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# *Indian Standard*

# SPECIFICATION FOR TOLUOLE, INDUSTRIAL

## *(First Revision)*

## 0. FOREWORD

0.1 This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 17 July 1968, after the draft finalized by the Coal Carbonization Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1955 as specification for toluole, industrial, solvent grade. While revising the standard, all changes found necessary after detailed study of the results and experience gained in the use of the standard during the last decade, have been incorporated. Particular mention should be made of the method of test for corrosive sulphur, which has replaced the earlier method based on the corrosion of copper strip. Also, the sampling plan and the conformity criteria have been revised. Opportunity has also been taken to modify all the methods on the basis of the latest versions of the earlier methods.

0.3 The toluole, industrial covered under this standard is derived by suitable fractionation and refining of crude benzole, obtained by extraction from the gas produced by carbonization of coal in coke ovens and retorts or recovered as a by-product in petroleum refining or petrochemical operations.

0.3.1 The material is generally used as an industrial solvent for gums, resins, oils and cellulose esters. It is also used in the manufacture of paints, varnishes, lacquers, benzoic acid, medicines, dyes, perfumes, explosives, and fine chemicals.

0.4 In the preparation of this standard, specifications of the National Benzole and Allied Products Association ( NBA ) 1960, Ed 4 ( England ); Standard methods of testing tar and its products, Ed 5 ( 1962 ) published by the Standardization of Tar Products Tests Committee, UK, and documents of the International Organization for Standardization ( ISO ) Technical Committee ISO/TC 78 Aromatic Hydrocarbons, have been drawn upon freely and the assistance so derived is acknowledged.

0.5 This standard is one of a series of Indian Standards on coal carbonization products.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for industrial toluole.

## 2. TERMINOLOGY

2.0 For the purpose of this standard, the definitions given in IS : 1303-1963† and in IS : 82-1950‡ along with the following definitions shall apply.

**2.1 Repeatability** — A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

**2.2 Reproducibility** — A quantitative measure of variability associated with operators working in different laboratories, each obtaining single results on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

## 3. REQUIREMENTS

**3.1 Description** — The material shall be clear and free from sediment, suspended matter and undissolved water.

**3.2 Neutrality** — The material shall not give an acid reaction when tested with mixed indicator as prescribed in Appendix A.

**3.3 Acid Wash Test** — When tested as prescribed under 10 of IS : 82-1950‡, the acid layer shall be clear and not darker than a solution of 1·5 g of potassium dichromate in 1 000 ml of the dilute sulphuric acid (1 : 1 by volume).

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

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

‡Methods of test for thinners and solvents for paints. (Since revised).

NOTE — Equivalent glass colour standards may be used but in that case these shall be frequently checked with the standard colour solutions prescribed.

**3.4 Corrosive Sulphur** — When tested as prescribed in Appendix B, the material shall not produce a stain darker than that produced by a standard sample containing 12 mg/l (13·7 mg/kg) of elemental sulphur in sulphur-free benzene.

3.5 The material shall also comply with the requirements given in Table 1.

### TABLE 1 REQUIREMENTS FOR TOLUOLE, INDUSTRIAL

| SL No. | CHARACTERISTIC | REQUIREMENT | METHOD OF TEST (REF TO CL No. IN) Appendix | Various Indian Standard |
|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) |
| i) | Colour* | Not darker than a freshly prepared solution of 0·8 ml of 0·1 N potassium dichromate and 12 ml of 0·1 N cobalt sulphate made up to 1 000 ml with water | — | 5 of IS : 82-1950† |
| ii) | Specific gravity at:<br>a) 15·5°/15·5°C<br>b) 27°/27°C | <br>0·860 to 0·875<br>0·849 to 0·864 | — | 6.3.3 of IS : 82-1950† |
| iii) | Distillation range:<br>a) Up to 105°C‡<br>b) Up to 120°C‡ | <br>5 ml *Max*<br>90 ml, *Min* | — | 7 of IS : 82-1950† |
| iv) | Residue on evaporation mg/100 ml, *Max* | 10·0 | — | 8.2 of IS : 82-1950† |
| v) | Total sulphur, percent by weight, *Max* | 0·2 | C | — |
| vi) | Hydrogen sulphide | To pass test | D | — |
| vii) | Mercaptans | Shall give no positive reaction | — | IS : 1448 [P:19]-1960§ |

*Equivalent glass colour standards may be used but in that case these shall be frequently checked with the standard colour solutions prescribed.

