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Methods of test for petroleum and its products

Part 306: Determination of oxidation stability of straight mineral oil

(Identical with IP 306/11)

Publishing and copyright information

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Contents

Summary of pages

This document comprises a front cover, an inside front cover, pages i to ii, pages 1 to 12, an inside back cover and a back cover.

Foreword

Publishing information

This part of BS 2000 is published by BSI and came into effect on 30 June 2011. 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This part of BS 2000 supersedes BS 2000-306:1994, which is withdrawn.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 2000-306 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 2000-306:2011 is thus identical with IP 306/11.

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 is designed to give a measure of the tendency of straight (i.e. plain) mineral lubricating oil to oxidize under specified conditions.

It employs the same apparatus and a similar procedure to those used to determine the oxidation stability of Inhibited Mineral Turbine Oil (BS 2000-280). It differs from this test in that (a) the test time is reduced to 48 h, (b) two conditions, namely no catalyst and solid copper metal catalyst are used, and (c) the degree of deterioration is expressed as total oxidation products (TOP) per cent.

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 (Identical with IP – Annex A)*

BS 3591, *Specification for industrial methylated spirits*

BS EN 13602, *Copper and copper alloys – Drawn, round copper wire for the manufacture of electrical conductors*

3 Summary of method

Dry oxygen is passed for 48 h into reaction tubes containing the oil, with, in one case no catalyst and in the other case a solid copper catalyst. The tubes and their contents are immersed in a suitable aluminium block heater (or oil bath) maintained at 120 °C. The volatile acids driven off by the flowing oxygen are absorbed in water. The volatile and oil-soluble acidities, and the sludge produced in the oil are determined at the completion of the test.

