Petroleum och relaterade produkter – 
Bestämning av åldringsegenskaper för inhiberade 
oljor och vätskor – TOST-provning – 
Del 2: Förfaringsätt för kategori HFC hydraulvätskor 
(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 
(ISO 4263-2:2003)
EN ISO 4263-2

Petroleum and related products - Determination of the ageing behaviour of inhibited oils and fluids - TOST test - Part 2:
Procedure for category HFC hydraulic fluids (ISO 4263-2:2003)

This European Standard was approved by CEN on 25 March 2003.

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Foreword

This document (EN ISO 4263-2:2003) has been prepared by Technical Committee ISO/TC 28 "Petroleum products and lubricants" in collaboration with Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 4263-2:2003 has been approved by CEN as EN ISO 4263-2:2003 without any modifications.

NOTE Normative references to International Standards are listed in Annex ZA (normative).
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 \((C_7H_{16})\), 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

A strong oxidizing acid cleaning solution, such as ammonium persulfate in concentrated sulfuric acid \((8 \text{ g/l})\), or other proprietary strongly oxidizing solutions used in accordance with the manufacturer’s instructions. A 10 % solution of three parts of hydrochloric acid \((1 \text{ mol/l})\) 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 \(1.60 \text{ mm} \pm 0.05 \text{ mm}\), made of carbon steel, soft bright annealed and free from rust.

4.7.2 Copper wire, of diameter \(1.63 \text{ mm} \pm 0.05 \text{ mm}\), 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 \(\mu\text{m} \) (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 hydraulic fluid 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.

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.

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 ± 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 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.