

INTERNATIONAL
STANDARD

ISO
10307-1

First edition
1993-09-15

**Petroleum products — Total sediment in
residual fuel oils —**

Part 1:

Determination by hot filtration
(standards.iteh.ai)

Produits pétroliers — Insolubles existants dans les fuel-oils résiduels —

Partie 1: Détermination par filtration à chaud
<https://standards.iteh.ai/catalog/standards/sist/6ef1eb7-9370-46e1-bd5r-df161919e800/iso-10307-1-1993>



Reference number
ISO 10307-1:1993(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 10307-1 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 10307 consists of the following parts, under the general title *Petroleum products — Total sediment in residual fuel oils*:

- Part 1: *Determination by hot filtration*
- Part 2: *Determination using standard procedures for ageing*

Petroleum products — Total sediment in residual fuel oils —

Part 1: Determination by hot filtration

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. The 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.

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1 Scope

This part of ISO 10307 specifies a method for the determination of total sediment in residual fuel oils having a maximum viscosity of 55 mm²/s at 100 °C, and for blends of distillate fuels containing residual components. The maximum total sediment covered by the precision evaluations of this method is 0,50 % (m/m) for residual fuels, and 0,40 % (m/m) for distillate fuels containing residual components. Some fuels may exceed the maximum filtration time specified in this method due to factors other than the presence of significant quantities of insoluble organic or inorganic material.

Sediment insoluble in toluene may be determined by ISO 3735.

NOTES

1 The method described in this part of ISO 10307 may be used for the assessment of total sediment after regimes of fuel pre-treatment designed to accelerate the ageing process (see part 2 of this International Standard).

2 Appreciable amounts of sediment in a residual fuel oil can cause fouling of facilities for handling, and give problems in burner mechanisms. Sediment may accumulate in storage tanks, on filter screens or on burner parts, resulting in obstruction to flow of oil from the tank to the burner.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10307. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10307 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 3735:1975, *Crude petroleum and fuel oils — Determination of sediment — Extraction method*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*.

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

3 Definition

For the purposes of this part of ISO 10307, the following definition applies.

3.1 total sediment: The sum of the insoluble organic and inorganic material which is separated from the bulk of the sample by filtration through a specified filter, and which is also insoluble in a predominantly paraffinic solvent.

4 Principle

An aliquot of the oil sample is filtered through the prescribed apparatus at 100 °C, and after solvent washing and drying the total sediment on the filter is weighed. The test is carried out in duplicate.

5 Materials

During the analysis, unless otherwise stated, use only reagents specified in ISO 6353-2 and ISO 6353-3, if listed there. If not, then use reagents of recognized analytical grade.

5.1 Heptane, $\text{CH}_3(\text{CH}_2)_5\text{CH}_3$.

WARNING — Heptane is a toxic volatile hydrocarbon, and shall only be used with adequate ventilation. Avoid inhalation of vapour or mist and prolonged skin contact.

5.2 Toluene, $\text{C}_6\text{H}_5\text{CH}_3$.

WARNING — Toluene is a toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through skin contact with the liquid. Use only in adequate ventilation and avoid skin contact.

5.3 Wash solvent, consisting of 85 % (V/V) heptane (5.1), and 15 % (V/V) toluene (5.2).

6 Apparatus

6.1 Filtration apparatus, an example of which is shown in figure 1, and the layout in figure 2. The construction is of brass, with copper steam coils attached, suitably supported, above a vacuum flask appropriately protected against the effects of implosion.

NOTE 3 Other apparatus configurations have been shown to be satisfactory, provided that the dimensional requirements and heating-medium capacity are strictly adhered to.

6.2 Thermometer, partial-immersion type, with immersion line at 50 mm to 80 mm, and a maximum length of 220 mm. The thermometer shall be able to record the temperature over the range 95 °C to 105 °C with a maximum graduation interval of 0,5 °C.

6.3 Electric oven, capable of maintaining a temperature of 110 °C \pm 1 °C. The oven shall be capable of evaporating the solvent without risk of fire or explosion.

