

## **Alternative proposal to ASTM C204 method for measurement of cement fineness, porosity, and density using automatic gas pycnometer**

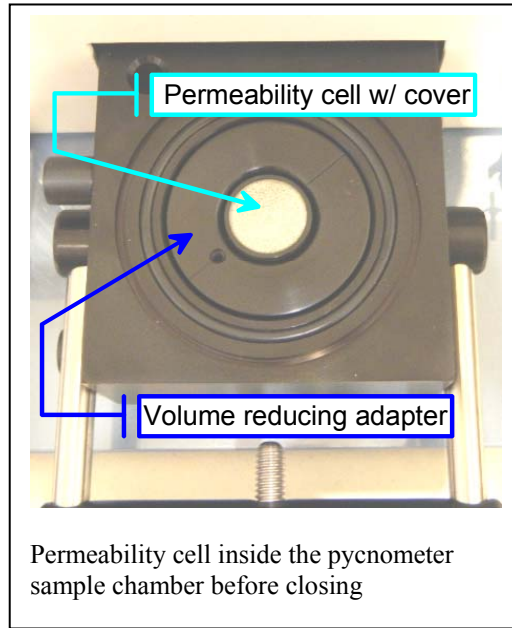
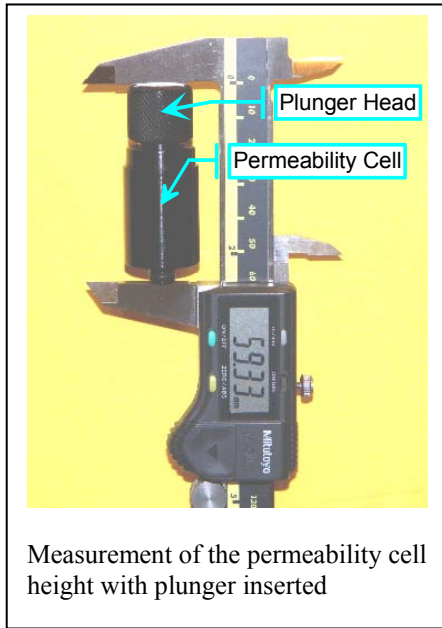
The purpose of this note is to present a more convenient approach to measurement of cement density, porosity, and fineness (specific surface area) using a specialized version of the HumiPyc series of gas pycnometers. The typical capability of true volume (density) measurements by a gas pycnometer is combined with proprietary design of sample cell for air-permeability and flow-through the sample technology. Additional features of the hardware and software allow for studies of other materials without some of the assumptions of the C204 method and to carry out the measurements more conveniently.

One of the objectives of the new approach was incorporation of the Blaine Air-Permeability apparatus design into the pycnometer and trying to preserve the experimental conditions used in the method. In the Blaine apparatus, a fixed amount of air passes through the packed bed of a cement sample. The initial pressure difference is about 2.3 kPa below atmospheric, and the time counting begins when the pressure difference is about 1.43 kPa below atmospheric pressure. The end time is recorded when the pressure value is about 0.31 kPa below atmospheric pressure. To mimic these conditions, but for the above atmospheric pressure levels to avoid creating the need of producing vacuum, the gas pycnometer is pressurized to a programmable pressure level. After stabilization, the process of recording of the pressure drop versus time takes place. To ensure the same amount of air volume flowing through the sample at these pressure limits, an optional auxiliary volume can be connected to the large reference chamber and it can be useful for other materials and conditions. Since the volumes of sample and reference chambers can be easily determined by prior calibrations, it is easy to calculate the time interval when a fixed air volume passes through the sample. The advantage of this approach is that any gas can be used and any pressure interval can be utilized for cementitious materials or other powders. The recorded pressure vs. time curve can serve for further studies of gas permeability and the method, in addition to yielding the needed time interval for passage of specific gas volume. The gas pycnometer replaces the U-Tube manometer and associated with it hardware. The calibrated temperature sensor (RTD based) provides the temperature of the test for exact calculation of viscosity of air or any other gas used.

### **Permeability Cell for Cement and other powders**

A similar in principle sample cell for holding the cement bed was designed to be used in the pycnometer. It can easily hold the 15 mm bed height and has the diameter close to the specified 12.7 mm. Cells with other diameters and bed heights can be used as well. The bottom of the cell has a ridge to support either the perforated disk stipulated by the C204 method or a fritted metal filter. The cell plugs into the bottom hole of the pycnometer sample chamber and can be covered with a special cover having large fritted filter

installed for protection against accidental powder removal during experiment. The plunger for forming the flat top and slightly compressing the bed has similar construction to the one described in the C204 method. The pictures below show the permeability cell with plunger inside during measurement of height without the sample, and after the permeability cell was installed inside the pycnometer sample chamber.



