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METHODS OF SAMPLING AND TEST FOR THINNERS AND SOLVENTS FOR PAINTS

(First Revision)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR THINNERS AND SOLVENTS FOR PAINTS

(First Revision)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR THINNERS AND SOLVENTS FOR PAINTS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 20 September 1973, after the draft finalized by the Paints and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1950 based largely on the interim co-ordinated draft produced with the assistance of the representatives of manufacturers and various departments and authorities of the Government of India, by the Co-ordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters (now Army Headquarters), India.

0.3 During the use of the earlier version of this standard a need was felt to revise the standard to introduce the modern developments in analytical techniques in the standard. While revising this standard the committee examined test methods for a number of individual thinners and solvents and felt that tests which are specific in nature may not be included in this revision. After careful consideration, it was decided to prescribe common tests applicable to a majority of solvents and thinners.

0.4 Test methods not described in this standard, and not specified in the standard for an individual material, are subject to agreement between the purchaser and the supplier.

0.5 Should any inconsistency exist between the requirements of this standard and the standard for an individual material, the latter shall prevail.

0.6 In the preparation of this standard, the committee has freely drawn upon the experience of all interests concerned in the use of the original standard and the assistance of relevant publications of the British Standards Institution and the American Society for Testing and Materials.

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes methods of sampling and test for thinners and solvents for paints. It covers the methods for determination of colour, relative density, distillation range, residue on evaporation, tests for corrosive sulphur, freedom from chlorinated hydrocarbon solvents and benzene, kauri butanol value, aniline point and mixed aniline point, acid wash test, test for hydrogen sulphide and mercaptans, freedom from lead, flash point and keeping quality.

2. TERMINOLOGY

2.0 For the purpose of this standard, the definitions given in IS : 1303-1963* and the following shall apply.

2.1 Approved Sample — The sample accepted by the indentor or inspection authority as the basis for supply. When a sample is tested and approved by the purchaser or inspection authority, the result of such tests as permits the supplier to meet the limits imposed by the specification for deliveries shall be made available to the supplier.

2.2 Tender Sample — The sample submitted by the supplier with his tender.

2.3 Freedom from Lead (Lead Free) — It shall mean that the material does not contain lead or compounds of lead or a mixture of both, calculated as metallic lead (Pb), exceeding 0·03 percent by mass.

2.4 Standard Atmospheric Conditions for Testing — An atmosphere with a relative humidity of 65 ± 2 percent and a temperature of 27 ± 2°C, provided that in a given series of experiments the temperature does not vary by more than ± 1°C (*see also* IS : 196-1966†).

2.5 Room Temperature — A temperature between 21 and 38°C unless otherwise specified.

2.6 Aniline Point — The minimum equilibrium solution temperature for equal volumes of aniline and solvent.

2.7 Mixed Aniline Point — The minimum equilibrium solution temperature of a mixture of two volumes of aniline, one volume of sample, and one volume of *n*-heptane of specified purity.

3. SAMPLING

3.1 General Requirements

3.1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

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*Glossary of terms relating to paints (*revised*).

†Specification for atmospheric conditions for testing (*revised*).

3.1.1 Samples shall not be taken in an exposed place.

3.1.2 The sampling instrument shall be clean and dry and shall be made of low or reduced spark-generating material.

3.1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

3.1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

3.1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour.

3.1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

3.1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, name of material, the date of sampling, the year of manufacture of the material, the batch number, the name of the sampler, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

3.1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

3.1.9 *Additional Precautions* — The following additional precautions shall be observed:

a) Rubber stoppers of composition corks shall not be used for closing the sample bottles;

b) Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened; and

c) Each sample container shall be protected by covers of oil proof paper, metal foil, viscous or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

3.2 Sampling Instrument

3.2.0 The following forms of sampling instrument may be used:

a) Sampling bottle or can for taking samples from various depths in large tanks, and

b) Sampling tube.

3.2.1 *Sampling Bottle or Can* — It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 1). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

3.2.2 *Sampling Tube* — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical having 5 to 10 mm internal diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

3.2.2.1 For small containers, the size of the sampling tube may be altered suitably.

3.3 Scale of Sampling

3.3.1 *Lot* — In a single consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of containers of different sizes or of different batches of manufacture, then the containers belonging to the same size and batch of manufacture shall be grouped together and each such group shall constitute a separate lot. In case consignment is in large tanks or vessels, the tanks or vessels belonging to the same batch of manufacture shall constitute a lot.

3.3.1.1 For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

3.3.2 *Sampling from Containers* — The number n of containers to be selected for sampling shall depend on the size N of the lot and shall be in accordance with Table 1.

TABLE 1 SCALE OF SAMPLING

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 100	5
101 to 200	6
201 ,, 300	7
301 ,, 400	8
401 ,, 500	9
501 and above	10

NOTE — When the size of the lot is five or less, all the containers shall be sampled.

