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Specification for

Calorimeter bombs

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Foreword

This revision of BS 4791 has been prepared under the direction of the Laboratory Apparatus Standards Committee. It supersedes BS 4791:1972, which is withdrawn.

The 1972 edition specified requirements for calorimeter bombs used for determining the calorific values and heat of combustion of various fuels and substances. Experience gained since then has justified the revision of this standard, with particular emphasis on safety requirements.

It is a matter of great concern that, from time to time, calorimeter bombs have been submitted for retest or overhaul in a deplorable condition. The faults range from serious damage to parts of the pressure vessel, to deep corrosion in the firing chamber. It is also evident that some combustion bombs have been withdrawn from service too soon. Bomb bodies are discarded because of worn closure threads, although these could be recut and fitted with a new smaller closure ring.

While some users fire their calorimeter bombs only occasionally and deterioration with wear is not likely to be noticeable for several years, other users fire frequently and require a simple, routine, assessment of the wear of the threads as the ring becomes a looser fit on the body. Appendix C describes a method of measurement of slackness of fit of these threads.

In this revision the specified quality of initial fit of the closure threads has been improved. A somewhat relaxed tolerance beyond which the used vessel has to be withdrawn from service has now been given.

The 1972 edition set a limit of 2 000 firings before overhaul: that figure may now be extended by steps, always provided that the maximum tolerance on slackness of fit is not exceeded. The period of validity of the report on a new calorimeter bomb has been correspondingly extended from three to four years.

Previously, rings were untested copies of an approved prototype. In this revision, the proving test on the body is applied to the body and its ring simultaneously. As the input valve has to be replaced at times, the leakage test for valves has been separated appropriately from the proving test.

In this edition, all the previous concern for safety in use has been retained and, further, the risk of damage to the vessel by chemically reactive products is now emphasized.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This British Standard specifies requirements for materials, construction, design and performance for calorimeter bombs used for the combustion of materials as a means of determining their calorific value and the elements present in the products of combustion.

Precautions for the safe use of the bomb and its ancillary equipment, together with routine maintenance procedures for keeping the calorimeter bomb in serviceable condition, are described in Appendix B, Appendix C and Appendix D.

CAUTION. This standard does not apply to calorimeter bombs for the determination of the calorific value of explosives or other samples if a pressure exceeding 50 bar¹⁾ could be generated.

NOTE 1 Calorimeter bombs in accordance with BS 4791:1972 were approved to tests different from those described in this revision. Retests should be made on the basis of the original test applied and reported accordingly. However, when bomb calorimeters are reconditioned and/or new rings are fitted, it is recommended that the requirements of this current edition be observed.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definition

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

calorimeter bomb

a high pressure vessel used for the determination of calorific values of combustible substances such as solid, liquid and gaseous fuels, foodstuffs and biological samples

NOTE The calorimeter bomb consists of a body, a cap and a closure ring. The test sample is contained in a crucible suspended from the cap. Oxygen is introduced through a valve in the cap and the sample is fired using electrodes that pass through the cap. WARNING NOTE. A known quantity of the given material is burned in the bomb and the heat released by the combustion is measured using a calorimeter. For most materials, it is necessary to introduce oxygen under pressure to ensure complete combustion. However, certain materials, such as pyrotechnics, generate sufficient oxygen for this purpose on combustion, and the introduction of gaseous oxygen in addition to that in the atmosphere is not therefore required and may be dangerous.

3 Design

3.1 The design shall be such that the bomb can be sealed and opened by hand without the use of tools of any kind.

3.2 The calorimeter bomb seal shall be so designed that, after tightening by hand, an increase of pressure in the bomb will tighten the seal and prevent leakage. Similarly, the fixings of the valve and the electrodes in the cap shall be so designed as to be leakproof when the bomb is fired or brought up to proving pressure.

4 Materials

4.1 Bomb cap and body

The bomb cap and body shall be made of a material that is capable of withstanding the pressures generated by the combustion process and that shall not be corroded by the products of combustion of the test samples.

The bomb cap and body shall each be machined from solid or hollow forgings or bar; they shall not be fabricated from components welded or brazed together.

