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To cite this article: Alberto M. Sereno , Maria A. Silva & Luis Mayor (2007) Determination of Particle Density and Porosity in Foods and Porous Materials with High Moisture Content, International Journal of Food Properties, 10:3, 455-469, DOI: 10.1080/10942910600880736

To link to this article: https://doi.org/10.1080/10942910600880736
DETERMINATION OF PARTICLE DENSITY AND POROSITY IN FOODS AND POROUS MATERIALS WITH HIGH MOISTURE CONTENT

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A simple gas pycnometer was developed to obtain the volume of porous food materials. The volume is obtained by measuring the change in pressure experienced by an amount of compressed gas filling a constant volume reference chamber when it expands into a second chamber containing a sample of the material to be tested. From such pressure change and the knowledge of the volumes of the two chambers the volume of the sample solid matrix is determined. The main difference between the proposed pycnometer and the typical helium pycnometer is the sequence of chambers; in the latter gas compression is made in the sample chamber, producing evaporation of water and volatiles when the gas is expanded into the reference chamber. For these reasons the helium pycnometer is only recommended for volume measurements of bone dry solids. The performance of the gas pycnometer was assessed by measuring the volume of different solids (non-porous metallic cylinder, glass spheres, porous metallic cylinder, sintered sand cylinder and apples) and by comparing the results with values from other methods. The gas pycnometer reproducibility of 0.019%, obtained with dried porous materials, is excellent when compared with a commercial helium pycnometer. The gas pycnometer proposed in this work can be easily built and offers reliable results of particle volume for any type of solids, specifically foods and other materials with high moisture content.

Keywords: Apple, Foodstuffs, Fruits, Gas pycnometer, Particle density, Porosity.

INTRODUCTION

One of the most important parameters of the macroscopic structure of porous media is porosity, generally defined as the ratio of pore or void volume to the apparent volume of the porous sample.\(^\text{[1]}\) It is common to add an “adjective” to define porosity more precisely,
such as “effective,” “interparticle,” “intraparticle,” “particle,” “powder.” In many aspects, the quality of porous products is dependent on their porosity. The porosity behavior during the processing of such products is a true concern when researching foods, inorganic catalysts, as well as powder processing in general.

In the case of foods, Rahman pointed out the following aspects as being dependent on material porosity: (i) prediction and understanding of heat and mass transfer phenomena in several food processing operations, such as drying, smoking, blanching, frying, and extrusion among others; (ii) mechanical and textural properties; (iii) sensory properties; and (iv) quality and acceptance by the consumer.

Porosity determination requires the knowledge of the total or apparent volume, and the void volume included in the material matrix; their difference is also known as the particle volume. Measurement of the apparent or total volume is a relatively easy task and has been performed by means of different experimental procedures, such as volume displacement of a liquid, dimensional determinations, and buoyant force measurements. Different methods have been proposed to measure the particle volume. These methods often imply changes in the structure and/or composition of the material, such as the measurement of the volume of ground and degasified samples and liquid infiltration by application of a vacuum pulse. Helium stereopycnometry and mercury porosimetry have also been used but require sample dehydration.

Some authors have proposed empirical and semiempirical equations for prediction of porosity. Few works have proposed methods to measure particle volume in solids with high moisture content, and consequently, porosity of these solids. Following the procedure described by Day and Mavroudis et al., a specially designed gas pycnometer was successfully used in this work to measure the particle volume of solids with any moisture content, and as a consequence, to determine the porosity of such solids, if apparent volume is also known. The performance of this pycnometer was compared with other methods used to measure the particle volume of solids.

**MATERIALS AND METHODS**

**Theory and Terminology**

Some definitions, previously reported by other authors, are relevant for the different measured properties in this work. These definitions assume that the material to be analyzed is made up of a solid matrix (sm), water (w) and a gas phase. The gas phase is present in open (op) and closed (cp) pores. The open or interconnected pores, as defined by Dullien, are the pores connected with each other and with the external surface of the material. Thus the total (apparent) volume of the material is defined as:

$$V_T = V_{sm} + V_w + V_{op} + V_{cp}. \tag{1}$$

The particle volume, given by Eq. (2), is the total volume of the sample excluding the volume of the open pores, but including the volume of the closed pores.

