



# SLOVENSKI STANDARD

## SIST EN 14832:2005

01-september-2005

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Petroleum and related products - Determination of the oxidation stability and corrosivity of fire-resistant phosphate ester fluids

Mineralölerzeugnisse und verwandte Produkte - Bestimmung der Oxidationsbeständigkeit und der Einwirkung auf Metallwerkstoffe von schwerentflammaren Flüssigkeiten auf der Basis von Phosphorsäureestern

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Pétrole et produits connexes - Détermination de la stabilité à l'oxydation et de la corrosivité des fluides difficilement inflammables à base d'esters phosphates

Ta slovenski standard je istoveten z: EN 14832:2005

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**ICS:**

75.080      Naftni proizvodi na splošno      Petroleum products in general

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en

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EUROPEAN STANDARD

EN 14832

NORME EUROPÉENNE

EUROPÄISCHE NORM

June 2005

ICS 75.080

English version

## Petroleum and related products - Determination of the oxidation stability and corrosivity of fire-resistant phosphate ester fluids

Pétrole et produits connexes - Détermination de la stabilité à l'oxydation et de la corrosivité des fluides difficilement inflammables à base d'esters phosphates

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This European Standard was approved by CEN on 25 May 2005.

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.

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## Foreword

This European Standard (EN 14832:2005) has been prepared 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 December 2005, and conflicting national standards shall be withdrawn at the latest by December 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.

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## 1 Scope

This European Standard specifies a method for assessing the oxidation stability of phosphate ester hydraulic fluids. These products fall into category HFDR of EN ISO 6743-4 [2] and into categories TSD, TGD and TCD of ISO 6743-5 [3]. The amount of acid developed during the test and the mass changes of the metal specimens are used to assess the level of oxidation stability.

The precision of the test method applies to changes in acid number up to 3,0 mg KOH/g and changes in mass of up to 3 mg per test piece. The change of acidity is determined using one of the test methods ISO 6618, ISO 6619, or ISO 7537. Results from two different test methods are not necessarily compatible and direct comparison needs caution.

NOTE This test method may also be applied to other non-aqueous fire-resistant fluids, such as those falling into category HFDU of EN ISO 6743-4 [2].

**WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

## 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.

EN 10277-2, *Bright steel products — Technical delivery conditions — Part 2: Steels for general engineering purposes*

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EN ISO 2160, *Petroleum products — Corrosiveness to copper — Copper strip test (ISO 2160:1998)*

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

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

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

ISO 6619, *Petroleum products and lubricants — Neutralization number — Potentiometric titration method*

ISO 7537, *Petroleum products — Determination of acid number — Semi-micro colour-indicator titration method*

## 3 Terms and definitions

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

### 3.1

#### **oxidation stability**

sum of the changes in acid number of the fire-resistant fluid and of water in the absorption vessel when subjected to the accelerated oxidizing conditions specified in this document

### 3.2

#### **corrosivity**

change in mass of specified metal test pieces subjected to the accelerated oxidizing conditions specified in this document

## 4 Principle

A test portion of fluid, of known acid number, is placed in a test vessel, which contains cleaned and weighed copper and steel test pieces. The fluid and test pieces are then subjected to a temperature of 120 °C under an oxygen flow rate of 1 l/h for 164 h. The exhausted gaseous acidic products of the oxidation are absorbed in water, and the acid number of this phase is determined. The acid number of the test portion and the mass of the test pieces are determined at the end of the test period, and the results are calculated as the sum of the changes in acid number, and/or the change in mass of the individual test pieces.

## 5 Reagents and materials

Unless otherwise specified, reagents shall be of recognized analytical grade.

**5.1 Water**, conforming to the requirements of grade 3 of EN ISO 3696.

**5.2 Oxygen**, Minimum purity 99,4 %.

**5.3 Phenolphthalein**, 1 g/l ethanolic solution.

**5.4 Potassium hydroxide**, 0,1 mol/l ethanolic solution.

**5.5 Wash solvent**, use either 2,2,4-trimethylpentane or petroleum spirit with a boiling range of 60 °C to 80 °C for the metal test pieces.

**5.6 Acetone**.

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**CAUTION — Acetone is a flammable liquid and should be handled with appropriate care.**

**5.7 Strong oxidizing cleaning solution**.

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The reference oxidizing cleaning solution, on which precision was based, is chromsulfuric acid (see the warning statement below), but alternative non-chromium containing solutions, such as ammonium persulfate in concentrated sulfuric acid (8 g/l) have also been found to give satisfactory cleanliness. Other non-alkaline laboratory cleaning agents that are demonstrated to give equivalent cleanliness to chromsulfuric acid, may be used for routine analysis.

**CAUTION — Chromsulfuric 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 chromsulfuric 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 waste (chromium is highly dangerous to the environment).**

Non-chromium containing, strong oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.

