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ISO 10307-2

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Petroleum products — Total sediment in residual fuel oils —

Part 2: Determination using standard procedures for ageing

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Produits pétroliers — Sédiment total dans les fuel-oils résiduels — Partie 2: Détermination à l'aide de méthodes de vieillissement de référence

Reference number
ISO 10307-2:1993(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.

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International Standard ISO 10307-2 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

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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*

Annex A forms an integral part of this part of ISO 10307.

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Introduction

Experience has shown that the precipitation of asphaltenes from a residual fuel oil in the form of sediment may occur during storage and handling. Such sediment may cause severe difficulties, and in extreme cases can render the fuel unfit for use. Once out of solution, it is extremely difficult to repeptize the asphaltenes into their original state.

Fuel pre-treatment designed to accelerate the ageing/sedimentation process, followed by filtration, is a well-established technique for testing whether sediment from residual fuel oils will precipitate during storage and handling. This could involve thermal ageing (heating to a specified temperature for a specified time) or chemical ageing (addition of a specified amount of a normal alkane to test whether the balance between the required aromaticity of the asphaltenes and the available aromaticity of the oil phase is disturbed to the extent that asphaltene precipitation occurs).

A means of predicting the presence of a reserve of stability to sedimentation in residual fuel oil during storage and handling is thus a useful tool in the petroleum products industry.

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Petroleum products — Total sediment in residual fuel oils —

Part 2:
Determination using standard procedures for ageing

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.

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1 Scope

This part of ISO 10307 specifies two procedures for accelerated ageing of residual fuel oils which, combined with the hot filtration method specified in ISO 10307-1, permit the prediction of fuel oil stability to sedimentation during storage and handling.

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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 1773:1976, *Laboratory glassware — Boiling flasks (narrow-necked).*

ISO 10307-1:1993, *Petroleum products — Total sediment in residual fuel oils — Part 1: Determination by hot filtration.*

ASTM D460:1990, *Method for sampling and chemical analysis of soaps and soap products.*

ASTM D850:1990, *Method for distillation of industrial aromatic hydrocarbons and related materials.*

ASTM D1015:1989, *Test method for freezing points of high-purity hydrocarbons.*

ASTM D4274:1988, *Method for testing polyurethane polyol raw materials — Determination of hydroxyl numbers of polyols.*

IP 17:1952, *Colour by Lovibond Tintometer.*

3 Definitions

For the purposes of this part of ISO 10307, the following definitions apply.

3.1 potential total sediment: Total sediment, determined by ISO 10307-1, after ageing a sample of residual fuel for 24 h at 100 °C under prescribed conditions.

3.2 accelerated total sediment: Total sediment, determined by ISO 10307-1, after dilution of a sample of residual fuel with hexadecane in the ratio of 1 ml per 10 g of sample under carefully controlled conditions, followed by storage for 1 h at 100 °C.

4 Principle

Total sediment is determined after accelerated ageing according to 4.1 or 4.2, followed by hot filtration as specified in ISO 10307-1.

4.1 Thermal ageing (Procedure A)

A sample of residual fuel oil is subject to ageing at 100 °C for 24 h under specified conditions.

4.2 Chemical ageing (Procedure B)

A sample of residual fuel oil is diluted with a specified amount of hexadecane (cetane) under carefully controlled conditions. It is then heated to 100 °C for 1 h.

5 Apparatus

5.1 Ageing bath, comprising an electrically heated oil bath, capable of maintaining a temperature of 100 °C ± 0,5 °C, fitted with air wells of i.d. 55 mm and depth 120 mm, as illustrated in figure 1.

