MAGNETIC SUSPENSION BALANCES

RUBOTHERM
PRÄZISIONSMESSTECHNIK GMBH

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Contents

Current information about new developments and activities as well as a list of Rubotherm's international distributors can be found on the internet at www.rubotherm.de.

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Rubotherm – your specialist for gravimetric analysis

Rubotherm Präzisionsmesstechnik develops, produces and sells measuring instruments and laboratory plants for process engineering.

The main emphasis of our work is placed on gravimetric measuring methods for recording mass changes under controlled environments.

In the 1980's the company founders from the Ruhr-Universität Bochum developed a new method whereby samples could be weighed in closed vessels without any contact. The principle is as follows: a sample is weighed by means of a new kind of magnetic suspension balance (which has been patented worldwide) from the outside and the suspension force is contactlessly transmitted from the pressurised measuring cell to a microbalance at ambient atmosphere. This means that mass changes of a sample can be recorded even under extreme conditions with the utmost accuracy.

Since the mid 80's these magnetic suspension balances have been applied to fundamental research work with enormous success.

Numerous requests, including many from industry, induced the developers to set up the company Rubotherm Präzisionsmesstechnik GmbH in 1990, located in the Ruhr Technology Centre in Bochum. Our weighing technology has since then been enjoying an even wider circle of application throughout the world in both research and industry.

Magnetic suspension balances – The renaissance of gravimetry

Far up until at least the middle of the last century gravimetry played a predominant role in the investigation of physical and chemical processes.

The decline of gravimetric measuring technology which has taken place since then results from two circumstances. The first one was due to modern spectroscopy. These instruments with their extremely high resolution have more or less taken the place of gravimetry for qualitative analysis. The second one we find in the fact that none of the gravimetric instruments developed up to the 1980's were really suitable for general use under controlled environments.

Only the development of new and reliable magnetic suspension balances in the late 80's with an extremely wide application range changed this situation. Now, mass changes can be recorded under nearly all conditions with utmost precision.

As a result all restrictions which prevented a broad application of gravimetry in the past have been eliminated. This is all the more important as none of the modern spectroscopic methods can achieve anywhere near the accuracy of gravimetry in the case of quantitative measurement.

We are talking about a difference of several decades in relative accuracy. Once being freed of its restricting bonds gravimetry can make an excellent contribution to the direct investigation of physical / chemical effects under process conditions and – coupled with spectroscopic instruments – for the quantitative connection of qualitative analytical data.

Apart from numerous special applications, three main areas have become apparent over the course of time:

- sorption measurements = material transport
- thermogravimetrics = investigation of chemical reactions
- density measurements = state quantities of fluids / density of porous material

Apparatus for the simultaneous measurement of viscosity and density at the Chair of Thermodynamics at the University of Bochum

Portable densitometer to calibrate vibrating gas density transducers in natural gas pipelines, Chair of Thermodynamics, University of Bochum

Cooperating partners

New developments of our measuring instruments are the direct result of suggestions from our distributors and customers in close cooperation with many research institutes both in Germany and abroad.

Those institutes in Germany which we would particularly like to mention are: the Chairs of Thermodynamics in Bochum and Siegen, the Chair of Separation Processes in Bochum, the Chair of Process engineering II at the Technical University Hamburg and the Institute for Non-Classical Chemistry in Leipzig.

Our Japanese partner company BEL Japan Inc. is also of particular importance since we are jointly developing new measuring techniques in our core area of sorption and thermogravimetrics.

Apparatus for the combined volumetric-gravimetric measurement of co-adsorption equilibria of binary mixtures. Development made in co-operation between Rubotherm and BEL Japan Inc.

BEL Japan Inc.

Magnetic suspension balances – weighing with utmost precision under extreme conditions

Magnetic suspension balances are able to weigh where all others fail.

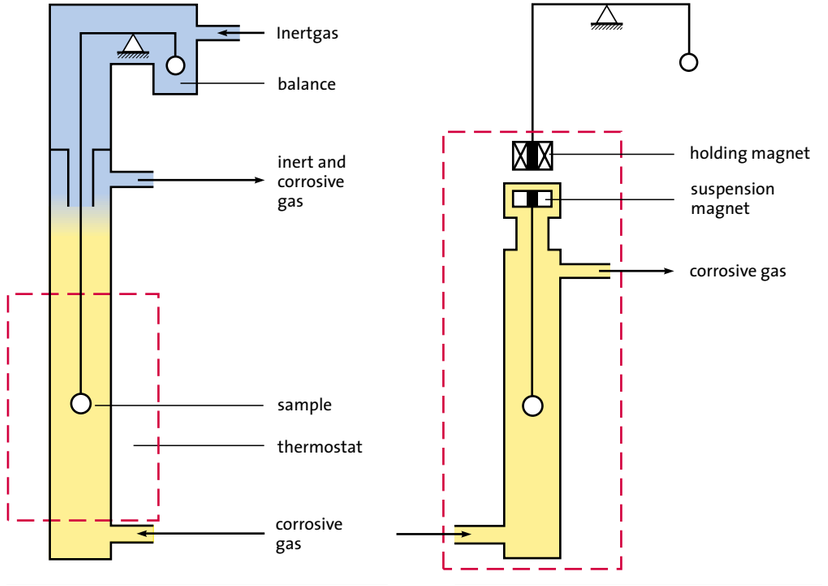
Magnetic suspension balances allow the changes in force and mass which act on samples under controlled environments (pressure, temperature, corrosive gases or fluids), to be measured with high accuracy. By means of these measurements it is possible to determine transport quantities and state quantities very easily and accurately (sorption, diffusion, surface tension, density), chemical reactions can be investigated (corrosion, decomposition, combustion etc.) or production processes can be simulated (polymerization, coating, drying etc.)

