BRITISH STANDARD

BS 1460:1967

Incorporating Amendment No. 1

Method for

Determination of apparent density after compaction of precipitated calcium carbonate

Co-operating organizations

The Chemicals Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives of the following Government departments and scientific and industrial organizations:

Board of Trade
British Iron and Steel Federation
Chemical Industries Association*
Fertilizer Manufacturers' Association
Gas Council
Institute of Vitreous Enamellers
Institution of Gas Engineers
Ministry of Defence, Army Department
Ministry of Health
National Sulphuric Acid Association
Royal Institute of Public Health and Hygiene

The industrial organization marked with an asterisk in the above list, together with the following, was directly represented on the committee entrusted with the preparation of this British Standard:

British Laboratory Ware Association
British Paper and Board Makers' Association (Incorporated)
British Polish Manufacturers' Association
British Waterworks Association
London Chamber of Commerce (Incorporated)
Proprietary Association of Great Britain
Toilet Preparations Federation
Welwyn Hall Research Association

This British Standard, having been approved by the Chemicals Industry Standards Committee and endorsed by the Chairman of the Chemicals Divisional Council, was published under the authority of the General Council on 18 January 1967

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First revision January 1967

The following BSI references relate to the work on this standard:
Committee reference CIC/9
Draft for comment 66/1471

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Amendments issued since publication

Amd. No.	Date of issue	Comments
6199	July 1967	Indicated by a sideline in the margin

Contents

Foreword

This standard makes reference to the following British Standards:

BS 410, *Test sieves.*

BS 903, *Methods of testing vulcanized rubber* — Part A7: *Determination of hardness.*

This British Standard was first issued in 1948, (under the authority of the Fine Chemicals Industry Standards Committee, now incorporated into the Chemicals Industry Standards Committee), when it was developed from a test which had been in industrial use for many years[1)]. The method consists of ascertaining the bulk of a known mass of a prepared sample of precipitated calcium carbonate after compaction by a standardized procedure.

Following a considerable amount, of discussion and joint testing by eleven collaborating laboratories, it proved possible at that time to set the method down in greater detail than had been previously possible and to declare the order of reproducibility of results which was to be expected for different grades of precipitated calcium carbonate.

In making these tests three grades of precipitated calcium carbonate, having apparent densities after compaction (g/ml) about 0.30 (Sample A), 0.50 (Sample B), and 0.80 (Sample C), were used.

The results of the statistical analysis of the data obtained are set out in the Appendix.

The inference drawn from these tests was that, provided the same operator made the determinations regularly without conscious variation of technique, the reproducibility of results obtainable, particularly in triplicate tests, was quite good, having regard to the known difficulties inherent in this type of test.

When comparison of results obtained in different laboratories was considered, difficulty arose particularly with precipitated calcium carbonate such as Sample A. This was due to the fact that some feature of the test (probably associated with the transfer of the sample to the cylinder, or to the methods of sieving the precipitated calcium carbonate), was not readily susceptible of description and standardization in such a way as to be perfectly repeatable by different operators.

It was felt, however, that the issue of this standard method would serve a useful purpose in eliminating avoidable discrepancies and, in conjunction with the statements made above, in drawing attention to the limitations inherent in the method and indicating the degree of accuracy that could be expected in practice.

In the present revision, details of the apparatus, in particular of the cylinder, are given more specifically, as it has been found that the supply of suitable cylinders has proved a difficulty. All measurements have been given in metric units. In Figure 1 these should not be interpreted as requiring the apparatus to be made with more precision than is usual in such carpentry work and apparatus made to the original drawings is still suitable for use.

Consideration has been given to the mechanization of the method, but no mechanical apparatus has been specified. Laboratories wishing to mechanize the procedure, should ensure that the apparatus faithfully copies the manual operations and that a suitable test programme is followed to show correspondence of results between the manual and mechanised methods.

[1)] Originally devised by Messrs. John and E. Sturge, Ltd.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 6 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 describes a method for the determination of apparent density after compaction of precipitated calcium carbonate.

