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Diesel and Burner Fuels

Power Test Codes

THE AMERICAN SOCIETY OF MECHANICAL ENGINEERS
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Diesel and Burner Fuels

POWER
TEST
CODES

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Foreword

THE edition of the ASME Power Test Codes issued in 1915 did not contain a Test Code for Liquid Fuels. When, however, the Society decided to revise these codes, a committee on this subject was organized for the purpose of formulating Test Codes on Solid Fuels, Liquid Fuels, and Gaseous Fuels.

After passing through the preliminary stages in the procedure prescribed by the Standing Committee, the Test Code for Liquid Fuels was printed in tentative form in the December, 1929, issue of *Mechanical Engineering*. It was then presented to the Society for discussion at a Public Hearing held during the Annual Meeting in New York in December, 1929. At the June 2, 1930, meeting of the Standing Committee it was approved in its finally revised form, and on August 4, 1930, was approved and adopted by the Council as a standard practice of the Society.

In the preparation of the original Test Code for Liquid Fuels and this recent revision, called the Test Code for Diesel and Burner Fuels, the Committee has worked in close cooperation with the ASTM Committee D-2 on Petroleum Products and Lubricants. It should be noted that certain of the ASTM specifications have been adopted as standards in the Test Code for Diesel and Burner Fuels.

This code was approved by the Power Test Codes Committee on December 6, 1957. It was approved and adopted by the Council as a standard practice of the Society by action of the Board on Codes and Standards on January 29, 1958.

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ASME POWER TEST CODES

Test Code for
Diesel and Burner Fuels

Contents

Introduction

1 The Test Code for Diesel and Burner Fuels is intended primarily to specify standard methods for the determination of those ascertainable chemical and physical properties which serve as indicators of the value of liquid fuels used in equipment for the generation of heat or of power.

It has been found advisable to adopt many standards already in use by other organizations. These are largely those of the American Society for Testing Materials which have been formulated by its Committee D-2 on Petroleum Products and Lubricants. Such modifications of these or other standards as have been made for the purpose of this code, are printed in italics.

NOTE: Certain words and phrases which are emphasized in the ASTM standards by the use of italic type, have been italicized and underscored in this publication. This has been done to avoid confusion with the modifications mentioned in the preceding paragraph.

Objects

2 The object of testing Diesel and burner fuels is to determine whether they meet specifica-

tions that the producer may establish for control in manufacture or for general sales classification, or that the consumer may establish to define the suitability of the fuels for his use in given burners, furnaces, or internal-combustion engines.

Generally speaking, the object of the Test Code for Diesel and Burner Fuels is not to formulate specifications of any of the above types. Their formulation is the province of the producer and consumer acting individually or in groups, separately or jointly. There are included in the code, however, two specifications for classification of fuel oils, (1) Specifications for Fuel Oils, and (2) Classification of Diesel Fuel Oils. These are broad specifications that have been found useful by industry after adoption by the American Society for Testing Materials and other bodies. They may be narrowed by the producer or consumer for specific applications of the fuel oil.

3 The specific objects of testing Diesel and burner fuels may include the determination of one or more of the following:

(*a*) Composition
(*b*) Content of foreign matter, solid or liquid
(*c*) Heating value
(*d*) Gravity
(*e*) Viscosity
(*f*) Ignition characteristics

4 Under the term, Diesel and burner fuels, gasoline as used in spark-ignition, internal-combustion engines, is not included.

Definition of Terms

5 The following definitions of terms relating to Diesel and burner fuels, which are excerpted from Standard Definitions of Terms Relating to Petroleum, ASTM Designation: D 288-53, are adopted for the purposes of this code.

Standard Definitions of Terms Relating to Petroleum

ASTM Designation: D 288-53

Adopted 1939; Revised 1947, 1948, 1949, 1951, 1952, 1953

These Definitions have been approved as "American Standard" by the American Standards Association

6 *The following are excerpts of standard definitions relating to petroleum.*

Crude Petroleum. A naturally occurring mixture, consisting predominantly of hydrocarbons, and/or of sulfur, nitrogen and/or oxygen derivatives of hydrocarbons, which is removed from the earth in liquid state or is capable of being so removed.

Note. Crude petroleum is commonly accompanied by varying quantities of extraneous substances such as water, inorganic matter and gas. The removal of such extraneous substances alone does not change the status of the mixture as crude petroleum. If such removal appreciably affects the composition of the oil mixture, then the resulting product is no longer crude petroleum.

Crude Shale Oil. The oil obtained as a distillate by the destructive distillation of oil-shale.

Engine Distillate. A refined or unrefined petroleum distillate similar to naphtha, but often of higher distillation range.

Fuel Oil. Any liquid or liquefiable petroleum product burned for the generation of heat in a furnace or firebox, or for the generation of power in an engine, exclusive of oils with a flash point below 100 F (38 C), Tag closed tester, and oils burned in cotton or woolwick burners. Fuel oils in common use fall into one of four classes:

(1) residual fuel oils, which are topped crude petroleums or viscous residuums obtained in refinery operations;
(2) distillate fuel oils, which are distillates derived directly or indirectly from crude petroleum;
(3) crude petroleums and weathered crude petroleums of relatively low commercial value;
(4) blended fuels, which are mixtures of two or more of the three preceding classes.

Gas Oil. A liquid petroleum distillate having a viscosity intermediate between that of kerosine and lubricating oil.

Note. It should be understood that oils, other than gas oil as defined above, may be and are used in the manufacture of gas.

Gasoline. A refined petroleum naphtha which by its composition is suitable for use as a carburant in internal combustion engines.

Kerosine. A refined petroleum distillate suitable for use as an illuminant when burned in a wick lamp.

Note. The flash point may not be below 73 F (23 C), as determined by the Abel Tester (which is approximately equivalent to 73 F (23 C) as determined by the Tag Closed Tester, ASTM Standard Method D 56). In the United States of America, local ordinances or insurance regulations require flash points higher than 73 F (23 C).

Oil-Shale. A compact rock of sedimentary origin, with an ash content of more than 33 per cent and containing organic matter that yields oil when destructively distilled but not appreciably when extracted with the ordinary solvents for petroleum.

Reid Vapor Pressure. The vapor pressure of a liquid fuel in pounds per square inch exerted at 100 F by the fuel in a Reid bomb of approved design. [Standard Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (ASTM Designation: D 323-55)].

Topped Crude Petroleum. A residual product remaining after the removal, by distillation, or other artificial means, of an appreciable quantity of the more volatile components of crude petroleum.

Tops. The unrefined distillate obtained in topping a crude petroleum.

Weathered Crude Petroleum. The product resulting from crude petroleum through loss, due to natural causes, during storage and handling, of an appreciable quantity of the more volatile components.

Collection of the Sample

7 Samples of Diesel and burner fuels to be tested may have to be taken from various types of containers, storage tanks, tank cars or trucks, drums, pipelines, or open streams. The method of sampling to be followed to obtain a sample must be varied according to the location from which it is taken. In all cases, precautions should be taken to insure that the liquid being sampled is homogeneous. A paint-mixing device is convenient for this purpose when sampling a small container. The Tentative Method of Sampling Petroleum and Petroleum Products, ASTM Designation: D 270-55T, with deletions of those sections referring to petroleum products other than Diesel and burner fuels, is adopted for the purposes of this code.

Tentative Method of Sampling Petroleum and Petroleum Products

ASTM Designation: D 270-55T

Issued 1950; Revised 1952, 1953, 1955

Scope

8 This method describes the standard procedures for obtaining representative samples of stocks or shipments of petroleum and its products.

Outline of Method

9 Samples of petroleum and its products are used in various ASTM Methods of Tests for determining physical and chemical characteristics. Therefore, it is necessary that they be truly representative of the products sampled. The precautions required to assure such a representative sample are numerous and depend on the type of product being sampled, the tank, carrier or container from which the sample is being obtained, the type and cleanliness of the sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their applications is presented in Table 1. Each procedure is suitable for sampling a number of specific products under definite storage, transportation, or container conditions. The basic principle of each procedure is to obtain a sample or a composite of several samples in such manner and from such locations in the tank or other container that the sample or composite will be truly representative of the product.

Terminology

10 (a) **Average Sample** is one that consists of proportionate parts from all sections of the container.

(b) **All-Levels Sample** is one obtained by submerging a closed sampler to a point as near as possible to the draw-off level, then opening the sampler and raising it at a rate such that it is nearly but not quite full as it emerges from the liquid. An all-levels sample is not an average sample because the tank volume may not be proportional to the depth, and because the operator cannot raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion. The tube sampling procedure, Par. 33, may be used to obtain an all-levels sample from a barrel or drum.

(c) **Upper Sample** is one obtained from the middle of the upper third of the tank contents, Fig. 1.

(d) **Middle Sample** is one obtained from the middle of the tank contents, Fig. 1.

(e) **Lower Sample** is one obtained from the middle of the lower third of the tank contents, Fig. 1.

(f) **Single Tank Composite Sample** is a blend of the upper, middle, and lower samples. For a tank of uniform cross-section, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal cylindrical tank, the blend consists of the three samples in the proportions shown in Table 2.

(g) **Multiple Tank Composite Sample (Ship, Barge, etc.)** is a blend of individual all-levels samples, from each compartment which contains the product being sampled, in proportion to the volume of material in each compartment.

(h) **Top Sample** is one obtained 6 in. below the top surface of the tank contents, Fig. 1.

TABLE 1 SUMMARY OF SAMPLING PROCEDURES AND APPLICABILITY

Procedure	Application	Tanks or Carriers	Par. No.
Bottle or Beaker Sampling	Liquids of 16 lb Rvp* or less, and semi-liquids that can be liquefied by heating	Tank cars and tank trucks, ship and barge tanks, and storage tanks	19
Tap Sampling	Liquids of 26 lb Rvp or less	Storage tanks	22
Continuous Sampling	Liquids of 2 lb Rvp or less	Filling, transfer, and pipelines	25
Dipper Sampling	Liquids of 2 lb Rvp or less, and semi-liquids	Free or open discharge streams	28
Tube Sampling	Liquids of 2 lb Rvp or less, and semi-liquids	Drums, barrels, and cans	31
Thief Sampling	Bottom samples of liquids of 2lb Rvp or less, and of semi-liquids	Tank cars and storage tanks	34

* *Reid vapor pressure.*

(i) **Outlet Sample** is one obtained at the level of the tank outlet (either fixed or a swing line outlet), Fig. 1.

(j) **Continuous Sample** is one obtained from a pipeline conveying the product in such manner as to give a representative average of the stream throughout the period of transit.

(k) **Dipper Sample** is one obtained by placing a dipper or other collecting vessel into the path of a free-flowing stream so as to collect a definite volume from the full cross-section of the stream at regular time intervals for a constant rate of flow, or at time intervals varied in proportion to the rate of flow.

(l) **Mixed Sample** is one obtained after mixing or vigorously stirring the contents of the original container, and then pouring out or drawing off the quantity desired.

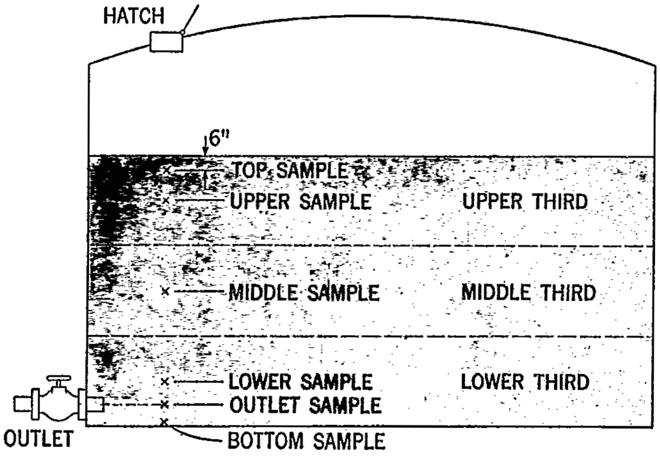


Fig. 1 Sampling Depths

(m) **Tube or Thief Sample** is one obtained with a sampling tube or special thief, either as a core sample, or spot sample from a specified point in the container.

(n) **Drain Sample** is one obtained from the draw-off or discharge valve. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

(o) **Bottom Sample** is one obtained from the material on the bottom surface of the tank, container, or line at its lowest point, Fig. 1. (Drain and bottom samples are usually taken to check for water, sludge, scale, etc.)

(p) **Borings Sample** is one obtained by collecting the chips made by boring holes with a ship auger from top to bottom of the material contained in a barrel, case, bag, or cake.

(q) **Grab Sample** is one obtained by collecting loose solids in equal quantities from each part or package of a shipment and in sufficient amount to be representative of all sizes and components.

Apparatus

Sample Containers

11 (a) **Container Specifications.** Sample containers may be clear or brown glass bottles, or cans. The clear glass bottle is advantageous because it may be examined visually for cleanliness, and also allows visual inspection of the sample for free water or solid impurities. The brown glass bottle affords some protection from light. The only cans permissible are those with the seams soldered on the exterior surfaces with a flux of rosin in a suitable solvent. Such a flux is easily removed with gasoline, whereas many others are very difficult to remove. Minute traces of flux may contaminate the sample so that results obtained on tests for dielectric strength, resistance to oxidation, and sludge formation may be erroneous.

(b) **Container Closure.** Cork or glass stoppers, or screw caps may be used for glass bottles; screw caps only for cans. Corks must be of good quality, clean and free from holes and loose bits of cork. Never use rubber stoppers. Contact of the sample with the cork may be prevented by wrapping tin or aluminum foil around the cork before forcing it into the bottle. Glass stoppers must be a perfect fit. Screw caps will endure harder usage if protected by a cork disk faced with tin or aluminum foil, or other material that will not affect petroleum products.

(c) **Cleaning Procedure.** All sample containers must be *absolutely* clean and free of water, dirt, lint, washing compounds, naphtha or other solvents, soldering fluxes or acids, corrosion, rust, and oil. Before using a container rinse it with Stoddard solvent or other naphtha of similar volatility. (It may be necessary to use sludge solvents to remove all traces of sediment and sludge from containers previously used.) Then wash the container with strong soap solution, rinse it thoroughly with tap water and finally with distilled water. Dry either by passing a current of clean, warm air through the container or by placing it in a hot dust-free cabinet at 40 C (104 F) or higher. When dry, stopper or cap the container immediately.

Sampling Apparatus

12 Sampling apparatus is described in detail under each of the specific sampling procedures. Clean, dry, and free all

Table 2 Sampling Instructions for Horizontal Cylindrical Tanks

Liquid Depth, Per cent of Diameter	Sampling Level, Per Cent of Diameter Above Bottom			Composite Sample, Proportionate Parts of		
	Upper	Middle	Lower	Upper	Middle	Lower
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70	..	50	20	..	6	4
60	..	50	20	..	5	5
50	..	40	20	..	4	6
40	..	..	20	..	..	10
30	..	..	15	..	..	10
20	..	..	10	..	..	10
10	..	..	5	..	..	10

sampling apparatus from any substance which might contaminate the material, using the procedure described in Par. 11(c).

Sampling Instructions and Precautions

Time and Place of Sampling

13 (a) **Finished Products.** When loading or discharging finished products, take samples from both shipping and receiving tanks, and from the pipeline if required.

(b) **Ship or Barge Tanks.** Sample each product immediately after the vessel is loaded, or just before discharging.

(c) **Tank Cars.** Sample the product immediately after the car is loaded, or just before unloading.

(d) **Package Lots (Cans, Drums, Barrels, or Boxes).** Take samples from a sufficient number of the individual packages to prepare a composite sample which will be representative of the entire lot or shipment. Select at random the individual packages to be sampled. The number of such random packages will depend upon several practical considerations such as:

(*1*) The tightness of the product specifications,
(*2*) The source and type of the material and whether or not more than one production batch may be represented in the lot, and
(*3*) Previous experience with similar shipments, particularly with respect to the uniformity of quality from package to package.

In most cases, the number specified in Table 3 will be satisfactory.

Obtaining Samples

14 (a) Directions for sampling cannot be made explicit enough to cover all cases. They must be supplemented by judgment, skill, and sampling experience, particularly as regards the type of sample to be taken. Extreme care and good judgment are necessary to insure samples which represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as in cold weather, or when handling materials at high temperatures, or for reasons of safety. Since many petroleum vapors are toxic and hazardous, avoid breathing them or igniting them from an open flame or a spark produced by metal equipment or tools. Do not wear hob-nailed shoes.

(b) When sampling volatile products, the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be up-ended into the opening of the sample container and remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

(c) When sampling other than volatile liquid products (2 lb Rvp or less), the sampling apparatus shall be filled and allowed to drain before drawing the actual sample. If the actual sample is to be transferred to another container, the sample container shall be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

Handling Samples

15 (a) **Volatile Samples.** It is necessary to protect volatile samples from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when the material is being transferred. When samples of more than 16 lb, Reid vapor pressure (Rvp) are being obtained, be sure to use containers strong enough to meet local safety regulations. After delivery to the laboratory, volatile samples should be cooled before the container is opened.

(b) **Light Sensitive Samples.** It is important that samples sensitive to light be kept in the dark, if the testing is to include the determinations of such properties as color, and inhibitor contents, sludge forming characteristics, stability tests, or neutralization value. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately. It is a definite advantage to use covered cardboard cartons into which the sample bottles may be placed immediately after collection.

(c) **Refined Materials.** Protect highly refined products from moisture and dust by placing paper, plastic, or metal foil over the stopper and the top of the container.

(d) **Container *Spillage*.** To allow for expansion and contraction, never completely fill a sample container.

Shipping Samples

16 To prevent loss of liquid and vapors during shipment, and to protect against moisture and dust, cover the stoppers of glass bottles with plastic caps which have been swelled in water, wiped dry, placed over the tops of the stoppered bottles and allowed to shrink tightly in place; or cover with paper tied securely at the neck. Seal paper-capped stoppers by dipping into paraffin wax or other suitable material so as to cover all of the paper cap and part of the neck. Never pour paraffin or sealing wax directly over stoppers. Be careful to avoid contamination of samples when removing such caps.

Labeling Sample Containers

17 Label the container immediately after a sample is obtained. Use waterproof and oil-proof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing, and handling. Include the following information:

(*1*) Date and time (and for continuous and dipper samples the hour and minute of collection),
(*2*) Name of the sampler,
(*3*) Name or number and owner of the vessel, car, or container,
(*4*) Brand and grade of material, and
(*5*) Reference symbol or identification number.

Sampling Procedures

General Information

18 The standard sampling procedures described in this method are summarized in Table 1. Alternative sampling

TABLE 3 MINIMUM NUMBER OF PACKAGES TO BE SELECTED FOR SAMPLING

No. of Packages in the Lot	No. of Packages to be Sampled	No. of Packages in the Lot	No. of Packages to be Sampled	No. of Packages in the Lot	No. of Packages to be Sampled
1 to 3	all	513 to 729	9	2 745 to 3 375	15
4 to 64	4	730 to 1 000	10	3 376 to 4 096	16
65 to 125	5	1 001 to 1 331	11	4 097 to 4 913	17
126 to 216	6	1 332 to 1 728	12	4 914 to 5 832	18
217 to 343	7	1 729 to 2 197	13	5 833 to 6 859	19
344 to 512	8	2 198 to 2 744	14	6 860 or over	20

procedures may be used if a mutually satisfactory agreement has been reached by the parties involved. Such agreement shall be put in writing and signed by authorized officials.

BOTTLE OR BEAKER SAMPLING

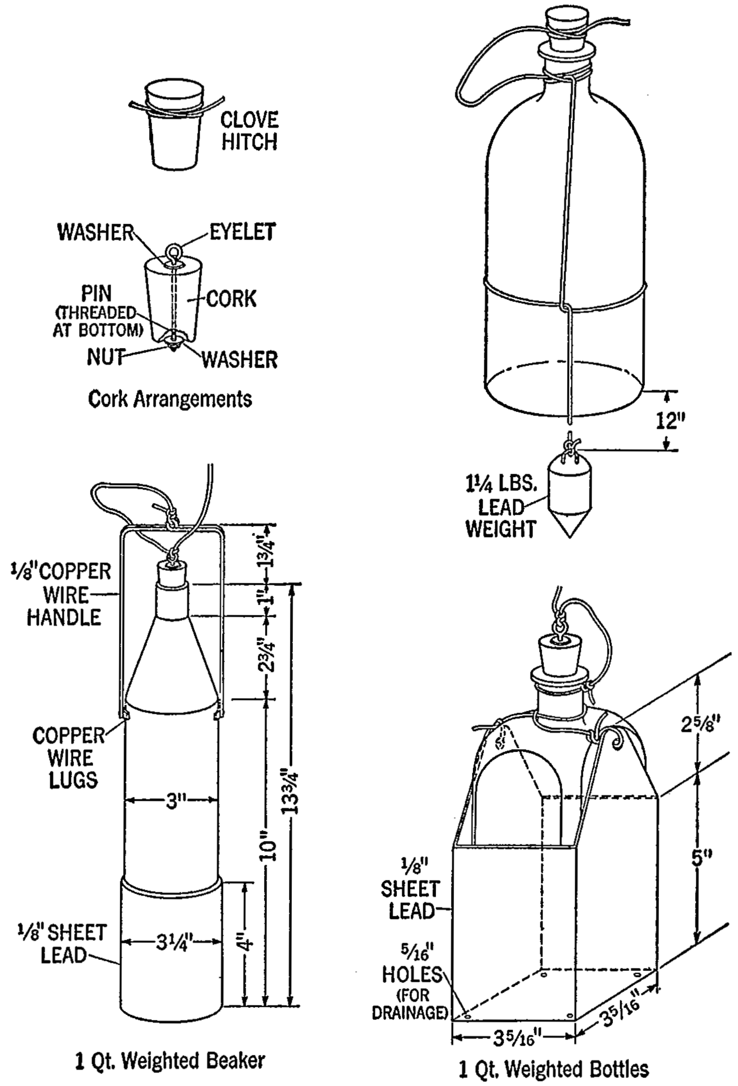
Application

19 The bottle or beaker sampling procedure is applicable for sampling liquids of 16 lb Rvp or less in tank cars, tank trucks, shore tanks, ship tanks and barge tanks. Semi-liquid materials which can be heated until sufficiently fluid may be sampled by this procedure.

Apparatus

20 A suitable sampling bottle or beaker, Fig. 2, is required. Recommended uses and sizes of openings are as follows:

Material	Diameter of Opening, In.	
	Weighted Sampling Bottle	Weighted Sampling Beaker
Light lubricating oils, kerosines, gasolines, transparent gas oils, *part cargoes* of Diesel fuels, distillates, or non-transparent gas oils	$\frac{3}{4}$	..
Heavy lubricating oils	$1\frac{1}{2}$	..
Light crude oils (less than 200 sec Saybolt universal viscosity at 100 F) and *full cargoes* of Diesel fuels, distillates, or non-transparent gas oils	$\frac{3}{4}$	$\frac{3}{4}$
Heavy crude and fuel oils	$1\frac{1}{2}$	$1\frac{1}{2}$



Cork Arrangements

1 Qt. Weighted Beaker

1 Qt. Weighted Bottles

FIG. 2 CONTAINER ASSEMBLIES FOR BOTTLE OR BEAKER SAMPLING

Procedure

21 (a) **All-Levels Sample.** Lower the weighted, stoppered bottle or beaker as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the cord or chain (nonsparking) and raise the bottle or beaker at such a rate that it is nearly but not quite full as it emerges from the liquid.

(b) **Upper, Middle, and Lower Samples.** Lower the weighted, stoppered bottle or beaker to the proper depths, Fig. 1, which are as follows:

Upper sample	middle of upper third of the tank contents.
Middle sample	middle of the tank contents.
Lower sample	middle of lower third of the tank contents.

Pull out the stopper with a sharp jerk of the cord or chain (nonsparking) and allow the bottle or beaker to fill completely at the selected level, as evidenced by the cessation of air bubbles. When full, raise the bottle or beaker, pour off a small amount, and stopper immediately.

(c) **Composite Sample.** Prepare a composite sample in the laboratory (not in the field) by mixing portions of the all-levels samples as specified in Par. 10 (g), or by mixing portions of the upper, middle, and lower samples as specified in Par. 10 (g).

(d) **Top and Outlet Samples.** Obtain these samples, Fig. 1, in the same manner as specified in Par. (b) but at the following depths:

Top Sample	6 in. below the top surface of the tank contents.
Outlet Sample	opposite the tank outlet (either fixed or swing line outlet).

(e) **Handling.** Immediately transfer *beaker samples* into clean, dry cans of convenient size, close and label the cans, and deliver them to the laboratory. Stopper and label *bottle samples* immediately after taking them, and deliver to the laboratory in the original sampling bottles.

TAP SAMPLING

Application

22 The tap sampling procedure is applicable for sampling liquids of 26 lb Rvp or less in tanks which are equipped with suitable sampling taps or lines. It is recommended for volatile stocks in tanks of the breather and balloon roof type, spheroids, etc. (Samples may be taken from the drain cocks of gage glasses, if the tank is not equipped with sampling taps.)

Apparatus

23 (a) **Tank Taps.** The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height and extending at least 3 ft inside the tank shell. A standard ¼-in. pipe with suitable valve is satisfactory.

(b) **Tube.** A delivery tube which will not contaminate or affect the product being sampled and of sufficient length to reach the bottom of the sample container is required to permit *submerged* filling.

(c) **Sample Containers.** Use clean, dry glass bottles of convenient size and strength to receive the samples. If the vapor pressure of the product to be sampled is between 16 and 26 lb Rvp, protect the bottle with a metal cover until the sample is discarded.

Procedure

24 Before a sample is drawn, flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

CONTINUOUS SAMPLING

Application

25 The continuous sampling procedure is applicable for sampling liquids of 2 lb Rvp or less and semi-liquids in pipelines, filling lines, and transfer lines.

Apparatus

26 (a) **Line Connection.** Construct and install the continuous sampling connection according to the form and dimensions shown in Fig. 3. To keep the orifice free from dirt, screens of larger area than the openings may be placed around the ¼-in. pipes, or ¼-in. screen fittings may be inserted in the drip lines. A pipe cap with a suitable orifice may be substituted for the cocks, provided the orifice size which will yield the correct amount of sample has been predetermined.

(b) **Receiver.** Use a clean, dry container of convenient size to receive the sample.

Procedure

27 Turn the three plug cocks to the same angle of opening, as nearly as possible, so that steady streams are drawn from the cocks and at such rates that about 0.1 per cent of the product, but not more than 40 gal, is diverted during its passage through the line. When sampling semi-liquids, heat the receiver with steam coils to a temperature just sufficient to keep the material in a liquid state. Label and deliver continuous samples to the laboratory in the containers in which they are collected.

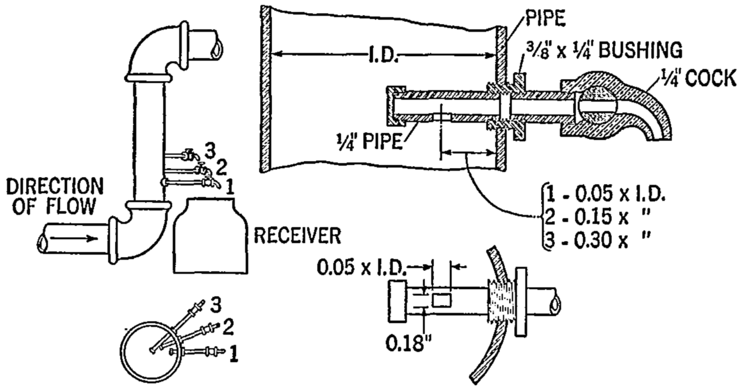


FIG. 3 ASSEMBLY FOR CONTINUOUS SAMPLING

DIPPER SAMPLING

Application

28 The dipper sampling procedure is applicable for sampling liquids of 2 lb Rvp or less and semi-liquids where a free or open discharge stream exists, as in small filling and transfer pipelines (2 in. diameter or less) and filling apparatus for barrels, packages, and cans.

Apparatus

29 (a) **Dipper.** Use a dipper with a flared bowl and a handle of convenient length, made of material such as tinned steel that will not affect the product being tested. It should have a capacity suitable for the amount to be collected, Par. 30, and must be protected from dust and dirt when not being used.

(b) **Sample Container.** Use a clean, dry container of the desired size.

Procedure

30 Insert the dipper in the free flowing stream so that a portion is collected from the full cross-section of the stream. Take portions at time intervals chosen so that a complete sample proportional to the pumped quantity is collected. The gross amount of sample collected should be approximately 0.1 per cent, but not more than 40 gal, of the total quantity being sampled. Transfer the portions into the sample container as soon as collected. Keep the container closed, except when pouring a dipper portion into it. As soon as all portions of the sample have been collected, close and label the sample container, and deliver it to the laboratory.

TUBE SAMPLING

Application

31 The tube sampling procedure is applicable for sampling liquids of 2 lb Rvp or less and semi-liquids in drums, barrels, and cans.

Apparatus

32 (a) **Tube.** Either a glass or metal tube may be used, designed so that it will reach to about 1/8 in. of the bottom and have a capacity of approximately 1 pt or 1 qt. A metal tube suitable for sampling 50 gal drums is shown in Fig. 4. Two rings soldered to opposite sides of the tube at the upper end are convenient for holding it by slipping two fingers through the rings, thus leaving the thumb free to close the opening.

(b) **Sample Containers.** Use clean, dry cans or glass bottles.

Procedure

33 (a) **Drums and Barrels.** Place the drum or barrel on its side with the bung up. If the drum does not have a side bung, stand it upright and sample from the top. If detection of water, rust, or other insoluble contaminants is desired, let the barrel or drum remain in this position long enough to permit the contaminants to settle. Remove the

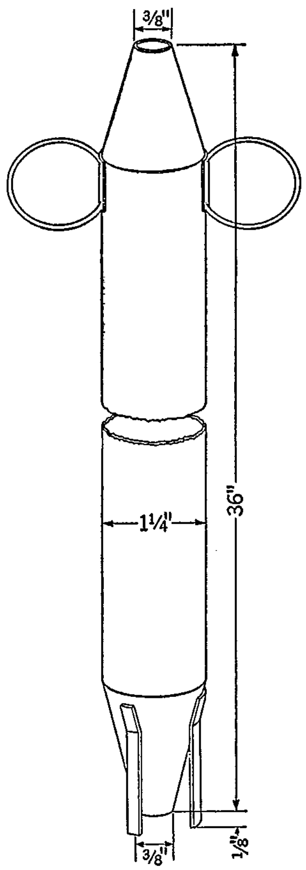
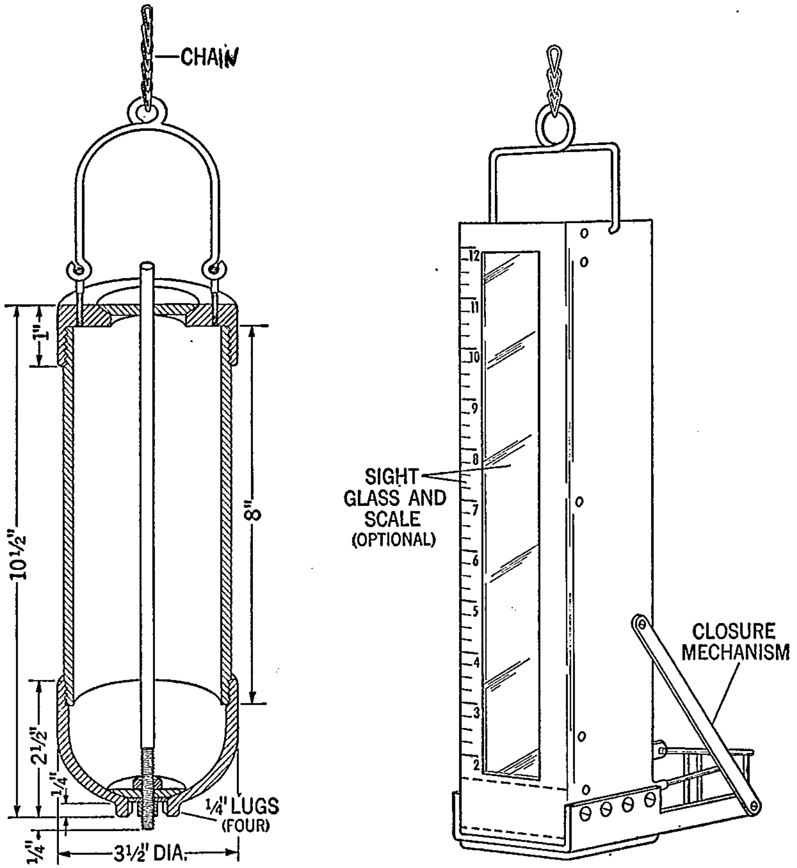


FIG. 4 SAMPLING TUBE



(a) *Bomb Type* (b) *Trap Type*

FIG. 5 TYPICAL SAMPLING THIEFS

bung and place it beside the bung hole with the oily side up. Close the upper end of the clean, dry sampling tube with the thumb, and lower the tube into the oil for a depth of about 1 ft. Remove the thumb, allowing oil to flow into the tube. Again close the upper end with the thumb and withdraw the tube. Rinse the tube with the oil by holding it nearly horizontal and turning it so that the oil comes in contact with that part of the inside surface which will be immersed when the sample is taken. Avoid handling any part of the tube which will be immersed in the oil during the sampling operation. Discard the rinse oil and allow the tube to drain. Insert the tube into the oil again, holding the thumb against the upper end. (If an all-levels sample is desired, insert the tube with the upper end open.) When the tube reaches the bottom, remove the thumb and allow the tube to fill. Replace the thumb, withdraw the tube quickly and transfer the contents to the sample container. Do not allow the hands to come in contact with any part of the sample. Close the sample container; replace and tighten the bung in the drum or barrel. Label the sample container and deliver it to the laboratory.

(b) **Cans.** Obtain samples from cans of 5-gal capacity or larger in the same manner as from drums and barrels, Par. (a), using a tube of proportionately smaller dimensions. For cans of less than 5-gal capacity, use the entire contents as the sample, selecting cans at random as indicated in Table 3 or in accordance with agreement between the purchaser and the seller.

THIEF SAMPLING

Application

34 The thief sampling procedure is applicable for obtaining bottom samples, Fig. 1, of liquids of 2 lb Rvp or less, or of semi-liquids in tank cars and storage tanks.

Apparatus

35 (a) **Thief.** The thief shall be designed so that a sample can be obtained within $\frac{1}{2}$ in. of the bottom of the car or tank. Two types of thiefs are illustrated in Fig. 5. One type is lowered into the tank with valves open to permit the oil to flush through the container. When the thief strikes the bottom of the tank, the valves shut automatically to trap a bottom sample. The other type has a projecting stem on the valve rod which opens the valves automatically as the stem strikes the bottom of the tank. The sample enters the container through the bottom valve and air is released simultaneously through the top. The valves snap shut when the thief is withdrawn.

(b) **Sample Containers.** Use clean, dry cans or glass bottles.

Procedure

36 Lower the clean, dry thief through the dome of the tank car or tank hatch until it strikes the bottom. When full, remove the thief and transfer the contents to the sample container. Close and label the container immediately, and deliver it to the laboratory.

Gravity

37 The specific gravity of a Diesel and burner fuel is in itself not a definite indication of the quality of the fuel. However, it provides a useful and convenient means of checking the uniformity of a product which is the result of blending operations and it must be known to calculate the weight of a given volume of oil for shipping purposes. It is also of value in calculating other properties of oil such as the approximate calorific value and Diesel index.

This code adopts the Standard Method of Test for API Gravity of Petroleum and Its Products, Hydrometer Method, ASTM Designation: D 287-55. This standard, in addition to defining the method for the determination of the specific gravity, includes the method for the calculation of the API gravity, an arbitrary scale that is widely used. It also includes an abbreviated table for conversion from one scale to the other and to pounds per gallon or gallons per pound.

Standard Method of Test for API Gravity of Petroleum and Its Products

Hydrometer Method

ASTM Designation: D 287-55

Adopted 1954; Revised 1955

This Method has been approved as "American Standard" by the American Standards Association

Scope

38 This method describes a procedure for the determination by means of a glass hydrometer of the API gravity (in vacuum) of crude petroleum and of petroleum products normally handled as liquids and having a Reid vapor pressure of 26 lb or less. Results are determined at 60 F, or converted to values at 60 F, by means of standard tables (Note 1).

NOTE 1. The procedure for measurement of specific gravity 60/60 F by hydrometer is described in ASTM Method D 1298.

Definition

39 API Gravity is defined by the following equation:

$$\text{API Gravity, deg} = \frac{141.5}{\text{sp gr } 60/60\text{ F}} - 131.5$$

Apparatus

40 The following apparatus is required:

(a) **Hydrometers**, of glass, graduated in degrees API (in vacuum) as listed in Table 4 and conforming to the Tentative Specifications for ASTM Hydrometers (ASTM Designation: E 100). For referee testing of petroleum products, the long plain form of hydrometer (1H to 10H) should be used.

TABLE 4 AVAILABLE API HYDROMETERS

ASTM Designation	Type	API Range	
		Total	Each Hydrometer
1H to 10H	long plain form	—1 to 101	12
21H to 40H	short plain form	0 to 101	6
51H to 60H	thermo-hydrometer	—1 to 101	12
71H to 74H	thermo-hydrometer	—1 to 41	12

(b) **Thermometers,** having a range of —5 to +215 F and conforming to the requirements for Thermometer 12 F as prescribed in ASTM Specifications E 1.

(c) **Hydrometer Cylinders,** made of metal, clear glass, or plastic. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25 mm than the portion of the hydrometer which is immersed beneath the surface of the sample.

Temperature of Test

41 The gravity determined by the hydrometer method is most accurate at or near the standard temperature of 60 F; use this or any other temperature between 0 and 195 F for test so far as it is consistent with the type of sample and necessary limiting conditions shown in Table 5.

Procedure

42 (a) Adjust the temperature of the sample in accordance with Table 5. The hydrometer cylinder and thermometer shall be at approximately the same temperature as the sample to be tested.

(b) Pour the sample into the clean hydrometer jar without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower-boiling constituents of the more volatile samples. For the more volatile samples, transfer to the hydrometer cylinder by siphoning. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper before inserting the hydrometer. Place the cylinder containing the sample in a vertical position in a location free from air currents. Take precautions to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. During this period, the temperature of the surrounding medium should not change by more than 5 F.

(c) Lower the hydrometer gently into the sample and, when it has settled, depress it about two scale divisions into the liquid and then release; keep the rest of the stem dry, as unnecessary liquid on the stem changes the effective weight of the instrument, and so affects the reading obtained. With samples of low viscosity, a slight spin imparted to the instrument on releasing assists in bringing it to rest, floating freely away from the walls of the hydrometer cylinder. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. This is particularly necessary in the case of the more viscous samples.

(d) When the hydrometer has come to rest, floating freely, and the temperature of the sample is constant to 0.2 F, read the hydrometer to the nearest scale division. The correct reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

(e) To make a reading with nontransparent oils, observe the point on the hydrometer scale to which the sample rises above its main surface, placing the eye slightly above the plane surface of the liquid. This reading requires a correction. Determine this correction for the particular hydrometer in use by observing the height above the main surface of the liquid to which the oil rises on the hydrometer scale when the hydrometer in question is immersed in a transparent oil having a surface tension similar to that of the sample under test.

(f) Observe the temperature of the sample to the nearest 0.25 F immediately before and after the observation of the gravity, the liquid in the cylinder being thoroughly but cautiously stirred with the thermometer, the whole of the mercury thread being immersed. Should these temperature readings differ by more than 1F, repeat the temperature and gravity observations when the temperature of the sample has become more stable. Record the mean of the thermometer reading before and after the final hydrometer reading, to the nearest degree Fahrenheit, as the temperature of the test (Note 2).

NOTE 2. When thermo-hydrometers are used, stir the sample by carefully raising and lowering the hydrometer. It is satisfactory in this case to read the thermometer scale after the hydrometer reading has been observed. Because the thermometer incorporated in the hydrometer possesses certain inherent defects of design, the precision of the reported value will be poorer than that described in Par. 44.

TABLE 5 LIMITING CONDITIONS AND TEST TEMPERATURES

Sample Type	Gravity Limits	Initial Boiling Point Limits	Other Limits	Test Temperature
Highly volatile	Lighter than 70° API	....	....	Cool to 35 F or lower in original closed container
Moderately volatile	Heavier than 70° API	Below 250 F	....	Cool to 65 F or lower in original closed container
Moderately volatile and viscous	Heavier than 70° API	Below 250 F	Viscosity too high at 65 F	Heat to minimum temperature for sufficient fluidity
Nonvolatile	Heavier than 70° API	Above 250 F	....	Any temperature between 0 and 195 F as convenient
Mixtures of non-petroleum with petroleum products	....	....	....	60 ± 0.25 F

Calculation

43 (a) When gravities have been observed on opaque

liquids by the procedure given in Par. 41(e), subtract the correction in degrees API from the hydrometer reading observed (Note 3).

(b) Correct all hydrometer readings to 60 F, using Table 5, "Reduction of Observed API Gravity to API Gravity at 60 F," of the ASTM-IP Petroleum Measurement Tables (American Edition).

NOTE 3. The interconversion of API gravity to specific gravity is given in Table 3, "API Gravity at 60 F to Specific Gravity at 60 F and to Density at 15 C" of the ASTM-IP Petroleum Measurement Tables (American Edition).

(c) Equivalent results can be obtained by determining specific gravity 60/60 F according to Method D 1298 and converting the results to API Gravity at 60 F by means of Table 3 of the ASTM-IP Petroleum Measurement Tables (American Edition).

Precision

44 The following criteria should be used for judging the acceptability of results obtained at temperatures differing from 60 F by less than 18 F:

Repeatability	**Reproducibility**
Duplicate Results by the Same Operator	Average of Two Results in Each of Two Laboratories
Deg API 0.2	Deg API 0.5

Appendix

The following table is an abridgement of Table 5 of the "National Standard Petroleum Oil Tables" as published in the National Bureau of Standards *Circular C 410** which contains similar data for each 0.1 deg API from 0 to 100 deg API, inclusive.

