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STANDARD

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**Petroleum-based lubricating oils — Ageing characteristics — Determination of change in Conradson carbon residue after oxidation**

*Huile de graissage à base de pétrole — Caractéristiques de vieillissement — Détermination de l'augmentation du résidu de carbone Conradson après oxydation*



Reference number  
ISO 6617:1994(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.

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 6617 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 4, *Classifications and specifications*.

Annex A forms an integral part of this International Standard.

# Petroleum-based lubricating oils — Ageing characteristics — Determination of change in Conradson carbon residue after oxidation

**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 procedure for determination of the ageing characteristics of petroleum-based lubricating oils. It is applicable to those oils which have an evaporation loss of less than 15 % mass fraction, as determined by the procedure specified in 9.3 of this standard.

## 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 3170:1988, *Petroleum liquids — Manual sampling*.

ISO 3585:1991, *Borosilicate glass 3.3 — Properties*.

ISO 6615:1993, *Petroleum products — Determination of carbon residue — Conradson method*.

## 3 Definition

For the purposes of this International Standard, the following definition applies.

**3.1 ageing characteristics:** Characteristics of a petroleum-based lubricating oil as determined by the increase in the Conradson carbon residue of the oil aged in accordance with this International Standard in comparison to that of the same non-aged oil.

## 4 Principle

The oil sample to be tested is aged for two 6-h periods at a temperature of 200 °C while air is passed through the oil. The value of the Conradson carbon residue of the aged sample is determined according to ISO 6615. The Conradson carbon residue of the non-aged oil, also determined according to ISO 6615, is subtracted from this value. The increase in the value of the carbon residue of the aged oil over that of the non-aged oil is reported as the result.

Under no circumstance may the procedure specified in ISO 6615 be modified, or any other procedure substituted for this procedure.

## 5 Apparatus

**5.1 Testing apparatus,** consisting of an ageing vessel and a holder with an air inlet tube and an air outlet tube (figure 1), all parts being manufactured from borosilicate glass in accordance with ISO 3585.

**5.1.1 Ageing vessel**, manufactured from 28 mm × 1,4 mm borosilicate glass pipe in accordance with ISO 3585, complete with a 29/32 conical ground glass joint.

**5.1.2 Vessel for temperature measurement**, constructed as specified for the ageing vessel (5.1.1), but without the ground glass joint.

**5.1.3 Holder**, with air inlet tube and air outlet tube.

The air inlet tube shall be manufactured from 7 mm × 1 mm borosilicate glass pipe in accordance with ISO 3585, and sealed into the holder as illustrated in figure 1. The air inlet tube shall be mounted in the centre of the holder and its lower end cut square. The length of the air inlet tube shall be such that, with the holder securely in place in the ageing vessel, its lower end is 1 mm ± 0,5 mm above the bottom of the ageing vessel.

Each ageing vessel (5.1.1) and holder shall be manufactured as a matched set and appropriately marked to protect against mixing between sets.

**5.2 Oven**, capable of being maintained at a temperature of 105 °C to 110 °C.

**5.3 Heating facility.**

An oil-bath heating facility equipped with a stirrer and temperature-control device, complete with a cover and adequate insulation to minimize heat loss, shall be provided. The stirring and heating effect shall produce a uniform temperature of 200 °C ± 1 °C in each sample. The quantity of oil in the bath should be such that at 200 °C ± 1 °C it expands to within 13 mm of the cover. A calibrated thermometer (5.4) shall be placed in a separate ageing vessel (5.1.2) and filled with oil to a depth of 110 mm. The ageing vessel provided for the thermometer shall not be fitted with a ground glass joint or air inlet tube.

