BRITISH STANDARD

BS 7591-1: 1992

Porosity and pore size distribution of materials —

Part 1: Method of evaluation by mercury porosimetry

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the General Mechanical Engineering Standards Policy Committee (GME/-) to Technical Committee GME/29, upon which the following bodies were represented:

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Contents

Foreword

This Part of BS 7591 has been prepared under the direction of the General Mechanical Engineering Standards Policy Committee and is one of a series which describe recommended methods for the evaluation of porosity and pore size distribution.

This Part of BS 7591 describes the evaluation of porosity by mercury porosimetry.

Other Parts of BS 7591 are as follows:

— *Part 2: Method of evaluation by gas adsorption*;

— *Part 3: Method of evaluation by challenge test*[1)];

— *Part 4: Method of evaluation by liquid expulsion*[1)].

CAUTION. Care should be taken when handling mercury and providing for the removal of mercury vapour from the vicinity of the porosimeter and from the general laboratory environment (see clause **5**).

The symbols and units used in this Part of BS 7591 do not necessarily conform to BS 5775, but are those widely used in the industry.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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

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.

[1)] In preparation.

Introduction

In general, different types of pores may be pictured as either apertures, channels or cavities within a solid body or as space (i.e. interstices or voids) between solid particles in a bed, compact or aggregate. Porosity is a term which is often used to indicate the porous nature of solid material and is more precisely defined as the ratio of the volume of accessible pores and voids to the total volume occupied by a given amount of the solid (see **3.9**). In addition to the accessible pores, a solid may contain closed pores which are isolated from the external surface and into which fluids are not able to penetrate. The characterization of closed pores, i.e. cavities with no access to an external surface, is not covered in this Part of this standard.

Porous materials may take the form of fine or coarse powders, compacts, extrudates, sheets or monoliths. Their characterization usually involves the determination of the pore size distribution as well as the total pore volume or porosity. For some purposes it is also necessary to study the pore shape and interconnectivity and to determine the internal and external surface area.

Porous materials have great technological importance, for example in the context of the following:

a) controlled drug release;

b) catalysis;

c) gas separation;

d) filtration including sterilization;

e) materials technology;

f) environmental protection and pollution control;

g) natural reservoir rocks.

It is well established that the performance of a porous solid (e.g. its strength, reactivity, permeability or adsorbent power) is dependent on its pore structure. Many different methods have been developed for the characterization of pore structure, in view of the complexity of most porous solids, it is not surprising to find that the results obtained are not always in agreement and that no single technique can be relied upon to provide a complete picture of the pore structure. The choice of the most appropriate method depends on the application of the porous solid, its chemical and physical nature and the range of pore size.

The most commonly used methods are as follows.

a) *Mercury porosimetry* where the pores are filled with mercury under pressure. This method is Suitable for many materials with pores in the approximate diameter range of 0.003 μm to 400 μm, and especially in the range 0.1 μm to 100 μm.

b) *Gas adsorption* where the pores are characterized by adsorbing a gas, such as nitrogen, at liquid nitrogen temperature. This method is used for pores in the approximate diameter range of 0.0004 μm to 0.04 μm (0.4 nm to 40 nm), and is an extension of the surface area estimation technique (see BS 4359-1).

c) *Challenge test* where the effective size of the through-pores in a structure is estimated by the passage of test particles or molecules of different sizes. This method is often used for pores in the approximate diameter range of 0.005 μm to 100 μm.

d) *Liquid expulsion* where the through-pores in a structure are characterised by the pressure required to empty them of a wetting fluid. This method is normally used for pores in the approximate diameter range of 0.05 μm to 50 μm.

1 Scope

This Part of BS 7591 describes a method of evaluation of pore size distribution by mercury porosimetry. It is a comparative test, usually destructive, in which the volume of mercury penetrating a pore or void is determined as a function of an applied hydrostatic pressure, which can be related to a pore diameter.

Practical considerations presently limit the maximum applied absolute pressure to about 400 MPa (60 000 psia)[2)] corresponding to a minimum equivalent pore diameter of approximately 0.003 μm. The maximum diameter will be limited for samples having a significant depth due to the difference in hydrostatic head of mercury from the top to the bottom of the sample. For most purposes this limit may be-regarded as 400 μm.

Inter-particle and intra-particle porosity are determined but the method does not distinguish between these porosities where they co-exist.

[2)] psia = pounds per square inch absolute,
1 lbf/in^2 = 6894 Pa and 1 Pa = 1 N/m^2.

