BRITISH STANDARD

BS EN ISO
7536 : 1996
BS 2000 :
Part 40 : 1996
ISO 7536 : 1994

Methods of test for

Petroleum and its products

Part 40. Petroleum products - Determination of oxidation stability of gasoline - Induction period method

(Identical with IP 40/96)

The European Standard EN ISO 7536 : 1996 has the status of a British Standard

BSi
British Standards

National foreword

This British Standard was published under the authority of the Materials and Chemicals Sector Board and comes into effect on 29 July 1996. It is the English language version of EN ISO 7536 : 1996 Petroleum products - Determination of oxidation stability of gasoline - Induction period method, published by the European Committee for Standardization (CEN), which endorses ISO 7536 : 1994, published by the International Organization for Standardization (ISO).

This British Standard supersedes BS 2000 : Part 40 : 1995 which is withdrawn.

There are no technical differences between this British Standard and the previous edition of BS 2000 : Part 40. A new edition has been made available to fulfil BSI's obligation to publish all approved European Standards.

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 40/BS EN ISO 7536 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 40 : 1996 is thus identical with IP 40/96.

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

The following BSI references relate to the work of this standard:
Committee reference PTI/13
Announcement in *BSI News*
July 1995

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 7536

February 1996

ICS 75.160.20

Descriptors: See ISO document

English version

Petroleum products - Determination of oxidation stability of gasoline - Induction period method (ISO 7536:1994)

Produits pétroliers - Détermination de la stabilité à l'oxydation de l'essence - Méthode de la période d'induction (ISO 7536:1994)

Mineralölerzeugnisse - Bestimmung der Oxidationsbeständigkeit von Ottokraftstoffen Induktionsdauerverfahren (ISO 7536:1994)

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europaisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard from ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO) has been taken over as a European Standard by the Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 1996, and conflicting national standards shall be withdrawn at the latest by August 1996. ,

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 7536:1994 has been approved by CEN as a European Standard without any modification.

IP 40/95 ISO 7536:1994 ASTM D525-94 BS 2000 : Part 40 : 1995

Petroleum products — Determination of oxidation stability of gasoline — Induction period method

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

1.1 This International Standard specifies a method for the determination of the stability of aviation and motor gasolines in their finished form only, under accelerated oxidation conditions, by measuring the induction period to breakpoint in a pressure bomb apparatus.

1.2 The method[1)] is not intended for the determination of the stability of gasoline components individually, particularly those with a high percentage of low-boiling unsaturated compounds, as they may cause explosive conditions within the apparatus. However, because of the unknown nature of certain samples, the specified bomb assembly includes a safety burst-disc in order to safeguard the operator.

1.3 The induction period may be used as an indication of the tendency of gasoline to form gum in storage. It should be recognized, however, that this correlation may vary markedly under different storage conditions and with different gasolines.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 breakpoint: Point in the pressure–time curve that is preceded by a pressure drop of exactly 14 kPa within 15 min and succeeded by a drop of not less than 14 kPa in 15 min.

2.2 induction period: Time elapsed between the placing of the bomb in the bath and the breakpoint at 100 °C.

3 Principle

The sample is oxidized in a pressure bomb initially filled at 15 °C to 25 °C with oxygen at 690 kPa and heated at a temperature between 98 °C and 102 °C. The pressure is read at stated intervals or recorded continuously until the breakpoint is reached. The time required for the sample to reach this point is the observed induction period at the temperature of test, from which the induction period at 100 °C may be calculated.

WARNING — To provide protection against the possible explosive rupture of the bomb, the bomb should be operated behind an appropriate safety shield.

4 Reagents and materials

4.1 Toluene, $C_6H_5CH_3$, 99 % minimum purity.

4.2 Acetone, CH_3COCH_3, 99 % minimum purity.

1) Further information can be found in the June 1978, January 1979 and June 1986 editions of the Institute of Petroleum Review.

4.3 Gum solvent, mixture of equal volumes of toluene (4.1) and acetone (4.2).

4.4 Oxygen, commercially available extra-dry, of not less than 99 % purity.

4.5 Detergent cleaning solution, able to clean used sample containers and covers to match the quality with regard to visual appearance and mass loss on heating under test conditions obtained by immersing similar used sample containers and covers in fresh chromic acid cleaning solution for 6 h followed by rinsing with water as specified in 6.1.

