

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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- British Cement Association
- British Coal Corporation
- British Laboratory Ware Association
- Coated Abrasives Manufacturers' Association
- Guild of Metal Perforators
- Institution of Chemical Engineers
- Institution of Mining and Metallurgy
- Mechanical Handling Engineers Association
- Ministry of Defence
- NABIM
- Society of Chemical Industry
- Society of Cosmetic Scientists
- Woven Wire Association

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- Aluminium Federation
- British Laboratory Ware Association
- China Clay Association
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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 test¹⁾*;
- *Part 4: Method of evaluation by liquid expulsion¹⁾*.

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.

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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.

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/in² = 6894 Pa and 1 Pa = 1 N/m².