6.4 Stirring rod, glass or PTFE (polytetrafluoroethylene), approximately 150 mm in length \times 3 mm in diameter.

6.5 Glass beaker, 30 ml capacity, either squat form with lip or conical.

6.6 Weighing bottles, with ground-glass stoppers, numbered, 80 mm in diameter \times 40 mm in height.

6.7 Electric hotplate, or other suitable heating device.

6.8 Steam-generator, to provide a source of steam at 100 °C \pm 1 °C. Alternative heating media to steam are acceptable where steam is either not available or not available at the specified temperature.

6.9 Vacuum source, capable of providing the specified absolute pressure of 40 kPa \pm 2 kPa (61,3 kPa vacuum).

6.10 Vacuum gauge, capable of recording the absolute pressure or vacuum specified in 6.9.

6.11 Glass-fibre filters, nominal porosity 0,001 6 mm, diameter 47 mm.

NOTE 4 A suitable type is the GF/A, manufactured by Whatman. Equivalent types are also suitable.

6.12 High-speed mixer, of any convenient type with a minimum speed of 400 rev/min.

6.13 Desiccator, tightly covered, with desiccant.

6.14 Cooling vessel, such as a desiccator-type or other type of tightly covered vessel for cooling the filters before weighing. Drying agent shall not be used.

6.15 Graduated syringe or wash bottle, minimum capacity 25 ml, graduated at 0,5 ml intervals.

6.16 Forceps, spade-ended.

6.17 Balance, single or double-pan, with a sensitivity of 0,1 mg for weighing the filters. A second balance with a lower sensitivity of 10 mg may be used for weighing the sample.

7 Sample preparation

Mix the whole sample thoroughly using a high-speed mixer (6.12), if practicable, for 30 s. A sample taken on a glass or PTFE rod (6.4) dipped to the bottom of the container shall show a homogeneous appearance. For fuels with a high wax content (high pour point) or of very high viscosity, heat the sample before stirring. The temperature shall be either 15 °C to 18 °C above the pour point for low-viscosity fuels, or sufficient to reduce the viscosity to between 150 mm²/s and

250 mm²/s for high-viscosity fuels. The temperature shall not exceed 80 °C during this preparation stage.

8 Filter preparation

For each test, dry two filters (6.11) for 20 min in the oven (6.3) at 110 °C. Transfer each filter separately and rapidly to a numbered weighing bottle (6.6) and allow to cool in the cooling vessel (6.14) to room temperature (5 min to 10 min). If a two-pan balance is used (see 6.17), weigh each weighing bottle plus filter by the tare method against an empty similar bottle, to the nearest 0,000 1 g.

NOTES

5 For convenience, it is useful to have a number of weighing bottles dedicated to the procedure, the lightest of which is chosen as the tare.

6 It has been shown that the same level of result is achieved by the use of a fine wire mesh support screen (see figure 1) in combination with a third, disposable, filter. Place this third filter below the two test filters on the support screen and follow the same pre-drying regime as the test filters, but do not weigh the third filter before placement, and discard it when filtration is complete.

The glass-fibre filters are fragile, and need to be handled with care. Before use, check each against a background light for consistency and the possible presence of small defects (holes).

Store all weighing bottles in a desiccator in the vicinity of the balance.

Do not place weighing bottles in the oven.

9 Procedure

9.1 General

The determination shall be carried out in duplicate.

9.2 Assembly of apparatus

Before use, check that the filter support screen is clean, and if necessary clean it by boiling it in a high-boiling aromatic solvent such as toluene (5.2). Renew the filter support screen if more than 2 % of the sinter area (i.e. a significant number of pores visible to the naked eye) is blocked by a particulate content after such cleaning.

