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UNITED STATES GOVERNMENT SPECIFICATION FOR TURPENTINE

(Gum Spirits and Wood Turpentine)

FEDERAL SPECIFICATIONS BOARD

STANDARD SPECIFICATION NO. 7

This specification was officially adopted by the Federal Specifications Board, on February 3, 1922, for the use of the Departments and Independent Establishments of the Government in the purchase of materials covered by it.

CONTENTS

1. GENERAL

These specifications apply both to the turpentine which is distilled from pine oleoresins, commonly known as gum spirits or spirits of turpentine, and to turpentine commonly known as wood turpentine, which is obtained from resinous wood, whether by steam or by destructive distillation. When ordering under these specifications, the purchaser shall specify whether (*a*) gum spirits or (*b*) wood turpentine is desired. When wood turpentine is specified, it may be stated whether steam or destructively distilled wood turpentine shall be furnished.

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Turpentine shall be pure and conform to the following requirements:

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

COLOR.—Shall be "standard" or better.

ODOR.—Shall be characteristic of the variety of turpentine specified and, if desired, shall conform to the odor of the sample agreed upon.

	Maximum	Minimum
Specific gravity, 15.5/15.5° C	0.875	0.862
Refractive index at 20° C, gum spirits	1.478	1.468
Refractive index at 20° C, wood turpentine	1.478	1.465
Residue after polymerization with 38 N H_2SO_4:		
Gum spirits—		
Volume (per cent)	2.0	....
Refractive index at 20° C	....	1.500
Wood turpentine—		
Volume (per cent)	2.5	....
Refractive index at 20° C	....	1.480
Initial boiling point at 760 mm pressure	160° C	150° C
Distilling below 170° C at 760 mm pressure (per cent)	....	90

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 turpentine 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 turpentine until the color matches as nearly as possible the color in the first tube. Read the difference in depth of the turpentine in the two tubes. If this difference is 50 mm or more, the turpentine is "standard" or better.

(*c*) **Odor.**—Determine by comparison with several samples of known purity, which have been kept in the dark in completely filled, well-stoppered bottles and are free from separated water.

(*d*) **Specific Gravity.**—Determine at 15.5/15.5° C, in a pycnometer accurately standardized and having a capacity of at least 25 cc, or by any other equally accurate method.

(*e*) **Refractive Index.**—Determine refractive index at 20° C with an accurate instrument. When the refractive index is determined at any other temperature, the readings obtained shall be corrected to 20° C by adding to or by subtracting from the actual reading 0.00045 for each degree centigrade that the temperature at which the determination was made is, respectively, above or below 20° C.

(*f*) **Distillation.**—APPARATUS.[1]—*Condenser.*—The type of apparatus (see Fig. 1) adopted by the American Society for Testing Materials for the distillation of paint thinners other than turpentine, substituting for the thermometer there described[2] an immersed thermometer such as is described below, is preferred. In case the A. S. T. M. distillation 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

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[1] Fig. 1.

[2] A. S. T. M. Standards, p. 607; 1918.

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 for 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. Or, support the flask in a metal cup, 15 to 20 cm in diameter, containing high-boiling mineral oil or glycerin and fitted with a concave cover having in the center a circular opening 5½ to 6 cm in diameter. In all cases take the necessary precautions to prevent fluctuation in temperature in the neck of the flask.

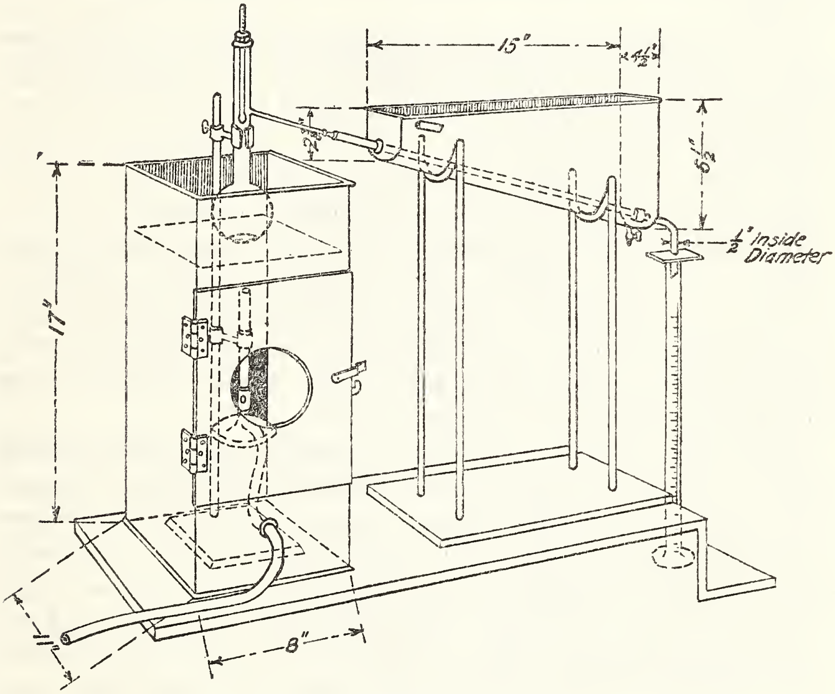

Fig. 1.—*Distillation apparatus*

Thermometer.—The thermometer used for turpentine distillation shall conform to the following specifications:

