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BUREAU OF STANDARDS

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RECOMMENDED SPECIFICATIONS FOR VOLATILE
MINERAL SPIRITS FOR THINNING PAINTS

PREPARED AND RECOMMENDED BY THE U. S. INTERDEPARTMENTAL COMMITTEE ON PAINT SPECIFICATION STANDARDIZATION, JULY 26, 1920; P. H. WALKER, BUREAU OF STANDARDS, CHAIRMAN; J. W. GINDER, TREASURY DEPARTMENT, SECRETARY.

[This committee was appointed at the suggestion of the Secretary of Commerce, and consisted of representatives of the War, Navy, Agriculture, Interior, Post Office, Treasury, and Commerce Departments, The Panama Canal, and the Educational Bureau of the Paint Manufacturers' Association of the United States. The committee submitted a preliminary draft of the specifications to a large number of representatives of the petroleum industries and the paint and varnish industries, and gave careful consideration to the replies which were received in time.]

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1. GENERAL

These specifications apply only to petroleum distillates, known as mineral spirits.

The oils delivered under these specifications shall be genuine and shall conform to the following requirements:

APPEARANCE.—Shall be clear and free from suspended matter and water;

COLOR.—Shall be “water white”;

SPOT TEST.—Shall evaporate completely from filter paper;

FLASH POINT.—Shall be not lower than 30° C (86° F) when tested in a closed cup tester;

SULPHUR.—Shall be absent, as determined by the white-lead test;

Distillate below 130° C (266° F) shall not exceed 5 per cent;

Distillate below 230° C (446° F) shall be not less than 97 per cent;

REACTION.—Shall be neutral.

2. DETECTION AND REMOVAL OF SEPARATED WATER

Draw a portion by means of a glass or metal container with a removable stopper or top, or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

3. SAMPLING.

The method of sampling given under (a) should be used whenever feasible. When method (a) is not applicable, method (b), (c), or (d) is to be used, according to the special conditions that obtain.

(a) WHILE LOADING TANK CAR OR WHILE FILLING CONTAINERS FOR SHIPMENT.—Samples shall be drawn by the purchaser's inspector at the discharge pipe where it enters the receiving vessel or vessels. The composite sample shall be not less than 5 gallons and shall consist of small portions of not more than 1 quart each taken at regular intervals during the entire period of loading or filling.

The composite sample thus obtained shall be thoroughly mixed, and from it three samples of not less than 1 quart each shall be placed in clean, dry glass bottles or tin cans, which must be nearly filled with the sample and securely stoppered with new clean corks or well-fitting covers or caps. These shall be sealed and distinctly labeled by the inspector; one shall be delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(b) FROM LOADED TANK CAR OR OTHER LARGE VESSEL.—The composite sample taken shall be not less than 5 gallons and shall consist of numerous small samples of not more than 1 quart each taken from the top, bottom, and intermediate points by means of a metal or glass container with removable stopper or top. This device attached to a suitable pole is lowered to the various desired depths, when the stopper or top is removed and the

container allowed to fill. The sample thus obtained is handled as in (a).

(c) **BARRELS AND DRUMS.**—Barrels and drums shall be sampled after gaging contents. Five per cent of the packages in any shipment or delivery shall be represented in the sample. Thoroughly mix the contents of each barrel to be sampled by stirring with a clean rod and withdraw a portion from about the center by means of a “thief” or other sampling device. The composite sample thus obtained shall be not less than 3 quarts, shall consist of equal portions of not less than one-half-pint from each package sampled, and shall be handled as in (a). Should the inspector suspect adulteration, he shall draw the samples from the suspected packages.

(d) **SMALL CONTAINERS, CANS, ETC., OF 10 GALLONS OR LESS.**—These should be sampled, while filling, by method (a) whenever possible; but in case this is impossible, the composite sample taken shall be not less than 3 quarts. This shall be drawn from at least five packages (from all when fewer), and in no case from less than 2 per cent of the packages. The composite sample thus taken shall be thoroughly mixed and subdivided as in (a).

4. LABORATORY EXAMINATION

Samples will, in general, be tested by the following methods; but the purchaser reserves the right to apply any additional tests, or use any available information to ascertain whether the material meets the specifications.