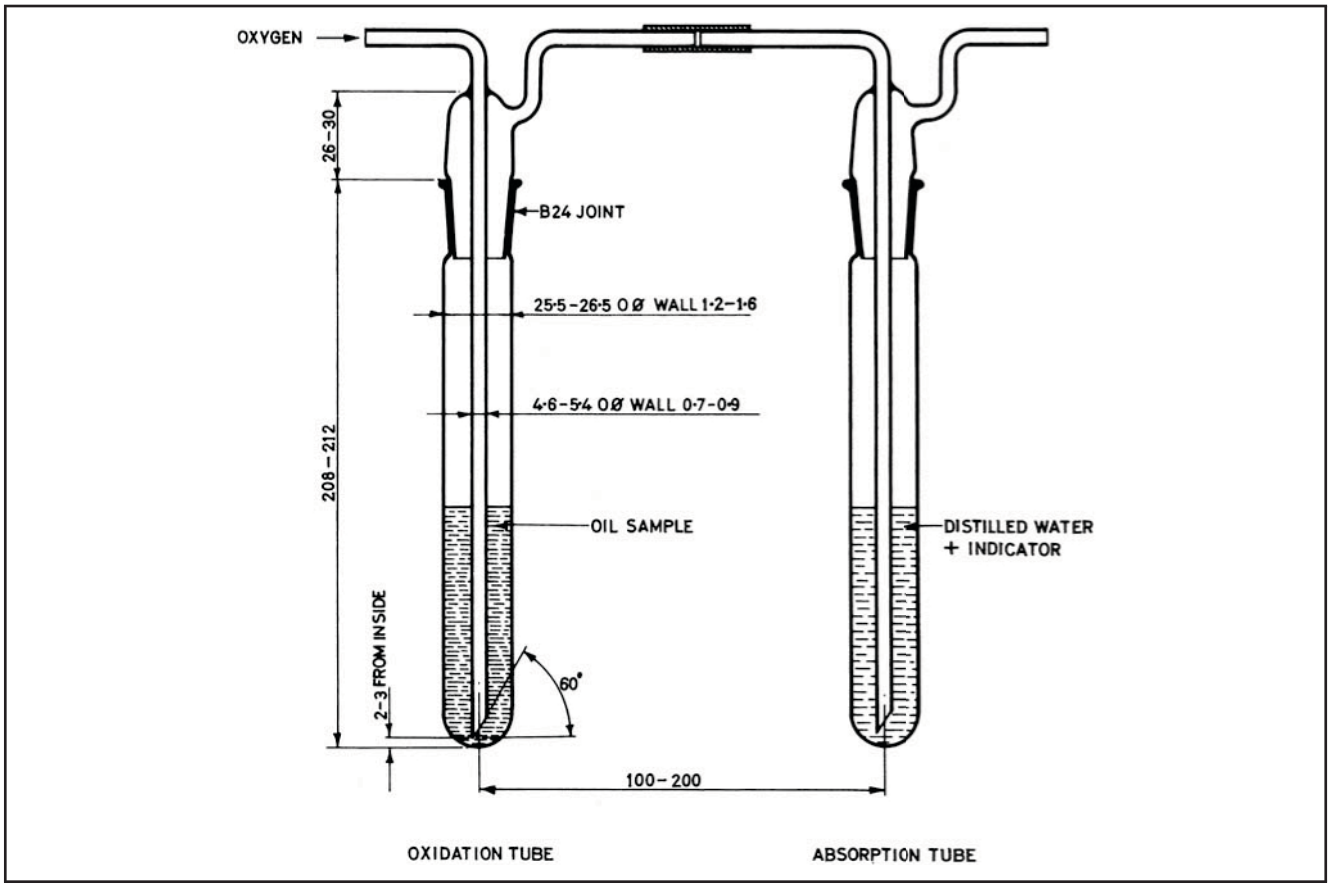
4 Apparatus

4.1 *Oxidation tubes*, manufactured from borosilicate or neutral glass having the dimensions shown in Figure 1.

4.2 *Absorption tubes*, these are the same as the oxidation tubes. The connection between oxidation and absorption tubes must be as short as possible and made of glass tubing butt-jointed to the vessels by means of short flexible sleeves (see Note 1) (length between axis of the two tubes 150mm ±50 mm). These tubes are mounted outside the bath.

NOTE 1 Silicone rubber sleeving has been found suitable for this purpose.