†Methods of test for thinners and solvents for paints. (Since revised).

‡The temperature being corrected for a pressure of 760 mm Hg.

§Methods of test for petroleum and its products: Doctor test.

## 4. PACKING AND MARKING

### 4.1 Packing

4.1.1 The material shall be packed as agreed to between the purchaser and the supplier, in suitable metal containers or in tank wagons.

4.1.2 All containers in which the material is packed shall be dry, clean, free from substances soluble in toluole and leak proof.

4.1.3 The containers shall be securely closed, protected from light, and stored in a cool place.

4.1.4 All containers for storage and transport of the material, since classified as flammable and dangerous goods, shall, in addition, comply with the requirements of the latest issue of Red Tariff and the requirements as laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids, and the Board of Trade Regulations as applicable thereon for transport by steamers.

### 4.2 Marking

4.2.1 Each container shall be marked with the name of the manufacturer; lot or batch number; weight of the material in the container; recognized trade-mark, if any; and the year of manufacture.

4.2.2 The containers shall have the caution label 'HIGHLY FLAMMABLE LIQUID' in red letters together with the corresponding symbol for labelling of dangerous goods as given in Fig. 3 of IS : 1260-1958*.

4.2.3 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 5. SAMPLING

**5.1 Preparation of Test Samples** — The method of drawing representative samples of the material shall be as prescribed in Appendix E.

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*Code of symbols for labelling of dangerous goods. (Since revised and split into various parts).

## 6. TEST METHODS

**6.1** Tests shall be conducted as prescribed in 3.2, 3.3, 3.4 and in col 4 and 5 of Table 1.

**6.2 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

# APPENDIX A

## (*Clause* 3.2)

## NEUTRALITY TEST

### A-1. APPARATUS

**A-1.1 Glass Cylinders** — Stoppered, of 100 ml capacity and made of good quality glass which is inert to contents under the conditions of the test.

### A-2. REAGENTS

**A-2.1 Mixed Indicator** — Dissolve 0·04 g of bromocresol green in 10 ml of rectified spirit and 0·2 g of alizarin red-S in freshly boiled and cooled water. Mix the two solutions and dilute to 100 ml with freshly boiled and cooled water. If necessary, add to the indicator solution either 0·01 N hydrochloric acid or 0·01 N sodium hydroxide solution until 0·3 ml of the indicator solution so treated gives the neutral shade (greyish green) when added to 10 ml of freshly boiled and cooled water.

A-2.1.1 The colour change of the indicator is yellow in acidic, greyish green in neutral and bluish violet in alkaline solution.

### A-3. PROCEDURE

**A-3.1** Place 10 ml of water in a clean stoppered cylinder and add 0·3 ml of indicator solution. Add 50 ml of the material and shake the stoppered cylinder thoroughly and allow to settle. Compare the colour of the aqueous layer with the colour of the contents of a similar cylinder prepared with the indicator but without the addition of the material.

### A-4. REPORTING

**A-4.1** Report the material as acidic, neutral or alkaline as the case may be.

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*Specification for water, distilled quality (*revised*).

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# APPENDIX B

( *Clause* 3.4 )

## DETERMINATION OF CORROSIVE SULPHUR

### B-0. GENERAL

**B-0.1 Outline of the Method** — The sample is freed from peroxides by the addition of quinol ( hydroquinone ). The sample thus prepared is shaken with mercury and the precipitate formed is transferred quantitatively to a filter paper to produce an even circular stain. This stain is compared with a standard stain and an indication of the elementary sulphur in the sample is thus obtained. This method is primarily intended for products containing less than 20 mg/l of elementary sulphur but the range of the test may be extended by diluting the sample with sulphur-free benzene.

