Anton Paar

Good Density Measurement™

Since 1967

Essential guidelines & helpful tips
for accurate density results

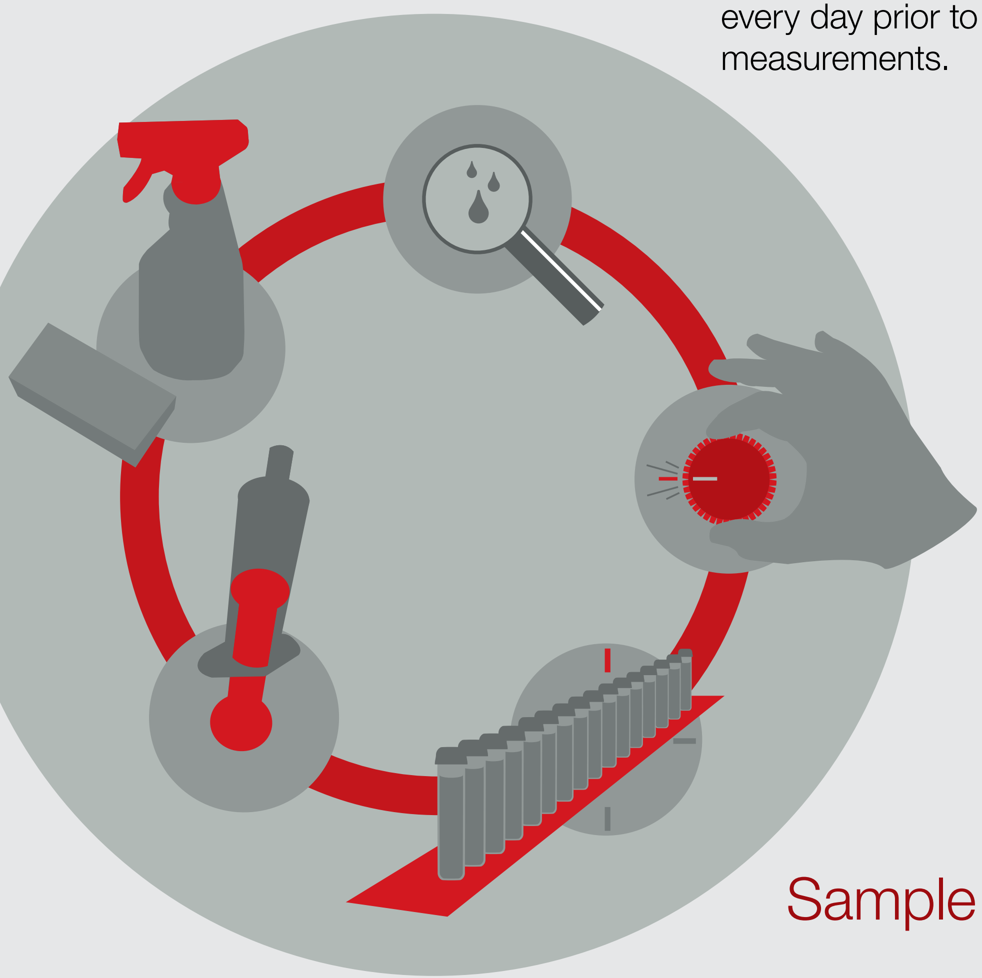
Prepared and published by Anton Paar GmbH

Since 1967 Anton Paar GmbH has specialized in providing highly accurate and reliable density meters for research and industry.

This brochure sums up our experience and insights into measurement practice gained in over forty years.

Good Density Measurement™ requires care and attention in five basic areas:

The water check, adjustment, sample preparation, sample filling, and cleaning. Follow these guidelines and you will be well on your way to accurate and reproducible density results.



Water Check

Perform a water check every day prior to your measurements.

Adjustment

If the water check fails and cleaning does not help, perform an air/water adjustment.

Sample Preparation

To get reproducible results prepare your samples with care and the same way every time.

Sample Filling

Fill the measuring cell carefully and without bubbles.

Cleaning

Remove the sample from the measuring cell right after the measurement, and clean your instrument regularly.

Water Check

If you carry out density checks at regular intervals you can ensure the high and stable accuracy of your density and concentration measurements.

Workflow

- Fill ultra-pure (e.g. bi-distilled or deionized), freshly degassed water into the measuring cell.
- Start a measurement.
- Compare the determined density with the reference value:
  $\rho_{Water} = 0.998203\ g/cm^3$ | for T = 20 °C

The water check fails if the measured density value is not within a required tolerance range. The tolerance range depends on the application and is more stringent in the pharmaceutical industry than in the soft drink industry, for instance.

Example:
A common tolerance limit for soft drinks is $\pm 1 \times 10^{-4}\ g/cm^3$. This means the water check is positive if the determined density is between 0.9981 g/cm³ and 0.9983 g/cm³.

If the water check fails, try the following:

- Use fresh ultra-pure water.
- Repeat the water check.

