SIEBTECHNIK TEMA

Pycnometry in the laboratory

What is the correct procedure and how can I avoid sources of error?
With standard gas pycnometers or the BULKINSPECTOR.

Pycnometry in the laboratory

What are points to remember?

Why does the quality of an analysis often depend on the person performing it?
And why are there certain tasks in laboratories that are often only (allowed to be) performed by a certain person?

IN MANY CASES, THE ANSWER IS EXPERTISE, PRACTICE & EFFICIENCY.

You need to know how to proceed and avoid mistakes in general (expertise). Routine and effective planning of the procedure likewise helps to avoid sources of error, and also saves time (practice & efficiency).

Density determination actually consists of two individual measurements on two different devices - on scales to determine the mass of the sample, and then on the actual gas pycnometer, the device for determining the volume.

Even the handling process between the two instruments can therefore lead to measurement inaccuracies, and even tiny contaminations falsify the result.

With a standard gas pycnometer, the laboratory technicians are additionally tied up for the entire duration of the measurement process.

To better explain the procedure for determining density, and to explain the differences with the BULKINSPECTOR, you can find all the necessary information here, including common potential errors.

LABORATORY SETUP

The entire density determination process is performed manually by trained laboratory technicians. Preferably always by the same person.

To avoid errors during handling and transport of the sample, or even a substitution of the sample, **it should be ensured that the scales and pycnometer are close together**. This keeps the travel distances short and minimizes risk.

Potential risks: Sneezing. The sample could be spilled, or other processes could cause it to be contaminated with other particles. As the sample containers often cannot be labeled, misidentification of samples is also conceivable.

SELECTION OF THE RIGHT MEASURING GAS

It is important to use the right measuring gas, which is selected based on the nature of the sample and the purpose of the measurement. **Wherever possible, this should be temperature-controlled to the measurement temperature.**

With conventional laboratory pycnometers, the temperature control of the gas is generally difficult. A longer line should therefore be laid in the room between the pressure reducer and the measuring device, to allow the measuring gas to reach room temperature before it flows into the device.

Helium has various qualities. Please never use cheaper helium balloon gas, because this has not been dehumidified.

PYCNOMETRY WITH STANDARD GAS PYCNOMETERS

THE SAMPLE

Strictly speaking, each sample should be dried and dehumidified before measurement to remove adsorbed water, both from the surface and from pores. This can be done using a vacuum in a desiccator, or in a drying oven at slightly above 100°C - if the sample can tolerate this.

However, for largely smooth sample surfaces, or for particles with few or no pores, this conditioning can be omitted.

In the case of very fine-grained material, potentially even with a high pore content, dehumidification of the sample before measurement is always recommended.

TEMPERATURE CONTROL & AIR PRESSURE

It is important that the sample is then allowed to cool down to room temperature (actually measurement temperature) **and keep it in the absence of air** because atmospheric moisture is generally absorbed very rapidly.

A pycnometer measurement is based on pressure measurements of a gas. **The thermodynamics of gases require exact temperatures to be maintained and special equipment for thermostatic control**. Not all instruments have this temperature control built in, but high-quality pycnometers should have it.

Devices **without electronic thermostatic control** must be operated in rooms with **a room temperature as consistent as possible**. This is important because otherwise there will be seasonal differences in measurement.

The ambient air pressure also fundamentally influences the measurement result of pycnometers.

The reason for this lies in the device technology of the valves and connecting lines. Pycnometers should therefore always be pressure compensated, so that they work equally well in different weather conditions (+/- 20...25 hPa), as well as at different geodetic heights (+4000 m altitude or sea level).

HANDLING

An often underestimated problem is the sample cup, which can warm up due to contact with fingers during handling processes between the scale and the pycnometer. Thin fabric gloves should therefore be worn if possible. **These additionally prevent contamination of the sample cup with e.g. greasy fingers**.

The sample can slip, get knocked over, and in the worst case fall to the floor.

However, because the surface of the sample cup (usually made of aluminum or stainless steel) is smooth, the gloves make handling more difficult. **The sample cup must be removed from the measuring cylinder by hand after the pycnometer measurement, and sits recessed in the measuring cell** or filler insert. This makes it necessary to be able to grip the cup by the inside lip. Most pycnometer manufacturers provide a small wire hook to help - the sample cup has holes at the top - for easier removal.

All in all, it takes familiarity and skill to handle the sample cup.

TARE WEIGHT OF THE SAMPLE CUP

Attention: If the scale is not electronically connected to the pycnometer, the mass of the sample must be entered manually. This creates potential for data transfer errors.

Many conventional pycnometers have a data interface, so that the data from the electronic analytical scale can be transferred directly. **Because the weight of the sample cup is irrelevant for the further measurement, it is set to 0 using the tare key of the scale.** In this way, only the weight of our sample is transmitted to the pycnometer.

