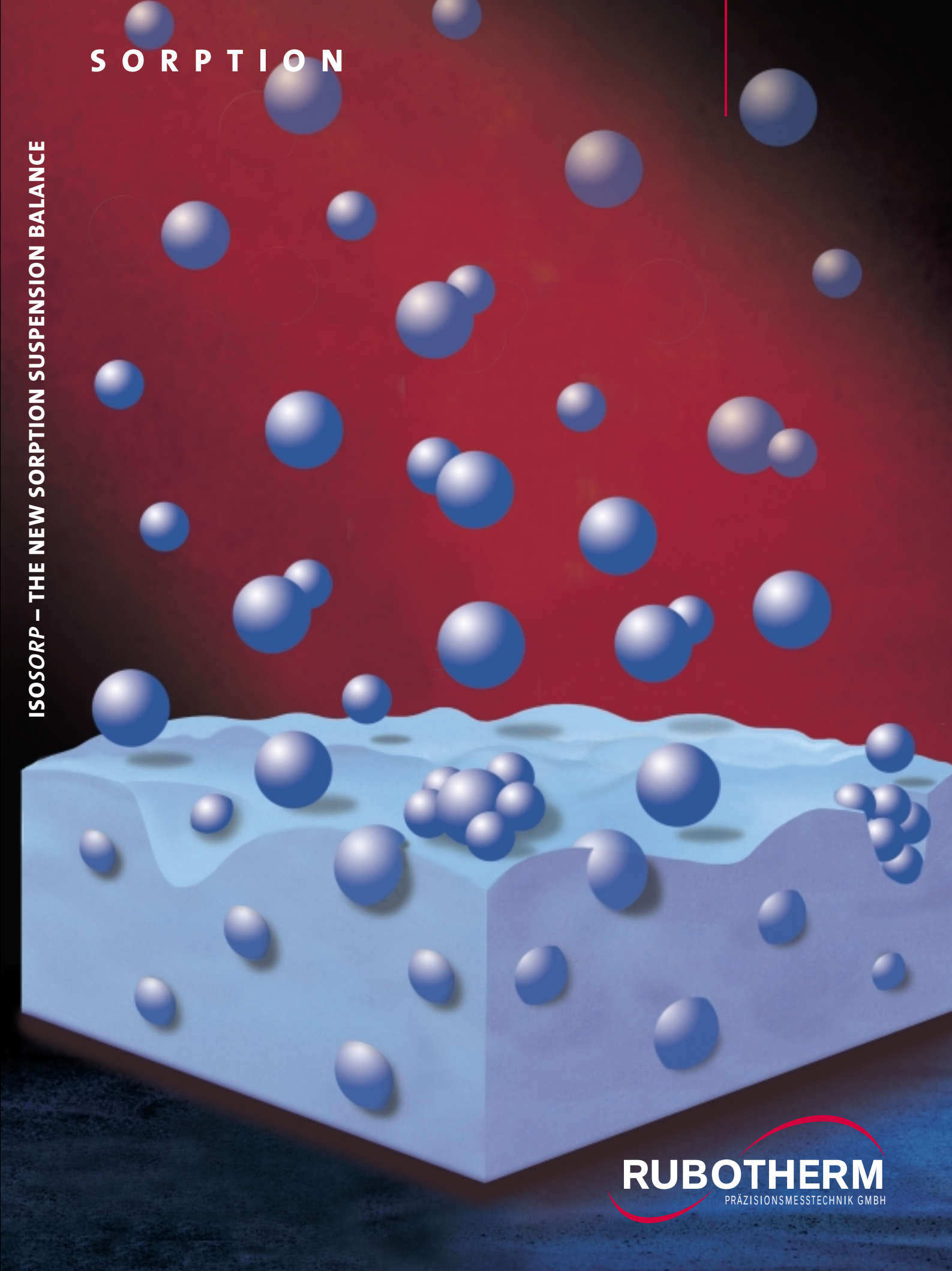
SORPTION

ISOSORP – THE NEW SORPTION SUSPENSION BALANCE



RUBOTHERM
PRÄZISIONSMESSTECHNIK GMBH

Contents

Current information about our instruments and forthcoming events can be called up on our internet page under www.rubotherm.de. The magnetic suspension balance technology is described in detail in the catalog "Magnetic Suspension Balances".
Special brochures are available for density measuring and thermogravimetry in addition to the current brochure "Sorption".