5.2 Thermometers.

5.2.1 For the ageing bath, as specified in A.1.

5.2.2 For general purpose, as specified in A.2.

5.3 Air condenser, glass, o.d. 8 mm, length 400 mm.

5.4 Stoppers, to provide a tight fit in the test jar.

5.4.1 Cork, bored to accommodate the air condenser.

5.4.2 Rubber, unbored.

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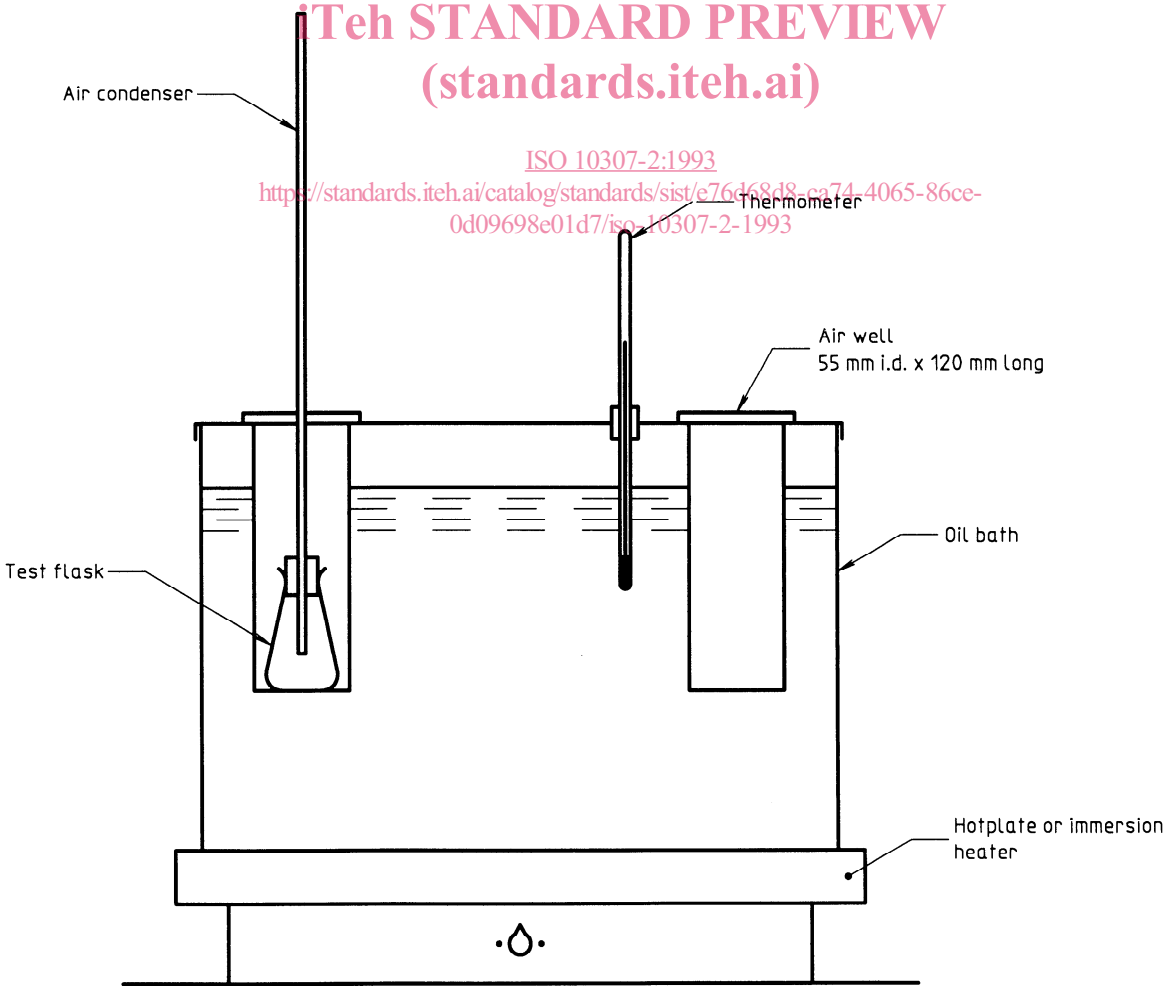

Figure 1 — Ageing bath

5.5 Microburette, of minimum capacity 5 ml, graduated in 0,02 ml subdivisions or less.

5.6 Magnetic stirrer/hotplate, capable of being controlled by a contact thermometer, or other suitable temperature control device, and with polytetrafluoroethylene (PTFE)-coated stirring bars, length 25 mm.

5.7 Contact thermometer, range 0 °C to 200 °C (see 5.6).

5.8 Metal block, of copper or brass, diameter 40 mm, height 40 mm, with a central hole of diameter 1 mm larger than the diameter of the lower part of the contact thermometer.

