minute analysis cycle has been supplied for fractionating column control, and it was later determined that a 5-minute cycle was adequate. Study can also reveal other factors, such as whether direct or cascade control is preferable, the settings for control modes, and other control functions.

The primary requirement for a chromatograph in closed loop control service is reliability. Inasmuch as it usually operates at high analysis frequency, good quality is mandatory in all elements, particularly in the programmer and columns. Timers, valves, gears, and other mechanical equipment must be of premium quality to withstand the accelerated usage to which they are subjected. The peak-picker, holding circuit, and other elements in the closed loop control system must be equally reliable. The columns in a control chromatograph are particularly vulnerable because, in addition to higher frequency analysis which shortens column life, a smaller diameter tubing normally is used for increased carrier velocity. This increases susceptibility to contamination which, although offset somewhat by smaller samples, demands the most efficient and reliable sampling system.

15.11.1 SAMPLING SYSTEMS, CLOSED LOOP CONTROL CHROMATOGRAPH

The sampling system for a control chromatograph follows the same basic design as other single sampling systems except that requirements are more stringent. Removal of all damaging contaminants normally present should be provided for, and the possibility of intermittent contamination should be explored thoroughly. Complete laboratory analyses of the sample should be made, and if they reveal any injurious materials that cannot be removed, a protective column must be provided. For continuous, reliable operation, filters, coalescers, and other apparatus subject to fouling should be installed dual-parallel so that one can be bypassed and cleaned while the other is in operation. They should be provided with purge facilities to prevent contamination on switching. At the same time, transportation lag in the system must be kept as low as feasible. This requires a very careful study of component volumes, line velocities, capacities, and so forth. Expensive sampling systems can be justified for a control chromatograph if they reduce dead time which is detrimental to closed loop control.

15.12 Operating Procedure

Startup and initial operation are properly a part of installation procedures because any possible need for modification is revealed during this period. Revision may be required, even when all known precautions have been observed in design, because of some condition not anticipated. No chromatograph that operates properly only part of the time should ever be turned over to, or accepted by, an operating department, because it will be a continuous source of trouble. Once the need for change has been verified, such change should be made promptly.

It is desirable to have a representative of the vendor at the user site to participate in activities such as checking out the analyzer installation, initial startup, and training user personnel on maintenance procedures.

15.12.1 PRELIMINARY TESTS

Many users employ the practice of witnessing performance and calibration tests at the vendor's factory prior to shipment. Some users conduct a series of preliminary tests on a chromatograph at the site before permanent installation is made. These procedures permit the user to make a complete test of an instrument under laboratory conditions and with clean samples of known composition. There are other advantages, one of which is instruction of personnel on the construction, limitations, and use of the instrument, and the importance of reference chromatograms.

In making such tests, startup procedures in the vendor's instruction book must be followed carefully. These will vary, not merely with different vendors, but also with different instruments from the same vendor.

While laboratory tests are being made, the sampling system can be tested simultaneously by circulation through the conditioning and venting apparatus. The connection to the chromatograph should be valved off except during the taking of samples to be examined in the laboratory. These should be taken periodically and tested for cleanliness and suitability. In the interim, filters, coalescers, and other sampling system devices can be opened and inspected. Once suitability is assured, samples can be taken and run on the instrument for calibration purposes as mentioned. One thing should be remembered: a sampling system should never be connected to the instrument until repeated tests assure that the sample is clean, representative, and measurable.

15.12.2 FINAL TESTS

If the preliminary test procedure outlined herein is not followed in the laboratory, a series of similar tests (and operator instruction) should be conducted at the site, despite the fact that conditions will be less favorable. If laboratory tests are conducted, they need not be repeated after the instrument is permanently installed, but it is necessary to verify that conditions at the permanent location have not altered the functioning of the analyzer elements. After verifying that operating conditions are normal, the initial sample run is made. First analyses should be made on a single sample even in a multisampling system, and successive chromatograms should be compared. Complete chromatograms should be made at first, regardless of the type of readout. After operation is stabilized, samples from the same source should be analyzed in the laboratory to verify calibration. The bar graph or other readout mechanism is then put in service and readings are compared to the chromatogram. In multistream sampling systems it may be advisable to repeat the procedure on each stream. The sample selector is then put in operation or, depending upon the duty of the instrument, the other auxiliary items are activated.

Closed loop control service calls for particular care. The peak-picker, holding circuits, and other elements in the control system should be carefully checked until reliability is established. It may be advisable to keep the instrument out of the control circuit initially and observe the records. When it is placed on control, its action should be limited by stops in the control system so that failure will not create a severe upset. Prior to placing the chromatograph on control, attenuator values, proportional band settings, and other control function adjustments should be compared to those determined previously (see 15.11). The calculated adjustments will probably require trimming after a period of operation, but they are the only criteria available for startup.

As startup proceeds, operator instruction should continue. Personnel should be asked to make chromatograms, switch the controller from automatic to manual control, and so forth. In short, the plant operator should discharge all future duties while under observation by expert personnel.

15.12.3 STANDARD SAMPLE

As operation proceeds, it is necessary periodically to analyze a sample in the laboratory to verify calibration. In addition, many users insist that means be provided for the operator at his discretion to check calibration. This normally is achieved by the standard samples referred to in Section 14. Several methods have been used to introduce such samples to the instrument. The methods shown in Figures 15-5 through 15-7 are probably the ultimate in operator convenience. A three-way solenoid valve is energized at will by the operator through a switch at the programmer. The sample container and connections are not shown on the illustrations because each installation is a separate problem. A comparatively large volume of sample is usually stored in a container near the analyzer. It may be synthetic if it approximates process sample composition, or it may be taken directly from the process sample stream and analyzed in the laboratory. The sample should always be taken downstream of the conditioning elements to assure cleanliness. Where this method is used, sampling systems are often equipped for this procedure. Because they must be stored at relatively high pressures, gas samples and volatile liquids should be taken ahead of the pressure-reducing valve. If there are conditioning elements after the reducing valve, special valving arrangements are required. If volatile components are not vaporized and lost, liquid samples can be taken after pressure reduction because they must be forced from the storage container into the instrument. Sometimes sampling is accomplished by pressurizing the container with carrier gas. With gas samples, the connection to the analyzer is taken from the top of the container, whereas it is taken near the bottom for all liquid samples. If volatile liquids require vaporization for analysis, the standard sample line may be equipped with an individual vaporizer and regulator. Common facilities may be unsatisfactory because the purge and vent dissipate stored sample too rapidly. Aside from the equipment used, problems sometimes arise with standard samples because of fractionation, stratification, and instability. As noted in Section 14, to be effective a standard sample must be uniform, representative, and stable.

It may be desirable to equip the solenoid with a timed actuator so that the sample supply is automatically switched back to the process after a period of time to conserve standard sample.

If the analyzer is used for a closed loop control system, the controller should be in manual operation when monitoring a standard sample.

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SECTION 16—VISCOMETERS

16.1 Scope

This section covers the installation of the more commonly used instruments for the continuous measurement of liquid viscosity.

16.2 Basic Principles of Viscosity Measurement

Viscosity can be defined as the internal friction of a fluid. It is a measure of the fluidity. When a fluid passes between two parallel plates of unit area and unit distance apart and the plates are moved in opposite directions at a given rate (rate of shear), a certain force must be applied to overcome the shearing stress of the fluid. The ratio of the shearing stress to the rate of shear is an expression of the viscosity.

The first intelligent definition of viscosity is credited to Isaac Newton. He assumed that the force required to maintain a difference of velocity between two planes in a liquid is proportional to their relative velocity divided by the distance between them, that is, the velocity gradient. If, for example, a plane of area A square centimeters is situated parallel to and at a distance of d centimeters from a fixed plane which is at rest and the space between the planes is completely filled with a fluid, the force F dynes required to move the plane area A at a velocity v centimeters per second in its own plane relative to the fixed plane is given by Formula 1.

$$F = \mu A \frac{v}{d} \qquad (1)$$

Where:

F = force, in dynes
μ = coefficient of viscosity of the fluid between the planes, in poises
A = area of plane, in square centimeters
v = velocity, in centimeters per second
d = distance from fixed plane, in centimeters

A more practicable definition is that of Hagan-Poiscuille who described viscosity in modern terms as the ratio of shearing stress to rate of shear at the wall of a tube. Mathematically viscosity may be expressed as in Formula 2.

$$\mu = \frac{\pi \Delta P R^4}{8QL} \qquad (2)$$

Where:

μ = viscosity, in poises
ΔP = pressure drop of fluid through tube, in dynes per square centimeter
R = radius of tube, in centimeters
Q = flow rate by volume, in cubic centimeters per second
L = length of tube, in centimeters

In the centimeter-gram-second system, μ is given in poises, the absolute unit of viscosity. (Centipoises are more commonly used for convenience.) The viscosity of water at 68 degrees Fahrenheit is one centipoise. Closely related are the kinematic viscosity units of stoke and centistoke, the viscosity obtained by expressing Q in mass flow rate. Hence, centipoise equals centistoke times density. Unfortunately, these relationships apply only to simple Newtonian liquids, wherein the viscosity is a constant at any given temperature. However, most petroleum products, such as lubricating oils where viscosity is a pertinent factor, are Newtonian. Notable exceptions are certain lube oils with additives, greases, tars, asphalts, and certain residuum fuels. These latter products are said to be thixotropic. A thixotropic substance possesses an internal structure which breaks down (that is, the viscosity decreases) when the substance is subjected to shear but will rebuild itself in time if allowed to rest.

The majority of laboratory viscosity-measuring devices are of the kinematic type, and results are usually calibrated in time units rather than in units of either absolute or kinematic viscosity.

A few of these relative or inferential scales are in general use today: the Saybolt scales in the United States, the Redwood scales in Great Britain, and the Engler scales in Europe. All three of these scales, based on Poiscuille's law, indicate the time of efflux under given conditions of a fixed volume of fluid through a specific capillary or aperture. Viscosity of a specified oil, for example, might be expressed as 105 Saybolt Universal seconds (SUS) at 130 degrees Fahrenheit. In this case, 105 seconds is the time required for 60 milliliters of the specified oil under its own head at 130 degrees Fahrenheit to flow through the orifice in a Saybolt Universal viscometer. The viscosity of lubricants is usually reported as Saybolt Universal seconds at 100, 130, or 210 degrees Fahrenheit and of fuel oils as Saybolt Furol seconds at 122 or 210 degrees Fahrenheit.

Kinematic viscosity, ν, may be determined from various scale values by means of an empirical formula in the form of the following:

$$\nu = At - \frac{B}{t} \qquad (3)$$

Where:

ν = viscosity, in stokes
A and B = constants applicable to the measuring apparatus (viscometer)
t = fluid efflux time, in seconds (scale time)

If the fluid density at the temperature of the test is known, absolute viscosity may then be calculated. Commonly accepted values for A and B in Formula 3 are listed in Table 16-1.

Table 16-1—Constants Applicable to Viscometers

Viscometer	Efflux Time (seconds)	A	B
Saybolt Universal	32-100	00226	1.95
	> 100	.00220	1.35
Saybolt Furol	25-40	.0224	1.84
	> 40	.0216	0.60
Redwood No. 1 (Standard)	34-100	.00260	1.79
	> 100	.00247	0.50
Redwood No. 2 (Admiralty)	32-90	.0246	1.00
	> 90	.0245	—
Engler	—	.00147	3.74

All of the continuous-process types of viscometers measure viscosity in absolute units but are usually calibrated to read out in time units in order to correlate with the laboratory instruments. Inasmuch as absolute viscosity is equal to kinematic viscosity multiplied by density, this calibration is valid as long as the density of the material being measured is constant. The density of most lubricating oil streams will not vary more than ±1 percent so that variations in calibration accuracy as a result of density changes are negligible. In measuring the viscosity of heavy fuels, however, large variations in density could occur which may necessitate plotting several calibration curves for the expected variation in density.

16.3 General

Several types of continuous viscometers are available. All may be mounted in conjunction with one or a combination of remote or close coupled, continuous recording, indicating, or controlling devices, thus lending themselves to automatic process control.

16.3.1 CAPILLARY

If a fluid of constant density is forced to move at a constant volumetric rate through a fixed constriction, the differential pressure across the restriction is a measure of the viscosity.

In viscometers based on this principle, the constriction ordinarily is a short tube or capillary of a cross-sectional area appropriate to the viscosity range to be measured. The constant volumetric rate is maintained with a positive displacement pump, and the differential pressure is measured with an instrument of the same type as is used for flow measurement. Constant fluid temperature is maintained with heat exchange equipment controlled by a fixed-temperature system.

Usually, only a small flowing bypass sample of the process stream is pumped through the instrument. Each viscosity range will require a specific selection of constriction and temperature. This type of viscometer has been used successfully for viscosities up to 2500 centipoises with accuracies of ±1 percent of span. Typical applications include fuel oils, hydraulic oils, lubricating oils, and various grades of asphalt.

16.3.2 ULTRASONIC PROBE

The damping effect of a surrounding fluid on the vibrations of a probe is a function of the viscosity of a constant-density fluid. In one such device based on this principle, the sensing probe is a metal blade a little over two inches long, a fraction of an inch wide, and a few thousandths of an inch thick. The blade is magnetostrictively driven in a longitudinal direction with periodic pulses of 28 kilohertz. The rate of decay of the damped oscillations is electronically measured and is a function of the product of the viscosity times the density of the fluid film in contact with the blade. A temperature bulb can be inserted in the fluid under test and connected into the electronic circuit so that the viscosity referred to some specific temperature may be directly indicated, recorded, or controlled. The ultrasonic viscometer is generally available in five ranges—0 to 5, 50, 500, 5000, and 50.000 in (centipoises) grams per cubic centimeter. Accuracy of the system is about +2 percent of full scale for Newtonian fluids of low viscosity (below 2000 centistokes). The ultrasonic probe viscometer is finding applications in fuel oil and asphalt blending, continuous asphalt conversion, and vacuum tower bottom fraction separation.

16.3.3 ROTATIONAL

The drag on a rotating cylinder is a function of the viscosity of the surrounding fluid. In one viscometer employing this principle, the cylinder is turned at a constant speed and the drag develops an electronically detected displacement of a spring section of the driving shaft. There are several other methods of measuring this drag. In a process viscometer application, the sample is continuously replaced and is subjected to a constant shear rate; thus measurement of apparent viscosity of non-Newtonian fluids is possible as well as measurement of absolute viscosity of Newtonian fluids. This instrument can operate within any range from 0-10 centipoises to 0-50,000 centipoises with an accuracy of ±1 percent full scale for ranges greater than 0-100 centipoises.

16.3.4 FLOAT

The plummet of a variable-area flowmeter (rotameter) normally is designed to minimize the effect of viscosity. If, on the contrary, the plummet is designed to maximize the effect of viscosity, and the flow rate through the tapered tube is maintained constant, the position of the viscosity-sensitive plummet becomes a measure of viscosity. Based on different applications, there are three types of float viscometers—single-float, two-float, and concentric.

The single-float viscometer is a direct reading viscosity instrument for continuous measurement. The constant sample flow is provided by a positive displacement pump. This type of an instrument can be used with non-Newtonian fluids with viscosities less than 400 centipoises and Newtonian fluids up to 10,000 centipoises. Expected accuracy is ±4 percent of reading.

The two-float viscometer incorporates a second float sensitive to fluid flowrate. This instrument is available for local indication only, and the flowrate must be adjusted manually to a predetermined value. This instrument is used for Newtonian fluids with viscosities between 0.3 and 250 centipoises. Accuracy is ±4 percent of reading for viscosities greater than 35 centipoises and ±2 percent of reading for lower viscosities.

The concentric viscometer is a variable area flowmeter with a viscosity-sensitive float and a differential pressure regulator. The flow rate of fluid past the viscosity sensitive float is held constant by the differential pressure regulator. This viscometer is commercially available in all metal construction with an extension coupled to the float and a magnetic pickup for display recording or control. The concentric viscometer can measure viscosities in the range of 0.5 to 550 centipoises with an accuracy of ±2 percent of reading for viscosities less than 35 centipoises and ±4 percent of reading for higher viscosities.

16.3.5 PISTON

The length of time required for a weighted piston to fall into a cylinder filled with fluid is a function of the fluid viscosity. Viscometers based on this principle raise and drop a measuring piston at 1- or 2-minute intervals and measure the time of drop by electrical means. The falling piston viscometer can be used for Newtonian and non-Newtonian fluids with viscosities ranging from 0.1 to 10^6 centipoises. The accuracy of this type is about ±1 percent of full scale.

16.4 Temperature Compensation

The variation of viscosity with temperature for petroleum materials is sufficiently great so that either the temperature of the sample measured must be held precisely constant or compensation of some sort must be provided. This compensation is generally provided by a temperature-measuring device which electrically, pneumatically, or mechanically applies a correcting factor to the indicated viscosity. This factor must be predetermined experimentally for the material being measured. Since accurate compensation is only possible over a narrow temperature range, temperature control of some sort is usually needed. This is especially true if the viscometer is to be used for automatic control to some viscosity related to a specific temperature.

For in-line blending of oils having known characteristics, the need for precise temperature control may be avoided through the use of a captive sample of the required blend. In the case of the ultrasonic probe viscometer, a captive sample (providing that the oils are not thixotropic) can be inserted in the flowing stream with one sensing element inserted in the captive sample. The other sensing element is inserted directly into the flowing stream and connected so that the electronic circuitry compares the two signals and transmits the difference or error signal to the appropriate control valve. The same thing can be accomplished with most viscometers provided that two sensing elements are used and that both the captive and the flowing samples are kept at the same temperature regardless of what that temperature might be.

16.5 Safety Considerations

16.5.1 SAMPLE MATERIAL PROBLEMS

Most viscometers require a small bypass stream from the process to the measuring element. This stream may be quite hot and the normal precautions should be taken to insulate sample lines and equipment for personnel protection. Where the material is extremely hot and sample coolers must be used to prevent damage to the viscometer, automatic overtemperature safety shutoff valves should be installed in the sample line to function in the event of cooling water failure.

For sample disposal, very small amounts of nontoxic materials can be discharged to convenient process sewers. Larger quantities can be returned to some low-pressure vessel or line or pumped to a high-pressure vessel or line. Toxic materials must be returned to some low-pressure vessel or line or pumped to a high-pressure vessel or line.

16.5.2 ELECTRICAL PRECAUTIONS

Viscometers are not generally used with particularly volatile or hazardous materials, but the normal electrical requirements for handling hydrocarbons should be followed.

All viscometers may have some electrical equipment associated with their operation either in the measuring circuit, temperature compensating circuit, or transmission system. The electrical system should be studied to determine whether it is intrinsically safe or what degree of hazard it might present.

The scope of electrical precautions necessary is outlined in Section 14—Analyzers.

16.6 Location and Housing

It is desirable to locate the viscometer so that sample lines are as short as possible. At the same time, the viscometer must be readily available for maintenance. This usually means that some sort of compromise must be made. Where a number of viscometers are to be installed in the same operating unit, it may be desirable to locate them adjacent to each other, especially if they are to be mounted in a walk-in type of enclosure. For additional information on analyzer location, see Section 14.

16.7 Sampling Systems

16.7.1 GENERAL

General design of sampling systems applicable to all analyzers is contained in Section 14.

Basically, sampling systems for viscometers may be divided into two types: one for sampling dirty materials of high viscosity, such as tars, asphalts, and heavy residual fuels; the other for sampling clean lubricating oils in the low to medium-high viscosity range.

A typical sampling system for high-viscosity liquids is shown in Figure 16-1. Note the use of a Y-type of strainer or rotating-disk filter connected into the high-velocity sample loop in such a manner as to continuously flush the filter face clean of entrained material. It may also be desirable to install a small in-line filter in the low-velocity sample line. All sample lines are connected to the sample taps on the process lines through the side connections of tees. This installation permits reaming out of the sample taps.

Viscometer and all piping, equipment, and so forth should be heat traced or cooled as required. Plugging of sample lines when the viscometer is shut down for a prolonged period of time can be minimized by the installation of wash oil connections for flushing out the system with a lighter oil. (Some processes, such as vacuum distillation units, have a wash oil system incorporated in their design.) If the wash oil cannot be returned to the process line, it may be necessary to install a wash oil effluent line.

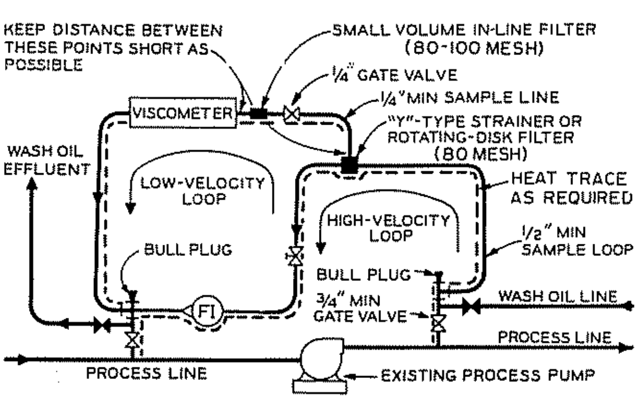

Figure 16-1—Typical Sampling System for High-Viscosity Liquids

A typical sampling system for measuring the viscosity of clean, light oils is shown in Figure 16-2. In those sections of the country where winter temperatures fall as low as 32 degrees Fahrenheit, it may be desirable to heat trace all sample lines. Line sizes shown in Figures 16-1 and 16-2 are minimum. Actual line size would be determined by desired sample loop speed of response, type and temperature of material handled, and operating practices in the particular refinery in which the installation is located.

16.7.2 SPECIFIC SAMPLING SYSTEMS

Specific sampling systems vary with the type of viscometer selected and the material which is to be sampled. Five types of viscometers are included in this discussion: the capillary, ultrasonic probe, rotational, float, and piston.

16.7.1.1 Capillary

The rate of sample flow through capillary-type viscometers is so small that unless the viscometer can be mounted within 10 feet of the process line, a controlled bypass sample loop should always be used. Figure 16-3 shows a typical installation of a capillary-type viscometer.

It is necessary to have a good filter in the sample line of capillary-type viscometers. Filtering can become a problem when measuring the viscosity of dirty material or material with suspended solids, such as tar, since the filter will plug up very rapidly. A cleanable disc-type filter is recommended (which in very dirty service may be motor driven to automatically rotate the movable discs). As an alternative a dual filter system

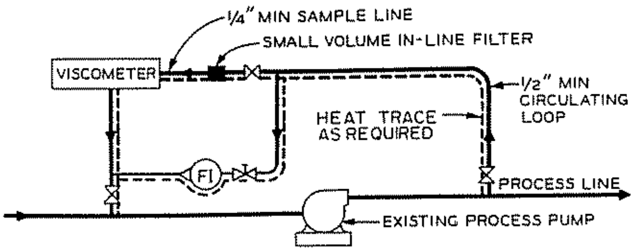

Figure 16-2—Typical Sampling System for Clean, Light Oils

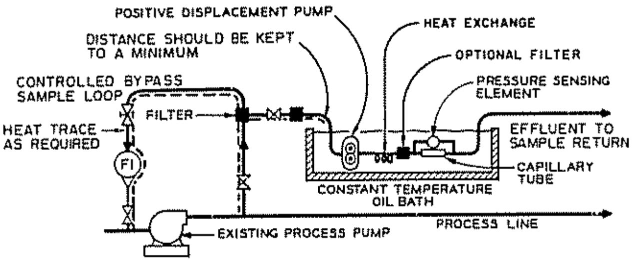

Figure 16-3—Typical Installation of a Capillary-Type Viscometer

may be used (connected in parallel with appropriate valving to allow one filter to be cleaned while the other is in service).

A Y-type filter with 60-mesh screen may be installed in the sample line followed by an additional 60-mesh screen installed ahead of the capillary system.

The capillary filter screen may be omitted when using the larger diameter capillary tubes usually required for narrow range measurements at high viscosities.

16.7.1.2 Ultrasonic Probe

Although the ultrasonic type of viscometer employs a probe as a sensing element, which can be inserted directly in process lines in about the same manner as a thermocouple or thermometer, generally it is advantageous to insert the probe in a circulating sample loop for several reasons:

1. Temperature compensation is available, but it is also usually necessary to temperature control the sample surrounding the probe.
2. Rate of flow can be controlled in the sample loop. The probe is not excessively sensitive to flow rate but does show some reaction to wide variations.
3. It is easier to remove the probe for cleaning or replacement from a sample loop than it is from a process line. (Some materials tend to deposit out on the probe necessitating cleaning the probe to restore calibration.)

A typical installation of the ultrasonic probe viscometer is shown in Figure 16-4.

16.7.1.3 Rotational

The rotational type of viscometer may be mounted directly in a vessel, or it can monitor viscosity in a flowing line. In either case, it is necessary to use a purge gas, such as instrument air, nitrogen, or carbon dioxide, to prevent corrosive fumes or explosive vapors from entering the upper chamber of the viscometer. A rate of 0.5 cubic feet per minute or greater and a positive pressure differential of 3 pounds per square inch are recommended. If viscosity is to be referred to some specific temperature, it is necessary to precisely control the temperature of the material being monitored by the viscometer or to compensate for the temperature changes.

