INTERNATIONAL STANDARD

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

Part 2:
Analysis of mesopores and macropores by gas adsorption

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

Partie 2: Analyse des mésopores et des macropores par adsorption de gaz

Reference number
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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.

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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 15901-2 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 *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

Generally speaking, different types of pores can be pictured as apertures, channels or cavities within a solid body, or as the space (i.e. an interstice or a void) 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. 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 (i.e. cavities with no access to an external surface) is not covered in this part of ISO 15901.

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 surface areas.

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;

h) building material properties;

i) polymer and ceramic industries.

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 do not always concur, 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.

Commonly used methods are as follows.

— **Mercury porosimetry**, where the pores are filled with mercury under pressure. This method is suitable for many materials with pores in the approximate diameter rang of 0,003 µm to 400 µm, and especially in the range of 0,1 µm to 100 µm.

— **Mesopore and macropore analysis by 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 0,002 µm to 0,1 µm (2 nm to 100 nm), and is an extension of the surface area estimation technique (see ISO 9277). (Discussion of other pore size distribution analysis techniques can be found in Recommendations for the Characterization of Porous Solids [1].)

— **Micropore analysis by 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 0,000 4 µm to 0,002 µm (0,4 nm to 2 nm).

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

Part 2: Analysis of mesopores and macropores by gas adsorption

1 Scope

This part of ISO 15901 describes a method for the evaluation of porosity and pore size distribution by gas adsorption. It is a comparative, rather than an absolute test. The method is limited to the determination of the quantity of a gas adsorbed per unit mass of sample at a controlled, constant temperature.

This part of ISO 15901 does not specify the use of a particular adsorptive gas, however nitrogen is the adsorptive gas most commonly used in such methods. Similarly, the temperature of liquid nitrogen is the analysis temperature most commonly used. Use is sometimes made of other adsorptive gases, including argon, carbon dioxide and krypton, and other analysis temperatures, including those of liquid argon and solid carbon dioxide. In the case of nitrogen adsorption at liquid nitrogen temperature, the basis of this method is to measure the quantity of nitrogen adsorbed at 77 K as a function of its relative pressure.

Traditionally, nitrogen adsorption is most appropriate for pores in the approximate range of widths 0,4 nm to 50 nm. Improvements in temperature control and pressure measurement now allow larger pore widths to be evaluated. This part of ISO 15901 describes the calculation of mesopore size distribution between 2 nm and 50 nm, and of macropore distribution up to 100 nm.

The method described in this part of ISO 15901 is suitable for a wide range of porous materials, even though the pore structure of certain materials is sometimes modified by pretreatment or cooling.

Two groups of procedures are specified to determine the amount of gas adsorbed:

— those which depend on the measurement of the amount of gas removed from the gas phase (i.e. gas volumetric methods), and

— those which involve the measurement of the uptake of the gas by the adsorbent (i.e. direct determination of increase in mass by gravimetric methods).

In practice, static or dynamic techniques can be used to determine the amount of gas adsorbed. To derive pore size distribution from the isotherm, it is necessary to apply one or more mathematical models, which entails simplifying certain basic assumptions.

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

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9277:1995, *Determination of the specific surface area of solids by gas adsorption using the BET method*

3 Terms and definitions

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

3.1
adsorbate
adsorbed gas

3.2
amount adsorbed
n_a
number of moles of gas adsorbed at a given pressure p

3.3
adsorbent
solid material on which adsorption occurs

3.4
adsorption
enrichment of the adsorptive gas at the external and accessible internal surfaces of a solid material

3.5
adsorptive
gas or vapour to be adsorbed

3.6
blind pore
dead-end pore
open pore having a single connection with an external surface

3.7
equilibrium adsorption pressure
p
pressure of the adsorptive gas in equilibrium with the adsorbate

3.8
ink bottle pore
narrow necked open pore

3.9
interconnected pore
pore which communicates with one or more other pores

3.10
isotherm
relationship between the amount of gas adsorbed and the equilibrium pressure of the gas, at constant temperature

3.11
macropore
pore of internal width greater than 50 nm