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IS 537
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BUREAU OF INDIAN STANDARDS

Draft Indian Standard

TOLUENE — SPECIFICATION

(*Third Revision* of IS 537)

(ICS 75.080.150)

Petroleum and their related products of synthetic or biological origin Sectional Committee, PCD 03

Last date for receipt of comment is **31 July 2022**

FOREWORD

(*Formal clauses will be added later*)

Toluene is used as raw material in manufacture of alkylated, nitrated and halogenated organic intermediates for petrochemical dyestuffs and fine chemical industries. It is also used as solvent in paint, coating, adhesive and ink formulations.

Toluene is derived by suitable fractionation and refining by washing with acid or hydro refining of crude benzoyl recovered from the gas produced during carbonization of coal in coke ovens and retorts or recovered as by-products in petroleum refining or petrochemical operations.

This standard was originally published in 1955 and was based on overseas national standards including the work accomplished till then by the Technical Committee, ISO/TC 78 'Aromatic Hydrocarbons, of International Organization for Standardization (ISO)'.

First revision of the standard was published in 1967. In first revision, toluene required with specially low content of non-sulphonable hydrocarbons was covered. Besides, the requirement of distillation range was tightened in order to take care of the possibility of admixture with benzene. Colorimetric tests were also prescribed for acid wash test and colour.

Second revision of the standards was published in 2011. In second revision, the requirements of residue on evaporation and corrosive sulphur were deleted, and Gas Chromatographic (GC) method was incorporated for determination of purity. Methods of test for distillation range and relative density were modified.

Earlier, toluene was essentially a coal base product which was being made available as by-product from coke ovens of steel plants. The requirements and methods of test were also stipulated on the basis of the publication by National Benzoyl and Allied Products Association (NBA) and the Standardization of Tar Products Tests Committee (STPC), U.K. in order to suit the prevailing quality of the product. However, during second revision, the Committee took cognizance of the fact that consequent upon exploration of oil fields especially the Bombay High, substantial quantities of crude was being made available which has completely changed the scenario. There was a distinct shift in the production of various aromatic hydrocarbons from the coal base to petroleum base, as a result of which

toluene was available in abundance which was more suitable for various purposes as compared to what was being made available from coal base.

Committee also observed this standards also covers the requirements of reagent grade toluene. Therefore, IS 1839 : 1961 'Toluene, reagent grade' was withdrawn.

In this third revision, new methods involving sophisticated instruments have been added as the refiners upgrade their testing methods in conformance to international standards. Significance of tests have been added. Thiophene and H_2S/SO_2 have been deleted from various international specifications of toluene, therefore, these two requirements have also been deleted in this standard. The purity, Pt-Co colour and non-aromatics specifications have been changed in line with international specifications with addition of acid wash colour. Vapor generation is maximum during distillation test and inhalation of benzene/ toluene vapors is all the more to be avoided. As in IS 534 : 2021 'Benzene — Specification (*fifth revision*)' toluene purity is part of the standard, hence distillation is kept as an optional test.

In this revision, considerable assistance has been drawn from the following standards published by International Organization for Standardization (ISO) and American Society for Testing and Materials (ASTM):

ASTM D841-19	Standard Specification for Nitration Grade Toluene
ISO 4626 : 1980	Volatile organic liquids – Determination of boiling range of organic solvents used as raw materials
ASTM D3505-18	Standard Test Method for Density or Relative Density of Pure Liquid Chemicals
ASTM D850-18	Standard Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Material
UOP 357	Trace Sulfur in Petroleum Distillates by the Nickel Reduction Method
ASTM D4052-18a	Density, relative density and API Gravity of liquids by digital density meter

The following alternate methods are available for the characteristic mentioned in Table 1, but in case of dispute the methods mentioned in Table 1 shall be the referee method.

Characteristic	*Methods of Test*
Colour, Pt-Co scale	ISO 2211[1)], ASTM D1209
Relative density, 15.56/15.56°C or Density 20°C	ISO 12185
Sulphur, mg/kg	ASTM D5453, ISO 13032, ISO 20846
Distillation	ASTM D850, ISO 918
Toluene, percent by mass	ASTM D7504
Non-aromatic hydrocarbons, percent by mass	
Benzene content, mg/kg	
Water content, mg/kg	ASTM D6304
Acid wash colour	ASTM D848

[1)]Automatic tintometer/spectrophotometer may also be used with compliance to ISO 2211.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in the standard.

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for toluene.

2 REFERENCES

The following standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
534 : 2021	Benzene — Specification (*fifth revision*)
1070 : 1992	Reagent grade water (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labeling of goods Part 1 Dangerous goods (*first revision*)
1446 : 2002	Classification of dangerous goods (*second revision*)
1448	Methods of test for petroleum and its products
(Part 160) : 2017/ISO 20846 : 2011	Determination of sulphur content of automotive fuels — Ultraviolet fluorescence method
(Part 161) : 2017/ISO 13032 : 2012	Determination of low concentration of sulfur in automotive fuels — Energy dispersive X-ray fluorescence spectrometric method
(Part 167) : 2018/ISO 12185 : 1996	Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method
(Part 175) : 2020/ISO 6296 : 2000	Petroleum products — Determination of water — Potentiometric Karl-Fischer titration method
IS 1448 (Part 182) : 2020/ISO 12937 : 2000	Petroleum products — Determination of water — Coulometric Karl Fischer titration method
4644 : 1968	Code of safety for benzene, toluene and xylene
4905 : 2015	Random sampling and randomization procedure (*first revision*)
5165 : 2017/ISO 383 : 1976	Laboratory glassware — Interchangeable conical ground joints (*first revision*)
8768 : 2000	Methods of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

3 REQUIREMENTS

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

4 PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed as agreed to between the purchaser and the supplier.

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

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

4.1.4 The 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 therein for transport by steamers.

Table 1 Requirements for Toluene
(*Clauses* 3, 7.1, E-5.1.2, E-5.2 and E-6.1.1)

Sl No.	**Characteristic**	**Requirement**	**Methods of Test, Ref to Annex/IS/ Parts of IS 1448**
(1)	(2)	(3)	(4)
(i)	Toluene, percent by mass, *Min*	99.8	D
(ii)	Appearance	Clear liquid free of sediment and haze when observed at 18.3 to 25.6°C	-
(iii)	Colour, platinum-cobalt scale, *Max*	20	IS 8768
(iv)	Relative density at 15.56/15.56°C or Density, 20°C, g/ml	0.869-0.873 0.865-0.870	A/(Part 167)[1)]
(v)	Total sulphur, mg/kg, *Max*	[2)]	B/(Part 160)[1)]/(Part 161)
(vi)	Distillation Range (1 to 96 percent volume) including the temperature 110.6°C at 101.3 kPa (760 mm of Hg pressure)[3)], °C, *Max*	0.6	C
(vii)	Non-aromatic hydrocarbons , percent by mass, *Max*	0.15	D
(viii)	Benzene content, mg/kg, *Max*	500	D
(ix)	Water content, mg/kg, *Max*	500	(Part 175)/(Part 182)[1)]
(x)	Acid Wash Colour	Passes with 2	Annex J of IS 534

[1)]In case of disputes, this method shall be the referee method.
[2)]As agreed between the purchaser and the supplier.
[3)]If purity by GC method is carried out [Sl No. (i)], distillation test is optional.

4.1.5 Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids (*see* IS 1446) shall be provided and all due precautions shall be taken [*see* IS 1260 (Part 1)] at all times to prevent accidents by fire or explosion.

4.1.6 Except when they are opened for the purpose of cleaning and rendering them free from toluene vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from toluene vapour.

4.2 Marking

4.2.1 Each container shall be marked with the following information:

a) Indication of the source of manufacture,
b) Net mass of the material in the container,
c) Batch number or code number, and
d) Date of manufacture.

4.2.2 Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods as given in IS 1260 (Part1).

4.2.3 *BIS Certification Marking*

The containers may also be marked with the BIS Standard Mark.

4.2.3.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 HANDLING

Toluene is toxic and therefore it shall be handled carefully. Exposure of toluene in atmosphere should be monitored regularly. Persons exposed to toluene shall be periodically checked according to factory rules and local state regulations (*see* IS 4644).

6 SAMPLING

Representative samples of the material shall be prepared as prescribed in Annex E.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in col 4 of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

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

ANNEX A
[*Table* 1, *Sl No.* (iv)]
DETERMINATION OF DENSITY OR RELATIVE DENSITY

A-1 GENERAL

Two methods, pyknometer method and digital density meter method, have been specified. Pyknometer method shall be taken as referee method.

A-2 SIGNIFICANCE

Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and petroleum products. It is used for calculating the mass when volume is known and for the conversion of measured volumes to volumes at the standard temperature of 15°C. It is also used to identify the stream/product to some extent. Though it gives a very uncertain indication of the fuel quality, density can indicate gross contamination increase if any. This property can also be used as an indication whether the product has been contaminated during the movement.

A-3 PYKNOMETER METHOD

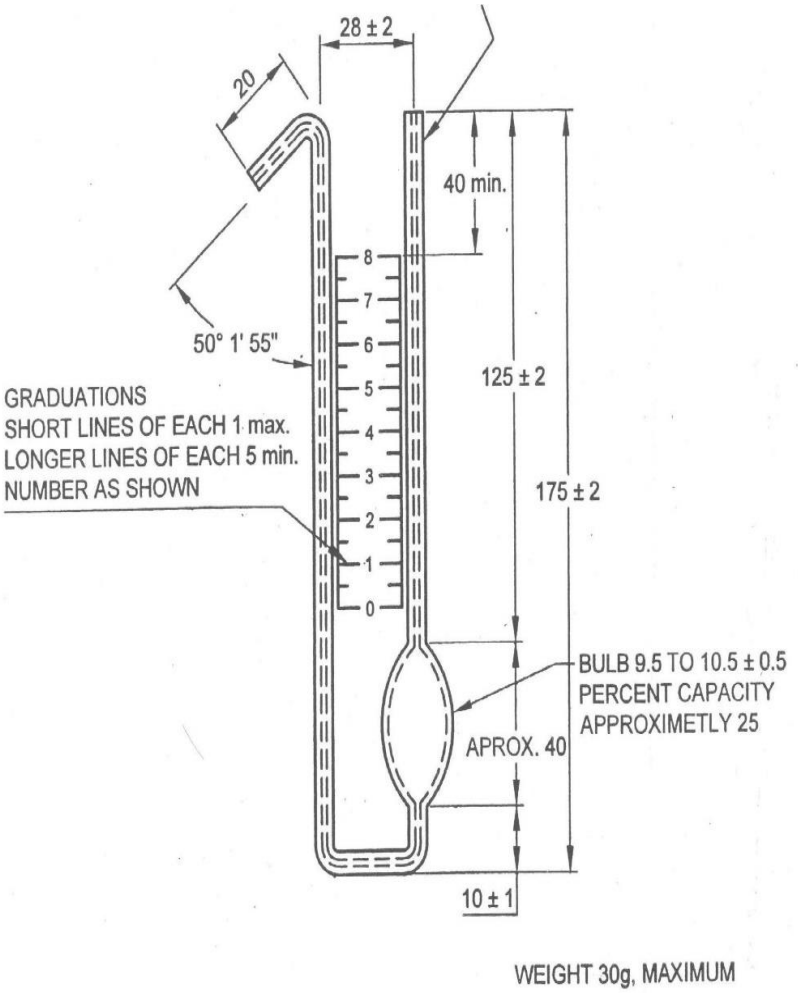
A-3.1 Outline of the Pyknometer Method

For materials listed in Table 2, the sample is drawn into a weighed and calibrated bicapillary pyknometer. The filler pyknometer is allowed to come to equilibrium at any convenient temperature between 10 and 30°C. The equilibrium temperature is measured to the nearest 0.02°C. The weight is determined by using a beam balance. The density, relative density, or commercial density at the desired reference temperature is then calculated from the sample weight, a calibration factor proportional to an equal volume of water, and a multiplier which corrects for the buoyancy of air and the change in volume of the pyknometer and the sample due to deviation from the chosen reference temperature.