NOTE 1 The type of detergent and condition of use need to be established in each laboratory on the basis of cleaning used sample containers and covers.

WARNING — Chromic acid is potentially hazardous in contact with organic materials and is toxic and highly corrosive. If used, wear full-face shield and full-length protective clothing including gloves.

5 Apparatus

5.1 Pressure bomb, of corrosion-resistant steel, with inside dimensions of the portion that encloses the reacting gasoline-oxygen mixture in accordance with those shown in figure 1.

The interior surfaces of the bomb and lid shall be highly polished to facilitate cleaning and to prevent corrosion.

Other structural details, such as method of closure (polygonal or knurled), gasket material and outside dimensions, are optional provided the limitations given in 5.1.1 and 5.1.2 are observed.

Carry out initial testing and periodic examination of the bomb to ensure its fitness for service.

5.1.1 The bomb shall be constructed to withstand a working pressure of 1 240 kPa at 100 °C, with an ultimate strength at least equal to that of a bomb constructed of 18 % mass fraction chromium and 8 % mass fraction nickel-alloy steel. A suitable material is an alloy steel conforming to the specification in annex A.

5.1.2 The closure shall be capable of making a seal that will not leak when the bomb is filled with oxygen to 690 kPa at 15 °C to 25 °C and plunged into a bath at 100 °C. It is preferable that the closure ring be constructed from an alloy different from that of the body if the mating threads of the two parts are to move with respect to each other when the tightening load is applied.

5.2 Gasket, of any suitable material that will pass the following test.

Place a gasket of the type under test in the bomb in the absence of gasoline and use a similar gasket to make the seal with the lid. Fill the bomb with oxygen at a pressure of 690 kPa and immerse in a bath at approximately 100 °C. If the pressure does not drop more than 14 kPa from the maximum in a 24 h period with the bath temperature constant at 100 °C ± 1,0 °C, the gasket shall be considered satisfactory.

5.3 Sample container and cover, in accordance with figure 2.

NOTE 2 The cover is intended to prevent the material refluxing back through the bomb stem from entering the sample, but not to prevent free access of oxygen to the sample.

5.4 Bomb stem, with a filler rod, constructed of the same material as the bomb lid and having dimensions in accordance with figure 1.

The filler rod and the inside of the stem shall have a high polish to facilitate cleaning and prevent corrosion. The stem shall be fitted, in the position shown in figure 1, with a circular metal plate 89 mm in diameter to serve as a closure for the bath when the bomb is in place.

5.5 Burst-disc assembly, of stainless steel, fitted to the bomb stem, which will rupture if subjected to a pressure greater than 1 530 kPa ± 10 %. Any expelled gas shall be directed away from the operator.

5.6 Connection for a pressure gauge and a tightly closing needle valve to the bomb stem as shown in figure 1. A quick-release air coupling fitted to the needle valve shall be used to facilitate oxygen entry to the bomb.

5.7 Needle valve, suitable for complete shutoff, fitted with a finely tapered needle and orifice.

NOTE 3 The needle valve should be used while purging, pressurizing and exhausting the bomb with oxygen.

5.8 Pressure gauge, indicating or recording type, reading to at least 1 380 kPa.

Any half of the scale interval between 690 kPa and 1 380 kPa (i.e. 345 kPa) shall be at least 25 mm in length measured along the arc of the scale. The

intervals of division shall be 35 kPa or less. The accuracy shall be 1 % or less of the total scale interval.

The gauge shall be connected to the bomb directly or by flexible metal or metal-sheathed gas-resistant polymeric tubing having a pressure rating to satisfy the above conditions. The total volume of the flexible tubing, connections and stem with the filler rod in place shall not exceed 30 ml.

When ordering equipment for this test, the manufacturer should be requested to ensure that the pressure gauge and needle valve are suitable for use with oxygen.

5.9 Oxidation bath, containing water, having a capacity of not less than 18 litres for one bomb, plus 8 litres for each additional bomb in multiple assemblies, and of such dimensions that the depth of the bath water is maintained at not less than 290 mm.