Application

In addition to fundamental research work and material testing these measuring methods can be applied to many processes in order to design or optimise a great number of industrial operations, for example:

- exhaust gas cleaning
- gasification of coal
- soil detoxification
- waste incineration
- foodstuff manufacturing
- purification and storage of natural gas and hydrogen
- production of plastic
- supercritical fluid extraction
- material synthesis

Gravimetric measurements in controlled environments



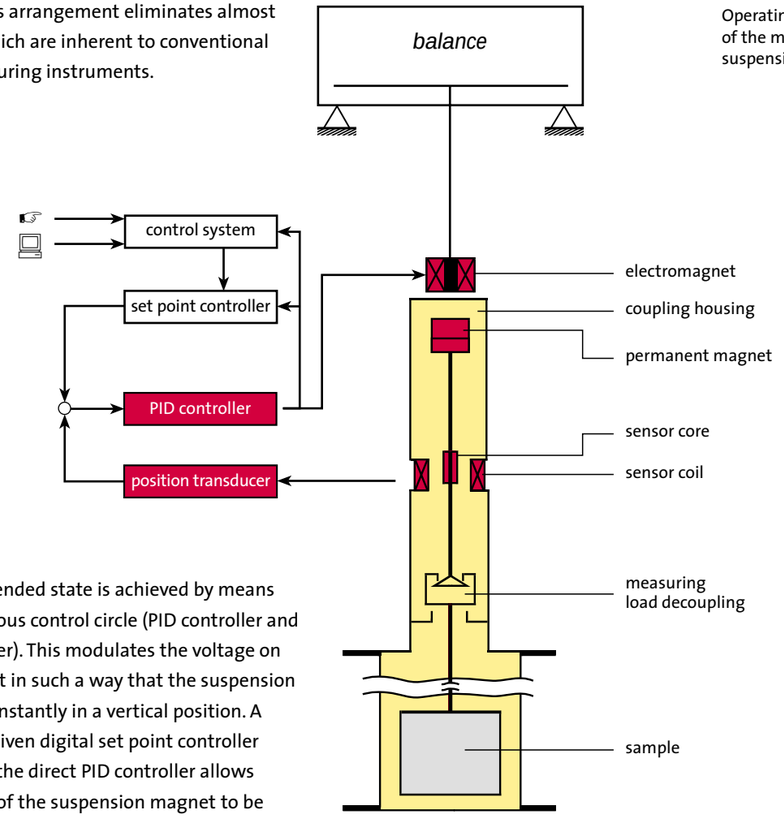
Conventional apparatus | Magnetic suspension balance

Conventional measuring apparatus

The main difficulty when using conventional gravimetric instruments is the direct contact between the measuring cell (sample atmosphere) and the weighing instrument. The balance can be damaged or disturbed by the measuring atmosphere and the measuring atmosphere can be adversely affected by flushing gases and pollution. These limitations considerably reduce the field of application of conventional measuring devices.

Magnetic suspension balances

These new, reliable suspension balances make it possible to weigh samples contactlessly under nearly all environments. Instead of hanging directly at the balance the sample to be investigated is linked to a so-called suspension magnet which consists of a permanent magnet, a sensor core and a device for decoupling the measuring load (sample). An electromagnet, which is attached to the underfloor weighing hook of a balance, maintains a freely suspended state of the suspension magnet via an electronic control unit. Using this magnetic suspension coupling the measuring force is transmitted contactlessly from the measuring chamber to the microbalance, which is located outside the chamber under ambient atmospheric conditions.
Consequently, this arrangement eliminates almost all restrictions which are inherent to conventional gravimetric measuring instruments.



Operating principle of the magnetic suspension balance

A controlled suspended state is achieved by means of a direct analogous control circle (PID controller and position transducer). This modulates the voltage on the electromagnet in such a way that the suspension magnet is held constantly in a vertical position. A microcontroller driven digital set point controller superimposed to the direct PID controller allows various positions of the suspension magnet to be set up.

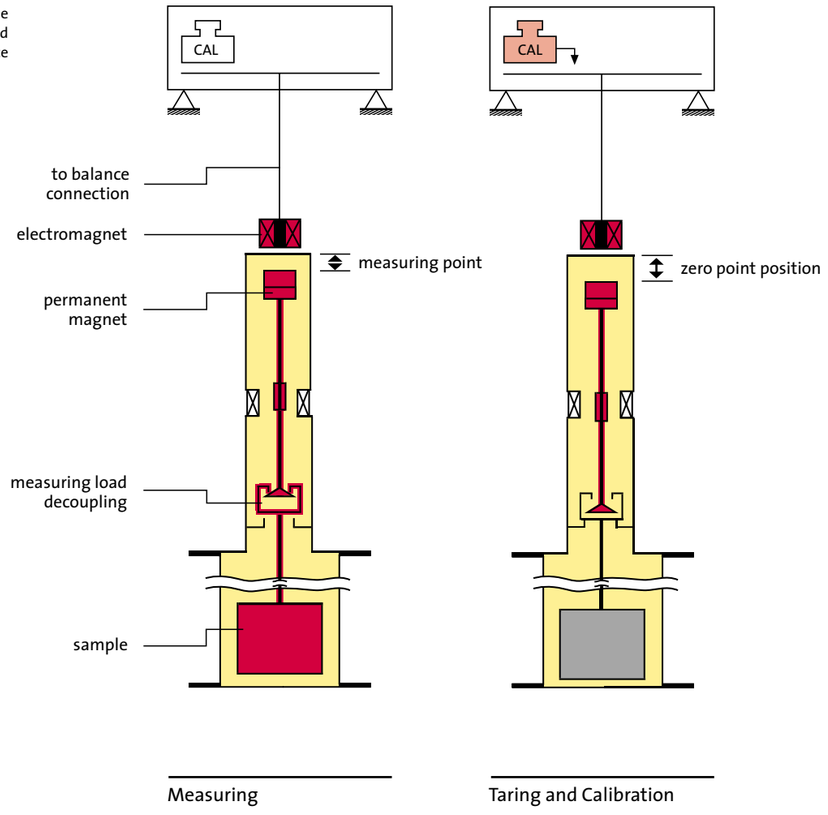
Automatic decoupling of the measuring load – utmost measuring accuracy

Modern, high resolution balances can be tared and calibrated automatically; a precondition for this correction of zero point and sensitivity drifts is an unloaded balance, that is to say, a decoupled measuring load.

The magnetic suspension balance offers the possibility of lowering the suspension magnet in a controlled way to a second stationary position a few millimetres below the measuring position. Then, a small carrier to which the sample is connected is set down on a support. Now the sample is decoupled from the balance. The suspension magnet alone is in a freely suspended state and only its weight is now transmitted to the balance. This so-called zero point position, which corresponds to an empty balance pan in a normal weighing procedure, allows a taring and calibration of the balance at all times, even when recording measurements under process conditions in the measuring cell (pressure, temperature).

