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

Part 2:
**Procedure for category HFC hydraulic
fluids**

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

ISO 4263-2:2003
Partie 2: Méthode pour les fluides hydrauliques de catégorie HFC

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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 4263-2 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 2: Procedure for category HFC hydraulic fluids

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 hydraulic fluids of category HFC as defined in ISO 6743-4 (see [2] in the Bibliography) and specified in ISO 12922 (see [3] in the Bibliography). The ageing is accelerated by the presence of oxygen, water and metal catalysts at elevated temperature, and the degradation of the fluid is followed by changes in pH value and insolubles content. Other parts of ISO 4263 specify similar procedures for the determination of the ageing behaviour of mineral oils and specified categories of fire-resistant fluids used in hydraulic and other applications.

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

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*

ISO 20843:—²⁾, *Petroleum and related products — Determination of pH of fire-resistant fluids within categories HFAE, HFAS and HFC*

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 pH value and insolubles content are measured. The test is continued until a pH of 4,0 is reached, an insolubles content of 4,0 % (m/m) is exceeded, or a 200 h duration has elapsed.

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

2) To be published.

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 (C₇H₁₆), of minimum purity 99,75 %.

4.3 Acetone (CH₃COCH₃), of general purpose reagent grade (GPR).

4.4 Propan-2-ol (CH₃CHOHCH₃), 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

A strong oxidizing acid cleaning solution, such as ammonium persulfate in concentrated sulfuric acid (8 g/l), or other proprietary strongly oxidizing solutions used in accordance with the manufacturer's instructions. 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.

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 $300 \text{ ml} \pm 1 \text{ ml}$, which applies to the test tube alone at $20 \text{ }^\circ\text{C}$. 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 $95 \text{ }^\circ\text{C} \pm 0,2 \text{ }^\circ\text{C}$. 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 $355 \text{ mm} \pm 10 \text{ mm}$. 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 $390 \text{ mm} \pm 10 \text{ mm}$. 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 $390 \text{ mm} \pm 10 \text{ mm}$.

5.3 Flowmeter, of minimum capacity 3 l/h and an accuracy of $\pm 0,1 \text{ l/h}$.

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 $\pm 0,1 \text{ }^\circ\text{C}$ and calibrated to better than $\pm 0,1 \text{ }^\circ\text{C}$. 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 $\pm 0,1 \text{ }^\circ\text{C}$ and calibrated to better than $\pm 0,1 \text{ }^\circ\text{C}$.

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.

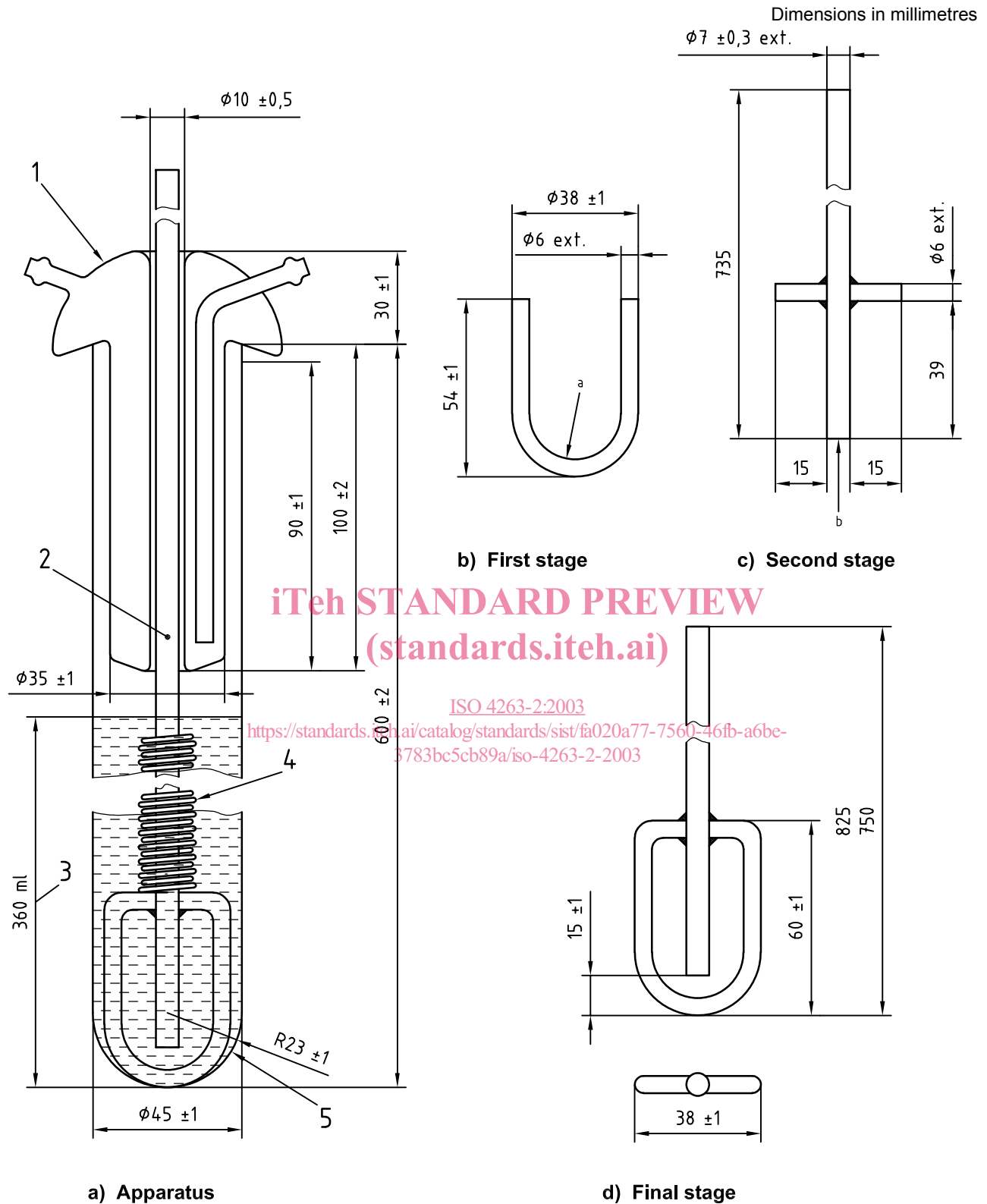
5.5 Wire-coiling mandrel. A mandrel, as illustrated in Figure 3, is used to produce the double spiral of copper and steel wire. The mandrel is included in a suitable winding device.

