INTERNATIONAL STANDARD

ISO 4263-3

Second edition
2010-10-15

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

Part 3:
Anhydrous procedure for synthetic hydraulic fluids

Pétrole et produits connexes — Détermination du comportement au vieillissement des fluides et huiles inhibés au moyen de l'essai TOST —

Partie 3: Méthode anhydre pour les fluides hydrauliques synthétiques

Reference number
ISO 4263-3:2010(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 4263-3 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 4263-3:2006), of which it is a minor revision with changes to the title, Clause 3 (last sentence), 8.8, 8.9 and 10.1 plus some minor editorial modifications.

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

NOTE As of the date of publication of this revised edition of ISO 4263-3, the current editions of Parts 1, 2 and 4 still retained the old four-part title: *Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test* — Part X: ....

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

Part 3:
Anhydrous procedure for synthetic hydraulic fluids

WARNING — The use of this part of ISO 4263 can 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 synthetic hydraulic fluids of categories HFDR, HFDU, HEES and HEPG as defined, for example, in ISO 12922[4] and ISO 15380[5]. The ageing is accelerated by the presence of oxygen and metal catalysts at elevated temperature, and the degradation of the fluid is followed by changes in acid number. Other parts of ISO 4263 specify similar procedures for the determination of ageing behaviour of mineral oils and specified categories of fire-resistant fluids used in hydraulic and other applications.

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

This test method can be used to compare the oxidation stability of fluids that are not 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 test method for synthetic hydraulic fluids is not known because interlaboratory data are not available. This method might not be suitable for use in specifications or in the event of disputed results as long as these data are not available. However, precision for inhibited turbine oils is given in Clause 11 for guidance as an indication of the precision that could be obtained for synthetic hydraulic fluids.

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 3170:2004, *Petroleum liquids — Manual sampling*

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

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 and a steel and copper catalyst coil. Small aliquots of the fluid are withdrawn at regular intervals and the acid number is measured (see the Note in Clause 1). The test is continued until an acid number increase 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 lifetime. For some requirements, the test may be discontinued at a fixed number of hours (e.g. 500 h or 1 000 h) when the value of the acid number has still not increased by 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 of ISO 3696:1987. 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 a 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 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.

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 cloth backing, or an equivalent grade of abrasive cloth.

