SLOVENSKI STANDARD
SIST EN ISO 4263-4:2006

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Nafta in sorodni proizvodi - Določanje staranja inhibiranih olj in tekočin - Preskus TOST - 4. del: Postopek za olja industrijskih naprav (ISO 4263-4:2006)

Petroleum and related products - Determination of the ageing behaviour of inhibited oils and fluids - TOST test - Part 4: Procedure for industrial gear oils (ISO 4263-4:2006)

Mineralölerzeugnisse und verwandte Produkte - Bestimmung des Alterungsverhaltens von inhibierten Ölen und Flüssigkeiten - TOST-Verfahren - Teil 4: Verfahren für Industriegetriebeöle (ISO 4263-4:2006)

Pétrole et produits connexes - Détermination du comportement au vieillissement des fluides et huiles inhibés - Essai TOST - Partie 4: Méthode pour les huiles pour engrenages industriels (ISO 4263-4:2006)

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Petroleum and related products - Determination of the ageing behaviour of inhibited oils and fluids - TOST test - Part 4: Procedure for industrial gear oils (ISO 4263-4:2006)

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This European Standard was approved by CEN on 9 January 2006.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Foreword

This document (EN ISO 4263-4:2006) 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of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

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

Endorsement notice

The text of ISO 4263-4:2006 has been approved by CEN as EN ISO 4263-4:2006 without any modifications.

INTERNATIONAL STANDARD

ISO 4263-4

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

Part 4: Procedure for industrial gear oils

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

Partie 4: Méthode pour les huiles pour engrenages industriels

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 4263-4 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 4: Procedure for industrial gear 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 gear oils of categories CKC, CKD, CKS and CKT as defined in ISO 6743-6[5]. The ageing is accelerated by the presence of air and elevated temperatures, and the degradation of the oil is assessed by the change in kinematic viscosity at 100 °C, the insoluble content (precipitation number), and possibly the acid number increase, the sediment content by filtration, the additives content variation and the infra-red oxidation.

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 2977:1997, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point*

ISO 3104:1994, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3170:2004, *Petroleum liquids — Manual sampling*

ISO 3405:2000, *Petroleum products — Determination of distillation characteristics at atmospheric pressure*

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

ISO 3734:1997, *Petroleum products — Determination of water and sediment in residual fuel oils — Centrifuge method*

ISO 12185:1996, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
insoluble content
precipitation number
number of millilitres of precipitate formed when 10 ml of lubricating oil are mixed with 90 ml of precipitation naphtha, and centrifuged under the conditions of the test

3.2
sediment by filtration
amount of insoluble matter, expressed in milligrams per 100 ml, retained on a filtration membrane of 1,2 µm pore size after filtration under vacuum

4 Principle

A test portion is reacted, in the absence of light, in the presence of air, and at a temperature appropriate to the type of industrial gear oil under test: 95 °C for CKC type; 121 °C for CKD type; 150 °C for CKS and CKT types. At the end of a 312 h period, the increase in kinematic viscosity at 100 °C, the insoluble content (precipitation number), the increase in acid number (if required), the sediment content by filtration, the additives depletion and the infra-red oxidation, are determined.

5 Reagents and materials

5.1 Water, unless otherwise specified, in accordance with the requirements of grade 2 as defined in ISO 3696:1987. Potable water means tap water, unless normal piped supplies are contaminated with particulate or highly soluble mineral content.

5.2 Heptane (C_7H_{16}), of reagent grade and of minimum purity 99,75 %.

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

5.4 Air, dried, oil-free, at constant pressure.

Supply from air cylinders or from a compressed air circuit is possible; this should be utilized via a regulation system to improve the consistency of the air flow.

5.5 Cleaning solutions

5.5.1 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 sulphuric 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 in contact with organic materials, but do not contain chromium which has special disposal problems.

5.5.2 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.

5.5.3 Laboratory detergent

The detergent shall be water soluble.

6 Apparatus

6.1 Oxidation cell, consisting of a large test tube of borosilicate glass, 600 mm in length, of 41 mm inside diameter, with a graduation mark to indicate a volume of 300 ml ± 1 ml at 20 °C, an air-delivery tube and a slotted cork stopper. The design and dimensions shall be as illustrated in Figure 1.

New corks should be used for each test.

6.2 Heating bath, consisting of a thermostatically controlled bath capable of maintaining the test oil portion in the oxidation cell at the specified temperature ± 0,2 °C. It shall be large enough to hold the required number of oxidation cells (6.1) immersed in the heat-transfer medium so that the bath liquid is at least 50 mm above the level of the oil sample. 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.

6.3 Flowmeters, capable of measuring a flow of air of 10 l/h with an accuracy of ± 0,5 l/h, one per oxidation cell is required.

6.4 Temperature-measurement devices

6.4.1 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 ± 0,2 °C and calibrated to better than ± 0,2 °C.

6.4.2 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 ± 0,2 °C and calibrated to better than ± 0,2 °C.

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

6.6 Air dryer. Between the air supply and the flowmeters, the air shall pass through a drying tower packed with indicating grade anhydrous calcium sulfate or equivalent. The quantity of desiccant shall be sufficient to last for the entire test.

7 Sampling

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