This unique option of the magnetic suspension coupling increases the measuring accuracy to levels not previously known, particularly in the case of long term measurements.

Automatic decoupling of the measuring load in order to tare and calibrate the balance



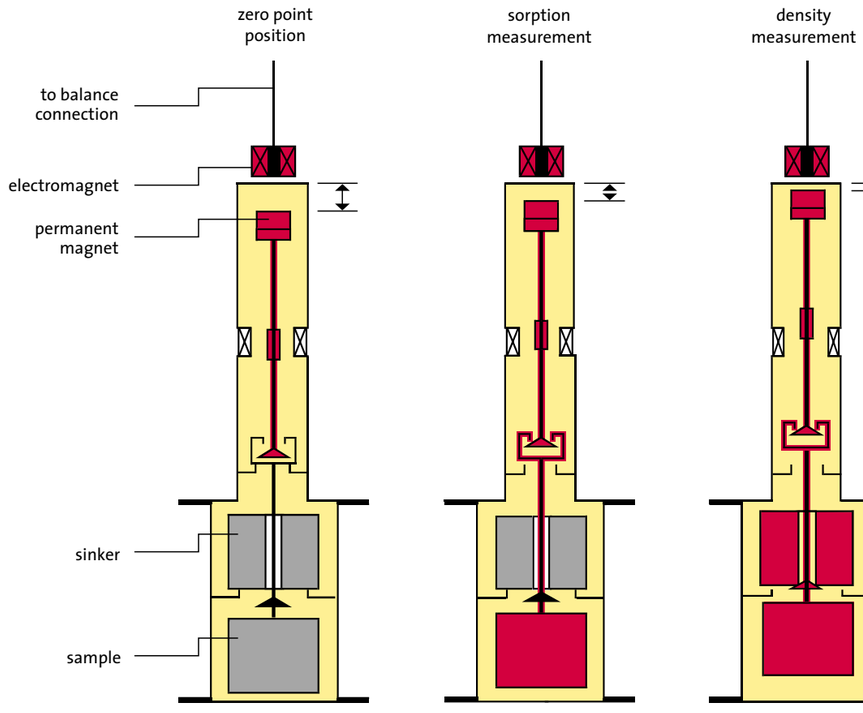
Simultaneous weighing of two samples with one magnetic suspension balance

A resulting further development of being able to decouple the measuring load is the possibility to measure the mass change of two samples with only one magnetic suspension balance. In addition to the first measuring load decoupling a second is arranged in the magnetic suspension coupling. Three different vertical positions of the suspension magnet, which can be arrived at in a controlled way, correspond to three different measuring positions:

Zero point: The permanent magnet alone is in a freely suspended state, allowing the balance to be tared and calibrated.
Measuring point 1: The first sample is lifted up and its mass is weighed.
Measuring point 2: The second sample is raised with the first and both masses are weighed together. By subtracting the first measuring point value from the second the mass of the second sample is given.

Application

Simultaneous sorption and density measuring as well as comparing measurements of two samples. Details regarding this measuring process can be found on page 20 of this brochure and also in the application catalog dealing with our new sorption analysers.



Example:
Simultaneous measurement of sorption and density

The new magnetic suspension balance – modular set up, diverse applications

Metal version from UHV up to 2000 bar

Many years of research and development work resulted in various types of suspension balances with very different working and measuring ranges. Based on these experiences, a standard measuring instrument has been developed whose different versions cover most applications. Standard suspension balances are being successfully used worldwide for a variety of gravimetric measuring tasks. This includes sorption and solubility investigations as well as thermogravimetric and density measurements of fluids.

The suspension balance enables samples to be weighed under almost all measuring conditions (eg vacuum, gas, fluid). The working range extends from ultra high vacuum to 2000 bar and from -200°C to +2000°C, also in corrosive and toxic media and is therefore far superior to the conventional gravimetric measuring apparatus available.

The standard magnetic suspension balance distinguishes itself by having a high resolution weighing cell integrated into the instrument. Thus, it was possible to achieve an exceedingly compact and robust device. In addition, the suspension control, which is microcontroller driven, makes it very easy to handle for the user, while at the same time the greatest reliability and accuracy are assured.

As a result, the setting up, maintenance and use of this high tech weighing instrument can be easily compared to that of a common analytical balance. The standard models are able to measure:

- weight changes of samples up to 80 g in mass with a resolution of 0.01 mg and a reproducibility of 0.02 mg.
- weight changes of samples up to 10 g in mass with a resolution of 1 µg and a reproducibility of 2 µg.

The suspension coupling is thermostated with a circulating fluid using a double tube thermostat. To connect the measuring cell the coupling housing is equipped with a flange corresponding to requirements.

Glass version for measuring in highly corrosive media

The standard suspension balance is available in glass for taking gravimetric measurements in highly corrosive media. All parts within the measuring cell (suspension magnet, measuring load coupling, and sample crucible) are completely fused in glass or are even made out of glass. All functions such as decoupling the measuring load whilst measuring are realized in the same way as with the metal version. Likewise, the measuring accuracy and resolution as well as the sample mass cover the same range. The pressure range however is limited as a result of the material. It ranges from vacuum to 1.3 bar. Depending on the application, the glass parts are made of either pyrex or quartz glass.

Thermostating

In the case of the metal version covering a range up to 350°C, both the suspension coupling area and also the measuring cell are thermostated with a circulating fluid using a double tube thermostat. With the glass version, numerous parts (coupling housing and measuring cell) are double sided and can be thermostated up to 250°C by means of a circulating fluid thermostat. Up to this temperature the suspension coupling and measuring cell form a thermic unit and a gradient free temperature level is achieved at all points.

