

INTERNATIONAL STANDARD

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

*Produits pétroliers et produits bitumineux — Dosage de l'eau — Méthode
par distillation*



Reference number
ISO 3733:1999(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

ISO 3733 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 6, *Bulk cargo transfer, accountability, inspection and reconciliation*.

This second edition cancels and replaces the first edition (ISO 3733:1976), of which it constitutes a technical revision.

Annex A forms an integral part of this International Standard.

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

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitation prior to use.

1 Scope

This International Standard specifies a method for determination of water up to 25 % in petroleum products, bitumens, tars and products derived from these materials, excluding emulsions, by the distillation method. The specific products considered during the development of this test method are listed in Table 3.

This International Standard may be used to determine water content in excess of 25 %. However, no precision data has been determined at levels greater than 25 %. Volatile water-soluble material, if present, is measured as water.

NOTE 1 A knowledge of the water content of petroleum products is important in the refining, purchase, sale and transfer of products.

NOTE 2 The amount of water determined by this method can be used to correct the volume involved in the custody transfer of product.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling*.

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*.

ISO 3405:—¹⁾, *Petroleum products — Determination of distillation characteristics at atmospheric pressure*.

3 Principle

The test material is heated under reflux with a water-immiscible solvent, which co-distills 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.

¹⁾ To be published. (Revision of ISO 3405:1988)

4 Reagents

NOTE For the purposes of this International Standard, the expression “% (V/V)” is used to represent the volume fraction of a material.

A solvent-carrier liquid appropriate to the materials being tested (see Table 3) shall be used.

4.1 Aromatic solvent

The following aromatic solvents, free of water (0,02 % maximum) are suitable:

- a) industrial grade xylene (mixed xylenes);
- b) a blend of 20 % (V/V) industrial grade toluene and 80 % (V/V) industrial grade xylene (mixed xylenes);
- c) petroleum distillate, yielding not more than 5 % (V/V) distillates at 125 °C and not less than 20 % (V/V) at 160 °C when tested in accordance with ISO 3405 and with a density not lower than 855 kg/m³ at 15 °C.

4.2 Petroleum distillate solvent

A petroleum solvent, free of water, of which 5 % (V/V) boils between 90 °C and 100 °C, and 90 % (V/V) distils below 210 °C.

4.3 Paraffinic solvents

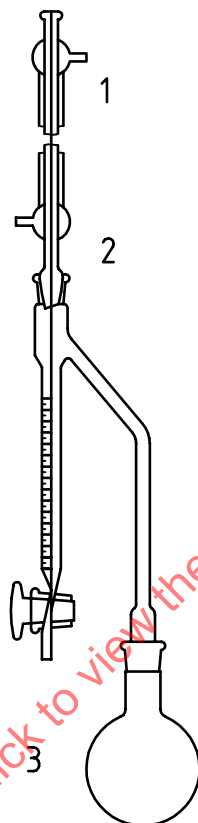
The following paraffinic solvents, free of water, are suitable:

- a) light petroleum with a boiling range of 100 °C to 120 °C;
- b) 2,2,4-trimethylpentane (*iso*-octane) of 95 % purity or better.