5.9 Conical flask, 50 ml capacity, narrow neck, complying with ISO 1773.

5.10 Spatula, of polypropylene, chamfered ends, 200 mm long.

5.11 Stirring rod, of PTFE, approximately 150 mm long and 3 mm in diameter.

5.12 Hexadecane distribution funnel, of borosilicate glass, capacity 5 ml to 10 ml, outlet orifice 0,4 mm ± 0,02 mm diameter.

NOTE 1 Standard funnels meeting the above criteria are available commercially.

5.13 Portable warm air blower, for heating the microburette to 20 °C minimum, if required.

5.14 Homogenizer, non-aerating, high-speed shear type.

6 Reagent

6.1 Hexadecane (cetane), conforming to the following requirements as determined by the specified or equivalent methods.

Freezing point (ASTM D1015)	16,2 °C min.
Hydroxyl number (ASTM D4274)	nil
Iodine number (ASTM D460)	nil
Colour (IP 17)	water white (1,0)
Distillation (ASTM D850)	
5 % *V* recovered at	286,6 °C ± 1,0 °C
Range	6,0 °C max.

As hexadecane starts to solidify at approximately 18 °C, store the bulk quantity at or above 20 °C, or bring to this minimum temperature before use.

NOTES

2 Portable warm air blowers and/or water baths at not more than 50 °C are suitable for raising the temperature of the microburette or bulk hexadecane container.

3 Commercially available hexadecane (cetane) with 99 % purity certification is suitable for use without further testing.

7 Sampling

The sample aliquot for these procedures shall be taken from the same container as that sample taken for ISO 10307-1 for determination of sediment by hot filtration.

NOTE 4 The samples will normally be taken at the same time, and thus be subjected to the same preparation procedure.

8 Sample preparation

Homogenize the whole sample thoroughly, using the homogenizer (5.14) if practicable, for 30 s. A sample taken on a glass or PTFE rod 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 above the pour point for low-viscosity fuels, or sufficient to reduce the viscosity to between 150 mm^2/s and 200 mm^2/s for high-viscosity fuels. The temperature shall not exceed 80 °C during this preparation stage.

9 Test procedures

9.1 Procedure A — Sediment determination using thermal ageing

Carry out the ageing procedure on duplicate aliquots.

9.1.1 Pour a 25 g ± 1 g aliquot of the homogenized sample into the conical flask (5.9), attach the air condenser (5.3) by means of the cork (5.4.1), and place the sample in the well of the ageing bath (5.1) at 100 °C ± 0,5 °C for 24 h ± 15 min.

9.1.2 Remove the flask from the bath, replace the air condenser by the rubber stopper (5.4.2), and shake vigorously until all the sludge has been uniformly suspended. To check this, invert the flask and examine the bottom and walls of the flask for any sludge deposits, after allowing the oil to drain down from the inside walls of the flask. Remove stubborn deposits from the walls or bottom of the conical flask by scraping with the spatula (5.10). Re-shake and within 1 min initiate the procedure specified in clause 9 of ISO 10307-1:1993 for sediment determination by hot filtration.

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9.2 Procedure B — Sediment determination using chemical ageing

Carry out the ageing procedure on duplicate aliquots.

9.2.1 Place the metal block (5.8) on the magnetic stirrer/hotplate (5.6), position the contact thermometer (5.7) in the hole in the block, supporting the contact thermometer with a clamp, and connect the thermometer to the stirrer/hotplate. Heat the stirrer/hotplate to a surface temperature corresponding to the temperature at which the sample under investigation has a kinematic viscosity of approximately 50 mm^2/s.

9.2.2 Weigh 25 g ± 0,2 g of the homogenized sample into the conical flask and add a PTFE-coated stirring bar. Place the flask in the centre of the stirrer/hotplate (controlled as described in 9.2.1) and adjust the stirring speed to approximately 200 rev/min. After 10 min, slowly add 2,5 ml ± 0,02 ml of hexadecane (6.1) from the microburette (5.5) to the flask at a maximum rate of 1,0 ml/min, while continuously stirring.