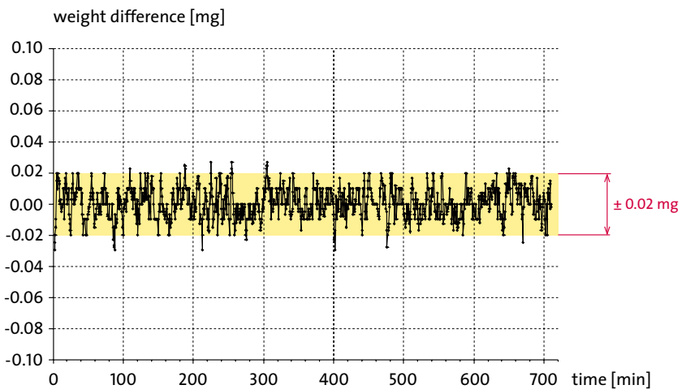
Should it be necessary that higher sample temperatures have to be carried out the measuring cell is then electrically heated. Consequently, temperatures up to 900°C for glass and 2000°C (low pressure metal version with ceramic cell) can be reached. The area for magnetic force transmission can still be thermostated up to 250°C. This temperature is normally sufficient to prevent the measuring fluid from condensing or reaction products from being precipitated.

Technical data and measuring accuracy

Application range for standard versions of the magnetic suspension balance

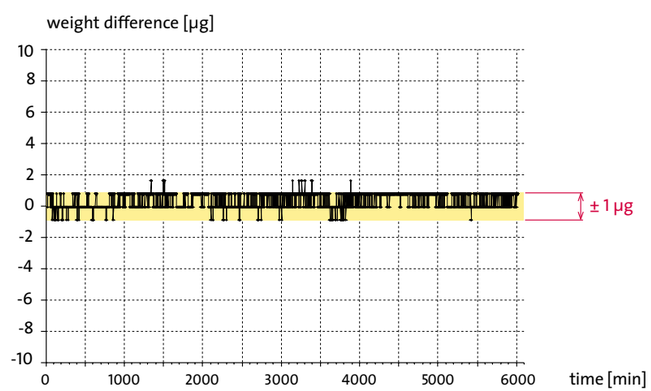
	metal version	glass version
pressure range	UHV ... 500 bar	vacuum ... 1.3 bar
temperature range (total measuring volume, ie. suspension coupling and measuring cell with sample)	-196 °C ... 350 °C	-60 °C ... 250 °C
by thermal decoupling (sample temperature)	up to 2000 °C (with ceramic measuring cell)	up to 900 °C
resolution	0.01 mg to 1 µg	
reproducibility (standard deviations)	±0.02 mg to ±2 µg	
relative error	⩽ 0.002 % of measured value	
maximum load	80 g or 10 g	

Special versions of the magnetic suspension balance considerably extend the measuring range given here. For more details see pages 20 - 24.



Weighing of a 20 g stainless steel calibrated weight in vacuum for 12 hours using a standard magnetic suspension balance with a resolution of 0.01 mg. (Measured deviations have been applied to the starting value in mg.)

The accuracy of the suspension balances is higher than that which can be achieved by manual weighing with a commercial balance alone. This results from an optimum control of the free suspension state, the gentle coupling and decoupling of the measuring load by the suspension coupling itself, the zero point correction and the calibration of the balances. The latter leads to a measuring accuracy with a 2 digit reproducibility which can hardly be bettered, particularly when long term measurements are carried out.

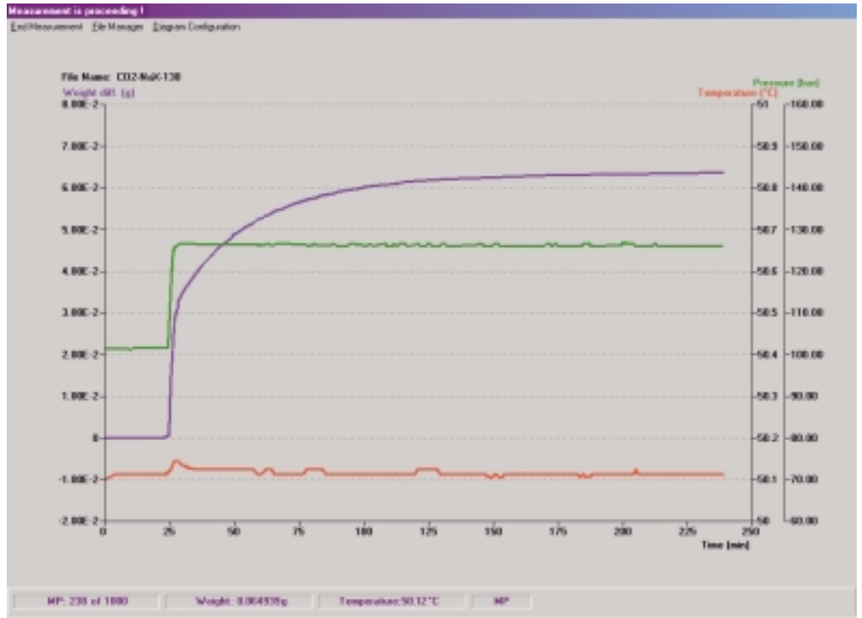


Weighing of a 3 g stainless steel calibrated weight in vacuum for 100 hours using a standard magnetic suspension balance with a resolution of 1 µg. (Measured deviations have been applied to the starting value in µg.)

Software for data acquisition

As a result of our long experience in producing and selling magnetic suspension balances we have developed software for data acquisition which can be universally applied. The software is used to control the measuring process (zero point – measuring point – switch, calibration, t-change, p-change) and to record data.

Its foremost advantages are its versatility, adaptability and modular design. The recording and on-line presentation of the data measured are stored in an ASCII format and are therefore available for evaluation on spreadsheet software.



Recording of mass change (blue), temperature (red) and pressure (green) using Rubotherm's MessPro software.

In addition to the mass values shown here the results from connected instruments (eg mass spectrometer) can also be displayed and stored.

The software enables the automatic run of several measuring sequences.

For adsorption measurements the measuring process can be monitored to see if the equilibrium has been reached. This is done by means of equilibrium criteria (mass change, pressure and temperature change) which are freely defined by the user. Once the equilibrium has been established the next point to be measured is achieved by means of the automatic gas dosing system and temperature control. Details concerning automated experimental systems are presented in the application brochures dealing with our sorption, thermogravimetric and density analyzers.

Magnetic suspension balances – well thought out and effective in every detail

Example of force transmission without contact using a cutaway model of a metal magnetic suspension balance

Sorption and density measurement with a magnetic suspension balance (sample basket and titanium sinker)

Suspension coupling housing with flow guide tube in a metal version for high pressure applications with two measuring gas connections.