Although the method has been drafted to meet the special requirements for testing precipitated calcium carbonate, it is recognized that it is often applied to other powders. In these cases it is necessary to assess the details of the test in relation to specific requirements, giving due consideration to the desired emphasis on discrimination between grades as against reproducibility. In this assessment it should be remembered that increasing the number of drops usually improves the reproducibility at the expense of discrimination, and consideration should be given to the possibility of using a different weight of test sample. For powders other than precipitated calcium carbonate it may be possible to relax some of the precisely defined conditions of handling the sample.

NOTE If test results are reported on such a modified test they should be accompanied by a statement in the form: "Tested generally in accordance with BS 1460, but using X g of sample and Y drops".

2 Apparatus[2)]

The apparatus required, the special parts of which are depicted diagrammatically in Figure 1, Figure 2 and Figure 3 comprises:

a) *Cylinder,* glass with a solid base ground flat at right angles to the axis of the cylinder. It shall be graduated by 2 ml subdivisions over the range 20 ml to 250 ml. The graduations shall be permanently numbered at 10 ml intervals. The maximum permissible error in the graduations at any point shall be ± 0.6 ml provided that the difference between error at any two points does not exceed 0.6 ml. The internal depth from the 250 ml graduation mark to the base shall be 220 mm to 240 mm. The empty cylinder together with its rubber bung, shall weigh 250 ± 5 g.

b) *Dropping box,* secured to a rigid bench or table. The distance between the flat ground base of the cylinder and the rubber base pad, when the cylinder is raised to the full height, shall be 25 ± 1 mm, i.e. the total lift of the cylinder shall be corrected to 25 ± 1 mm by suitable adjustment of the shelf.

The rubber base pad shall have a nominal thickness of 5–6 mm and a hardness of 70 – 85 I.R.H.D.[3)]

c) *Timing device,* to indicate seconds audibly.

NOTE This may conveniently be either a metronome or a stop clock.

d) *Balance* or *scales,* of a type providing easy access to the pan or pans, which should be at least 100 mm in diameter. It is necessary that the pointer should show a significant deflection for a change in load of 0.25 g.

e) *Sampling paper,* black glazed.

f) *Finger stalls,* smooth rubber.

g) *Sieve*[4)], nominal aperture width 500 microns, known as mesh number 30.

3 Procedure

a) Weigh out approximately 42 g of a fresh portion of the material on a piece of paper.

b) Fold a piece of blank glazed sampling paper 250 × 250 mm with two parallel creases to form a channel 13 mm wide down the middle of the paper on its glazed side. Place this paper on the scale and counterpoise either with a similar sheet or by other suitable means.

c) Place the sieve on the prepared paper on the bench, in such a manner that there is a 50 mm space between the gauze and the paper.

d) Transfer the precipitated calcium carbonate to the sieve and with the back of the fingers fitted with rubber finger-stalls lightly rub all the calcium carbonate through the sieve, using a short stroke. If the sieve tends to clog, raise it about 25 mm and tap the edge lightly with the fingers (but not against the bench)

e) Transfer the paper with the sieved sample to the balance and adjust the mass of the sample to 40 ± 0.25 g.

f) Pick up the paper with the sieved sample and form it into a chute.

[2)] The special parts of the apparatus are available commercially from Messrs. Jencons (Scientific) Limited, Hemel Hempstead, England.
[3)] BS 903, *"Methods of testing vulcanized rubber"* — Part A7: *"Determination of hardness"*.
[4)] BS 410, "*Test sieves*".

Allow the paper to lie between the thumb and fingers on the palm of the hand and introduce it for about 13 mm into the cylinder, which should be held in the other hand and inclined at an angle of about 45° to the horizontal. Slip the calcium carbonate into the cylinder smoothly and without jerking. Any tendency towards sticking may be overcome by gently tapping the bottom end of the chute with a finger. On no account shall the cylinder be knocked or jolted, nor shall the calcium carbonate in the paper be squeezed during the filling of the cylinder.

g) Fit the rubber bung into the cylinder without jolting.

h) Gently place the cylinder in the dropping-box and secure it.

j) Start the timing device.