RUBOTHERM
PRÄZISIONSMESSTECHNIK GMBH

UNIVERSITÄTSSTRASSE 142
D-44799 BOCHUM
PHONE: 00 49 - 2 34 - 70 99 6-0
FAX: 00 49 - 2 34 - 70 99 6- 22
E-MAIL: INFO@RUBOTHERM.DE

WWW.RUBOTHERM.DE

ARTWORK: DESIGNBÜRO SCHÖNFELDER ESSEN

Rubotherm –
your specialist for gravimetric analysis

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

With this brochure we would like to introduce our ISO*SORP* instruments for measuring mass transfer under controlled environments. In addition to the instruments for sorption measurements shown here there are two further brochures dealing with our thermogravimetric and density analysers.

The special feature of all our measuring devices lies in the contactless weighing of a sample through the walls of the measuring cell by means of a magnetic suspension balance. The principle is explained in detail in the Rubotherm catalog "Magnetic Suspension Balances".

Magnetic suspension balances

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.

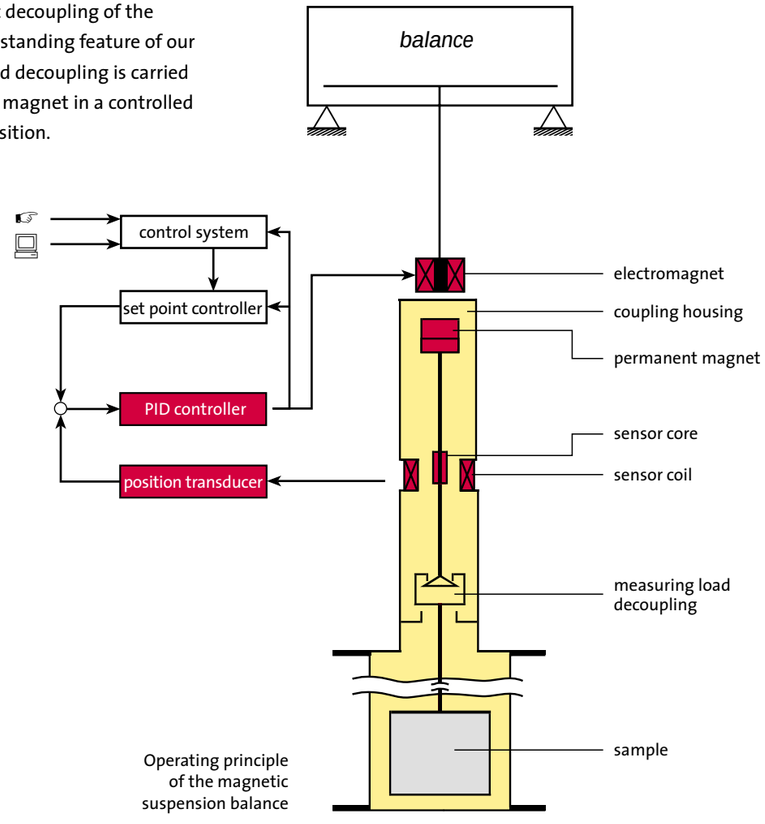
The main difficulty when using conventional gravimetric instruments is the direct connection of the measuring cell (sample atmosphere) and the weighing instrument. The balance can be damaged or disturbed by the measuring atmosphere and the measuring atmosphere can be adversely affected by flushing gases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

Using our magnetic suspension balances it is 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). The 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. The 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. 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.

Besides the complete separation of measuring cell and weighing system the automatic decoupling of the measuring load is one more outstanding feature of our measuring instruments. The load decoupling is carried out by lowering the suspension magnet in a controlled way to a so-called zero point position.

This position, where only the weight of the suspension magnet is transmitted to the balance, corresponds to an empty balance pan in a normal weighing procedure. Now the balance can be tared and calibrated even when recording measurements under process conditions. This unique feature of our magnetic suspension balances increases the measuring accuracy to levels not previously known, particularly in the case of long term measurements.