A-3.2 Apparatus

A-3.2.1 *Pyknometer*

9 to 10 ml capacity, conforming to the dimensions given in Fig. 1, constructed of borosilicate glass, and having a total weight not exceeding 30 g.



All dimensions in millimeters.

FIG. 1 PYKNOMETER

Table 2 20°C Reference Temperature Multiplier, F_{20}, for Use in Computing Density

(*Clause* A-3.1 and A-3.6)

Temperature, °C	**Benzene**	**Toluene**	**Mixed Xylenes**	**o-xylene**	**m-xylene**	**p-xylene**
(1)	(2)	(3)	(4)	(5)	(6)	(7)
10	0.98822	0.989 41	0.990 28	0.99052	0.99028	0.99011
10.2	0.98845	0.989 62	0.99047	0.9907	0.99047	0.9903
10.4	0.98865	0.98983	0.99066	0.99089	0.99066	0.99049
10.6	0.93891	0.99003	0.99085	0.991 07	0.99085	0.99069
10.8	0.989 14	0.99024	0.99104	0.991 26	0.99104	0.99066
11	0.98937	0.9904	0.991 23	0.99144	0.991 23	0.991 07
11.2	0.9896	0.99066	0.991 42	0.991 63	0.99142	0.99126
11.4	0.98962	0.99036	0.99161	0.991 81	0.991 61	0.991 46
11.6	0.99005	0.991 07	0.991 79	0.992	0.991 79	0.99165
11.8	0.99028	0.991 28	0.991 98	0.99218	0.99198	0.991 84
12	0.99051	0.991 48	0.992 17	0.992 37	0.992 17	0.99204
12.2	0.99074	0.991 69	0.992 36	0.99255	0.99236	0.99223
12.4	99097	0.9919	0.99255	0.99274	0.99253	0.99242

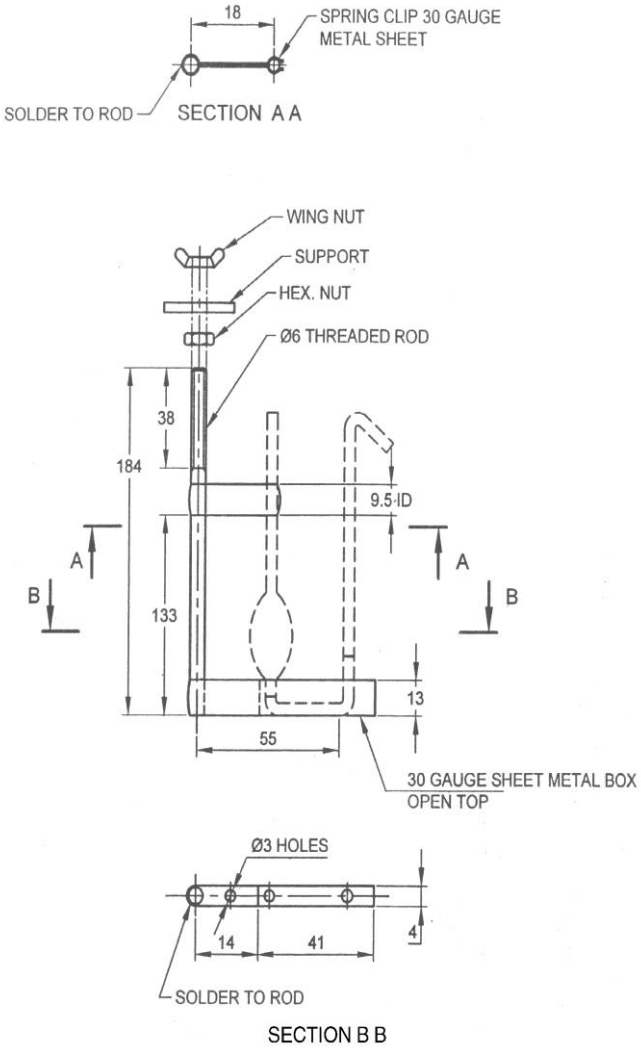
12.6	0.991 20	0.992 11	0.99274	0.99292	0.99274	0.99262
12.8	0.991 44	0.99231	0.99293	0.993 11	0.992 93	0.992 81
13	0.991 67	0.99252	0.993 12	0.99329	0.993 12	0.993 00
13.2	0.991 90	0.99273	0.993 31	0.99348	0.99331	0.993 20
13.4	0.99213	0.992 94	0.9935	0.993 67	0.9935	0.993 39
13.6	0.99236	0.993 15	0.99369	0.993 85	0.99369	0.99358
13.8	0.99259	0.993 35	0.993 89	0.99404	0.99389	0.993 78
14	0.99282	0.993 56	0.994 08	0.99422	0.99408	0.99397
14.2	0.99305	0.993 77	0.99427	0.99441	0.99427	0.994 17
14.6	0.99353	0.994 19	0.99464	0.99478	0.99465	0.994 56
14.8	0.993 75	0.9944	0.994 84	0.99497	0.99484	0.99475
15	0.99398	0.99441	0.99503	0.99516	0.99503	0.99495
15.2	0.994 21	0.99481	0.995 22	0.99534	0.99522	0.995 14
15.4	0.99445	0.995 02	.0.99541	0.99553	0.995 41	0.99534
15.6	0.99468	0.995 23	0.99561	0.99572	0.99561	0.995 53
15.8	0.99491	0.99544	0.9958	0.9959	0.995 80	0.99573
16	0.99515	0.99565	0.99599	0.99609	0.99599	0.99592
16.2	0.995 38	0.99586	0.99618	0.99628	0.99618	0.996 12
16.4	0.99561	0.99607	0.99637	0.99646	0.99637	0.99631
16.6	0.995 85	0.99628	0.99657	0.99665	0.99657	0.99651
16.8	0.99608	0.99649	0.99676	0.99684	0.99676	0.996 70
17	0.99632	0.99670	0.99695	0.99703	0.99695	0.9969
17.2	0.99655	0.99691	0.99714	0.99721	099714	0.997 10
17.4	0.996 79	0.99712	0.99734	0.9974	0.99734	0.99729
17.6	0.99702	0.99733	0.99753	0.99759	0.99753	0.99749
17.8	0.99726	0.99754	0.99772	0.99778	0.99772	0.99768
18	0.997 49	0.99775	0.99791	0.99797	0.99791	0.997 88
18.2	0.99773	0.99796	0.99811	0.99815	0.99811	0.99808
18.4	0.99796	0.99817	0.998 30	0.99834	0.998 30	0.99327
18.6	0.9982	0 99838	0.99849	0.998 53	0.99849	0.998 47
18.8	0.99843	0.99859	0.99869	0.99872	0.99869	0 998 67
19	0.99867	0.99880	0.99888	0.998 91	0.99888	0.998 86
19.2	0.9989	0.99901	0.999 07	0.999 10	.0.99907	0.99906
19.4	0.99914	0.99922	0.99927	0.99928	0.99927	0.99926
19.6	0.999 38	0.99943	0.999 46	0.99947	0.99944	0.999 46
19.8	0.99961	0.999 64	0.99966	0.99966	0.99966	0.99965
20	0.99985	0.99985	0.999 85	0.999 85	0.99985	0.99985
20.2	1.00009	1.00006	1.00004	1.00004	1.00004	1.00005
20.4	1.00032	1.00027	1.00024	1.00023	1.000 24	1.000 25
20.6	1.00056	1.00048	1.00043	1.000 42	1.00043	1.00044
20.8	1.0008	1.00069	1.00063	1.00061	1.00003	1.000 64
21	1.001 04	1.000 91	1.00082	1.0008	1.000 82	1.000 84
21.2	1.001 28	1.00112	1.001 02	1.00099	1.00102	1.001 04
21.4	1.001 51	1.001 33	1.001 21	1.001 18	1.001 21	1.001 24
21.6	1.001 75	1.001 54	1.00141	1.001 37	1.001 41	1.00143
21.8	1.00199	1.001 75	1.001 60	1.00156	1.0016	1.00163
22	1.002 23	1.001 96	1.0018	1.00175	1.001 80	1.001 63

22.2	1.00247	1.00218	1.00199	1.001 94	1.001 99	1.00203
22.4	1.00271	1.002 39	1.00219	1.002 13	1.00219	1.00223
22.6	1.00295	1.002 60	1.00238	1.00232	1.00238	1.002 43
22.8	1.003 19	1.00281	1.00258	1.06251	1.00253	1.00263
23	1.00342	1.00302	1.002711	1.002 70	1.00278	1.002 33
23.2	1.00366	1.00324	1.00297	1.00289	1.00297	1.003 03
23.4	1.0039	1.00345	1.00317	1.00308	1.003 17	1.00322
23.6	1.004 14	1.00366	1.00336	1.00327	1.00336	1.00342
23.8	1.00438	1.00387	1.00356	1.00346	1.00356	1.003 62
24	1.00462	1.00409	1.00376	1.00365	1.00376	1.00382
24.2	1.00487	1.0043	1.00395	1.003 84	1.00395	1.00402
24.4	1.00511	1.00451	1.00415	1.00403	1.00415	1.00472
24.6	1.00535	1.00413	1.00435	1.00422	1.00435	1.00442
24.8	1.00559	1.00494	1.00454	1.00442	1.00454	1.00462
25	1.005 83	1.005 15	1.00474	1.00461	1.00474	1.00482
25.2	1.00507	1.005 37	1.00494	1.0048	1.00494	1.00502
25.4	.1.00631	1.00558	1.00514	1.00499	1.00514	1.00522
25.6	1.00656	1.00579	1.00533	1.00518	1.00533	1.00542
25.8	1.0068	1.00601	1.00553	1.99537	1.00553	1.00563
26	1.007 04	1.00622	1.00573	1.005 57	1.00513	1.005 83
26.2	1.007 28	1.00643	1.00593	1.00576	1.00593	1.00603
26.4	1.007 53	1.00665	1.00612	1.00595	1.00612	1.00623
26.6	1.007 77	1.00686	1.00632	1.006 14	1.00632	1.00643
26.8	1.00801	1.00707	1.006 52	1.00634	1.00652	1.00663
27	1.00825	1.00729	1.00672	1.00653	1.00672	1.00683
27.2	1.0085	1.007 50	1.00692	1.00672	1.00692	1.007 03
27.4	1.00874	1.00772	1.00711	1.00691	1.00711	1.00724
27.6	1.00899	1.00793	1.00731	1.007 11	1.007 31	1.00744
27.8	1.00923	1.008 15	1.00751	1.0073	1.007 51	1.00764
28	1.00947	1.00836	1.00771	1.007 49	1.00771	1.00784
28.2	1.00972	1.00858	1.00791	1.00769	1.00791	1.00804
28.4	1.00996	1.00879	1.008 11	1.00788	1.008 11	1.00825
28.6	1.01021	1.00901	1.00831	1.00807	1.00831	1.00845
28.8	1.01045	1.00922	1.00851	1.00827	1.00851	1.008 65
29	1.0107	1.00944	1.00871	1.00846	1.00871	1.00885
29.2	1.01094	1.00965	1.90891	1.00866	1.00891	1.00906
29.4	1.011 19	1.00987	1.009 11	1.00885	1.00911	1.00926
29.6	1.01143	1.01008	1.00931	1.00904	1.00931	1.00946
29.8	1.011 68	1.0103	1.00951	1.00924	1.00951	1.00966
30	1.01192	1.01051	1.00971	1.00943	1.00971	1.00987

NOTE — Choose a multiplier for the material being measured corresponding to the bath Temperature at which the pyknometer is equilibrated.

