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Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test —

Part 1:
Procedure for mineral oils

Pétrole et produits connexes — Détermination du comportement au vieillissement des fluides et huiles inhibés — Essai TOST —

Partie 1: Méthode pour les huiles minérales

Reference number
ISO 4263-1:2003(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4263-1 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 4263 consists of the following parts, under the general title *Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test*:

- *Part 1: Procedure for mineral oils*
- *Part 2: Procedure for category HFC hydraulic fluids*
- *Part 3: Anhydrous procedure for synthetic hydraulic fluids*
- *Part 4: Procedure for industrial gear oils*

Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test —

Part 1:
Procedure for mineral oils

WARNING — The use of this part of ISO 4263 may involve hazardous materials, operations and equipment. This part of ISO 4263 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 4263 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 4263 specifies a method for the determination of the ageing behaviour of rust- and oxidation-inhibited mineral oils having a density less than that of water, used as turbine oils (categories TSA, TGA, TSE, TGE of ISO 6743-5, see [4] in the Bibliography), hydraulic oils (categories HL, HM, HR, HV, HG of ISO 6743-4, see [3] in the Bibliography), and circulating oils (category CKB of ISO 6743-6, see [5] in the Bibliography). Oils containing synthetic components can be tested by this procedure, but no precision statement is available yet for such fluids.

NOTE 1 For the purposes of this part of ISO 4263, the term "% (m/m)" is used to represent the mass fraction of a material.

NOTE 2 Other signs of oil deterioration, such as the formation of insoluble sludge, catalyst coil corrosion or decrease in pH value, may occur, which indicate oxidation of the oil, but are not reflected in the calculated oxidation lifetime. The correlation of these occurrences with field service is under investigation.

This test method is widely used in specifications and is considered of value in comparing the oxidation stability of oils that are prone to contamination with water. However, because of the large number of individual field-service applications, the correlation between the results of this test and actual service performance can vary markedly, and is best judged on experience.

The precision of this part of ISO 4263 for oxidation life was only determined on inhibited turbine oils, and applies to oxidation lives of 700 h to 3 900 h.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170:—[1], *Petroleum liquids — Manual sampling*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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[1] To be published. (Revision of ISO 3170:1988)

ISO 6618:1997, *Petroleum products and lubricants — Determination of acid or base number — Colour-indicator titration method*

ISO 6619:1988, *Petroleum products and lubricants — Neutralization number — Potentiometric titration method*

ISO 7537:1997, *Petroleum products — Determination of acid number — Semi-micro colour-indicator titration method*

3 Principle

A test portion is reacted, in the absence of light, at 95 °C with oxygen in the presence of water and a steel and copper catalyst coil. Small aliquots of the oil are withdrawn at regular intervals and the acid number is measured (see Note 2 in Clause 1). The test is continued until an acid number of 2,0 mg of potassium hydroxide (KOH) per gram of test portion is reached and the number of hours is recorded as the oxidation life. For some requirements, the test may be discontinued at a fixed number of hours (e.g. 1 000 h) when the value of the acid number is still below 2,0 mg of KOH per gram of test portion.

4 Reagents and materials

4.1 Water, unless otherwise specified, in accordance with the requirements of grade 2 as defined in ISO 3696. Potable water means tap water, unless normal piped supplies are contaminated with particulate or highly soluble mineral content.

4.2 Heptane (C_7H_{16}), of minimum purity 99,75 %.

4.3 Acetone (CH_3COCH_3), of general purpose reagent grade (GPR).

4.4 Propan-2-ol ($CH_3CHOHCH_3$), of general purpose reagent grade (GPR).

4.5 Oxygen, of minimum purity 99,5 %. Supplied through a pressure-regulation system adequate to maintain the specified flow rate throughout the test duration.

Supply from an oxygen cylinder should be via a two-stage regulation system and a needle valve to improve the consistency of gas-flow regulation.

WARNING — Use oxygen only with equipment validated for oxygen service. Do not allow oil or grease to come into contact with oxygen and clean and inspect all regulators, gauges and control equipment. Check the oxygen-supply system regularly for leaks. If a leak is suspected, turn off immediately and seek qualified assistance.

4.6 Cleaning solutions

4.6.1 Strong oxidizing acid solution

The reference strong oxidizing cleaning solution on which precision was based, is chromosulfuric acid (see the following warning), but alternative non-chromium containing solutions, such as ammonium persulfate in concentrated sulfuric acid (8 g/l) have been found to give satisfactory cleanliness. A 10 % solution of three parts of hydrochloric acid (1 mol/l) and one part of orthophosphoric acid (concentrated GPR grade) removes iron oxide deposits.