The method is suitable for the study of most porous materials. Samples that amalgamate with mercury, such as certain metals, may be unsuitable for this technique or may require a preliminary passivation. Other materials may deform or compact under the applied pressure. In some cases it may be possible to apply sample compressibility corrections and useful comparative data may still be obtained. (See **7.2.7**)

The mercury porosimetry technique should be considered to be comparative, as for most porous media a theory is not available to allow an absolute calculation of results of pore size distribution.

NOTE The determination of pore size distribution of refractory materials by mercury porosimetry is described in BS 1902-3.16.

2 References

2.1 Normative references

This Part of BS 7591 incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this Part of BS 7591 only when incorporated in it by updating or revision.

2.2 Informative references

This Part of BS 7591 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Definitions (see also Figure 1)

For the purposes of this Part of BS 7591, the following definitions apply.

3.1
blind pore (dead-end pore)

an open pore having a single connection with an external surface

3.2
ink bottle pore

a narrow necked open pore

3.3
interconnected pore

a pore which communicates with one or more other pores

3.4
open pore

a cavity or channel with access to an external surface

3.5
pore diameter

the diameter of a pore, assumed to be cylindrical, just penetrated by mercury at a given pressure as calculated by the Washburn equation (see clause **8**)

3.6
porosimeter

an instrument for measuring porosity and pore size distribution

3.7
porosimetry

methods for the estimation of porosity and pore size distribution

3.8
open porosity

the ratio of the volume of open pores and voids, which are accessible using the method employed, to the total volume occupied by a given amount of solid

3.9
total porosity

the ratio of the volume of voids plus the volume of open and closed pores to the total volume occupied by a given amount of porous solid

3.10
right cylindrical pore

a cylindrical pore perpendicular to the surface

3.11
through pore

a pore which passes all the way through the sample

3.12
void

the space between particles in a bed,
i.e. inter-particle pore

3.13
pore size distribution

the distribution of pore width in a porous body as calculated by application of the Washburn equation (see clause **8**) which assumes a cylindrical pore model

4 Principles

The pore size distribution of a porous solid can be determined by forcing mercury into an evacuated sample under increasing pressure and measuring the volume of mercury intruded as a function of pressure. The determination may proceed either with the pressure being raised in a step-wise manner and the volume of mercury intruded measured after an interval of time (equilibration time) when stability has been achieved, or by raising the pressure in a continuous (progressive) manner.

