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Methods of test for petroleum and its products

Part 48: Determination of oxidation characteristics of lubricating oil

(Identical with IP 48/11)

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Summary of pages

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Foreword

Publishing information

This part of BS 2000 is published by BSI and came into effect on 30 June 2010. It was prepared by Technical Committee PTI/13, *Petroleum Testing and Terminology*. A list of organizations represented on this committee can be obtained on request to its secretary.

Supersession

This part of BS 2000 supersedes BS 2000-48:1997, which is withdrawn.

Information about this document

This new edition has been updated to be in line with changes implemented by the Energy Institute.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of "Standard Methods for Analysis and Testing of Petroleum Products and British Standard 2000 Parts" which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as parts of BS 2000.

Under the terms of the agreement between BSI and the Institute of Petroleum, the revised version of BS 2000-48 will be published by BSI and by the IP (in "Standard Methods for Analysis and Testing of Petroleum Products and British Standard 2000 Parts" and as a separated publication). The numbering of the parts of BS 2000 follows that of the corresponding IP methods. BS 2000-48:2011 is thus identical with IP 48/11.

WARNING. This part of BS 2000 calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

It has been assumed in the preparation of this part of BS 2000 that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is "shall".

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

1 Scope

This method indicates the tendency of a lubricating oil to deteriorate on oxidation under specified conditions. A measure of the deterioration is obtained by comparison of the viscosity and carbon residue before and after oxidation. The test is not suitable for additive-type oils (other than those containing ashless additives) or those which form solid products or lose more than 10% by evaporation during the test.

WARNING. This part of BS 2000 calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

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.

BS 2000-0.1:1999, *Methods of test for petroleum and its products – Part 0: General introduction – Section 0.1: Specifications – IP standard for thermometers*

BS 2000-0.2, *Methods of test for petroleum and its products – Part 0: General introduction – Section 0.2: Specifications for IP standard reference liquids (Identical with IP Appendix B)*

BS 2000-14, ISO 4262, *Methods of test for petroleum and its products – Part 14: Petroleum products – Determination of carbon residue – Ramsbottom method (Identical with IP 14/94)*

BS EN ISO 3104, BS 2000-71.1, *Methods of test for petroleum and its products – Part 71, Section 71.1: Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity (Identical with IP 71 Section 1/96)*

3 Principle

The sample is subjected to relatively severe oxidation conditions by heating to 200 °C and passing air through it at 15 L/h for two periods of 6 h. After standing for 15 h to 30 h, the kinematic viscosity and Ramsbottom carbon residue of the oxidized oil are determined and compared with those from corresponding texts on the unoxidized oil. An alternative test procedure for the shortened version with a 12 h continuous oxidation period is given in Annex A.

4 Reagents and materials

4.1 *Petroleum spirit*, 60/80 conforming to BS 2000-0.2.

4.2 *Sulfuric acid*, concentrated, of general purpose reagent grade or a non chromium containing strongly-oxidizing acid cleaning solution.

WARNING. Concentrated sulfuric acid and non chromium containing strongly oxidizing cleaning solutions are highly corrosive and potentially hazardous in contact with organic materials. Wear a full face shield and full length protective clothing including suitable gloves. Avoid breathing vapour.

5 Apparatus

5.1 *Oxidation tubes*, manufactured from borosilicate or neutral glass and conforming to the dimensions shown in Figure 1.