WARNING — Chromosulfuric acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr(VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromosulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solution down the drain, but neutralize it with great care owing to the concentrated sulfuric acid present, and dispose of it in

accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).

Strongly oxidizing acid cleaning solutions that are chromium-free are also highly corrosive and potentially hazardous when in contact with organic materials, but do not contain chromium which has special disposal problems.

4.6.2 Surfactant cleaning fluid

A proprietary strong surfactant cleaning fluid is a preferred alternative to the strong oxidizing cleaning solution, whenever the condition of the glassware permits this.

4.6.3 Laboratory detergent

The detergent shall be water soluble.

4.7 Catalyst wires

4.7.1 Low-metalloid steel wire, of diameter 1,60 mm ± 0,05 mm, made of carbon steel, soft bright annealed and free from rust.

4.7.2 Copper wire, of diameter 1,63 mm ± 0,05 mm, made of either electrolytic copper wire of 99,9 % minimum purity or soft copper wire of an equivalent grade.

4.8 Abrasive cloth, made of silicon carbide of 150 µm (100-grit) with a cloth backing, or an equivalent grade of abrasive cloth.

4.9 Absorbent cotton

5 Apparatus

5.1 Oxidation cell, consisting of a large test tube of borosilicate glass with a graduation mark at 300 ml ± 1 ml, which applies to the test tube alone at 20 °C. A mushroom condenser and oxygen-delivery tube, also of borosilicate glass, fit into the test tube. The design and dimensions shall be as illustrated in Figure 1.

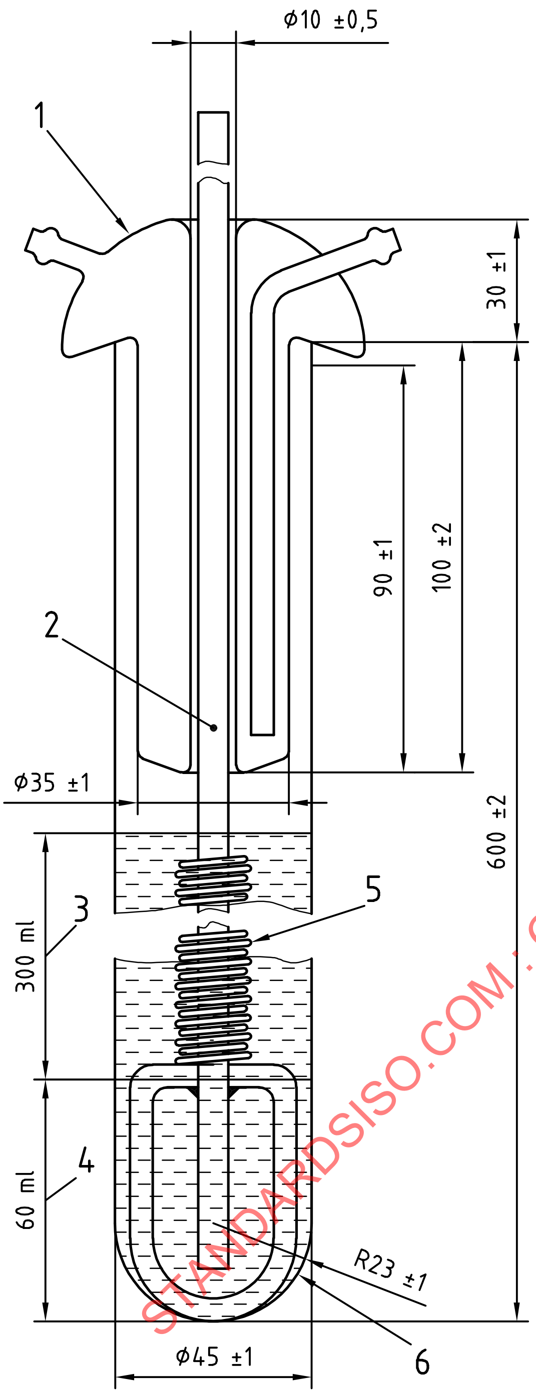
5.2 Heating bath, consisting of a thermostatically controlled bath capable of maintaining the oil test portion in the oxidation cell at 95 °C ± 0,2 °C. It shall be large enough to hold the required number of oxidation cells (5.1) immersed in the heat-transfer medium to a depth of 355 mm ± 10 mm. It shall be constructed to ensure that light is excluded from the test portions during the test. If a fluid bath is used, it shall be fitted with a suitable stirring system to provide a uniform temperature throughout the bath. If the fluid bath is fitted with a top, the total length of the oxidation cell within the bath shall be 390 mm ± 10 mm. If a metal-block bath is used, the heaters shall be distributed so as to produce a uniform temperature throughout the bath, and the holes in the block shall have a minimum diameter of 50 mm and a depth, including any insulating cover, of 390 mm ± 10 mm.

