INTERNATIONAL STANDARD

ISO 9030

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Crude petroleum — Determination of water and sediment — Centrifuge method

Pétrole brut — Détermination de la teneur en eau et en sédiments — Méthode par centrifugation

Reference number
ISO 9030:1990(E)

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.

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.

International Standard ISO 9030 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annexes A and B form an integral part of this International Standard.

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Crude petroleum — Determination of water and sediment — Centrifuge method

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

1 Scope

This International Standard specifies a method for the laboratory determination of water and sediment in crude oils by means of a centrifuge procedure. The precision data have only been determined for water contents up to 1 % (V/V).

NOTE 1 It has been observed that centrifugal methods of determination of water and sediment may, in many cases, give erroneous results. This is especially so when use of a high-speed mixer has been employed to obtain a representative sample. The method is therefore not entirely satisfactory and the amount of water determined is almost always lower than the actual water content.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

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

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity.*

ISO 5272:1979, *Toluene for industrial use — Specifications.*

ISO 9029:1990, *Crude petroleum — Determination of water — Distillation method.*

3 Significance

A knowledge of the water content of crude oil is important in the refining, purchase, sale and transfer of products.

The amount of water as determined by this method is used to correct the volume involved in the custody transfer of oil.

4 Principle

Equal volumes of crude oil and water-saturated toluene are placed in a cone-shaped centrifuge tube. After centrifugation, the volume of the higher-density water and sediment layer at the bottom of the tube is read.

5 Apparatus

Usual laboratory apparatus, together with the following:

5.1 Centrifuge.

5.1.1 The centrifuge shall be capable of spinning two or more filled, cone-shaped 203 mm centrifuge tubes at a speed that can be controlled to give a relative centrifugal force (rcf) of a least 600 at the tip of the tubes (see 5.1.5).

5.1.2 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 in a metal shield or case strong enough to eliminate danger if any breakage occurs.

5.1.3 The centrifuge shall be heated so that the sample temperature can be maintained at 60 °C ± 3 °C during the entire run (see 8.7). If a thermostatic control is provided, it shall be capable of maintaining the temperature within these limits and operate safely if there is a flammable atmosphere.

5.1.4 Electric-powered and heated centrifuges shall meet all safety requirements for use in hazardous areas.

5.1.5 The necessary minimum speed of the rotating head in revolutions per minute (rpm) is calculated as follows:

$$\text{rpm} = 1\,335\sqrt{\frac{\text{rcf}}{d}}$$

where

rcf is the relative centrifugal force;

d is the diameter of swing, in millimetres, measured between the tips of opposite tubes when in the rotating position.

5.2 Centrifuge tubes.

5.2.1 Each centrifugal tube shall be a 203 mm cone-shaped tube, 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 shall be constricted in shape for closure with a cork. Scale-error tolerances and the smallest graduations between various calibration marks are given in table 1 and apply to calibration made with air-free water at 20 °C, when reading the bottom of the meniscus.

5.2.2 The accuracy of the graduations on the centrifuge tube shall be volumetrically verified, in accordance with ISO 4787, before use of the tube. The verification shall include calibration at each mark up to the 0,25 ml mark (see figure 2), and at the 0,5, 1,0, 1,5, 2,0, 50,0 and 100 ml marks. The tube shall not be used if the scale error at any mark exceeds the applicable tolerance from table 1.

5.3 Bath.

The bath shall be either a solid-metal-block bath or a liquid bath of sufficient depth for immersing the centrifuge tube in the vertical position to the 100 ml mark. Means shall be provided for maintaining the temperature at 60 °C ± 3 °C.

NOTE 2 By agreement between parties, a temperature of 49 °C ± 3 °C may be used, but the precision achieved may not be the precision indicated in this International Standard.

6 Reagents

6.1 Toluene, conforming with the requirements for grade 1 toluene as specified in ISO 5272.

The solvent shall be water-saturated at 60 °C ± 3 °C (see note) but shall be free of suspended water (see annex B for the solvent/water saturation procedure).