Figure 16-5 shows a typical installation in a vessel where a definite liquid level is maintained in the vessel.

This type of viscometer can also be used to monitor viscosity in flowing lines through the use of a sample chamber suitably connected to the flowing stream. The piping arrangements for connecting the sample chamber to the flowing line are shown in Figure 16-6. Unless long sample lines are necessary or the material is extremely dirty, no strainers are required in this installation.

The installation, as shown in Figure 16-6, can be used to directly indicate viscosity referred to a specific base temperature. If this type of analysis is required, precise temperature control of the material in the sample chamber is necessary.

As shown in Figure 16-5 and 16-6, it is desirable to include a flow indicator (FI) in the purge gas line. When handling explosive materials, even though the electrical system is intrinsically safe, it may be desirable to install a pressure switch to automatically deenergize the electrical circuits if the purge gas pressure drops below a safe level.

16.7.1.4 Float

The float viscometer can only be used to continuously monitor viscosity in a flowing stream. Temperature compensation is available on this model, but the

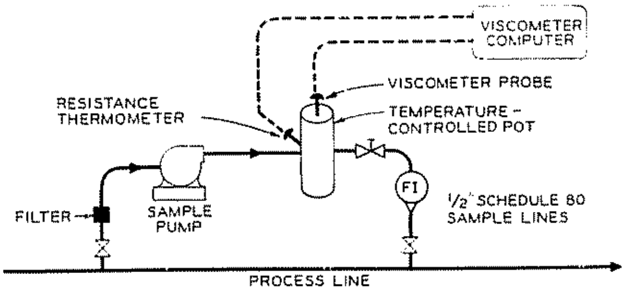

Figure 16-4—Typical Installation of an Ultrasonic Probe Viscometer

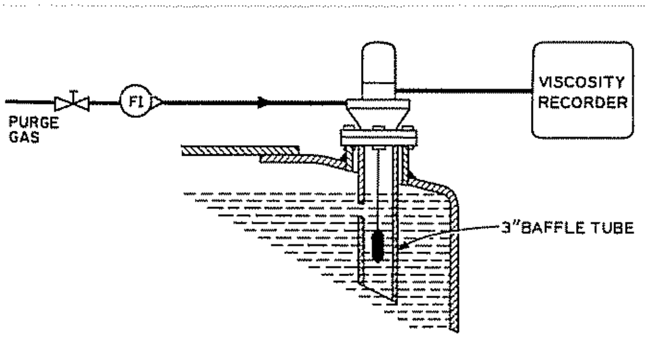

Figure 16-5—Tank Mounting for Rotational Viscometer

sample stream should be temperature controlled if variations in temperature of greater than ±10 degrees Fahrenheit can occur.

It is important that the flow rate be kept constant in float-type viscometers. The various sampling arrangements to obtain constant flow rate are shown in Figures 16-7 and 16-8. The sampling system shown in Figure 16-7 uses a precision positive displacement pump to ensure constant flow rate through the viscometer. A strainer, located in the sample line ahead of the pump, is an absolute necessity as any amount of solids entrained in the flowing liquid would destroy the close clearances essential for the operation of the pump. This sampling system is applicable where the viscometer can be conveniently located close to the process line. Where it is not convenient to locate the viscometer near the process line, sampling time or transportation lag can be reduced by installing a relatively high-velocity sample loop as shown in Figure 16-8.

If it is not practical to connect the sample loop around an existing process pump, as shown in Figure 16-8, an orifice in the process line or an existing control valve can be used to create a pressure drop to force the fluid through the sample loop. If pressure drop is not available, it may be necessary to use a sample pump. Sample line size is shown as ¼ inch, but line size and viscometer size will vary with the viscosity range of the material to be measured.

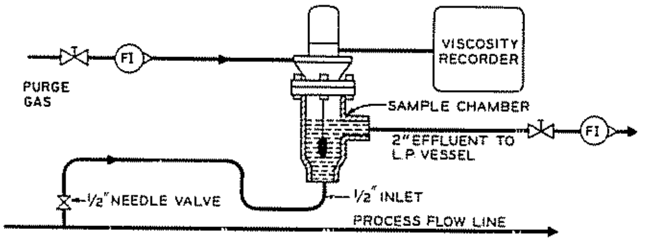

Figure 16-6—Flowing Line Installation for Rotational Viscometer

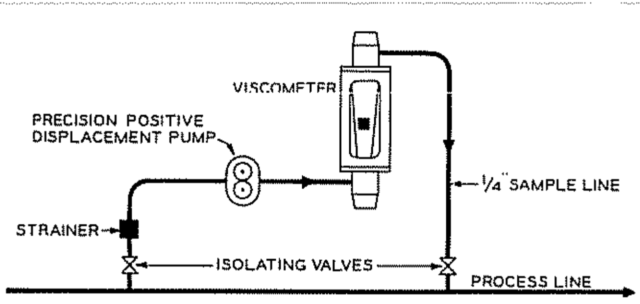

Figure 16-7—Viscometer Located Close to Process Line

Use of the concentric viscometer eliminates the need for a positive displacement metering pump since flow rate of fluid past the viscosity sensitive float is held constant by maintaining a constant pressure drop across the viscometer tube.

16.7.1.5 Piston

The sample flow rate through a piston type of viscometer, as shown in Figure 16-9, is not critical, and a simple sample pump normally is satisfactory.

The normal flow for the sample (see Figure 6-9) is from the process line through Valve 1, the pump, and then into the viscosity measuring tube. From the measuring tube, the sample flows through the temperature bulb chamber and then back to the line through Valve 2. The bypass line is used to calibrate the viscometer and adjust the temperature compensator. During this procedure, Valve 3 is opened and either Valve 1 or 2 is closed. For complete information on calibration, refer to manufacturer's literature.

Filters or strainers are not essential in the sample line unless the material to be measured is known to be dirty or contain solids. If the viscometer cannot be mounted close to the process line, it may be desirable to install a sample loop as illustrated in Figure 16-8. One manufacturer claims that temperature compensation is effective over a range of ±15 degrees Fahrenheit. Any variations in temperature greater than this will require temperature control of the sample. Use of the

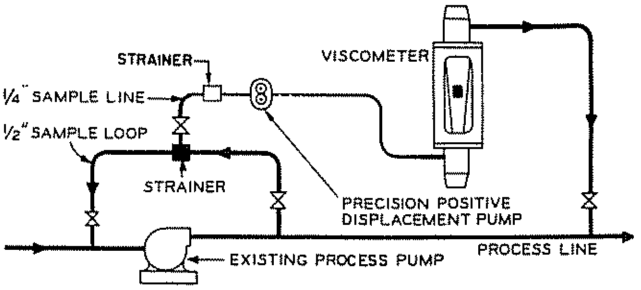

Figure 16-8—Viscometer Sample Line from Circulating Loop

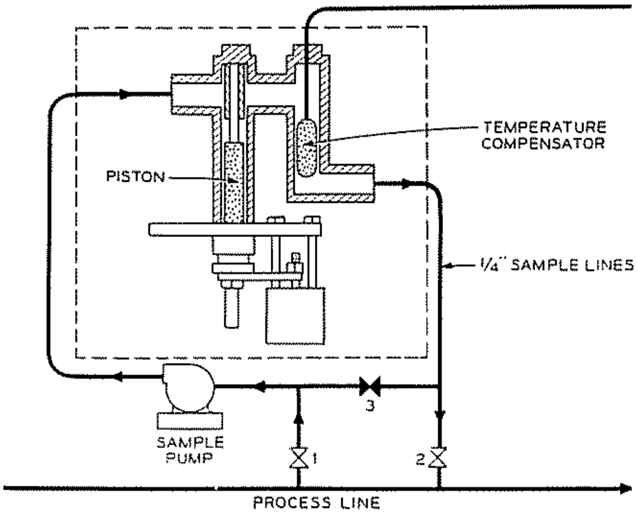

Figure 16-9—Installation of Piston-Type Viscometer

piston type of viscometer should be avoided where vibration is expected or where fast response time (less than 1 minute) is required.

16.8 Calibration

Calibration of viscometers in lubricating oil service does not present any problems since lubricating oils are usually stable and nonvolatile Furthermore, the following items should be noted during viscometer calibration:

1. The viscometer can be calibrated from control laboratory analyses of a representative sample taken near the viscometer or a standard sample can be run through the analyzer. (The control laboratory should be alerted that the samples to be analyzed are for viscometer calibration and where possible the laboratory analyzer should be operated at the same temperature as the process analyzer.)
2. Viscometers can sometimes be calibrated from standard samples which are available commercially.
3. A chart should be marked when a sample is taken for calibration.

The calibration of viscometers measuring materials, such as residual fuels, which exhibit some non-Newtonian characteristics can be quite difficult. These materials tend to be thixotropic. A chemist in the control laboratory or the manufacturer should be consulted as to the method used to analyze samples of such materials.

Check calibration daily at first, decrease the number of checks as reliability is established. Refer to manufacturer's literature for calibration methods.

16.9 Readout

All of the viscometers described in this section can be adapted to read out on most of the standard pneumatic or electrical indicating or recording instruments for measurement or control. Refer to manufacturer's literature for special adaptations of peak-picking devices for batch-type viscometers.

16.10 Startup

Startup procedures for the continuous measurement of liquid viscosity are as follows:

1. Turn on heat tracing lines (if any).
2. Circulate sample. (In the rotational type of viscometer, it is necessary to turn on the purge gas and note that it is at the correct pressure before circulating sample.)
3. Check for leaks.
4. Place heat exchangers, coolers, temperature controls, pressure controls, and so forth in operation and check for stability.
5. Apply step change and check speed response.
6. Calibrate.

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SECTION 17—DISTILLATION, FLASH, AND VAPOR PRESSURE INSTRUMENTS

17.1 Scope

Instruments covered in this section are used in refineries to duplicate certain American Society for Testing and Materials (ASTM) tests which formerly could be made only in the laboratory. These units are usually plant stream analyzers, that is, they are connected to the process by a permanently installed sample system. Devices used in control rooms or operating areas for occasional quality control tests on samples obtained by the operators are sometimes known as access testers. Those devices in which the operator initiates the sample injection manually are known as batch-type analyzers.

17.2 Definitions

The instruments discussed in this section are defined as follows:

1. *Distillation instruments*—This group is comprised of those devices which operate on a liquid (or condensed vapor) sample by volatilizing and, usually, condensing part or all of the sample. Distillation instruments may be used to analyze mixtures of liquids (or gases) if the components differ at least in vapor pressure and are not decomposed by heat. Types of distillation analyzers are (a) initial boiling point, (b) end point, and (c) other boiling points, such as 50-percent point.
2. *Flash point instruments*—This group is comprised of those devices which determine the lowest temperature at which the vapors above the sample may be ignited by application of a test flame or other source of ignition.
3. *Vapor pressure instruments*—This group is comprised of those devices which determine the pressure exerted over the sample within specified conditions of temperature and in the presence or absence of other gases.

17.3 General

Some of the instruments described in 17.2 continuously measure the desired property of the sample producing an unbroken output signal or curve. This signal may be used as input to a controller. Other instruments, such as the semicontinuous type, operate on automatically sequenced, discrete quantities and produce sawtooth, spike, or bar records from which the desired values are read from the peaks or valleys. The intermittent signal from these instruments must be stored in order to be useful as a controller input.

17.3.1 DISTILLATION INSTRUMENTS

Distillation-type instruments may be of the continuous, semicontinuous, or batch type. Most of these devices correlate with ASTM Designation D 86 *Test for Distillation of Petroleum Products*, or ASTM Designation D 1160 *Test for Distillation of Petroleum Products at Reduced Pressures*.

17.3.1.1 Initial Boiling Point Instruments

The initial boiling point analyzer automatically controls continuous distillation of a sample so that it is split into a constant ratio of overhead vapor fraction to bottoms liquid fraction. The vapor temperature in the top of the distillation tower is measured and can be correlated with ASTM D 86 distillation data. An initial boiling point analyzer is illustrated schematically in Figure 17-1.

A preconditioned stream sample passes through a preheater coil in the boiling pot and enters the top of the tower through a variable inlet valve. A float in the boiling pot which responds to liquid level changes controls the volume of sample as it enters the analyzer. The sample flows through the float guide tube to the feed zone of the tower and to the boiling pot. As fractionation progresses, the sample feed is split into overhead and bottoms cuts. Overhead vapor exits at the top of the tower. Bottoms liquid exits from the boiling pot and is combined with overhead vapor. A ball-type check valve in the bottoms outlet line prevents overheads from flowing back into the boiling pot. Pressure in the process line and tower is controlled by the pressure control system.

An orifice in the overhead line and an "equivalent orifice" in the bottoms line maintains the overhead to bottoms ratio. Overhead flow is restricted by the orifice; the "equivalent orifice" creates resistance to flow, thus the ratio remains constant. Temperature of vapor in the top of the tower is measured and represents readout temperature which can be correlated with ASTM D 86 distillition data.

A fixed wattage heater provides necessary heat to the boiling pot. Heat input requirements are based on

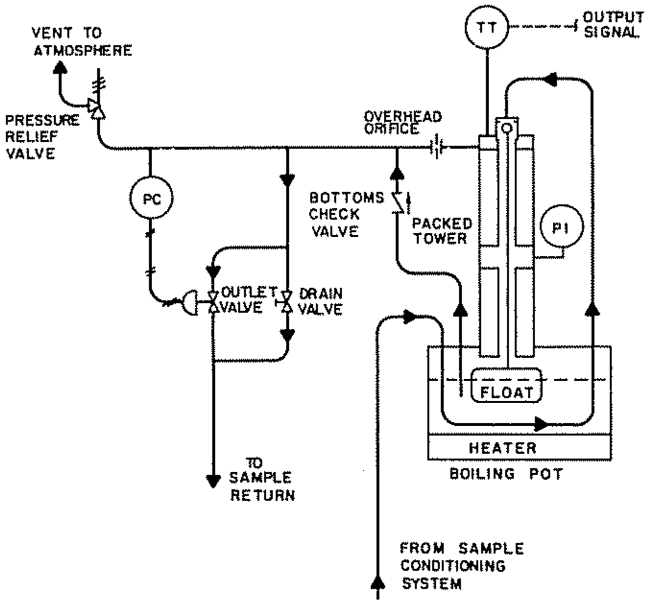


Figure 17-1—Initial Boiling Point Analyzer

the boiling range of the sample. Proper heater wattage is determined by the manufacturer based on the customer's sample distillation data. Heater selection is made in the range of 800 to 3200 watts.

17.3.1.2 End Point Instruments

The end point analyzer automatically controls continuous distillation of a sample so that it is split into a constant ratio of overhead vapor fraction to bottoms liquid fraction. The bottoms boiling temperature is measured and can be correlated with ASTM D 86 distillation data. The end point analyzer is illustrated schematically in Figure 17-2.

The operation of the end point analyzer is basically similar to the initial boiling point analyzer with a few minor exceptions. The major difference in operation lies in the fact that in the end point analyzer the split between overhead and bottoms cuts is accomplished with the orifice in the bottoms line, and the temperature monitored is the bottoms boiling temperature.

For very high boiling point applications, vacuum operation may be required.

17.3.1.3 Other Boiling Point Instruments

Additional points on the distillation curve are usually obtained by adapting the initial boiling point or end point instruments. The only modification required in these cases is to change the orifice size and possibly the heater wattage. Changing the orifice size will change the overhead to bottoms ratio and, in this way, will change the operating point.

17.3.2 FLASH POINT INSTRUMENTS

A typical flash point instrument, as shown in Figure 17-3, operates cyclically on a sample which is flowing at a constant rate and which is first cooled to below the flash point. At the start of the test cycle, a heater turns on and begins to raise the temperature of the sample. Air is added to the sample at a constant rate as it leaves the heater and overflows into the flash chamber. The temperature of the sample entering the flash chamber is recorded. The controlled air/vapor mixture in the flash chamber is exposed to a periodic high-voltage spark. Ignition of the mixture is detected by a thermocouple located in the flash vapor chamber. Upon ignition, the thermocouple output voltage activates a sensitive relay shutting off the heater and the spark. The peak liquid temperature recorded is the flash point. The temperature recorder is driven downscale as the sample in the flash chamber cools, and at the completion of the cooling cycle another flash point cycle is initiated.

17.3.3 VAPOR PRESSURE INSTRUMENTS

Vapor pressure instruments are usually the continuous type. The product is pumped, without interruption, through the instrument yielding a continuous output signal which is recorded or used as an input to a controller, or both.

Instruments which determine Reid vapor pressure (see Figure 17-4) are built to simulate the procedure described in ASTM Designation D 323 *Test for Vapor Pressure of Petroleum Products (Reid Method)* wherein

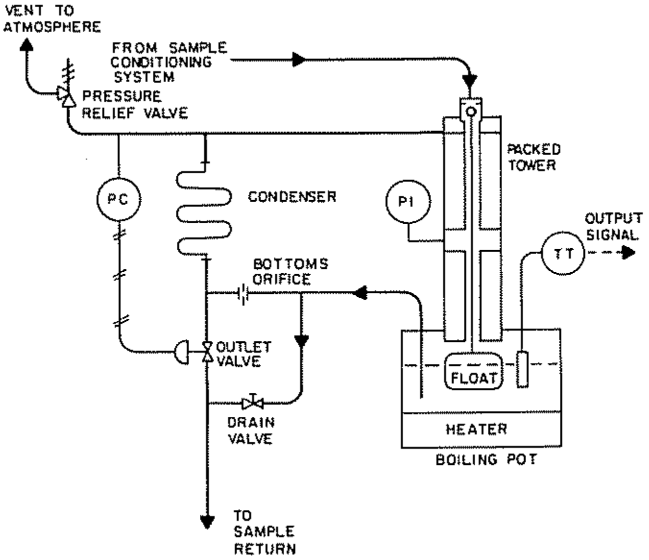


Figure 17-2—End Point Analyzer

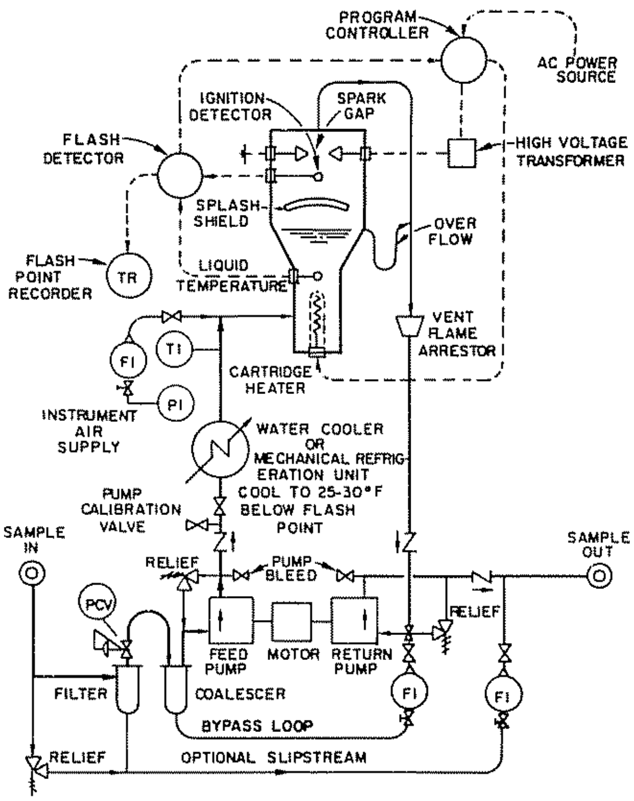


Figure 17-3—Flash Point Instrument

the sample is brought to equilibrium with air in a constant-temperature chamber. The pressure in the chamber is the signal to the recorder, which is calibrated in pounds of Reid vapor pressure.

Another type of vapor pressure analyzer makes use of an ejector-type nozzle. The ejector device works on the principle of increasing the kinetic head until the static head at the nozzle discharge reaches the vapor pressure of the liquid (see Figure 17-5). When the pressure at the tip of the nozzle attains the vapor pressure of the fluid being educted at the stream pumping temperature, a cut-off or cavitation limit occurs which determines the maximum quantity pumpable for a given suction condition To take advantage of this, the sample is forced through the nozzle of the analyzer at a preset constant high pressure and at a corresponding velocity so that the existing liquid velocity will be large. Under these conditions, pressure in the diffuser chamber will approach and be limited by the vapor pressure of the sample. This pressure is measured at the vapor chamber surrounding the nozzle tip and is identical to that in the diffuser chamber. The analyzer can be calibrated for either absolute or Reid vapor pressure monitoring.

17.4 Safety Considerations

17.4.1 SAMPLE MATERIAL PROBLEMS

Many of the materials analyzed by distillation, boiling point, flash point, and vapor pressure instruments are flammable and have low flash points, which make them particularly susceptible to ignition if released to the atmosphere. If a sample is taken from a process line carrying material at relatively high pressure and at a temperature above its autoignition temperature, the slightest leak in the sample line can be the cause of a fire. Even though the sample material does not flash as it emerges from a leak, it may vaporize and create a potentially explosive atmosphere. The sample is analyzed in a case or housing which contains the electrical equipment of the analyzer and where it is possible to have a source of ignition. Therefore, sample lines and all connections must be made leakproof.

Hot sample lines, in addition to being leakproof, should also be insulated or shielded for personnel protection. Sample coolers, where used, should be equipped with automatic over-temperature shutoff valves in the sample line.

17.4.2 ELECTRICAL AND MECHANICAL CONSIDERATIONS

All electrical wiring should be installed in accordance with the *National Electrical Code* as well as applicable national, state, and local codes Electrical sample heaters should be of the low-watt density type and should be equipped with overheat protection.

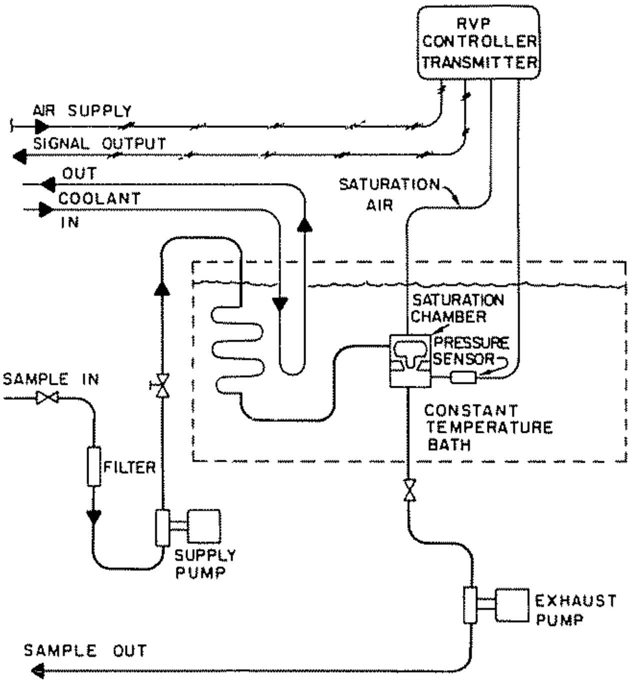


Figure 17-4—Reid Vapor Pressure Monitor

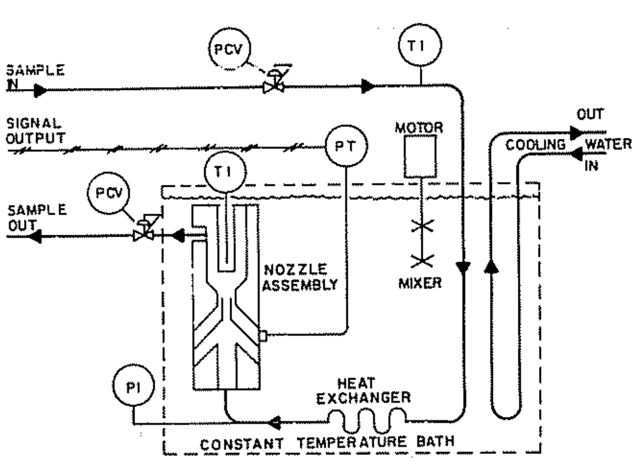


Figure 17-5—Vapor Pressure Monitor

Pumps and piping are the prime sources of mechanical leaks. Where pumps are used, provision for disposal of leakage should be considered when designing the installation. If an analyzer is equipped with its own pumps, the proper provision for collecting and draining leakage should be an integral part of the apparatus. Where pumps are installed separately, good-quality leakproof pumps, such as "canned" pumps, will help to ensure a tight system.

The use of screwed joints should be minimized to avoid piping leaks. Tubing and high-quality tubing fittings should be used wherever practicable.

17.4.3 EFFLUENT DISPOSAL

Sample disposal is a problem common to all analyzer installations whether the sample is unchanged, vaporized, or distilled and condensed. The end products in any case must be disposed of to a safe location. Practices for hydrocarbon drains in the particular refinery should be followed. Overhead vapors may be condensed and disposed of as liquid. Bottoms should be cooled, if required, for disposal.