4.9 Absorbent cotton

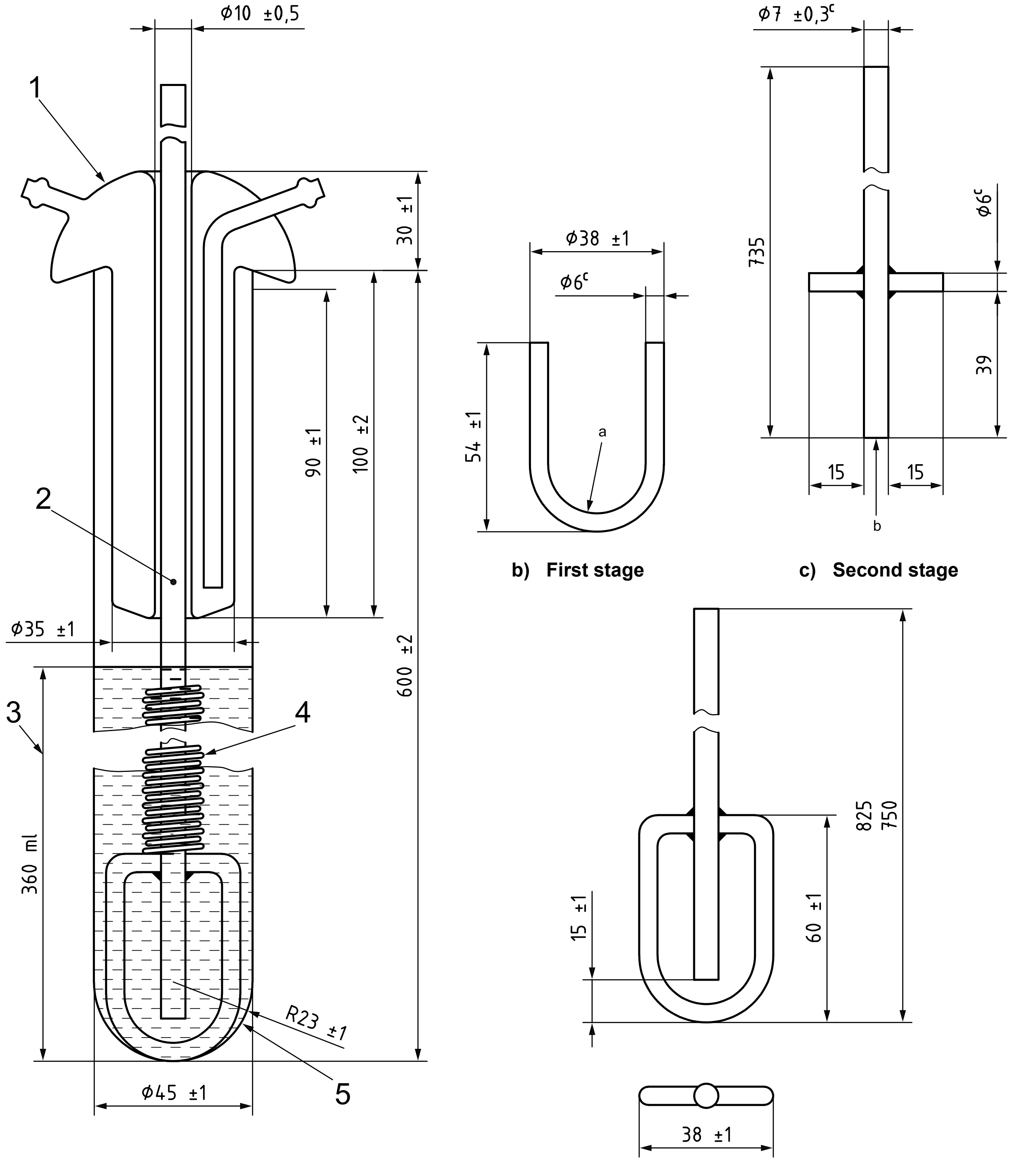
5 Apparatus

5.1 Oxidation cell, consisting of a large test tube of borosilicate glass with a graduation mark to indicate a volume of 300 ml ± 1 ml 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 hydraulic fluid test portion in the oxidation cell at 95,0 °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, capable of measuring 3,0 l/h with an accuracy of ± 0,1 l/h.

Dimensions in millimetres (unless otherwise indicated)



a) Apparatus

b) First stage

c) Second stage

d) Final stage

Key

1	glass condenser	4	catalyst coil
2	oxygen-delivery tube	5	radius of bottom cell
3	fluid sample		

[a] Bend over mandrel of diameter 26.

[b] Grind end of tube.

[c] External diameter.

Figure 1 — Oxidation cell

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 graduated in 0,1 °C increments. For metal-block heating baths, a temperature-measurement system, with possibly more than one device of the same readability and accuracy, is required.

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 thin, stainless steel wire.

5.5 Wire-coiling 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.

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 (5.9) fitted through the condenser. Aliquot sizes are generally 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.