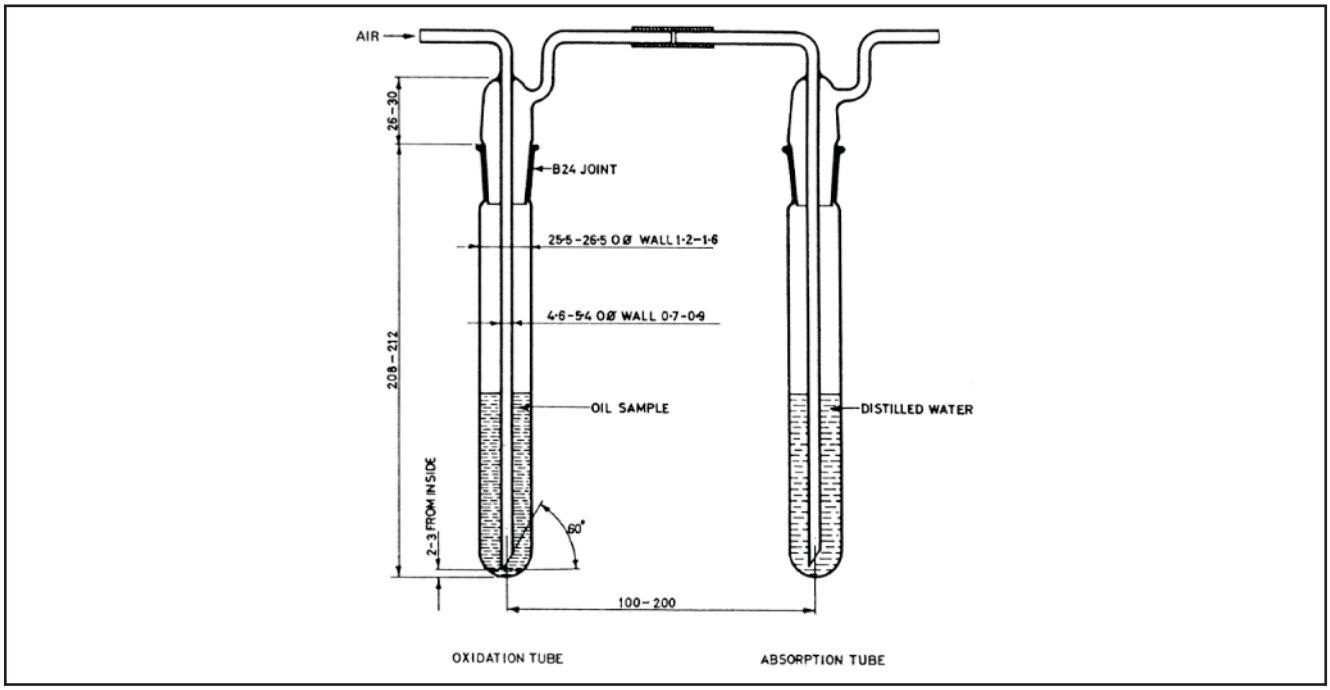
5.2 *Absorption tubes*, if required for reduction of objectionable atmospheric odours.

NOTE 1 The oxidation tube used in this method vents to the atmosphere and results in an unpleasant smell in some laboratories. This can be avoided by passing the issuing vapours through a tube containing water.

Absorption tubes shall conform to **5.1** and contain approximately 25 mL of water. The absorption tubes shall be mounted outside the bath and connected to the oxidation tubes. The length between the axis of the two tubes shall be 150 mm ±50 mm. The connection between the oxidation and absorption tubes shall be as short as possible and jointed to the vessels by means of short flexible sleeves (see note 2). To avoid evaporation of water, ensure the absorption tube is protected from the heating bath by insulation.

NOTE 2 Silicone rubber sleeving has been found to be suitable for this purpose.

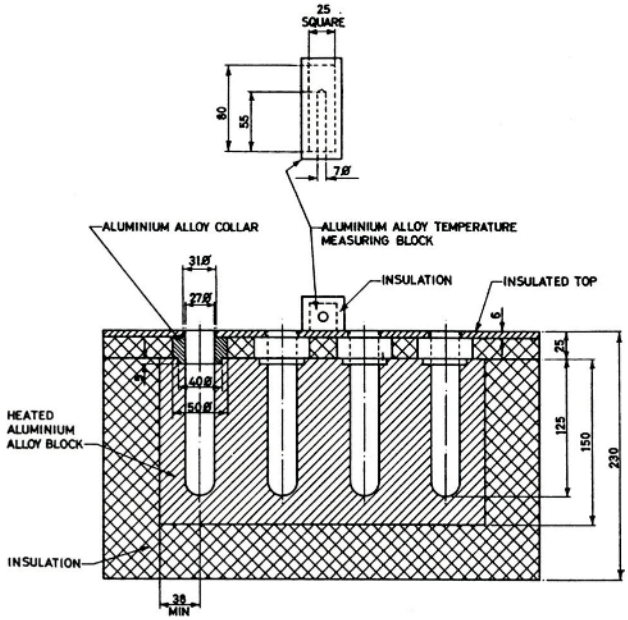
Figure 1 **Oxidation and absorption tubes**