(a) **APPEARANCE.**—Examine to determine compliance with the specifications.

(b) **COLOR.**—Fill a 200 mm perfectly flat-bottomed colorimeter tube, graduated in millimeters, to a depth of from 40 to 50 mm with the oil to be examined. Place the tube in a colorimeter and place on or under it a No. 2 yellow Lovibond glass. Over or under a second graduated tube in the colorimeter place a No. 1 yellow Lovibond glass and run in the same oil until the color matches as nearly as possible the color in the first tube. Read the difference in depth of the oil in the two tubes. If this difference is 150 mm or more, the oil is “water white.”

(c) **SPOT TEST.**—Place five drops of the oil on clean white filter paper and allow the liquid to evaporate at room temperature, away from direct sunlight. There should be no oily spot left after 30 minutes.

(d) FLASH POINT.—Determine with either the "Tag" or Elliott closed-cup tester. The former is preferred, and directions for its use are to be found in A. S. T. M., standards D-56-19. For methods of determining with the Elliott cup, see Proceedings A. S. T. M., paragraph I, page 414, 1917.

(e) SULPHUR.—Place 25 g of dry white lead in a small porcelain dish and mix thoroughly with 50 cc of mineral spirits. Cover with a watch glass, place on a steam bath for 2 hours, remove, and observe the color after 18 hours. There should be no appreciable darkening of the white lead. This test must be performed in an atmosphere free from hydrogen sulphide.

(f) DISTILLATION.—*Apparatus.*—See Fig. 1.

Condenser.—The type of apparatus adopted by the American Society for Testing Materials for the distillation of paint thinners other than turpentine is preferred. In case this apparatus is not available, use an ordinary straight glass tube condenser, about 22 inches long, with 16 inches in contact with the cooling water. The end of the condenser tube should be fitted with an adapter, or should be bent down to a nearly vertical position, and the tip should be cut off or ground down at an acute angle. The tip should extend a short distance into the receiving cylinder.

Flask.—Comparable results can be obtained only by using flasks of the same dimensions. The distilling flask used shall be the standard Engler flask, as used for petroleum distillation, having the following dimensions: Diameter of bulb, 6.5 cm; cylindrical neck, 15 cm long, 1.6 cm internal diameter; side or vapor tube, 10 cm long, 0.6 cm external diameter, attached to neck at an angle of 75° , so that when the flask contains its charge of 100 cc of oil, the surface of the liquid shall be 9 cm below the bottom of the junction of the side tube and neck.

Support of Flask.—Support the flask on a plate of asbestos 20 cm in diameter, having an opening 4 cm in diameter in its center, and heat with an open flame. Surround the flask and burner with a shield to prevent fluctuation in the temperature of the neck of the flask.

Thermometer.—A thermometer such as the one adopted by the American Society for Testing Materials for distillation of mineral thinners should be used.

Receiving Cylinder.—Collect the distillates in accurately graduated 50 or 100 cc cylinders. The so-called normal or precision cylinder of 50 cc capacity, having an internal diameter of 1.5 cm and graduated in 0.2 cc, is preferred. If a cylinder with larger

inside diameter is used, a pasteboard cover should be placed over the top and surround the condenser tube.

Operation.—Place 100 cc of the spirits and several small pieces of pumice (or glass) in the distilling flask and fit the thermometer so that the top of the mercury bulb is level with the bottom of the side tube. Place the flask in position on the asbestos board and connect with the condenser. Apply the heat cautiously at first, and when distillation begins regulate the heat so that the spirits distil at the rate of not less than 4 nor more than 5 cc per