**5.8 Abrasives**, silicon carbide paper or cloth of 65 µm (grade 240) and of 30 µm (grade 600) grit size.

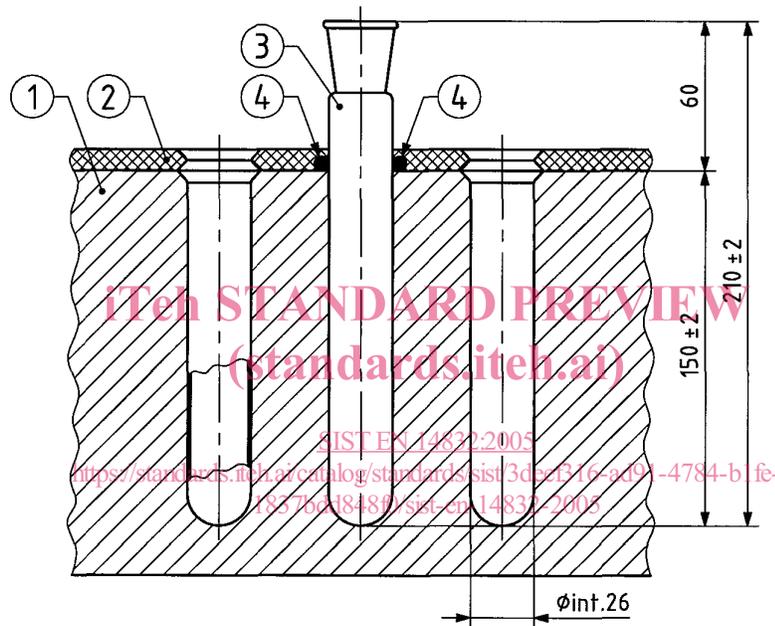
**5.9 Drying agent**, molecular sieve with a pore size of  $4 \times 10^{-4}$  µm or other equivalent means for drying the oxygen (5.2).

## 6 Apparatus

### 6.1 Heating block or heating bath

Consisting of an aluminium block (see Figure 1) or an oil bath (see Figure 2), capable of maintaining a temperature in the test portion of  $120\text{ °C} \pm 0,5\text{ °C}$ . It shall be fitted with an outer cover of non-asbestos thermal insulating material such that the surface temperature does not exceed  $60\text{ °C}$ . The design of the bath and test vessel supports shall ensure that the test vessel is heated over a length of 150 mm.

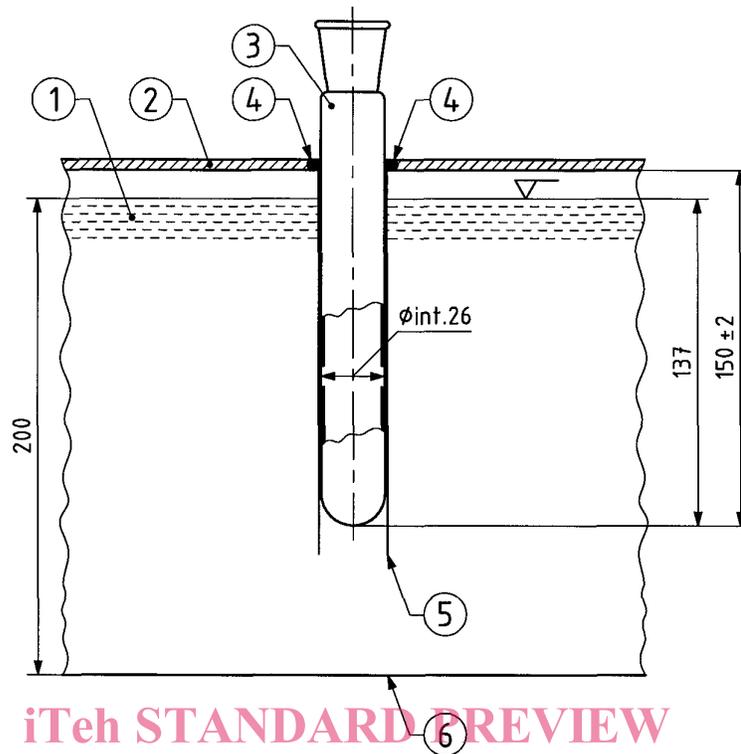
In the case of an aluminium heating block, there shall not be a gap between the test vessel and the thermal insulation. In the case of an oil bath, the test vessel shall be immersed to a depth of 137 mm, and the distance from the bottom of the test vessel to the underside of the bath cover shall be  $150\text{ mm} \pm 2\text{ mm}$ . The gap between test vessel and bath cover shall be sealed by means of an O-ring.



#### Key

- |                      |               |
|----------------------|---------------|
| 1 Aluminium block    | 3 Test vessel |
| 2 Thermal insulation | 4 O-ring      |

Figure 1 — Heating block



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#### Key

- |   |             |   |                     |
|---|-------------|---|---------------------|
| 1 | Oil         | 4 | O-ring              |
| 2 | Bath cover  | 5 | Test vessel support |
| 3 | Test vessel | 6 | Bottom heating bath |

Figure 2 — Oil bath

### 6.2 Test vessels

Constructed of borosilicate glass, of length  $210 \text{ mm} \pm 2 \text{ mm}$ , outer diameter  $28 \text{ mm} \pm 0,5 \text{ mm}$ , wall thickness  $2 \text{ mm} \pm 0,2 \text{ mm}$ , and fitted with a female tapered ground-glass joint of either 24/29 or 24/39 size. A second test vessel is used as the absorption vessel.

### 6.3 Inserts

Constructed of borosilicate glass, with a male tapered ground-glass joint to fit into the test vessel and absorption vessel. They are equivalent to a wash-bottle insert, carrying a glass inlet tube of outside diameter  $5 \text{ mm} \pm 0,4 \text{ mm}$  and a wall thickness of  $0,8 \text{ mm} \pm 0,1 \text{ mm}$ , with the bottom tip bevelled at  $30^\circ$ . The distance between the tip of the inlet tube and the bottom of the test vessel shall be  $2,5 \text{ mm} \pm 0,5 \text{ mm}$  when fitted. The inserts shall be fitted with a joint connector of suitable gas-tight design, such that two inserts fitted together give a horizontal distance of 100 mm to 200 mm between the test vessel and absorption vessel. Other joint connectors or connections like PTFE tubing may be used if they fulfil the requirements of this clause. The general arrangement is illustrated in Figure 3.