For a bomb of the design illustrated in Figure 1 and with the minimum dimensions and dimensional ratios given in Table 1, the material shall have the following mechanical properties.

- a) A tensile strength of not less than 490 N/mm², when tested in accordance with BS 18-1.
- b) A yield stress of not less than 210 N/mm 2 , when tested in accordance with BS 18-1.
- c) An elongation at break of not less than 30 %, when tested in accordance with BS 18-1.
- d) An Izod impact value of not less than 68 J, when tested in accordance with BS 131-1.

If the bomb cap and body are made of stainless steel, the steel shall have passed the 90° bend test described in clause **23** of BS 1503:1980.

NOTE If the bomb cap and body are made of a material other than stainless steel, the material supplier should certify that the material has passed a correspondingly suitable test for resistance to intercrystalline corrosion.

4.2 Closure ring

The closure ring shall be made of material, such as aluminium bronze, capable of withstanding the pressure generated by the combustion process.

 ${\bf NOTE}\quad {\bf Materials\ should\ be\ chosen\ to\ minimize\ galling\ or\ seizing\ of\ the\ thread\ engaging\ the\ bomb\ body.}$

¹⁾ 1 bar = 100 kPa.

The closure ring shall be machined from solid or hollow forgings or bar. It shall not be fabricated from components welded or brazed together.

For a bomb designed to the minimum dimensions and dimensional ratios given in Table 1, the material of the closure ring shall have the following mechanical properties.

a) A tensile strength of not less than $520\ N/mm^2$, when tested in accordance with BS 18-1.

b) An elongation at break of not less than 20 %, when tested in accordance with BS 18-1.

4.3 Plating

The mating screw threads of the body and closure ring shall not be electroplated.

 NOTE The external surfaces of the closure ring may be electroplated to provide protection against corrosion.

Table 1 — Dimensions for bomb body, cap and closure ring: thread specification

Detail	Symbol (see Figure 1)	Requirement
Body External diameter (excluding any knurled section) Internal diameter Capacity of the chamber Thickness of side wall Thickness of base Internal radius	D t T E	Uniform to within $0.03~\mathrm{mm}$ Not less than $55~\mathrm{mm}$ $325 \pm 25~\mathrm{mL}$ Not less than $0.10D$ Not less than $0.165D$ Not less than t
Closure ring Overall height Dimensions of closure ring if made of aluminium bronze:		Uniform to within 0.03 mm
Thickness of wall Thickness of flange	$A \\ B$	Not less than 0.14D Not less than 0.17D
Screw thread Nominal pitch and thread form Fit		Either ISO metric threads of 1.5 mm pitch complying with BS 3643, or Unified threads of 16 tpi complying with BS 1580-1 and BS 1580-2. The roots shall be radiused The difference in pitch diameter between the two assembling screw threads shall not exceed 0.10 mm before the calorimeter is put into service initially
Length of engagement	C	Not less than $0.25D$
Cap Thickness of cap flange Total thickness of cap	F	Not less than $0.10D$ Not less than $0.25D$
Crucible position related to cap Position of base of crucible to underside of cap		Not less than 90 mm
Total mass		Not greater than 3.25 kg

4.4 Sealing ring

The ring used to seal the cap to the body shall be inert to the materials which may come into contact with the calorimeter bomb (see 4.1).

NOTE The ring should be made of suitable resilient material.

4.5 Valves

The valve or valves which are used for the admission and/or release of oxygen shall be suitable for use with pressures of up to 100 bar within the bomb.

NOTE Self-sealing valves fitted with a high pressure valve core have been proved by experience to be suitable. For information on the availability of suitable self-sealing valves, apply to Enquiry Section (London), British Standards Institution, enclosing a stamped addressed envelope for reply.

If a self-sealing valve is fitted, it shall be provided with a screw threaded cap to cover its inlet.

4.6 Electrodes and sample crucible

The electrodes and sample crucible shall be made of materials that are capable of withstanding the heat generated by the combustion process and that will not be corroded by the products of combustion of the test samples.

NOTE Details of the crucible to use for a given sample material will usually be given in the appropriate material test method.

