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## DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

George K. Burgess, Director

## CIRCULAR OF THE BUREAU OF STANDARDS, No. 86

[3d. ed. issued October 11, 1926]

**UNITED STATES GOVERNMENT MASTER SPECIFICATION  
FOR TURPENTINE (GUM SPIRITS OF TURPENTINE AND  
STEAM-DISTILLED WOOD TURPENTINE)**

## FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 7b

[Revised August 7, 1926]

This specification was officially promulgated 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 turpentine (gum spirits of turpentine and steam-distilled wood turpentine).

[The latest date on which the technical requirements of this revision shall become mandatory for all departments and independent establishments of the Government is November 7, 1926. They may be put into effect, however, at any earlier date, after promulgation.]

## CONTENTS

	Page
I. General specifications.....	1
II. Types.....	1
III. Material.....	2
IV. General requirements.....	2
V. Detail requirements.....	2
VI. Methods of inspection, tests, and basis of purchase.....	2
1. Detection and removal of separated water.....	2
2. Sampling.....	2
3. Laboratory examination.....	3
4. Basis of purchase.....	9
VII. Packing and marking.....	10
VIII. Notes.....	10

## I. GENERAL SPECIFICATIONS.

There are no general specifications applicable to this specification.

## II. TYPES

This specification applies to two types of turpentine or spirits of turpentine as follows:

A. Gum spirits of turpentine, commonly known as gum spirits. This is the product distilled from the oleoresin exuding from living pine trees.

B. Steam-distilled wood turpentine, which is distilled with steam from the oleoresin within the wood.

### III. MATERIAL

No details specified.

### IV. GENERAL REQUIREMENTS

There are no general requirements applicable to this specification.

### V. DETAIL REQUIREMENTS

Gum spirits of turpentine or steam-distilled wood turpentine, as specified in contract, shall be pure and shall 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 mild, aromatic, and characteristic of the type of turpentine specified. If desired, shall conform to the odor of the sample agreed upon.

	Maximum	Minimum
Specific gravity, 15.5/15.5° C. ....	0.875	0.860
Refractive index at 20° C. ....	1.478	1.465
Residue after polymerization with 38 N H <sub>2</sub> SO <sub>4</sub> .....		
Volume (per cent).....	2.0	1.500
Refractive index at 20° C. ....		
(This residue shall be viscous and its color straw or darker.)		
Initial boiling point at 760 mm. pressure.....	160° C.	150° C.
Distilling below 170° C. at 760 mm. pressure (per cent).....		90

### VI. METHODS OF INSPECTION, TESTS, AND BASIS OF PURCHASE

*Deliveries will, in general, be inspected and tested by the following methods, but the purchaser reserves the right to use any additional available information to ascertain whether the material meets the specification.*

#### 1. 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.

#### 2. 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 gauging 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).

### 3. LABORATORY EXAMINATION

(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 the agreed-upon sample, which shall have been kept in the dark in completely filled, well-stoppered bottles and free from separated water.

(d) SPECIFIC GRAVITY.—Determine at 15.5/15.5° C. by any convenient method that is accurate within 2 points in the fourth decimal place.

(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.*<sup>1</sup>—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<sup>2</sup> 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 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,

<sup>1</sup> Figure 1.

<sup>2</sup> A. S. T. M. Standards, p. 607; 1913.



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  $5\frac{1}{2}$  to 6 cm in diameter. In all cases take the necessary precautions to prevent fluctuation in temperature in the neck of the flask.

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

It shall be graduated from  $145^{\circ}$  to at least  $200^{\circ}$  C. in  $0.2^{\circ}$  intervals. Thermometers graduated above  $200^{\circ}$  C. may be used, pro-

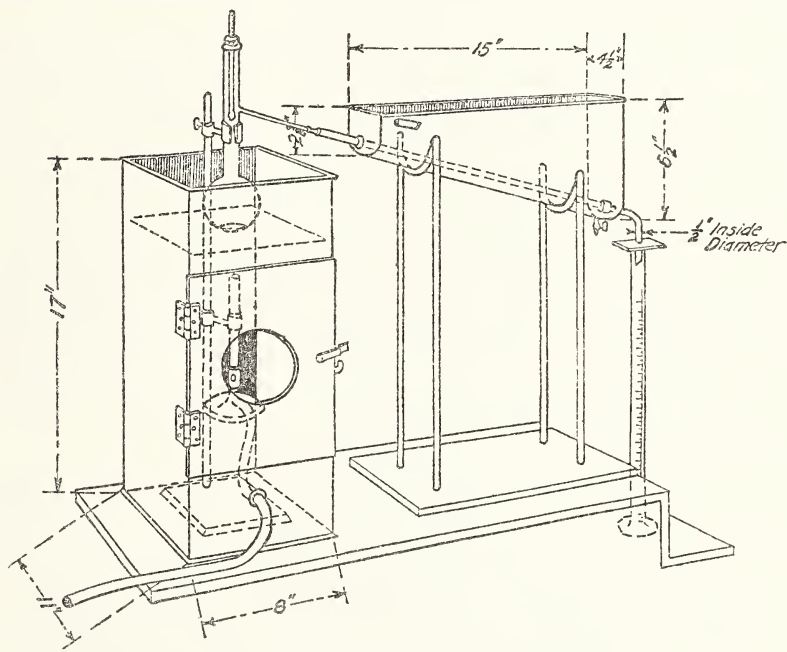


FIG. 1.—Distillation apparatus

vided they also comply with the following requirements: Length, bottom of thermometer to  $175^{\circ}$  mark, not more than 8 nor less than 6.5 cm. Length, top of bulb to  $145^{\circ}$  mark, not less than 1.5 cm. Length,  $145$  to  $175^{\circ}$  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  $\pm 0.5^\circ \text{C}$ . 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 with a hole for the condenser tube should be placed over the top.