5.3 *Heating bath*, an aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the required number of oxidation tubes at a temperature of 200 °C ±0.5 °C (see Figure 2). This temperature shall be read on a thermometer, or a suitable temperature-measuring device (**5.5**), inserted to within 5 mm from the bottom of an oxidation tube containing 40 mL of oil (without an air supply) and placed in the heating bath. When using an aluminium block heater, the tubes shall be inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, ensure heating over the 150 mm length of the tube. In the case of oil baths, the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath. For both types of heating bath the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In case of slackness, a 25 mm diameter 0-ring may be placed around the tube and pressed against the heater surface.

WARNING. The heating bath should be provided with a safety cut-out to prevent bath overheating.

Figure 2 **Typical metal heating bath**



5.4 *Air supply*, from a suitable source of dry air, passed through a cotton wool or glass wool filter into a pressure stabiliser and then to the required number of oxidation tubes through any system capable of delivering 15 L/h ±0.25 L/h to each oxidation tube, the rate being measured by a suitably calibrated flow meter. The flow rate shall be checked periodically by the use of a soap bubble flow meter, or an equivalent electronic device, connected to the exit of the oxidation tube or absorption tube if being used.

5.5 *Thermometer*, conforming to the specification Type IP 22C as specified in BS 2000-0.1:1999 or a suitable temperature measuring device readable to ±0.1 °C and calibrated to better than ±0.1 °C.

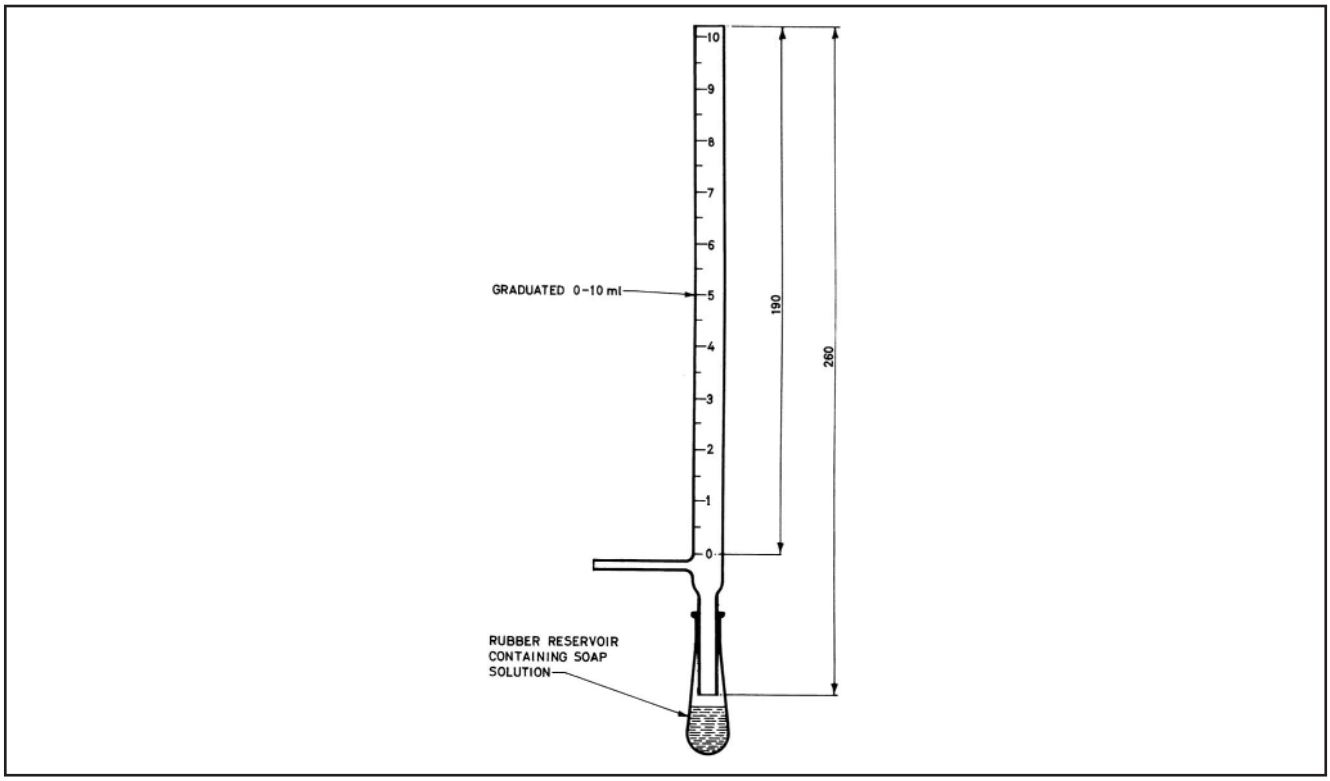
5.6 Soap bubble flow meter or an equivalent electronic device, capable of measuring the required flow rate. An example of a suitable soap bubble flow meter is shown in Figure 3.

