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AMERICAN WOOD-PRESERVERS' ASSOCIATION
STANDARD

A1-91

STANDARD METHODS FOR ANALYSIS OF CREOSOTE AND OIL-TYPE PRESERVATIVES

Note: Standard A1-89 consists of eighteen pages dated as follows: Pgs. 1-2, 1989; Pgs, 3-4, 1974; Pgs. 5-8, 1989: Pgs. 9-12, 1973; Pgs. 13-16, 1976; Pgs. 17-18, 1980.

Page Number

Note: The ASTM Standards referred to herein may be obtained from the American Society for Testing Materials, 1916 Race Street, Philadelphia, Pa. 19103.

1. STANDARD METHOD FOR THE DETERMINATION OF COKE RESIDUE

Scope

This method of test covers the determination of the coke residue (fixed carbon) Of creosote, coal tar, and creosote solutions. As coal tars vary in their coke residue values, the method is therefore not a quantitative index of the percentage of coal tar in creosote solutions. However, by specifying a maximum amount of coke residue as done in AWPA Standard P2, the method does serve to limit the amount of coal tar in such solutions. Formerly the test was also used as a check on the cleanliness of AWPA P1/P13 creosote (absence of coal tar).

Outline of Method

After subjecting the creosote-coal tar solution (oil) under test to the distillation method (Standard A1, Section 2), the weighed residue at 355°C in the distillation flask is thoroughly mixed and a one gram sample transferred to a special platinum crucible with vented cover. The crucible is then placed in a furnace maintained at 950° ± 20°C for exactly seven minutes. A final weighing gives the amount of coke of the distillation residue. The coke residue of the creosote-coal tar solution under test is calculated from (a) the percentage of distillation residue at 355°C, (b) the weight of the sample of distillation residue transferred to the platinum crucible, and (c) the weight of coke remaining in the platinum crucible after the coking operation.

Apparatus

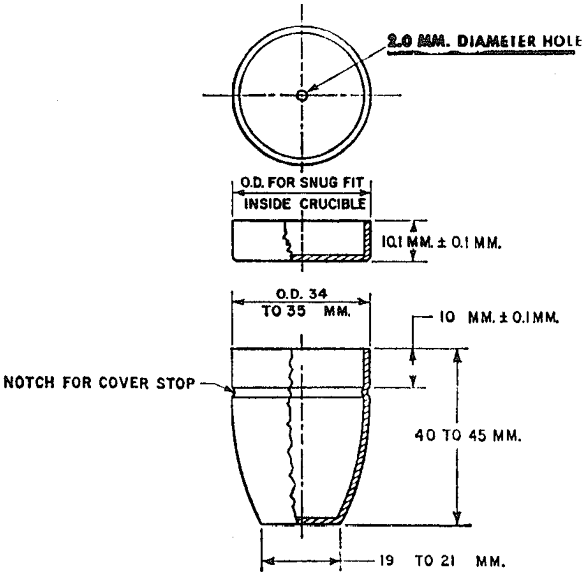


FIGURE 1.—Platinum Crucible for Use in Determining Coke Residue.

1. **Crucible and Cover**—Platinum crucible and cover with dimensions as shown in Figure 1. The cover shall be tightly fitting, shall have a depth of between 10.0 and 10.2 mm, and shall have a hole 2.0 mm in diameter at its center. The crucible without cover shall have a capacity of 25 to 30 ml. It shall have an outside diameter at the top of 34.0 to 35.0 mm, and outside diameter at the bottom of 19 to 21 mm, and a height of 40 to 45 mm. The weight of crucible with cover shall be 25 to 35 g.

2. Crucible Holder—The crucible holder shall be made from No. 20 nichrome wire and a brass ring as shown in Figure 2. The holder serves as a support for the platinum crucible during the coking operation.

3. Furnace—A vertical electric tube furnace having a heating chamber 1½" (38 mm) in diameter, by 6" (150 mm) in depth. The power input shall be 300 to 400 watts at 115 volts. The furnace shall be equipped with a suitable rheostat or variable transformer and pyrometer, for control at 950° ± 20°C. A thermocouple shall be installed through a central hole in the bottom of the furnace, and sealed in place so that its junction is located 2 to 5 mm below the eventual position of the bottom of the platinum crucible.

Note: This is the Fieldner-type furnace designed for the determination of volatile matter in coal (Bureau of Mines Bulletin 85). The Hoskins Type FA-120 electric furnace has proven satisfactory.

Procedure

Heat and adjust the temperature of the uncovered, empty furnace to 950° ± 20°C.

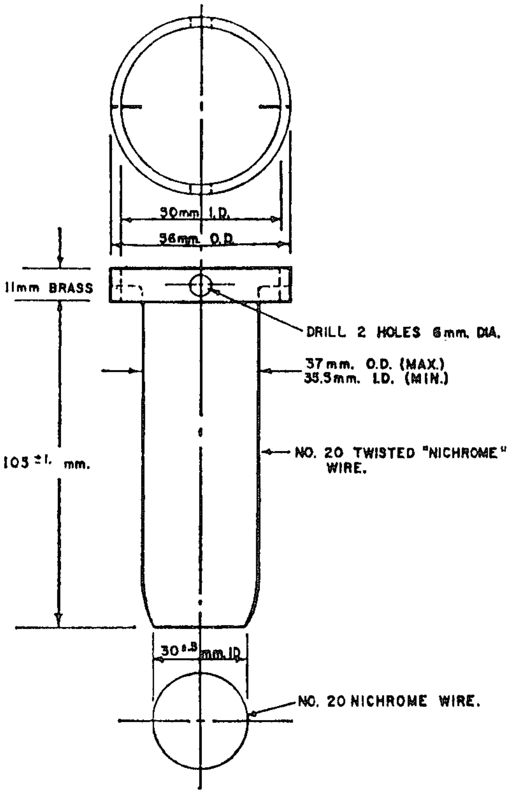


FIGURE 2.—Crucible Holder.

Clean the platinum crucible and cover, place them in the crucible holder, and suspend them in the temperature-adjusted furnace for at least five minutes. Remove the holder with contents from the furnace and cool the crucible and cover to about 150°C, being careful not to contaminate the bottom of the crucible. Then place the crucible with cover in a desiccator until they have cooled to room temperature and weigh (P). Record all weighings to the nearest mg.

If the coke residue determination is made shortly after completion of the distillation test (AWPA Standard A1, Section 2) transfer a 1 ± 0.1 g portion of the well-stirred distillation residue at 355°C directly from the distillation flask to the tared platinum crucible. Since the tare weight (P) includes the weight of the platinum cover, the cover must also be on the balance pan during this operation.

If the residue is in a storage container, carefully heat the container and contents in a water bath or on a steam bath until sufficiently liquid for pouring. Then transfer a 1 ± 0.1 g portion to the platinum crucible as directed in the preceding paragraph.

After the desired amount of distillation residue has been transferred to the platinum crucible insert its cover firmly and weigh (Q). Weight of sample of distillation residue taken for coking: $Q - P = W$.

Place the covered crucible with sample in the crucible holder and suspend in the furnace at 950 ± 20°C for exactly seven minutes. At the end of this period immediately remove the crucible holder from the furnace, cool the crucible and contents to about 150°C as before, transfer to a desiccator. When at room temperature, weigh (R). Weight of coke in sample of distillation residue: $R - P = A$.

Calculations

Calculate the percentage of coke residue on the basis of original oil (creosote-coal tar solution) as follows:

$$\text{Coke Residue} = \frac{A \times B}{W}$$

where:

W = Weight of sample of distillation residue taken for coking

A = Weight of coke in sample of distillation residue

B = Percentage of distillation residue at 355°C of original oil (creosote-coal tar solution)

Example—$W = 1.100$ g; $A = 0.308$ g; $B = 29.0\%$

$$\text{Coke Residue} = \frac{29.0 \times 0.308}{1.100} = 8.1\%$$

Report

Report the calculated amount of coke residue to the nearest 0.1%.

Precision

Coke Residue, 0–2.5% Range

Repeatability: Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.3 percent absolute.

Reproducibility: The values reported by each of two laboratories should not be considered suspect unless they differ by more than 0.5 percent absolute.

NOTE: The estimated standard deviation of repeatability is 0.12 percent absolute at 16 D.F. The estimated standard deviation of reproducibility is 0.18 percent absolute at 14 D.F.

Coke Residue, 2.5–12% Range

Repeatability: Duplicate values by the same operator should not be considered suspect unless they differ by more than 12 percent relative.

Reproducibility: The values reported by each of two laboratories should not be considered suspect unless they differ by more than 14 percent relative.

NOTE: The estimated standard deviation of repeatability is 4.1 percent relative at 24 D.F. The estimated standard deviation of reproducibility is 4.9 percent relative at 21 D.F.

2. STANDARD METHOD OF DISTILLATION

Scope

This method of test is suitable for the distillation of creosote and creosote-coal tar solution.

Sampling

Thoroughly stir or otherwise mix the sample immediately before removing the portion for testing, to ensure that such portion will be representative of the sample. If crystallized solids are present, warm the sample to dissolve the crystals, taking care to avoid loss of volatile material.

Apparatus

1. Flask.—A side-neck distillation flask, as shown in Fig. 3, having the following dimensions:

Diameter of bulb, outside	86.0 ± 1.5 mm.
Diameter of neck, inside	22.0 ± 1.0 mm.
Diameter of tubulature, inside	10.0 ± 0.5 mm.
Height of flask, outside	131.0 ± 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature, inside	93.0 ± 1.5 mm.
Length of tubulature	220 ± 5 mm.
Angle of tubulature	75 ± 2 deg.
Thickness of tubulature wall	1.0 to 1.5 mm.

