

BRITISH STANDARD

**BS 2000 :
Part 14 : 1994
ISO 4262 : 1993**

Methods of test for

Petroleum and its products

Part 14. Petroleum products - Determination of carbon residue - Ramsbottom method

(Identical with IP 14/94)

National foreword

This British Standard, having been prepared under the direction of the Petroleum Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 31 March 1994. It is identical with ISO 4262 : 1993, prepared by Technical Committee 28, Petroleum products and lubricants, of the International Organization for Standardization (ISO).

This British Standard supersedes BS 4451 : 1980, which is withdrawn.

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 'IP Standard methods for analysis and testing of petroleum and related products' 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.

The numbering of the Parts of BS 2000 follows that of the corresponding methods published in 'IP Standard methods for analysis and testing of petroleum and related products'. Under the terms of the agreement between BSI and the Institute of Petroleum, BS 2000 : Part 14 will be published by the IP (in 'Standard methods for analysis and testing of petroleum and related products' and as a separate publication). BS 2000 : Part 14 : 1994 is thus identical with IP 14/94. Square brackets marked in the margin of this IP Standard indicate text that differs from the previous edition.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

© The Institute of Petroleum
& BSI 1994

The following BSI references
relate to the work of this
standard:
Committee reference PTC/13
Draft for comment 92/50648 DC

Petroleum products — Determination of carbon residue — Ramsbottom method

WARNING — The use of this International Standard may involve hazardous materials, 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 determining the amount of carbon residue, in the range of 0,01 % (*m/m*) to 30,0 % (*m/m*), left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming tendency. The method is generally applicable to relatively non-volatile petroleum products which partially decompose on distillation at atmospheric pressure.

NOTES

1 The term "carbon residue" is used throughout this International Standard to designate the carbonaceous residue formed during evaporation and pyrolysis of a petroleum product. The residue is not entirely composed of carbon, but is a coke which can be further changed by pyrolysis. The term "carbon residue" is retained in this method only in deference to its widespread use.

2 The carbon residue of distillate and residual fuel oils gives an approximate ranking of such fuels in terms of their propensity to form deposits in specific applications.

3 The presence of alkyl nitrates in distillate fuels, or ash-forming additives in either distillate or residual fuels, will give carbon residue results that are higher than the corresponding values of the fuel without additives. These values may not correlate with the propensity of a fuel to form deposits.

4 The carbon residue of base lubricating oils may give an indication of the propensity of the oil to lay down deposits in combustion chambers, and/or of the relative chemical constitution of oils of similar viscosity. Most finished lubricating oils contain ash-forming additives, and thus the carbon residue of finished lubricants cannot be used in this manner.

5 The carbon residue of a gas oil is a useful guide in the manufacture of gas.

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 683-13:1986, *Heat-treatable steels, alloy steels and free-cutting steels — Part 13: Wrought stainless steels*.

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

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*.

ISO 3405:1988, *Petroleum products — Determination of distillation characteristics*.

3 Principle

3.1 The test portion is weighed into a glass coking bulb having a capillary opening, and is placed in a metal furnace maintained at a temperature of approximately 550 °C. The test portion is thus quickly heated to the point at which all volatile matter is evaporated out of the bulb with or without decomposition, while the heavier residue remaining in the bulb undergoes cracking and coking reactions. In the later stages of the heating period, the coke or carbon residue is subject to further slow decomposition or slight oxidation due to the possibility of air being drawn into the bulb. After a specified heating period, the bulb is removed from the furnace, cooled in a dessicator, and again weighed. The residue remaining is calculated as a mass percentage of the test portion.

3.2 Provision is made for determining the proper operating characteristics of the furnace with a control bulb containing a thermocouple, which gives a specified time-temperature relationship.

3.3 For light products where over 90 % (V/V) distils at below 370 °C, the procedure may be carried out on the residue remaining after 90 % (V/V) has been distilled. The procedure for obtaining this residue is given in annex A.

4 Apparatus

4.1 Glass coking bulb, made of heat-resistant glass, and conforming to the dimensions and tolerances shown in figure 1.

Prior to use, check the diameter of the capillary to see that it is greater than 1,5 mm and not more than 2,0 mm. Pass a 1,5 mm diameter drill rod through the capillary and into the bulb; attempt to pass a 2,0 mm diameter drill rod through the capillary. Reject bulbs that do not permit the insertion of the smaller rod and those whose capillaries are larger than the larger rod.