Operating principle of the magnetic suspension balance

ISO*SORP 2000 – the sorption measuring standard*

Based on our magnetic suspension balances, a compact almost universally applicable sorption-measuring standard has been developed. These instruments are notable for several reasons: they are easy to handle, offer excellent measuring accuracy, have a wide range of application, and provide a high degree of automation. They are the results of many years of co-operation with numerous applicants and continued further development of our sorption measuring devices.

Fully automatic process – MessPro-Software

In order to control and monitor the fully automatic process, a comprehensive software package is available. This software can take care of the recording, in-situ presentation and storing of data as well as monitor the entire system. The temperature of the sample is regulated and the pressure in the measuring cell is set corresponding to the configuration of the measuring sequence. It is possible either to supervise equilibrium criteria or to control the carrying out of measurements purely over time.

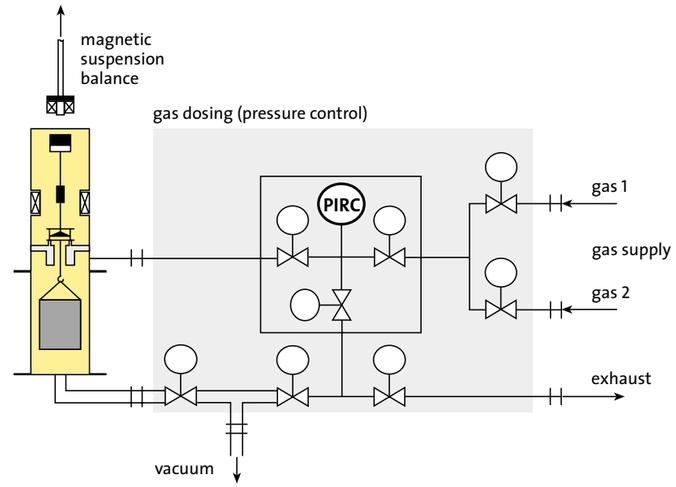
Thermostating

The entire interior volume of the magnetic suspension balance can be thermostated to the same temperature, up to 620 K as a thermal unit. With the metal version this is achieved by means of double-walled thermostating tubes which completely surround the measuring cell and suspension balance.

At sample temperatures of more than 620 K, the measuring cell is heated electrically allowing therefore temperatures of up to 1020 K to be reached. At even higher temperatures up to approximately 2300 K our thermo-gravimetric devices are used. These are described in detail in our special brochure ”Thermogravimetry“.

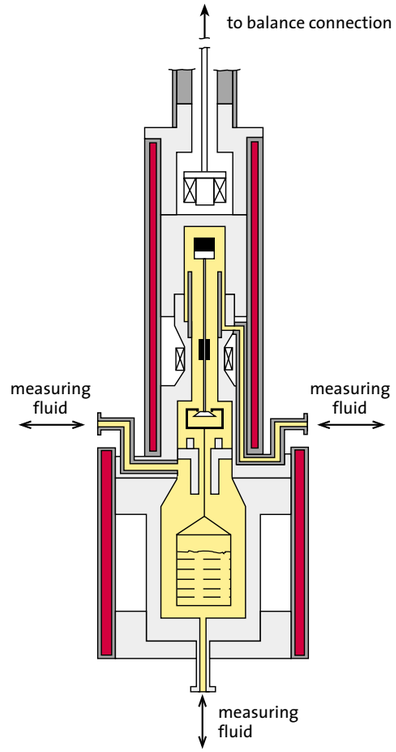
Automatic gas dosing

For the fully automatic measuring of sorption isotherms or isobars, besides thermostating, a gas dosing is also necessary. Various types of gas dosing systems are available for the ISO*SORP* – to measure in static atmospheres (see figure on the right), to measure in flowing atmospheres and a gas dosing system with a gas manifold volume for a combined volumetric-gravimetric measuring process.



Magnetic suspension balance

Measurements to obtain the target value of the sorption experiment i.e. the mass of the material adsorbed, are carried out with a magnetic suspension balance. A special outstanding feature of this instrument is the complete separation of the balance from the measuring atmosphere (see figure on the right). This means that the sample can be exposed to almost all measuring fluids even corrosive ones without the need for protective measures and can be applied in a very large pressure and temperature range for gravimetric measurements. Besides this the magnetic suspension balances stand out as a result of the long term stability which can be achieved using their unique load decoupling mechanism.