Dimensions in millimetres

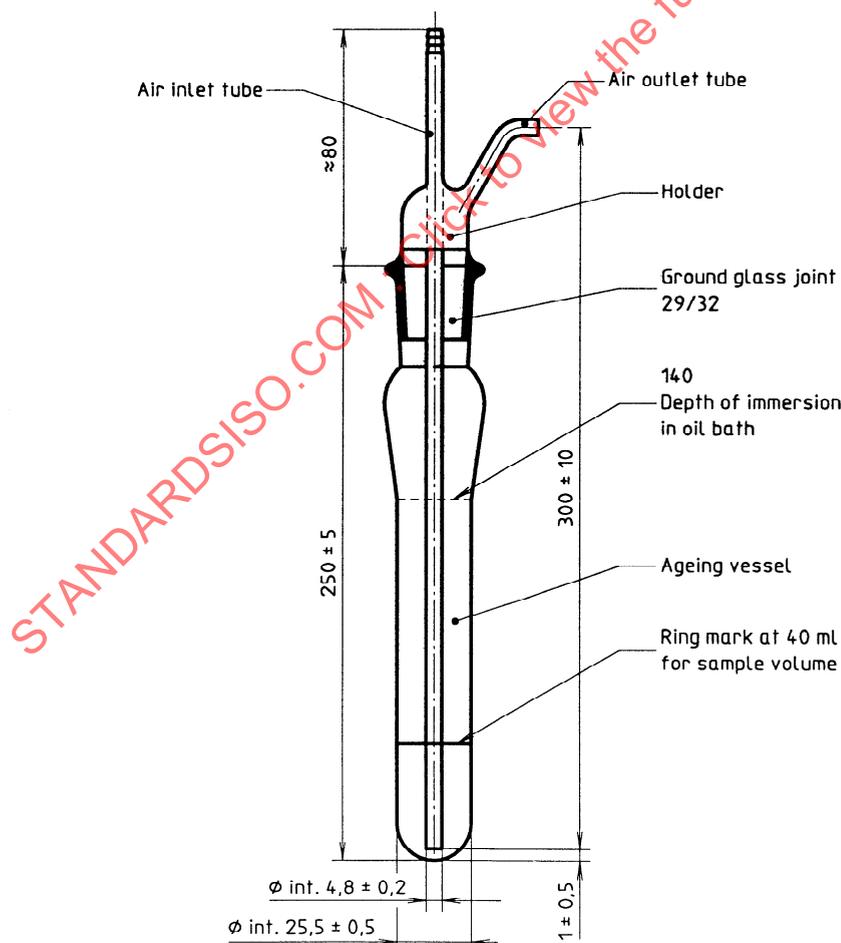


Figure 1 — Ageing vessel and holder

A support for the ageing vessels shall be fixed in the oil bath so that the ageing vessels are immersed in the oil to a depth of 140 mm. The oil-bath heating facility shall be provided with round openings properly sized to securely accommodate one ageing vessel per opening. The parts of the ageing vessels and the thermometer which protrude above the cover shall be protected from draught by extending the walls of the oil bath to a height of up to 150 mm beyond the cover. An acceptable alternative to extending the walls of the oil bath is to place a protective cap of the same height over the cover.

In lieu of an oil-bath heating facility, a metal-block heating device may be utilized, providing that it uniformly maintains the temperature of the oil under test in the ageing vessel at  $200\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ . If a metal-block heating device is used, protection from draught shall be provided by placing a cap 155 mm high over the metal block and ageing vessel.

NOTE 1 To obtain proper heat transfer, the space between the metal block and the ageing vessel may be filled with a suitable heat-transfer fluid, such as silicone.

**5.4 Thermometer**, meeting the specification given in annex A.

## 5.5 Air supply equipment.

**5.5.1 Gas cleaning vessel**, containing glass wool.

**5.5.2 Pressure regulator**, electrically or mechanically controlled.

For example, a pressure regulator consisting of 24 mm  $\times$  1,8 mm borosilicate glass pipe in accordance with ISO 3585, approximately 1 200 mm long, sealed at the bottom, filled with a glycerin/water mixture (in a 2/1 ratio by volume) to a height of approximately 1 000 mm.

A glass T-piece, made from 6 mm  $\times$  1 mm borosilicate glass pipe in accordance with ISO 3585, is also required. The vertical section, approximately 1 200 mm long, shall be fitted with a slotted cork to support the T-piece.

**5.5.3 Flowmeter**, for each ageing vessel, having an average measuring range suitable to cope with a flowrate of approximately 15 l/h, calibrated for air at 20  $^{\circ}\text{C}$ , with maximum error of  $\pm 0,25$  l/h.

**5.5.4 Soap-bubble flowmeter**, having a capacity of 150 ml and a scale marked in 1-ml divisions, or any other suitable flowmeter.

## 6 Reagents and materials

**6.1 Chromiumsulfuric acid cleaning solution**, or alternatively a non-chromium-containing, strongly oxidizing acid cleaning solution.<sup>1)</sup>

**WARNING — Chromiumsulfuric 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 chromiumsulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solutions down the drain, but neutralize them with great care owing to the concentrated sulfuric acid present and dispose of them in accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).**

**Non-chromium-containing, strongly 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.**

A solution of chromiumsulfuric acid may be produced by dissolving 50 g of  $\text{K}_2\text{Cr}_2\text{O}_7$  in 1 litre of concentrated sulfuric acid (mass fraction of  $\text{H}_2\text{SO}_4 = 96\%$ , density = 1,84 g/ml). This solution shall be kept in glass bottles with ground necks and stoppers.

Alternatively, commercially available chromiumsulfuric acid may be used.

**6.2 Oil-free dry compressed air**, from a cylinder.

**6.3 Acetone** (propan-2-one), analytical grade.

## 7 Sampling

Sampling shall be carried out as specified in ISO 3170.

1) Hot Nochromix<sup>®</sup> solution or a 24-h soak in Micro<sup>®</sup> solution gives results statistically similar to those of hot chromiumsulfuric acid. Nochromix<sup>®</sup> and Micro<sup>®</sup> are examples of a suitable non-chromium-containing product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Similar products may be used if it has been established that they give equivalent results.

