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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 40]

WATER BY DISTILLATION

(*Third Revision*)

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1. SCOPE

1.1 This method covers the determination of water in petroleum products, tars and products derived from tars.

1.1.1 The specific products listed in **7.1.1** represent the range of materials considered in developing the details of this method.

2. SUMMARY OF METHOD

2.1 The material is heated under reflux with a water-immiscible solvent which co-distils with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap and the solvent returning to the still.

3. APPARATUS

3.1 The apparatus comprises of a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap and condenser may be connected by any suitable method for producing a leak-proof joint. Preferred connections are ground joints for glass and O-rings for metal to glass. Typical assemblies are illustrated in Fig. 1 to 3.

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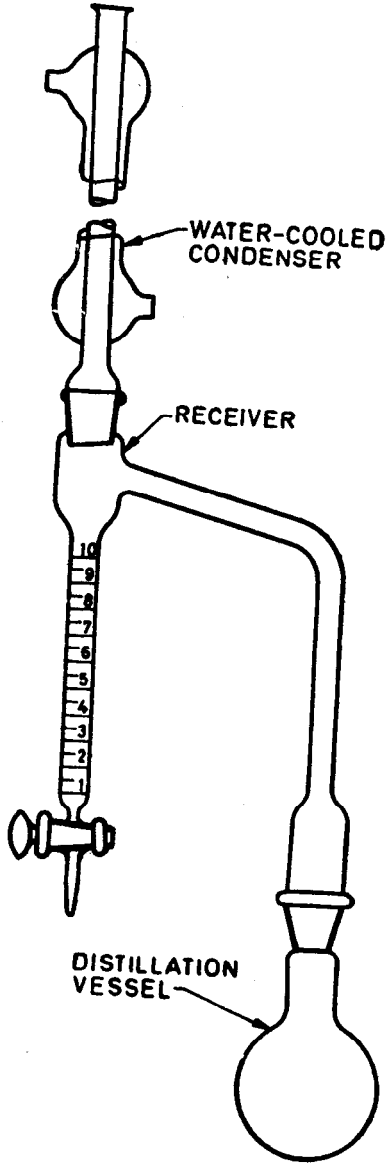
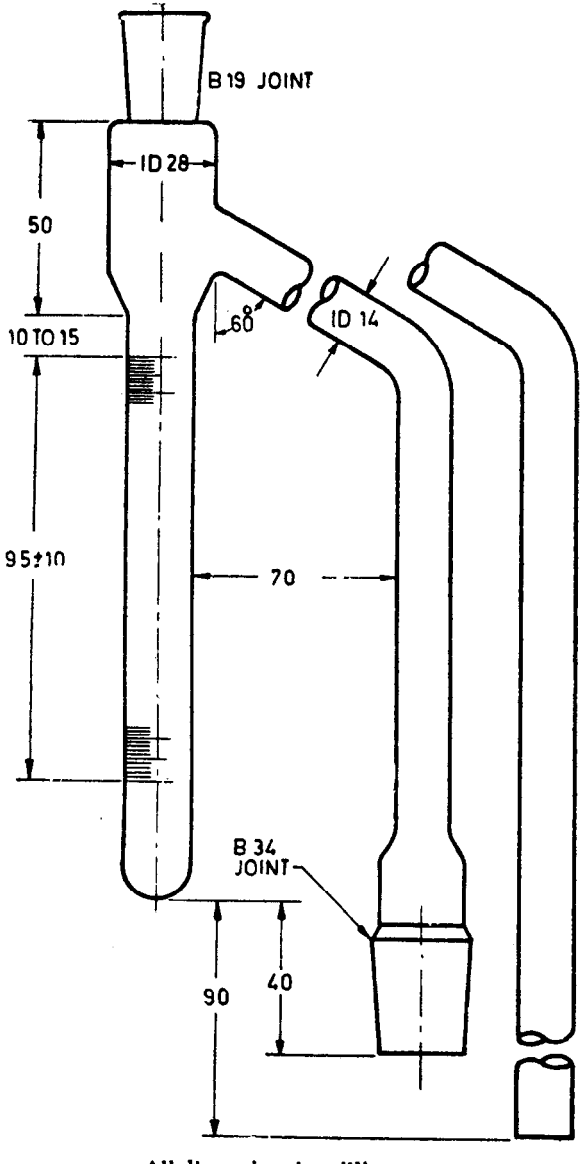


FIG. 1 TYPICAL ASSEMBLY WITH GLASS STILL
(DEAN AND STARK APPARATUS)



All dimensions in millimetres.