### B-1. APPARATUS

**B-1.1 Test Tube** — 150 mm long, 20 mm outside diameter, fitted with a ground-glass stopper.

**B-1.2 Filter** — comprising a filter holder (*see* Fig. 1) made of a material not attacked by the liquid under test. Suitable materials are stainless steel and superpolyamide plastics material like nylon and fitted to a Buchner flask by means of an adaptor with ground socket ( *see* Fig. 2 ). Strips of close-textured filter paper, approximately 24 mm wide, are fitted through the slot of the holder.

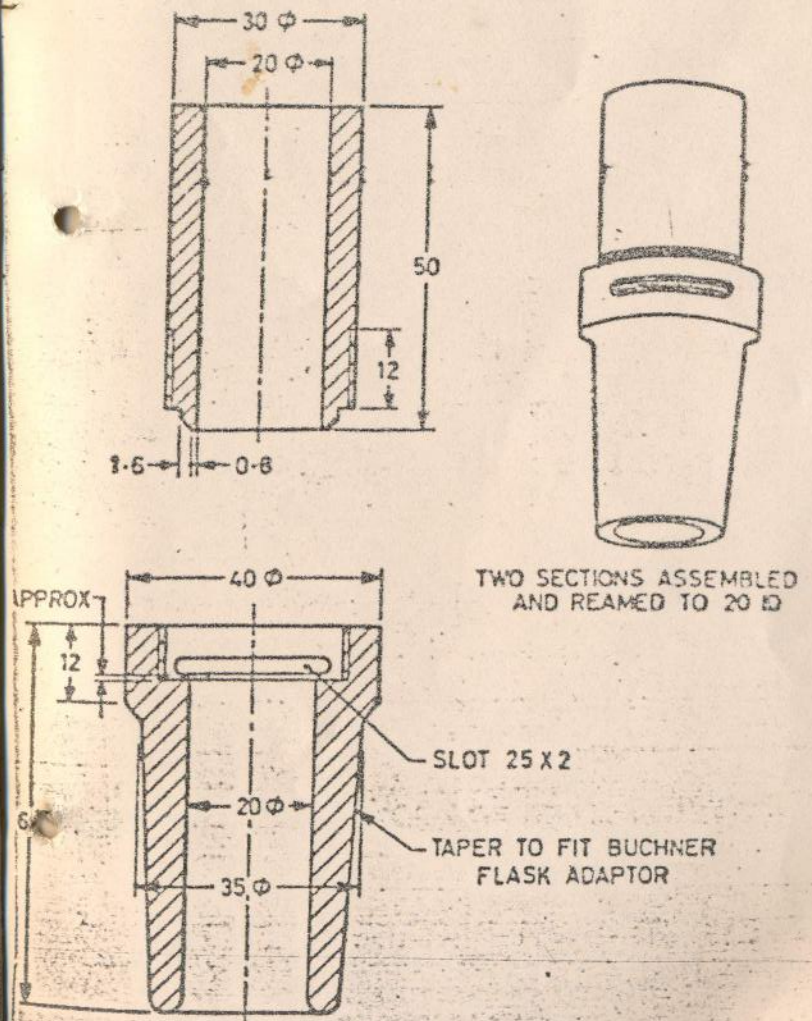
### B-2. REAGENTS

**B-2.1 Lead Acetate Reagent Solution** — Mix 300 g of lead acetate with 100 g of lead oxide ( litharge ), add 50 ml of water and heat on a water-bath until the colour of the mixture becomes uniformly white or pink. Add 950 ml of water slowly. Allow the suspension formed to settle and filter the supernatant layer.

**B-2.2 Hydroquinone** — 10 percent ( *w/v* ) solution in anhydrous methanol. The solution should be stored in an amber-glass bottle and should be not more than 14 days old.

