BRITISH STANDARD

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14832:2005

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

The European Standard EN 14832:2005 has the status of a British Standard

ICS 75.080

National foreword

This British Standard is the official English language version of EN 14832:2005.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

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Summary of pages

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14832

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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

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

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

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

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

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Contents

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of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.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.

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*

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.

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

5.7 Strong oxidizing cleaning solution.

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.

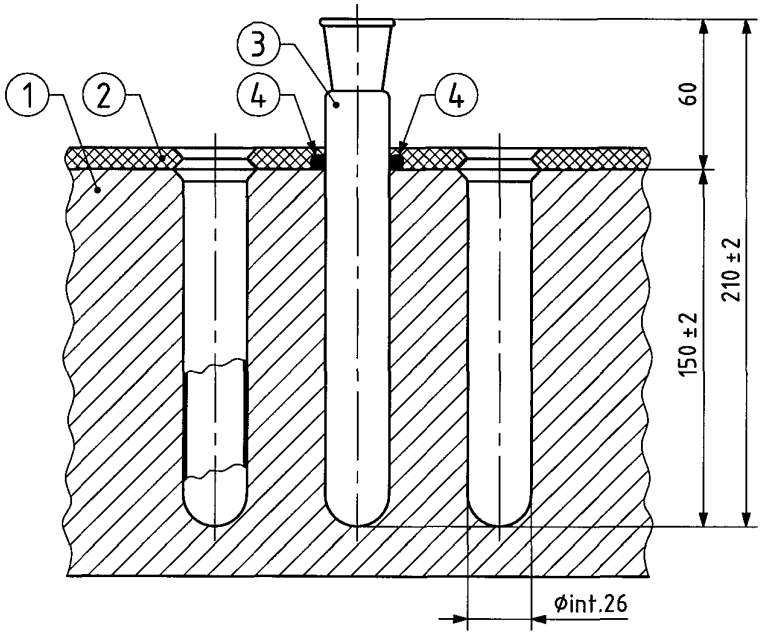
5.9 Drying agent, molecular sieve with a pore size of 4×10^{-4} μm or other equivalent means for drying the oxygen (5.2).

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 °C ± 0,5 °C. It shall be fitted with an outer cover of non-asbestos thermal insulating material such that the surface temperature does not exceed 60 °C. The design of the bath and test vessel supports shall ensure that the test vessel is heated over a length of 150 mm.

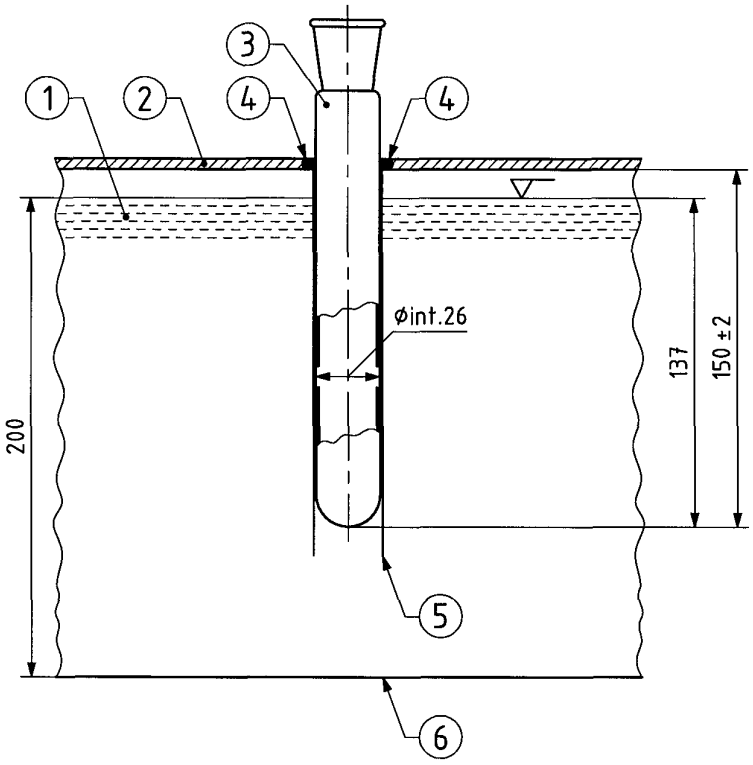
In the case of an aluminium heating block, there shall not be a gap between the test vessel and the thermal insulation. In the case of an oil bath, the test vessel shall be immersed to a depth of 137 mm, and the distance from the bottom of the test vessel to the underside of the bath cover shall be 150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