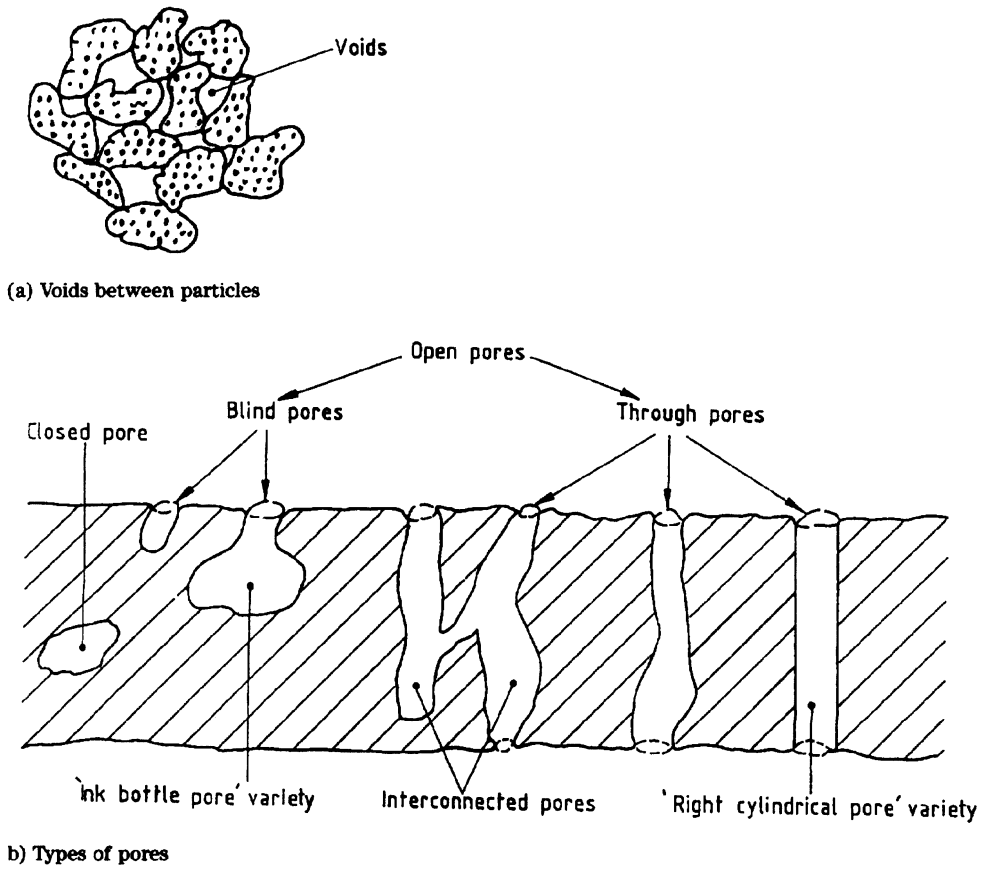

Figure 1 — Types of pore space

The technique of mercury porosimetry is based on the relationship between pore size and applied pressure as described in clause **8**. There are several assumptions inherent in the calculations and the values of contact angle and surface tension used in the calculation should be stated in the test report (see clause **9**).

5 Apparatus and material

WARNING NOTE. It is important that proper precautions for the protection of laboratory personnel are taken when mercury is used. Samples and penetrometers are contaminated with mercury and hydraulic fluid after testing. Attention is drawn to the relevant regulations and guidance documents which include the Department of Employment Technical Data Note No. 21, published by HMSO[1].

5.1 *Sample holder*

The sample holder, referred to as a penetrometer or dilatometer, should have a uniform bore capillary tube through which the sample can be evacuated and through which mercury can enter. The capillary tube is attached to a wider bore tube in which the test sample is located. If precise measurements are required the internal volume of the capillary tube should be between 20 % and 90 % of the expected pore and void volume of the sample. Since different materials exhibit a wide range of open porosities a number of penetrometers with different diameter capillary tubes and sample volumes may be required. A special design of sample holder is often used with powdered samples to avoid the loss of powder (see **7.2.3**).

5.2 *Porosimeter*

For convenience of operation the test may be carried out as two sequential measurements, a low pressure test up to 0.2 MPa (30 psia) and a high pressure test up to the maximum operating pressure of the porosimeter. The porosimeter may have separate ports for high and low pressure operation, or the low pressure test may be carried out on a separate unit.

Prior to any porosimetry measurement it is necessary to evacuate the sample to a maximum residual pressure of 7 Pa (0.05 torr)[3)], then fill the sample holder with mercury to a given low pressure. To cause intrusion of mercury there should be a means of generating pressure. It is necessary to use a hydraulic pump to generate high pressures of up to 400 MPa (60 000 psia). It is possible to use compressed gases to generate lower pressures.

A means to reduce and verify the reduction of pressure to atmospheric is necessary.

Several pressure transducers are required to cover the total pressure range.

A means of detecting the change in the volume of mercury intruded to a resolution of 1 mm^3 or less is desirable. This is usually done by measuring the change in capacitance between the mercury column in the capillary tube and a metal sleeve around the outside of the penetrometer.

5.3 *Mercury purity*

The mercury used in the porosimeter should be of analytical quality (at least 99.99 % (*m*/*m*) purity).

6 Procedures for calibration and performance

6.1 General

Sample preparation and the filling of the penetrometer with mercury require vacuum, the level of which is usually recorded using a transducer. For the porosity evaluation, two signals are required to be measured in a porosimeter; the applied pressure and the corresponding volume change of mercury as it fills pores in the sample. The volume of mercury displaced from a precision glass capillary tube is most commonly determined as a function of electrical capacitance change.

6.2 Pressure signal calibration

Pressure is usually measured with electronic pressure transducers which will have been factory calibrated. The accuracy of the pressure measurement should be within ± 1 % of the full scale transducer reading or ± 2 % of the actual reading, whichever is the lower.

NOTE It is recommended that verification of calibration should be performed every six months by an organization specializing in this type of work.

6.3 Volume signal calibration

The accuracy of the volume measurement should be within ± 1 % of the total volume to be measured.

NOTE It is recommended that verification of calibration should be performed every six months by an organization specializing in this type of work.

6.4 Vacuum transducer calibration

The accuracy of the indicated vacuum is generally not critical. The vacuum manifold system, without a sample, should be capable of achieving at least 3 Pa (approximately 10^{-2} torr), and if possible it should be calibrated to within 1 Pa at this level.

6.5 Verification of porosimeter performance

It is recommended that a stable reference material selected by the user should be tested on a regular basis to monitor instrument calibration and performance.

7 Procedure

7.1 Sampling

NOTE 1 The sample for test should be representative of the bulk material and should be of an appropriate quantity (see **7.1.2**). Particular precautions should be taken when its properties are directionally orientated (see **7.1.1**).