NOTE 3 By agreement between parties, a temperature of 49 °C ± 3 °C may be used, but the precision achieved may not be the precision indicated in this International Standard.

6.2 Demulsifier, to promote the separation of water from the test portion and to prevent its clinging to the walls of the centrifuge tube. The recommended stock solution is 25 % demulsifier to 75 % toluene. For some crude oils, a different ratio of demulsifier to toluene may be required.

Demulsifiers used in the concentration and quantity given in this International Standard will not add to the water and sediment volume determined. Store the solution in a dark bottle that is tightly closed.

The type of demulsifier, and the concentration and quantity used, shall be agreed between the parties.

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 into the laboratory test container.

7.2 Laboratory sample

Only representative samples obtained as specified in ISO 3170 or ISO 3171 shall be used in this International Standard. Before taking a test portion from the sample received by the laboratory, homogenize the sample using the procedure described in annex A.

8 Procedure

8.1 Fill each of two centrifuge tubes (5.2) to the 50 ml mark with sample directly from the sample container. Then, with a pipette, add 50 ml of toluene (6.1), which has been water-saturated at 60 °C (see note). Read the top of the meniscus at both the 50 ml and 100 ml marks. Add 0,2 ml of demulsifier solution (6.2) to each tube, using a 0,2 ml pipette or an automatic pipettor. Stopper the tubes tightly and invert the tubes ten times to ensure the oil and solvent are uniformly mixed.

NOTE 4 By agreement between parties, a temperature of 49 °C ± 3 °C may be used, but the precision achieved may not be the precision indicated in this International Standard.

8.2 If the crude oil is very viscous and mixing of the solvent with the oil is difficult, the solvent may be added to the centrifuge tube first to facilitate mixing. Take care not to fill the centrifuge tube past the 100 ml mark with the sample.

8.3 Loosen the stoppers slightly and immerse the tubes to the 100 ml mark for at least 15 min in the bath (5.3) maintained at 60 °C ± 3 °C.

NOTE 5 By agreement between parties, a temperature of 49 °C ± 3 °C may be used, but the precision achieved may not be the precision indicated in this International Standard.

Secure the stoppers and again invert the tubes ten times to ensure uniform mixing of oil and solvent.

CAUTION — The vapour pressure at 60 °C is approximately double that at 40 °C.

8.4 Place the tubes in the trunnion cups on opposite sides of the centrifuge (5.1) and establish a balanced condition. (If the tubes cannot be counter-balanced by eye, place them, in their trunnion cups, on either side of a balance and equalize their masses by the addition of water to the trunnion cups.) Retighten the corks and spin for 10 min at a minimum relative centrifugal force of 600 as calculated using the equation given in 5.1.5.

8.5 Immediately after the centrifuge comes to rest following the spin, read and record the combined volume of water and sediment at the bottom of each tube. Take this reading to the nearest 0,05 ml for graduations from 0,1 ml to 1 ml and to the nearest 0,1 ml for graduations above 1 ml. Below 0,1 ml, estimate the reading to the nearest 0,025 ml (see figure 2). Return the tubes without agitation to the centrifuge and spin for another 10 min at the same rate.

8.6 Repeat this operation until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two spinnings are required.

8.7 Maintain the temperature of the sample at 60 °C ± 3 °C during the entire centrifuging procedure.

NOTE 6 By agreement between parties, a temperature of 49 °C ± 3 °C may be used, but the precision achieved may not be the precision indicated in this International Standard.

CAUTION — To avoid the danger of tubes breaking in the cups, take care that the tubes are bedded on to the bottom cushion so that no part of the tube is in contact with the rim of the cup.

9 Expression of results

9.1 Record the final volume of water and sediment in each tube. If the difference between the two readings is greater than one subdivision 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.

9.2 Report as the result the sum of two admissible readings, expressed as the percentage by volume of water and sediment (see table 2).