$$V_p = V_{sm} + V_w + V_{cp} \tag{2}$$

Finally, the substance volume, given by Eq. (3), is the volume of the material excluding the air volume:
As a consequence, three different density definitions arise: apparent, particle and substance density, obtained by substituting in Eq. (4) by total (apparent) volume, particle volume and substance volume, respectively.

$$\rho_l = \frac{m}{V_i}$$  \hspace{1cm} (4)

Two different types of porosity can be defined: total porosity, which is the ratio of the air volume (that is, open and closed pore volumes) to the total (apparent) volume:

$$\varepsilon_T = \frac{\rho_{sb} - \rho_b}{\rho_{sb}} = \frac{V_T - V_{sb}}{V_T}$$  \hspace{1cm} (5)

and open pore porosity, also called effective porosity,\textsuperscript{[1]} which is the ratio of the volume of pores connected to the outside to the total (apparent) volume:

$$\varepsilon_{op} = \frac{\rho_p - \rho_b}{\rho_p} = \frac{V_T - V_p}{V_T}.$$ \hspace{1cm} (6)

In this work, an apparatus and a procedure are proposed to determine the particle volume of wet and dry solids, allowing the calculation of the particle density and open pore porosity, if the apparent density of such materials is known. The next section describes the proposed apparatus.

**Apparatus Description**

The apparatus used in this study was a gas pycnometer built in the Laboratory of Rheology and Food Engineering, Department of Chemical Engineering, University of Porto. The pycnometer measures the change in pressure experienced by a known amount of compressed gas filling a constant volume reference chamber when it expands into a second chamber containing a sample of the material.

Basically, the difference between the gas pycnometer presented in this work and a commercial helium pycnometer is the sequence of the chambers, since in the latter the gas compression is done in the sample chamber followed by its expansion into a reference chamber. Such sequence of operations implies that when the solid contains moisture, even in small quantities, its partial vaporization occurs during the gas expansion, changing the composition of the solid sample and affecting the pressure in the system. Since pressure is what is actually measured by the instrument, any variation in pressure measurement will be reported as a volume variation. Thus, the commercial helium pycnometers are only recommended to measure the volume of bone dry solids\textsuperscript{[21,22,23]} A schematic diagram of the experimental gas pycnometer and its picture are shown in Figure 1 and Figure 2, respectively.

\[
V_{sb} = V_{sm} + V_w. \hspace{1cm} (3)
\]
This gas pycnometer consists of two sections with volumes $V_1$ (ca 44 cm$^3$) and $V_2$ (ca 47 cm$^3$), four valves, one pressure transducer and one thermocouple. The first section includes the reference chamber and all piping connections between valve 1 and valve 2. The pressure transducer and the thermocouple are placed after the reference chamber and before valve 2. The second section includes the sample chamber and all piping connections between valves 2 and 3. The pressure transducer measures the absolute pressure within the range 0–7 bar (0–0.7 MPa) and resolution of 0.001 bar (100 Pa). Although the apparatus was located in a room with controlled temperature, a thermocouple was installed to check any variation in temperature during the analysis. The K-type thermocouple was connected to a digital display with resolution of 0.1°C. The gas used in the experiments was compressed air.

**Gas Pycnometer Operation**

To obtain reliable measurements the apparatus should be always operated in two subsequent modes: (i) calibration and (ii) analysis. In the calibration mode, the measurements are done using a calibration cylinder made of nonporous steel, having a known
constant volume, in order to evaluate $V_1$ and $V_2$. In the analysis mode, the calibration cylinder is substituted by the sample. The operation steps are shown in Table 1.

The system was considered to have reached mechanical equilibrium when no pressure change was observed for 1 min. It was also assumed that the solid matrix and liquid phase were incompressible in the range of pressures used (up to seven bars).

