INTERNATIONAL STANDARD 3733

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Petroleum products and bituminous materials — Determination of water — Distillation method

Produits pétroliers et produits bitumineux — Détermination de la teneur en eau — Méthode par distillation

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3733 was drawn up by Technical Committee ISO/TC 28, *Petroleum products,* and circulated to the Member Bodies in October 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Iran	Sweden
Brazil	Ireland	Turkey
Bulgaria	Israel	United Kingdom
Canada	Japan	U.S.A.
Chile	Netherlands	U.S.S.R.
Czechoslovakia	Poland	Yugoslavia
France	Portugal	
Germany	Romania	

No Member Body expressed disapproval of the document.

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Petroleum products and bituminous materials — Determination of water — Distillation method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of water in crude petroleum, tars and products derived from these materials, excluding bituminous emulsions.

The specific products listed in 8.1.1 represent the range of materials considered in developing the details of this method.

2 REFERENCES

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling.*

ISO 3171, *Petroleum products — Liquid hydrocarbons — Automatic pipeline sampling.*

ISO 5272, *Toluene — Specifications.* [1]

ISO 5280, *Xylene — Specifications.* [1]

ISO . . . , *Solid petroleum products — Sampling.* [1]

3 PRINCIPLE

The test portion is heated under reflux with a water-immiscible solvent which co-distils with the water in the test portion. 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.

4 SOLVENT-CARRIER LIQUID

4.1 Any suitable hydrocarbon solvent, free from water, boiling in the range 100 to 200 °C may be used. With asphaltic crude oils, residual fuel oils and bitumens, aromatic solvents are desirable to avoid separation of asphaltenes. For the determination of water in certain lubricating greases, petroleum distillates of close boiling range (4.3) have been shown to be necessary.

4.2 The following solvents have been found suitable :

a) Toluene — ISO 5272, grade 2.

b) Xylene — ISO 5280.

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

CAUTION — Toluene vapour is toxic and care should be taken to avoid breathing it. The danger of eye splashes from toluene in particular, or any of the other specified solvents should be avoided.

4.3 Petroleum distillates of close boiling range. Use either :

petroleum spirit with a boiling range from 100 to 120 °C, or

iso-octane, 95 % purity or better.

1) In preparation.

5 APPARATUS

5.1 The apparatus comprises a glass or metal still, a heater, a reflux condenser, minimum length of jacket 400 mm, 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 figures 1 and 2.

5.1.1 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.

5.1.2 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 that are likely to foam or solidify in the still.

5.2 Dimensions and descriptions of typical glassware for use in this method are shown in figure 3.

5.3 The stills and traps should be chosen to cover the range of materials and water contents expected. If the amount of water collected is likely to exceed 25 ml, the 25 ml trap fitted with a stopcock may be used and the excess water drained off into a graduated cylinder.

NOTE – A given apparatus will be deemed satisfactory when accurate results are obtained by the calibration technique described in clause 7.