The top of the bath shall have openings of diameter suitable to accommodate the bomb and to fit the cover plate fastened to the bomb stem, and shall be provided with a thermometer fixed in such a position that the 97 °C mark of the thermometer is above the cover of the bath.

When in place, the top of the bomb lid shall be submerged at least 50 mm below the surface of the bath water.

Auxiliary lids are needed to cover the openings when the bombs are not in the bath. The bath shall be provided with a condenser and source of heat to maintain the bath water boiling vigorously.

5.10 Thermometer, having a range of 95 °C to 103 °C, in accordance with the requirements in annex B.

5.11 Forceps, of corrosion-resistant steel, spade-ended.

6 Preparation of apparatus

6.1 Wash the glass sample container (5.3) with gum solvent (4.3) until free from gum. Rinse thoroughly with water and immerse the sample container and cover in hot detergent cleaning solution (4.5). Remove from the cleaning solution by means of the forceps (5.11) and handle only with forceps thereafter. Wash the container and cover thoroughly, first with tap water, then with distilled water, and dry in an oven at 100 °C to 150 °C for at least 1 h.

6.2 Drain any gasoline from the bomb (5.1) and wipe the inside of the bomb and lid, first with a clean cloth moistened with gum solvent (4.3) and then with a clean dry cloth.

Remove the filler rod from the stem and carefully clean any gum or gasoline from the stem, rod, and needle valve with gum solvent (4.3). Clean the quick-release air coupling, and all lines leading to the bomb.

WARNING — Ensure all components of the equipment are thoroughly cleaned before storage and re-use to avoid possible formation of volatile peroxides during a test. Any cleaning solutions shall be disposed of in accordance with procedures for the disposal of toxic waste.

The bomb and all connecting lines shall be thoroughly dry before each test is started.

7 Procedure

7.1 Bring the bomb (5.1) and the gasoline to be tested to a temperature of 15 °C to 25 °C. Place the glass sample container (5.3) in the bomb and add 50 ml $\pm$ 1 ml of test sample.

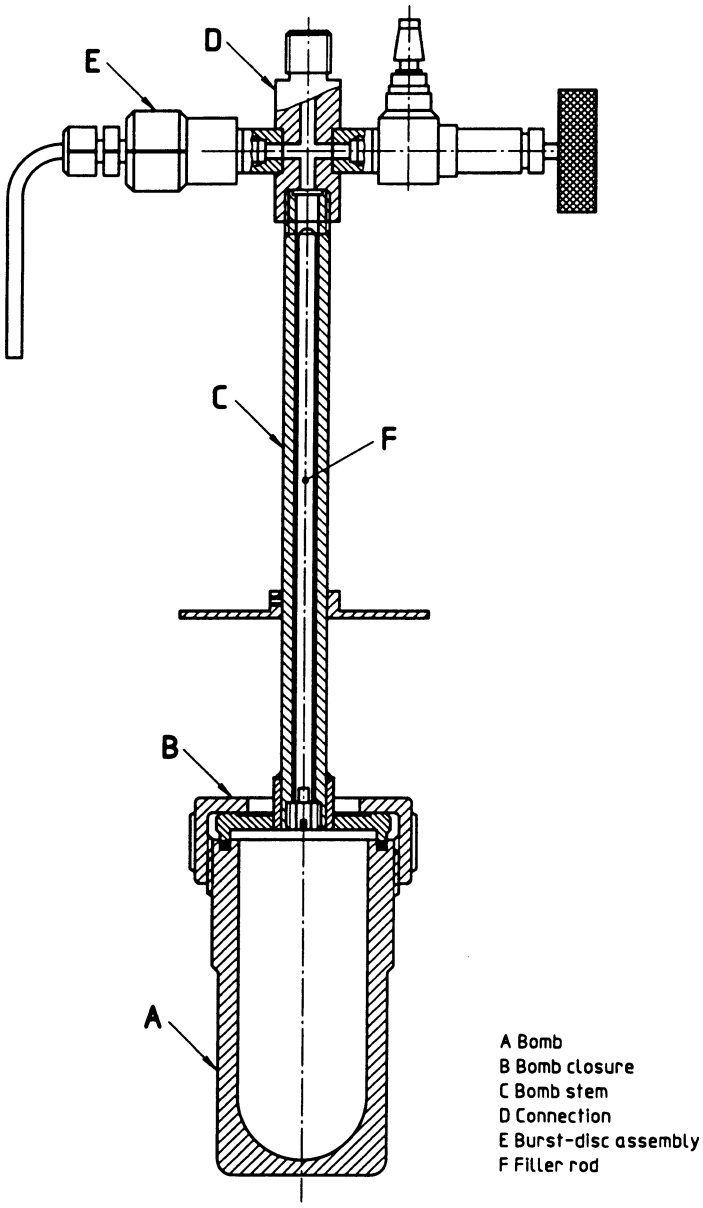
Cover the sample container, close the bomb, and introduce oxygen until a pressure of 690 kPa to 705 kPa is attained. Allow the gas in the bomb to escape slowly at a rate not exceeding 350 kPa per min in order to flush out the air originally present.