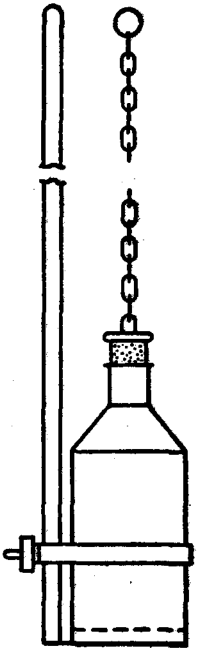

FIG. 1 SAMPLING BOTTLE OR CAN

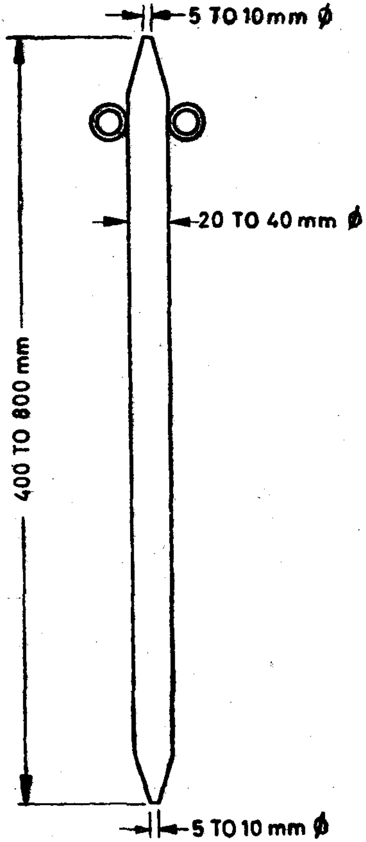

FIG. 2 SAMPLING TUBE

3.3.2.1 The containers shall be selected at random and to ensure the randomness of selection, random number tables may be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them in one order as 1, 2, 3,......, etc, up to r and so on, where r is the integral part of N/n (N being the lot size and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to constitute the sample for test.

3.3.3 *Sampling from Tanks or Vessels* — Each of the tanks or vessels in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the specification.

3.4 Preparation of Test Samples

3.4.1 *Test Samples from Container* — To ensure that the sample taken from each container is fairly representative, the contents shall be mixed thoroughly, when possible, by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of the sampling tube (*see* Fig. 2). The approximate quantity of the material to be drawn from a container shall be nearly equal to thrice the quantity required for testing purposes.

3.4.1.1 Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics specified in the material specification. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

3.4.1.2 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier, and the third for the referee.

3.4.1.3 All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **3.1.7**.

3.4.1.4 The referee test sample, consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

3.4.2 *Test Samples from Tanks or Vessels*

3.4.2.1 For drawing a sample from a tank or vessel lower the closed sampling bottle or can (*see* **3.2.1**) slowly to the required depth, open and fill it at that depth. Four samples shall be obtained at levels of one tenth of the depth of the liquid from the top surface (top sample), one half of the depth (middle sample), nine-tenths of the depth of the liquid from the top surface (lower sample) and one sample from the bottom (bottom sample). All the four samples thus obtained from a tank or vessel shall be mixed together in a clean dry container, and shall be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. Each of the tanks or vessels in the lot shall be sampled in the above manner and separate samples obtained for each of the tanks or vessels. The approximate quantity of the material to be drawn from a tank or vessel shall nearly be equal to thrice the quantity required for carrying out tests for all the requirements prescribed in the material specification.

3.4.2.2 All the samples thus obtained from tanks or vessels in the lot shall be transferred to separate sample containers. The containers shall then be sealed air-tight with stoppers and labelled with full indentification particulars given in **3.1.7**.

3.4.2.3 The referee test samples, consisting of the samples from the tanks or vessels in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

4. PRELIMINARY EXAMINATION OF SAMPLES

4.1 The original sealed sample containers shall be opened and the condition of the contents noted paying particular attention to homogenity, clarity (freedom from haziness and turbidity), presence of foreign matter, and visible impurities.

5. COLOUR

5.0 Outline of the Method — The colour of the material in cylinders is compared against the specified standard solution in Nessler cylinders.

5.1 Apparatus

5.1.1 *Nessler Cylinders* — of 50 ml capacity, conforming to IS : 4161-1967*.

5.2 Procedure — Filter the material through a 150 mm diameter Whatman No. 31 filter paper and reject first 10 ml of filtrate. Pour 50 ml of the material in one of the two cylinders. Pour into the other Nessler cylinder 50 ml of the standard solution as prescribed in the individual material specification. Hold the Nessler cylinders in a comparator above the surface of an opaque glass sheet reflecting diffused daylight and examine visually.

6. RELATIVE DENSITY†

6.1 General — For the purpose of this standard, the relative density of the material is the ratio of the mass of a given volume of the material at a specified temperature to that of an equal volume of distilled water at the same temperature.

6.1.1 If the relative density is to be determined at a temperature other than the specified temperature, the value at the specified temperature shall be calculated as given below:

a) If the temperature of testing is higher than the specified temperature, add 0·000 65 to the value for relative density obtained for each degree Celsius of the difference between the two temperatures.

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*Specification for Nessler cylinders.

†Synonymous with specific gravity.

b) If the temperature of testing is lower than the specified temperature, subtract 0·000 65 from the value of relative density obtained for each degree Celsius of the difference between the two temperatures.

6.2 Apparatus — Any one of the following may be used:

a) Standard hydrometer,

b) Westphal hydrostatic balance, and

c) Relative density bottle or pyknometer.

6.2.1 In case of dispute, the relative density bottle or pyknometer shall be used.

6.3 Procedure

6.3.1 *With Standard Hydrometer* — Pour the material to be tested into a clean hydrometer jar without splashing so as to avoid the formation of air bubbles. The diameter of the jar shall be at least 2·5 cm greater than the diameter of the hydrometer to be used therein. If air bubbles are formed, remove them after they have collected on the surface by touching them with a piece of clean blotting paper or filter paper. Keep the jar in a vertical position in a place free from air currents and in a bath maintained at the specified temperature. Lower the hydrometer gently into the sample, and when it has settled, depress it about two scale divisions into the liquid, the unimmersed portion of the stem being kept dry, as any unnecessary liquid on the stem changes the effective mass of the instrument, and affects the reading obtained. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. Read the point on the hydrometer scale to which the material rises with the eye placed at the principal surface of the liquid.