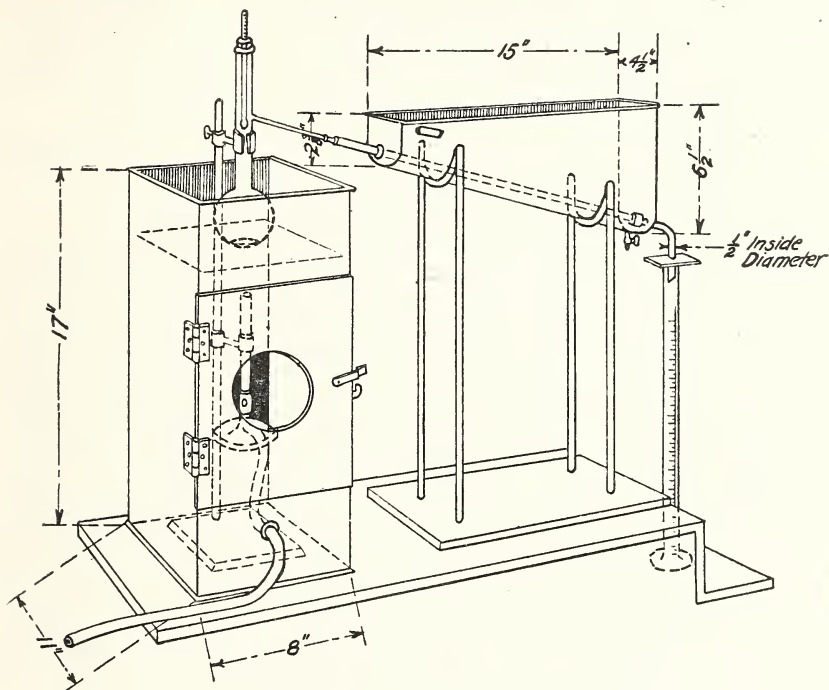


FIG. 1.—Distillation apparatus

minute (approximately two drops per second). The initial boiling point is the thermometer reading at the instant when the first drop falls from the end of the condenser. Discontinue distillation when the temperature reaches 230°C (446°F). Let the condenser drain and read the percentage distilled.

The percentage distilled below successive selected temperatures and the temperature at which each successive 10 cc distils may also be determined if desired.

(g) REACTION.—Shake 50 cc of the mineral spirits and 50 cc of water in a half-filled stoppered cylinder for one minute, allow to

settle, draw off 25 cc of the water with a pipette, add several drops of phenolphthalein solution and one drop of $\frac{N}{10}$ alkali, which should give the water an alkaline reaction.

5. BASIS OF PURCHASE

(a) UNIT.—Mineral spirits shall be purchased either (1) by volume, the unit being a gallon of 231 cubic inches at 15.5° C (60° F), or (2) by weight. A gallon of mineral spirits at 15.5° C (60° F) weighs 6.3 to 6.8 pounds. The exact weight in pounds per gallon of any sample can be determined by multiplying the specific gravity at 15.5/15.5° C (60/60° F) by 8.33. Example—If the specific gravity at 15.5° C is 0.7642, the weight per gallon at this temperature will be $0.7642 \times 8.33 = 6.366$ pounds.

When purchased by weight, quotations shall be by the pound or by the 100 pounds. The request for bids will state whether quotations shall be by the gallon, pound, or 100 pounds.

(b) CORRECTION OF VOLUME.—The gallonage paid for shall be the volume corrected to a standard temperature of 15.5° C (60° F). The correction shall be made by deducting from (when the temperature of gaging is above 15.5° C) or adding to (when the temperature of gaging is below 15.5° C) the gallonage as gaged. Such deduction or addition shall be computed on the basis of a coefficient of expansion of 0.000945 per degree centigrade (or 0.000525 per degree Fahrenheit). Example—If the temperature at which the spirits is gaged is 75° F, and the volume delivered (at that temperature) is 8000 gallons, then $0.000525 \times 15 \times 8000$ equals the quantity in gallons which must be subtracted from 8000 gallons to give the true gallonage at 60° F, or, if the temperature at which the spirits is gaged is 10° C, then $0.000945 \times 5.5 \times 8000$ equals the quantity in gallons which must be added to the gaged volume of 8000 gallons to give the true gallonage at 15.5° C.

(c) CERTIFICATION.—Mineral spirits delivered in barrels, drums, or tank cars shall either be accompanied by an official gager's certificate showing the net contents of each container and also the temperature of contents at the time of gaging, or shall be subject to gaging by the purchaser's inspector. In the absence of a statement of the temperature at the time of gaging on the official gager's certificate, or in case the barrels show evidence of loss by leakage or other shortage, the delivery shall be subject to reinspection and regaging by the purchaser's inspector.

WASHINGTON, July 26, 1920.