ISO*SORP 2000 –*
solves almost all measuring problems

The ISO*SORP* apparatus described on the previous pages is made up of various components. These components can be specially selected according to the application. This exceedingly flexible set-up of the ISO*SORP* can cover almost all applications by means of the corresponding combination of our standard components. This guarantees even for unusual applications a reliable and exact measuring device with a high degree of automation.

In the following pages, the components of the ISO*SORP* are described, and finally – on pages 14 and 15 – a suggestion is made of how to go about selecting the right components.

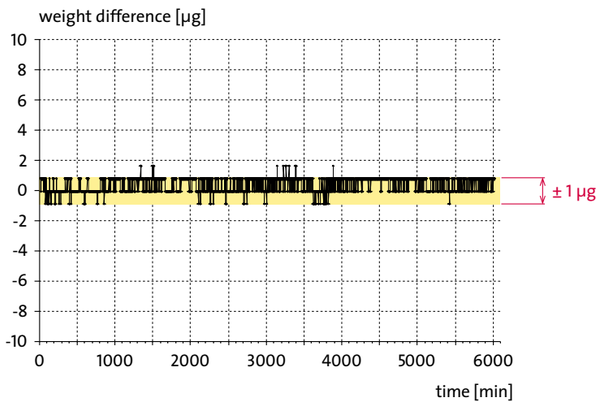
ISO*SORP* – an instrument tailor-made for your application and not just mass-produced

Basic component – magnetic suspension balance

The magnetic suspension balance forms the main part of the ISO*SORP* 2000 and is a unique, high precision gravimetric measuring device. The fundamental technology and suitable form of the instrument can be referred to on pages 6 - 16 in the catalog "Magnetic Suspension Balances".

For the ISO*SORP* both metal and glass suspension balances are used depending on the measuring task. Measuring ranges and accuracies of these instruments are given on page 12 of the catalog "Magnetic Suspension Balances".

Weighing of a 3 g stainless steel weight in vacuum for 100 hours (values shown are the deviations from the initial value)



The magnetic suspension balance enables a highly accurate gravimetric measuring process of both sorption equilibrium as well as sorption kinetics. The application range for pressure extends from UHV up to 500 bar. The temperature in the whole measuring cell (magnetic coupling and sample) can be set up from 77 K up to 620 K. Measurements at higher temperatures can be achieved by electrically heating the sample cell whilst thermally de-coupled from the magnetic suspension coupling.

Two resolutions can be obtained by using two different weighing instruments:

A resolution of 0.01 mg with samples up to 80 g and a reproducibility of ± 0.02 mg

or a resolution of 1 µg with samples up to 10 g and a reproducibility of ± 2 µg.

Thermostating I – no cold ends during the measuring process

One main advantage of the magnetic suspension balance is its wide working temperature range. Therefore, the entire volumes which are in contact with the measuring fluid can be thermostated as a thermal unit at temperatures ranging from 77 K to 620 K. As a result, a gradient free temperature level is achieved at all points in the measuring fluid. For thermostating, the metal version of the magnetic suspension balance is combined with double-walled tubes through which a thermostated liquid is circulating (see figure on the right). For the same purpose the housing of the glass suspension balance is double-walled.

Thermostating II – heating up the sample for pretreatment

Many kinds of samples can or must be activated by a certain temperature program in preparation for the measuring process. If the temperature range achieved by the fluid thermostating is not sufficient for the activation, the measuring cell can be electrically heated. This is done up to 770 K with the same measuring cell but using an adapter for a thermal separation. To reach temperatures up to 1020 K elongated measuring cells made of special high temperature alloys are provided. The area of the magnetic force transmission – the magnetic suspension coupling – can still be heated with a fluid thermostating process up to temperatures of 620 K. In addition to using the electrical heating process for activation, it can be used for sorption measurements at temperatures of over 620 K. For sample temperatures of more than 1020 K special magnetic suspension balances for high temperature measurements are used. These instruments are described in detail in the special brochure on "Thermogravimetry".