Metal magnetic suspension balance with double-walled thermostation jacket and a measuring cell for high pressure applications eg. material transport measurements.

Sample crucibles for various applications and samples

Double walled jacket for thermostating a high pressure measuring cell with a circulating fluid

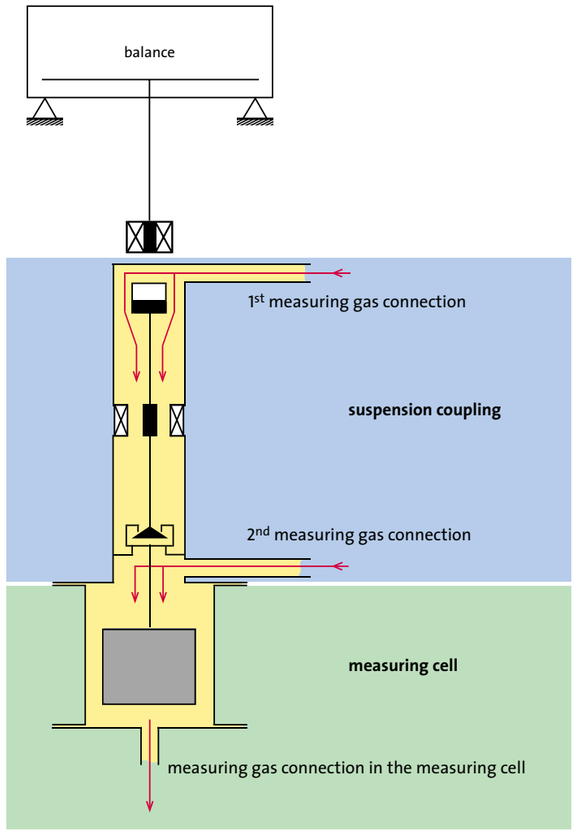
Measuring gas connections

Both metal and glass versions of the magnetic suspension balance can be filled or flushed, depending on application, by up to three measuring gas connections.

One measuring gas connection is so applied that the entire inner volume of the coupling is flushed free of dead volumes. With the metal version the flushing of the permanent magnet used is achieved by means of a flow guide tube. In the case of the glass version, the measuring gas connection is placed directly above the permanent magnet. This flushing free of dead volumes is particularly useful for pumping measuring fluid mixtures in order to avoid having differences in concentration in the measuring room.

A further measuring gas connection is in the coupling housing flange, or in the case of the glass version, in the bottom part of the glass coupling housing. This is used to feed large volume gas flows to the sample. Therefore, the permanent magnet and the measuring load decoupling remain more or less untouched by the flow which leads to a clear reduction in noise level caused by high flow rates. Both gas connections can either be used singly or together.

Always a gas connection at the bottom of the measuring cell is provided.

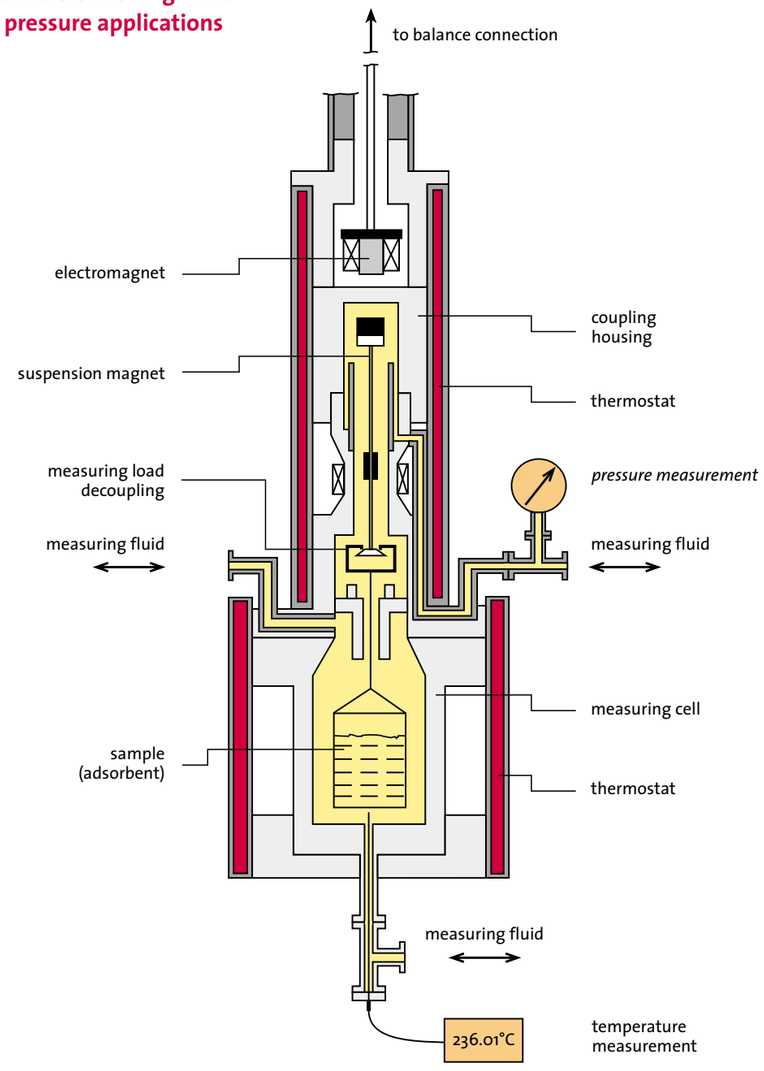


Measuring gas connections of the magnetic suspension balance

ISO*SORP* 2000 – the new sorption measuring standard

- adsorption
- absorption
- desorption
- supercritical fluid extraction
- diffusion
- drying
- polymerization

