Flask Calibration & Use

Analyzing binder density in a ceramic slurry is essential in order to control the concentration of particles (i.e., SiO_2, polymers, surfactants) in the binder. If the concentration increases too much, it can promote particle collisions, which can lead to instability of the colloidal silica. Or, it can result in agglomeration or gelation of the SiO_2, which causes irreversible damage to the binder.

In any case, if the binder density is not held within the recommended specification, it will result in shells that are either too weak or too strong. For these reasons, it is very important to measure binder density in a repeatable, reliable and consistent manner.

In this tech tip, we will describe the calibration and use of volumetric and pycnometer flasks, both commonly used to measure binder density.

Equipment

Volumetric Flask

These affordable flasks are commonly used for density analysis in combination with a scale (up to 0.01 gram accurate). This method is sensitive to human error due to the visual interpretation of the liquid face (meniscus) against the fine reference line in the flask.

Pycnometer Flask

A pycnometer flask works similarly to the volumetric flask, but the visual interpretation is taken out of the equation due to the use of a drilled cap. Although more expensive, the results are typically more repeatable and consistent than the volumetric flask.

Flask Calibration

1. Place empty flask on scale (up to 0.01 gram accurate).
2. Tare scale.
3. Fill flask with deionized (DI) water, according to instructions (see sections below on using/filling volumetric or pycnometer flasks).
4. Weigh flask with water on scale.
5. The weight of the water (grams) equals the flask's volume (ml).
6. Label the flask with the volume that was obtained.

Calculations

- Flask volume (ml) = (weight full flask (gram) – weight empty flask (gram))
- Liquid density (g/ml) = (weight full flask (gram) – weight empty flask (gram)) / flask volume (ml)

Example Calculation

- Weight of water in 10 ml flask → 9.94 g = 9.94 ml volume of flask
- Weight of centrifuged binder in 10 ml flask → 11.50 g
- Divide weight of binder by volume of flask → 11.50 g ÷ 9.94 ml = 1.157 g/ml

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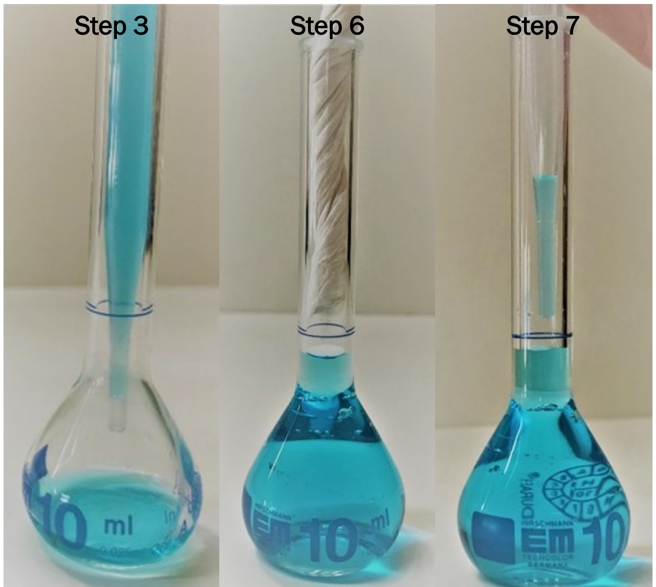
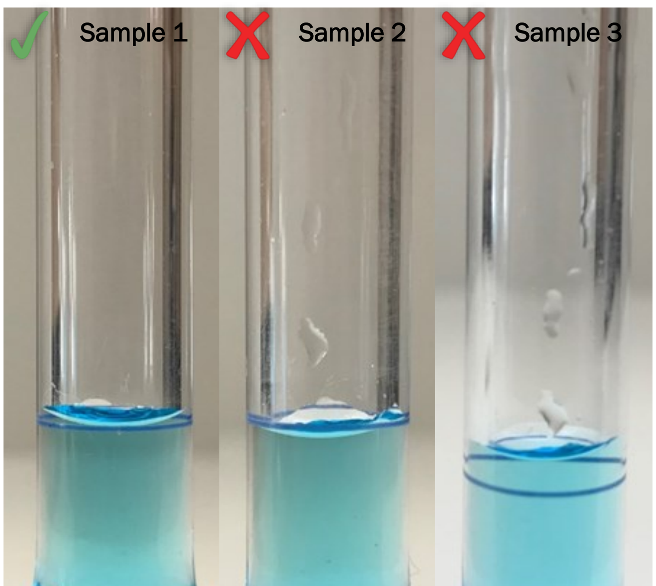


Using/Filling a Volumetric Flask

1. Secure a clean, dry volumetric flask.
2. Use a clean pipette to fill the flask.
3. Immerse the pipette under the flask's reference line before emptying.
4. Fill the flask just under the reference line.
5. Tap the flask to release entrapped air bubbles.
6. Clean off any liquid residue in the flask neck with paper/ tissue.
7. Complete filling the flask by carefully applying droplets of liquid until the bottom of the liquid meniscus touches the top end of the reference line.
8. Use paper/tissue again to clean any residue in the neck or to absorb the excess liquid out of the flask.