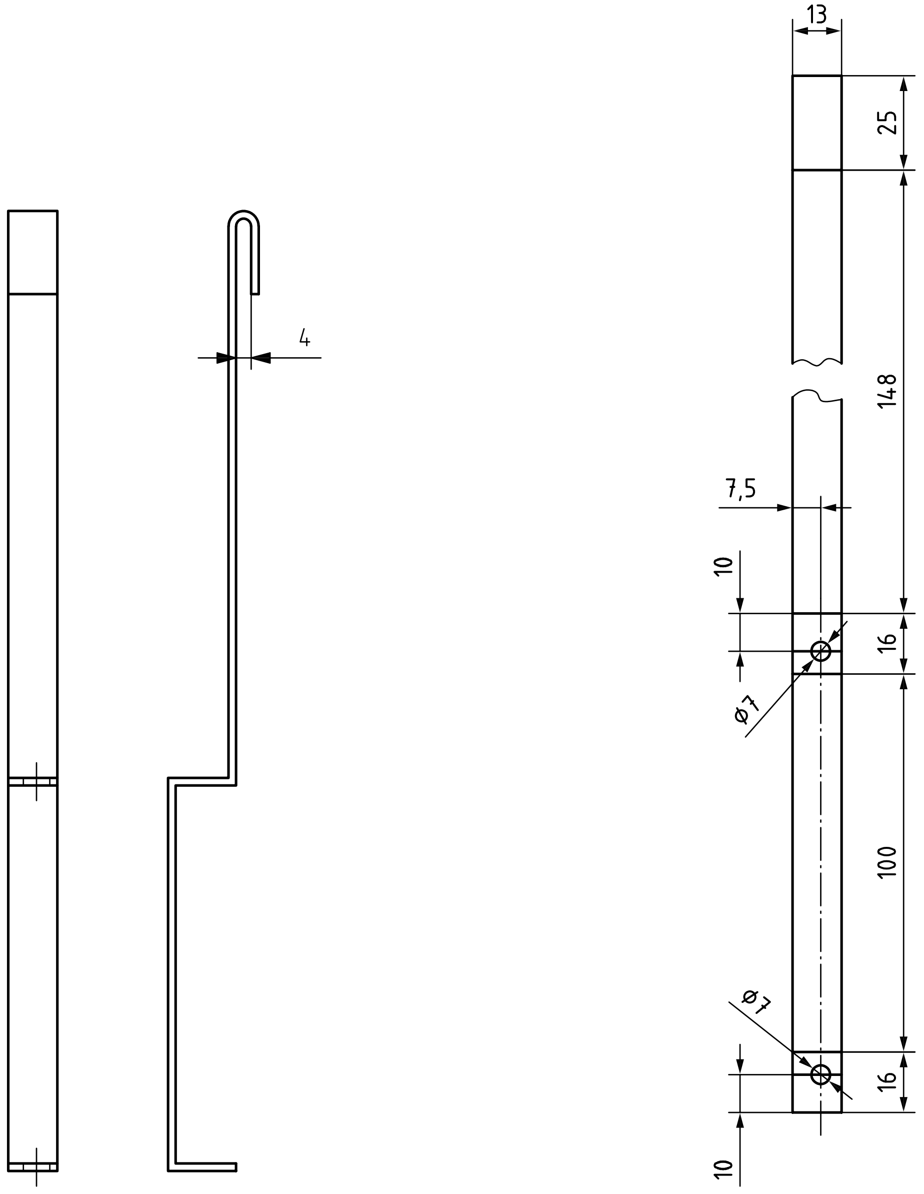
5.9 Sampling tube. Stainless steel tubing, 2,11 mm in outside diameter, 1,60 mm in inside diameter, 610 mm ± 2 mm long, with one end finished at 90° and the other end fitted with an optional female Luer connector [if using syringes as aliquot-removal devices (5.7)]. The optional connector is preferably of elastomeric material such as poly(fluorovinyl chloride) to provide a good seal with the syringe.

5.10 Stopper, for the optional Luer fitting of the sampling tube (5.9), made of polytetrafluoroethylene (PTFE) or poly(fluorovinyl chloride).

5.11 Sampling tube holder, for supporting the sampling tube (5.9), made of methyl methacrylate resin, having the dimensions shown in Figure 4.

5.12 Sampling tube spacer, for positioning the end of the sampling tube (5.9) above the sampling tube holder (5.11), made of plastic tubing of poly(vinyl chloride), polyethylene, polypropylene, or polytetrafluoroethylene, having an inside diameter of approximately 3 mm and 51 mm ± 1 mm in length.

Dimensions in millimetres



a) Bracket (finished) **b) Development of bracket**

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

Figure 2 — Thermometer bracket

Dimensions in millimetres
(unless otherwise indicated)