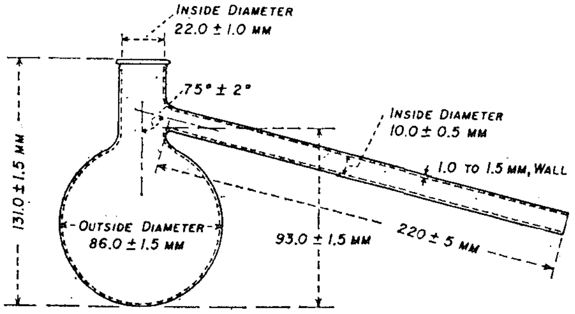


Figure 3.—Distillation flask.

2. Condenser Tube.—A tapered glass condenser, as shown in Figs. 4 and 5, having the following dimensions:

Diameter of small end, outside	12.5 ± 1.5 mm.
Diameter of large end, outside	28.5 ± 3.0 mm.
Length	360 ± 4 mm.
Length of tapered part	100 ± 5 mm.

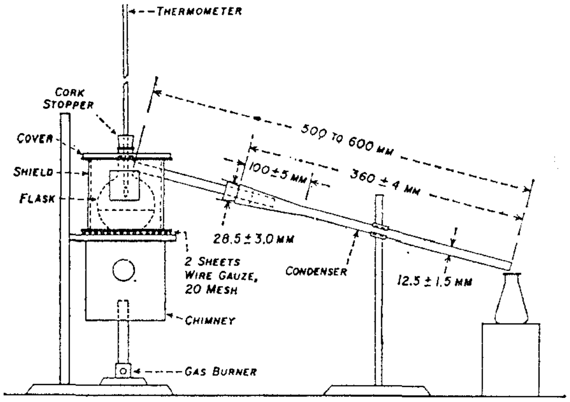


Figure 4.—Apparatus assembly for flame distillation.

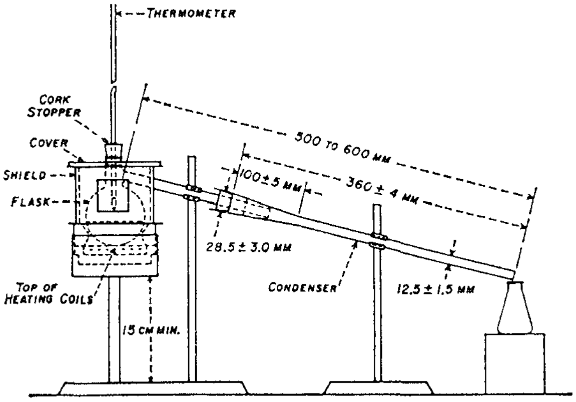


Figure 5.—Apparatus assembly for electric heater distillation.

3. Source of Heat

(a) *Flame.*—Bunsen or Meker type burner, or

(b) *Electric Heater.*—Output variable to 600 or to 750 watts with removable upper and lower refractories, as illustrated in Fig. 6, provided with variable transformer or rheostat suitable for the voltage used[1] and fitted with clamp for mounting on vertical support rod.

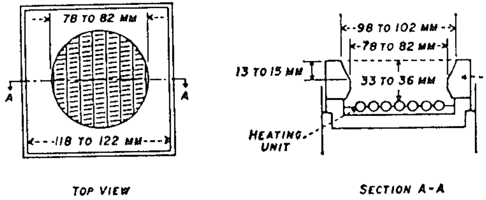
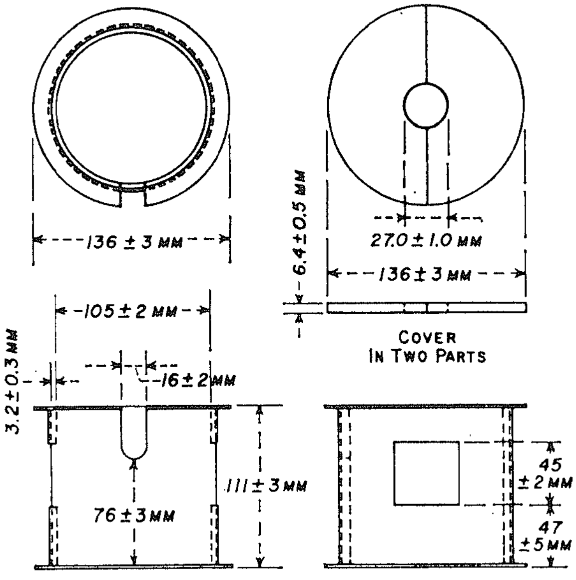


Figure 6.—Upper part of electric heater.

4. Shield and Cover

(a) *For Flame Distillation.*—A metal shield, fitted with mica windows, and lined with $1/8$-in. asbestos, of the form and dimensions shown in Fig. 7; with two-part cover made from $1/4$-in. "Transite" board, also shown in Fig. 7.



FLANGED OPEN-END CYLINDER MADE OF 22 GAGE STAINLESS STEEL WITH 1/8-IN. ASBESTOS LINING.

TWO MICA WINDOWS ARE PROVIDED AT RIGHT ANGLES TO THE END SLOT.

Figure 7.—Shield and cover for flame distillation.

(b) *For Electric Heater Distillation.*—A metal shield, fitted with mica windows, and cover of the same construction and dimensions as those used for flame distillation (4(a)), except for the height of the shield. See Fig. 8.

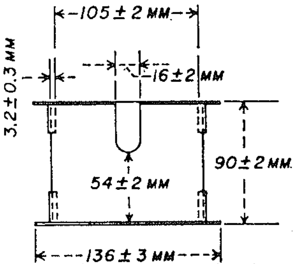


Figure 8.—Shield for use with electric heater.

5. **Burner Chimney for Flame Distillation.**—A cylindrical metal chimney approximately 100 mm. high, 95 to 105 mm. in diameter, and having a peephole 25 mm. in diameter centered about 32 mm. below the ring support, used to protect the flame from air currents. The top of the shield shall be flanged to permit its being suspended from the ring support. See Fig. 9.

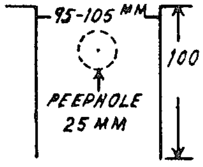


Figure 9.—Burner chimney for flame distillation.

6. **Gauzes for Flame Distillation.**—Two 6 inch by 6 inch sheets of 16 mesh nichrome wire gauze made of 0.02 inch diameter wire.

7. **Receivers.**—Erlenmeyer flasks or beakers having a capacity of 50 to 125 ml. tared to the nearest 0.05 g.

8. **Balance and Weights.**—A balance and weights accurate to 0.05 g.

9. **Thermometer.**—An ASTM High Distillation thermometer having a range of −2° to 400° C. and conforming to the requirements for Thermometer 8 C as prescribed in the Specifications for ASTM Thermometers (ASTM Designation: E 1).

10. Some chemical supply houses do not stock all of the above apparatus. Burrell Company, W. H. Curtin & Co., and Scientific Glass Apparatus Co. usually carry complete stocks.

Apparatus Assembly

(a) *For Flame Distillation.*—Suspend the burner chimney by its flange from a support ring, place the specified two sheets of wire gauze on the burner chimney, and set the flask shield on the upper sheet of gauze.

Insert the thermometer through a regular-length, tightly-fitting cork, rolled prior to use, in the neck of the distillation flask so that the bottom of the cork is from 23 to 28 mm. above the lowest point of the juncture between the tubulature and the neck of the flask, and the bottom of the bulb is 12 to 13 mm. from the surface of the liquid in the flask. The exact location of the thermometer bulb may be determined by calculating the number of divisions on the thermometer which are equal to 12 to 13 mm., lowering the thermometer through the cork until the tip of its bulb touches the surface of 100 g. of sample in the flask, and then raising the thermometer the calculated number of degrees to give the correct distance from the surface of the liquid. Align the stem on the axis of the bulb through the neck of the flask.

Place the distillation flask in the flask shield with its bulb resting on the gauze and connect the condenser tube to the tubulature of the flask with a tight cork joint, having the tubulature project 30 to 50 mm. through the cork. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. Support the condenser tube in a position such that it is in alignment with the tubulature of the flask, and the thermometer is vertical. Place the shield cover on the flask shield around the neck of the flask.

(b) *For Electric Heater Distillation.*—Attach the electric heater to a vertical support so that, at the end of the distillation, it can be lowered for a distance of at least six inches (15 cm.). Place the upper refractory on the heater with its larger opening facing upwards. Set the flask shield on the upper refractory.

Position the thermometer in the distillation flask as described in the second paragraph of (a).

Connect the condenser tube to the tubulature of the distillation flask with a tight cork joint, having the tubulature project 30 to 50 mm. through the cork. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. Place the distillation flask in the flask shield and support the assembly so that the bottom of the flask is between 4 and 7 mm. above the heating coils, the condenser tube is in alignment with the tubulature of the flask, and the thermometer is vertical. Place the shield cover on the flask shield around the neck of the flask.

Procedure

1. **Dehydration.**—If not more than 3.0 percent of water is present, the sample may be tested without previous dehydration. If the water content exceeds 3.0 percent, dehydrate a representative portion of the sample before distillation in accordance with the "Standard Method for the Dehydration of Oil-Type Preservatives", Section 7. In case of dispute the determination shall be carried out on a dehydrated sample.

