

TT-R-271C  
 March 12, 1975  
 SUPERSEDING  
 Fed. Spec. TT-R-271B  
 April 27, 1967

FEDERAL SPECIFICATION

RESINS, PHENOL-FORMALDEHYDE  
 (PARA-PHENYL AND PARA-BUTYL)

This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers synthetic resins for use in the manufacture of paints, varnishes, and etc.

1.2 Classification. The resins shall be of the following types:

- Type I - Phenol-formaldehyde, para-phenyl.
- Type II - Phenol-formaldehyde, modified para-tertiary butyl phenol.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

Federal Specifications:

- TT-T-775 - Tung Oil, Raw (China Wood) (For Use In Organic Coatings).
- TT-X-916 - Xylene (For Use In Organic Coatings).
- VV-G-109 - Gasoline, Unleaded.
- PPP-B-35 - Bags, Textile, Shopping, Burlap, Cotton and Waterproof Laminated.
- PPP-D-723 - Drums, Fiber.

Federal Standards:

- Fed. Std. No. 123 - Marking for Domestic Shipment (Civil Agencies).
- Fed. Test Method Std. No. 141 - Paint, Varnish, Lacquer and Related Materials; Methods of Inspection, Sampling, and Testing.

(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(Single copies of this specification and other Federal Specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Washington, DC, Atlanta, Chicago, Kansas City, MO, Fort Worth, Denver, San Francisco, Los Angeles, and Seattle, WA.

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

Military Standards:

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-129 - Marking for Shipment and Storage.

(Copies of Military Specifications and Standards required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

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### 3. REQUIREMENTS

3.1 Material. The material shall be as specified herein and of good commercial quality entirely suitable for the purpose intended. The resins, type I and type II, shall be free from foreign matter or adulterant. A certificate of compliance to this effect is necessary.

#### 3.2 Qualitative requirements.

3.2.1 Condition in container. The resin as received shall show no evidence of deterioration, putrefaction or any other defects when examined as in 4.3.1.

3.2.2 Storage stability. When stored for 2 years in a closed container at temperature range of 32° to 100°F. as described in 4.3.2. The materials shall be useable and shall show no evidence of deterioration or other defects.

#### 3.2.3 Appearance.

3.2.3.1 Type I. The resin shall be in solid, lump form; the lumps shall be uniform and homogeneous in appearance when tested as in 4.3.3.

3.2.3.2 Type II. The resin shall be in solid, granulated form, the granules shall be uniform in appearance when tested as in 4.3.3. <sup>1/</sup>

3.2.4 Color. When tested as in 4.3.4, the color of the resin solution shall not be darker than the potassium dichromate solution.

3.2.5 Rosin and rosin derivatives. Rosin and rosin derivatives shall not be present when tested as in 4.3.5.

#### 3.2.6 Identification.

3.2.6.1 Type I. The resin, when tested as in 4.3.6.1, shall indicate the presence of para-phenyl phenolic resin.

3.2.6.2 Type II, the Resin, when tested as in 4.3.6.2, shall indicate the presence of para-butyl phenolic resin.

3.2.7 Water resistance. When tested as in 4.3.7 the resins shall show no discoloration or otherwise visible change in 72 hours.

3.2.8 Effect on tung oil. When tested as specified in 4.3.10 the resin shall show an accelerating effect on the increase in viscosity of tung oil at 260°C. (500°F.); that is, a 25-gallon oil length of the resin-oil combination shall show a faster bodying rate and time of gelation, respectively, than the tung oil heated alone at the same temperature.

### 3.3 Quantitative requirements (type I and type II).

3.3.1 Quantitative requirements shall be as specified in table I.

TABLE I. Quantitative requirements

Characteristics	Type I		Type II	
	Minimum	Maximum	Minimum	Maximum
Specific Gravity	1.19	1.24	1.07	1.09
Volatile Content, percent by weight	----	6	----	6
Softening Point, degree F.	195	225	290	315
Ash, percent by weight	----	0.2	----	0.2

### 4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other

<sup>1/</sup> Industry may furnish the material in solution. The solution shall be clear and homogeneous, free from lumps, gells, skins and shall not thicken.

facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Sampling and inspection. Sampling and inspection shall be in accordance with method 1031 of Fed. Test Method Std. No. 141.