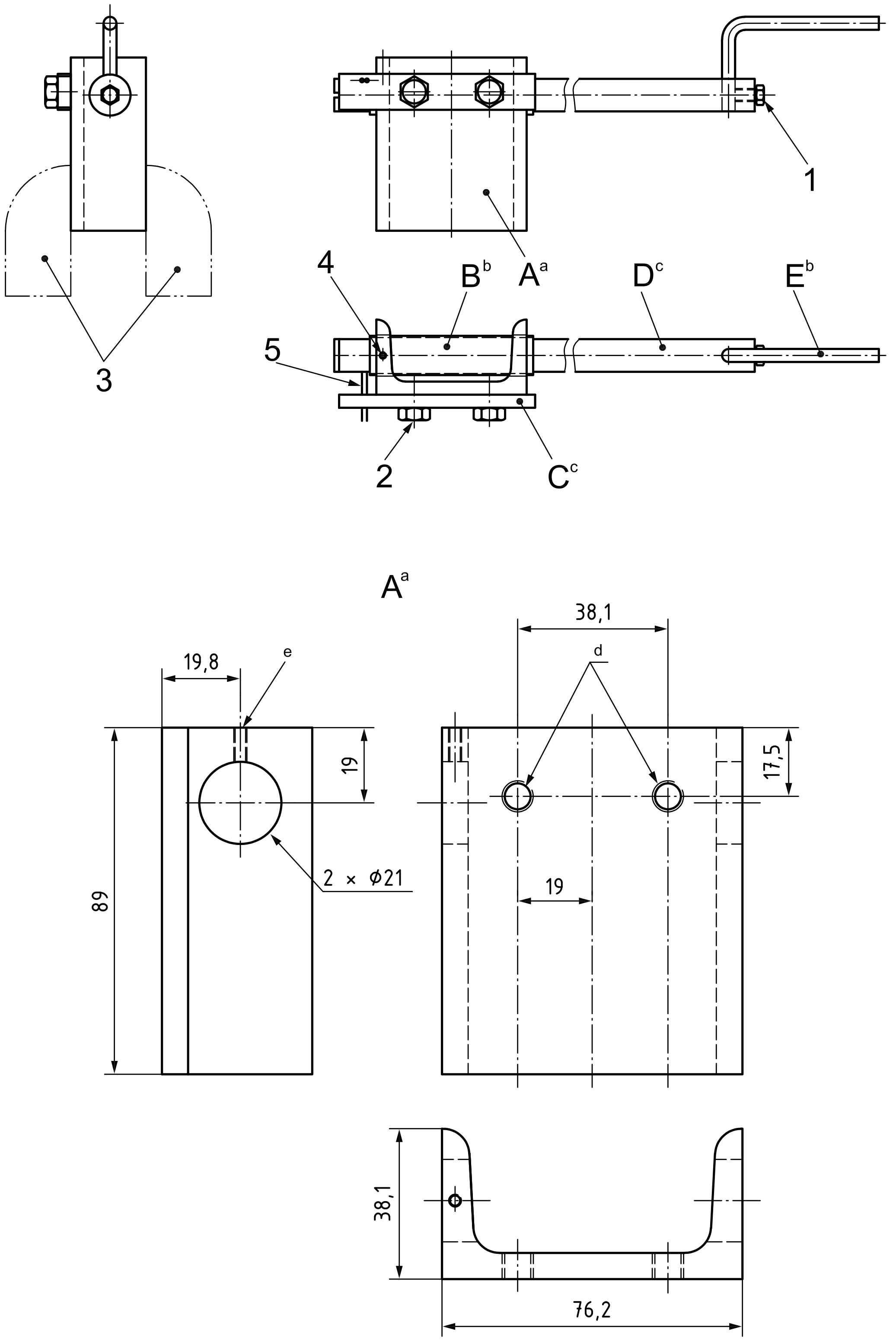


Figure 3 — Catalyst coil mandrel

Dimensions in millimetres
(unless otherwise indicated)