2. **Distillation Test.**—Weigh the distillation flask to the nearest 0.05 g. and then weigh 100.0 ± 0.1 g. of the sample into it. Assemble the apparatus as described under Apparatus, Item 10.

Apply heat to the flask so that, 45 seconds after the first drop of distillate falls from the end of the condenser, the distillation rate is 80 to 100 drops per minute.[2] Maintain this rate throughout the distillation. If the sample contains over 1% of water, heat the flask and contents slowly until the vapor temperature reaches 170° C. to distill over water before continuing the distillation as described above. Warm the condenser tube whenever necessary to prevent accumulation of solid distillates in the tube.

Collect the distillate fractions in tared receivers at the points designated by the specifications, changing receivers as the thermometer indicates the maximum temperature, corrected as described in the following paragraph, for each fraction. The following fractions are specified:

Up to 210° C.
210° to 235° C.
235° to 270° C.
270° to 315° C.
315° to 355° C.

Do not change the position of the thermometer during the distillation. Make no correction for emergent steam of the thermometer, but if the laboratory's altitude is over 250 feet above sea level, adjust (but do not report) the temperatures in accordance with the text table.

When the maximum temperature specified for the test—corrected for altitude—is indicated by the thermometer, immediately remove the flame and the flask shield cover; or when the electric heater is used, immediately remove the flask shield cover and drop the electric heater a distance of at least six inches. Allow the apparatus to cool for at least 5 min., or until no vapors are visible. Drain any material remaining in the condenser tube into the receiver containing the last fraction. Weigh the receivers containing the distillate fractions to the nearest 0.05 g. Remove the cork and thermometer and weigh the flask and residue to the nearest 0.05 g.

Should the fraction to 210° C. contain water, determine the water volume[8] and calculate the net weight of oil distillate, assuming that 1 ml. of water weighs 1 g.

If tests of the residue are required, replace the cork and thermometer in the flask and lower the thermometer until its bulb is in the liquid residue. If the residue is not completely fluid, heat it carefully to a temperature not exceeding 150° C. by holding the bulb of the flask over a wire gauze heated by a gas burner or by immersion in a suitable bath whose temperature does not exceed 150° C. Incline the flask and rotate it so that the fluid residue will flow around the side, and collect any oils that have condensed on the upper surfaces of the flask. Mix the contents of the flask until they are homogeneous. Allow the residue to cool to a temperature at which it can be readily poured from the flask without loss of volatile material and then pour it into the desired testing equipment or into a suitable receptacle. Cover the receptacle.

Calculations

1. If the laboratory's altitude is more than 250 feet above sea level, change the temperatures listed in (2) under Procedure, above, to the temperatures in the following table: "Adjustment of Distillation Test Temperatures for Altitude."

Adjustment of Distillation Test Temperatures for Altitude to the Nearest One Degree (C)*

Altitude vs. Temperature of Cut

Ft. Above Sea Level	Temperature Degree C				
	210	235	270	315	355
500	209	234	269	314	354
1000	208	233	268	313	353
1500	208	232	267	312	352
2000	207	232	266	311	351
2500	206	231	265	310	350
3000	205	230	264	309	349
3500	204	229	263	308	347
4000	203	228	263	307	346
4500	203	227	262	306	345
5000	202	226	261	305	344
5500	201	225	260	304	343
6000	200	225	259	303	342
6500	199	224	258	302	341
7000	198	223	257	301	340

* Average barometer assumed

2. Convert the distillation results to the water-free basis by means of the following formulas:

I. Fraction to 210° C.:

Percentage (water-free basis) = (Wt. of fraction — W) × $\frac{100}{100 - W}$

II. Second and subsequent fractions including residue:

Percentage (water-free basis) = Wt. of fraction × $\frac{100}{100 - W}$

where W = ml. water, expressed as grams, in fraction distilling to 210° C.

3. Add the percentages (water-free basis) of the fractions to obtain cumulative percentages to the specified temperatures.

Report

Report the cumulative percentages to the specified temperatures and distillation residue, on a water-free basis, to the nearest 0.1 percent.

Precision

The following criteria should be used for judging the acceptability of results at the 95 percent probability level:

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 1.1 percent absolute.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 2.9 percent absolute.

Note

The estimated standard deviation of repeatability is 0.41 percent absolute at 272 degrees of freedom. The estimated standard deviation of reproducibility is 1.05 percent absolute at 352 degrees of freedom.

Notes

[1] The "Precision" Ful-Kontrol, 750-watt heater with built-in variable transformer control has been found satisfactory. This heater is available for 115 volts, 50/60 cycles only.

[2] If an electric metronome is not available to measure the distillation rate, not less than 20 drops nor more than 25 drops of distillate shall fall from the end of the condenser in the time interval 45 to 60 seconds after the first drop or in any given 15-second time interval thereafter.

[3] The amount of water contained in this fraction may be determined by either of the following methods:

(a) Transfer the fraction, after weighing, to a tube or cylinder graduated in 0.1 ml. Rinse the receiver several times with benzene, adding the rinsings to the tube or cylinder containing the fraction or,

(b) Collect the fraction to 210° C. in a tared 5-ml graduated cylinder having a flared top. After weighing, add benzene which will result in a clear separation of the water and oil distillate.

3. STANDARD METHOD FOR THE DETERMINATION OF THE AMOUNT OF MATERIAL INSOLUBLE IN XYLENE

Scope

This method covers the determination of the Xylene insoluble matter in creosote and creosote-coal tar solution. Since this method is empirical, strict adherence to all details of the procedure is necessary for close agreement of results among laboratories.

Apparatus and Reagents

1. **Filtering crucibles,** porcelain with fine-porosity bottom 40 ml. capacity, high form, maximum pore diameter, 7 μ. (Selas Grade 01, Size FC 40, or equivalent.)

2. Filter apparatus, filter flask and tube with crucible adapter, and means for producing a vacuum.

3. Balance and weights accurate to 0.5 mg.

4. Xylene—5-degree distillation range, conforming to ASTM Standard D-845.

5. Acetone, boiling at a pressure of 760 mm. Hg. within a range of 1.0° C., which shall include the temperature of 56.1° C.

6. Colite Analytical Filter Aid (CAFA), dry to constant weight at 105° C. and store in tightly stoppered container. (Do not use any other grade of filtering medium, because porosities differ.)

Safety Precautions

1. Use fresh, clean, moisture free xylene for each determination.

2. Xylene is toxic and extremely flammable, all operations with xylene must be carried out under an efficient head.

Procedure

1. Make and record all weighings to the nearest 0.5 mg.

2. Clean a crucible, if used for less than six determinations as follows: Remove the mat, wash the crucible with distilled water, dry and ignite in a muffle furnace for one hour at about 800° C. Cool the crucible slowly to prevent cracking and place it in a desiccator while still warm.

After a crucible has been used for six determinations, remove any residual ash from pores in the filtering area by boiling in 1:1 hydrochloric acid. Then boil the crucible in distilled water, thoroughly back wash with distilled water, dry, and ignite as above.

3. Transfer 0.45 to 0.55 g of Celite to a clean, filtering crucible. Distribute the Celite evenly over the bottom. Dry in an oven at 105 to 110° C. for 30 minutes. Cool in a desiccator and weigh. Record the weight of crucible plus Celite.

4. Take the original, undehydrated sample, and if necessary, heat and stir until any crystalline material is in solution and the sample is homogeneous. Determine the percentage of water in accordance with the "Standard Method for the Determination of Water in Oil-Type Preservatives."

5. Weigh the following size sample, into a 100 ml beaker:

New Creosote	10 ± 1 g
Used Creosote	5.0 ± 0.5 g
Creosote Solutions	2.0 ± 0.1 g

Calculate and record the weight of the sample.

6. Warm 50 ml. of xylene to a temperature of 50° to 60° C., and immediately add it to the sample while stirring thoroughly. Continue stirring until the sample is dispersed and the bottom of the beaker is clean. Bring the beaker or flask containing the solution to boiling on a hot plate.

7. Insert the filter tube with adapter in the filter flask and place the previously prepared and tared crucible in the adapter. Fill the crucible halfway with boiling xylene, and with the suction turned on, slowly pour the mixture containing the sample into the crucible before the xylene has been drawn entirely through the Celite. Take care that the Celite is never free from liquid, either during the addition of the solution containing the sample, or during the subsequent washing with xylene.

8. Wash the beaker thermometer, or stirring rod, and crucible with hot xylene. Pass all washes through the filter. Use a suitable policeman to sweep the insoluble particles into the crucible. Wash the crucible and contents with hot xylene, allowing each wash to pass almost through the filter before the next is added, until the washings are colorless.

9. Reduce the suction and wash the contents with acetone until the washings are colorless. Four additions of 5 ml. each are usually sufficient. Remove the crucible, and wipe the outside clean with a tissue moisture with xylene

Place the filtering crucible in the drying oven at 105° ± 5° C. and dry to constant weight (±0.5 mg). Record the weight of the filtering crucible and its contents.

Calculations

Calculate the xylene insoluble content as follows:

$$\text{xylene insoluble, percent} = 100 \times \frac{A - B}{C} \times \frac{100}{100 - D}$$

Where A = Total weight of filtering crucible, Celite plus matter insoluble in xylene.
B = Initial weight of filtering crucible containing dried Celite.
C = Weight of sample taken for determination.
D = Percentage water in the sample.

Precision

The following criteria should be used for judging the acceptability of results:

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.2.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 0.5.