It shall be graduated from 145° to at least 200° C in 0.2° intervals. Thermometers graduated above 200° C may be used, provided they also comply with the following requirements: Length, bottom of thermometer to 175° mark, not more than 8 nor less than

6.5 cm. Length, top of bulb to 145° mark, not less than 1.5 cm. Length, 145 to 175° mark, not more than 6 cm.

The thermometer shall be made of suitable thermometric glass and thoroughly annealed, so that the scale errors will not increase after continued heating.

The thermometer shall be filled above the mercury with an inert gas, with sufficient pressure above the mercury column to prevent breaking of the column. It shall have a reservoir at the top, so that the pressure will not become excessive at the highest temperature.

Every fifth graduation shall be longer than the intermediate ones, and the marks shall be numbered at each interval of 5°. The graduation marks shall be clear-cut and fine and the numbering clear-cut and distinct.

The error at any point on the scale shall not exceed ±0.5° C when tested for total immersion of the mercury column.

Receiving Cylinder.—Collect the distillate in an accurately graduated 50 or 100 cc cylinder. 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 turpentine and several small pieces of pumice (or glass) in the distilling flask, fit the thermometer so that the top of the mercury bulb is level with the bottom of the side tube, and the 175° C (347° F) mark is below the cork. Place the flask in position on the asbestos board or oil bath and connect with the condenser. Apply the heat cautiously at first, and, when distillation begins, regulate the heat so that the turpentine distills at the rate of not less than 4 nor more than 5 cc per 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 170.0° C (338° F), or an equivalent thereof, depending on the atmospheric pressure, as outlined below; 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. distills may also be determined, if desired, making the necessary correction of the temperature for variations in atmospheric pressure.

CORRECTION FOR VARIATION IN ATMOSPHERIC PRESSURE.—Since distillation results are comparable only when obtained under exactly the same pressure conditions, turpentine shall be distilled at that pressure which, at room temperature, is equivalent to a pressure of 760 mm of mercury at 0° C. Whenever the atmospheric pressure after correcting to 0° C is other than 760 mm, a correction must be made. Since alteration of the pressure in the distilling system requires rather complicated apparatus, it is simpler to alter the temperature observation points to correspond to the prevailing pressure.

To determine what the atmospheric pressure at the prevailing room temperature, or at the temperature of the barometer, would be at 0° C, read the barometer and thermometer alongside when about to begin distillation. Refer to Table 1, page 11. Under the column nearest the observed pressure reading, and on the line nearest the observed temperature of the barometer will be found the correction which must be subtracted from the observed pressure reading to obtain the equivalent, or true, reading at 0° C.

The distilling temperature of turpentine is affected plus (+) or minus (−) 0.057° C for each millimeter variation of the barometer above or below the normal 760 mm at 0° C.[3] If the barometer reading, after correcting to 0° C, is below 760 mm, the turpentine will distill at a slightly lower temperature than under normal pressure. Therefore, the temperature recorded at the beginning of distillation (and any others observed during the course of the distillation) must be corrected to get its equivalent at normal pressure. The final temperature observation point (170° C of the specifications) must be altered accordingly to get its equivalent at the pressure (corrected to 0° C) at which distillation is made.

For example, if the barometer reading, after correcting to 0° C, is 750 mm, the correction of the observed initial distilling temperature will be $0.057 \times 10 = 0.6°$ C approximately. If the reading of the thermometer when the turpentine begins to distill is 155.6° C, the corrected initial distilling temperature will be $155.6° + 0.6° = 156.2°$ C. Furthermore, the temperature observation point at end of distillation (170.0° C at 760 mm) must be altered to the same extent. Since the turpentine is distilling 0.6° C below what it would at normal pressure, distillation must

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[3] Landolt-Börnstein Physikalisch-Chemische Tabellen, Ed. 4, Table 127, p. 435.

be discontinued at 0.6° C below the specified limit of 170.0° C to determine the percentage distilling below 170.0° C.