* *Par. 197, covering Petroleum-Measurement Tables refer to recently published tables of the American Society for Testing Materials and the Institute of Petroleum, Great Britain, which are more complete than those of the National Bureau of Standards.*

TABLE SHOWING SPECIFIC GRAVITIES, POUNDS PER GALLON, AND U.S. GALLONS PER POUND AT 60 F CORRESPONDING TO DEGREES API

Deg API	Specific Gravity at 60/60 F	Pounds per Gallon at 60 F[a]	Gallons per Pound at 60 F[a]
0	1.076	8.962	0.1116
1	1.068	8.895	0.1124
2	1.060	8.828	0.1133
3	1.052	8.762	0.1141
4	1.044	8.698	0.1150
5	1.037	8.634	0.1158
6	1.029	8.571	0.1167
7	1.022	8.509	0.1175
8	1.014	8.448	0.1184
9	1.007	8.388	0.1192
10	1.0000	8.328	0.1201
11	0.9930	8.270	0.1209
12	0.9861	8.212	0.1218
13	0.9792	8.155	0.1226
14	0.9725	8.099	0.1235
15	0.9659	8.044	0.1243
16	0.9593	7.989	0.1252
17	0.9529	7.935	0.1260
18	0.9465	7.882	0.1269
19	0.9402	7.830	0.1277
20	0.9340	7.778	0.1286
21	0.9279	7.727	0.1294
22	0.9218	7.676	0.1303
23	0.9159	7.627	0.1311
24	0.9100	7.578	0.1320
25	0.9042	7.529	0.1328
26	0.8984	7.481	0.1337
27	0.8927	7.434	0.1345
28	0.8871	7.387	0.1354
29	0.8816	7.341	0.1362
30	0.8762	7.296	0.1371
31	0.8708	7.251	0.1379
32	0.8654	7.206	0.1388
33	0.8602	7.163	0.1396
34	0.8550	7.119	0.1405
35	0.8498	7.076	0.1413
36	0.8448	7.034	0.1422
37	0.8398	6.993	0.1430
38	0.8348	6.951	0.1439
39	0.8299	6.910	0.1447
40	0.8251	6.870	0.1456
41	0.8203	6.830	0.1464
42	0.8155	6.790	0.1473
43	0.8109	6.752	0.1481
44	0.8063	6.713	0.1490
45	0.8017	6.675	0.1498
46	0.7972	6.637	0.1507
47	0.7927	6.600	0.1515
48	0.7883	6.563	0.1524
49	0.7839	6.526	0.1532
50	0.7796	6.490	0.1541
51	0.7753	6.455	0.1549
52	0.7711	6.420	0.1558
53	0.7669	6.385	0.1566
54	0.7628	6.350	0.1575
55	0.7587	6.316	0.1583
56	0.7547	6.283	0.1592
57	0.7507	6.249	0.1600
58	0.7467	6.216	0.1609
59	0.7428	6.184	0.1617

[a] The data in this column are based upon apparent weights in air, the weight of one gallon of water at 60 F in air being assumed to be 8.32828 lb. Consequently, a correction for the buoyancy of air is required in calculating these figures from the specific gravities in the second column.

(*Continued on next page*)

TABLE SHOWING SPECIFIC GRAVITIES, POUNDS PER GALLON, AND U.S. GALLONS PER POUND AT 60 F CORRESPONDING TO DEGREES API (*Continued*)

Deg API	Specific Gravity at 60/60 F	Pounds per Gallon at 60 F[a]	Gallons per Pound at 60 F[a]
60	0.7389	6.151	0.1626
61	0.7351	6.119	0.1634
62	0.7313	6.087	0.1643
63	0.7275	6.056	0.1651
64	0.7238	6.025	0.1660
65	0.7201	5.994	0.1668
66	0.7165	5.964	0.1677
67	0.7128	5.934	0.1685
68	0.7093	5.904	0.1694
69	0.7057	5.874	0.1702
70	0.7022	5.845	0.1711
71	0.6988	5.817	0.1719
72	0.6953	5.788	0.1728
73	0.6919	5.759	0.1736
74	0.6886	5.731	0.1745
75	0.6852	5.703	0.1753
76	0.6819	5.676	0.1762
77	0.6787	5.649	0.1770
78	0.6754	5.622	0.1779
79	0.6722	5.595	0.1787
80	0.6690	5.568	0.1796
81	0.6659	5.542	0.1804
82	0.6628	5.516	0.1813
83	0.6597	5.491	0.1821
84	0.6566	5.465	0.1830
85	0.6536	5.440	0.1838
86	0.6506	5.415	0.1847
87	0.6476	5.390	0.1855
88	0.6446	5.365	0.1864
89	0.6417	5.341	0.1872
90	0.6388	5.316	0.1881
91	0.6360	5.293	0.1889
92	0.6331	5.269	0.1898
93	0.6303	5.246	0.1906
94	0.6275	5.222	0.1915
95	0.6247	5.199	0.1924
96	0.6220	5.176	0.1932
97	0.6193	5.154	0.1940
98	0.6166	5.131	0.1949
99	0.6139	5.109	0.1957
100	0.6112	5.086	0.1966

[a] The data in this column are based upon apparent weights in air, the weight of one gallon of water at 60 F in air being assumed to be 8.32828 lb. Consequently, a correction for the buoyancy of air is required in calculating these figures from the specific gravities in the second column.

Viscosity

45 Viscosity of a fluid is a term applied to the inner friction which tends to bring to rest portions of the fluid which are moving relative to one another. It may be defined as the force required to move a plane surface of unit area at a unit velocity with reference to another plane surface separated from the first by a layer of the fluid of unit thickness. The unit for the expression of viscosity is the poise or centipoise which equals 1/100 poise. The kinematic viscosity of a fluid is the viscosity divided by the density; the unit for expression is the stoke or centistoke. These values are normally expressed in cgs units.

Expressed simply, the rate of flow of a fluid at a definite temperature through an orifice of definite size and shape is a measure of its viscosity. The viscosity of Diesel and burner fuels decreases with increase in temperature. Hence the viscosity of an oil affects the rate of flow through pumps, piping, valves, orifices, and nozzles, and affects the degree of atomization at the nozzle of a burner, and the angle of the atomized spray. For these reasons, a knowledge of the viscosity of a liquid fuel and of its relation to temperature is of the utmost importance to the design of a system for the handling and burning of the fuel.

The Standard Method of Test for Saybolt Viscosity, ASTM Designation: D 88-56, is adopted for the purpose of this code. It includes the determination by the Saybolt Universal viscosimeter for less viscous fluids and the Saybolt Furol viscosimeter for more viscous fluids. A method for the experimental determination of the kinematic viscosity is available as ASTM Designation: D 445-53T. It is not included in this code, but tables for the calculation of this value from either the Saybolt Universal or Saybolt Furol values are given on pages 30 and 31.

The rate of change of the viscosity of a fuel oil with change in temperature, which is termed the viscosity index, is an important characteristic of an oil as it determines the temperature to which an oil must be heated to obtain a given pumpability or the proper viscosity for atomization. The Standard Method for Calculating Viscosity Index, ASTM Designation: D 567-53, and Standard Viscosity Charts for Liquid Petroleum Products, ASTM Designation: D 341-43, are adopted by this code. By the use of the charts specified in the latter standard, the viscosity of an oil at a given temperature may be determined when the viscosities at two other temperatures are known.

Standard Method of Test for Saybolt Viscosity

ASTM Designation: D 88-56

Adopted 1926; Revised 1930, 1933, 1936, 1938, 1944, 1953, 1955, 1956

This Method has been approved as "American Standard" by the American Standards Association

Scope

46.1 This method describes procedures for the empirical measurement of Saybolt viscosity of petroleum products at specified temperatures between 70 and 210 F. There is also included a special procedure for waxy and resinous materials.

Definition

46.2 (a) **Saybolt Universal Viscosity.** The efflux time in seconds of 60 ml of sample flowing through a calibrated Universal orifice under specified conditions.

(b) **Saybolt Furol Viscosity.** The efflux time in seconds of 60 ml of sample flowing through a calibrated Furol orifice under specified conditions. The Furol viscosity is approximately one-tenth the Universal viscosity, and is recommended for those petroleum products having viscosities

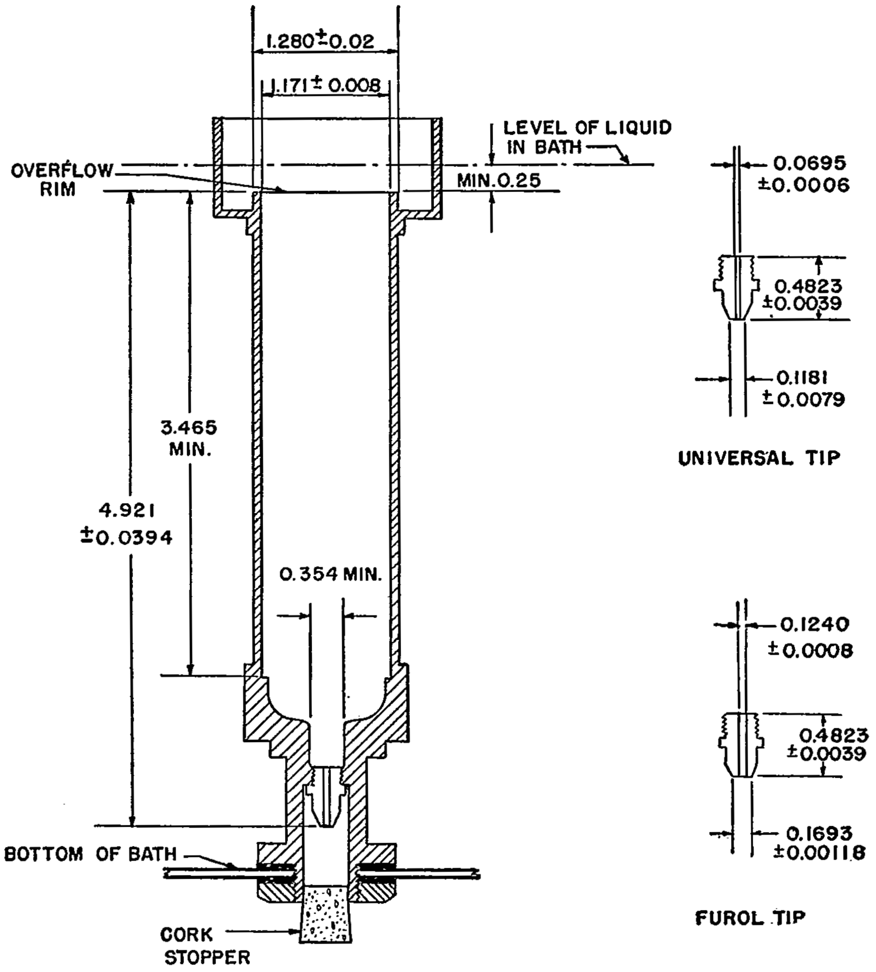


FIG. 6A SAYBOLT VISCOMETER WITH UNIVERSAL AND FUROL ORIFICE

All Dimensions in Inches

greater than 1000 sec (Saybolt Universal), such as fuel oils, and other residual materials. The word "Furol" is a contraction of fuel and road oils.

Outline of Method

46.3 The efflux time in seconds of 60 ml of sample, flowing through a calibrated orifice, is measured under carefully controlled conditions. This time is corrected by an orifice factor, and reported as the viscosity of the sample at that temperature.

Apparatus

47.1 (a) **Saybolt Viscometer and Bath,** as described in Appendix I. (See Fig. 6A.)

(b) **Withdrawal Tube** as shown in Fig. 6B.
(c) **Thermometer Support** as shown in Fig. 6C.
(d) **Filter Funnel** as shown in Fig. 7A.
(e) **Receiving Flask** as shown in Fig. 7B.
(f) **Timer,** graduated in tenths of a second, and accurate to within 0.1 per cent when tested over a 60-min interval. Electric timers are acceptable if operated on a controlled frequency circuit.

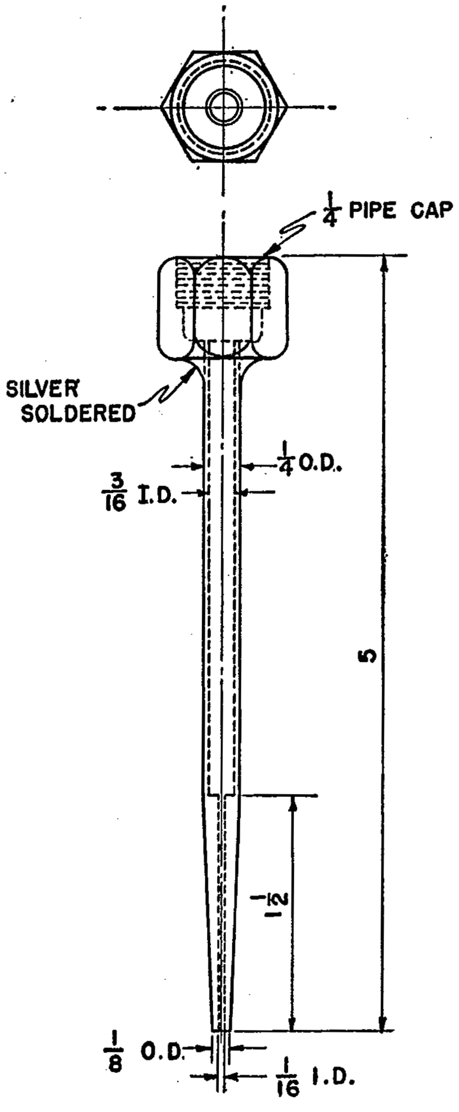


Fig. 6B Withdrawal Tube for Use with Saybolt Viscometer

All Dimensions in Inches

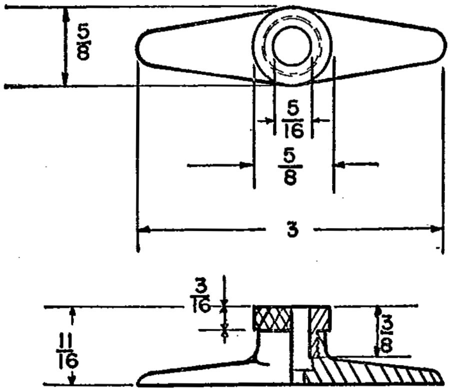


Fig. 6C Thermometer Support

All Dimensions in Inches

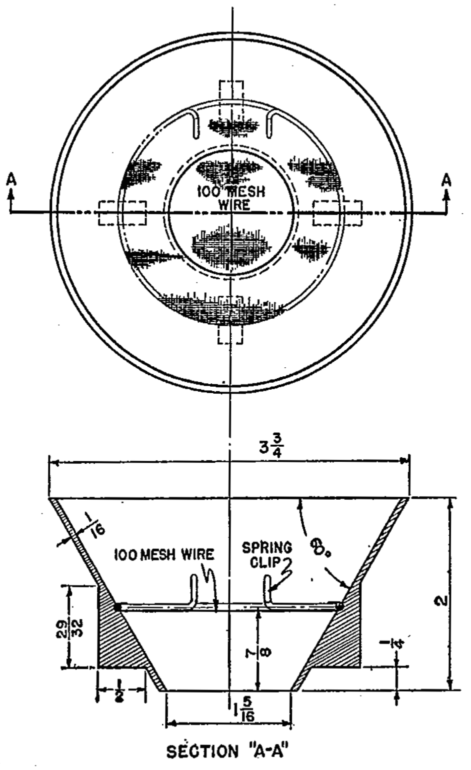


Fig. 7A Filter Funnel for Use with Saybolt Viscometer

All Dimensions in Inches

(g) **Viscosity Thermometers**, for reading the test temperature of the sample. The ASTM Saybolt Viscosity Thermometers shown in Table 6 shall conform to the requirements in ASTM Specifications for Thermometers, E 1.

(h) **Bath Thermometers.** Viscosity thermometers, or any other means of equivalent accuracy.

Preparation of Apparatus

47.2 (a) Use a Universal orifice for lubricants and distillate materials with efflux times greater than 32 sec to give the desired accuracy. Liquids with efflux times over 1000 sec are not conveniently tested with this orifice.

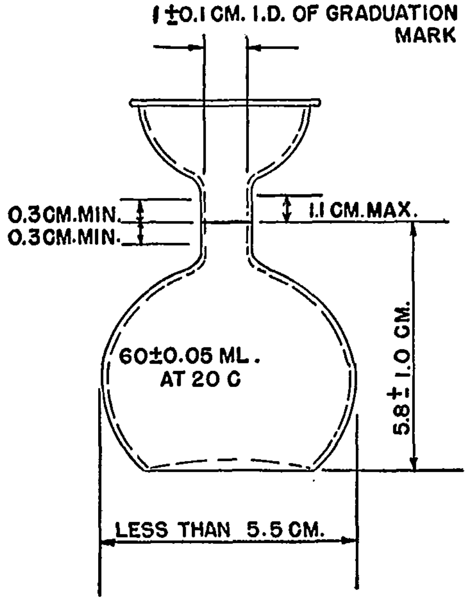


FIG. 7B RECEIVING FLASK

All Dimensions in Millimeters

(b) Use a Furol orifice for residual materials with efflux times greater than 25 sec. The Furol efflux time is approximately one-tenth the Universal efflux time.

NOTE 2. The Universal orifice is used at 70, 100, 130, and 210 F. The Furol orifice is used at 77, 100, 122, and 210 F.

(c) Set up the viscometer and bath where they will be free from drafts and rapid changes in air temperature. Locate them so that the sample will not be contaminated by dust or vapors during the test.

(d) Viscosity determinations shall not be made at temperatures below the dew point of the room's temperature. Room temperatures up to 100 F will not introduce errors in excess of 1.0 per cent. For standardization and referee tests, the room temperature shall be kept between 68 and 86 F, and the actual temperature recorded.

TABLE 6 ASTM SAYBOLT VISCOSITY THERMOMETERS

Standard Test Temperature, F	Temperature Range, F	Sub-divisions, F	ASTM Thermometer No.
70	66 to 80	0.2	17 F
77	66 to 80	0.2	17 F
100	94 to 108	0.2	18 F
122	120 to 134	0.2	19 F
130	120 to 134	0.2	19 F
140	134 to 148	0.2	20 F
180	174 to 188	0.2	21 F
210	204 to 218	0.2	22 F

(e) Fill the bath at least ¼ in. above the overflow rim of the viscometer. Table 7 lists recommended bath media for each test temperature.

(f) Provide adequate stirring and thermal control for the bath so that the sample will not fluctuate more than ±0.05 F after reaching the test temperature.

(g) Clean the viscometers with an effective nontoxic solvent and remove all solvent from the gallery and viscometer.

NOTE 3. The plunger commonly supplied with the viscometer should never be used for cleaning as the overflow rim and walls of the viscometer may be damaged by its use.

Calibration of Viscometers

48 (a) Calibrate the Saybolt Universal viscometer at periodic intervals by measuring the efflux time at 100 F of an appropriate viscosity standard, following the procedure given in Par. 49. See Appendix II.

TABLE 7 BATH TEST TEMPERATURES

Standard Test Temperature, F.	Recommended Bath Medium	Temperature Differential,[a] F Maximum	Functional Precision
70	Water	±0.1	±0.05
77	Water	±0.1	±0.05
100	Water, or Oil of viscosity 50 to 70 sec S.U. at 100 F	+0.25	±0.05
122	Water, or Oil of viscosity 120 to 150 sec S.U. at 100 F	+0.35	±0.05
130	Water, or Oil of viscosity 120 to 150 sec S.U. at 100 F	+0.05	±0.05
140	Water, or Oil of viscosity 120 to 150 sec S.U. at 100 F	+1.0	±0.1
180	Water, or Oil of viscosity 330 to 370 sec S.U. at 100 F	+1.5	±0.1
210	Oil of viscosity 330 to 370 sec S.U. at 100 F	+2.0	±0.1

S.U. = Saybolt Universal.

[a] Maximum difference allowed between bath temperature and test temperature to maintain thermal equilibrium while stirring sample in viscometer with test thermometer.

NOTE 4. Saybolt viscometers can be certified at the National Bureau of Standards by the use of oil standards having viscosity values established using distilled water at 68 F as the basic standard.

(b) The efflux time shall equal the certified Saybolt viscosity value. If the efflux time differs from the certified value by more than 0.2 per cent, calculate a correction factor, F, for the viscometer as follows:

$$F = \frac{V}{t}$$

where:

V = certified Saybolt viscosity of the standard, and
t = efflux time in seconds at 100 F.

NOTE 5. The correction factor applies at all viscosity levels and for all temperatures, provided the calibration is based on a viscosity standard having an efflux time between 200 and 600 sec.

(c) Calibrate the Saybolt Furol viscometer at 122 F in the same manner as above, using a viscosity standard having a minimum efflux time of 90 sec.

(d) Viscometers or orifices which have corrections in excess of 1.0 per cent shall not be used for referee testing.

Procedure

49 (a) If the test temperature is above room temperature, the test may be expedited by preheating the sample to not more than 3 F above the test temperature. Never preheat any sample to within 50 F of its flash point (see ASTM Method D 93, Test for Flash Point by Means of the Pensky-Martens Closed Tester), as volatility losses may alter its composition.

(b) Insert a cork stopper, having a cord attached for its easy removal, into the air chamber at the bottom of the viscometer. The cork shall fit tightly enough to prevent the escape of air, as evidenced by the absence of oil on the cork when it is withdrawn.

(c) Filter the prepared sample through a 100-mesh screen directly into the viscometer until the level is above the overflow rim.

(d) Stir the sample until its temperature remains constant within 0.05 F of the test temperature during 1 min of continuous stirring. Stir with a viscosity thermometer equipped with a thermometer support (Fig. 6C). Use a circular motion at 30 to 50 rpm in a horizontal plane.

NOTE 6. Never adjust the temperature by immersing hot or cold bodies into the sample. Such thermal treatment may affect the sample and the precision of the test.

(e) Remove the thermometer from the sample. Quickly remove the oil from the gallery until its level is below the overflow rim. This is done by placing the tip of the withdrawal tube (Fig. 6B) at one point in the gallery and applying suction. Do not touch the overflow rim with the withdrawal tube, or the effective head of the sample will be reduced.

(f) Place the receiving flask (Fig. 7B) where the stream of oil from the bottom of the viscometer will just strike the neck of the flask. The graduation mark on the flask shall be between 10 and 13 cm from the bottom of the viscometer tube.

(g) Snap the cork from the viscometer using the attached cord. *At the same instant start the timer.* Stop the timer the instant the bottom of the meniscus reaches the graduation mark. Record the efflux time in seconds.

Special Procedure for Waxy or Resinous Materials

50 (a) Steam-refined cylinder oils, black lubricating oils, residual fuel oils, and other materials which may possibly contain waxes or resins require special handling to obtain reproducible results. In order to control the possible anomalies in testing such material, the thermal history of the sample shall be controlled. This special procedure will aid in the control of wax crystals and lattice structure formations.

(b) Heat the filtered sample in a loosely stoppered container for 1 hr *in an oven maintained at 212 to 215 F.*

(c) Immediately pour the sample into the clean, stoppered viscometer without refiltering. Place a thermometer with support into the sample. Do not disturb the sample until it is 1 F above test temperature.

(d) When the sample reaches this temperature, stir it with the thermometer using a circular motion, at 30 to 50 rpm, until the temperature of the sample remains constant within 0.05 F of the test temperature during 1 min of continuous stirring.

(e) Complete the test as described in Par. 49 (e), (f), and (g).

(f) Viscosity determinations of these materials shall be completed within 1 hr after filling the viscometer with sample. Do not run repeat determinations on the same portion of these materials.

Calculation and Report

51.1 (a) Multiply the efflux time by the calibration factor for the viscometer used.

(b) Report the corrected time in seconds as the Saybolt Universal viscosity or Saybolt Furol viscosity of the oil at the temperature at which the test was made.

(c) Report the values below 200 sec to the nearest 0.1 sec. Report all values of 200 sec or higher to the nearest whole second.

Editorial Note

The following section on precision is under review and study by Section A of Research Division VII of Committee D 2 on Petroleum Products and Lubricants, and is published here as information only.

51.2 Precision. Results in different laboratories with different operators under referee or standardization conditions of testing should not differ by more than 0.5 per cent.

Appendix I

Saybolt Viscometer and Accessories

Viscometer

A1 The viscometer, illustrated in Fig. 6A, shall be constructed entirely of corrosion-resistant metal, conforming to dimensional requirements shown in Fig. 6A. The orifice tip, Universal or Furol, may be constructed as a replaceable unit in the viscometer. Provide a nut at the lower end of the viscometer for fastening it in the bath. Mount vertically in the bath and test the alignment with a spirit level on the plane of the gallery rim. Provide a cork or other suitable means to prevent the flow of sample until the start of the test; a small chain or cord may be attached to the cork to facilitate rapid removal.

Bath

A2 The bath serves both as a support to hold the viscometer in a vertical position as well as the container for the bath medium. Equip the bath with effective insulation and with an efficient stirring device. Provide the bath with a coil for heating and cooling and with the thermostatically controlled heaters capable of maintaining the bath within the functional precision given in Table 7. The heaters and coil should be located at least 3 in. from the viscometer. Provide a means for maintaining the bath medium at least $\frac{1}{4}$ in. above the overflow rim. The bath media are given in Table 7.

Appendix II

Viscosity Standards

A3 Viscosity standards are available from two sources. These standards may be used with equal confidence provided they are used immediately after opening and not stored for re-use as permanent viscosity standards.

API Viscosity Standards

A4 (a) American Petroleum Institute liquid viscosity standards having certified Saybolt viscosity values established by annual cooperative determinations of kinematic viscosity values. The kinematic viscosity values are converted to Saybolt Universal and Furol viscosity values by means of conversion tables given in ASTM Methods in D 446 and D 666, respectively.

(b) The "alpha" standard has a Saybolt Universal viscosity of approximately 300 sec at 110 F. The "beta" standard has a Saybolt Furol viscosity of approximately 90 sec at 122 F. Exact values are supplied with each sample.

Note 7. The API viscosity standards are available in 8-oz containers, priced $2.50 each, and in 1-qt containers, priced $5.00 each. Purchase orders should be addressed to Lacey Walker, Secretary, American Petroleum Institute, 50 West 50th Street, New York 20, N. Y. Remittance payable to the American Petroleum Institute, with the order, will expedite shipments. Shipment will be made by express, collect, to points within the United States. The American Petroleum Institute will not accept an order requiring shipment outside the United States. Purchasers outside the United States may order oils shipped to their designated agents in this country. The API will accept no further responsibility than delivery to such agents.

NBS Viscosity Standards

A5 National Bureau of Standards liquid viscosity standards having accurate values supplied with each sample. Standard SB has a Saybolt Universal viscosity of approximately 300 sec at 100 F. Standard SF has a Saybolt Furol viscosity of approximately 200 sec at 122 F.

Note 8. The NBS viscosity standards are available only as 1-pt samples at $5.00 per sample FOB Washington, D. C. Purchase orders should be addressed to the National Bureau of Standards, Washington 25, D. C.

Routine Calibrations

A6 The viscosity standards may also be used for routine calibrations at other temperatures as shown in Table 8.

Table 8 Saybolt Viscosity Standards
(Approximate Values)

Designation	Universal, Sec			Furol Sec
	At 100 F	At 130 F	At 210 F	At 122 F
Alpha	305	...	53	...
Beta	1810	...	128	91
Gamma	37	...	...	...
SB	300	...	...	...
SC	....	300	...	...
SF	....	...	...	200

Other Viscosity Standards

A7 Other reference liquids, suitable for routine calibrations, may be established by selecting stable oils covering the desired range and determining their viscosities in a viscometer calibrated with an API or NBS standard as described in Par. 48.

Standard Method for Calculating Viscosity Index

ASTM Designation: D 567-53

Adopted 1941; Revised 1953

This Method has been approved as "American Standard" by the American Standards Association

Scope

52 (a) The tables and equations included in this method provide a means for calculating the viscosity index of a petroleum product or lubricant from its viscosity at 100 and 210 F. This method provides tables for oils with viscosities at 210 F between the values of 2.0 and 75.0 centistokes (cs). Equations are provided for calculating basic values for oils having viscosities at 210 F below 2.0 cs or above 350 sec Saybolt Universal at 210 F.

(b) The kinematic viscosity values are based on the standard value for distilled water being 1.0038 cs at 68 F.

Definition

53 Viscosity Index. The viscosity index is an empirical number indicating the effect of change of temperature on the viscosity of an oil. A low viscosity index signifies relatively large change of viscosity with temperature.

Formulas and Tables

54 (a) Viscosity index is calculated from the following equations:

$$\text{V.I.} = \frac{L - U}{L - H} \times 100 \quad \ldots\ldots\ldots\ldots\ldots (1)$$

$$\text{V.I.} = \frac{L - U}{D} \times 100 \quad \ldots\ldots\ldots\ldots\ldots (2)$$

where:

U = viscosity at 100 F of the oil whose viscosity index is to be calculated,

L = viscosity at 100 F of an oil of 0 viscosity index having the same viscosity at 210 F as the oil whose viscosity index is to be calculated,

and

H = viscosity at 100 F of an oil of 100 viscosity index having the same viscosity at 210 F as the oil whose viscosity index is to be calculated. This value is not shown in the tables because its use is not necessary when D is known. If it is desirable to know H, it can be determined as follows $H = L - D$.

$D = L - H$

(b) Table 9 contains the basic kinematic viscosity values in centistokes for L and D which are defined above. The values shown in this table are the official basis for Viscosity Index system. Table 10 contains L and D values for Saybolt Universal viscosity and are included for convenient reference.

Note 1. Unnecessary interpolations can be avoided by use of expanded tables based on this method. ASTM Viscosity Tables for Kinematic Viscosity Conversions and Viscosity Index Calculations are published separately as *ASTM Special Technical Publication No. 43A.* Copies may be obtained from Society Headquarters, 1916 Race St., Philadelphia 3, Pa.

(c) If the kinematic viscosity of the oil at 210 F is below 2.0 cs at 210 F obtain the values for L and D from the following equations:

$$L = X(1.655 + 1.2665X) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

$$D = X(0.1725 + 0.34984X) \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

where:

X = kinematic viscosity at 210 F.

(d) If the Saybolt viscosity of the oil at 210 F is above 350 sec Saybolt Universal, then obtain the values for L and D from the following equations:

$$L = 0.216Y^2 + 12.07Y - 721.2 \quad \ldots\ldots\ldots\ldots (5)$$

$$D = 0.1752Y^2 - 0.498Y - 245.8 \quad \ldots\ldots\ldots\ldots (6)$$

where:

Y = Saybolt Universal viscosity at 210 F, in seconds.

Note 2. Tables containing "Viscosity Indices Calculated from Basic Kinematic Viscosity Values" are based upon the values shown in Table 9 and can be used to avoid necessary calculation. These tables are to be published separately and will be available early in 1954 from ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

Tables containing "Viscosity Indices Calculated from Saybolt Universal Viscosity Values" are based upon values shown in Table 10 and can be used to avoid necessary calculation. These tables are published separately as "ASTM Viscosity Index Tables," and can be obtained from ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

Calculation and Reporting

55 (a) Determine the viscosity of the oil at 100 F and 210 F.

Enter the appropriate Table 9 or 10 with the viscosity value at 210 F and obtain the values for L and D. Basic values for viscosities not listed, but which are within the range of the tables, may be obtained by linear interpolation.

(b) Calculate the basic values for kinematic viscosities below 2.0 cs at 210 F by use of Eq (3) and (4).

Calculate the L and D values for Saybolt viscosity above 350 sec Saybolt Universal at 210 F by use of Eq (5) and (6).

Apply the 100 F viscosity value and the L and D values to Eq (2) to obtain the Viscosity Index of the oil.

(c) Report the Viscosity Index to the nearest half number.

Examples:

(*1*) Given Kinematic Viscosity at 100 F = 82.50 cs
and Kinematic Viscosity at 210 F = 9.100 cs
From Table 9: $L = 138.18$
From Table 9: $D = 60.44$
Substituting in Eq. (*2*):

$$\frac{138.18 - 82.50}{60.44} \times 100 = 92.12$$

V.I. = 92.0

(*2*) Given Kinematic Viscosity at 100 F = 38.87 cs
and Kinematic Viscosity at 210 F = 5.860 cs
From Table 9 (by interpolation): $L = 59.81$
From Table 9 (by interpolation): $D = 20.59$
Substituting in Eq. (*2*):

$$\frac{59.81 - 38.87}{20.59} \times 100 = 101.7$$

V.I. = 101.5

(*3*) Given Kinematic Viscosity at 100 F = 5.538 cs
and Kinematic Viscosity at 210 F = 1.805 cs
From Eq. (*3*):
$L = 1.805(1.655 + 1.2665 \times 1.805) = 7.114$
From Eq. (*4*):
$D = 1.805(0.1725 + 0.34984 \times 1.805) = 1.451$
Substituting in Eq. (*2*):

$$\frac{7.114 - 5.538}{1.451} \times 100 = 108.61$$

V.I. = 108.5

(*4*) Given Saybolt Universal Viscosity at 100 F = 776 sec
and Saybolt Universal Viscosity at 210 F = 77.0 sec
From Table 10: $L = 1489$
From Table 10: $D = 755$
Substituting in Eq. (*2*):

$$\text{V.I.} = \frac{1489 - 776}{755} \times 100 = 94.5$$

(*5*) Given Saybolt Universal Viscosity at 100 F = 12,540 sec
and Saybolt Universal Viscosity at 210 F = 378
From Eq. (*5*):
$L = 0.216 \times 378^2 + 12.07 \times 378 - 721.2 = 34704$
From Eq. (*6*):
$D = 0.1752 \times 378^2 - 0.498 \times 378 - 245.8 = 24599$
Substituting in Eq. (*2*):

$$\frac{34704 - 12540}{24599} \times 100 = 90.10$$

V.I. = 90.0

Reproducibility of Results

56 The reproducibility of results depends upon the accuracy in determining viscosities at 100 F and 210 F. Assuming an error not greater than 0.2 per cent for determined Kinematic viscosities and 0.5 per cent for determined Saybolt Universal viscosities the results of duplicate determinations will not differ by more than the following amounts:

Viscosity at 210 F		Possible Deviations in Viscosity Index units			
		Oil of V.I. = 0		Oil of V.I. = 100	
Kinematic, cs	Saybolt Universal, sec	Saybolt Viscosity	Kinematic Viscosity	Saybolt Viscosity	Kinematic Viscosity
4.0	39.45	12	5	9	3
6.0	45.8	8.5	3.5	5	2
8.0	52.4	7	2.5	3	1.5
12.0	66.4	5.5	2.5	2.5	1.0
20.0	98.2	5.0	2.0	2.0	0.5

TABLE 9 BASIC VALUES OF *L* AND *D* FOR KINEMATIC VISCOSITY

Kinematic Viscosity at 210 F, cs	*L*	*D* (*L*−*H*)	Kinematic Viscosity at 210 F, cs	*L*	*D* (*L*−*H*)	Kinematic Viscosity at 210 F, cs	*L*	*D* (*L*−*H*)
2.00	8.376	1.745	**7.50**	96.80	39.03	**13.00**	257.32	125.96
2.10	9.061	1.905	**7.60**	99.23	40.26	**13.10**	260.81	127.95
2.20	9.771	2.073	**7.70**	101.69	41.51	**13.20**	264.36	129.97
2.30	10.507	2.249	**7.80**	104.21	42.80	**13.30**	267.94	132.01
2.40	11.267	2.429	**7.90**	106.67	44.05	**13.40**	271.47	134.03
2.50	12.053	2.617	**8.00**	109.16	45.32	**13.50**	275.04	136.08
2.60	12.865	2.814	**8.10**	111.75	46.66	**13.60**	278.61	138.13
2.70	13.702	3.017	**8.20**	114.38	48.01	**13.70**	282.21	140.20
2.80	14.563	3.225	**8.30**	116.98	49.36	**13.80**	285.85	142.29
2.90	15.451	3.442	**8.40**	119.63	50.75	**13.90**	289.45	144.36
3.00	16.364	3.667	**8.50**	122.24	52.08	**14.00**	293.07	146.45
3.10	17.302	3.898	**8.60**	124.85	53.44	**14.10**	296.75	148.58
3.20	18.265	4.135	**8.70**	127.48	54.82	**14.20**	300.45	150.73
3.30	19.254	4.379	**8.80**	130.14	56.22	**14.30**	304.11	152.85
3.40	20.268	4.630	**8.90**	132.85	57.64	**14.40**	307.78	154.98
3.50	21.307	4.889	**9.00**	135.51	59.04	**14.50**	311.51	157.15
3.60	22.373	5.156	**9.10**	138.18	60.44	**14.60**	315.26	159.33
3.70	23.462	5.428	**9.20**	140.88	61.87	**14.70**	318.97	161.49
3.80	24.578	5.708	**9.30**	143.63	63.33	**14.80**	322.75	163.70
3.90	25.718	5.994	**9.40**	146.33	64.76	**14.90**	326.54	165.92
4.00	26.89	6.30	**9.50**	149.04	66.20	**15.00**	330.34	168.14
4.10	28.14	6.69	**9.60**	151.76	67.65	**15.10**	334.26	170.44
4.20	29.45	7.12	**9.70**	154.53	69.13	**15.20**	338.19	172.75
4.30	30.83	7.60	**9.80**	157.34	70.64	**15.30**	342.09	175.05
4.40	32.29	8.14	**9.90**	160.09	72.10	**15.40**	345.89	177.24
4.50	33.85	8.75	**10.00**	162.89	73.59	**15.50**	349.87	179.59
4.60	35.49	9.43	**10.10**	165.78	75.17	**15.60**	353.97	182.06
4.70	37.21	10.17	**10.20**	168.71	76.76	**15.70**	357.92	184.39
4.80	39.00	10.95	**10.30**	171.59	78.32	**15.80**	361.94	186.77
4.90	40.85	11.78	**10.40**	174.51	79.90	**15.90**	365.97	189.16
5.00	42.74	12.63	**10.50**	177.48	81.52	**16.00**	370.02	191.56
5.10	44.68	13.52	**10.60**	180.39	83.10	**16.10**	374.19	194.04
5.20	46.64	14.42	**10.70**	183.33	84.70	**16.20**	378.37	196.53
5.30	48.62	15.34	**10.80**	186.30	86.32	**16.30**	382.52	198.99
5.40	50.61	16.27	**10.90**	189.30	87.95	**16.40**	386.68	201.47
5.50	52.61	17.21	**11.00**	192.30	89.61	**16.50**	390.91	204.02
5.60	54.61	18.15	**11.10**	195.36	91.29	**16.60**	395.15	206.56
5.70	56.61	19.09	**11.20**	198.47	93.00	**16.70**	399.35	209.07
5.80	58.61	20.03	**11.30**	201.62	94.74	**16.80**	403.63	211.64
5.90	60.61	20.97	**11.40**	204.71	96.45	**16.90**	407.92	214.22
6.00	62.61	21.91	**11.50**	207.82	98.17	**17.00**	412.22	216.81
6.10	64.61	22.85	**11.60**	210.95	99.90	**17.10**	416.60	219.45
6.20	66.61	23.79	**11.70**	214.12	101.67	**17.20**	421.04	222.12
6.30	68.61	24.72	**11.80**	217.32	103.44	**17.30**	425.50	224.82
6.40	70.62	25.66	**11.90**	220.48	105.21	**17.40**	429.92	227.49
6.50	72.66	26.61	**12.00**	223.68	106.99	**17.50**	434.36	230.18
6.60	74.77	27.60	**12.10**	226.99	108.84	**17.60**	438.81	232.88
6.70	76.99	28.67	**12.20**	230.33	110.71	**17.70**	443.33	235.61
6.80	79.31	29.82	**12.30**	233.63	112.57	**17.80**	447.87	238.38
6.90	81.73	31.03	**12.40**	236.95	114.44	**17.90**	452.37	241.11
7.00	84.22	32.31	**12.50**	240.31	116.33	**18.00**	456.9	243.9
7.10	86.77	33.69	**12.60**	243.70	118.24	**18.10**	461.5	246.7
7.20	89.36	35.12	**12.70**	247.06	120.14	**18.20**	466.1	249.5
7.30	91.93	36.55	**12.80**	250.46	122.07	**18.30**	470.6	252.2
7.40	94.37	37.80	**12.90**	253.89	124.01	**18.40**	475.2	255.0

(*Continued on next page*)

TABLE 9 BASIC VALUES OF L AND D FOR KINEMATIC VISCOSITY (*Continued*)