Pressure and temperature measurement

In order to be able to assign the measured sorption processes with the thermodynamic quantities of state, the ISO*SORP* is equipped with pressure and temperature measuring facilities. For the pressure measurement, depending on the pressure range and accuracy requirements, instruments and sensors produced by Desgranges et Huot, Paroscientific, MKS, Kistler or Druck are used. According to which sensor type is used, a relative measuring accuracy of between 0.005% and 0.05% can be obtained. For measuring temperature, a platinum resistant sensor is used. By using a calibrated Pt 25 sensor with an ASL measuring bridge, accuracies of ±0.001 K can be obtained. When dealing with usual requirements a Pt 100 is used, which provides a typical measuring accuracy of ±0.03 K at 273 K. Likewise, thermocouples of various types can be used.

Simultaneous weighing of two samples

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

Due to continued development of the decoupling system it is now possible to weigh two samples simultaneously with only one balance (see catalog on Magnetic Suspension Balances pages 8 and 9). Besides the first measuring load decoupling a second is arranged in the magnetic suspension coupling (see figure). Three different vertical positions of the suspension magnet, which can be arrived at in a controlled way, correspond to the three different measuring positions.

Zero point: the permanent magnet alone is in a freely suspended state, allowing the balance to be tared and calibrated.

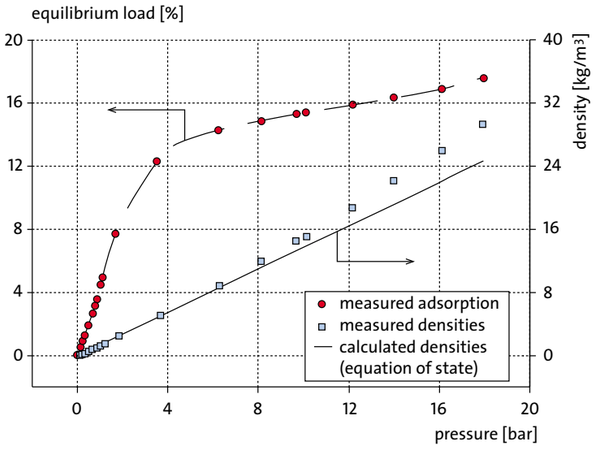
Measuring point 1: the first sample is lifted up and its mass is weighed.

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 I: simultaneous sorption and density measurement

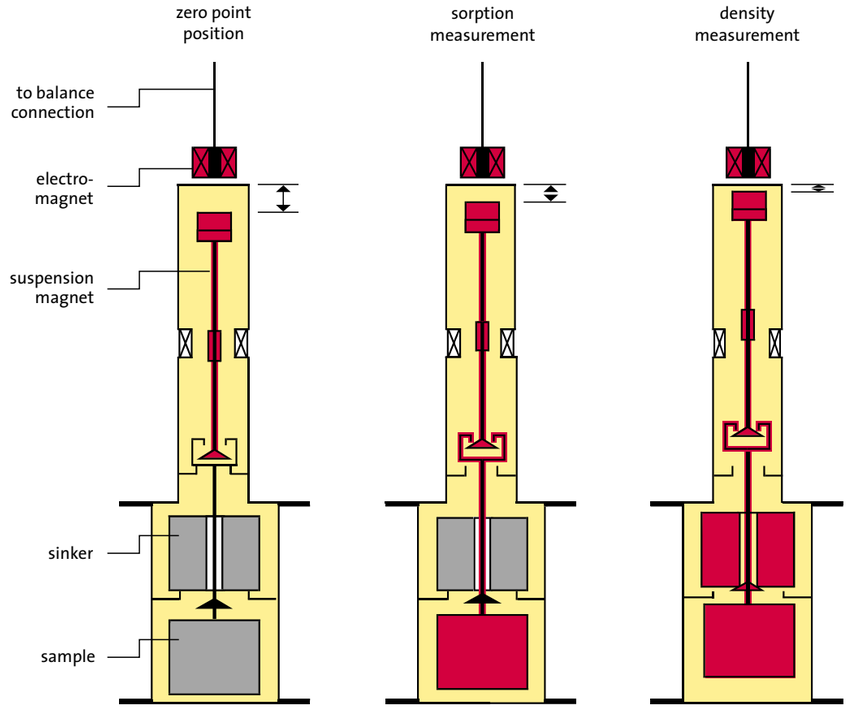
The first step is to weigh the sorbent to be examined as the first sample. Then, a titanium sinker with a calibrated volume is weighed as the second sample. This measurement allows the density of the measuring fluid surrounding the sample to be ascertained, which is required for the buoyancy correction of the measured sorption values. An example for this can be seen on the left – an adsorption measurement with H_2S on zeolite, together with the H_2S densities measured simultaneously. The line represents the calculation of the density with an equation of state for pressure and temperature, which is obviously incorrect.