6.3.2 *With Westphal Hydrostatic Balance* — Suspend the plummet in the cylinder filled with recently boiled distilled water at the specified temperature and place the largest rider on the hook. Adjust the screw on the base until the pointer is exactly opposite the fixed indicator point. Wipe the plummet and the cylinder to remove the water. Fill the cylinder with the material at the same temperature and dip the plummet into the material removing air bubbles, if any, formed in the eyehole of the plummet, by lifting it from the material. Re-immerse the plummet in the material and adjust the height of the balance till the plummet is nearly in the middle of the column of the liquid. Place riders on the beam till the pointer and the fixed indicator are exactly opposite each other. Read the relative density from the position of the riders on the beam, beginning with the largest and ending with the smallest.

6.3.3 *With a Relative Density Bottle or Pyknometer*—Use a relative density bottle or pyknometer of 25 ml capacity. Weigh it in a clean and dry condition. Fill it up to the mark with distilled water at the specified temperature and weigh. Remove the water, dry and fill with the material at the same temperature and weigh.

6.3.4 *Calculation*

$$\text{Relative density} = \frac{c - a}{b - a}$$

where

c = mass in g of relative density bottle or pyknometer filled with the material,

a = mass in g of the dry relative density bottle or pyknometer, and

b = mass in g of relative density bottle or pyknometer filled with recently distilled water.

7. DISTILLATION RANGE

7.1 The determination of distillation range shall be done as prescribed in IS : 5298-1969*, except that the constructional details of the thermometers used shall be as given in Table 2. If the thermometers of prescribed range are not available, the thermometers of nearest equivalent range may be used.

TABLE 2 CONSTRUCTIONAL DETAILS OF THERMOMETERS

Ange	Graduation at Each	Longer Lines at Each	Fully Figured at Each	Maximum Overall Length	Minimum Length of Main Scale	Maximum Bulb Length	Maximum Error	Maximum Distance from Bottom of Bulb to Top of Contraction Chamber
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
°C	°C	°C	°C	mm	mm	mm	°C	mm
65 to 90	0·1	1	1	400	215	12	± 0·4	25
70 to 130	0·2	1	2	380	200	15	± 0·6	25
130 to 160	0·1	1	1	420	240	12	± 0·6	25
50 to 210	0·5	5	5	430	240	12	± 0·8	25

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*Method for determination of distillation range and of distillation yield.

8. RESIDUE ON EVAPORATION

8.0 Outline of the Method — Two methods are employed. In one method the material is evaporated on a water-bath without preliminary distillation. In the second method a preliminary distillation is carried out. The second method shall be used for coal tar distillates like light naptha, heavy naptha, benzole and xylole, etc.

8.1 Estimation Without Preliminary Distillation

8.1.1 *Procedure* — Evaporate 100 ml of the sample to dryness in a weighed glass or silica crucible of 150 ml capacity on a water-bath. Dry the residue further in an air-oven at a temperature of 100 ± 2°C for the period prescribed in Table 3. Cool in a desiccator, weigh and calculate the residue as milligrams per 100 ml of the material.

8.2 Estimation with Preliminary Distillation

8.2.1 *Apparatus* — The assembly shall consist of the following:

a) A round-bottomed flask of about 250 ml total capacity with ground joint;
b) A distillation head with a ground-glass joint to fit the flask;
c) A 100-ml graduated receiver; and
d) Dishes, hemispherical in shape (without lip), 66 ±2 mm in external diameter and 18 ± 3 g in mass.

8.2.2 *Procedure* — Measure 100 ml of the sample into the flask and connect the flask to the distillation head. Attach the distillation head to an efficient condenser which does not hold up liquid and which is fitted with a bent end. Place the flask on an asbestos sheet of 150 × 150 mm, and having a central hole 40 mm in diameter. Pass the delivery end of the condenser through a cork inserted in the 100-ml graduated receiver, care being taken that the cork is provided with another tube through which the gases and uncondensed vapour evolved during the distillation can be led away from any source of ignition, Bring the contents of the flask cautiously to boiling over a boiling water-bath or a hot-plate or a bunsen flame, avoiding any sudden evolution of vapour, and distil at the rate of 4 to 5 ml per minute (about 2 drops per second) until 75 ml of distillate collects in the receiver. Disconnect the flask from the distillation head and pour the contents into one of the dishes which has been dried previously in a steam-oven, cooled in a dust-free atmosphere for 15 to 20 minutes and weighed. Allow the flask to drain into the dish in an inverted vertical position for 15 seconds. Place the dish at once on a vigorously boiling water-bath, supported in a hole having a diameter of 64 ± 1 mm. Protect the dish from dust. After the dish has been kept on the water-bath for 90 minutes, transfer it to an oven and keep at a temperature of 100 ± 2°C for the period specified in Table 3. Allow it to cool for 15 to 20 minutes in a desiccator, weigh and express the result as grams of residue per 100 ml of sample.

TABLE 3 DURATION OF OVEN HEATING FOR THE DETERMINATION OF RESIDUE ON EVAPORATION

(*Clauses* 8.1.1 *and* 8.2.2)

Sl No.	Material	Duration in Minutes for Drying of Residue in the Oven
(1)	(2)	(3)
i)	Turpentine (*see* IS : 6646-1972[*])	120
ii)	White spirit and thinners for paints and enamels conforming to IS : 1872-1961[†]	90
iii)	Coal tar naphtha light and thinner conforming to IS : 1873-1961[‡] and IS : 5667-1970[§]	90
iv)	Benzol (*see* IS : 358-1964[‖])	60
v)	Xylole (*see* IS : 359-1965[¶])	60
vi)	Ethyl alcohol (*see* IS : 321-1964[**])	15
vii)	Acetone (*see* IS : 170-1966[††])	30
viii)	Amyl acetate (*see* IS : 231-1957[‡‡])	60
ix)	Amyl alcohol (*see* IS : 360-1964[§§])	60
x)	Trichloroethylene (*see* IS : 245-1971[‖‖])	30
xi)	Methylene chloride (*see* IS : 4566-1968[¶¶])	30

[*] Specification for oil of turpentine, solvent grade.