Asphaltenes which are flocculated during the addition of hexadecane owing to lack of homogeneity are not easily peptized afterwards, and can produce faulty results. Therefore it is imperative that local over-dilution does not occur during the addition step. This is dependent on the maximum rate of addition, which can be controlled by positioning the microburette above a funnel (5.12), discharging the 2,5 ml ± 0,02 ml directly into the funnel neck, and allowing the hexadecane to drip into the stirred sample by gravity. Direct addition of the hexadecane from the microburette is not recommended, but if carried out it is essential that the orifice diameter of the microburette does not exceed 1,0 mm, and the overall tip diameter does not exceed 5,0 mm.

NOTE 5 The rate of mass addition can be determined by plotting mass transferred as calculated from the volume delivered by the burette (hexadecane density at 20 °C is 773,4 kg/m^3) against time of delivery.

9.2.3 Pour the sample + hexadecane mixture into a fresh 50 ml flask and attach the air condenser by means of the cork. Place the flask in the well of the oil bath at 100 °C ± 0,5 °C. Allow the flask to remain in the bath for 60 min ± 2 min.

9.2.4 Remove the flask from the bath, replace the air condenser by the rubber stopper and shake vigorously until all the sludge has been uniformly suspended. To check this, invert the flask and examine the bottom and walls of the flask for any sludge deposits, after allowing the oil to drain down from the inside walls of the flask. Remove stubborn deposits from the walls or bottom of the flask by scraping with the spatula. Re-shake and within 1 min initiate the procedure specified in clause 9 of ISO 10307-1:1993 for sediment determination by hot filtration.

10 Expression of results

10.1 The results obtained after either of the above ageing procedures shall be compared with results obtained on the same sample in an unaged (existent) condition. These results shall then be reported in the manner described in 10.2 or 10.3.

10.2 Report **potential total sediment** to the nearest 0,01 % (*m/m*) as the average of duplicate determinations carried out on samples subjected to thermal ageing.

10.3 Report **accelerated total sediment** to the nearest 0,01 % (*m/m*) as the average of duplicate determinations carried out on samples subjected to chemical ageing.

When calculating the results of determinations of accelerated total sediment, take into account the hexadecane diluent by using a divisor of 9,28 M_1 in the formula in clause 10 of ISO 10307-1:1993 instead of 10 M_1.

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11 Precision

It was established in 1989, by the statistical examination of interlaboratory results, that neither of the ageing procedures described in this part of ISO 10307 affects the precision of ISO 10307-1.

12 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 10307, including the procedure (A or B) followed;

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

c) the results of the test after ageing, including the description (potential or accelerated) (see clause 10);

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

e) the date of the test;

Annex A
(normative)

Thermometer specifications

A.1 Thermometer for ageing bath

The thermometer (5.2.1) shall meet the specifications given in table A.1 below.

Table A.1 — Ageing bath thermometer specifications

Temperature range, °C	95 to 103
Immersion	Total length
Scale marks subdivisions, °C long lines at each, °C numbers at each, °C maximum line width, mm	 0,1 0,5 1 0,15
Scale error, max., °C	0,1
Expansion chamber permits heating to, °C	 155
Total length, mm	270 to 280
Stem o.d., mm	6,0 to 8,0
Bulb length, mm	25 to 35
Bulb o.d., mm	5,0 to stem o.d.
Scale location distance from bottom of bulb to line at 95 °C, mm length of scale range, mm	 135 to 150 70 to 100
NOTE — An IP 24C/ASTM 22C thermometer conforms to this specification.	

A.2 General purpose thermometer

The general purpose thermometer (5.2.2) shall meet the specifications given in table A.2 below.

Table A.2 — General purpose thermometer specifications

Nominal range, °C	0 to 100
Immersion, mm	75
Scale marks subdivisions, °C long lines at each, °C numbers at each, °C maximum line width, mm	 1 5 10 0,25
Scale error, max., °C	1,0
Total length, mm	295 to 315
Bulb o.d.	Stem o.d. max.
NOTE — An ISO 1770 Type D/75 thermometer conforms to this specification.	

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