A-3.2.2 *Bath*

Having a depth of at least 300 mm, capable of being maintained constant to ±0.02°C at any convenient temperature between 10°C and 30°C. Provide a support for the pyknometer (*see* Fig. 2) constructed of any suitable non-corrosive metal.



All dimensions in millimeters.
FIG. 2 PYKNOMETER HOLDER

A-3.2.3 *Bath Thermometer* — Having a range from -8 to +32°C.

A-3.3 Preparation of Apparatus

A-3.3.1 *Acid Cleaning*

When liquid fails to drain cleanly from the walls of the pyknometer or its capillary. Clean with hot chromic acid solution, thoroughly and rinse well with water. Dry at 105°C to 110°C for at least 1 h, preferably with a slow current of filtered air passing through the pyknometer.

A-3.3.2 *Solvent Cleaning*

For use between determinations. Rinse with toluene and then with anhydrous acetone, drying with a filtered stream of dry air.

A-3.4 Calibration of Apparatus

A-3.4.1 Using the procedure described in **A-3.5**, determine the weight of freshly boiled reagent water held by the pyknometer with the water level at each of three different scale points on the graduated arms. Make all weighings on the same day, using the same balance and weights.

A-3.4.2 Calculate the volume, V_T^p, at each scale point tested by means of the following equation, carry all calculations in 6 non-zero digits and round to 4 decimal places:

$$Pyknometer\ capacity, V_T^p, ml = A \times \left(\frac{W^w}{d_t^w}\right) + B \times (T - t)$$

where

A = air buoyancy coefficient, a constant for the temperature range involved = 1.001 064;
V_T^p = volume of pyknometer at reference temperature, T, in ml;
W^w = weight of water in air, contained in the pyknometer, in g;
d_t^w = density of water at t (*see* Table 3);
t = temperature, °C;
T = reference temperature, 20°C or 15.56°C; and
B = volumetric coefficient of expansion of 9.5 ml of a borosilicate glass pyknometer, 9.262 76 × 10^{-5} ml/°C.

A-3.4.3 Prepare a calibration curve by plotting apparent volume, V_A (the sum of the scale readings on the two arms of the pyknometer) against the corresponding calculated volume, V_T^p. If a straight line cannot be drawn through the three points, discard the data and determine three additional points so that a straight calibration line can be drawn, such that no data point lies more than 0.000 2 ml units from the line. If neither set of data meets the condition, the diameters of the graduated capillary arms are not sufficiently uniform, and the pyknometer shall be discarded.

A-3.4.4 From the curve obtained, prepare a table of apparent volume, V_A (sum of scale readings of both arms), as apparent volume against corresponding calculated volumes, V_T^p, in increments of 0.000 1 ml. Label Table 3 with the reference temperature to which it applies.

Table 3 Density of Water, g/ml
(*Clause* A-3.4.2 and A-3.4.4)

***t*,°C**		**0.0**	**0.1**	**0.2**	**0.3**	**0.4**	**0.5**	**0.56**	**0.6**	**0.7**	**0.8**	**0.9**
15	0.999	13	11	10	08	07	05	04	04	02	00	99
16	0.998	97	96	94	92	91	89		87	86	84	82
17		80	79	77	75	73	72		70	68	66	64
18		62	61	89	57	55	53		51	49	47	45
19		42	42	40	38	36	34		32	30	27	25
20		23	21	19	17	15	13		11	09	07	04
21		02	00	98	96	93	91		89	87	85	82
22	0.997	80	78	75	73	71	69		66	64	62	59
23		57	64	52	50	47	45		42	40	38	35

24		33	30	28	25	23	20		18	15	13	10
25		08	05	02	00	97	95		92	89	87	84
26	0.996	81	79	76	73	71	68		65	63	60	57
27		54	52	49	46	43	41		38	35	32	29
28		26	24	21	18	15	12		09	06	03	00
29	0.995	98	95	92	89	86	83		80	77	74	72
30		68	65	62	59	56	53		50	46	43	40

A-3.5 Procedure

A-3.5.1 Weigh the clean, dry pyknometer to 0.1 mg and record the weight.

A-3.5.2 With the sample at approximately the test temperature, fill the pyknometer by holding it in an upright position and placing the hooked tip in the sample; the liquid will then be drawn over the bend in the capillary by surface tension. Allow the pyknometer to fill by siphoning (about 1 min) and break the siphon when the liquid level in the bulb arm of the pyknometer reaches the lowest graduation mark.

A-3.5.3 Thoroughly dry the wet tip. Wipe the body of the pyknometer with a chemically clean, lint-free cloth slightly damp with water (*see* Note) and weigh the filled pyknometer to the nearest 0.1 mg.

NOTE—In atmospheres below 60 percent relative humidity, drying the pyknometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg or more in the weight of the pyknometer. This charge may not be completely dissipated in less than 30 min, and can be detected by touching the pyknometer to the wire hook in the balance and then drawing it away slowly. If the pyknometer exhibits an attraction for the wire hook, it may be considered to have a static charge.

A-3.5.4 Place the pyknometer in the holder in a constant temperature bath held at any convenient temperature 10°C and 30°C within ±0.02°C. When the liquid level has reached temperature equilibrium (usually in about 10 min) and while still in the water bath, read the scale to the nearest 0.2 small divisions at the liquid level in each arm.

A-3.6 Calculation

Compute the density or relative density, or both, by means of the following equations:

$$\text{Density, g/ml at 20°C} = (W^s/V_{20}{}^p) \times F_{20} + 0.001\ 21$$

$$\text{Relative density at 20/20°C} = [(W^s/V_{20}{}^p) \times F_{20} + 0.001\ 21] \times 1.000\ 96$$

where

W^s = observed weight of sample, corrected for variation of weights, in g,
$V_{20}{}^p$ = calculated volume, $V_T{}^p$, of sample at 20°C obtained from the pyknometer calibration table, in ml, and
F_{20} = constants taken from Table 2, corresponding to the test temperature.

A-4 DIGITAL DENSITY METER METHOD

A-4.1 Outline of the Digital Density Meter Method

A small volume (approximately 0.7 ml) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

A-4.2 Apparatus

A-4.2.1 *Digital Density Analyzer*

A digital analyzer consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer must accommodate the accurate measurement of the sample temperature during measurement or must control the sample temperature as described in **A-4.2.2**. The instrument shall be capable of meeting the precision requirements described in this test method.

A-4.2.2 *Circulating Constant-Temperature Bath (Optional)*

Capable of measuring the temperature of the circulating liquid constant to ± 0.05°C in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.

A-4.2.3 *Syringes*

At least 2 ml in volume with a tip or an adapter tip that will fit the opening of the oscillating tube.

A-4.2.4 *Flow-Through of Pressure Adapter*

For use as an alternative means of introducing the sample into the density analyzer either by a pump or by vacuum.

A-4.2.5 *Thermometer*

Calibrated and graduated to 0.1°C, and a thermometer holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore connections should be estimated to the nearest 0.05°C.

A-4.3 Reagents and Materials

A-4.3.1 *Water* — Redistilled freshly boiled and cooled reagent water for use as a primary calibration standard.

A-4.3.2 *Petroleum Naphtha* — For flushing viscous petroleum samples from the sample tube (extremely flammable).

A-4.3.3 *Acetone* — For flushing and drying the sample tube (extremely flammable).

A-4.3.4 *Dry Air* — For blowing the oscillator tube.

A-4.4 Preparation of Apparatus

Set up the density analyzer and constant temperature bath following the manufacturer's instructions. Adjust the bath or internal temperature control so that the desired test temperature is established and maintained in the sample compartment of the analyzer. Calibrate the instrument at the same temperature at which the density of the sample is to be measured.

NOTE — Precise setting and control of the test temperature in the sample tube is extremely important. An error of 0.1°C can result in a change in density of one in the fourth decimal.

A-4.5 Calibration of Apparatus

A-4.5.1 Calibrate the instrument when first set up and whenever the test temperature is changed. Thereafter, conduct calibration checks at weekly intervals during routine operation.

A-4.5.2 Initial calibration, or calibration after a change in test temperature, necessitates calculation of the values of the constants *A* and *B* from the periods of oscillation (*T*) observed when the sample cell contains air and redistilled, freshly boiled and cooled reagent water. Other calibrating materials such as n-nonane, n-tridecane, cyclohexane, and n-hexadecane (for high temperature applications) can also be used as appropriate.

A-4.5.2.1 While monitoring the oscillator period, *T* flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. Contaminated or humid air can affect the calibration. When these conditions exist in the laboratory, pass the air used for calibration through a suitable purification and drying train. In addition, the inlet and outlet ports for the U-tube must be plugged during measurement of the calibration air to prevent ingress of moist air.

A-4.5.2.2 Allow the dry air in the U-tube to come to thermal equilibrium with the test temperature and record the *T*-value for air.

A-4.5.2.3 Introduce a small volume (about 0.7 ml) of redistilled, freshly boiled and cooled reagent water into the sample tube from the bottom opening using a suitable syringe. The test portion must be homogeneous and free of even the smallest air or gas bubbles. The sample tube does not have to be completely full as long as the liquid meniscus is beyond the suspension point. Allow the display to reach a study reading and record the *T*-value for water.

A-4.5.2.4 Calculate the density of air at the temperature of test using the following equation:

$$D_a, \frac{g}{ml} = 0.001293 \times (\frac{273.15}{T}) \times (\frac{P}{760})$$

where

T = temperature, in K; and
P = barometric pressure; in torr.

A-4.5.2.5 Determine the density of water at the temperature of test by reference to Table 4.