The filtration unit (6.1) shall be clean and dry before assembly. Stack the two previously dried and weighed filters on top of the sinter support with the mesh imprint side down using forceps (6.16), placing the one from the lower-numbered weighing bottle on the bottom. Apply slight vacuum to aid the centralization of the filters and place the top portion of the filtration apparatus carefully on to the filters before clamping. Shut off the vacuum suction and press

steam, or the alternative heating medium (see 6.8), at 100 °C ± 1 °C through the unit for 10 min prior to addition of the test portion and throughout the addition and filtration stages.

9.3 Addition of test portion

Pour into a 30 ml beaker (6.5) approximately 11 g of the residual fuel sample or approximately 10,5 g of the blended distillate fuel sample prepared as in clause 7, and weigh to the nearest 0,01 g (see note 7). Connect the vacuum source (6.9) and apply vacuum to an absolute pressure of 40 kPa ± 2 kPa (61,3 kPa vacuum). Transfer the contents of the beaker, unheated for blended distillate fuel or at 100 °C ± 2 °C for residual fuel (see note 8) to the centre of the filter, taking care that none of the test portion touches the walls of the filtration cell during transfer. Residual-fuel test portions which overheat to above 105 °C shall be discarded, and not re-used. Re-weigh the beaker to the nearest 0,01 g. The quantity transferred shall be 10 g ± 0,5 g.

For samples of high viscosity and/or high sediment level, filtration will be aided by small-stage or even dropwise addition. It is expedient to use the maximum filter area available, but care shall be taken to avoid unfiltered oil coming into contact with the walls of the filtration cell. For samples of low filtration rate, the pressure of 40 kPa ± 2 kPa shall be maintained for 25 min. If filtration is not complete in 25 min, discontinue the test and repeat the procedure using a 5 g ± 0,3 g test portion. If the second filtration is still not complete in 25 min, report the result as "filtration time exceeds 25 min".

NOTES

7 When testing residual fuels, it may be expedient to weigh the beaker plus stirring rod plus thermometer (6.2) before and after transfer, to avoid errors incurred by attempting to obtain a net mass.

8 Any convenient means of heating the test portion to 100 °C ± 2 °C may be used, such as a hotplate, a water or oil bath or an oven (see 6.7), if equipped with a suitable stirrer.

9.4 Filter washing

When the filtration is complete, and the upper filter appears dry, continue the steam and vacuum for a further 5 min. Discontinue the steam supply and cool the apparatus by passing tap water through the coils. Wash the upper filter carefully with two portions of 25 ml ± 1 ml of wash solvent (5.3) dispensed from a graduated syringe or a graduated wash bottle with a fine nozzle (6.15), taking care to remove any adhered sample from the wall of the upper part of the apparatus. If the test portion filters very rapidly, release the vacuum before the addition of the first portion of wash solvent, to ensure complete coverage of the

filter area by solvent. Then gently re-apply the vacuum for the subsequent operations.

Carefully remove the top portion of the filtration unit and wash the rim of the filter with a further 10 ml ± 0,5 ml of wash solvent in a similar manner. Finally, wash the whole of the filter area with 10 ml ± 0,5 ml of heptane (5.1).

9.5 Apparatus disassembly

When the upper filter appears dry, discontinue the vacuum. Using forceps, remove each filter separately and transfer them to the oven at 110 °C. Dry for 20 min and quickly transfer them to the same numbered weighing bottles as used in clause 8. Take care to place the lighter-coloured (i.e. the lower) filter back in the lower-numbered weighing bottle. Allow to cool in the cooling vessel to room temperature (5 min to 10 min), and re-weigh (against tare) to the nearest 0,000 1 g.

10 Expression of results

Calculate the mass percentage of total sediment to the nearest 0,01 % (*m/m*) using the equation:

$$S = \frac{(m_5 - m_4) - (m_3 - m_2)}{10m_1}$$

where

- S* is the total sediment, expressed as a percentage by mass;
- m*₁ is the mass, in grams, of the test portion;
- m*₂ is the mass, in milligrams, of the lower filter before filtration;
- m*₃ is the mass, in milligrams, of the lower filter after filtration;
- m*₄ is the mass, in milligrams, of the upper filter before filtration.
- m*₅ is the mass, in milligrams, of the upper filter after filtration.