## 8 Preparation of the apparatus

Thoroughly clean the ageing vessels with their holders using suitable solvents and cleaning solutions (6.1 and 6.3) and subsequently dry them in an air oven. If stubborn deposits are present on the surface of the glassware, remove them using chromiumsulfuric acid, in a safe manner observing the warning in 6.1. After removing the chromiumsulfuric acid, rinse the ageing vessels first with tap water, and thereafter with demineralized water until the water shows a neutral reaction. Subsequently dry the glassware in an air oven (5.2) at 105 °C to 110 °C for a minimum of 3 h. After drying, allow the parts to cool to room temperature in a desiccator in which they are stored until used.

Adjust the heating device to a temperature of 200 °C ± 1 °C as registered in the temperature-measuring vessel in accordance with 5.3.

## 9 Test procedure

### 9.1 General

Carry out two determinations simultaneously as described in 9.2 to 9.4 .

### 9.2 Ageing process

At an ambient temperature of 18 °C to 28 °C, fill two ageing vessels with samples of the oil to be tested up to the 40 ml mark and close off with the holder. To determine the evaporation loss as described in 9.3, weigh the ageing vessels together with the sample and holder to the nearest 0,01 g. After weighing, place the ageing vessels in the pretreated heating device and connect the air inlet tube to the flowmeter. Then pass an air stream through the sample for 6 h at a rate of 15 l/h ± 0,25 l/h, maintaining the temperature at 200 °C ± 1 °C, as registered in the vessel for temperature measurement. After 6 h stop the ageing process by removing the ageing vessels from the heating device.

During the ageing process, the air flow shall be checked several times at the air outlet tube by means of a soap-bubble flowmeter (5.5.4).

After storing the two samples for 12 h to 18 h at ambient temperature, repeat the ageing process as described above.

NOTE 2 For routine batch control purposes, the two 6 h ageings at 200 °C ± 1 °C may be carried out consecutively without the interval of 12 h to 18 h standing time at ambient temperature. However, this modified procedure cannot be used for reference purposes.

### 9.3 Evaporation loss

After the aged samples have cooled to room temperature, determine the evaporation loss by weighing the sample and holder to the nearest 0,01 g and comparing the mass fraction of sample lost with the limiting value of 15 % indicated in clause 1.

### 9.4 Determination of the Conradson carbon residue

Individually stir each of the two aged samples thoroughly. Determine the Conradson carbon residue in accordance with ISO 6615, using a 5 g test portion weighed to the nearest 5 mg.

If the two aged samples have been standing for a long period before determining the Conradson carbon residue, heat them for 20 min in a water bath at 100 °C and mix well in the ageing vessel directly prior to determination.

Calculate the arithmetic mean of the results obtained from the Conradson carbon residue determination for the two aged samples.

## 10 Expression of results

**10.1** Calculate the increase in the carbon residue as the difference between the value obtained for the Conradson carbon residue of the non-aged sample in accordance with ISO 6615 and the mean value for the Conradson carbon residue of the two aged samples. This value represents the test result.

**10.2** The absolute increase in the Conradson carbon residue of the test oil on ageing is stated with reference to this International Standard as a mass fraction rounded up to the nearest 0,05 %. The evaporation loss is also given as mass fraction, expressed as a percentage.

## 11 Precision

The precision of the method, as obtained by statistical analysis of interlaboratory test results, is as follows.

### 11.1 Repeatability

The difference between successive 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 shown in table 1 only in one case in 20.

## 11.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values shown in table 1 only in one case in 20.

**Table 1**

	<b>Repeatability</b> % of average value of results	<b>Reproducibility</b> % of average value of results
Ash-free lubricants	20	30
Ash-containing lubricants	25 <sup>1)</sup>	40 <sup>1)</sup>
1) Determined in round-robin tests using ash-containing lubricants whose Conradson carbon residue was between 0,9 g and 1,9 g per 100 g (average values of round-robin test).		

## 12 Test report

The test report shall contain the following information:

- a) the type and complete identification of the petroleum-based lubricating oil tested;
- b) a reference to this International Standard;
- c) the results of the test (see 10.2);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

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## Annex A (normative)

### Thermometer specification

Range: ..... 195 °C to 205 °C

Immersion, mm: ..... 100

#### Graduation

Subdivisions: ..... 0,1 °C

Long lines at each: ..... 0,5 °C

Numbers at each: ..... 1 °C

Scale error, max.: ..... 0,2 °C

Expansion chamber to permit heating: ..... Required

Total length, mm: .....  $300 \pm 10$

Stem outer diameter, mm: .....  $6,5 \pm 1,5$

Bulb length, mm: .....  $30 \pm 5$

Bulb outer diameter: ..... Not greater than stem

Minimum distance, in mm, from bottom of bulb to line at 195 °C: ..... 165

Length of scale range, mm: .....  $90 \pm 20$

#### Stem enlargement

Diameter, mm: .....

Length, mm: .....

Distance to bottom, mm: .....

#### Thread reference temperature

Over entire length: ..... 40 °C

Expansion extension (safety bubble) for a volume equivalent of: ..... 50 °C

Thermometer filling: ..... Mercury and protective gas

Head of thermometer: ..... Equipped with ring or button

NOTE 3 Thermometers to DIN 12785 and IP 22 C oxidation test are suitable. There is no equivalent International Standard at this time.

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