Key

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

Figure 1 — Heating block



Key

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

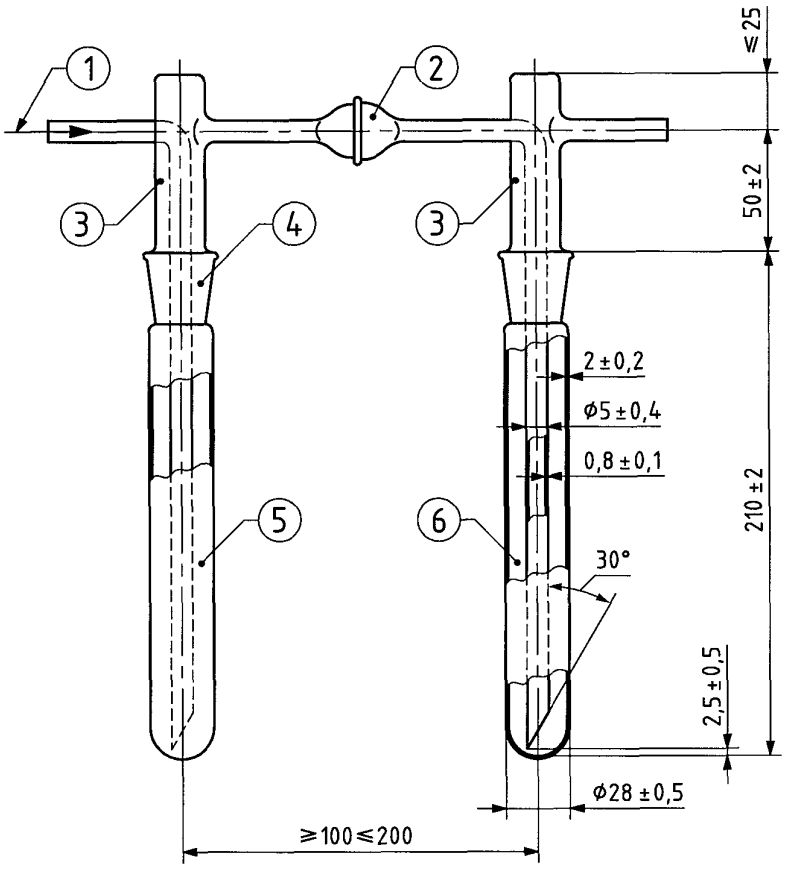
Figure 2 — Oil bath

6.2 Test vessels

Constructed of borosilicate glass, of length 210 mm ± 2 mm, outer diameter 28 mm ± 0,5 mm, wall thickness 2 mm ± 0,2 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 mm ± 0,4 mm and a wall thickness of 0,8 mm ± 0,1 mm, with the bottom tip bevelled at 30°. The distance between the tip of the inlet tube and the bottom of the test vessel shall be 2,5 mm ± 0,5 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.



Key

1 Oxygen inlet
2 Joint connector
3 Insert
4 Female tapered ground glass joint
5 Test vessel
6 Absorption vessel

Figure 3 — General arrangement of apparatus

6.4 Flowmeter, capable of measuring a flow rate of 1 l/h to the nearest 0,1 l/h.

NOTE A suitable soap bubble flowmeter is illustrated in Figure 4.

6.5 Analytical balance, capable of weighing to the nearest 0,1 mg or better.

6.6 Copper test pieces, constructed from the copper specified in EN ISO 2160, of dimensions 60 mm × 10 mm × 2 mm.

6.7 Steel test pieces, manufactured from carbon steel of quality EN 10277-2 or equivalent, of dimensions 60 mm × 10 mm × 2 mm.

6.8 Cotton wool

6.9 Cleaning brushes, small, hard and nylon-bristled.

NOTE A toothbrush is suitable.

6.10 Oven, capable of being controlled at 100 °C ± 2 °C.

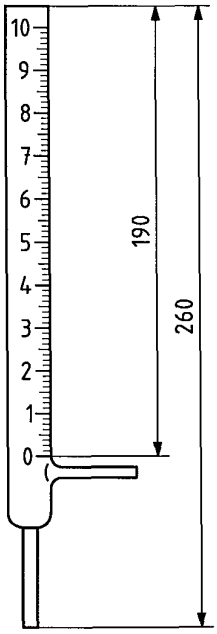


Figure 4 — Soap bubble flowmeter

7 Samples and sampling

7.1 Unless otherwise specified samples shall be taken as described in EN ISO 3170 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

7.2 Laboratory samples shall be mixed thoroughly by shaking vigorously for 30 s before taking the test portion. The test portion shall not be filtered.

8 Preparation of glassware

8.1 The pre-cleaned test vessels shall be stored for at least 6 h in cleaning solution (5.7).

8.2 Rinse thoroughly in tap water, followed by water (5.1).

8.3 Dry for 30 min in the oven (6.10), and cool to room temperature before weighing.

9 Preparation of metal test pieces

9.1 Carefully segregate the polishing materials and solvent to ensure that there is no transfer of removed material between the two metals. Wear gloves or use forceps for all handling of the test pieces.

9.2 Polish the metal surfaces and edges with 65 μm silicon carbide paper or cloth (5.8) until there is no more significant roughness and a fresh metal surface is exposed.

9.3 Polish the metal surfaces and edges with 30 μm silicon carbide paper or cloth (5.8) until they are smooth and even. Use a new piece of paper or cloth for the final polish.