Metal version for high and low pressure applications



High pressure sorption measuring

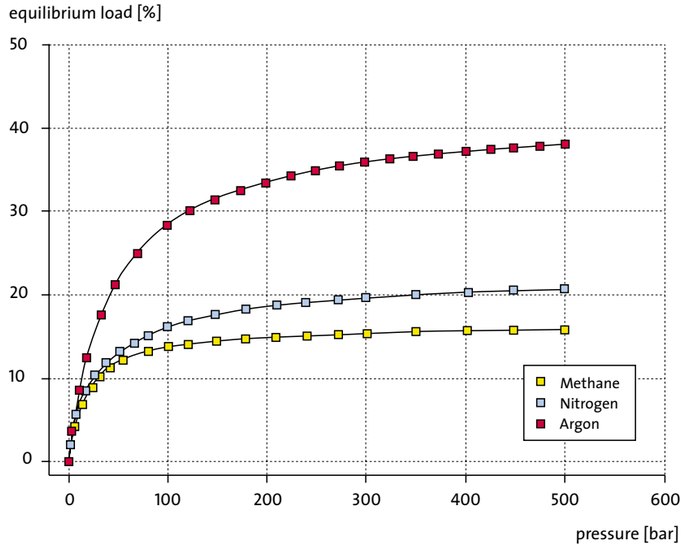
Pressure range UHV up to 500 bar

Temperature range with measuring cell and coupling housing as thermal unit: -196 °C up to +350 °C

Temperature range with thermally separated measuring cell: -267 °C up to 750 °C

For the operating principle and technical data of the suspension balances see pages 6ff and the table on page 12.

Application example: Sorption measurements on gases in activated carbon



Adsorption equilibria of various gases on activated carbon at 25 °C

Measurements were carried out at the Institute for Non-Classical Chemistry at the University of Leipzig using a magnetic suspension balance with a resolution of 0.01 mg and a measuring load of up to 30 g. Pressure and temperature range reached 500 bar and 150 °C.

Measuring temperatures up to 350°C

Up to this temperature both the measuring cell and coupling housing form a thermal unit. A gradient-free temperature level is achieved at all points in the measuring fluid. In this case both cell and coupling housing are thermostated by a circulating liquid (see fig on the right).

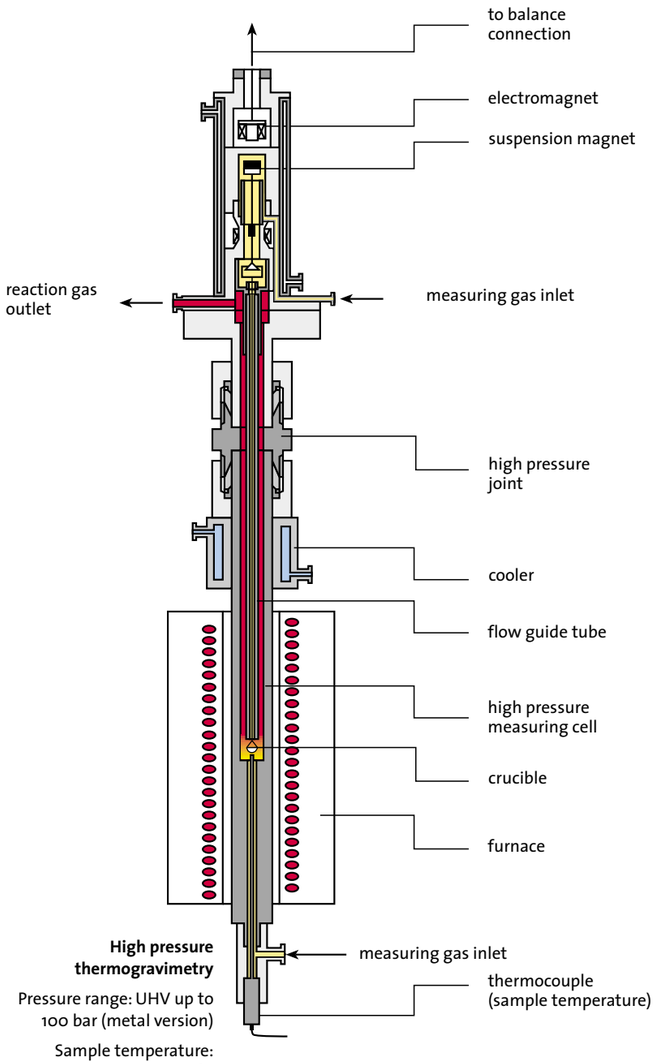
Measuring temperatures up to 750°C

To obtain higher measuring temperatures the measuring cell and suspension coupling are thermally separated and electrically heated. The coupling housing is still thermostated by a circulating fluid up to a maximum of 350 °C.

All possibilities the new ISO*SORP* sorption measuring standard offers are shown in our brochure dealing with sorption analysers.

Metal version with a liquid thermostat enclosing the measuring cell.

Thermogravimetry



High pressure thermogravimetry

Pressure range: UHV up to 100 bar (metal version)

Sample temperature: up to 1000°C

Suspension coupling temperature: up to 250°C

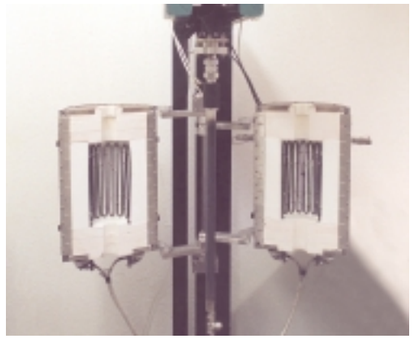
Low pressure versions

Pressure range: vacuum to 1.3 bar (quartzglass and ceramic measuring cell)

Sample temperature: up to 900°C (quartzglass version) up to 1700°C (ceramic measuring cell)

Applications

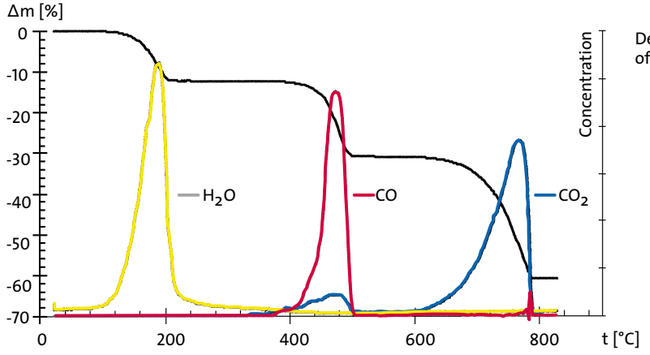
- decomposition
- coating
- pyrolysis
- gasification
- reaction kinetics
- corrosion



Special versions

- Low pressure measuring cell for temperatures up to 2000°C
- High pressure version with integrated heating element for pressures over 100 bar and temperatures over 1000°C

See page 6ff and the table on page 12 for more information about the function and technical data. Detailed description and technical data of our thermogravimetric analyzers are given in the special brochure "Thermogravimetry".

