**MICROTRAC**
MRB

Density Measurement of Fine Powders
BELPYCNO L

Helium Pycnometry with BELPYCNO L

Helium pycnometry is used to determine the true density of a wide variety of materials, like powders, porous materials or bodies with a complex shape. The volume of a material is measured by the displacement of an inert gas (helium) calculated from the pressures by expanding the gas from a reference volume into a sample chamber or vice versa.

Unfortunately, powders, and here especially fine, porous powders, were carried with the expanded gas into valves and the tubing of the instruments. This is the reason, why in the BELPYCNO L the sample chamber is placed after the reference chamber, so a contamination of the reference chamber with sample is not possible. Additionally, there is the option to release the pressure from the sample chamber through a restriction. Therefore, the parameter -- **Restriction delta pressure** -- has to be set to a pressure below the normal delta pressure between atmosphere and the pressure after expansion from the reference into the sample chamber.
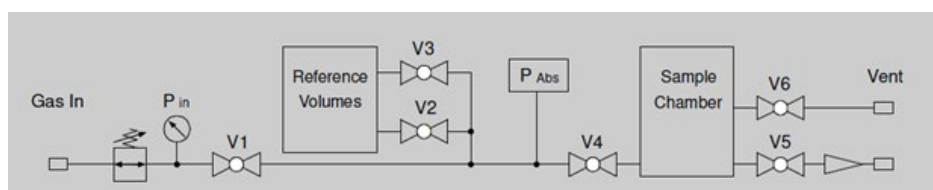
Fig. 1: *Flow diagram of the BELPYCNO L (left) and BELPYCNO L instrument (right)*

In Fig.1 the flow diagram of the instrument is shown, where valve 6 opens the normal line for degassing and valve 5 opens the sample chamber through a restriction against the atmosphere.

Below, the analytical parameters for the measurement of a fine powder are listed, the -- `Restriction delta pressure` -- is set to 0.25 bar, which means, that the pressure from the sample chamber is released by valve 5 to atmosphere till an absolute pressure of about 1.25 bar is reached, after this the valve 6 opens to release the residual pressure more quickly to the atmosphere.

ANALYTICAL PARAMETERS

- Reference: III
- Reduction: Large
- Reference volume: 61.97250 cc
- Cell volume: 60.45888 cc
- Filler volume: 0.00000 cc
- Repeated analyses nr.: 3
- Flow cleaning time: 0: sec
- Number of cleaning cycles: 5
- Sample cleaning time: 5 sec
- Atm. stabilization time: 10 sec
- Restriction delta pressure: 2.00000 bar
- Equilibrium delta pressure: 0.00020: bar
- Equilibrium delta time: 15 sec
- % Standard deviation: 0.020 %
- Nr. of good measurements: 5
- Nr. of max. measurements: 25

- High precision: Yes
- Temperature set: 20.00: °C

Additional to these standard settings there is the possibility, to protect the instrument against fine particles by an optional filter kit. Fig.2 shows a set of filters with a sample vessel and a calibration ball.

For the first measurement with the filter, the system (the height of the volume reduction and the sample vessel) has to be adapted and then recalibrated. The use of the filter is shown in Fig. 3 and Fig. 4, it is placed on top of the volume reduction. When working with powders, the -- **Restriction delta pressure** -- can be set to higher values and in some cases can be left at a value of 2.4 bar like for monolithic samples.

Fig. 2: *Set of filters, vessel and calibration ball.*

Fig. 3: *Use of filters*

Fig. 4: *Use of filters*

There are two different ways to handle the volume of the filter, first and recommended way if using a filter only from time to time, you can calibrate the system without filter and do a measurement with the calibration ball and the filter. The difference of the measured volume compared to the specified calibration ball volume can be used as filler volume for the measurement parameters. Or second and recommended, if measuring mostly powders, is to calibrate the system with filter. When changing the filter, the volume must be checked by measuring a calibration ball.

Calibration of the instruments should always be done carefully, because it is the basis for a good measurement. Therefore, first of all the suitable calibration ball should be measured sometimes to see if a constant value is obtained (if this value is equal to the given volume of the reference ball the system is calibrated and should be kept like it is). If not, now a calibration procedure with parameters as shown below could be started.

PARAMETERS

- Calibration ball volume: 14.36838 cc
- No. of cleaning cycles: 12
- Cleaning time: 5 sec
- Atm stabilization time: 20 sec
- Equilibrium delta press.: 0.00020 bar
- Equilibrium delta time: 15 sec
- No. of measurements: 8
- Deviation limit: 0.050 %

At the end, the calibration ball should be measured again, to see if the deviation of the measured volume and the given calibration ball volume is within the desired accuracy.

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