5.3 Flowmeter, of minimum capacity 3 l/h and an accuracy of ± 0,1 l/h.

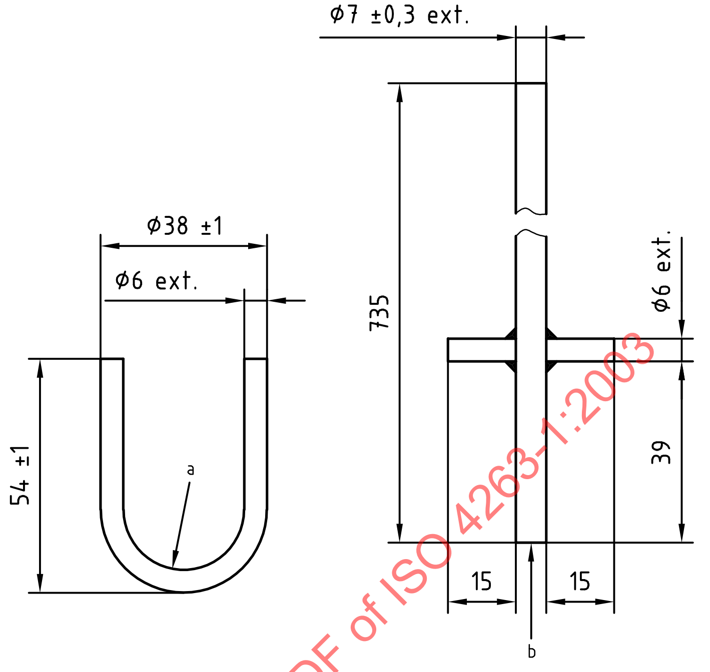
5.4 Temperature-measurement devices

5.4.1 Heating bath. The temperature in liquid heating baths shall be measured by either a liquid-in-glass thermometer meeting the requirements of the specification given in Annex A, or an equivalent temperature-measurement system readable to ± 0,1 °C and calibrated to better than ± 0,1 °C. For metal-block heating baths, a temperature-measurement system, with possibly more than one device of the same readability and accuracy, is required.

