INTERNATIONAL STANDARD 3735

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

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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 3735 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	Germany	Portugal
Austria	Ghana	Romania
Belgium	Hungary	South Africa, Rep. of
Brazil	India	Spain
Bulgaria	Iran	Sweden
Canada	Israel	Turkey
Chile	Japan	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
France	Poland	Yugoslavia

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of sediment in crude petroleum and fuel oils by extraction with toluene.

2 REFERENCES

ISO 4793, *Laboratory apparatus — Filters — Porosity grading.* [1)]

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

3 PRINCIPLE

A test portion, in a refractory thimble, is extracted with hot toluene until the residue reaches constant mass. The mass of residue, calculated as a percentage, is reported as "sediment by extraction".

4 SOLVENT

Toluene, conforming to ISO 5272, grade 2.

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

5 APPARATUS

Extraction apparatus (see figures 1 and 2), consisting of the following :

5.1 Extraction flask : a wide-neck conical flask of 1 litre capacity.

5.2 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 to 8 mm and a wall thickness of approximately 1,5 mm. The tin coating shall have a minimum thickness of 0,075 mm. The exposed surface of the coil for cooling purposes is about 115 cm^2.

5.3 Extraction thimble, of a refractory porous material, pore size index P 16 (see 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 and the bottom of the condenser coil.

5.4 Thimble basket, corrosion resistant, made of platinum, stainless steel, nickel-chromium alloy, or similar material and meeting the requirements of figure 2.

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

In this procedure the thimble basket is suspended either as shown in figure 1 a), 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 1 b), where the wire supports of the basket are attached to hooks soldered to the underside of the condenser lid.

5.6 Source of heat, suitable to vaporize toluene.

1) In preparation

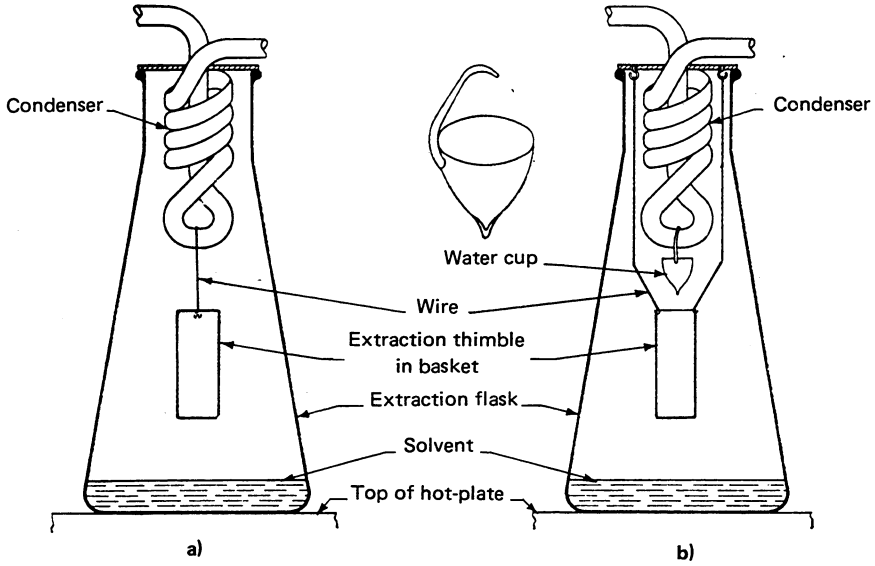
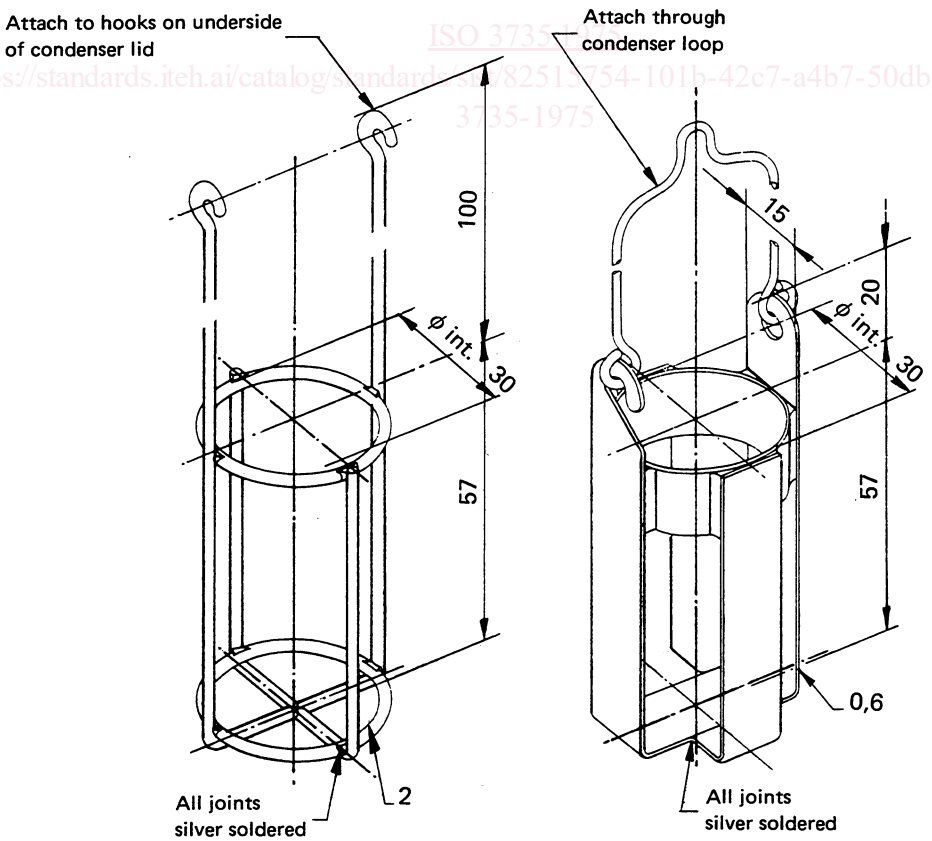