[†] Specification for thinner for synthetic paints and varnishes for aircrafts.

[‡] Specification for thinner, antichill for cellulose nitrate based paints, dopes and lacquers for aircrafts.

[§] Specification for thinner for cellulose nitrate based paints and lacquers.

[‖] Specification for benzole, industrial (*revised*).

[¶] Specification for xylole, industrial solvent grade (*revised*).

[**] Specification for absolute alcohol (*revised*).

[††] Specification for acetone (*first revision*).

[‡‡] Specification for amyl acetate.

[§§] Specification for amyl alcohol (*revised*).

[‖‖] Specification for trichloroethylene, technical (*second revision*).

[¶¶] Specification for methylene chloride (dichloromethane), technical.

In the case of fractions distilling less than 50 percent at 100°C and less than 95 percent at 145°C, continue evaporation on the water-bath if necessary, for a period longer than 90 minutes and until the evaporation is complete.

9. KAURI-BUTANOL VALUE DETERMINATION

9.0 Outline of the Method — This test is intended for determination of relative solvent power of the material. The material is titrated against a standard kauri-butanol solution to an end point where the sharp outlines of 10 point print placed below the water-bath become blurred and observed through the liquid. From the volume of the material consumed, the kauri-butanol value is calculated.

9.1 Apparatus

9.1.1 *Water-Bath* — of clear glass vessel.

9.1.2 *Flasks* — one 200 ml capacity volumetric flask and one 250 ml capacity long-necked flask.

9.1.3 *Burette* — of 50 ml capacity.

9.1.4 *Print Specimen* — A sheet of white paper having on it black 10 to 12 point print, monotype Baskerville; a specimen of the print is given below:

> The volume in millilitres at 25°C of the solvent, corrected to a defined standard, required to produce a defined degree of turbidity when added to 20 g of standard solution of kauri resin in *n*-butyl alcohol. For kauri-butanol values of 60 and above, the standard is toluene and has an assigned value of 105. For kauri-butanol values below 60, the standard is a blend of 75 percent *n*-heptane and 25 percent toluene and has an assigned value of 40.

9.2 Reagents

9.2.1 *Kauri-Butanol Solution* — Place in a 3-litre flask 400 g of clean pale, bold kauri resin of ASTM grade XXXX, XXX or XX ground to pea-size. Add, while shaking vigorously, 2 000 g of *n*-butyl alcohol conforming to IS : 361-1962*. Reflux the contents over a water-bath till the resin goes into solution. Allow the solution to stand for 48 hours and then filter through a Buchner funnel using double filter paper.

9.2.2 *Toluene* — conforming to IS : 537-1967†.

9.2.3 *Heptane-Toluene Blend* — consisting of 25 ± 0·1 percent toluene and 75 ± 0·1 percent *n*-heptane.

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*Specification for normal butyl alcohol, technical (*revised*). (Since revised).

†Specification for toluene, pure, nitration grade (*first revision*).

9.3 Procedure

9.3.1 *Standardization of Kauri-Butanol Solution* — Weigh accurately 20 ± 0·10 g of kauri-butanol solution in a 250-ml long-necked flask and keep it in the water-bath maintained at 25°C. Titrate the contents with standard toluene with constant swirling keeping the temperature at 25°C. Continue the titration with successive reduced amount of toluene as the end point is approached. The end point is reached when the sharp outlines of 10 point print placed below the water-bath and observed through the liquid are obscured or blurred. Alternatively, if the titration is done in a room whose temperature is controlled at 25 ± 1°C, the 10 point print is placed below the flask itself and end point is viewed through the contents of the flask. Check the temperature of the bath immediately and repeat the experiment if the variation is more than 1°C from 25°C. Note the volume of toluene used and this represents the actual titre for the particular kauri-butanol solution.

NOTE — This value should preferably lie between 100 to 110 ml. Adjust the concentration of the solution by dilution if these limits are exceeded.

9.3.2 *Standardization of Heptane-Toluene Blend* — Weigh accurately 20 ± 0·10 g of kauri-butanol solution (**9.2.1**) in a 250-ml long-necked flask, keep in the water-bath maintained at 25°C and titrate with heptane-toluene blend. Alternatively, the titration may be carried out in a room whose temperature is controlled at 25 ± 1°C. Note the volume of heptane-toluene blend required to give a blurred vision of the 10 point print when viewed through the liquid.

NOTE — If the composition of the blend is known to differ from 25 ± 0·1 percent toluene but is within the range 22 to 28 percent toluene, the constant in the blend factor equation (*see* **9.4**) will differ from 40·0 by 0·60 units for each one percent toluene. For example, at 28 percent toluene, the constant is 41·8.

9.3.3 *Final Titration* — Weigh accurately 20 ± 0·10 g of the kauri-butanol solution in a 250-ml long-necked flask. Place the flask on a water-bath maintained at 25 ± 1°C. Fill a 50-ml burette with the material and titrate this with kauri-butanol solution with constant stirring. The end point is reached when the sharp outlines of 10 point print placed directly below the water-bath or the flask, in case the titration is done in a room whose temperature is controlled at 25 ± 1°C, and observed through the liquid are blurred. Check the temperature immediately and repeat the experiment if the variation is more than 1°C. Note the volume of the material consumed.

9.4 Calculation

$$\text{Kauri-butanol value} = \frac{65\,(C-B)}{A-B} + 40$$

where

C = volume in ml of the material required to titrate 20 g of kauri-butanol solution (**9.3.3**),

B = volume in ml of heptane-toluene blend required to titrate 20 g of kauri-butanol solution (**9.3.2**), and

A = volume in ml of toluene required to titrate 20 g of kauri-butanol solution (**9.3.1**).