Dimensions in millimetres

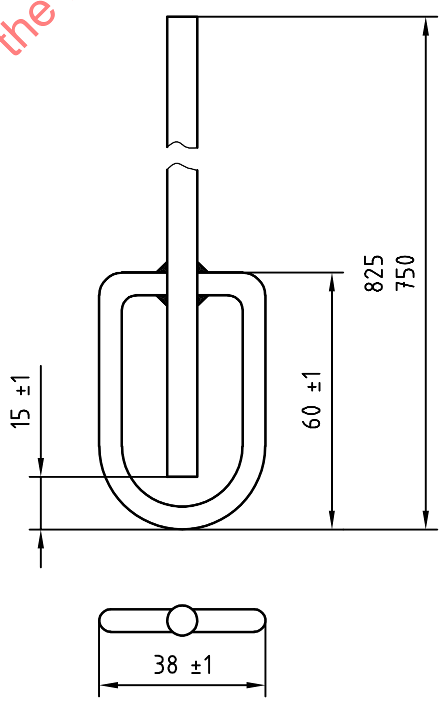


a) Apparatus



b) First stage

c) Second stage



d) Final stage

Key

1 glass condenser

2 oxygen-delivery tube

3 oil sample

4 water

5 catalyst coil

6 radius of bottom of cell

[a] Bend over mandrel of diameter 26

[b] Grind end of tube

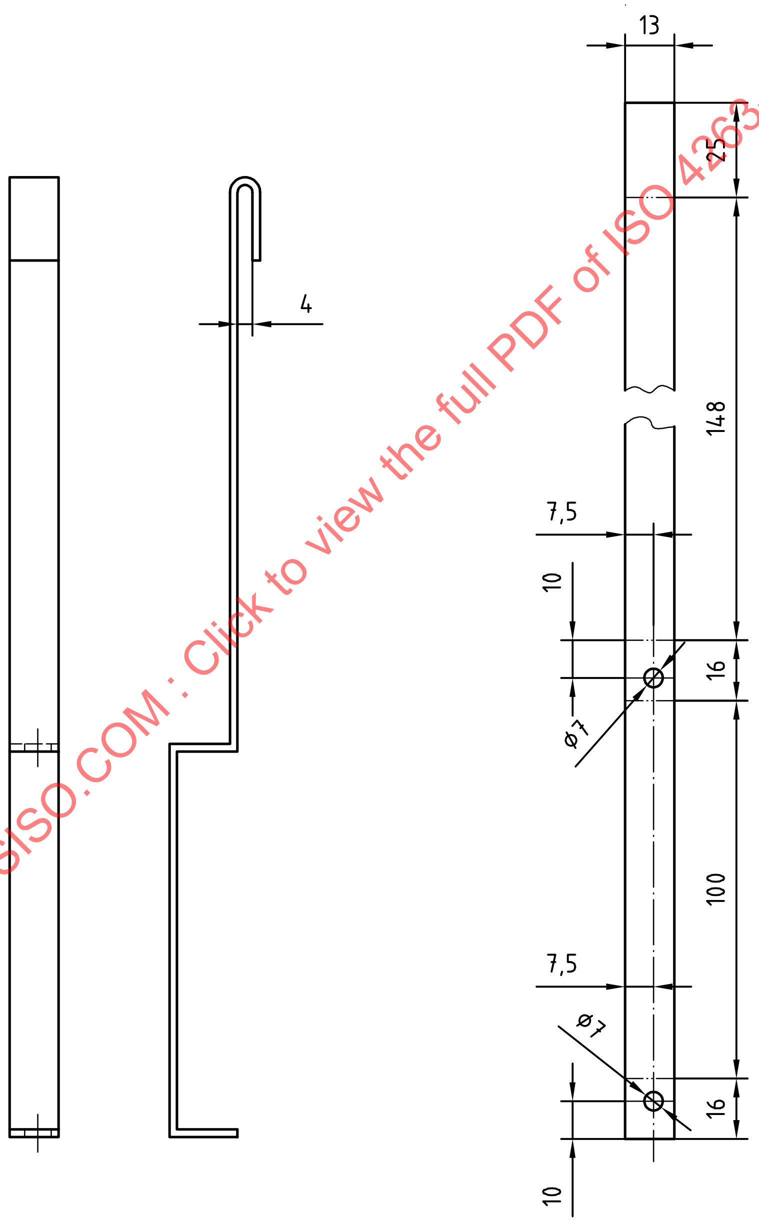
Figure 1 — Oxidation cell

5.4.2 Oxidation cell. The temperature in the oxidation cell shall be measured by either a liquid-in-glass thermometer meeting the requirements of the specification given in Annex A, or an equivalent temperature-measurement system readable to ± 0,1 °C and calibrated to better than ± 0,1 °C.

5.4.3 Thermometer bracket. If a liquid-in-glass thermometer is used in the oxidation cell, it shall be suspended by means of a bracket as illustrated in Figure 2. The thermometer is held in the bracket by either two fluoro-elastomer O-rings of approximately 5 mm diameter, or by the use of a thin stainless steel wire.

5.5 Wire-coiling mandrel. A mandrel, as illustrated in Figure 3, is used to produce the double spiral of copper and steel wire. The mandrel is included in a suitable winding device.

