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Determination of density by volumetric displacement — Skeleton density by gas pycnometry

Détermination de la masse volumique par déplacement volumétrique — Masse volumique du squelette mesurée par pycnométrie à gaz

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Contents

Page

Foreword

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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

Introduction

The true solid state density of a material is defined as the ratio of the mass to the volume occupied by that mass. Therefore, the contribution to the volume made by pores or internal voids and also interparticle voids (in the case of granulated or highly dispersed samples) shall be subtracted when calculating the true density.

If the material has no porosity, the true density can be measured by displacement of any fluid in which the solid remains inert. The accuracy of the method is limited by the accuracy with which the fluid volume can be determined. Usually, however, the pores, cracks, or crevices of the material will not easily be completely penetrated by a displaced liquid. In these instances, the true density can be measured by using a gas as the displaced fluid if the material does not contain closed pores, which cannot be penetrated by the analysis gas. Therefore, the density experimentally determined by gas pycnometry generally is the so called skeleton density of the material which equals the true solid state density only for samples without closed pores.

Apparatus used to measure solid volumes are often referred to as pyknometers or pycnometers after the Greek “pyknos”, meaning thick or dense. With gas pycnometry, materials of irregular shape can be analysed.

Once the volume of solid skeleton of the sample and the sample mass have been determined, the skeleton density is readily calculated.

Determination of density by volumetric displacement — Skeleton density by gas pycnometry

1 Scope

This International Standard specifies a method for rapid and efficient determination of the skeleton density of solid material samples of regular or irregular shape, whether powdered or in one piece, by means of a gas displacement pycnometer.

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

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

ISO 15901-3, *Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption — Part 3: Analysis of micropores by gas adsorption*

3 Terms and definitions

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

3.1
density
ratio of the mass of a certain amount of a sample to the volume occupied by that mass

3.2
true solid state density
ratio of the sample mass to the volume of the compact solid skeleton of the sample which excludes the volume of open and closed pores or internal voids and also interparticle voids as in the case of granulated or highly dispersed samples

3.3
skeleton density
ratio between sample mass and the 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

3.4
closed pore
pore totally enclosed by its walls and hence not interconnecting with other pores and not accessible to fluids

3.5
open pore
pore not totally enclosed by its walls and open to the surface either directly or by interconnecting with other pores and therefore accessible to fluids

3.6
gauge pressure sensor
because gauge pressure is defined relative to atmospheric conditions, the signal or reading of a gauge pressure sensor is the total pressure minus atmospheric pressure

3.7
absolute pressure sensor
absolute pressure sensor measures the pressure relative to an absolute vacuum that means the reference is full vacuum (zero pressure)

4 Symbols and abbreviated terms

Table 1 — Symbols

Symbol	Name	Unit
ρ_s	skeleton density	g cm^{-3}
m_s	sample mass	g
V_s	skeleton volume of the sample	cm^3
V_{cell}	sample chamber volume	cm^3
V_{ref}	reference chamber volume	cm^3
V_{cal}	volume of the calibrated reference sample	cm^3
p_1	equilibrated gauge pressure prior to expansion [a]	Pa
p_2	equilibrated gauge pressure after expansion [a]	Pa
p_{A1}	equilibrated gauge pressure before expansion (calibration step A) [a]	Pa
p_{A2}	equilibrated gauge pressure after expansion (calibration step A) [a]	Pa
p_{B1}	equilibrated gauge pressure before expansion (2nd calibration step) [a]	Pa
p_{B2}	equilibrated gauge pressure after expansion (2nd calibration step) [a]	Pa
p_a	pycnometer pressure at start of analysis	Pa
p_i^*	pycnometer absolute gas pressure i (i = 1, 2, A1, A2, B1, or B2)	Pa
p_i	pycnometer excess gas pressure i (i = 1, 2, A1, A2, B1, or B2)	Pa

[a] gauge pressure (excess gas pressure) p_i is defined as the difference between the absolute pressure p_i^* and the pycnometer pressure p_a at start of analysis, i.e. $p_i = p_i^* - p_a$ (see 3.6, 3.7, and 6.3.2.1)

5 Principle of the method

The skeleton density will be determined volumetrically in a gas expansion pycnometer. This technique is based on the displacement of a volume of gas by the solid space. The measurement is performed by expanding gas from one chamber to another (see Figure 1) under isothermal conditions. First, the weight of the dry sample is to be determined and the sample loaded into the sample chamber. The sample chamber then is pressurized to a set value when using the experimental configuration 1 of Figure 1. In a further step, the analysis gas will be expanded into a second chamber, the reference volume. The equilibrated pressures for both steps will be recorded by the instrument. Density is calculated using these values.

For gas pycnometers according to the experimental configuration 2 of Figure 1, the first step consists in pressurizing the reference chamber to a set value followed by the expansion into the sample chamber having a lower initial pressure than the set value. It is important for both experimental pycnometer configurations, that every chamber of the pycnometer is at the same pressure p_a prior to starting the

analysis steps (see Clause 6) Furthermore, all parts of the pycnometer shall have the same controlled temperature.

The analysis gas of sufficient purity (see 6.1) shall be nonreactive and also non-adsorbing onto the solid sample. It has to behave as ideally as possible. Therefore, helium is used for most applications. Another reason for the preferred use of helium as the analysis gas for gas pycnometry is that it is able to penetrate even the smallest pores or cracks of a material.

NOTE 1 Because of its pronounced ability to permeate thin inner walls of samples with closed cells, helium can cause difficulties if permeable samples are to be analysed. Therefore, as described in Annex A.6, gas pycnometric measurements using helium can be erroneous in the case of organic samples like cellulose and cellular polymers with low density. For density measurements of those samples, the use of alternative inert gases such as nitrogen, argon, or sulfur hexafluoride as well as dry air is recommended.

NOTE 2 If the sample contains no closed pores, then the volume measured by gas pycnometry is the true volume. To test the presence of closed pores, after a first density determination the sample can be powdered revealing any possible closed pores accessible to the test gas. An increased density value of the ground sample material indicates closed pores in the original sample.

6 Apparatus and procedure

6.1 Apparatus

6.1.1 Gas expansion pycnometer, with fixed-volume sample chamber (see Figure 1).[1] [2] [3]

6.1.2 Calibrated reference sample, (in general calibration spheres made of stainless steel with known traceable volume).

6.1.3 Analysis gas, in general helium (see Clause 5) with a minimum purity of 99,996 % (by volume).

6.1.4 Analytical balance.

6.1.5 Drying oven, for pre-treatment of samples preferably with the option of purging during heating or heating in a vacuum.

Figure 1 is a schematic diagram of the two principal configurations of automatic gas expansion pycnometers having fixed sample chamber size. Main components of such instruments are two chambers connected by tubes (a sample chamber which can be sealed for inserting the sample or the calibration spheres and a reference chamber), a pressure-measuring sensor, and three valves. The difference between the pycnometer configurations is in the sequence of the sample chamber and the reference chamber.

Sample volumes of commercially available gas expansion pycnometers vary from 0,1 cm^3 to about 500 cm^3. This is accomplished either by having fixed-volume sample chambers of different sizes, or by means of volume-filling inserts placed into a sample chamber. These variations in sample chamber