A-4.5.2.6 Using the observed *T* values and the reference values for water and air, calculate the values of the constants *A* and *B* using the following equations:

$$A = \frac{(T_w^2 - T_a^2)}{(d_w - d_a)}$$

$$B = T_a^2 \times A \times d_a$$

where

T_w = observed period of oscillation for cell containing water;
T_a = observed period of oscillation for cell containing air;
d_w = density of water at test temperature; and
d_a = density of air at test temperature.

Alternatively, use the *T* and *d* values for the other reference liquid, if one is used.

A-4.5.2.7 If the instrument is equipped to calculate density from the constants *A* and *B* and the observed *T*-value from the sample, then enter the constants in the instrument memory in accordance with the manufacturer's instructions.

A-4.5.2.8 Check the calibration and adjust if needed by performing the routine calibration check described in **A-4.5.3**.

A-4.5.2.9 To calibrate the instrument to display relative density, that is, the density of the sample at a given temperature referred to the density of water at the same temperature, follow sections **A-4.5.2.1** through **A-4.5.2.7**, but substitute 1.000 for d_w in performing the calculations described in **A-4.5.2.6**.

A-4.5.3 Weekly calibration adjustments to constants *A* and *B* can be made if required, without repeating the calculation procedure.

NOTE — The need for a change in calibration is generally attributable to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition can be compensated by adjusting *A* and *B*, it is good practice to clean the tube with warm chromic acid solution whenever a major adjustment is required. Chromic acid solution is the most effective cleaning agent; however, surfactant cleaning fluids have also been used successfully. Care should be taken while handling warm chromic acid solution as it causes severe burns and it is a recognized carcinogen.

A-4.5.3.1 Flush and dry the sample tube as described in **A-4.5.2.1** and allow the display to reach a steady reading. If the display does not exhibit the correct density for air at the temperature of test, repeat the cleaning procedure or adjust the value of constant *B* commencing with the last decimal place until the correct density is displayed.

A-4.5.3.2 If adjustment to constant *B* was necessary in **A-4.5.3.1**, then continue the recalibration by introducing redistilled, freshly boiled and cooled reagent water (*see* IS 1070) into the sample tube as described in **A-4.5.2.3** and allow the display to reach a steady reading. If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature (*see* Table 4) by changing the value of constant *A*, commencing with the last decimal place. If the instrument has been calibrated to display the relative density, adjust the reading to the value 1.000 0.

NOTE — In applying this weekly calibration procedure, it can be found that more than one value each for *A* and *B*, differing in the fourth decimal place, will yield the correct density reading for the density of air and water. The

setting chosen would then be dependent upon whether it was approached from a higher or lower value. The setting selected by this method could have the effect of altering the fourth place of the reading obtained for a sample.

A-4.5.4 Some analyzer models are designed to display the measured period of oscillation only (*T*-values) and their calibration requires the determination of an instrument constant *K*, which must be used to calculate the density or relative density from the observed data.

A-4.5.4.1 Flush and dry the sample tube as described in **A-4.5.2.1** and allow the display to reach a steady reading. Record the *T*-value for air.

A-4.5.4.2 Introduce redistilled, freshly boiled and cooled reagent water into the sample tube as described in **A-4.5.2.3**, allow the display to reach a steady reading and record the *T*-value for water.

A-4.5.4.3 Using the observed *T*-values and the reference values for water and air (*see* **A-4.5.2.4** and **A-4.5.2.5**), calculate the instrument constant *K* using the following equations:

For density:

$$K_1 = (d_w - d_a)/(T_w^2 - T_a^2)$$

For relative density:

$$K_2 = (1.0000 - d_a)/(T_w^2 - T_a^2)$$

where

T_w = observed period of oscillation for cell containing water,
T_a = observed period of oscillation for cell containing air,
d_w = density of water at test temperature, and
d_a = density of air at test temperature.

A-4.6 PROCEDURE

A-4.6.1 Introduce a small amount (about 0.7 ml) of sample into the clean, dry sample tube of the instrument using a suitable syringe.

A-4.6.2 The sample can also be introduced by siphoning. Plug the external TFE-fluorocarbon capillary tube into the lower entry port of the sample tube. Immerse the other end of the capillary in the sample and apply suction to the upper entry port using a syringe or vacuum line until the sample tube is properly filled.

A-4.6.3 Turn on the illumination light and examine the sample tube carefully. Make sure that no bubbles are trapped in the tube, and that it is filled to just beyond the suspension point on the right-hand side. The sample must be homogeneous and free of even the smallest bubbles.

NOTE — If the sample is too dark in colour to determine the absence of bubbles with certainty, the density cannot be measured within the stated precision limits.

Table 4 Density of Water
(*Clause* A-4.5.2.5 and A-4.5.3.2)

Temperature, °C	Density, g/ml
(1)	(2)
0	0.999 840

3	0.999 964
4	0.999 972
5	0.999 964
10	0.999 699
15	0.999 099
15.56	0.999 012
16	0.998 943
17	0.998 774
18	0.998 595
19	0.998 404
20	0.998 203
21	0.997 991
22	0.997 769
23	0.997 537
24	0.997 295
25	0.997 043
26	0.996 782
27	0.996 511
28	0.996 231
29	0.995 943
30	0.995 645
35	0.994 029
37.78	0.993 042
40	0.992 212
45	0.990 208
50	0.988 030
55	0.985 688
60	0.983 191
65	0.980 546
70	0.977 759
75	0.974 837
80	0.971 785
85	0.968 606
90	0.965 305
100	0.958 345

A-4.6.4 Turn the illumination light off immediately after sample introduction, because the heat generated can affect the measurement temperature.

A-4.6.5 After the instrument displays a steady reading to four significant figures for density and five for *T*-values, indicating that temperature equilibrium has been reached, record the density or *T*-value.

A-4.7 CALCULATION

A-4.7.1 *Calculating Density Analyzers*

The recorded value is the final result, expressed either as density, in g/ml or kg/m^3, or as relative density.

NOTE — 1 000 kg/m^3 = 1 g/ml.

A-4.7.2 *Non-calculating Density Analyzers*

Using the observed *T*-value for the sample and the *T*-value for water and appropriate instrument constants determined in **A-4.5.4.3**, calculate the density or relative density using the equation given below. Carry out all calculations to six significant figures and round the final results to four.

For density:

$$\text{Density, g/ml (kg/dm}^3\text{) at } t = d_w + K_1 \times (T_s^2 - T_w^2)$$

For relative density:

$$\text{Relative density, } t/t = 1 + K_2 \times (T_s^2 - T_w^2)$$

where

T_w = observed period of oscillation for cell containing water;
T_s = observed period of oscillation for cell containing sample;
d_w = density of water at test temperature;
K_1 = instrument constant for density;
K_2 = instrument constant for relative density; and
t = temperature of test, °C.

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF TOTAL SULPHUR CONTENT

B-1 OUTLINE OF THE METHOD

The sample is reacted with Raney nickel. The hydrogen sulphide liberated from the nickel sulphide thus formed is absorbed and titrated with mercuric acetate. Certain oxygenated sulphur compounds are not completely determined and some olefins interfere with the test.

NOTE — Stringent precautions shall be taken to avoid sulphur contamination from atmosphere, apparatus, and reagents or other sources. Care should be taken not to allow sodium hydroxide reagent or apparatus 'wet' with this reagent to be exposed to laboratory atmosphere.

B-2 APPARATUS

B-2.1 *Reduction Apparatus*

The reduction apparatus shall be of the shape, dimensions and assembly as shown in Fig. 3. The cone and delivery tube is connected to a supply of nitrogen via a Dreschel bottle and the luted venting device. Rubber tubing shall not be used for nitrogen supply connection; PVC or other sulphur-free

plastics tubing is suitable. The 100-rol flash is heated electrically by means of a mantle. The apparatus consists of the following:

B-2.1.1 *Reduction Flask*

100 ml round-bottomed flask with two short upright necks having respectively 10/19 and 14/23 ground-glass sockets (*see* IS 5165). The flask is also fitted with a 10/19 ground-glass socket inclined to centre-bottom.

B-2.1.2 *Glass Delivery Tube*

A 10/19 (*see* IS 5165) cone and stem with the end drawn out to a 1 mm hole, of such a length that the tip is within 5 mm of the centre of the bottom of the flask when the cone is in position in the inclined socket.

B-2.l.3 *Tap Funnel* — Capacity 20 ml with 10/19 cone and socket (*see* IS 5165).

B-2.1.4 *Adaptor* — Right-angle connection with 10/19 cone.

B-2.1.5 *Condenser*

A Liebig condenser, effective length 150 mm, with a 14/23 cone and socket.

B-2.1.6 *Absorber*

A delivery tube of 6 ± 0.5 mm outer diameter bent at 110° and containing a small expansion chamber in the upright section. The lower end of the tube has a 1 mm hole, and fits into a covered 200 mm x 32 mm outer diameter boiling tube. The upper end of the delivery tube is bent at an angle of 70° and fitted with a 14/23 cone at a distance of approximately 135 mm from the longer arm.

B-2.1.7 *Gas Washing Bottle* — Dreschel bottle having a dip-tube of about 6 mm outer diameter.

B-2.1.8 *Microburette* — Capacity 10 ml arranged so that the liquid in the absorber can be titrated.

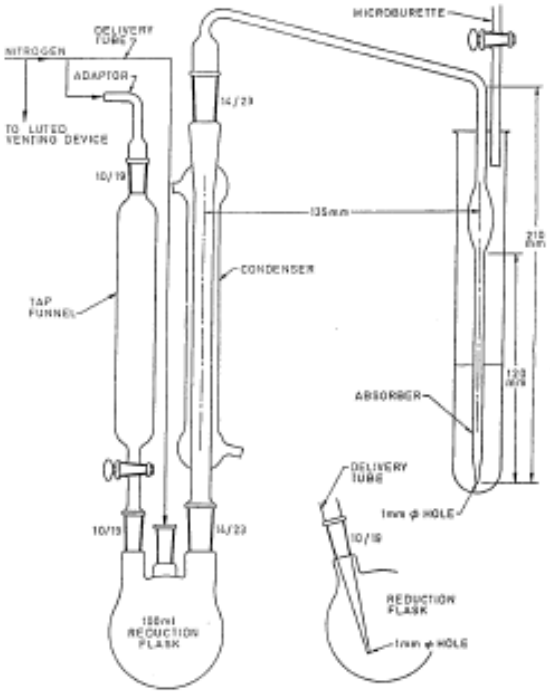


FIG. 3 REDUCTION APPARATUS

B-2.2 Flask

A 500 ml stoppered conical flask, marked at the 400 ml level with the cone and stopper lubricated with silicone grease.

B-2.3 Measuring Cylinder, capacity 10 ml.

B-2.4 Pipette (Not to be Operated by Mouth)

B-2.5 Thermometer — Any suitable thermometer including the interval 75 to 80°C.

B-3 REAGENTS

B-3.1 Acetone

B-3.2 Propan-2-ol

B-3.3 Nitrogen

B-3.4 Raney Nickel, 50 percent nickel, 50 percent aluminium.