If the water check still fails:

- Clean the measuring cell thoroughly.
- Repeat the water check using fresh ultra-pure water.

If the water check still fails:

- Perform an air/water adjustment.

Perform a water check every day before your measurements.

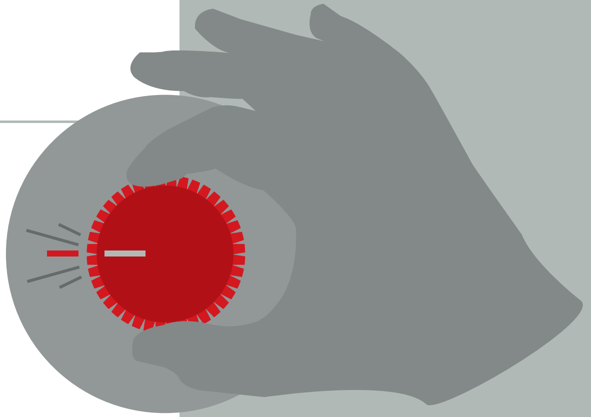
My water check:

The tolerance limit for my water check is ______________________________

____

____

Adjustment



An adjustment changes the instrument constants. For the consistency and comparability of results an adjustment should only be performed if the water check fails and using fresh water and cleaning the measuring cell does not help. Poor cleaning is the cause of the majority of measuring errors – an adjustment should be seen as a last resort.

Workflow

- Common adjustment media are dry air and ultra-pure (e.g. bi-distilled), freshly degassed water.
- Follow the automatic adjustment procedure on your instrument.
- Make a record in your adjustment logbook.

In the past it was recommended to perform an adjustment once a week, but this is no longer the case. Adjust your instrument only if it is out of range.

Reasons why the side effects of cleaning need to be compensated by an adjustment:

Cause	Effect	Influence on density
Aggressive cleaning	Volume of the measuring cell is increased	Artificial density decrease
Ineffective cleaning	Volume of the measuring cell is decreased	Aritificial density increase

If the water check fails and cleaning does not help, perform an air/ water adjustment.

My adjustment media are:

Adjustment medium 1 ______________________

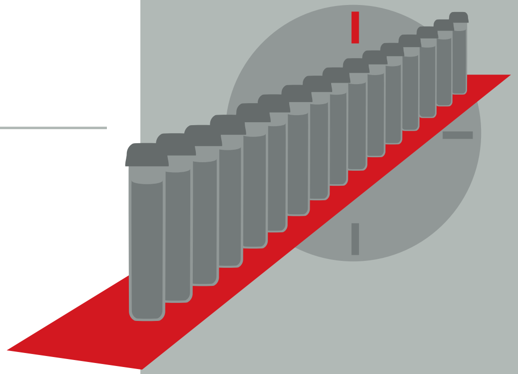
Density 1 ______________________

____

Adjustment medium 2 ______________________

Density 2 ______________________

Sample Preparation



Your sample contains gases.

There are different methods for the degassing of liquid samples. The best method for your application depends on the kind of sample, the kind of gas, and the amount of gas that is dissolved in the sample. Be aware of the fact that you may slightly change the composition of many samples during sample preparation due to evaporation of volatile components.

Stirring: Applicable for CO_2

- Stir your sample vigorously for 5 to 15 minutes until no more bubbling occurs.
- You can also pour the sample through a paper filter after stirring to get an even more efficient degassing effect.

- Do not boil flammable liquids because of a high risk of fire.
- If your sample contains volatile compounds that are toxic, always handle the sample in an appropriate environment, like a fume hood.

Ultrasonic bath: Applicable for air, but not for CO_2

- Put your sample into an ultrasonic bath for approximately 5 to 10 minutes until the bubble formation stops.

Boiling: Applicable for air, but not for CO_2

- Boil the liquid for several minutes to remove dissolved air.
- Fill a clean glass flask full with the boiled liquid and cover it.
- Wait until the liquid has cooled down to the approximate measuring temperature.

Your sample is aggressive.

- Observe all safety regulations regarding the handling of the samples, cleaning, rinsing and waste liquids (e.g. use of safety glasses, gloves, respiratory protection, etc.).
- Check the chemical resistance of all materials which come into contact with the sample before starting the measurement.

Your sample is viscous.

- Heat up your sample to get lower viscosities.
- For highly viscous samples use a heating attachment to prevent sample freezing in the sample inlet and outlet.
- If you use a sample filling system, check whether the viscosity corresponds to the given specifications.

Your sample is volatile.

- Close the sample vials with caps.
- Gently swirl the vials to bring condensed droplets back into the bulk liquid.
- For highly volatile samples use a sample filling unit which supports sample filling under pressure.

Contact your local Anton Paar representative to find out the best way of preparing your sample.

To get reproducible results prepare your samples with care and the same way every time.

My sample preparation:

Sample Filling

Fill the measuring cell carefully and without bubbles.