5 Apparatus

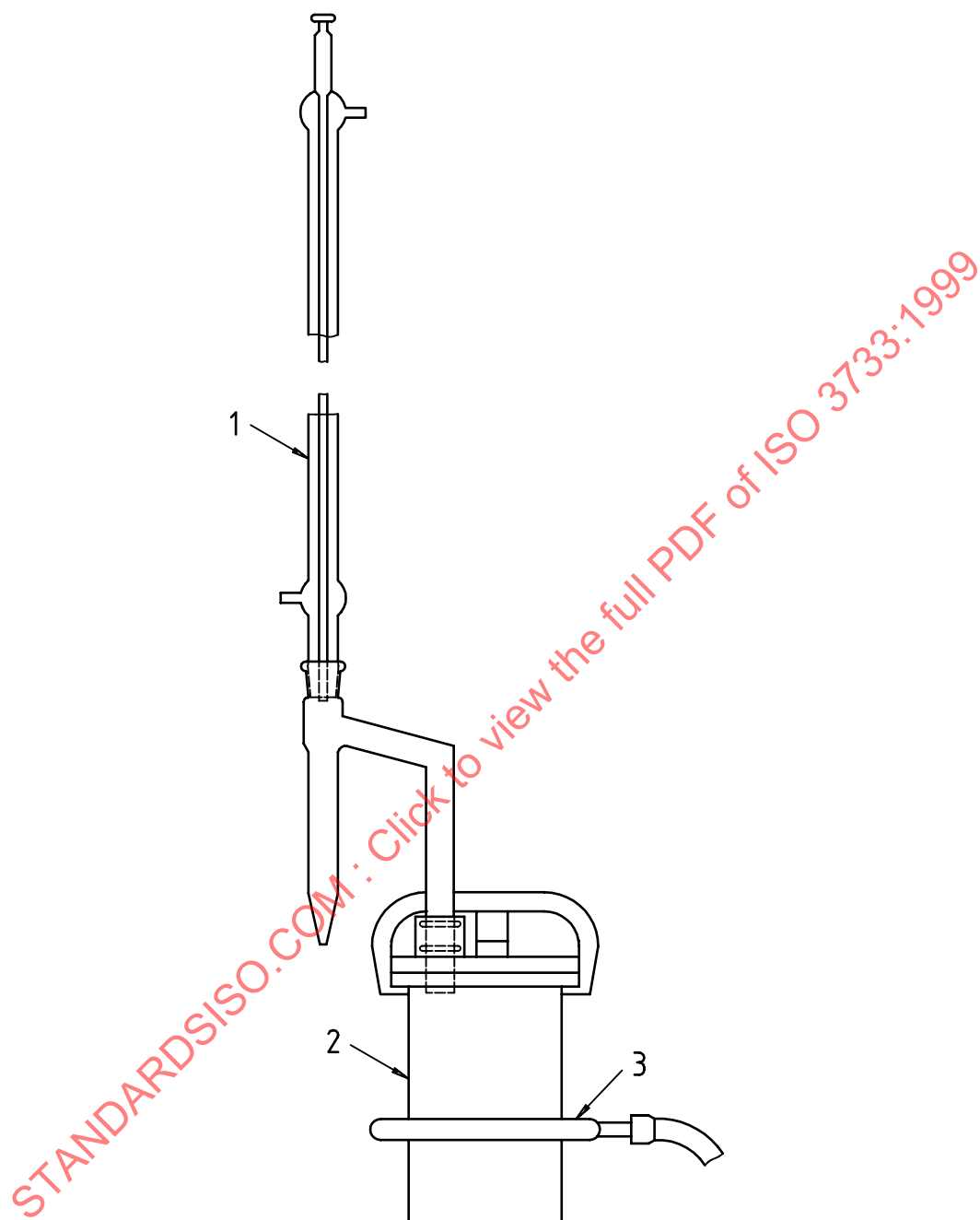
5.1 General

The apparatus comprises a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap and condenser shall be connected by any suitable method that produces a leak-proof joint. Preferred connections are ground joints for glass, and O-rings for metal to glass. Typical assemblies of the glassware are shown in Figures 1 to 3 and the dimensions are given in Table 1. Any given testing apparatus will be deemed suitable, if accurate results are obtained using the methods described in 6.2 and 6.3. However, a straight water-cooled condenser with a length of 400 mm is recommended. The stills and traps shall be chosen to cover the range of materials and water contents expected. If the amount of water collected is likely to exceed 25 ml, a 25 ml trap fitted with a stopcock shall be used and the excess water shall be drained off into a graduated cylinder meeting the requirements of 6.2 and 6.3.