Report the total sediment by hot filtration as the average of the duplicate determinations to the nearest 0,01 % (*m/m*). If a 5 g sample was used, report the result as "total sediment (5 g) by hot filtration". If filtration is not complete within the specified 25 min, report the result as "filtration time exceeds 25 min".

11 Precision

11.1 Repeatability

The difference between successive test results, expressed as the average of duplicate determinations,

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 below only in one case in twenty:

$$r = 0,123\sqrt{x} \text{ for residual fuels}$$

and

$$r = 0,048\sqrt{x} \text{ for distillate fuels containing residual components,}$$

where *x* is the average of the test results, expressed as a percentage by mass.

11.2 Reproducibility

The difference between two test results, expressed as the average of duplicate determinations independently obtained by different operators operating in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values below only in one case in twenty:

$$R = 0,341\sqrt{x} \text{ for residual fuels}$$

and

$$R = 0,174\sqrt{x} \text{ for distillate fuels containing residual components,}$$

where *x* is the average of the test results, expressed as a percentage by mass.

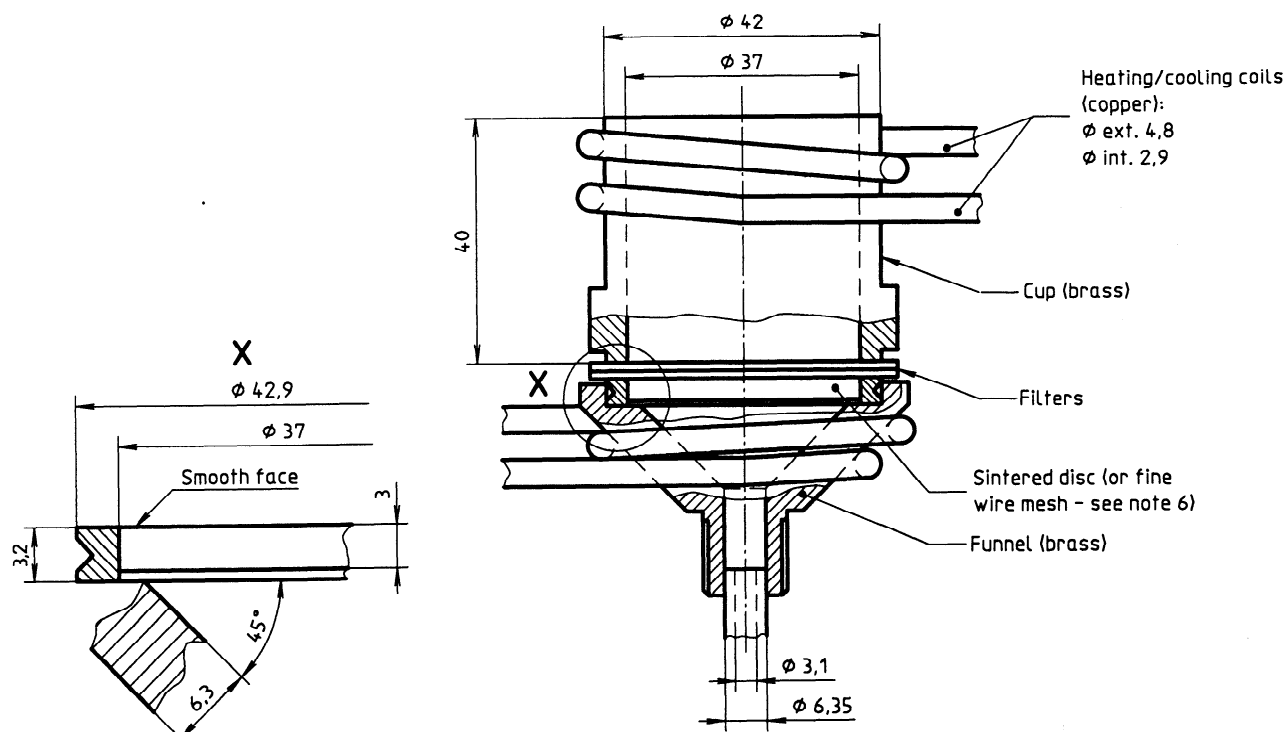
NOTE 9 These precision values have been obtained by statistical examination of the results of inter-laboratory tests on matrices of samples tested between 1986 and 1989, and were first published in 1990.