### Obtaining the Volumes Containing the Reference (V1) and Sample (V2) Chambers

As air was the gas used in the experiments and pressure could reach up to seven bars, the ideal gas equation and the Beattie-Bridgeman equation for air \(^{[24]}\) were initially used to describe air behavior, but no significant differences were found. So, the ideal gas equation was used for all calculations. Eqs. (7–13) correspond to steps 1–4 described above (see Table 1), that is, with empty pycnometer. The ideal gas model applied to step 1 provides Eqs. (7) and (8).

\[
P_{\text{atm}}V_1 = n_1R_{\text{atm}}T_{\text{atm}} \tag{7}
\]

\[
P_{\text{atm}}V_2 = n_2R_{\text{atm}}T_{\text{atm}} \tag{8}
\]

The temperature and pressure measured in step 3 are related by Eq. (9) while Eq. (10) is related to step 4.

\[
P_1V_1 = n_1R_{\text{atm}}T_1 \tag{9}
\]

and

\[
P_2(V_1 + V_2) = (n_1 + n_2)R_{\text{atm}}T_2, \tag{10}
\]

where $n_1$ corresponds to the amount of air in $V_1$, that is, the air that entered after opening valve 1 plus the amount of air initially in $V_1$ at atmospheric pressure.
Rearranging Eqs. 9 and 10:

\[
\frac{V_1}{V_1 + V_2} = \frac{n_1}{n_1 + n_{2e}} \frac{T_1 P_2}{T_2 P_1}
\]

(11)

Making \( A = \frac{T_1 P_2}{T_2 P_1} \) and inverting Eq. 11:

\[
\frac{V_1 + V_2}{V_1} = \left(1 + \frac{n_{2e}}{n_1}\right) \frac{1}{A}
\]

(12)

Combining Eqs. 8 and 9 with Eq. 12 and solving for \( V_1 \):

\[
V_1 = V_2 \left( \frac{P_{\text{atm}}}{P_2} \frac{T_2}{T_{\text{atm}}} - 1 \right) \frac{A}{A - 1}
\]

(13)

Eqs. (14–22) correspond to steps 6–7 (Table 1), that is, with the calibration cylinder inside the sample chamber.

\[
P'_{\text{atm}} (V_2 - V_k) = n_{2k} R T'_{\text{atm}}
\]

(14)

Eq. (15) relates the variables measured in steps 2 and 3 with the calibration cylinder in the pycnometer:

\[
P_1' V_1 = n_1' R T_1'
\]

(15)

where \( n_1' \) corresponds to a new amount of air contained in \( V_1 \), that is, the air that entered volume \( V_1 \) after opening valve 1 plus the air initially present in \( V_1 \) at atmospheric pressure. Letting the air expand to fill the sample cell, the following relation is obtained:

\[
P_2' (V_1 + V_2 - V_k) = (n_1' + n_{2k}) R T'_2.
\]

(16)

Dividing the members of Eq. (15) by the corresponding members of Eq. (16):

\[
\frac{V_1}{V_1 + V_2 - V_k} = \frac{n_1'}{n_1' + n_{2k}} \frac{T_1'}{T_2'} \frac{P_2'}{P_1'}
\]

(17)

Making \( B = \frac{T_1'}{T_2'} \frac{P_2'}{P_1'} \) and inverting Eq. (17):

(17a)
Combining Eqs. (14) and (15) with Eq. (18) and rearranging:

\[
\frac{V_1 + V_2 - V_k}{V_1} = \frac{n_1' + n_{zk}1}{n_1'} B.
\]  

(18)

or

\[
\frac{V_1}{V_2 - V_k} = \left(\frac{P'_{atm} T_2'}{P'_{2} T'_{atm}} - 1\right) B \quad \text{B - 1}
\]

(19)

Inserting Eq. (13) into Eq. (19) and solving for \(V_2\):

\[
V_2 = \frac{V_1}{\left(\frac{P'_{atm} T_2'}{P'_{2} T'_{atm}} - 1\right) B \quad \text{B - 1}} + V_k.
\]

(19a)

Comparing Eqs. (21) with Eq. (13), \(V_1\) can be obtained by:

\[
V_1 = V_2 EF.
\]

(22)

During calibration, the gas pycnometer was operated according to the steps 1–6 (Table 1) and Eqs. (20) and (22) were used to calculate \(V_1\) and \(V_2\).