Table 1 — Centrifuge-tube calibration tolerances

Range ml	Subdivision 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	25,0	± 1,00

Table 2 — Expression of results

Volume of water and sediment in tube 1 ml	Volume of water and sediment in tube 2 ml	Total percentage of water and sediment % (*V*/*V*)
No visible water and sediment	No visible water and sediment	0
No visible water and sediment	0,025	0,025
0,025	0,025	0,05
0,025	0,05	0,075
0,05	0,05	0,10
0,05	0,075	0,125
0,075	0,075	0,15
0,075	0,10	0,175
0,10	0,10	0,20
0,10	0,15	0,25

10 Precision

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

10.1 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 following values in only one case in twenty:

from 0,0 % (*V*/*V*) to 0,3 % (*V*/*V*) water and sediment: see figure 3;

from 0,3 % (*V*/*V*) to 1,0 % (*V*/*V*) water and sediment: 0,12 % (*V*/*V*).

10.2 Reproducibility

The difference between two single and independent 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 following values in only one case in twenty:

from 0,0 % (*V*/*V*) to 0,3 % (*V*/*V*) water and sediment: see figure 3;

from 0,3 % (*V*/*V*) to 1,0 % (*V*/*V*) water and sediment: 0,28 % (*V*/*V*).

11 Test report

The test report shall contain at least the following information:

a) all details necessary for the identification of the product tested;

b) a reference to this International Standard;

c) the result of the test (see clause 9);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