## Procedure

It is assumed that the pycnometer reference chambers (and any additional volume if one is being attached) are calibrated as per pycnometer standard procedure. The internal diameter of the permeability cell should also be measured and recorded. The actual sequence of the measurements can differ from the description below, but the mass of the cement used and the height of the bed must be determined. It is more convenient to measure first the initial volume of the pycnometer sample chamber with the permeability cell installed without the cement sample. Next, after forming the cement bed, the permeability data are obtained. In the final step, the volume of pycnometer sample chamber with the permeability cell installed with the cement sample is carried out. It is also assumed, that appropriate experiment definition template has been prepared (or provided) and one of the specialized version of the gas pycnometer and computer for automatic control are available.

1. After placing the perforated disk or fritted filter and two paper filter circles of diameter 12.7 mm into the permeability cell, insert the cell with the special cover into the pycnometer sample chamber and insert the volume reducing adapter. Close the pycnometer sample chamber.
2. Start the software experimental procedure, which in the first step will determine the sample chamber volume of the pycnometer without the cement sample.

3. Remove the permeability cell and record the initial height of the permeability cell (with the plunger inserted).

4. Remove one of the paper filters from the permeability cell, prepare the cement sample appropriately, weigh about 2.8 – 3.0 g of the cement sample, tap lightly the wall to settle the cement in the cell, and put the paper filter on top of the cement bed. Using the plunger, form the flat and firm bed using only thumb pressure. (*This is the most critical aspect of the measurements and some discussion will follow*). Record the actual mass of cement used to form the bed.

5. With the plunger being inserted in the permeability cell, record the height. The difference between this step measurement and the step 3, will yield the cement bed height.

6. Insert the cell permeability cell into the pycnometer and continue the experimental procedure. In the next step, after the requested level of pressurization is achieved, the auxiliary exhaust valve will open and the pressure versus time curve will be recorded. Depending on particular template design, the step can be repeated a number of times. After that, the usual sequence of measurements of sample chamber volume will take place, yielding the volume of the cement sample. After completing the measurements and calculations, the permeability hardware should be cleaned and the paper filters discarded.

7. With all data at hand, the permeability calculator that is provided with the HumiPyc software can be used for further calculations.

- The pycnometer measures the sample volume and knowing its mass, the sample **true density** is calculated. There is no need to assume some specific density value like 3.15 g/cm<sup>3</sup> for the Portland cement

- Knowing the cement volume determined by the pycnometer (true volume) and the geometrical volume of the cement bed, the **porosity** can be calculated and recorded. There is no need to assume a specific porosity value, like 0.53 or 0.50 , as even for different cements, porosity can differ quite a bit from these values.

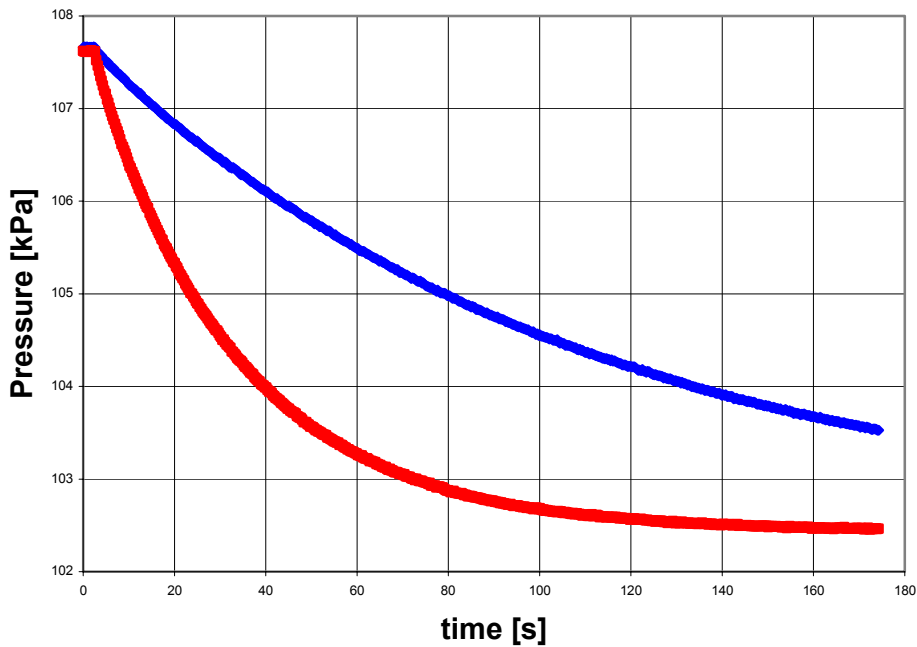
- Depending on the pressure differential or the start or end pressure value and the declared gas volume passed through the bed, the **time interval** can be calculated and recorded.