FIG. 2 5 ml RECIEVER SHOWING ALTERNATIVE CONNECTIONS TO DISTILLATION VESSEL

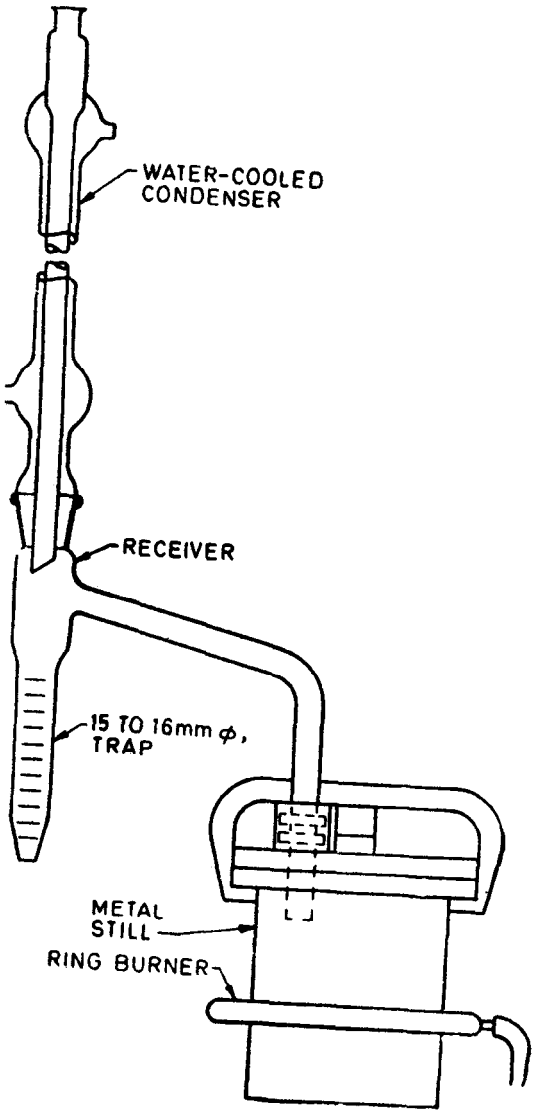


FIG. 3 TYPICAL ASSEMBLY WITH METAL STILL

3.1.1 *Still* — A glass or metal vessel with a short neck and suitable joint for accommodating the reflux tube of the trap. Vessels of 500, 1 000 and 2 000 ml nominal capacities have been found satisfactory.

3.1.2 *Heater* — Any suitable gas burner or electric heater may be used with the glass still. A gas ring burner with ports on the inside circumference shall be used with the metal still, and shall be of such dimensions that it may be moved up and down the vessel when testing materials which are liable to foam or to solidify in the still.

3.2 Dimensions and description of typical glassware for use in this method are given in relevant Indian Standards on glass apparatus. A straight water-cooled condenser with a length of 400 mm is recommended. The stills and traps should be chosen to cover the range of materials and water content expected (*see* Note).

NOTE — Instead of standardizing on a particular apparatus with respect to dimensions and style, a given apparatus will be deemed as satisfactory when accurate results are obtained by the standard addition technique obtained in 6.

4. SOLVENT-CARRIER LIQUID

4.1 Any suitable hydrocarbon boiling in the range of 100 to 200°C may be used. With residual fuel oils and bitumens, aromatic solvents are desirable in order to avoid separation of asphaltenes. For the determination of water in certain lubricating greases, close boiling range petroleum distillate (*see* **4.3**) has been found to be necessary.

4.2 The following solvents have been found suitable:

a) *Commercial toluene* — industrial grade,

b) *Commercial xylene* — industrial grade, and

c) Petroleum distillate fractions in the boiling range of 100 to 200°C.

4.3 Petroleum spirit in the boiling range of 100 to 120°C. *Iso*-octane 95 percent purity or better.

CAUTION — Toluene and xylene are toxic, volatile hydrocarbons which are absorbed by inhaling the vapour or through the skin by contact with the liquid. Use adequate ventilation and avoid skin contact.

5. SAMPLE

5.1 The portion of the sample used for the test shall be thoroughly representative of the total sample. If the material is liquid, thoroughly

mix the sample as received, warming if necessary, to ensure uniformity. Crush the solid materials that are sufficiently brittle, mix thoroughly, and take a representative sample for analysis. When there is doubt as to the uniformity of the material, run a number of samples and average the data. Sampling procedure is described in the appropriate clauses of IS : 1447-1966*.

5.2 A sample size of 100 ml or 100 g is recommended. Should however the quantity of water exceed the capacity of the largest permitted trap, the quantity of sample may be decreased or the trap fitted with a stopcock used, where excess water may be withdrawn into a graduated cylinder.