12 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 10307;
- b) all details necessary for the complete identification of the sample 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.

Dimensions in millimetres



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Figure 1 — Detail of filtration cell
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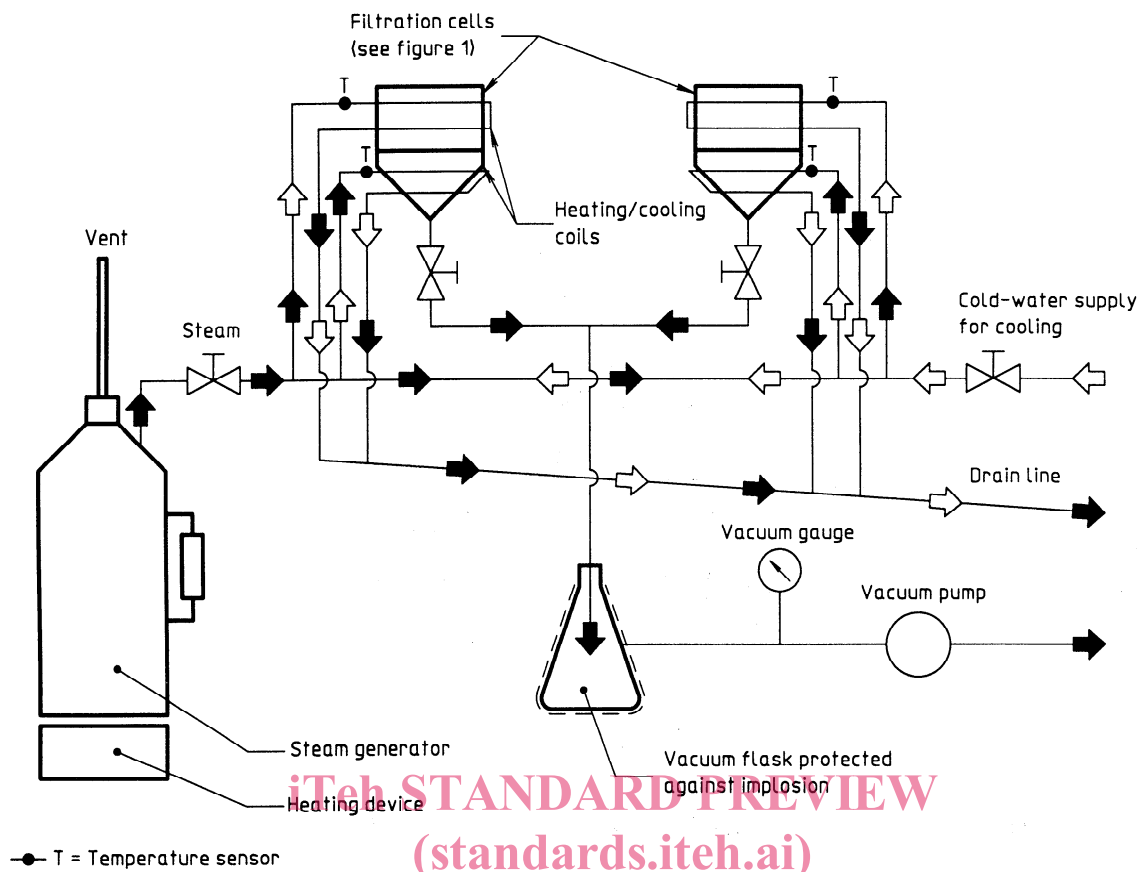


Figure 2 — Arrangement of filtration apparatus

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