
**Petroleum products and lubricants —
Inhibited mineral turbine oils —
Determination of oxidation stability**

*Produits pétroliers et lubrifiants — Huiles minérales inhibées pour
turbines — Détermination de la stabilité à l'oxydation*



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.

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.

International Standard ISO 7624 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annexes A, B and C form an integral part of this International Standard.

© ISO 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

Petroleum products and lubricants — Inhibited mineral turbine oils — Determination of oxidation stability

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

1 Scope

This International Standard specifies a method for the determination of the resistance to oxidation under specified conditions of unused inhibited mineral turbine oils. The method is also applicable to other types of oil, such as hydraulic oil.

NOTE — This method may be used for specification purposes, and is of value in estimating the oxidation stability of turbine lubricants in the laboratory. It should be recognized, however, that correlation between the results of any single method of test for oxidation stability and the stability of an oil in service will vary with service conditions.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*.

3 Principle

Oxygen is passed for 164 h through a sample of the oil with added soluble metal catalysts (iron and copper) and maintained at 120 °C. The volatile acid products, the acidity of the oil, and the sludge formed are determined. If it is required to measure the length of time to obtain a pronounced increase in the rate of evolution of volatile acids (induction period), an acidity/time curve is obtained by daily determinations of the volatile acids.

4 Reagents and materials

During the analyses, use only reagents of recognized analytical grade and water meeting the requirements of ISO 3696 Grade 2 or water of equivalent purity, unless otherwise specified.

4.1 Catalysts

Prepare 1 % (*m/m*) solutions of copper naphthenate and iron naphthenate by dilution of master solutions which conform to annex A. After dilution, the solutions shall have a total trace metals content (other than sodium) of not greater than 300 mg/kg. Use mineral oil containing approximately 5 % aromatic carbon as the diluent for the working solutions. Store this solution in the dark.

NOTE — High boiling turbine base oil of ISO VG 32 viscosity is recommended.

Do not use white paraffinic oils because of the lower solubility of the iron naphthenate.

4.2 Oxygen, commercial product (minimum purity 99,4 %).

The oxygen shall be dried by passing through a suitable desiccant. The oxygen delivery system shall be constructed such that a smooth flow of oxygen is delivered to the measuring device. At the beginning of each test-run, the oxygen flow-rate given by the measuring device, shall be set to $1,0 \text{ l/h} \pm 0,1 \text{ l/h}$ and checked with the soap bubble flowmeter (5.6), and from there on, when carrying out daily flow checks, the rate shall be maintained at $1,0 \text{ l/h} \pm 0,1 \text{ l/h}$ for the duration of the test, and checked by the soap bubble meter if considered necessary.

WARNING — Use only with equipment conditioned for oxygen service by careful cleaning to remove oil, grease and other combustibles. Do not use oils or grease on regulators, gauges or control equipment. If a leak in the oxygen supply pipework is suspected, turn off immediately and seek qualified assistance. Depressurize the pipework prior to investigating a leak. The oxygen supply system should be checked regularly.

4.3 Alkali blue, 20 g/l solution in ethanol (4.10).

To obtain a good end-point with alkali blue indicator, after dissolving in ethanol (4.10), add 0,03 ml of HCl (4.6) to each 1 ml of indicator solution. After 24 h, carry out a check acid value determination to ensure that sensitization has occurred, i.e. a distinct colour change from a blue to a red which is comparable to that of a 10 % solution of cobalt nitrate $[\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$.

4.4 Phenolphthalein, 10 g/l alcoholic indicator solution.

4.5 Heptane, analytical grade, or of equal or greater purity.

4.6 Hydrochloric acid, aqueous solution of 0,1 mol/l HCl.

4.7 Potassium hydroxide, standard volumetric ethanolic solution of $c_{\text{KOH}} = 0,1 \text{ mol/l}$. Standardize daily or as appropriate.

