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## Evaluation of pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption —

### Part 1: Mercury porosimetry

*Distribution de taille des pores et la porosité des matériaux solides par porosimétrie à mercure et l'adsorption des gaz —*

*Partie 1: Porosimétrie à mercure*

[Revision of first edition (ISO 15901-1:2005) and ISO 15901-1:2005/Cor 1:2007]

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15901-1 was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

ISO 15901 consists of the following parts, under the general title *Evaluation of pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption*:

- *Part 1: Mercury porosimetry*
- *Part 2: Analysis of mesopores and macro-pores by gas adsorption*
- *Part 3: Analysis of micropores by gas adsorption*

## Introduction

In general, different pores (micro-, meso-, and macropores) 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 the accessible pores and voids to the total volume occupied by an amount of the solid. 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 is not covered in 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 specific surface area.

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

- controlled drug release,
- catalysis,
- gas separation,
- filtration including sterilization,
- materials technology,
- environmental protection and pollution control,
- natural reservoir rocks,
- building materials properties,
- polymers and ceramic.

It is well established that the performance of a porous solid (e.g. its strength, reactivity, permeability) 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 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 appropriate diameter range of 0,004  $\mu\text{m}$  to 400  $\mu\text{m}$ .
- b) meso- and macropore analysis by gas adsorption, where the pores are characterized by adsorbing a gas, such as nitrogen, at liquid nitrogen temperature. The method is used for pores in the approximate diameter range of 0,002  $\mu\text{m}$  to 0,1  $\mu\text{m}$  (2 nm to 100 nm).
- c) micropore analysis by gas adsorption, where the pores are characterized by adsorbing a gas, such as nitrogen, at liquid nitrogen temperature. The method is used for pores in the approximate diameter range of 0,4 nm to 2 nm.

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# Evaluation of pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption — Part 1: Mercury porosimetry

**WARNING** — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

## 1 Scope

This international standard describes a method for the evaluation of the pore size distribution and the specific surface area of pores in solids by mercury porosimetry according to the method of Ritter and Drake [1] [2]. It is a comparative test, usually destructive due to mercury contamination, 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) corresponding to a minimum equivalent pore diameter of approximately 4 nm. 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 the most purposes, this limit can be regarded as 400  $\mu\text{m}$ . The measurements cover inter-particle and intra-particle porosity. In general, without additional information from other methods it is difficult to distinguish between these porosities where they co-exist. The method is suitable for the study of most porous materials non-wettable by mercury. Samples that amalgamate with mercury, such as certain metals, e.g. gold, aluminium, reduced copper, reduced nickel and silver, can be unsuitable for this technique or can require a preliminary passivation. Under the applied pressure some materials are deformed, compacted or destroyed, whereby open pores may be collapsed and closed pores opened. In some cases it may be possible to apply sample compressibility corrections and useful comparative data may still be obtainable. For these reasons, the mercury porosimetry technique is considered to be comparative.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14488, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

JIS K 8572, *Mercury*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

- 3.1 porosimeter**  
instrument for measuring pore volume and pore size distribution
- 3.2 porosimetry**  
methods for the estimation of pore volume, pore size distribution, and porosity
- 3.3 porous solid**  
solid with cavities or channels which are deeper than they are wide
- 3.4 powder**  
porous or nonporous solid composed of discrete particles with maximum dimension less than about 1 mm, powders with a particle size below about 1 µm are often referred to as fine powders
- 3.5 pore**  
pores in solid materials are cavities or channels which are deeper than they are wide otherwise they are part of material's roughness
- 3.6 void/interstice**  
space between particles, i.e. interparticle pore
- 3.7 macropore**  
pore of internal width greater than 50 nm
- 3.8 mesopore**  
pore of internal width between 2 nm and 50 nm
- 3.9 micropore**  
pore of internal width less than 2 nm
- 3.10 closed pore**  
a pore totally enclosed by its walls and hence not interconnecting with other pores and not accessible to fluids
- Note 1 to entry: Pores with apertures smaller than approx. 4 nm are not accessible to mercury porosimetry, and therefore considered closed pores in this standard.
- 3.11 open pore**  
a pore not totally enclosed by its walls and open to the surface either directly or by interconnecting with other pores and therefore accessible to fluid
- 3.12 ink bottle pore**  
narrow necked open pore



**3.13****pore size**

internal pore width (for example the diameter of a cylindrical pore or the distance between the opposite walls of a slit) which is a representative value of various sizes of vacant space inside a porous material

**3.14****pore volume**

volume of open pores unless otherwise stated

**3.15****pore diameter**

diameter of a pore in a model in which the pores typically are assumed to be cylindrical in shape and which is calculated from data obtained by a specified procedure

**3.16****median pore diameter**

diameter that corresponds to the 50th percentile of pore volume, i.e. the diameter for which one half of the pore volume is found to be in larger pores and one half is found to be in smaller pores

**3.17****modal pore diameter (mode)**

pore diameter of the maximum in a pore size distribution curve

**3.18****hydraulic pore diameter**

average pore diameter, calculated as the ratio of pore volume multiplied by four to pore area

**3.19****bulk volume**

volume of powder or solids, including all pores (open and closed) and interstitial spaces between particles

Note 1 to entry: For mercury porosimetry: volume of the sample plus pores not filled by mercury at the applied pressure of interest, generally that at the start of the analysis or filling of the sample holder with mercury.

**3.20****bulk density**

ratio of sample mass to bulk volume

**3.21****skeleton volume**

volume of the sample including the volume of closed pores (if present) but excluding the volumes of open pores as well as that of void spaces between particles within the bulk sample (ISO 12154)

Note 1 to entry: Open pores with apertures smaller than approx. 4 nm are not accessible to mercury porosimetry, and therefore if such pores are present, a skeleton volume cannot be determined but rather an apparent volume is assessed.

**3.22****skeleton density**

ratio of sample mass to skeleton volume

**3.23****apparent volume**

total volume of the solid constituents of the sample including closed pores and pores inaccessible or not detectable by the stated method

Note 1 to entry: In mercury porosimetry the accessibility of pores is a function of applied pressure.

**3.24**

**apparent density**

mass of a material divided by the apparent volume

**3.25**

**envelope volume**

total volume of the particle, including closed and open pores, but excluding void space between the individual particles

**3.26**

**envelope density**

ratio of the mass of a particle to the envelope volume of the particle

**3.27**

**porosity**

ratio of the volume of the accessible pores and voids to the bulk volume occupied by an amount of the solid

**3.28**

**interparticle porosity**

ratio of the volume of void space between the individual particles to the bulk volume of the particles or powder

**3.29**

**intraparticle porosity**

ratio of the volume of open pores inside the individual particles of a particulate or divided solid sample to the bulk volume occupied by the sample

**3.30**

**surface area**

extent of available surface area as determined by given method under stated conditions

**3.31**

**surface tension**

work required to increase a surface area divided by that area

**3.32**

**contact angle**

angle at which a liquid/vapour interface meets the surface of a solid material