If the barometer reading corrected to 0° C is above 760 mm, subtract the temperature correction from the observed thermometer reading to determine the initial distilling point, and continue distillation to 170.0° C plus the correction to determine the percentage distilling below 170.0° C.

(*g*) **Polymerization.**—Place 20 cc of 38 *N* (equivalent to 100.92 per cent H_2SO_4) sulphuric acid in a graduated, narrow-necked Babcock flask, stopper, and place in ice water to cool. Add slowly, from a pipette, 5 cc of the turpentine to be examined. Gradually mix the contents, keeping warm, but being very careful that the temperature does not rise above 60° C. When the mixture no longer warms up on shaking, agitate thoroughly and place the flask in a water bath and heat at 60 to 65° C for not less than 10 minutes, keeping the contents of the flask thoroughly mixed by vigorous shaking for one-half minute each time, six times during the period. Do not stopper the flask after the turpentine has been added, as it may explode. Cool to room temperature, fill the flask with concentrated sulphuric acid until the unpolymerized oil rises into the graduated neck and centrifuge from four to five minutes at not less than 1200 r. p. m., or for 15 minutes at 900 r. p. m., or allow to stand, lightly stoppered, for 12 hours. Calculate the percentage, note the consistency and color, and determine the refractive index (at 20° C) of the unpolymerized residue. The consistency should be viscous and the color straw or darker.

REAGENT FOR TESTING.—In a weighed glass-stoppered bottle (the regular 2½-liter acid bottle is of a convenient size) mix ordinary concentrated sulphuric acid (sp. gr. 1.84) with fuming sulphuric acid. If the fuming acid used contains 50 per cent excess SO_3, the ratio of one part, by weight, of the former to three-fourths of a part, by weight, of the latter will give a mixture slightly stronger than the required strength. To determine the exact strength of this mixture in terms of H_2SO_4, weigh exactly, in a weighing pipette of about 10 cc capacity, approximately 20 g of the acid. Allow it to flow down the sides of the neck into a 1000-cc volumetric flask containing about 200 cc of distilled water. When the pipette has drained, wash all traces of the acid remaining in the pipette into the flask, taking precautions to prevent loss of SO_3, and make up to the mark. Titrate

20-cc portions, drawn from a burette, against half normal alkali. Calculate the concentration in terms of the percentage of H_2SO_4 in the sample taken.

In the same way determine the percentage of H_2SO_4 in the stock of ordinary concentrated acid (sp. gr. 1.84). From these data calculate the quantity of the latter which must be added to the quantity of mixed acid in the weighed bottle to bring it to a concentration, in terms of H_2SO_4, of 100.92 per cent.

After adjusting the concentration by the addition of the ordinary sulphuric acid, thoroughly shake the bottle of mixed acid and again determine its concentration. The allowable variation is ± 0.05 per cent H_2SO_4. Finally as a check run a polymerization test on gum turpentine known to be pure. The residue should fall below 2 per cent.

Special precautions must be taken to prevent dilution of this acid by the absorption of atmospheric moisture. The arrangement shown in Fig. 2 is most suitable for storing and delivering measured quantities of this reagent.

With the three-way stopcocks *A* and *B* in the position shown, acid is siphoned into the pipette *P*, the displaced air passing into *R*. To empty the pipette, *A* and *B* are turned to the position shown by the broken lines, air passing in at *a*. The acid adhering to the walls of the pipette dries this air so that when it passes into *R* on again filling the pipette there is no accumulation of moisture in the acid remaining in the reservoir. If such arrangement is not to be had, the acid should be kept in well-fitting glass-stoppered bottles of not more than one-half liter capacity.