4.3 Test procedures. The tests shall be conducted in accordance with test methods of Fed. Test Method Std. No. 141, except as otherwise specified in table II.

TABLE II. Index

Characteristics	Requirements reference	Applicable tests	
		Fed. Test Method Std. No. 141	Paragraph reference
Condition in container	3.2.1	----	4.3.1
Storage stability	3.2.2	----	4.3.2
Appearance	3.2.3	----	4.3.3
Color	3.2.4	----	4.3.4
Rosin and rosin derivatives	3.2.5	5031	4.3.5
Identification	3.2.6	----	4.3.6
Water resistance	3.2.7	----	4.3.7
Effect on tung oil	3.2.8	----	4.3.8
Specific gravity	Table I	----	4.3.9
Volatile content	Table I	----	4.3.10
Melting (softening) point	Table I	----	4.3.11
Ash	Table I	----	4.3.12

4.3.1 Condition in container. Open the container as received and examine for compliance with 3.2.1.

4.3.2 Storage stability. Store the resin in a bag conforming to PPP-B-35, type II, style A for 2 years at the temperature range of 32° to 100°F. and evaluate for compliance with 3.2.2 to avoid long period of test it is necessary that the supplier submit a certified statement that the material has been tested and found to comply the requirement.

4.3.3 Appearance. Examine the resin as received and evaluate for compliance with 3.2.3.

4.3.4 Color. Make a solution of the resin in xylene conforming to TT-X-916 (50 percent by weight). Make a solution of 0.0322 gram of reagent grade potassium dichromate in 100 milliliters of concentrated sulfuric acid, then evaluate for compliance with 3.2.4.

4.3.5 Rosin and rosin derivatives. Run the test in accordance with method 5031 of Fed. Test Method Std. No. 141 and observe for compliance with 3.2.5.

4.3.6 Identification. (Qualitative test).

4.3.6.1 Type I. Run the test in accordance with method 5141.1 Procedure B of Fed. Test Method Std. No. 141 for compliance with 3.2.6.1.

4.3.6.2 Type II. The resin shall show similar spectra as shown in figure 4 by using nuclear magnetic resonance (NMR) Spectrometry (see 6.4).

4.3.7 Water resistance. Select a piece of resin weighing approximately 5 grams, rub free of dust and place in approximately 25 milliliters of distilled water in a clean test tube. Stopper the test tube and allow the resin to remain in water for 72 hours at 23°C. + 1.1°C. (73.5° + 2°F.). At the end of the immersion, remove the resin and allow to dry then observe for compliance with 3.2.7.

4.3.8 Effect on tung oil. The apparatus shall conform to figure 1, a modification of the apparatus (commonly used for the Browne test). With the apparatus shown it is possible to quickly attain the desired temperature and to hold this temperature constant within 1°C. (1.8°F.). Any sources of heat, however, which may be readily controlled may be used for heating the bath in place of bunsen burner.

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4.3.8.1 Preparation of sample. Weigh a sample of approximately 34 grams of resin and 66 grams of tung oil conforming to TT-T-775 into a 250-milliliter Erlenmeyer flask. Heat to 180°C. (356°F.) in 15 minutes and stir to insure uniformity, cool to room temperature, and stopper.

4.3.8.2 Procedure. Heat the oil bath to 260°C. (500°F.) and hold constant by means of the burner fitted with the needle valve. Weigh 1.5 grams of tung oil (Fed. Spec. TT-T-775) into the test tube, insert the glass rod and place in the oil bath. Determine the length of time from the insertion of the tube in the oil bath to the point when the tung oil forms a gel strong enough to allow the tube to be lifted out of the bath by the glass rod. Repeat the procedure with the same weight of previously prepared oil-resin mixture. Determine if the resin has accelerated the increase in viscosity of the tung oil, as evidenced by a shorter time for the oil-resin mixture to reach the end point of the test.

4.3.9 Specific gravity. Select a piece of resin weighing about 5 grams which is free from bubbles, cracks, dust, and abrupt depressions. Suspend the resin on a loop of fine wire and weigh it accurately in air and in water at 25°C. (77°F.). Calculate the specific gravity. Make three such determinations on separate pieces of resin, average the results, and report as the specific gravity.