NOTE — If the burette is maintained at a temperature other than 25°C, correct the volume of the titrant used to standard temperature as follows:

$$\text{Correction, per ml} = V\,(25 - T) \times 0{\cdot}0009$$

where

V = volume in ml of titrant used, and

T = temperature of titrant in the burette in degree centigrade.

10. ANILINE POINT AND MIXED ANILINE POINT OF HYDROCARBON SOLVENTS

10.1 Aniline Point

10.1.1 *Outline of the Method* — Known and equal volumes of aniline and dry sample are taken into an air-jacketed tube and stirred rapidly till the mixture is completely miscible, if necessary by heating. Then the mixture is allowed to cool slowly and the temperature at which the mixture becomes cloudy is recorded as aniline point.

10.1.2 *Apparatus*

10.1.2.1 *Aniline point apparatus* — It shall consist of the following main components (*see also* Note 1 under **10.1.2.5** and Fig. 3):

a) *Test-tube* — of heat resistant glass, approximately 25 mm in diameter and 150 mm in length.

b) *Jacket tube* — of ~~heat resistant glass,~~ approximately 37 to 42 mm in diameter and 175 mm in length.

c) *Stirrer* — manually operated suitable metal stirrer of soft iron wire, as shown in Fig. 3. A concentric ring having a diameter of approximately 19 mm shall be at the bottom, and the length of the stirrer to a right-angle bend shall be approximately 200 mm. The right angle bend shall be approximately 55 mm in length. A glass sleeve, approximately 65 mm in length and having an inside diameter of 3 mm, shall be used as a guide for the stirrer. Any suitable mechanical device for operating the specified stirrer is an approved alternate for the manual operation.

d) *Thermometers* — ASTM aniline point thermometers having ranges of −38 to +42°C, 25 to 105°C, and 90 to 170°C respectively, or equivalent thermometers of suitable range.

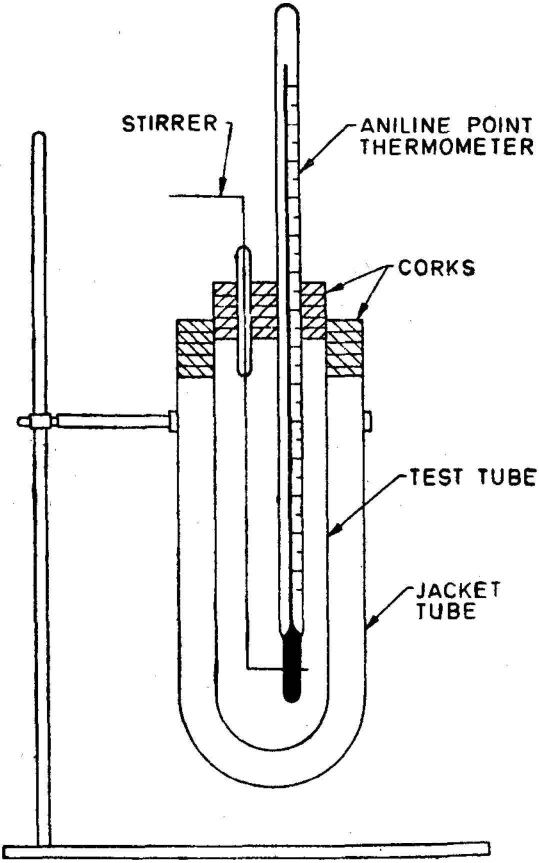

FIG. 3 ANILINE POINT APPARATUS

10.1.2.2 *Cork stoppers* — numbers 12 and 24.

10.1.2.3 *Pipettes* — Two of 10 ± 0·04 ml size, and two of 5 ± 0·02 ml size. A rubber ~~suction~~ bulb shall be provided for use with one of the 10-ml pipettes when measuring aniline.

10.1.2.4 *Heating-bath* — a suitable non-aqueous, nonvolatile, transparent liquid-bath. An open flame, or an infrared lamp (250 to 375 W) provided with suitable means for temperature control may be substituted for the liquid-bath. A hot water-bath shall not be used (*see* Note 2 under **10.1.2.5**).

10.1.2.5 *Cooling-bath* — A nonaqueous cooling-bath shall be substituted for the heating-bath when the aniline point or mixed aniline point is below room temperature (*see* Note 2).

NOTE 1 — Acceptable alternate apparatus may be used, provided it has been shown to give results of the same accuracy and precision as those described in this method.

NOTE 2 — Water should not be used as either a heating or cooling medium, since aniline is hygroscopic and moist aniline will give erroneous test results. For example, the aniline point of the *n*-heptane reagent as measured with dry aniline is increased from that specified by approximately 0·5°C if the aniline reagent contains 0·1 percent water.

10.1.3 *Reagents*

10.1.3.1 *Aniline* — Aniline meeting the following requirements on the day of use shall be satisfactory. Determine the aniline point of standard *n*-heptane in duplicate. The individual results shall not differ by more than 0·1°C, and the average shall be between 69·1°C and 69·5°C. If these requirements are not met, purify the aniline by drying over potassium hydroxide (KOH) pellets, decant, and distil (*see* Notes 1 and 2) discarding the first and last 10 percent. Retest the aniline.

NOTE 1 — Purified aniline can be stored by collecting the distillate in clean, dry ampules, sealing the ampules under vacuum or dry nitrogen, and storing in a cool, dark place for future use.

NOTE 2 — *Caution*: Aniline is highly toxic. Care shall be taken not to get it on the skin or clothing, even in very small quantities. It shall not be pipetted directly by mouth suction because of its extreme toxicity. Avoid breathing fumes. Aniline is also toxic by absorption through skin, even in very small quantities, and shall be handled with caution.

10.1.3.2 *Calcium sulphate* (*drierite*) — anhydrous ($CaSO_4$).