6 Preparation of apparatus

The oxidation and absorption tubes shall be chemically cleaned.

*NOTE 3 A satisfactory method is to wash out with petroleum spirit (**4.1**) and after drying, soak with concentrated sulfuric acid for a minimum of 16 h. If the concentrated sulfuric acid does not remove all deposits, repeat the acid cleaning with a non chromium containing strongly-oxidising acid cleaning solution (**4.3**). Drain and completely remove sulfuric acid by washing, first with tap water, then with distilled water. Dry the tubes in an air oven at 105 °C to 110 °C for at least 3 h and then allow to cool to room temperature in a desiccator in which they are kept until use.*

Figure 3 **Soap bubble flowmeter**



7 Procedure

7.1 Weigh the oxidation tube and the ground-glass head to the nearest 0.1 g. Transfer 40 mL of the sample at room temperature into the oxidation tube, insert the ground-glass head and weigh to the nearest 0.1 g. Place the oxidation tube, ground-glass head and the sample in the heater at 200 °C ±0.5 °C (see note 4). If the absorption tube is being used, add 25 mL of distilled water and connect to the oxidation tube. Connect the air supply and adjust the air flow to 15 L/h ±0.25 L/h. To avoid evaporation of the water, the absorption tube should be protected from the bath by insulation.

NOTE 4 If a number of tubes are to be placed simultaneously in the bath, the tubes may be preheated for 5 min to 10 min at 180 °C to 200 °C in order to reduce the cooling effect of the oxidation bath.

7.2 When the oxidation bath has again reached 200 °C ±0.5 °C, and not less than 10 min after introducing the oxidation tube, adjust the air flow to 15 L/h ±0.25 L/h. Maintain this flow for the duration of the test. After 6 h ±10 min, remove the oxidation tube from the bath.

7.3 Allow it to stand at ambient temperature for 12 h to 18 h. Repeat the heating procedure for a further 6 h ±10 min. At the end of this time, remove the oxidation tube from the bath and allow to cool to ambient temperature.

7.4 Weigh the oxidation tube, ground-glass and the oxidized sample to the nearest 0.1 g. Calculate the evaporation loss as described in **8.1**. The test is not suitable for evaluating a sample which loses more than 10% by evaporation during the test. Between 15 h and 30 h after completion of the oxidation periods, heat the oxidation tube in a boiling water bath and mix thoroughly to ensure the uniformity of the contents.

7.5 Determine, on the unoxidized oil, the kinematic viscosity at 40 °C by BS 2000-71.1 and the Ramsbottom carbon residue by BS 2000-14.

7.6 Immediately after heating and mixing of the oxidized oil, charge a viscometer through a 75 µm filter as specified in BS 2000-71.1 and a Ramsbottom carbon residue coking bulb as specified in BS 2000-14. Determine the kinematic viscosity at 40 °C and the Ramsbottom carbon residue.

7.7 If successive determinations of the kinematic viscosity on the oxidized oil do not give consistent results within the determinability specified in BS 2000-71.1, report the oil as unsuitable for testing using BS 2000-48.

NOTE 5 The use of the same viscometer for the determination of the kinematic viscosity of the oxidized and unoxidized oil is recommended where practicable.