4.3.10 Volatile content at 293°C. (560°F.). Crush a sample of the resin into pieces the size of wheat grains or less. Weigh accurately 10 grams of the resin into a tared 100-milliliter pyrex beaker. Place the beaker on a stand and insert it in a muffle furnace at 293°C. (560°F.) or in an oven at the same temperature. Maintain the sample at this temperature for 15 minutes, then remove, cool, and weigh it accurately. Report the loss in weight as volatile content.

4.3.11 Softening point. Determine softening point in accordance with Method 4495 of Fed. Test Method Std. No. 141.1/

4.3.11.1 Apparatus. (See figure 2).

4.3.11.1.1 Two circular brass plates, 1/16 inch thick and 2-3/8 inches in diameter; one plate has three holes equally spaced about its center, each 5/8 inch in diameter. A shoulder 1/32 inch deep and 9/128 inch wide is cut around each hole so that the molds will fit snugly without danger of sliding off. These plates shall be attached to one end of a threaded steel rod, 1/8 inch by 12 inches, through their centers, by means of 3/16 inch hexagonal nuts, in such a manner that their opposing faces are 7/8 inch apart and parallel.

4.3.11.1.2 Three corrosion-resisting steel molds, 3/4 inch in outside diameter and 5/32 inch deep; diameter of hole at top 11/16 inch, diameter of hole at bottom 3/8 inch; permissible variation in all dimensions of 0.01 inch.

4.3.11.1.3 Steel ball bearings, 5/16 inch in diameter, and weighing between 1.97 and 2.07 grams.

4.3.11.1.4 Glass beaker, 1.5-liter capacity.

4.3.11.1.5 Well-annealed glass test tube, 8 inches long by 2-1/2 inches in inside diameter.

4.3.11.1.6 Two standardized thermometers, 0 to 400°F. range, 0.25 inch in diameter, and 16 inches long.

4.3.11.2 Preparation of sample. Powder the sample in a mortar or break into pieces the size of wheat grains. Place the empty molds on an amalgamated brass plate, fill with the ground resin down into the mold with a spatula. Continue until the mold is filled level with the top and there are no bubbles showing. Separate the mold from the amalgamated plate and trim off the excess resin with a knife. It is convenient to make an indentation of not more than 1 milliliter in the center of the molded resin to prevent the steel ball from rolling off. The resin in the molds should be a clear, fused mass. Only enough heat is applied during filling to melt the resin sufficiently to be packed down smooth. All this operation should be done as rapidly as possible to avoid excess loss of easily volatilized substances in the resin.

4.3.11.3 Procedure. Assemble the apparatus as illustrated by figure 3. See that the bottom of the inner thermometer is even with the bottom of the steel ball on top of the molded resin and the bulb of the outer thermometer is so suspended as to be opposite the lower brass plate. Fill the beaker with water-white glycerine to within 1/2 inch of the top when the test tube is immersed. Heat the beaker containing the glycerine, with test tube and outer thermometer in place, to 150°F. (66°C.). Introduce the resin rings and inner thermometer so that the bottom plate is 1-1/4 inches above the bottom of the test tube. Continue heating and regulate it so that there will be a rise in temperature of 4° to 5°F. (2° to 3°C.) per minute in the bath. The temperature recorded by the inner thermometer at the instant the ball drops through the ring and touches the bottom brass plate shall be taken as the softening point of the sample.

1/ Industry shall be allowed to use any other method provided the product meets the requirement specified herein.

4.3.12 Ash. Weigh 5 to 6 grams of resin into a tared porcelain crucible. Heat the crucible with a gas flame of sufficient intensity to promote slow burning. When the sample has been burned sufficiently to char, transfer to a muffle furnace and ignite at 1200°F. (648° to 704°C.) until all organic matter is destroyed. Cool in a desiccator and weigh. (Make all weighings to the nearest milligram).

4.3.13 Inspection of preparation for delivery. The packaging, packing, and marking shall be examined and tested to determine compliance with section 5 of this specification. Defects shall score in accordance with table III. Sampling shall be in accordance with MIL-STD-105. Defects of closure listed shall be examined on shipping containers fully prepared for delivery. The lot size shall be the number of shipping containers in the end item inspection lot. The inspection level shall be II and the AQL shall be 4.0 defects per hundred units.

TABLE III. Classification of preparation for delivery defects

Examination	Defects
Markings (exterior)	Omitted; incorrect; illegible; improper size; location, sequence, or method of application.
Materials	Any component missing or damaged.
Workmanship	Inadequate application of components such as incomplete closure, inadequate sealing, bulging or distortion of containers.