The simultaneous measurement of sorption and density is especially needed if the buoyancy effects caused by the density of the adsorptive fluid are large; (high pressure and / or low temperatures); likewise, if the density of the adsorptive cannot be calculated as there are no equations of state available (as is almost always the case with mixed gases). In addition, density measuring in the case of a binary gas mixture offers the possibility of determining the concentration of the gas mixture without further analysis since the density is a function of the mixture composition.



Adsorption equilibrium of H_2S on DAY Zeolite
(circles, left axis)
simultaneously measured densities of H_2S gas
(squares, right axis) calculated density using an equation of state (line)

Chair of Thermodynamics, University of Siegen



Example:
Simultaneous measurement of sorption and density

Application II: density measurements of porous solids

To determine its density the sample and the sinker are pressurized in one or several pressure steps using gases which adsorb as little as possible (He, Ar). There is no need to calculate the gas density which may produce larger assignment errors, since this is gained with utmost precision directly from the measured buoyancy acting on the sinker.

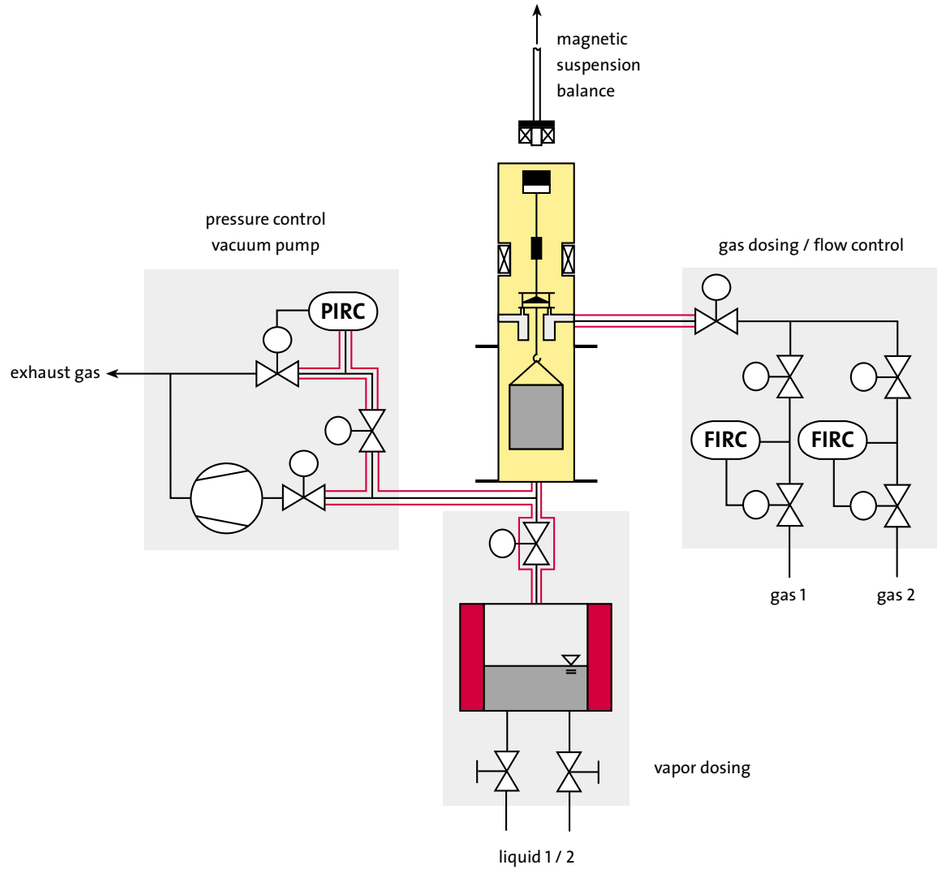
Application III: reference measuring method

The comparative measuring of two samples (e.g. sample and reference sorbent or sample and inert sample for comparison) is carried out in the same way as the sorption and density measuring process described above. A second sample is weighed instead of the Ti sinker. Thus, this kind of measuring procedure can be used to determine the buoyancy force acting on an inert sample which can then be used as a corrective of the measured data with the active sample. In addition, a comparison can be directly made with a standardised reference sorbent. In this way, the relation between the sample data recorded and the tracing back to a reference standard is ascertained both directly and experimentally.