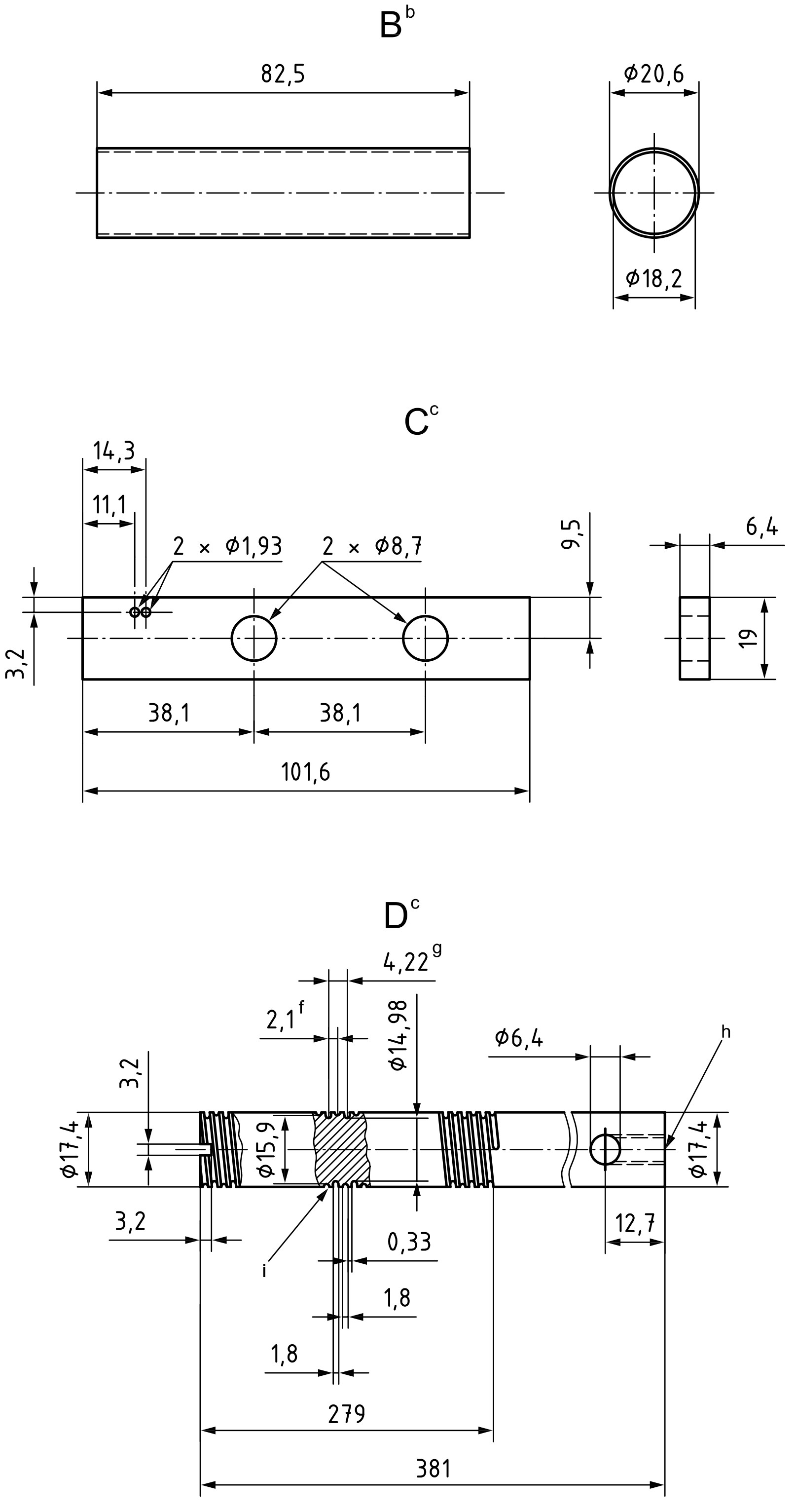
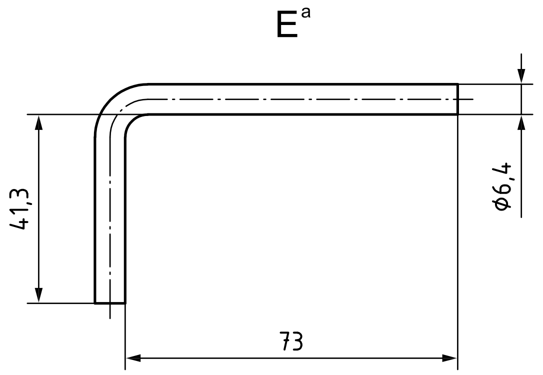


Figure 3 — Catalyst coil mandrel (*continued*)

Dimensions in millimetres
(unless otherwise indicated)



Key

1 1/4″ Whitworth × 12,7 hexagon head steel screw (or equivalent)
2 5/16″ Whitworth × 12,7 hexagon head steel screw (or equivalent)
3 vice jaws
4 2BA steel grub screw (or equivalent)
5 catalyst wires

[a] Material: steel.
[b] Material: brass.
[c] Material: bronze.
[d] Holes (2) tap 5/8″ Whitworth (or equivalent).
[e] Hole tap 2BA (or equivalent).
[f] Pitch.
[g] Pitch height.
[h] Hole tap 1/4″ Whitworth (or equivalent).
[i] 6 TPI double thread.