**Key**

- 1 Condenser
- 2 Receiver
- 3 Distillation vessel

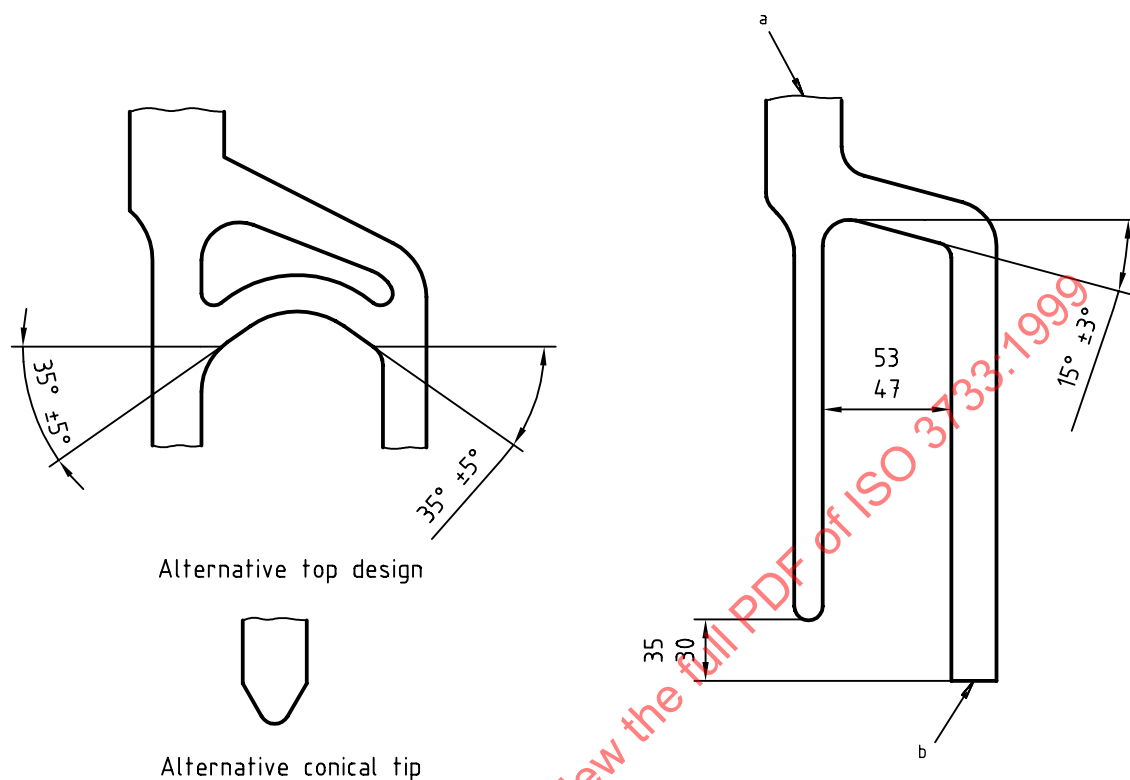
Figure 1 — Typical assembly with glass still (Dean and Stark apparatus)

**Key**

- 1 Water-cooled condenser
- 2 Metal still
- 3 Ring burner

Figure 2 — Typical assembly with metal still

Dimensions in millimetres



NOTE 1 Dimensions than those shown other in Table 1 are for guidance only.

NOTE 2 25 ml traps may be fitted with a stopcock if required.

- a Top finish:
beaded edge;
or
conical ground-glass joint, ISO 383/24/29 socket;
or
spherical ground-glass joint, ISO 641/35/20 cup.
- b Bottom finish:
fire polished;
or
conical ground-glass joint, ISO 383/24/29 cone;
or
spherical ground-glass joint, ISO 641/35/20 ball.