Automatic gas dosing

There are three types of automatic gas dosing devices: for experiments in static atmospheres purely as a pressure regulator, for experiments in a flushing gas as a mass flow controller with reaction pressure regulation and for experiments with gas or vapor mixtures using a volumetric gas dosing instrument with a calibrated gas manifold volume and highly accurate pressure measurement (see also page 18).

For the pressure regulation of a static measuring atmosphere we use a calibration pressure controller produced by Druck. A set value both from low pressures as well as from high pressures can be obtained with the highest accuracy using this particular control unit. It is equally possible to achieve a constant pressure change over time (rise / fall), a so-called ramp function. An example for such a gas dosing can be seen in the adjacent figure (two pressure regulators for the low and high pressure range).



To measure in flowing media the pressure and mass flow control is carried out as shown in the flow scheme below left. Using this device gravimetric experiments with gas mixtures of highly defined composition can be carried out in flowing atmospheres with regulated pressures.

All three gas dosing systems mentioned here can be heated from 350 K to 450 K in order to prevent condensation of the measuring fluid.

A thermostated vapor pressure cell is used for the automatic dosing of vapors in the magnetic suspension balance. It is connected to the measuring cell by means of an automatic valve which is quickly regulated and electrically heated tubes. With the help of the pressure measurement in the measuring volume the control valve is regulated by the software in such a way that defined pressures can be set. The vapor dosing part is shown underneath the magnetic suspension balance in the illustration.

MessPro-Software

The MessPro-Software allows the storing and presentation of all data measured as well as the fully automatic controlling of all instruments connected. As a result of its modular set up it can be easily adapted to the configuration of the measuring system required and besides this enables the data recording of several measuring sequences to be automatically carried out.

In the case of adsorption measurements for example it can be tested if an equilibrium value has been reached. This is possible with the help of equilibrium criteria (mass change over time, pressure and temperature change), which can be freely defined by the user. After the equilibrium has been determined, the next point to be measured is set by means of the automatic temperature control and gas dosing system. A further available option is a purely time controlled run through of a measuring sequence. The temperature and pressure points which have to be measured are defined either before or during the measuring process by the user in a table form.

During the measurement, current data are shown as online diagrams. Mass, pressure, temperature, and other measured signals (e.g. mass spectrometer, FTIR) are displayed in relation to the measuring time. Correction of the balance drift or the buoyancy force, which acts on the sample by the surrounding fluid, can be carried out directly online. The determination of the buoyancy force is calculated using an equation of state or is ideally measured directly using the simultaneous density measurement.

All measuring valves are stored in an ASCII-File and are therefore available for evaluation on spreadsheet software.

ISO*SORP 2000 – instrumentation choice*

The ISO*SORP* 2000 can be compiled from several briefly described individual units mentioned before, to suit perfectly your application. In the following, the individual selection possibilities have been summarised.

Choice of balance resolution

- 0.01 mg resolution (± 0.02 mg reproducibility) with up to 80 g sample load and 1 or 2 samples (e.g. simultaneous density measuring).
- 1 µg resolution (± 2 µg reproducibility) with up to 10 g sample load and 1 or 2 samples (e.g. simultaneous density measuring).

Choice of material for the magnetic suspension coupling

Stainless steel and special alloys for UHV up to highest pressures, glass in the pressure range from vacuum up to 1.3 bar for highly corrosive media.

Choice of pressure range for the metal version

- Low pressure (LP): UHV up to 3 bar
- Medium pressure (MP): UHV up to 150 bar
- High pressure (HP): UHV up to 500 bar

Choice of temperature range for the magnetic suspension coupling

- **Glass version:**
  - Standard (ST): up to 420 K
  - High temperatures (HT): up to 520 K
- **Metal version:**
  - Standard (ST): up to 420 K
  - High temperatures (HT): up to 520 K
  - Specially high temperatures (SHT): up to 620 K
  - Low temperatures (LT): up to 77 K