Automatic filling with sample filling units

Using sample filling units is the only way of eliminating filling errors due to the operator. As sample filling units repeat measurements in the same way every time, this is the best way to get repeatable results.
Even critical samples, like highly viscous samples or samples with volatile components, are filled smoothly. Some sample filling units additionally support automatic cleaning.

Workflow

- Fill your samples in the corresponding sample vials and prepare the magazine.
- If your sample unit supports automatic cleaning, make sure that sufficient cleaning liquid is available.
- Empty the waste container before starting a series of measurements.
- Check the instrument settings.
- Prepare the sample list for your instrument.
- Start the measurement.

⚠ Before filling any sample into the density meter, make sure that all wetted parts are resistant to it.

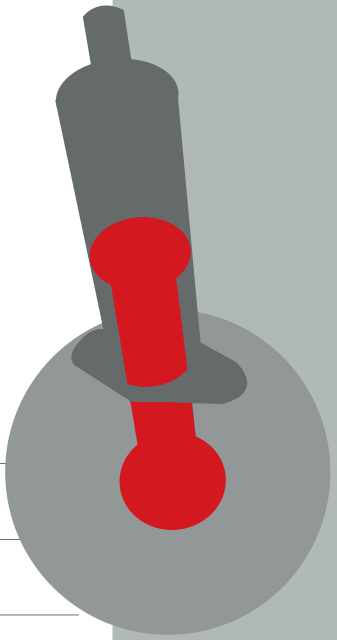
Manual filling with syringe

Using a syringe is the traditional way of filling a sample into a density meter. It requires some training to achieve repeatable results and to avoid bubbles in the measuring cell.

Workflow

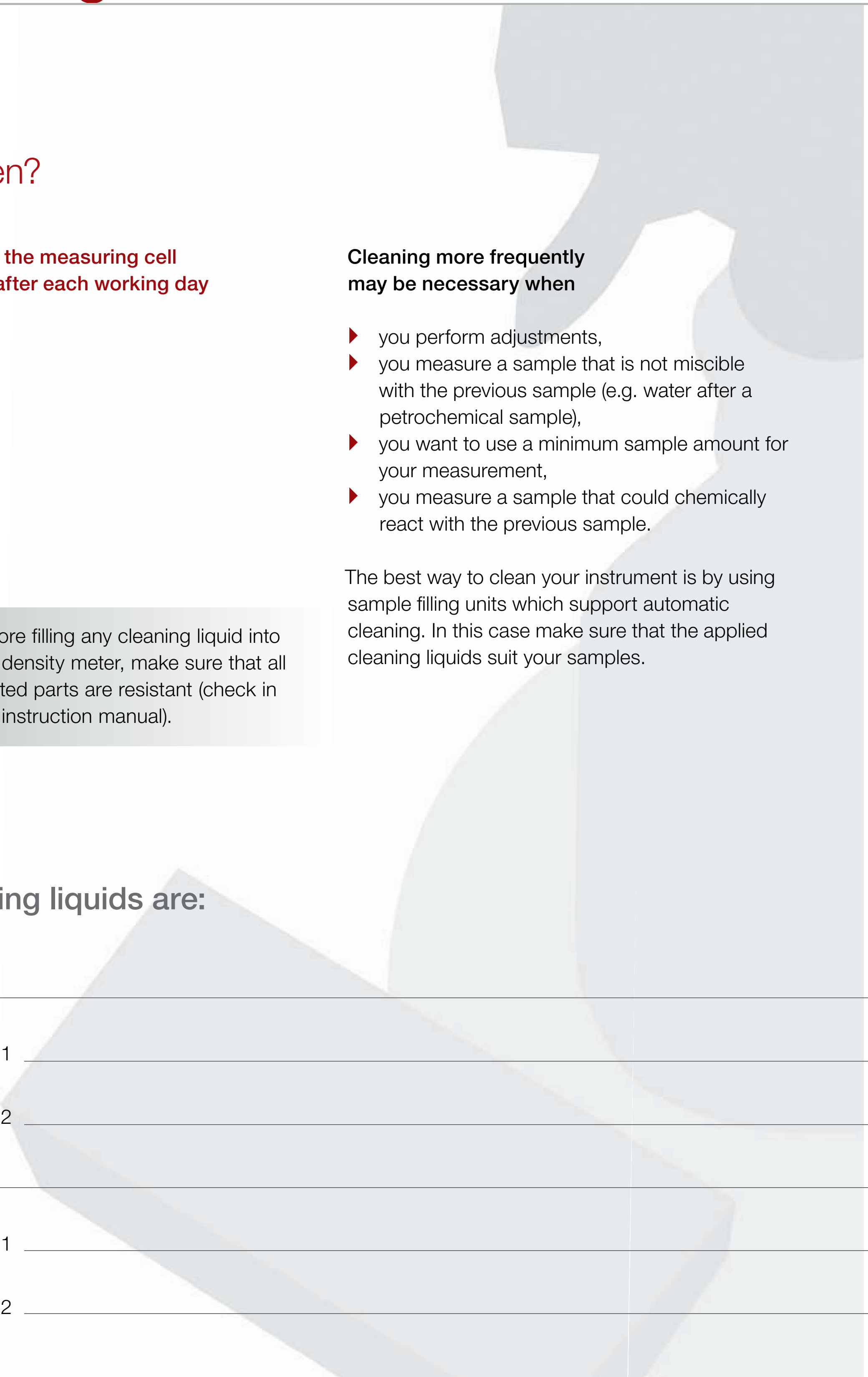
- Press the plunger smoothly and slowly without stopping.
- Check whether the measuring cell is filled without bubbles.
- Check the instrument settings.
- Start the measurement.