FIGURE 1 – **Extraction apparatus for determination of sediment showing in b) the water cup in position**

Dimensions in millimetres



a) **Wire support** b) **Ribbon support**

FIGURE 2 – **Basket thimble support**

6 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 thimble. Cold samples of oil should be warmed to facilitate mixing. The difficulties in obtaining representative samples for this determination are unusually great; hence, the importance of sampling cannot be too strongly emphasized.

7 PROCEDURE

7.1 For referee tests a new extraction thimble should be used.

For routine tests, thimbles may be used for a number of successive determinations on different samples, the extraction to constant mass for one determination being considered as the preliminary extraction for the succeeding determination. When the amount of accumulated sediment becomes objectionable, remove the combustible portion by heating to a dull red heat (preferably in an electric furnace) and subject the thimble to a preliminary extraction before using for another determination.

7.2 Before using a new thimble, rub the outside surface with fine sandpaper and remove all loosened material with a stiff brush. Give the thimble a preliminary extraction with the toluene, allowing the solvent to drip from the thimble for at least 1 h. Then dry the thimble for 1 h at a temperature of 115 to 120 °C, cool in a desiccator, without desiccant, for 1 h and weigh to the nearest 0,1 mg. Repeat this extraction until the masses of the thimble after two successive extractions do not differ by more than 0,2 mg.

7.3 Place an estimated 10 g test portion of the sample in the thimble as soon as possible after the sample has been thoroughly mixed. Do not attempt to adjust this estimated 10 g portion to any exact predetermined amount. Weigh to the nearest 0,01 g. Place the thimble in the extraction apparatus and extract with the toluene for 30 min after the solvent dropping from the thimble is 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 within 20 mm of the top.

7.4 When testing samples having a high water content, use the assembly shown in figure 1 b). 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.

7.5 After the extraction is completed, dry the thimble for 1 h at 115 to 120 °C, cool in a desiccator, without desiccant, for 1 h and weigh to the nearest 0,2 mg.

7.6 Repeat the extraction, allowing the solvent to drip from the thimble for at least 1 h but not longer than 1,25 h, dry, cool, and weigh the thimble as described in 7.5. 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.

8 CALCULATION

Calculate the mass of the sediment as a percentage of that of the original sample.

9 PRECISION

The precision of the method, as obtained by statistical examination of inter-laboratory test results in the range 0 to 0,4 %, is as follows :

9.1 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 value only in one case in twenty :

$$(0{,}017 + 0{,}255\,S)$$

where S is the average result in % (m/m).

9.2 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 following value only in one case in twenty :

$$(0{,}033 + 0{,}255\,S)$$

where S is the average result in % (m/m).

10 TEST REPORT

Report the results to the nearest 0,01 % as the percentage by mass of sediment by extraction. The test report shall make reference to this International Standard.