NOTE 2 It is recommended that a second sample is taken and held in reserve in case a repeat determination is necessary.

7.1.1 *Obtaining a test sample*

Since the material from which the sample for test is taken may be in a variety of forms, different sub-sampling methods are appropriate as follows.

a) *From a block*

Several pieces about 1 cm^3 may be taken in order to represent different zones from within the block.

NOTE 1 Test samples greater than 1 cm^3 may require an increase in analysis time.

The pieces may be cut with a saw or core drill, but there is a possibility that saw marks can be interpreted as pores. If coarse pores are of particular interest, polish the surfaces of the pieces with a medium of 10 μm maximum particle size. If fine pores are of particular interest, test the sample in the as-sawn condition and ignore data from pore diameters greater than 125 μm.

Polished test pieces should be washed to remove adhering particles, which can affect the sample mass and block its pores. The sample should be dried to constant mass.

[3)] 1 torr = 133.322 Pa.

NOTE 2 For materials subject to hydration, wash with a non-aqueous liquid. For materials affected by heat, use an alternative drying procedure.

b) *From a powder*

Powdery or granular material samples which are free-flowing should be subdivided by rotary sampling or chute riffling. Non-free-flowing powders may be sampled by coning and quartering. Suitable apparatus and procedures are given in BS 3406-1:1986.

To help to distinguish between inter- and intra-particle pores, it may be beneficial to sieve the sample to a particle size range which allows a clearer distinction between the two, but it is important to establish that this does not make the sampling un-representative.

c) *From a film or sheet*

Film and sheet material may be sampled by either cutting a strip, or by stamping discs, to fit the appropriate penetrometer.

NOTE Difficulties in testing material in this form may arise due to proximity between adjacent faces. This can be overcome by interposing pieces of woven steel wire gauze to keep discs separate, or by rolling a strip of gauze together with the sample strip.

7.1.2 *Quantity of sample*

The quantity of test sample required is dependent upon its nature. The largest possible sample size commensurate with the size of cell should be taken. However, the total pore volume should lie within the recommended measuring range of the capillary tube and the apparatus. In the case of unknown specimens, a preliminary test will usually be necessary to ascertain the optimum quantity of test sample. The test sample is placed preferably in a penetrometer having a bulk sample volume of between 3 cm^3 and 15 cm^3, although larger cells may be used.

7.2 Method

7.2.1 *Sample pretreatment*

The sample should be pretreated to remove adsorbed material which can obscure its accessible porosity: this includes adsorbed water, and other materials such as organic molecules used in the manufacture or operation of the porous solid.

NOTE In order to optimize pretreatment, it is advisable to study the thermal behaviour of the material by, e.g. thermogravimetric analysis and differential scanning calorimetry, to determine the temperatures at which materials are evolved from the sample, together with any phase changes which could affect the history of the sample. In many cases, heating to 110 °C in a vacuum oven at 3 Pa (10^{-2} torr) for 4 h will be suitable.

When a satisfactory pretreatment regime has been established, the sample can be out-gassed by heating and/or evacuation, or by flowing inert gas. If the sample is in a form which allows amalgamation with, or wetting by, mercury it may be possible to passivate the surface e.g. by producing a thin layer of oxide, or by coating with stearate.

Care should be taken to ensure that the treatment does not affect the porous nature of the sample. The mass of the test sample to be used should be recorded.

7.2.2 *Filling of sample cells*

After sample pretreatment, the sample should be transferred to a clean and dry penetrometer. To minimize recontamination by, for example, readsorption of water vapour, it may be prudent to effect the transfer in a purged glove box, and to dose the penetrometer with nitrogen for final transfer to the porosimeter.

7.2.3 *Evacuation*

The object of sample evacuation is to remove the majority of vapours and gases from the sample, prior to filling the penetrometer with mercury. Fine powders with relatively high surface areas may tend to fluidize under vacuum with loss of sample into the vacuum system. This effect may be avoided by selection of penetrometers designed specifically for powders, and by controlling the rate of evacuation.

A vacuum approximating to 15 Pa (5×10^{-2} torr) is generally desirable. The evacuation time is considerably reduced for pre-dried samples. The achieved vacuum should be reported.

7.2.4 *Filling the penetrometer with mercury*

The vacuum of **7.2.3** is also required to ensure the transfer of mercury from its reservoir to the penetrometer. It is advantageous if the mercury is de-aerated during filling, thus maintaining the sample vacuum and avoiding air-bubble entrapment.