Liquids which are virtually unchanged in the analyzer (such as gasoline in the vapor pressure instruments) or liquids which will cause no contamination (such as fractionated liquids and bottoms) may be pumped back to the process line. Liquids which cannot be returned to the process line should be pumped to the blowdown system or other safe disposal point. Relief valves in the sampling system or at pumps should be piped to vent or drain.

The low-back-pressure discharge requirements for most analyzers can be met by installing a vented receiver with a level controller and pump. The level controller can be arranged to automatically start and stop a pump which has sufficient head to deliver the liquid into the process line. Such an arrangement is shown in Figure 17-6. With some boiling point analyzers the low-back-pressure effluent can be returned to the process by an educator included in the sample conditioning package.

17.5 Analyzer Location

The ideal location for an analyzer is seldom found; therefore, a compromise is nearly always necessary. The following considerations affect the decision as to where to place the instrument:

1. Proximity to the sample tap is desirable. Most analyzers furnish either an electrical or pneumatic output which can be transmitted to the control room.
2. The analyzer should be installed in a nonhazardous area if practicable. Even though the analyzer, or any part of it, is built into an explosionproof housing and is perfectly safe during operation in *National Electrical Code* Class I areas, unsafe conditions could exist during maintenance and electrical calibration because the electrical and electronic elements must be exposed. Soldering of components should not be done in locations where hydrocarbon vapors may be present. If the analyzer cannot be located in a nonhazardous area, the analyzer or faulty plug-in type of units should be removed for soldering or for checking (refer to Section 14).
3. The control room is the least desirable location for an analyzer because of the necessity of piping the sample to it. It is not considered good practice to pipe hydrocarbons into the control room.
4. In their present stage of development, many analyzers must be maintained and calibrated in place and, therefore, should be enclosed in a walk-in type of housing. In areas of cold winters, heating of the shelter is

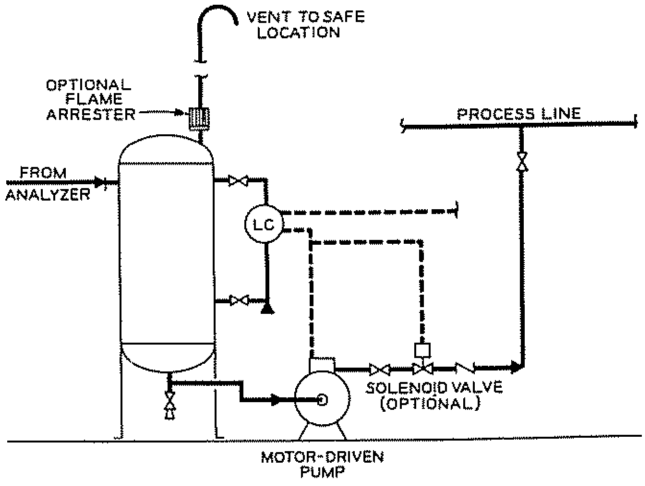


Figure 17-6—Liquid Sample Disposal System

required for the benefit of maintenance personnel even though some instruments are capable of operating under extremes of ambient temperature. In hot areas, protective shading should be provided for the instrument as well as for personnel. In addition, air conditioning should be considered in areas where ambient air temperatures can exceed the operating temperature limits of the analyzer. In all other areas, rain protection at least is necessary. Proper ventilation of analyzer shelters should always be considered.

17.6 Sampling Systems

17.6.1 SAMPLE-POINT LOCATION

The location of the sample tap is important. Refer to the discussion in Section 14 for general considerations.

17.6.1.1 Representative Sample

A representative sample must be obtained. This requires special care where the sample point is in the overhead vapor line from a fractionator because under some conditions liquid may be condensing on the pipe wall. In such instances, a sample probe extending into the pipe should be used. Adaptations of the ASTM and American Society of Mechanical Engineers (ASME) steam sampling nozzles may be used (see Section 14). The openings in the probe should be so oriented that impinging droplets will not be picked up. For volatility-type instruments, however, the sample is usually liquid as it is withdrawn from the process, thus, a horizontal tap in the side of a horizontal line is preferable. Taps into the top of a line should be avoided because of the possibility of withdrawing noncondensables or vapor. Also, taps into the bottom of a line should be avoided because of the possibility of withdrawing water or sediment. Free water is especially troublesome in a distillation type of instrument. It is usually better to withdraw the sample from a flowing line rather than from a vessel. However, if it is necessary to withdraw the sample from a vessel, the same precaution should be observed as for sample taps in piping. In addition, regions of stagnation should be avoided.

17.6.1.2 Pressure and Temperature

Pressure and temperature conditions at the sample point must be selected in order to give a manageable sample. For liquids, the pressure should be high enough and the temperature low enough so that there is no danger of flashing in the sample line. If the sample must be taken at or near its bubble point, it should flow through a small cooler to prevent flashing when the pressure is reduced. In some analyzers the pressure ahead of the inlet pumps must be low enough to avoid uncontrolled flow through the pumps yet high enough to afford positive suction. In addition, the temperature must be reduced enough to prevent vaporization of the sample in the suction side of the pump.

17.6.1.3 System Lag

In many cases, improper selection of the sample point can introduce as much lag as excessively long sample lines. Sample-point location is discussed in detail in Section 14. The consideration of lag becomes more important if the analyzer is to be used as a control instrument or to transmit to a computer.

17.6.2 SAMPLE SYSTEM PIPING DESIGN

It is necessary to design the sampling system piping so that it has small volume, minimum length, and sufficiently high flow rate to keep the distance/velocity lag reasonably short. In general, the excess flow must be circulated back to the process line. Usually sufficient pressure differential for satisfactory sample loop circulation is available across a control valve or a pump.

Practical velocities in small-diameter liquid systems are much lower than for gas systems. For example, ½-inch steel pipe or ½-inch OD stainless steel tubing are good choices for liquid sample lines. At a flow of 2 gallons per minute of a hydrocarbon with 0.7 specific gravity and 1 centipoise viscosity, the pressure drop is about 7.5 pounds per square inch per 100 feet of straight pipe and the velocity is approximately 4 feet per second.

If the viscosity under these conditions rises to 9 centipoises, the pressure drop rises to 15 pounds per square inch per 100 feet. Therefore, in some cases, it may be necessary to heat the line to reduce the viscosity. It is frequently necessary to use smooth drawn tubing or a larger size pipe for the sample loop in order to achieve the desired velocity. Pressure reducers and flow indicators are generally needed to maintain the required sample flow into the analyzer.

If a sample line containing liquid must be heated to maintain a lower viscosity for better flowing conditions, precautions must be taken to prevent upsetting the analyzer temperature control or overheating and vaporizing volatile components (when these are to be measured). If steam heating is used, the steam pressure may be reduced and/or proper spacing of the tracer line from the sample line may be chosen to maintain the required temperature of the sample.

Electric heating is often used because of its flexibility and other inherent advantages. Sometimes satisfactory temperature regulation can be achieved by selecting heating cable with proper voltage and resistance characteristics. If closer temperature regulation is required, it may be accomplished by either a thermostatic control or an appropriate modulating controller.

17.6.3 SAMPLE CONDITIONING

A fine mesh strainer to remove particles, such as pipe scale, from the sample stream may be sufficient for most instruments. However, if the sample must pass through capillaries or other small restrictions, or if deposits of fine particles may be detrimental to the performance of the instrument, a suitable filter of small volume should be installed ahead of the analyzer.

Some instruments require the sample stream to enter the analyzer section at a temperature which is within certain prescribed limits. If sample preheating or precooling is required, the heater or cooler may be furnished separately or as part of the instrument. In either case, appropriate utilities must be furnished. If the heater or cooler is furnished separately, installation of a temperature control system to maintain the proper sample temperature may be necessary. Coolers can be a source of considerable trouble. Sample and effluent coolers are usually so small that fouling becomes a problem. Small, self-contained recirculating coolant systems are now available which overcome these cooler problems, but these systems require electrical power. Plant drinking water should not be used for sample conditioning due to the possibility of drinking water contamination. It is advisable to use water filters if plant cooling water or cold condensate is used for cooling purposes. The water temperature in the cooler should not be allowed to rise above the point at which mineral deposits tend to accumulate. In general, this maximum temperature is several degrees lower than that allowed for larger heat exchangers using the same water supply. If liquid water is present in the sample, it must be eliminated. Removal of entrained liquid is discussed in Section 14. It may be necessary to remove other undesirable components, such as corrosive materials from the streams, as discussed in 17.9. For suggested installations of sample conditioning systems, see Section 14.

17.6.4 CONNECTIONS FOR STANDARD SAMPLES

If the instrument calibration is to be checked by means of standard samples, piping provisions must be made for valving the standard sample into the instrument either manually or automatically, as required.

17.7 Readout

The readout from most distillation devices is temperature. The same precautions must be observed as for any other temperature-recording installation with additional attention to sensitivity and accuracy. Flash point instruments also read out in values of temperature, usually producing sawtooth chart curves. To provide a continuous readout a suitable peak holder may be added to the analyzer output.

Vapor pressure instruments usually read out in pressure. Generally, the principles for the installation of pressure instruments apply to the readout end of the vapor pressure instruments as well.

17.8 Checking and Calibration

17.8.1 USE OF STANDARD SAMPLES

Almost any analytical instrument can be checked by means of a standard sample. However, the practicality of standard samples for high flow rate analyzers should be considered. This is a basic check which is analogous to checking a micrometer with a gage block. The assistance of laboratory personnel is generally required to develop the standard sample necessary to achieve the optimum calibration.

17.8.2 GENERAL PRACTICES

The practices generally followed in checking a plant stream analyzer are listed here.

1. A sample should be obtained which corresponds closely in measured properties to the normal plant stream. Such a sample may be a quantity of the plant stream or may be obtained from other sources. A 55-gallon-drum quantity is an appropriate size sample. Several laboratory analyses, according to the appropriate ASTM method, should be run on the sample. The statistical average of these analyses should be taken as the comparison value for the instrument, if the instrument is to duplicate the method.
2. The sample should be kept under conditions which will not cause changes in the analyzed properties. These values can change because of evaporation, polymerization, stratification, contamination, and so forth. Storage conditions should be selected for each particular type of sample. This is especially important if standard samples are used to check volatility measuring instruments which are calibrated for high volatility.
3. The sample should be admitted into the instrument for the proper interval of time and the results compared with the value which should be recorded for the sample.
4. If the recorded value differs from the predicted value, proper adjustments should be made. Some such adjustments may be made in the recording or transmitting instrument; however, it may be necessary to clean or replace faulty parts of the analytical section of the instruments.

5. After adjustments are made and the instrument is lined out on the process stream, the standard sample should be readmitted and the actual and predicted readings again compared.

6. If it is impossible to obtain agreement between an instrument reading and a standard sample (and if agreement has been obtained previously), it is best to obtain a new standard sample.

Standard samples may be admitted to the plant stream analyzer by pressurization from a suitable vessel with a suitable gas, usually nitrogen, or by pumping into the unit with a sample pump.

17.8.3 COMPARISON WITH LABORATORY ANALYSES

If the use of a standard sample is impracticable or impossible, the recorded reading of the analytical instrument may be checked against laboratory analysis. For a check of this type, at least three separate portions of the same sample should be submitted and the statistical average of the sample results should be used for comparison of the results recorded by the instrument. If the agreement is beyond tolerances and if it seems reasonable that the instrument is out of calibration, adjustments should be made. After appropriate adjustment or maintenance, or both, are performed and the instrument is lined out, a recheck should be made against another set of laboratory results. This method works best under conditions in which the properties of the sampled stream are changing slowly. Recognized, accepted procedures for obtaining and handling laboratory samples should be followed.

Many plant stream analyzers are designed to correlate with tests based on specified ASTM methods (refer to ASTM D 2891, *Validation of Results of Process Distillation Analyzers* and ASTM D 2984, *Test for Validation of Results of Process Vapor Pressure Analyzers*). Where this is the case, it is essential to use the proper ASTM method for checking the grab sample or for certifying a standard sample.

17.8.4 CALIBRATION BY MEANS OF TEMPERATURE

Temperature checks may be used on some instruments in which the readout is directly dependent upon temperature measurement. These checks may include putting a simulated signal into the system, checking the temperature sensor in a standard bath, or replacing the sensor with a certified replacement.

17.9 Special Precautions

It should be determined whether the plant stream contains or may contain any contaminants which will adversely affect the analyzer. The instrument manufacturer should be advised of contaminants which may be present in the sample. Such contaminants may include corrosive agents (such as chlorides, caustics, hydrogen sulfide, sulfur dioxide, and sulfur trioxide) as well as dyes, salts, or residues which cause deposits. If the analyzer design is such that these contaminants cannot be tolerated, or if suitable materials of construction are not avalaible, it will be necessary to remove the undesirable materials from the sample stream. For suggested installations for sample conditioning, see Section 14.

In some cases, it may be impossible to arrive at an ideal solution, and periodic cleaning, inspection, and part replacement will be required.

17.10 Startup

The manufacturer's instructions should be followed in startup; however, regardless of the make of instrument, the following points must be observed:

1. Check all connections for tightness. Visually trace all sample lines from sample point to sample disposal. Check for continuity, proper slope, and absence of pockets.

2. Check out all utility and power supplies for proper connections, correct voltages or pressures, and for safety.

3. Check out analyzer circuit for loose connections and for function. Make certain that all field connections have been made in accordance with the manufacturer's appropriate wiring diagram.

4. Start flow through the sample loop and establish proper pressures and flow rates. Make certain that there are no leaks.

5. A sampling system should never be connected to the instrument until repeated tests assure that the sample is clean, representative, and measurable.

6. Admit sample to analyzer. Establish proper flow rates, pressures, and levels.

7. Check all controls for operability and stability. Check all valves for operability.

8. Turn on heaters. It is advisable to watch the instrument closely during this warm-up period. Observe the instrument at frequent intervals until equilibrium seems to have been established.

9. After operation seems satisfactory and stability has been achieved (this may be hours or even days) the results may be checked by the appropriate method selected (see 17.8).

17.11 Shutdown

Shutdown should be made in accordance with the manufacturer's instructions. The following steps should be observed, where applicable:

1. Turn off power to heaters.
2. Allow sample to flow until analyzer is cool.
3. Shut off sample at source.
4. Drain analyzer and flush out if required.
5. Take precautions to prevent freezing in cold weather by heating or flushing with a lighter oil.
6. Lock the door of the analyzer housing.

REFERENCES

Manufacturer's literature
Precision Scientific Development Co., Chicago, Ill.
TOTCO, Division of Baker Oil Tools, Glendale, Calif.

SECTION 18—MOISTURE ANALYZERS

18.1 Scope

This section covers installation of the more commonly used types of continuous stream moisture analyzers. Portable analyzers, as well as those types of instruments whose primary function is laboratory analysis, will not be discussed.

18.2 General

The measurement of the moisture content of a process stream can be accomplished by several types of moisture analyzers. This discussion covers only the more commonly used techniques in both liquid- and gas-phase analysis. All variations of each technique will not be described since the installation practices are similar. These analyzers can be equipped with control devices, transmission systems, and alarm contacts and safety devices (see RP 550, *Manual on Installation of Refinery Instruments and Control Systems,* Part I).

18.2.1 DEW-POINT PRINCIPLE

Dew point is commonly defined as the temperature at which vapor begins to condense out of a gas. It can also be defined as the saturation temperature corresponding to the partial pressure of water vapor. In moisture measurements, the dew point is that temperature at which the water in the sample gas begins to condense.

18.2.1.1 Mirror Type

One type of instrument for measuring dew point, illustrated in Figure 18-1, utilizes a chamber fitted with an inlet nozzle. The inlet nozzle causes a small stream of gas to impinge on a mirror which has been cooled to a temperature below that of the sample dew point. The mirror temperature is then adjusted automatically to the dew point of the gas.

In this method, the two ports in the gas chamber allow a beam of light to reflect from the mirror and be measured by a phototube. A second phototube monitors the light source directly. The difference between the two signals represents the size of the dew spot on the mirror. Electronic circuits maintain the difference in phototube output at a preset value by varying the amount of power to a heater coil. By controlling mirror temperature and the subsequent size of the dew spot, equilibrium is maintained. The mirror temperature is then recorded as the dew point of the gas.

18.2.1.2 Hygroscopic-Salt Type

The hygroscopic-salt type of element, shown in Figure 18-2, is used to measure the dew point of natural gas, carbon dioxide, nitrogen, hydrogen, oxygen, and other gases which will not react chemically with the salt.

The basis of operation is the behavior of a hygroscopic salt, normally lithium chloride, in the presence of water vapor. Water vapor is absorbed, forming a salt solution, and the solution is heated until the rate of evaporation of moisture equals the rate of absorption. For a known pure salt, the temperature at which equilibrium occurs may be related to dew point.

The sensing element normally consists of a thermometer bulb inside a thin-walled metal tube covered with salt-impregnated woven-glass tape. The tape is wound with two separate but parallel wire windings, and a relatively constant voltage is applied causing a current to flow through the salt solution between the wires. This increases its temperature until equilibrium is reached between absorption and evaporation.

Equilibrium temperature may be recorded directly on an instrument which reads in either dew point, grains of moisture per standard cubic foot, percent by volume of water vapor, or grains of moisture per pound of dry gas.

18.2.2 ELECTROLYSIS

With the electrolysis type of measuring instrument, see Figure 18-3, the analysis is accomplished by continuously and quantitatively absorbing and electrolyzing all water present in a gaseous sample stream entering a cell. The electrolysis current, which is related directly by Faraday's law of chemical equivalents to the mass rate of flow of water into the cell, is used as the indication of water content.

The heart of the instrument is the electrolysis cell, in which both absorption and electrolysis take place simultaneously. It consists of a tube which has a pair of closely spaced, usually platinum, wires wound in a double helix on its inner surface. The space between these wires is filled with phosphorous pentoxide. A dc voltage is applied to the elements of the cell and the electrolysis current is measured with an indicator or recorder. With a constant gas flow, the amount of current required for electrolysis is a precise measure of the amount of water present in the parts-per-million range.

This type of instrument can be used to measure low concentrations of moisture in liquids. The first step is to strip water from the sample with a dry gas and the second to pass water vapor and stripping gas to the electrolytic cell. Figure 18-4 illustrates the equipment for this purpose, showing the dryer for stripping gas, the stripping column, and the gas outlet which is connected to an electrolysis cell similar to that shown in Figure 18-3. The liquid flow rate to the column and column temperature must be precisely controlled to obtain a quantitative measurement of moisture. The flow rate of the stripping gas is not critical; however, the measurement of the moisture content of the gas after stripping is subject to limitations. For example, electrolytic-type analyzers cannot always be used successfully under the following conditions:

1. Streams containing materials which will polymerize in the cell, such as butadiene or other olefins or diolefins, cannot be analyzed.
2. Streams containing basic substances, such as ammonia, which will react with the phosphorous pentoxide to form phosphates cannot be analyzed.
3. Hydrogen-rich mixtures sometimes cause erroneous readings because hydrogen will react with the oxygen produced by the electrolysis process, resulting in a higher concentration of water than is present in the sample.
4. Streams which contain more than 2,000 parts per million of water will saturate the phosphorous pentoxide causing a short in the electrodes or will wash the phosphorous pentoxide from the cell. Even below this concentration the probe exhibits a great deal of hysteresis when there is a change in the moisture content of the process stream. Therefore, this analyzer may not be suitable when tight moisture control is required.

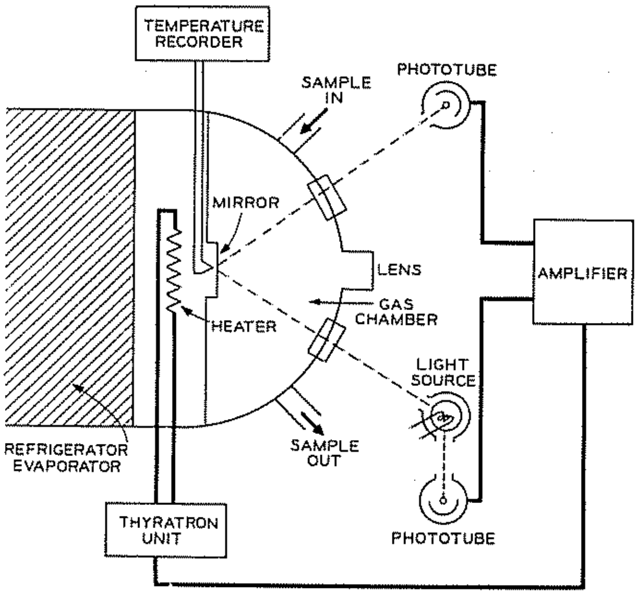


Figure 18-1—Mirror-Type Instrument for Measuring Dew Point

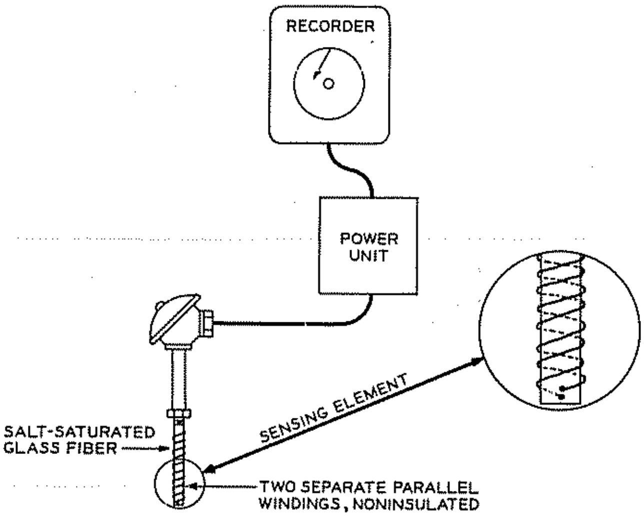


Figure 18-2—Hygroscopic-Salt Type of Measuring Instrument

These limitations should be carefully reviewed and the manufacturer should be consulted for information concerning components specifically designed to minimize operating problems.

18.2.3 INFRARED

With the infrared analyzer, see Figure 18-5, two beams of infrared radiation are directed to travel through parallel cells. One beam traverses the sample cell and the other traverses a comparison cell. The emergent radiation from both cells is directed into a comparison detector. When the stream to be analyzed is introduced into the sample cell, the stream absorbs and thus reduces the radiation reaching the detector from the sample beam. As a result, the relative intensities of the two beams are unbalanced, and the detector generates an electrical signal which is proportional to the difference between the two radiation beams. This signal is then amplified and recorded as a measure of difference in sample composition or moisture content. Infrared techniques are adaptable to either liquid- or vapor-phase analysis in concentrations from low parts per million to several percent.

18.2.4 CAPACITANCE

In the capacitance-type instrument, see Figure 18-6, the sensing unit is a rugged capacitance cell with dessicant filling the space between the plate electrode surfaces. Moisture content of the sample stream changes

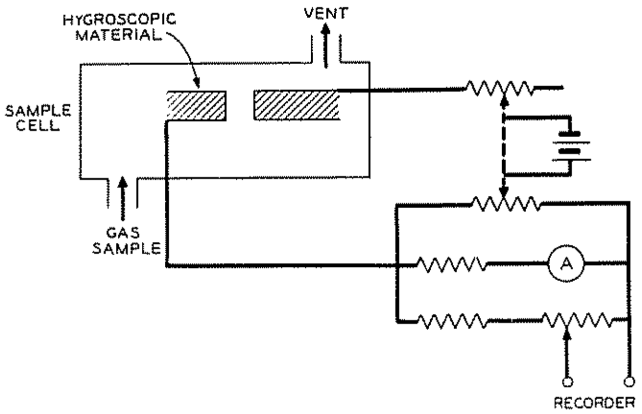


Figure 18-3—Electrolysis Measuring Instrument

the electrical capacity of the system. This change is electronically detected, amplified, and converted to a signal which can be used to operate indicators, recorders, controllers, or alarms.

The capacitance type of analyzer has been successfully used in measuring the moisture content of a wide variety of liquids, such as propanes, butanes, benzene, gasoline, kerosine, and transformer oils as well as gas streams, such as hydrogen, natural gas, nitrogen, and air streams. Polar compounds added to these streams may adversely affect the analyzer results. Temperature control of the sample is essential. The instrument can be calibrated to read out in percent moisture or in parts per million. For gaseous samples, the analyzer can be calibrated in ranges from 1 to 1000 parts per million of moisture. For liquid samples, the analyzer will measure moisture from 5 to 60 percent of solubility of water in the liquid.