B-3.5 Sodium Hydroxide Solutions, 2.5 N and 1 N

Clean the conical flask with nitric acid/potassium dichromate mixture [prepared by dissolving 5 g of potassium dichromate in 5 ml of water and adding 100 ml of concentrated (15 N) nitric acid, stirring continuously], rinse thoroughly with water. Fill with water to the 400 ml mark and add the mass of

sodium hydroxide pellets appropriate for preparation of the 2.5 N or 1 N solution. Swirl gently until dissolution is complete and allow to cool.

B-3.6 Hydrochloric Acid, 5N

B-3.7 Potassium Hydroxide — 40 g/l solution in ethanol.

B-3.8 Mercuric Acetate Solution

Dissolve 0.675 g of mercuric oxide, previously dried at 100°C, in 50 ml of water containing 2 ml of glacial (17 M) acetic acid. Dilute to 1 000 ml with water and mix well. Dilute 50 ml of the solution thus prepared to 250 ml with water and mix well. One milliliter of the diluted solution is equivalent to 0.02 mg of sulphur.

B-3.9 Dithizone Indicator Solution — 1 g/1 in acetone, prepared fresh daily, or every 3 days, if stored in a refrigerator.

NOTE — When experience has been gained with the concentration of indicator required, a few grains of the solid indicator may be added to the absorber. In this way any instability of indicator solution is overcome.

B-4 PROCEDURE

B-4.1 Clean the apparatus thoroughly with a mixture of nitric acid and potassium dichromate. Rinse thoroughly with water and acetone and dry in an oven which has not been contaminated with sulphur or sulphur containing materials in previous use. The apparatus is self-scouring and, when in constant use, shall not be cleaned between determinations, except for rinsing the flask, delivery tube, absorber and thermometer with water. When not in constant use, it shall be cleaned between determinations with water and acetone.

NOTE — For activating nickel perfectly, the weighed Raney nickel is kept in caustic solution for at least 4 h.

B-4.2 Weigh accurately about 0.5 g of Raney nickel and put it in the reduction flask using a cone made from glazed paper, and add 10 ml of sodium hydroxide solution (2.5 N) from the measuring cylinder. Care shall be taken at this stage because there is a vigorous reaction.

B-4.3 When the reaction has subsided, swirl the liquid in the flask to bring the nickel adhering to the sides of the flask to the bottom. Set the flask aside for 10 min and then decant the supernatant liquid. Wash down both necks of the flask with 10 to 15 ml of water. Swirl the water vigorously to disturb the nickel residue, but avoid entrainment of air and, with minimum delay for settling, decant the water as completely as possible without too much attention to removing the last drop. Repeat the water wash three more times, and follow with a wash with 10 ml of propan-2-ol. Decant most of the propan-2-ol leaving enough to cover the catalyst, and add a further 10 ml of propan-2-ol.

NOTE — Incrustations around the stoppers and necks of sodium hydroxide bottles contain sufficient quantities of sulphur to affect test results. Such incrustations should be removed without allowing material to fall into the bottle. Before using solution from the bottle, pour a little to waste. Replace the stopper promptly.

B-4.4 Assemble the apparatus except for the tap funnel, lightly greasing all the joints with silicone grease. Add 50 ml of a mixture of equal parts of sodium hydroxide solution (1 N) and acetone to the boiling tube and add 5 drops of the dithizone indicator solution.

B-4.3.1 Calculate the appropriate size of sample as follows:

$$\frac{100\ ml}{Specified\ or\ expected\ sulphur\ content\ (\frac{mg}{kg})}$$

(with a maximum of 50 ml) and pipette this volume into the flask through the 10/19 socket.

B-4.4 Complete the assembly of the apparatus. Measure 10 ml of the hydrochloric acid solution into the tap funnel. Pass nitrogen at the rate of 2 or 3 bubbles/second as shown in the Dreschel bottle containing the ethane-di-ol potassium hydroxide solution. Note the burette reading and titrate the contents of the absorber with the mercuric acetate solution to a pale pink colour.

B-4.4.1 Heat the flask at such a rate that the contents boil gently in about 10 min. Maintain the heating for a further period of 30 min at such a rate that small bubbles rise copiously from the nickel and gentle refluxing occurs.

B-4.4.2 Increase the input to the heating mantle slightly and allow the hydrochloric acid solution to drip slowly (10 ml in 5 to 10 min) into the flask. Vigorous generation of hydrogen will occur, but little or no hydrogen sulphide will be evolved until about half the hydrochloric acid has entered the flask. Titrate the absorbing solution to a pink colour. As hydrogen sulphide is evolved and absorbed and the colour of, the absorbing solution reverts to yellow, titrate in more mercuric acetate solution in order to restore the pink colour. After the addition of the acid, open the tap of the funnel occasionally to sweep forward any hydrogen sulphide that may have collected below it. When the evolution of hydrogen sulphide has almost ceased, increase the nitrogen flow rate to about 5 bubbles/second in the bottle to improve the transfer of hydrogen sulphide to the absorber.

B-4.4.3 When the evolution of hydrogen sulphide has apparently ceased, turn off the nitrogen temporarily and cool the flask by reducing the heat input and by blowing a little air on to it or by applying a damp cloth. The reduction in pressure will cause the absorbing solution to rise up the delivery tube. Restore the nitrogen flow before the absorbing solution reaches the bend above the cone. Repeat this operation at about 2 min intervals until no more hydrogen sulphide is washed down. If any liquid enters the cone, the test shall be abandoned.

B-4.4.4 Boil the contents of the flask vigorously and continue the titration to the end point.

B-4.4.5 Carry out a blank test on the reagents omitting the sample. Once a day is normally sufficient but the blank shall always be re-determined, if there is any change in the reagents, apparatus, or laboratory atmosphere which could conceivably affect the blank value. This value should not exceed 0.6 ml.

B-5 CALCULATION

$$\text{Total sulphur content, mg/kg} = \frac{20\times(V_1 - V_2)}{V\times D}$$

where

V_1 = volume of mercuric acetate solution used for the sample titration, in ml;
V_2 = volume of mercuric acetate solution used for the blank test, in ml;
V = volume of sample taken for the test, in ml; and
D = density of the sample at the temperature at which it was measured, in g/ml.

ANNEX C
[*Table* 1, *Sl No.* (vi)]
DETERMINATION OF DISTILLATION RANGE

C-1 OUTLINE OF THE METHOD

The method includes distillation of 100 ml test portion under prescribe conditions, which are equivalent to simple batch distillation, systematic observation of thermometer readings and volumes of condensate and calculation of the results from these data with correction to standard atmospheric pressure.

C-1.1 Significance

Distillation indicates volatility of the product. This is an important characteristics not only for the identification product, but also for its application. Its also used as an internal quality control tool, and in R&D work on hydrocarbons and related materials. It gives a broad indication of general purity of the product.

C-2 DEFINITIONS

For the purpose of this Annex the following definitions shall apply:

C-2.1 Initial Boiling Point

The temperature noted (corrected, if required) at the moment when the first drop of condensate falls from the tip of the condenser during a distillation carried out under standardization conditions.

C-2.2 Dry Point

The temperature note (corrected, if required) at the moment of vaporization of the last drop of liquid at the bottom of the flask during a distillation carried out under standardized conditions, discarding any liquid on the side of the flask and on the thermometer.

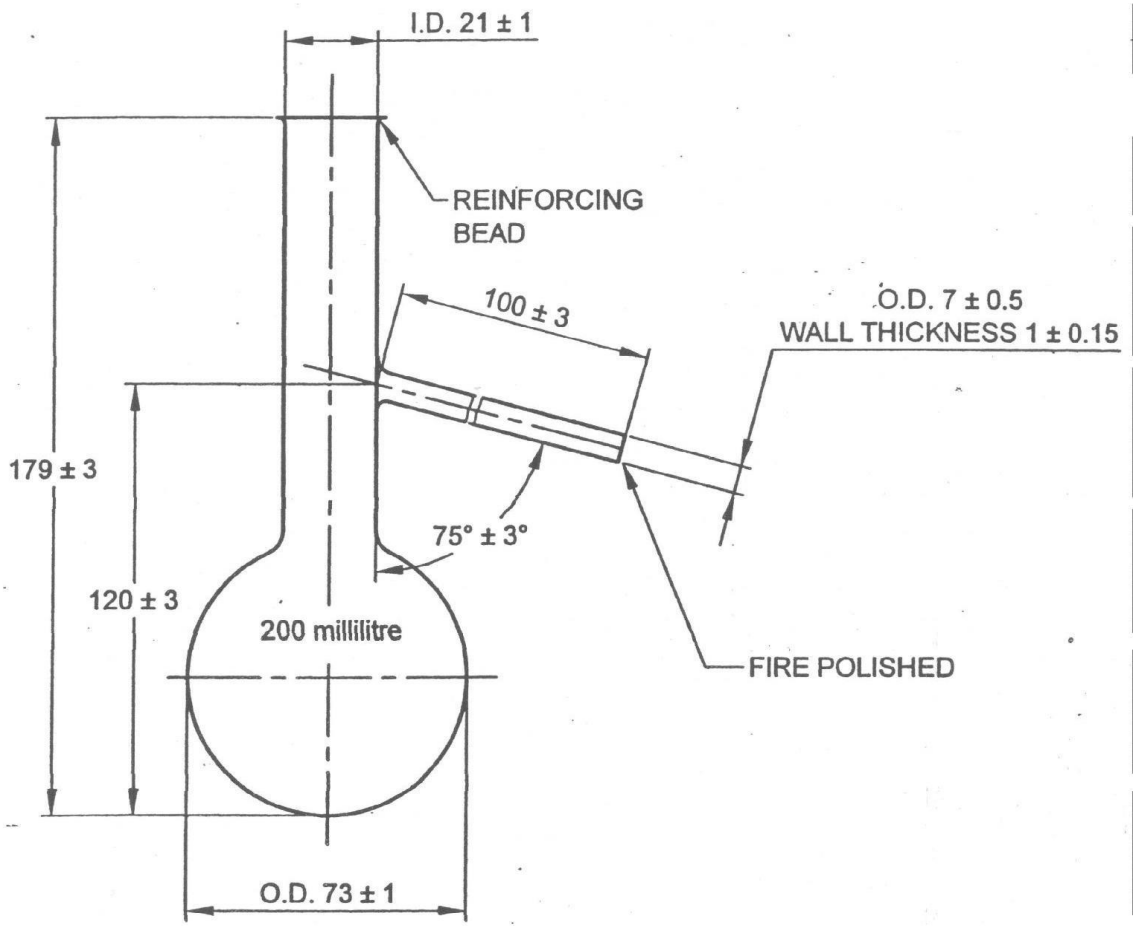
C-2.3 Boiling Range

The temperature interval between the initial boiling point and dry point.

C-3 APPARATUS

The apparatus, a suitable form of which is shown in Fig. 4 to 7, shall comprise the following items:

C-3.1 Distillation Flask — Made of heat resistant glass, of capacity 200 ml, conforming to the dimensions shown in Fig. 4.



All dimensions in millimeters.
FIG. 4 DISTILLATION FLASK

C-3.2 Thermometer

Mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to Table 5.