Kinematic Viscosity at 210 F, cs	L	D ($L-H$)	Kinematic Viscosity at 210 F, cs	L	D ($L-H$)	Kinematic Viscosity at 210 F, cs	L	D ($L-H$)
18.50	479.9	257.9	**28.00**	1021.9	605.8	**52.5**	3289.5	2196.7
18.60	484.5	260.7	**28.20**	1035.5	614.8	**53.0**	3348.9	2239.7
18.70	489.2	263.6	**28.40**	1047.8	623.0	**53.5**	3409.8	2283.8
18.80	493.9	266.5	**28.60**	1061.6	632.2	**54.0**	3470.6	2328.0
18.90	498.6	269.4	**28.80**	1075.4	641.4	**54.5**	3531.2	2372.0
19.00	503.3	272.3	**29.00**	1089.3	650.6	**55.0**	3592.1	2416.3
19.10	508.2	275.3	**29.20**	1103.3	660.1	**55.5**	3653.7	2461.1
19.20	513.1	278.4	**29.40**	1117.2	669.3	**56.0**	3715.7	2506.3
19.30	517.9	281.3	**29.60**	1131.4	678.8	**56.5**	3778.3	2551.9
19.40	522.8	284.4	**29.80**	1144.2	687.5	**57.0**	3841.8	2598.3
19.50	527.7	287.4	**30.0**	1159.2	697.5	**57.5**	3906.8	2645.8
19.60	532.6	290.4	**30.5**	1195.2	721.7	**58.0**	3971.6	2693.1
19.70	537.5	293.4	**31.0**	1230.2	745.3	**58.5**	4036.2	2740.4
19.80	542.5	296.5	**31.5**	1267.1	770.2	**59.0**	4101.2	2788.0
19.90	547.5	299.7	**32.0**	1304.6	795.6	**59.5**	4166.8	2836.1
20.00	552.4	302.7	**32.5**	1342.5	821.3	**60.0**	4233.2	2884.8
20.20	562.5	309.0	**33.0**	1381.0	847.5	**60.5**	4301.3	2934.8
20.40	572.7	315.4	**33.5**	1419.8	874.0	**61.0**	4369.1	2984.5
20.60	582.4	321.5	**34.0**	1459.2	901.0	**61.5**	4436.7	3034.3
20.80	593.3	328.3	**34.5**	1499.0	928.2	**62.0**	4504.7	3084.4
21.00	603.0	334.4	**35.0**	1539.3	955.9	**62.5**	4573.3	3134.9
21.20	614.0	341.3	**35.5**	1582.0	985.3	**63.0**	4642.8	3186.0
21.40	624.7	348.0	**36.0**	1623.3	1013.8	**63.5**	4713.9	3238.5
21.60	635.1	354.6	**36.5**	1665.0	1042.6	**64.0**	4784.8	3290.8
21.80	646.3	361.7	**37.0**	1707.3	1071.9	**64.5**	4855.4	3342.9
22.00	657.3	368.7	**37.5**	1749.9	1101.4	**65.0**	4926.3	3395.3
22.20	667.8	375.4	**38.0**	1793.1	1131.5	**65.5**	4997.9	3448.3
22.40	679.2	382.6	**38.5**	1838.7	1163.2	**66.0**	5070.6	3502.1
22.60	690.4	389.7	**39.0**	1882.9	1194.0	**66.5**	5144.7	3556.9
22.80	701.1	396.6	**39.5**	1927.5	1225.2	**67.0**	5218.6	3611.7
23.00	712.8	404.1	**40.0**	1972.7	1256.9	**67.5**	5292.2	3666.3
23.20	724.2	411.4	**40.5**	2018.4	1288.9	**68.0**	5366.1	3721.1
23.40	735.1	418.4	**41.0**	2064.6	1321.4	**68.5**	5440.7	3776.5
23.60	747.0	426.0	**41.5**	2112.3	1354.9	**69.0**	5516.4	3832.8
23.80	758.6	433.5	**42.0**	2159.9	1388.4	**69.5**	5593.6	3890.2
24.00	769.7	440.7	**42.5**	2207.5	1422.0	**70.0**	5670.5	3947.4
24.20	781.5	448.3	**43.0**	2255.7	1456.1	**70.5**	5747.1	4004.4
24.40	793.7	456.2	**43.5**	2305.4	1491.3	**71.0**	5824.8	4062.2
24.60	804.9	463.4	**44.0**	2355.6	1526.8	**71.5**	5904.1	4121.3
24.80	817.2	471.4	**44.5**	2407.4	1563.6	**72.0**	5983.0	4180.1
25.00	829.3	479.2	**45.0**	2459.1	1600.3	**72.5**	6061.6	4238.7
25.20	841.8	487.4	**45.5**	2510.7	1637.1	**73.0**	6141.4	4298.3
25.40	853.3	494.9	**46.0**	2562.7	1674.2	**73.5**	6222.6	4358.9
25.60	865.9	503.1	**46.5**	2615.3	1711.7	**74.0**	6303.5	4419.3
25.80	878.7	511.5	**47.0**	2668.6	1749.8	**74.5**	6384.1	4479.5
26.00	891.5	519.8	**47.5**	2723.4	1789.0	**75.0**	6464.7	4539.7
26.20	904.1	528.1	**48.0**	2778.2	1828.3			
26.40	917.1	536.6	**48.5**	2832.8	1867.4			
26.60	930.1	545.2	**49.0**	2887.7	1906.8			
26.80	943.1	553.7	**49.5**	2943.3	1946.8			
27.00	955.0	561.5	**50.0**	2999.3	1987.1			
27.20	968.3	570.3	**50.5**	3055.9	2027.9			
27.40	981.6	579.1	**51.0**	3113.3	2069.3			
27.60	994.9	588.0	**51.5**	3172.2	2111.8			
27.80	1008.4	596.9	**52.0**	3230.9	2154.3			

TABLE 10 VALUES OF *L* AND *D* FOR CALCULATING VISCOSITY INDEX FROM SAYBOLT UNIVERSAL VISCOSITY

Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)
40.0	137.9	30.8	**65.0**	976.1	462.1	**90.0**	2115	1129
40.5	147.3	34.2	**65.5**	996.2	473.3	**90.5**	2141	1145
41.0	157.9	38.6	**66.0**	1016.3	484.5	**91.0**	2166	1160
41.5	169.4	43.5	**66.5**	1036.7	495.9	**91.5**	2192	1176
42.0	181.7	49.0	**67.0**	1057.1	507.3	**92.0**	2217	1191
42.5	194.7	55.0	**67.5**	1077.7	518.8	**92.5**	2244	1208
43.0	208.4	61.5	**68.0**	1098.3	530.5	**93.0**	2270	1224
43.5	222.4	68.2	**68.5**	1119.2	542.2	**93.5**	2296	1240
44.0	236.5	75.0	**69.0**	1140.1	554.0	**94.0**	2322	1256
44.5	250.9	82.0	**69.5**	1161.1	565.9	**94.5**	2349	1272
45.0	265.1	88.8	**70.0**	1182	578	**95.0**	2375	1288
45.5	279.4	95.6	**70.5**	1204	590	**95.5**	2402	1305
46.0	293.9	102.6	**71.0**	1225	602	**96.0**	2428	1321
46.5	308.2	109.5	**71.5**	1247	615	**96.5**	2455	1337
47.0	322.4	116.2	**72.0**	1268	627	**97.0**	2481	1353
47.5	337.0	123.0	**72.5**	1290	639	**97.5**	2509	1371
48.0	352.3	130.3	**73.0**	1311	651	**98.0**	2536	1388
48.5	368.5	138.2	**73.5**	1333	664	**98.5**	2564	1406
49.0	386.2	147.2	**74.0**	1355	677	**99.0**	2591	1423
49.5	404.2	157.0	**74.5**	1377	689	**99.5**	2619	1440
50.0	422.0	166.9	**75.0**	1399	702	**100.0**	2646	1457
50.5	439.2	175.8	**75.5**	1422	715	**101.0**	2701	1491
51.0	456.1	184.4	**76.0**	1444	728	**102.0**	2757	1526
51.5	473.1	193.1	**76.5**	1468	743	**103.0**	2814	1562
52.0	490.5	202.1	**77.0**	1489	755	**104.0**	2870	1597
52.5	507.7	210.9	**77.5**	1512	768	**105.0**	2928	1634
53.0	525.2	219.9	**78.0**	1534	781	**106.0**	2985	1670
53.5	543.0	229.2	**78.5**	1557	794	**107.0**	3043	1706
54.0	560.5	238.3	**79.0**	1580	808	**108.0**	3102	1744
54.5	578.1	247.4	**79.5**	1604	822	**109.0**	3161	1782
55.0	596.0	256.8	**80.0**	1627	836	**110.0**	3220	1819
55.5	614.1	266.3	**80.5**	1651	850	**111.0**	3280	1858
56.0	632.3	275.9	**81.0**	1674	864	**112.0**	3340	1896
56.5	650.2	285.3	**81.5**	1698	878	**113.0**	3400	1934
57.0	668.6	295.1	**82.0**	1721	892	**114.0**	3462	1974
57.5	687.0	304.8	**82.5**	1745	906	**115.0**	3523	2013
58.0	705.5	314.7	**83.0**	1769	920	**116.0**	3585	2053
58.5	723.9	324.5	**83.5**	1793	934	**117.0**	3648	2094
59.0	742.8	334.6	**84.0**	1817	949	**118.0**	3711	2135
59.5	761.7	344.8	**84.5**	1841	963	**119.0**	3774	2176
60.0	780.6	355.0	**85.0**	1865	977	**120.0**	3838	2218
60.5	799.7	365.4	**85.5**	1890	992	**121.0**	3902	2259
61.0	818.9	375.8	**86.0**	1914	1007	**122.0**	3966	2301
61.5	838.1	386.2	**86.5**	1939	1022	**123.0**	4031	2343
62.0	857.4	396.8	**87.0**	1964	1037	**124.0**	4097	2387
62.5	876.9	407.4	**87.5**	1989	1052	**125.0**	4163	2430
63.0	896.5	418.1	**88.0**	2014	1067	**126.0**	4229	2473
63.5	916.2	429.0	**88.5**	2039	1082	**127.0**	4296	2517
64.0	936.2	440.1	**89.0**	2064	1098	**128.0**	4363	2561
64.5	955.9	450.9	**89.5**	2090	1114	**129.0**	4430	2605

(*Continued on next page*)

TABLE 10 VALUES OF *L* AND *D* FOR CALCULATING VISCOSITY INDEX FROM SAYBOLT UNIVERSAL VISCOSITY (*Continued*)

Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)
130	4498	2650	**180**	8450	5341	**230**	13481	8907
131	4567	2696	**181**	8540	5404	**231**	13593	8988
132	4636	2742	**182**	8630	5467	**232**	13705	9069
133	4705	2787	**183**	8721	5530	**233**	13818	9150
134	4775	2834	**184**	8813	5595	**234**	13930	9230
135	4845	2880	**185**	8904	5658	**235**	14044	9313
136	4915	2927	**186**	8997	5723	**236**	14158	9395
137	4986	2974	**187**	9089	5787	**237**	14272	9477
138	5058	3022	**188**	9182	5853	**238**	14387	9560
139	5130	3070	**189**	9276	5919	**239**	14502	9643
140	5202	3118	**190**	9370	5985	**240**	14617	9726
141	5275	3167	**191**	9464	6050	**241**	14733	9810
142	5348	3216	**192**	9559	6117	**242**	14850	9895
143	5422	3266	**193**	9654	6184	**243**	14966	9978
144	5496	3316	**194**	9750	6252	**244**	15084	10064
145	5570	3365	**195**	9846	6319	**245**	15201	10148
146	5645	3416	**196**	9942	6387	**246**	15319	10234
147	5721	3467	**197**	10039	6455	**247**	15438	10320
148	5796	3518	**198**	10137	6524	**248**	15557	10406
149	5873	3570	**199**	10235	6594	**249**	15676	10492
150	5949	8621	**200**	10333	6663	**250**	15796	10579
151	6026	3673	**201**	10431	6732	**251**	15917	10667
152	6104	3726	**202**	10531	6803	**252**	16037	10754
153	6182	3779	**203**	10630	6873	**253**	16158	10842
154	6260	3832	**204**	10730	6944	**254**	16280	10931
155	6339	3886	**205**	10831	7015	**255**	16402	11020
156	6418	3940	**206**	10931	7086	**256**	16524	11108
157	6498	3995	**207**	11033	7159	**257**	16647	11198
158	6578	4049	**208**	11134	7230	**258**	16771	11288
159	6659	4105	**209**	11237	7304	**259**	16894	11377
160	6740	4160	**210**	11339	7376	**260**	17019	11469
161	6821	4215	**211**	11442	7449	**261**	17143	11559
162	6903	4272	**212**	11546	7523	**262**	17268	11650
163	6985	4328	**213**	11649	7596	**263**	17394	11742
164	7068	4385	**214**	11754	7671	**264**	17520	11834
165	7151	4442	**215**	11858	7745	**265**	17646	11926
166	7234	4499	**216**	11964	7821	**266**	17773	12018
167	7318	4557	**217**	12069	7896	**267**	17900	12111
168	7403	4615	**218**	12175	7972	**268**	18028	12205
169	7488	4674	**219**	12282	8048	**269**	18156	12298
170	7573	4733	**220**	12389	8125	**270**	18284	12392
171	7659	4792	**221**	12496	8201	**271**	18413	12486
172	7745	4852	**222**	12604	8279	**272**	18542	12580
173	7832	4912	**223**	12712	8356	**273**	18672	12676
174	7919	4972	**224**	12820	8433	**274**	18802	12771
175	8006	5032	**225**	12930	8512	**275**	18933	12867
176	8094	5094	**226**	13039	8590	**276**	19064	12963
177	8182	5155	**227**	13149	8669	**277**	19196	13060
178	8271	5217	**228**	13259	8748	**278**	19328	13156
179	8360	5278	**229**	13370	8828	**279**	19460	13253

(*Concluded on next page*)

TABLE 10 VALUES OF *L* AND *D* FOR CALCULATING VISCOSITY INDEX FROM SAYBOLT UNIVERSAL VISCOSITY (*Concluded*)

Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)	Saybolt Universal Viscosity at 210 F, sec	*L*	*D* (*L*−*H*)
280	19593	13351	305	23054	15901	330	26784	18669
281	19726	13448	306	23198	16007	331	26939	18784
282	19860	13547	307	23342	16114	332	27094	18900
283	19994	13645	308	23487	16221	333	27250	19016
284	20128	13743	309	23632	16328	334	27406	19132
285	20263	13843	310	23778	16436	335	27563	19249
286	20399	13943	311	23924	16545	336	27720	19366
287	20535	14043	312	24071	16654	337	27877	19483
288	20671	14143	313	24218	16762	338	28035	19601
289	20808	14244	314	24366	16872	339	28193	19719
290	20945	14344	315	24513	16981	340	28352	19838
291	21082	14445	316	24662	17092	341	28511	19956
292	21220	14547	317	24808	17199	342	28671	20076
293	21359	14649	318	24960	17313	343	28831	20195
294	21498	14752	319	25110	17424	344	28991	20315
295	21637	14854	320	25260	17536	345	29152	20435
296	21777	14958	321	25410	17647	346	29314	20556
297	21917	15061	322	25561	17759	347	29475	20677
298	22057	15164	323	25712	17871	348	29638	20799
299	22198	15268	324	25864	17984	349	29800	20920
300	22340	15373	325	26017	18098	350	29963	21042
301	22482	15478	326	26169	18211			
302	22624	15583	327	26322	18325			
303	22767	15688	328	26476	18440			
304	22910	15794	329	26630	18554			

Standard Viscosity-Temperature Charts for Liquid Petroleum Products

ASTM Designation: D 341-43

Adopted 1939; Revised 1943

(Reapproved in 1949 without change)

These Charts have been approved as "American Standard" by the American Standards Association

Scope

57 The viscosity-temperature charts covered by this standard are a convenient means for ascertaining the viscosity of a petroleum oil at any temperature, within a limited range, provided viscosities at two temperatures are known. Conversely, the charts may be used to ascertain the temperature at which a desired viscosity is attained. (See Figs. 8 and 9.)

Precaution

58 The charts should be used only in that temperature range in which the petroleum oil is a liquid. A safe range is that between initial boiling point and cloud point. Few data are available for temperatures above 300 F; therefore, it is probable that in this range the charts will not give as accurate results as at the lower temperatures.

Description

59 (*a*) The charts are so constructed that for any given petroleum oil the viscosity-temperature points lie on a straight line. Five charts are available, as follows:[1]

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[1] The Viscosity-Temperature Charts are available as follows from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.:

Chart A. Saybolt Viscosity (20 by 16 in.): Single copy, 35 cents; pad of 25, $2.25; five pads, any assortment except Chart B, $8.50.
Chart B. Saybolt Viscosity, Abridged (8½ by 11 in.): Pad of 50, $2.75; three pads, $6.50.
Chart C. Kinematic Viscosity, High Range (20 by 16 in.): Single copy, 35 cents; pad of 25, $2.25; five pads, any assortment except Chart B, $8.50.
Chart D. Kinematic Viscosity, Low Range (20 by 20 in.): Single copy, 35 cents; pad of 25, $2.25; five pads, any assortment except Chart B, $8.50.
Chart E. Kinematic Viscosity, Low-Temperature Range (26 by 20 in.): Single copy, 35 cents; pad of 25, $2.25; five pads, any assortment except Chart B, $8.50.

Chart A. Saybolt Universal Viscosity:
  Viscosity: 33 to 100,000,000 Saybolt Universal seconds
  Temperature: —30 to +450 F
  Size: 20 by 16 in.

Chart B. Saybolt Universal Viscosity, Abridged:
  Viscosity: 33 to 100,000 Saybolt Universal seconds
  Temperature: —30 to +350 F
  Size: 8½ by 11 in.

Chart C. Kinematic Viscosity, High Range:
  Viscosity: 2 to 20,000,000 centistokes
  Temperature: —30 to +450 F
  Size: 20 by 16 in.

Chart D. Kinematic Viscosity, Low Range:
  Viscosity: 0.40 to 100 centistokes
  Temperature: —30 to +450 F
  Size: 20 by 20 in.

Chart E. Kinematic Viscosity, Low-Temperature Range:
  Viscosity: 2.0 to 20,000,000 centistokes
  Temperature: —100 to +450 F
  Size: 24 by 16 in.

(b) Charts A and B have Saybolt Universal viscosity lines only, whereas Charts C, D and E have kinematic viscosity lines only. Charts C, D and E are on the same scale so that they may be superimposed on one another over the range 2 to 100 centistokes to make one continuous chart from 0.40 to 20,000,000 centistokes. Chart A is also on the same scale. The ranges covered by Chart B are those most generally used in viscosity determinations and this portion of Chart A has been reproduced on a slightly reduced scale as a convenience for certain types of plotting where the accuracy of Chart A is not considered essential.

(c) Kinematic charts C and D may be modified easily for use at low temperatures by substituting the temperatures in column B in the following table for the temperatures in column A which are printed on the bottom of the charts. This modification is based on the mathematical formulas used in the development of the charts. This procedure does not change the scale, therefore the modified charts can be superimposed on the original charts in the overlapping temperature range. Chart E has been prepared according to this procedure and is an extension of Chart C down to —100 F.

Column A	Column B	Column A	Column B
Temperature Printed on Chart, F	Low Temperature to be Substituted for those in Column A, F	Temperature Printed on Chart, F	Low Temperature to be Substituted for those in Column A, F
+5	—150	+200	—20
+20	—140	+215	—10
+35	—130	+230	0
+50	—120	+245	+10
+65	—110	+260	+20
+80	—100	+275	+30
+95	—90	+290	+40
+110	—80	+305	+50
+125	—70	+320	+60
+140	—60	+335	+70
+155	—50	+350	+80
+170	—40	+365	+90
+185	—30	+380	+100

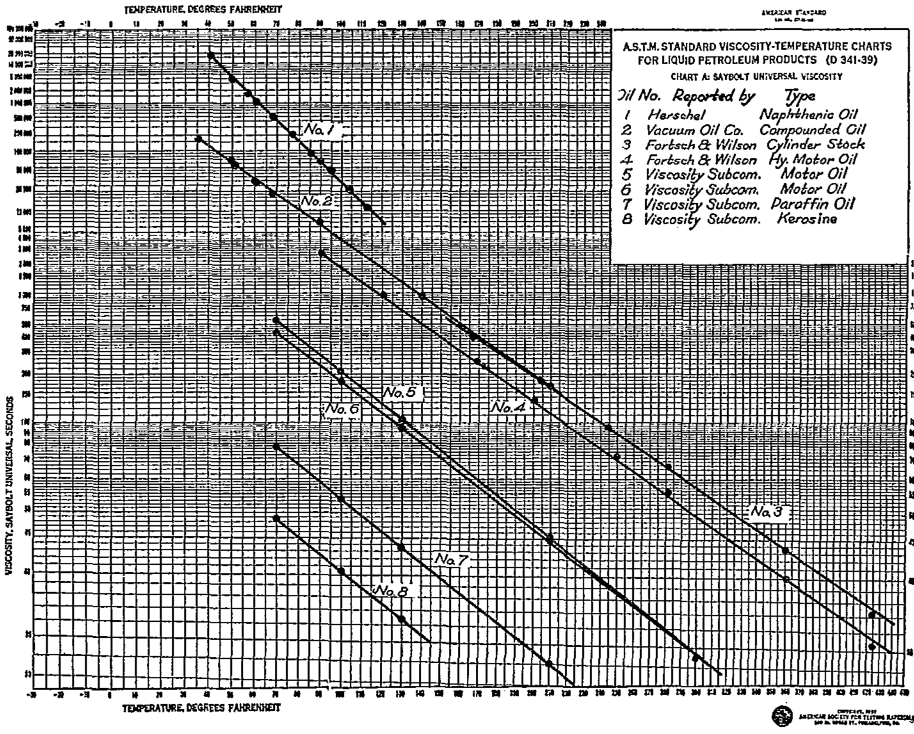

FIG. 8 FACSIMILE OF SAYBOLT UNIVERSAL VISCOSITY-TEMPERATURE CHART A ON WHICH SEVERAL TYPICAL EXPERIMENTAL CURVES HAVE BEEN PLOTTED (Chart Size: 20 by 16 in.).

Procedure

60 Plot the two known viscosity-temperature points[2] on the chart. With great care draw accurately a sharply defined, straight line through them. A point on this line, within the range defined in Par. 58, shows the viscosity at the corresponding desired temperature, and *vice versa.*

Extrapolation

61 Viscosity-temperature points on the extrapolated portion of the line, but still within the range defined in Par. 58, are satisfactory provided the viscosity-temperature line is located quite accurately. For purpose of extrapolation it is especially important that the two known viscosity-temperature points be far apart. If these two viscosity-temperature points are not sufficiently far apart, experimental errors in the viscosity determinations and in drawing the line may seriously affect the accuracy of extrapolated points, particularly if the difference between an extrapolated temperature and the nearest temperature of determination is greater than the difference between the two temperatures of determination. In extreme cases a viscosity determination at a third temperature is advisable.

Appendix

In addition to their primary purpose, these viscosity-temperature charts furnish a convenient means for estimating the viscosity at a given temperature of blends of petroleum liquids, provided viscosities of the component liquids at the given temperature are known. For this purpose the 0 F line may be assumed to represent 100 per cent of the light component and the 100 F line to represent 100 per cent of the heavy component. Plot the known viscosities of the two components on the proper lines at points corresponding to the values on the viscosity scale, and draw a straight line through them. Points on this line show viscosities at the given temperature of blends of the particular components, depending upon the proportion of the light and heavy component.

Example. As an example, estimate the viscosity of a blend of 15 per cent of an oil having a viscosity of 38 sec, Saybolt Universal at 100 F and 85 per cent of an oil having a viscosity of 153 sec, Saybolt Universal at 100 F. Draw a straight line between the 38-sec Saybolt point on the 0 F line and the 153-sec Saybolt point on the 100 F line. The printed figures between 0 and 100 F represent the percentage of the heavy component. The line crosses the 85 F line at a value corresponding to 105 sec. Thus the estimated viscosity of the blend is 105 sec, Saybolt Universal at 100 F. The experimental value was 108 sec.

Note. In case one of the components is motor gasoline, plot the gasoline point on the extension of the 0 F line 3.75 in. below its intersection with the 33-sec Saybolt Universal line and plot the point corresponding to the heavy component on the 100 F line. Points on the straight line drawn between these two points show viscosities of blends of motor gasoline and oil at the same temperature as the components.

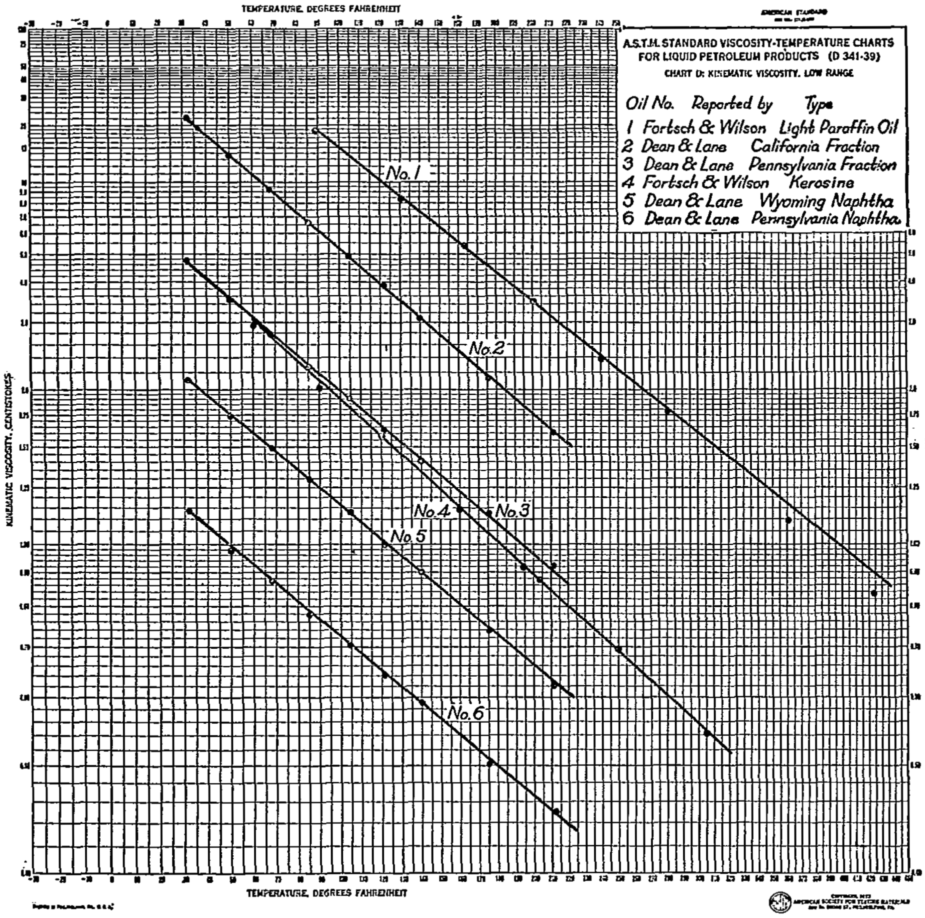

Fig. 9 Facsimile of Kinematic Viscosity-Temperature Chart D, Low Range on Which Several Typical Experimental Curves Have Been Plotted (Chart Size: 20 by 20 in.)

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[2] If viscosities are not known they should be determined in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (ASTM Designation: D 88).

VALUES FOR CONVERTING SAYBOLT UNIVERSAL VISCOSITY TO KINEMATIC VISCOSITY

Saybolt Universal Viscosity, Sec	Kinematic Viscosity, Centistokes, When Saybolt Universal Viscosity is at		
	100 F	130 F	210 F
34	2.4	2.4	2.3
36	3.0	3.0	2.9
38	3.7	3.6	3.6
40	4.3	4.3	4.2
42	4.9	4.9	4.8
44	5.6	5.5	5.4
46	6.2	6.1	6.1
48	6.8	6.8	6.7
50	7.4	7.4	7.3
52	8.0	8.0	7.9
54	8.6	8.6	8.5
56	9.2	9.2	9.1
58	9.8	9.7	9.6
60	10.3	10.3	10.2
62	10.9	10.9	10.8
64	11.5	11.5	11.4
66	12.0	12.0	11.9
68	12.6	12.5	12.4
70	13.1	13.1	13.0
72	13.6	13.6	13.5
74	14.2	14.1	14.0
76	14.7	14.6	14.6
78	15.2	15.2	15.1
80	15.7	15.7	15.6
82	16.2	16.2	16.1
84	16.7	16.7	16.6
86	17.2	17.2	17.1
88	17.7	17.7	17.5
90	18.2	18.1	18.0
92	18.7	18.6	18.5
94	19.2	19.1	19.0
96	19.7	19.6	19.5
98	20.1	20.0	20.0
100	20.6	20.5	20.5
105	21.8	21.7	21.4
110	22.9	22.9	22.8
115	24.1	24.1	23.9
120	25.3	25.2	25.1
125	26.4	26.4	26.2
130	27.5	27.5	27.3
135	28.7	28.6	28.4
140	29.8	29.7	29.6
145	30.9	30.9	30.7
150	32.1	32.0	31.8
155	33.2	33.1	32.9
160	34.3	34.2	34.2
165	35.4	35.3	35.2
170	36.5	36.4	36.3
175	37.6	37.5	37.4
180	38.7	38.7	38.5
185	39.9	39.8	39.6
190	41.0	40.9	40.7
195	42.1	42.0	41.8
200	43.2	43.1	42.8
205	44.3	44.2	44.0
210	45.3	45.2	45.0
215	46.4	46.3	46.1
220	47.5	47.4	47.2
225	48.6	48.5	48.3
230	49.7	49.6	49.4
235	50.8	50.7	50.4
240	51.9	51.8	51.5
245	53.0	52.8	52.6
250	54.0	53.9	53.6
260	56.2	56.1	55.9
270	58.4	58.3	58.0
280	60.6	60.5	60.2
290	62.8	62.6	62.3
300	64.9	64.8	64.5
310	67.1	66.9	66.6
320	69.3	69.1	68.8
Over 320, centistokes = seconds ×	$\frac{1}{4.620}$	$\frac{1}{4.629}$	$\frac{1}{4.652}$

VALUES FOR CONVERTING SAYBOLT FUROL VISCOSITY AT 122 F TO KINEMATIC VISCOSITY

Saybolt Furol Viscosity, Sec	Kinematic Viscosity, Centistokes	Saybolt Furol Viscosity, Sec	Kinematic Viscosity, Centistokes
26	50.0	70	146.8
27	52.2	72	151.1
28	54.4	74	155.4
29	56.6	76	159.8
		78	164.2
30	58.9		
31	61.1	80	168.4
32	63.4	82	172.7
33	65.6	84	177.0
34	67.8	86	181.3
		88	185.6
35	70.0		
36	72.2	90	190.0
37	74.4	95	200.6
38	76.6	100	211.5
39	78.9	105	222.2
		110	232.8
40	81.1		
41	83.3	115	243.6
42	85.6	120	254.2
43	87.8	125	264.8
44	90.0	130	275.6
		135	286.4
45	92.2		
46	94.4	140	297.2
47	96.6	145	307.8
48	98.9	150	318.5
49	101.1	155	329.1
		160	339.8
50	103.3		
52	107.7	165	350.4
54	112.1	170	361.1
56	116.5	175	371.9
58	120.9	180	382.5
		185	392.1
60	125.2		
62	129.6	Over 185 centistokes $= \frac{\text{seconds}}{0.470}$	
64	134.0		
66	138.1		
68	142.4		

Heating Value

62 Inasmuch as the purpose in burning any fuel is that of producing energy in the form of heat or power, the heating value of a fuel oil is of paramount importance. Fortunately, the variations in heating value within the high and low range of a given grade of fuel oil are small, and variations from one grade to the next are not large. For general purposes, the heating value of fuel oils may be calculated as follows:

Distillate fuels—
High heat value, Btu per lb = 18,250 + (API Gravity × 40)
Residual fuels—
High heat value, Btu per lb = 17,690 + (API Gravity × 58)

Experience has shown that the heating values calculated in this way may be expected to agree with experimentally determined values within ±0.5 per cent of the Btu value determined.

When greater accuracy is required as, for example, for the determination of the thermal efficiency of a process, the heating value should be determined experimentally. For this purpose, the Standard Method of Test for Heat of Combustion of Liquids by Bomb Calorimeter, ASTM Designation: D 240-50, is adopted for this code.

Standard Method of Test for Heat of Combustion of Liquids by Bomb Calorimeter

ASTM Designation: D 240-50

Adopted 1927; Revised 1930, 1950

This Method has been approved as "American Standard" by the American Standards Association

Scope

63 This method describes procedures for the determination of heat of combustion of aviation gasoline and the thermal value of fuel oils by means of a bomb calorimeter.

Apparatus

64 The apparatus shall consist of the following:

(a) **Vessel.** A calorimeter vessel equipped with a vacuum jacket or a water jacket having a cover to protect it from air currents. When a water jacket is used, provision shall be made for continuous stirring by means of a mechanical stirring device.

(b) **Stirrer.** A suitable device for stirring the water in the calorimeter vessel at a uniform rate. It shall be capable of stirring the water thoroughly and at the same time not so rapidly as to cause appreciable heating of the water (Note 1). A motor-driven screw or turbine stirrer is recommended. The portion of the stirring device immersed in the calorimeter vessel shall be connected to the outside portion with material of low heat conductivity.

Note 1. When the temperature of the calorimeter vessel and its contents is in equilibrium with the atmosphere, continuous stirring for 10 min should *not* cause an increase in temperature of the calorimeter vessel of more than 0.01 C.

(c) **Bomb.** A calorimeter bomb having a capacity of not less than 300 ml. It shall be constructed so that liquids may be easily drained out and so that it will not leak during the test. The inner surface of the bomb shall be of platinum, gold, porcelain enamel, stainless steel, or similar material that will not be affected by the combustion process or products. Other materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and they shall not undergo any reaction which will affect the sulfur content of the liquids in the bomb.

(d) **Oil Cup.** An oil cup made of platinum, glazed silica, or other suitable material, with a capacity of not less than 2.5 nor more than 5.0 ml.

(e) **Firing Wire.** A firing wire of pure iron or platinum. If a platinum oil cup is used, the firing wire shall be of platinum; if a glazed silica cup is used, the firing wire may be either platinum or iron. If iron wire is used, it should be about No. 34 B. & S. gage.

(f) **Thermometer.** Mercurial, electrical resistance, or thermoelectric thermometers, for determining the rise in temperature of the calorimeter water. The thermometers shall be certified by the National Bureau of Standards and used with the corrections given on their certificates. Corrections shall also be made for the temperature of the emergent stem of mercurial thermometers, and for the "setting" of Beckmann thermometers. For accurate testing, either Beckmann or special calorimetric thermometers, graduated to 0.01 or 0.02 C shall be used. Such thermometers should be tapped lightly just before each reading to avoid errors caused by the sticking of the mercury meniscus, particularly when the temperature is falling. A convenient method is to mount a small electric buzzer directly on the top of the thermometer and connect it with a dry cell and a push button. The button should be pressed for a few seconds immediately before each reading.

(g) **Oxygen.** Oxygen under pressure, free from combustible material.

Procedure

65 (a) **Temperatures of Room and Jacket Water.** The room in which the calorimeter is located shall be free from drafts and sudden changes in temperature. In case the calorimeter vessel has a water jacket, it shall be filled with water within 2 or 3 C of room temperature.

(b) **Preparation of Bomb and Sample.** The firing wire shall be clean and so arranged that it will touch the oil or standard sample when the oil cup is placed in position in the bomb. If iron wire is used a piece not more than 10 cm (preferably 5 cm) in length shall be cut, coiled in a small spiral, and connected to the terminals, using, if necessary, a piece of platinum wire of somewhat larger diameter than the iron wire to make the connection.

About 5 ml of distilled water shall be placed in the bomb to saturate with water vapor the oxygen used for combustion.

A quantity of oil sufficient to give a temperature rise of from 3 to 4 C shall be placed in the weighed oil cup and the weight of the charge determined to the nearest 0.1 mg. The cup shall then be placed in the bomb and the bomb closed. It is important that the cover of the bomb be screwed on firmly, and that the bomb does not leak under pressure.

NOTE 2. Acceptable procedure for handling volatile liquids include those described in the following reports: Richards and Barry, *Journal,* American Chemical Society, Vol. 37, p. 993 (1915); Jessup, *Journal of Research,* National Bureau Standards, Vol. 18, p. 115 (1937); Proson and Rossini, *Journal of Research,* National Bureau of Standards, Vol. 27, p. 289 (1941); Dean, Williams and Fisher, *Industrial and Engineering Chemistry* Analytical Edition, Vol. 16, p. 182 (1944); LeTourneau and Matteson, *Analytical Chemistry,* Vol. 20, p. 663 (1948).

(c) **Addition of Oxygen.** Oxygen from the supply cylinder shall be admitted to the bomb slowly, in order to avoid blowing the oil from the cup, until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure, atm
300 to 350	40
350 to 400	35
400 to 450	30
450 to 500	27.5
Over 500	25

(d) **Addition of Water.** The required quantity of distilled water determined by measurement or by weighing to an accuracy of plus or minus 0.1 per cent shall be added to the calorimeter vessel.

(e) **Temperature Adjustments.** The initial temperature of the water in the calorimeter vessel shall be adjusted so that the final temperature after combustion will be not more than 1 and preferably about 0.5 C above that of the jacket. Under these conditions the total correction for heat gained from or lost to the surroundings will be small when the temperature rise is 3 to 4 C, and the effect of evaporation will also be small.

(f) **Firing Current.** The voltage of the electric current used in firing the charge shall not exceed 12 volts and the switch controlling the current shall remain closed not more than 2 sec. When iron wire is used, the use of an ammeter to indicate when the wire has burned through is recommended.

(g) **Combustion.** The loaded bomb shall be placed in the calorimeter vessel, the electric connections made, the cover placed in position, and the stirrer and thermometer so arranged as not to be in contact with the bomb or vessel. The stirrer shall then be started. After the temperature has become steady, which shall be not less than 2 min after the stirrer was started, the temperature shall be read at 1-min intervals for 5 min. The charge shall then be fired and the time noted. Observations of temperature and time shall then be made to enable the operator to fix the time when the temperature is equivalent to 60 per cent of the total rise (Note 3). When the temperature has reached a maximum and is falling gradually, a series of readings shall be taken at 1-min intervals for 5 min to determine the cooling rate. The bomb shall be removed from the vessel and the pressure released at a uniform rate, such that the operation shall require not less than 1 min. The bomb shall then be opened and the inside examined for traces of unburned oil and sooty deposit. If either is found, the determinations shall be discarded.

NOTE 3. In practice, the interval of time between firing and this 60 per cent value will be found nearly constant for a given calorimeter vessel with the usual quantities of oil samples, and it will be necessary only occasionally to determine the time for the 60 per cent rise of temperature.

(h) **Titration.** The interior of the bomb including the oil cup shall be rinsed with a fine jet of distilled water. The wash water, not to exceed 350 ml in volume, shall be collected in a beaker. Special care shall be taken not to lose, by splashing or otherwise, any of the wash water. The wash water shall be titrated with a standard alkali solution (1 ml of which is equivalent to 0.02173 g of HNO_3 or to 5 cal) using methyl orange or methyl red as an indicator, to determine the quantity of acid.

Water Equivalent of Calorimeter

66 The water equivalent of the calorimeter shall be determined by the combustion of standard samples obtained from the National Bureau of Standards following the procedure described by the Bureau. Not less than five combustions of each of two of the standard materials (sucrose, naphthalene, and benzoic acid) shall be made. The conditions under which the combustions of the standard samples are made, including the quantity of water, oxygen, and firing wire, and the method of correcting for radiation, shall be the same as for the combustions of oil samples.

Radiation Correction

67 The rise in temperature of the calorimeter water shall be corrected for loss and gain of heat as follows:

$$t = t_c - t_a - r_1(b - a) + r_2(c - b)$$

where:

t = corrected temperature rise in deg C
a = time of firing
b = time when rise of temperature has reached six-tenths of total amount
c = time when temperature has reached a maximum after firing
t_a = temperature at time a, corrected for thermometer errors
t_c = temperature at time c, corrected for thermometer errors
r_1 = rate of temperature rise in deg C per minute for 5 min before firing
r_2 = rate of temperature drop in deg C per minute for 5 min after reaching the maximum temperature, and
b-a and c-b = time intervals expressed in minutes.

Calculation

68 (a) Calculate the *high* heat of combustion of the sample by means of the following equation:

High Heat of Combustion, Btu per lb =

$$\frac{1.8(tw - e_1 - e_2 - e_3)}{g} + 1.6(t_c - 25)$$

where:

t = corrected temperature rise, deg C, as calculated in Par. 67
t_c = maximum temperature, deg C, reached after firing corrected for thermometer errors
w = water equivalent of calorimeter
e_1 = corrections, calories, of nitric acid formed (230 cal per g)
e_2 = corrections, calories, of sulfur content (1300 cal per g) for differences in heats of formation of sulfur dioxide and aqueous sulfuric acid (see Note 4)
e_3 = corrections, calories, for iron firing wire (1600 cal per g), and
g = weight of sample in grams.

NOTE 4. This represents an additional correction as sulfuric acid has been titrated and calculated as nitric acid.

(b) *Report the high heat of combustion of liquid fuel as calculated in Paragraph (a).*

(c) *Report the low heat of combustion of liquid fuel, calculated from the high heat of combustion by the following equation:*

Low heat of combustion, Btu per lb =
High heat of combustion —1030 $(H \times 9)$
where H = *total lb hydrogen per lb oil.*

NOTE: *The low calorific value (low heat of combustion) is calculated from the high calorific value (high heat of combustion) at 68 F (20 C) by making a deduction of 1030 Btu per lb (572 cal per g) of water derived from unit quantity of fuel, including both the water originally present as moisture and that formed by combustion. The deduction is not equal to the latent heat of vaporization of water (1055 Btu per lb at 68 F (20 C)) because the calculation is made to reduce from high value at constant volume to low value at constant pressure, for which the appropriate factor under these conditions is 1030 Btu per lb.*

Precision

69 Results should not differ from the mean by more than the following amounts:

Repeatability (Same Operator and Apparatus), per cent of mean	**Reproducibility** (Different Operator and Apparatus), per cent of mean
High or *Low* Heat of Combustion, Btu per lb 0.3	*High* or *Low* Heat of Combustion, Btu per lb 0.5

Carbon, Hydrogen, and Sulfur Contents

70 For the calculation of the heat losses in the waste gases from any process in which fuel oil is burned, a knowledge of the carbon and hydrogen contents is required. Also, for the calculation of the low heating value of a fuel oil, a knowledge of the hydrogen content is necessary. For the determination of these values, the applicable sections of Standard Methods of Sampling and Analysis of Coal and Coke, ASTM Designation: D 271-48, which is also a part of the Test Code for Solid Fuels, PTC 3.2—1954, are adopted for this code. (See Pars. 71-73, inclusive.)

The total sulfur content of a fuel oil is of importance as a measure of difficulty that may be expected from corrosion by sulfur acids if the waste gases are cooled below the dewpoint of the gas. Also, in certain industrial applications such as glass making, enameling, heat treating, and steel making, a high content of sulfur may be detrimental to the quality of the product. For the purpose of this code, the Standard Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method, ASTM Designation: D 129-52, Tentative Method of Test for Sulfur in Petroleum Products by the Lamp Gravimetric Method, ASTM Designation: D 90-55T are adopted. For information only for those who may find it useful for routine control purposes, a method for the volumetric determination of sulfur is presented.

Corrosion of certain parts of a system handling fuel oil, such as copper and brass pump seals, diaphragms, and pressure-regulating valve bellows, has been found to be related, not to the total sulfur content of the oil but to the content of elemental sulfur or other corrosive sulfur compounds. For the determination of the presence of these compounds, the Tentative Method of Test for Copper Corrosion by Petroleum Products (Copper Strip Test), ASTM Designation: 130-56, is adopted for this code.

