INTERNATIONAL STANDARD

ISO 3735

Second edition
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Crude petroleum and fuel oils — Determination of sediment — Extraction method

Pétrole brut et fuel-oils — Détermination de la teneur en sédiments — Méthode par extraction

Reference number
ISO 3735:1999(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 3735 was developed by Technical Committee TC 28, *Petroleum products and lubricants*, Subcommittee 6, *Bulk cargo transfer, accountability, inspection and reconciliation*.

This second edition cancels and replaces the first edition (ISO 3735:1975), of which it constitutes a technical revision.

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Crude petroleum and fuel oils — Determination of sediment — Extraction 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 limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of sediment in crude petroleum and fuel oils by extraction with toluene. The precision applies to a range of sediment levels from 0,01 % (m/m) to 0,40 % (m/m), although higher levels may be determined.

NOTE 1 If this International Standard is applied to crude petroleum samples containing significant amounts of salts, an overestimation of the sediment content may be obtained because a proportion of the inorganic salts may be trapped in the extraction thimble. This problem is generally not significant for crude petroleum samples containing less than 0,1 % (m/m) total salts.

NOTE 2 For the purposes of this International Standard, the terms "% (m/m)" and "% (V/V)" are used to represent the mass and volume fractions of materials, respectively.

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 1773:1997, *Laboratory glassware — Narrow-necked boiling flasks.*

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

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

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

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

3 Principle

A test portion, in a refractory thimble, is extracted with hot toluene until the residue reaches constant mass.

4 Reagents and materials

4.1 Methylbenzene (toluene), ($C_6H_5CH_3$), conforming to grade 2 of ISO 5272.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Extraction apparatus, as illustrated in figures 1 and 2, and consisting of the elements described in 5.1.1 to 5.1.6.

5.1.1 Extraction flask, consisting of a wide-necked conical (Erlenmeyer) flask of 1 litre capacity, conforming generally to the requirements of ISO 1773, but with a minimum external neck diameter of 50 mm.

5.1.2 Condenser, in the form of a metal coil approximately 25 mm in diameter and 50 mm in length attached to, and with the ends projecting through, a lid of sufficient diameter to cover the neck of the flask as shown in figure 1. The coil shall be made from stainless steel, tin, tin-plated copper or tin-plated brass tubing having an outside diameter of 5 mm to 8 mm and a wall thickness of approximately 1,5 mm. If constructed of tin-plated copper or brass, the tin coating shall have a minimum thickness of 0,075 mm.

NOTE The exposed area of the coil for cooling purposes is approximately 115 cm^2.

5.1.3 Extraction thimble, of a refractory porous material, pore size index P 16 according to ISO 4793, 25 mm in diameter by 70 mm in height, weighing not less than 15 g and not more than 17 g. The thimble shall be suspended from the condenser coil by means of a basket so that it hangs approximately mid-way between the surface of the extracting solvent (4.1) and the bottom of the condenser coil (see 5.1.2).

5.1.4 Thimble basket, corrosion-resistant, made of platinum, stainless steel, nickel-chromium alloy, or similar material. It shall meet the design and dimension requirements of figure 2.

5.1.5 Water cup, for use when testing a sample with a water content in excess of 10 % (*V/V*) [see figure 1b)]. The cup shall be made of glass, conical in shape, approximately 20 mm in diameter and 25 mm deep, having a capacity of approximately 3 ml. A glass hook shall be fused onto the rim on one side and shaped so that, when hung on the condenser, the cup hangs with its rim reasonably level.

In these procedures, the thimble basket is suspended either as shown in figure 1a), by means of the corrosion-resistant wire looped over the bottom of the condenser coil and attached to the basket supports, or as in figure 1b), where the wire supports of the basket are attached to hooks soldered to the underside of the condenser lid.

5.1.6 Heater, suitable for vaporizing the toluene.

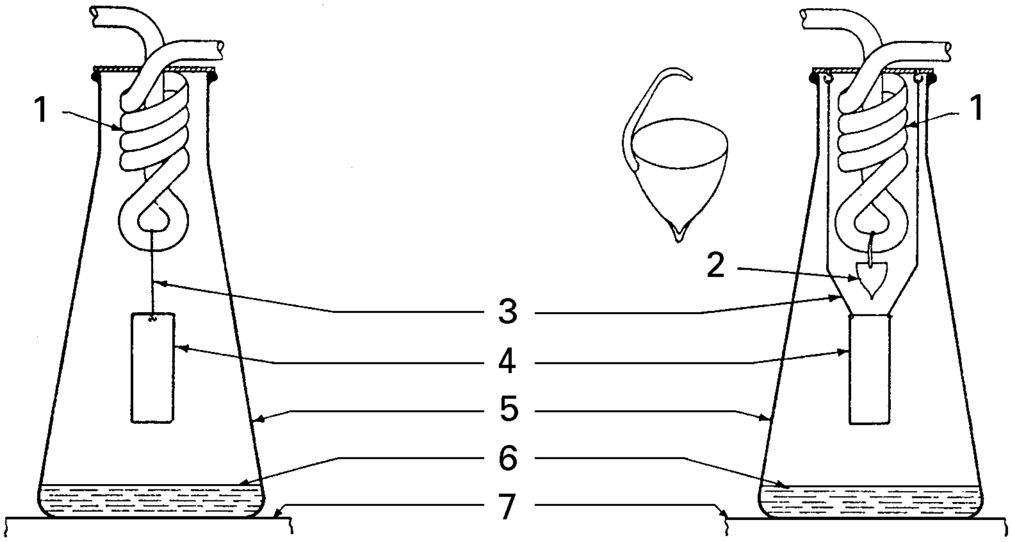
NOTE A hotplate is preferred.