Dimensions in millimetres



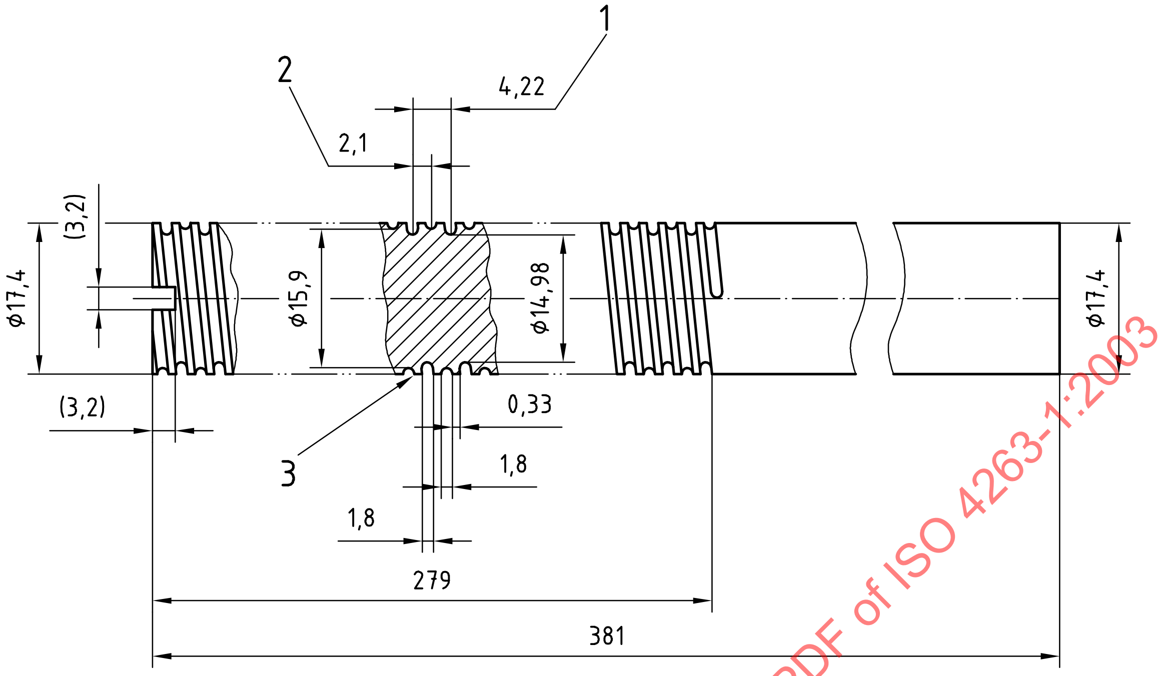
a) **Bracket finished**

b) **Development of bracket**

Material: 18-8 stainless steel (0,792 mm)

Figure 2 — Thermometer bracket

Dimensions in millimetres



Material: bronze

Key

1	lead
2	pitch
3	double thread

Figure 3 — Catalyst coil mandrel

5.6 Oxygen-supply tube. Flexible polyvinylchloride (PVC) tubing of approximately 6,4 mm inside diameter and 1,5 mm wall thickness, is required to deliver oxygen to the oxidation cell.

5.7 Aliquot-removal devices. Depending on the size and frequency of removal of aliquots of the test portion for analysis, a selection of devices are required. Glass syringes, fitted with Luer connectors and stainless steel needles, or long pipettes fitted with suitable pipette fillers, are suitable. These may be inserted via a sampling tube fitted through the condenser. Aliquot sizes will generally be in the range of 2 ml to 10 ml, and the devices shall be capable of removing the required aliquot ± 0,2 ml.

5.8 Aliquot containers. Small, dark glass vials of 5 ml to 10 ml capacity, fitted with close-fitting polyethylene caps, are required.

6 Sampling

Unless otherwise specified, samples shall be obtained by the procedures described in ISO 3170.

7 Preparation of materials and apparatus

7.1 Cleaning catalysts

Immediately prior to winding a catalyst coil, clean a 3,00 m ± 0,01 m length of steel wire (4.7.1) and an equal length of copper wire (4.7.2) with wads of absorbent cotton (4.9) soaked in heptane (4.2), and then abrade with the abrasive cloth (4.8) until a fresh metal surface is exposed. Wipe with dry absorbent cotton until all the loose particles of metal and abrasive have been removed. In all subsequent operations, handle the catalyst wires with clean gloves (cotton, rubber or plastic) to prevent contact with the skin.

7.2 Preparation of catalyst coil

Twist the steel and copper wires together tightly at one end for three turns and then wind them simultaneously alongside each other on a threaded mandrel (5.5 and Figure 3), inserting the steel wire in the deeper thread. Twist the free ends of the steel and copper wires together for three turns and bend the twisted ends to conform to the shape of the spiral coil. Remove the coil from the mandrel by reversing the winding action. Ensure that the overall length of the coil is 225 mm ± 5 mm by stretching or compression if necessary.

7.3 Catalyst storage

Store the catalyst coil in a dry inert atmosphere prior to use, in accordance with the procedures described in Annex B. Inspect before use to ensure that no corrosion products or contaminating materials are present. For storage of less than 24 h, storage of the coil in heptane that is free from traces of water and corrosive materials is satisfactory.

NOTE Redistilled heptane (4.2), stored in a tightly sealed bottle, is suitable for overnight storage of the catalyst coil.

7.4 Cleaning new glassware

Wash new oxygen-delivery tubes, condensers and test tubes with hot detergent solution (see 4.6.3) and rinse thoroughly with potable water (4.1). Clean the interiors of the test tubes, the exteriors of the condensers, and both interiors and exteriors of the oxygen-delivery tubes by either soaking for 24 h in a 10 % solution of the surfactant cleaning fluid (4.6.2), or by washing in strong oxidizing acid solution (4.6.1). Rinse all parts thoroughly with potable water followed by water (4.1) and allow to dry, either in an oven at approximately 100 °C or with a final rinse of propan-2-ol (4.4) or acetone (4.3) followed by air drying at ambient temperature.