4.8 Toluene.

4.9 Dichloromethane. Dispose of waste dichloromethane in an approved manner.

4.10 Ethanol, denatured, containing approximately 5 % (V/V) water.

4.11 Sulfuric acid, concentrated, 98 % (*m/m*) minimum H_2SO_4 .

5 Apparatus

Ordinary laboratory apparatus and

5.1 Oxidation tubes, borosilicate or neutral glass conforming to the dimensions shown in figure 1.

Dimensions in millimetres

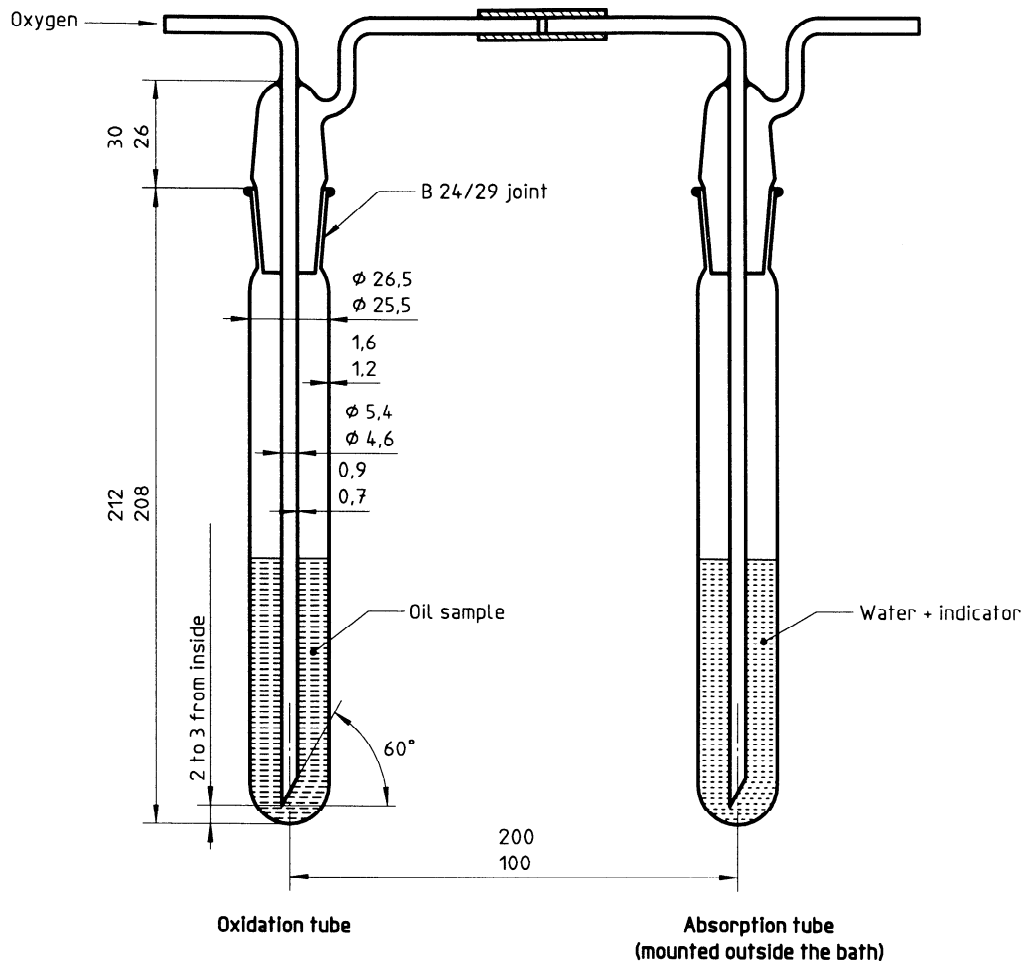


Figure 1 — Oxidation and absorption tubes

5.2 Absorption tubes, identical to the oxidation tubes (5.1).

The connection between oxidation and absorption tubes shall be as short as possible and made by means of short flexible silicone rubber sleeves (length between axis of the two tubes $150 \text{ mm} \pm 50 \text{ mm}$). These tubes shall be mounted outside the bath.

5.3 Heating bath, aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the desired number of oxidation tubes at a temperature of $120^\circ\text{C} \pm 0,5^\circ\text{C}$ (see figure 2).

Read the temperature from a type B thermometer or a temperature measuring device (5.8), inserted in a test tube to within 5 mm from the bottom. Fill the test tube up to the immersion line of the thermometer with the oil under test, and place in the heating bath.

The temperature of the upper surface shall be kept at $60^\circ\text{C} \pm 5^\circ\text{C}$ (see note) and measured using a type A thermometer or a temperature measuring device (5.8) (see figure 2). The surfaces of the block, other than that against the upper surface of the heating bath, shall be protected by suitable insulation. This block shall be placed as near to the holes as practicable and within the area of the aluminium heater block.

NOTE — $60^\circ\text{C} \pm 5^\circ\text{C}$ applies for an ambient temperature of 20°C , which should be used for assessing equipment compliance. Operation outside $60^\circ\text{C} \pm 5^\circ\text{C}$ due to extremes of ambient temperature has only a slight effect on test results.