For paste-like materials, always use a syringe. If the samples have a very high viscosity, you can fill them into the syringe by pulling the plunger completely out of the syringe, filling it from the back using a spoon and then inserting the plunger again.



My sample filling:

Cleaning



How often?

Clean and dry the measuring cell at least once after each working day or work shift.

Before filling any cleaning liquid into the density meter, make sure that all wetted parts are resistant (check in the instruction manual).

Cleaning more frequently may be necessary when

- you perform adjustments,
- you measure a sample that is not miscible with the previous sample (e.g. water after a petrochemical sample),
- you want to use a minimum sample amount for your measurement,
- you measure a sample that could chemically react with the previous sample.

The best way to clean your instrument is by using sample filling units which support automatic cleaning. In this case make sure that the applied cleaning liquids suit your samples.

Workflow

- Clean the measuring cell using two cleaning liquids:
  - Cleaning liquid 1: Cleaning liquid 1 dissolves and removes sample residues in the measuring cell. It needs to be a good solvent for all sample components.
  - Cleaning liquid 2: Cleaning liquid 2 removes cleaning liquid 1 and is easily evaporated by a stream of dry air in order to accelerate drying of the cell. Cleaning liquid 2 needs to be a good solvent for cleaning liquid 1.
- Dry the measuring cell by activating the internal air pump.
- Check whether the cleaning and drying was successful by measuring the density of air (= air check).
- Compare the determined density with the reference value:
  $\rho_{\mathrm{Air}} = 0.001199\ \mathrm{g/cm^3}$ | for T = 20 °C, p = 1013 mbar

Remove the sample from the measuring cell immediately after the measurement, and clean your instrument regularly.

My cleaning liquids are:

Sample ____________________

Cleaning liquid 1 ____________________

Cleaning liquid 2 ____________________

Sample ____________________

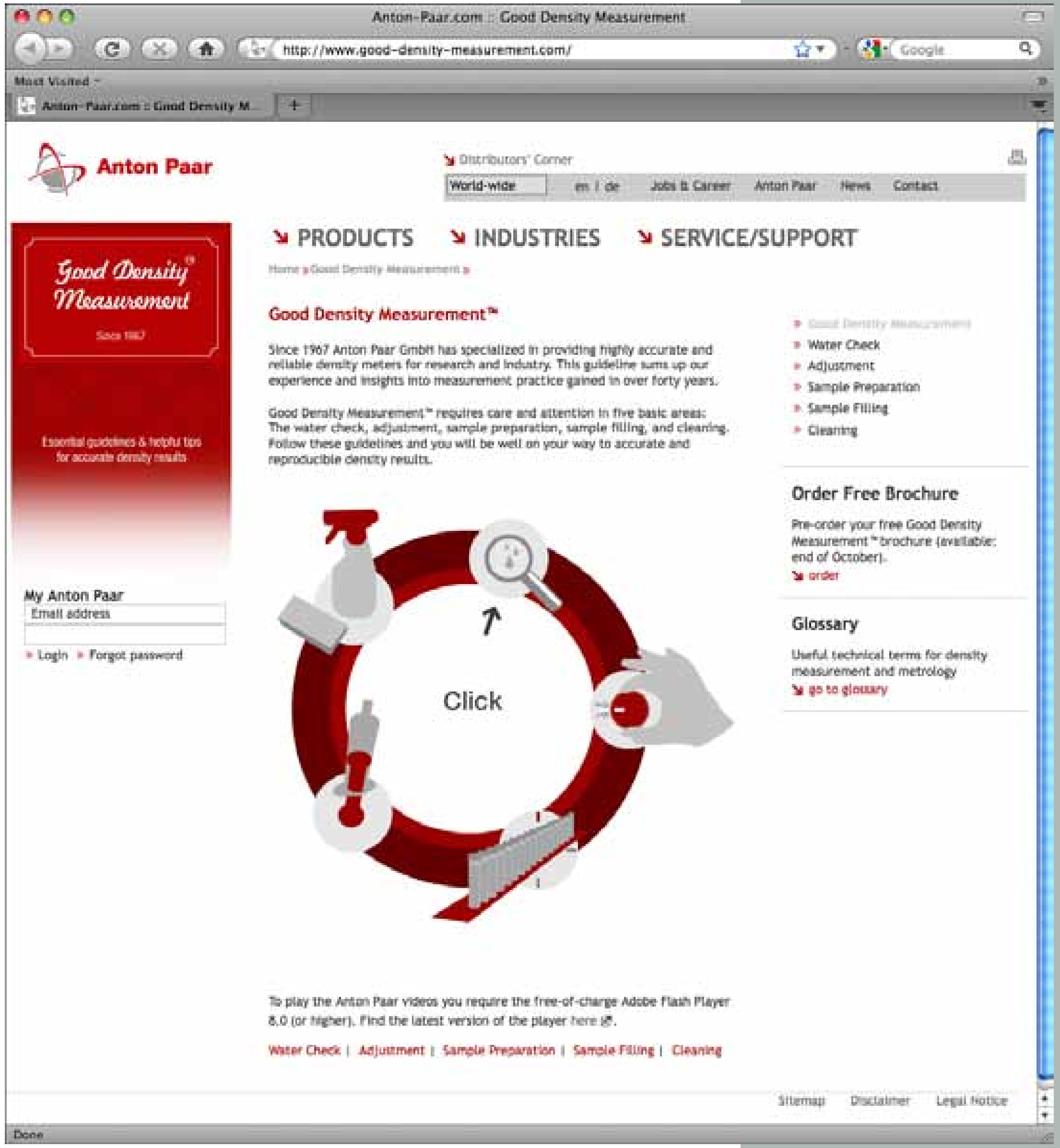
Cleaning liquid 1 ____________________

Cleaning liquid 2 ____________________

List of typical samples and suggested cleaning liquids:

Sample	Suggested cleaning liquid 1	Suggested cleaning liquid 2
After-shave, perfume	Alcohol	-
Beer	Water, enzymatic lab cleaner*	Alcohol
Beer wort	Water, enzymatic lab cleaner*	Alcohol
Fuel	Petroleum naphtha	Acetone
Liquid soap & detergent	Water	Alcohol
Lubricating oil	Petroleum naphtha	Acetone
Milk, cream	Water, enzymatic lab cleaner*	Alcohol
Motor oil	Petroleum naphtha	Acetone
Orange juice	Water	Alcohol
Schnapps	Alcohol	-
Soft drinks	Water	Alcohol
Salad dressing, mayonnaise	Petroleum naphtha	Alcohol
Shampoo	Water	Alcohol
Suntan lotion	Petroleum naphtha	Alcohol
Wood protection/white spirit-based (water-based)	Petroleum naphtha (water)	Alcohol

* Using enzymatic lab cleaner is recommended for special cleaning.
After using the lab cleaner you need to rinse the measuring cell with water.



Good Density Measurement™

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Good Density Measurement™ requires care and attention in five basic areas: The water check, adjustment, sample preparation, sample filling, and cleaning. Follow these guidelines and you will be well on your way to accurate and reproducible density results.

Order Free Brochure

Pre-order your free Good Density Measurement™ brochure (available: end of October).

Glossary

Useful technical terms for density measurement and metrology

To play the Anton Paar videos you require the free-of-charge Adobe Flash Player 8.0 (or higher). Find the latest version of the player here.

www.good-density-measurement.com

Glossary

Density (“True Density”)

The density ρ is defined as mass divided by volume:

$$\rho = \frac{m}{V}$$

ρ … density

m … mass

V … volume

The unit of density is kg/m³ or g/cm³.
1 g/cm³ = 1000 kg/m³.

Mass is independent of external conditions, such as buoyancy in air or gravity. It corresponds to weight in vacuo.

True density of liquids and gases is measured with the oscillating U-tube method.

The density of liquids and gases is highly temperature-dependent. Therefore, density measurement always requires accurate temperature measurement or control. Typically, density decreases with increasing temperature as the individual molecules require more space due to their thermal motion.

***Note:** Water is a unique liquid. The density maximum is reached at a temperature of 4 °C with ρ = 0.999972 g/cm³.*

Apparent Density

The apparent density ρ_{app} of a sample is defined as weight in air divided by volume:

$$\rho_{app} = \frac{W}{V}$$

ρ_{app} … apparent density

W … weight

V …volume

Units applied for apparent density are typically kg/m³ or g/cm³. Please note that the values of (true) density and apparent density differ. Apparent density is smaller than true density.