7.5 Cleaning used glassware

Immediately following the termination of a test, drain the oil completely from the test tube and rinse all glassware with heptane (4.2) to remove traces of oil. Wash with hot detergent solution (see 4.6.3) using a long-handled brush and rinse thoroughly with potable water.

NOTE If adherent deposits are still present, these may be removed by filling the test tube with detergent solution, inserting the oxygen-delivery tube and fitting the condenser, and replacing the tube in the heating bath at test temperature. Often, after several hours of soaking, all adhering deposits except iron oxide have loosened, and this can be removed by a subsequent soaking in the hydrochloric/orthophosphoric acid mixture (see 4.6.1).

After all deposits have been removed, follow the cleaning procedure described in 7.4.

Store all cleaned glassware in a dry, dust-free condition until required.

7.6 Cleaning aliquot-removal device

Completely drain the tube of the sampling device and/or any other devices used and rinse any surfaces that have contacted the oil with heptane (4.2) to remove traces of oil. Soak the device to above the contact level for 24 h in the surfactant cleaning fluid (4.6.2), or wash in strong oxidizing acid solution (4.6.1), rinse with potable water, followed by water (4.1), and dry in the manner described in 7.4.

8 Procedure

8.1 Adjust the heating bath to a temperature that will maintain the temperature in the oil in the oxidation cells (5.1) at 95 °C ± 0,2 °C throughout the duration of the test, with oxygen passing through the cells at 3 l/h ± 0,5 l/h.

NOTE The temperature of the heating bath (5.2) will be above 95 °C due to the cooling effect of the oxygen flow, but the specific temperature will depend on the bath medium, capacity and circulation efficiency.

It is recommended that the heating bath be always filled with oxidation cells, using dummy (ballast) cells for positions not occupied by test cells. In this way, the heating levels will be consistent, and the level of fluid medium in liquid baths will be maintained at the correct immersion depth.

8.2 Measure the temperature in each cell containing 360 ml of fluid (which may be 300 ml of oil plus 60 ml of water), by means of the liquid-in-glass thermometer located on the thermometer bracket (5.4.3) with the immersion line at the fluid surface, or by means of an alternative temperature-measurement device at the same point. If the liquid-in-glass thermometer is used, correct the temperature reading by a 0,1 °C subtraction to allow for emergent-stem heating.

8.3 When a uniform temperature of 95,0 °C ± 0,2 °C is obtained in all the test cells in the heating bath, record the heating bath temperature and maintain it at this level throughout tests carried out under the same conditions. Any change in condition, such as a change in the number of cells in the bath, or dramatic change in the test fluid type, necessitates a check on the level and uniformity of temperature control. All temperature measurements are carried out on new, undepleted test portions and/or dummy cells. Immediately the temperature control has been established, remove the temperature-measurement devices.

8.4 Fill the empty oxidation test tube with 300 ml of mineral oil (up to the graduation mark). Slide the catalyst coil over the oxygen-inlet tube. If the wires are uneven at one end of the coil, position the coil so that this end is down. Place the oxygen-inlet tube with the coil in the test tube, and place the condenser over the oxygen-delivery tube and test tube. If used, a sampling tube and holder are inserted at this time. Check the immersion depth of the oxidation cell, and adjust to 355 mm ± 10 mm as necessary. Connect the condenser to the cooling water and adjust the flow rate such that the temperature of the outlet water does not exceed 32 °C at any time during the test.

8.5 Connect the oxygen-delivery tube to the oxygen supply through the flowmeter (5.3) using new flexible tubing (5.6) no more than 600 mm in length. Before use, flush the interior of the tubing with heptane (4.2) and blow dry with air. Adjust the rate of oxygen flow to 3 l/h ± 0,1 l/h.

8.6 Maintain the conditions for 30 min, and then add 60 ml ± 2 ml of water (4.1) to the oxidation cell by raising the condenser. Mark the level of the water and of the oil plus water on the outside of the test tube.

8.7 Throughout the duration of the test, maintain the temperature of the heating bath at that determined in 8.3 to maintain the oil-water mixture at 95 °C ± 0,2 °C in each test cell.