**B-2.3 Mercury** — free from moisture.

**B-2.4 Benzene** — free from elementary sulphur and visible water. Benzene may be freed from elementary sulphur by shaking it for 10 minutes with approximately 1 percent by volume of mercury. Decant the benzene from the mercury and filter, using a hardened close-textured filter paper. Shake the filtered benzene with fresh



All dimensions in millimetres.

Fig. 1 Filter Holder

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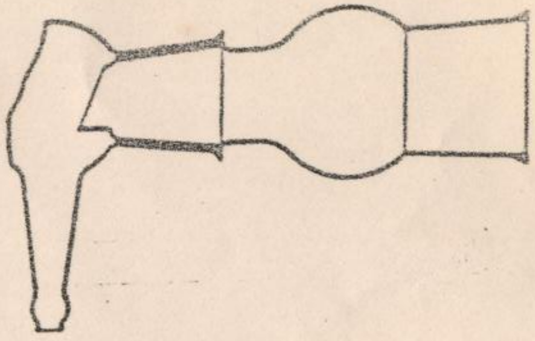

Fig. 2 Adaptor

mercury and repeat the procedure until the elementary sulphur reduced to the required level. The mercury that has been used in th test, or that used for treating the benzene, may be rendered suitable f re-use by filtration and cleaning with nitric acid by one of the standa procedures.

**B-2.5 Standard Elementary Sulphur Solution** — containing 100 mg/l pure crystalline sulphur in benzene that has been prepared by shakin with mercury as described under B-2.4 above. Each millilitre of th solution diluted to 100 ml with the benzene is equivalent to 1 mg/l elementary sulphur in the dilute solution. Prepare the crystallin sulphur by recrystallizing pure sulphur twice from benzene and dryin it in an oven at 100°C.

Note — To ensure that the reagents are satisfactory, the procedure des is carried out using the benzene in place of the sample. The stain prod the filter paper should be negligible.

## B-3. PROCEDURE

**B-3.0** If hydrogen sulphide is present and it is required to estima elementary sulphur in its absence, the hydrogen sulphide may b removed by shaking the sample for 2 minutes in a separator with a equal volume of lead acetate solution. After allowing the mixture t settle, run the reagent off and wash the sample twice with wate Decant the sample through a filter paper to free it from water.

**B-3.1** Pipette 1·0 ml of the hydroquinone solution into a 25 ml volum tric flask and dilute to 25 ml with the sample under test. Shake unt the contents of the flask are well mixed.

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**B-3.1.1** Shake 5·0 ml of this mixture for one minute with 2 ml of the mercury in the glass-stoppered test tube. The shaking should be vigorous at about 180 shakes per minute. Decant the sample quickly through the filter, being taken to prevent the mercury passing into the filter (*see* Fig. 3). The sample should be poured directly on to the filter paper to ensure an even stain.

Note — The suction applied to the filter should be between 20 mm and 30 mm of mercury below atmospheric pressure. The filter should be covered during application of the suction.

**B-3.1.2** Wash the mercury by shaking it with 10 ml of benzene and decant this benzene through the filter in the same way. Repeat the washing with another 10 ml of the benzene. Dry the filter paper by drawing filtered air through it for 2 minutes.

**B-3.1.3** At the same time as, or immediately before the determination, prepare a standard stain equivalent to the specified maximum concentration of corrosive sulphur, using the appropriate quantity of standard corrosive sulphur solution in place of the sample.

Cut the stain produced and the standard stain in half and compare visually by placing the halves together on a white background.

Note — For convenience in routine work, the test may be carried out using printed stains which have been standardized against the standard elementary sulphur solution, but in case of dispute the stain obtained in the test is to be compared directly with the stain produced by the appropriate quantity of standard elementary sulphur solution.