9.4 Rub the surface with cotton wool (6.8) soaked in wash solvent (5.5) until all loose particles are removed.

9.5 Wipe the surfaces with cotton wool soaked in acetone (5.6).

9.6 Air-dry the test pieces, and weigh to the nearest 0,1 mg.

NOTE Prepared test pieces may be kept for up to 8 h in stoppered bottles of wash solvent before final preparation, drying and weighing.

10 Procedure

10.1 Determine the acid number of the sample in accordance with the procedure specified in ISO 6618, ISO 6619 or ISO 7537.

10.2 Neutralize the water (5.1) by titration, using phenolphthalein (5.3) as indicator.

10.3 Add 25 ml ± 0,2 ml of neutralized water (10.2) to the (absorption) test vessel (6.2), and mark the level on the outside of the vessel.

10.4 Weigh the test vessel (6.2) to the nearest 0,1 mg, and add 25 g ± 0,2 g of the test portion. Re-weigh.

10.5 Assemble the apparatus with one of each of the test pieces inserted into the test vessel so that they are separated from each other by the oxygen inlet tube.

10.6 Place the test vessel in the pre-heated heating bath (6.1), with the test vessel connected to the absorption vessel outside the bath.

10.7 Connect the oxygen inlet tube to the oxygen supply, and set the oxygen flow rate to 1 l/h ± 0,1 l/h. Note the time of the start of oxygen delivery as the start of the test.

10.8 During the test, check at least daily, the oxygen flow rate and the level of the water in the absorption vessel. If water has evaporated, make up to the mark with neutralized water (10.2). Re-adjust the oxygen flow rate as necessary.

10.9 After 164 h ± 2 h, remove the test vessel from the heating bath, leaving the apparatus assembled to cool to room temperature.

10.10 After cooling, remove a test portion of the aged test portion and determine the acid number in accordance with the procedures specified in one of the titration methods mentioned in Clause 1.

10.11 Remove the water from the absorption vessel and titrate it back to the same point of neutralization (10.2) with ethanolic potassium hydroxide (5.4). Calculate the acid number of the water related to the mass of fluid test portion according to Clause 11.

10.12 Remove the test pieces from the test vessel, rinse with acetone (5.6), brush under running tap water with the cleaning brush (6.9), rinse again with acetone, air-dry and weigh to the nearest 0,1 mg.

11 Calculation

11.1 Calculate the oxidation stability, OS, expressed in milligrams of KOH per gram, as the sum of the difference in acid number of the sample before and after ageing, and the acid number before and after ageing of the water (10.11) according to

$$OS = (A_2 - A_1) + (A_4 - A_3) \quad (1)$$

where

— A_1 is the acid number of the sample before ageing (10.1), expressed to the nearest 0,01 mg KOH per gram;

— A_2 is the acid number of the sample after ageing (10.10), expressed to the nearest 0,01 mg KOH per gram;

— A_3 is the acid number of the water before ageing (10.2), expressed to the nearest 0,01 mg KOH per gram, which should be close to zero after correct execution of the neutralization step described in (10.2);

— A_4 is the acid number of the water after ageing (10.11), expressed to the nearest 0,01 mg KOH per gram.

11.2 Calculate the change in mass of the test pieces by weighing to the nearest 0,1 mg.

12 Expression of results

12.1 Report the following:

a) the acid numbers A_1, A_2, A_3 and A_4 to the nearest 0,01 mg KOH/g (see 11.1);

b) the oxidation stability expressed in milligrams of KOH per gram as calculated in 11.1.

12.2 Report, to the nearest 0,1 mg, the following:

a) the change in mass of the copper test piece;

b) the change in mass of the steel test piece.

13 Precision

13.1 General

The precision, as obtained by statistical examination of inter-laboratory test results according to EN ISO 4259 [1] is given in 13.2 and 13.3. The precision of the acid value determination is given in the corresponding titration test method.

13.2 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in twenty.

13.3 Reproducibility

The difference between two single and independent test results obtained by different operators working in different laboratories on identical material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in twenty.

Table 1 — Precision values

Oxidation stability *mg KOH/g*		**Repeatability**	**Reproducibility**
up to 0,3		0,10	0,20
above 0,3 to 1,5		0,15	0,40
above 1,5 to 3,0		0,30	0,70
Mass change of test pieces *mg*			
Copper	up to 2	0,8	1,0
Copper	above 2 to 3	1,0	2,0
Steel	up to 1	0,4	0,5

14 Test report

The test report shall contain at least the following information:

a) reference to this document;

b) type and complete identification of the product tested;

c) results of the test (see Clause 12);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) date of the test.

NOTE The results of a visual inspection of the test pieces, like discoloration, etching, occurrence of pits, etc. can also provide valuable information.

Bibliography

[1] EN ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor 1:1993)*

[2] EN ISO 6743-4, *Lubricants, industrial oils and related products (class L) — Classification— Part 4: Family H (Hydraulic systems) (ISO 6749-4:1999)*

[3] ISO 6743-5, *Lubricants, industrial oils and related products (class L) — Classification — Part 5: Family T (Turbines)*

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