Carbon and Hydrogen

Method with Electrically Heated Combustion Furnace

71 *The determination of carbon and of hydrogen shall be made with a weighed quantity of the sample in a combustion furnace of the Heraeus type.*[1] *The products of combustion shall be thoroughly oxidized by passing them over red-hot CuO and* $PbCrO_4$*, and shall be fixed by absorbing the water in a weighed Marchand tube filled with granular* $CaCl_2$ *and by absorbing the* CO_2 *in a Liebig bulb containing KOH (30 per cent).*

Apparatus

72 *The apparatus used shall consist of a purifying train in duplicate, a combustion tube in the furnace, and an absorption train.*

(a) ***Purifying Train.*** *The purifying train shall consist of the following purifying reagents, arranged in order of passage of air or oxygen through them:* H_2SO_4 *KOH solution, soda lime, and granular* $CaCl_2$*. One of the trains is for air and one for oxygen. In the* H_2SO_4 *and KOH scrubbing bottles, the air or the oxygen shall be made to bubble through about 5 mm of the purifying reagent. Both purifying trains shall be connected to the combustion tube by a Y-tube, the joint being made tight by a rubber stopper which has been heated at 150 C for 24 hr.*

(b) ***Combustion Tube.*** *The combustion tube shall be made of fused quartz. Its external diameter shall be about 21 mm and its total length 1000 mm. The first 300 mm of the tube shall be empty; following this empty space is a 10 mm long asbestos plug (acid-washed and ignited). The next 340 mm shall be filled with "wire" CuO; a second asbestos plug, 5 mm long, separates the copper oxide from 100 mm of fused* $PbCrO_4$*, which shall be held in place by another asbestos plug, 10 mm long and 200 mm from the end of the tube. The end of the tube shall be drawn out for rubber-tubing connection with the absorption train.*

(c) ***Absorption Train.*** *The absorption train shall consist of a Marchand tube filled with granular* $CaCl_2$ *to absorb moisture. The* $CaCl_2$ *should be saturated with* CO_2 *before using. The Marchand tube shall be followed by a Liebig bulb containing KOH solution (30 per cent), in which any possible impurities, as ferrous iron or nitrates, have been oxidized by a little* $KMnO_4$*. A guard tube containing granular* $CaCl_2$ *and soda lime shall be attached to the Liebig bulb to absorb any* CO_2 *escaping the KOH solution and any water evaporating from that solution.*

(d) ***Furnace.*** *The furnace as provided by the manufacturer does not include the small heater. This may be made in the laboratory by winding an alundum tube 120 mm in length with No. 20 nichrome II wire and enclosing it in a cylinder packed with magnesia-asbestos. The movable heaters have very thin platinum foil, weighing about 9 g in all, wound on porcelain tubes of 30 mm internal diameter. The larger one, which heats the CuO, is 350 mm in length, ana*

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[1] U.S. Bureau of Mines Technical Paper No. 8, p. 19 (1939).

the smaller one, which heats the sample in the boat, is 200 mm in length.

Procedure

73 (*a*) *Connect the train to an aspirator which draws the products of combustion through the entire train. A guard tube of* $CaCl_2$ *prevents moisture from running back into the absorption train. Maintain the suction constant by means of a Mariotte flask. The advantage of aspirating the gases through the train rather than forcing them through by pressure is that the pressure on the rubber connections is from the outside, so that gas-tight connections are more easily maintained than if the pressure is on the inside of the tube. Make the connections as tight as possible. The usual test for tightness is to start aspiration at the rate of about three bubbles of air per second through the potash bulb, and then to close the inlet for air and oxygen at the opposite end of the train; if there is no more than one bubble per minute in the potash bulb, the apparatus shall be considered tight.*

(*b*) *Before starting a determination when the train has been idle some hours, or after any changes in chemicals or connections, run a blank by aspirating about 1 liter of air through the train, which is heated in the same manner as if a determination on the sample were being made. If the Liebig bulb and the tube containing calcium chloride show a change in weight of less than 0.5 mg each, the apparatus is in proper condition for use.*

(*c*) *Use a porcelain or platinum boat provided with a glass weighing tube of suitable size, which is fitted with an accurately ground glass stopper. Weigh the tube and empty boat. Quickly place in the boat approximately 0.2 g of the air-dry sample (passing a 250-micron (No. 60) sieve and finer, or better, passing a 149-micron (No. 100) sieve if much free impurity is present). Place the boat at once in the weighing tube, which is quickly stoppered to prevent moisture change in the sample while weighing, and transfer to the furnace. Connect the absorption tubes and transfer the boat and sample from the weighing tube to the combustion tube, which should be cool for the first 300 mm. The* CuO *should be red hot and the* $PbCrO_4$ *at a dull-red heat. Transfer the boat from the weighing tube to the combustion tube as rapidly as possible.*

(*d*) *As soon as the boat is in place near the asbestos plug at the beginning of the copper oxide, insert the stopper connecting with the purifying train and start the aspiration with pure oxygen gas at the rate of three bubbles per second. Move small furnace to within not less than 30 mm of the rubber stopper, then switch it on and continue the aspiration carefully until practically all the moisture is expelled from the sample. Then increase the heat and advance the furnace very gradually until the volatile matter has been driven off. In driving off the volatile matter, the heat shall be applied gradually in order to prevent a too rapid evolution of gas and tar, which may either escape complete combustion or may be driven back into the purifying train. Increase the heat slowly until the sample is ignited, then the temperature may be increased rapidly, but care should be taken not to damage the combustion tube.*

(*e*) *Any moisture collecting in the end of the combustion tube or in the rubber connection joining it to the* $CaCl_2$ *tube may be driven over into the* $CaCl_2$ *tube by carefully warming with a piece of hot tile. Continue the aspiration with oxygen for 2 min after the sample ceases to glow, then turn off the heat and aspirate about 1200 cu cm of air. Disconnect the absorption bulbs, wipe with a clean cloth, and allow to cool to the temperature of the balance room before weighing.*

(*f*) **Calculation.** *Calculate the percentages of hydrogen and carbon as follows:*

$$\text{Hydrogen, per cent} = \frac{11.19 \times (\text{increase in wt of } CaCl_2 \text{ tube})}{\text{wt of sample}}$$

$$\text{Carbon, per cent} = \frac{27.27 \times (\text{increase in wt of KOH bulb})}{\text{wt of sample}}$$

(*g*) *Weigh the ash in the boat and carefully inspect it for any unburned carbon, which would destroy the value of the determination.*

NOTE: *Indicating "Drierite" (8 mesh) or "Anhydrone" may be used in place of* $CaCl_2$ *"Ascarite" (8-20 mesh) may be used in place of soda lime and 30 per cent KOH. When an absorbent is substituted in one unit of the apparatus, it must be substituted throughout the train. Stetser-Norton or Johnson absorption bulbs are satisfactory for containing these absorbents.*

Standard Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method

ASTM Designation: D 129-52

Adopted 1927; Revised 1933, 1934, 1939, 1944, 1949, 1950, 1951, 1952

This Method has been approved as "American Standard" by the American Standards Association

Scope

74 This method describes the procedure for the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases, that cannot be burned completely in a wick lamp. The method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat.

Outline of Method

75 (a) The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb washings, is determined gravimetrically as barium sulfate.

(b) **Safety.** *Strict adherence to all of the provisions prescribed hereafter insures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least ½-in. thick, or equivalent protection be provided against unforseeable contingencies.*

Apparatus and Materials

76 The apparatus and materials shall consist of the following:

(a) **Bomb,** having a capacity of not less than 300 ml, so constructed that it will not leak during the test and that quantitative recovery of the liquids from the bomb may be readily achieved. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the bomb.

(b) **Sample Cup,** platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 to 11 g.

(c) **Firing Wire,** platinum, approximately No. 26 B. & S. gage.

(d) **Caution.** *The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.*

(e) **Ignition Circuit,** capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire.

(f) **Cotton wicking or Nylon Sewing Thread,** white.

Reagents

77 Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

(a) **Barium Chloride Solution (85 g per l).** Dissolve 100 g of $BaCl_2 \cdot 2H_2O$ in distilled water and dilute to 1 liter.

(b) **Bromine Water (saturated).**

(c) **Hydrochloric Acid (sp gr 1.18).**

(d) **Oxygen,** free of combustible material and sulfur compounds, available at a pressure of 40 atmospheres.

(e) **Sodium Carbonate Solution (50 g per l).** Dissolve 135 g of $Na_2CO_3 \cdot 10H_2O$ or its equivalent in distilled water and dilute to 1 liter.

(f) **White Oil,** refined, sulfur-free.

Procedure

78 (a) **Preparation of Bomb and Sample.** Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 ml of Na_2CO_3 solution in the bomb (Note 1) and rotate the bomb in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of sample and white oil (Note 2) specified in the following table, weighing the sample to the nearest 0.2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

Caution. *Do not use more than 1 g total of sample and white oil or other sulfur-free combustible material.*

Sulfur Content, per cent	Weight of Sample, g	Weight of White Oil, g
5 or under	0.6 to 0.8	0.0
Over 5	0.3 to 0.4	0.3 to 0.4

NOTE 1. After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness should be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with Grit No. 2/0, or equivalent paper,* coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide* and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before using the bomb it should be washed with soap and water to remove oil or paste left from the polishing operation.

NOTE 2. If the sample is not readily miscible with white oil, some other nonvolatile, sulfur-free combustible diluent may be employed in place of the white oil. However, the combined weight of sample and nonvolatile diluent shall not exceed 1 g.

(b) **Addition of Oxygen.** Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely.

Caution. *Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.* Admit oxygen slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure,** Atm	Maximum Gage Pressure,** Atm
300 to 350	38	40
350 to 400	35	37
400 to 450	30	32
450 to 500	27	29

** The minimum pressures are specified to provide sufficient oxygen for complete combustion and the *maximum pressures represent a safety requirement.*

(c) **Combustion.** Immerse the bomb in a cold distilled-water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination and thoroughly clean the bomb before again putting it in use (Note 1).

(d) **Collection of Sulfur Solution.** Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of distilled water, and collect the washings in a 600-ml beaker having a mark to indicate 75 ml. Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to a suitable indicator. (The volume of the washings is normally in excess of 300 ml.) Place the sample cup in a 50 ml beaker and add 2 ml of HCl and enough distilled water just to cover the cup. Heat the contents of the beaker to just below its boiling point for 3 or 4 min and add to the beaker containing the bomb washings. Wash the sample cup and the 50-ml beaker thoroughly with distilled water. Remove any precipitate in the cup by means of a rubber policeman. Add the washings from the cup and the 50-ml beaker, and the precipitate, if any, to the bomb washings in the 600-ml beaker. Do not filter any of the washings, since filtering would remove any sulfur present as insoluble material.

(e) **Determination of Sulfur.** To the combined washings add 10 ml of saturated bromine water and evaporate to approximately 200 ml on a hot plate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 ml of $BaCl_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml as indicated by a mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow to cool for 1 hr before filtering. Filter the supernatant liquid through an ashless, quantitative filter paper. Wash the precipitate with water, first by decantation and then on the filter, until free from chloride. Transfer the paper and precipitate to a suitable weighed crucible and dry (Note 3) at low heat until the moisture has evaporated. Char the paper completely without igniting it, and

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* Emery Polishing Paper Grit No. 2/0 may be purchased from the Behr-Manning Co., Troy, N. Y. Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, N. J.

finally ignite at a bright red heat until the residue is white in color. After ignition is complete, allow the crucible to cool to room temperature, and weigh.

NOTE 3. A satisfactory means of drying, charring, and igniting the paper and precipitate is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition usually will occur at the desired rate.

(f) **Blank.** Make a blank determination on 0.3 to 0.4 g of white oil by following the normal procedure, omitting the sample. Repeat this blank whenever new reagents or white oils are used (Note 4).

NOTE 4. This procedure measures the sulfur in the white oil and the reagents as well as that introduced from contamination.

Calculation

79 Calculate the sulfur content of the sample as follows:

$$\text{Sulfur, per cent by weight} = \frac{(P - B)\ 13.73}{W}$$

where:
P = grams of $BaSO_4$ obtained from sample,
B = grams of $BaSO_4$ obtained from blank, and
W = grams of sample used.

Precision

80 Results should not differ from the mean by more than the following:

Sulfur Content, per cent	Repeatability, Same Operator and Apparatus	Reproducibility, Different Operator and Apparatus
0 to 0.5	0.015	0.03
Over 0.5 to 1.0	0.020	0.04
Over 1.0 to 2.0	0.025	0.05
Over 2.0 to 3.0	0.035	0.07
Over 3.0 to 4.0	0.06	0.09
Over 4.0 to 5.0	0.08	0.12
Over 5.0	0.10	0.14

Volumetric Method of Determination of Sulfur* for Routine Control Purposes

Scope

81 The following volumetric method for the determination of sulfur, while not accurately enough reproducible among different laboratories to be accepted as a standard, has been found acceptable in some laboratories for routine control purposes and is included here for information only.

Reagents

82 (a) **Barium Chloride.** Dissolve 3.8200 g of $BaCl_2 \cdot 2H_2O$ in 1000 ml of distilled water. The solution is standardized gravimetrically, 1 ml equivalent to approximately 0.0005 g S.

(b) **Phenolphthalein.** Dissolve 0.05 g of phenolphthalein in 100 ml of 50 per cent by volume neutral isopropyl alcohol.

(c) **Hydrochloric Acid.** Mix 500 ml of HCl (sp gr 1.19), and 500 ml of distilled water.

(d) **Hydrochloric Acid** (Approximately 0.005 N). Dilute 0.5 ml of HCl (sp gr 1.19) to 1 liter with distilled water.

(e) **Isopropyl Alcohol** (99 per cent).

(f) **Sodium Hydroxide** (Approximately 0.1 N). Dissolve 4.0 g of sodium hydroxide in 1000 ml of distilled water.

(g) **Saturated Bromine Water.** Add an excess of bromine to 1000 ml of distilled water.

(h) **Ammonium Hydroxide** (sp gr 0.90).

(i) **Tetrahydroxyquinone.**

(j) **Standard Sulfate Solution for Routine Control of Method.** Dissolve 1.0871 g of K_2SO_4, which has been dried at 110 C, in distilled water and dilute to 2000 ml. 1 ml is equivalent to 0.1 mg S.

Procedure

83 (a) **Ignition.** Sulfur is determined in the ashings from the oxygen-bomb calorimeter following the calorimetric determination. The procedure through titration of the washings shall be as specified in Pars. 63, 64, and 65.

(b) **Titration for Sulfur.** To the washings after titration for "acid correction," add 1 ml of NH_4OH (sp gr 0.90), heat the solution to boiling, and filter through qualitative filter paper. Wash the residue and filter paper thoroughly five or six times with hot distilled water. To the filtrate and washings, amounting to about 250 ml, add 1 ml of saturated bromine water and sufficient HCl to make it slightly acid. Boil the solution to expel the excess bromine. Evaporate to a volume of approximately 75 ml and cool to below 35 C. Add 2 or 3 drops of phenolphthalein indicator and then add 0.1 N sodium hydroxide until the solution is faintly pink. Transfer the solution to a 100 ml volumetric flask and dilute to 100 ml with distilled water. Pipette a 25-ml aliquot into a 250-ml Erlenmeyer flask and neutralize by adding 0.005 N hydrochloric acid until the pink color just disappears. Add 25 ml of isopropyl alcohol and 0.5 g of tetrahydroxyquinone. Swirl the flask until the indicator is dissolved and titrate slowly with standard barium chloride solution until a permanent rose color is imparted to the mixture. The titration is best accomplished when performed over a white surface.

(c) **Blanks and Corrections.** In all cases a correction must be applied by running a blank exactly as described above, using the same amount of all reagents that were employed in the regular determination.

(d) **Calculation.** Calculate the sulfur content as follows:

$$\text{Sulfur, per cent} = \frac{(\text{Ml of standard barium chloride} - \text{Ml blank}) \times 0.2}{\text{Weight of sample}}$$

(e) **Routine Control of Method.** When this sulfur determination is used routinely, the standard sulfate solution may be titrated at regular intervals of time to control these details: (1) Ability of the operator to reproduce the end point of the titration, (2) Maintenance of proper strength of standard barium chloride solution, and (3) Maintenance of other reagents without excessive interferences. Procedure: Transfer 25 ml of standard sulfate solution to a 250-ml Erlenmeyer flask. Add 25 ml of isopropyl alcohol and 0.5 g of tetrahydroxyquinone. Titrate with standard barium chloride solution to same end point as above. Determine blank correction on reagents as above. Titration minus blank correction shall be 5.0 ml.

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* Industrial and Engineering Chemistry, Analytical Edition, vol. 14, 1942. "Volumetric Determination of Sulfur in Coal and Coke," S. S. Tomkins, pp. 141-145.

Tentative Method of Test for Sulfur in Petroleum Products by the Lamp-Gravimetric Method

ASTM Designation: D 90-55T

Issued 1921; Revised 1924, 1926, 1929, 1930, 1934, 1941, 1946, 1947, 1950, 1955

Scope

84 (a) This method describes procedures for determination of total sulfur in concentrations above 0.002 per cent by weight in liquid petroleum products (Note 1).

NOTE 1. Results obtained by this method, where applicable, are equivalent to those obtained by ASTM Method D 1266, Test for Sulfur in Petroleum Products and Liquified Petroleum Gases by the CO_2-O_2 Lamp Method, within the limits of precision and accuracy of the two methods.

(b) The direct burning procedure (Par. 89) is applicable to such materials as gasoline, kerosine, naphtha, and other liquids that can be burned completely and without smoking in a wick lamp. The blending procedure (Par. 70) is applicable to aromatics, gas oils, and distillate fuel oils, naphthenic acids, alkyl phenols, and many other materials that cannot be burned satisfactorily by the direct burning procedure.

Outline of Method

85 The sample is burned in a closed system, using a wick-type lamp and an atmosphere of sulfur-free air. The oxides of sulfur produced are absorbed and oxidized to sulfuric acid by means of hydrogen peroxide and determined gravimetrically as barium sulfate.

Apparatus

86 *The apparatus shall consist of the following:*

(a) **Chimneys, Absorbers, Spray Traps, and Lamps** (Fig. 10), as specified in the Appendix.

(b) **Cotton Wicking.** Clean, unused, uniform, two-strand twisted cotton wicking of good quality, weighing 5 to 6 mg per cm per strand.

(c) **Vacuum System.** A vacuum manifold equipped with a vacuum regulating device, valves, etc. A convenient arrangement is shown in Fig. 11, but any other similar system may be used. The system shall be connected to a vacuum pump of sufficient capacity to permit a steady gas flow of 3 liters per min through each absorber and to maintain a constant manifold pressure of approximately 40 cm of water below atmospheric.

(d) **Air Purifying System.** A device to supply purified air to the burner manifold at a nearly constant pressure of approximately 20 cm of water and to the chimney manifold at a pressure of 1 to 2 cm of water. A convenient arrangement is shown in Fig. 12, but any other similar system may be used. The tubing which connects the chimneys to the manifold shall have an internal diameter not smaller than 0.25 in. (0.63 cm) in order to prevent unnecessary restriction of gas flow.

Reagents and Materials

87 Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. References to water shall be understood to mean distilled water.

(a) **Barium Chloride Solution (85 g per liter).** Dissolve 100 g of $BaCl_2 \cdot 2H_2O$ in water and dilute to 1 liter.

(b) **Diluent.** The diluent used shall have a sulfur content less than 0.001 per cent, be completely miscible with the sample to be analyzed, and permit burning at a moderate rate without smoking. Normal heptane, *iso*octane, and absolute ethyl alcohol have been found suitable.

(c) **Hydrochloric Acid** (sp gr 1.18).

(d) **Hydrogen Peroxide** (30 per cent).

(e) **Hydrogen Peroxide** (3 per cent). Dilute H_2O_2 (30 per cent) of low sulfur content with water. Store in a dark-colored glass-stoppered bottle.

(f) **Sodium Hydroxide Solution (100 g per liter).** Dissolve 100 g of technical grade NaOH pellets in distilled water, and dilute to 1 liter.

(g) **Sulfuric Acid (1:16).** Mix 60 ml of H_2SO_4 (sp gr 1.84) with 960 ml of water.

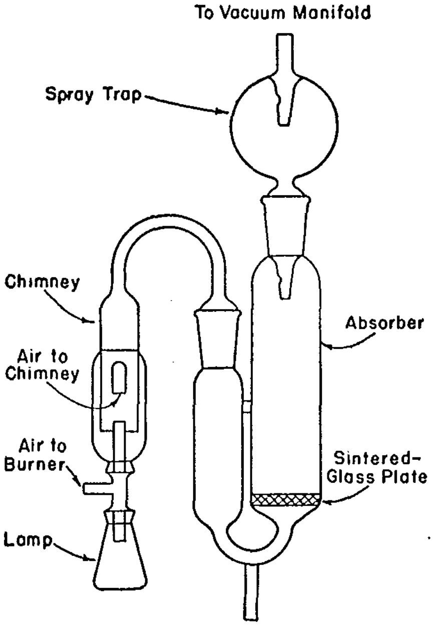

FIG. 10 DIAGRAMMATIC SKETCH OF COMBUSTION AND ABSORPTION APPARATUS FOR SULFUR DETERMINATION

Preparation of Apparatus

88 (a) Place 300 to 400 ml of NaOH (100 g per liter) in the first scrubber (Fig. 12) and the same amount of H_2O_2-H_2SO_4 solution (300 ml of water, 30 ml of H_2SO_4 (1:16), and 30 ml of H_2O_2 (30 per cent)) in the second scrubber. For apparatus in daily use, replace these solutions twice each week or whenever the volume becomes less than two thirds of the original.

(b) When the apparatus is first assembled, charge the absorber with 30 ± 2 ml of water, and adjust the individual valves between the vacuum manifold and the spray traps so that approximately 3 liters of air per minute will be drawn through each absorber when the chimney outlets are open to the atmosphere, while maintaining the pressure in the vacuum manifold at approximately 40 cm of water below atmospheric. When all adjustments have been made, remove the water from the absorbers.

(c) Introduce 30 ± 2 ml of H_2O_2 (3 per cent) into the larger bulb of each absorber. In addition to one absorber for each test sample to be burned, prepare an extra absorber for use as a blank. Attach the spray traps and chimneys and connect them to their respective manifolds by means of rubber tubing. Close the chimney openings by means of corks.

(d) With the burner control valves closed, the valve to the vacuum regulator fully open, and the pressure in the vacuum manifold adjusted to approximately 40 cm of water below atmospheric, turn on the purified air. Adjust the chimney manifold control valve so that, at the required flow through the absorbers, only a small stream of gas escapes at the pressure regulator (Note 2), a small stream of air enters at the vacuum regulator, and the pressure in the chimney manifold is 1 to 2 cm of water. Minor adjustment of the vacuum regulator and vacuum control valve may be necessary to achieve this condition.

Note 2. It is convenient to balance the air flow system by regulating the pressure in the vacuum manifold. This is done by raising or lowering the air inlet tube in the vacuum regulator by sliding it in a rubber sleeve.

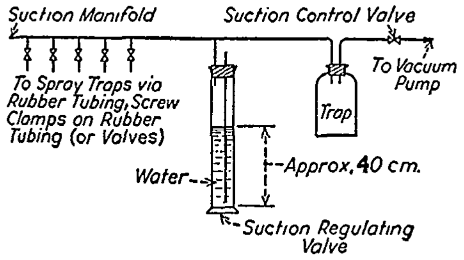

Fig. 11 Suction System for Sulfur Determination

(e) Thread the required number of burners with four 15-cm lengths of cotton wicking by doubling two 30-cm lengths, inserting the looped ends into the top of the inner tube of the burner, and drawing the wicking through by means of a metal hook. Trim the wick as close as possible to the top of the burner with a pair of sharp scissors; then draw the wick down until the trimmed edge is flush with the top of the burner (see Note 4). It is essential that thoroughly cleaned burners and new wicking be used for each test.

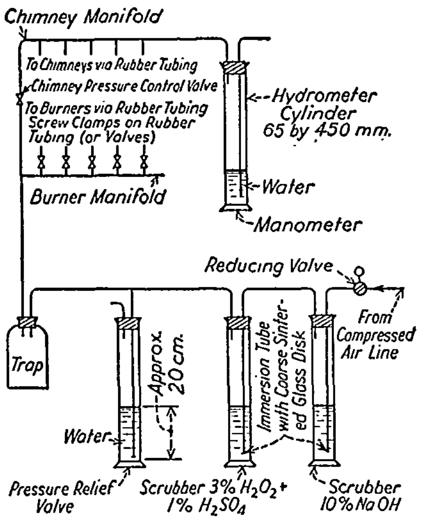

Fig. 12 Purified Air System for Sulfur Determination

Procedure for Direct Combustion

89 (a) By means of a pipet, introduce into the flask of each burner an approximate quantity of sample as indicated in Table 11. Stopper the flasks with clean numbered corks. Weigh each flask and its burner to 0.005 g (Note 3).

Table 11

Sulfur Content, per cent by weight	Sample Size	
	g	ml
0.5 and under	5 to 10	10
Over 0.5	3 to 5	5

Note 3. While the stoppered flasks and prepared burners may all be weighed separately, it is usually more convenient to place each flask and its burner on the balance pan and obtain the combined weight in a single weighing.

(b) Handling each lamp individually, insert the burner in the flask. As soon as the sample has risen by capillary action to the top of the wick, connect the side tube of the burner to the burner manifold by means of rubber tubing. Light the burner with a sulfur-free flame (such as an alcohol lamp) and insert into the chimney, pinching off the connection between the chimney and the chimney manifold during the insertion if the flame tends to be blown out. At the same time, adjust the air flow to the burner so that the flame is maintained at a point just below smoking and has a steady, symmetrical appearance. Continue in this manner until all lamps have been placed in the chimneys. Make any minor adjustment of the chimney control valve necessary to maintain the required pressure (see Par. 88 (d)). During the burning, and particularly during the latter stages when the flame becomes small, decrease the air supply to the burners in order to prevent extinction of the flames.

Note 4. Most types of samples burn with a luminous yellow flame, the size and shape of which is dependent upon the air flow to the burner, the volatility of the material, the tightness of the fit of the wick in the burner tube, and the position of the top of the wick relative to the top of the burner. It is preferable that the latter two variables be fixed with relation to the first before burning is started so that the flame can be controlled by variation in rate of air flow. Highly volatile samples require a tight-fitting wick, the top of which may need to be several millimeters below the top of the burner. In extreme cases the flask may have to be cooled in ice during the burning. Less volatile materials require a more loosely fitting wick and may require warming. In any case, it is essential that the flame burn smoothly and symmetrically and without jets in the inner cone or smoke on the outer fringes. Materials that cannot be burned properly under these conditions shall be burned by blending as described in Par. 90.

(c) When the burning of each sample is completed (as evidenced by the flame becoming small owing to depletion of the sample), remove the lamp from the chimney, extinguish the flame, shut off the air supply to the burner, and stopper the chimney opening. Immediately reweigh the lamp, burner, and numbered cork. When all combustions have been completed, turn off the air supply.

(d) Rinse the chimneys and spray traps with 30 to 40 ml of water, adding the rinsing to the absorbers, and proceed as directed in Par. 91. When the sample burned contains tetraethyllead, use hot water to rinse the chimneys.

(e) **Blank.** Leave the chimney of the blank absorber (Par. 88 (c)) stoppered, and allow the purified air stream to pass through that absorber until all samples started at one time have finished burning. Analyze the absorber liquid as directed in Par. 91.

Procedure for Blending and Combustion

90 (a) Add 6 ml of sulfur-free diluent to each flask. Stopper the flasks with numbered corks and weigh to 0.005 g. By means of a pipet, introduce into the flask of each burner

an approximate quantity of sample as indicated in Table 12, swirl to mix thoroughly, and reweigh (Note 3).

TABLE 12

Sulfur Content, per cent by weight	Sample Size	
	g	ml
1 and under	3 to 5	5
Over 1	2 to 3	4

(b) Insert the burner and burn as described in Par. 89 (b). Remove each lamp from its chimney as the flame nears extinction and extinguish the flame. Add 2 ml of diluent, allowing the diluent to rinse down the walls of the flask. Burn the additional diluent, and repeat the addition of diluent and burning once more so that a total of 10 ml of diluent has been burned.

(c) After all lamps have completed burning, turn off the air supply. Rinse the chimneys and spray traps with 30 to 40 ml of water, adding the rinsings to the absorbers, and proceed as directed in Par. 91.

(d) **Blank.** Prepare a separate flask containing 10 ml of diluent for use as a diluent blank, and allow to burn while the samples are being burned. Analyze the absorber liquid as directed in Par. 91.

Analysis of Absorbent

91 (a) Transfer the absorber liquid to a 400-ml beaker. Rinse the absorber thoroughly with water, and add the rinsings to the beaker. Filter the solution to remove any

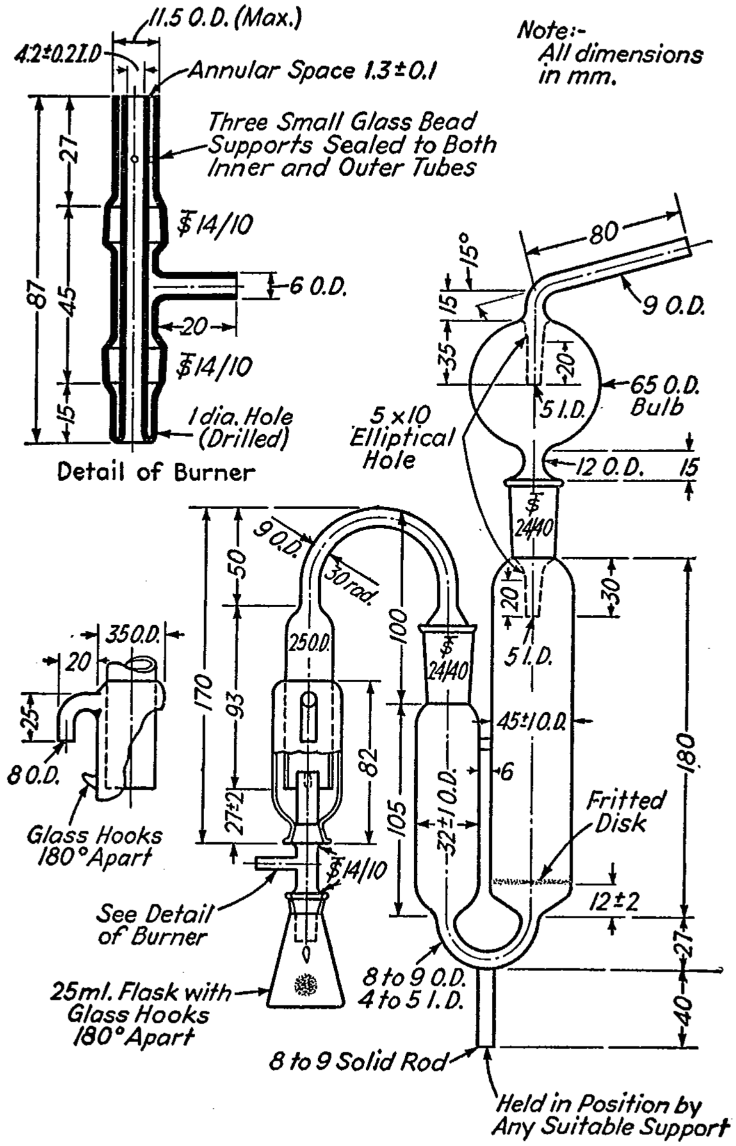

FIG. 13 DETAILED DRAWING OF COMBUSTION AND ABSORPTION APPARATUS FOR SULFUR DETERMINATION

NOTE. In the case of those dimensions for which no specific tolerances are designated above, the permissible variation is ±10 per cent to the nearest millimeter, provided, however, that in no case shall the deviation be greater than 5 mm.

foreign material, receiving the filtrate in a 400-ml beaker having a mark to indicate 75 ml. Add 2 ml of HCl, heat to boiling, and add 10 ml of $BaCl_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml as indicated by the mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow to cool 1 hr before filtering.

(b) Filter the supernatant liquid through a close-texture, ashless filter paper. Wash the precipitate with water, first by decantation and then on the filter paper, until free of chloride. Transfer the paper and precipitate to a suitable weighed crucible, and dry at low heat until the moisture has evaporated. Char the paper completely without igniting it, and finally ignite at a bright red heat until the precipitate is burned white (Note 5). After ignition is complete, allow the crucible to cool to room temperature and weigh.

NOTE 5. A satisfactory means of accomplishing these operations is to place the uncovered crucible containing the wet filter paper in a cold electric muffle furnace and turn on the current. Drying, charring, and ignition usually occur at the desired rate.

Calculation

92 Calculate the sulfur content as follows:

$$\text{Sulfur, per cent by weight} = \frac{(w - b) \times 13.73}{W}$$

where:

w = grams of $BaSO_4$ from sample,

b = grams of $BaSO_4$ from the corresponding blank (Note 6), and

W = grams of sample used.

NOTE 6. The determination should be discarded if the blank correction used in the calculation exceeds 1.5 mg of $BaSO_4$. Frequently, impure reagents are the cause of this difficulty.

Precision and Accuracy

93 (a) Results should not differ from the mean by more than the following amounts:

Sulfur Content, per cent by weight	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
0.002 to 0.25	0.001	0.002
Over 0.25	0.5 per cent of mean	1 per cent of mean

(b) Results should not differ from the true value by more than the following amounts:

Sulfur Content, per cent by weight	Accuracy
0.002 to 0.25	0.002
Over 0.25	1 per cent of true value

Appendix

Apparatus

A1 The apparatus shall consist of the following:

(a) **Lamp.** A lamp of chemically resistant glass, consisting of a 25-ml Erlenmeyer flask and a burner that conforms to the dimensions shown in Fig. 13. The burner consists of two concentric glass tubes, the external one having an arm, provided with standard-taper glass joints for connection with the flask and the chimney. The upper ends of both burner tubes shall be polished and shall have plane surfaces that are in the same horizontal plane. The burner shall have a 1-mm opening near its base to allow equalization of pressure between the chimney and the flask. When connected with the chimney, the lamp shall be held in position by rubber bands or metal springs stretched between glass hooks on the flask and chimney.

(b) **Chimney.** A chimney of chemically resistant glass conforming to the dimensions shown in Fig. 13, provided with standard-taper glass joints for connection with the burner and absorber.

(c) **Absorber.** An absorber of chemically resistant glass conforming to the dimensions shown in Fig. 13, provided with standard-tapered glass joints for connection with the chimney and spray trap. A fritted disk with average pore diameter from 150 to 200 microns shall be sealed in the larger of two bulbs of the absorber. The fritted disk should be of such a porosity that, when 50 ml of water is placed in the absorber and air is passed through at the rate of 3.0 liters per minute in the forward direction, the pressure differential between the two sides of the absorber is between 15 and 23 cm of water and the air is dispersed uniformly.

(d) **Spray Trap.** A spray trap of chemically resistant glass conforming to the dimensions shown in Fig. 13, provided with a standard-taper glass joint for connection with the absorber.

Standard Method of Test for Copper Corrosion by Petroleum Products

(Copper Strip Test)

ASTM Designation: D 130-56

Adopted 1956

This Method has been approved as "American Standard" by the American Standards Association

Scope

94 This method describes procedures for the detection of the corrosiveness to copper of aviation gasoline, automotive gasoline, farm tractor fuel, cleaners (Stoddard) solvent, diesel fuel, fuel oil (distillate) and certain other petroleum products.

NOTE 1. For copper strip tests applicable to electrical insulating oils, see ASTM Methods D 989 and D 1275; to greases, see Method D 1261; to mineral (petroleum) spirits, see Specifications D 235 and D 965; to industrial aromatic hydrocarbons, see Method D 849.

Outline of Method

95 A polished copper strip is immersed in a given quantity of sample and heated at a temperature and for a time characteristic of the material being tested. At the end of this period, the copper strip is removed, washed, and compared with the ASTM Copper Strip Corrosion Standards.

Apparatus

96 (a) **Low Pressure Bomb and Bath** (for test pressures up to 100 psi) constructed according to the dimensions and materials shown in Fig. 14. Alternative designs for the bomb cap and synthetic rubber gasket may be used provided that the internal dimensions of the bomb are the same as those shown in Fig. 14. Provide a 25- by 150-mm test tube as a liner for holding the sample. Provide a boiling water bath in which the bomb can be completely submerged vertically and capable of being maintained at 212 ± 2 F (100 ± 1 C).

(b) **Test Tube Bath, Oil or Water,** for maintaining a constant temperature of 122 ± 2 F (50 ± 1 C) or 212 ± 2 F (100 ± 1 C). Provide a support to hold a 25 by 150-mm test tube upright and submerged to a depth of about 100 mm.

(c) **Strip Vise** for holding the copper strip firmly without marring the edges while polishing. The types shown in Figs. 15 and 16 have been found convenient, but a small commercial vise can be adapted to this use by fitting it with stainless steel shields.

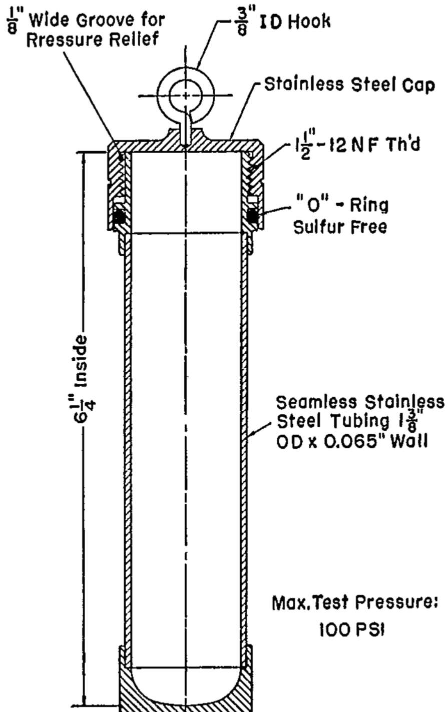

FIG. 14 COPPER STRIP CORROSION TEST BOMB

(d) **Test Tube,** flat, as shown in Fig. 17, for protecting corroded strips for close inspection.

(e) **Thermometer.** An ASTM Gravity Thermometer having a range of —5 to +215 F or —20 to +102 C, graduated in 0.5 F or 0.2 C subdivisions, and conforming to the requirements for thermometer 12 F or 12 C as prescribed in ASTM Standard Specifications E 1.

Materials

97 (a) **Wash Solvent,** use knock test grade *iso*octane.

(b) **Copper Strip,** 3 in. long, cut from $\frac{1}{2}$-in. wide, $\frac{1}{16}$ to $\frac{1}{8}$-in. thick, soft-temper, annealed bus bars for electrical conductors that conform to ASTM Specification B 187, for Copper Bus Bar, Rod, and Shapes and are drawn from electrolytic copper ingots or bars conforming to ASTM Specifications B 5, for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars. The strips may be used repeatedly but should be discarded when they show pitting or deep scratches that cannot be removed or when the surfaces become deformed.

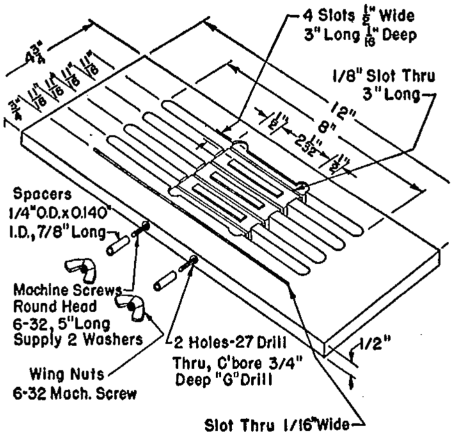

FIG. 15 COPPER STRIP HOLDER

(c) **Polishing Materials,** silicon-carbide or alumina grit paper of varying degrees of fineness including 240-grit silicon-carbide paper or cloth; also a supply of 150-mesh silicon-carbide grain and pharmaceutical grade absorbent cotton.

(d) **Copper Corrosion Standards,** consisting of reproductions of typical strips representing increasing degrees of tarnish and corrosion as listed under "Description" in Table 13. These reproductions are lithographically printed on an aluminum sheet and are encased in plastic for protection. Keep covered when not in use to avoid fading (see Note 2) due to light.

NOTE 2. Evidence of fading can be detected by comparing two different sets, one of which has preferably been carefully protected from light; the one which shows evidence of fading should be replaced. Alternately a $\frac{1}{2}$-in. opaque strip may be placed across the top of the colored strips and removed at intervals to inspect for fading.

Preparation of Strips

98 (a) **Surface Preparation.** Remove all surface blemishes from all six sides of the strip with silicon carbide or alumina grit paper of such degrees of fineness as are needed to accomplish the desired results efficiently (Note 3). Finish with 240-grit silicon-carbide paper, removing all marks that may have been made by other grades of paper used previously. Immerse the strip in knock test *iso*octane, from which it may be withdrawn immediately for final polishing or in which it may be stored for future use.

NOTE 3. As a practical manual procedure, for surface preparation, place a sheet of the paper on a flat surface, moisten it with kerosine or *iso*octane, and rub the strip against the paper with a rotary motion, protecting the strip from contact with the fingers with an ashless filter paper. Alternatively, the strip may be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth.

(b) **Final Polishing.** Remove a strip from the *iso*octane. Holding it in the fingers protected with ashless filter paper, polish first the ends and then the sides with the 150-mesh silicon-carbide grains picked up from a clean glass plate with a pad of absorbent cotton moistened with a drop of *iso*octane. Wipe vigorously with fresh pads of absorbent cotton and subsequently handle only with stainless steel forceps; *do not touch with the fingers.* Clamp in a vise and polish the main surfaces with silicon-carbide grains on absorbent cotton. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing the direction (see Note 4). Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

NOTE 4. It is important to polish the whole surface of the strip uniformly to obtain a uniformly stained strip. If the edges show wear (surface elliptical) they will likely show more corrosion than the center. The use of a vise will facilitate uniform polishing.

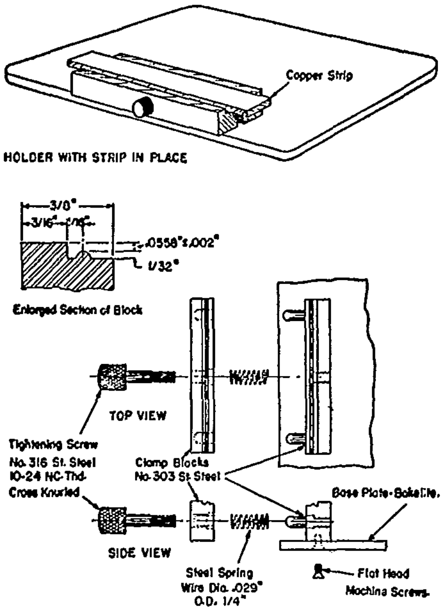

FIG. 16 OPTIONAL COPPER STRIP HOLDER

Sample

99 (a) It is particularly important that gasoline and fuel samples for this test be collected in clean dark bottles or cleaned tinned cans; to clean new tinned cans, rinse free of rosin or soldering flux with the sample to be tested. Care should be taken during sampling to protect the samples from exposure to direct or even diffused daylight (see Note 5). Make the test as soon as possible after sampling.