## 5. PREPARATION FOR DELIVERY

5.1 Packaging, packing, and marking. The resins shall be packaged, packed, and marked in accordance with industry's practices. The resins shall be furnished in standard commercial containers of type, size, and kind commonly used for the purpose, so constructed as to permit acceptance by carrier for transportation at the lowest applicable rate, and to afford maximum protection from normal hazards of transportation. The shipping containers shall be marked with the name of the material, type, and the quantity contained therein and as specified in the contract or purchase order (see 6.2).

5.2 General. The packaging, packing, and marking specified herein apply only to direct purchases by or direct shipments to the Government.

## 6. NOTES

6.1 Intended use. The phenol-formaldehyde resins covered by this specification is intended for use in the manufacture of a wide range of vehicle types and is noted particularly for its ability to produce fast-drying, durable, water- and chemical-resistant finishes.

6.2 The phenol-formaldehyde resins should be purchased by weight, the unit being 1 pound avoirdupois.

6.3 Ordering data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents.

- (a) Title, number, and date of this specification.
- (b) Selection of type (see 1.2).
- (c) Selection of applicable level of packaging, packing, and marking required (see 5.1).
- (d) Size of container (see 5.1).

6.4 The identification of this resin (and similar types) can be accomplished by nuclear magnetic resonance (NMR) Spectrometry. The NMR spectrum exhibits a small resonance peak at position A, 1.68 ppm ( ) due to the geminal dimethyl group of the modifying phenol and a large singlet at position B, 1.27 ppm ( ), due to the tertiary butyl group of the t-butyl phenol moiety (see figure 4). The signal at position A is somewhat complex due to the incorporation of a spinning side band.

Instrument: Varian EM - 360 (Varian Instruments, Palo Alto, California) or equivalent 60 MHz NMR Spectrometer.

Reference: Tetramethyl Silane (TMS).

Identification: Average the signal heights  $I_A$ , and  $I_B$ , of two integration sweeps (upfield and downfield) for the resonance peaks at 1.68 ppm(A) and at 1.27 ppm(B).

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**Civil Agency Coordinating Activities:**

GSA - FSS  
VA - DM & S  
HEW - NIH  
JUSTICE

**Preparing Activity:**

GSA - FSS

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Orders for this publication are to be placed with General Services Administration, acting as an agent for the Superintendent of Documents. See section 2 of this specification to obtain extra copies and other documents referenced herein.