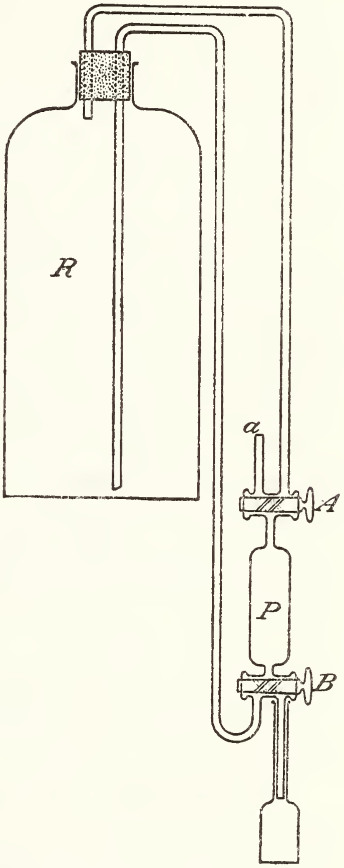

Fig. 2.—*Acid bottle and pipette*

5. BASIS OF PURCHASE

(*a*) **Unit.**—Turpentine shall be purchased (*a*) by volume, the unit being a gallon of 231 cubic inches at 15.5° C (60° F), or (*b*) by weight. A gallon of turpentine at 15.5° C (60° F) weighs 7.19 to 7.30 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 8.34. Example: If the specific gravity at 15.5° C is 0.8642, the weight per gallon at this temperature will be $0.8642 \times 8.34 = 7.207$ 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 deducted from (when the temperature of gaging is above 15.5° C) or added 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 for turpentine of 0.000945 per degree centigrade (or 0.000525 per degree Fahrenheit). Example: If the temperature at which the turpentine is gaged is 75° F and the volume delivered (at that temperature) is 8000 gallons, then $0.000525 \times 15^\circ \times 8000$ gallons 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 turpentine is gaged is 10° C, then $0.000945 \times 5.5^\circ \times 8000$ gallons 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.**—Turpentine 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 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.

TABLE 1.—Correction to Barometer Readings [a]

[From circular F, instrument division, Weather Bureau, U. S. Department of Agriculture]

Temperature, °C	Observed reading of barometer, in millimeters														
	640	650	660	670	680	690	700	710	720	730	740	750	760	770	780
15.0	1.56	1.59	1.61	1.64	1.66	1.69	1.71	1.74	1.76	1.78	1.81	1.83	1.86	1.88	1.91
16.0	1.67	1.69	1.72	1.75	1.77	1.80	1.83	1.85	1.88	1.90	1.93	1.96	1.98	2.01	2.03
17.0	1.77	1.80	1.83	1.86	1.88	1.91	1.94	1.97	1.99	2.02	2.05	2.08	2.10	2.13	2.16
18.0	1.88	1.91	1.93	1.96	1.99	2.02	2.05	2.08	2.11	2.14	2.17	2.20	2.23	2.26	2.29
19.0	1.98	2.01	2.04	2.07	2.10	2.13	2.17	2.20	2.23	2.26	2.29	2.32	2.35	2.38	2.41
20.0	2.08	2.12	2.15	2.18	2.21	2.25	2.28	2.31	2.34	2.38	2.41	2.44	2.47	2.51	2.54
21.0	2.19	2.22	2.26	2.29	2.32	2.36	2.39	2.43	2.46	2.50	2.53	2.56	2.60	2.63	2.67
22.0	2.29	2.33	2.36	2.40	2.43	2.47	2.51	2.54	2.58	2.61	2.65	2.69	2.72	2.76	2.79
23.0	2.40	2.43	2.47	2.51	2.54	2.58	2.62	2.66	2.69	2.73	2.77	2.81	2.84	2.88	2.92
24.0	2.50	2.54	2.58	2.62	2.66	2.69	2.73	2.77	2.81	2.85	2.89	2.93	2.97	3.01	3.05
25.0	2.60	2.64	2.68	2.72	2.77	2.81	2.85	2.89	2.93	2.97	3.01	3.05	3.09	3.13	3.17
26.0	2.71	2.75	2.79	2.83	2.88	2.92	2.96	3.00	3.04	3.09	3.13	3.17	3.21	3.26	3.30
27.0	2.81	2.85	2.90	2.94	2.99	3.03	3.07	3.12	3.16	3.20	3.25	3.29	3.34	3.38	3.42
28.0	2.91	2.96	3.00	3.05	3.10	3.14	3.19	3.23	3.28	3.32	3.37	3.41	3.46	3.51	3.55
29.0	3.02	3.06	3.11	3.16	3.21	3.25	3.30	3.35	3.39	3.44	3.49	3.54	3.58	3.63	3.68
30.0	3.12	3.17	3.22	3.27	3.32	3.36	3.41	3.46	3.51	3.56	3.61	3.66	3.71	3.75	3.80
31.0	3.22	3.27	3.32	3.37	3.43	3.48	3.53	3.58	3.63	3.68	3.73	3.78	3.83	3.88	3.93
32.0	3.33	3.38	3.43	3.48	3.54	3.59	3.64	3.69	3.74	3.79	3.85	3.90	3.95	4.00	4.05
33.0	3.43	3.48	3.54	3.59	3.64	3.70	3.75	3.81	3.86	3.91	3.97	4.02	4.07	4.13	4.18

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[a] These corrections apply to a mercurial barometer with brass scale. They can, however, be used for a mercurial barometer with glass scale, since the errors introduced thereby are negligible as applied to the work contemplated in this Circular. For exact correction to be applied to such a barometer see Smithsonian Physical Tables, p. 119; 1914. An aneroid barometer should not be relied on.

For barometer readings below 640 mm the correction can be interpolated, since the difference, at any particular temperature, for each 10 mm variation in barometer reading is practically constant.

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