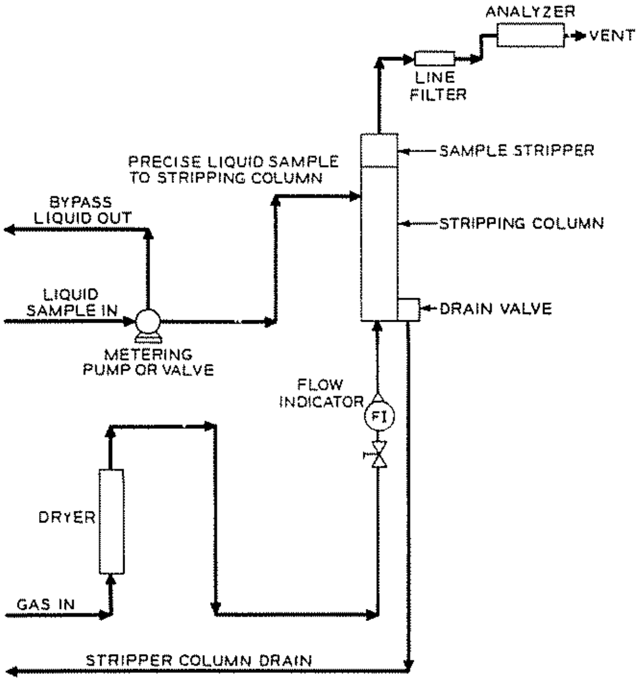


Figure 18-4—Liquid Sample Dry-Gas Stripping System

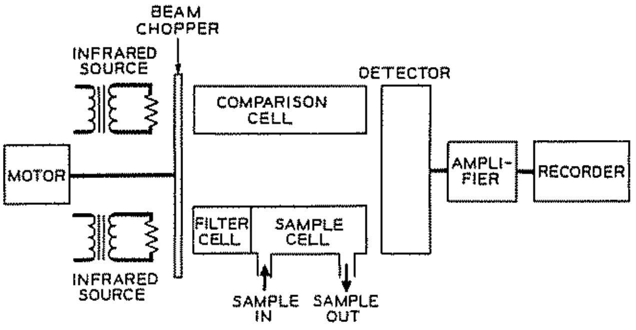


Figure 18-5—One Form of Infrared Analyzer

18.2.5 VIBRATING CRYSTAL

In the vibrating crystal analyzer, see Figure 18-7, moisture content is measured by comparing the changes in frequency of two hygroscopically coated quartz crystal oscillators which when dry vibrate at a nominal 9 million hertz. Water vapor is alternately absorbed and desorbed on each crystal, resulting in a mass difference with a corresponding change in frequency. These changes in mass of the coating are compared electronically, and moisture content is indicated on the analyzer scale in parts per million by volume. This analyzer can be calibrated to readout in ranges from 0 to 5 parts per million full scale up to 0 to 25,000 parts per million full scale.

This type of analyzer overcomes the problem of measuring dynamic changes of moisture concentration by employing two crystals and a system for flow switching. One crystal is exposed alternately to the moist sample gas and then to a dry reference gas every 30 seconds. The other crystal is kept dry and used as a reference.

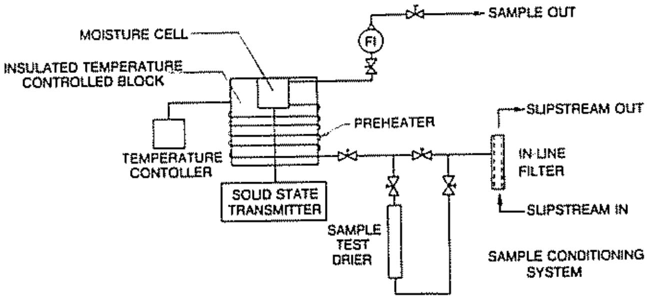
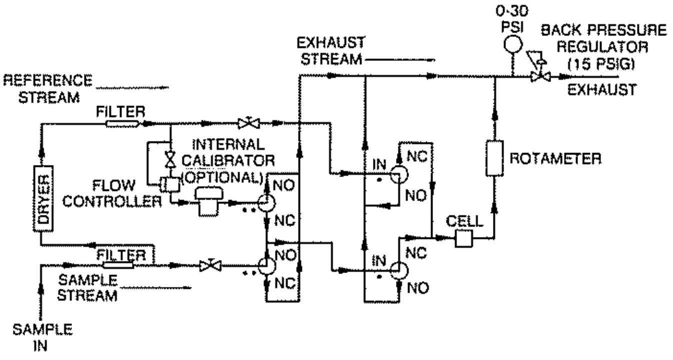


Figure 18-6—Capacitance Cell Type Moisture Analyzer



* energized
* * de-energized

Figure 18-7—Vibrating Crystal Moisture Analyzer

18.2.6 ALUMINUM-OXIDE SENSOR PROBE

The aluminum-oxide moisture analyzer, see Figure 18-8, employs a sensor probe which is inserted into a liquid or gaseous slipstream for measuring moisture content. The sensor consists of an aluminum strip which is anodized by a special process to provide a porous oxide layer. A very thin coating of gold is deposited over this structure. The aluminum base and the gold layer form the two electrodes of what is essentially an aluminum-oxide capacitor. Water molecules transport through the gold layer and equilibrate on the pore walls in a manner which is functionally related to the moisture content of the sample stream. The number of water molecules absorbed on the oxide structure determines the conductivity of the pore wall. Each value of pore wall resistance provides a distinct value of electrical impedance which in turn is a direct measure of the water vapor pressure. Each sensor probe has its own calibration curve and is capable of measuring water content of less than 1 part per million up to 20,000 parts per million.

The aluminum-oxide probe is susceptible to severe corrosion in the presence of certain acids and bases and in process streams with high-moisture content. If these components are present in the stream to be analyzed, the manufacturer should be consulted on the suitability of this analyzer for the application. Where these conditions exist only during special times (for example, the catalyst regeneration cycle on a hydrogen reformer) a bypass system can be provided as shown in Figure 18-8 to protect the analyzer. A dry gas purge is recommended while the analyzer is bypassed to protect the probe and prevent moisture accumulation in the sample system. Also, the aluminum-oxide probe is somewhat fragile and requires a protective sleeve in excessively turbulent streams.

18.2.7 OTHER INSTRUMENTS

There are several analytical instruments other than those mentioned here which are capable of determining moisture. Near-infrared, ultraviolet, and chromatographic analyzers have been used successfully. As these instruments are generally more complex and expensive, they are not used extensively for moisture determination in refinery processes.

18.3 Sampling System

The function of the analyzer is the designer's first consideration. The degree of sample preparation must be determined in order to supply a representative sample to the analyzer.

Careful considerations must be given to the design and construction of the sampling system. The designer should know from exploratory analysis the condition, composition, and contaminants of the sample and should provide necessary components to prepare a sample compatible with the analyzer without changing the moisture content. Special attention must be paid to any stream which contains a component which will tend to coat the cell. Pertinent information concerning construction materials, sample line velocities, component volumes, and other factors concerning good sampling practice is presented in Section 14.

18.3.1 SAMPLING SYSTEM FOR PARTS-PER-MILLION ANALYZER

Proper design and construction of the sampling system are the most important features in the installation of a parts-per-million moisture analyzer. Some of the important features the designer should take into consideration are the following:

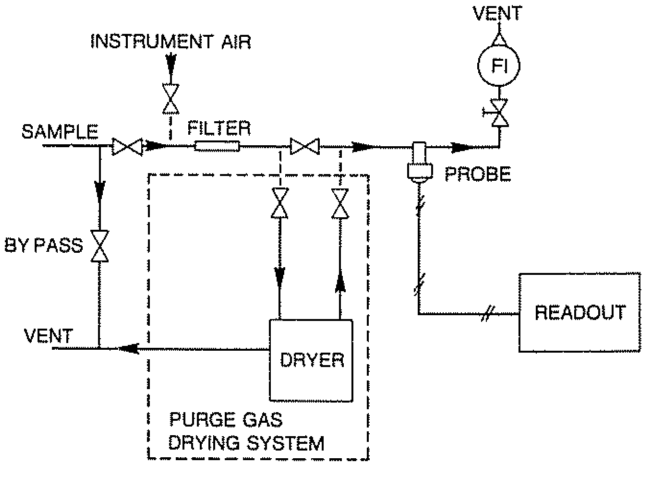


Figure 18-8—Aluminum-Oxide Probe Type Moisture Analyzer

1. *Moisture holdup*—To minimize moisture holdup, the smallest and shortest possible lines should be used for sample loops. Only minimum-volume scrubbers and filters should be employed and, when necessary, pressure should be reduced as near the sample point as possible.
2. *Particle contamination*—The use of sintered stainless steel filter elements will minimize particle contamination. In some installations, it is necessary to install large filters in the main bypass line to remove the bulk of the larger particles.
3. *Adsorption, desorption, and moisture penetration*—The use of stainless steel lines and equipment properly installed (pressure tight) will minimize penetration of moisture from the atmosphere. In addition, sample lines can be passivated with a solution of 20 percent nitric acid and 2 percent hydrochloric acid for 30 minutes. The acid is removed with water and the line is then dried with alcohol. All components in the system, such as filters, pressure regulators, valves, and flow controllers, should be examined for materials which may absorb moisture from the sample. The use of rubber or plastic regulator diaphragms, oil-base pipe dope, and bourdon-type pressure gages should be avoided.
4. *Vaporizing and heating*—Frequently, liquid samples must be analyzed in instruments designed to handle vapors only. For features pertinent to vaporizing, see Section 14. Sometimes it is necessary to heat the entire sampling system to avoid condensation which will cause erratic analyzer operation.

18.3.2 SAMPLING SYSTEM FOR HIGHER RANGE MOISTURE ANALYZER

In general, the same considerations should be given to the design of the sampling system for higher range moisture analyzers as outlined herein. The degree of accuracy required may permit relaxation of certain of these requirements.

Some types of moisture analyzers do not require sample preparation systems. Normally, this type of instrument uses a sturdy stationary probe installed in the process medium.

18.3.3 SAMPLE-POINT LOCATION

A sample point should be chosen which requires the least sample preparation equipment to provide a representative sample in the proper physical state for the analyzer. The location should be chosen as outlined in Section 14.

18.3.4 CELL PROTECTION FOR PARTS-PER-MILLION RANGE

In certain types of moisture analyzers, such as the electrolysis type, serious cell damage can occur when excessively high moisture content is experienced. In the vibrating crystal type, liquid droplets can cause damage to the cell coating. The manufacturer of these analyzers should be consulted on what type of protective system is required to prevent cell damage.

18.3.5 MATERIALS OF CONSTRUCTION

The important factors in the selection of material for a sampling system are corrosion resistance, safety, and error prevention (see Section 14).

18.4 Location and Housing

It is desirable to locate the analyzer as close to the sample point as possible and in an area which is vibration-free. Care should be taken to provide easy access to the instrument for proper maintenance. In order to minimize sample handling problems special attention should be given to the location of parts-per-million analyzers.

To obtain maximum efficiency, accuracy, and stability, analytical instruments should be afforded housing in addition to the weatherproof or explosionproof enclosure provided by the manufacturer. It is sometimes necessary to thermostatically control analyzer shelters when analysis is being made in the parts-per-million range. See Section 14 for a more detailed description of analyzer shelters.

18.5 Effluent Disposal

Consideration should be given to the safe disposal of the sampled process material which cannot be economically returned to the process system. Small amounts of nontoxic liquids often can be vented to convenient process sewers. Lighter materials may be vented to the atmosphere. Large samples of nontoxic and all toxic materials should be vented or pumped to vessels or other closed systems. Caution should be exercised to ensure that the analyzer back pressure remains constant and does not exceed the manufacturer's recommendations.

18.6 Safety Considerations

The installation of the analytical instrument and the data presentation equipment should be made in accordance with national and local codes which are applicable to the respective locations. Generally, the analyzer sections should meet the area classification requirements given in *National Electrical Code*, Article 500.

18.7 Readout

The moisture monitors described in this section can be adapted to read out on most standard pneumatic or electrical indicating or recording instruments for data presentation or control. In some cases, transducers may be required to obtain a compatible signal.

18.8 Startup

Before attempting to put the analyzer onstream, the following steps should be taken:

1. Train the operating and maintenance personnel prior to startup, making certain that everyone is thoroughly familiar with the limitations of the analyzer and with checkout and startup procedures.
2. Make a final check of the system to assure that all electrical and sample line connections are tight and connected properly.
3. Check sampling system by putting a glass or plastic tube, or container, in the position of the analyzer. Pass the sample through the container for several hours to determine that the sampling system is working properly and that no contamination will reach the analyzer.
4. Check and adjust temperature of heating equipment on sampling systems requiring controlled temperatures.
5. After the system has been flushed and purged of contaminating particles and excess moisture, the next step is to dry out the lines and bring them into equilibrium with the material being sampled. Often the parts-per-million sampling system will require several days to reach equilibrium. Flow through the analyzer cell should not be started until the moisture concentration is less than the maximum scale reading. To assure analyzer availability and reduce dry-down time, it is sometimes necessary to connect a cylinder of dry gas to the system for a day or so of operation.
6. On certain types of analyzers, it is desirable to electrically energize the instrument for a period of time in order to stabilize it prior to introducing the sample.

18.9 Calibration

Most moisture analyzers require periodic calibration. Some analyzers can be calibrated by analyzing a sample of known composition or by laboratory analysis of a sample from the process stream. However, as moisture tends to absorb or desorb from the walls of a typical cylinder, neither method is entirely satisfactory when precise measurements are desired. Specially coated aluminum cylinders should be used. There is also a problem in withdrawing a representative sample from the sample containers.

18.9.1 CALIBRATION OF PARTS-PER-MILLION ANALYZER

Some analyzers in the parts-per-million range (for example, the electrolysis type) are not equipped with calibration adjustments. With these analyzers, the primary consideration is the precise control of sample flow rate. The actual flow rate can be measured by a soap-film flow technique (time soap bubble through a buret) or a wet test meter.

In many installations, trend recordings only are required (for dryer regeneration) and absolute calibration is unnecessary. Some installations will require accuracy checks. Figure 18-9 illustrates a blending system used in one method for checking accuracy. The sample is split into two streams; one is dried and the other is saturated at a controlled temperature and pressure. The moisture content of the saturated stream can be controlled by regulating the saturation temperature or pressure. The two streams are then blended to give the desired moisture level. The success of this will depend upon precise control of flow rates and the allowance of ample time (usually several hours) for the system to come into equilibrium after a change in moisture content has been made. Another method of calibration is the use of standard moisture gases stored in specially treated aluminum pressurized cylinders. A special lining prevents absorption and desorption of moisture on the cylinder wall so that moisture content remains constant after more than 2 years of storage. Standard samples containing up to 500 parts per million of moisture can be obtained.

Precise calibration of an analyzer can also be done using a moisture generator. The moisture generator method of calibration is based on the principle of controlled permeation of water through a membrane for

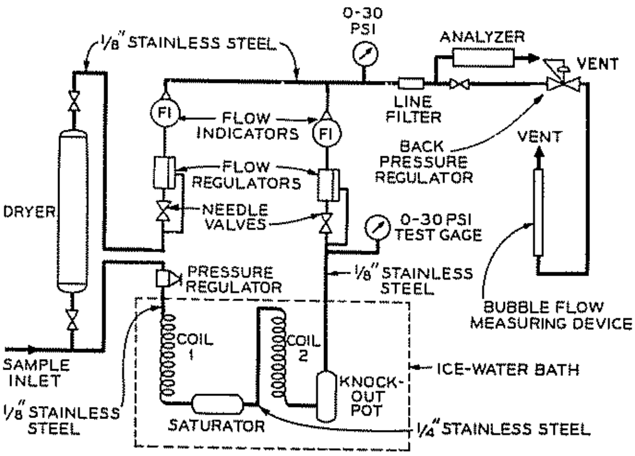


Figure 18-9—Moisture Blender for Calibration Check

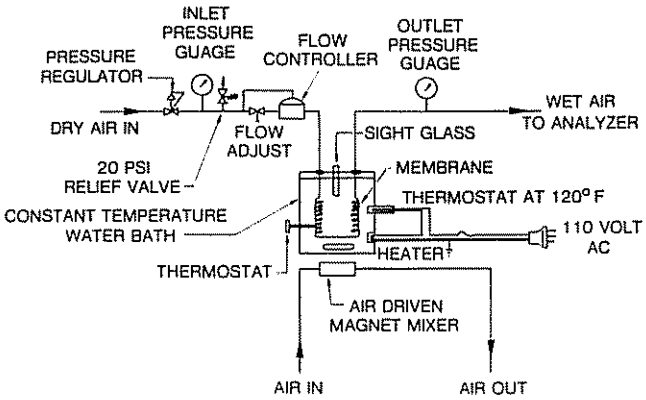


Figure 18-10—Moisture Generator for Calibration Check

example, polytetrafluoroethylene[1] tubing to produce a stream of air or nitrogen at constant moisture. One type of moisture generator is shown in Figure 18-10 In this moisture generator, dry air is passed through polytetrafluoroethylene tubing immersed in a constant temperature water bath. The quantity of water passing into the air stream is a function of bath temperature and membrane geometry. Higher transfer rates can be obtained by increasing bath temperature or by using thinner walled tubing and larger surface areas. This method is used for calibrating analyzers operating in a range of 2 to 125 parts per million of moisture

In the case of liquid analysis, it is possible to prepare a standard sample if special precautions are taken to dry the sample vessel completely.

18.9.2 CALIBRATION OF PERCENT-RANGE ANALYZER

Standard samples are practical for the calibration of percent-range analyzers. A laboratory analysis of a representative sample withdrawn from the process stream usually provides satisfactory information for calibration. The manufacturers of the various types of analyzers should be consulted for any special procedures.

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Halter, R. C., and Johnson, L. M., "Measurement of Trace Water in Refinery Process Streams," *Proc. API*, **41** **[III]**, pp 46-51, 1961.

Schall, William C., "Selecting Sensible Sampling Systems," *Chem. Eng.* **69** **[10]**, pp 157-72, May 14, 1962.

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Mine Safety Appliance Co.
Panametrics, Inc.
TOTCO Instruments

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[1] For example, Teflon.

SECTION 19—OXYGEN ANALYZERS

19.1 Scope

Common practices for the installation of continuous process stream oxygen analyzers are presented in this section. Portable and laboratory types of analyzers will not be discussed. Some of the more general applications for oxygen analyzers in the refining industry are briefly outlined.

19.2 General

19.2.1 COMBUSTION EFFICIENCY ANALYSIS

The oxygen analyzer has become one of the leading combustion efficiency guide instruments primarily because oxygen has a direct relationship to excess air. By sampling flue gases, the operator can use the minimum amount of air for complete combustion and yet obtain the fullest utilization of the fuel burned. Unlike other combustion efficiency measurement methods, percent of oxygen is considered to have a direct relationship to the amount of excess air in the flue gases regardless of the hydrogen-to-carbon ratio of the fuel burned (see Figure 19-1).

Another combustion application is the monitoring of the oxygen content of the fuel gases from fluid catalytic cracking regenerators.

19.2.2 PERCENT OF OXYGEN IN HYDROCARBON STREAMS

Because oxygen is sometimes used as a reactant in refinery processes, it has become customary for some refiners to install oxygen analyzers on these processes to control the amount injected or to sound an alarm before unsafe limits have been reached. Some processes are subject to accidental admission of oxygen which must be guarded against to avoid explosions or fires. Oxygen analyzers have been used as alarms on these systems or to activate safety devices before unsafe limits have been reached.

19.2.3 PERCENT OF OXYGEN IN INERT PURGE OR BLANKETING GAS

Where an inert gas is manufactured or used to purge or blanket vessels, it may be desirable to have a continuous analysis of the oxygen content of the purging gas prior to admission of the gas to the system.

It is also good practice to monitor the oxygen content of the inert gas blanket inside of processing equipment or storage tanks when inadvertent admission of air could cause degradation of the contents of the tank or permit an explosive mixture to exist.

19.2.4 CATALYST REGENERATION

In some catalytic processes, the catalyst is regenerated when the catalytic activity reaches a certain level. This is usually accomplished by purging the reactors with inert gas, starting circulation of the inert gas, and raising the temperature to some required level to promote combustion upon admission of air. The air flow to the system is carefully controlled and the circulated gas is monitored by oxygen analyzers. This allows a controlled burning of the carbon deposits from the catalyst. Control of the burning rate is important in order to avoid temperatures which will cause permanent damage to the catalyst.

19.3 Basic Types

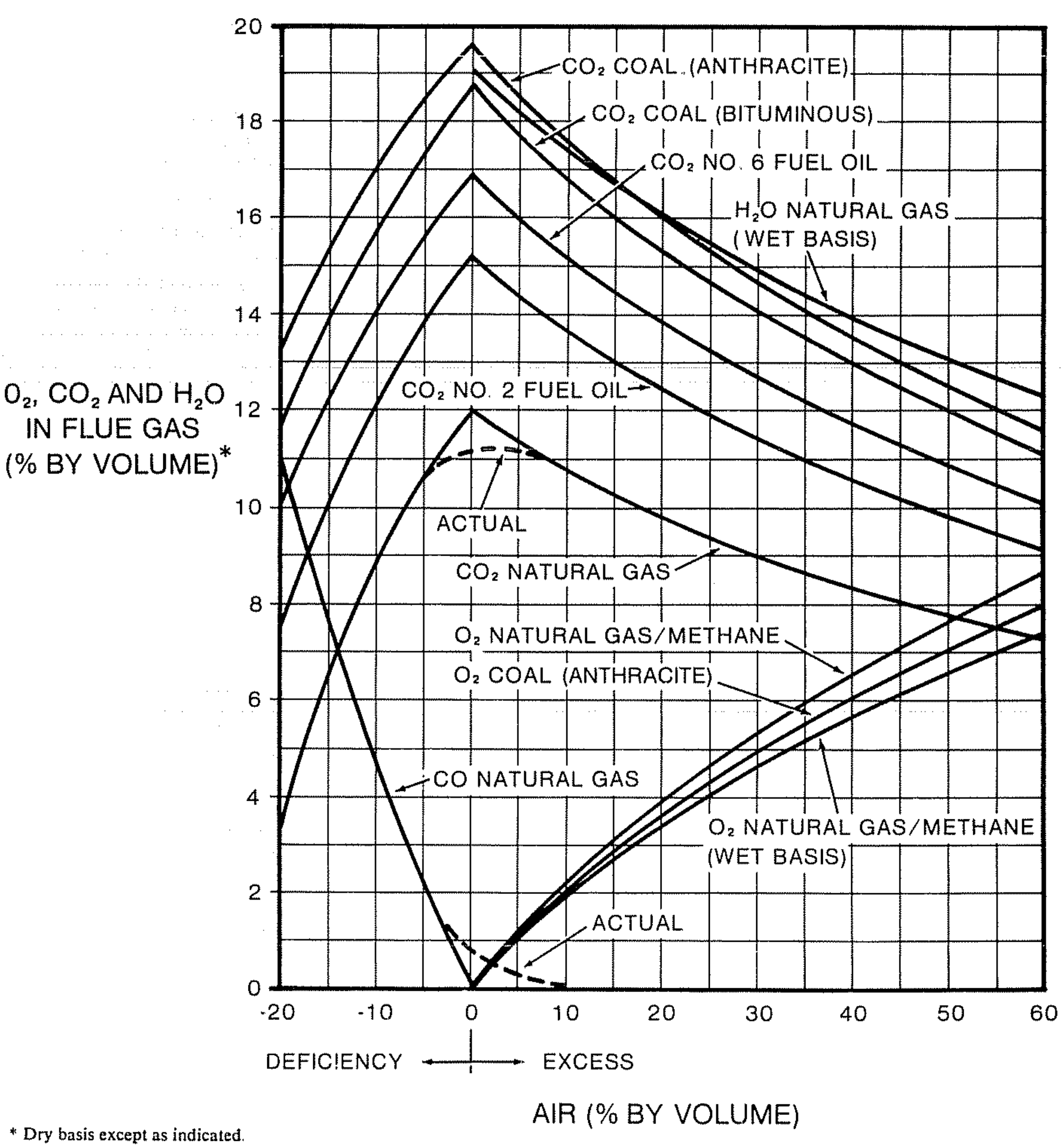
There are two basic types of oxygen analyzers currently suitable for refinery process stream applications: electrochemical and paramagnetic. Oxygen analyzers can be equipped with alarm contacts, transmission systems, and control and safety devices.

19.3.1 ELECTROCHEMICAL PRINCIPLE

Electrochemical oxygen analyzers consist of at least two metallic electrodes in good electrical contact with an appropriate electrolyte. The chemical reactions occurring in the cell produce an output current or voltage which is a function of the number of oxygen molecules diffusing into the interface between the electrolyte and the measuring electrode. The various types of electrochemical analyzer cells are conveniently classified by the type of electrolyte used: aqueous solutions of various metal salts and soluble organic compounds or zirconia ceramic doped with calcia, yttria, and scandia.

19.3.1.1 Aqueous Electrochemical Type

Figure 19-2 illustrates a typical aqueous electrochemical oxygen analyzer cell. Oxygen molecules diffuse through the membrane to the cathode where they are reduced to OH— ions upon acceptance of four electrons produced by an appropriate reaction at the anode. The materials used in these cells depend upon the individual manufacturers' particular designs. The cells may be operated in a variety of modes at temperatures between 0 and 50 degrees Celsius.