Figure 1 **Oxidation and absorption tubes**

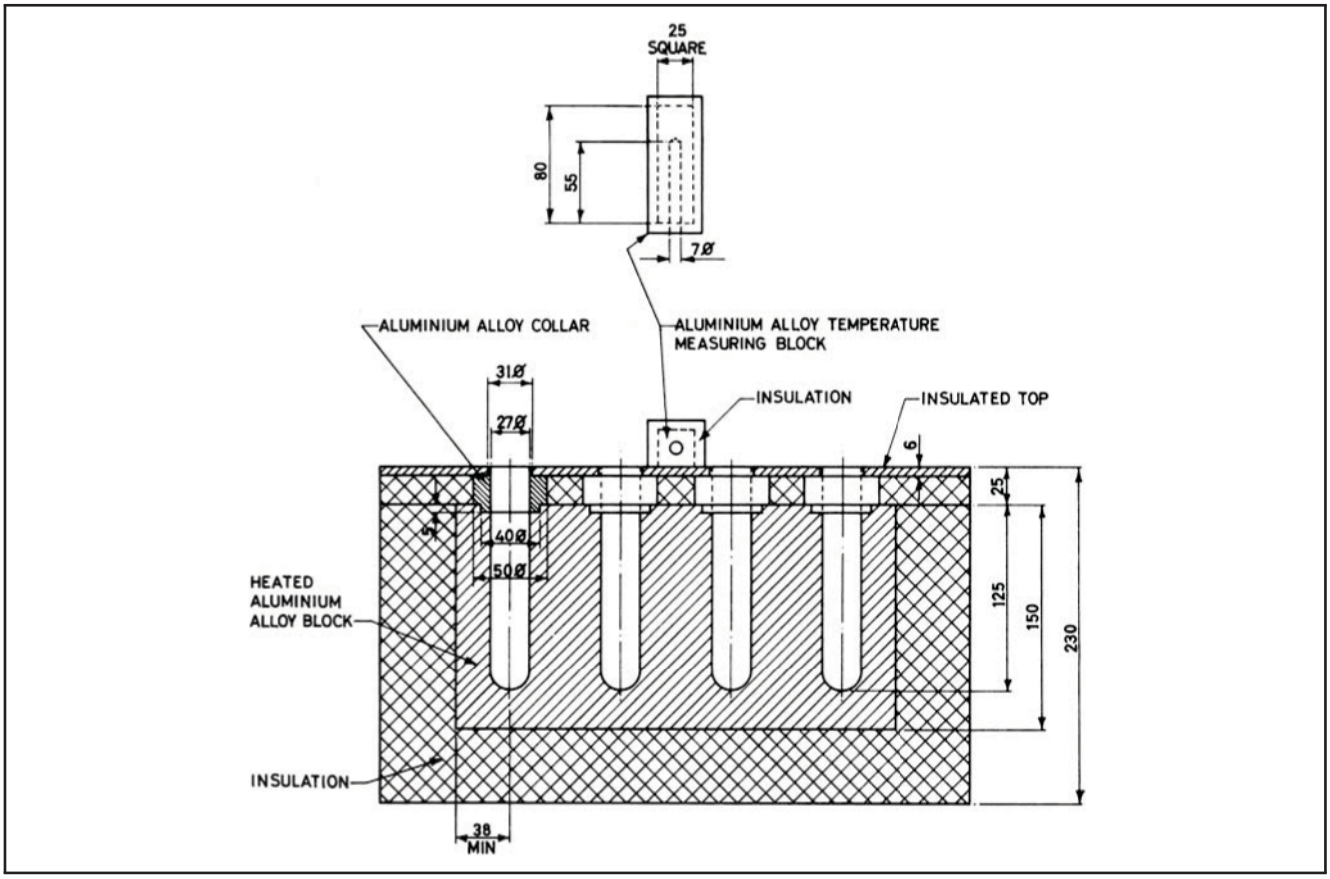


4.3 *Heating bath,* an aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the desired number of oxidation tubes at the required temperature of 120 ±0.5 °C, see Figure 2. This temperature shall be read on a thermometer Type IP 81C as specified in BS 2000-0.1:1999 inserted in a test tube to within 5 mm from the bottom; this test tube shall be filled with oil up to the immersion line of the thermometer and placed in the heating bath. The temperature of the upper surface must be kept at 60 ±5 °C (see Note 2). Measure this temperature by the use of a thermometer in a drilled aluminium block, see Figure 2; the surfaces of this block, other than that against the upper surface of the heating bath, are protected by suitable insulation. This block should be placed as near to the holes as practicable and within the area of the aluminium heater block. When using an aluminium block heater the test tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block must be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube. In the case of oil baths the oxidation tubes must 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 tube above the upper surface must be 60 mm and the diameter of the holes must be just sufficient to allow insertion of the specified tube. In case of slackness a 25 mm diameter 0-ring may be placed round the tube and pressed against the heater surface.

NOTE 2 This temperature of 60 ±5 °C applies for an ambient temperature of 20 °C which should be used for assessing equipment compliance. Operation outside 60 ±5 °C due to extremes of ambient temperature only has a slight effect on test results.

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

Figure 2 **Typical metal heating bath**



4.4 *Filtering crucibles*, Gooch type crucibles with sintered filter disc of Grade P10 porosity (4-10 microns) 35 mL capacity (see Note 3). See Annex A.

NOTE 3 The maximum pore size can be determined in accordance with the method described in BS 1752 Laboratory sintered or fritted filters.

4.5 *Porcelain crucibles*, 50 mL capacity.

4.6 *Soap bubble flowmeter*, for checking the oxygen flow rate (see Figure 3).

4.7 *Burette*, volume 10 mL with graduations of 0.01 mL.