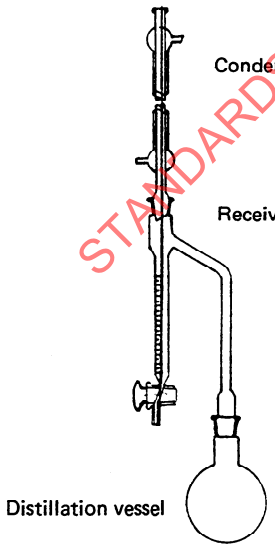


FIGURE 1 – **Typical assembly with glass still (Dean and Stark apparatus)**

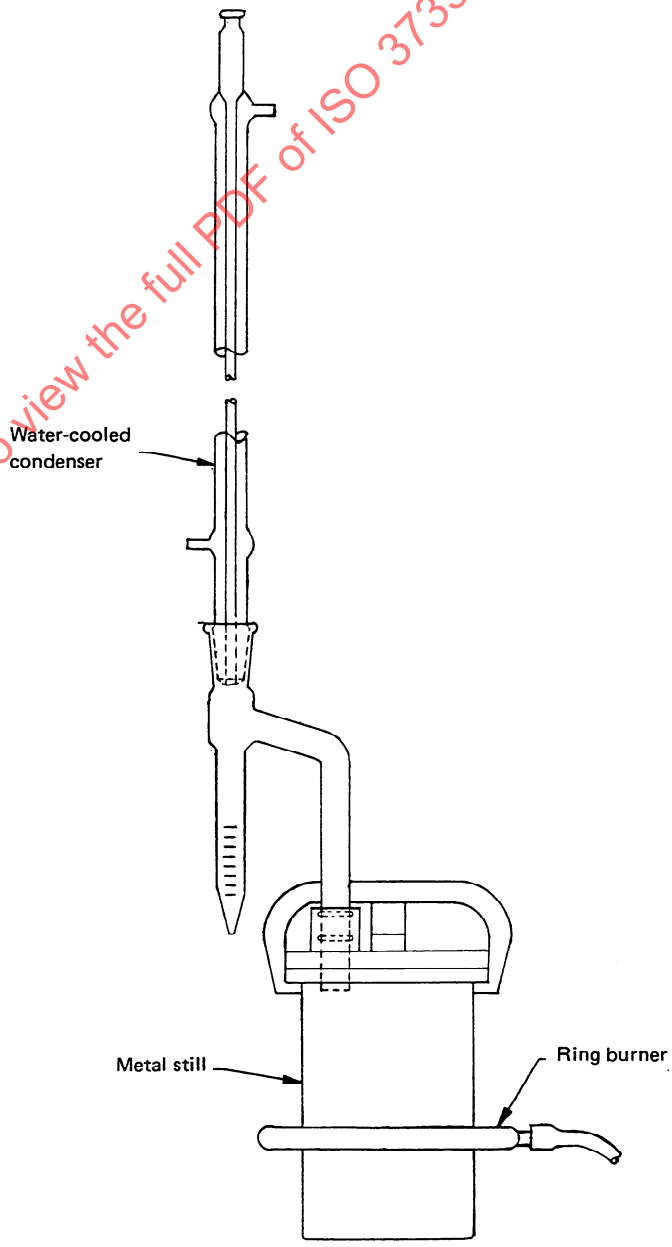
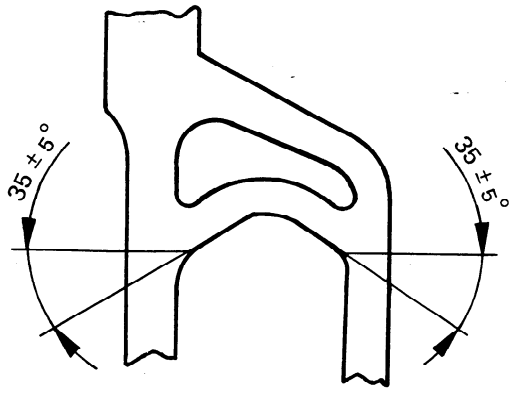


FIGURE 2 – **Typical assembly with metal still**

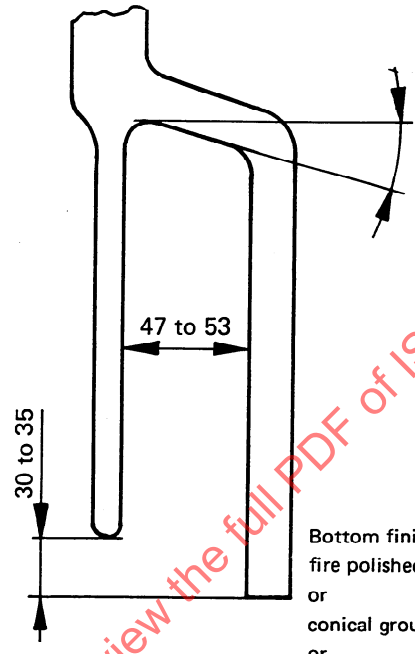
Top finish :
beaded edge;
or
conical ground glass joint, ISO/R 383/24/29 socket;
or
spherical ground glass joint, ISO 641/35/20 cup.