* Dry basis except as indicated.

Figure 19-1—Distribution of Products from Combustion of Various Fuels

a. POTENTIOMETRIC MODE

The open circuit output voltage of the cell is a function of the log of the concentration of oxygen in the cathode/electrolyte interface. While nominally responsive to oxygen only, the electrodes also respond in varying degrees to other oxidizing and reducing gases which may be present, such as Cl_2, SO_2, H_2S, NO, and so forth. Sulfur bearing gases may also poison the electrode surfaces if insoluble sulfur-bearing compounds are formed.

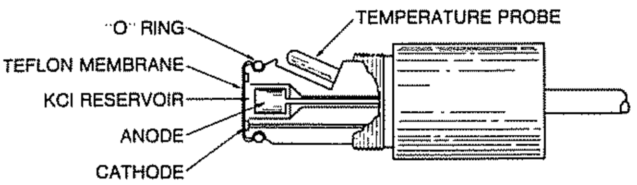


Figure 19-2—Typical Aqueous Electrochemical Cell

b. AMPEROMETRIC MODE

The output of this mode of operation is a current proportional to the number of oxygen molecules reaching and being reduced at the cathode/electrolyte interface. This mode is subject to the same interferences as is the potentiometric mode. The linear calibration, however, is obtained at the expense of increased sensitivity to the diffusion rate of oxygen molecules to the cathode/electrolyte interface and, therefore, to the temperature of the cell and the condition of the membrane.

c. POLAROGRAPHIC MODE

The output of the cell is a current proportional to the concentration of oxygen at the cathode/electrolyte interface which flows in the external circuit when a bias voltage is applied between cathode and anode. This bias voltage is just below the oxidation/reduction potential of the oxygen/electrode system of the cell and suppresses the effects of other electrode reactions with lower oxidation/reduction potentials. Diffusion rate and electrode poisoning are still serious problems.

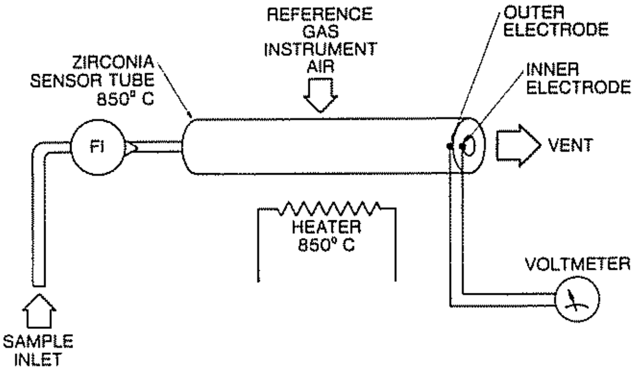
Specially modified polarographic-type electrochemical cells are also widely used for determining carbon monoxide concentrations below 4,000 parts per million.

19.3.1.2 Zirconia Electrochemical Type

These high-temperature zirconia electrochemical cells consist of a thin section of doped zirconia, usually a disc or a tube whose opposite surfaces are coated with porous platinum electrodes. When operated at a temperature of about 850 degrees Celsius, the potential difference between the two electrodes is proportional to the log of the ratio of the partial pressures of oxygen existing at the two electrode/electrolyte interfaces. Figure 19-3 shows a typical arrangement of such a cell.

Instrument-quality dry air is normally used as the reference gas on one side of the cell while the other is exposed to the sample gas. The temperature of the zirconia must be controlled very accurately if significant errors are to be avoided. For this reason, care must be taken to minimize changes of the heat load supplied by the heater. These changes are caused primarily by the temperatures and flow rates of reference and sample gases at the electrode surfaces.

Any combustible gases present in the sample or reference gases will be burned completely before the gases reach the surface of the electrodes since 850 degrees Celsius is well above the ignition temperature of fuel gases. Thus, the partial pressure of oxygen sensed by the zirconia cell will be that remaining after all combustible gases have been burned. This characteristic becomes quite important because flue gases contain both excess oxygen and combustible gases. When determining low residual oxygen concentrations in inert gases, care must be taken to remove all organic contaminants, which will act either as fuel or release oxygen under other than equilibrium conditions, from the sample tubing.



NOTE: Voltage output is proportional to the logarithm of reference and test gas oxygen partial pressure ratio.

Figure 19-3—Zirconia Electrochemical Analyzer Schematic Diagram

Figure 19-4 shows a typical installation of a probe-type zirconia analyzer directly into a flue gas stream. In this probe the zirconia cell is enclosed by a porous ceramic cup through which the sample gas must diffuse. Most other oxygen analyzers using zirconia cells are located immediately outside of the flue and use simple open probes and air aspirators to transport the sample gas over the measuring electrode and back into the stack.

More elaborate sampling systems are used in many instances particularly for dirty, wet applications and when the flue temperature exceeds the temperature limit of the analyzer.

19.3.2 PARAMAGNETIC PRINCIPLE

Oxygen has the unusual characteristic of paramagnetism which is defined as the property of being attracted by a magnetic field. Most other gases are diamagnetic and are repelled by a magnetic field. Other low background gases, such as nitric oxide and nitrogen dioxide, having appreciable paramagnetic properties are not normally encountered in significant concentrations in refinery operations. When analyzers with a range of 2 percent or less are used, these low background gases

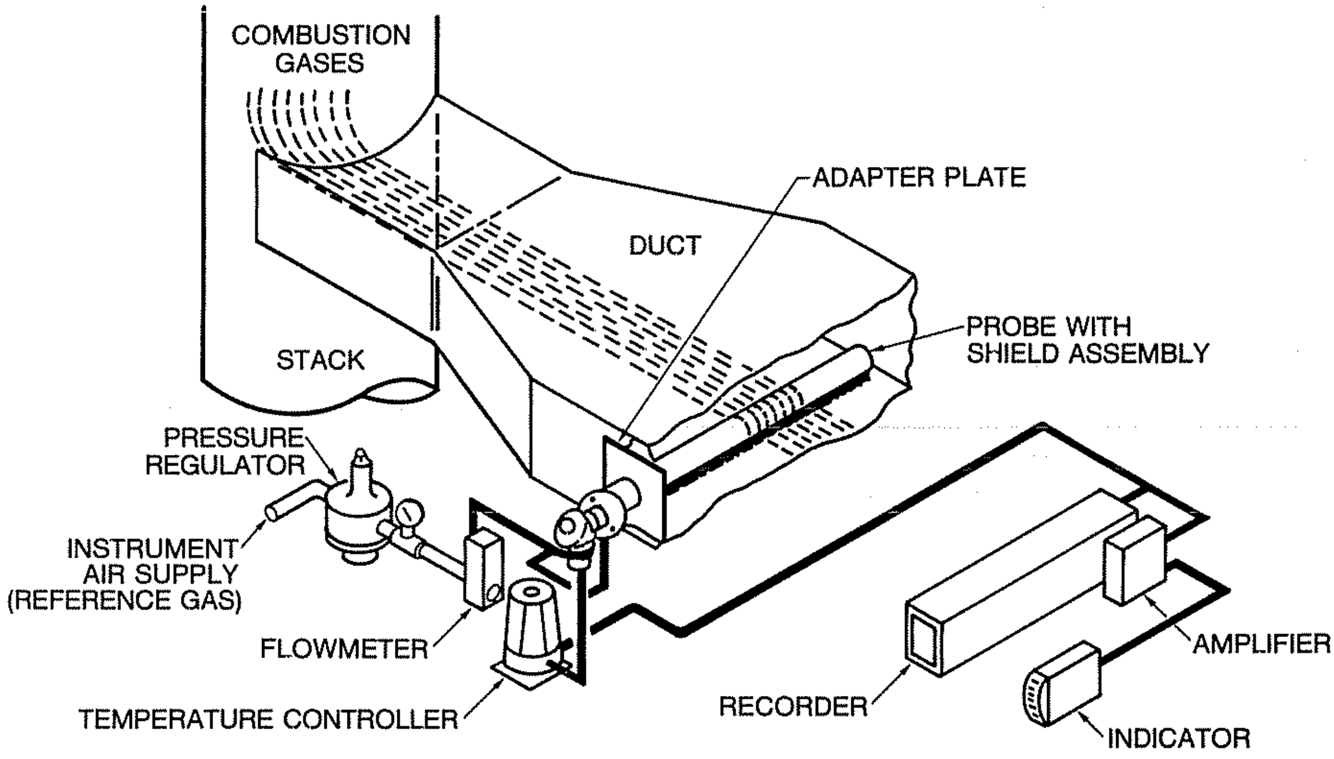


Figure 19-4—Probe-Type Zirconia Electrochemical Oxygen Analyzer

and certain hydrocarbon gases may cause predictable errors if their concentrations vary. All paramagnetic gases become less susceptible to magnetic attraction as the temperature of the gas is increased.

Two methods which have been developed to utilize the paramagnetic properties of oxygen as a measure of oxygen content in a gaseous stream are described in 19.3.2.1 and 19.3.2.2.

19.3.2.1 Magnetodynamic Type

One method, illustrated in Figure 19-5, measures the degree of deflection of a precison-built test body of definite magnetic susceptibility and counters the deflection by means of an electrostatic force. The test body, in the shape of a dumbbell, is suspended in a manner similar to a galvanometer in a nonuniform magnetic field. The force tending to deflect the test body is a function of the difference in magnetic susceptibilities of the test body and the surrounding gas. If the gas is more paramagnetic than the test body, the magnetic force tends to expel the test body. If the gas is less paramagnetic, the test body is drawn toward the magnetic field. The magnitude of this force is proportional to the oxygen content of the gas being sampled. Another design counters the deflection by means of a current feedback loop attached to the dumbbell.

19.3.2.2 Thermal Paramagnetic Type

The flow of gas which results from paramagnetism has been utilized in the thermal paramagnetic oxygen analyzer. The cooling effect of the gas flow is used to cool the filament of a thermal conductivity cell, thus changing its resistance. The flow of gas through the cell is a product of magnetic and thermal convection currents, and it is necessary to eliminate that flow created by thermal convection. This is usually accomplished with a reference or compensating cell which also eliminates or minimizes other interfacing signals not of magnetic origin. The difference in resistance caused by convection cooling of the two cells is a direct measurement of the oxygen content of the gas. There are various modifications to obtain special effects, but it is not the intent of this section to describe each method used. Some of the more commonly used thermal paramagnetic types of analyzer cells are shown in Figure 19-6.

19.4 Sampling

19.4.1 SAMPLE-POINT LOCATION

When sampling of the gas is required, it is important

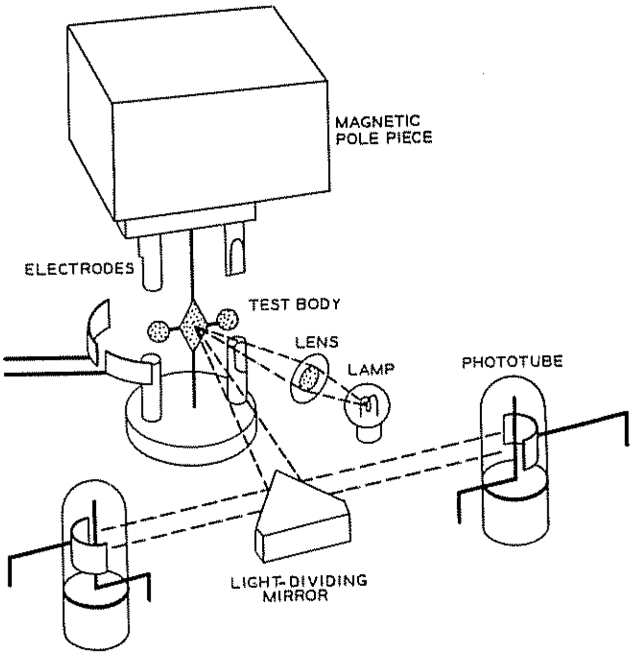


Figure 19-5—Magnetodynamic Paramagnetic Oxygen Analyzer

that the sample point should be located to provide (a) a clean and representative sample, (b) a system requiring minimum maintenance, and (c) a system which is readily accessible for servicing. Many of the principles discussed in Section 14 are applicable.

19.4.1.1 Combustion Systems

In locating a sample point where the oxygen analyzer is to be used for monitoring combustion efficiency, it is necessary to consider the adverse effect of air leakage. In fired heaters and boilers, air infiltration through open peepholes, leaky brickwork, or air preheaters might cause the analyzer to indicate excess combustion air while the actual firing zone may be starving for air, thus resulting in inefficient operation.

To assure efficient combustion, the analyzer should measure the gases leaving the actual combustion zone. A common sample-point location is in the heater stack or breeching. These locations represent small areas through which the combustion gases must pass and, therefore, tend to assure a representative sample for analysis. Another sample-point location often advocated by burner and heater design engineers is the firebox area where the gases more nearly represent the combustion product. For samples from such areas to be meaningful, it is important to seal off the burners which are not being used as well as other sources of air infiltration.

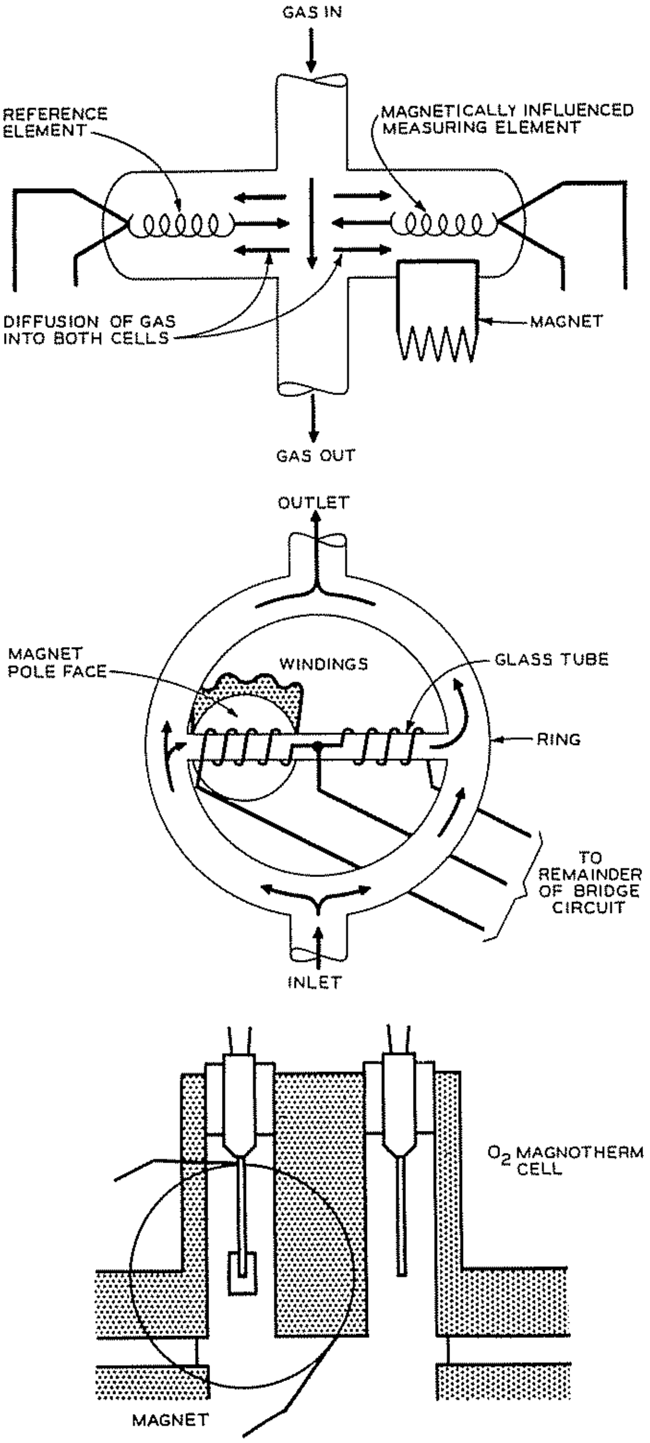


Figure 19-6—Thermal Paramagnetic Oxygen Analyzers

In some large boilers or heaters, detection of air leakage and prompt repair is important to avoid excess fuel consumption. In such installations, samples from both the breeching or stack and the combustion zone may be taken. The difference in oxygen content between the samples will indicate excessive air infiltration and the need for corrective measures.

19.4.1.2 Other Process Systems

Where positive pressure streams (such as enumerated in 19.2.2 through 19.2.4) are sampled, the normal considerations as outlined in Section 14 should be followed. To minimize lag, the sample point should be located as close to the point of stream origin as practicable and yet obtain a well-mixed, representative sample of the material to be analyzed.

19.4.2 TRANSPORTING SAMPLE

19.4.2.1 Positive Pressure Systems

To obtain a sample from a system under pressure, the method, as outlined in Section 14, is much the same as for other analyzers. The main consideration is the transportation of a clean sample to the analyzer with minimum lag and without having changed the oxygen content. Figure 19-7 is a typical example of the positive pressure system.

Normal requirements of this system are the following:

1. An adequate pressure-reducing system
2. A filtering system for removal of particulate matter
3. A cooler, if required
4. Water wash or water removal system, or both, where needed.

On some systems, such as a fluid catalytic cracking regenerator sampling system, use of a cyclone before the filter generally improves the filter life.

19.4.2.2 Negative Pressure Systems

In negative pressure systems, water or steam aspirators are commonly used to deliver the sample to the analyzer, although vacuum pumps are used in some applications. Special care must be practiced to ensure a leakproof sampling system wherever negative pressure is encountered. Any leak will allow air to enter the system and result in an analysis error. On an installation monitoring combustion-product flue gases, a leak would create oxygen readings in excess of the actual amount of oxygen and could lead to firebox operation with oxygen deficiency. This deficiency would result in unburned fuel, lowered efficiency, and the possible hazard of explosion or release of toxic carbon monoxide (CO), or both.

Some users have overcome this problem with systems as shown in Figure 19-8 in which a steam or water aspirator is located inside of the vessel or chamber being sampled. This solution provides a positive pressure sampling system from the point of sample origin to the analyzer. Where sampling systems of this type are considered desirable, it should be ascertained that the oxygen (desorbed into) or the carbon dioxide and oxygen (absorbed from the sample by the water) required in this method of sample induction does not substantially affect the accuracy of analysis. Some of the more commonly used systems for negative pressure sampling are shown in Figures 19-9 through 19-11.

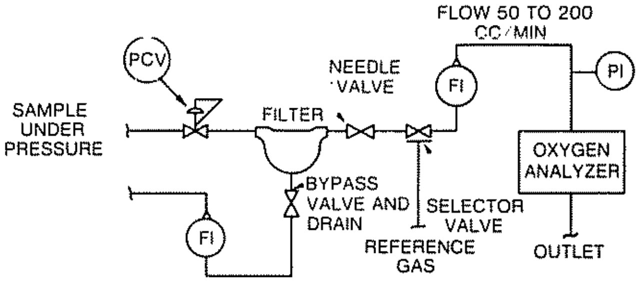


Figure 19-7—Positive Pressure Sample Preparation

19.4.2.3 Corrosion

The sampling system piping and fittings, as well as other components, should be of materials suitable for the sample gases being handled. Selecting material for flue gas sampling is quite difficult, especially in a refinery where several fuels may be burned. Some fuels may be high in hydrogen sulfide, sulfur dioxide, and similar acidic materials whereas other fuels may contain caustic or alkaline materials. These materials, being released in the flue gases, will be drawn into the sampling system where they will, when contacted with water, become agents of corrosion. Materials such as stainless steels and higher alloys have been used successfully.

Heat-resistant glass[1] fitted with rubber connectors has been used but is susceptible to breakage and, for that reason, is not acceptable for some installations. Several refiners report excellent results with ½-inch ID steam hose, polypropylene, polyvinyl chloride, and polytetrafluoroethylene[2] tubing for sample lines in corrosive flue gas service. Temperature limitations of the materials being used should be reviewed with the manufacturer prior to selection.

Material and design specifications for filters, separators, dryers, regulators, and other sample-handling components should be carefully reviewed with the manufacturer to ensure proper selection for the service requirements (see Section 14.).

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[1] For example, Pyrex

[2] For example, Teflon

19.5 Analyzer

19.5.1 ANALYZER LOCATION

In the following order of importance, the analyzer should be—

1. Properly located for satisfactory sampling,
2. Easily readable by the operator,
3. Accessible for maintenance within economical considerations, and
4. Protected against mechanical damage, vibration, weather and corrosive ambient conditions.

Most process analyzers consist of a measuring cell with associated sample flow rate controls, the necessary electronic signal conditioning circuitry, and power supplies. An indicating meter is normally the only local readout provided. Remote indication is normally accomplished via standardized output signals such as 0 to 10 millivolt, 0 to 1 volt, or 4 to 20 milliampere which are compatible with most standard process control transmitters, recorders, and controllers. Alarm functions may be included in the analyzer, or standard process control system alarms may be utilized.

If the analyzer is located in the best possible place for accessibility, the sampling system may be so complex that it will not operate satisfactorily. On the other hand, if the analyzer is located in a nonaccessible location, the usefulness of the instrument will be limited regardless of how accurately and dependably it operates.

19.5.2 ANALYZER HOUSING

Most presently available stack analyzers are housed in weatherproof, purgeable, and/or explosionproof enclosures and are self-standing. They are also normally designed for severe ambient temperature excursions (typically −32 to 60 degrees Celsius) without damage. In those cases where water or steam aspirators are used or where the sample gas contains condensate, winterizing or tracing methods outlined in API RP 550, Part I, Section 8—Seals, Purges, and Winterizing should be followed.

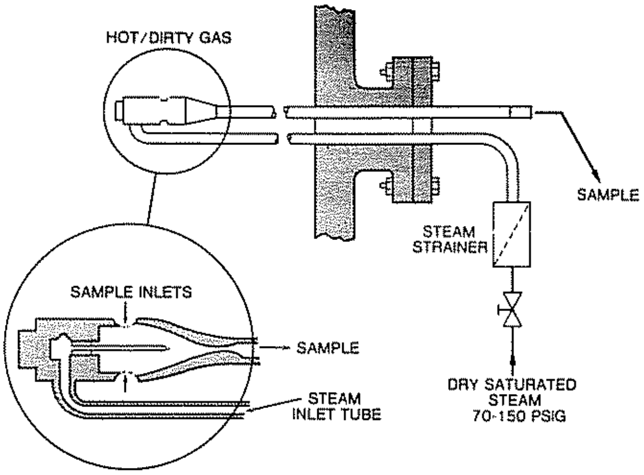


Figure 19-8—Negative Pressure Sample System with Internal Ejector

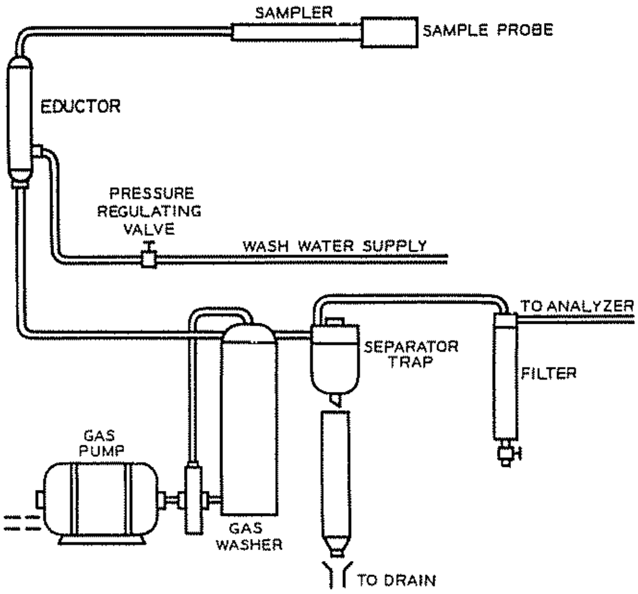


Figure 19-9—Negative Pressure Sample Preparation with Water Wash

Some types of analyzers cannot maintain good calibration stability unless the temperature inside the analyzer is maintained within certain limits. It is recommended that these analyzers located out-of-doors be provided with a weather shield of some type, preferably a total enclosure type of housing. Figure 19-12 illustrates one type of enclosure.

19.6 Safety Considerations

19.6.1 SAMPLE DISPOSAL

Samples of refinery heater flue gases should be disposed of to a safe location for reasons of personnel safety and also explosion hazard. Normally, heater flue sample gas is noncombustible, and therefore no explosion hazard exists. However, consideration must be given to the possibility of mechanical failures and operational upsets which might cause the sample gas to become flammable. Positive measures should be taken to protect against the hazards of this abnormal condition.