3A. ALTERNATE METHOD FOR THE DETERMINATION OF THE AMOUNT OF MATERIAL INSOLUBLE IN XYLENE

Scope

This method is intended as an alternate procedure from that specified in the previous section. In case of a dispute, the referred method shall be Standard A1, Section 3.

Apparatus and Reagents

1. Refer to reagent specifications outlined in A1, Section 3.
2. Scale or balance accurate to three decimal places.
3. Filtering crucibles, porcelain with 40 ml capacity (top diameter 1-5/8″, bottom 1″) and bottom perforations 1/32″ (0.6 mm) diameter.
4. Glass microfibre filter pads with 1.2 um nominal pore retention and 2.4 cm in diameter.
5. 500 ml side-arm vacuum filtering flask, tapered rubber crucible holder with flared glass collection stem and a means of producing a vacuum.

Safety Precautions as Outlined in A1, Section 3

Procedures

1. Place a glass microfiber filter into a clean filtering crucible. Dry in oven for 1/2 hour at 105°C, then remove and cool in desiccator.
2. Weigh and record tare weights of crucible with glass filter and a clean 100 ml beaker.
3. Pour a well mixed, crystal free sample (10 g new oil, 5 g used oil) into the tared 100 ml beaker and record actual weight. A 2 g sample may be used for creosote solutions.
4. Heat 250–300 ml of xylene to 50–60°C and add 40–50 ml of this warm xylene to the sample in the beaker. Stir to dissolve the sample completely and heat to boiling (leave stir rod or thermometer in beaker).
5. Place crucible in holder on the vacuum flask and be sure filter pad is correctly positioned in the bottom.
6. Fill crucible one-half full with warm xylene and simultaneously turn on suction, then begin to pour sample mixture before crucible is empty (use only a partial vacuum if warm xylene empties too quickly from the crucible). Add sample to crucible, not letting the crucible be free of liquid. Care must be taken to be sure the filter mat does not float free from the bottom of the crucible.
7. Rinse beaker and stir rod with warm xylene and pass all washes through the crucible until washings are colorless.
8. Reduce or turn off suction and wash the contents with acetone until all washings are colorless. Four additions of 5 ml each are usually sufficient. Again avoid dislodging filter pad from its position.
9. Turn off vacuum and remove crucible from holder. Wipe outside of crucible with a clean tissue and place in oven at 105°C and dry to a constant weight (1/2 hour).
10. Cool crucible in a desiccator, then weigh and return to oven for 1/2 hour, cool and reweigh. Repeat until a constant weight is obtained.
11. Calculate xylene insoluble content:

$$\% \times \text{XI} = \frac{\textit{Weight of residue}}{\text{Weight of sample}} \times 100\%$$

12. Dispose of glass fibre filter and rinse crucible with solvent and wipe clean.

13. After several tests, carbon deposits may appear inside the crucible. These can be cleaned by firing over a bunsen flame.

Precision

Precision on statements for this method have not yet been determined, however a round robin comparison of results by this method to results of the standard method showed no statistical difference to exist.

4. STANDARD METHOD FOR THE DETERMINATION OF THE SPECIFIC GRAVITY OF OIL-TYPE PRESERVATIVES

Scope

This method is suitable for the determination of the specific gravity of oil-type preservatives. To determine the specific gravities of distillation fractions, see Section 5.

Apparatus

1. A set of three hydrometers covering the range 1.000 to 1.150 and conforming to the requirements for hydrometers 125H (range 1.000 to 1.050), 126H (range 1.050 to 1.100), and 127H (range 1.100 to 1.150) as prescribed in the specifications for ASTM Hydrometers (ASTM Designation: E 100).

If not available, hydrometers (60°/60° F.) in the range 1.000 to 1.150 of similar accuracy and having a scale length not less than 9.5 mm. per 0.010 units of specific gravity may be used.

2. Hydrometer cylinder of glass having the following approximate dimensions:

Length	Approximately 300 mm.
Diameter	Not less than 32 mm.

3. ASTM Low Softening Point Thermometer having a range of —2° to 80° C. (30° to 180° F.) conforming to the requirements for Thermometer 15 C (15 F) as described in the Specifications for ASTM Thermometers (ASTM Designation: E 1); or other thermometer of suitable range and precision.

Procedure

Place the oil in the cylinder to a depth sufficient to float the hydrometer and, with thorough stirring to ensure uniformity, adjust the temperature to 38° C. (100° F.) or, if not entirely liquid at that temperature, to the lowest temperature at which the oil is completely liquid.[1] Insert the hydrometer and allow the instrument to settle until it is in floating equilibrium. Then read the height of the meniscus on the stem of the hydrometer and the temperature to the nearest 0.1° C. (0.2° F.). Add to the hydrometer reading an increment of specific gravity equivalent to 1 mm. on the scale. Then correct this observed specific gravity at the observed temperature to the specific gravity at 38°/15.5° C. using Table II of Standard F1, entitled "Factors to be used for determining the Specific Gravity at 100° F. When Observed at Temperatures Ranging from 60° to 220° F."

Report

Report the specific gravity at 38°/15.5° C. to the nearest one thousandth unit of specific gravity.

Precision

The following criteria should be used to judge the acceptability of results at the 95 percent probability level:

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.002.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 0.004.

Note

The estimated standard deviation of repeatability is 0.0003 at 14 degrees of freedom. The estimated standard deviation of reproducibility is 0.0014 at 22 degrees of freedom.

5. STANDARD METHOD FOR THE DETERMINATION OF THE SPECIFIC GRAVITY OF DISTILLATION FRACTIONS AND RESIDUE

Scope

This method of test is suitable for the determination of the specific gravities of the distillation frac-

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Note

[1] It is advantageous to have the cylinder immersed in a bath maintained at the temperature of test.

tions and residue of Creosote and Creosote Solutions. It is also suitable for determining the specific gravity of quantities of oil-type preservatives too small to be determined by the hydrometer method. Section 4.

Apparatus

1. Pycnometers:
    - (a) Gay–Lussac type, 10 ml. and 25 ml. capacity.
    - (b) Hubbard–Carmick type, 25 ml. capacity.
2. Water bath maintained at 38.0° ± 0.1° C.
3. Balance and weights accurate to 0.5 mg.
4. ASTM Low Softening Point Thermometer having a range of —2° to 80° C. and conforming to the requirements for Thermometer 15 C as prescribed in the specifications for ASTM Thermometers (ASTM Designation: E 1).

Calibration of Pycnometers

Before calibration, grind the stopper into the neck of the pycnometer by partially filling the pycnometer with water, inserting the stopper into the neck, and rotating the stopper by hand with very light pressure. During this operation, keep the ground surfaces wet and occasionally flush with water. When the wet stopper can be rotated freely with no tendency for the ground surfaces to stick together, using no more pressure than the weight of the stopper, the pycnometer is in condition for calibration.

Thoroughly clean the pycnometer with hot chromic acid cleaning solution. Empty the acid from the pycnometer, flush thoroughly with distilled water, dry in an oven at about 110° C., cool in a desiccator for 15 to 30 minutes, and weigh to the nearest 0.001 g. Designate this weight P.

Fill the pycnometer with freshly boiled distilled water at room temperature. (A pipette or drawn-out medicine dropper facilitates this operation.) Insert the stopper with a rotary motion to secure a firm seat, making sure that no air is entrapped, then completely immerse the pycnometer in the water bath at 38.0° C. for 30 minutes.

While still in the water bath, raise the pycnometer so the top of the stopper is slightly above the water level in the bath and wipe off the water on the flat top surface of the stopper with soft absorbent paper, taking precautions not to remove water from the capillary tube. Remove the pycnometer from the water bath, immerse its lower half momentarily in cold water, dry its surface and weigh it to the nearest 0.001 g. Then remove the stopper, add distilled water and reinsert the stopper as described above. Return the pycnometer to the water bath for 30 minutes, remove, and weigh as before. (It is advisable to have the previously determined weights on the balance pan to expedite the reweighing). Repeat this operation until three successive weighings check within 0.010 g.; if this proves impossible, regrind the stopper and repeat the calibration until three weighings agree within 0.010 g. Designate this weight W_1. Remove the water and thoroughly dry the pycnometer in an oven at about 110° C.

Procedure

1. **Fractions Entirely Liquid Below 38° C.**—Carefully heat the fraction to a temperature below 38° C. in a water bath or by direct heat with an asbestos board under the container until the distillation fraction is entirely liquid. Fill the dry Gay–Lussac pycnometer by means of a warm pipette or warm drawn-out medicine dropper until the neck is about one-half full, avoiding the inclusion of air bubbles. Insert the stopper with a rotary motion to secure a firm seat, making sure no air is entrapped. Place the filled pycnometer in the water bath at 38.0° C. so that the top of the stopper is slightly above the water level, and allow to remain for at least 30 minutes. All this time the capillary tube should be completely filled with oil. Carefully wipe off with soft absorbent paper any oil from the top flat surface of the stopper while the pycnometer is still in the water bath. Remove the pycnometer from the water bath, immerse its lower half momentarily in cold water, dry its surface and weigh it to the nearest 0.001 g. Remove the stopper, refill the pycnometer with liquid fraction, and repeat the determination as described above until two successive weighings agree within 0.010 g. Designate this weight W_2.