Since the Blaine method is rather relative than absolute and requires a standard material for comparison, like the Standard Reference Material Number 114 (from NIST) for Portland cement, the steps from 1 to 7 need to be repeated for the standard material. Then, after obtaining the porosity and time interval measurement for the standard material, the calculation of the **specific surface area** for the sample can be made.

The usage of a pycnometer and the ancillary hardware associated with the permeability cell allow for carrying out automatic, fast, and convenient experimentation without the involvement of mercury or any other liquids, which can be harmful to the operator health or environment, and are harder to obtain due to regulatory reasons. Unlike at the time when the method was created, having all data recorded and easily transferable to spreadsheets, further mathematical calculations and the method study can be carried out.

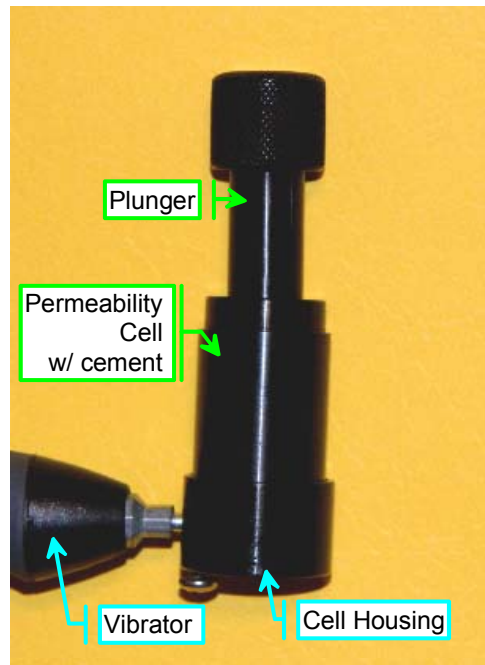
Perhaps the most crucial factor in the applicability of this method is the ability to form the cement beds in the same way all the time, for the sample and for the standard material. The many other detailed mechanical dimensions or specific hardware is not as important. As an example to show disparate results, and old cement sample was used as is, without the appropriate shaking to fluff the cement. The following chart shows pressure drop curve versus time for two runs, one after producing the cement bed by applying the thumb pressure on the plunger for compaction (red line), and the second run, when the same bed was re-settled using a special vibrator and applying similar thumb pressure during vibrating process (blue line).

Pressure vs. time curves for **slightly packed (red)** and **vibrated (blue)** cement bed



The consecutive runs are very repeatable for either way of the cement bed preparation. However, the way the bed was prepared can yield very different times for a given pressure interval. The atmospheric pressure value was 102.45 kPa. It is worth to mention that the use of vibrations to settle the cement under thumb pressure reduced the height of the bed by less than 1 mm on about 15 mm bed height, but the flow characteristics are substantially different.

To diminish the effect of sample preparation and compaction variation from sample to sample, especially if different operators are involved, it is recommended to use a designated vibrator for compaction of the powdered material. The next picture shows a simple solution by attaching a specially designed housing to a commonly available hand held engraver, that produces 7200 strokes per minute. The housing allows easy insertion of the permeability cell. Thumb pressure can be applied to the plunger during the compaction process.



Alternatively, an adapter assembly can be attached to the plunger head (either a fixed weight or a spring construction) to exhibit constant force downward. Applying vibrations of the same force for a fixed time with constant force pushing the plunger, more uniform powder bed construction can be achieved.

The above methodology of determining the cements fineness (specific surface area) can also be utilized for characterization of other powdered materials of similar properties. Considering the simplicity and convenience of the method, low cost of instrumentation and very low cost of analysis, it can be of interest to many material research scientists and QC testing.