Where ignition of the sample gas is possible (for instance, when a zirconia electrochemical cell at 850 degrees Celsius is left operating in a shut down heater not free of fuel gas), consideration should be given to de-

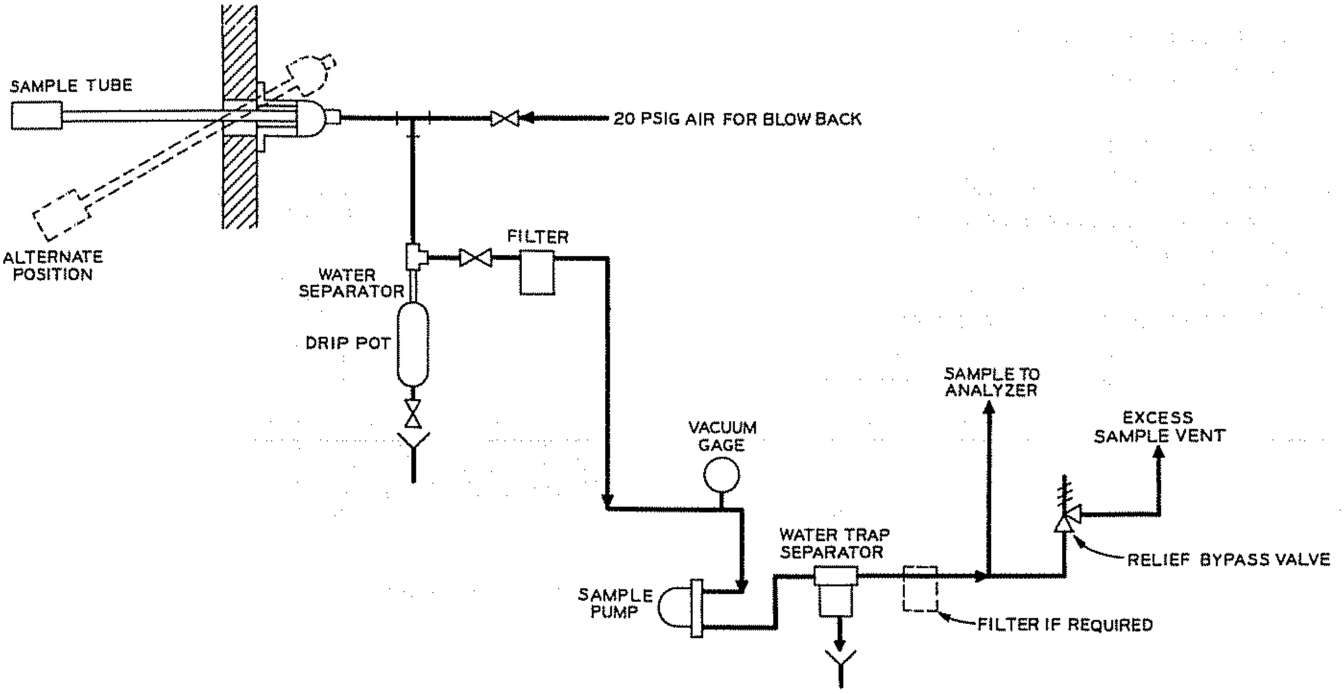


Figure 19-10—Negative Pressure Sample Preparation with Sample Pump

sign principles that prevent back flashing or to the addition of a safey interlock system that shuts down the analyzer upon flame failure.

When sampling a hydrocarbon stream, it is recommended that an analyzer be chosen that can be vented to a safe location outside of the analyzer enclosure. Consideration should be given to such principles as (a) normal sample line velocities in excess of the flame velocity, (b) minimum practical sample volume at the ignition source, (c) installation of a flame arrester near the ignition point, or (d) positive sample dilution below the flammable range. Each such system requires special consideration and preventative measures.

When steam tracing or heating is used, insulation or shielding must be provided for the protection of personnel.

19.6.2 ELECTRICAL HAZARDS

Analyzers and recorders located in classified hazardous areas should have all electronic gear, open contacts, and any other arcing devices enclosed in explosionproof or properly purged cases unless the analyzer or recorder has been determined to be intrinsically safe.

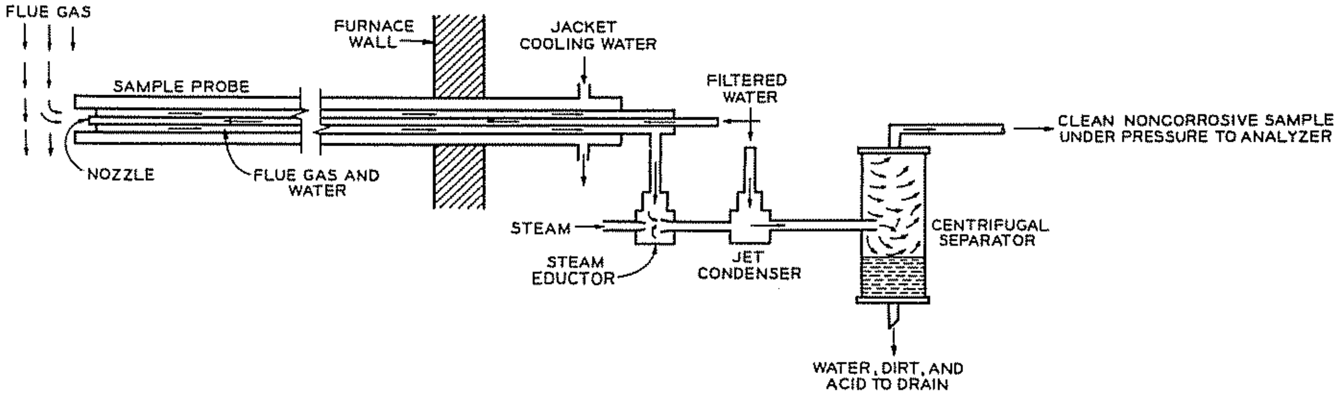


Figure 19-11—Negative Pressure Sample Preparation with External Eductor

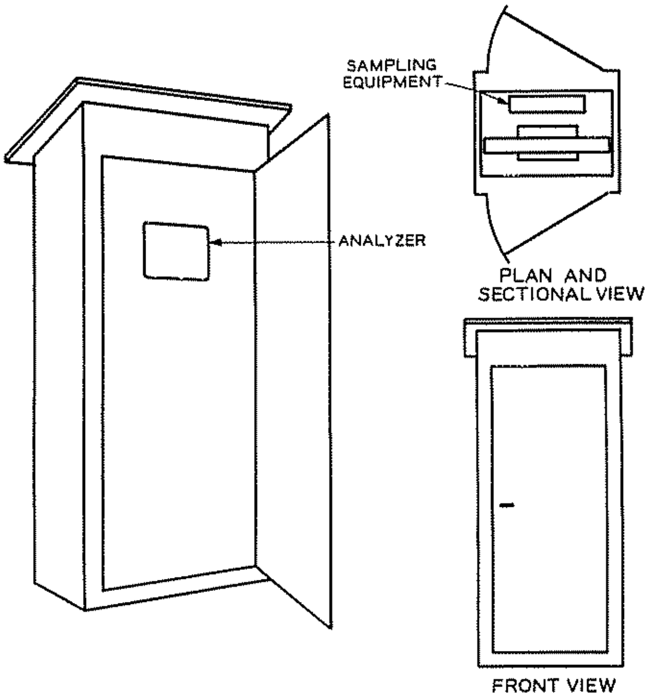


Figure 19-12—Typical Analyzer Housing

19.7 Readout

The signals from the oxygen analyzers described in this section can be adapted to read out on most of the standard pneumatic or electrical indicating or recording instruments for measurement or control.

19.8 Calibration

Calibration methods may vary with the type or range of analyzer and with local conditions.

19.8.1 ZERO AND SPAN CHECKS

Analyzers with a range which exceeds 21 percent of oxygen can use instrument-quality, dry, compressed air as a span gas. Some low-range analyzers have a special calibration range to permit this. Also, zero check may be made on some thermal, paramagnetic instruments by removing the magnet from across the measuring cell, thereby stopping the magnetic convection action.

19.8.2 ORSAT

Orsat analysis, a common laboratory technique, can be made of the gases being monitored by analyzers used in flue gas service. This method can be reliable provided the apparatus and chemicals are in good condition. It is recommended that the sample for the Orsat apparatus be taken from the sample line at the inlet to the analyzer. The instant the sample is taken, the chart should be marked to indicate a reference point for use in comparisons after the results of the Orsat test are known.

19.8.3 PORTABLE ANALYZERS

Because of the time involved in Orsat readings, as well as the potential for human errors, portable analyzers have become popular as working devices. Extreme care must be exercised to ensure that these battery-operated units have been calibrated against a laboratory standard immediately prior to their use in the field, and that they meet electrical safety requirements. Only under these circumstances should the portable analyzers be used to calibrate the analyzer. Corrections for changes of ambient temperature may be required.

19.8.4 STANDARD SAMPLE

A more accurate and dependable method of calibration is the use of a standard sample which can be manufactured or purchased for this purpose. A standard sample gas generally is made up of a given percentage of oxygen in a nitrogen background gas. In cases where the background gases of the process stream differ too greatly from nitrogen in their effect on the analyzer, it is recommended that the standard be made up from background gases which match the process stream. A suggested procedure is to obtain at least one certified sample bottle with an oxygen content of approximately 75 percent of the analyzer range and another bottle with oxygen content at 25 percent of the analyzer range. Some authorities prefer that the second sample be approximately the same as the normally expected sample conditions. When analyzing inert gas, a standard sample gas containing no oxygen is helpful to check analyzer zero.

With those analyzers equipped with automatic calibration, it is good practice to have the analyzer manufacturer submit complete specifications for the calibration gas to be used as well as the type of container and fittings required to adapt the container to the analyzer.

19.9 Installation

19.9.1 STEAM TRACING

In areas where freezing weather is encountered, sample lines should be steam traced if liquid water or condensate is formed in the sampling system. When the steam tracing is installed, care must be taken to avoid evaporating the water which previously had been condensed to condition the sample for analysis. A common method of steam tracing a sample line is to install a spacer to keep the steam tracing line from physical contact with the sample line and to use a thermostatic type of steam trap. In some cases, electrical heating may be used instead of steam tracing.

19.9.2 PROPER SLOPE OF SAMPLE LINE

Where steam or water aspirators are used, the sample line should slope toward the water separator system which is normally at the analyzer location. Condensate collecting in low spots in the sample line can cause the analyzer to be inoperable and can also contribute to corrosion problems. On some installations where steam aspirating is used at the sample point, a drip leg or pot can be installed at a low point to collect the condensate. Also, heat tracing may be used to keep the sample temperature above the dew point in order to eliminate condensation. Material selection for lines and equipment must be carefully considered when condensation occurs.

Where low spots in the sample line are unavoidable, liquid traps should be provided to prevent water damage to the analyzer. A filter and drip-pot assembly is sometimes installed at the sample source to remove water and dirt from the sample.

19.9.3 EROSION

Erosion normally is encountered only in the catalyst reject system which should be designed to minimize this effect. In sampling systems containing a high content of dust or catalyst, filters or cyclones should be used at the sample source and/or automatic backflush facilities added. To use these devices, the temperature of the gas must be above the dew point and the pressure must be greater than that required to overcome the pressure drop of the device used. Generally, a cyclone will require a minimum pressure drop of 3 pounds per square inch to operate satisfactorily. The pressure drop in filters varies depending upon material and element porosity selected.

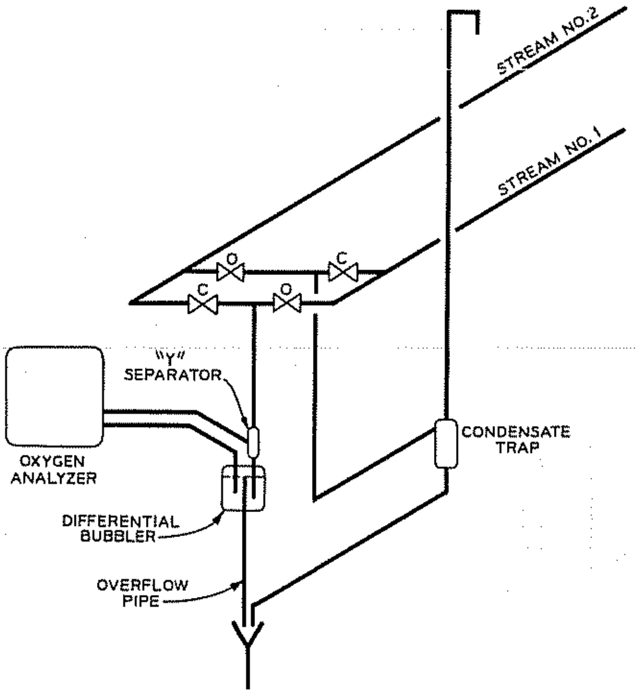
19.9.4 HOSE

When plastic or rubber hose is used for sample lines, it is important to provide adequate support on horizontal or slightly pitched lines to avoid pockets where condensate can settle out and restrict the flow. A satisfactory method of support is to run the hose through a pipe or in a trough. The line should be given as much pitch as possible. Running hose in close proximity to hot lines should be avoided.

19.9.5 LOCATION OF REGULATORS AND MANIFOLDS

On analyzers with pressurized samples, the sample regulator should be located as close to the sample point as practicable to decrease the transport time. A course filter or strainer should be installed ahead of the regulator.

Manifolds for multistream installations should be located at the analyzer. The sample-switching manifold should be designed to permit a continuous flow of sample regardless of whether it flows to the analyzer or past it. This design will ensure that the sample to be analyzed is fresh. A typical installation method is illustrated in Figure 19-13.



NOTE: Tubing down stream of the vent tees should be as short as possible.

Figure 19-13—Multistream Sampling System

19.9.6 POWER REGULATION

In locations where the power supply voltage and frequency are likely to vary, power regulation may be required to ensure stable operation of the analyzer.

19.9.7 VIBRATION EFFECTS

Most analyzers, especially those having suspended moving elements similar to a galvanometer, are adversely affected by vibration. Mounting of analyzers in areas around reciprocating machinery should be avoided (see Section 14).

19.10 Startup Procedures

After the analyzer installation is completed, a procedure should be developed and followed to check out the complete system and to ascertain that all necessary items are properly installed and operating in accordance with design.

19.10.1 SAMPLING SYSTEM

The sampling system should be placed in service and flushed to remove scale and dirt before it is connected to the analyzer. During this operation, filters and orifices should be removed from the line to prevent plugging.

19.10.2 SAMPLING SYSTEM LEAKS

After lines have been flushed and the filters, orifices, and so forth have been replaced in the sample line, the system should be pressure tested for leaks. This testing is especially important on negative pressure systems. A recommended testing procedure to ensure tightness of the system is outlined in Instrument Society of America RP7.1 *Pneumatic Control Circuit Pressure Test*. An Orsat apparatus can also be used to locate leaks in negative pressure sampling systems. This test method is time-consuming but effective if properly conducted.

19.10.3 ELECTRIC POWER AND WIRING

The power source to the instrument should be checked to make sure that the voltage is correct, that the installed fuses are of the correct rating, and that the switch overload heater is of the right value. If these requirements have been met, the power to the analyzer and recorder can be turned on.

19.10.4 START SAMPLE

The sampling system should be activated and the sample started through the instrument in accordance with the manufacturer's instruction manual.

19.10.5 CALIBRATION

Analyzer calibration should be checked. One of the calibration methods outlined in 19.8 or the method suggested by the manufacturer should be used.

19.10.6 TRANSMISSION AND CONTROL SYSTEMS

Where transmission or control is employed, procedures outlined in the manufacturer's manual for check out and calibration of systems should be followed. Pneumatic systems should be checked and all leaks repaired; a preferred method is outlined in ISA RP7.1.

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SECTION 20—pH ANALYZERS

20.1 Scope

Suggested practices for the installation of continuous process stream pH analyzers of the electrometric (glass electrode) type are presented herein. Colorimetric or other electrometric types of analyzers will not be discussed in this section nor will laboratory or portable analyzers.

The use of pH instrumentation is well established as a means for measuring and controlling hydrogen ion concentration (pH) in refinery streams. Some of the more common applications are for the following:

1. Refinery waste water
2. Cooling tower water
3. Boiler feedwater
4. Condensate in fractionator reflux systems
5. Corrosion control in processing facilities
6. Chemical neutralization

20.2 pH Measurement

The term *pH* is a convention used to indicate the acidity or alkalinity of a solution. It is obtained by determining the logarithm (base 10) of the reciprocal of hydrogen ion concentration expressed in grams per liter.

The pH index, from a practical standpoint, can be considered to range from 0 pH to 14 pH (see Figure 20-1). The smaller the pH number the greater the free acidity or hydrogen ion concentration. A solution having a pH number of 7 at 25 degrees Celsius is neutral. Solutions with a pH below 7 are acidic and those with a pH above 7 are basic or alkaline. As the pH value increases in a given solution, the hydroxyl ions increase; the hydrogen ions correspondingly decrease, inasmuch as the product of the respective ion concentrations is a constant value.

An important fact to consider is that, as the pH decreases from 14 to 1, the hydrogen ion concentration increases ten times for each unit drop in the pH value. This means that 7 pH denotes ten times the acidity of 8 pH; also, 6 pH denotes ten times the acidity of 7 pH. In practical terms, this means that to reduce the pH of a solution from 6 to 5 requires ten times the acid needed to reduce the pH of the same solution from 7 to 6.

With so large a change in acidity or alkalinity taking place with a change of only one pH unit, the need for a sensitive and precise pH measuring and control installation cannot be overemphasized. Also, it is important to keep in mind that the pH of a solution usually varies with the solution temperature. Industrial pH meters read the pH of the solution in contact with the electrodes at the temperature existing when the measurement is made. They do not compensate to a base solution temperature.

20.3 Electrode Measuring System

The electrode system is comprised of three elements: the glass electrode, the reference electrode, and the sensing element for the automatic temperature compensator. A schematic arrangement of these elements is shown in Figure 20-2.

20.3.1 GLASS ELECTRODE

The primary function of the glass electrode is to develop a potential proportional to the hydrogen ion concentration (pH value) of the process stream. For practical purposes, the pH value is determined by measuring the potential or voltage developed between the process stream and the standard solution contained within the glass electrode. This potential is developed because of the difference in hydrogen ion concentration of the two solutions.

If the hydrogen ion concentration in the process stream is higher than that inside the glass electrode, there will be a positive potential. If the process stream is lower in hydrogen ion concentration, there will be a negative potential. The potential generated is amplified and fed to the measuring instrument where it is translated into a direct reading of pH.

20.3.2 REFERENCE ELECTRODE

The reference electrode is designed to produce a constant potential regardless of the solution in which it is immersed. This constant potential is used as a base or reference point from which to measure the variable potential produced by the glass electrode. It is essential to maintain a conductive path between the electrode and the process stream. There are two designs of reference electrodes: diffusion and flowing.

20.3.2.1 Diffusion Electrode

In the diffusion-type electrode, the electrolyte diffuses through a large porous area to contact the process. These electrodes contain a salt slurry within a gel that resists contamination and drying. They are generally a

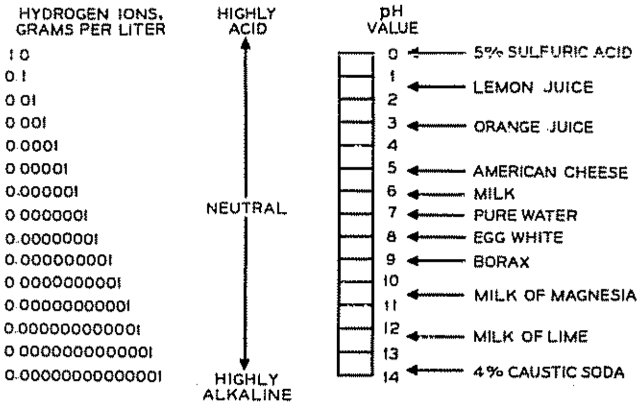


Figure 20-1—The pH Scale

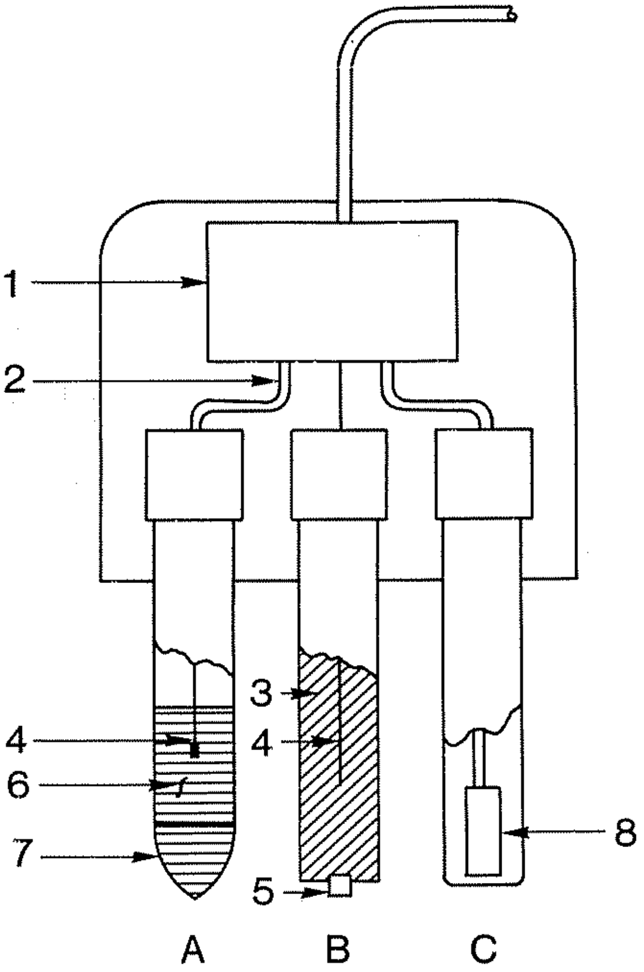
sealed system that permits diffusion without the need for external pressure. The diffusion-type reference electrodes are recommended for nearly all applications. In some high-conductivity processes (sugars, particularly), the porous area may become plugged resulting in a very high resistance and a poor measurement.

20.3.2.2 Flowing Electrodes

With the flowing-type electrode, a conductive path between the electrode and the process stream is maintained by having a small amount of the electrolyte flow out of the reference electrode. When it is necessary to measure the pH of a solution under pressure, the effect of the stream pressure is overcome by the use of a pressurized reference electrode which prevents backflow. Note that if the flowing-type electrode is used, it is necessary to maintain a positive pressure at all times to insure outflow of the electrolyte.

20.3.2.3 Automatic Temperature Compensator

The temperature compensator is usually necessary because the electromotive force developed by the glass electrode will be affected by the temperature of the electrode itself. The element consists of a resistance thermometer built into a probe which is immersed in the process stream. The thermometer automatically alters circuit constants so that a given electromotive force will indicate the correct pH at the process stream temperature. The pH meter is not compensated to read to a base temperature. The temperature compensator corrects for the temperature coefficients of the electrodes, but it does not compensate for changes in solution pH caused by temperature variations. Automatic temperature compensation is usually unique to a specific measuring instrument. Consult manufacturer's specifications for details.



Legend:
1—Transmitter
2—Shielded cable and cap
3—Potassium chloride salt or gel slurry
4—Silver-silver chloride element
5—Liquid junction (porous plug)
6—Internal buffer and electrolyte
7—pH sensitive glass membrane
8—Temperature sensitive resistor

NOTES:
Arrangement A shows a glass electrode.
Arrangement B shows a reference electrode.
Arrangement C shows a temperature compensator.

Figure 20-2—Schematic Diagram of Typical Electrode System

20.4 Types of Service

Application and process requirements usually dictate the type of equipment for each specific pH installation. The majority of pH measuring systems utilize sensing elements which are located in one of four ways:

1. In a piped sample stream at atmospheric pressure (see Figure 20-3)

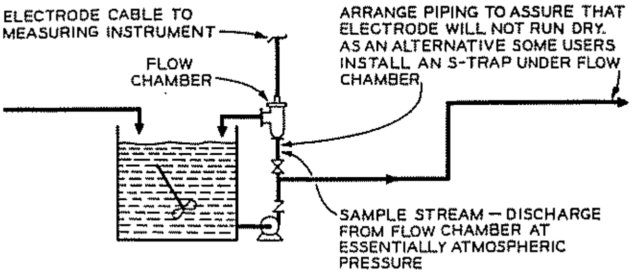


Figure 20-3—Piped Sample Stream at Atmospheric Pressure

2. In a piped main process stream or piped sample stream at positive pressure (see Figure 20-4)
3. In a tank, trough, or stream at constant level (see Figure 20-5)
4. In a tank, trough, or stream at variable level (see Figure 20-6)
5. Sampling system for heavily contaminated oily samples containing light oil and heavy sludge (see Figure 20-7).

Electrode flow chambers are used to admit pH sensitive electrode assemblies directly into piped process streams or into bypass sample streams. They may be used in streams under pressures of 150 pounds per square inch gage or less.

In some instances, individual glands may be used to advantage instead of a flow chamber. These permit mounting the electrodes directly into a large process line.

A more convenient method is to provide a flow chamber in a bypass. The bypass can easily be shut off and the electrode system serviced.

Immersion assemblies are used in a tank, trough, or open stream at constant level. The tips of the electrodes are immersed in the process fluid.

If submersion assemblies are used in a tank, trough, or open stream where the level is variable, the assembly is submerged to a point where the electrodes are still immersed when the level drops to its lowest point.