2. **Fractions Containing Solids at 38° C.**—Carefully heat the fraction in a water bath or by direct heat with an asbestos board under the container until the distillation fraction is entirely liquid. By means of a warm pipette or warm drawn-out medicine dropper, transfer a sufficient amount of the fraction to the dry Gay–Lussac or Hubbard–Carmick pycnometer until it is approximately one-half full, avoiding the inclusion of air bubbles and contact of the oil with the ground glass surface of the neck of the pycnometer. (Permitting the stream of liquid to impinge on the side of the pycnometer below the ultimate liquid level aids in preventing inclusion of air bubbles.) Cool the pycnometer to room temperature and weight with the stopper. Designate this weight W_3.

Add freshly boiled distilled water to the partially filled pycnometer until it is about three-quarters full. Partially immerse the pycnometer without the stopper in a small water bath, maintained at 90° to 95° C., and allow to remain until the fraction is liquid and free of air bubbles. (Any entrapped air bubbles can be removed with a heated fine wire loop.)

Cool the pycnometer and contents to a temperature of about 25° C. and then add cool freshly boiled distilled water until the neck is about one-half full. Insert the stopper with a rotary motion to secure a firm seat, making sure that all air is excluded. Completely immerse the pycnometer in the water bath at 38.0° C. and allow sufficient time for an equilibrium crystal state to be established. One hour is usually sufficient. While still in the water bath, raise the pycnometer until the top of the stopper is above the water level and wipe off the water on the flat surface of the stopper with soft absorbent paper, taking precautions not to remove water from the capillary tube. Remove the pycnometer from the water bath, immerse its lower half momentarily in cold water, dry its surface and weigh it to the nearest 0.001 g. Remove the stopper, add freshly distilled water, reinsert the stopper, and return the pycnometer to the water bath at 38.0° C. After 30 minutes, remove and weigh the pycnometer as described above. Repeat these operations until two successive weighings agree within 0.010 g. Designate this weight W_4.

3. **Distillation Residue.**—Melt the distillation residue as described in the Standard Method of Distillation (see Section 2, last paragraph of Procedure 2). Carry out the determination as described in Procedure 2 above, using the Hubbard-Carmick pycnometer, but omitting the liquefaction step in the second paragraph of that procedure.

Calculations

1. Fractions Entirely Liquid Below 38° C.
Specific Gravity at 38.0°/15.5° C. =

$$\frac{W_2 - P}{W_1 - P} \times 0.99393$$

2. Fractions Containing Solids at 38° C. and distillation residue

Specific Gravity at 38.0°/15.5° C. =

$$\frac{W_3 - P}{(W_1 - P) - (W_4 - W_3)} \times 0.99393$$

Where, P = Weight of empty pycnometer
W_1 = Weight of pycnometer full of water
W_2 = Weight of pycnometer full of liquid fraction
W_3 = Weight of pycnometer partially filled with solid fraction or distillation residue
W_4 = Weight of pycnometer full of solid fraction or residue and water
0.99393 the ratio of the density of water at 38.0° C. to the density of water at 15.5° C., i.e.,

$$\frac{0.99299}{0.99905}$$

Report the specific gravity at 38.0°/15.5° C. to the nearest one thousandth unit of specific gravity.

Precision

The following criteria should be used to judge the acceptability of results at the 95 percent probability level:

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.005.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 0.010.

Note

The estimated standard deviation of repeatability is 0.0018 at 54 degrees of freedom. The estimated standard deviation of reproducibility is 0.0036 at 27 degrees of freedom.

6. STANDARD METHOD FOR THE DETERMINATION OF WATER IN OIL-TYPE PRESERVATIVES

Scope

This method is suitable for determining water in oil-type preservatives. The sample is distilled with a volatile solvent, therefore the dehydrated material cannot be used for other required tests. (See Section 7 for dehydration procedure).

Apparatus

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 assem-

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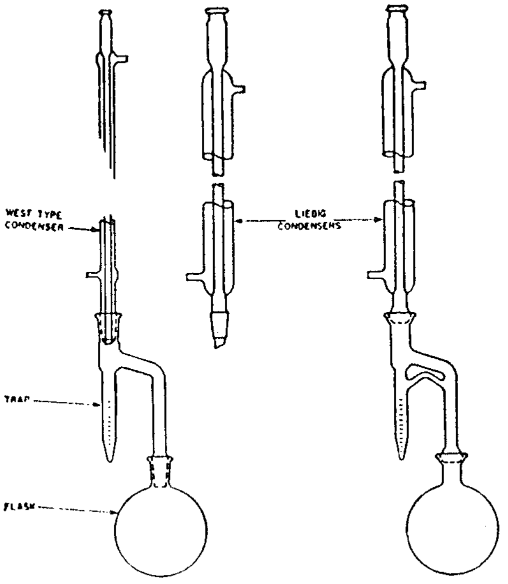
Note

[1] Calibration of the pycnometer is not necessary prior to making each determination. Dependent on usage, recalibration should be done at sufficient time intervals to insure that the calibration is accurate. In case of dispute, calibration is mandatory.

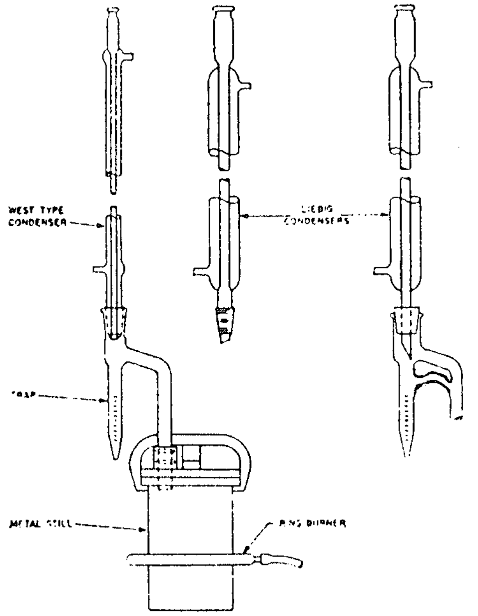


Note: Trap shall be 15 to 16 mm. inside diameter.
Figure 10.—Typical assemblies with glass flask.

blies are illustrated in figures 10 and 11 reproduced from ASTM Standard D95.

1. **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.

2. **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.



Note: Trap shall be 15 to 16 mm. inside diameter.
Figure 11.—Typical assemblies with metal still.

3. **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.

4. **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.

Reagents

For general use an aromatic solvent is preferred since it has high solvency and dispersing power for most bituminous materials. Xylene, or a blend of 20 percent benzene and 80 percent xylene is recommended.

Sample

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 ensure uniformity, 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.

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

1. Transfer 100 ml. of sample, measured with an accuracy of ± 1 percent, to the still. Measure 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. When the sample to be tested contains more than 10 percent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 ml. of water.

2. Assemble the components of the apparatus as illustrated in Figures 10 and 11, 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.

3. 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.

4. 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.

Calculation

Calculate the water in the sample as percent by volume as follows:

$$\text{Water, percent} = \frac{\text{vol. of water in trap}}{\text{vol. of sample}} \times 100$$

Report

Report the determined percentage of water to the nearest 0.1 percent.

Precision

The following criteria should be used for judging the acceptability of results:

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 0.2 percent absolute.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 0.4 percent absolute.

Variations: The type of apparatus joint, method of heating the still, solvent used, trap size and type of still within the limits given in the section on apparatus do not affect this precision statement.

7. STANDARD METHOD FOR THE DEHYDRATION OF OIL-TYPE PRESERVATIVES

Scope

This method is suitable for the dehydration of oil-type preservatives prior to a distillation test.

Sampling

The portion of sample removed for dehydration must be representative of the total sample. Thoroughly stir the sample as received, warming if necessary to ensure liquidity and uniformity, and remove a representative portion for dehydration.

Apparatus

1. **Still.**—A vertical, cylindrical copper still, with removable flanged top and yoke, of the form and approximate dimensions shown in Figs. 12 and 13.

2. **Thermometer.**—A thermometer conforming to the specification given in "Standard Method of Distillation" (see Section 2).

3. **Condenser.**—Copper trough condenser with straight-walled glass tube, having approximately the form and dimensions shown in Fig. 12.

4. **Separatory Funnel.**—A separatory funnel of the form shown in Fig. 12 having a total capacity of 120 ml. with outlet graduated in fifths of a milliliter, or a separatory funnel of the Squibb type having a capacity of about 100 ml.

5. **Assembling Apparatus.**—Assemble the apparatus as shown in Fig. 12.

Procedure

Transfer 200 to 300 ml. of the portion of the sample removed for dehydration to the copper still.[1] Clamp the lid on, using a paper gasket slightly wet with the oil under test around the flange of the still. Apply heat by means of the ring burner, which shall be placed just above the level of the oil in the still at the beginning of the dehydration and gradually lowered when most of the water has distilled over. Continue the distillation until the vapor temperature reaches 205° C., as indicated by the thermometer with the bulb opposite the off-take of the connecting

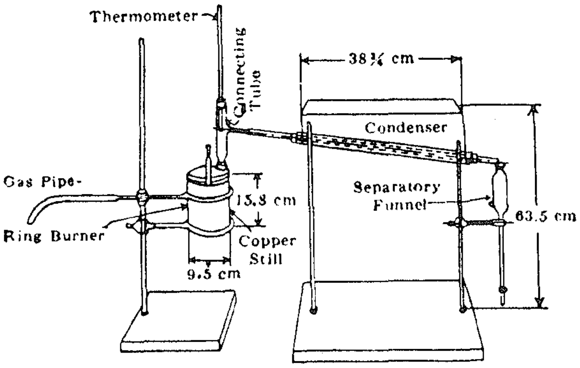


Figure 12.

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[1] When free water is present in the portion of the sample removed for dehydration, separate, if possible, before the transfer.