Figure 3 — Catalyst coil mandrel (*continued*)

Dimensions in millimetres
(unless otherwise indicated)

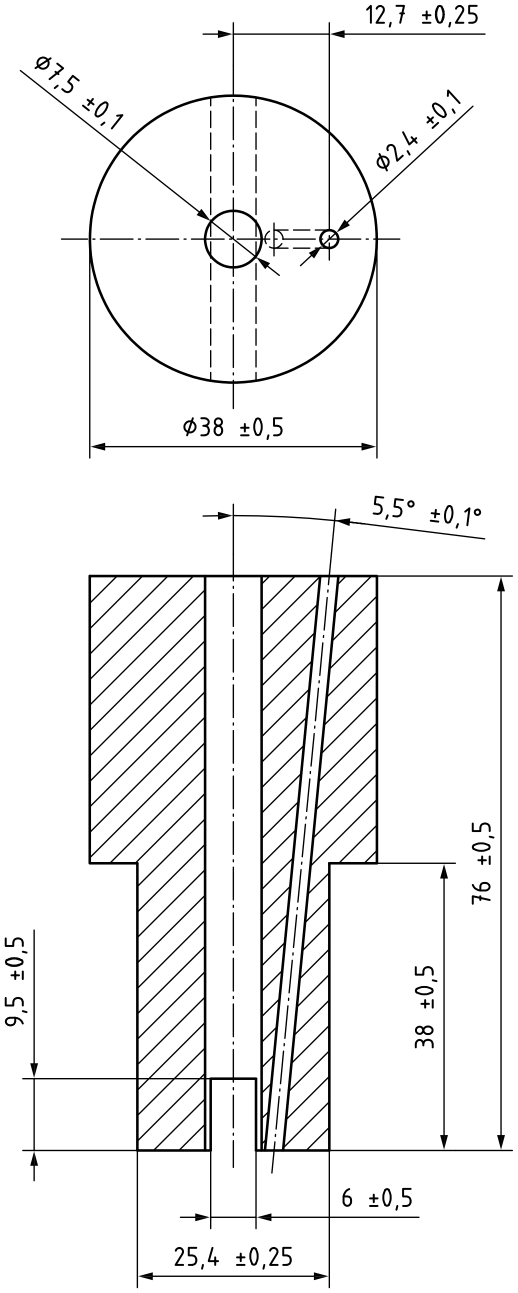


Figure 4 — Sampling tube holder

6 Sampling

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

7 Preparation of materials and apparatus

7.1 Cleaning the catalyst wire

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 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 Storage of the catalyst coil

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 or by 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 hydraulic fluid completely from the test tube and rinse all glassware with heptane (4.2) to remove traces of hydraulic fluid. 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 can 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 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 tube (5.9) and/or any other devices used and rinse any surfaces that have contacted the hydraulic fluid with heptane (4.2) to remove traces of hydraulic fluid. 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 hydraulic fluid in the oxidation cells (5.1) at 95,0 °C ± 0,2 °C throughout the duration of the test, with oxygen passing through the cells at 3,0 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, 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 subtracting 0,1 °C 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 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 after the temperature control has been established, remove the temperature-measurement devices.

8.4 Fill the empty oxidation test tube with 360 ml of hydraulic fluid. 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 into the test tube, and place the condenser over the oxygen delivery tube and test tube. If used, a sampling tube (5.9) and holder (5.11) 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.

NOTE As an alternative to using the sampling tube holder (5.11) and sampling tube spacer (5.12), the sampling tube (5.9) can be secured in position by taping to the oxygen-delivery tube using a suitable adhesive tape. The sampling tube is taped to the oxygen-delivery tube at a distance of approximately 25 mm above the top of the condenser. The bottom of the sampling tube is positioned at (150 ± 5) mm from the bottom curved end of the oxygen-delivery tube.

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 3,0 l/h ± 0,1 l/h.

8.6 Maintain the conditions for 30 min. Mark the level of the hydraulic fluid 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 hydraulic fluid at 95,0 °C ± 0,2 °C in each test cell.

8.8 At determined intervals (see the third 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 hydraulic fluid in the oxidation cell. Determine the acid number of the aliquot in accordance with the procedure described in ISO 7537, and if required, record the condition of the hydraulic fluid and catalysts.

Replace the condenser if removed, connect the oxygen-supply tube, and re-establish the test conditions.