NOTE 5. Short wavelengths of light (violet and ultraviolet) have a marked effect on reducing, or removing entirely, the coloration tendency of the corrosive sulfur present in the sample on copper.

(b) If suspended water (haze) is observed in the sample (see Note 6), dry by filtering a sufficient volume of sample through a medium rapid qualitative filter (for example, Whatman No. 4), into the prescribed clean, dry test tube. Carry out this operation in a darkened room or under a light-protected shield.

NOTE 6. Contact of the copper strip with water before, during, or after the completion of the test run, will cause staining, making it difficult to evaluate the strips.

Procedure

100 (a) **Tests at 122 F (50 C) and 212 F (100 C) on Less Volatile Materials.** Place 30 ml of sample, completely clear and free of any suspended or entrained water (Note 6), into a chemically clean, dry 25- by 150-mm test tube and, within 1 min after completing the final polishing, slide the copper strip into the sample tube. Stopper with a vented cork and place in a bath maintained at the required temperature within ± 2 F (1 C). After 3 hr ± 5 min in the bath, examine the strip as described in Paragraph (c).

(b) **Tests at 212 F (100 C) For Aircraft Engine Fuels.** Place 30 ml of sample, completely clear and free of any suspended or entrained water (Note 6) into a chemically clean, dry 25- by 150-mm test tube, and within 1 min after completing the final polishing, slide the copper strip into the sample tube. Carefully slide the test tube into the sample bomb (Fig. 14) and screw the lid on tight. Completely immerse the bomb in a boiling water bath (212 ± 2 F or 100 ± 1 C). After 2 hr ± 5 min in the bath, withdraw the bomb, immerse for a few minutes in tap water. Open, withdraw the test tube and examine the strip as described in Paragraph (c).

(c) **Examination of Strip.** Empty the contents of the test tube into a 150-ml tall-form beaker, letting the strip slide in gently so as to avoid breaking the beaker. Immediately withdraw the strip with stainless steel forceps and immerse in knock test *iso*octane. Withdraw the strip at once, dry with quantitative filter paper (by blotting and not by wiping), and inspect for evidences of tarnishing or corrosion by comparison with the ASTM Copper Strip Corrosion Standards. Hold both the test strip and the standard strip in such a manner that light reflected from them at an angle of approximately 45 deg will be observed. In handling the test strip during the inspection and comparison, the danger of marking or staining can be avoided if it is inserted in a flat tube (see Fig. 77) which is stoppered with absorbent cotton.

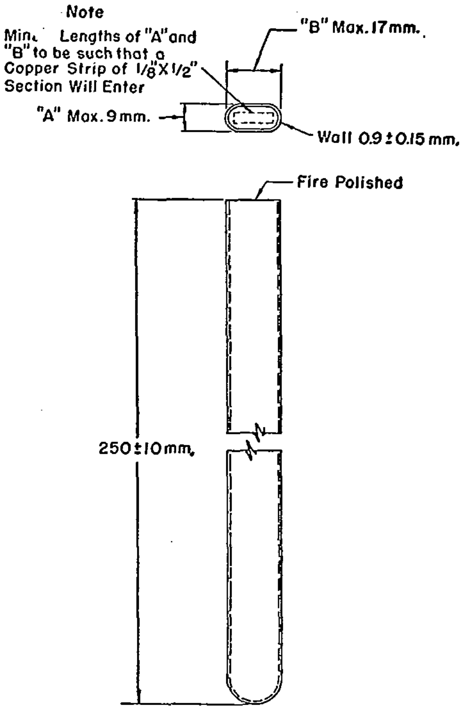

FIG. 17 FLAT GLASS TEST TUBE

Interpretation

101.1 Interpret the corrosiveness of the sample accordingly as the appearance of the test strip agrees with one of the strips of the ASTM Copper Corrosion Standards (Par. 97(d)).

(a) The freshly polished strip is included in the set of Corrosion Standards only as an indication of appearance of a properly polished strip before a test run; it is not possible to duplicate this appearance after a test even with a completely non-corrosive sample.

(b) When a strip is in the obvious transition state between that indicated by any two adjacent Corrosion Standard Strips, judge the sample by the more tarnished Standard Strip.

(c) A claret red strip in Classification 2 can be mistaken for a magenta overcast on brassy strip in Classification 3 if the brassy underlay of the latter is completely masked by a magenta overtone. To distinguish, immerse the strip in knock test *iso*octane; the former will appear as a dark orange strip while the latter will not change.

(d) To distinguish multicolored strips in Classifications 2 and 3, place a test strip in a 20- by 150-mm test tube and bring to a temperature of 600 to 700 F (315 to 355 C) in 4 to 6 min with the tube lying on a hot plate. Adjust to temperature by observing an ASTM high distillation thermometer in a second test tube. If the strip belongs in Classification 2, it will assume the color of a silvery and then a gold strip. If in Classification 3 it will take on the appearance of a transparent black, etc., described in Classification 4.

(e) Repeat the test if blemishes due to finger prints are observed, or due to spots from any particles or water droplets that may have touched the test strip during the digestion period. Repeat the test also if the sharp edges along the flat faces of the strip appear to be in a classification higher than the greater portion of the strip; in this case it is likely that the edges were burnished during polishing.

Report

101.2 Report the corrosiveness in accordance with one of the four numbered classifications listed in Table 13.

TABLE 13 ASTM COPPER STRIP CLASSIFICATIONS

Classification	Designation	Description[a]
1	......Slight Tarnish	Light orange, almost the same as a freshly polished strip Dark orange
2	......Moderate Tarnish	Claret red Lavender Multicolored with lavender blue and/or silver overlaid on claret red Silvery Brassy or Gold
3	......Dark Tarnish	Magenta overcast on brassy strip Multicolored with red and green showing (peacock), but no gray
4	......Corrosion	Transparent black, dark gray or brown with peacock green barely showing Graphite or lusterless black Glossy or jet black

[a] The ASTM Copper Corrosion Standard is made up of strips characteristic of these descriptions.

Ash Content

102 Although the ash content of distillate fuel oils and of Diesel fuels is usually so low that it can be considered negligible, the determination may frequently be considered desirable. The ash content of residual fuel oils will be of higher order because the ash content of the original oil is concentrated in this fraction and because contamination by chemicals used in refining, by solids from pipe lines or tanks, or by sea water may occur.

For the purpose of this code, the Standard Method of Test for Ash Content of Petroleum Oils, ASTM Designation: D 482-46, with modifications, is adopted.

Standard Method of Test for Ash Content of Petroleum Oils

ASTM Designation: D 482-46

Adopted 1946

This Method has been approved as "American Standard" by the American Standards Association

NOTE. A new Par. 104 was added editorially in May, 1954.

Scope

103 This method of test is intended for use in the determination of the ash content of fuel oils and other petroleum oils (Note 1).

NOTE 1. When this method is applied to used oils from engines operating on leaded fuels and to new and used oils containing metallo-organic addition agents, large portions of the metal compounds may be lost by vaporization. In such cases the ash is not a measure either of inorganic contaminating material or of the metallo-organic compounds in the oil. This method is not recommended for use with such oils.

Outline of Method

104 The sample contained in a suitable dish or crucible is ignited and allowed to burn until only carbonaceous mate-

rial remains. The residue is ignited to constant weight by heating with a flame or in a muffle furnace.

Procedure

105 A silica dish of 50- to 100-ml capacity shall be heated to redness and weighed after cooling in a desiccator. A 30- to 50-g sample of the oil shall be placed in the dish and weighed to the nearest 0.01 g. The sample shall then be heated cautiously by means of a bunsen burner until the oil ignites at the surface (Note 2).

NOTE 2. The burner shall then be removed and the oil allowed to burn completely.

The rate of burning usually decreases after the oil is partly consumed and, when this occurs, heat may again be applied by means of the bunsen burner, or by setting the dish at the opening of an electrically heated muffle, care being taken to avoid loss through excessive bubbling or frothing of the sample (Note 3). Heating shall be continued, at a temperature not exceeding 1100 F, until the carbonaceous matter is completely consumed. The dish shall then be cooled in a desiccator and weighed. The heating shall then be repeated until a constant weight is obtained. The weighing and handling of the dish shall be in accordance with accepted quantitative technique.

NOTE 3. Caution. Use of a platinum dish should be avoided if the sample contains lead, zinc, or other metals which attack platinum at high temperatures.

Report

106 Report the weight of the residue expressed as a percentage of the original sample as the percentage ash content.

Precision

107 Duplicate determinations by the same operator, or the average results obtained by two different operators should agree within 10 per cent *of the ash content.*

Ash Composition

108 Despite the relatively low ash content of fuel oils, the composition of the ash is often such that more or less serious difficulties arise in the operation of oil-fired equipment. Virtually every metal has been found in the ash of various fuel oils. The presence and the amount of some are, to some extent, characteristic of the source of the crude petroleum. The major constituents are usually compounds of the alkalis, nickel, iron, and vanadium. Vanadium and the alkalis, as oxides or in combination, are considered to be the principal offenders in their corrosive attack on refractories and on steel at temperatures as low as 1100 F. At lower temperatures, they frequently form adherent, strongly bonded deposits.

Preparation of Ash Sample

109 The preparation of the ash from the sample of oil and the analysis of the ash shall be carried out as follows:

A laboratory sample of oil-ash for analysis of ash composition shall be prepared by burning a sufficient quantity of the original oil sample to produce a minimum quantity of 0.5 to 1.0 g of ash. The practice described for burning off the carbonaceous matter at a temperature not exceeding 1100 F shall be strictly observed in order to minimize the loss of volatile inorganic constituents. Heating shall be repeated until constant weight is obtained.

Methods of Analysis

110 Standard methods of analysis[1] shall be used for the determination of vanadium, nickel, sulfur, iron, calcium, magnesium, silicon, and total alkali in the sample of ash, expressing each as a percentage by weight of the element in the sample. Spectrographic analysis may be used for confirmation and for the identification of additional elements in the sample.

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[1] "Scott's Standard Methods of Chemical Analysis," N. H. Furman (Editor); D. Van Nostrand Co., Inc., New York, Fifth Ed., 1947.
"Applied Inorganic Analysis," W. F. Hillebrand and G. E. F. Lundell; John Wiley & Sons, Inc., New York, 1929.

Water and Sediment

111 Immiscible impurities that may occur in Diesel and burner fuel are water and sediment. Water enters through contamination in transit or in storage tanks; it may also enter through leaks in steam heating coils. It may cause difficulty through interference with the combustion process and it may lead to corrosion of storage tanks. Solid sediment is not likely to be a problem with the

lighter distillate fuels but may be appreciable in the heavier, more viscous residual fuels. If the sediment is abrasive in nature it can cause wear on piping and burner parts. It will also clog strainers and burner tips and will build up as deposits in tanks and piping. Carbonaceous sediment which results from cracking during refining is not likely to be as harmful as the noncarbonaceous solids.

For the purposes of this code, three methods for the determination of water or sediment or both are adopted.

The Tentative Method of Test for Water and Sediment by Means of Centrifuge, ASTM Designation: D 96-52T, is a rapid method for estimation of the combined water and sediment. The centrifuge does not usually separate all of the water, hence, the Standard Method of Test for Water in Petroleum Products and other Bituminous Materials, ASTM Designation: D95-56T, should be used for the determination of the total water. The sediment in oil may be separately determined by the Standard Method of Test for Sediment in Fuel Oil by Extraction, ASTM Designation: D 473-48.

Tentative Method of Test for Water and Sediment by Means of Centrifuge

ASTM Designation: D 96-52T

Issued 1947; Revised 1950, 1952

Scope

112 This method describes a procedure for the determination of water and sediment in crude mineral oils and fuel oils by means of the centrifuge (Note 1).

NOTE 1. A centrifuge method for determining water and sediment is not entirely satisfactory because the amount of water obtained is nearly always lower than the actual water content. Nevertheless, on account of the wide use of the centrifuge for this purpose, it is desirable that the method of making the determination be standardized as far as possible. It must be clearly understood that the total volume as read from the centrifuge tube includes both the sediment and the precipitated water. Accurate determination of water content, if desired, should be made in accordance with the Standard Method of Test for Water in Petroleum Products and other Bituminous Materials (ASTM Designation: D 95) of the American Society for Testing Materials.

Apparatus

113 (a) **Centrifuge**, capable of whirling two or more filled centrifuge tubes at a speed which can be controlled to

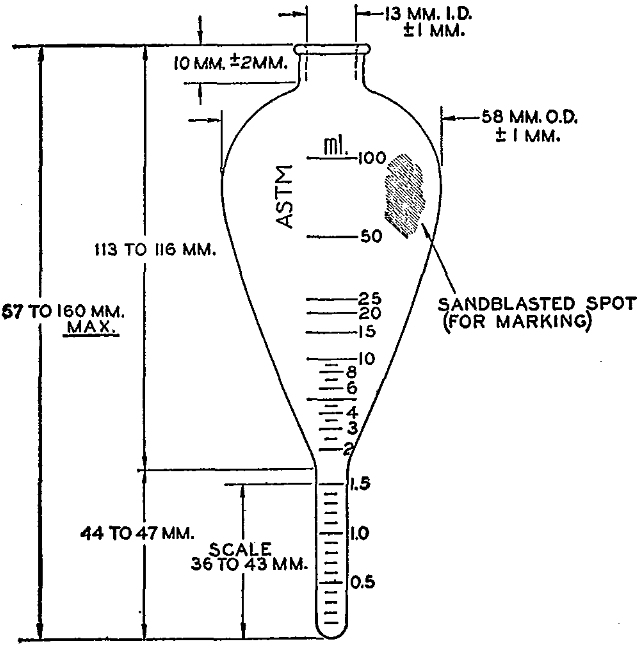

FIG. 18 ASTM PEAR-SHAPED CENTRIFUGE TUBE

give a relative centrifugal force (rcf) of between 500 and 700 at the tip of the tubes. The revolving head, trunnion rings and trunnion cups, including the cushion, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head as follows:

$$\text{rpm} = 265 \sqrt{\frac{\text{rcf}}{d}}$$

where:
rcf = relative centrifugal force, and
d = diameter of swing, inches, measured between tips of opposite tubes when in rotating position.

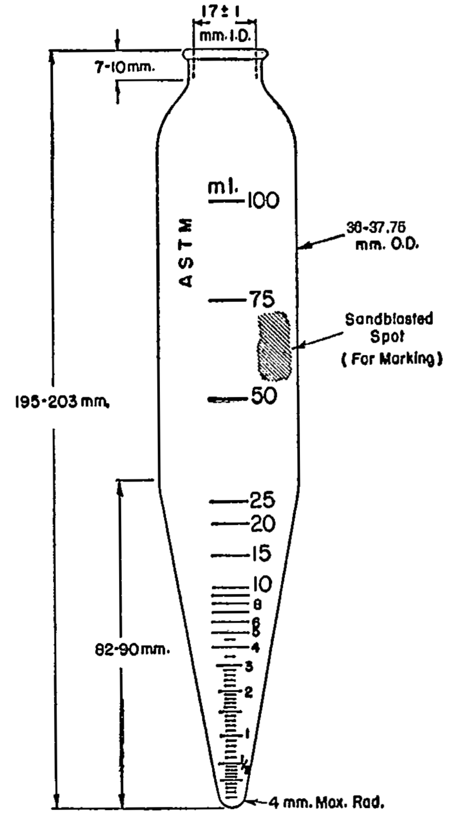

Fig. 19 ASTM Cone-Shaped Centrifuge Tube

(b) **Centrifuge Tube,** pear-shaped or cone-shaped, conforming to dimensions given in Figs. 18 and 19, respectively, and made of thoroughly-annealed glass. The graduation, numbered as shown in Figs. 18 and 19, shall be clear and distinct, and the mouth constricted in shape for closure with a cork. Scale error tolerances and smallest graduations between various calibration marks are given in Tables 14 and 15, and apply to calibrations made with air-free water at 20 C.

(c) **Bath.** The bath shall be either a solid metal block bath or a liquid bath of sufficient depth for immersing the centrifuge tube in a vertical position to the 100-ml mark. Means shall be provided for heating this bath to 120 F (49 C).

Table 14 Pear-Shaped Centrifuge Tube Calibration Tolerances

Range, ml	Smallest Scale Division, ml	Maximum Scale Error, ml
0 to 1.5	0.1	0.03
Over 1.5 to 3.0	0.5	0.20
Over 3.0 to 5.0	0.5	0.30
Over 5.0 to 10.0	1	0.50
Over 10 to 25	5	1.0
Over 25 to 100	25	2.0

Sample

114 The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself. Deviation from this rule shall not be permitted. The difficulties in obtaining representative samples for this determination are unusually great; hence, the importance of sampling cannot be too strongly emphasized.

Procedure

115 (a) Exactly 50 ml of industrial 90 benzene (ASTM Specifications D 837), shall be measured into each of two centrifuge tubes, and exactly 50 ml of the oil to be tested shall then be added to each (Note 2). The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed. The temperature of the bath shall be maintained at 120 F (49 C) and the centrifuge tubes shall be immersed therein to the 100-ml mark for 10 min.

Table 15 Cone-Shaped Centrifuge Tube Calibration Tolerances

Range, ml	Smallest Scale Division, ml	Maximum Scale Error, ml
0 to 0.1	0.05	0.02
Over 0.1 to 0.3	0.05	0.03
Over 0.3 to 0.5	0.05	0.05
Over 0.5 to 1.0	0.1	0.05
Over 1.0 to 2.0	0.1	0.10
Over 2.0 to 3.0	0.2	0.10
Over 3.0 to 5.0	0.5	0.20
Over 5.0 to 10.0	1	0.50
Over 10 to 25	5	1.0
Over 25 to 100	25	1.0

Note 2. When testing a sample containing waxy material, preheat the oil-benzene mixture to 140 F (60 C) before each whirling; the final temperature of the oil-benzene mixture shall not drop below 115 F (46 C).

(b) The two centrifuge tubes or pairs of tubes shall then be shaken vigorously for 10 sec, placed in their respective trunnion cups and balanced against each other. They shall then be placed on opposite sides of the head and whirled for 10 min at a rate, calculated from the equation in Par. 113 (a), sufficient to produce a relative centrifugal force (rcf) of between 500 and 700 at the tips of the whirling tubes (see Table 16 for relationship between diameter swing, rcf, and rpm). The combined volume of water and sediment at the bottom of each tube shall be read and recorded, reading to the nearest graduation mark from 0 to 1.5 ml on the pear-shaped tube and from 0 to 2 ml on the cone-shaped tube. Readings above the 1.5 and 2.0-ml graduations should be estimated to 0.1 ml. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 min at the same rate, and removed for reading the volume of water and sediment as before. This operation shall be repeated until the combined volume of water and sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required.

Water and Sediment

116 The combined total volume of water and sediment shall be read on each tube, estimating to 0.1 ml. The sum of the two readings shall be recorded as percentage of water and sediment, centrifuge method.

Reproducibility of Results

117 With care and proper attention to details, duplicate determinations of water and sediment by this method should not differ by more than 0.2 ml.

TABLE 16 ROTATION SPEEDS APPLICABLE FOR CENTRIFUGES OF VARIOUS DIAMETERS

Diameter of Swing, in.[a]	Rpm at 500 rcf	Rpm at 700 rcf
12	1710	2030
13	1650	1950
14	1590	1880
15	1530	1810
16	1480	1760
17	1440	1700
18	1400	1660
19	1360	1610
20	1330	1570
21	1300	1530
22	1270	1500
23	1240	1460
24	1210	1430

[a] Measured in inches between tips of opposite tubes when in rotating position.

Tentative Method of Test for Water in Petroleum Products and Other Bituminous Materials

ASTM Designation: D 95-56T

Scope

118 This method of test is used to determine the water content of bituminous materials by distillation with a water-immiscible, volatile solvent. The method is suitable for a variety of bituminous materials, but it is especially applicable to crude petroleums and tars and products derived from them, such as fuel oils, road oils, creosotes, road tars, and asphalts.

Outline of Method

119 The material is heated under reflux with a water-immiscible solvent which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap and the solvent returning to the still.

Apparatus[1]

120 The apparatus comprises a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap, and condenser may be connected by any suitable method for producing a leak-proof joint. Preferred connections are ground joints for glass to glass and O-rings for metal to glass. Typical assemblies are illustrated in Figs. 20 and 21.

(a) **Still.** A glass or metal vessel having a nominal capacity of 500 to 1000 ml and a short neck accommodating the reflux tube of the trap.

(b) **Heater.** Any suitable gas burner or electric heater may be used with the glass flask. A gas ring burner with ports on the inside circumference shall be used with the metal still.

(c) **Condenser.** A straight tube condenser, having a jacket at least 400 mm long and an inner tube whose outside diameter is 9.5 to 12.7 mm.

(d) **Trap.** A glass trap of 10- or 25-ml capacity. The traps shall be graduated in 0.1-ml divisions with a ±0.05-ml maximum error below 1 ml, and in 0.2-ml divisions with a ±0.1-ml maximum error above 1 ml.

Solvent

121 (a) For general use an aromatic solvent is preferred since it has high solvency and dispersing power for most bituminous materials. Xylol, or a blend of 20 per cent benzol and 80 per cent xylol, is recommended.

(b) For asphalts and similar petroleum products, a petroleum distillate, 5 per cent boiling between 194 and 221 F, and 90 per cent distilling below 410 F, may be used.

(c) For coal-tar, water-gas tar, and similar materials, the aromatic solvent must be used.

Sample

122 (a) The portion of the sample used for the test must be thoroughly representative of the total sample. If the material is liquid, thoroughly stir the sample as received, warming if necessary, to insure uniformity. Crush the solid materials that are sufficiently brittle, mix thoroughly, and take a representative sample for analysis. When there is doubt as to the uniformity of the material, run a number of samples and average the data.

(b) Base the size of the test portion on the estimated water content of the sample, such that the water yield does not exceed the capacity of the trap.

Procedure

123 (a) Transfer a suitable amount of sample, measured with an accuracy of ±1 per cent, to the still. Measure

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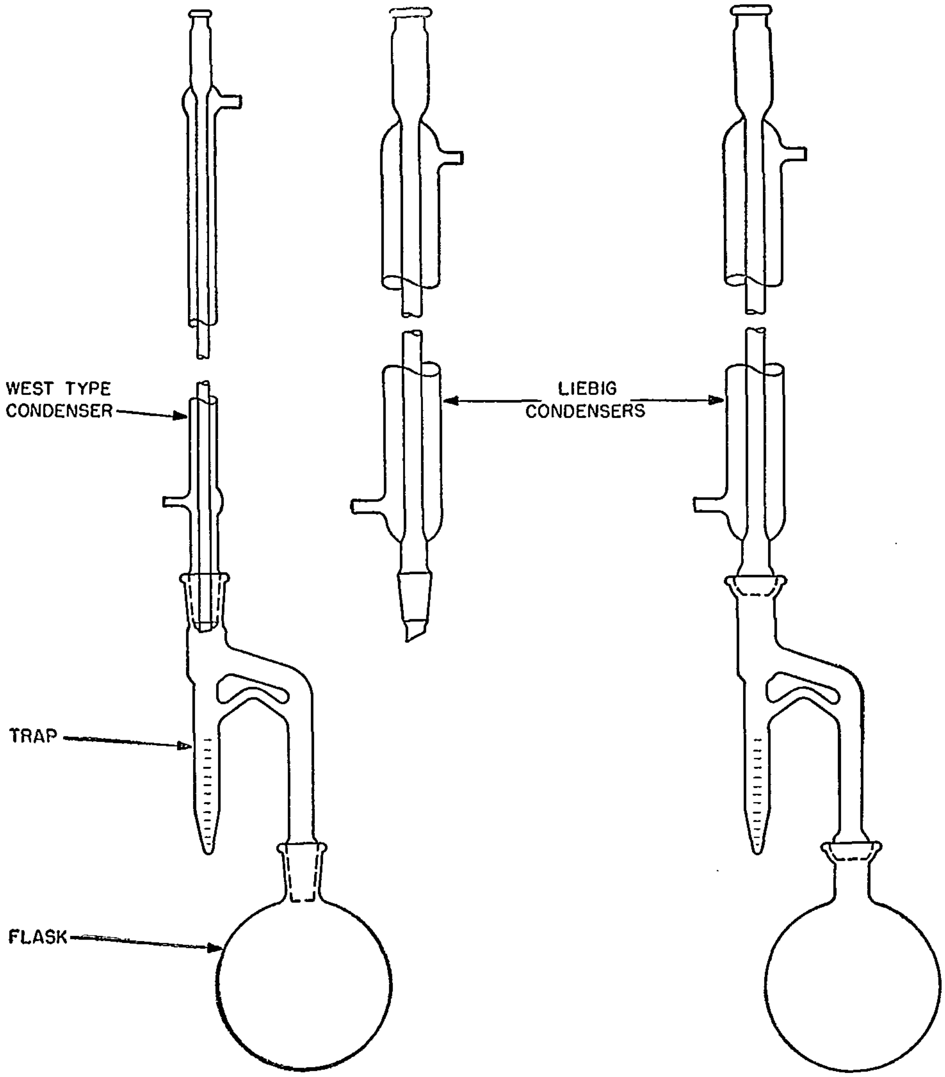
[1] Constructional details of the apparatus are prescribed in ASTM Specifications E 123, for Apparatus for Determination of Water by Distillation.

ordinary liquid samples in a graduated cylinder of appropriate size. Rinse the material adhering to the cylinder into the still with one 50-ml and two 25-ml portions of the solvent. Drain the cylinder thoroughly after the sample transfer and each rinsing. Weigh solid or semi-solid materials or bituminous emulsions directly into the still and add 100 ml of solvent. When large samples are being analyzed, a solvent volume in excess of 100 ml may be necessary.

(b) Assemble the components of the apparatus as illustrated in Figs. 20 and 21, making all connections vapor- and liquid-tight. If a metal still with removable cover is used, insert a gasket of heavy paper, moistened with solvent, between the still body and cover. The condenser tube and trap must be chemically clean to assure free drainage of water into the bottom of the trap. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture inside it. Circulate cold water through the jacket of the condenser.



Note. Trap shall be 15 to 16 mm inside diameter.

Fig. 20 Typical Assemblies with Glass Flask

(c) Apply heat to the still, adjusting the rate of boiling so that condensed distillate discharges from the condenser at the rate of 2 to 5 drops per second. If the metal still is used, start heating with the ring burner about 3 in. above the bottom of the still and gradually lower the burner as the distillation proceeds. Continue distillation until no water is visible in any part of the apparatus except in the trap. If there is a persistent ring of water in the condenser tube, increase the rate of distillation or cut off the condenser water for a few minutes.

(d) When the evolution of water is completed, allow the trap and contents to cool to room temperature. Dislodge any drops of water adhering to the sides of the trap with a glass or polytetrafluorethylene rod or other suitable means and transfer them to the water layer. Read the volume of the water in the trap to the nearest scale division.

Report

124 Report the water in the sample as per cent by weight

or volume, according to the basis on which the sample was taken, calculated as follows:

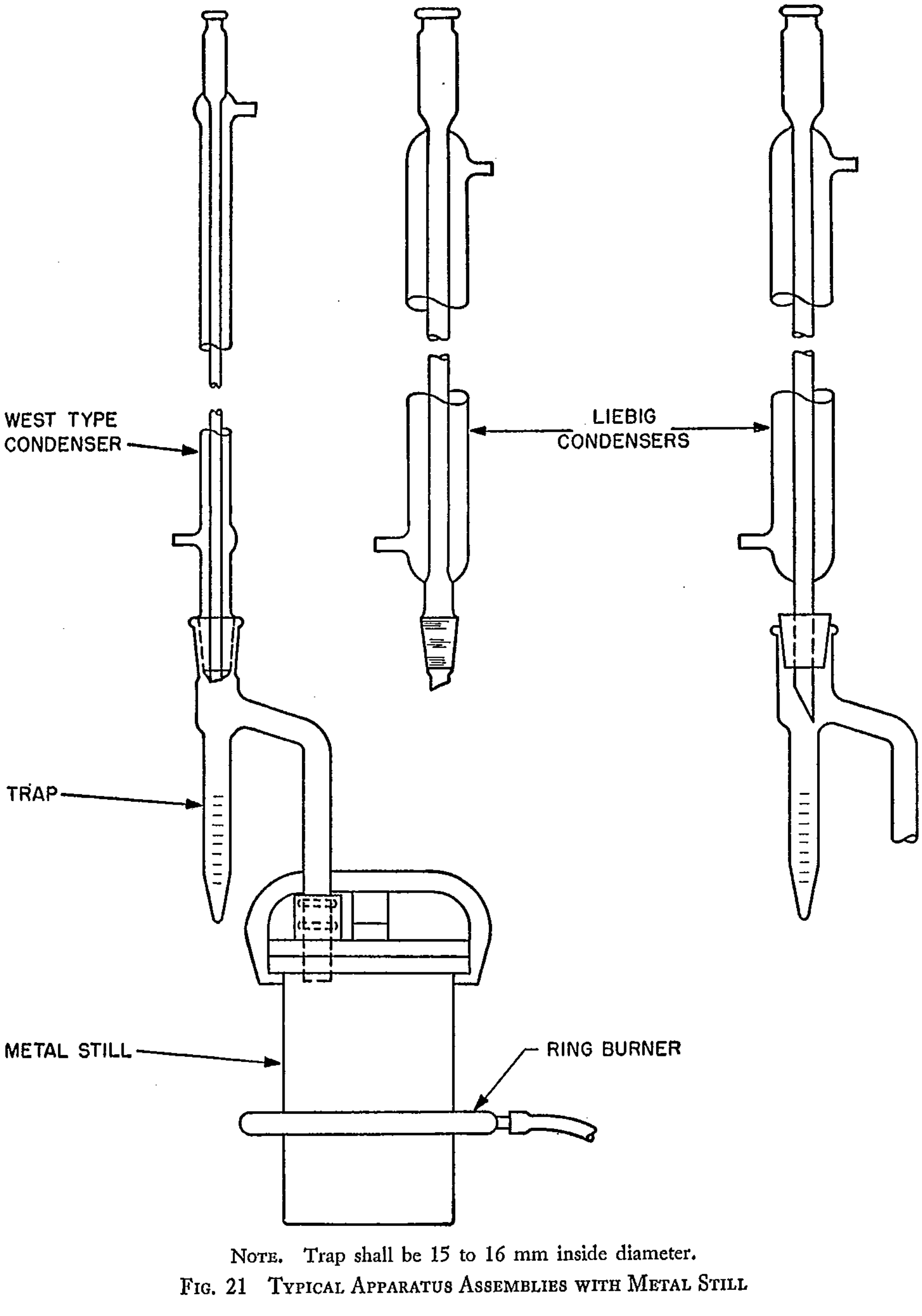
$$\text{Water, per cent} = \frac{\text{vol of water in trap}}{\text{wt (or vol) of sample}} \times 100$$

Volatile water-soluble material present may also be measured as water.

Precision

125 Two tests of the same material should agree within the greater of the following limits:

Volume of Water Collected, ml	Repeatability (Same Apparatus and Operator)	Reproducibility (Different Apparatus and Operator)
0 to 1	0.1 ml	0.2 ml
Over 1 to 25	0.1 ml or ±1 per cent of the mean	0.2 ml or ±5 per cent of the mean



NOTE. Trap shall be 15 to 16 mm inside diameter.

FIG. 21 TYPICAL APPARATUS ASSEMBLIES WITH METAL STILL

Standard Method of Test for Sediment in Fuel Oil by Extraction

ASTM Designation: D 473-48

Scope

126 The method of test is intended for the determination of sediment in fuel oils by extraction with industrial 90 benzene (ASTM Specifications D 837).

Apparatus

127 The extraction apparatus, Fig. 22 shall consist of the following:

(a) **Extraction Flask.** An Erlenmeyer flask of 1-liter capacity or a wide-mouth Erlenmeyer flask of approximately 500-ml capacity.

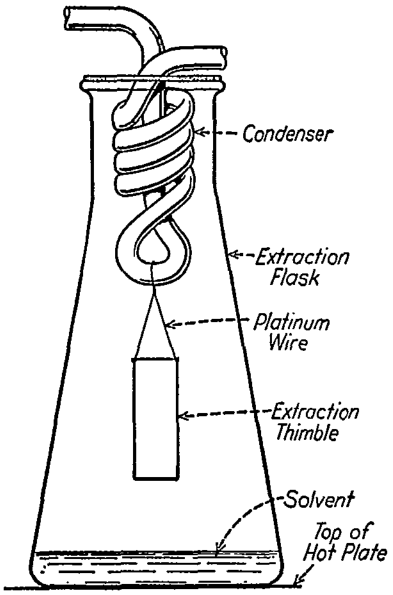


FIG. 22 EXTRACTION APPARATUS FOR DETERMINATION OF SEDIMENT

(b) **Condenser.** Underwriters' form, comprising a block-tin coil approximately 1 in. in diameter and 2 in. in length, attached to and with the ends projecting through a lid of sufficient diameter to cover the neck of the flask as shown in Fig. 22.

(c) **Extraction Thimble.** An alundum (porous grade) thimble[1] 1 in. in diameter by 2¾ in. in height, weighing not less than 15 nor more than 17 g. The thimble shall be suspended from the condenser coil by means of a platinum wire so that it hangs approximately 1¾ in. above the extracting solvent and 1¾ in. below the condenser coil.

(d) **Source of Heat.** A suitable source of heat to vaporize benzol, such as a steam bath or electric hot plate.

Procedure

128 The alundum thimble before use shall be given a preliminary extraction, allowing the solvent to drip from the thimble for at least 1 hr. The thimble shall then be dried for 1 hr at a temperature of 105 to 110 C, cooled to room temperature, and weighed to the nearest 0.0001 g. This extraction shall be repeated until the weights of the thimble after two successive extractions, do not differ by more than 0.0002 g. Approximately 10 g of the sample[2] shall be placed in the thimble and the weight determined to the nearest 0.01 g. The thimble shall be placed in the extraction apparatus and the sample extracted with industrial 90 benzene (ASTM Specifications D 837)[2] until the solvent dropping from the thimble is colorless.[3] The rate of extraction shall be such that the surface of the mixture of oil and benzol in the thimble does not rise higher than to within ¾ in. of the top. After the extraction is completed the thimble shall be dried for 1 hr at 105 to 110 C, cooled, and weighed to the nearest 0.0001 g. The extraction shall be repeated, allowing the solvent to drip from the thimble for at least 1 hr but not longer than 1¼ hr, the thimble dried, cooled, and weighed as above. This extraction for a 1-hr period shall be repeated, if necessary, until the weights of the dried thimble and sediment after two successive extractions do not differ by more than 0.0002 g. For referee tests a new extraction thimble shall be used. For routine tests thimbles may be used for a number of successive determinations on different samples, the extraction to constant weight for one determination being considered as the preliminary extraction for the succeeding determination. When the accumulation of sediment becomes objectionable and the combustible portion is removed by heating to a dull red heat (preferably in an electric furnace), the thimble shall be subjected to a preliminary extraction before being used for another determination.

Reproducibility of Results

129 With proper attention to details of methods of procedure, check results by the same operator should not deviate from the mean value by more than the following amounts:

Sediment, per cent	Deviation from Mean Value
0.00 to 0.25	±0.01
0.26 to 0.50	±0.02

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[1] Alundum thimbles listed as No. 5163 RA 98 by the Norton Co., Worcester, Mass., have proved satisfactory.

[2] The portion of the sample taken for test shall be thoroughly representative of the sample. An estimated 10-g portion of the sample should be placed in the thimble as soon as possible after the sample has been thoroughly mixed. No attempt should be made to adjust this estimated 10-g portion to any exact predetermined amount.

[3] When testing oils having high water content, difficulty due to clogging of the thimble with moisture may be overcome by interrupting the extraction when this occurs and drying the thimble in an oven before proceeding further with the extraction.

Carbon Residue

130 Petroleum products are mixtures of many compounds which differ widely in their physical and chemical properties. Some oils may be vaporized at atmospheric pressure in the absence of air without leaving appreciable residue, but other oils upon distillation leave a nonvolatile carbonaceous residue. The residue, when determined in accordance with a prescribed procedure, is designated carbon residue although it also contains the ash-forming constituents of the oil and thus will be erroneously high as a measure of the carbon residue. Ash can be deducted.

The carbon residue is considered of minor importance in evaluating the performance characteristics of residual fuel oils or distillate oils which are to be used in atomizing burners. The degree of atomization and the intensity of mixing of the combustion air with the fuel spray are of much greater importance. However, for oils to be used in vaporizing burners in which the oil is converted to a vapor by contact with a hot surface, the carbon residue will be of importance. Accumulations of carbon may cause operating difficulties from poor oil distribution, clogged oil inlets, shorted ignition electrodes, or delayed ignition.

For the purpose of this code, the Tentative Method of Test for Carbon Residue of Petroleum Products, Ramsbottom Coking Method, ASTM Designation: D 524-52T, is adopted.

Tentative Method of Test for Carbon Residue of Petroleum Products

Ramsbottom Coking Method

ASTM Designation: D 524-52T

Issued 1951; Revised 1952

This Method has been approved as "American Standard" by the American Standards Association

NOTE. An editorial change in Par. 133(a) was made in June, 1955.

Scope

131 This method describes the test procedure for determining the amount of carbon residue (Note 1) left after evaporation and pyrolysis of an oil, and is intended to provide some indication of the relative coke-forming propensity. The method is generally applicable to relatively nonvolatile petroleum products which partially decompose on distillation at atmospheric pressure. Petroleum products containing ash-forming constituents as determined by ASTM Method D 482, Test for Ash Content of Petroleum Oils, will have an erroneously high carbon residue, depending upon the amount of ash formed (Note 2).

NOTE 1. The term "carbon residue" is used throughout this method to designate the carbonaceous residue formed during evaporation and pyrolysis of a petroleum product. The residue is not entirely composed of carbon, but is a coke which can be further changed by pyrolysis. The term "carbon residue" is continued in this method only in deference to its wide common usage.

NOTE 2. Values obtained by this method are not numerically the same as those obtained by the Conradson coking method (ASTM Designation: D 189), nor have satisfactory correlations been found between the results by the two methods for all materials which may be tested, because the carbon residue test is applied to a wide variety of petroleum products.

Outline of Method

132 (a) The sample, after being weighed into a special glass bulb having a capillary opening, is placed in a metal furnace maintained at approximately 1020 F. The sample is thus quickly heated to the point at which all volatile matter is evaporated out of the bulb with or without decomposition while the heavier residue remaining in the bulb undergoes cracking and coking reactions. In the latter portion of the heating period, the coke or carbon residue is subject to further slow decomposition or slight oxidation due to the possibility of breathing air into the bulb. After a specified heating period, the bulb is removed from the bath, cooled in a desiccator, and again weighed. The residue remaining is calculated as a percentage of the original sample.

(b) Provision is made for determining the proper operating characteristics of the furnace with a control bulb containing a thermocouple, which must give a specified time-temperature relationship.

Apparatus

133 (a) **Glass Coking Bulb,** of heat resistant glass, conforming to the dimensions and tolerances shown in Fig. 23 (Note 3). Prior to use, check the diameter of the capillary to see that the opening is greater than 1.5 and not more than 2.0 mm. Pass a No. 53 Twist Drill and Steel Wire Gage drill rod through the capillary and into the bulb; attempt to pass a No. 47 Twist Drill and Steel Wire Gage drill rod through the capillary. Reject bulbs that do not permit the insertion of the smaller rod and those whose capillaries are larger than the larger rod (Note 3).

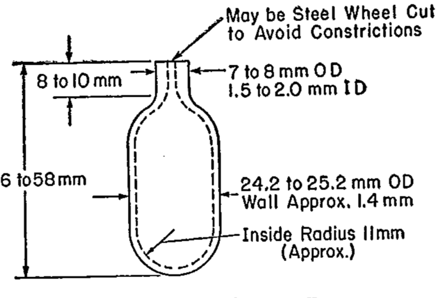

FIG. 23 GLASS COKING BULB

NOTE 3. Do not use the stainless steel bulbs heretofore specified for ASTM Method D 524-42 or earlier versions, because unpredictable weight changes are likely to occur, causing erratic results.

(b) **Control Bulb,** stainless steel, containing a thermocouple and conforming to the dimensions and tolerances shown in Fig. 24, for use in determining compliance of furnace characteristics with the performance requirements (Par. 133). The control bulb shall be provided with a dull finish, as specified in Fig. 24, and must not be polished thereafter. A polished bulb has different heating characteristics from one with a dull finish. A suitable thermocouple pyrometer for observing true temperature within ±2 F is also required.

(c) **Sample Charging Syringe,** 5 or 10-ml glass hypodermic (Note 4), fitted with a No. 17 needle (1.5 mm OD) for transfer of the sample to the glass coking bulb.

NOTE 4. A syringe having a needle which fits on the ground-glass tip of the syringe is not recommended, as it may be blown off when pressure is applied to the syringe plunger. The Luer-Lok type syringes are more satisfactory, as the needle locks on the bottom of the syringe barrel, and cannot be blown off by pressure.[1]

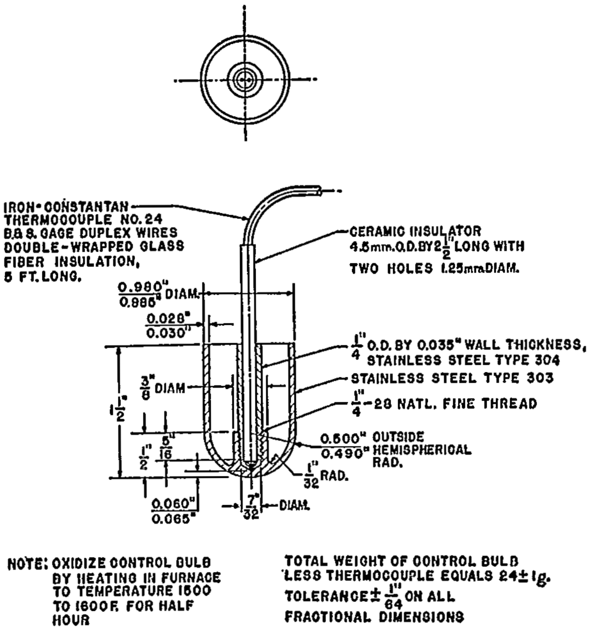

FIG. 24 CONTROL BULB

(d) **Metal Coking Furnace** of solid metal, having coking bulb wells, 1.002 ± 0.004 in. in internal diameter and 3 in. deep to the center of the well bottom, with suitable arrangements for heating to a uniform temperature of 1020 F. The bottom of the well shall be hemispherical to accommodate the bottom of the glass coking bulb. Do not cast or otherwise form the furnace with unnecessary voids which will impede heat transfer. If a molten metal furnace is used, provide it with a suitable number of bulb wells, the internal dimensions of which correspond to the internal dimensions of holes in the solid metal furnace. The bulb wells shall be immersed in the molten metal to leave not more than $\frac{1}{8}$ in. of the bulb well exposed above the molten metal at operating temperatures (Note 5).