Results

<u>Sample 1 - Right</u>
There is no excess of liquid above the reference line. The bottom of the meniscus touches the top end of the reference line.

<u>Sample 2 - Wrong</u>
There is an excess of liquid above the reference line. The meniscus is below the reference line.

<u>Sample 3 - Wrong</u>
There is an excess of liquid above the reference line. The meniscus is above the reference line.

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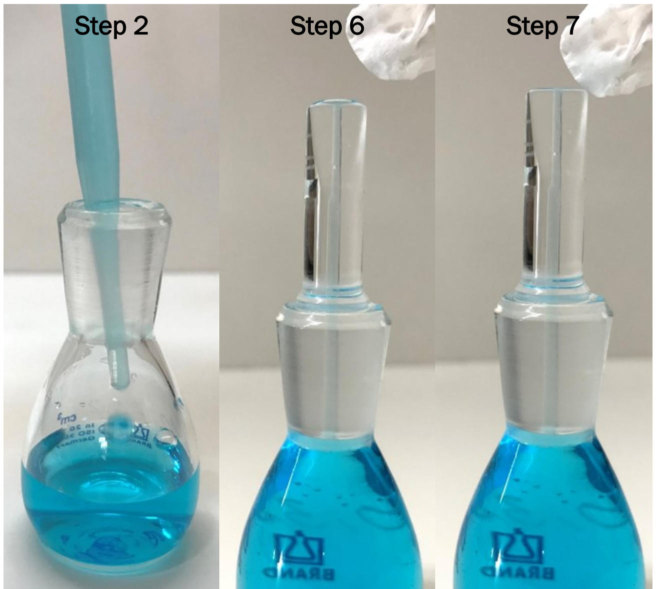
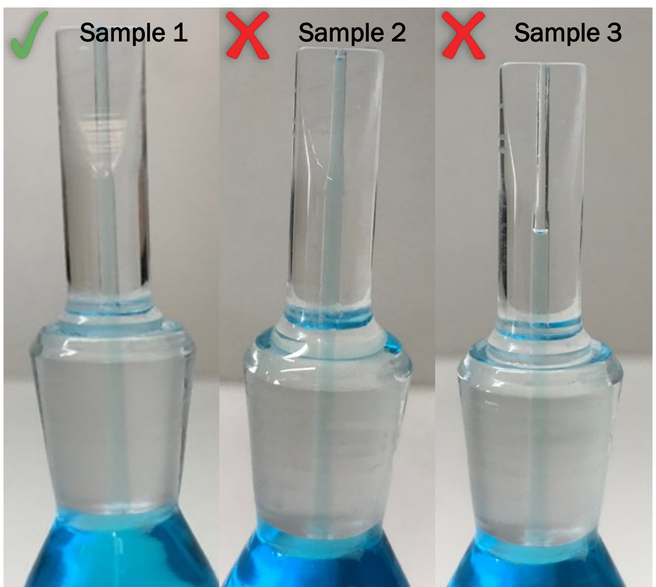


Using/Filling a Pycnometer Flask

1. Secure a clean, dry pycnometer flask.
2. Fill the pycnometer flask with a clean pipette until halfway to its neck.
3. Release entrapped air by taping the flask.
4. Apply the cap by dropping it gently into the neck.
5. Do not force the cap into the neck.
6. Carefully absorb any excess liquid from the flask and cap surface with tissue/paper.
7. Be careful not to absorb any liquid out of the cap bore.



Results

<u>Sample 1 - Right</u>
The cap bore is full. There is no excess of liquid on the flask surface.

<u>Sample 2 - Wrong</u>
There is excess liquid between the cap and the flask. Absent of liquid in cap bore (absorbed).

<u>Sample 3 - Wrong</u>
Absent of liquid in cap bore (absorbed).

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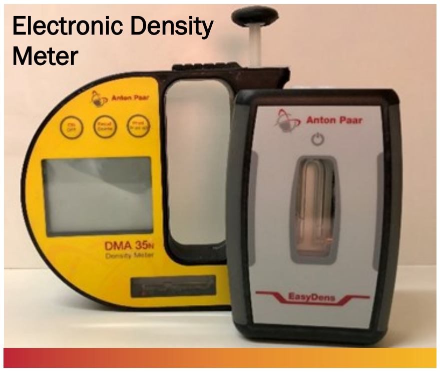

Important Tips

- Have multiple flasks available (i.e., every slurry has its own flask), as this will prevent having to clean a single flask between several measurements.
- Calibrate flasks before every use or register the flasks in a calibration system.
- Calibrate scales regularly with a reference weight.
- Rinse flasks immediately with DI water after every use.
- An electronic density meter can be used in place of volumetric or pycnometer flasks, as it reduces the risk of process failure to a minimum. After the binder is injected, the density is displayed up to 10^{-3} accurate. It is a proven method and highly recommended for repeatable and numerous analyses.