BSI

BS 2882 : 1980
ISO 3734-1976

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Method for Determination of water and sediment in crude petroleum and fuel oils (centrifuge method)

ISO title : Crude petroleum and fuel oils — Determination of water and sediment — Centrifuge method

Technically equivalent to method ASTM D 1796-68 : IP 75/69

Méthode de détermination de la teneur en eau et en sédiments du pétrole brut et des fuel-oils (méthode par centrifugation)

Methode zur Bestimmung des Wasser- und Bodensatzgehaltes von Roherdöl und Heizölen (Zentrifugalmethode)

British Standards Institution

Gr 3

Contents

National foreword

This British Standard, which is published under the direction of the Petroleum Standards Committee, was first published in 1957, with the title 'Method for determination of water and sediment in crude and fuel oils—Centrifuge method', as the result of an agreement between the British Standards Institution and the Institute of Petroleum, and was technically equivalent to IP 75/57.

The present revision is identical with ISO 3734-1976 and is technically equivalent to ASTM D 1796-68 :IP 75/69.

This method was prepared by Subcommittee 3, Static petroleum measurement, of Technical Committee 28, Petroleum products, of the International Organization for Standardization (ISO) as a result of discussions in which the United Kingdom participated.

The principal changes from the previous edition of BS 2882 are as follows.

(a) The substitution of toluene for 'benzole' as the solvent.

(b) The increase of the range of relative centrifugal force from 600 to 800.

(c) The addition of a metal heating block as an alternative to a liquid bath for heating the centrifuge tube.

(d) The inclusion of a requirement that the temperature of the test portion shall not be allowed to drop below 46 °C during centrifuging.

(e) The provision of precision data for water and sediment contents greater than 1.0 % and presentation of the precision in the form of continuous curves.

Terminology and conventions. The text of the international standard has been approved as suitable for publication, without deviation, as a British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Where the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

NOTE. *Textual error.* In clause **4**, **6.4** and table 2, the unit 'rev/min' should be read as 'r/min'.

Cross-references

International standard	British Standard
ISO 3733-1976	BS 4385 : 1980 Method for determination of water in petroleum products and bituminous materials (distillation method) (Identical)
ISO 3735-1975	BS 4382 : 1980 Method for determination of sediment in crude petroleum and fuel oils (extraction method) (Identical)

A suitable British Standard specification for toluene, corresponding to grade 2 of ISO 5272*, is BS 805/2 incorporated in BS 135, 458, 805 : 1977 'Specifications for benzene, xylenes and toluenes'.

CAUTION Attention is drawn to the Health and Safety at Work etc. Act, 1974, and the need for ensuring that the method of test specified in this standard is carried out with suitable precautions.

The procedure described in this standard method is intended to be carried out by qualified chemists or other suitably trained and/or supervised personnel. Normal safety precautions should be observed throughout the use of the method. Attention is drawn in the text to some specific hazards.

*ISO 5272 has been published since the publication of ISO 3734.

British Standard Method for

Determination of water and sediment in crude petroleum and fuel oils (centrifuge method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of water and sediment in crude petroleum and fuel oils by means of a centrifuging procedure.

NOTE – Experience has shown that with some types of oils it is difficult to obtain complete separation of the water and sediment with this method. When this situation is encountered ISO 3733 and ISO 3735 may be used.

The criterion for the abandonment of this method shall be failure to obtain either a clear interface or the repeatability or reproducibility given in clause 8.

2 REFERENCES

ISO 3733, *Petroleum products and bituminous materials – Determination of water – Distillation method.*

ISO 3735, *Crude petroleum and fuel oils – Determination of sediment – Extraction method.*

ISO 5272, *Toluene – Specifications.* [1)]

3 SOLVENT

3.1 Toluene, conforming to ISO 5272, grade 2, shall be used as the solvent, except as provided in 3.2. The solvent shall be water saturated at ambient temperature, but shall be free of suspended water. This may be accomplished by the addition of 2 ml of water to 1 000 ml of solvent. Shaking will aid saturation, but adequate settling time is necessary to ensure that the solvent is free from suspended water before use.

CAUTION – Toluene is toxic. In particular, take precautions to avoid breathing the vapour, and protect the eyes.