k) With the thumb and forefinger of one hand gently take hold of the upper part of the cylinder and during one second lift it to the full extent of its travel. Avoid any undue impact against the upper stop so that no jar is given to the calcium carbonate.

l) At the commencement of the next second, smartly release the cylinder by quickly and completely withdrawing the thumb and forefinger.

m) Continue the process of lifting and dropping until 50 counted drops are completed, the cylinder falling once every two seconds. Rotation through an arc of about 10° should be given during the lifting that precedes each drop, since this will help to impart a level surface to the calcium carbonate for the final volume reading.

n) Immediately the 50 drops are completed remove the cylinder from the dropping-box, raise it to eye level and note volume to the nearest 1 ml (V). Any further drop in level after standing should be ignored.

4 Calculation

Apparent density after compaction,

$$\text{grammes per millilitre} = \frac{40}{V}$$

where V = volume occupied by the 40 g of sample.

This result shall be expressed to two significant figures.

Appendix Summary of results

	Sample A	Sample B	Sample C
i) Mean result over all laboratories (See Note 2.)	**118.3**	**73.3**	**49.0**
ii) Standard deviation within laboratories	1.21 × 1.03 %	0.10 = 0.55 %	0.25 = 0.45 %
iii) Standard deviation of mean of three tests within laboratories	0.70 × 0;6 %	0.23 × 0.3 %	60.13 = 0.3 %
iv) Standard deviation of mean of three tests between laboratories	6.5 = 5.5 %	1.71 = 2.3 %	1.04 = 2.1 %

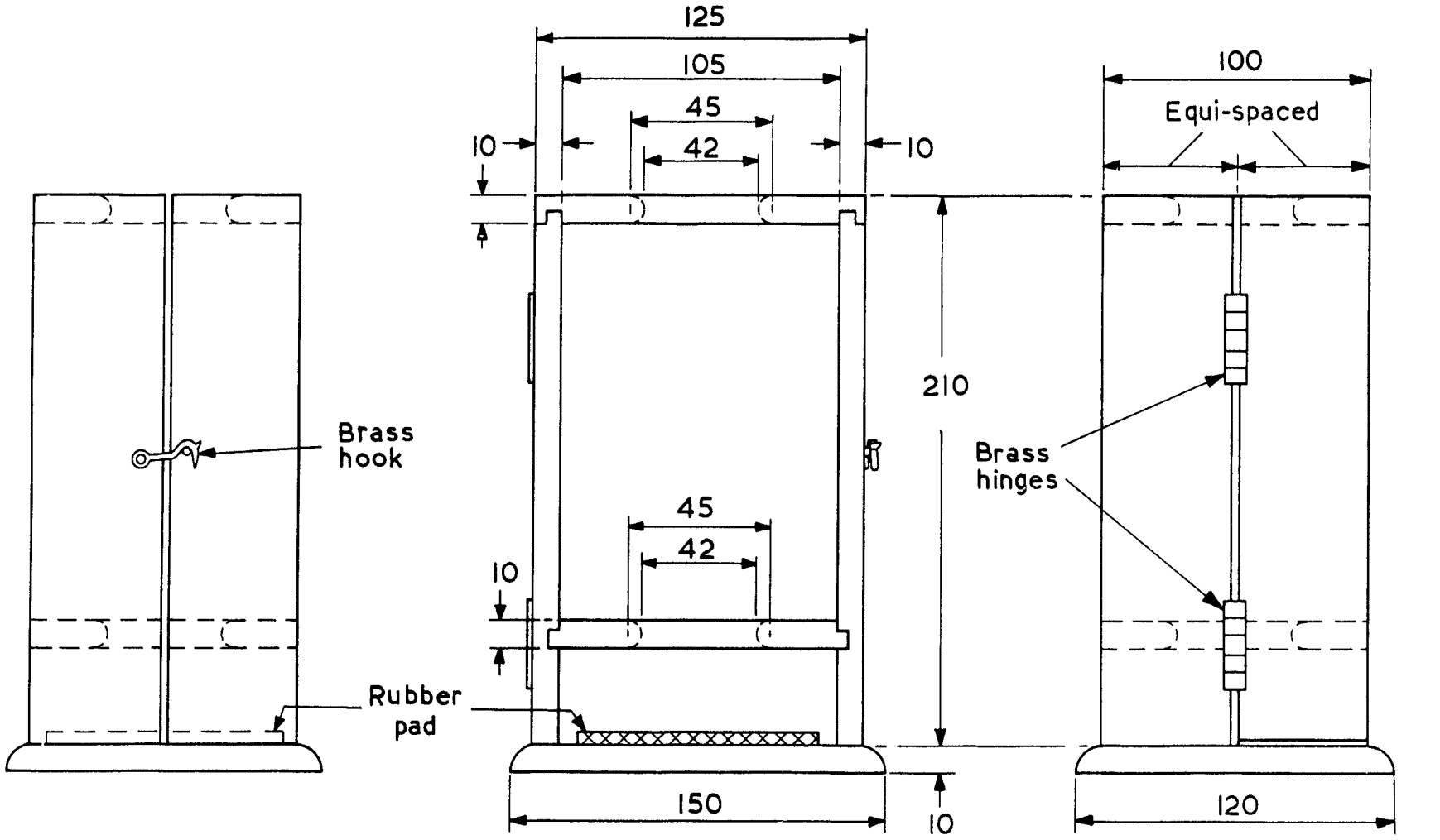
NOTE 1 The standard deviation is a measure of variation. A useful property of the standard deviation is that 95 % of the results are likely to fall within the limits of ± 2 × standard deviation. Thus the 95 % limits of error are represented by ± 2 × standard deviation.

