INTERNATIONAL STANDARD

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

Part 1: Mercury porosimetry

Evaluation de la 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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

This second edition cancels and replaces the first edition (ISO 15901-1:2005), which has been technically revised. It also incorporates the Corrigendum ISO 15901-1:2005/Cor 1:2007.

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 macropores 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 in this International Standard is more precisely defined as the ratio of the total pore volume of the accessible pores and voids to the volume of the particulate agglomerate. 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 International 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 accessible 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;

— 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 approximate diameter range of 0,004 µm to 400 µm.

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 µm to 0,1 µm (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.

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 psi) corresponding to a minimum equivalent pore diameter of approximately 4 nm. The maximum diameter is 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 µm. 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, copper, nickel and silver, can be unsuitable with 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*

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

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