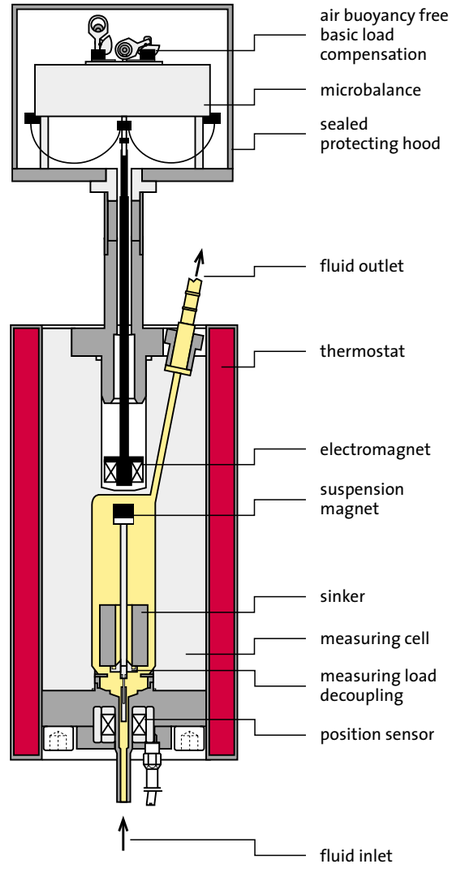


Decomposition of Ca-Oxalate

Thermophysical properties

Single sinker densitometer

Density measurements of gases and fluids with reference data quality are a precondition for formulating thermal equations of state. Highly accurate density measurements are necessary for the calibration of industrial densitometers which for example are used in pipelines in order to account the supplied natural gas quantities. The buoyancy method using Archimedes' principle is used to obtain high precision density measurements whereby a sinker, whose volume is known, is weighed in a measuring fluid. The buoyancy which the sinker experiences indicates the density of the fluid. By using a magnetic suspension balance this process can be applied in an extremely large pressure and temperature range to almost all substances with the utmost accuracy.



Compact design – widely applicable

This extemely compact instrument, where the coupling housing and measuring cell including sinker are designed as a unit, is nonetheless optimal for flushing and easy to thermostate. The maximum measuring temperature is restricted to the working temperature of the suspension coupling (≤ 350°C). Apart from this, the operating principle and most specifications do not differ from the standard suspension balances. But with the main advantage of a very high pressure range of up to 2000 bar.

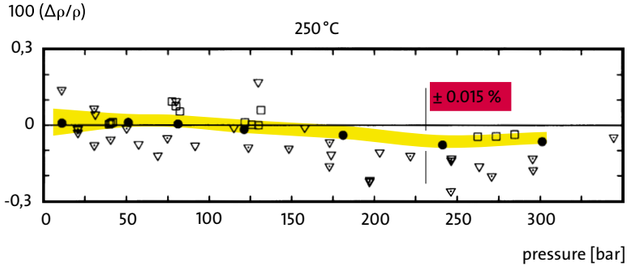
High pressure single sinker densitometer

pressure range: UHV up to 500 bar (Special version up to 2000 bar)

temperature range: -195 °C up to +350 °C

density range: up to 2000 kg/m³

density uncertainty: 0.02 % and 0.01 kg/m³

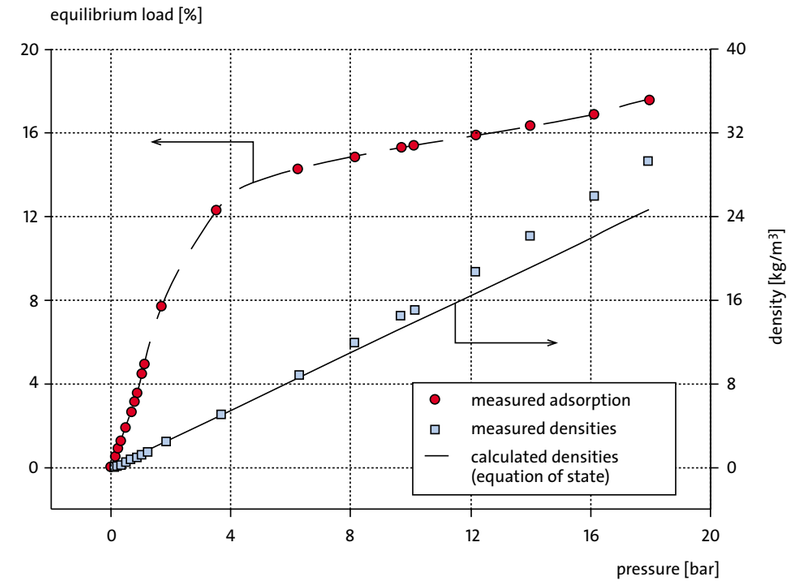


Reference density measurements on carbon dioxide at 250°C up to 300 bar (sinker weight 60 g / Resolution 0.01 mg), Chair of Thermodynamics, University of Bochum

Simultaneous measurement of mass transfer and density

Using the magnetic suspension balance to measure two samples, which has already been mentioned on page 9, simultaneous sorption and density measurements can be carried out. By weighing the Ti sinker as the second sample according to Archimedes' principle, the density of the fluid phase is determined.

Particularly under high pressure or in gas mixtures this cannot be calculated accurately using equations of state. However, the density influences via the buoyancy correction the measured sorption values enormously and can cause clearly falsified measuring results.