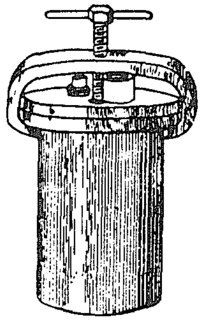

Figure 13.

tube. Collect the distillate in the separatory funnel. When the distillation is completed, and a clear separation of water and oil in the funnel has taken place,[2] return any light oil distilled over with the water to the oil in the still and mix thoroughly before further testing.

8. STANDARD METHOD FOR DETECTING THE PRESENCE OF SMALL AMOUNTS OF PETROLEUM OIL IN COAL TAR CREOSOTE

Scope

This method of test is suitable for showing the presence of two percent or more "paraffinic" petroleum oil or three percent or more "aromatic" petroleum oil in coal tar creosote. The results obtained are qualitative, not quantitative.

Outline of Method

The sample of creosote is dehydrated, after which 25 ml. is distilled to coke. A 4.0 ml. portion of the total distillate is shaken with 6.0 ml. of a mixture of triethyleneglycol (95 percent by volume) and furfural (5 percent by volume) in a 15-ml centrifuge tube. After centrifuging the tube and contents, the volume of insoluble material in the top layer is read and the amount of insoluble calculated.

Apparatus

1. **Distillation Flask.**—As used in AWPA A1, Section 2.

2. **Distillation Flask.**—50-ml. capacity with side tube 77 mm. below mouth at 75° angle. A. H. Thomas Co. 5397. *Use only once.*

3. **Condenser Tube.**—As used in AWPA A1, Section 2.

4. **Centrifuge Tubes.**—15-ml. capacity and graduated in 0.1. ml. Length 120 mm., outside diameter 17 mm. A. H. Thomas Co. 2994–H10.

5. **Centrifuge.**—Motor driven, to hold 15-ml. centrifuge tubes. "Precision" Centricone, Clinical Model, which costs less than $100.00, is satisfactory.

6. **Burner.**—Bunsen type.

7. **Magnifier.**—Pocket type, low power.

Reagents

1. Triethyleneglycol (TEG); Matheson, Coleman & Bell #5809. Must be stored over Drierite or other suitable desiccant.

2. Furfural (F); purified and free of water (Baker & Adamson Code 1796). Redistill if discolored discarding first 10% by volume and using the next 50% by volume distilled.

3. Mixture of 95 percent by volume of TEG and 5 percent by volume of F; store over suitable desiccant such as Drierite.

Procedure

1. **Dehydration.**—Heat the creosote sample to complete liquidity at a temperature not exceeding 75°C. Pour about 100 ml. of the limpid sample into the AWPA distillation flask, stopper the flask with a rolled cork stopper, and attach the condenser by means of another cork stopper.

Heat the sample to boiling by means of a 20-cm long flame, holding the burner by hand and intermittently heating the top of the unprotected flask and the side tube to vaporize condensed water. Boil the sample for a few minutes. If more than 5 ml of distillate is collected, separate the water that has co-distilled, using salt if necessary to effect separation (see A1, Section 7). Return the dry distillate to the cool dehydrated sample.

2. **Distillation to Coke.**—Transfer 25 ml of the dehydrated sample to a 50-ml distillation flask. Stopper the flask with a rolled cork and connect the condenser by means of another cork. Suspend the assembly so that the bottom of the bulb is 7 to 8 cm above the top of the gas burner.

Heat the sample to boiling by means of a small (6cm) flame, holding the burner by hand and occasionally heating the top of the unprotected flask. When boiling starts, place the burner on the table and adjust the flame so that the rate of distillation is maintained at between 100 and 120 drops per minute. Collect the distillate in a small Erlenmeyer flask suspended from the end of the condenser. Towards the end of the distillation maximum gas input must be used, with the tip of the inner cone directly under the bottom of the flask. Remove the flame the moment the flask is dry. Gently heat the condenser tube and drain its contents into the receiver. Mix the distillate thoroughly and *use within 6 hours* for solubility testing.

3. **Solubility Test.**—Transfer 4.0 ml (4.3 g) of the cooled total distillate to a centrifuge tube. Centrifuge the tube and contents for a few minutes. Then clamp the tube in a vertical position before a strong light source and read the sample volume

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[2] Separation of water and oil in the separatory funnel may be more readily accomplished by the addition of a concentrated solution of sodium chloride and slight heating.

to the nearest 0.05 ml at the top of the clearly defined vertical light streaks in the oil, using a small magnifier to facilitate the reading. If the volume is outside the range 3.90 to 4.10 ml, adjust by adding or removing distillate sample. Then add 5.8 to 6.2 ml of solvent solution and stopper the tube with a rolled cork that must not enter more than 8 mm into the tube. Shake the tube and contents vigorously for 30 seconds, remove the cork stopper, and wipe its bottom on the top of the tube. Fill another centrifuge tube partly with water to counterbalance the tube with sample and solvent, then place the tubes in the centrifuge. Run the latter for 30 minutes or longer, until the difference between meniscus levels no longer changes. Again clamp the tube in a vertical position before a strong light source and read and record the volume sample plus solvent at the highest horizontal meniscus line to the nearest 0.05 ml. If an interface between two layers is visible, read and record its position to the nearest 0.05 ml. In case crystals deposit near the liquid surface, heat this location momentarily with a small flame and slightly shake the vertically held tube in a horizontal motion before making the readings.

Calculations

$$\text{Insoluble matter, percent volume} = \frac{a-b-0.35^{*}}{4.0} \times 100$$

where:

a = Reading at horizontal line at top of the meniscus, ml.

b = Reading at interface between layers, ml.

Traces of insoluble matter appear as tiny globules and should be so reported. Absence of such traces and of a light-colored top layer indicates complete miscibility of the total distillate in the solvent.

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* A factor to correct the reading at the top of the meniscus to the true volume of sample plus solvent.

9. METHOD FOR DETERMINING CONFORMANCE OF DISTILLATION FRACTIONS OF CREOSOTE OR CREOSOTE SOLUTION TO SPECIFIC GRAVITY REQUIREMENTS

Scope

This method is suitable for determining whether or not the specific gravity of a distillate fraction or the distillation residue of creosote or creosote–coal tar solution is greater than a given specific gravity. In cases of dispute, the pycnometer method shall be used.

General

This proposed method employs the principle of sink or float of a droplet of the distillation fraction in a salt solution of predetermined specific gravity and temperature set as a minimum requirement for the fraction under test.

Apparatus and Equipment

1. **Constant temperature bath**—Controlled bath for maintaining temperature at 38° C. ± 0.2° C.
2. **Volumetric flasks**—glass stoppered.
   1–500 ml.
   2–200 ml.
3. Beakers 100 ml.
4. Two glass-stoppered bottles 500 ml.
5. Test tubes (125x15mm.).
6. Rack or clamps for holding test tubes in the bath.
7. Medicine droppers drawn out to moderately fine point.
8. Thermometers of suitable range, i.e., 38° C.
9. Polyethylene squeeze bottle—500 ml.

Chemicals and Reagents

1. Potassium chloride (CP).
2. Detergent Aerosol AY.
3. Reagent water meeting the requirements of ASTM Standard D 1193 for nonreferee reagent water.

Preparation of Stock Solutions

1. Solution A to 500 ml. of distilled water, add 0.5 grams of Aerosol AY and mix thoroughly.

2. Solution B, transfer about 125 ml. of Solution A to a 200 ml. volumetric flask and place in bath at 38° C. Weigh 10.4 grams of potassium chloride in a tared 100 ml. beaker. Add 40 to 50 ml. of Solution A and dissolve the salt by warming to about 38° C. Add the salt solution to the volumetric flask in the bath and rinse the beaker and thermometer with Solution A from a squeeze bottle. Stopper flask and mix. Bring flask and contents to equilibrium temperature in the bath. Add Solution A to the mark and mix again. Store in a clean, glass-stoppered bottle. This bottle should be labeled "For Specific Gravity of 235° to 315° C. Fraction—1.027."

3. Solution C, repeat as in 2 above, except that 33.3 grams of potassium chloride are used. Label the stock bottle "For Specific Gravity of 315° to 355° C. Fraction—1.095."

4. Stock solutions should be checked by pycnometer comparing distilled water with stock solutions at 38° C.

Test Procedure

1. Fraction 235° to 315° C., fill a test tube with Solution B. Hold the tube by means of a clamp or rack in the bath at 38° C. Warm a clean, dry medicine dropper and partially fill with the liquefied fraction. Insert the tip about one to two inches below surface of Solution B and force out a drop of the oil. If droplet clings to dropper, gently jar it loose. Let stand for five minutes; if drop sinks, the requirement is met.

2. Fraction 315° to 355° C. Repeat "Procedure 1" with Solution C and the Fraction 315° to 355° C. after liquefying. Use a clean, fresh dropper. Again, if the drop sinks after five minutes, the requirement is met.

10. STANDARD 5-BALL COLUMN METHOD OF DISTILLATION OF CREOSOTE, CREOSOTE SOLUTION AND CREOSOTE PETROLEUM SOLUTION

Scope

This method of test is suitable for the distillation of creosote, creosote-coal tar solution, creosote-petroleum solution—either new or working oils—or the extracted preservative residuals from treated materials. It is a method for use in characterization of these preservatives or preservative residuals by their distillation properties when such characteristics are important in connection with laboratory or field evaluation tests. The distillation pattern obtained by the method correlates closely with those obtainable by fractional distillation and by gas liquid chromatography.