It is normal to omit acid number determinations for the first 350 h of test, and thereafter to carry out determinations at intervals of every 150 h to 330 h. For hydraulic fluids 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 fluid under test is nearing the end of its oxidation life, when more frequent observations may be introduced. If the acid number has still not increased by 2,0 mg of KOH per gram of test portion at 10 000 h, it is unreasonable to continue the test, as the fluid will have been seriously distorted, and the catalyst coil might have become exposed.

8.9 Discontinue the test when the measured acid number has increased by 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:

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

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 the fluid, 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 increase of less than 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 of this test method for synthetic hydraulic fluids is not known because interlaboratory data are not available. This method may not be suitable for use in specifications or in the event of disputed results as long as these data are not available.

However, precision for inhibited turbine oils is given for guidance as an indication of the precision that could be obtained for synthetic hydraulic fluids. The precision for inhibited turbine oils was determined by statistical examination in accordance with ISO 4259[1] of interlaboratory test results. 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 ISO 6618[3] 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 20.

For inhibited turbine oils, $r = 0{,}192X$

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 20.

For inhibited turbine oils, $R = 0{,}332X$

where X is the average of the results being compared.

11.4 Reproducibility with duplicate tests

When testing inhibited turbine oils, 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 20.

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 described 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 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 and anhydrous synthetic fluids

C.1 General

This annex describes a procedure for the determination of the insolubles content of mineral oils and anhydrous synthetic fluids that have been the subject of the accelerated oxidation described in ISO 4263-1 and this part of ISO 4263, respectively.

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 l. 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/fluid 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 rotating speed of the centrifuge, n, in revolutions per minute, and the relative centrifugal force, F_{rc}, is given by the following equation:

$$n = 1\,337\sqrt{\left(F_{\text{rc}}/d\right)}$$

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.

C.3.9 **Analytical balance**, capable of weighing to the nearest 0,1 mg.

C.4 Procedure

C.4.1 At the end of the specified test period, disconnect the oxygen supply, remove the condenser and remove the oxidation apparatus from the heating bath. Wash the outside of the oxidation cell with heptane (4.2). Pour as much as possible of the hot contents of the oxidation cell into the separating funnel (C.3.3) and allow to cool and separate. Add sufficient heptane (approximately 300 ml) to the oxidation cell (5.1) to cover the catalyst coils.

C.4.2 If examination of the catalyst coil is required, remove it and the inlet tube from the heptane, and wash with clean heptane, allowing the washings to fall into the oxidation cell. Then inspect the coil, using the rating system given in Annex D if required. Return the coil to the oxidation cell until required (see C.4.5).

C.4.3 Using only the forceps (C.3.6), place a filter medium (C.3.2) into a weighing dish (C.3.7) and place in the oven (C.3.8), with the lid of the weighing dish open, for a minimum of 1 h. Cool in a covered vessel for 30 min and weigh to the nearest 0,1 mg. Place the membrane in the filter assembly (C.3.1).

C.4.4 If analysis of the oil/fluid is required, decant the required volume (normally 100 ml), through the neck of the separating funnel into a centrifuge tube (C.3.5) and centrifuge for 1 h at a relative centrifugal force of approximately 1 100. Decant the centrifuged oil/fluid from any deposit for analysis, and transfer the deposit to the separating funnel using small portions of heptane when the bulk of the oil/heptane or fluid/heptane mixture has been passed through the filter membrane.

C.4.5 Transfer the contents of the oxidation cell to the separating funnel and mix well. If any deposits remain on the inlet tube or catalyst coil, remove these carefully with a non-abrasive implement while washing with heptane, and transfer these deposits and washes to the funnel. Allow to stand out of direct sunlight for a minimum of 16 h.

NOTE The waiting period allows equilibrium to be established between the insoluble material and the oil/heptane or fluid/heptane, and allows the coalescence of the deposit into a more filterable form.

C.4.6 Filter the oil/heptane mixture or fluid/heptane mixture through the membrane filter, decanting the clearer mixture from the upper part of the contents first. Wash the separating funnel, centrifuge tube, oxidation cell and inlet tube with heptane and pass these washings through the filter. Wash the filter with heptane until free of oil/fluid.

NOTE Two or more membrane filters can be used if required.