NOTE 5. Ramsbottom coke furnaces now in use may have dimensional differences from those given above; however, it is essential that new furnaces obtained after the adoption of this method conform to the requirements outlined in Paragraph (d). A description of one type of furnace which has been found to be satisfactory is given in the Appendix.

(e) **Temperature-Measuring Devices.** A removable iron-constantan thermocouple with a sensitive pyrometer, or other suitable temperature-indicating device, located centrally near the bottom portion of the furnace and arranged to measure the temperature of the furnace so that the performance tests specified in Par. 134 can be obtained (Note 6). It is desirable to protect the temperature-indicating device with a quartz or thin metal sheath when a molten bath is used.

NOTE 6. It is good practice to calibrate the thermocouple or other temperature-measuring device against a standard thermocouple or reference standards about once a week, when the furnace is in constant use, the actual frequency depending on experience.

Checking Performance of Apparatus

134 Periodically check the performance of the furnace and temperature-measuring devices as described in Paragraphs (a), (b), and (c) to make certain that as used they conform to the requirements of the method. Consider the furnace as having standard performance, and use it with any degree of loading, when the operating requirements described for each coking bulb well are met, while the bath is fully loaded as well as singly loaded. Use only a furnace that has successfully passed the performance or control tests given in this section.

(a) **Thermocouple.** At least once every 50 hr of use of the control bulb, calibrate the thermocouple in the control bulb against a standard thermocouple (Note 7).

NOTE 7. In use at the high temperature of the test iron-constantan thermocouples oxidize and their calibration curves change.

(b) **Fully Loaded Furnace.** When the furnace temperature is within a previously chosen 4 F temperature range (which range is to be used thereafter with that particular furnace for both standardization and routine operation) and within the general range 1020 ± 10 F, insert the control bulb in one well and, within 15 sec, insert in each of the other wells a glass coking bulb containing 4 ± 0.5 g of a viscous neutral petroleum lubricating oil with a viscosity within the SAE 30 range. With a suitably accurate potentiometer or millivoltmeter (sensitive to 2 F or less), observe the temperature rise in the control bulb at 1-min intervals for 20 min. If the temperature in the control bulb reaches 1015 F in not less than 4 and not more than 6 min from the instant of its insertion in the furnace, and remains within the range 1020 ± 5 F for the remaining portion of the 20-min test, consider that particular coking bulb well suitable for use as a "standard performance well" when the furnace is used fully loaded. Inspect each well in similar fashion with the furnace fully loaded each time (Note 8).

NOTE 8. It is possible that not all of the wells in old furnaces will meet the requirements when fully loaded and singly loaded; and, when this is the case, inspect each well for any degree of furnace loading which may be used. For example, if not more than three wells of a six-well furnace can be used at any one time, the three wells to be used should be chosen from the performance data obtained with fully loaded and singly loaded furnaces. Then each of the three wells should be inspected for triple loading, two of the wells for double loading, and one for single loading. Use the wells tested and no others in applying the test procedure.

(c) **Singly Loaded Furnace.** When the furnace temperature is within a previously chosen 4 F temperature range (which range is to be used thereafter with that particular furnace for both standardization and routine operation) and within the general range 1020 ± 10 F, insert the control bulb in one well, with the remaining wells unoccupied. With a

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[1] A suitable syringe of this type is manufactured by Becton, Dickinson and Co., Rutherford, N. J.

suitably accurate potentiometer or millivoltmeter (sensitive to 2 F or less), observe the temperature rise in the control bulb at 1-min intervals for 20 min. If the temperature in the control bulb reaches 1015 F in not less than 4 and not more than 6 min from the instant of its insertion in the furnace, and remains within the range 1020 ± 5 F for the remaining portion of the 20-min test, consider that particular coking bulb well suitable for use as a "standard performance well" when only a single test is made. Inspect each well in similar fashion with the furnace singly loaded each time.

Procedure

135 (a) Place a new glass coking bulb (Note 9) in the coking furnace at 1020 F for about 20 min to decompose any foreign matter and to remove water. Place in a closed desiccator over $CaCl_2$ for 20 to 30 min and then weigh to the nearest 0.1 mg.

NOTE 9. Do not reuse a glass coking bulb, as unpredictable results are sometimes obtained in such cases. For routine testing, new bulbs may be used without preignition provided they are visibly free from particles or other contamination. Such bulbs, at least, should be heated in an oven to 300 F, placed in a desiccator, and then weighed.

(b) Shake thoroughly the sample to be tested, first warming if necessary to reduce its viscosity. Strain the sample through a 100-mesh wire screen. Fill a hypodermic syringe with the sample and carefully charge the coking bulb with 4.0 ± 0.5 g of sample, making sure that no oil remains on the exterior surface or on the inside of the neck of the bulb. Reweigh the bulb and contents to the nearest milligram. In those cases where spattering of the sample is known to take place, for example, with some oils having a carbon residue value greater than 6 per cent, reduce the size of the sample to 1.0 ± 0.3 g. In reporting the results, include the size when such small samples are used (Note 10).

NOTE 10. In sampling oils containing sediment (for example, used oils), it is important to make the transfer of sample in the shortest possible time to avoid segregation of the sediment. Samples containing sediment which settles quickly after stirring can be placed in the coking bulbs more expeditiously by using an arrangement such as that shown in Fig. 25. This sampling device consists of a three-way 2-mm stopcock to which has been fused two lengths of capillary tubing (1.5 mm ID). Connect the third leg of the stopcock by means of pressure tubing to a vacuum line. Secure the glass coking bulb to the short arm of capillary tubing by a 1-in. length of rubber hose, taking care that the capillary of the glass bulb is butted up against the capillary tubing. Immerse the long end of the capillary tubing in the sample. After evacuating the coking bulb, manipulate the stopcock to cause the stirred sample to flow freely into the bulb through the two lengths of capillary tubing. It is necessary to use tubing with the same size capillary as that in the neck of the coking bulb to prevent accumulation of any sediment during transfer.

(c) Place the coking bulb in a "standard performance well" with the furnace at the checking temperature (Note 11), and allow to remain for 20 ± 2 min. Remove the bulb with metal tongs, the tips of which have just been heated. Duplicate the furnace and bulb conditions used when standardizing that bulb well (Par. 134 and Note 8). If there is appreciable loss of oil from frothing, discard the test (Note 12).

NOTE 11. On making a test, it is important to adhere rigorously to the temperature conditions chosen for Checking Performance of Apparatus Par. 134; for example, if the bath was at a temperature of 1025 ± 2 F when inserting the control bulb, then it is necessary to use similar temperature conditions in the coking test. When maintained in normal operation, the temperature of an electrically heated furnace with automatic controls will generally fluctuate within a specific temperature range. Therefore, when making a coking test, it is generally important that the test bulbs be inserted when the furnace is at the same temperature or at the same position in the temperature cycle as it was when the inspection test was started, unless it has been proven that the temperature variations are inappreciable.

NOTE 12. Frothing may be due to water which can be removed by heating gently in a vacuum and sweeping out the vapor with nitrogen prior to filling the bulb.

(d) After removal, cool the bulb in a desiccator under the same conditions (including time for weighing) used befor filling the bulb (Paragraph (b)). When removing the bulb from the desiccator, examine to make sure there are no foreign particles adhering to the bulb; if any are found, as black particles sometimes are on the capillary neck, brush them off with a piece of sized paper or camel's hair brush. Weigh to the nearest 0.1 mg. Discard the used glass coking bulb (Note 13).

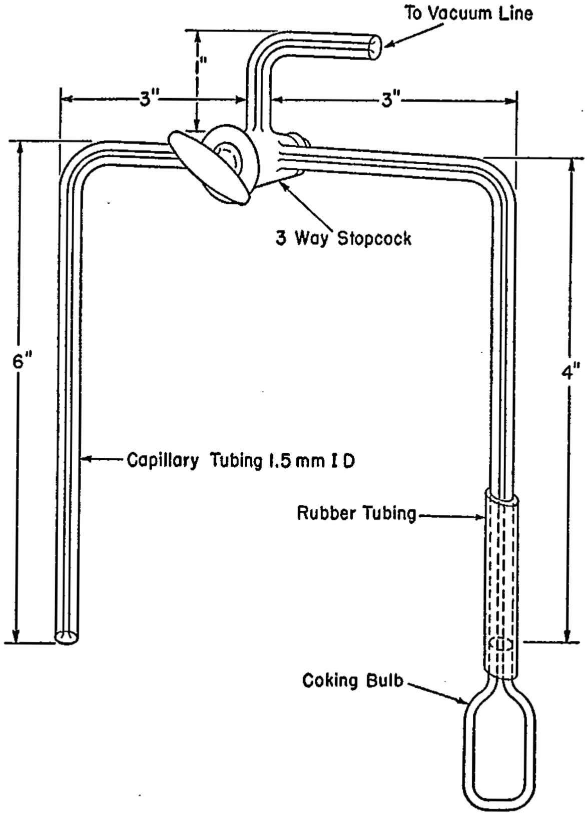

FIG. 25 SAMPLING DEVICE

NOTE 13. In studies of oil characteristics, useful information can often be gleaned from a simple visual examination of the coking bulb after the test. Thus, significance can be attached to noting with the results, such findings as: coke more or less fills the bulb; liquid material is present, either as liquid residue or drops; the residue is not black and flaky, but is colored and pulverulent (presumably from presence of inorganic materials).

Test on 10 Per Cent Distillation Residue

136 This test is intended for testing light distillate oils having Ramsbottom coke residue less than 0.05 per cent, such as No. 1 and No. 2 fuel oils.

(a) Make a distillation using a clean flask for each test as described in ASTM Method D 158, Test for Distillation of Gas Oil and Similar Distillate Fuel Oils, using the 200-ml graduate to measure the initial sample, and, without cleaning, to receive the distillate. Maintain the condenser temperature at 32 to 40 F throughout the distillation, in the case of products having distillation end points below 600 F; use the standard condenser temperature of 90 to 100 F for products having end points above 600 F or for samples that yield waxy distillates.

(b) Carry out the distillation at the rate prescribed in Method D 158 until 178 ± 0.5 ml have been collected in the graduate, then discontinue the heating and allow the condenser to drain until 180 ml (90 per cent of the charge to the flask) has been collected in the graduate. Replace the graduate with a small Erlenmeyer flask and catch any final drainage in this flask.

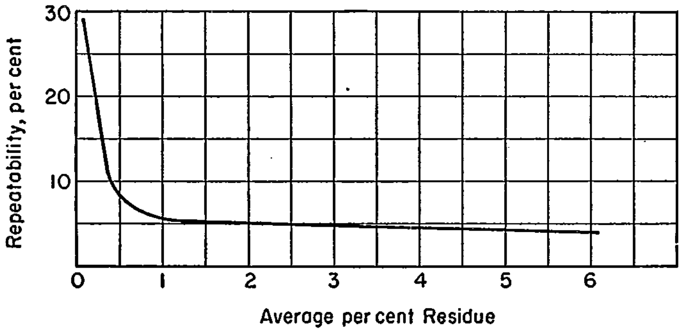

FIG. 26 REPEATABILITY *versus* AVERAGE PER CENT RESIDUE, RAMSBOTTOM CARBON RESIDUE

(c) To the distillate in the Erlenmeyer flask add, while still warm, the residue left in the distillation flask and shake well (Note 14). While warm enough to flow freely, place 4.0 ± 0.5 g of sample in a previously weighed coking bulb, using a hypodermic syringe for this operation, and proceed as described in Par. 135.

NOTE 14. The contents of the Erlenmeyer flask represent a 10 per cent residue from the original product.

Calculation and Report

137 (a) Calculate the carbon residue of the sample or of the 10 per cent residue as follows:

$$\text{Carbon residue} = \frac{A \times 100}{W}$$

where:

A = weight of carbon residue, in grams, and
W = weight of sample, in grams.

(b) Report the values obtained as "ramsbottom carbon residue, per cent," or as "ramsbottom carbon residue on 10 per cent distillation residue, per cent," indicating when a 1-g sample is used.

Precision

138 The criteria below should be used for judging the acceptability of the results when testing homogeneous samples. Carbon residue values of less than 0.1 per cent are not reliable, as shown by the curves in Figs. 26 and 27.

(a) **Repeatability.** Duplicate results by the same operator should not differ by more than the corresponding percentage as shown in Fig. 26 for repeatability *versus* average per cent residue.

(b) **Reproducibility.** The average of two results obtained in each of two laboratories should not differ by more than the corresponding percentage as shown in Fig. 27 for reproducibility *versus* average per cent residue.

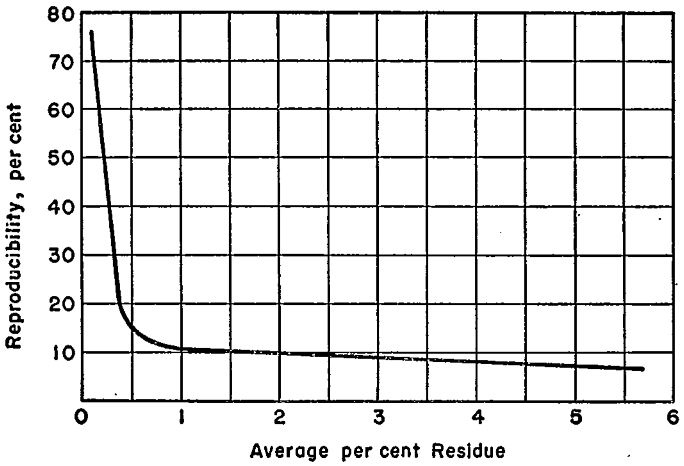

FIG. 27 REPRODUCIBILITY *versus* AVERAGE PER CENT RESIDUE, RAMSBOTTOM CARBON RESIDUE

Appendix

Ramsbottom Coking Furnace

A1 The greatest difficulty in achieving satisfactory precision for this method is to obtain a uniformly operating furnace. The type of furnace described below meets the performance characteristics prescribed in Par. 134.

A2 Solid Metal Furnace.[2] A solid metal furnace may be constructed as illustrated in Fig. 28. It may be constructed of cast iron or other suitable metal for use under the high-temperature conditions which are employed in this method of test. It is desirable to cast the metal without any unnecessary voids. Use of a substantial mass of metal for the block avoids the requirement for an excessive amount of electrical heating which could cause wide fluctuations in block temperature unless very sensitive controls were used.

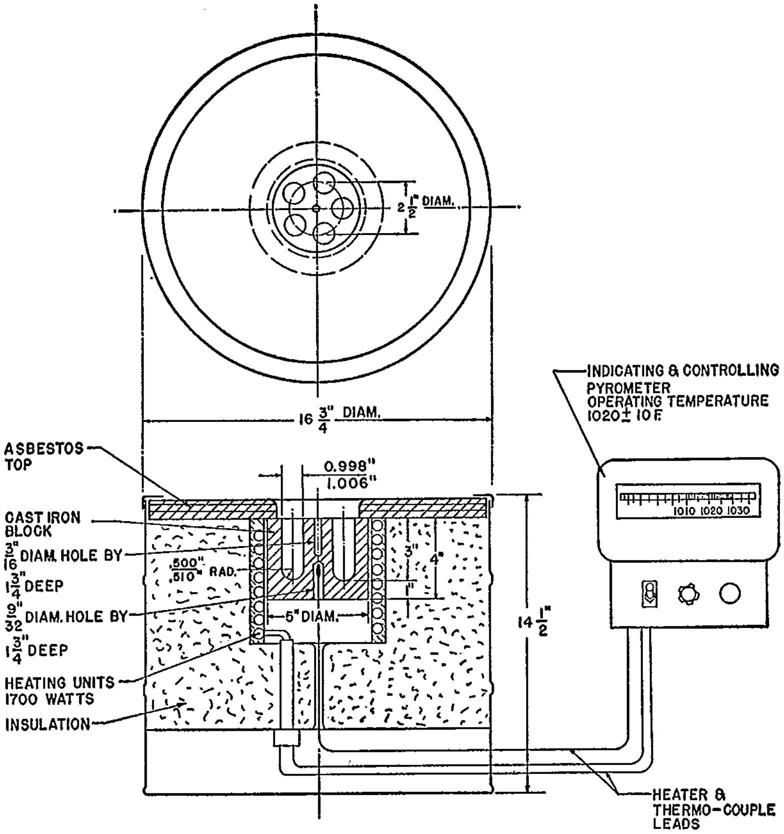

Fig. 28 Solid Metal Furnace

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[2] A suitable solid metal furnace is available commercially from the Precision Scientific Co., 3737 West Cortland Street, Chicago 47, Ill.

Distillation Range

139 The principal purpose of a distillation test of a fuel oil, in which the fractions of the oil which distill at various temperature ranges are determined, is to determine the composition of the oil. That is, the results will demonstrate whether the fuel is a distillate oil or whether it is a blend of heavy and light fractions.

The results also indicate the ease with which the fuel may be vaporized and are thus of value in indicating the ease of ignition and the suitability of the oil for cold starts in vaporizing-type burners. For atomizing burners, the fineness of atomization is much more important than the volatility of the fuel. For either vaporizing or atomizing burners, the distillation end point, in itself, has a relatively minor effect on the performance because the components of the fuel represented by the end point usually constitute only a small percentage of the total.

To cover the range of this code, two standards are adopted: The Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, ASTM Designation: D 86-56, and the Standard Method for Test for Distillation of Gas, Oil and Similar Distillate Fuel Oils, ASTM Designation: D 158-54.

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products

ASTM Designation: D 86-56

Adopted 1927; Revised 1930, 1935, 1938, 1940, 1945, 1946, 1952, 1953, 1954, 1956

This Method has been approved as "American Standard" by the American Standards Association

Scope

140 This method of test is intended for use in the distillation of gasoline, naphtha, kerosine, and similar petroleum products.

Apparatus

141 The apparatus shall consist of the following:

(a) **Flask.** A standard 100-ml Engler distillation flask, as shown in Fig. 29, conforming to the following dimensions and permissible variations:

	Centimeters	Inches	Permissible Variations	
			Centimeters	Inches
Diameter of bulb, outside	6.5	2.56	±0.2	±0.08
Diameter of neck, inside	1.6	0.63	±0.1	±0.04
Length of neck	15.0	5.91	±0.4	±0.16
Length of vapor tube	10.0	3.94	±0.3	±0.12
Diameter of vapor tube, outside	0.6	0.24	±0.05	±0.02
Diameter of vapor tube, inside	0.4	0.16	±0.05	±0.02
Thickness of vapor tube wall	0.1	0.04	±0.015	±0.0006

The position of the vapor tube shall be 9 ± 0.3 cm (3.55 ± 0.12 in.) above the surface of the liquid when the flask contains its charge of 100 ml. The tube shall be approximately in the middle of the neck and set at an angle of 75 ± 3 deg with the vertical.

(b) **Condenser.** A condenser (Fig. 30A shows one of the approved types) $\frac{9}{16}$ in. (14.29 mm) in outside diameter with 0.031 to 0.036-in. wall thickness, made of seamless brass tubing, 22 in. (55.88 cm) in length. It shall be set so that approximately 15.5 in. (39.4 cm) of the tube will be in contact with the cooling medium contained in the condenser jacket, subsequently described, with about 2 in. outside the cooling bath at the upper end, and 4.5 in. outside the cooling bath at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of

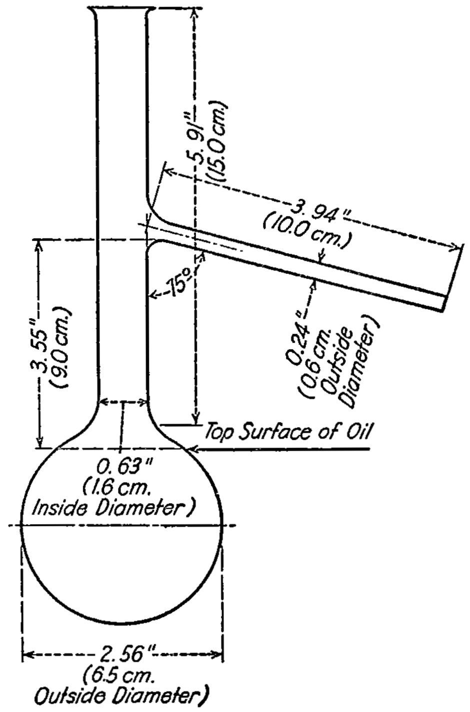

FIG. 29 DIMENSIONS OF 100-ML ENGLER FLASK

75 deg with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.26 in. per linear inch of condenser tube (sine of angle of 15 deg) and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. nor more than 0.28 in. per linear inch of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to 1¼ in. (2.54 to 3.18 cm) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle. The capacity of the cooling bath shall be not less than 340 cu in. (5.55 liters) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than 1¼ in. below the plane of the top of the bath at its point of entrance and not less than ¾ in. above the floor of the bath at its exit. Clearances between the condenser tube and the walls of the bath shall be at least ½ in. except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 cu in. per tube.

(c) **Shield.** Two types of shields can be used, as follows:

Type 1 Shield. A shield (Fig. 30A) 19 in. high, 11 in. long and 8 in. wide, made of sheet metal approximately No. 22 gage, with a door on one narrow side, two openings 1 in. in diameter equally spaced in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be 8½ in. below the top of the shield. There shall be three ½-in. holes in each of the four sides with their centers 1 in. above the base of the shield.

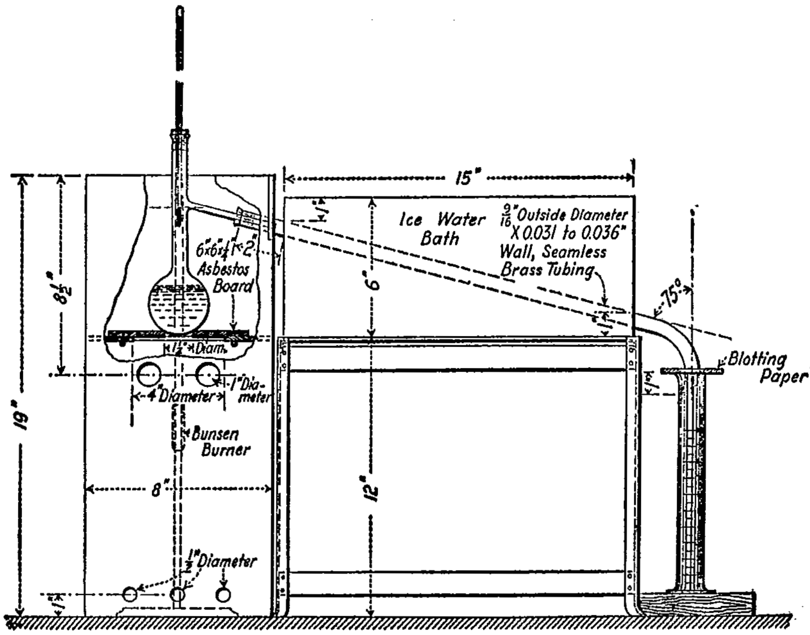

Fig. 30A Apparatus for Distillation Test

Type 2 Shield. A shield 17½ in. high, 8 in. long and 8 in. wide made of sheet metal of approximately No. 22 gage, with window on front side. Open bottom of shield spaced approximately 2 in. from base of unit. Rear of shield to have eliptical hole for vapor tube. A flask adjusting knob located on the front of the shield for adjusting the flask support. Also heat adjusting indicating dial to provide stepless heat control when electric heater is used. All mechanism built into bottom portion of shield.

(d) **Flask Support and Ceramic or Asbestos Boards.** A ring support of the ordinary laboratory type, 4 in. or larger in diameter, supported on a stand inside the shield, or a flask support platform that is adjustable from the outside of the shield may be used. Two ceramic or hard asbestos boards, the first having a hole 1¼-in.[1] in diameter in the center and the thickness of the board at the 1¼-in. diameter opening shall be ⅛ to ¼-in.; the second board to fit tightly inside the shield with an opening 3 to 4 in. in diameter, concentric with the flask support. These shall be arranged as follows: The second board shall be placed on the support, and the first or smaller board on top so it may be moved in accordance with the directions for placing the distillation flask. Direct heat shall be applied to the flask only through the 1¼-in. diameter opening in the first board.

(e) **Gas Burner.** A burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in Par. 143. The flame shall never be so large that it spreads over a circle of diameter greater than 3½ in. (8.89 cm) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

(f) **Electric Heater.** An electric heater may be used instead of a gas burner and shall be capable of bringing over the first drop within the time specified in Par. 143 (a) when started cold, and of continuing the distillation at a uniform rate. (A heater unit of low-heat capacity, adjustable from 0 to 750 w, has been found satisfactory.) The electric heater shall be fitted with a ceramic or hard asbestos board top having a hole 1¼ in. in diameter in the center, and the thickness of the board at the 1¼-in. diameter opening shall be ⅛ to ¼ in. When an electric heater is employed, the portion of the shield above the board shall be the same as with the gas burner, but the part below the board may be omitted.

(g) **Thermometer.** An ASTM Low-Distillation Thermometer, total immersion, graduated in either Centigrade or

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[1] When distilling petroleum products having an end point above 470 F (243.3 C), the hole in the asbestos or ceramic board shall be 1½ in. (3.81 cm) in diameter.

Fahrenheit degrees, having a range of —2 to +300 C or 30 to 580 F and conforming to the requirements for thermometer 7 C or 7 F, respectively, as prescribed in the Standard Specifications for ASTM Thermometers (ASTM Designation: E 1) (Note 1).

(h) **Thermometer.** An ASTM High-Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of —2 to +400 C or 30 to 760 F and conforming to the requirements for thermometer 8 C or 8 F, respectively, as prescribed in the Standard Specifications for ASTM Thermometers (ASTM Designation: E 1).

Note 1. Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements applicable to the Institute of Petroleum thermometers IP 5 C (Low Distillation) and IP 6 C (High Distillation) may be used, provided calibration corrections are used in temperature ranges where IP requirements for scale accuracy are less stringent than those of ASTM.

(i) **Graduate.** A graduate of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top (Note 2). The cylinder shall be graduated to contain 100 ml, and the graduated portion shall be not less than 7 in. (17.78 cm) nor more than 8 in. (20.32 cm) in length. It shall be graduated in single milliliters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 ml. The over-all height of the graduate shall be not less than $9\frac{3}{4}$ in. (24.8 cm), nor more than $10\frac{1}{4}$ in. (26.0 cm). The graduations shall not be in error by more than 1 ml at any point on the scale.

Note 2. Cylinders lacking the bottom 1-ml line will not be rejected.

Preparation of Apparatus

142 (a) The condenser bath shall be filled with cracked ice,[2] and enough water added to cover the condenser tube. The temperature shall be maintained at 32 to 34 F (0 to 1.1 C) for gasolines higher than 9.5 lb Reid vapor pressure and 32 to 40 F (0 to 4.5 C) for other products.

(b) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(c) A 100-ml sample shall be measured in the 100-ml graduated cylinder at 55 to 65 F (12.8 to 18.3 C) and transferred directly to the distillation flask which has been cooled at 55 to 65 F (12.8 to 18.3 C) for gasolines higher than 9.5 lb Reid vapor pressure. The flask may be at room temperature for other products. None of the liquid shall be permitted to flow into the vapor tube.

(d) The thermometer (Note 3) provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet at its junction with the neck of the flask. The thermometer shall be approximately at room temperature when placed in the flask.

Note 3. Either the ASTM High- or Low-Distillation Thermometer shall be used depending upon the initial boiling point and the end point of the product. Use the ASTM Low-Distillation Thermometer for all materials except those found to have both the initial boiling points higher than 212 F (100 C) and final boiling points higher than 482 F (250 C) as determined using that thermometer.

(e) The charged flask shall be placed in the $1\frac{1}{4}$-in. (3.18-cm) opening in the 6 by 6-in. (15.24 by 15.24-cm) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm) nor more than 2 in. (5.08 cm).

(f) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm) but not below the 100-ml mark. Unless the room temperature is between 55 and 65 F (12.8 and 18.3 C) the receiving graduate shall be immersed up to the 100-ml mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

Procedure

143 (a) When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 min. The distillation thermometer shall be read 2 min after heat is applied and the indication recorded as the "correction temperature." This figure is of significance only in cases when there is a question as to the accuracy[3] of the initial boiling point, as subsequently determined. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point.*

(b) The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation proceeds at a uniform rate of not less than 4 ml per min nor more than 5 ml per min. The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 ml, when the mercury of the thermometer reaches each point that is a multiple of 10 C or the Fahrenheit equivalent of this point (30 C, 40 C, 50 C, 60 C, etc., or 86 F, 104 F, 122 F, 140 F, etc.). If preferred, the reading of the distillation thermometer may be observed and recorded when the level of the distillate reaches each 10-ml mark on the graduate. In case a product is being tested to ascertain whether or not it conforms with a given specification, all necessary observations shall be made and recorded, whether or not they are included in the series ordinarily employed by the laboratory making the test.

(c) No adjustment of the heat shall be made after the liquid residue in the flask is approximately 5 ml unless the time required to bring over the last 5 ml of distillate and

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[2] Any other convenient cooling medium may be used.

[3] The initial boiling point is one of the observations which it is customary to report as indicative of the quality of gasoline. It is a point which is very difficult to check, especially when duplicate tests are made at different room temperatures, and it has not been found practical to devise an accurate method of correcting for this unavoidable variation in operating conditions. The practical significance of initial boiling point is not regarded as sufficient to warrant requiring that tests be conducted under regulated conditions of room temperature. The following directions are included as an expedient intended to obviate some of the difficulties that develop on account of the practice of including initial boiling point limits in specifications for the purchase and sale of gasoline:

If the "correction temperature" is below 70 F (21 C) or above 80 F (27 C), the observed initial boiling point, if it is below 150 F (66 C), may be considered incorrect by an amount not less than one third the difference between the "correction temperature" and 75 F (24 C). In case there is a dispute between the purchaser and the seller, the observed initial boiling point figures obtained by all parties shall be revised according to the following formula:

$$R = O - \frac{\text{Correction Temperature (deg Fahr)} - 75}{3}$$

where $R =$ the revised initial boiling point, and
$O =$ the observed initial boiling point.

If all the revised figures fall within the specification limit the gasoline in question shall be considered as passing; otherwise arrangements shall, if possible, be made to conduct a distillation test with the room temperature maintained between the limits of 70 and 80 F (21 and 27 C), inclusive.

reach the end point exceeds 5 min. The *end point* is the maximum temperature observed on the distillation thermometer and is usually reached after the bottom of the flask has become dry (Note 4). If the bottom of the flask is not dry the operator shall record this fact.

Note 4. When distilling certain naphthas manufactured for specific purposes, such as for use in paints and varnishes, the temperature indicated by the distillation thermometer when the last drop evaporates from the lowest point in the flask shall be recorded as the *dry point.* Any liquid hanging to the side of the flask or the thermometer should be disregarded.

(d) In case the time required to bring over the last 5 ml of distillate and reach the end point exceeds 5 min the test shall be repeated and the heat shall be adjusted when the liquid residue reaches 5 ml. This adjustment may be either an increase or a decrease but must accomplish the purpose of bringing the period required to vaporize the last 5 ml of distillate and reach the end point within the limits of 3 and 5 min.

Table 17 Values of the Constants "A" and "B" Used in Obtaining Corrected Distillation Loss

Observed Barometric Pressure, mm	A	B
560	0.231	0.384
570	0.240	0.380
580	0.250	0.375
590	0.261	0.369
600	0.273	0.363
610	0.286	0.357
620	0.300	0.350
630	0.316	0.342
640	0.333	0.333
650	0.353	0.323
660	0.375	0.312
670	0.400	0.300
680	0.428	0.286
690	0.461	0.269
700	0.500	0.250
710	0.545	0.227
720	0.600	0.200
730	0.667	0.166
740	0.750	0.125
750	0.857	0.071
760	1.000	0.000

(e) The total volume of the distillate collected in the receiving graduate shall be observed at the conclusion of the distillation, and thereafter at not less than 2-min intervals, until successive observations agree. This volume shall then be recorded as the *recovery.*

(f) The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 ml, measured when cool and the volume recorded as *residue.*

(g) The sum of the recovery and the residue shall be calculated and recorded as *total recovery.*

(h) The difference between 100 ml and the total recovery shall be recorded as *distillation loss* (Note 5).

Note 5. Abnormal distillation losses, when caused by low barometric pressure, may be corrected by the equation given below to an approximation of the loss to be expected under normal pressure. The corrected losses shall not be used in the calculation of percentages evaporated.

$$\text{Corrected loss} = AL + B$$

where

L = observed distillation loss, and
A and B = empirical constants, as given in Table 17 for various values of the observed barometric pressure.

(i) The sum of the volume collected in the cylinder at any specified temperature, including the initial boiling point, and the distillation loss may be recorded as the percentage evaporated at the temperature in question. Clear distinction shall be made between the inclusion or exclusion of the distillation loss figure in the report of the volume of distillate obtained at any prescribed temperature. In the first case the figure shall be reported as the volume *evaporated,* in the second, as the volume *recovered.*

Correction of Temperature Points to Standard Atmospheric Pressure

144 The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm (29.92 in.), by the use of the Sydney Young equation, as follows:

For Centrigrade readings:

$$C_c = 0.00012\ (760 - P)\ (273 + t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760 - P)\ (460 + t_f)$$

where C_c and C_f = the corrections to be added to the observed temperature t_c or t_f respectively, and
P = the actual barometric pressure in millimeters of mercury.

Table 18 is a convenient approximation of the corrections as calculated by the above equations for those cases where the temperature for a specified volume is required.

Precision

145 The following criteria, based on 95 per cent probability, should be used for judging the acceptability of results:

(a) Duplicate results obtained by the same operator and apparatus should be considered suspect if they differ by more than the repeatability which, according to Fig. 30B, is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.

(b) The results obtained by each of two laboratories should be considered suspect if they differ by more than the reproducibility which, according to Fig. 30B is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.

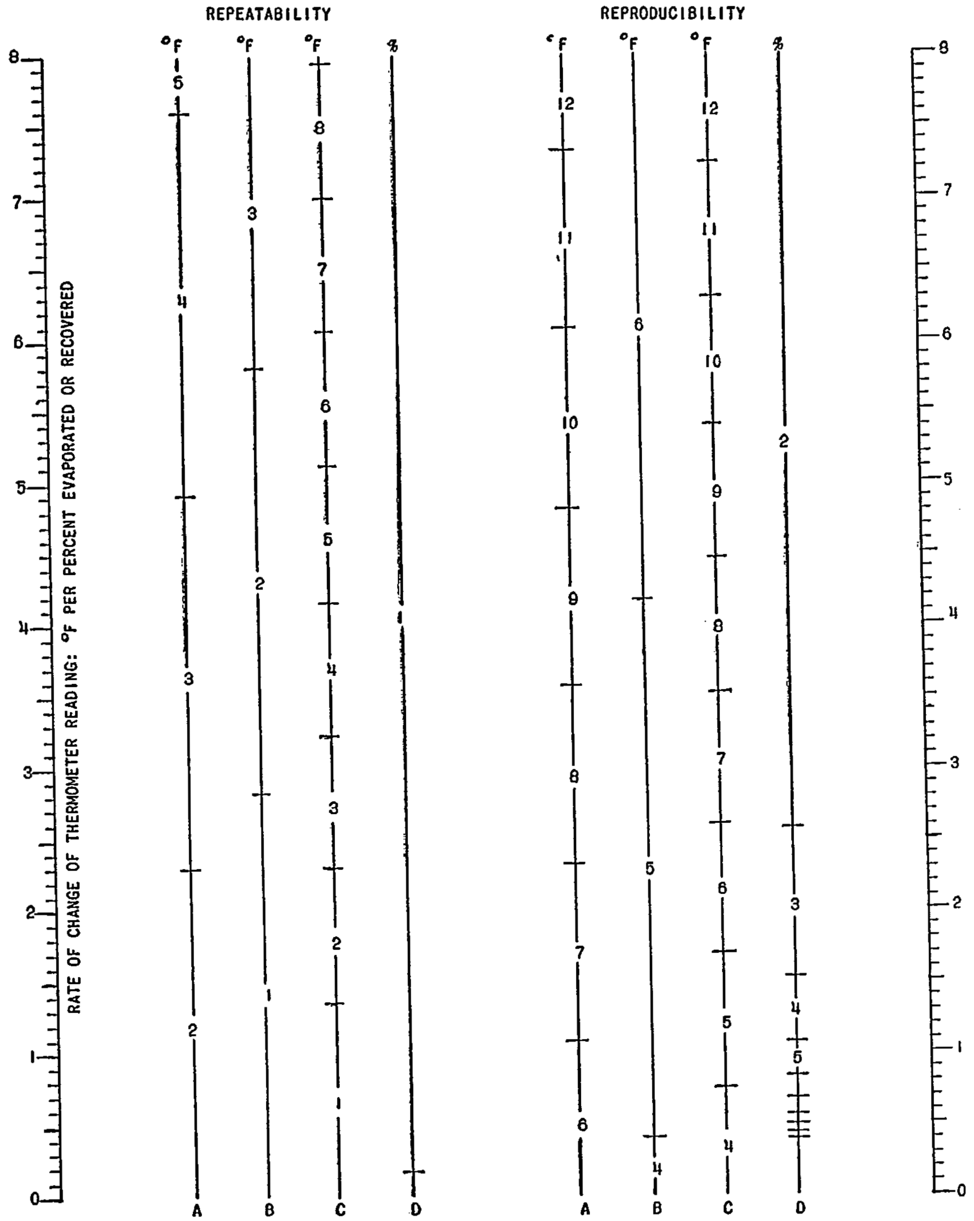
(c) To facilitate the use of Fig. 30B, the rate of change in thermometer reading in degrees Fahrenheit per the percentage evaporated or recovered, at any point between the initial boiling point and the end point or dry point, should be

Table 18

Temperature Range		Correction[a] per 10-mm Difference in Pressure	
deg C	deg F	deg C	deg F
10 to 30	50 to 86	0.35	0.63
30 to 50	86 to 122	0.38	0.68
50 to 70	122 to 158	0.40	0.72
70 to 90	158 to 194	0.42	0.76
90 to 110	194 to 230	0.45	0.81
110 to 130	230 to 266	0.47	0.85
130 to 150	266 to 302	0.50	0.89
150 to 170	302 to 338	0.52	0.94
170 to 190	338 to 374	0.54	0.98
190 to 210	374 to 410	0.57	1.02
210 to 230	410 to 446	0.59	1.06
230 to 250	446 to 482	0.62	1.11
250 to 270	482 to 518	0.64	1.15
270 to 290	518 to 554	0.66	1.19
290 to 310	554 to 590	0.69	1.24
310 to 330	590 to 626	0.71	1.28
330 to 350	626 to 662	0.74	1.32
350 to 370	662 to 698	0.76	1.37
370 to 390	698 to 734	0.78	1.41
390 to 410	734 to 770	0.81	1.45

[a] To be added in case barometric pressure is below 760 mm; to be subtracted in case barometric pressure is above 760 mm.

assumed to be the same as the average rate between two data points which are equidistant above and below the point in question, provided that the span from the point in question to either of the other data points does not represent more than 10 per cent evaporated or recovered in any case, nor more than 5 per cent if the point in question is not included in the 10 to 90 per cent range. For the initial boiling point, end point, or dry point, the rate of change should be assumed to be the same as the average rate over an interval, not to exceed 5 per cent evaporated or recovered, between the extreme point and the next data point above or below it.



A. INITIAL BOILING POINT
B. END POINT OR DRY POINT
C. THERMOMETER READING AT PRESCRIBED PERCENT EVAPORATED OR RECOVERED
D. PERCENT EVAPORATED OR RECOVERED AT PRESCRIBED THERMOMETER READING

Fig. 30B Precision of Distillation Method D 86

Standard Method of Test for Distillation of Gas Oil and Similar Distillation Fuel Oils

ASTM Designation: D 158-54

Adopted 1928; Revised 1938, 1941, 1952, 1953, 1954

This Method has been approved as "American Standard" by the American Standards Association

Scope

146 This method of test is intended for use in the distillation of liquid petroleum distillates having viscosities intermediate between kerosine and lubricating oil.

Apparatus

147 The apparatus shall consist of the following:

(a) **Flask.** A Saybolt distilling flask conforming to the following dimensions and permissible variations:

	Centimeters	Inches	Permissible Variations	
			Centimeters	Inches
Diameter of bulb, outside	8.57	3.37	±0.3	±0.12
Diameter of neck, inside	1.50	0.59	±0.1	±0.04
Over-all length, neck plus bulb	21.50	8.46	±0.3	±0.12
Length of vapor tube	10.0	3.94	±0.3	±0.12
Diameter of vapor tube, outside	0.60	0.24	±0.05	±0.02
Diameter of vapor tube, inside	0.40	0.16	±0.05	±0.02
Thickness of vapor tube wall	0.10	0.04	±0.015	±0.006

The center of the vapor tube shall be 6.5 ± 0.3 cm (2.56 ± 0.12 in.), below the top of the neck. The tube shall be approximately in the middle of the neck and set at an angle of 75 ± 3 deg with the vertical.

(b) **Condenser.** A condenser $\frac{9}{16}$ in. (14.29 mm) in outside diameter, with 0.031- to 0.036-in. wall thickness, made of seamless brass tubing, 22 in. (55.88 cm) in length. It shall be set so that approximately 15.5 in. (39.4 cm) of the tube will be in contact with the cooling medium contained in the condenser jacket, subsequently described, with about 2 in. outside the cooling bath at the upper end, and 4.5 in. outside the cooling bath at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75 deg with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.26 in. per linear inch of condenser tube (sine of angle of 15 deg) and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. nor more than 0.28 in. per linear inch of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to 1¼ in. (2.54 to 3.18 cm) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle. The capacity of the cooling bath shall be not less than 340 cu in. (5.55 liters) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than 1¼ in. below the plane of the top of the bath at its point of entrance and not less than ¾ in. above the floor of the bath at its exit. Clearances between the condenser tube and the walls of the bath shall be at least ½ in. except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 cu in. per tube.