5.2 Analytical balance, with an accuracy of 0,1 mg.

5.3 Non-aerating mixer, meeting the verification of mixing efficiency requirements specified in ISO 3170 and ISO 3171.

NOTE Both insertion mixers and circulating external mixers are acceptable, provided they meet the above criteria.

5.4 Oven, capable of maintaining a temperature of 115 °C to 120 °C.



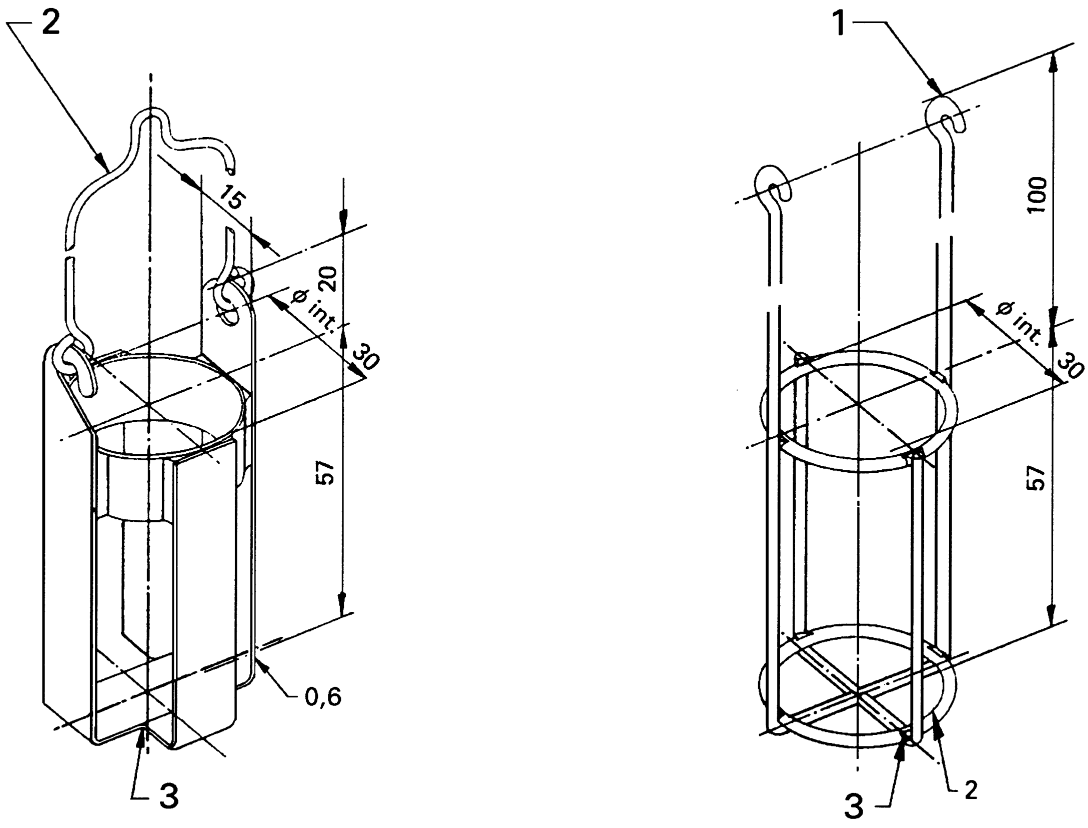
a) **Water content ⩽ 10 %** (*V*/*V*) b) **Water content > 10 %** (*V*/*V*)

Key

1 Condenser
2 Water cup
3 Wire
4 Extraction thimble in basket
5 Extraction flask
6 Solvent
7 Top of hotplate

Figure 1 — Extraction apparatus showing in b) the water cup in position

Dimensions in millimetres



a) Ribbon support

b) Wire support

Key

1 Attach to hooks on underside of condenser lid
2 Attach through condenser loop
3 All joints are silver soldered

Figure 2 — Thimble basket support

5.5 Cooling vessel, consisting of a desiccator without desiccant.

5.6 Thermometer, capable of measuring the temperature of the sample to the nearest 1 °C.

6 Samples and sampling

6.1 Unless otherwise specified, samples shall be taken by the procedures specified in ISO 3170 or ISO 3171.

6.2 Test portions from the laboratory samples shall be drawn immediately after thorough mixing. Heat viscous samples to a temperature which renders the sample liquid, and homogenize, using the mixer (5.3) as necessary (see 6.3).