If possible the mercury should not fall directly onto the sample during the filling process. This is particularly important if large pores are to be measured. The hydrostatic pressure of the mercury over the sample may be minimized by filling the penetrometer in the horizontal position; a typical filling pressure would then be about 5 kPa (approx. 1 psia).

In the case of a penetrometer filled in the vertical position, the filling pressure comprises any applied pressure plus the pressure contribution, in Pa, created by the vertical head of mercury, in mm, contacting the sample.

NOTE 1 mm Hg head of pressure = 133.3 Pa.

Mercury in excess of the requirement to fill the penetrometer is re-directed to the mercury reservoir. The fill pressure should be reported.

7.2.5 ***Measurement***

7.2.5.1 *Low pressure*

Admit air or nitrogen in a controlled manner to increase the pressure either in stages corresponding to particular pore sizes of interest, or continuously at a slow rate. The pressure and volume intruded should be recorded either at each pressure stage, or for continuous increase in pressure at points sufficiently close together to give the required resolution in the pore size distribution. Data can be recorded either graphically or via a suitable computer. When the maximum required pressure has been reached, reduce the pressure to ambient and transfer the penetrometer to the high pressure port or unit.

7.2.5.2 *High pressure*

Increase the pressure in the system until it is the same as the highest pressure measured in the low pressure port. Record the intrusion volume at this pressure as the initial reading from which subsequent intrusion volumes are calculated. Increase the pressure either in stages or continuously, up to the maximum required pressure. If the pressure is continuously increased, care should be taken to ensure that the intrusion curve is independent of the rate of pressure increase. Record the pressure and intruded volume as described in **7.2.5.1**. If required, the pressure may then be decreased either in stages or continuously to determine the mercury extrusion curve.

7.2.6 ***Completion of test***

Before finally removing the penetrometer from the porosimeter ensure that the pressure in the apparatus has been returned to ambient. A visual check to ascertain that the mercury has penetrated the mass of the sample is advisable.

7.2.7 ***Blank and sample compression correction***

7.2.7.1 *General*

The mercury, the sample and its cell, and other components of the volume detector system are compressed to different degrees under elevated pressures. Compressibility corrections may be justified where the porosity is low, the sample is relatively compressible, or where high precision is required.

7.2.7.2 *Measurement of correction*

A test in accordance with clause **6** is carried out without using a test sample, or preferably using a control sample which is non-porous but of similar size and heat capacity as the test sample. The test is made under exactly the same conditions as are employed for the actual test sample.

7.2.7.3 *Applying the correction*

The result of the test described in **7.2.7.2** is a series of apparent volume changes. Apparent intrusions are to be subtracted from the measured intrusions on the test sample. Apparent extrusions are to be added to the measured intrusions on the test sample.

8 Presentation of results (see Table 1)

The following calculations show how pore diameter D (in μm) is calculated from the Washburn equation.

$$D = (-4\gamma \cos\theta)/P$$

e.g: $$D = \frac{180}{P} \text{ (approximately)}$$

where

θ is the contact angle of 130°;

γ is the surface tension of 485×10^{-3} N/m;

P is the corrected pressure for hydrostatic mercury head in sample penetrometer expressed in psia.

NOTE Mercury density is 13.5335 g/ml and sample mass is 0.3340 g.

9 Test report

A summary of the measurement conditions and constants used in the calculation should be provided with each result as follows.

a) analysis laboratory;

b) sample identification;

c) sample source;

d) sample mass;

e) instrument used;

f) pretreatment conditions;

g) evacuation pressure;

h) fill pressure;

i) equilibration time or rate of change;

j) corrected pressure and corrected volume intruded at each pressure stage;

k) penetrometer calibration constant;

l) contact angle used;

m) surface tension value used;

n) operator's name;

p) date of analysis.

The results should be reported at least as specific cumulative volume against corrected pressure. Other formats are possible, for example see Figure 3 and Figure 4.