Dimensions in millimetres

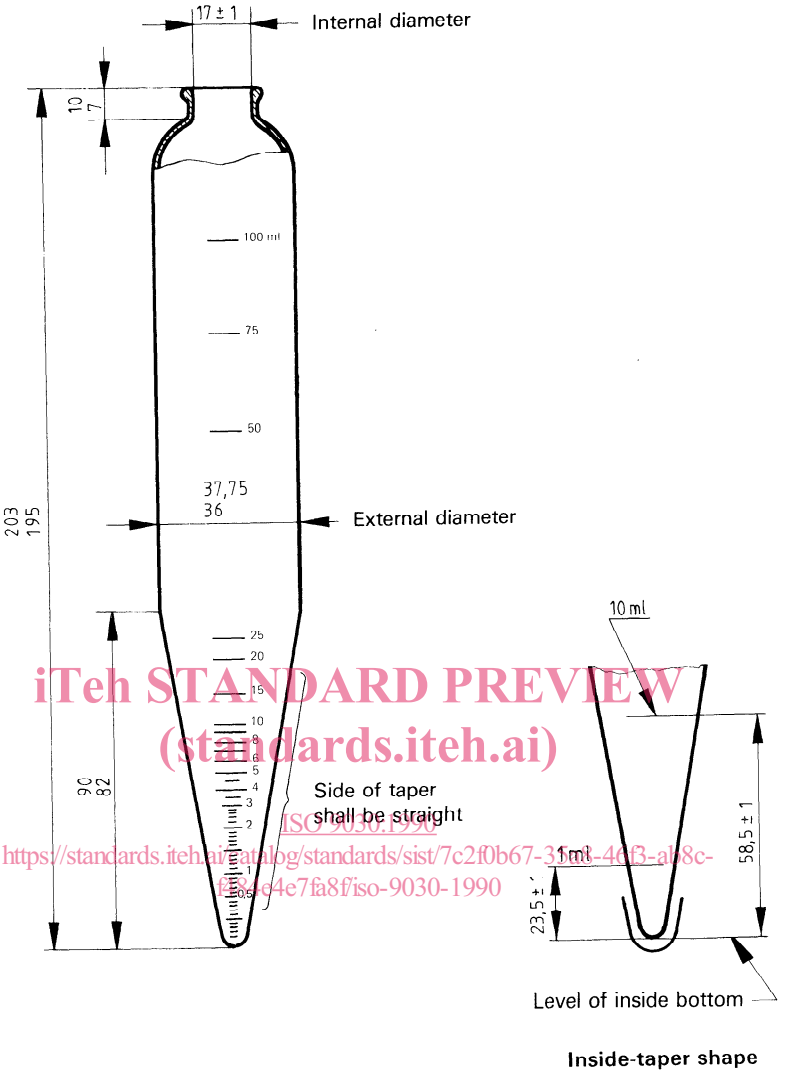

Figure 1 — 203 mm centrifuge tube

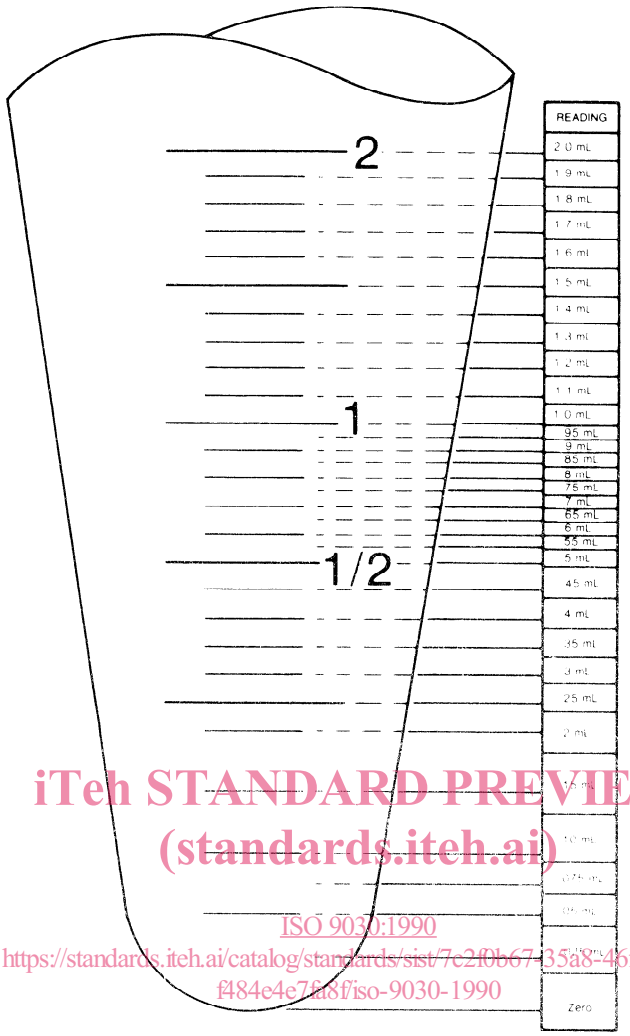

Figure 2 — Procedure for reading the volume of water and sediment at low levels

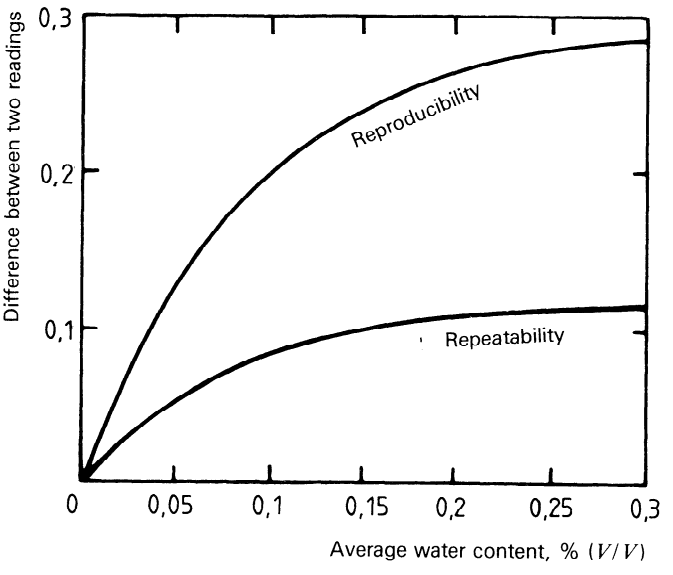

Figure 3 — Precision of the method for water and sediment contents within the range 0,0 % (*V*/*V*) to 0,3 % (*V*/*V*)