Apparent density can be calculated from true density by taking into account the buoyancy in air of the sample and the weight and density of a reference weight. Steel is nowadays defined as the material of choice for the weights. Earlier, brass was used.

$$\rho_{app} = \frac{\rho_{true,\ sample} - \rho_{air}}{1 - \dfrac{\rho_{air}}{\rho_{steel\ or\ brass}}}$$

ρ_{app} … apparent density

$\rho_{true,\ sample}$ … true density of sample

ρ_{air} … true density of air

$\rho_{steel\ or\ brass}$ … true density of brass or steel

- ρ_{brass} = 8.4 g/cm³
- ρ_{steel} = 8.0 g/cm³

Specific Gravity

The specific gravity SG (sometimes referred to as relative density D) is calculated by dividing the density of a sample ρ_{sample} by the density of pure water ρ_{water} at defined temperatures:

$$D^{20}/_4 = SG^{20}/_4 = \frac{\rho_{sample} \text{ at } 20\ °C}{\rho_{water} \text{ at } 4\ °C}$$

$D^{T1}/_{T2} = SG^{T1}/_{T2}$ … specific gravity related to defined temperatures

ρ_{sample} … density of sample

ρ_{water} … density of pure water

The specific gravity is dimensionless, which means it has no unit.

- At 4 °C: ρ_{water} = 0.999972 g/cm³
- At 20 °C: ρ_{water} = 0.998203 g/cm³

Apparent Specific Gravity

The apparent specific gravity SG_{app} (sometimes referred to as apparent relative density D_{app}) is calculated by dividing the apparent density of a sample $\rho_{app,\ sample}$ by the apparent density of pure water $\rho_{app,\ water}$ at defined temperatures:

$$D_{app}\ ^{20}/_{20} = SG_{app}\ ^{20}/_{20} = \frac{\rho_{app,\ sample} \text{ at } 20\ °C}{\rho_{app,\ water} \text{ at } 20\ °C}$$

$$D_{app}\ ^{20}/_{20} = \frac{\text{weight}_{\ sample+bottle} - \text{weight}_{\ bottle}}{\text{weight}_{\ water+bottle} - \text{weight}_{\ bottle}}$$

$D_{app}\ ^{T1}/_{T2} = SG_{app}\ ^{T1}/_{T2}$ … specific gravity related to defined temperatures

$\rho_{app,\ sample}$ … apparent density of sample

$\rho_{app,\ water}$ … apparent density of pure water

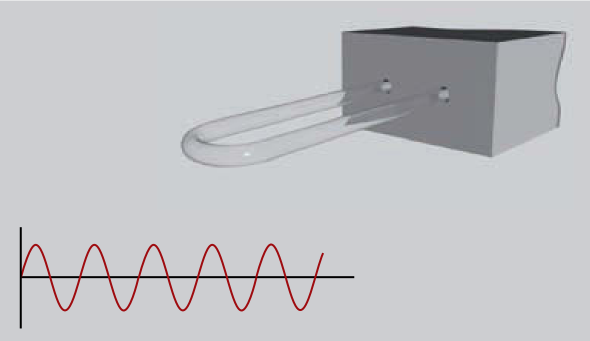
The apparent specific gravity is dimensionless, which means it has no unit.

Sample	Air at T = 20 °C, p = 1013 mbar	Water at T = 20 °C
Specific gravity $SG^{20}/_{20}$	0.00120	1
Specific gravity $SG^{20}/_4$	0.00120	0.99823
Apparent specific gravity $SG_{app}\,^{20}/_{20}$	0	1

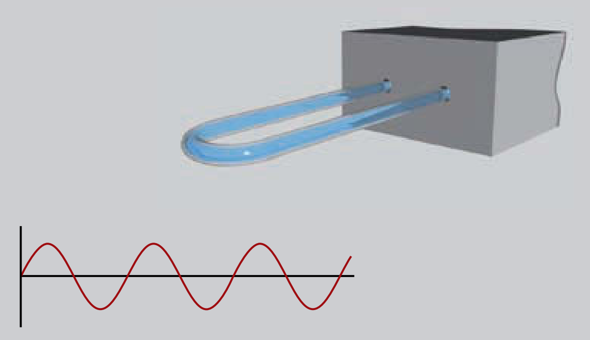
Glossary

Oscillating U-tube Method

The oscillating U-tube method is used to measure the true density of fluids. The sample is introduced into a U-shaped tube that is electronically excited to oscillate at its characteristic frequency. The characteristic frequency changes depending on the density of the sample. Via a precise determination of the characteristic frequency and an appropriate adjustment, the density of the sample is determined. Due to the high temperature dependency of density, the measuring cell has to be accurately thermostatted.



Oscillations of a U-tube filled with air



Oscillations of a U-tube filled with water

Modern high-precision density meters additionally provide:

- Viscosity correction: to enable accurate results over a large viscosity range,
- Reference oscillator: to enable accurate results over a large temperature range following just one adjustment.

Concentration Measurement

The concentration of a mixture of two components (binary mixture) can be determined by means of density measurement. Mixing two samples with known densities A and B gives a sample whose density lies between value A and B. The exact value depends on the mixing ratio and therefore on the concentration.

Concentration measurement is also possible for so-called quasi binary mixtures:

- Mixtures containing two major components. Some additional ingredients exist in small concentrations, but due to the small impact on the bulk density, these components can be ignored.