Table 1 — Example of part of a computation of pore size distribution

Measured pressure	Corrected pressure	Pore diameter	Measured cumulative volume	Corrected cumulative volume	Specific cumulative volume	Mean pore diameter	Differential volume	Logarithmic differential volume
P' psia	P[a] psia	D µm	V'' ml	V[b] ml	V ml/g	D' µm	dV/dD' ml/g · µm	$dV/d\log D$ ml/g
1 000.0	1 003.5	0.1802	0.1065	0.1045	0.3128			
1 250.0	1 253.4	0.1443	0.1165	0.1144	0.3425	0.1623	0.826	0.307
1 500.0	1 503.3	0.1203	0.1245	0.1221	0.3656	0.1323	0.961	0.292
2 000.0	2 003.1	0.0903	0.1390	0.1365	0.4087	0.1053	1.436	0.346
2 500.0	2 503.0	0.0723	0.1516	0.1489	0.4458	0.0813	2.059	0.384
3 000.0	3 003.0	0.0602	0.1604	0.1576	0.4719	0.0662	2.165	0.329
4 000.0	4 003.0	0.0452	0.1763	0.1733	0.5189	0.0527	3.124	0.377
5 000.0	5 003.0	0.0362	0.1882	0.1850	0.5539	0.0407	3.879	0.362
6 000.0	6 002.0	0.0301	0.2132	0.2099	0.6287	0.0331	12.440	0.947
7 000.0	7 001.0	0.0258	0.3229	0.3195	0.9566	0.0280	76.240	4.903
8 200.0	8 201.0	0.0221	0.3528	0.3488	1.0443	0.0239	23.210	1.277
8 605.0	8 606.0	0.0210	0.3588	0.3545	1.0614	0.0215	16.440	0.815
9 028.0	9 029.0	0.0200	0.3632	0.3585	1.0734	0.0205	12.160	0.575

[a] Corrected pressure for hydrostatic mercury head in sample penetrometer (see **7.2.4**).
[b] Intended volume corrected for compression of mercury, penetrometer and sample, V'(ml).