Table 5 Requirements of Thermometer
(*Clause* C-3.2)

Sl No.	Characteristic	Requirement
(1)	(2)	(3)
(i)	Immersion, mm	100
(ii)	Range, °C	72 to 126
(iii)	Graduation, °C	0.2
(iv)	Longer lines at each. °C	1
(v)	Figured at each, °C	2
(vi)	Scale error not to exceed, °C	0.2

(vii)	Overall length, mm	395 ±5
(viii)	Stem diameter, mm	6 to 7
(ix)	Bulb length, mm	15 to 20
(x)	Distance from bottom of bulb, mm	
	to 72 °C	125 to 145
	to 127 °C	335 to 360
(xi)	Expansion chamber to allow heating to, °C	150

C-3.3 Draught Screen

C-3.3.1 *For Use with a Gas Burner*

C-3.3.1.1 The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in Fig. 5 and be made of sheet of metal of thickness approximately 0.8 mm.

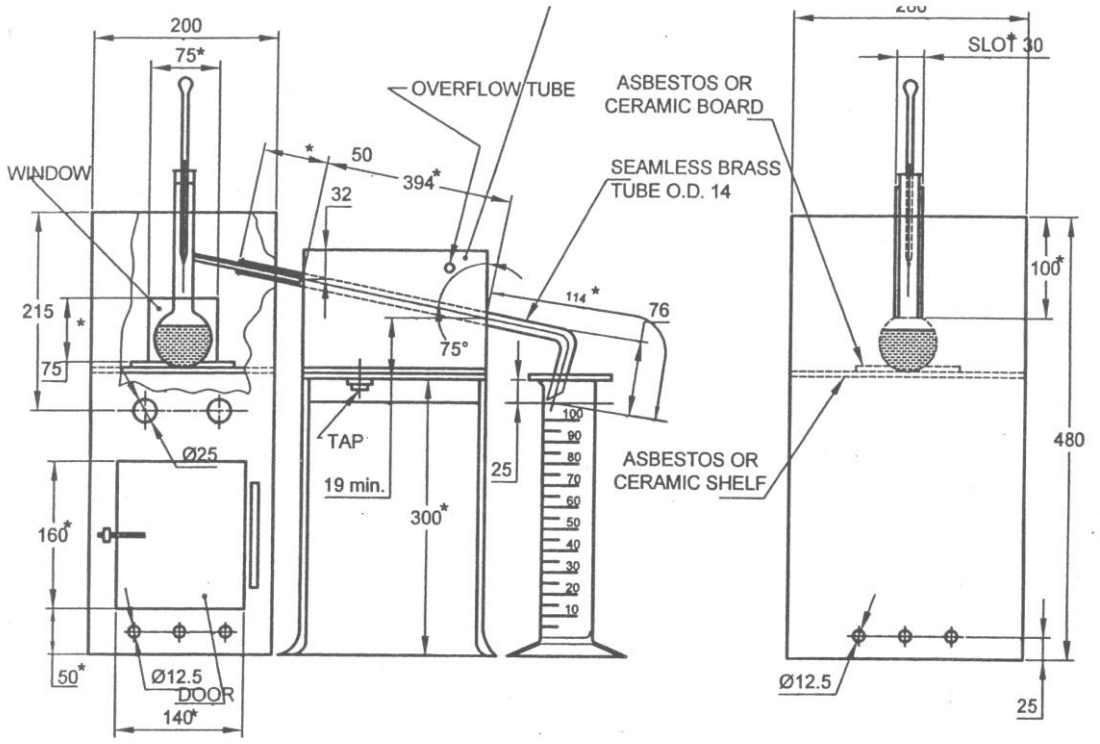
C-3.3.1.2 In each of the two narrower sides of the draught screen, there shall be two circular holes of diameter 12.5 mm, the centers of which are situated 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Fig. 5.

C-3.3.1.3 At the middle of each of the wider sides, a vertical slot for the condenser tube, dimensioned approximately as shown in Fig. 5, shall be cut downwards from the top of the screen. A removable shutter of suitable dimensions shall be provided for closing whichever vertical slot is not in use. This arrangement enables the condenser (*see* **C-3.6**) to be placed on either side of the draught screen.

C-3.3.1.4 A shelf of ceramic material, of thickness 3 mm to 6 mm and possessing a centrally cut circular hole of diameter 75 mm to 100 mm, shall be supported horizontally in the screen and shall fit closely to the sides of the screen, to ensure that hot gases from the source of heat (*see* **C-3.5**) do not come in contact with the sides or neck of the flask (*see* **C-3.1**). The supports for this shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

C-3.3.1.5 A board as described in **C-3.4** shall rest on this shelf.

C-3.3.1.6 In one of the narrower sides of the screen, a door shall be provided having the approximate dimensions shown in Fig. 5 and overlapping the opening in the screen by approximately 5 mm all round.



All dimensions in millimeter
FIG. 5 DISTILLATION ASSEMBLY

C-3.3.2 *For Use with an Electric Heater*

When an electric heater is employed, the portion of the draught screen above the shelf shall be as described in **C-3.3.1**, but the lower portion (including the shelf) may be modified or omitted, provided that the changes does not expose the distillation flask to draughts (*see* Fig. 6). Provision shall be made for adjustment of the shelf to facilitate fitting of the flask.

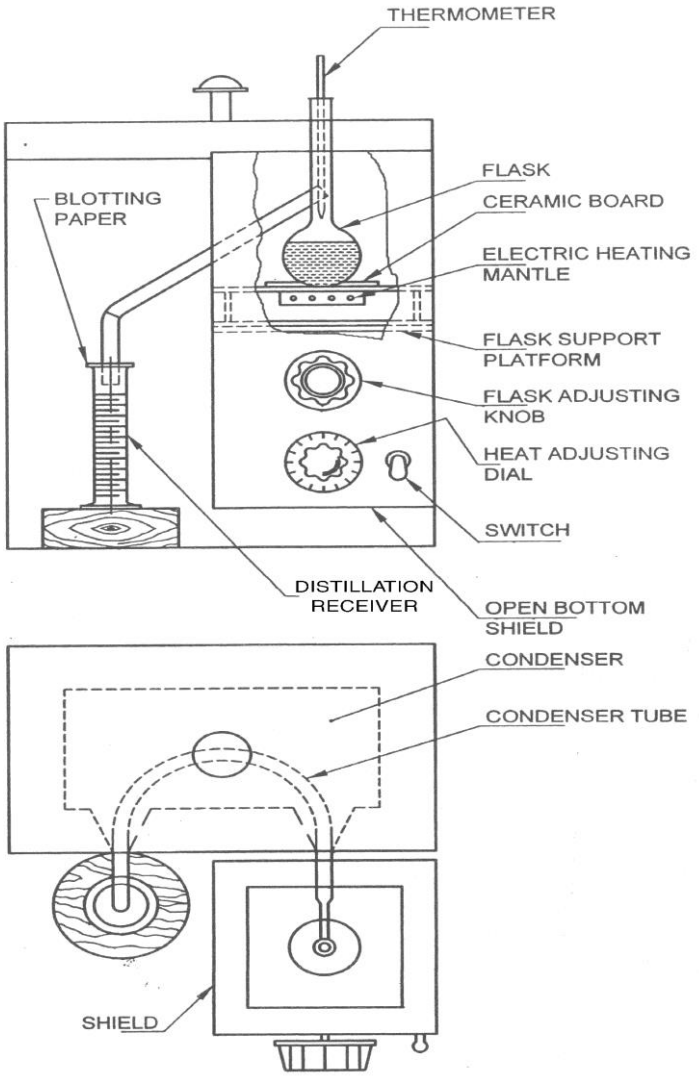


FIG. 6 DISTILLATION APPARATUS USING ELECTRIC HEATER

C-3.4 Ceramic Boards

C-3.4.1 It shall be of thickness 3 to 6 mm, with central holes of diameter 32 mm or 38 mm respectively and overall dimensions not less than 150 mm^2. When a gas heater is employed, this board shall rest on the shelf described in **C-3.3.1** when an electric heater is employed, the same arrangement shall be adopted, if the shelf is present; alternatively, the board may be placed directly on the heater or it may form the top of the heater. Provision shall be made for adjusting the height of the heater.

C-3.4.2 Whichever type of heater is employed, direct heat shall only be applied to the flask through the central hole in the ceramic board.

C-3.5 Source of Heat

Comprising either a gas burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in **C-5.3**. A sensitive regulating valve or governor is desirable adjuncts or

an electric heater capable of complying with the same requirements (A heater of low heat retention, adjustable from 0 to 1 kW, has been found satisfactory).

C-3.6 Condenser

C-3.6.1 A seamless brass tubing, of length 560 mm, outside diameter 14 mm and wall thickness 0.8 to 0.9 mm, surrounded by a metal cooling bath, preferably of copper or brass. The tube shall be set so that a length of approximately 390 mm is in contact with the cooling medium in the cooling bath, with about 50 mm outside the cooling bath at the upper end, and about 115 mm outside at the lower end. The length of the tube projecting at the upper end shall be straight and set at an angle of 75 degree to the vertical. The section of the tube inside the cooling bath may be either straight shall be 0.26 mm per linear millimeter of the condenser tube (sin 15^{o}), and no part of it shall have a gradient less than 0.24 mm nor more than 0.28 mm per linear millimeter of the tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and slightly backward so as to ensure contact with the wall of the receiver (*see* **C-3.7**) at a point 25 mm to 32 mm below the top of the receiver when it is in a position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle so that the tip may be brought into contact with the wall of the receiver.

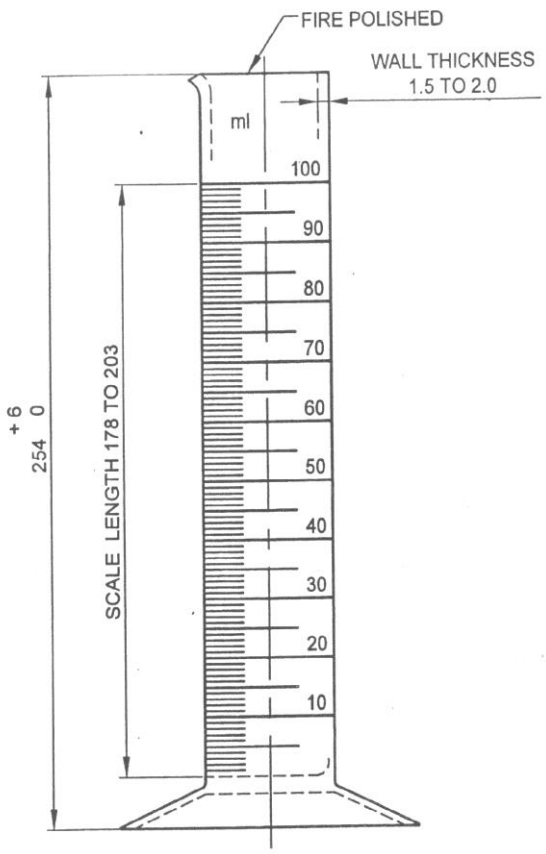
C-3.6.2 The capacity of the cooling bath shall be not less than 5.5 liter of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line is not less than 32 mm below the plane of the top of the bath at its point of entrance, and not less than 19 mm above the floor of the bath at its exist. Clearances between the condenser tube and the walls of the bath shall be at least 13 mm, except for the section adjacent to the points of entrance and exit.