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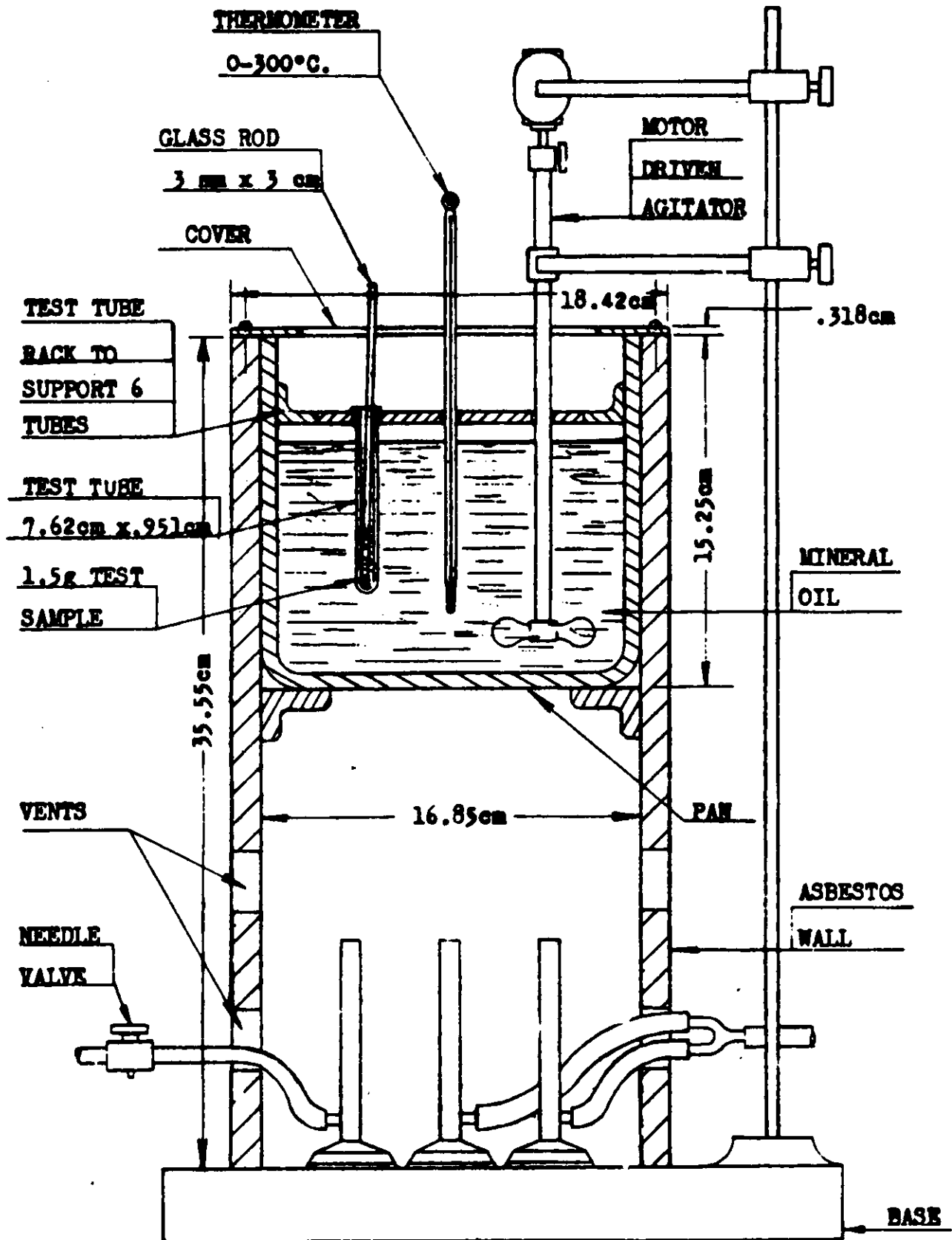


FIGURE 1.--Effect on tung oil test apparatus.

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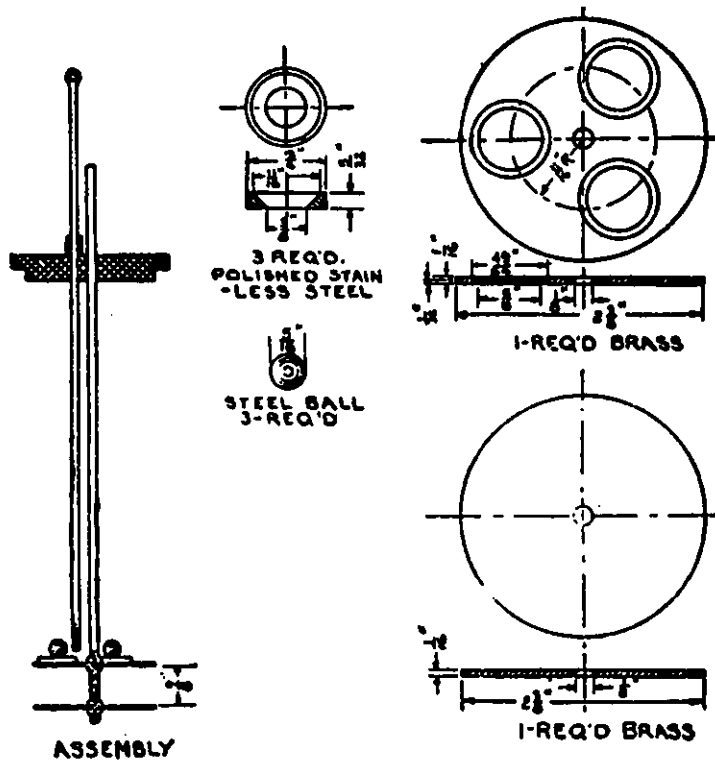


FIGURE 2. Detail of softening apparatus.

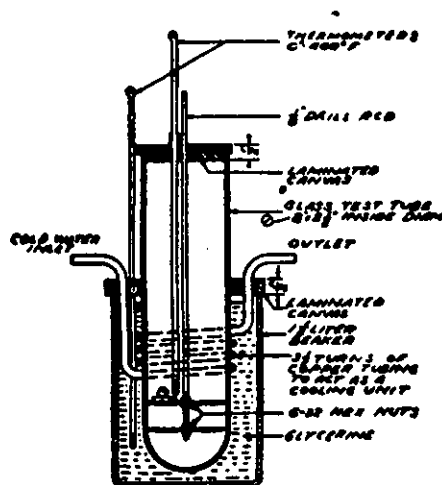


FIGURE 3. Cross section of softening point apparatus assembly.