5 Dimensions and construction

5.1 The essential dimensions shall be as given in Table 1.

NOTE The symbols for these dimensions are also shown in Figure 1, which illustrates a typical design of calorimeter bomb

- **5.2** The roots of the knurling and the closure threads and any undercuts at the end of the threaded portions shall not bring the bomb below the minimum wall thickness, as given in Table 1.
- **5.3** Apart from the knurling, the surface of all metal parts shall have a smooth finish throughout. The inside of the combustion chamber shall be free from ledges or crevices that could retain contaminating material.
- **5.4** All sharp edges shall be removed and corners shall be radiused to 0.75 mm minimum.

6 Performance

6.1 Resistance to hydraulic deformation and leakage

6.1.1 Hydraulic deformation (proving test). When tested in accordance with **A.1.1**, the bomb shall not show visible signs of leakage. The closure ring shall couple and uncouple smoothly and shall not show signs of galling.

When tested in accordance with A.1.2 and A.1.3, the application and release of the pressure shall not cause permanent deformation of the body or of the closure ring, subject to a tolerance of 0.02 mm either on the increase in diameter of the body or on the increase in height of the ring to allow for the estimated measurement uncertainty.

6.1.2 *Leakage*. If the requirements of **6.1.1** are met, the bomb shall be tested in accordance with **A.2**, and during the test period no bubbles attributable to leakage shall appear.

A similar leakage test shall be made whenever the valve is replaced; see also **D.2.4**.

NOTE This test may be undertaken by the user.

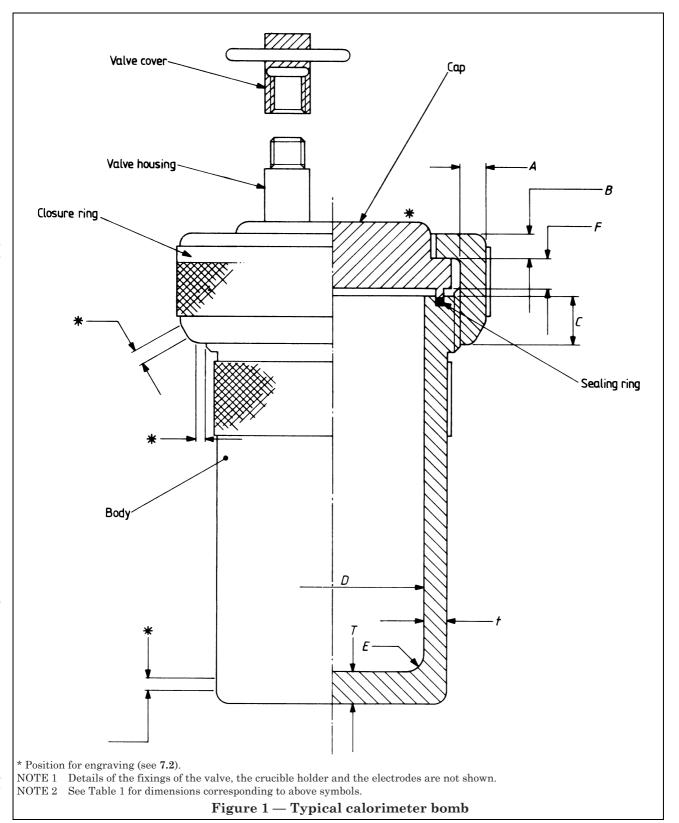
6.2 Fit of screw threads

When tested in accordance with Appendix C, the calorimeter bomb shall either not have an axial slackness of more than 0.06 mm initially or 0.32 mm finally or shall not have a diametral slackness of more than 0.55 mm finally (see Figure 2).

7 Marking

- **7.1** Each calorimeter bomb shall be marked with the following:
 - a) number and date of this British Standard, i.e. BS 4791:1985²⁾;
 - b) identification number, marked indelibly on the bomb body, cap and closure ring in the position indicated by asterisks in Figure 1;
 - c) test pressure of 300 bar;
 - d) date of pressure test and initials of testing authority.

²⁾ Marking BS 4791:1985 on or in relation to a product is a claim by the manufacturer that the product has been manufactured in accordance with the requirements of the standard. The accuracy of such a claim is therefore solely the manufacturer's responsibility. Enquiries as to the availability of third party certification to support such claims should be addressed to the Director, Quality Assurance Division, BSI, PO Box 375, Milton Keynes MK14 6LO in the case of certification marks administered by BSI or to the appropriate authority for other certification marks.