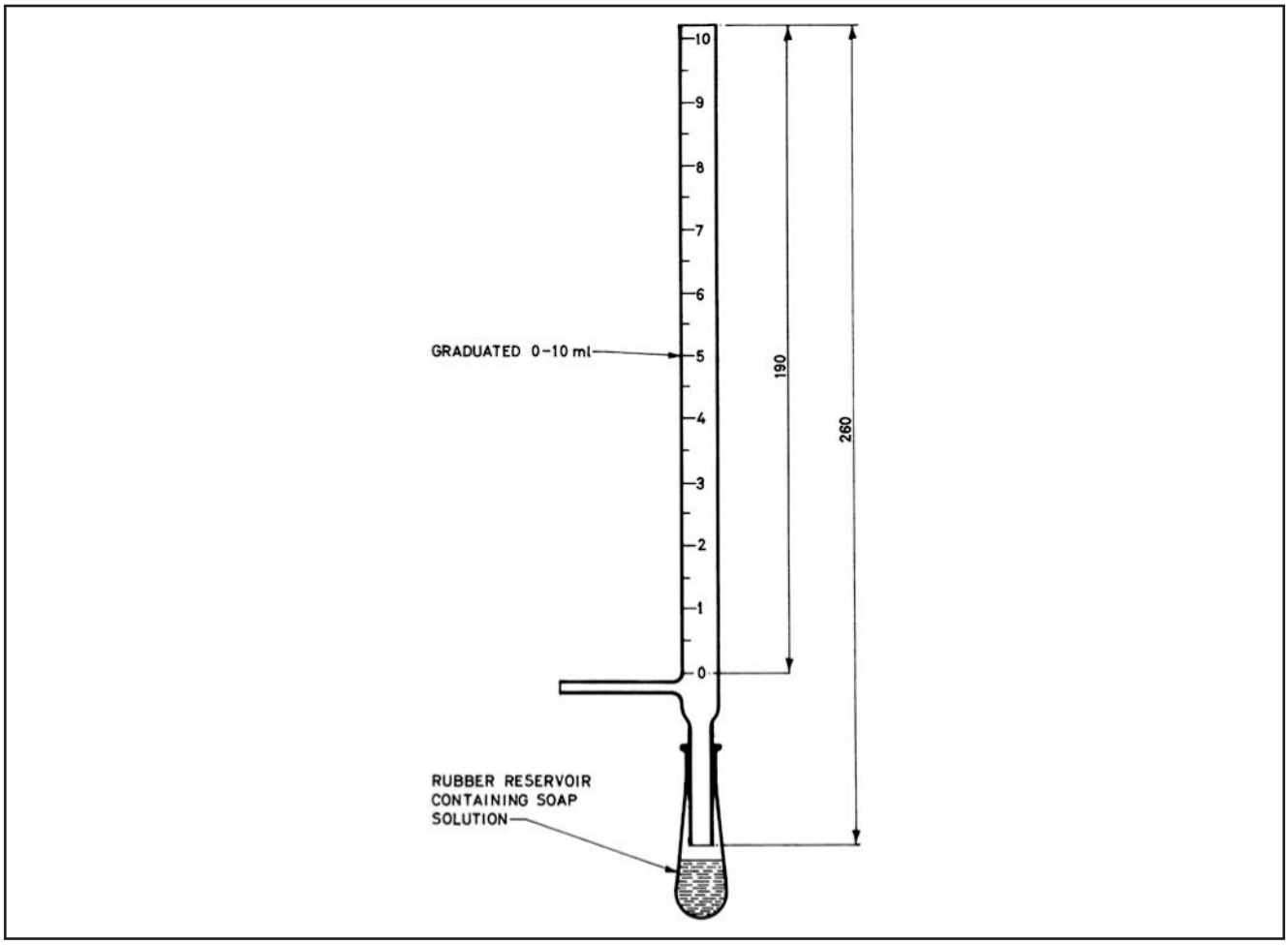
4.8 *Thermometers*, Types IP 81C and IP I5C conforming to BS 2000-0.1:1999, or others of suitable range and equal or greater accuracy.

5 Reagents and materials

5.1 *Oxygen*, commercial product obtained from liquid air (minimum purity 99.4%). The oxygen must be dried by passing through suitable desiccant. A 10 L flask, acting as a surge vessel, smooths the oxygen flow, excess of which bubbles through mineral oil contained in a test tube. At the beginning of each test-run, the oxygen flow-rate given by the measuring device should be checked with the soap bubble flow-meter. From thereon, at the daily flow checks, the rate shall be adjusted to the measurement obtained initially when checked by the soap-bubble meter, or by directly checking with the soap-bubble meter, if considered necessary.

5.2 *Alkali blue solution*, 2 g/100 mL IMS to BS 3591 (66 op) (see Note 4).

Figure 3 **Soap bubble flowmeter**



NOTE 4 To obtain a good end point with Alkali Blue indicator it should be dissolved in ethanol containing at least 5% of water and 0.03 mL of 0.1 M HCI should be added to each 1 mL of indicator solution. After 24 h make 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 Co(NO3)2.6H2O.

5.3 *Phenolphthalein solution*, 1 g/100 mL alcoholic solution.

5.4 *n-Heptane*, IP Specification. (see Note 5)

NOTE 5 n-Heptane as described above is exactly the same as that used as a reference fuel in determining the octane number of gasoline.

5.5 *Hydrochloric acid*, 0.1 M aqueous solution.

5.6 *Potassium hydroxide*, 0.1 M alcoholic solution.

5.7 *Toluene pure*, sulphur free.

5.8 *Chloroform*, GPR.

5.9 *Ethanol*, IMS to BS 3591, 66 op.

5.10 *Sulphuric acid (concentrated)*, GPR.

5.11 *Copper wire*, annealed; composition designated C101 as described in BS EN 13602. Diameter 1.04 mm ±0.01 mm (see Note 6).