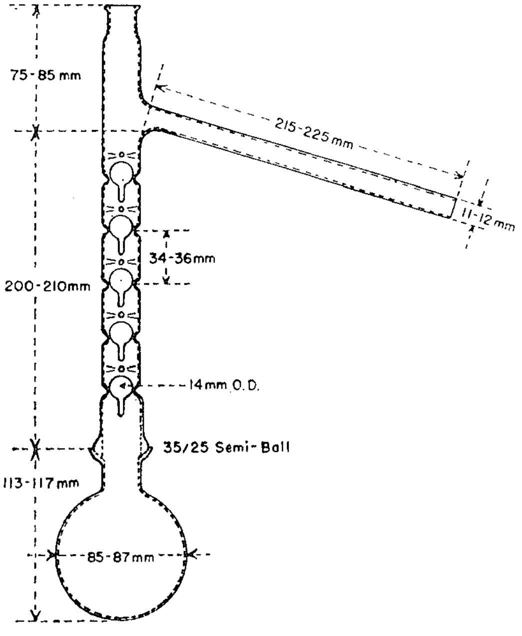

Figure 1.—5-Ball distillation column and flask.

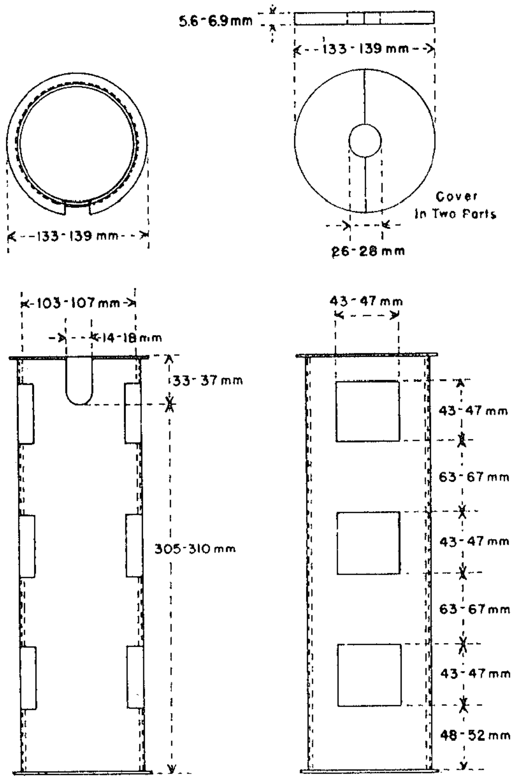

Figure 2.—Distillation shield and cover.

Apparatus

1. Distillation Flask. Round bottom, outside diameter 86.0 ± 1.0 mm., with 35/25 outer semi-ball connection in short neck; 300 ml. capacity.

2. Fractionating Column. Snyder 5-ball, 25 mm. outside diameter with 35/25 inner semi-ball connection at lower end; shown in Figure 1.

3. Condenser Tube. Tapered, conforming to the following dimensions:

Outside diameter of small end	12.5 ± 1.5 mm
Outside diameter of large end	28.5 ± 3.0 mm
Length	360 ± 4.0 mm
Length of tapered part	100 ± 5.0 mm

4. Flask and Column Shield. A cylindrical metal shield line with 3.2 mm. (1/8 in.) asbestos, riveted to the metal; provided with a cover comprising two pieces of 6.4 mm. (1/4-in.) asbestos cement board; and fitted with transparent windows as shown in Figure 2.

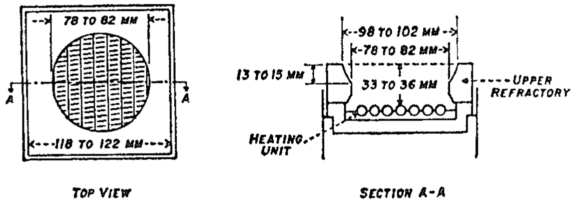

Figure 3.—Upper part of electric heater.

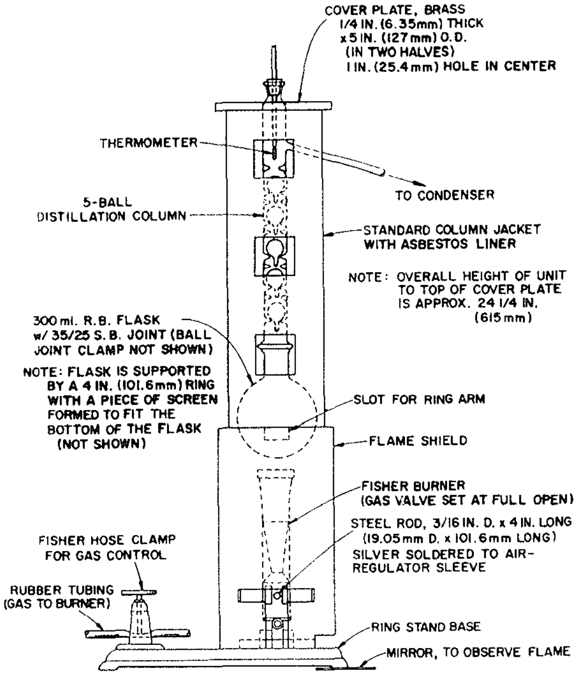

Figure 4—Burner shield for flame distillation

5. Source of Heat

(a) Flame. Bunsen or Meker type burner, or

(b) Electric Heater. Output variable to 600 or 750 watts with removable upper and lower refractories, as illustrated in Fig. 3, provided with variable transformer or rheostat suitable for the voltage used (Note 1) and fitted with clamp for mounting on vertical support rod.

6. Burner Shield for Flame Distillation. A cylindrical metal shield, 95 to 105 mm. in diameter, and having a peephole 25 mm. in diameter centered about 32 mm. below the ring support. The top of the shield shall be flanged to permit its being suspended from the ring support. The height of the burner shield shall be appropriate for the type of burner used. See Fig. 4.

7. Gauzes for Flame Distillation. Two sheets of 20-mesh wire gauze made of No. 26 B and S gauge nichrome wire, and 125 to 150 mm. in diameter or square.

8. Shield for Electric Heater Distillation. A metal shield fitted with mica windows, and cover of the same construction and dimensions as those used for flame distillation (Section 6) except for the height of the shield. See Fig. 5.

9. Receivers. Erlenmeyer flasks or beakers having a capacity of 50 to 125 ml. tared to the nearest 0.05 g.

10. Balance and Weights. A balance and weights accurate to 0.05 grams.

11. Thermometer. An ASTM High Distillation thermometer having a range of —2 to 400C. and conforming to the requirements for Thermometer 8 C as prescribed in the Specifications for ASTM Thermometers (ASTM Designation: E1).

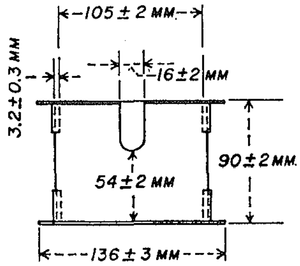

Figure 5.—Shield for use with electric heater.

12. Some chemical supply houses do not stock all of the above apparatus. The Scientific Glass Apparatus Co., Bloomfield, N. J. usually carry complete stocks.

13. Usual laboratory apparatus as required.

14. Apparatus Assembly

(a) For Flame Distillation. Suspend the burner shield by its flange from the support ring. Place the flask and column shield on the upper sheet of gauze. Insert the thermometer through a cork in the top of the 5-ball column so that the top of the thermometer bulb is level with the lowest point of juncture between the tubulature and the column, inside, and the stem is aligned on the axis of the column. Connect the 5-ball column to the charged distilling flask by means of a semi-ball clamp, using a heavy silicone grease to lubricate the semi-ball connections. Place the assembled column and flask in the flask and column shield with the bottom of the flask resting on the gauze. Place the pieces of the cover on the flask shield around the neck of the column. Connect the condenser tube to the tubulature of the column with a tight cork joint, having the tubulature project 30 to 50 mm. through the cork. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. Support the condenser tube in a position such that it is in alignment with the tubulature of the column and the thermometer is vertical.

(b) For Electric Heater Distillation. Attach the electric heater to a vertical support so that, at the end of the distillation, it can be lowered for a distance of at least 15 cm. (6 in.). Place the upper refractory on the heater with its large opening facing upwards. Set the flask and column shield on the upper refractory. Place the distillation flask in the flask and column shield and support the assembly so that the bottom of the flask is between 4 and 7 mm. above the heating coils. Connect the condenser tube as in (a) above.

Procedure

1. Preparation of the Sample

(a) Thoroughly stir or otherwise mix the sample immediately before removing the portion for testing, warming if necessary to insure a solution free from crystallized solids. Take care to avoid loss of volatile material.

(b) If the sample contains more than 1.0 percent of water, dehydrate a representative sample before distillation in accordance with AWPA Standard Method for the Dehydration of Oil-type Preservatives (A1, Section 7).

2. Distillation Test. Weigh the distilling flask, and also the condenser tube, to the nearest 0.1 gram. Weigh 100.0 ± 0.1 grams of the sample into it. If insufficient material is available for a 100-gram charge, the test may be carried out with not less than 60 grams weighed to the nearest 0.05 gram. If the distillation residue of the creosote is known to be 15 percent or less, carry out the test with not less than 150 grams nor more than 200 grams, weighed to the nearest 0.1 gram. Assemble the apparatus as described in Section 14 under Apparatus.