8 Calculation

8.1 Evaporation loss

Calculate the percentage (*m*/*m*) evaporation loss, *E*, using the following equation:

$$E = \frac{(S - O)}{S - W} \times 100$$

where

W is the mass of the oxidation tube and ground-glass head in grams;

S is the mass of the new sample, oxidation tube and the ground-glass head in grams;

O is the mass of the oxidized sample, oxidation tube and ground-glass head in grams.

8.2 Viscosity ratio

Calculate the viscosity ratio, v_R, using the following equation:

$$v_R = \frac{v_2}{v_1}$$

where

v_1 is the kinematic viscosity of the unoxidized oil in millimetres squared per second at 40 °C;

v_2 is the kinematic viscosity of the oxidized oil in millimetres squared per second at 40 °C.

8.3 Carbon residue increase

Calculate the percentage (*m*/*m*) Ramsbottom carbon residue increase, $C_{R,I}$, using the following equation:

$$C_{R,I} = C_{R,O} - C_{R,U}$$

where

$C_{R,U}$ is the percentage (*m*/*m*) Ramsbottom carbon residue of the unoxidized oil;

$C_{R,O}$ is the percentage (*m*/*m*) Ramsbottom carbon residue of the oxidized oil.

9 Expression of results

Report the following:

a) the kinematic viscosity of the unoxidized oil in square millimetres per second (mm^2/s) at 40 °C (see **7.5**);

b) the kinematic viscosity of the oxidized oil in square millimetres per second (mm^2/s) at 40 °C (see **7.6**);

c) the viscosity ratio to the nearest 0.01 (see **8.2**);

d) the Ramsbottom carbon residue of the unoxidized oil (see **7.5**);

e) the Ramsbottom carbon residue of the oxidized oil (see **7.6**);

f) the increase in carbon residue after oxidation (see **8.3**);

If consistent results are not obtained the oil shall be reported as being unsuitable for testing.

10 Precision

The following criteria should be used for judging the acceptability of results (95% probability) (see Notes 6 and 7).

Viscosity ratio:

Level of result X 1.08 to 2.4

Repeatability 0.2883 (X – 1.0)

Reproducibility 0.5759 (X – 1.0)

Carbon residue increase:

Level of result X 0.2 to 2.4

Repeatability 0.1258 (X + 0.38)

Reproducibility 0.3392 (X + 0.38)

where X is the average of two results.

NOTE 6 These precision values, as defined in BS 2000-0.3, have been obtained by statistical examination of inter-laboratory results and were first published in 1980.

NOTE 7 These precision data were derived from programmes in which plain mineral oils were tested and are not applicable to other types of oil.

11 Test report

The test report shall contain at least the following information:

a) a reference to this standard;

b) the type and identification of the product tested;

c) the result of the test (see Clause **9**);

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

e) the date of the test.

Annex A (normative) An alternative test procedure for a continuous, 12 h oxidation period

A.1 Scope

The full test period is three days and this may cause difficulties in refineries where production and laboratory facilities are continuous. In these circumstances, where the test is being used for production control, it is permissible to use a shortened version with the 12 h continuous oxidation period, followed by immediate determination of the viscosity and Ramsbottom carbon residue of the oxidized oil. However, it is necessary for the test to be carried out in full in cases of dispute.

A.2 Apparatus

Use the same apparatus as specified in Clause **5**.

A.3 Procedure

A.3.1 Follow the test procedure as given in **7.1**.

A.3.2 Follow the procedure as outlined in **7.2**, except use a continuous 12 h ±10 min oxidation period, followed by immediate determination of the viscosity and Ramsbottom carbon residue of the oil (as specified in **7.4**, **7.5** and **7.6**). Omit procedure given in **7.3**.

A.4 Calculation

Determine the evaporation loss, viscosity ratio and Ramsbottom carbon residue in accordance with **8.1**, **8.2** and **8.3**.

A.5 Precision

Practical experience has shown that results obtained by the abbreviated version are normally within the repeatability and reproducibility of the full test procedure.

Bibliography

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 2000-0.3, *Methods of test for petroleum and its products – Part 0, Section 0.3: Significance and usage of IP precision data (Identical with IP Appendix E)*

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