6. STANDARDIZATION

6.1 A given assembly of apparatus will be considered satisfactory when accurate readings are obtained from addition of known amounts of water from a calibrated burette or pipette to a clear hydrocarbon oil and tested in accordance with **7**.

6.2 The readings will be judged accurate if conformity to the permissible limits given in Table 1 for the various sized graduated traps are not exceeded.

TABLE 1 PERMISSIBLE LIMITS

Capacity of Receiver in ml at 20°C	Volume of Water in ml Added to Flask at 20°C	Permissible Limits for Recovered Water in ml at 20°C
5	1	$1 \pm 0{\cdot}1$
10	1	$1 \pm 0{\cdot}1$
10	5	$5 \pm 0{\cdot}2$
25	12	$12 \pm 0{\cdot}2$

6.3 A reading outside the permissible limits suggests malfunctioning due to vapour leaks, too rapid boiling, inaccuracies in calibration of trap, or ingress of extraneous moisture. Eliminate these factors before repeating the standardization.

7. PROCEDURE

7.1 Measure a suitable amount of sample (*see* **5.2**) to an accuracy of ± 1 percent of sample and transfer it to the still.

*Methods of sampling of petroleum and its products.

7.1.1 Measure ordinary liquid samples in a graduated cylinder of appropriate size. Rinse the material adhering to the cylinder into the still with one 50 ml and two 25 ml portions of the solvent-carrier liquid; the latter being one selected from those described in **4** and corresponding to the type suggested in Table 2 for the specific material under test. Drain the cylinder thoroughly after the sample transfer and each rinsing.

TABLE 2 SOLVENT CARRIER TO BE USED FOR VARIOUS SAMPLE MATERIALS

Type of Solvent-Carrier Liquid	Materials
Aromatic	Asphalt, tar, coal tar, water gas tar, road tar, cutback bitumen, liquid asphalt and tar acid
Petroleum distillate	Road oil, fuel oil, lubricating oil and petroleum sulphonates
Volatile spirits	Lubricating grease

7.1.2 Weigh solid or viscous materials directly into the still and add 100 ml of the selected solvent-carrier liquid.

7.1.3 In case of low water content material when samples larger than 100 g or 100 ml may be used, a solvent-carrier volume in excess of 100 ml may be necessary.

7.1.4 Glass beads or other boiling aids may be added, if necessary, to reduce bumping.

7.2 Assemble the components of the apparatus as illustrated in Fig. 1 to 3 choosing the trap in accordance with the expected water content of the sample and making all connections, vapour and liquid tight. If a metal still with removable cover is used, insert a gasket of heavy paper, moistened with solvent, between the still body and cover. The condenser tube and trap shall be chemically clean to ensure free drainage of water into the bottom of the trap. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture inside it. Circulate cold water through the jacket of the condenser.

7.3 Apply heat to the still, adjusting the rate of boiling so that condensed distillate discharges from the condenser at the rate of 2 to 5 drops per second. If the metal still is used, start heating with the ring burner about 76 mm above the bottom of the still and gradually lower the burner as the distillation proceeds. Continue distillation until no

water is visible in any part of the apparatus except in the trap and the volume of water in the trap remains constant for 5 minutes. If there is a persistent ring of water in the condenser tube, carefully increase the rate of distillation or cut off the condenser water for a few minutes.

7.4 When the evolution of water is complete, allow the trap and contents to cool to room temperature. Dislodge any drops of water adhering to the sides of the trap with a glass rod or other suitable means and transfer them to the water layer. Read the volume of the water in the trap to the nearest scale division.

8. CALCULATION

8.1 Calculate the water in the sample, as mass or volume percent according to the basis on which the sample was taken, as follows:

$$\text{Water, percent} = \frac{\text{volume of water in trap}}{\text{mass (or volume) of sample}} \times 100$$

8.1.1 Volatile water-soluble material, if present, may be measured as water.

9. REPORT

9.1 Report the result as the water content to the nearest 0·1 percent, if 100 ml or 100 g of sample has been used for results 1·0 percent and above. Report to the nearest 0·05 percent for results below 1·0 percent.

10. PRECISION

10.1 Results of duplicate tests shall not differ by more than the following amounts:

Water Collected	*Repeatability*	*Reproducibility*
0 to 1·0 ml	0·1 ml	0·2 ml
1·1 to 25 ml	0·1 ml or 2 percent of the mean, whichever is greater	0·2 ml or 10 percent of the mean, whichever is greater

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