C-3.6.3 The cooling bath may be provided with a tap at the bottom for drainage or inlet, and with an overflow tube near the top.

C-3.6.4 The main dimensions of the tube and cooling bath are shown in Fig. 5.

C-3.7 Receiver

It shall be of capacity 100 ml, complying with the details shown in Fig. 7. None of the graduation lines shall be in error by more than 1 ml. The shape of the base is optional but it shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 15 to the horizontal.



All dimensions in millimetres.
FIG. 7 DISTILLATION RECEIVER

C-3.8 Barometer — Accurate to the nearest 1 mbar, 0.1 kPa or 1 mmHg.

C-4 ASSEMBLY AND PREPARATION OF APPARATUS

C-4.1 Assembly

Assemble the apparatus, swabbing out the condenser with a piece of lint-free cloth attached to a wire cord or by any other suitable means, and paying attention to the following details:

C-4.1.1 *Position and Choice of Thermometer*

Use the thermometer as indicated at **C-3.2**. Centre the thermometer into the neck of the flask through a tight-fitting silicone-rubber or cork stopper so that the upper end of the contraction chamber is level with the lower wide of the vapour tube at its junction with the neck of the flask.

C-4.1.2 *Support for Flask*

If a draught screen with ceramic shelf is used, place the appropriate ceramic board (*see* **C-3.4** and **C-5.3**) on top of the shelf so that the two holes are concentric.

C-4.1.3 *Connection of Flask to Condenser*

C-4.1.3.1 Make a leak-proof connection of the flask (*see* **C-3.1**) to the tube of the condenser (*see* **C-3.6**) by means of a tight-fitting silicone-rubber or cork stopper through which the vapour tube of the flask passes. Connect the flask to the condenser so that the flask is in a vertical position; the end of the vapour tube shall extend at least 25 mm and not more than 50 mm beyond the cork into the condenser tube and shall be co-axial with it.

C-4.1.3.2 Place the flask in such a position on the board that the base completely closes the hole in the board.

C-4.2 Filling of Cooling Bath

Fill the bath with water or with water and crack ice in sufficient quantity to cover the condenser tube, so as to ensure that the temperature of the bath at the start of and during distillation remains between 25°C to 30°C.

C-4.3 Adjustment of Temperature of Sample

Adjust the temperature of the sample to between 20°C to 30°C, to prevent excessive evaporation of the product.

C-5 PROCEDURE

C-5.1 Test Portion

Using the graduated receiver (*see* **C-3.7**), measure 100 ± 0.5 ml of the sample at the temperature to which it has been adjusted as specified in **C-4.3**. Remove the flask (*see* **C-3.1**) from the apparatus and transfer the test portion directly to the flask, allowing the receiver to drain for 15 to 20s. Do not allow any of the test portion to enter the vapour tube.

C-5.2 Apparatus Assembly

C-5.2.1 Connect the flask to the condenser (*see* **C-3.6**) and insert the thermometer (*see* **C-3.3**) as described in **C-4.1.1** and position the flask as described in **C-4.1.3**. Place the receiver (*see* **C-3.7**), without drying, at the outlet of the condenser tube in such a position that the condenser tube extends into the receiver at least 25 mm but does not extend below the 100 ml mark.

C-5.2.2 Place a flat cover on the top of the receiver to prevent entry of condensed moisture.

C-5.3 Operating Conditions

A certain amount of judgement is necessary in choosing the best operating conditions to obtain acceptable accuracy and reproducibility. As a general guide it is recommended that the following conditions shall be established:

(a) Flask support — Hole diameter, 32 mm
(b) Heating rate — Time from application of heat to collection of first drop of distillate 5 to 10 min, and time for rise of vapour column in neck to flask to side arm, 2.5 min to 3.5 min.

C-5.4 Initial Boiling Point

Record the temperature at the instant the first drop of distillate falls from the tip of the condenser as the initial boiling point (*see* **C-2.1**).

C-5.5 Distillation

Adjust the heat input so that the distillation proceeds at a rate of 4 to 5 ml/min (approximately 2 drops per second), and move the receiver so that the tip of the condenser tube touches one side of the cylinder after the first drop falls.

C-5.6 Dry Point

Without changing the heater setting, continue distillation beyond the 95 percent point until the dry point (*see* **C-2.2**) is observed. Record the temperature at this moment as the dry point. If a dry point is not obtained (that is, if active decomposition occurs before the dry point is reached, as shown by a rapid evolution of vapour or heavy fumes, or if there is liquid remaining on the bottom of the flask when the maximum temperature is observed in the distillation thermometer), record this fact.

C-5.7 Atmospheric Pressure

Read and record the barometric pressure to the nearest 1 mbar, 0.1 kPa or 1 mmHg.

C-6 CALCULATIONS

C-6.1 Thermometer Bore Correction

Apply the correction for any variation in the bore of the thermometer as given by the calibration certificate.

C-6.2 Thermometer Bulb Shrinkage Correction

Apply the correction for shrinkage of the mercury bulb of the thermometer as determined by any change in its ice or steam point, where applicable. Other means can be employed, such as the use of a platinum-resistance thermometer or a recognized standard thermometer.

C-6.3 Barometer Correction

After applying the corrections for thermometer error, correct each reading for deviation of the barometric pressure from normal by adding algebraically the correction, calculated as follows:

$$K\times(760-p_0)$$
or $$K'\times(1\ 013-p_1)$$
or $$K'\times(1\ 013-p_2)$$

where

K = rate of change of boiling point with pressure, in (°C/mmHg) (*see* Table 6);

K' = rate of change of boiling point with pressure, in per millibar or per 0.1 kPa (°C/ mbar or °C/kPa) (*see* Table 6);
p_0 = barometric pressure during the test, in mmHg;
p_1 = barometric pressure during the test, in millibars;
p_2 = the barometric pressure, in kilopascals, during the test.

Table 6 Boiling Point
(*Clause* C-6.3)

Sl No.	Characteristic	Requirement
(1)	(2)	(3)
(i)	Boiling point °C (at 1.013 bar, 101.3 kPa, 760 mmHg)	110.6
(ii)	Rate of change of boiling point with pressure *K'* (°C/mbar, °C/0.1kPa)	0.035
(iii)	Rate of change of boiling point with pressure *K* (°C/mmHg)	0.046

ANNEX D
[*Table* 1, *Sl No*. (i), (vii) and (viii)]
DETERMINATION OF NON-AROMATIC HYDROCARBONS, BENZENE AND PURITY OF TOLUENE BY GAS CHROMATOGRAPHY AND EFFECTIVE CARBON NUMBER

D-1 GENERAL

D-1.1 This test method covers the determination of total nonaromatic hydrocarbons and monocyclic aromatic hydrocarbons in toluene gas chromatography.

D-1.2 The limit of detection (LOD) is 0.000 2 mass percent and limit of quantitation (LOQ) is 0.000 6 mass percent for impurities in toluene.

D-2 OUTLINE OF THE METHOD

The sample to be analyzed is injected into a gas chromatograph equipped with a Flame Ionization Detector (FID) and a capillary column. The peak area of each component is measured and adjusted using Effective Carbon Number (ECN) correction factors. The concentration of each component is calculated based on its relative percentages of total adjusted peak area and normalized to 100 percent.

D-3 APPARATUS

D-3.1 Chromatographic data system is required.

D-3.2 Columns

The choice of column is based on resolution requirements. Any column may be used that is capable of resolving all significant impurities from the major component. The column and conditions described in Table 7 have been used successfully and shall be used as a referee in cases of dispute.

Table 7 Recommended Method Parameters
(*Clause* D-3.2 and D-5)

Inlet	Split
Temperature, °C	270
Column:	
Tubing	fused silica
Length, m	60
Internal diameter, mm	0.32
Stationary phase	crosslinked polyethylene glycol
Film thickness, µm	0.25
Column temperature program	
Initial temperature, °C	60
Initial time, min	10
Programming rate, °C/min	5
Final, °C	150
Time 2, min	10
Carrier gas	Helium or hydrogen
Linear velocity, cm/s at 145°C	20 helium or 45 hydrogen
Split ratio	100:1
Sample size, µl	0.6
Detector:	Flame ionization
Temperature, °C	300
Analysis time, min	38

NOTE — In case of requirement, above parameters can be modified to achieve sensitivity and separation of peaks.

D-3.3 Gas Chromatograph

Any instrument having a flame ionization detector and a splitter injector suitable for use with a fused silica capillary column may be used, provided the system has sufficient sensitivity, linearity, and range to determine 0.000 1 mass percent, while not exceeding the full scale of either the detector or the electronic integration for the major component. It shall have a split injection system that will not discriminate over the boiling range of the samples analyzed.

D-3.4 Injector

The specimen must be precisely and repeatably injected into the gas chromatograph. An automatic sample injection devise is highly recommended.

D-3.5 Syringe — Chromatographic, capable of delivering appropriate µl volumes.

D-4 REAGENTS AND MATERIALS

D-4.1 Purity of Reagent

Reagent grade chemicals shall be used in all tests.

D-4.2 Carrier Gas, Makeup Gas and Detector Gases, 99.999 Percent Pure

Oxygen in carrier gas shall be less than 1 ppm (less than 0.5 ppm is preferred). Purify carrier, makeup and detector gases to remove oxygen, water, and hydrocarbons.

D-4.3 Air for the FID should contain less than 0.1 ppm total hydrocarbon.

D-5 PREPARATION OF APPARATUS

Follow manufacturer's instructions for mounting and conditioning the column into the chromatograph and adjusting the instrument to the conditions described in Table 7, allowing sufficient time for the equipment to reach equilibrium.

D-6 CALIBRATION

D-6.1 Prior to implementation of the ECN method, a laboratory shall demonstrate that the equipment is set up properly using an equipment known check sample. This known check sample can be prepared by using toluene with benzene, ethyl benzene, *m*-xylene, *p*-xylene etc. (impurities as per requirement). This mixture shall be used to determine retention times of each component, and that the separation of *m*-xylene from *p*-xylene is satisfactory. Laboratory may use commercially available mixture also.

D-6.2 The LOD for this standard is 0.000 2 mass percent. The equipment set-up check sample contains 0.000 4 mass percent toluene. Acceptable results are 0.000 1 mass percent to 0.000 8 mass percent.

D-7 PROCEDURE

D-7.1 Bring the sample to room temperature.

D-7.2 Analyze the equipment set-up check sample as needed to ensure adequate resolution of *m*-xylene from *p*-xylene, that the gas chromatograph has the sensitivity specified by this standard, and that all the peaks are properly identified.

D-7.3 Inject an appropriate amount of sample into the instrument.

D-7.4 Review the chromatographic data system result. Measure the area of all peaks. The non-aromatics fraction includes all peaks up to ethylbenzene except for the peaks assigned to benzene and toluene. Sum together all the non-aromatic peaks as a total area. When either benzene or toluene is analyzed and 1,4-dioxane is required to be reported, the non-aromatic fraction does not include the peak assigned to 1,4-dioxane.

D-7.5 See Fig. 8 for representative chromatograms.