8.8 At determined intervals (see the fourth paragraph of this subclause), disconnect the oxygen-supply tube and allow the contents of the oxidation cell to settle for 30 min. Remove the condenser if necessary and, using the selected aliquot-removal device, withdraw an aliquot of 2 ml to 5 ml from the mid-point of the oil in the oxidation cell, taking care not to include any water. Determine the acid number of the aliquot by the procedures described in ISO 6618, ISO 6619 or ISO 7537, and if required, record the condition of the oil, water and catalysts.

Add additional water to the oxidation cell to restore the level to the mark established in 8.6. Replace the condenser if removed, connect the oxygen-supply tube, and re-establish the test conditions.

Acid numbers determined in accordance with ISO 6618, ISO 6619 and ISO 7537 may not be exactly comparable, and thus care should be taken, when comparing results, that the same acid number test method has been used in those results being compared.

It is normal to omit acid number determinations for the first 500 h of test, and thereafter to carry out determinations at intervals of every 150 h to 330 h. For oils with an expected oxidation life of less than 500 h, earlier removal of aliquots is at the discretion of the operator. For very long duration tests, aliquots should be taken less frequently, and after 3 000 h, at 500 h ± 25 h intervals until observation indicates that the oil is nearing the end of its oxidation life, when more frequent observations may be introduced. If the acid number is still below 2,0 mg of KOH per gram of test portion at 10 000 h, it is unreasonable to continue the test, as the water/oil ratio will have been seriously distorted, and the catalyst coil may have become exposed.

NOTE The objective is to maintain 60 ml of water present throughout the test, but in some instances it will not be possible to see clearly the oil/water interface. It may be helpful to note the level difference for one aliquot removal and make appropriate adjustments for the number of removals and the original mark at 360 ml.

8.9 Discontinue the test either when the measured acid number exceeds 2,0 mg of KOH per gram of test portion, or at a predetermined fixed period of hours.

8.10 At the end of long fixed-hour tests, other requirements than the acid number may also be specified. A procedure for the determination of the insolubles content is given in Annex C, a means of assessment of the degradation of catalyst coil condition is given in Annex D, and an outline procedure for metals analysis is given in Annex E. Any formation of disagreeable odour should be noted and reported.

9 Calculation

Calculate the oxidation life, L, in hours, from the following equation:

$$L = A + (B - A)\ \left[(2{,}0 - C)/(D - C)\right]$$

where

A is the number of test hours when the acid number was last determined to be less than 2,0 mg of KOH per gram of test portion;

B is the number of test hours when the acid number was first determined to be above 2,0 mg of KOH per gram of test portion;

C is the acid number at A h;

D is the acid number at B h.

10 Expression of results

10.1 Report the oxidation life of mineral oils, in hours, to the nearest 1 h up to 1 000 h, and to the nearest 10 h above 1 000 h. When the test has been discontinued at a fixed number of hours, with the acid number below 2,0 mg of KOH per gram of test portion, report the acid number in parentheses following the fixed period of test.

10.2 Report any degradation of the condition of the catalyst coils and/or the formation of disagreeable odour at the completion of the test.

11 Precision

11.1 General

The precision, as determined by statistical examination in accordance with ISO 4259 (see [1] in the Bibliography) of interlaboratory test results on a matrix of mineral oils only, with an oxidation life range of

700 h to 3 900 h, is given in 11.2 and 11.3. There was enough data generated to provide an estimate of reproducibility when comparing the results of the average of two successive tests by a single operator, and this is also reported. The precision was determined using only ISO 6618 as the test method for acid number determination.

11.2 Repeatability, r

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 following value in only one case in twenty.

$$r = 0{,}192X$$

where X is the average of the results being compared.

11.3 Reproducibility, R

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 in only one case in twenty.

$$R = 0{,}332X$$

where X is the average of the results being compared.

11.4 Reproducibility with duplicate tests

If two successive tests are performed by each operator as specified in 11.3, the difference between the averages of the two sets of results from the tests would exceed the value of $0{,}302X$, where X denotes the mean value of the averaged results from the two laboratories, in only one case in twenty.

12 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 4263;

b) the type and complete identification of the product 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.

Annex A
(normative)

Liquid-in-glass thermometer specifications

The liquid-in-glass thermometers given in 5.4.1 and 5.4.2 shall meet the specifications given in Table A.1. Alternative temperature-measurement systems shall meet the performance criteria given in these specifications.