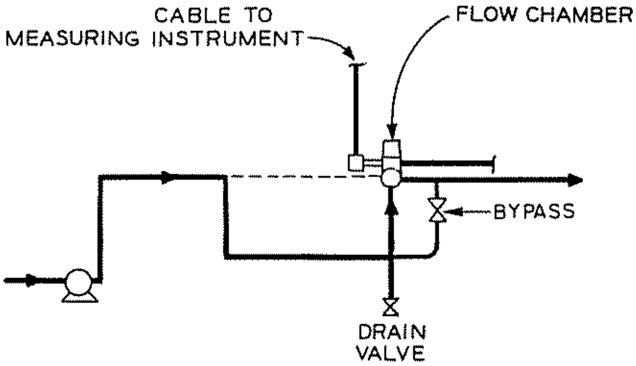


Figure 20-4—Piped Main Process (or Sample) Stream

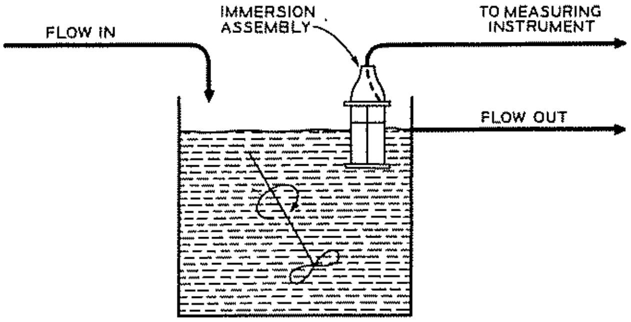


Figure 20-5—Tank at Constant Level

A flow-type system can be utilized as long as its discharge point is below the lowest level expected.

20.5 Installation

20.5.1 ELECTRODE LOCATION

The electrode assembly should be located so as to permit quick and easy standardization, cleaning, and replacement. However, it must be remembered that the most important decision concerning electrodes is to select the most desirable point of measurement in the stream. The assembly and associated piping should be arranged so that the reference electrode will remain immersed in the process solution at all times. In some instances, it may be necessary to "pocket" the piping near the flow assembly or to draw a slipstream through a pocketed flow assembly. Reference electrodes should not be allowed to become dry. Some companies have installed a source of city water adjacent to the flow

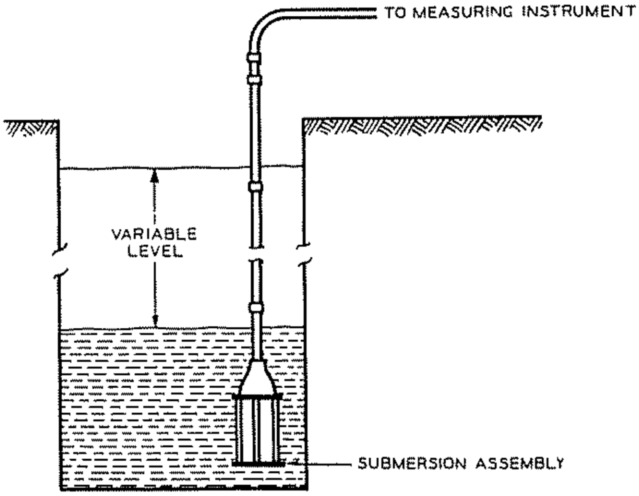


Figure 20-6—Tank at Variable Level, Submersion Assembly

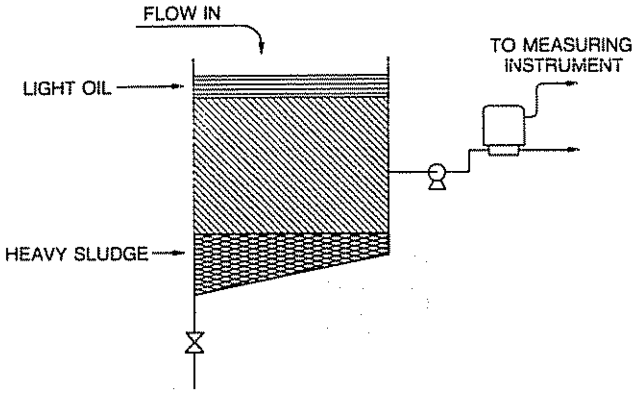


Figure 20-7—Sampling Technique for Heavily Contaminated Oily Systems

assembly so that when the sample is shut off, water may be substituted, thus keeping the electrodes wet.

If the reference electrode (flowing type) in a flow chamber is not pressurized, the sample stream must be at atmospheric pressure. For this reason, the discharge flow from the assembly must not be—

1. Located at a higher elevation than the flow assembly itself
2. Piped through pipe or tubing of such small diameter that a positive pressure will be created within the discharge from the flow chamber
3. Discharged into a pressurized system.

In neutralization systems, the electrodes must be located far enough downstream of the point of injection of the neutralizing material to ensure proper mixing and reaction. In recording applications, where lag is a minor factor, locating the electrode assembly far enough downstream for complete mixing is permissible. In control applications, where lag must be minimized, better mixing by use of an agitator will reduce the process lag and make control easier. Control of the pH of a process stream flowing in small pipes can only be maintained if the pH change of the material to be treated is small and changes slowly. If there are large or rapid fluctuations, a retention tank is necessary.

Because the electrode measures the pH of only the thin layer of solution in contact with the electrode, it is essential that this layer be representative of the bulk of the solution being measured. It is important to obtain the correct flow rate around the electrode to minimize stagnation and to ensure a representative measurement.

When oil-contaminated water must be measured, the assembly may be submerged (as in Figure 20-6) or a tank may be used (as in Figure 20-7) to allow the oil to float up and out of the sample stream or to allow the heavy sludge to settle to the bottom. A flow chamber with high sample velocity is desirable. The washing action of the process will remove oil or sludge clinging to the electrodes. In this case, the mixer should be omitted. It is recognized that such an installation may introduce an undesirable time lag; however, oil cannot be tolerated on the electrode surface.

20.5.2 pH ELECTRODE CLEANING

pH electrodes are vulnerable to coating and subsequent signal degradation in installations where precipitates, oils, and fine suspended matter adhere to electrode surfaces. Large junction reference electrodes minimize coating problems by offering a relatively large surface area to complete the electrode measuring circuit.

Manual cleaning usually consists of a routine procedure of dipping or scrubbing the electrodes in acid, detergent, or solvent, depending on the nature of the coating. In flow-type electrode mountings, this can be partially automated by periodic flushing with a cleaning agent. Cleaning can be facilitated by installing a three-way valve which selects either the process sample or the cleaning agent.

Some self-cleaning is achieved by electrode mountings that develop a high flow velocity past the electrode surfaces. This technique is especially effective for water/oil mixtures.

Ultrasonic cleaning is of greatest benefit in preventing solid particles from adhering to electrodes. It can eliminate or greatly reduce the need for manual cleaning. Some care is required in adjusting the power output of an ultrasonic cleaner since too much power can crack measuring electrode glass membranes.

Where frequent cleaning is necessary, some users install twin flow chambers in control applications thus switching the stream from one chamber to the other on a regular schedule. The idle assembly should be cleaned promptly and must be kept wet until returned to service.

20.5.3 pH METER

It is desirable to locate the pH meter (or amplifier converter) as close as practicable to the electrode assembly to facilitate calibration and standardization and to reduce electrode lead lengths. However, most manufacturers have transmitters located in or near the electrode mounting head. These transmitters convert the high-impedance signal to a current signal that can be transmitted over long distances. Refer to manufacturer's specifications for details. When it is necessary to locate the readout device remotely, a separate indicating meter installed near the electrode assembly may be desirable.

20.5.4 CABLE AND CONDUIT

If a local transmitter is not used, the electrode cables between the assembly and the meter should be installed in rigid conduit which contains no other leads. This will help to avoid any flexing of the coaxial cable and to keep out stray electromotive force as well. The leads should be pulled in one continuous length. Where this is impossible and splices are necessary, they should be made by well-qualified personnel. Only connections and junction boxes specifically designed for coaxial cable should be used. This is necessary to provide and maintain, by minimizing the entrance of moisture into the coaxial cable, the high impedance required for glass electrode assemblies. Care must be taken in the installation of coaxial cable not to contaminate the surface of any of the insulating materials. Even the salts present on the finger tips, if transferred to the insulator surface, can cause the formation of a bridging condensate film.

The special cable precautions apply specifically to the shielded cable for the glass electrode. Normal wiring methods and precautions apply to the reference electrode lead and the temperature compensator. Every precaution should be followed to keep junction boxes and conduits dry. Special attention should be given to sealing the raceway at the recorder, electrode housing, and at each junction box used.

For additional information on cable and conduit installations, see API RP 550, *Manual on Installation of Refinery Instruments and Control Systems*, Part I—Process Instrumentation and Control, Section 7—Transmission Systems.

20.5.5 GROUNDING

Proper grounding of the components of the pH system is essential. One of the major sources of startup difficulties is improper grounding. The manufacturer's instructions should be carefully followed.

If the system uses all plastic pipe and the flow chamber is plastic, it is very important that process origin and discharge points are at the same potential. Otherwise, an extraneous voltage will be impressed between the electrodes and indicated on the readout. Locating a section of metal pipe before and after the assembly and grounding both ends will reduce this effect.

For additional information on grounding, see RP 550, Part I, Section 7.

20.5.6 WEATHER PROTECTION

All flow assemblies must be designed to withstand and seal out moisture at the electrode caps and cable connections. It is good practice to locate the electrode assembly in a clean, dry housing to protect it from atmospheric conditions. In humid locations, the assembly should be housed and heated to avoid moisture condensation on the electrodes and connecting leads. Routine maintenance work should be scheduled only during clear, dry weather if cable junction boxes or the electrode housing are to be opened. In some areas, protection against freezing may be necessary.

Conduit connections should be made watertight. All leads should be sealed. Special care must be taken to prevent capillary action in the coaxial cable. Desiccant should be installed in accordance with the manufacturer's recommendations.

20.6 Safety Considerations

A discussion of safety factors, which may require consideration, is presented in Section 14.

20.7 Readout

The pH components described in this section can be combined with potentiometric or current-type recorders and indicators for measurement or control.

20.8 Calibration

20.8.1 BUFFERING SOLUTION

A buffered solution is a standard solution of a known hydrogen ion concentration. This standard solution has been buffered with salts so that it resists changes in its pH from dilution or from the addition of either acids or alkalis.

There are three National Bureau of Standards buffering solutions: 4.01, 6.86, and 9.18 pH. Many more are available from manufacturers.

20.8.2 CALIBRATION PROCEDURES

The instrument should be calibrated by immersing the electrodes in at least two different buffering solutions bracketing the range of intended use.

The buffers should be a minimum of 3 pH units apart and at, or near, operating temperature. If the instrument and electrodes do not check the buffers within 0.1 pH units after instrument adjustment, trouble exists in electrodes, wiring, or recorder. Temporarily electrodes may be connected directly at the recorder to isolate wiring troubles. Electrodes may be separately tested and buffered on a laboratory instrument. A color comparator is inadequate for calibration purposes and should not be used. When checking against a laboratory pH meter, it is sometimes advisable to draw the samples in preheated thermos jugs to permit laboratory pH checks at operating temperatures and thereby minimize temperature errors.

20.9 Startup

It is generally recommended that the manufacturer's operating manual be used as a reference for starting a continuous process stream pH analyzer.

In general, the following steps apply:

1. Turn instrument power on.
2. Standardize potentiometer circuit.
3. Check to make sure electrode assembly is properly installed and the electrodes are clean. If the electrodes are to be used immediately, soak the lower half of the glass measuring electrode in approximately 0.1 normal hydrochloric acid at 50 degrees Celsius for half an hour, then wash thoroughly with distilled water. If time is available, this soaking period should be extended overnight at room temperature and followed by washing the electrode thoroughly with distilled water. This procedure should be used with all new glass electrodes and with electrodes that have become dry between periods of use.

It is also very important to use glass electrodes within the strict design limits set by the manufacturer. Glass measuring electrodes designed specifically for high temperature generally are unusable at temperatures lower than those indicated by the manufacturer. Their source resistance may be so high that it causes low recorder sensitivity and completely erratic readout.

4. Make a final check of electrical wiring and connections, and make sure all gaskets and seals have been carefully made up in strict accordance with the manufacturer's instructions.
5. Check that electrolyte reference electrode is not dried.
6. Start sample flow; use manufacturer's recommended rate.
7. Calibrate electrically and chemically.

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SECTION 21—DENSITOMETERS

21.1 Scope

This section covers the installation of the more commonly used instruments for the continuous measurement of fluid density or specific gravity. Excluded from the discussion are those instruments or devices primarily used in laboratory analysis of given samples.

Various methods are used for the determination of fluid density or specific gravity. It is the intent of this section to cover only those methods that are more commonly employed with remote or close coupled, continuous recording, indicating, and/or controlling devices; thus lending themselves to automatic process control. Densitometers are available for measurement of both liquids and gases. The two types will be covered separately in this discussion.

21.2 Density or Specific Gravity Measurement

The density of a material may be defined as the weight per unit volume at given conditions of temperature and pressure. Generally, it is expressed in pounds per cubic foot, pounds per gallon, or grams per cubic centimeter.

21.2.1 LIQUID

The specific gravity of a liquid is the term indicating its density with reference to water. The specific gravity of water is usually defined as 1.0 at 60 degrees Fahrenheit.

21.2.2 GAS

The specific gravity of a gas is the weight of a cubic foot of gas compared to the weight of a cubic foot of dry air under the same pressure and temperature conditions. Air is defined as base 1.0 at 60 degrees Fahrenheit and 760 millimeters of mercury absolute.

21.3 Liquid Densitometers

21.3.1 BALANCED FLOW VESSEL

In this method of determining liquid density, a fixed-volume vessel through which the sample flows is balanced by a scale beam (see Figures 21-1 and 21-2). The displacement of the scale beam caused by a change in weight of the fixed volume of sample is proportional to density.

21.3.2 BALANCED FLOW TUBE

Another version of the fixed-volume vessel instrument (see Figures 21-3 and 21-4) employs either a straight or U-shaped hollow beam. In operation, fluid is circulated through the hollow beam which is attached to a pneumatic null-balance system. Changes in weight of the fluid are frequently transmitted in the form of a pneumatic signal which can be used to record, indicate, control, or a combination of these functions. No interpolation or indirect measuring is involved. Density is read directly, and errors which result from velocity, viscosity, pressure, and product composition are precluded. The relatively large flow rate through the hollow tube permits such materials as slurries, drilling mud, and cement-water mixtures to be measured.

21.3.3 INDUSTRIAL SPECIFIC GRAVITY DISPLACER

With the industrial specific gravity displacer (see Figures 21-5 and 21-6), the sample is passed through a chamber containing a completely submerged, externally counterbalanced float. The change in buoyancy of the float with change in density of the liquid within the chamber is proportional to density. The signal may be transmitted by pneumatic, mechanical, or electrical means. This instrument is similar to the widely used displacement types of level controls except that sample connections to the chamber are made in such a manner as to minimize velocity effects.

21.3.4 CHAIN-BALANCED-FLOAT DENSITY INSTRUMENT

With the chain-balanced-float density instrument (see Figures 21-7 and 21-8), a float in a continuous-flow chamber is weighted to carry half the weight of a light chain when submerged in medium-density liquid at midpoint in its range. The chain is attached to the bottom of the float and to the vessel wall at half height. Any increase in density causes the float to rise, supporting more chain; any decrease in density causes the float to sink, supporting less chain. The float position is transmitted by a differential transformer.

21.3.5 GAMMA-RAY DENSITY GAGE

In the gamma-ray density gage (see Figure 21-9), a gamma-ray source, such as a radium salt or radioisotope, is arranged so that the gamma rays pass through the pipe or vessel walls and through the fluid to a radia-

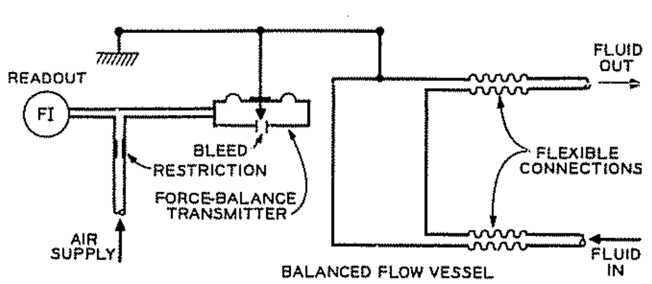
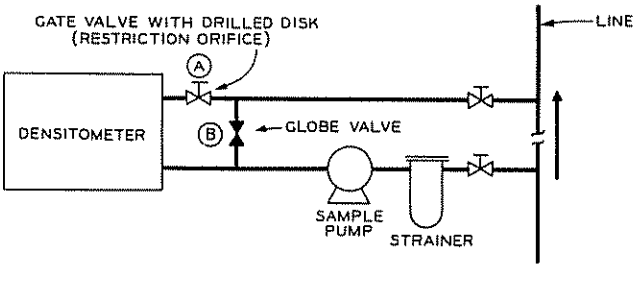


Figure 21-1—Balanced Flow Vessel

tion detector. The detector is mounted on the outside of the pipe or vessel. The source may be located on the opposite side of the line or vessel, inside the line or vessel, or arranged to traverse a diagonal or longitudinal portion of the line or vessel. The selection of the proper arrangement depends upon the ratio of gamma-ray absorption by the fluid and metal through which the rays pass. The manufacturer should be consulted as to the most desirable type to use for adequate results.

In a commonly used arrangement, the radioisotope source is located on one side of the line and the radiation detector is located on the other side of the line. This type, illustrated in Figure 21-9, is supplied as an assembly and bolts in place around the section of line. Any material passing through the pipe or contained in the vessel will absorb part of the gamma rays from the source. The amount of energy reaching the detector varies inversely with the density of the process stream. The gamma-ray density gage may be used to take continuous density measurements of solutions, liquids, slurries, or divided solids. It requires no sampling system as the radiation source and the detector may be mounted directly on the outside of the process line. It can be used to indicate, record, or control, or for a combination of these functions.



NOTE: The pump is used only when adequate pressure drop cannot be obtained between the process connections.

Pump supplies slipstream of several times the required volume. Under normal operation, Valve A is closed and Valve B is adjusted to avoid introducing error as a result of high flow velocity. To flush, open Valve A and close Valve B.

Figure 21-2—Typical Liquid Densitometer Sampling System

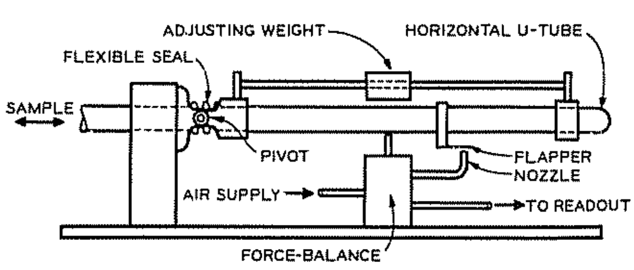
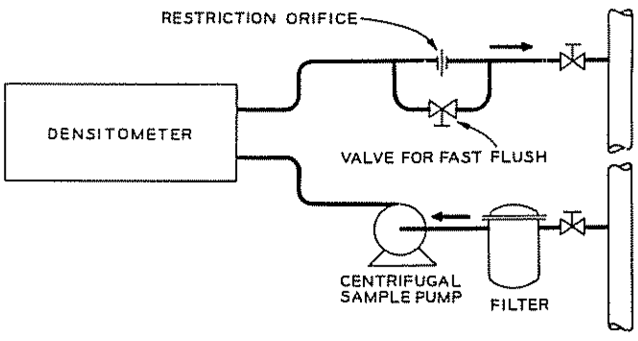


Figure 21-3—Balanced Flow Tube

21.3.6 VIBRATING-PROBE LIQUID DENSITOMETER

In one form of vibrating-probe densitometer (see Figure 21-10), a detector consists of a driver end connected through a vibrational path to a paddle and returned to a pickup end, thus completing a loop through the liquid to be tested. The driver coil receives 60-cycle ac input and produces 120-cycle vibrations in the loop. The pickup end contains a permanent magnet and coil which generate an output signal proportional to the amplitude of the paddle vibration. The electromotive force of this output signal is nominally 500 millivolts. Inasmuch as changing liquid density will increase or decrease the amplitude of the paddle vibration, the output signal will also increase or decrease, thus becoming a measure of the liquid density. A temperature element may be connected into the electrical output circuit for temperature correction.

The detector may be installed directly in the flow line or vessel and is adaptable to a wide range of liquids or slurries (see Figure 21-11). If normal process pressure, temperature, or velocity do not exceed manufacturer's recommendations, no special sample handling system is required.



NOTE: The pump is used only when adequate pressure drop cannot be obtained between the process connections.

Figure 21-4—Typical Sampling System for Balanced Flow Tube

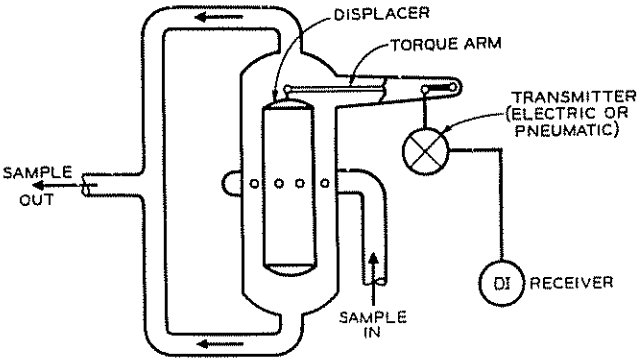


Figure 21-5—Industrial Specific Gravity Displacer

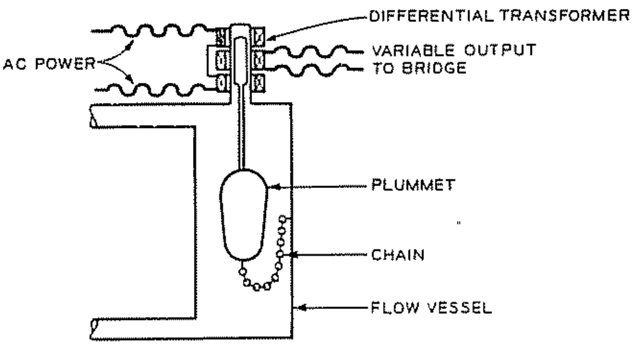


Figure 21-7—Chain-Balanced-Float Densitometer

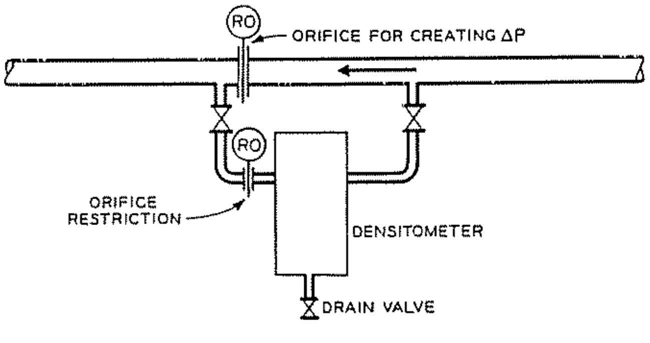
21.3.7 VIBRATING-TUBE LIQUID DENSITOMETER

This twin-tube densitometer is designed to provide continuous on-line measurement of liquid density. The operation of the system can be compared to that of a tuning fork. Two parallel tubes carrying the process liquid are maintained in mechanical vibration by an electromagnetic coil placed centrally between them. This causes the tubes to vibrate at their natural frequency which is a function of the density of the liquid they contain. Then the output frequency is detected and converted to a direct density reading by a frequency-to-current converter. If desired, a temperature sensor is used to enable the output reading to represent the density at a specific temperature.

This instrument is immune to vibration and can be mounted at any angle. Vertical mounting is preferred as this prevents solids from precipitating onto the tube walls and air bubbles from being trapped.

21.3.8 VIBRATING-SPOOL DENSITY METER

Another type of density meter incorporates a vibrating, thin-wall cylinder or spool (see Figure 21-12). The spool is maintained in circumferential oscillation by an electromagnetic field. The frequency of oscillation depends only upon the density of the fluid surrounding the spool and is independent of temperature, viscosity, and static pressure effects. Measurement of this frequency allows determination of the fluid density. This type of density meter is suitable for liquids, gases, or cryogenic fluids.



