SLOVENSKI STANDARD

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Ocena porazdelitve velikosti por in poroznosti materialov z živosrebrovo porozometrijo in plinsko adsorpcijo - 1. del: Živosrebrova porozometrija

Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption - Part 1: Mercury porosimetry

Distribution des dimensions des pores et porosité des matériaux solides par porosimétrie au mercure et par adsorption de gaz - Partie 1: Porosimétrie au mercure

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INTERNATIONAL STANDARD

ISO 15901-1

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

Part 1:
Mercury porosimetry

Distribution des dimensions des pores et porosimétrie des matériaux solides par porosimétrie au mercure et par adsorption de gaz —

Partie 1: Porosimétrie au mercure

Reference number
ISO 15901-1:2005(E)

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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, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 15901 consists of the following parts, under the general title *Evaluation of pore size distribution and porosimetry 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) can 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 a given amount of the solid. In addition to the accessible pores, a solid can 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 can 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 of 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 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 of 0,003 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 (2,0 nm to 100 nm), and is an extension of the surface area estimation technique;

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 2,0 nm, and is an extension of the surface area estimation technique.

Evaluation of pore size distribution and porosimetry 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 in pores of 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 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 the most purposes, this limit can be regarded as 400 µm. The measurements cover interparticle and intraparticle porosity. In general, it cannot distinguish between these porosities where they co-exist.

The method is suitable for the study of most non-wettable, by mercury, porous materials. 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 can be collapsed and closed pores opened. In some cases, it is possible to apply sample compressibility corrections and useful comparative data can still be obtained. 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 3165, *Sampling of chemical products for industrial use — Safety in sampling*

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

M 024 4/85, Quecksilber und seine Verbindungen. Merkblatt der Berufsgenossenschaft der chemischen Industrie, Postfach 101480, D-69004 Heidelberg, Germany

3 Terms and definitions

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

3.1
bulk density
powder density under defined conditions

3.2
blind pore
dead-end-pore
open pore having a single connection with an external surface

3.3
closed pore
cavity not connected to the external surface

NOTE Closed pores are not covered in this International standard.

3.4
contact angle
angle that a non-wetting liquid makes with a solid material

3.5
external surface area
area of external surface including roughness but outside pores

3.6
ink bottle pore
narrow necked open pore

3.7
interconnected pore
pore which communicates with one or more other pores

3.8
internal surface area
area of internal pore walls

3.9
intraparticle porosity
ratio of the volume of open pores internal to the particle to the total volume occupied by the solid

3.10
interparticle porosity
ratio of the volume of space between particles in a powder to the apparent volume of the particles or powder

3.11
macropore
pore of internal width greater than 50 nm

3.12
mesopore
pore of internal width between 2 nm and 50 nm

3.13
micropore
pore of internal width less than 2 nm which is accessible for a molecule to be adsorbed

3.14
open pore
cavity or channel with access to an external surface

3.15
open porosity
ratio of the volume of open pores and voids to the total volume occupied by the solid

3.16
pore size
pore width (for example, the diameter of a cylindrical pore or the distance between the opposite walls of a slit) that is a representative value of various sizes of the vacant space inside a porous material

NOTE One of the methods to determine pore sizes is by mercury porosimetry.

3.17
pore volume
volume of pores determined by stated method

3.18
porosimeter
instrument for measuring porosity and pore size distribution

3.19
porosimetry
methods for the estimation of porosity and pore size distribution

3.20
porosity
ratio of total pore volume to apparent volume of particle or powder

3.21
porous solid
solid with cavities or channels which are deeper than they are wide

3.22
skeletal density
mass of a powder divided by the total volume of the sample, including closed pores but excluding open pores

3.23
apparent density
mass of a powder divided by the total volume of the sample, including closed and inaccessible pores, as determined by the stated method

3.24
powder density
mass of a powder divided by its apparent volume, which is taken to be the total volume of the solid material, open and closed pores and interstices

3.25
surface area
extent of available surface area as determined by given method under stated conditions

3.26
surface tension
force required to separate a film of liquid from either a solid material or a film of the same liquid