*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  $175^\circ \text{C}$ . ( $347^\circ \text{F}$ .) 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  $170.0^\circ \text{C}$ . ( $338^\circ \text{F}$ .), 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 should be distilled at that pressure which, at room temperature, is equivalent to a pressure of 760 mm of mercury at  $0^\circ \text{C}$ . Whenever the barometric reading after correcting to  $0^\circ \text{C}$ . 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 barometric reading at the prevailing room temperature, or at the temperature of the barometer, would be at  $0^\circ \text{C}$ ., read the barometer and thermometer alongside when about to begin distillation. Refer to Table 1, page 7. 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.<sup>3</sup> 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  $0.057 \times 10 = 0.6^\circ \text{C.}$ , approximately. If the reading of the thermometer when the turpentine begins to distill is 155.6° C. the corrected initial distilling temperature will be  $155.6^\circ + 0.6^\circ = 156.2^\circ \text{C.}$  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 be discontinued at 0.6° C. below the specified limit of 170.0° C. to determine the percentage distilling below 170.0° C.

TABLE 1.—*Correction to barometer reading*<sup>1</sup>

[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

<sup>1</sup> 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.

<sup>3</sup> Landoit-Börnstein Physikalisch-Chemische Tabellen, Ed. 4, Table 127, p. 435.



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  $\text{H}_2\text{SO}_4$ ) 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 4 to 5 minutes at not less than 1,200 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.

*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  $\text{SO}_3$ , 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  $\text{H}_2\text{SO}_4$ , 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 1,000 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  $\text{SO}_3$ , 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  $\text{H}_2\text{SO}_4$  in the sample taken.

In the same way determine the percentage of  $\text{H}_2\text{SO}_4$  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  $\text{H}_2\text{SO}_4$ , 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  $\pm 0.05$  per cent  $\text{H}_2\text{SO}_4$ . 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 Figure 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.

#### 4. BASIS OF PURCHASE

(a) UNIT.—Turpentine shall be purchased (a) by volume, the unit being a gallon of 231 cubic inches at  $15.5^\circ \text{C}$ . ( $60^\circ \text{F}$ .), or (b) by weight. A gallon of turpentine at  $15.5^\circ \text{C}$ . ( $60^\circ \text{F}$ .) 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  $15.5/15.5^\circ \text{C}$ . ( $60/60^\circ \text{F}$ .) by 8.33. Example: If the specific gravity at  $15.5^\circ \text{C}$ . is 0.8642, the weight per gallon at this temperature will be  $0.8642 \times 8.33 = 7.199$  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  $15.5^\circ \text{C}$ . ( $60^\circ \text{F}$ .).

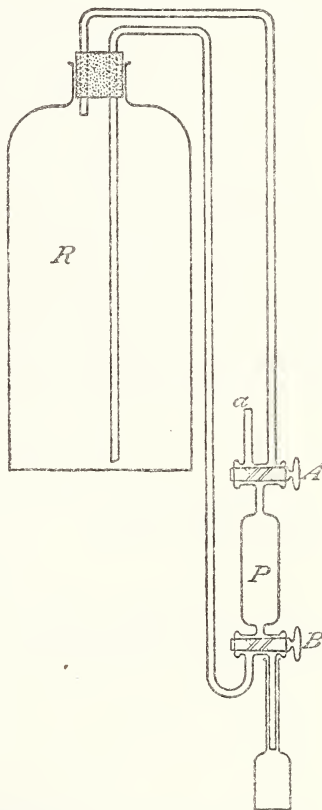


FIG. 2.—Acid bottle and pipette

The correction shall be deducted from (when the temperature of gauging is above 15.5° C.) or added to (when the temperature of gauging is below 15.5° C.) the gallonage as gauged. 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 gauged is 75° F. and the volume delivered (at that temperature) is 8,000 gallons, then  $0.000525 \times 15^\circ \times 8,000$  gallons equals the quantity in gallons which must be subtracted from 8,000 gallons to give the true gallonage at 60° F., or if the temperature at which the turpentine is gauged is 10° C., then  $0.000945 \times 5.5^\circ \times 8,000$  gallons equals the quantity in gallons which must be added to the gauged volume of 8,000 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 gauger's certificate showing the net contents of each container and also the temperature of contents at time of gauging, or shall be subject to gauging by the purchaser's inspector. In the absence of a statement of the temperature at the time of gauging on the official gauger's certificate, or in case the barrels show evidence of loss by leakage or other shortage, the delivery shall be subject to reinspection and regauging by the purchaser's inspector.

## VII. PACKING AND MARKING

Packing shall be in accordance with commercial practice, unless otherwise specified.

## VIII. NOTES

This specification does not cover what is known as destructively distilled wood turpentine.

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