10.1.3.3 *n-Heptane* — conforming to the requirements given below:

ASTM motor octane number	0·0 ± 0·2
Density at 20°C, g/ml	0·683 80 ± 0·000 15
Refractive index at 20°C	1·387 70 ± 0·000 15
Freezing point, °C	— 90·710, *Min*
Distillation:	
50 percent recovered, °C (at 760 mmHg)	98·427 ± 0·025°C
Differential, 80 percent recovered minus 20 percent recovered, °C	0·020, *Max*

10.1.4 *Procedure*

10.1.4.1 Clean and dry the apparatus. The sample to be tested shall be dried by shaking for at least 2 minutes with anhydrous $CaSO_4$ (Drierite). Pipette 10 ml of aniline (*see* Note 2 under **10.1.3.1**) and 10 ml of the dry sample into the air-jacketed tube fitted with stirrer and thermometer. If the material is too volatile or too viscous for pipetting, weigh a quantity of the sample corresponding to 10 ml at room temperature in a test tube to the nearest 0·01 g. Centre the thermometer in the test tube so that the immersion mark is at the liquid level, making sure that the bulb does not touch the side of the tube.

10.1.4.2 Stir the sample rapidly, using a 5-cm stroke and avoiding the inclusion of air bubbles, and if necessary, heat until complete miscibility is obtained. If the aniline-sample mixture is completely miscible at room temperature, substitute a non-aqueous cooling-bath for the heating-bath. continue stirring and allow the mixture to cool slowly at a rate of 0·5 to 1°C/min. Continue cooling to a temperature 1 to 2°C below the first appearance of turbidity and observe whether the turbidity increases markedly. Record the temperature at which the mixture becomes cloudy throughout with complete separation of aniline and sample. This will be the minimum equilibrium solution temperature and not the temperature of separation of minor portions of the sample (*see* Note).

NOTE — The true aniline point is characterized by a turbidity which increases sharply as the temperature is lowered.

10.1.4.3 Repeat the observation of aniline point temperature by heating and cooling to obtain three successive determinations. If these do not differ by more than the stated repeatability of the method average, apply the thermometer correction, and round to the nearest 0·1°C.

10.2 Mixed Aniline Point

10.2.1 *Procedure* — This procedure is applicable to samples having aniline points below the temperature at which aniline crystallizes from the mixture, and to samples for which the mixed aniline point is a specification requirement. Pipette 10 ml of aniline (*see* Note 2 under **10.1.3.1**), 5 ml of sample, and 5 ml of *n*-heptane into a clean and dry apparatus. Determine the aniline point of the mixture as given in **10.1**.

10.3 Report

10.3.1 *Aniline Point* — Report the minimum equilibrium solution temperature, to the nearest 0·1°C, of the mixture of equal volumes of aniline and sample as the aniline point of the sample.

10.3.2 *Mixed Aniline Point* — Report the minimum equilibrium solution temperature, to the nearest 0·1°C, of the mixture of two volumes of aniline, one volume of sample, and one volume of *n*-heptane as the mixed aniline point of the sample.

NOTE — If a ratio other than one volume of heptane to one volume of sample is used, the ratio used must be stated when reporting the mixed aniline point.

10.4 Precision

10.4.1 *Repeatability* — Duplicate results, that is, two average temperatures obtained by the same operator in a series of observations as described in **10.1.4** shall not differ by more than 0·2°C.

10.4.2 *Reproducibility* — The result obtained by each of the two laboratories shall not differ by more than 0·5°C and 0·7°C for aniline point and mixed aniline point respectively.

11. TEST FOR CORROSIVE SULPHUR

11.0 General

11.0.1 *Outline of the Method* — A clean copper foil is dipped into the material for two hours at specified temperature and examined for change in colour.

11.1 Materials

11.1.1 *Copper Foil* — of electrolytic copper, size 40 × 30 × 0·8 mm.

11.1.2 *Test Tubes* — thick walled, about 40 mm in diameter and 175 mm in length, fitted by means of a cork to a Liebeg condenser having a waterjacket.

11.2 Procedure — Clean the copper foil on both sides until the surface is highly polished, by rubbing it with a cork moistened with distilled water and dipped in dry emery powder passing 75 micron IS Sieve. Wipe the foil with filter paper moistened with acetone (*see* IS : 170-1966*), care being taken that it is not touched by fingers. Cut the cleaned foil into two strips of 30 × 20 mm each. Place one strip in a clean test tube and cork the tube. Introduce the other strip into the thick walled test tube, containing 50 ml of the material under examination. Fit this test tube to the reflux condenser and allow it to remain for two hours in a water-bath maintained at 50 ± 2°C.

11.3 Comparison and Report — Compare the appearance of the two copper strips and report the result of exposure to the material under one or other of the following descriptions:

a) No change;
b) Slight decolouration;
c) Brown or peacock shade;
d) Steel grey;
e) Black, not scaled; and
f) Black, scaled.

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*Specification for acetone (*first revision*).

11.4 Both the free sulphur and hydrogen sulphide discolour the copper under the conditions of the test. The extent to which corrosion takes place may be affected by the presence of various substances, including peroxides, which will inhibit the corrosion. The presence or absence of peroxides may be ascertained by shaking the material with 25 percent by volume of a freshly prepared 5 percent (*m/v*) solution of potassium iodide to which a few drops of starch solution have been added. Peroxides will immediately produce a strong blue colouration.

12. TEST FOR FREEDOM FROM CHLORINATED HYDROCARBON SOLVENTS AND BENZENE

12.0 Outline of the Method — The material is steam distilled and solvents collected. Solvents are subjected to test for benzene and chlorinated hydrocarbons qualitatively.

12.1 Apparatus

12.1.1 *Steam Distillation Apparatus* — any suitable apparatus.

12.1.2 *Distillation Flask* — 100-ml capacity.