**Sample Volume Determination**

Equation (23) relates the variables when the sample with unknown volume is in the sample chamber and valves 1 and 2 are closed (system at atmospheric pressure),

\[
P_{atm}(V_2 - V_s) = n_2 s RT'_{atm}.
\]

(23)

After steps 2 and 3 (Table 1) with the sample,

\[
P_{1s}V_1 = n_1 s RT_{1s},
\]

(24)

where \(n_1^s\) corresponds to the amount of air in \(V_1\), that is, the air that entered after opening valve 1 plus the air mass initially in \(V_1\) at atmospheric pressure when the sample is in the gas pycnometer. After expanding the air into the sample cell:
Combining Eqs. (23–25) and rearranging, the expression for the sample volume is given by:

\[ V_S = V_1 \left( \frac{P_{1S} T_{2S}}{P_{2S} T_{1S}} - 1 \right) \frac{P_{1S}}{P_{2S}} T_{2S} - \frac{P_{2S}}{P_{2S}} T_{1S} \right) + V_2 \]  

(26)

In the case of an isothermal operation:

\[ V_S = V_1 \left( \frac{P_{1S} - P_{2S}}{P_{atm} - P_{2S}} \right) + V_2. \]  

(27)

It is observed in Eq. (27) that only pressure differences are involved and thus gauge pressures can be used instead of absolute pressures, leading to the following equation:

\[ \frac{V_2 - V_S}{V_1} = \frac{P_{1S} - P_{2S}}{P_{2S}}. \]  

(28)

For a non-isothermal situation where gauge pressures are measured, a suitable equation can also be found from Eq. (26). The same principle used in this work was used by Day\textsuperscript{[19]} to determine the interstitial volume of seeds in chambers with volume of 1000 cm\textsuperscript{3} and pressures slightly higher than the atmospheric. Equation (28) is identical to the one obtained by Day\textsuperscript{[19]} that used gauge pressures in his final equation. Mohsenin\textsuperscript{[25,26]} cited Day’s paper and presented the initial derivation, where absolute pressures were considered, and obtained a final equation similar to Eq. (28) without specifying that gauge pressures need to be used. Mavroudis et al.\textsuperscript{[18]} also used the same principle to determine particle volume and porosity during osmotic dehydration of apples. They presented an identical equation but they didn’t specify whether they used absolute or gauge pressures.

When absolute pressure is measured, Eq. (26) or (27) must be used, since these equations take into account the amount of air contained initially in the chambers, before the compression. For the same reason, Eqs. (13) and (19) must be used for the calibration. Table 2 illustrates what happens if the adequate equations are not used, that

<table>
<thead>
<tr>
<th>Absolute applied pressure (P₁), MPa</th>
<th>Reference chamber (V₁), cm³</th>
<th>Sample chamber (V₂), cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>n₂=k=0 in Eq. 13</td>
<td>n₂=0 in Eq. 18</td>
</tr>
<tr>
<td>0.3489</td>
<td>44.5</td>
<td>46.6</td>
</tr>
<tr>
<td>0.5042</td>
<td>44.3</td>
<td>46.6</td>
</tr>
<tr>
<td>0.6550</td>
<td>44.3</td>
<td>46.5</td>
</tr>
</tbody>
</table>

Table 2 Volume of reference and sample chambers with applied pressure.
is, if absolute pressure is measured but the amount of air contained in the chamber at atmospheric pressure is not considered in the calculations. The chamber volumes calculated without considering the amount of air initially in them at atmospheric pressure (Eqs. 12 and 18), varied considerably with the applied pressure; this fact would produce important errors in the measurement of particle volumes, so Eqs. (13) and (19) must be used.

The commercial helium gas pycnometer measures gauge pressures. The equation derived considers gauge pressures and isothermal process.\[21\] As the gas compression occurs in the sample chamber followed by its expansion into the reference chamber, the final equation is similar to Eq. (28) with inverted numerator and denominator in the right-hand term, as shown in Eq. (29).

\[
\frac{V_2 - V_S}{V_1} = \frac{p_{ae}}{p_{be} - p_{ae}}
\]  

(29)

Experimental Design, Preparation of Samples and Analytical Methods

In order to assess the reliability of the gas pycnometer, the particle volume of different solid samples was measured. The samples used were: a non-porous metallic cylinder, glass spheres and a sintered sand cylinder. The results were compared with the particle volume values obtained using other methods. Calipers were used to determine the dimensions of the non-porous metallic cylinder, and the particle volume was obtained from such dimensions. Liquid pycnometry was used to determine the volume of the glass spheres. A helium pycnometer (AccuPyc 1330TM from Micromeritics®) was used to measure the particle volume of the sintered sand cylinder.