7.2 If engraving or stamping is used for marking the calorimeter body, it shall be confined to the position indicated by the asterisk in Figure 1 and shall be completely within the thickness of the base.

NOTE Marking by electric etching is also permissible.

8 Supply of information

- **8.1** The following information shall be supplied with the calorimeter bomb.
 - a) The identification number of the bomb.
 - b) The British Standard reference (if any), the analysis, and the mechanical properties of the material from which the bomb body and cap were made. If the material is stainless steel, it shall be confirmed that it has satisfied the 90° bend test for intercrystalline corrosion (see 4.1).
 - c) The date of the proving test and the gas leakage test, the test pressures (i.e. 300 bar and 40 bar respectively), the testing authority and the result of the test.
 - d) The British Standard reference (if any), the analysis, and the mechanical properties of the material from which the closure ring was made.
- **8.2** The report for the pressure proving test shall be valid for not more than four years. A new report shall be issued on re-testing.

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Appendix A Methods of test

A.1 Proving test

A.1.1 *General.* Subject the bomb to an internal pressure of 300 bar and maintain this pressure for 10 min. If the oxygen inlet valve is of the self-sealing type, remove the valve for this test.

NOTE After the proving test it may be necessary to use tools to open the bomb. So doing does not constitute failure to comply with 3.1 because the proving pressure is much higher than normal operating pressures.

A.1.2 Measurement of body diameter

Measure the diameter of the body at a minimum of eight located positions, evenly distributed. Check the consistency of reading and continuity, taking readings on a setting rod at the outset of the test and during a series of measurements.

In order to permit the necessary accurate measurements on body and ring, ensure that their external surfaces are protected from damage at all times.

NOTE If a setting rod is not available, a solid part of the body, such as that indicated by *T* in Figure 1, may be used as control. **A.1.3** *Measurement of height of closure ring*. With the lower narrow face of the ring standing on a datum surface, such as a surface plate complying with the requirements of grade 1 of BS 817, explore the height at a minimum of eight positions, four along each of two diameters at 90° and alternately positioned as near as practicable to the outer and inner circumferences of the top annular face. Check the consistency of reading and continuity with gauge blocks or other precision setting pieces at the initial and later stages of the proving test.

A.2 Gas leakage test

After the proving test, subject each calorimeter bomb to a gas leakage test using air or oxygen at a pressure of 40 bar. When the bomb is at this pressure, remove it from the gas supply and immerse it in a bath of cold water for a period of 10 min.

Take care not to confuse air escaping from intercomponent spaces with leakage. If leakage is suspected, empty the bomb, examine the suspect region and tighten or replace seals as necessary, and then repeat the test.

Appendix B Safeguarding, periodic re-checking, overhaul and retest

B.1 The calorimeter bomb is a high pressure vessel and should be handled with care at all times to avoid damage.

The materials described in clause 4 have proved to be suitable for a wide variety of test samples. However, some samples, such as those which release chlorine, have been found to corrode steel bombs. Users are advised to check beforehand the likely reactions of test samples so as to avoid running the risk of damaging their combustion bombs

Deterioration of the vessel can be minimized by keeping the parts clean. In particular, the inside of the body should be cleansed from the products of combustion as soon as the observations are completed. Any deterioration of the surface should be reported by the operator so that the circumstances may be investigated. The screw threads should be cleaned carefully and thoroughly with a tooth brush. Any damage to the threads should be rectified by a skilled operator.

B.2 It is recommended that the user of the calorimeter bomb should keep a log of the number of firings and record the dates and results of inspections and measurement checks.

The outside diameter of the body should be measured regularly so that any distortion, which would necessitate withdrawal of the bomb from use, is detected when it occurs. The frequency of such measurements should be related to the frequency of firing; they should be made weekly if a bomb is in continual daily use.