  Example: The major components of regular soft drinks are water and sugar. All other ingredients can be ignored when measuring the sugar concentration (°Brix).

- Mixtures containing several components, but only the amount of one component varies. All other ingredients are kept precisely constant.

  Example: For the production of infusions several base ingredients are precisely weighed according to a receipe. In a second step this mixture is diluted with water. The concentration can be controlled by means of density measurement.

Calibration

A calibration is the comparison of achieved measurement results with a standard reference value. A calibration is performed to validate the quality of measurements and adjustments. The standard reference value is given by the certificate of density standard liquids, for instance.

Recommendation: *1 to 2 calibrations should be performed per year with certified standards.*

Adjustment

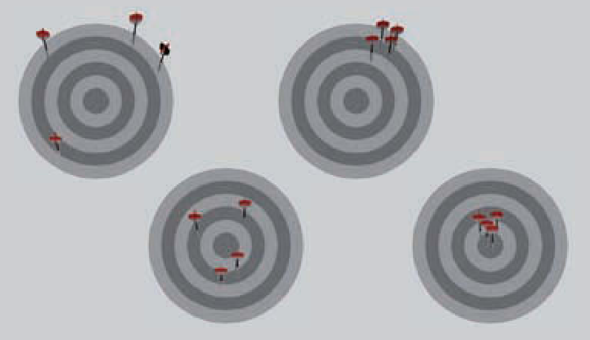
An adjustment is the modification of instrument constants to enable correct measurements and eliminate systematic measurement errors.
An adjustment is carried out after a calibration, unless the stated deviation is within the tolerance range.

For the adjustment the density meter uses the density values of the standards and the measured oscillation periods to calculate the instrument constants. Usually two standards are required for an adjustment, like dry air and pure (e.g. bi-distilled), freshly degassed water.

Accuracy

Accuracy expresses qualitatively how close the measurement result comes to the true value of a measurand.

In contrast, the quantitative measure of accuracy is uncertainty of measurement.



Accuracy and precision states (from left to right):
Not accurate, not precise
Accurate, not precise
Not accurate, precise
Accurate, precise

Precision

Precision expresses qualitatively how close the measurement results come to each other under given measurement conditions.

Precision can be stated under repeatability or reproducibility conditions.

Glossary

Uncertainty of Measurement

The uncertainty of a measurement specifies an interval within which the true value of the measurand is expected.

Uncertainty of measurement includes instrumental measurement uncertainty (arising from the measuring instrument), uncertainty of the calibration standards and uncertainty due to the measurement process (sample preparation, sample filling,…).

Uncertainty of measurement generally comprises many components:

- Some of these components may be evaluated from the statistical distribution of the results or series of measurements and can be characterized by standard deviations (Type A evaluation according to ISO/IEC Guide 98-3:2008).
- The other components, which can also be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information (Type B evaluation according to ISO/IEC Guide 98-3:2008).

Repeatability

The repeatability is the closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement.
Such ideal conditions lead to a minimum dispersion of measurement results.

The repeatability conditions are:

- The same measurement procedure
- The same operator
- The same measuring instrument, used under the same conditions
- The same location
- Repetition over a short period of time

Repeatability may be expressed with the repeatability standard deviation. This standard deviation is calculated from measurements carried out under repeatability conditions.

Reproducibility

Reproducibility is the closeness of the agreement between the results of measurements of the same measurand carried out under changed conditions of measurement.

Such conditions lead to a maximum dispersion of measurement results.

These reproducibility conditions may include:

- Measuring principle
- Measuring method
- Operator
- Measuring instrument
- Reference standard
- Location
- Conditions of use
- Time

The changed measurement conditions have to be stated.

Reproducibility may be expressed with the reproducibility standard deviation. This standard deviation is calculated from measurements carried out under defined reproducibility conditions.

Measurement Error

The measurement error is a measured quantity value minus a reference quantity value. Random measurement errors and systematic measurement errors can be distinguished.

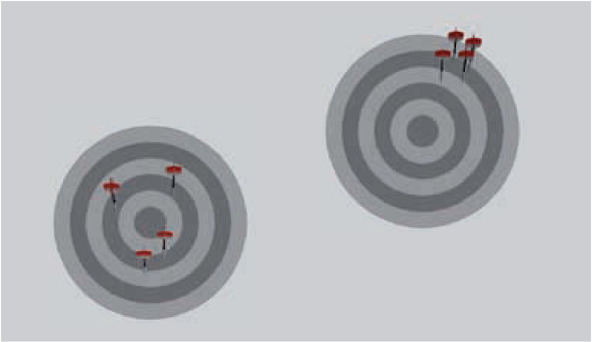
Random Measurement Error

The random measurement error is the component of a measurement error that varies in an unpredictable manner in replicated measurements. Many measurements have to be carried out to eliminate random measurement errors. The mean value of these measurements tends towards the true value.

Systematic Measurement Error

The systematic measurement error is the mean value that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions, minus the true value of the measurand.

