
**Petroleum products — Electrical insulating
oils — Detection of corrosive sulfur**

*Produits pétroliers — Huiles d'isolation électrique — Détection du soufre
corrosif*



Foreword

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International Standard ISO 5662 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 5662:1978), which has been technically revised.

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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

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Introduction

Mineral insulating oils may contain substances that cause corrosion under certain conditions of use. The test method specified in this International Standard is designed to detect unacceptable quantities of free sulfur and corrosive sulfur compounds.

In most of their uses, insulating oils are continually in contact with metals that are subject to corrosion. Since the presence of detrimental corrosive sulfur compounds will result in deterioration of these metals to an extent dependent upon the quantity and type of corrosive agent and the time and temperature factors, the detection of these undesirable impurities, even though not in quantitative terms, is a means for recognizing the hazard involved.

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Petroleum products - Electrical insulating oils - Detection of corrosive sulfur

WARNING - The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this 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 detection of corrosive sulfur compounds in electrical insulating oils of petroleum origin.

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 2160: —¹⁾, *Petroleum products - Corrosiveness to copper - Copper strip test*.

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

3 Principle

A strip of polished pure copper is subjected to contact with the oil under test at 140 °C in the absence of air. At the end of the test period, the strip is examined for colour change, and assessed on the basis of pass or fail by reference to a table of potential discolorations.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade.

4.1 Wash solvent: 2,2,4-trimethylpentane of minimum purity 99,75 %.

NOTE 1 Other sulfur-free volatile hydrocarbon solvents which show no tarnish when tested by ISO 2160 for 3 h at 50 °C, are suitable.

4.2 Acetone, sulfur-free.

4.3 Diethyl ether.

4.4 Nitrogen, commercial grade, oxygen-free.

4.5 Water: Unless otherwise described, water shall be of a purity equivalent to grade 3 of ISO 3696.

5 Apparatus

5.1 Heating medium, consisting of a hot-air oven or oil bath, capable of being heated to, and controlled at, 140 °C ± 2 °C.

NOTE 2 A circulating hot-air oven is preferred.

5.2 Bottles, nominally 250 ml, made of chemically resistant glass, with narrow mouths and fitted with ground glass stoppers, capable of holding 270 ml to 280 ml when filled completely to the stopper.

1) To be published. (Revision of ISO 2160: 1985)

NOTE 3 Bottles of such capacity are required in order to allow sufficient space for expansion of the oil. Borosilicate glass bottles have been found satisfactory.

5.3 Electrolytic sheet copper, 0,125 mm to 0,250 mm in thickness.

5.4 Polishing materials, consisting of 65 μm silicon-carbide paper or cloth, 105 μm silicon-carbide powder and pharmaceutical grade absorbent cotton (cotton wool).

5.5 Drying oven, capable of being controlled at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

5.6 Forceps, stainless steel, spade-ended.

6 Preparation of apparatus

6.1 Cleaning of bottles

Bottles (5.2) shall be chemically clean. Remove any oil from the bottles by rinsing them with solvents, then wash them with a phosphate-type cleaning agent. Rinse the bottles with tap water, then with grade 3 water (4.5), and dry them in the oven (5.5).

6.2 Preparation of test strips

From the sheet copper (5.3) cut a strip 6 mm x 25 mm (see note 4) and remove blemishes from the surfaces with the 65 μm silicon-carbide paper (5.4). Strips may be stored in wash solvent (4.1) at this point for future use. Do the final polishing of the strip by removing it from the wash solvent, holding it between the fingers protected with ashless filter paper, and rubbing with 105 μm silicon-carbide powder (5.4) picked up from a glass plate with a pad of absorbent cotton moistened with a drop of the wash solvent. Wipe the strip with fresh pads of cotton (5.4) and subsequently handle it only with the forceps (5.6), avoiding all contact with the fingers. Rub the strip in the direction of its long axis. Clean all metal dust and abrasive from the strip by rubbing vigorously with clean pads of cotton until a fresh pad remains unsoiled. Bend the clean strip in a V-shape to provide two limbs approximately 12,5 mm long at an angle of approximately 60° , and wash it successively in acetone (4.2), water (4.5), acetone and diethyl ether (4.3). Dry the strip in a stream of warm air for a maximum of 5 min and then proceed immediately as described in 7.1 (see notes 5 and 6).

NOTE 4 It has been found convenient to polish a larger piece of copper from which, after the final polishing, several strips of the required size may be cut.

NOTE 5 This method of cleaning has been adopted from ISO 2160.

NOTE 6 The strip may be dried in the oven briefly (less than 3 min), but if so, should be examined carefully for tarnish before use.

7 Procedure

7.1 Use the oil to be tested as received, and do not filter through a filter paper.

Promptly place the prepared copper strip (6.2) in a clean bottle (6.1) to which has been added 250 ml of the oil to be tested. Place the bent copper strip standing on its long edge so that no flat surface lies along the glass bottom of the vessel. Lubricate the ground glass stopper with a small amount of the sample. Bubble nitrogen (4.4) through the oil in the bottle by means of a glass tube connected to the reduction or needle valve of the cylinder (rubber connections shall be sulfur-free) for 2 min, and quickly put the stopper loosely in place.

7.2 Place the stoppered bottle in the heating medium (5.1) controlled at $140\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, immersed to the neck if an oil bath is used. When the oil in the bottle has reached approximately $140\text{ }^{\circ}\text{C}$, tighten the stopper more firmly. Remove the bottle after heating for 19 h at $140\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Let the oil in the bottle cool down to room temperature. Carefully withdraw the copper strip and wash it with wash solvent (4.1) to remove all the oil. Dry the copper strip by laying it edge down on a piece of filter paper and allowing the wash solvent film to evaporate. Then inspect it as described in 7.3. Handle the strip only with the forceps (5.6).

7.3 To inspect the test strip, hold it in such a manner that light reflected from it at an angle of approximately 45° will be observed.

8 Interpretation of results

The oil shall be classified as corrosive or non-corrosive in accordance with table 1.