3.2 It is recognized that some oils may require the use of solvents other than toluene. Solvents agreed upon between the purchaser and seller may be used provided these do not contribute to the water and sediment.

3.3 Demulsifiers. A demulsifier may only be used in transactions where tests have demonstrated a need. If a demulsifier is required, the type and amount to be used shall be agreed between the purchaser and seller.

4 APPARATUS

4.1 Centrifuge, capable of whirling two or more filled centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (rcf) of between 500 and 800 at the tips of the tubes. The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to prevent danger if any breakage occurs.

The centrifuge drive shall not be capable of causing ignitions of solvent vapour during the operation of the test.

Calculate the speed of the rotating head as follows :

$$\text{rev/min} = 1\,336\sqrt{\frac{\text{rcf}}{d}}$$

where

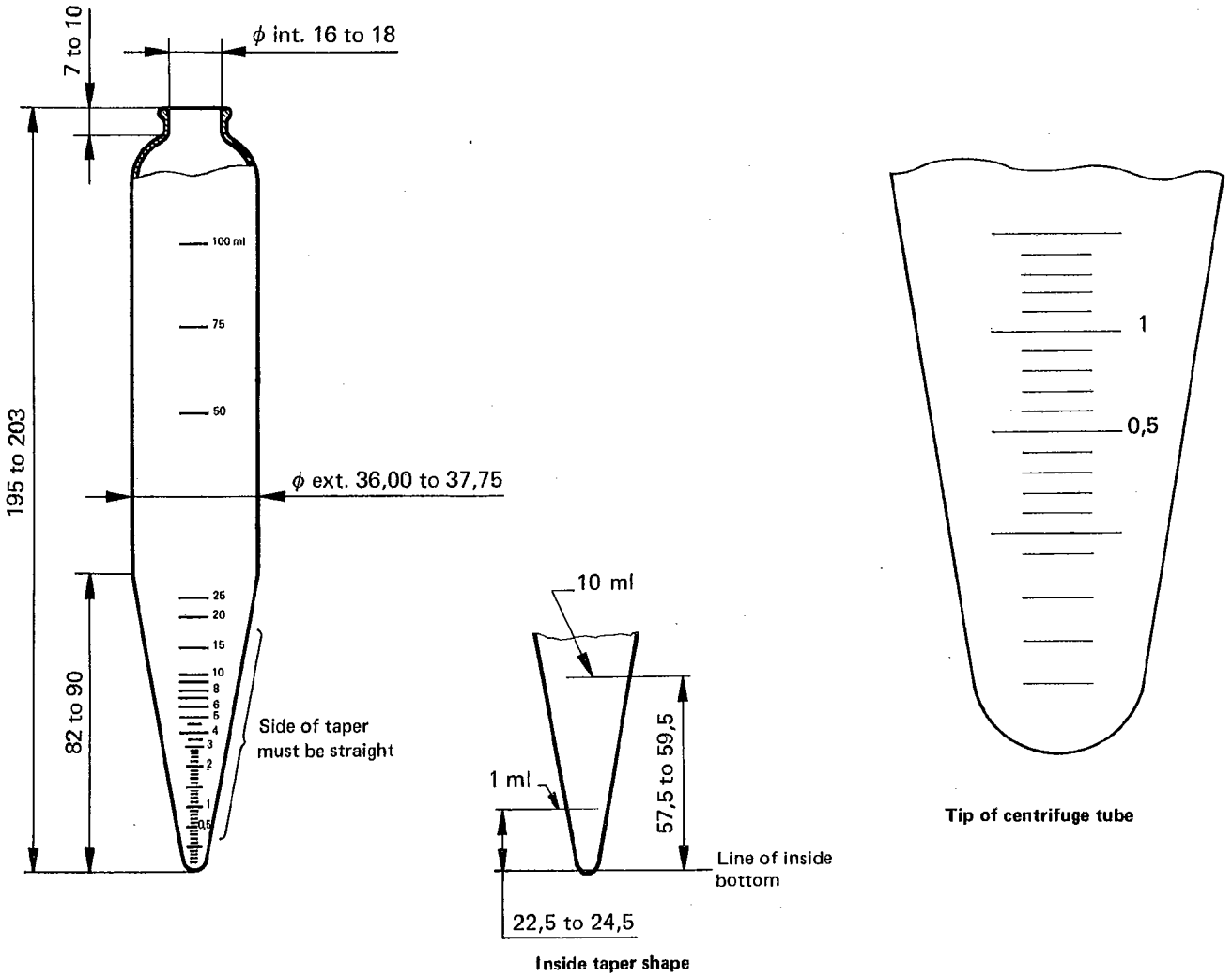
rcf is the relative centrifugal force;

d is the diameter of swing, i.e. twice the radius of swing, in millimetres, measured between axis of rotation and tip of tube when in rotating position.