Systematic measurement errors, and their causes, are either known or unknown. A correction can be applied to compensate for a known systematic measurement error.



Measurement errors (from left to right):
Random measurement error
Systematic measurement error

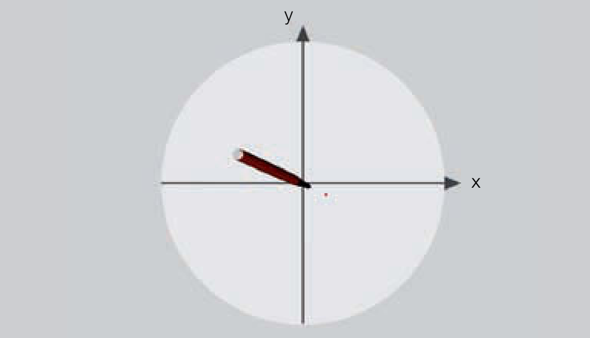
Glossary

Resolution

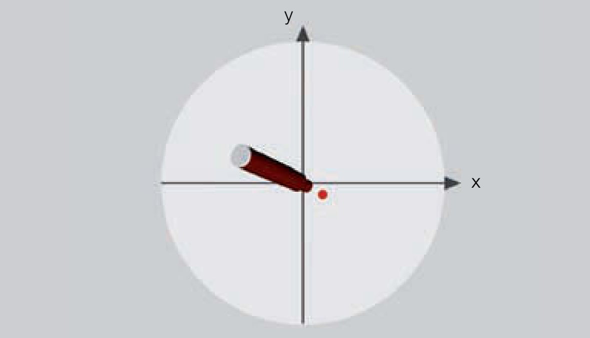
Resolution is the ability to resolve differences, i.e. to draw a distinction between two things. High resolution means being able to resolve small differences. In a digital system, resolution means the smallest increment or step that can be taken or seen. In an analog system, it means the smallest step or difference that can be reliably observed.

The most common mistake is the assumption that instruments with high resolution give more accurate results. High resolution does not necessarily mean high accuracy.

The accuracy of a system can never exceed its resolution!



Allegory for fine resolution:
With a fine marker it is possible to draw small dots.



Allegory for coarse resolution:
With a thick marker it is not possible to make fine drawings.

Arithmetic Mean Value

The arithmetic mean value x_0 is the sum of the measurement values divided by the number of measurements n:

$$x_0 = \frac{1}{n}\sum_{i=1}^{n} x_i = \frac{x_1 + x_2 + \ldots + x_n}{n}$$

x_0 … mean value
x_i … measurement value of the i^{th} measurement
n … number of measurements

The mean value does not give any information about the scattering of measurement results.

Example:
A series of density measurements gives the following results:
$x_1 = 0.998203$ g/cm³
$x_2 = 0.998203$ g/cm³
$x_3 = 0.998204$ g/cm³
$x_4 = 0.998203$ g/cm³
$x_5 = 0.998204$ g/cm³
$x_6 = 0.998205$ g/cm³

Arithmetic mean value (with n = 6):
$x_0 = 0.9982037$ g/cm³

***Tip:** In Microsoft Excel you can use the function AVERAGE(number1, number2, ...)*

Experimental Standard Deviation (s.d.)

For a series of n measurements of the same measurand, the experimental standard deviation s characterizes the dispersion of the results.
It is given by the formula:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - x_0)^2}$$

s … empirical standard deviation
n … number of measurements
x_i … measurement value of the i^{th} measurement
x_0 … arithmetic mean value

The mean value is often quoted along with the standard deviation. The mean value describes the central location of the data, the standard deviation describes the scattering.

The example from the arithmetic mean value is used to calculate the experimental standard deviation:

$x_0 = 0.9982037$ g/cm³
n = 6

$$s = \sqrt{\frac{(x_1-0.9982037)^2 + (x_2-0.9982037)^2 + \ldots + (x_6-0.9982037)^2}{5}}$$

$s = 0.00000008$ g/cm³

***Tip:** In Microsoft Excel you can use the function STDEV(number1, number2,...)*

All in all, what makes for Good Density Measurement™?

Awareness of the entire measurement workflow, from start to finish – from the moment you start working in your lab to the moment you are cleaning your equipment. Just keep your eyes trained on the five basic areas – and you're well on your way …

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Our product range

Density, concentration and temperature for lab and process applications
- Fluid density and concentration meters
- Beverage analyzers
- Alcohol meters
- CO_2 meters
- High-precision thermometers

Rheometry and viscometry
- Rotational and oscillatory rheometers
- Capillary viscometers
- Stabinger viscometers

Chemical and analytical techniques
- Sample preparation
- Microwave synthesis

Material characterization
- X-ray structure analysis
- Colloid science

Refractometry and Polarimetry
- Refractometers and polarimeters
- Sugar colorimeters

Subcontracting
- Precision mechanics
- Mechanical/electronic subassemblies

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