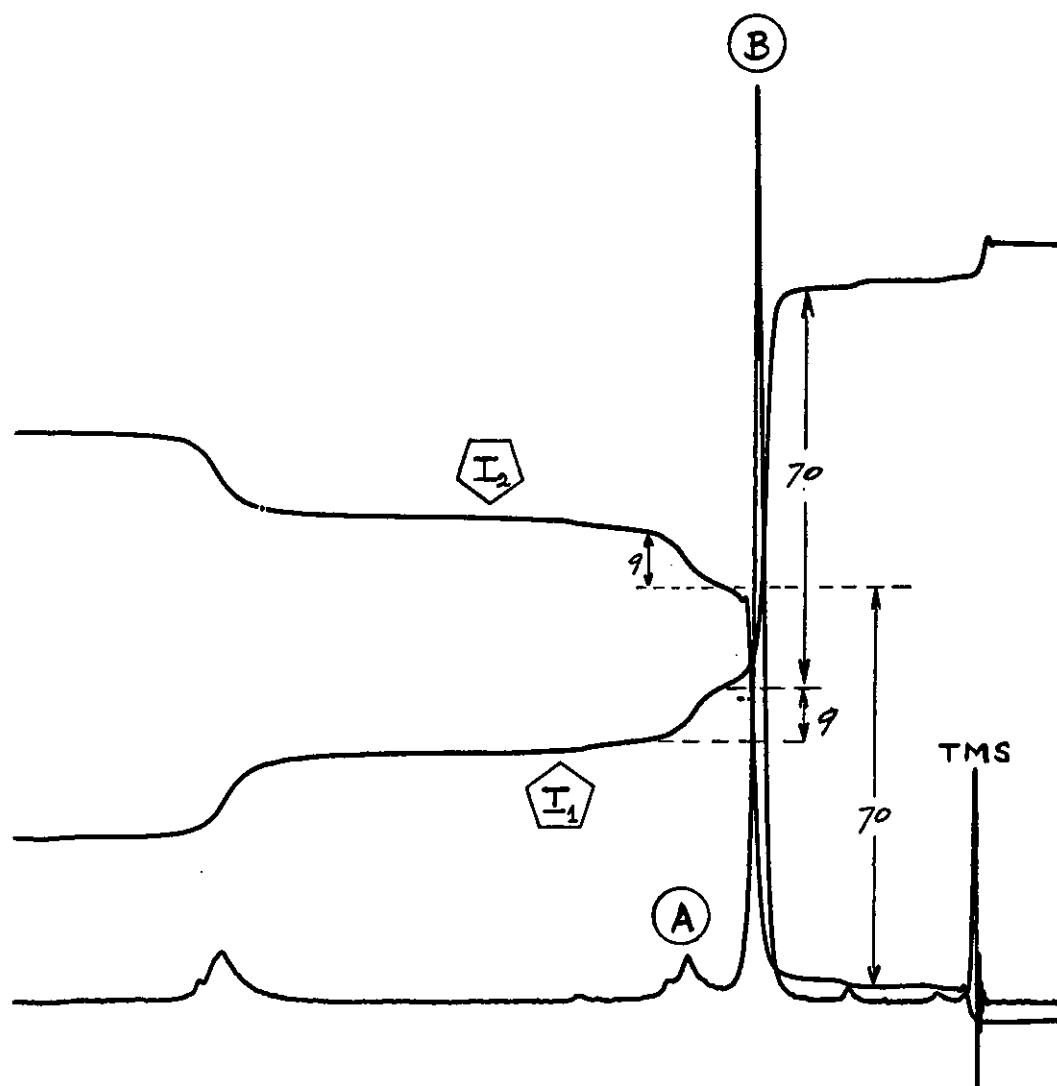


FIGURE 4

NMR SPECTRUM OF CKM-2400

A: gem dimethyl resonance (6 protons)

B: t-butyl resonance (9 protons) $I_1$ , and  $I_2$ : integration sweeps

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