Introduce oxygen again until a pressure of 690 kPa to 705 kPa is attained and observe for leaks, ignoring an initial rapid drop in pressure (generally not greater than 40 kPa) which may be observed as a result of dissolution of oxygen in the sample.

If the rate of pressure drop does not exceed 7,0 kPa in 10 min, assume the absence of leaks and proceed with the test without repressuring.

7.2 Place the charged bomb in the vigorously boiling water bath (5.9), taking care to avoid shaking, and record the time of immersion as the starting time. Maintain the temperature of the water bath between 98 °C and 102 °C.

Observe the temperature to the nearest 0,1 °C at intervals during the test, calculate the average temperature to the nearest 0,1 °C and record this as the temperature of the test.



Complete assembly

Figure 1 — Oxidation bomb and burst-disc assembly

Dimensions in millimetres
Tolerance ± 0,25 mm
unless otherwise stated

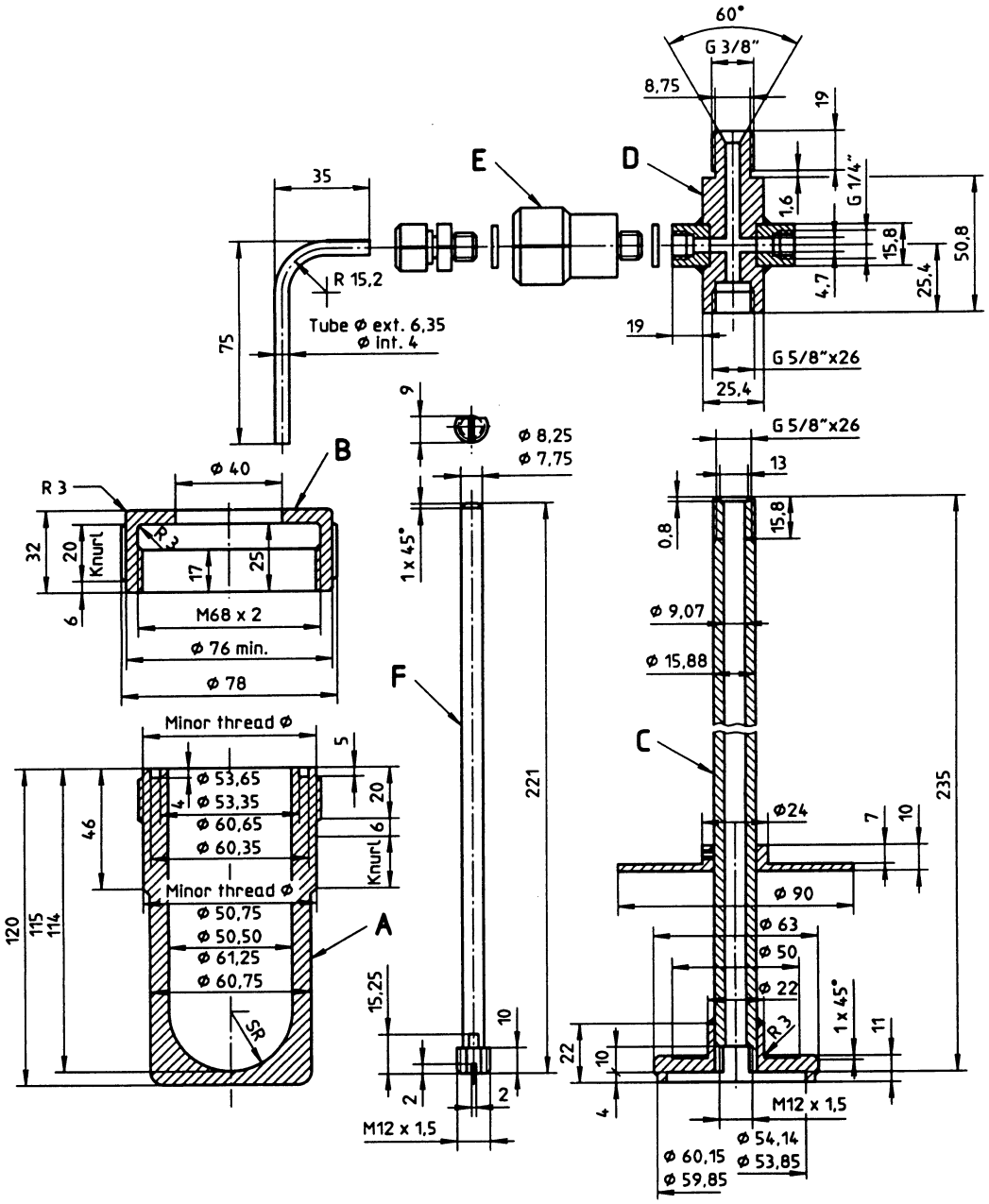


Figure 1 — Oxidation bomb and burst-disc assembly (*end*)

Dimensions in millimetres

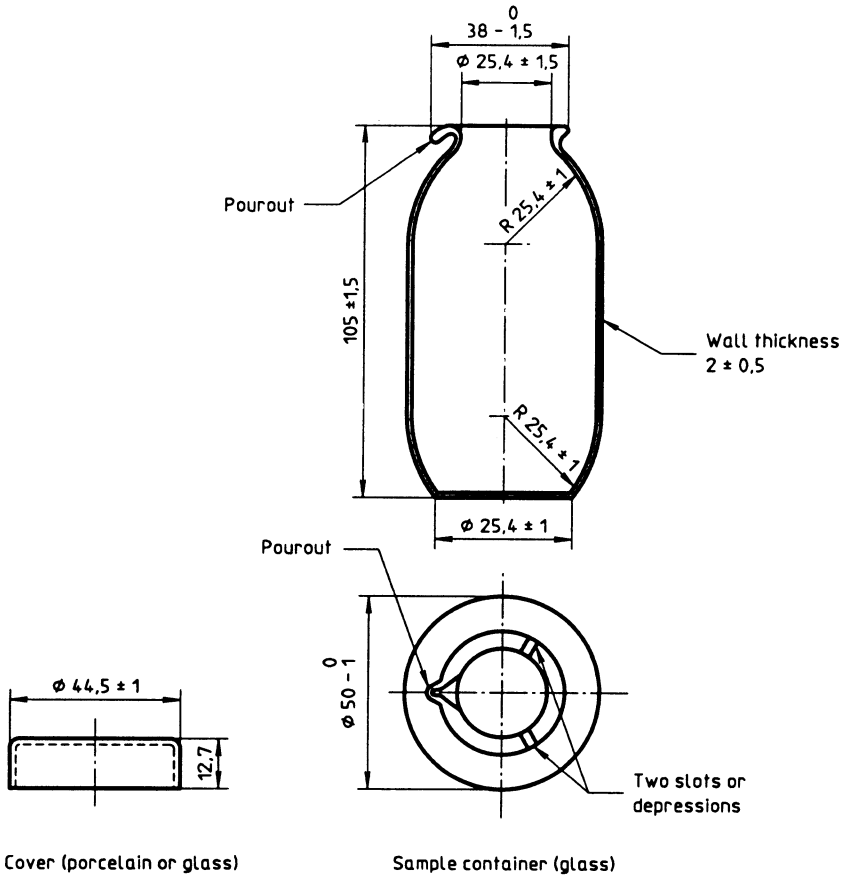


Figure 2 — Sample container (glass) and cover (glass or porcelain)

Make a continuous record of the pressures in the bomb or, if an indicating pressure gauge is used, take pressure readings at intervals of 15 min or less.