Table A.1 — Thermometer specifications

Thermometer		Heating bath	Oxidation bath
Temperature range	°C	72 to 126	80 to 100
Immersion	mm	100	76
Scale marks:			
Subdivisions	°C	0,2	0,1
Longer lines at each	°C	1	—
Numbers at each	°C	2	1
Maximum line width	mm	0,15	—
Scale error, maximum	°C	0,2	0,1
Total length	mm	390 to 400	235 to 245
Stem OD	mm	6,0 to 8,0	6,0 to 7,0
Bulb length	mm	15 to 20	—
Bulb OD	mm	< stem OD	—
Scale location:			
Bottom of bulb to line at	°C	72	—
Distance	mm	125 to 145	—
Scale length	mm	190 to 235	—

NOTE An ASTM 40C/IP 80C thermometer meets the specification for the heating bath thermometer.

Annex B
(normative)

Procedure for packaging and storage of catalyst coils

B.1 Materials

B.1.1 Test tubes, made of borosilicate glass, 250 mm long, with an outer diameter of 25 mm and an inner diameter of approximately 22 mm.

B.1.2 Test tube caps, cylindrical, made of polyethylene and designed to grip closely the outside surface of the test tube.

B.1.3 Desiccant bags, made of paper, approximately 76 mm long, 51 mm wide and 3 mm thick, containing 3 g ± 0,5 g of silica gel.

B.1.4 Flushing tube, made of stainless steel or glass, 305 mm long and with an outside diameter of approximately 5 mm, to deliver nitrogen to the bottom of the test tube.

B.1.5 Nitrogen, of minimum purity 99,7 %, oxygen free, and regulated to a suitable pressure.

The pressure should be sufficient to dislodge loose particles from the inside of the test tube, but not so high as to cause a vortex and entrain air within the tube.

B.2 Procedure

B.2.1 Flush a new test tube (B.1.1), that is visibly clean and dry, with nitrogen (B.1.5), using the flushing tube (B.1.4) to blow out any loose particles.

B.2.2 Hold the empty tube at an angle and, wearing gloves, gently slide the catalyst coil into the tube. Add a desiccant bag (B.1.3) folded lengthwise to fit into the tube. Insert the flushing tube down the middle of the test tube, to the bottom, and blow nitrogen through the tube for several seconds.

B.2.3 Withdraw the flushing tube slowly, with nitrogen still flowing, and immediately seal the test tube with a cap (B.1.2).

Annex C
(informative)

Method for the determination of the insolubles content of mineral oils

C.1 Scope

This Annex describes a procedure for the determination of the insolubles content of mineral oils that have been the subject of the accelerated oxidation described in this part of ISO 4263.

NOTE A similar procedure is available for the determination of the insolubles content of category HFC hydraulic fluids subjected to the same accelerated oxidation, but there are significant differences in terms of the filter porosity, aliquot size and preparation. This procedure is given in Annex C of ISO 4263-2:2003 [2]. The results of the two procedures are not comparable.

C.2 Principle

At the end of a fixed period (normally 1 000 h), the oxygen-tube contents are filtered through a membrane filter medium of nominal porosity 5 µm, and the mass of retained material is determined.

C.3 Apparatus

C.3.1 Standard filter assembly, capable of holding the filter medium (C.3.2) securely on a filter support between the funnel and a vacuum flask of minimum capacity 1 litre. The vacuum flask shall be protected against implosion.

C.3.2 Filter medium, consisting of membrane filters of diameter 47 mm, made of a material compatible with the oil under test, and of nominal pore diameter 5 µm.

C.3.3 Separating funnel, of capacity 500 ml, made of borosilicate glass.

C.3.4 Centrifuge, capable of giving a relative centrifugal force (F_{rc}) of approximately 1 100 at the tips of the tubes. The centrifuge shall meet all the safety requirements for normal use, and shall be fitted with trunnion cups and cushions that firmly support the tubes in position when the centrifuge is in motion, and together with the revolving head, shall withstand the maximum centrifugal force applied. The centrifuge shall be enclosed by a metal shield or case strong enough to withstand breakage during operation.

NOTE The relationship between the rotational frequency of the centrifuge, n, in revolutions per minute, and the relative centrifugal force, F_{rc}, is given by the following equation:

$$n = 1\ 337\sqrt{F_{\text{rc}}/d}$$

where d is the diameter of swing of the rotating tubes, expressed in millimetres.

C.3.5 Centrifuge tubes, suitable for use with the centrifuge (C.3.4).

C.3.6 Forceps, with unserrated tips.

C.3.7 Weighing dish, covered, of suitable dimensions to contain the filter medium (C.3.2).

C.3.8 Oven, controlled at 80 °C ± 5 °C, for drying the filter medium.