To do this, the cleaned and empty cup is first placed in the scale.

Once the empty sample cup has been weighed and the tare weight of the scale has been zeroed, the cup can be removed from the scale and filled with the sample.

FILLING THE SAMPLE CUP

Caution: Unless the entire partial sample is placed in the sample cup, and if residues are simply wiped away, sampling errors will result.

For bulk materials, ensure that the entire quantity of the sample from the previous splitting process is placed in the cup.

Please also ensure that potential equalization takes place between the analytical scale and the pycnometer. It can happen that electrostatic charging “sprays out” samples from the sample cup during transport between the scale and the measuring device. This would result in a weighing loss that falsifies the measurement result.

A funnel makes this filling process easier. However, powders often have **problems with electrostatic charging** (triboelectric effect). Toner, powder coating and other extremely fine non-conductive plastic particles are particularly sensitive. The particles may “jump and spray” around in an uncontrolled manner, sticking to random locations, e.g. also the outside of the sample cup.

In any case, **adhering particles must be removed with a brush**. In addition, it must be ensured that **no particles adhere to the outside or bottom of the sample cup**. These would otherwise be carried over into the measuring cell. This problem could again occur when emptying the sample from the sample cup; care should be taken here to ensure appropriate cleaning.

Recommendation: The sample cup should be filled with the sample to 75% of volume, otherwise the proportion of gas volume in the measuring cell will be too large relative to the sample volume.




WEIGHING THE SAMPLE

To avoid the influence of air currents, always make sure that the doors of the high-precision analytical scale are closed again after opening and inserting the sample.

The sample cup and sample can now be weighed and - **after the settling time** - a key can be pressed to transfer the data to the pycnometer.

FILLING THE PYCNOMETER

To determine density, carefully insert the sample cup into the pycnometer. The size of the measuring cell in commercially available pycnometers is such that the largest of three possible sample cups fits directly into it. For smaller samples, there are two smaller sample cups available. **In order to minimize measurement errors resulting from excessive empty gas volume, two matching cylindrical filler inserts are provided for each of the smaller sample cups**, which should be inserted into the measuring cell before measurement. For instruments without temperature control, it should be ensured that the inserts are stored in the same place and can reach the same temperature as the pycnometer. Likewise important: The cover of the measuring cell should always remain mounted, and should also only be opened directly before inserting the sample cup, so that it also remains at the measuring temperature.

If different sample sizes are used, the inserts and corresponding sample cup sizes need to be changed frequently. At each change, calibration with the calibration sphere is required. This could easily be forgotten. Standard pycnometers do not automatically detect which insert / sample cup is currently being used.

The inserts should be inserted long enough before the measurement so that they can reach the measurement temperature before the measurement takes place. The temperature control of the inserts can take some time.

PYCNOMETRY WITH STANDARD GAS PYCNOMETERS

Attention: The cover has a significant influence on the quality of the measurement. In addition to the temperature errors already mentioned, an improperly fitted cover can lead to a measurement error because of changes in the measurement volume. Possible reasons for this: The cover is not properly screwed down as far as it will go (e.g. a dirty thread requires more force to turn) or there are particles between the cover and the measuring cell (contamination of the contact surface).

After opening the cover of the measuring cell, the sample cup can be inserted and the measuring cell can be closed tightly again. There are steel screw fasteners for the cover, which need to be turned multiple times to tighten them, until the direct contact between the steel surfaces of the cover and the measuring cell results in increased resistance. The contact surface between the measuring cell and the cover is open at the top, and must be clean and absolutely free of particles.

A cover with a bayonet lock has the advantage that it is tightened with a partial rotation. The rest position of the bayonet ensures that the cover cannot be overtightened with force. The geometry of the bayonet thus determines the axial closing motion of the cover and, provided the contact surfaces are clean, also the closing force. This is an advantage over threaded covers.

MEASUREMENT & DATA OUTPUT

The actual measuring routine of the pycnometer can now begin, the parameters for which have been defined and entered in advance. This also includes a unique sample reference number. Once the measurement is completed, a measurement report is provided - depending on the pycnometer model, e.g. on the PC- which can be printed out or saved in a database.

As it is necessary to concentrate on the measurement and the sequences of actions for the entire duration of the measurement process, no other activities can be performed simultaneously.

CLEANING

After use, the sample container and filling funnel are completely cleaned and freed from residues. An inspection of the cover and the contact surfaces of the pycnometer may also be useful.

This overview “Pycnometry in laboratories with standard pycnometers” was developed in cooperation with Prof. Dr. Ing. Dieter Claus Schwechten. Until his retirement, Professor Schwechten taught at the HTWG University of Konstanz, and regularly used gas pycnometers both professionally and as part of his teaching activities.