(c) **Shield.** Two types of shields can be used as follows:

Type 1 Shield. A shield 19 in. high, 11 in. long and 8 in. wide, made of sheet metal approximately No. 22 gage, with a door on one narrow side, two openings 1 in. in diameter equally spaced in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be 8½ in. below the top of the shield. There shall be three ½-in. holes in each of the four sides with their centers 1 in. above the base of the shield.

Type 2 Shield. A shield 17½ in. high, 8 in. long and 8 in. wide made of sheet metal of approximately No. 22 gage, with window on front side. Open bottom of shield spaced approximately 2 in. from base of unit. Rear of shield to have eliptical hole for vapor tube. A flask adjusting knob located on the front of the shield for adjusting the flask support. Also heat adjusting indicating dial to provide stepless heat control when electric heater is used. All mechanism built into bottom portion of shield.

(d) **Flask Support and Ceramic or Asbestos Boards.** A ring support of the ordinary laboratory type, 4 in. or larger in diameter, supported on a stand inside the shield, or a flask support platform that is adjustable from the outside of the shield may be used. Two ceramic or hard asbestos boards, the first having a hole 2¾ in. in diameter in the center and the thickness of the board at the 2¾-in. diameter opening shall be ⅛ to ¼ in.; the second board to fit tightly inside the shield with an opening 3 to 4 in. in diameter, concentric with the flask support. These shall be arranged as follows: the second board shall be placed on the support, and the first or smaller board on top so it may be moved in accordance with the directions for placing the distillation flask. Direct heat shall be applied to the flask only through the 2¾-in. diameter opening in the first board.

(e) **Gas Burner.** A burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in Par. 149. The flame shall never be so large that it spreads over a circle of diameter greater than 4½ in. (11.43 cm) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

(f) **Electric Heater.** An electric heater may be used instead of a gas burner and shall be capable of bringing over the first drop within the time specified in Par. 148 (d) when started cold, and of continuing the distillation at a uniform rate. (A heater unit of low-heat capacity, adjustable from 0 to 750 w, has been found satisfactory.) The electric heater shall be fitted with a ceramic or hard asbestos board top having a hole 2¾ in. in diameter in the center, and the thickness of the board at the 2¾-in. in diameter opening shall be ⅛ to ¼ in. When an electric heater is employed, the portion of the shield above the board shall be the same as with the gas burner, but the part below the board may be omitted.

(g) **Thermometer.** An ASTM High Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of —2 to 400 C or 30 to 760 F and conforming to the requirements for thermometer 8 C or 8 F, respectively, as prescribed in the Standard Specifications for ASTM Thermometers (ASTM Designation: E 1) (Note 1).

NOTE 1. Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for The Institute of Petroleum Thermometer "IP 6 C High Distillation" may be used, provided calibration corrections are used in temperature ranges where IP requirements for scale accuracy are less stringent than those of ASTM.

(h) **Graduate for Sample.** A graduate or pipet graduated to deliver 200 ± 1.0 ml, for measuring the sample to be tested.

(i) **Graduates for Fractions Taken on Temperature Basis.** Graduates of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. They shall be graduated to contain 100 ml and the graduated portion shall be not less than 7 in. (17.78 cm) nor more than 8 in. (20.32 cm) in length. They shall be graduated in single milliliters and each fifth mark shall be distinguished by a longer line. They shall be numbered from the bottom at intervals of 10 ml. The over-all height of the graduates shall be not less than 9¾ in. (24.8 cm) nor more than 10¼ in. (26.0 cm). The graduations shall not be in error by more than 1 ml at any point on the scale.

(j) **Graduates for 10 Per Cent Fractions.** Graduates of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. They shall be graduated to contain 25 ml and the graduated portion shall be not less than 3¼ in. (8.25 cm) nor more than 3¾ in. (9.52 cm) in length. They shall be graduated in half milliliters and each tenth mark (indicating multiples of 5 ml) shall be distinguished by a longer line. They shall be numbered from the bottom at intervals of 5 ml. The over-all height of the graduates shall be not less than 5⅜ in. (13.65 cm) nor more than 5⅝ in. (14.29 cm). The graduations shall not be in error by more than 0.25 ml at any point on the scale.

Preparation of Apparatus

148 (a) Provision shall be made for circulating water through the condenser.

(b) Swab the condenser tube to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(c) Place a volume of water-free oil equivalent to 200 ml at 55 to 65 F (12.8 to 18.3 C) in the flask.

(d) Fit the thermometer, provided with a cork, tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(e) Place the charged flask over the 2¾-in. (6.99-cm) opening in the asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. Adjust the position of the flask so that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm) nor more than 2 in. (5.08 cm).

(f) Place a clean, dry graduated cylinder (see Par. 147 (i) and (j) at the outlet of the condenser tube in such position that the condenser tube shall extend into the graduate approximately 1 in. (2.54 cm) but not below the upper graduation mark.

Procedure

149 (a) When everything is in readiness, circulate water through the condenser bath at such a rate that the water overflowing the condenser shall be at a temperature of 90 to 100 F (32.2 to 37.8 C). Apply heat to the contents of the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 min. When the first drop falls from the end of the condenser, record the reading of the distillation thermometer as the *initial boiling point.* Move the receiving cylinder so that the end of the condenser tube shall touch the side of the cylinder. Then regulate the heat so that the distillation will proceed at a uniform rate of not less than 8 nor more than 10 ml per min. Separate a fraction at every temperature point above the initial boiling point that is a multiple of 50 F. In case the product is being tested to determine its conformity to specifications involving limits that are not multiples of 50 F, any readings may be taken, or fractions separated, which are called for by the specification requirements. If desired, 20-ml (10 per cent) fractions may be separated and readings made on the thermometer just as each graduate is withdrawn.

(b) When the temperature of the distilling vapors reaches 500 F (260 C), adjust the temperature of the water in the condenser bath to approximately 140 F (60 C) and the bath maintained between 140 and 160 F (60 to 71.1 C) for the remainder of the test. Continue the distillation until a vapor temperature of 700 F (371.1 C) is reached or until the maximum temperature point is reached. The maximum temperature point may result from complete distillation of the oil below 700 F (371.1 C), or from cracking (Note 2).

NOTE 2. Cracking will be evidenced by an increase in distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. When this condition is observed, it is advisable to discontinue the distillation.

(c) In case the oil distills completely below 700 F (371.1 C), make the final adjustment of heat when a quantity of distillate amounting to 90 per cent of the sample has been collected. Continue the heating until the thermometer reaches a maximum reading and starts to fall consistently; but in no case should the period between the 90 per cent point and the maximum temperature be more than 10 min. Record the highest temperature observed on the distillation thermometer as the *maximum temperature.* Allow sufficient time after distillation is completed for any distillate in the condenser to drain into the final fraction. Bring each fraction to room temperature, read and record the volume. Calculate and record the percentage of distillate in each fraction. Calculate and record the difference between 100 and the sum of the distillate percentages as *residue and loss.*

TABLE 19

Temperature Range		Correction[a] per 10-mm Difference in Pressure	
deg C	deg F	deg C	deg F
10 to 30	50 to 86	0.35	0.63
30 to 50	86 to 122	0.38	0.68
50 to 70	122 to 158	0.40	0.72
70 to 90	158 to 194	0.42	0.76
90 to 110	194 to 230	0.45	0.81
110 to 130	230 to 266	0.47	0.85
130 to 150	266 to 302	0.50	0.89
150 to 170	302 to 338	0.52	0.94
170 to 190	338 to 374	0.54	0.98
190 to 210	374 to 410	0.57	1.02
210 to 230	410 to 446	0.59	1.06
230 to 250	446 to 482	0.62	1.11
250 to 270	482 to 518	0.64	1.15
270 to 290	518 to 554	0.66	1.19
290 to 310	554 to 590	0.69	1.24
310 to 330	590 to 626	0.71	1.28
330 to 350	626 to 662	0.74	1.32
350 to 370	662 to 698	0.76	1.37
370 to 390	698 to 734	0.78	1.41
390 to 410	734 to 770	0.81	1.45

[a] To be added in case barometric pressure is below 760 mm; to be subtracted in case barometric pressure is above 760 mm.

Reproducibility of Results

150 With proper care and strict attention to details, duplicate determinations by the same operator for end point should not differ from each other by more than 6 F (3.3 C) and differences in volumes of fractions separated at the various temperature limits should not exceed 4 ml. When temperatures are observed for specified percentages distilled the differences should not exceed 4 F (2.2 C). When duplicate determinations are made by different operators normal differences in observations are one and a half times those which are expected with a single operator.

Correction for Barometric Pressure

151 When it is agreed to make allowance for the effect of barometric pressure, correct the temperature points to 760 mm (29.92 in.), by the use of the Sydney Young equation as follows:

For Centigrade readings:

$$C_o = 0.00012\ (760 - P)\ (273 + t_o)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760 - P)\ (460 + t_f)$$

where:

C_o and C_f = the correction to be added to the observed temperature t_o or t_f, respectively, and

P = actual barometric pressure in millimeters of mercury.

Table 19 is a convenient approximation of the corrections as calculated by the above equations for those cases where the temperature for a specified volume is required.

Flash Point

152 An element of danger incident to the handling and storage of Diesel and burner fuels is the explosive ignition of vapors in the unfilled portions of tanks, drums, or other containers. A flash-point test in which a determination is made of the temperature to which oil must be heated to give off enough vapor to create an explosive mixture in a closed system is designed to indicate the degree of danger from the oil and the temperature at which the produce may be stored or handled without serious fire hazard.

In addition, the flash point may serve as a guide to determine if a fuel can be ignited from an open spark as in domestic oil burners. It can be used, in some cases, to differentiate between heavy grades of fuel oil. The flash point is not a reliable measure of the volatility, ignition characteristics, or burning qualities of an oil.

For the purpose of this code, the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester, ASTM Designation: D 93-52, is adopted.

It should be noted that to detect explosive hazards in confined spaces from the evaporation of fuel oil, a number of analyzers or combustible gas indications are available, such as: the MSA Explosimeter, MSA Combustible Gas Indicator, the J. W. Combustible Gas Indicator, the Davis Vapotester, and the G-E Leak Detector. In the first four of these instruments, a sample is drawn into the apparatus and catalytically burned on a hot wire. The heat of combustion changes the resistance of the wire and is used to measure the combustible content of the sample. The G-E Detector works on the principle of the absorption of ultra-violet light.

Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester

ASTM Designation: D 93-52

Adopted 1922; Revised 1936, 1940, 1942, 1946, 1952

This Method has been approved as "American Standard" by the American Standards Association

NOTE. Extensive editorial changes in Pars. 155, 156, 157, and 160 were made in July, 1954.

Scope

153 (a) This method of test is intended for determining the flash point of fuel oils.

(b) Provision is made in Pars. 159 to 161 of this method for determining the flash point of cut-back asphalts and other viscous materials and suspensions of solids.

Pensky-Martens Tester

154 The ASTM Pensky-Martens tester, a diagram of which appears in Fig. 31, shall include the following major parts:

(a) **Cup.** The cup shall be made of brass and shall conform to the dimensional requirements prescribed in Table 20. The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange but the wall thickness at the upper edge shall be not less than 0.04 in. (0.102 cm). The flange should be approximately 0.5 in. (1.27 cm) in width and approximately 0.125 in. (0.318 cm) in thickness. It shall be equipped with devices for locating the position of the lid on the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory. There may be a fillet approximately $\frac{5}{32}$ in. (0.397 cm) in radius inside the bottom of the cup.

(b) **Lid:**

(1) STIRRING DEVICE. The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0.1 in. (0.254 cm) nor more than 0.125 in. (0.318 cm) in diameter, mounted in the center of the cup, and carrying two two-bladed brass propellers. The blades of both propellers shall be approximately 0.313 in. (0.795 cm) wide and

TABLE 20 DIMENSIONAL REQUIREMENTS OF PENSKY-MARTENS FLASH TESTER

	Inches			Centimeters		
	Min	Normal	Max	Min	Normal	Max
Inside diameter below filling mark	1.950	2.000	2.050	4.953	5.080	5.207
Difference, inside and outside diameters below filling mark	0.120	0.125	0.130	0.305	0.318	0.330
Inside height	2.150	2.200	2.250	5.461	5.588	5.715
Thickness of bottom	0.070	0.095	0.120	0.178	0.241	0.305
Distance from rim to filling mark	0.845	0.860	0.875	2.146	2.184	2.223
Distance lower surface flange to bottom of cup	1.780	1.795	1.810	4.521	4.559	4.597

shall be set at an angle of approximately 45 deg. The smaller (upper) propeller shall have an over-all diameter of approximately 0.75 in. (1.905 cm). The larger (lower) propeller shall have an over-all diameter between 1.25 and 1.75 in. (3.175 and 4.445 cm). The thickness of the propeller blades shall be not less than 0.057 in. (0.145 cm) nor more than 0.081 in. (0.206 cm), which limits correspond respectively to No. 15 and No. 12 B & S gage sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0.4 in. (1.016 cm).

The plane of the center of the upper propeller shall be 0.4 in. (1.016 cm) below the level of the rim of the cup. The plane of the center of the lower propeller shall be 2.0 in. (5.08 cm) below the level of the rim of the cup. The level of the rim of the cup is, in effect, the level of the plane part of the portion of the lower surface of the lid inside the rim.

(2) COVER PROPER. The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim, shall be not less than 0.031 in. (0.079 cm) nor more than 0.078 in. (0.198 cm). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

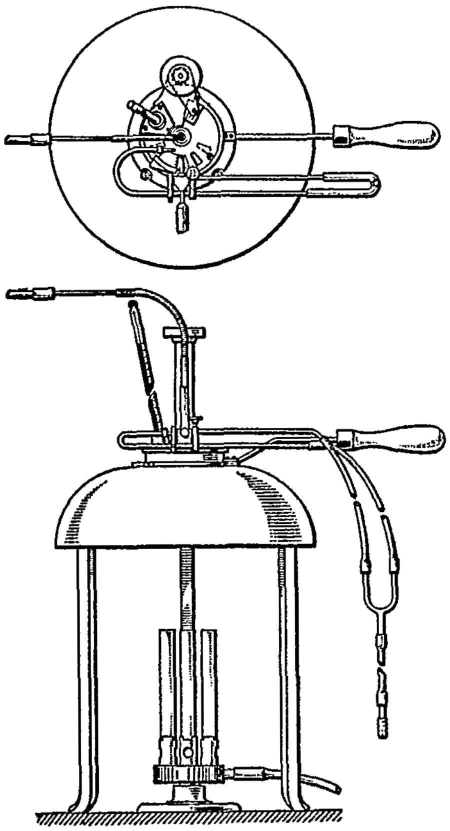
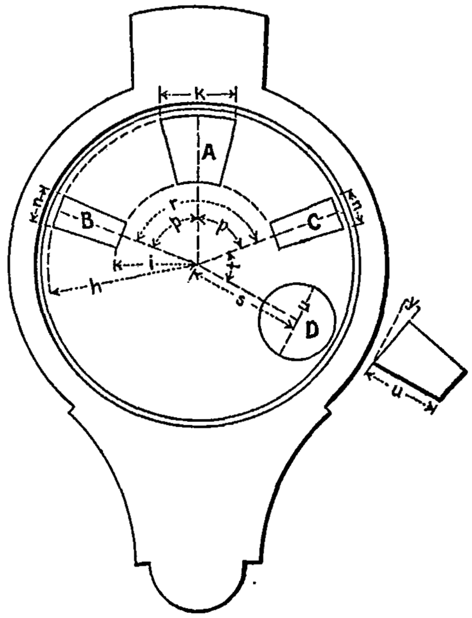


FIG. 31 ASTM STANDARD PENSKY-MARTENS TESTER

There shall be four openings in the cover, as indicated in Fig. 32.



h	0.938 to 0.969 in.
i	0.531 to 0.563 in.
k	0.500 to 0.540 in.
n	0.187 to 0.219 in.
s	approximately 0.75 in.
u	approximately 0.5 in.
Angles *p*	equal
Angle *r*	135 to 140 deg
Angle *t*	50 to 60 deg
Angle *y*	10 to 15 deg

FIG. 32 COVER FOR ASTM STANDARD PENSKY-MARTENS TESTER

Opening *A* is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0.938 in. (2.383 cm) nor more than 0.969 in. (2.461 cm). The radius of the inner circle shall be not less than 0.531 in. (1.349 cm) nor more than 0.563 in. (1.430 cm). The chord of the arc of the outer circle shall be not less than 0.500 in. (1.270 cm) nor more than 0.540 in. (1.372 cm).

Openings *B* and *C* are equal areas, each of the same general form as opening *A* but of approximately half the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partly define opening *A*. The chord of the outer arc for opening *B* or opening *C* shall be not less than 0.187 in. (0.475 cm) nor more than 0.219 in. (0.556 cm). The sum of the areas of openings *B* and *C* shall be not less than 75 per cent nor more than 100 per cent of the area of opening *A*. Openings *B* and *C* shall be equally distant from opening *A* and radii drawn through each of their centers shall be at an angle of not less than 135 deg nor more than 140 deg.

Openings *A, B,* and *C* need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of Fig. 32.

Opening *D* is for a thermometer collar. Its center is approximately 0.75 in. (1.905 cm) from the center of the lid and on a radius at an angle of not less than 50 deg nor more than 60 deg from a radius passing through the center of opening *C*. The thermometer collar shall have an inside diameter of approximately 0.5 in. (1.27 cm). It shall be set at an angle of not less than 10 deg nor more than 15 deg from the perpendicular.

(3) Shutter. The lid shall be equipped with a brass shutter, approximately 0.094 in. (0.239 cm) in thickness, operating on the plane of the upper surface of the lid. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the lid between two stops so placed that when in one extreme position the openings *A, B,* and *C* of the lid are completely closed and when in the other extreme position these orifices are completely opened.

(4) Flame Exposure Device. The flame exposure device shall have a tip with an opening 0.027 in. (0.069 cm) to 0.031 in. (0.079 cm) in diameter. The flame exposure device (Note 1) shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper at a point on a radius passing through the center of the larger opening *A* and approximately 0.1 in. (0.254 cm) from the outer edge of the opening.

Note 1. A pilot flame for automatic relighting of the exposure flame should be provided.

A bead 0.156 in. (0.398 cm) in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

(c) **Stove.** Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests.

(1) Air Bath. The air bath shall have a cylindrical interior 1.625 in. (4.128 cm) to 1.656 in (4.206 cm) in depth and a diameter not less than 0.125 in. (0.317 cm) nor more than 0.156 in. (0.396 cm) greater than the outside diameter of the cup. The air bath may be either a flame-heated metal casting (Note 2) or an electric-resistance element (Note 3).

Note 2. If the heating element is a flame-heated metal casting, it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0.25 in. (0.635 cm) in thickness. The casting shall be designed so that products of combustion of the flame cannot pass up and in contact with the cup.

Note 3. If the air bath is of the electric-resistance type, it shall be constructed so that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

(2) Top Plate. The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to the bottom of the cup by not less than 0.063 in. (0.160 cm) nor more than 0.125 in. (0.317 cm).

The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing bushings. The spacing bushings should be of proper thickness to define the air gap which shall be not less than 0.125 in. (0.317 cm) nor more than 0.187 in. (0.475 cm). The spacing bushings shall be not more than 0.375 in. (0.952 cm) in diameter.

(d) **Thermometers.** Two standard thermometers shall be used with the ASTM Pensky-Martens tester.

For tests in which the indicated reading falls within the limits 20 to 200 F, a low-range ASTM PM and Tag Thermometer having a range of −7 to +110 C or +20 to +230 F and conforming to the requirements for thermometers 9 C or 9 F as prescribed in ASTM Specifications E 1 shall be used (Note 4).

For tests in which the indicated reading falls within the limits 230 to 700 F, a high-range ASTM PM High Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 90 to 370 C or 200 to 700 F and conforming to the requirements for thermometers 10 C or 10 F as prescribed in Specifications E 1 shall be used.

For the range 200 to 230 F, either thermometer may be employed, depending upon the convenience of the operator.

Thermometers shall be mounted so that the bottom of the bulb is 1.75 in. (4.445 cm) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim).

Note 4. Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for The Institute of Petroleum thermometer IP 15 F (or 15 C) PM-Low, or IP 16 F (or 16 C) PM-High, may be used, provided calibration corrections are used in temperature ranges where the IP requirements for scale accuracy are less stringent than those of ASTM.

Procedure

155 (a) Thoroughly clean and dry all parts of the cup and its accessories before starting the test. Take particular care to avoid the presence of any gasoline or naphtha used to clean the apparatus after a previous test.

(b) Fill the cup with the oil to be tested up to the level indicated by the filling mark.

(c) Place the lid on the top and set the latter in the stove. Take care to have the locating devices properly engaged. Insert the thermometer. If it is known that the toll will flash above 220 F, the ASTM PM High Thermometer may be selected; otherwise, it is preferable to start with the ASTM PM and Tag Thermometer and change in case a temperature of 220 to 230 F is reached.

(d) Light the test flame and adjust it, making it the size of a bead $\frac{5}{32}$ in. (3.97 mm) in diameter.

(e) Supply heat at such a rate that the temperature read on the thermometer increases not less than 9 nor more than 11 F per min. Turn the stirrer at a rate of from 1 to 2 revolutions per second.

(f) Apply the test flame at each temperature reading that is a multiple of 2 F up to 220 F. For the temperature range above 220 F, apply the flame at each temperature reading that is a multiple of 5 F. Make the first application of the test flame at a temperature at least 30 F below the actual flash point. Apply the test flame by operating the device controlling the shutter and test flame burner so that the flame is lowered in 0.5 sec, left in its lowered position for 1 sec, and quickly raised to its high position. Discontinue stirring while applying the test flame.

Flash Point

156 Record as the flash point the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. Do not confuse the true flash with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

Barometric Pressure

157 Observe and record the barometric pressure. No corrections shall be made except in case of dispute, when the flash point figure shall be corrected according to the following rule:

For each inch (25 mm) below 29.92 in. (760 mm) barometric reading, add 1.6 F to the flash point.

For each inch (25 mm) above 29.92 in. (760 mm) barometric reading, subtract 1.6 F from the flash point.

Precision

158 Results shall not differ from each other by more than the following:

Flash Point, deg F	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
200 and under	2 F	4 F
Over 200	5 F	10 F

DETERMINATION OF FLASH POINT OF CUT-BACK ASPHALTS AND OTHER VISCOUS MATERIALS AND SUSPENSIONS OF SOLIDS

Apparatus

159 The apparatus shall consist of the following:

(a) **Flash Point Tester.** Pensky-Martens tester as described in Par. 154 (a), (b), and (c), except that the stirrer shall be mechanically operated to stir in a downward direction at a speed of 70 to 80 rpm.

(b) **Thermometer.** Low-range thermometer as specified in Par. 154 (d) (Note 5).

Procedure

160 Bring the material to be tested and the tester to a temperature 30 F lower than the estimated flash point. Completely fill the air space between the cup and the interior of the air bath with water at the temperature of the tester and material. Raise the temperature throughout the duration of the test at a rate of not less than 2 nor more than 3 F per min.

NOTE 5. Solid carbon dioxide (Dry Ice) shall, in no case, be used to obtain the proper rate of temperature rise, since CO_2 has a blanketing effect which leads to a false flash point.

The following paragraphs are from Standard Method of Test for Flash Point by Tag Closed Tester (ASTM Designation: D 56).

Initial Test

161 (a) Place the test cup in position in the liquid bath and measure 50 ml of sample into it, using an accurate graduate or other measuring device for the purpose. The temperature of the sample shall be such that, when "thermal equilibrium" (Note 6) has been established, the reading of the sample cup thermometer is at least 16 F below the expected flash point. When the expected flash point is at or below room temperature, the oil may need to be cooled sufficiently to compensate for the heat which will be absorbed from the metal of the apparatus. Destroy air bubbles on the surface of the sample. Wipe the inside of the cover with a clean cloth or absorbent tissue paper, then attach the lid to the bath collar. Insert the thermometer.

(b) Record the equilibrium temperature (Note 6) of the sample and record the barometric pressure.

(c) Light the test flame, adjusting it to the size of the small white bead on the cover. Adjust the heat so that the temperature of the sample in the test cup rises at the rate of 1.8 ± 0.2 F per min.

(d) When the temperature of the sample in the test cup is 9 F below its expected flash point, operate the mechanism on the cover in such a manner as to introduce the test flame into the vapor space of the cup, and immediately bring it up again. The time consumed for the full operation shall be about 1 sec, or the time required to pronounce distinctly the words "thousand and one." Avoid any jerkiness in the operation of depressing and raising the test flame. Record the time at which the first introduction of the test flame is made and the temperature of the sample at that time.

NOTE 6. "Thermal equilibrium" is considered to exist when the difference between the readings of the bath and sample-cup thermometers has been nearly constant for at least 2 min with the bath liquid temperature slightly higher than the sample temperature.

(e) Repeat the application of the test flame after each 1 F rise in temperature of the sample in the test cup until a distinct flash in the interior of the cup is observed. Do not confuse the true flash with the bluish halo which sometimes surrounds the test flame during applications immediately preceding the actual flash. Record the time and the temperature of the sample when the flash point is reached.

(f) If, at any time between the first introduction of the test flame and the observation of the flash point, the rise in temperature is more rapid than 2 F per min or slower than 1.6 F per min, repeat the test, adjusting the source of heat to obtain the proper rate of temperature rise.

Repeat Test

162 (a) After completing the initial test, temporarily remove the source of heat, lift the lid, and carefully wipe off the thermometer bulb (Note 7). Remove the sample cup, empty, and carefully wipe dry.

NOTE 7. It is not necessary to turn off the test flame during this change; it may be left adjusted to the proper size of flame.

(b) Lower the temperature of the bath by the addition of cold water (or by other suitable means if a bath liquid other than water is used) until its "equilibrium temperature" (Note 6) is at least 16 F below the flash point of the sample found by the initial test.

(c) Replace the sample cup and measure into it a fresh 50-ml sample, suitably precooled if necessary. Repeat the test procedure (Note 8) as described in Par. 161, introducing the test flame for the first time, however, when the sample temperature is 10 F below the flash point obtained by the initial test.

NOTE 8. Never make a repeat test on the same portion of sample once used; always take a fresh portion of sample for each test.

Average Value of Flash Point

163 Discard the result of the initial test if it is lower than the estimated probable flash. Report as the flash point, so indicating if corrected for barometric pressure, the following:

(*1*) The average of the first two test results if they do not differ by more than 1 F, or

(*2*) The average of the first three test results if the difference between extremes is not more than 2 F, or

(*3*) The average of the first five test results, subject to the proviso that if one, and only one, of the figures varies from the average by more than 3 F, it shall be discarded and a new average calculated from the other four results.

Correction for Barometric Pressure

164 In cases of dispute, or when the barometric pressure differs from 760 mm by more than 13 mm, correct the observed flash points by means of the following equation:

$$\text{Corrected flash point} = F + 0.06\,(760 - P)$$

where:

F = observed flash point, in degrees Fahrenheit, and
P = barometric pressure, in millimeters of mercury.

Precision

165 Duplicate tests should not differ by more than 5 F.

Cetane Number

166 The ignition characteristics of a fuel oil for diesel engines are important as they govern the degree of roughness with which the engine will operate. The ignition quality may only be determined by operation of an engine of given characteristics under specified conditions; the quality is expressed as the cetane number. The cetane number requirements of an engine depend on its design, its size, the nature of the variations in speed and load, and on the starting and atmospheric conditions. For a given engine and operating conditions, the minimum cetane number of the oil required for the desired performance should be determined. The user should remember that an oil with a higher cetane number than that specified by the manufacturer will not necessarily improve the performance of the engine.

To determine the ignition characteristics of Diesel fuels, the Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method, ASTM Designation: D 613-48T, is adopted for the purposes of this code.

When equipment is not available for determining the cetane number, it may be approximated by calculation. Appendix II of ASTM Specification Designation: D 975-53T contains details of this method for obtaining a calculated Cetane Index. This method is not applicable to fuels containing additives for raising Cetane number. The formula given is:

$$\begin{aligned}\text{Calculated Cetane Index} = {} & 97.833\,(\log \text{mid-boiling point, F}) \\ & + 2.2088\,(\text{API Gravity})\,(\log \text{mid-boiling point, F}) + 0.01247\,(\text{API Gravity})^2 \\ & - 423.51\,(\log \text{mid-boiling point, F}) - 4.7808\,(\text{API Gravity}) + 419.59\end{aligned}$$

where the mid-boiling point is the temperature in degrees F for 50 per cent recovery at 760 mm of Hg as determined in ASTM Method, Designation: D 86-56. If many calculations are to be made, it will be found to be much more convenient to use a nomograph based on the formula given. Such a nomograph is included in ASTM Specification Designation: D 977-53T.

Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method

ASTM Designation: D 613-48T

Issued 1941; Revised 1943, 1947, 1948

Scope

167 This method describes the test for determining the ignition quality of Diesel fuels in terms of ASTM cetane number.

Definition

168 **ASTM Cetane Number** of a Diesel fuel is the whole number nearest the percentage by volume of normal cetane in a blend with α-methylnaphthalene that matches the ignition quality of the fuel when compared by this method.

Outline of Method

169 The cetane number of a Diesel fuel is determined by comparing its ignition quality with those for blends of reference fuels of known cetane numbers under standard operating conditions. This is done by varying the compression ratio for the sample and each reference fuel to obtain a fixed "delay period," that is the time interval between injection and ignition. When the compression ratio for the sample is bracketed between those for two reference fuel blends differing by not more than five cetane numbers, the rating of the sample is calculated by interpolation.

Apparatus

170 The ignition test unit illustrated in Fig. 33 consists of a single cylinder engine of continuously variable compression ratio, with suitable loading and accessory equipment and instruments, mounted on a stationary base. The engine and equipment specified in Supplement I* on Apparatus, shall be used without modification and installed as directed in Supplement VI* on Installation and Assembly. It is important to avoid vibration difficulties by providing a firm foundation for the unit as described in Section 506 of Supplement V* on Building and Utility Requirements. It is necessary to keep the apparatus in good mechanical condition, as described in Supplement IV on Maintenance.

Reference Fuels

171 **ASTM Cetane Reference Fuels,** conforming to the specifications and requirements in Supplement II* on Refer-

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* 1952 ASTM Manual of Engine Test Methods for Rating Fuels, American Society for Testing Materials, 1916 Race St., Philadelphia, Pa., 320 pp., $8.00.

ence Materials and Blending Accessories, are the following:

(a) ASTM *n*-cetane,
(b) ASTM *α*-methylnaphthalene.

Normally, secondary reference fuels which have been calibrated against these standards as described in Section 204 of Supplement II* are used for routine testing.

Operating Conditions

172 The following standard operating conditions (see Supplement III* on Operation, for further details) are mandatory:

(a) **Engine Speed,** 900 ± 9 rpm, with a maximum variation of 9 rpm during a test.

(b) **Injection Advance,** 13.0 deg btdc.

(c) **Injection Opening Pressure,** 1500 ± 50 psi.

(d) **Injection Quantity,** 13.0 ± 0.2 ml per min (that is, 13.0 ml in 60 ± 1 sec). Make this adjustment for each sample.

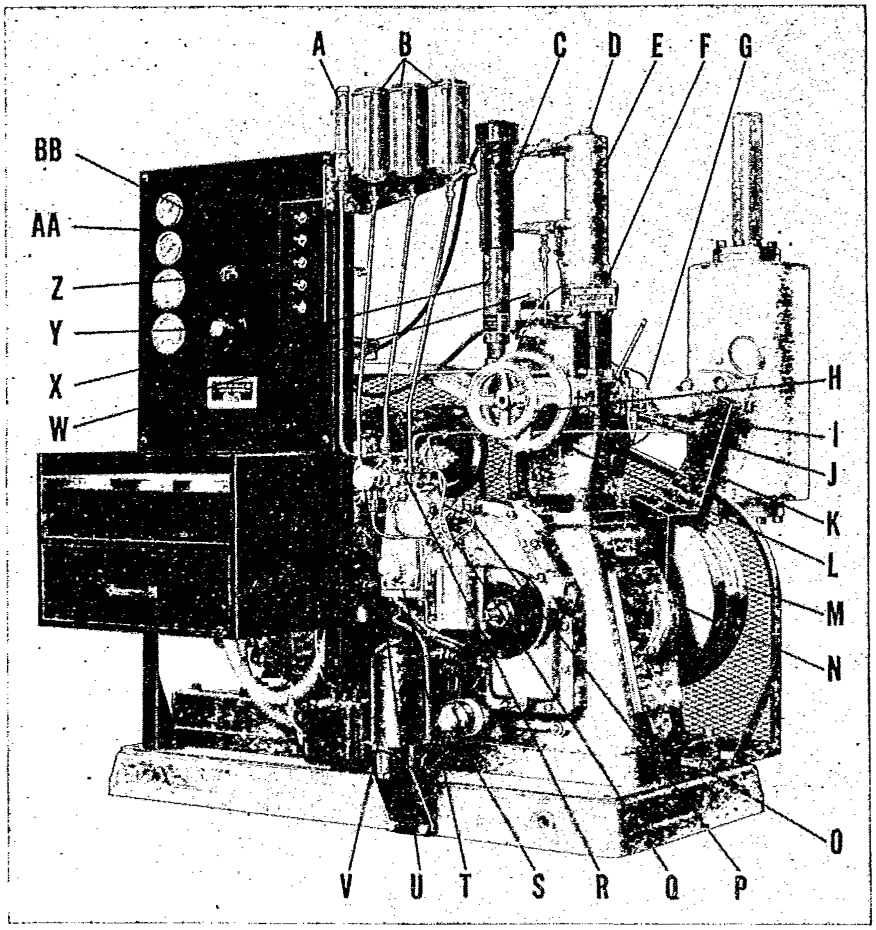
(e) **Injection Pump Setting,** such that the inlet port in the barrel of the injection pump closes when the plunger has moved 0.083 ± 0.007 in. from the base circle of the cam. See Section 447 of Supplement IV,* for checking and adjusting the setting of the injection pump.

(f) **Injector Pintle Valve Lift,** 0.005 ± 0.001 in.

(g) **Injector Water Jacket Temperature,** 100 ± 5 F.

(h) **Valve Clearances,** 0.008 ± 0.001 in., measured with the engine hot and running under standard operating conditions on a reference fuel of 50 cetane number.

(i) **Crankcase Lubricating Oil,** SAE 30, having a Saybolt Universal viscosity of 185 to 255 sec at 130 F determined by ASTM Method D 88.



A—Buret
B—Fuel Tanks
C—Air Heater Housing
D—Coolant Filler Cap
E—Coolant Condenser
F—Combustion Indicator
G—Injector Indicator
H—Locking Handwheel
I—Injector By-pass Valve
J—Water Connections for Fuel Injector
K—Sight Tube
L—Handwheel for Adjusting Compression Ratio
M—Flywheel Ring
N—Oil Filler Cap
O—Oil Drain Cap
P—Fuel Drain Valve
Q—Injector Bleed Line
R—Tank Drain Valve
S—Oil Heater Switch
T—Fuel Injection Pump
U—Fuel Selector Valve
V—Oil Filter
W—Water Heater Switch
X—Air Intake Silencer
Y—Air Heater Rheostat
Z—Field Rheostat for 115-v d-c Generator
AA—Instrument Panel
BB—Neon Indicating Switch

FIG. 33 CETANE UNIT

(j) **Oil Pressure,** 25 to 30 psi under operating conditions.

(k) **Oil Temperature,** 135 ± 15 F with the temperature sensitive element completely immersed in the crankcase oil.

(l) **Coolant Temperature,** 212 ± 3 F, constant within ± 1 F during a test.

(m) **Intake Air Temperature,** 150 ± 1 F.

(n) **Compression Ratio Adjustment,** micrometer set to read 2.000 in. (10.0 to 1 compression ratio) when the clearance volume is 72 ± 0.5 ml. Using water, this volume is measured to the seat for the combustion indicator, with the piston at top dead center, and with its over-travel past the top of the cylinder adjusted to 0.015 ± 0.001 in. by cylinder shims.

Starting the Engine

173 While the engine is being motored, open the by-pass and pump drain valves of the fuel bowls in use, long enough to purge the fuel system of entrapped air, then close the valves and increase the compression ratio until the engine fires.

Stopping the Engine

174 Open the fuel by-pass valve, turn off the neon indicators, and then stop the synchronous motor. To avoid possible corrosion and warping of the intake and exhaust valves and seats between operating periods, close both valves by turning the flywheel to tdc on the compression stroke.

Adjustment of Injection Indicator

175 The procedure for setting the injection indicator, Fig. 34, is as follows:

(a) With the engine stopped, and with no force applied to the leaf spring, *F*, by the adjusting screw, *E*, turn the center adjusting screw, *B*, counterclockwise to obtain a small gap between the contact points of the leaf springs.

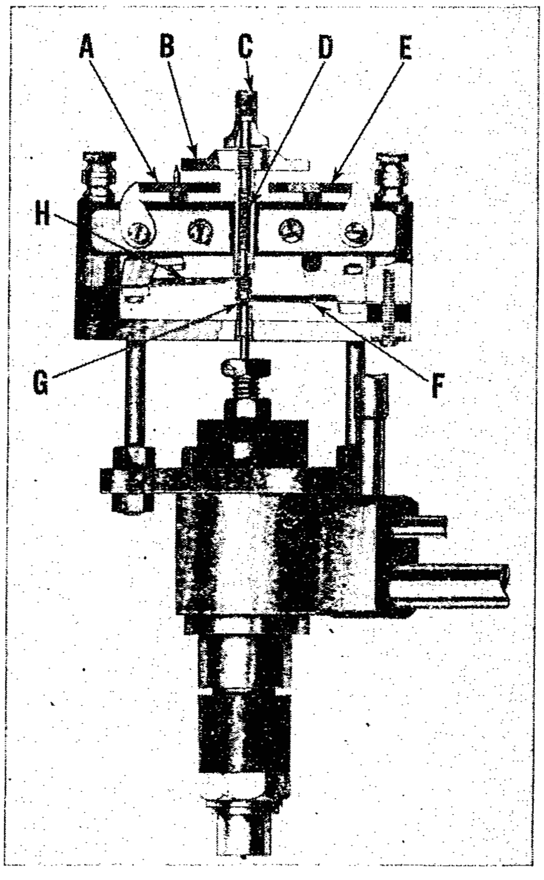
(b) Adjust the mounting bracket of the indicator until there is a clearance of about 0.030 in. between the lower leaf spring, *F*, and the injector pin, *G*.

(c) Turn the adjusting screw, *E*, clockwise until the leaf spring, *F*, just touches the injector pin, *G*. Then increase the tension by continuing the rotation of the adjusting screw one complete turn.

(d) Adjust the leaf spring, *H*, by turning its adjusting screw, *A*, clockwise until the contact points just touch. Make certain that they touch as a result of spring tension, and not because of force applied by the screw. Should the latter be the case, it is necessary to remove the spring and bend it slightly downward at the outer end. Turn the adjusting screw counterclockwise one turn from this position.

(e) Turn the adjusting screw, *C*, of the bumper spring, *D*, in the center adjusting screw, *B*, clockwise to lock it. This gives the correct bumper spring tension and permits the two screws to move as a unit.

(f) With the engine operating under standard conditions, set the fuel pump to inject 13.0 ± 0.2 ml per min. Close



A—Upper Spring Adjusting Screw
B—Center Adjusting Screw
C—Bumper Spring Adjusting Screw
D—Bumper Spring
E—Lower Spring Adjusting Screw
F—Lower Leaf Spring
G—Injector Pin
H—Upper Leaf Spring

Fig. 34 Injection Indicator

the switch for the neon indicators. Adjust the gap between the contact points by turning the dial, *B,* just enough for the injector neon tube to make a solid band of light around the flywheel. Then rotate the dial counterclockwise 2 divisions to give a gap of 0.002 in. Lock the spring by means of the clamp screws.

Adjustment of Combustion Indicator

176 (a) The basic setting of the combustion indicator, Fig. 35, is made with the engine stopped. Turn the center adjusting screw, *B,* counterclockwise to obtain a small gap between the contact points of the leaf springs. Then adjust the leaf spring, *F,* until it just touches the end of the indicator pin, *G.* Make certain that this contact is made as a result of spring tension and not because of force applied by the adjusting screw, *E.* Apply tension by rotating the adjusting screw, *E,* clockwise one complete turn. For the next steps follow the procedure described in Par. 175 (d) and (e). Then obtain a preliminary gap of about 0.010 in. between the contact points by turning the dial, *B,* clockwise.

(b) It is necessary to make the final gap setting for each test fuel. With the engine operating under standard conditions, including correct fuel quantity and injection angle, this is done by the "lingering flash" procedure, as follows:

First, adjust the compression ratio until the engine fires normally. This is at least two ratios above the lowest ratio at which the engine operates without misfiring.

Second, close the switch for the neon indicators.

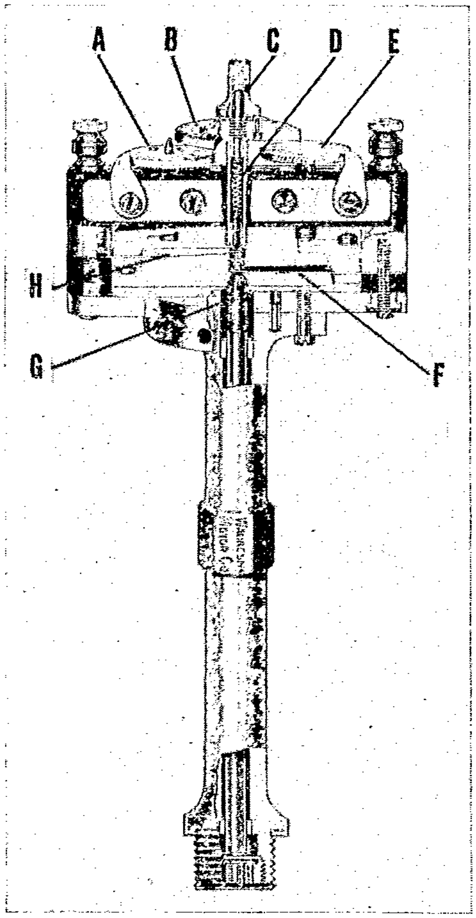
Third, adjust the injection timing at the fuel pump so that the injection flash begins at the reference line in the peering tube, *A* in Fig. 36. The injector neon tube is displaced 13 deg on the flywheel to make it flash at tdc when injection actually occurs 13 deg btdc.