With use, the threads of the body and closure ring wear, particularly on the first turns of thread to engage. However, when the quality of fit at partial assembly has declined to the limit of 7H/8g (free fit)³⁾, it is essential that the bomb be taken out of service. It may then be checked for suitability for correction of the body thread to within the side wall thickness given in Table 1, and matching a new closure ring of correspondingly reduced thread diameter.

NOTE 1 $\,$ Testing the screw thread for wear is described in Appendix C.

NOTE 2 For a thread of about 50 mm diameter \times 1.5 mm pitch, the maximum difference of pitch diameter for 7H/8g free fit is 0.547 mm.

³⁾ See BS 3643-2, which tabulates the limits of tolerance on diametral difference between internal and external threads in assembly.

B.3 It is essential that development of slackness of fit of a bomb be checked after not more than 1 000 firings and subsequently at intervals not exceeding 500 firings and the vessel should be withdrawn as soon as the threads are too slack; this surveillance may be undertaken within the user's organization provided that the equipment and expertise are available and responsibility is defined. It is imperative that a calorimeter bomb be re-certified by the manufacturer or by an independent testing authority at intervals not exceeding 4 years (see **8.2**).

B.4 When the calorimeter bomb is overhauled and a new ring fitted, it is imperative that the assembly be subjected before use to a further proving pressure test as described in **A.1**, a report recording the latest information be supplied, and the bomb be marked with the latest date of test [see **7.1** d) and **8.1** c)].

Appendix C Method of measurement of slackness of fit of closure ring and body

NOTE This appendix gives recommendations for users of calorimeter bombs on checking wear of the threads.

C.1 Preliminaries

The threads should first be cleaned and inspected for damage such as burrs, bruising of the metal or roughness due to galling. Local damage should be corrected by skilled attention before further tests.

C.2 Direct measurement of slackness of fit in the diametral or axial directions

C.2.1 The closure ring and cap should be assembled fully with the body and the number of turns of the ring required to close the bomb counted. The ring and cap should be removed, and the ring, without the cap, should then be assembled on to the body using four turns less than the full number of turns previously counted.

C.2.2 The bomb body should be stood on a surface plate and held down firmly. An engineer's dial gauge capable of reading to an accuracy of at least 0.02 mm and mounted on a sturdy stand should be set in contact with a finished surface of the ring as shown in Figure 2. With the bomb body held immovably, the closure ring should be moved between its two extreme positions diametrally or axially and the amount of slackness observed on the dial gauge.

C.2.3 If there is a plain surface on the side of the ring, it is preferable to measure diametral slackness. If, however, knurling of the side prevents side registration, then axial slackness may be measured.

C.2.4 The ring should not be rotated; when conducting the axial test a plastics ring should be used with a free-running fit on the bomb body to displace the closure ring.

C.2.5 To measure the diametral slackness (where this is possible), five readings should be taken at a position on the periphery of the ring and then a further five readings at a position 90° to the original. The mean of the total of 10 readings should then be taken in order to establish the diametral slackness.

C.2.6 To measure the axial slackness, readings should be taken on the top surface of the closure ring, with one reading at each of 10 positions evenly distributed around the ring. The mean of the 10 readings should be ascertained for the purpose of establishing the axial slackness.

Appendix D Safe use of a calorimeter bomb and its ancillary equipment

D.1 General

It is essential that attention be paid to the safety measures given in any method for the determination of a calorific value and particular attention be paid to prescribed precautions when testing volatile liquid fuels.

Emphasis is laid on the following procedures in the use of oxygen filling equipment and firing equipment.

D.2 Oxygen filling equipment

D.2.1 It is imperative that the filling system includes control valve, a pressure-operated safety device and a pressure gauge.

D.2.2 The control valve may be of the single stage diaphragm type or of the needle type.

D.2.3 The safety device may be either a valve or a bursting disc. Set the device to operate at 2.5 bar above the working pressure specified in the test method for the sample, provided that the setting does not exceed 42.5 bar.

D.2.4 The pressure gauge should be of the safety pattern described in BS 1780 with a range of at least 50 bar and an accuracy of \pm 2 bar at 35 bar.

It should be checked annually and also at any time if its accuracy is suspect.

It is essential that oil should not be used when checking this gauge, which should be marked "USE NO OIL".