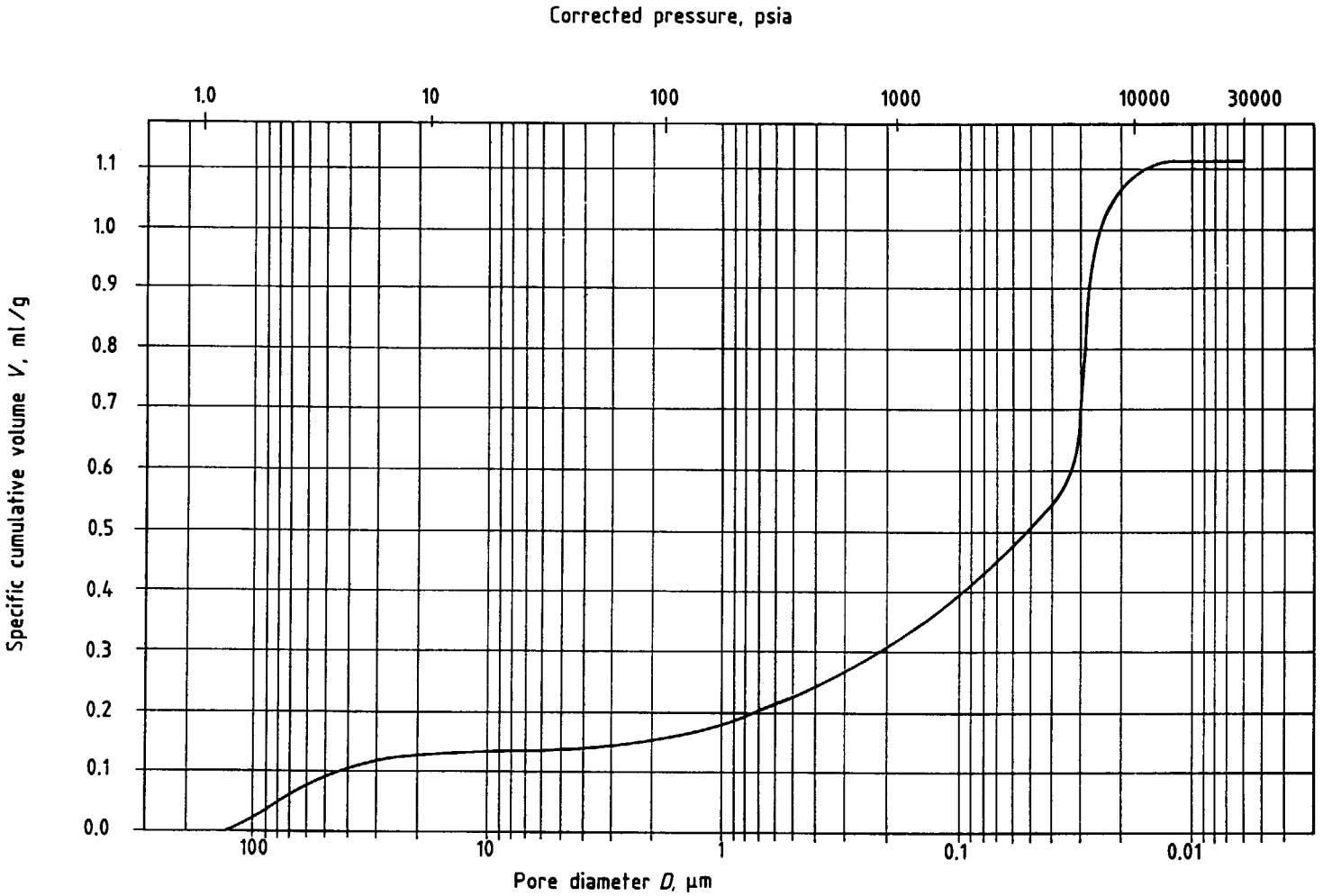

Figure 2 — Example of cumulative pore size distribution

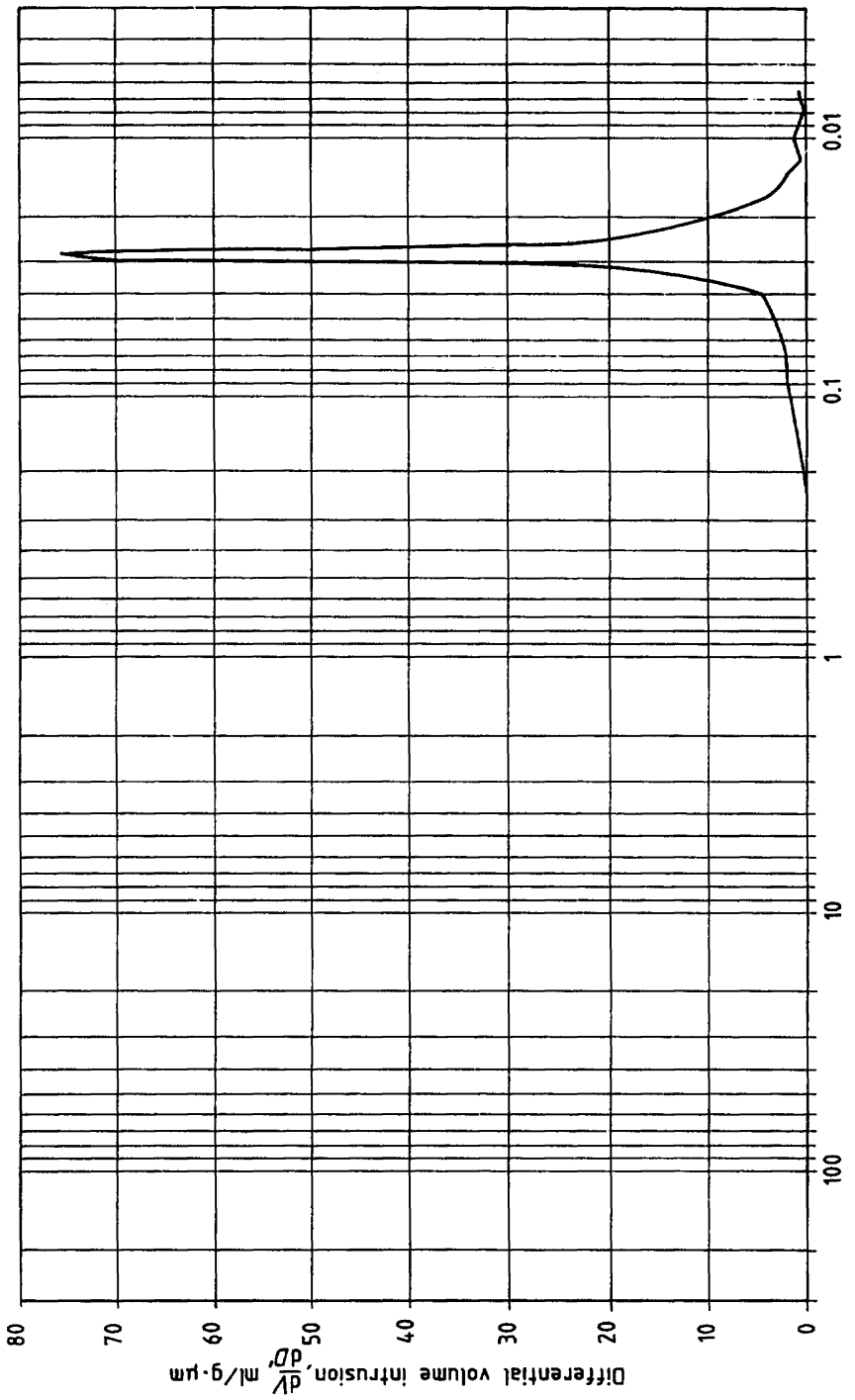

Figure 3 — Example of differential pore size distribution on a linear diameter basis