At a glance

Pycnometry in the laboratory = manual process

1. Selection of the right measuring gas
   generally helium
2. Selection of the appropriate sample container
   incl. insert & calibration, sample should fill it up to 75%
3. Set the tare weight of the specimen holder
   in the scale to 0
4. Fill sample container
   Caution: avoid loss of volume and electrostatic charging
5. Weigh sample
   avoid common weighing errors
6. Filling the gas pycnometer
   ensure that the cover is properly closed
7. Volume measurement & data output
8. Cleaning

LABORATORY TECHNICIAN

Is occupied for the duration of the measurement
Influences the duration & quality of the measurement process
can perform approx. 4 - 5 measurements per hour if trained / approx. 2- 3 measurements per hour if untrained

SCALE

To avoid transmission errors, use a device with electronic data transfer
Should ideally be located immediately adjacent to the gas pycnometer to keep transport distances short

PYCNOMETER

Conventional gas pycnometers vary greatly in their equipment; there are devices with membrane keypads or graphic control panels. Some have an integrated temperature control, others do not. In most cases, these are tabletop units of small size with a plastic housing. The measuring cell is closed by means of a screw or bayonet lock.

LABORATORY SETUP

The complete process of density determination takes place automatically inside the BULKINSPECTOR itself, spatial proximity to other devices is therefore not essential.

It only requires sufficient setup space. If you have decided on further automation with the **optional sample magazine**, additional space should be planned for this.

Since the BULKINSPECTOR itself is only 85.5 cm high, we can also supply **base frames** (with or without sound insulation).

If compressed air is not available at the installation site, a **low-noise compressor** is also available, which can be integrated e.g. into the base frame.

SELECTION OF THE RIGHT MEASURING GAS

It is important to use the right measuring gas. This should be selected based on the nature of the sample and the purpose of the measurement.

BEFORE COMMISSIONING

After switching on, the axes are referenced and it should be ensured that the **compressed air and helium supply are connected**. In order to achieve precise measurements, start the temperature control process and wait until the measurement temperature is reached.

SOFTWARE SETTINGS

For the measurements, the following **major parameter settings** can be adjusted: Measurement temperature, measuring cup size, measurement repetitions, measurement pressure, rinsing time, and the measurement accuracy desired from the measurements.

Apart from the measurement, a large number of other machine parameters can be individually configured.

To use the BULKINSPECTOR, you do not have to stand directly next to the machine. You can also use the tablet for remote operation, thus saving walking distances. For example: If you are working with the sample magazine, you can fill the magazine and adjust the settings on the spot.

THE SAMPLE | FILLING THE BULKINSPECTORS

The measuring cups should be **70-75%** filled with sample material.

When the sample magazine is connected, the pycnometer can analyze samples automatically for hours.

For this purpose, the sample cups are all removed and separately filled.

No manual intervention is then required after each sample.

Use the time for other activities, or perform the measurements outside laboratory hours!

The samples are also already temperature-controlled in the sample magazine, which leads to higher measurement precision.

Alternatively, you can of course also fill the BULKINSPECTOR individually by hand. To do this, the sample is placed in the filling device.

An insert is included to facilitate filling of the 10ml vessel.

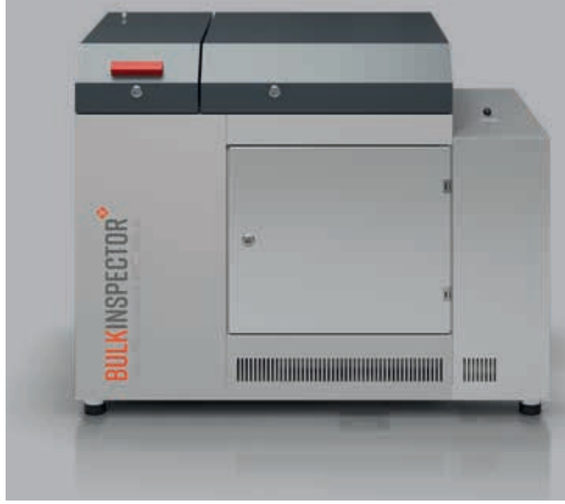
PYCNOMETRY WITH THE BULKINSPECTOR

TEMPERATURE CONTROL & AIR PRESSURE

The BULKINSPECTOR is a self-contained system. The interior of the device is tempered by means of Peltier elements. Neither the ambient air pressure (weather conditions (+/- 20...25 hPa) nor the geodetic altitude (+4000 m altitude or sea level)) has any influence on the measurement result.

MEASUREMENT & DATA OUTPUT

The software automatically saves the measurement results. These can be accessed via the app, saved to the network, or transmitted (Bluetooth, WLAN).