D.2.5 If a calorimeter bomb has been inadvertently overcharged with oxygen, abandon the test and DO NOT FIRE THE CHARGE.

D.2.6 DO NOT fire the charge if there are gas leaks when the bomb is immersed in the water in the calorimeter.

D.2.7 It is strongly recommended that the oxygen cylinder be situated outside the room or enclosure containing the bomb to reduce the risk of explosion.

D.3 Quantity of sample

In every case the quantity of sample placed in the bomb should be kept within the limit specified in the relevant test method. As a general guide the heat release should not exceed 100 J for each mL capacity of the bomb.

D.4 Ignition circuit

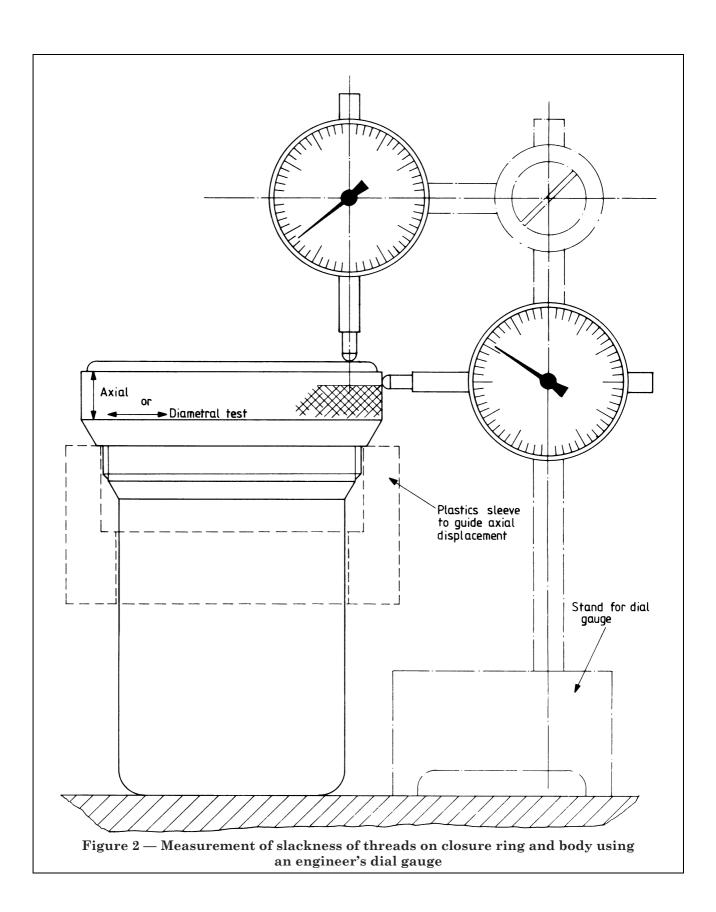
D.4.1 The firing circuit should be controlled by a switch that is spring-loaded (biased) to return to the "contacts open" condition when its operating lever is released.

D.4.2 The ignition voltage should not exceed 24 V. If the ignition voltage is derived from the mains supply, a double wound (isolating) transformer with an earthed interwinding screen should be used.

D.4.3 The firing button should be in such a position that the operator can stand back and fire the bomb without having to reach over it. A remote firing position, e.g. behind a protective wall or in another room, is recommended, especially when testing liquid fuels.

D.4.4 It is desirable to include an ammeter or a pilot light in the firing circuit to indicate when current is flowing. A 5A fuse should also be included.

D.4.5 Do not approach the bomb until 20 s after firing.



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Publications referred to

- BS 18, Methods for tensile testing of metals.
- BS 18-1, Non-ferrous metals.
- BS 131, Methods for notched bar tests.
- BS 131-1, The Izod impact test on metals.
- BS 817, Specification for surface plates.
- BS 1503, Specification for steel forgings (including semi-finished forged products) for pressure purposes.
- BS 1580, Specification for Unified screw threads.
- BS 1580-1 and BS 1580-2, Diameter ¼ in and larger.
- BS 1780, Specification for Bourdon tube pressure and vacuum gauges.
- BS 3643, ISO metric screw threads.
- BS 3643-1, Principles and basic data.
- BS 3643-2, Specification for selected limits of size.

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