After the performance of the pycnometer had been assessed, particle density and porosity of a biological material (apple) with different moisture contents were measured. Apples (Golden Delicious variety) were purchased in a local market and stored in a refrigerator at 4°C until use. An electric slicing machine was used to cut slices perpendicularly to the apple axis with a thickness of 2.60 ± 0.1 mm; from these slices, discs with diameter of 25.7 ±0.1 mm were subsequently cut with a cork borer. To obtain samples of different moisture contents, samples were partially dried in an oven at 70°C under natural convection; they were removed from the oven at different drying times and their mass, dimensions, particle volume and apparent volume were determined. Dimensions were measured with a caliper. Particle volume was determined with the gas pycnometer presented in this work. Apparent volume was obtained by two different methods: from the dimension values, using calipers (experiment 1) and measuring buoyant forces\[17\] using n-heptane as liquid (experiment 2). From the apparent and particle volume, and using Eq. (6), porosity was calculated.

Moisture content of the samples was determined by oven drying at 70°C under a pressure lower than 10 kPa, during 24 h.\[27\] Concerning particle volume measurements with the proposed gas pycnometer, for all series of tests the effect of the applied pressure was studied as well as different modes of opening valve 2: fast, slowly and by steps. The sensitivity of the results with respect to the uncertainty of pressure and temperature measurements was also assessed. In the case of tests involving apple, mass of samples was determined before and after the analysis in order to check any possible mass loss.
RESULTS AND DISCUSSION

The calibration procedure was repeated more than 50 times in order to verify the reproducibility of the results. The volume (V2) that contains the sample chamber was found to be 46.505 ± 0.009 cm³ with reproducibility of 0.019%. The reproducibility of a helium gas pycnometer (AccuPyc 1330TM from Micromeritics®) was determined as 0.018%, slightly higher than the reproducibility indicated by the manufacturer, typically within ± 0.01%.[28] In spite of the difference between the accuracy of the pressure transducers used in both pycnometers, the reproducibility of the gas pycnometer studied in this work is excellent when compared to a commercial one.

The results obtained for the different solids tested in the gas pycnometer are shown in Table 3. Such results are quite good when compared with other methods. All tests were performed using applied pressures of 0.20, 0.25, 0.30, 0.35, 0.50, and 0.65 MPa and similar results were obtained in all cases. The operating mode of valve 2 had a slight influence in the results. When slow or step opening were used the measured volume was slightly smaller than when the valve was opened very quickly, apparently suggesting that in the former a more intense air penetration in the solid is observed. Nevertheless, it is too difficult to control and reproduce the way the valve is opened in the slow or step modes, when this is conducted manually, leading to results with increased standard deviations for such runs (data not shown).

The sensitivity of the results with respect to the uncertainty in pressure and temperature measurements is presented in Table 4. As expected the higher the pressure the lower the deviation of the measured volume. This is why higher pressures, around 0.65 MPa, were used for the analyses, leading to the results presented in Tables 2 and 4. Some tests were done using samples of fresh Golden Delicious apple (X=87% w.b.). All porosity values were between 22.5 % and 24.5% in agreement with results in the range of 20% to 25% presented by Hills and Remigereau,[29] as well as with the values determined by Lozano.

Table 3 Comparison of tests performed in the gas pycnometer and with other methods of particle volume analysis.

<table>
<thead>
<tr>
<th>Material</th>
<th>Gas pycnometer</th>
<th>Other methods(1)</th>
<th>Relative deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>V_S   σ</td>
<td>V_{om} σ</td>
<td></td>
</tr>
<tr>
<td>Non-porous Metallic Cylinder</td>
<td>29.390 0.019</td>
<td>29.4053 0.0004</td>
<td>Callipers</td>
</tr>
<tr>
<td>Glass spheres</td>
<td>19.635 0.015</td>
<td>19.486 0.005</td>
<td>Liquid Pycnometry</td>
</tr>
<tr>
<td>Sintered sand Cylinder</td>
<td>2.80   0.02</td>
<td>2.811 0.003</td>
<td>Helium Pycnometry</td>
</tr>
</tbody>
</table>

Table 4 Sensitivity of the results related to the accuracy of the pressure and temperature measurements.