12.1.3 *Bunsen Burner*

12.2 Reagents

12.2.1 *Nitric Acid* — conforming to IS : 264-1968*.

12.2.2 *Sulphuric Acid* — conforming to IS : 266-1961†.

12.2.3 *Copper Wire* — 0·914 to 1·21 mm in diameter.

12.3 Procedure — Steam distil 100 ml of the material using a receiver cooled in ice. Separate the solvent layer, wash with several times its volume of distilled water, discard the washings, dry and redistil, collecting all solvent within the agreed specified boiling range, subject this to the tests prescribed in **12.3.1** and **12.3.2**.

12.3.1 *Test for Benzene* — Mix equal volumes of nitric acid and sulphuric acid in a test tube and cool. To 2 ml of this solution add 10 drops of the solvent and shake. Warm, if necessary and examine for the odour of nitrobenzene which is given by 5 percent benzene or larger amounts of toluene. Boil the contents for at least 30 seconds, allow to cool and add 10 ml of water. Mix the contents and examine for odour fo nitrobenzene, which should be no longer present if the contents were boiled enough. Allow the tube to stand for some time. A yellow cloudy solution or flocculant precipitate changing to yellow needles is evidence of the presence of benzene. It may further be confirmed by separating the yellow needles and finding out their melting point which shall be within 89 to 90°C.

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*Specification for nitric acid (*first revision*)

†Specification for sulphuric acid (*first revision*)

12.3.1.1 In case of dispute gas chromotography may be employed to detect the presence of benzene using analytical grade benzene as standard.

12.3.2 *Test for Chlorinated Hydrocarbons*—Form a small loop in the copper wire and heat in a small Bunsen flame until it no longer colours the flame. Allow the loop to cool and then dip it into the test solution. Immediately place in the outer part of the flame and when the first luminous flame disappears, examine for the green colouration due to chlorinated compounds.

12.3.3 The material shall be deemed to have passed the test if the solution when tested as prescribed in **12.3.1** does not give off smell of nitrobenzene or forms yellow crystals and when tested as prescribed in **12.3.2** does not impart a green colouration to the Bunsen flame.

13. ACID WASH TEST

13.0 Outline of the Method—The material is shaken with an equal volume of 95 percent (*m/m*) sulphuric acid and the colour of the acid layer is compared with the specified standard.

13.1 Reagent

13.1.1 *Colour Standards*—Dissolve the specified amount of potassium bichromate (conforming to IS:250-1964*) in the appropriate volume of a mixture of equal volumes of concentrated sulphuric acid relative density 1·84 and distilled water.

13.2 Procedure—Filter about 20 ml of the material, reject the first 10 ml of the filtrate, and introduce 5 ml of the remainder into a dry, stoppered, coloured glass cylinder about 15 mm in diameter and about 100 mm in height to the shoulder. Introduce into the cylinder 5 ml of 95 percent (*m/m*) sulphuric acid, shake the cylinder for exactly two minutes and allow it to stand undisturbed for exactly 10 minutes. Make the comparison immediately with the specified standard contained in a cylinder identical in all respects, including colour, with the cylinder containing the sample and acid.

13.2.1 A suitable Lovibond comparator may be used.

14. TEST FOR HYDROGEN SULPHIDE AND MERCAPTANS

14.0 Outline of the Method—The material is shaken with sodium plumbite solution in a stoppered glass cylinder. The formation of black precipitate confirms the presence of hydrogen sulphide. Opalescence followed by darkening in colour indicates the presence of both mercaptans and elementary sulphur.

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*Specification for potassium bichromate, technical and analytical reagent (*revised*).

14.1 Reagents

14.1.1 *Sodium Plumbite Solution* — Dissolve about 125 g of sodium hydroxide in 1 litre of distilled water, add to the solution 60 g of litharge (PbO) passing through a 150-micron IS Sieve, and boil the mixture for 30 minutes. Allow it to settle and decant or siphon the clear liquid. Alternatively, add a filtered solution of 25 g of lead acetate crystals in 200 ml of water to a solution of 60 g of sodium hydroxide in 100 ml of water, heat the combined aqueous solutions on a water-bath for 30 minutes and then dilute to 1 litre. Keep in a tightly corked bottle and filter before use, if not clear.

14.1.2 *Sulphur* — Dry flowers of sulphur, chemically pure.

14.2 Procedure

14.2.1 Shake vigorously, for 15 seconds, 10 ml of the material with 5 ml of sodium plumbite solution in a glass-stoppered cylinder of 150 ml capacity and about 25 mm internal diameter. If hydrogen sulphide is present in the sample, there shall be an immediate black precipitate.

14.2.2 Opalescence followed by darkening in colour at this stage, that is, before adding the sulphur indicates the presence of both mercaptans and elementary sulphur.

14.2.3 If hydrogen sulphide is absent, add a small quantity of sulphur (0·05 to 0·1 g), such that practically all of it floats on the interface between the sample and the sodium plumbite solution. Shake the cylinder for 15 seconds and allow to settle for one minute before making observations on the contents of the cylinder.

14.3 Interpretation of Results — If the sample and the sodium plumbite solution show no change of colour and the sulphur film remains bright yellow, report the test as 'Negative'. If the sample or the sodium plumbite solution is discoloured, or the yellow colour of the sulphur is at all masked, report the test as 'Positive'. If the discolouration be very slight, report the test as 'Positive but discolouration very slight'.

14.3.1 Oxidized samples may contain peroxides giving a brown precipitate not darkend by the addition of sulphur; this must not be confused with the effect of mercaptans. The presence or absence of peroxides may be ascertained by the method described in **11.4**.