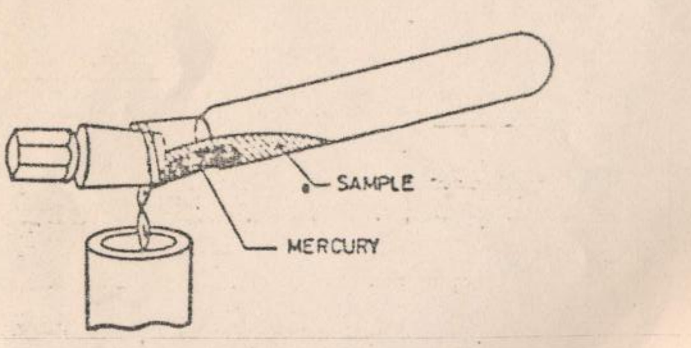


Fig. 3 Filtration

**-3.2 Reporting of Results** — Report the test stain as darker, or not arker than the appropriate standard stain. If the sample has been iluted, report the procedure used.

## APPENDIX C

[ *Table* 1, *Item* ( v ) ]

## DETERMINATION OF TOTAL SULPHUR

### C-1. OUTLINE OF THE METHOD

**C-1.1** The material is diluted with methanol or rectified spirit and known quantity of the diluted solution is burnt in a lamp. The products of combustion on being drawn through neutral hydrogen peroxide solution produce acidity which is determined by titration with sodium carbonate solution.

### C-2. APPARATUS

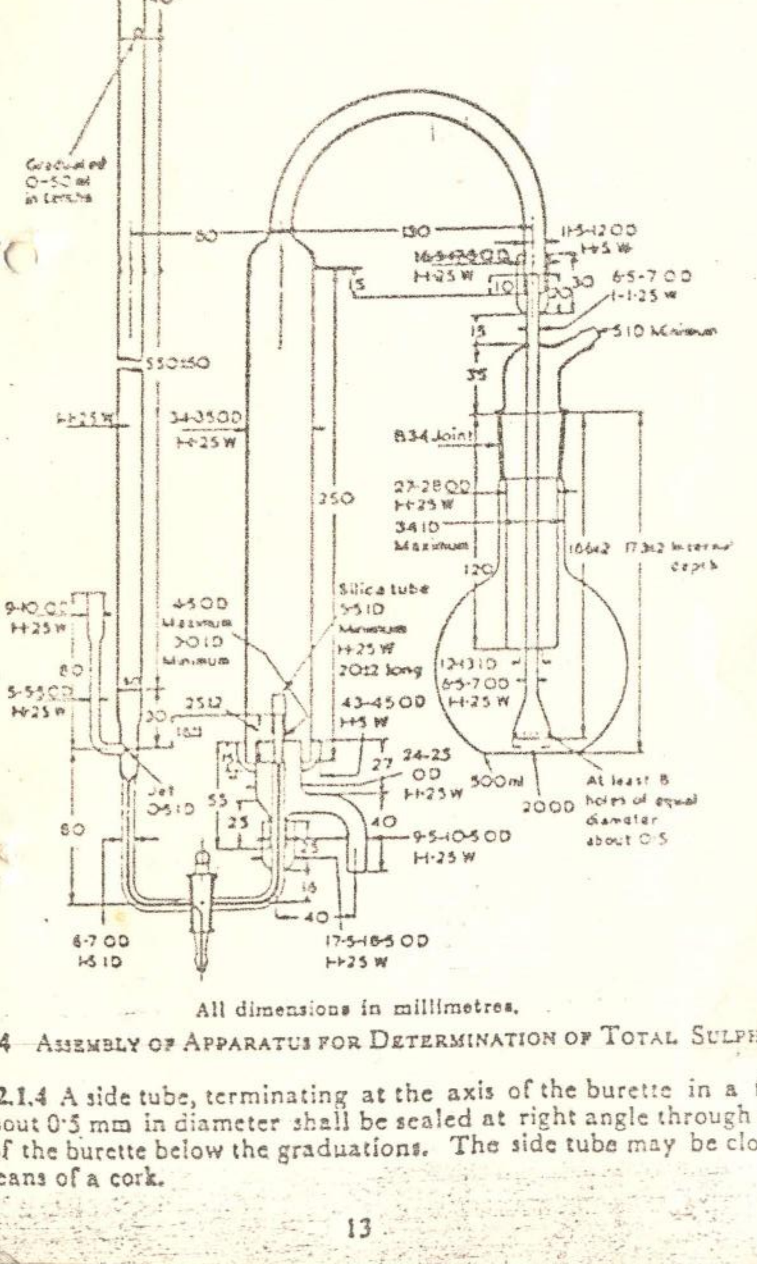
**C-2.0** The apparatus described below with the shape, dimensions and assembly shown in Fig. 4, 5 and 6 shall be used.