Figure 3 — Details of typical traps

Table 1 — Essential requirements of traps

Trap size, ml	2	5	10		25	
Scale range, ml	0 to 2	0 to 5	0 to 1	>1 to 10	0 to 10	0 to 1 >1 to 25
Smallest scale division, ml	0,05	0,05	0,1	0,2	0,1	0,1 0,2
Maximum scale error, ml	0,025	0,05	0,05	0,1	0,1	0,05 0,1
Bottom of graduated tube	round	round	conical		round	conical
Length of graduated portion, mm	85 to 105	120 to 140	120 to 140		120 to 140	140 to 160

5.2 Still, comprising a glass or metal vessel with a short neck and a suitable joint for accommodating the reflux tube of the trap. Vessels of nominal capacity 500 ml, 1 000 ml and 2 000 ml have proved satisfactory. The metal still shall be fitted with a heat-resistant gasket of material other than asbestos.

5.3 Heater, a suitable electric heater may be used with the glass still. A heater combined with a magnetic stirrer has proved to be effective to avoid bumping. A gas ring burner with ports on the inside circumference shall be used with the metal still. The gas ring burner shall be of such dimensions that it can be moved up and down the vessel when testing materials that are likely to foam or solidify in the still.

6 Verification and recovery test

6.1 General

Before initial use, verify the accuracy of the trap in accordance with 6.2. Before using the apparatus, carry out a recovery test in accordance with 6.3.

6.2 Verification

Before initial use, verify the accuracy of the graduation marks on the trap by adding 0,05 ml increments of distilled water from a 5 ml microburette or a precision micropipette readable to the nearest 0,01 ml. If there is a deviation of more than 0,05 ml between the water added and water observed, reject the trap or recalibrate.

6.3 Recovery test

Before using the apparatus for a water determination, test the overall recovery of water in the entire apparatus by introducing 250 ml to 1 000 ml of dry (0,02 % water maximum) xylene, according to the size of the vessel, to the apparatus (500 ml to 2 000 ml vessel as specified in 5.2) and proceed as described in clause 8. When complete, discard the contents of the trap, allow the contents of the still to cool and add the appropriate quantity of distilled water from a burette directly to the distillation flask in accordance with Table 2, and proceed as described in clause 8. The readings shall be judged accurate if the permissible limits given in Table 2 for graduated traps of different sizes are not exceeded.

Table 2 — Permissible limits for water recovery

Values in millilitres

Capacity of trap at 20 °C	Volume of water added to flask at 20 °C	Permissible limits for recovered water at 20 °C
2	1,00	$1,0 \pm 0,025$
5	1,00	$1,0 \pm 0,025$
5	4,50	$4,5 \pm 0,025$
10	1,0	$1 \pm 0,1$
10	5,0	$5 \pm 0,25$
25	12,0	$12 \pm 0,25$

A reading outside the permissible limits indicates malfunction due to vapour leaks, too rapid boiling, or ingress of extraneous moisture. Eliminate these factors and repeat the recovery test.

7 Sampling (see annex A)

7.1 General

Sampling is defined as all steps required to obtain a representative sample of the contents of any pipe, tank or other system and to place the sample in the laboratory test container.

7.2 Laboratory sample

Only representative samples obtained as specified in ISO 3170 and ISO 3171 shall be used in this International Standard.

The size of the test portion shall be based on the expected water content of the sample, such that the water yield does not exceed the capacity of the trap, unless a trap with a stopcock is used, since excess water can then be withdrawn into a graduated cylinder.

7.3 Preparation of the test samples

The following sample-handling procedures shall apply in addition to those covered in ISO 3170 and ISO 3171.

7.3.1 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.

7.3.2 Liquid samples shall be mixed, after warming, if necessary, in the original container. The mixing time, mixing power (speed) and height above the bottom of the container shall be satisfactory for the product being analysed as established in the procedure given in annex A. The volume and water content of the sample shall not exceed the maximum validated in annex A.

NOTE If there is any doubt about the homogeneity of the mixed samples prepared in 7.3.1 or 7.3.2, determinations should be made on the total volume of the sample if the sample size is compatible with the expected water content. If this is not possible, a determination should be made on at least three test portions. All these results should be included in the test report and their average should be recorded as the water content of the sample.