The centrifuge may require a heating device to ensure that the temperature does not fall below 46 °C during centrifuging.

1) In preparation.

Dimensions in millimetres unless otherwise specified



NOTE – For volumetric tolerances see table 1.

FIGURE 1 – **Centrifuge tube**

4.2 Centrifuge tube, cone-shaped, conforming to the dimensions given in figure 1 and made of thoroughly annealed glass. The graduations, numbered as shown in figure 1, shall be clear and distinct, and the mouth constricted in shape for closure with a cork. Scale error tolerances and smallest graduations between various calibration marks are given in table 1, and apply to calibrations made with freshly boiled water at 20 °C, reading the bottom of a shaded meniscus.

4.3 Bath : either a solid metal block bath or a liquid bath of sufficient depth for immersing the centrifuge tube, in a vertical position, to the 100 ml mark. Means shall be provided for maintaining temperatures of 50 ± 1 °C and 60 ± 1 °C.

TABLE 1 – **Centrifuge tube calibration tolerances**

Range ml	Sub-division ml	Volume tolerance ml
0 to 0,1	0,05	± 0,02
Above 0,1 to 0,3	0,05	± 0,03
Above 0,3 to 0,5	0,05	± 0,05
Above 0,5 to 1,0	0,10	± 0,05
Above 1,0 to 2,0	0,10	± 0,10
Above 2,0 to 3,0	0,20	± 0,10
Above 3,0 to 5,0	0,5	± 0,20
Above 5,0 to 10	1,0	± 0,50
Above 10 to 25	5,0	± 1,00
Above 25 to 100	25	± 1,00

5 SAMPLING

The sample shall be thoroughly representative of the material to be tested and the test portion shall be thoroughly representative of the whole sample. Vigorously agitate the sample immediately before transferring the test portion to the tube. Cold samples of oils should be warmed to facilitate mixing. The difficulites in obtaining representative samples for this determination are unusually great; hence, the importance of sampling cannot be too strongly emphasized.

6 PROCEDURE

6.1 Fill each of two centrifuge tubes (4.2) to the 50 ml mark with water-saturated solvent; then immediately pour the well-shaken sample directly from the sample container into the centrifuge tubes until the total volume in each tube is 100 ml (see note). If a demulsifier is required, add the appropriate amount at this point. The centrifuge tubes and contents shall be weighed and balanced to within 0,5 g by attaching masses, for example copper wire, to the outside of the lighter tube; balancing by addition of solvent to the lighter tube shall not be used. Stopper the tubes tightly and shake vigorously until the contents are thoroughly mixed. Immerse the tubes to the 100 ml mark for 10 min in the bath maintained at the appropriate temperature specified in 6.2.

NOTE — The volume of both the test portion of oil and the solvent shall be read at the top of the meniscus.

6.2 The bath temperature shall be regulated so as to give the condition as appropriate in a) or b) below :

a) in the case of wax-free oils, the temperature of the sample at the commencement of each centrifuging shall be 50 ± 1 °C;

b) in the case of waxy oils and oils having a pour point above 10 °C, the temperature of the sample at the commencement of each centrifuging shall be 60 ± 1 °C.

The temperature of the test portion during centrifuging shall not be allowed to fall below 46 °C in either case a) or case b).

6.3 Remove the tubes from the bath and invert them to ensure that the contents are uniformly mixed.

CAUTION — A large increase in pressure may develop in the tube due to the increase of vapour pressure of the solvent with rise in temperature.