Apply heat to the flask by means of flame or electric heater. When the condensate forms at the bottom of the column reduce the heat so that the ring of condensate continues to rise slowly at a uniform rate. Adjust the rate of distillation, (Note 2) and subsequently maintain that rate, so that from 35 to 45 drops per minute fall from the end of the take-off and condenser tubes. Warm the condenser tube whenever necessary to prevent accumulation of solid distillates in the tube.

Calculations

1. Collect the distillate fractions in tared receivers, changing receivers as the thermometer indicates the maximum temperature for each specified fraction. The following fractions shall be collected:

Up to 200C
200 to 210C
210 to 235C
235 to 270C
270 to 315C
315 to 355C

Do not correct for emergent stem of the thermometer, but if the barometric pressure is not within the range 756 to 765 mm. of mercury adjust the temperature according to the table.

Adjustment of Distillation Test Temperatures for Barometric Pressure

Barometric Pressure mm of Mercury	Fractionation Temperatures for Various Barometric Pressure Ranges, deg. Cent.				
786 to 795	212	237	272	317	357
776 to 785	211	236	271	316	356
766 to 775	211	236	271	316	356
756 to 765	**210**	**235**	**270**	**315**	**355**
746 to 755	209	234	269	314	354
736 to 745	209	234	269	314	354
726 to 735	208	233	268	313	353
716 to 725	208	233	267	312	352
706 to 715	207	232	267	312	351
696 to 705	207	231	266	311	351
686 to 695	206	231	265	310	350
676 to 685	205	230	265	309	349
666 to 675	205	230	264	309	348
656 to 665	204	229	264	308	348
646 to 655	204	228	263	307	347
636 to 645	203	228	262	307	346
626 to 635	202	227	262	306	345
616 to 625	202	226	261	305	345
606 to 615	201	226	260	305	344
596 to 605	201	225	260	304	343

When the maximum temperature of 355C is indicated by the thermometer, immediately remove the heat source and the flask-shield cover.

Drain any oil remaining in the condenser tube into the receiver containing the last fraction. Weigh the condenser tube to the nearest 0.1 gram and add the positive difference, if any, between the before and after weights to the last fraction. Weigh the receivers containing the distillate fractions to the nearest 0.05 gram and record the weight of each fraction.

2. Convert the distillation results to the water-free basis by means of the following formulas:

I. Fraction to 210° C.:

$$\text{Percentage (water-free basis)} = (\text{Wt. of fraction} - W) \times \frac{100}{100 - W}$$

II. Second and subsequent fractions including residue:

$$\text{Percentage (water-free basis)} = \text{Wt. of fraction} \times \frac{100}{100 - W}$$

where W = ml. water, expressed as grams, in fraction distilling to 210° C.

3. Add the percentages (water-free basis) of the fractions to obtain cumulative percentages to the specified temperatures.

Report

Report the cumulative percentages to the specified temperatures and distillation residue, on a water-free basis, to the nearest 0.1 percent. Report the residue by difference.

Precision

The following criteria should be used for judging the acceptability of results at the 95 percent probability level: (Note 3)

Repeatability

Duplicate values by the same operator should not be considered suspect unless they differ by more than 1.1 percent absolute.

Reproducibility

The values reported by each of two laboratories should not be considered suspect unless the values differ by more than 2.9 percent absolute.

Notes

1. The "Precision" Ful Kontrol 750-watt heater with built-in variable transformer control has been found satisfactory. This heater is available for 115 volts, 50/60 cycles only.

2. An electric metronome is preferred.

3. The figures given below are the same as those for standard A1 flask distillation. More precise figures are not currently available for the 5-ball column method or for relating precision to degrees of freedom.

11. STANDARD METHOD OF DETERMINING LIQUIDITY OF CREOSOTE AND CREOSOTE SOLUTIONS

1.0 Application of Test

1.01 The determination of liquidity by this method is applicable to creosote oils, creosote-coal tar solutions, and similar tar oils at specified temperatures between the range of 5°C and 38°C.

1.02 This method is also applicable for determining the percent dry salts in such oils at the specified temperature.

2.0 Apparatus

2.01 The apparatus shall consist of the following: Erlenmeyer flasks, 200 ml, 100 ml, 50 ml capacity. Dewar flask, approximately 4300 ml capacity, A.H.T. Co. No. 5385-K or equivalent. Asbestos or corrugated paper board cover for the Dewar flask, with an opening for a thermometer.

Wire screen approximately 4 inches square. Copper or other metal wire, approximately 12 gauge, 4 pieces approximately 10 inches in length each, suitable for use as hooks in hanging the wire screen as a platform in the Dewar flask.

Crucible, monel metal, Gooch type, approximately 4.4 cm width at top, tapering to 3.6 cm at bottom, and covered with 200 mesh wire cloth at the bottom, Newark Wire Cloth Co., Newark, N.J.

Filtering flask with crucible holder for filtering under vacuum. Water bath maintained at the specified temperature ± 0.2°C.

Balance, accurate to 0.1 gm, Thermometer, —2 to 80°C, ASTM Low Softening Point.

Laboratory press or other suitable means for pressing wet salts. Absorbent filter paper.

3.0 Preparation of Sample

3.01 The sample shall be truly representative of the oil to be tested.

3.02 Heat the sample carefully until completely liquid, without loss of volatile components by overheating, and mix thoroughly until completely uniform.

4.0 Preparation of the Dewar Flask

4.01 Trim the corners of the wire screen with tin snips, or bend them over so that the screen will freely fit into the Dewar flask to form a shelf or platform.

4.02 Loop the pieces of 12 gauge wire into a hook at one end and fasten one length of wire at each of the four rounded corners of the wire screen, and hang in the Dewar flask by bending the free ends of the wire hooks over the top rim of the flask so that the screen forms a level shelf or platform approximately 7.5 inches from the top.

4.03 Fill the Dewar flask with water at 10°C below the temperature specified for the liquidity test, to a point approximately one-half inch below the level of the screen platform before adjusting the screen in place.

5.0 Procedure

5.01 Determination of percent liquidity.

5.011 Weigh 100 grams ±0.1 (Note 9.01) of the well-mixed, completely liquid sample into a 200 ml Erlenmeyer flask, and adjust temperature to 30°C above the temperature specified for the liquidity test.

5.012 Place the Erlenmeyer flask in the previously prepared Dewar flask by resting it on the screen platform. Adjust the asbestos or corrugated board cover over the Dewar flask, with the thermometer in place in the oil and extending out through the hole provided for it in the cover.

5.013 Allow the oil to cool slowly by stirring 15 seconds with the thermometer at 15 minute intervals. When the oil reaches the temperature specified for the liquidity test in 1 hour ± 10 minutes, immediately transfer the Erlenmeyer flask to the water bath maintained at the specified test temperature ± 0.2°C. Stir the contents of the Erlenmeyer flask 15 seconds at 15 minute inervals for a period of 3 hours in the water bath maintained at the proper temperature.

5.014 Tare the monel metal crucible to the nearest 0.1 gm and set up for vacuum filtration. Remove the Erlenmeyer flask from the water bath at the end of the 1-hour period and immediately filter its contents, using a partial vacuum at the start, and increasing the suction as the filter cake forms, to a full suction within one minute. Maintain the portion of oil remaining in the Erlenmeyer flask at the specified temperature during filtration, swirling the flask prior to each addition to the crucible until the entire test portion has been filtered. Remove any salts that may adhere to the flask by rinsing with a portion of the filtrate maintained at the specified temperature.

5.015 Maintain full suction on the crucible for 5 minutes after filtration has been completed. Remove the crucible and weigh to the nearest 0.1 gm.

5.02 Determination of percent dry salts.

5.021 Remove a 4 to 5 gram portion of wet salts from the filter cake obtained in Section 5.015, weigh to the nearest 0.1 gm, and transfer to a large sheet of absorbent filter paper. Fold into a square approximately 4 by 4 inches, place in a press, and press out oil. Renew the filter paper, and repeat the pressing operation until the salts are substantially free of oil. This is indicated when only a trace of oil appears as a stain on fresh filter paper. Weigh the dry salts recovered to the nearest 0.1 gm.

6.0 Calculations and Report

6.01 Calculations.

6.011 Calculate liquidity as follows:

$$\% \text{ Liquidity} = \frac{\text{Weight of Sample} - \text{Weight of Wet Salts}}{\text{Weight of Sample}} \times 100$$

6.012 Calculate dry salts content as follows:

$$\% \text{ Dry Salts} = \frac{a\ (100 - C)}{b}$$

Where a = Weight of dry salts recovered.
b = Weight of portion of wet salts used in the test.
c = Percent Liquidity of the sample.

6.02 Report the test results as follows:

Liquidity, 3 hours @ °C = %
Dry Salts, 3 hours @ °C = %

7.0 Reproducibility

7.01 The determination should be reproducible to 0.1 + 0.05 × (percent wet salts) expressed in terms of the percent liquidity.

8.0 Precautions

8.01 Make sure that the temperature of the oil does not drop below the specified test temperature during the preliminary cooling in the Dewar flask, and that the oil and water bath do not vary by more than ± 0.2°C from the specified temperature during the test period.

8.02 Make sure that the filtration is as rapid as possible once it is started, and the unfiltered portion of the oil in the Erlenmeyer is maintained at the specified temperature until filtration is complete.

9.0 Notes

9.01 If the liquidity is known or estimated to be less than 80 percent, use a 50 gram portion in a 125 ml. Erlenmeyer flask; if the liquidity is less than 70 percent, use a 25 gram portion in a 50 ml. Erlenmeyer flask.