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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 (ISO 4263-1:2003)

Mineralölerzeugnisse und verwandte Produkte - Bestimmung des Alterungsverhaltens von inhibierten Ölen und Flüssigkeiten - TOST-Verfahren - Teil 1: Verfahren für Mineralöle (ISO 4263-1:2003)

Pétrole et produits connexes - Détermination du comportement au vieillissement des fluides et huiles inhibées - Essai TOST - Partie 1: Méthode pour les huiles minérales (ISO 4263-1:2003)

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 4263-1

December 2004

ICS 75.100

English version

**Petroleum and related products - Determination of the ageing
behaviour of inhibited oils and fluids - TOST test - Part 1:
Procedure for mineral oils (ISO 4263-1:2003)**

Pétrole et produits connexes - Détermination du
comportement au vieillissement des fluides et huiles
inhibées - Essai TOST - Partie 1: Méthode pour les huiles
minérales (ISO 4263-1:2003)

Mineralölerzeugnisse und verwandte Produkte -
Bestimmung des Alterungsverhaltens von inhibierten Ölen
und Flüssigkeiten - TOST- Verfahren - Teil 1: Verfahren für
Mineralöle (ISO 4263-1:2003)

This European Standard was approved by CEN on 21 December 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.



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

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

EN ISO 4263-1:2004 (E)**Foreword**

The text of ISO 4263-1:2003 has been prepared by Technical Committee ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 4263-1:2004 by 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 June 2005, and conflicting national standards shall be withdrawn at the latest by June 2005.

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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 4263-1:2003 has been approved by CEN as EN ISO 4263-1:2004 without any modifications.

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INTERNATIONAL STANDARD

ISO
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2003-03-15

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

Part 1: Procedure for mineral oils

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

Partie 1: Méthode pour les huiles minérales

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Reference number
ISO 4263-1:2003(E)

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Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Normative references	1
3 Principle	2
4 Reagents and materials.....	2
5 Apparatus	3
6 Sampling	6
7 Preparation of materials and apparatus	6
8 Procedure	8
9 Calculation	9
10 Expression of results.....	9
11 Precision	9
12 Test report	10
Annex A (normative) Liquid-in-glass thermometer specifications	11
Annex B (normative) Procedure for packaging and storage of catalyst coils	12
Annex C (informative) Method for the determination of the insolubles content of mineral oils	13
Annex D (informative) Appearance rating of catalyst coil wires.....	15
Annex E (informative) Determination of metals content	16
Bibliography	17

ISO 4263-1:2003(E)

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*

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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

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*

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

ISO 4263-1:2003(E)

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 (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**

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 $1,60 \text{ mm} \pm 0,05 \text{ mm}$, 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 $150 \text{ }\mu\text{m}$ (100-grit) with a cloth backing, or an equivalent grade of abrasive cloth.

4.9 Absorbent cotton

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5 Apparatus <https://standards.iteh.ai/catalog/standards/sist/8e28d5c8-49ef-4f34-9f5c-5c891a4b453b/sist-en-iso-4263-1-2005>

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 oil 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.