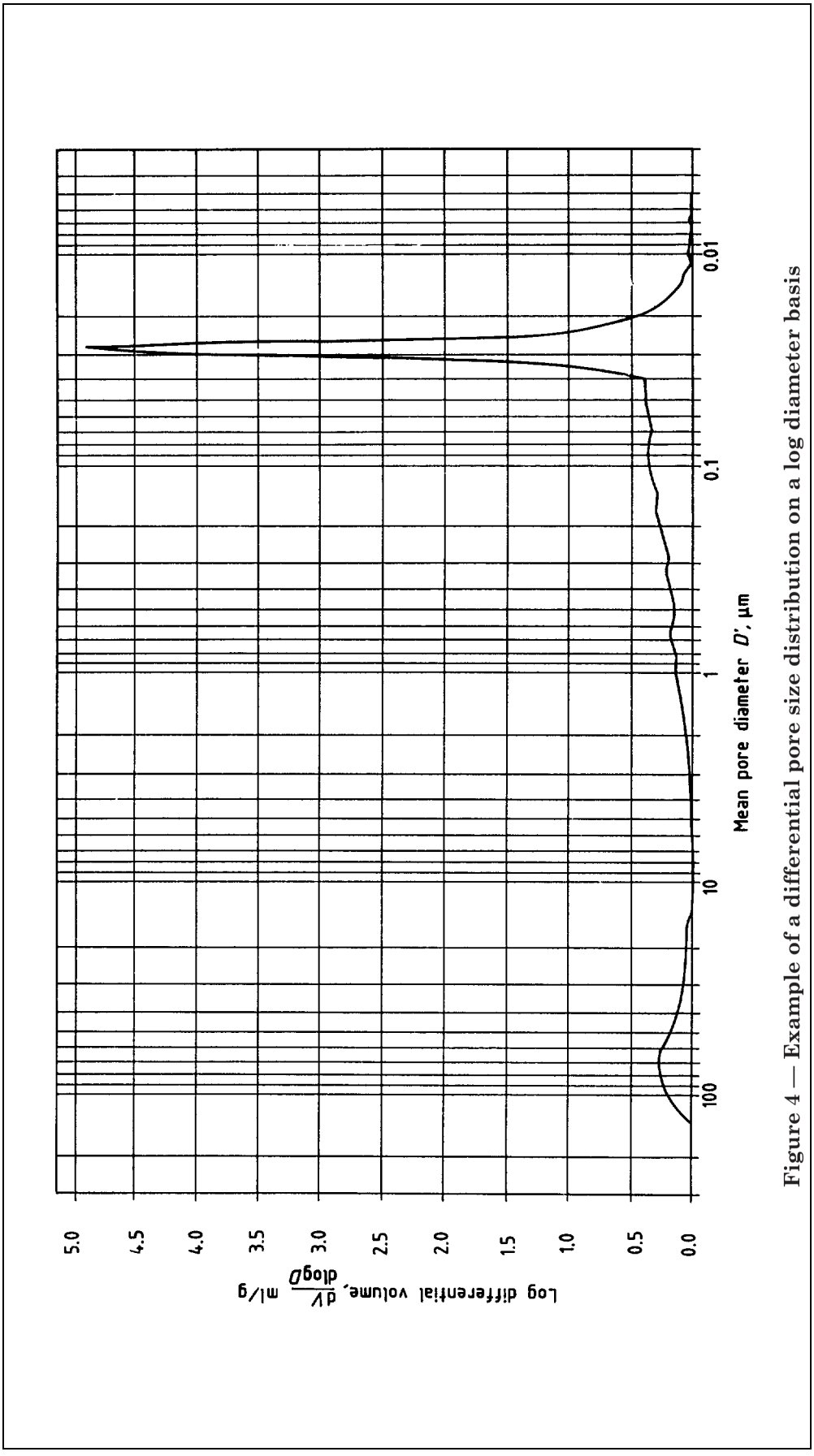

Figure 4 — Example of a differential pore size distribution on a log diameter basis

List of references (see clause 2)

Normative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 3406, *Methods for determination of particle size distribution.*
BS 3406-1:1986, *Guide to powder sampling.*
BS 4359, *Determination of the specific surface area of powders.*
BS 4359-1:1984, *Recommendations for gas adsorption (BET) methods.*

Informative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 1902, *Methods of testing refractory materials.*
BS 1902-3, *General and textural properties.*
BS 1902-3.16:1990, *Determination of pore size distribution (method 1902–316).*
BS 5775, *Specification for quantities, units and symbols.*

Other references

[1] Department of Employment Technical Data Note No. 21. HMSO.

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