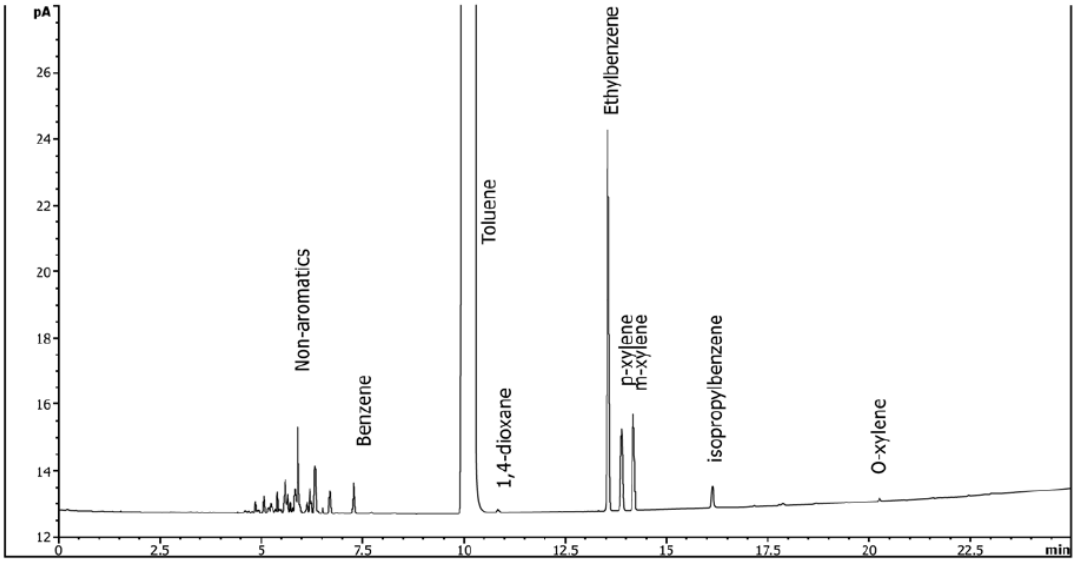


FIG. 8 TYPICAL CHROMATOGRAM OF TOLUENE

D-8 CALCULATION

D-8.1 Using the ECN mass correction factors listed in Table 8, calculate the concentration of each component as follows:

$$C_i = 100 \times (A_i \times R_i) / \sum_{i=1}^{n} (A_i \times R_i)$$

where

C_i = concentration for component *i*, in mass percent;
A_i = peak area of component *i*; and
R_i = ECN correction factor for component *i*.

D-8.2 Calculate the volume percent concentration of each component using the density in Table 8 as follows:

$$V_i = 100 \times (C_i/D_i) / \sum_{i=1}^{n} (C_i/D_i)$$

where

V_i = calculated volume percent concentration of component *i*;
C_i = calculated mass percent concentration of component *i* from **D-8.1**; and
D_i = density of component *i*.

Table 8 Effective Carbon Number Correction Factors and Density
(*Clauses* D-8.1 and D-8.2)

Component	*ECN Correction Factor*[1)]	*Density at* 20°C, *in g/ml*
Non-Aromatics	1.0000	0.7255
Benzene	0.9095	0.8780
Toluene	0.9195	0.8658
1,4-dioxane	3.0774	1.0329

Ethylbenzene	0.9271	0.8658
p-xylene	0.9271	0.8597
m-xylene	0.9271	0.8630
Cumene	0.9329	0.8605
o-xylene	0.9271	0.8786
n-propylbenzene	0.9329	0.8620
C_9 Aromatics	0.9329	0.8715
tert-butylbenzene	0.9376	0.867
sec-butylbenzene	0.9376	0.863
Styrene	0.9210	0.9048
Ethyltoluene	0.9329	0.861
C_{10} Aromatics	0.9376	0.8694
p-diethylbenzene (PDEB)	0.9376	0.8620
Phenylacetylene	0.8296	0.9300
Alpha-methylstyrene (AMS)	0.9276	0.9077
Trimethylbenzen	0.9329	0.8637
2-propenylbenzene	0.9276	0.893
Unknown	0.931[2)]	

[1)]Correction factors are relative to n-heptane.
[2)]This is the average of correction factors for components with retention times greater than 1,4-dioxane and does not include compounds with a triple bond.

D-9 REPORT

D-9.1 Calculate concentration of individual impurities and total non-aromatics, to the nearest 0.000 1 mass percent, C_i.

D-9.2 Report benzene and total non-aromatics, to the nearest 0.000 1 mass percent.

D-9.3 Report toluene, percent by mass = 100.00 - C_i , (C_i = total concentration of all impurities, percent by mass)

D-9.4 Report toluene purity as "purity (by GC)" to the nearest 0.01 mass percent.

D-10 PRECISION AND BIAS

Repeatability and Reproducibility are as below:

Component	*Average mass percent[1)], $\bar{x}$*	*Repeatability limit[2)],r*	*Reproducibility limit[3)],R*
Non-Aromatics	0.0052	0.0032	0.0047
Benzene	0.0159	0.0008	0.0022
Toluene	99.9345	0.0068	0.0131
Ethylbenzene	0.0204	0.0014	0.0029
p-xylene	0.0097	0.0018	0.0029
m-xylene	0.0108	0.0020	0.0037

o-xylene	0.0010	0.0016	0.0020
C9+Aromatics	0.0024	0.0026	0.0028

[1)]The average of the laboratories' calculated averages.
[2)]Average of four levels of r.
[3)]Average of four levels of R.

ANNEX E
(*Clause* 6)
SAMPLING OF TOLUENE

E-1 GENERAL REQUIREMENTS OF SAMPLING

E-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed:

a) Samples shall not be taken in an exposed place;
b) Sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material;
c) Samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination;
d) 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;
e) Samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour;
f) Sample container shall be sealed air-tight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of material, the batch number, the name of the sample, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample; and
g) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

E-1.2 Additional Precautions

The following additional precautions shall be observed:

a) Rubber stoppers or 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, viscose or other suitable impervious material over the stopper to keep away moisture and dusts from the mouth of the bottle and to protect it while being handled.

E-2 SAMPLING INSTRUMENT

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.

E-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. 9). 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.

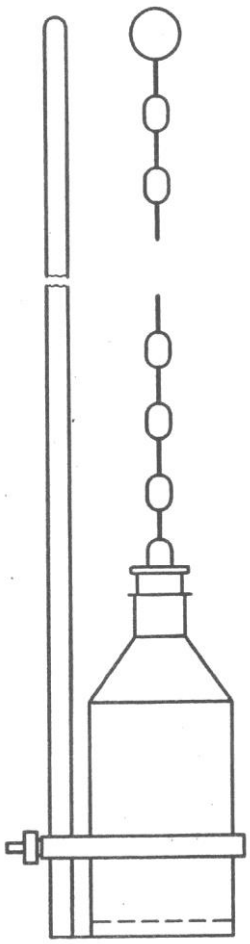
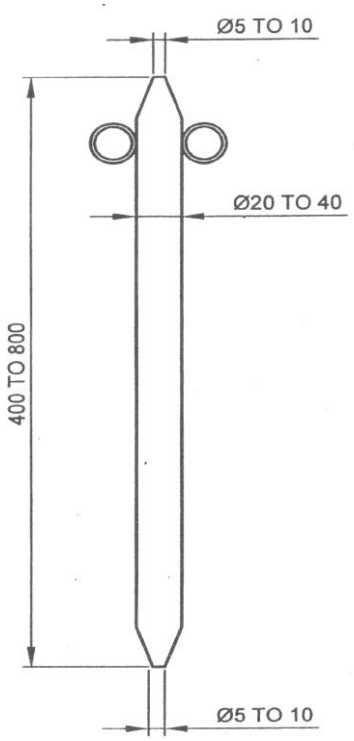


FIG. 9 SAMPLING BOTTLE OR CAN

E-2.2 Sampling Tube

E-2.2.1 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. 10). The upper and lower ends are conical and reach 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.



All dimensions in millimeters
FIG. 10 SAMPLING TUBE

E-2.2.2 For small containers, the size of the sampling tube may be altered suitably.

E-3 SCALE OF SAMPLING

E-3.1 Lot

E-3.1.1 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 the consignment is in large tanks or vessels, the tanks or vessels belonging to the same batch of manufacture shall constitute a lot.

E-3.1.2 For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

E-3.2 Sampling from Containers

The number of containers to be selected for sampling shall depend on the size of the lot and shall be in accordance with Table 9.

Table 9 Scale of Sampling
(*Clause* E-3.2)

Sl No.	Lot size	No. of containers to be selected
(1)	(2)	(3)

(i)	Up to 100	5
(ii)	101 to 200	6
(iii)	201 to 300	7
(iv)	301 to 400	8
(v)	401 to 500	9
(vi)	501 and above	10

NOTE — In the case of very small lots where the selection of the five containers may be uneconomical, all the containers shall be selected

E-3.3 The containers shall be selected at random in order to ensure the randomness of selection, procedure given in IS 4905 may be adopted.

E-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 standard.

E-4 PREPARATION OF THE TEST SAMPLES

E-4.1 Test Samples from Containers

To ensure that the sample taken from each container is fairly representative, the containers 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. 10). The approximate quantity of the material to be drawn from a container shall be nearly equal to thrice the quantity required for testing purposes as indicated in **E-5.1**.

E-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 under **E-5**. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

E-4.1.2 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

E-4.1.3 All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed airtight with stoppers and labeled with full identification particulars given in **E-1.1** (f).

E-4.1.4 The referee test sample, consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

E-4.2 Test Samples from Tanks or Vessels

E-4.2.1 For drawing a sample from a tank or vessel, lower the closed sampling bottle or can (*see* **E-2.1**) slowly to the required depth, open and fill it at that depth. Three 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) and nine-tenths of the depth of the liquid from the top surface (lower sample). All the three samples thus obtained from a tank/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 a vessel shall nearly be equal to thrice the quantity required for carrying out tests for all the requirements prescribed in **E-5**.

E-4.2.2 All the samples thus obtained from the tanks or vessels in the lot 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 **E-1.1** (f).

E-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 purchaser and the supplier, to be used in case of any dispute.

E-5 NUMBER OF TESTS

E-5.1 For Samples from Containers

E-5.1.1 Tests for the determination of distillation range and residue on evaporation shall be conducted on each of the individual samples separately (*see* **E-4.1.2**).

E-5.1.2 Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite samples separately (*see* **F-4.1.1**).

E-5.2 For Samples from Tanks or Vessels

Tests for the determination of all the characteristics given in Table 1 shall be conducted on the samples from different tanks or vessels separately

E-6 CRITERIA FOR CONFORMITY

E-6.1 For Containers

E-6.1.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of the distillation range if test results for each of the individual samples tested in respect of distillation range satisfy the requirements as given in Table 1.

E-6.1.2 *For Composite Samples*

In respect of all other characteristics, the lot shall be considered as conforming to the composite sample satisfies each one of these requirements.

E-6.2 For Tanks or Vessels

The lot shall be declared as confirming to the standard requirements of various characteristics, if each of the test results satisfies the relevant requirements specified in the standard individually.