C.4.7 With the vacuum applied, remove the funnel from the filter assembly and rinse the membrane on the base with a gentle stream of heptane, directing the stream from the edge towards the centre in order to remove final traces of oil/fluid without disturbing the deposit. Maintain the vacuum for a short time to remove the heptane. Transfer the membrane to the weighing vessel used in the initial weighing and dry for a minimum of 1 h in the oven (C.3.8). Cool for 30 min in a covered vessel and weigh to the nearest 0,1 mg.

C.5 Calculation

Subtract the initial mass of the membrane filter plus weighing vessel from the final mass.

C.6 Expression of results

Report the total mass of deposit at the end of the specified oxidation period to the nearest 1 mg, and the oxidation period to the nearest 1 h.

Annex D
(informative)

Appearance rating of catalyst coil wires

The rating system given in Table D.1 can be used to provide a qualitative indication of the corrosion properties of the fluid under test. Only the outside surface of the coil is rated.

Table D.1 — Appearance of catalyst coil wires

Rating	Description
Steel	
Bright	Freshly polished appearance
Tarnished	No red-brown rust specks of corrosion, but overall discoloration
Light rust	Rusting confined to not more than 6 spots
Moderate rust	Rusting confined to 7 to 12 spots
Severe rust	Rusting with 13 or more spots
Etched	Dull steel colour
Coated	Deposits on the wire prevent an estimate of the surface corrosion
Copper	
Bright	Freshly polished appearance
Tarnished	Moderate discoloration
Brown	Uniform light or dark brown colour
Green	Distinct green colour
Black	Very dark colour
Etched	Dull copper colour
Coated	Deposits on the wire prevent an estimate of the surface corrosion

Annex E
(informative)

Determination of metals content

E.1 General

Some manufacturers of equipment specify maximum levels of iron and copper in the three phases of material at the end of a test period. This may be in terms of the level in each, or one particular, phase, or in terms of total metal dissolved and/or absorbed. This annex does not give a definitive method for the determination of the metals content, which may be carried out by any suitable analysis technique agreed between supplier and user, but gives some guidelines for the preparation of the phases for analysis. Analysis techniques include atomic absorption spectrometry (AAS), inductively coupled plasma spectrometry (ICP), and X-ray fluorescence spectrometry (XRF).

E.2 Liquid phases

E.2.1 Carry out the procedure given in C.4. Retain the sludge deposits from the oil/heptane mixture or fluid/heptane mixture on the membrane in a separate flask.

E.2.2 If the liquid phases are homogenous and clear, determine the copper and iron contents directly by the agreed analysis technique. If the phases are non-homogenous or cloudy, evaporate the liquid in each safely, to a moist residue and follow the procedure given in E.3.

E.3 Sediment and residues

E.3.1 Digest the sediment on the filter membrane and/or the moist residues, in the minimum quantity of concentrated sulfuric acid and transfer to a porcelain, fused silica or platinum evaporating crucible. Heat at a low temperature, avoiding spattering, until no fumes evolve. Heat the crucible in a furnace at 775 °C ± 25 °C until no carbon is visible. Allow the crucible to cool, add three drops of water plus 10 drops of sulfuric acid (1 + 1) and move the crucible so that the entire residue is moistened. Repeat the low temperature heating and replace in the furnace for 30 min. Cool to room temperature.

E.3.2 The sulfated ash produced in accordance with E.3.1 can now be dissolved and digested for analysis for copper and iron content by the agreed analytical technique.

E.4 Expression of results

E.4.1 Separately calculate the copper content and iron content of each of the phases analysed, to the nearest 1 mg/kg up to 100 mg/kg, the nearest 10 mg/kg between 100 mg/kg and 1 000 mg/kg, and to the nearest 50 mg/kg for values in excess of 1 000 mg/kg.

E.4.2 Report the results in the format required, e.g. total copper plus iron or copper plus iron in oil/fluid.

Bibliography

[1] ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*

[2] ISO 4263-2:2003, *Petroleum and related products — Determination of the ageing behaviour of inhibited oils and fluids — TOST test — Part 2: Procedure for category HFC hydraulic fluids*

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

[4] ISO 12922:1999, *Lubricants, industrial oils and related products (class L) — Family H (Hydraulic systems) — Specifications for categories HFAE, HFAS, HFB, HFC, HFDR and HFDU*

[5] ISO 15380:2002, *Lubricants, industrial oils and related products (class L) — Family H (Hydraulic systems) — Specifications for categories HETG, HEPG, HEES and HEPR*

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