4.2 Control bulb, for use in determining compliance of furnace characteristics with the performance requirements (see clause 5). Made of stainless steel, containing a thermocouple and conforming to the tolerances shown in figure 2. The control bulb shall be provided with a dull finish by heating in a furnace at a temperature of 850 °C to 900 °C for 30 min, and shall not be polished thereafter, as a polished bulb has different heating characteristics from one with a dull finish.

A suitable thermocouple pyrometer for observing true temperature within ± 1 °C is also required.

4.3 Syringe, for test portion charging. A 5 ml or 10 ml glass hypodermic syringe fitted with a needle of 1,5 mm outside diameter or a serum needle of 1,45 mm to 1,47 mm outside diameter, for transfer of the test portion to the glass coking bulb.

NOTE 6 A syringe having a needle which fits on the ground glass tip of the syringe is not recommended, as it may be blown off when pressure is applied to the syringe plunger. Locking-type syringes are more satisfactory, as the needle locks on the bottom of the syringe barrel, and cannot be blown off by pressure.

4.4 Metal coking furnace, constructed of solid metal, having coking bulb wells 25,45 mm $\pm$ 0,1 mm in internal diameter and 76 mm deep to the centre of the well bottom, with suitable arrangements for heating to a uniform temperature of 550 °C. The bottom of the well shall be hemispherical to accommodate the bottom of the glass coking bulb (4.1).

Do not cast or otherwise form the furnace with unnecessary voids which will impede heat transfer.

If a molten metal furnace is used, provide it with a suitable number of bulb wells, the internal dimensions of which correspond to the internal dimensions of holes in the solid metal furnace. The bulb wells shall

Dimensions in millimetres

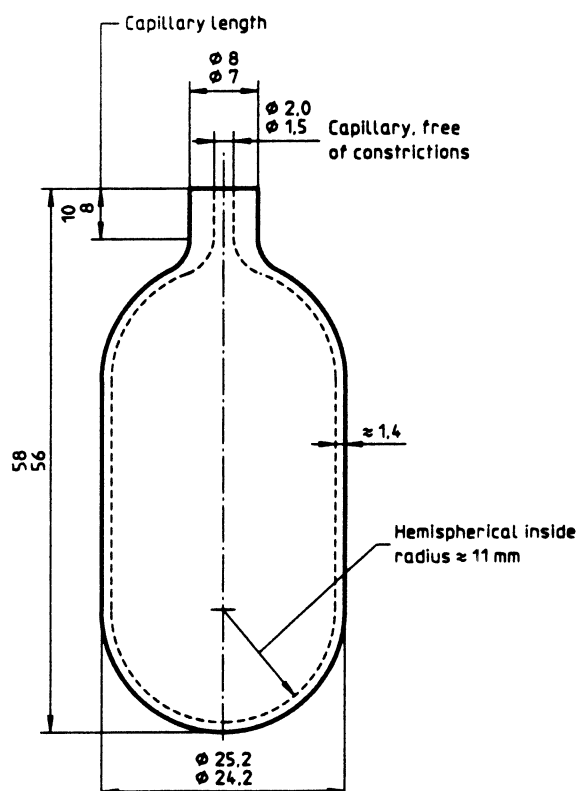


Figure 1 — Glass coking bulb

be immersed in the molten metal to leave not more than 3 mm of the bulb well exposed above the molten metal at operating temperatures.

NOTE 7 A description of one type of furnace which has been found to be satisfactory is given in annex B.

4.5 Temperature-measuring devices. A removable iron-constantan thermocouple with a sensitive pyrometer, or other suitable temperature-indicating device shall be used, located centrally near the bottom portion of the furnace and arranged to measure the temperature of the furnace, thus enabling the performance tests specified in clause 5 to be carried out.

NOTES

8 When a molten bath is used, it is desirable to protect the temperature-indicating device with a quartz or thin metal sheath.

9 It is good practice to calibrate the thermocouple or other temperature-measuring device against a standard thermocouple or a reference standard approximately once a week when the furnace is in constant use, the actual frequency depending on experience.



5 Verification of apparatus performance

coking bulb well while the bath is fully loaded as well as singly loaded. Once these criteria have been met, use the furnace with any degree of loading. Use only a furnace that has successfully undergone the performance or control tests given in this clause.

5.2 Thermocouple

Calibrate the thermocouple in the control bulb against a standard thermocouple at least once every 50 h of use of the control bulb (4.2).