If, during the initial 30 min of the test, a leak develops (as indicated by a steady drop in pressure considerably in excess of 14 kPa in 15 min), discard the test.

Continue the test until the breakpoint is reached, which is that point preceded by a pressure drop of exactly 14 kPa within 15 min and succeeded by a drop of not less than 14 kPa in 15 min.

Record the number of minutes from the time the bomb is placed in the bath until the breakpoint has been reached as the observed induction period at the temperature of the test.

NOTE 4 If the test is made in an environment where the atmospheric pressure is consistently below normal (101,3 kPa), it is permissible to add a higher-boiling liquid such as ethylene glycol to the water in order to maintain the operating temperature of the bath at approximately 100 °C.

7.3 Cool the bomb by submerging it in cool tap water. Remove the bomb from the water when cooled and release the pressure slowly from the bomb through the needle valve at a rate not exceeding 350 kPa/min. Clean the bomb and sample container in preparation for the next test.

8 Calculation

Calculate the induction period at 100 °C, IP_{100}, expressed in minutes, from one of the following equations.

— When the test temperature is above 100 °C:

$$IP_{100} = (IP_t)[1 + 0{,}101(T_a - 100)]$$

— When the test temperature is below 100 °C:

$$IP_{100} = (IP_t)/[1 + 0{,}101(100 - T_b)]$$

where

IP_t is the observed induction period, in minutes, at the temperature of the test;

T_a is the test temperature, in degrees Celsius, when above 100 °C;

T_b is the test temperature, in degrees Celsius, when below 100 °C.

9 Expression of results

Report the induction period at 100 °C, IP_{100}, calculated as in clause 8 to the nearest whole minute.

10 Precision

The precision of the method, obtained by statistical examination of interlaboratory test results, is given in 10.1 and 10.2 .

NOTE 5 The precision reported was determined using equipment not incorporating a burst-disc assembly. However, a consideration of the design, and some limited testing indicate that any effect of such an assembly is likely to be minimal.

10.1 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, differ from the mean by more than 5 % only in one case in twenty.

10.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, differ from the mean by more than 10 % only in one case in twenty.

11 Test report

The test report shall contain at least the following information:

a) sufficient details for the complete identification of the product tested;

b) a reference to this International Standard;

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)

Chromium, nickel-alloy steel — Specification

The corrosion-resistant steel used for the construction of the oxidation bomb (5.1) shall meet the ultimate strength requirements of a similar bomb constructed with a steel meeting the specification given in table A.1.

Table A.1 — Corrosion-resistant steel specification

Steel component	Mass fraction %
Carbon	0,08 max.
Manganese	2,00 max.
Phosphorus	0,045 max.
Sulfur	0,030 max.
Silicon	1,00 max.
Chromium	18,00 to 20,00
Nickel	8,00 to 10,50
Nitrogen	0,10 max.

Annex B
(normative)

Thermometer specification

The thermometer (5.10) shall meet the specifications given in table B.1.

NOTE 6 Thermometer ASTM 22C/IP 24C meets these requirements.

Table B.1 — Oxidation stability thermometer specifications

Range	95 °C to 103 °C
For test at	100 °C
Immersion	Total
Total length	270 mm to 280 mm
Stem o.d.	6,0 mm to 8,0 mm
Bulb length	25 mm to 35 mm
Bulb o.d.	> 5,0 mm and < stem
Scale location:	
Length from bottom of bulb to line at 95 °C	135 mm to 150 mm
Length of graduated portion	70 mm to 100 mm
Graduations:	
Subdivisions	0,1 °C
Long lines at each	0,5 °C
Numbers at each	1 °C
Scale error	0,1 °C max.
Expansion chamber, of volume to permit heating to	155 °C
Contraction chamber:	
Distance to top	60 mm max.
Stem enlargement:	
o.d.	8,0 mm to 10,0 mm
Length	4,0 mm to 7,0 mm
Distance to bottom	112 mm to 116 mm

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