14.3.2 Carbon disulphide, particularly in concentrations exceeding 0·4 percent of sulphur present as carbon disulphide, causes a darkening of the aqueous layer on standing. The test is, therefore, unreliable for samples containing high amounts of carbon disulphide; and caution shall always be exercised not to confuse this darkening with the immediate blackening due to hydrogen sulphide or that due to mercaptans. Similarly, certain phenolic substances (which may have been introduced as inhibitors) cause colouration of the aqueous layer and, if their presence be expected, a blanck test with sodium hydroxide in place of sodium plumbite solution shall first be carried out for the purpose of comparison.

15. FREEDOM FROM LEAD

15.0 Outline of the Method — Determination of lead is carried out by treating the material with bromine, nitric acid and then ammonium acetate and estimated colourimetrically. If the amount of lead precipitate is large, a gravimetric determination is followed precipitating lead as lead sulphide and finally oxidizing to lead sulphate.

15.1 Apparatus

15.1.1 *Beaker* — tall, about 400 ml capacity.

15.1.2 *Sintered Glass Filter Crucible* — No. 2 porosity.

15.1.3 *Cooling Jar* — containing broken ice and water, of a size in which the beaker can be conveniently placed.

15.1.4 *Measuring Jar* — 100 ml capacity.

15.1.5 *Nessler Cylinders* — two, of 50 ml capacity.

15.2 Reagents

15.2.1 *Bromine Water* — saturated.

15.2.2 *Sodium Sulphide Solution* — 10 percent (m/v).

15.2.3 *Nitric Acid* — relative density 1·42, lead free.

15.2.4 *Dilute Nitric Acid* — 10 percent by volume, lead free.

15.2.5 *Concentrated Sulphuric Acid* — relative density 1·84, lead free.

15.2.6 *Ammonium Acetate Solution* — prepared by mixing one volume of ammonium hydroxide (1 : 1) with two volumes of 35 percent acetic acid.

15.2.7 *Standard Lead Solution* — Dissolve in distilled water 0·1598 g of lead nitrate which has been finely ground previously and dried at 100 ± 2°C. Make up the volume to 1000 ml. Each ml of this solution contains 0·1 mg of lead.

15.3 Procedure

15.3.1 Pour 100 ml of the sample into the beaker placed in the cooling jar. Add bromine water until a permanent red brown colour is obtained.

Filter quickly the precipitate of lead bromide through the sintered glass filter crucible and proceed as described in **15.3.2**. If no precipitate of lead bromide is formed with the addition of bromine water, transfer the contents of the beaker to a glass-stoppered bottle and shake with 20 ml of sodium sulphide solution. Filter the precipitate of lead sulphide through the sintered glass filter crucible and proceed as described in **15.3.2**.

15.3.2 Thoroughly wash the precipitate with petroleum ether. Place the crucible in the beaker in which the precipitation was made and pour 2 ml of concentrated nitric acid into it. Then add dilute nitric acid into the beaker so that the crucible is completely immersed. Boil the contents for about 3 minutes. Remove the crucible and rinse it into the beaker with distilled water. Evaporate the contents of the beaker to about 3 ml. Proceed as described in **15.3.3** unless a large quantity of precipitate is obtained on bromination, in which case proceed as described in **15.3.4**.

15.3.3 Add 10 ml of ammonium acetate solution to the residue obtained in the beaker in **15.3.2**, transfer the contents to the measuring jar and add distilled water to make up the volume to 100 ml. Take 10 ml of this solution in one of the Nessler cylinders, add 5 drops of sodium sulphide solution and make up the volume to 50 ml mark. In the other Nessler cylinder take 5 ml of standard lead solution, add 5 ml of sodium sulphide solution and make up the volume to 50 ml mark. Compare the colour produced in the two cylinders.

15.3.4 If a very large precipitate is obtained on bromination, add 3 ml of concentrated sulphuric acid to the residue in the beaker and evaporate the solution on a sand-bath till fumes appear. Cool, add 100 ml of distilled water and allow to stand on a boiling water-bath for 15 minutes. Then dilute the contents to about 150 ml and allow to stand overnight at room temperature. Filter the insoluble matter on a No. 42 Whatman filter paper (9 cm) and wash thoroughly with 2 percent solution of lead-free sulphuric acid. Transfer the filter paper and residue to a small beaker, cover with 20 ml of distilled water and add 1 to 2 g of solid ammonium acetate. Heat the beaker on a water-bath for not less than half an hour, stirring the contents occasionally. Decant the liquid through No. 42 Whatman filter paper (9 cm). Repeat the extraction with ammonium acetate solution. Transfer all the insoluble matter including the filter pulp to the filter paper and wash thoroughly with warm water, collecting the filtrate and washings in a 150-ml beaker. Pass hydrogen sulphide through the liquid for 10 to 15 minutes and filter the precipitated lead sulphide at once through a No. 40 Whatman filter paper (9 cm). Wash thoroughly but quickly with hydrogen sulphide water. Transfer the precipitate and filter paper to a tared silica crucible. Dry carefully, ignite to sulphate, cool and weigh. From the difference in mass of crucible, calculate the mass of lead and express it as percentage of the material taken for test.

16. FLASH POINT (ABEL)

16.0 Outline of the Method — The sample suitably cooled, is placed in the cup of the Abel apparatus and heated at a prescribed rate. A small test flame is directed into the cup at regular intervals and the flash point is taken as the lowest temperature at which application of the test flame causes the vapour above the sample to ignite with a distinct flash inside the cup.

16.1 Procedure — The determination is carried out as prescribed in **24** of IS : 101-1964*.

17. KEEPING QUALITY

17.1 When stored under cover in a dry place in the original sealed containers under normal temperature conditions, the material shall retain the properties prescribed in the material specifications for the specified period after the date of manufacture which shall be subsequent to the date of placing contract.

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*Methods of test for ready mixed paints and enamels (*second revision*).

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