NOTE 6 If 1.04 mm dia wire is not available then the next size may be used but the length must be recalculated to give the same copper surface area to oil weight ratio: i.e. 100.6 ±1 mm^2/g.

5.12 *Silicon carbide cloth*, 100 grade.

5.13 *Acetone*, GPR.

6 Safety precautions

6.1 *Oxygen*, vigorously accelerates combustion, ensure your safety regulations for handling and working with oxygen are followed. See **B.1**.

6.2 *n-Heptane*, flammable, harmful if inhaled. See **B.2**.

6.3 *Isopropyl alcohol*, (alcoholic potassium hydroxide) – flammable. See **B.3**.

6.4 *Toluene*, flammable, harmful if inhaled. See **B.4**.

6.5 *Chloroform*, may be fatal if swallowed, harmful if inhaled. See **B.5**.

6.6 *Ethyl alcohol*, IMS flammable. See **B.6**.

6.7 *Acetone*, extremely flammable, harmful if inhaled. See **B.7**.

6.8 *Sulphuric acid (concentrated)*, poison, corrosive, strong oxidizer, harmful if inhaled, when used wear approved face protection and protective clothing. See **B.8**.

6.9 *Oxidized oil*, avoid contact with skin. See **B.9**.

7 Preparation of apparatus

7.1 Cleaning the test tubes

The oxidation and absorption tubes shall be chemically cleaned. A satisfactory method of cleaning is to wash with acetone, followed by distilled water. Drain and then soak in concentrated sulphuric acid for a minimum of 16 h. Drain and complete removal of the acid by washing, first with tap water, then with distilled water. Dry the tubes in an air oven at 105 to 110 °C for at least 3 h, and then allow them to cool to room temperature in a desiccator in which they are kept until they are used.

7.2 Preparation of solid metal catalyst

Immediately before required, clean a 770 mm (see Note 7) length of copper wire using absorbent cloth wet with n-heptane, and follow by rubbing with the abrasive cloth until a fresh metal surface is exposed. Wipe with dry absorbent cotton wool until all loose particles of metal and abrasive have been removed. In subsequent operations handle the wire with cotton gloves to prevent it coming into contact with the skin. Roll the wire into a spiral 20 mm external diameter and 50 mm long, and store in n-heptane until insertion in the oil sample.

NOTE 7 If 1.04 mm dia wire is not available then the next size may be used but the length must be recalculated to give the same copper surface area to oil weight ratio: i.e. 100.6 ±1 mm^2/g.

8 Procedure

8.1 Weigh 25 ±0.04 g of sample into each of two oxidation tubes, cleaned according to **7.1**.

8.2 Place a copper coil catalyst in one of the tubes (see Note 8).

NOTE 8 Use a drop of oil under test to seal the ground-glass joint of the test vessel.

8.3 Put the oxidation tubes in the heating bath and maintain at 120 ±0.5 °C. Connect the oxidation tube to the absorption tube, into which has been placed 25 mL of neutral distilled or deionised water and 5 or 6 drops of the phenolphthalein indicator solution. To avoid evaporation of water the absorption tube must be protected from the heating bath by insulation. Connect the oxygen supply and adjust the flow of oxygen to 1 ±0.1 L per hour, which must be checked daily. After 48 h stop the oxygen flow and remove the oxidation tubes from the heating bath.

Absorption tube: treat as per **8.4**.

Oxidation tube: treat as per **8.5** and **8.6**.

8.4 Volatile acids

As quickly as possible after the test, titrate the water in the absorption tube with the alcoholic potassium hydroxide solution.

8.5 Sludge determination

Cool the sample of 25 g of artificially aged oil in the dark for 1 h and then pour it into a conical flask of 500 mL capacity, fitted with a ground glass stopper. Use 300 mL of n-heptane (see Note 9) to recover the oil adhering to the test tube and oxygen lead-in tube and add the washings to the oil in the flask.

*NOTE 9 If the n-heptane recovered from previous test is used it must be acid-free and comply with the original specification (see **5.4**).*

Allow the mixture to stand in the dark for 24 h, at a temperature of 20 ±2 °C, then filter through the filtering crucible previously dried to constant weight. (See Annex A.)

At the start of filtering only a small pressure drop should be used to prevent the sludge passing through the filter. Cloudy filtrates should be passed through a second time.