Adsorption equlibrium of H_2S on DAY Zeolith (circles on the left axis)

simultaneously measured density results of H_2S gases (squares on the right axis) calculated data using an equation of state (line)

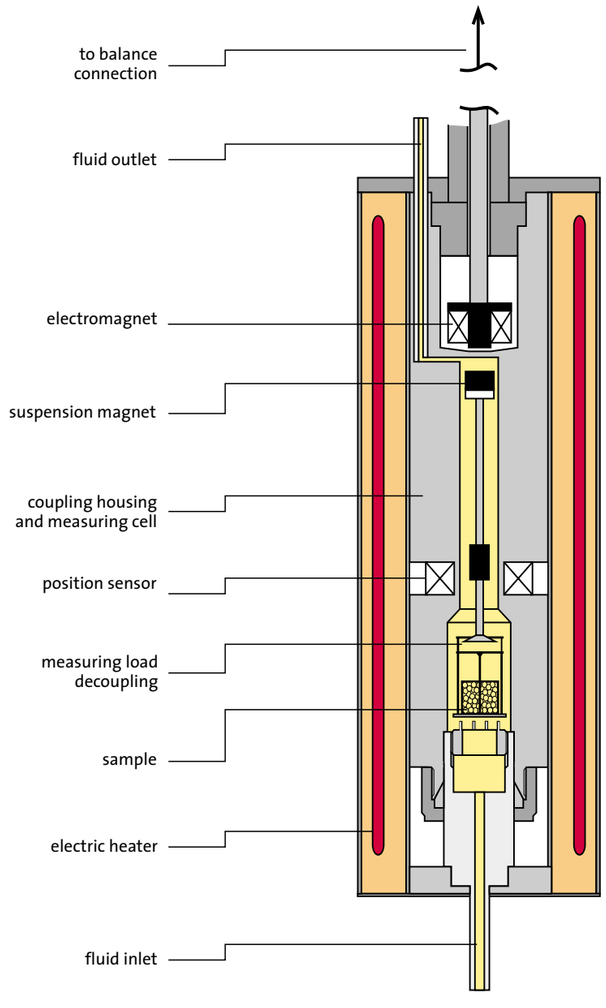
Chair of Thermodynamics, University of Siegen

Furthermore, the density measuring of binary mixtures enables their composition to be calculated without the necessity of further analysis (eg gas chromatograph).

Therefore, the magnetic suspension balance offers two devices in one: gravimetric measurements of mass transfer through sorption and also the gravimetric measuring of gas compositions.

High temperature and high pressure magnetic suspension balances

For the examination of extraction properties of supercritical water, eg for the decontamination of polluted soils, a special magnetic suspension balance was developed. Similar to the case for density measuring, the measuring cell and the suspension coupling form a unit to make a very compact device. The entire measuring volume is thermostated with an electrical heater to a maximum temperature of 500°C and has a gradient free temperature level at all points so that the measuring fluid does not condense. In order to achieve an extremely wide pressure range up to 500 bar at such high temperatures, the entire suspension coupling has been produced with a special heat proof alloy of low magnetic permeability.



High temperature-high pressure magnetic suspension balance for mass transfer measurements in supercritical water (up to 500°C and 500 bar)

Pressure range:
UHV to 500 bar

Temperature range:
up to 500°C
(entire measuring room incl. magnetic suspension coupling)

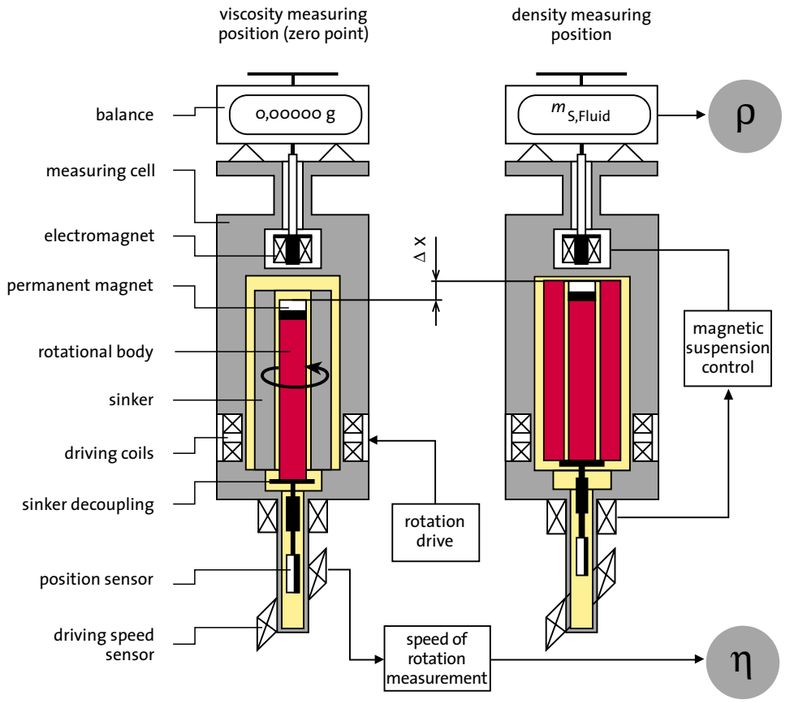
Sample mass: up to 10 g

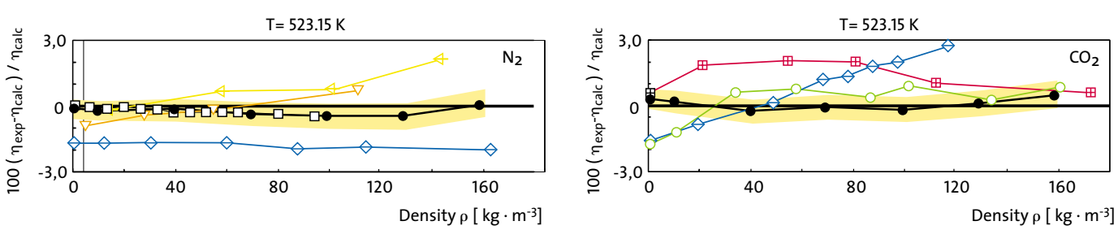
Resolution: 0.01 mg

Simultaneous density and viscosity measurements with gaseous media

With this new type of magnetic suspension balance, the density and dynamic viscosity of gases can be measured almost simultaneously. The density measuring procedure follows the principle of the one sinker density measuring method (see page 19) and the viscosity measuring by means of a rotation technique. In this process, the magnetic suspension coupling acts as a contactless and frictionless hold for the rotating part which is set in motion from the outside. After turning off the driving field, the dynamic viscosity of the measuring fluid can be determined from the damping of the rotation of the freely suspended rotating body.

Magnetic suspension balance for simultaneous density and viscosity measurement with gaseous media





● Simultaneous density and viscosity measurement, Chair of Thermodynamics, University of Bochum

□▽○◇⊞◁ Other authors

Literature on magnetic suspension balances

Magnetic suspension balances

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