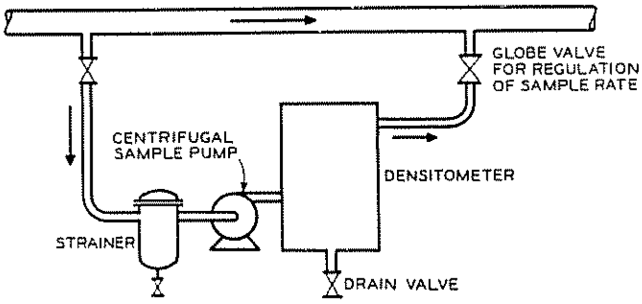
NOTE: The sample pump system of Figure 21-8 is also applicable

Figure 21-6—Typical Hookup for Industrial Specific Gravity Displacer

21.4 Gas Densitometers

21.4.1 GAS SPECIFIC GRAVITY BALANCE

The gas balance densitometer (see Figures 21-13 and 21-14) is comprised of a test chamber with a tall vent stack or column inside of which is a floating, liquid-sealed bell. The floating bell is scale- or force-balanced. Gas, which is admitted to the test chamber at atmospheric pressure, fills the chamber and exits at the top of the vent stack. The column of gas acting on the outside of the floating bell is thus weighed against the air acting against the inner side of the floating bell. The resultant vertical movement of the floating bell is, therefore, proportional to the specific gravity of the gas compared to the specific gravity of air.



NOTE: The hookup shown in Figure 21-6 may also be used.

Figure 21-8—Typical Hookup for Chain-Balanced-Float Density Instrument

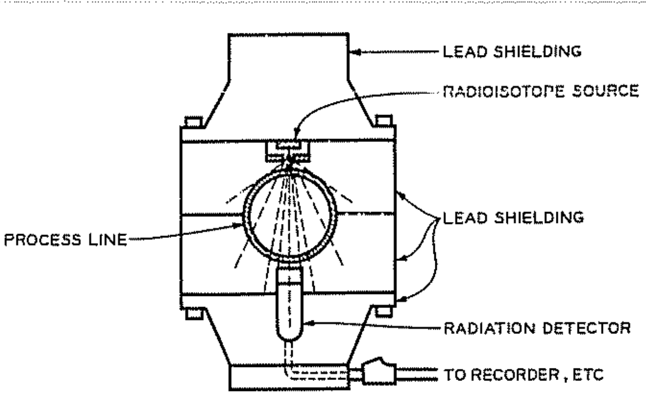


Figure 21-9—Gamma-Ray Density Gage

21.4.2 GAS DENSITY BALANCE

The gas density balance instrument (see Figures 21-15 and 21-6) is of the electrical null-balance type. The gas density is measured by the buoyancy of one ball of a dumbbell as compared with the other. The reference ball is punctured; therefore, it is not subject to the buoyancy effects. Rotation of the dumbbell about a horizontal suspension produces an electrostatic force between electrodes and the suspension. A light source is arranged to project a beam of light onto a small mirror mounted on the dumbbell. This light beam is reflected by the mirror to a prism which divides it into two beams which fall on two identical phototubes in a bridge circuit. Balancing potential is obtained and measured by the amount of light received by each of the phototubes. The rebalance potential nulls the balance and is recorded as specific gravity relative to air. The instrument is compensated for barometric pressure changes. Calibration is made with a known reference gas. Narrow spans may be obtained.

21.4.3 FLUID-DRIVE GAS GRAVITOMETER

With the fluid-drive gas density meter (see Figures 21-17 and 21-18), two opposing fluid drives develop a torque which is proportional to fluid density. The difference in the opposing torques aligns a pointer which indicates specific gravity.

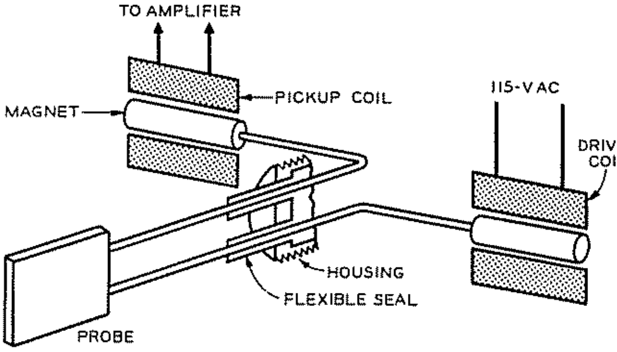


Figure 21-10—Vibrating-Probe Densitometer

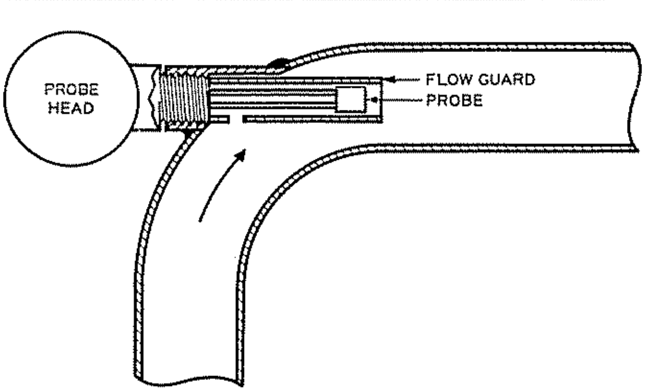


Figure 21-11—Typical Line-Mounted Vibrating Probe

21.4.4 BLOWER-TYPE GAS DENSITOMETER

In the blower-type gas densitometer (see Figures 21-19 and 21-20) a constant-speed rotating element in a bypass line produces a differential pressure which is proportional to the gas density. A differential pressure transducer connected between the inlet and the outlet of the rotating element measures the differential produced and sends signals to indicate, record, or control. This instrument does not require correction for gas compressibility and may be used in conjunction with a gas volume flowmeter as a mass flow device.

21.4.5 VIBRATING-PROBE GAS DENSITOMETER

The vibrating-probe liquid densitomer, described in 21.3.6 and shown in Figure 21-10, may also be adapted to the measurement of gas density. Two models are

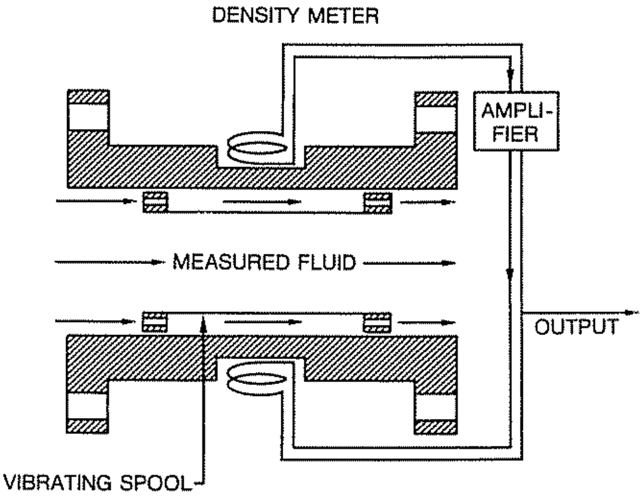


Figure 21-12—Vibrating Spool Principle

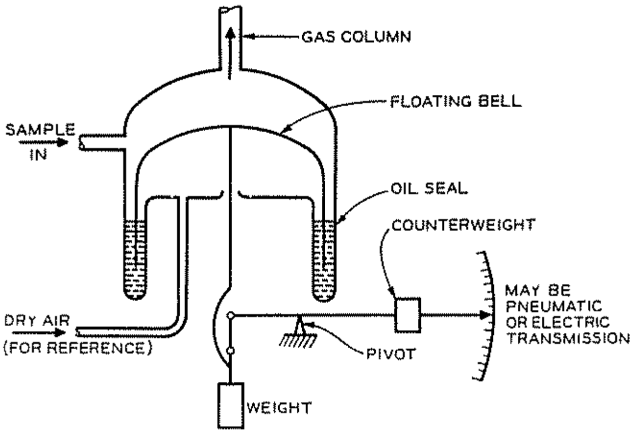


Figure 21-13—Gas Specific Gravity Balance

available. The first employs a single probe. The second uses two probes, one of which is immersed in a reference gas at the same temperature and pressure as the sample. The latter type is usually more stable and more easily calibrated.

21.4.6 THERMAL CONDUCTIVITY GAS DENSITOMETER

The thermal conductivity gas densitometer (see Figure 21-21) consists of a pneumatic Wheatstone bridge mounted in the vertical plane. The difference in molecular weight between the reference gas and sample gas will cause an unbalanced flow past the two thermistors. The temperature difference between the two thermistors is a measure of the sample gas molecular weight which is correlated to density. Alternating the sample with air will give results which can be correlated to specific gravity.

The reference gas is selected for having sufficient difference in density from the sample and is preferably of high molecular weight, high heat capacity, and low viscosity. Nitrogen, argon, and carbon dioxide tend to satisfy these requirements. The sample flow is maintained at about 10 cubic centimeters per minute while the reference flow is set at about ten times that to avoid back-diffusion.

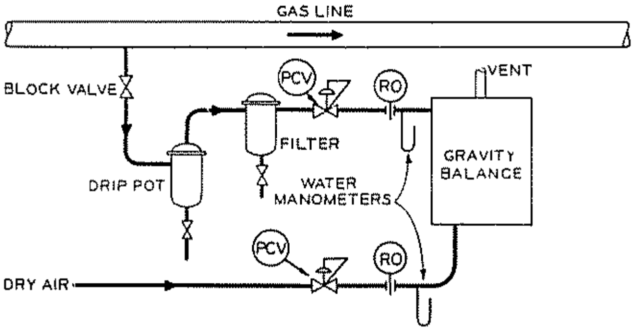


Figure 21-14—Typical Hookup for Gas Specific Gravity Balance

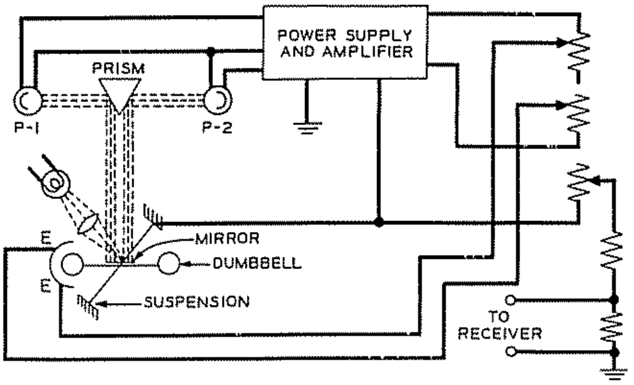


Figure 21-15—Gas Density Balance

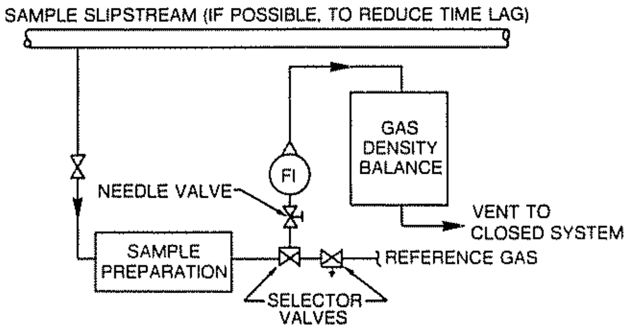
The main advantage of this design is that the sample gas never comes in contact with the detectors and, therefore, they cannot become coated, coked, or contaminated by the sample. Another desirable feature is that the cell contains no moving parts.

Variations in ambient temperature can introduce errors and, therefore, the cell temperature is normally controlled within ±1 degree Fahrenheit.

21.5 Compensation

21.5.1 PRESSURE COMPENSATION

Where dissolved gases have no effect, liquid densitometers usually require no pressure compensation. Where dissolved gases are present in such quantity as to affect accuracy, a careful study should be made prior to installation to determine the type and extent of corrective steps required to minimize their effect.



NOTE: For a discussion of sample preparation see Section 14.

Figure 21-16—Typical Gas Densitometer Sampling System

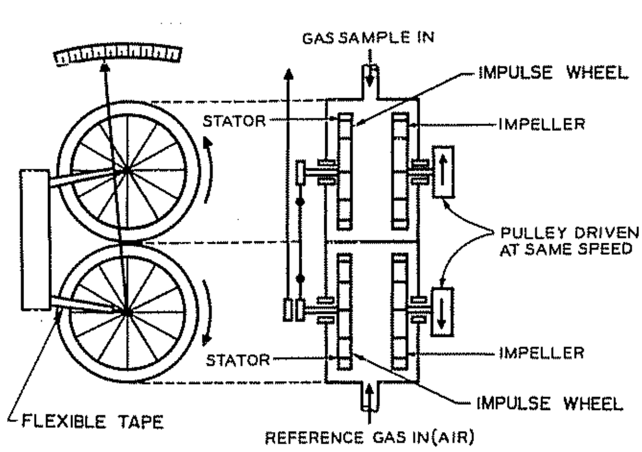


Figure 21-17—Fluid Drive Gas Gravitometer

21.5.2 TEMPERATURE COMPENSATION

On liquids where variation in the temperature of the liquid at the measuring instrument does not exceed 10 degrees Fahrenheit, no temperature compensation is normally employed. For high accuracy, temperature compensation must be provided when temperature variations exceed this value.

The change in density of gases with change in temperature is significant and must be corrected by employing a suitable means of temperature compensation.

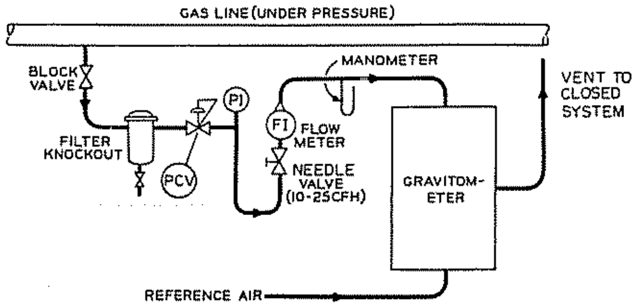
On most densitometers, temperature compensation can be obtained by providing a temperature-measuring device which electrically, mechanically, or pneumatically acts upon the primary signal from the density-measuring element to correct for the effect of temperature variation.

21.5.3 EFFECTS OF DEPOSITS, MOISTURE, AND FOREIGN MATERIALS

The accuracy of all liquid densitometers may be adversely affected by the deposit of foreign materials in the measuring chamber or on the displacer or float. If deposits or buildup are likely to occur, a system of flushing or cleaning will aid in obtaining the desired accuracy. With the gamma-ray instruments, a decrease in thickness of the pipe or vessel wall will adversely affect the instrument.

The gas specific gravity balance of Figure 21-13 will give inaccurate results if dust, dirt, or other foreign materials are allowed to accumulate on the floating bell.

Gas densitometers of the types shown in Figures 21-13, 21-15, and 21-17 are affected by the moisture content of the air used as a reference. For this reason the reference air must be thoroughly dried before introduction to the instrument or both the gas and reference air streams must be saturated.



NOTE: Install filter, reducing valve, rate valve, meter, and so forth in gas sample line.

Figure 21-18—Typical Sampling System for Gas Under Pressure

The viscous-drag gas gravitometer (see Figure 21-17) can be supplied with a built-in, two-compartment humidifier through which the gas and air sample pass before entering the impeller chambers. If it is desirable to obtain the density of the gas on a dry basis, the reference air must pass through suitable drying equipment prior to its entry into the instrument.

21.6 Safety Considerations

21.6.1 SAMPLE MATERIAL HAZARDS

With the exception of the balanced flow vessel described in 21.3.1, the liquid densitometers covered herein are usually mounted in the line or vessel or very close to the line or vessel. With this type of installation, the connecting lines are relatively large and mechanically strong. The same precautions should be followed as when installing other equipment handling the fluid involved.

The liquid densitometer described in 21.3.1 is used most often for pipeline sampling of crude oil and refined oil products. Temperatures are near ambient and no special precautions other than those normally ob-

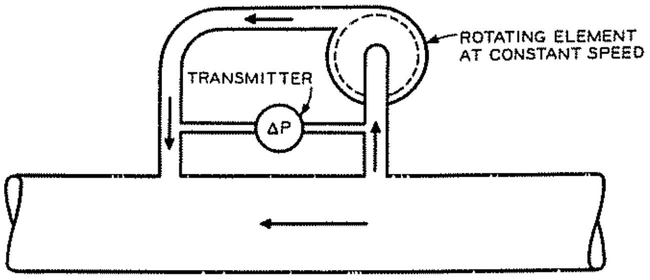


Figure 21-19—Principle of the Rotating-Element Type of Gas Densitometer

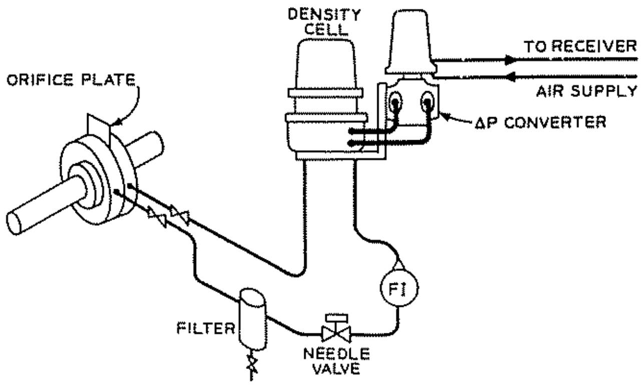


Figure 21-20—Typical Hookup for One Form of Rotating-Element Densitometer

served when handling hydrocarbons at line temperatures and pressures are involved

Gas densitometers like other gas sampling instruments may handle gases that are flammable, toxic, or irritating to the eyes or mucous membranes, or both. Precautions should be taken to vent such gases to a safe location—either sufficiently high, so that they will readily disperse in the atmosphere, or to a convenient, low-pressure line, vessel, or stack.

21.6.2 ELECTRICAL HAZARD

Liquid or gas densitometers may be used with volatile and other flammable materials. Generally, some electrical equipment is associated with their operation, either in the measuring circuit, temperature compensating circuit, or transmission system The electrical system should be studied to determine what degree of hazard it might present The scope of electrical precautions necessary is outlined in Section 14—Analyzers.

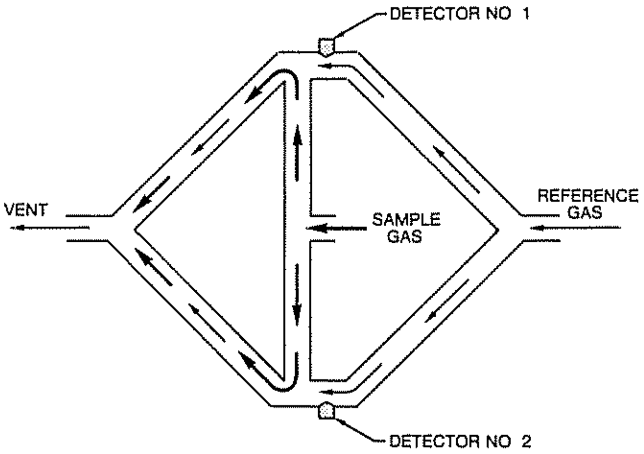


Figure 21-21—Thermal Conductivity Gas Densitometer

21.6.3 RADIATION HAZARD

Densitometers using radium salts or radioisotopes present an additional hazard because of the possible exposure of personnel to radiation Before installing or using a device of this type, all personnel should be thoroughly familiar with state and federal regulations which may be applicable. The manufacturer will usually assist in obtaining license application forms and copies of other pertinent information. It is desirable that personnel servicing equipment of this type be provided with film badges or dosemeters. These devices are essential for monitoring and maintaining within safe limits the radiation dosage received by service personnel.

21.7 Installation

21.7.1 LOCATION

It is desirable to locate the densitometer so that sample lines, if used, are as short as possible. The densitometer must also be located so that it can be serviced readily. This usually means that a compromise must be made. When a number of instruments are to be installed on the same operating unit, it may be desirable to locate them adjacent to each other in a common walk-in type of enclosure.

For additional information on analyzer location, see Section 14.

21.7.2 HOUSING

In most cases, it is desirable or even mandatory to house the densitometer to protect it from the weather and to maintain the ambient temperature within reasonable limits It is recommended that the instruments shown in Figures 21-1, 21-3, 21-12, 21-14, and 21-16 be enclosed in weatherproof housings. The instruments shown in Figures 21-5, 21-7, 21-9, and 21-10 generally are mounted in the line or vessel and require no housing. The readout equipment should be located in the control room or at some protected location.

21.7.3 EFFECTS OF VIBRATION

The densitometers shown in Figures 21-1, 21-3, 21-10, 21-13, and 21-15 can all be adversely affected by vibration. Precautions should be taken to locate the instrument and arrange piping so that vibration will not be transmitted to the device.

21.7.4 SAMPLING SYSTEMS

General design of sampling systems applicable to all analyzers is contained in Section 14.

Densitometers differ from other types of process stream analyzers. Most types of densitometers use much larger samples and some may be mounted in the line or vessel. Also densitometers may handle material such as slurries. In order to give a better understanding of the specific sampling system required this discussion will be referenced to the appropriate figure numbers.

1. *Figure 21-1*. The balanced flow vessel design generally is used for crude oil and refined products. The sample connections to the flow vessel are relatively small. The sampling system auxiliaries should be designed to deliver a reasonably clean stream to the instrument. The sample should be free of solid or foreign material which might plug or coat the connections or settle out in the balanced vessel, thus contributing to false information. The sample is usually supplied by a small-volume displacement pump. A restriction orifice in the main line can sometimes be used to promote sample flow.

2. *Figure 21-3*. The balanced flow tube may be obtained with a tube diameter of ¾ inch or larger. It can be used on any liquid and is suitable for measuring slurries. No special sampling system is required. If the process line is small, the instrument may be installed directly in the line; suitable block valves and a bypass must be provided so that the instrument may be taken out of service for maintenance.

3. *Figure 21-5*. The industrial specific gravity displacer has large sample connections and no special sampling system is required. A valve or restriction orifice should be provided on the outlet for regulating the flow rate so that the velocity of the flowing stream will not affect the displacer. Location of the valve or restriction on the inlet is not advisable due to possible partial vaporization across the restriction when used on some types of liquids.

4. *Figure 21-7*. The chain-balance-float densitometer requires the same precautions as the balanced flow tube (see Item 2 and Figure 21-3).

5. *Figure 21-9*. Inasmuch as the gamma-ray density gage is entirely external to the line and has no contact with the fluid, no sampling system is required. It is suitable for interface detection or for use on any material which can be moved through a line; for example, liquids, divided solids, or slurries. Coaxial cable is used from the radiation detector to the remotely located amplifier. Refer to RP 550, Part II, Section 20—pH Analyzers for precautions to be followed when using coaxial cable.

6. *Figure 21-10*. The vibrating-probe densitometer may be mounted on the line or vessel or in a slipstream. It employs no sampling system and may be used on numerous liquids and some slurries. As this instrument is somewhat sensitive to flow effects, it should be provided with a sleeve-type guard around the probe.

7. *Figure 21-13*. The gas specific gravity balance is suitable for clean, dry gases such as natural gas. It is not suitable for gases that contain dust, dirt, or other impurities which would settle out on the floating bottom or on the surface of the sealing liquid. The sample must be supplied at near atmospheric pressure. Therefore, a suitable pressure reducer and filter are required in the sample supply line.

8. *Figure 21-15*. The gas density balance is probably one of the most accurate instruments available and is capable of measuring very low gas densities. However, it is one of the most delicate instruments and requires the most sophisticated sampling system. The sample must be extremely clean and dry and must be only slightly above atmospheric pressure. Refer to Section 14, for details of sample preparation and handling systems.

9. *Figure 21-17*. The fluid drive gravitometer is a rugged instrument requiring minimum maintenance. This type of densitometer has been successfully used on gases, such as flue gas, sulfur dioxide, nitrogen, hydogen sulfide, and hydrogen recycle gas. Ambient air is generally used as a reference gas. To prevent errors in measurement and to avoid damage to the instrument, the gas sample may require a simple conditioning system. Gas should enter the instrument at essentially atmospheric pressure (within ½ inch of water column) or at a flow rate of 10 to 25 cubic feet per hour. If the sample is at high pressure, a primary reducing valve should be used followed by a differential pressure regulator with a rotameter for final pressure reduction. With noncorrosive gases, a simple filter should be installed in the sample line along with a knockout pot to remove free water. For use on noncorrosive gases, the instrument has a built-in, double-compartment humidifier through which both the sample gas and reference air pass before entering the measuring chambers. The temperature of the sample gas should be ambient. Usually if the sample piping is 15 feet or more in length, the sample will reach ambient temperature before reaching the instrument and no auxiliary cooling or heating devices are necessary. If the gas sample is corrosive, it is usually measured on a dry basis and reference gas dryers as well as dryers for the sample must be provided.

10. *Figure 21-19*. One form of the rotating-element densitometer is installed directly on the line and no sample handling equipment is required. A typical sampling system for another form of the densitometer is shown in Figure 21-20. Measurement is density at line conditions.

11. *Figure 21-21*. The thermal conductivity gas densitometer requires a clean, dry sample only slightly above

atmospheric pressure. Refer to Section 14 for details of sample preparation and handling systems.

21.8 Calibration

With so many different types of densitometers currently available, no attempt will be made to detail the methods of calibration. Calibration may be in terms of API gravity or specific gravity, as desired. Refer to the manufacturer's instruction manuals for this information.

21.9 Readout

All of the densitometers described in this section can be adapted to read out on most of the standard pneumatic or electrical indicating or recording instruments for measurement or control. Refer to manufacturer's literature for special adaptations.

21.10 Startup

Startup procedures are discussed in RP 550, Part II Sections 16 through 20. For additional information, refer to manufacturer's instruction manuals.

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