Carefully remove all traces of oil by repeated washing of the sludge with n-heptane. The total volume of the n-heptane used for the washing of the sludge shall be 150 mL. Dry the crucible containing the sludge at 110 °C to constant weight. The filtrate shall be used for the determination of soluble acidity (see Note 10).

NOTE 10 Because of the small amount of sludge produced in the uncatalysed test, care must be taken to ensure that the inside and outside of the filter crucible are washed free from oil. Weighing should be carried out as quickly as possible to prevent absorbance of moisture by the filter. Dissolve any sludge adhering to the test tubes, the oxygen lead-in tube and the catalyst coil, where appropriate, in small quantities of chloroform (a total of 30 mL) and transfer the solution into a tared porcelain crucible. After the evaporation of the chloroform dry at 100 °C to constant weight.

8.6 Soluble acidity

Collect the heptane solution obtained after filtering off the sludge in a 500 mL measuring flask and make up to the mark with n-heptane. Mark three determinations of the neutralization value on 100 mL samples of the n-heptane/oil solution (see Note 11).

NOTE 11 A single determination of acidity is normally adequate but three determinations should be made for referee purposes. Immediately before use prepare the titration solvent as follows: Add 2 mL of the alkali blue solution to 100 mL mixture of 60 mL of toluene and 40 mL of IMS. Neutralise the mixture with the alcoholic potassium hydroxide solution to give a red colour comparable to that of a 10% solution of cobalt nitrate Co(NO3)2.6H2O, and this colour shall persist for at least 15 seconds. Add this neutralised solvent with swirling, to 100 mL of the heptane solution, then titrate with the alcoholic potassium hydroxide solution at a temperature not exceeding 30 °C.

9 Calculation and report

9.1 Calculate and report as follows.

9.2 Volatile acidity:

$$\text{VA, mg KOH/mg} = \frac{A \times 56.1 \times M}{25}$$

where

A is the volume of alcoholic KOH (0.1M) solution, mL; and

M is the molarity of the alcoholic KOH used.

9.3 Total sludge:

S%=(a+b) x 4

where

a is the weight of sludge insoluble in n-heptane, g; and

b is the weight of sludge recovered by chloroform, g.

9.4 Soluble acidity of oil:

$$\text{SA, mg KOH/g} = \frac{A \times 56.1 \times M}{25}$$

where

A is the volume of alcoholic KOH (0.1 M) solution necessary to neutralise the n-heptane/oil solution mL, and

M is the molarity of the alcoholic KOH used.

9.5 Calculate the total oxidation products (TOP) as follows:

$$\text{TOP}\% = S + \frac{180(SA + VA)}{561}$$

The figure 180 in the numerator is the average molecular weight of oil oxidation acids.

9.6 Report the TOP to the nearest 0.0% uncatalysed or solid copper catalysed as appropriate, BS 2000-306.

9.7 Where required report the ratio of sludge to TOP as a percentage to the nearest whole number, BS 2000-306.

10 Precision

10.1 General

The following criteria should be used for judging the acceptability of results (95% confidence).

10.2 Repeatability

Duplicate results by the same operator should be considered suspect if they differ by more than the amounts given below.

10.3 Reproducibility

The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the amounts given below.

Test	Repeatability	Reproducibility
Total Oxidation Products - uncatalysed	0.217 (x+0.05)	0.695 (x+0.05)
Total Oxidation Products - catalysed	0.19	0.33
Ratio, Sludge/Total Oxidation Products - catalysed	0.080 (x+22)	0.268 (x+22)

In the above formulae, x is the mean of 2 results. The following tables give typical values of repeatability and reproducibility obtained from the above formulae, for the range of results covered.

Total oxidation products – Uncatalysed

Level of results	Repeatability	Reproducibility
0.05	0.02	0.06
0.10	0.03	0.10
0.15	0.04	0.13

Total oxidation products – Catalysed

Level of result	Repeatability	Reproducibility
0.5 to 1.2	0.19	0.33

Ratio: Total sludge/total oxidation products, % – Catalysed (see Note 12)

Level of results	Repeatability	Reproducibility
5	2	7
10	2	8
20	3	11
30	4	13
40	4	16

NOTE 12 No precision data can be quoted for the Ratio: Total sludge/total oxidation products — uncatalysed, because the Total sludge contents - uncatalysed in the precision evaluation programme were all less than 0.05%w. These precision values were obtained by statistical examination of inter-laboratory test results and were first published in 1974.