**C-2.1** Lamp

**C-2.1.1** The burner shall be a piece of glass tubing loosely fitted with a wick consisting of a piece of recently ignited asbestos string, 20 to 25 mm in length and 2·5 to 3 mm in diameter. The wick shall project about one millimetre above the top of the glass tubing. Round the burner shall be fitted a short sleeve of silica tubing, which shall rest on a copper spiral, the size of the flame being controlled by the height of the sleeve. The burner shall be attached by means of a capillary tube and a three-way tap to a 50 ml burette, reading to 0·1 ml. A small cup containing mercury for sealing the chimney mounting (*see* C-2.2) shall be sealed to the capillary tube below the burner. The three-way tap shall be situated at the lowest point of the capillary tubing and shall have an outlet for draining the burette. The tap shall be reasonably free from leakage.

**C-2.1.2** To test the three-way tap for leakage, free the key from all traces of grease, dip it in water and replace in the barrel. Close both the side tubes and the burner by any suitable means. Fill the burner, the burette and the outflow from the tap with water. Do not insert a cork in the top of the burette. Adjust the water level in the burette at 700 mm above the lowest portion of the outflow of the tap, which, on turning, connects the burette and the burner

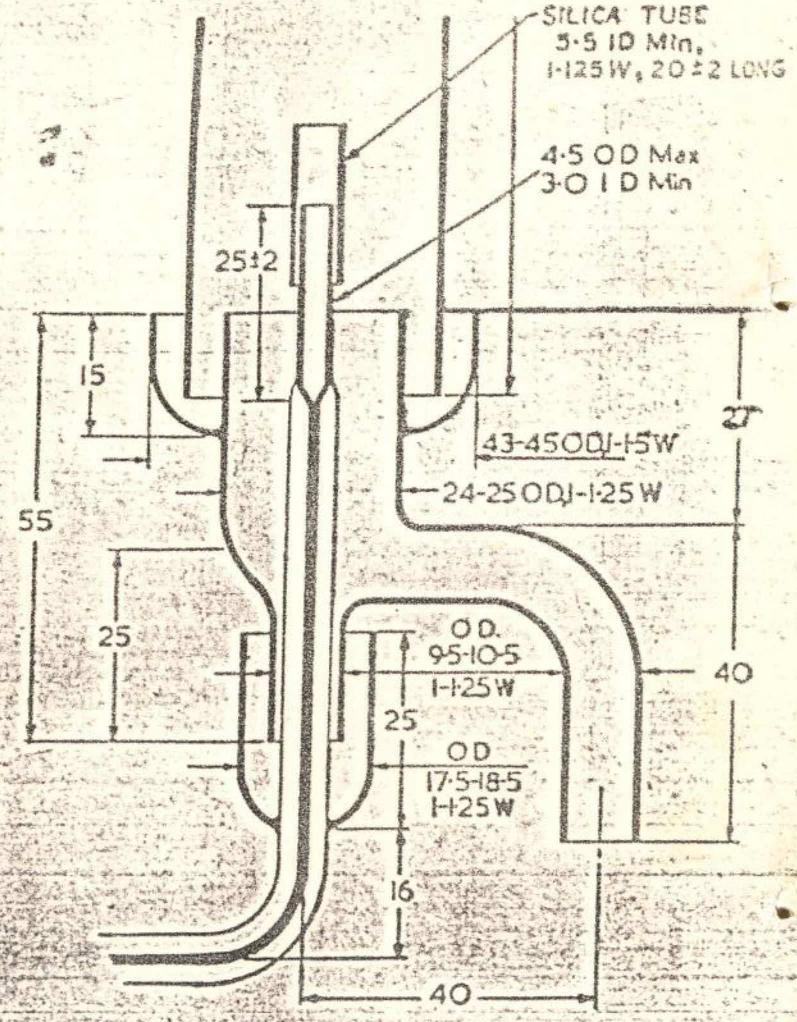
**C-2.1.3** The tap shall be taken to be leak-free if, after standing for three hours, the fall in level of the water in the burette does not exceed 0·7 ml.