Fourth, make a preliminary adjustment of the compression ratio with the handwheel, so that the combustion flash begins slightly below the reference line in the peering tube, *B* in Fig. 36. It is desirable that the combustion flash begin at this point in each of the intermediate adjustment steps so the final precise adjustment can be made by raising the compression ratio with the handwheel.

Fifth, turn the dial, *B,* so that the combustion flash lingers for one or two cycles when the by-pass valve of the injection nozzle is opened quickly. If the flash disappears immediately, the gap between the contact points is too large; if the flash continues, the gap is too small. In either case, readjust the gap.

Sixth, rotate the dial, *B,* counterclockwise 0.5 divisions to increase the gap by 0.0005 in. This is the operating position for testing.

Seventh, the combustion flash should now be such that raising the compression ratio slightly with the handwheel will bring the flash into coincidence with that for injec-



A—Upper Spring Adjusting Screw
B—Center Adjusting Screw
C—Bumper Spring Adjusting Screw
D—Bumper Spring

E—Lower Spring Adjusting Screw
F—Lower Leaf Spring
G—Pin
H—Upper Leaf Spring

FIG. 35 COMBUSTION INDICATOR

tion, *C* in Fig. 36. If excessive readjustment of the handwheel setting is necessary to obtain this coincidence, change the compression ratio adjustment, and repeat the fifth and sixth steps until coincidence of the combustion and injection flashes is obtained. Then record the combustion chamber length (handwheel setting).

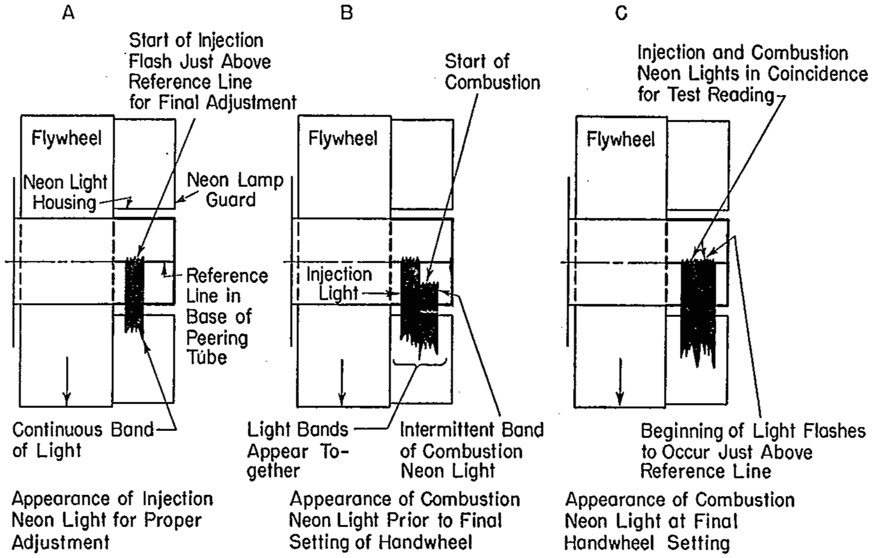
(c) Throughout the test maintain the combustion indicator adjustment as determined for the test fuel in Paragraph (b).

Bracketing the Test Fuel

177 **(a)** Operate the engine on a trial blend of reference fuels, based on the estimated cetane number of the sample. Check and, if necessary, adjust the injection rate to 13 ± 0.2 ml per min and the injection timing to 13 deg btdc. Adjust the handwheel so that the combustion flash begins at the reference line in the peering tube.

(b) Operate the engine on a second trial blend of the reference fuels, differing from the first by not more than five cetane numbers, and repeat the procedure described in Paragraph (a).

(c) If the handwheel setting for the sample is bracketed by those for the reference fuels, continue the test; otherwise try additional blends until this requirement is satisfied. Repeat the operation on both the sample and each of the final reference blends at least twice, adjusting the injection rate and timing as necessary to maintain standard conditions. In changing fuels, always allow at least 5 min before taking readings, to insure thorough flushing of the injection system and to let the engine reach equilibrium.



Appearance of Injection Neon Light for Proper Adjustment
A

Appearance of Combustion Neon Light Prior to Final Setting of Handwheel
B

Appearance of Combustion Neon Light at Final Handwheel Setting
C

FIG. 36 INJECTION AND COMBUSTION NEON-FLASH PATTERNS

Reporting

178 **(a)** Average the handwheel settings obtained in accordance with Par. 177 for the sample and each of the final reference fuels. Find the ASTM cetane number by interpolation from the averages so obtained.

(b) Report the cetane number to the nearest integer. When the interpolated figure ends with 0.5, round off to the nearest even number; for example, report 68.5 as 68, not 69.

Reproducibility

179 Extensive data from independent laboratories over a number of years for many samples of conventional Diesel fuels have shown a standard deviation of 1.4 cetane numbers (average deviation of about 1.0 cetane number). Based on this standard deviation, the number of tests required to yield a rating of desired accuracy is given in the following table:

Accuracy Desired Cetane Number, Plus or Minus	Number of Tests Required to Obtain the Desired Accuracy		
	9 Times Out of 10	19 Times Out of 20	99 Times Out of 100
2	1	2	3
1	5	8	13

This table shows that a single rating can normally be expected to be within plus or minus 2 cetane numbers of the true value in about 9 cases out of 10. On the other hand, if an accuracy of plus or minus 1 cetane number is desired with a certainty of 90 per cent (9 times out of 10), it is necessary to obtain an average of five ratings in five different engines (preferably in five independent laboratories).

Although the above degree of reproducibility applies to conventional Diesel fuels when careful attention is given to the details of test procedure and engine condition, it does not necessarily apply to fuels which differ materially from finished Diesel fuels. In such cases the reproducibility is likely to be lower and result in higher standard deviations.

Cloud and Pour Points

180 Petroleum oils become more or less plastic solids when cooled to low enough temperatures, either because of partial separation of wax as a solid phase or because of the large increase in the viscosity of the hydrocarbons of the oil. If the separated wax becomes visible, the temperature at which this occurs is known as the cloud point. The temperature at which the oil will just begin to flow is known as the pour point.

The cloud point has little signficance in the use of fuel for the generation of heat or power other than the tendency of separated wax to clog fine filters.

The pour point is an indication of the ability to discharge an oil from a container by pouring or by gravity flow to pumping equipment. However, as may be inferred from the test method adopted, oil may flow at an acceptable rate at temperatures below the pour point under a high pressure head when being discharged from a large vessel or when being pumped mechanically. Also, the previous history of the oil with respect to temperature changes before testing may materially affect the pour point determination. Hence, many oils may be discharged or pumped at temperatures well below their pour points.

The Standard Method of Test for Cloud and Pour Points, ASTM Designation: D 97-47, is adopted for the purposes of this code.

Standard Method of Test for Cloud and Pour Points

ASTM Designation: D 97-47

Adopted 1928; Revised 1930, 1933, 1934, 1939, 1947

This Method has been approved as "American Standard" by the American Standards Association

Scope

181 (a) The test for cloud point is intended for use only on oils which are transparent in layers 1½ in. in thickness.

Definitions

182 (a) **Cloud Point.** The cloud point of a petroleum oil is the temperature at which paraffin wax or other solid substances begin to crystallize out or separate from solution when the oil is chilled under definite prescribed conditions.

(b) **Pour Point.** The pour point of a petroleum oil is the lowest temperature at which the oil will pour or flow when it is chilled without disturbance under definite prescribed conditions.

Apparatus

183 The following apparatus shown in Fig. 37 shall be used:

(a) **Test Jar.** A test jar, *a*, of clear glass, cylindrical form, flat bottom, approximately 1$\frac{3}{16}$ to 1$\frac{5}{16}$ in. in inside diameter and 4½ to 5 in. in height. An ordinary 4-oz oil sample bottle may be used if it meets these requirements, and no test jar is available.

(b) **Thermometer.** An ASTM Cloud and Pour Test Thermometer having a range of —38 to +50 C or —36 to +120 F, as specified, and conforming to the requirements for thermometer 5 C or 5 F, respectively, as prescribed in ASTM Specifications E 1,[1] or an ASTM Low Cloud and Pour Thermometer having a range of —80 to +20 or —112 to +70 F, as specified, and conforming to the requirements for thermometer 6 C or 6 F, respectively as prescribed in Specifications E 1.

(c) **Cork.** A cork, *c*, to fit the test jar, bored centrally to take the test thermometer.

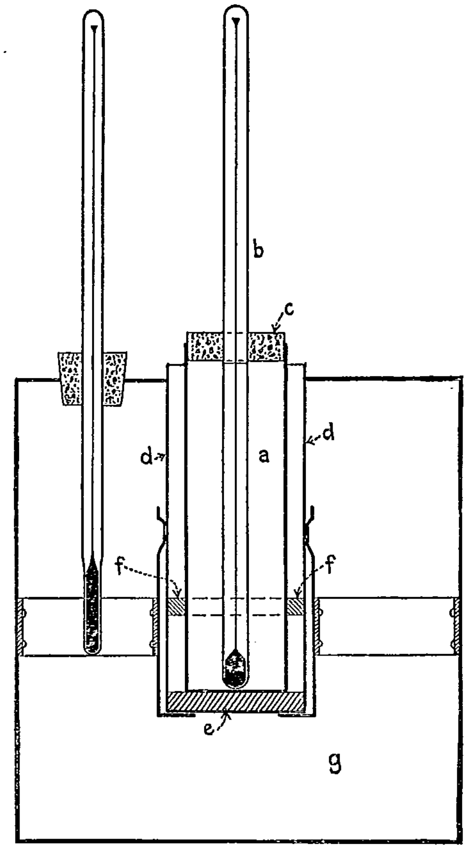


FIG. 37 APPARATUS FOR CLOUD AND POUR TEST
(As Assembled for Cloud Test)

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[1] For tests above —65 F it is permissible to use the ASTM Low Cloud and Pour Thermometers having ranges of —60 to +20 C and —70 to +70 F, as previously prescribed for thermometers 6 C-39 and 6 F-39 in the Standard Specifications for ASTM Thermometers E 1-46, 1946 Book of ASTM Standards, Part III-A, p. 718.

(d) **Jacket.** A jacket, *d,* of glass or metal, water tight, of cylindrical form, flat bottom, about $4\frac{1}{2}$ in. in depth, with inside diameter $\frac{3}{8}$ to $\frac{1}{2}$ in. greater than the outside diameter of the test jar.

(e) **Disk.** A disk of cork or felt, *e,* $\frac{1}{4}$ in. in thickness and of the same diameter as the inside of the jacket.

(f) **Gasket.** A ring gasket, *f,* about $\frac{3}{16}$ in. in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. This jacket may be made of cork, felt or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

(g) **Bath.** A cooling bath, *g,* of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a vertical position, is essential. For determinations of pour points below 50 F, two or more baths should be at hand. The required bath temperatures may be maintained by refrigeration if available, otherwise, by suitable freezing mixtures.

Note. The freezing mixtures commonly used are as follows:

	For Temperatures Down To
Ice and water	50 F
Crushed ice and sodium chloride	10 F
Crushed ice and calcium chloride crystals	—15 F
Solid carbon dioxide and acetone or gasoline[a]	—70 F

[a] This mixture may be made as follows: In a covered metal beaker chill a suitable amount of acetone or gasoline to 10 F, or lower, by means of an ice-salt mixture. Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid. Then add to the chilled acetone or gasoline enough of the solid carbon dioxide to give the desired temperature.

Procedure for Cloud Point

184 (a) Bring the oil to be tested to a temperature at least 25 F above the approximate cloud point. Remove moisture, if present, by any suitable method, as by filtration through dry filter paper until the oil is perfectly clear, but make such filtration at a temperature at least 25 F above the approximate cloud point.

(b) Pour the clear oil into the test jar, *a,* to a height of not less than 2 nor more than $2\frac{1}{4}$ in. Mark the test jar to indicate the proper level.

(c) Tightly close the test jar by the cork, *c,* carrying the test thermometer, *b,* in a vertical position in the center of the jar with the thermometer bulb resting on the bottom of the jar.

(d) Place the disk, *e,* in the bottom of the jacket, *d,* and insert the test jar with the ring gasket, *f,* 1 in. above the bottom into the jacket. The disk, gasket, and inside of jacket shall be clean and dry.

(e) Maintain the temperature of the cooling bath, *g,* at 30 to 35 F. Support the jacket, containing the test jar, firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

(f) At each test thermometer reading that is a multiple of 2 F, remove the test jar from the jacket, quickly but without disturbing the oil, inspect for cloud, and replace in the jacket. This complete operation shall require not more than 3 sec. If the oil does not show a cloud when it has been cooled to 50 F, place the test jar in the jacket in a second bath maintained at a temperature of 0 to +5 F. If the oil does not show a cloud when it has been cooled to 20 F, place the test jar in the jacket in a third bath maintained at a temperature of —30 to —25 F.

(g) When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, record the reading of the test thermometer, corrected for error if necessary, as the cloud point.

Procedure for Pour Point

185 (a) Pour the oil into the test jar, *a,* to a height of not less than 2 nor more than $2\frac{1}{4}$ in. Mark the jar to indicate the proper level. When necessary, heat the oil in a water bath just sufficiently for pouring into the test jar.

(b) Close the test jar tightly by the cork, *c,* carrying the test thermometer, *b,* in a vertical position in the center of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be $\frac{1}{8}$ in. below the surface of the oil.

(c) Heat the oil, without stirring, to a temperature of 115 F in a bath maintained at not higher than 118 F. Cool the oil to 90 F in air in a water bath approximately 77 F in temperature. Heat oils on which a pour point below —30 F is expected as above with the high cloud- and pour-test thermometer in position, cool to 60 F, place the low cloud- and pour-test thermometer in position, and then place the assembly in the jacket. Heat oils on which a pour point of above 90 F is expected to 115 F or to a temperature 15 F above the expected pour point, with the high cloud- and the pour-test thermometer in position, and immediately introduce the test jar into the jacket.

(d) Place the disk, *e,* in the bottom of the jacket, *d,* and insert the test jar, with the ring gasket, *f,* 1 in. above the bottom, into the jacket. The disk, gasket and inside of jacket shall be clean and dry.

(e) After the oil has cooled enough to allow the formation of paraffin wax crystals, take great care not to disturb the mass of the oil nor to permit the thermometer to shift in the oil. Any disturbance of the spongy network of wax crystals will lead to low and fictitious results.

(f) Maintain the temperature of the cooling bath, *g,* at 30 to 35 F. Support the jacket, containing the test jar, firmly in a vertical position in the cooling bath so that not more than 1 in. of the jacket projects out of the cooling medium.

(g) Beginning at a temperature 20 F before the expected pour point, at each test thermometer reading that is a multiple of 5 F, remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall require not more than 3 sec. If the oil has not ceased to flow when its temperature has reached 50 F, place the test jar in the jacket in a second bath maintained at a temperature of 0 to +5 F. If the oil has not ceased to flow when its temperature has reached 20 F, place the test jar in the jacket in a third bath maintained at a temperature of —30 to —25 F. For determinations of very low pour points, additional baths should be maintained with successively lower temperature differentials of about 30 F. In each case transfer the test jar when the temperature of the oil reaches a point 50 F above the temperature of the new bath. At no time place the cold test jar directly in the cooling medium. As soon as the oil in the test jar does not flow when the jar is tilted, hold the test jar in a horizontal position for exactly 5 sec, as noted by a stop watch or other accurate timing device, and observe carefully. If the oil shows any movement under these conditions, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature 5 F lower.

(h) Continue the test in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for exactly 5 sec. Certain lubricating oils tend to move as a whole and should be very closely observed. Record the reading of the test thermometer at this temperature, corrected for error if necessary. Take the pour point as the temperature 5 F above this solid point.

Special Procedure for Black Oils, Cylinder Stocks and Nondistillate Fuel Oils

Special Procedure for Pour Point

186 (a) In those cases where it is known that a sample has been subjected to some temperature higher than 115 F

during the preceding 24 hr or where the history of the sample in this respect is not known, hold the sample in the laboratory 24 hr before testing, unless three consecutive tests in accordance with Par. 185 (c) of the same sample in the same test jar give check results. For these particular oils, results obtained by one or the other of the alternative procedures shall be called the upper (maximum) pour point.

(b) Determine the lower (minimum) pour point by heating a sample, while stirring, to 220 F. Then pour the oil into the test jar, cooled to 90 F as before, and determine the pour point as described in Par. 185.

(c) Report both the upper and lower pour points.

Reproducibility of Results

187 Individual results of the pour test on the same oil in any one laboratory may vary by 5 F and in different laboratories by 10 F, although the average of three or more results in different laboratories should show a difference between averages no greater than 5 F. For oils tested by the special procedure described in Par. 186, reproducibility of this order cannot be expected, as these oils show anomalous pour points depending on their previous thermal history.[2]

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[2] It is a recognized property of these oils that the temperature to which they have been subjected before testing influences their pour points. Although the lower pour points as determined by the special procedure will show approximately the reproducibility given above, yet the upper pour points will show greater variations depending on the previous thermal history of the oils. Further information on this subject is contained in *Proceedings,* American Society for Testing Materials, Vol. 31, Part I, pp. 468 to 470 (1931), and Vol. 32, Part I, pp. 402 to 405 (1932).

Stability of Fuel Oils

188 Distillate heating oils and Diesel fuels when held in storage over relatively long periods of time sometimes have a tendency to form sediment and/or soluble gums. Unless such deterioration products are well dispersed by means of additives or naturally occurring dispersants, their presence in the fuel may lead to filter and nozzle plugging or injector sticking. Distillate fuels vary widely in storage stability, depending on such factors as the type of crude from which they are derived, hydrocarbon composition, refinery treatment and processing, and compatibility with other stocks. There is no standardized or widely accepted laboratory test for predicting the storage stability of distillate fuels. Numerous tests have been developed for this purpose, and some have been found quite reliable when applied to certain types of blending stocks (those produced by a single refiner, for example), but in general no known test, short of storage under actual field conditions, is considered applicable for assessing the storage stability of all types of distillates.

189 Certain residual fuel oils, because of thermal instability, will, when heated, deposit a film on the surface of the heater. As this film increases in thickness, the rate of heat transfer will decrease and the pressure drop across the heater may be seriously increased. The Test for the Thermal Stability of Boiler Fuel Oil (NBTL Heater), Method 346.1.1, November 15, 1948, as published in Federal Specification VV-L-791d, November 15, 1948, is adopted for the purposes of this code to measure this tendency of fuel oils to foul heaters.

The Military Specification for Fuel Oil, Boiler, MIL-F-859A, July 31, 1951, requires that Navy special fuel oil, which corresponds approximately to No. 5 fuel oil, shall show a No. 1 tube or better under this test and that Navy heavy fuel oil, which corresponds to No. 6 fuel oil, shall show a No. 2 tube or better.

Thermal Stability of Boiler Fuel Oil (NBTL* Heater)

Scope

190 This method of test is intended for the determination of the thermal stability of fuel oil under prescribed conditions of heating.

Apparatus

191 The apparatus shall consist of the following: (See Fig. 38.)

(a) Heater Element consists essentially of a steel tube 31 in. long, 0.540 in. in outside diameter, and 0.049 in. in wall thickness, with internal electric heating, and enclosed in a thermally insulated length of 1½-in. pipe through which the test fuel oil flows at a specified rate. The heater element shall conform to the design details given in Fig. 38 and the method of construction is described briefly below.

Cut a length of 1½-in. wrought-iron standard pipe such that when a 1½-in. iron coupling is screwed on one end and a 1½-in. by 1½-in. by 1-in. iron tee is screwed on the other end (with side outlet vertical), the over-all length will be 26 in. Assemble the coupling and tee on the 1½-in. pipe in such a manner that the connections are tight at fuel-oil temperatures of 200 to 300 F.

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* NBTL = Naval Boiler Testing Laboratory.

From a piece of 2-in. (across flats) hexagonal brass rod, machine two $1\frac{1}{2}$-in. pipe plugs and through each drill a clearance hole for the 0.540-in. OD steel tube. Bore out the hexagonal end of each plug to the proper diameter for tapping with a $\frac{3}{4}$-in. standard pipe tap and to an appropriate depth so that adequate space is left for the packing material after a $\frac{3}{4}$-in. pipe plug is inserted. The bottom shoulder of the recess should be flat in order to retain the packing satisfactorily. Tap each plug with a $\frac{3}{4}$-in. standard pipe tap.

Make two packing gland nuts by machining a piece of $1\frac{1}{2}$-in. (across flats) hexagonal brass rod to form $\frac{3}{4}$-in. pipe plugs. Through each plug, drill a clearance hole for the 0.540-in. OD steel tube.

Halfway along the length of the iron coupling on one end of the $1\frac{1}{2}$-in. tube, drill a hole to accommodate a length of $\frac{1}{8}$-in. brass pipe and braze. This fuel-oil inlet pipe should be pointing downwards when the side outlet of the tee at the other end is pointing upwards (Fig. 38).

Into the 1-in. side outlet of the tee on the other end of the $1\frac{1}{2}$-in. tube, screw a 1-in. standard iron close nipple and a 1-in. standard iron coupling. Assemble in such a manner that the connections are tight at fuel-oil temperatures of 200 to 300 F. Halfway along the length of the coupling, drill a hole to accommodate a length of $\frac{1}{8}$-in. brass pipe, which acts as the fuel-oil outlet, and braze in place. To the other end of the brass pipe, connect a $\frac{1}{8}$-in. brass elbow and brass valve. The oil returns from this valve to the fuel-oil reservoir.

Close the top of the coupling on the side outlet from the T with a 1-in. by $\frac{3}{4}$-in. brass reducing bushing, and insert a special steel fitting, threaded with $\frac{3}{4}$-in. standard pipe thread, and fitted with a re-entrant tube closed at the bottom to act as a thermometer well. The bottom of the thermometer well should be on a level with the top of the straight-through bore of the T (Fig. 38). This special fitting and the reducing bushing should be assembled in such a manner that the connections are tight at fuel-oil temperatures of 200 to 300 F.

The assembled heater shell ($1\frac{1}{2}$-in. tube, $1\frac{1}{2}$-in. coupling, and the $1\frac{1}{2}$-in. by $1\frac{1}{2}$-in. by 1-in. T, including the fitting and thermometer well in the side outlet of the T) shall be insulated with a 1-in. layer of asbestos as shown in Fig. 38, leaving exposed only the ends of the heater tube for insertion of the packing glands, and the opening to the thermometer well. A suitable support shall be constructed for holding the insulated heater shell in a horizontal position.

In order to assemble the heater, center the externally polished steel heater tube (31 in. long, 0.540-in. outside diameter and 0.049-in. wall thickness) in the insulated heater shell by means of the brass packing gland at each end, with approximately an equal length of steel tube projecting at the two ends. Insert the packing material (such as $\frac{1}{4}$-in. rope asbestos) which shall be capable of withstanding the high temperature involved, and insert and tighten the brass packing nuts. The assembly of the heater element is now complete, except for the glass-enclosed internal heater which is inserted into the steel heater tube through one end.

The glass-enclosed internal heater shall be made in the following manner. Cut a length of No. 22 B. & S. gage chromel heating wire (about 1 ohm per ft) such that the total resistance is approximately 27 ohms. Thread a steel rod with 10-24 thread for an appropriate length and wind the wire tightly around the threaded rod. Clamp the wire in place at the ends, heat to 1,000 F and quench in order to preserve the coil form. Remove the coiled wire from the threaded rod and insert in the center of a 32-in. length of pyrex glass tubing, $\frac{7}{16}$-in. outside diameter and $\frac{1}{16}$-in. wall thickness, which has been flared out slightly at one end to a diameter greater than the inside diameter of the steel heater tube into which the glass tube fits. The coiled resistance wire should be centered longitudinally in the glass tube, and should cover a length of approximately 24 in.

The iron-constantan thermocouple shall be made by butt-welding the ends, and inserted in a series of four single-hole porcelain insulators, 4 in. long by $\frac{1}{8}$-in. in outside diameter, in such a manner that the thermocouple junction will be separated from the heating coil by the wall of the insulator.

Symbol	Name of Part	Material
A	Heater shell, $1\frac{1}{2}$-in. standard pipe	Wrought iron
B	Tee, $1\frac{1}{2}$-in. by $1\frac{1}{2}$-in. by 1 in.	Iron
C	Coupling, $1\frac{1}{2}$-in.	Iron
D	Nipple, 1-in.	Iron
E	Coupling, 1-in.	Iron
F	Bushing, 1-in. by $\frac{3}{4}$-in.	Brass
G	Thermometer well, $\frac{3}{4}$-in.	Steel
H	Pipe, $\frac{1}{8}$-in. standard	Brass
I	Elbow, $\frac{1}{8}$-in.	Brass
L	Valve, $\frac{1}{8}$-in.	Brass
M	Gland bushing	Brass
N	Packing ring	Brass
O	Packing, $\frac{1}{4}$-in. rope	Asbestos
P	Gland body	Brass
Q	Heater element, 31 in. long, 0.540-in. outside diameter, 0.049-in. wall thickness	Steel tube
R	Glass tube, 32 in. long, $\frac{7}{16}$-in. outside diameter, $\frac{1}{16}$-in. wall thickness	Pyrex
S	Heating wire, No. 22 B. & S.	Chromel
T	Insulators, single hole, $\frac{1}{8}$-in. outside diameter, 4 in. long	Porcelain
U	Insulators, double hole, $\frac{5}{32}$-in. outside diameter, 4 in. long	Porcelain
V	Thermocouple	I.C.

The thermocouple assembly, including the $\frac{1}{8}$-in. porcelain insulators, shall be inserted in the glass tube with the junction in the geometrical center of the heating coil.

The leads from the thermocouple junction shall be brought out, one at each end; through double-hole porcelain insulators, 4 in. long by $\frac{5}{32}$-in. in outside diameter. The leads from the heating coil shall be brought out, one at each end, through opposite holes of the same insulators containing the thermocouple leads.

The internal heater is then ready for insertion in the steel heater tube.

(b) **Test Assembly.** The test assembly shall be as illustrated in the lower right corner of Fig. 38. A suitable oil reservoir of 2 to 2.5 gal. capacity shall be fitted with an outlet in the bottom, and this outlet shall be connected through a throttling valve to the inlet of a small motor-driven pump. From the pump, a line runs to a T connection, one branch of which leads through a bypass valve back to the fuel oil reservoir, and the other branch leads through a throttling valve to the fuel-oil heater assembly. From the fuel oil heater, the oil returns to the reservoir.

The leads from the internal electric heater are connected through an ammeter to a voltage regulator or rheostat. The leads from the iron-constantan thermocouple are connected to a potentiometer.

Preparation of Apparatus for Test

192 Clean the oil reservoir and introduce about $1\frac{1}{2}$ gal. of cleaning agent, such as kerosine. Drain the oil used in the previous test from the heater assembly, remove the internal electric heater and the steel tube from the heater assembly, and close the ends with pipe plugs. Start the pump and circulate the cleaning fluid for one hour through the entire circulating system, including the bypass line. Drain the cleaning fluid from the system, and replace with a sample of the test oil. Circulate the test oil throughout the entire circulating system for one hour, and then drain and discard.

Clean the steel heater tube from any residues left from the previous run, and install in the heater assembly. Insert the internal electric heater, and connect the leads to the heating circuit. Connect the thermocouple leads to the potentiometer.

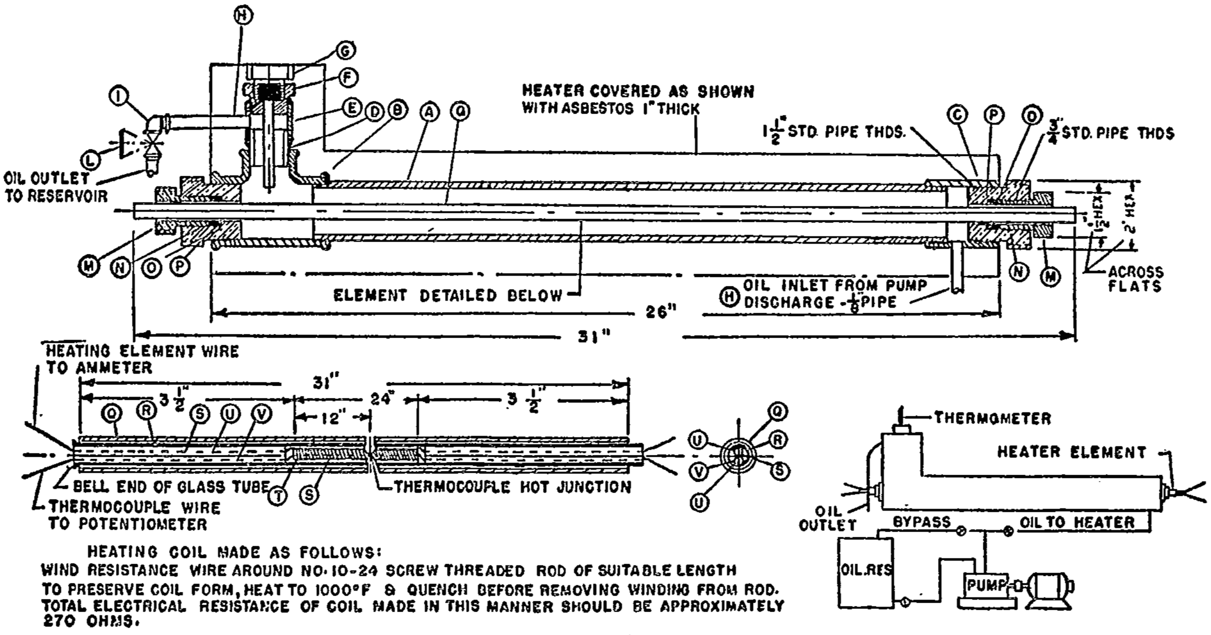
The apparatus is now ready for a thermal stability test of the fuel oil sample.

Test Procedure

193 After preparation of the apparatus for test as outlined in Par. 192, transfer $1\frac{1}{2}$ gal. of the test fuel to the fuel oil reservoir. Close the bypass valve, and open all other valves. Start the pumping unit and allow the heater shell to fill and start overflowing back into the reservoir.

The rate of oil shall be reduced by adjusting the valves until it is approximately 0.25 gal. per hr, the excess fuel oil being allowed to return to the reservoir through the bypass connection. Determination of the rate of oil flow can be made by calculation based on the time required to fill a 60-ml receiving flask (method 30.4) with oil from the heater outlet. This rate shall be maintained until the outlet oil temperature, as indicated by the thermometer in the thermometer well of the heater assembly, reaches the applicable test temperature.

The internal electric heater shall be connected to the heating circuit, and by means of the voltage regulator or rheostat, the heating current shall be controlled so that a temperature of 600 F ± 10 F is indicated by the thermocouple in the center of the electric heating coil. This temperature shall be maintained for the duration of the test.



HEATING COIL MADE AS FOLLOWS:
WIND RESISTANCE WIRE AROUND NO. 10-24 SCREW THREADED ROD OF SUITABLE LENGTH TO PRESERVE COIL FORM, HEAT TO 1000°F & QUENCH BEFORE REMOVING WINDING FROM ROD. TOTAL ELECTRICAL RESISTANCE OF COIL MADE IN THIS MANNER SHOULD BE APPROXIMATELY 270 OHMS.

FIG. 38 FUEL OIL HEATER

When the outlet oil temperature, as indicated by the thermometer in the thermometer well of the heater assembly, reaches the applicable test temperature, the valves on the circulating oil system shall be adjusted, and kept adjusted, so that the outlet oil temperature is maintained for the duration of the test within ±5 F of the applicable test temperature. Due to the necessity for compensating for the temperature rise of the oil in the reservoir, some adjustment of the rate of oil flow may be required for approximately the first 4 hr of the test. The applicable test temperature shall be:

(1) *200 F for No. 5 fuel oils, and*
(2) *A temperature 25 F above that at which the viscosity of the test oil is 150 Saybolt Universal seconds for No. 6 or bunker C fuel oils.*

The thermocouple temperature and the outlet oil temperature shall be maintained within the prescribed limits for a total period of 20 hr. The rate of oil flow shall be determined at hourly intervals during this period, and shall be recorded.

At the end of the 20-hr period, the current to the electric heater shall be turned off but the circulation of oil shall be continued for at least 5 min to preclude possible over-heating of the oil. After stopping the oil circulation, the oil in the heater shell shall be allowed to drain by gravity into the reservoir.

When drainage of the oil has ceased and the heater shell has cooled sufficiently, the internal heating element shall be removed, and the steel tube disconnected and removed, care being taken not to disturb any deposit on its outside surface.

The steel tube shall then be inspected in accordance with Par. 194.

Inspection and Rating

194 After the steel tube has cooled approximately to room temperature, after careful removal from the heater assembly as outlined in Par. 193, an 8-in. section terminating approximately 3 in. from the outlet oil connection shall be rinsed by flushing with a small stream of benzol. After the benzol has drained off, approximately one-half (4 in.) of the benzol-washed section shall be wiped by hand with a clean soft cloth. The latter step serves principally to permit visual estimation of the thickness of the films obtained in the other sections. In order to clarify the inspection and rating system, the above sections of the tube will be designated as follows:

(A) Benzol-washed section (4 in.) after wiping with cloth.
(B) Benzol-washed section (4 in.) unwiped.
(C) Unwashed section.

The test fuel oil shall be rated by examination of the appearances of the deposit on the steel tube in the unwashed section C, and in the unwiped, benzol-washed section B. The appearance of the deposit in these two sections shall be recorded. While not used for rating, the appearance of the wiped section A shall also be noted and recorded.

The oil shall be classed as stable or unstable on the basis of the above examination and in accordance with the following criteria:

A stable oil is characterized by the presence only of a clean oil film in the unwashed section C and of negligible discoloration or film in the unwiped, washed section B.

An unstable oil is identified by the presence of an asphaltic smear or emulsion layer in the unwashed section C, and discoloration and carbon film (dry or wet due to inability to wash oil-free) in the unwiped, washed section B.

Specifically, the oil stability shall be rated numerically in accordance with the fuel oil stability classification chart in Fig. 39, or on the basis of Table 21. In both of these, the information on the wiped, washed section A is included as a matter of record only.

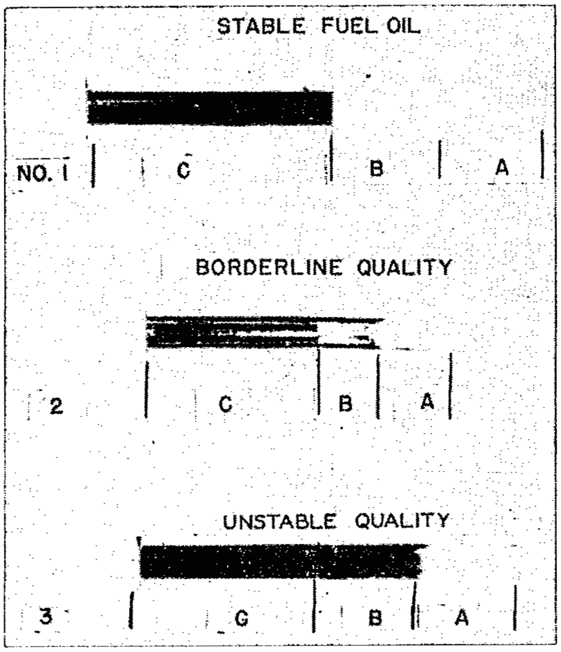


FIG. 39 FUEL OIL STABILITY CLASSIFICATION CHART

TABLE 21

Steel Tube (See Fig. 39)	Rating	Tube Sections		
		Wiped, Washed A	Unwiped, Washed B	Unwashed C
1	Stable	Metal bright. No discoloration.	Metal bright. Negligible discolorations or films.	Clean oil film or lightly peppered only.
2	Borderline	Slightly darkened.	Thin oil-free layer of carbon varying in color from a light tan to iridescent blue.	Heavily peppered oil film or annular streaked lines of sediment or evidence of the beginning of the formation of a film of asphaltic smear.
3	Unstable	Definite discoloration or darkened.	Definite black, carbon film which, depending on its thickness, may prove difficult to wash oil-free.	Thin to thick film of an asphaltic smear. This film may assume the appearance of concentric annular rings for very unstable products.

Compatibility of Fuel Oils

195 Certain fuel oils, each of which will by itself be acceptable by the thermal stability test, will when mixed together deposit objectionable coatings when subjected to the thermal stability test. An approximate indication, which is adopted by the Navy for its test of compatibility of oils is as follows:

Equal volumes of the two or more oils that are to be used together shall be thoroughly mixed and a sample of the mixture shall be subjected to the thermal stability test of Par. 193. As a minimum, one test of equal volumes may be used.

Specifications for Diesel and Burner Fuels

196 For the convenience of the purchaser, seller, and supplier of Diesel and burner fuels, Table 22, Detailed Requirements for Fuel Oils, from Tentative Specifications for Fuel Oils, ASTM Designation: D 396-48T, and Table 23, Tentative Classification of Diesel Fuel Oils, ASTM Designation: D 975-53T, are included in this code. The methods of test for the various characteristics are those presented in this Test Code for Diesel and burner fuels. These specifications may be accepted as written or may be modified by mutual agreement. The term Bunker C oil, which, although widely used, is not a recognized standard, includes all residual fuels heavier than No. 5.

Petroleum Measurement Tables

197 For various calculations having to do with conversion of the gravity of oil from one scale to another and, for the reduction or weight to volume or volume to weight in a variety of units, The ASTM-IP Petroleum Measurement Tables issued in 1953 as a result of joint effort of the American Society for Testing Materials and The Institute of Petroleum, Great Britain, will be found of great value. These tables constitute a great expansion of the tables issued as the National Bureau of Standards Circular C 410 which have previously been the standard table of reference. The American edition of 544 pp. is available from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa., at $8.75 or for $6.50 to members of the ASTM. A British edition is also available at $7.00 or $5.25 to members of the ASTM and a Metric edition in three languages, English, French, and Spanish, is available at $7.70 per copy or $5.80 to members of the ASTM.

(*Tables 22 and 23 are on the following pages.*)

TABLE 22 DETAILED REQUIREMENTS FOR FUEL OILS[a]

Grade of Fuel Oil[b]		Flash Point, F	Pour Point, F	Water and Sediment, Per Cent by Volume	Carbon Residue on 10 Per Cent Bottoms, Per Cent	Ash, Per Cent by Weight	Distillation Temperatures, F			Saybolt Viscosity, Sec				Kinematic Viscosity, Centistokes				Gravity, Deg API	Corrosion at 122 F (50 C)[d]
							10 Per Cent Point	90 Per Cent Point	End Point	Universal at 100 F		Furol at 122 F		At 100 F		At 122 F			
		Min	Max	Max	Max	Max	Max	Max	Max	Max	Min	Max	Min	Max	Min	Max	Min	Min	
No. 1	A distillate oil intended for vaporizing pot-type burners and other burners requiring this grade of fuel	100 or legal	0	Trace	0.15	...	420	...	625	...	...	...	..	2.2	1.4	...	..	35	pass
No. 2	A distillate oil for general purpose domestic heating for use in burners not requiring No. 1 fuel oil	100 or legal	20[c]	0.10	0.35	...	[e]	675	...	40	...	...	..	(4.3)	...	...	..	26	....
No. 4	An oil for burner installations not equipped with preheating facilities	130 or legal	20	0.50	...	0.10	...	...	...	125	45	...	..	(26.4)	(5.8)	...	..	..	....
No. 5	A residual-type oil for burner installations equipped with preheating facilities	130 or legal	..	1.00	...	0.10	...	...	...	...	150	40	..	...	(32.1)	(81)	..	..	....
No. 6	An oil for use in burners equipped with preheaters permitting a high viscosity fuel	150	..	2.00[f]	...	...	...	...	...	...	...	300	45	...	...	(638)	(92)	..	....

[a] Recognizing the necessity for low-sulfur fuel oils used in connection with heat-treatment, non-ferrous metal, glass and ceramic furnaces and other special uses, a sulfur requirement may be specified in accordance with the following table:

Grade of Fuel Oil	Sulfur, Max, Per Cent
No. 1	0.5
No. 2	1.0
No. 4	no limit
No. 5	no limit
No. 6	no limit

Other sulfur limits may be specified only by mutual agreement between the purchaser and the seller.

[b] It is the intent of these classifications that failure to meet any requirement of a given grade does not automatically place an oil in the next lower grade unless in fact it meets all requirements of the lower grade.

[c] Lower or higher pour points may be specified whenever required by conditions of storage or use. However, these specifications shall not require a pour point lower than 0 F under any conditions.

[d] Report as passing when the copper test strip shows no gray or black deposit.

[e] The 10 per cent point may be specified at 440 F maximum for use in other than atomizing burners.

[f] The amount of water by distillation plus the sediment by extraction shall not exceed 2.00 per cent. The amount of sediment by extraction shall not exceed 0.50 per cent. A deduction in quantity shall be made for all water and sediment in excess of 1.0 per cent.

TABLE 23 LIMITING REQUIREMENTS FOR DIESEL FUEL OILS[a]

Grade of Diesel Fuel Oil		Flash Point, F	Pour Point, F	Water and Sediment, Per Cent by Volume	Carbon Residue on 10 Per Cent Residuum, Per Cent	Ash, Per Cent by Weight	Distillation Temperatures, F		Viscosity at 100 F		Sulfur, Per Cent by Weight	Copper Strip Corrosion	Cetane Number[d]
							90 Per Cent Point	End Point	Kinematic, Centistokes (or Saybolt Universal, Sec.)				
		Min	Max	Max	Max	Max	Max	Max	Min	Max	Max	Max	Min
No. 1-D	A volatile distillate fuel oil for engines in service requiring frequent speed and load changes.	100 or legal	20[b]	Trace	0.15	0.01	...	625	1.4	...	0.50	No. 3	40[c]
No. 2-D	A distillate fuel oil of low volatility for engines in industrial and heavy mobile service.	125 or legal	..[b]	0.10	0.35	0.02	675	...	1.8 (32.0)	5.8 (45)	1.0	No. 3	40[c]
No. 4-D	A fuel oil for low and medium speed engines.	130 or legal	..[b]	0.50	...	0.10	...	...	5.8 (45)	26.4 (125)	2.0	.....	30[c]

[a] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller and supplier.

[b] For cold weather operation the pour point should be specified 10 F below the ambient temperature at which the engine is to be operated except where fuel oil heating facilities are provided.

[c] Low-atmospheric temperatures as well as engine operation at high altitudes may require use of fuels with higher cetane ratings.

[d] A Proposed Method of Determining Calculated Cetane Index is given in Par. 166.

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