Annex A (normative) Alternative method for sludge determination

A.1 Scope

This method describes an alternative procedure for determining the sludge content of the oil/n-heptane mixture. For referee tests or in the case of dispute, the sludge content determined using GOOCH FILTERING CRUCIBLES shall be used.

A.2 Summary of method

The oil/n-heptane mixture from **8.3** is filtered through a dried and pre-weighed 47 mm dia. 5 μm membrane filter and the weight of sludge determined after washing with heptane and drying.

A.3 Apparatus

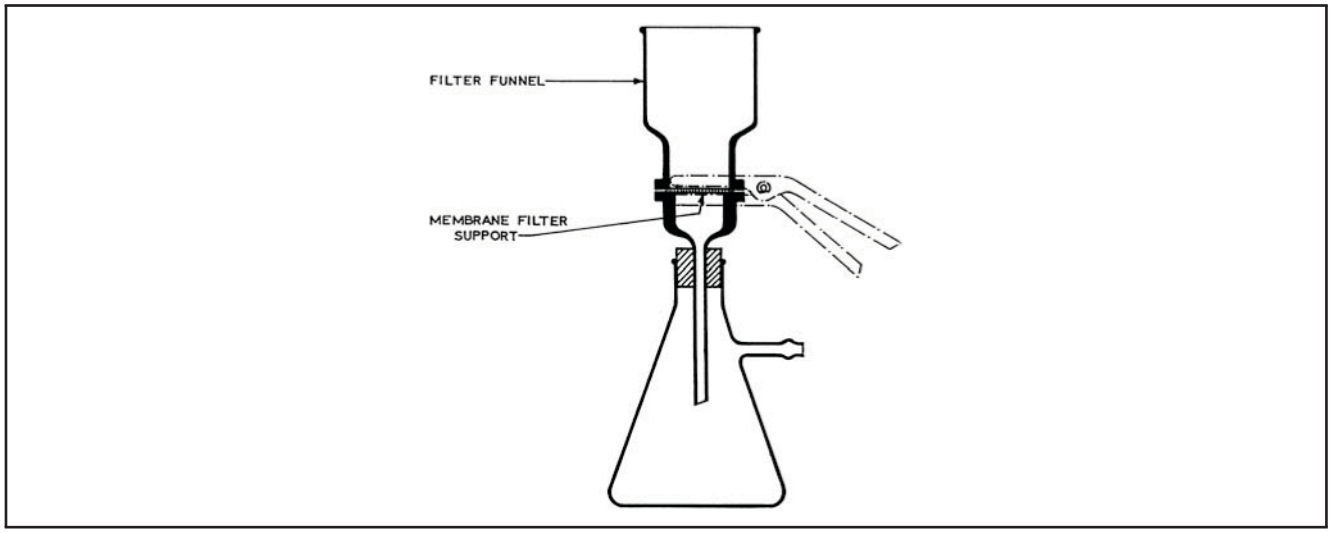
A.3.1 *Membrane filters*, 47 mm diameter, nominal pore size 5.0 μm.

A.3.2 *Forceps*, flat bladed with non-serrated, non-pointed tips.

A.3.3 *Petri dishes*, suitable to hold 47 mm diameter membrane filters.

A.3.4 *Filtration apparatus*, of the type shown in Figure A.1. It consists of a funnel and funnel base with a filter support, such that the membrane filter can be clamped between the sealing surfaces of the funnel and its base by means of a metal clamp.

Figure A.1 **Apparatus for the alternative method of sludge determination**



A.3.5 *Oven*, of the static type (without fan-assisted circulation), controlled at 80 ±5 °C. Not greater than 85 °C.

NOTE 13 If a fan assisted oven has to be used, place the petri dishes in a suitable container with a vented lid.

A.4 Procedure

A.4.1 Using clean forceps, place the membrane filter into the petri dish and place in the oven at 80 °C. After one hour, remove from the oven, allow to cool in a dessicator and weigh.

A.4.2 Place the weighed membrane filter on to the filter support and install and clamp the funnel.

A.4.3 Filter the 300 mL of oil/heptane mixture from **8.5** through the membrane filter. Cloudy filtrates should be passed through a second time. Carefully remove all traces of oil by repeated washing of the sludge, flask and funnel with n-heptane. The total volume of n-heptane used for the washing shall be 150 mL.

A.4.4 With the vacuum applied, carefully remove the clamp and funnel. Wash the periphery of the membrane filter with a small volume of n-heptane by directing it in a gentle stream from the edge towards the centre, taking care not to wash any of the sludge from the surface of the membrane filter. Maintain the vacuum after the final washing only for a few seconds necessary to remove excess n-heptane from the membrane filter.