The difficulties of obtaining a representative test portion for this determination are unusually great, hence the need for great attention to the mixing and aliquoting stages.

6.3 Record the temperature of the sample before mixing. Mix the laboratory sample in its original container, using the mixing time, mixing power (speed) and position relative to the bottom of the container appropriate to the crude petroleum or fuel oil being analysed, and as established by the verification of mixing efficiency requirements given in ISO 3170 and ISO 3171. To avoid the loss of light ends from crude petroleum or other samples containing volatile material, operate the mixer through a gland in the closure of the sample container. Take the test portion for analysis immediately after mixing. Record the temperature of the sample immediately after mixing. If the rise in temperature during mixing exceeds 10 °C, cool the sample, and repeat the mixing at a lower energy input.

NOTE A rise in temperature of greater than 10 °C may result in a decrease of viscosity that is sufficient for the sediment to settle.

7 Selection of thimbles and preparation

7.1 Selection of thimbles

Select either a new or used thimble for the test. Requirements for new thimbles are given in 5.1.3.

New thimbles shall be used in the event of dispute.

NOTE Used thimbles may be employed when carrying out routine tests. When reusing thimbles, the extraction to constant mass for one determination should be considered as the preliminary extraction for the succeeding determination. After several determinations, the accumulated sediment may be sufficient to interfere with further determinations, and the procedure described in 7.3 should then be followed.

7.2 Preparation of a new thimble

Rub the outside surface of the thimble with fine sandpaper and remove all loosened material with a stiff brush. Place the thimble in the extraction flask (5.1) and extract with toluene (4.1), allowing the solvent to drip from the thimble for at least 1 h. Dry the thimble for 1 h in the oven (5.4), cool in the cooling vessel (5.5) for 1 h , and weigh to the nearest 0,1 mg. Repeat the extraction and cooling steps until the masses of the thimble after two successive extractions do not differ by more than 0,2 mg.

7.3 Preparation of a used thimble

Remove the combustible portion of the accumulated sediment by heating the thimble to dull red heat for 20 min. Extract, cool and weigh to constant mass by following the procedure described in 7.2.

NOTE An electric furnace maintained at approximately 750 °C is suitable for heating the thimble.

8 Procedure

8.1 Place a test portion of approximately 10 g in the thimble (5.1.3) immediately after the laboratory sample has been mixed. Do not attempt to adjust the estimated 10 g portion to any exact predetermined mass.

Weigh the thimble plus test portion to the nearest 0,01 g. Add 150 ml to 200 ml of toluene (4.1) to the flask, place the thimble in the extraction apparatus (5.1), place on the heater (5.1.6) and extract with hot toluene for 30 min after the solvent dripping from the thimble becomes colourless.

Ensure that the rate of extraction is such that the surface of the mixture of oil and toluene in the thimble does not rise higher than to 20 mm of the top.

8.2 When the water content of the sample is known or suspected to be above 10 % (V/V), use the assembly illustrated in figure 1b).

NOTE In this procedure, any water in the test portion is removed as its toluene azeotrope and collects in the water cup, where it separates as a bottom layer. The toluene layer overflows into the thimble.

If the cup becomes full of water, allow the apparatus to cool and empty the cup.

8.3 After the extraction is completed, dry the thimble for 1 h in the oven (5.4), cool for 1 h in the cooling vessel (5.5), and weigh to the nearest 0,1 mg.

8.4 Repeat the extraction, allowing the solvent to drip from the thimble for at least 1 h but not longer than 1 h 15 min. Then dry, cool and weigh the thimble as described in 8.3. Repeat this extraction for further 1 h periods, if necessary, until the masses of the dried thimble plus sediment after two successive extractions do not differ by more than 0,2 mg.

9 Calculation

Calculate the sediment content of the sample, S, as a percentage by mass, using the equation:

$$S = \frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the thimble;

m_2 is the mass, in grams, of the thimble plus test portion;

m_3 is the mass, in grams, of the thimble plus sediment.

10 Expression of results

Report the sediment content of the sample, as “sediment by extraction”, as a percentage by mass, rounded to the nearest 0,01 % (m/m).

Report that “the sample mixing procedure was performed in accordance with the procedures specified in ISO 3170 or ISO 3171”.

Report the temperature of the sample before and after mixing.

11 Precision

11.1 General

The precision of the method, as obtained by statistical examination of interlaboratory test results of a matrix of samples with sediment contents in the range of 0,01 % (m/m) to 0,40 % (m/m), is given in 11.2 and 11.3.

11.2 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 value below only in one case in 20.

$$r = 0{,}017 + 0{,}255\,\overline{S}$$

where $\overline{S}$ is the average result, as a percentage by mass, of the values being compared.

11.3 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 value below only in one case in 20.

$$R = 0{,}033 + 0{,}255\ \overline{S}$$

where $\overline{S}$ is the average result, as a percentage by mass, of the values being compared.

12 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the product tested;

c) the results 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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