Dimensions in millimetres



Alternative top design

Alternative conical tip



25 ml traps may be fitted with a stopcock if required

Bottom finish :
fire polished;
or
conical ground glass joint, ISO/R 383/24/29 cone;
or
spherical ground glass joint, ISO 641/35/20 ball.

Essential requirements of traps

Trap size, ml	5	10			25	
Scale range, ml	0 to 5	0 to 1	>1 to 10	0 to 10	0 to 1	>1 to 25
Smallest scale division, ml	0,1	0,1	0,2	0,1	0,1	0,2
Maximum scale error, ml	0,05	0,05	0,1	0,1	0,05	0,1
Bottom of graduated tube	round	conical		round	conical	
Length of graduated portion, mm	120 to 140	120 to 140		120 to 140	140 to 160	

Dimensions shown other than in the table are for guidance only.

FIGURE 3 — **Details of typical traps**

6 SAMPLING

6.1 Laboratory sample

According to the nature of the material, laboratory samples should be taken in accordance with ISO 3170, ISO 3171 or ISO . . .

6.2 Preparation of test samples

6.2.1 Solid samples which are sufficiently brittle shall be crushed and mixed thoroughly. A representative test portion shall be drawn from the crushed and mixed samples.

6.2.2 Liquid samples shall be thoroughly mixed, after warming if necessary, in order to ensure uniformity. A representative test portion shall be drawn from the mixed sample.

NOTE — If there is any doubt about the uniformity of the mixed test sample prepared as in 6.2.1 or 6.2.2, determinations should be made on a number of test portions and the average results reported as the water content.

7 CALIBRATION

7.1 A given assembly of apparatus will be considered satisfactory if accurate readings are obtained after the addition of known amounts of distilled or demineralized water from a calibrated burette or pipette to a clear hydrocarbon oil and testing the mixture in accordance with clause 8.

7.2 The readings shall be judged accurate if the permissible limits given in table 1 for the graduated traps of different sizes are not exceeded.

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

TABLE 1 — **Permissible limits**

Capacity of trap at 20 °C ml	Volume of water added to flask at 20 °C ml	Permissible limits for recovered water at 20 °C ml
5	1	1 ± 0,1
10	1	1 ± 0,1
10	5	5 ± 0,2
25	12	12 ± 0,2

8 PROCEDURE

8.1 Measure a test portion of 100 ml or 100 g to an accuracy of ± 1 % and transfer it to the still.

8.1.1 Measure mobile liquid materials in a graduated cylinder of appropriate size. Rinse the material adhering to the cylinder with one 50 ml portion and two 25 ml portions of the solvent-carrier liquid, the solvent being one selected from those described in clause 4 and corresponding to the type suggested in table 2 for the specific material under test. Drain the cylinder thoroughly after the test portion transfer and each rinsing.

TABLE 2 — **Types of solvent-carrier liquid**

Type of solvent-carrier liquid	Materials
Aromatic	bituminous materials excluding emulsions
Petroleum distillate	crude petroleum, road oil, fuel oil, lubricating oil, petroleum sulphonates
Close boiling petroleum distillate	lubricating greases

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

8.1.3 In cases of material with a low water content, when large test portions may be used, a solvent-carrier volume in excess of 100 ml may be necessary.

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

8.2 Assemble the components of the apparatus as shown in figures 1 and 2, 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 a removable cover is used, insert a gasket or heavy paper, moistened with solvent, between the still body and cover. The condenser tube and trap must 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.

8.3 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 75 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 the water in the trap remains constant for 5 min. 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.

8.4 When the carry-over 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 by other suitable means and transfer them to the water layer. Read the volume of the water in the trap to the nearest scale division.

9 EXPRESSION OF RESULTS

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

$$\text{water content \% } (m/m) = \frac{V_0}{m} \times 100$$

$$\text{or water content \% } (V/V) = \frac{V_0}{V} \times 100$$

where

V_0 is the volume, in millilitres, of water in the trap;

m is the mass, in grams, of the test portion;

V is the volume, in millilitres, of the test portion.

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