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 is 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 $\pm 0,2 \text{ ml}$.

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 Standard filter assembly, capable of holding the filter medium (5.10) securely on a filter support between the funnel and the vacuum flask. The vacuum flask shall be protected against implosion.

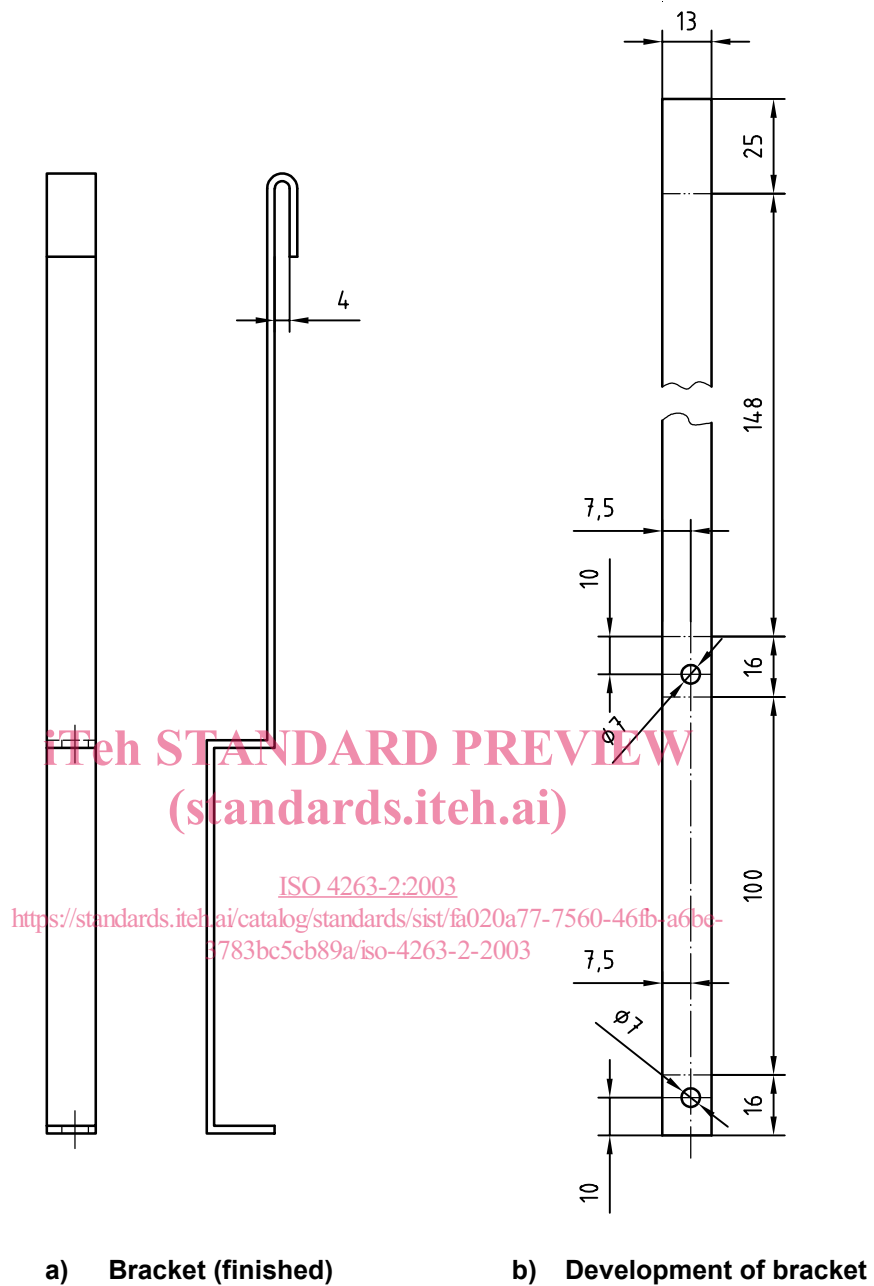


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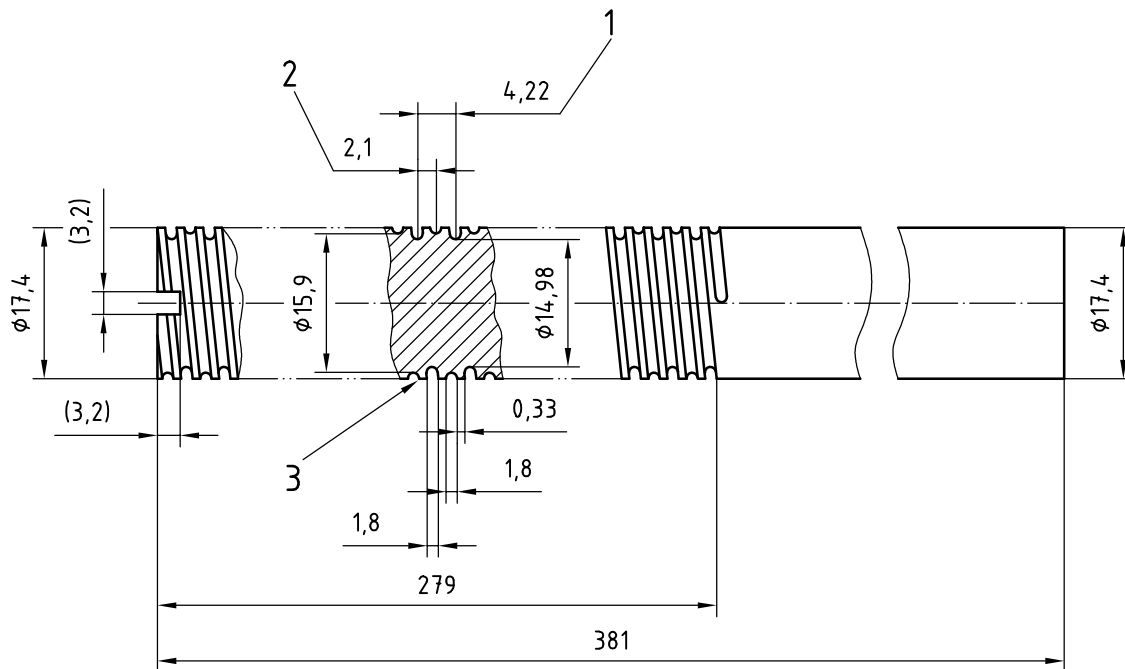
Figure 1 — Oxidation cell

Dimensions in millimetres



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

Figure 2 — Thermometer bracket



Material: bronze

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Key

- 1 lead
- 2 pitch
- 3 double thread

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Figure 3 — Catalyst-coil mandrel

5.10 Filter medium, of diameter 47 mm, consisting of membrane filters inert to the fluid under test and water, with a nominal pore diameter of 0,4 µm.

NOTE Nylon or glass fibre filters are likely to be most suitable. Cellulose ester membranes may not be inert to all fluids.

5.11 Drying oven, controlled at 105 °C ± 3 °C for drying the filter medium (5.10) and optionally for the glassware.

5.12 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.13 Graduated cylinder, of capacity 500 ml, capable of measuring 360 ml ± 5 ml.

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 \pm 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 \pm 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 the 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 the oven (5.11) 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 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 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 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.