NOTE 2 The numerical values quoted in the above table are based on the volume in millilitres occupied by 40 g of the powder after being subjected to the standard procedure, and not on the bulk densities calculated from these volumes. For convenience the standard deviations have also been given as percentages of the means.

NOTE 3 Each sample of precipitated calcium carbonate was tested on three separate days by all eleven laboratories. The variation within these sets of three repeat results represents the extent to which the laboratories can reproduce their own results on each sample and is measured by the standard deviation within laboratories, i.e., Item ii) of, the above table.

NOTE 4 Item iii) represents the expected reproducibility within laboratories of the mean of three repeat tests on separate days and is derived from Item ii) by dividing the latter by $\sqrt{3}$. This has been done in order to compare the variation between laboratories [i.e. Item iv) of the above table] with the variation within laboratories. Both Item iii) and Item iv) are thus based on the means of three tests.

It is seen from the above table that the bulkier the precipitated calcium carbonate the less reproducible are the results both within and between laboratories. For all three samples, the variation between laboratories is considerably greater than the variation within laboratories.



All dimensions in mm

Figure 1 — Dropping box

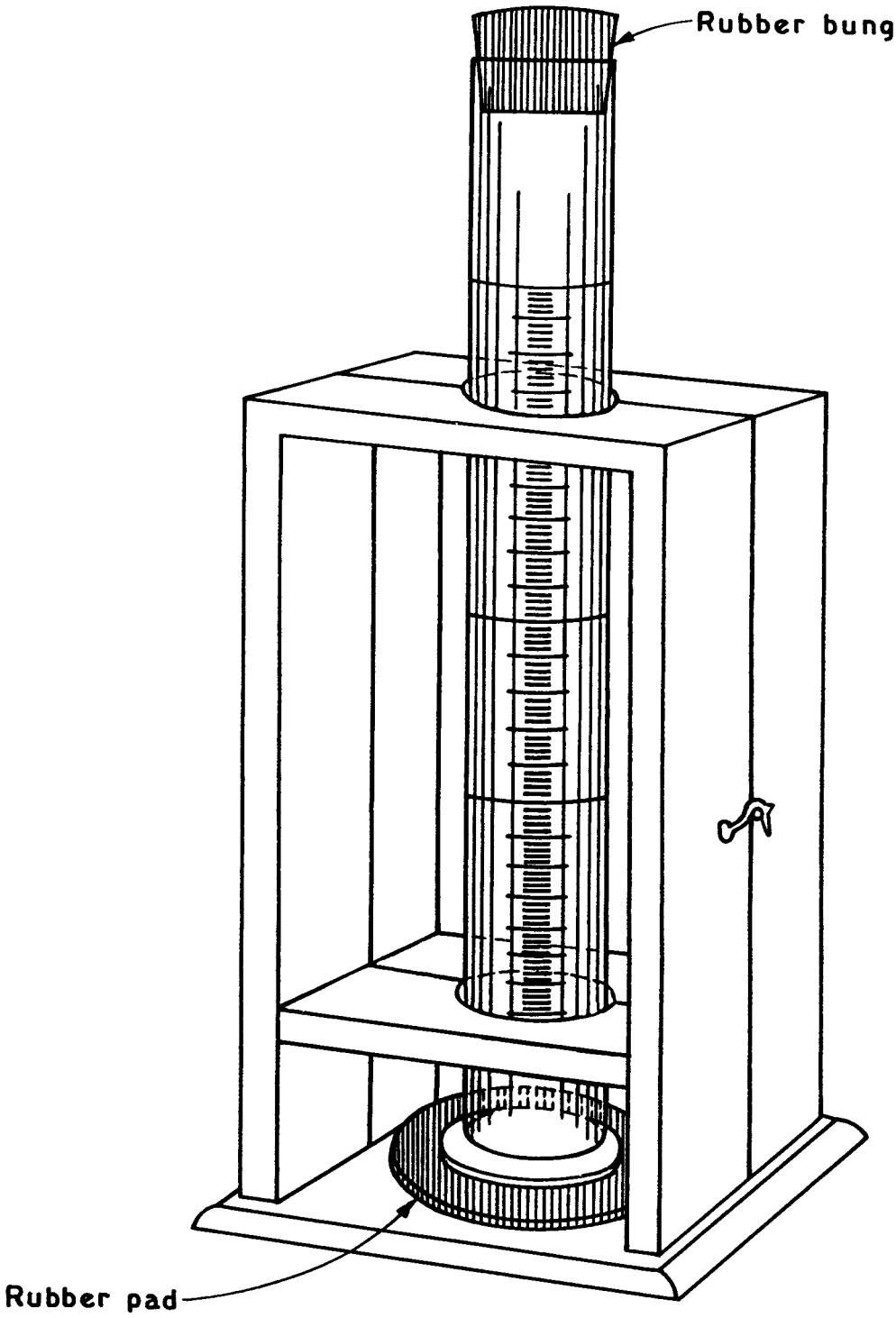


Figure 2 — Apparatus (assembled)

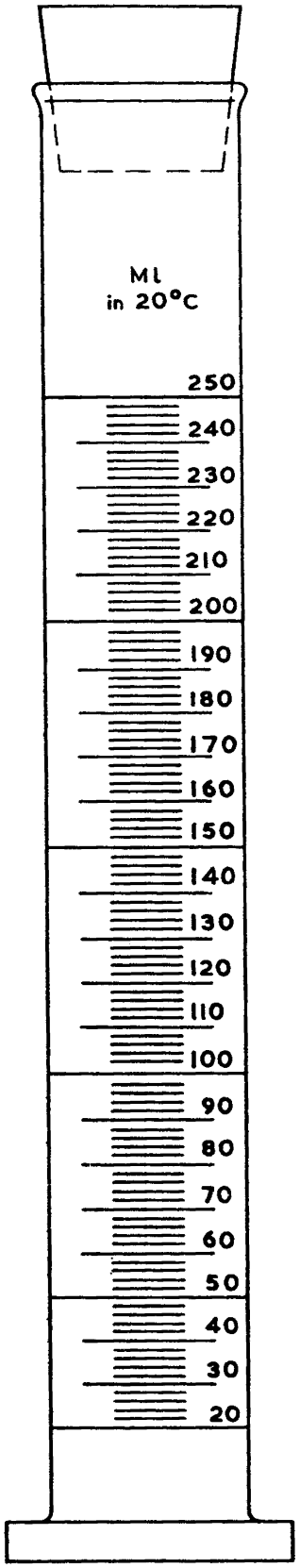


Figure 3 — Cylinder

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London
W4 4AL