At a glance

BULKINSPECTOR = fully automatic

1. Setup & commissioning
2. Filling the pycnometer
   or sample magazine
3. Automatic density measurement
   tare weight of sample container, filling, weighing the sample, volume measurement, emptying & cleaning of sample containers

LABORATORY TECHNICIAN

Only fills the BULKINSPECTOR or optional sample magazine.
Empties the collection container as required.
As the handling & measurement are completely automatic, other tasks can be performed in the meantime. The measurement can also be performed outside laboratory hours.

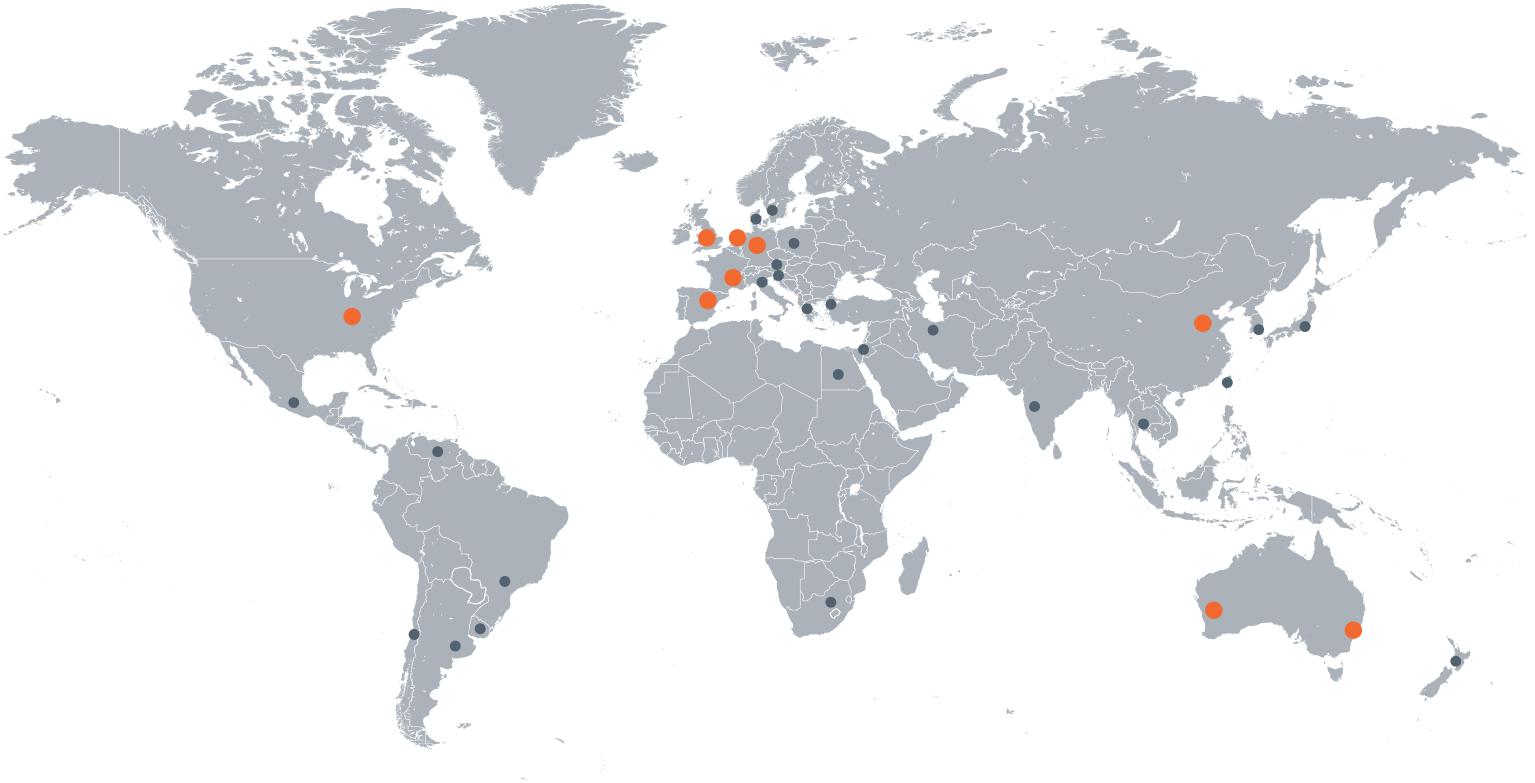
SCALE

The weighing process takes place automatically in the BULKINSPECTOR. Weighing & transmission errors can be excluded, as can contamination.

PYCNOMETER

Approx. 12 measurements per hour
Mobile operating unit, data transfer to external systems, optionally available sample magazine for 25/50 samples for automatic processing, temperature control of the interior, precision scale, steel housing, weight 190 kg, dimensions W1100 x D675 x H855 mm.

One Solution. Worldwide.



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