6.4 Place the tubes in trunnion cups on opposite sides of the centrifuge to establish a balance condition and whirl for 10 min at a rate, calculated from the equation given in 4.1, sufficient to produce a relative centrifugal force (rcf) of between 500 and 800 at the tip of the whirling tubes (see table 2 for the relationship between diameter of swing, rcf and rev/min). Read and record the combined volume of water and sediment at the bottom of each tube to the nearest 0,05 ml from 0,1 to 1 ml graduation lines and to the nearest 0,1 above 1 ml graduation line. Below 0,1 ml, estimate to the nearest 0,025 ml (see figure 1). Return the tubes without agitation to the centrifuge and whirl for 10 min at the same rate. Repeat this operation until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two whirlings are required.

NOTE — In order to avoid the danger of tubes breaking in the cups, care must be taken that the tubes are bedded onto the bottom cushion so that no part of the tube is in contact with the rim of the cup.

TABLE 2 — **Rotation speeds applicable for centrifuges of various diameters of swing**

Diameter of swing mm	rev/min at 500 rcf	rev/min at 800 rcf
450	1 410	1 780
500	1 340	1 690
550	1 280	1 610
600	1 235	1 542

7 EXPRESSION OF RESULTS

7.1 Record the final volume of water and sediment in each tube. If the difference between the two readings is greater than one sub-division on the centrifuge tube (see table 1), or 0,025 ml for readings of 0,10 ml and below, the readings are inadmissible and the determination shall be repeated.

7.2 Express the sum of the two admissible readings (see 7.1) as the percentage by volume of water and sediment; report the results as shown in table 3.

TABLE 3 — **Expression of results**

Reading in ml			Percentage by volume of water and sediment
Tube 1	Tube 2	Total	
No visible water and sediment	No visible water and sediment	—	0 (Zero)
No visible water and sediment	0,025	0,025	0,05
0,025	0,025	0,05	0,05
0,025	0,05	0,075	0,10
0,05	0,05	0,10	0,10
0,05	0,075	0,125	0,15
0,075	0,075	0,15	0,15
0,075	0,10	0,175	0,20
0,10	0,10	0,20	0,20
0,10	0,15	0,25	0,25
etc.			

8 PRECISION

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows :

8.1 Repeatability

The difference between successive 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 for repeatability shown in figure 2 only in one case in twenty.

8.2 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 for reproducibility shown in figure 2 only in one case in twenty.

9 TEST REPORT

The rest report shall include the following information :

a) reference to this International Standard;

b) results of test, in accordance with clause 7;

c) solvent used, if other than toluene;

d) name and amount of demulsifier, if used;

e) bath temperature.

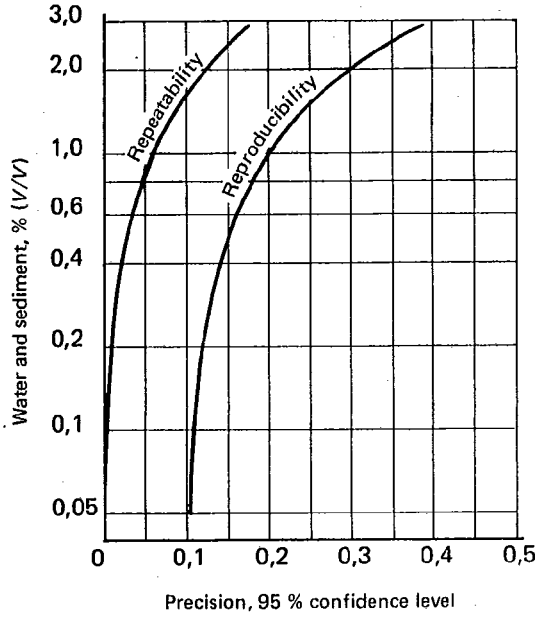


FIGURE 2 — **Precision curves**

Standards publications referred to

See national foreword.

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This British Standard, having been prepared under the direction of the Petroleum Standards Committee, was published under the authority of the Executive Board and comes into effect on 30 June 1980.

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Attention is drawn to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

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The following BSI references relate to the work on this standard :
Committee reference PTC/-Draft for comment 74/54300 DC

Cooperating organizations

The Petroleum Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Chemical Industries Association
British Lubricants Federation Limited
British National Oil Corporation
*Department of Industry (National Engineering Laboratory)
Hydrocarbon Solvents Association
*Institute of Petroleum
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The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Gas Corporation
Institution of Gas Engineers
Liquefied Petroleum Gas Industry Technical Association (UK)

Amendments issued since publication

Amd. No.	Date of issue	Text affected

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