A.4.5 Using clean forceps, carefully remove the membrane filter from the filter base, place in the petri dish and dry in the oven at 80 °C. (Not greater than 85 °C). After one hour remove from the oven, allow to cool in a dessicator and reweigh.

A.4.6 Collect the n-heptane solution obtained, after filtering off the sludge, in a 500 mL measuring flask and make up to the mark with n-heptane. Use this solution to determine the soluble acidity as described in **8.6**.

Annex B (informative) Precautionary statements

B.1 Oxygen

WARNING – Vigorously accelerates combustion. Keep oil and grease away. Do not use oil or grease on regulators, gauges, or control equipment. Use only with equipment conditioned for oxygen service by careful cleaning to remove oil, grease and other combustibles. Keep combustibles away from oxygen and eliminate ignition sources. Keep surfaces clean to prevent ignition or explosion, or both, on contact with oxygen. Always use a pressure regulator. Release regulator tension before opening cylinder valve. All equipment and containers used must be suitable and recommended for oxygen service. Never attempt to transfer oxygen from the cylinder in which it is received to any other cylinder. Do not mix gases in cylinders. Do not drop cylinder. Make sure cylinder is secured at all times. Keep cylinder valve closed when not in use. Stand away from outlet when opening cylinder valve. For technical use only. Do not use for inhalation purposes. Keep cylinder out of sun and away from heat. Keep cylinders from corrosive environment. Do not use cylinder without label. Do not use dented or damaged cylinders.

B.2 n-Heptane

WARNING – Flammable. Harmful if inhaled. Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid prolonged breathing of vapour or spray mist. Avoid prolonged or repeated skin contact.

B.3 Isopropyl alcohol (propan-2-ol)

WARNING – Flammable.

Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid prolonged breathing of vapour or spray mist. Avoid contact with eyes and skin. Do not take internally.

B.4 Toluene

WARNING – Flammable. Vapour harmful. Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Avoid breathing of vapour or spray mist. Avoid prolonged or repeated contact with skin. The use of protective gloves and suitable eye protection is recommended.

B.5 Chloroform

DANGER! – May be fatal if swallowed. Harmful if inhaled. May produce toxic vapours if burned. Keep container closed. Avoid prolonged breathing of vapour or spray mist. Avoid contact with eyes and skin. Do not take internally. Use with adequate ventilation. Keep vapours away from open flame.

Do not mix acetone and chloroform as this could result in an explosive mixture.

B.6 Ethyl alcohol (ethanol, IMS)

WARNING – FLAMMABLE.

Denatured alcohol cannot be made non-toxic. Keep away from heat, sparks and open flame. Keep container closed. Use with adequate ventilation. Avoid prolonged breathing of vapour or spray mist. Avoid contact with eyes and skin. Do not take internally.

B.7 Acetone

DANGER! – Extremely flammable. Vapours may cause flash fire. Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Vapours may spread long distances and ignite explosively. Avoid build up of vapours and eliminate all sources of ignition, especially electrical apparatus and heaters. Avoid prolonged breathing of vapour or spray mist. Avoid contact with eyes and skin.

Do not mix acetone and chloroform as this could result in an explosive mixture.

B.8 Sulphuric acid (concentrated)

DANGER! – POISON. Corrosive. Strong oxidizer. Contact with organic material may cause fire. May be fatal if swallowed. Liquid and vapour cause severe burns. Harmful if inhaled. Contact with water liberates large amounts of heat. Spillage may cause fire. Do not get in eyes, on skin, on clothing. Do not breath vapour, spray or mist. Dilute by addition of acid to water. Keep in tightly-closed container in approved acid storage cabinet. Keep cool. Loosen closure carefully when opening. Use with adequate ventilation. Do not allow water to get into container because of violent reaction. Keep container closed when not in use. Use protective clothing and goggles when handling. Wash thoroughly after handling.

B.9 Oxidized oil

Avoid contact with skin. Wear protective gloves when handling. Wash thoroughly after contact.

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

BS 1752, ISO 4793, *Specification for laboratory sintered or fritted filters including porosity grading*

BS 2000-280, *Methods of test for petroleum and its products – Part 280: Petroleum products and lubricants – Inhibited mineral turbine oils – Determination of oxidation stability (Identical with IP 280/1999)*

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