<table>
<thead>
<tr>
<th>P_1 (MPa)</th>
<th>ΔP_2 (MPa)</th>
<th>ΔV_S (cm³)</th>
<th>ΔT (°C)</th>
<th>ΔV_S (cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.20</td>
<td>±0.0001</td>
<td>±0.10</td>
<td>±0.1</td>
<td>±0.054</td>
</tr>
<tr>
<td>0.25</td>
<td>±0.0001</td>
<td>±0.07</td>
<td>±0.1</td>
<td>±0.045</td>
</tr>
<tr>
<td>0.35</td>
<td>±0.0001</td>
<td>±0.04</td>
<td>±0.1</td>
<td>±0.038</td>
</tr>
<tr>
<td>0.50</td>
<td>±0.0001</td>
<td>±0.03</td>
<td>±0.1</td>
<td>±0.034</td>
</tr>
<tr>
<td>0.65</td>
<td>±0.0001</td>
<td>±0.02</td>
<td>±0.1</td>
<td>±0.032</td>
</tr>
</tbody>
</table>
et al.\textsuperscript{[17]} (X=88\% w.b.). If any water was lost in wet samples, then a mass loss would be observed during the analysis. Some mass loss (less than 1\%) was indeed detected for samples of fresh apple only due to their high moisture content (87\% wb). Such loss can be considered negligible compared to the loss of about 5\% observed during the same time for control samples resting on the laboratory bench.

Figure 3 shows particle and apparent density of dehydrated apple samples at different moisture contents. The values for particle density obtained in experiments 1 and 2 are similar, whereas values of apparent density show differences due to the different experimental method used to obtain the apparent volume of samples. Figure 4 presents the porosity of Golden Delicious apples with different moisture contents, which were obtained in the drying experiments. The differences between the results of experiments 1 and 2 are directly associated to the different ways used to obtain the apparent volume, since there was no difference between their particle volumes. The results for porosity are in good agreement with those obtained by Lozano et al.\textsuperscript{[17]} for Granny Smith apples, as can be observed in Fig. 4. Each point in Figure 4 is the average value of five runs and all points are presented in Table 5 with the corresponding standard deviation.

**CONCLUSIONS**

The gas pycnometer and procedure described in this work were successfully used to determine particle volume of solids with high moisture content, that is, the volume occupied by the solid matrix, water and closed pores. Therefore, the porosity and particle density of foods and other solids with high moisture content can be easily and accurately obtained from measurements which require a precise pressure sensor and with the
proposed mathematical models. The gas pycnometer is easily built offering reliable results of particle volume for solids with high moisture content. It should be stressed that the use of the helium pycnometer is only recommended for bone dry solids. Further investigation needs to be done in order to verify the influence of the air humidity in the pressure measurements and in the sample mass loss. The operation mode of the expansion valve should be further investigated to assure reproducible results, as well.

**ACKNOWLEDGMENTS**

The author M.A. Silva wishes to acknowledge CAPES (Brazilian Agency for Improvement of Graduate Personnel, Brazil, proc. n. BEX0956/02-3) for the financial support during her sabbatical stay at
the University of Porto (UP), Portugal, as well as the facilities provided by Requimte/Department of
Chemical Engineering at UP. The author Luis Mayor wishes to acknowledge SFRH/BD/3414/2000
PhD grant to Fundação para a Ciência e a Tecnologia, Portugal.

NOMENCLATURE

A Defined in Eq. (11a)
B Defined in Eq. (17a)
E Defined in Eq. (21)
F Defined in Eq. (21)
G Defined in Eq. (21)
H Defined in Eq. (21)
m Mass (kg)
n Number of moles
p Gauge pressure (Pa)
P Absolute pressure(Pa)
R Universal gas constant (8314.41 J kgmol$^{-1}$ K$^{-1}$)
T Temperature (K)
V Volume (m$^3$)
ε Porosity
ρ Density (kg m$^{-3}$)
σ Standard deviation

Subscripts and Superscripts

1 refers to the volume that contains the reference chamber
2 refers to the volume that contains the sample chamber
′ refers to the measurements done with known solid volume in the sample chamber
* refers to the measurements done with sample in the gas pycnometer
a ex after expansion
a tm atmospheric
b apparent
b ex before expansion
cp closed pores
e empty
k known solid volume
om other methods
op open pores
p particle
S sample
sb substance
sm solid matrix
T total
w water

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