8 Procedure

8.1 Homogenize the sample in accordance with 7.3 before taking the test portion.

8.2 Measure the required test portion to an accuracy of $\pm 1\%$ and transfer it to the still in accordance with 8.3 or 8.4, depending on the sample type.

8.3 Measure mobile liquid samples in a graduated cylinder of an appropriate size. Rinse the material adhering to the cylinder with one 50 ml and two 25 ml portions of the solvent-carrier liquid (see clause 4 and Table 3). Drain the cylinder thoroughly after the sample transfer and each rinsing.

Table 3 — Type of solvent-carrier liquid versus material to be tested

Type of solvent-carrier liquid	Material
Aromatic	Bitumen, coal tars, preparations from bitumen and coal tars excluding emulsions.
Petroleum distillate	Road oil, fuel oil, lubricating oil, petroleum sulfonates.
Paraffinic	Lubricating grease.

8.4 Weigh solid or viscous materials directly into the still and add 100 ml of the selected solvent-carrier liquid (see Table 3). When testing material of low water content where large test portions are necessary, a solvent-carrier liquid volume in excess of 100 ml may be used.

8.5 A magnetic stirrer has been found to be most effective, but glass beads or other boiling aids may be added, if necessary, to reduce bumping.

8.6 Assemble the components of the apparatus as illustrated 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 of heavy paper, moistened with solvent, between the body and the cover of the still. The condenser tube and trap shall be chemically clean to assure 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.

8.7 Apply heat to the still, adjusting the rate of boiling so that condensed distillate discharges from the condenser at a rate of 2 drops per second to 9 drops per second. If the metal still is used, start heating with the ring burner approximately 75 mm above the bottom of the still and gradually lower the burner as the distillation proceeds. Continue the 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 min. If there is a persistent ring of water in the condenser tube, carefully increase or cut off the condenser water for a few minutes.

8.8 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 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.

8.9 Whenever a new solvent batch is used, a solvent blank shall be established by placing the volume of the solvent to be used during the actual determinations in the distillation flask and testing as outlined in 8.5 to 8.8.

9 Calculation

Calculate the water in the sample, φ as a percentage (volume/volume), or w as a percentage (mass/mass), in accordance with the basis on which the sample was taken as follows:

$$\varphi = \frac{V_1 - V_2}{V_0} \times 100 \quad (1)$$

$$\varphi = \frac{V_1 - V_2}{m/\rho} \times 100 \quad (2)$$

$$w = \frac{V_1 - V_2}{m} \times 100 \quad (3)$$

where

V_0 is the volume of the test portion, in millilitres;

V_1 is the volume of water, in millilitres, in the trap when the measurement is made on the unknown sample;

V_2 is the volume of water, in millilitres, in the trap when the measurement is made on the solvent blank;

m is the mass of test sample, in grams;

ρ is the density of sample, in grams per millilitre at 15 °C.

It is assumed that the density of water is 1 g/ml.

NOTE Volatile water-soluble material, if present, is measured as water.

10 Expressions of results

Report the result as the water content to the nearest 0,05 % if the 2 ml trap has been used, and to the nearest 0,1 % if the 10 ml or 25 ml traps have been used with a 100 ml or 100 g sample.

11 Precision

The criteria described in 11.1 and 11.2 shall be used to judge the acceptability of results (95 % probability) when using the 10 ml or 25 ml traps. The precision when using the 2 ml trap has not been established.

11.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 4 in only one case in 20.

11.2 Reproducibility

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 4 in only one case in 20.

Table 4 — Precision

Values in millilitres

Water collected	Repeatability	Reproducibility
0,0 to 1,0	0,1	0,2
1,1 to 25	0,1 ml or 2 % of the mean, whichever is greater	0,2 ml or 10 % of the mean, whichever is greater

12 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the result of the test (see clause 10);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

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