All dimensions in millimetres.

Fig. 4 Assembly of Apparatus for Determination of Total Sulphur

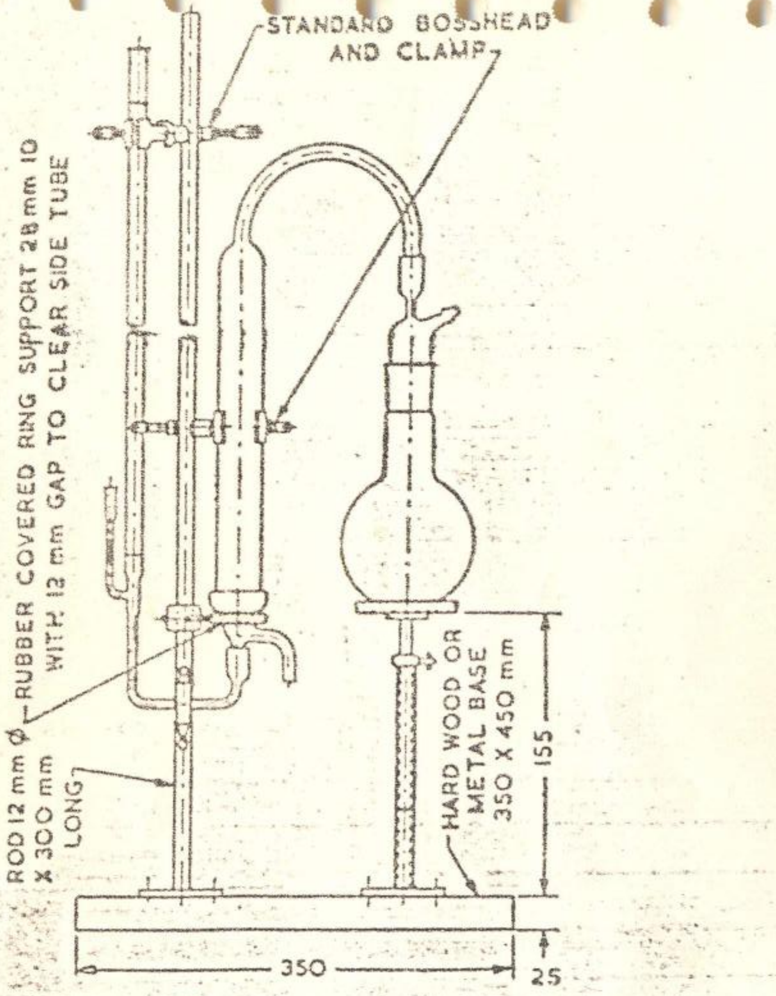
**C-2.1.4** A side tube, terminating at the axis of the burette in a fine at about 0·5 mm in diameter shall be sealed at right angle through the ide of the burette below the graduations. The side tube may be closed y means of a cork.



All dimensions in millimetres.

FIG. 5 DETAILS OF CHIMNEY MOUNTING

C-2.1.5 The burner and the burette shall both be vertical when in operation and the top of the jet shall be 16±1 mm below the top of the burner. It is important, when constructing the lamp, to ensure that this dimension is within the limits indicated.



All dimensions in millimetres.

FIG. 6 TOTAL SULPHUR IN REFINED LOWER BOILING PRODUCTS—SUPPORT

C-2.1.6 The principle of operation of the lamp is that when the top of the burette is closed with a cork during use, air can then only enter through the jet as liquid from the burette burns. This point is thus maintained at atmospheric pressure and hence the level of the liquid in the burner remains constant.