Choice of the measuring gas connections

- **Glass version:**
  - Standard: One connection at the upper end of the magnetic suspension coupling housing and one connection at the bottom of the measuring cell
  - Additional connection: One additional connection below the measuring load decoupling for flushing the measuring cell with higher flow rates
- **Metal version:**
  - Standard: One measuring gas connection at the bottom of the measuring cell
  - Measuring gas connection I: An additional connection with a flow guide tube for the dead volume free flushing of the entire inner volume of the coupling housing
  - Measuring gas connection II: An additional connection in the flange of the coupling housing for the measuring gas inlet directly above the sample in case of higher flow rates

Choice of measuring cell

- **Glass version:**
  - Standard: Double-walled glass measuring cell for thermostating with a circulating fluid up to 520 K
  - Low temperatures: Elongated single-walled measuring cell for use at low temperatures (e.g. LN_2 cooling).
  - High temperatures: Elongated single-walled measuring cell and an exterior electrical heating unit ($T \leqslant 1070$ K)
- **Metal version:**
  - Standard: Sorption measuring cell for thermostating from the outside in a temperature range from 200 K up to 620 K. For thermostating in a temperature range up to 620 K a double-walled thermostat which can be flushed through with thermostating fluid is used. In this case a constant temperature of ± 0.1 K is achieved. At higher temperatures up to 720 K an electrically heated thermostat and an adapter for thermal decoupling are used.
  - Low temperatures: Elongated, thin walled measuring cell for use at low temperatures (e.g. LN_2 cooling or LHe cooling).
  - High temperatures: Elongated measuring cell with an outer electrical heater ($T \leqslant 1020$ K). Here a constant temperature of ±0.5 K is reached.

Choice of gas supply device

- Static: Pressure regulator for the fully automatic dosage of measuring gas up to a certain set value (stipulate pressure range)
- Gas flow: Gas flow and pressure regulator for fully automatic measuring in flowing gas mixtures
- Volumetric: Gas dosage with a calibrated volume for a defined dosing of a certain quantity of a gas mixture. This allows adsorption processes of binary mixtures to be measured without having to measure the gas composition (see page 18).

All three mentioned gas dosing systems can be heated from 350 K up to 450 K in order to prevent condensation of the measuring fluid.

Choice of pressure and temperature measurement

- Temperature: Temperature measurement beneath the sample in the measuring cell, according to temperature range and accuracy requirements with Pt 25, Pt 100 or a thermocouple sensor. Measuring accuracies lie between ±0.001 K and ±2 K.
- Pressure: Pressure measurement in the measuring cell, choice of pressure sensor according to pressure and temperature range and accuracy requirements (±0.005% and 0.5%).
  Alternatively the pressure sensor can be arranged outside the measuring cell at one of the measuring gas connections by means of a shut off valve.

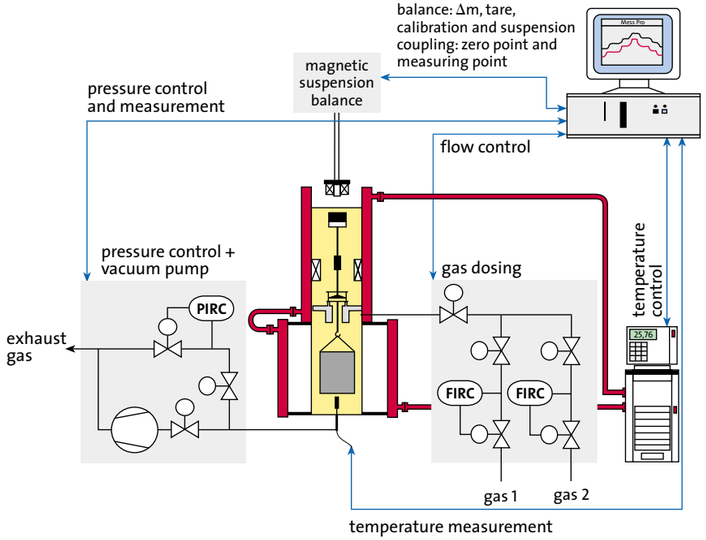
ISO*SORP 2000 instrumentation examples*

The principal set up of ISO*SORP* and the choice of individual devices or modules available have been discussed previously. In the following, we would like to describe several devices which we have put together from the numerous combinations possible. The intention here is to show instruments which are more frequently used and which allow a large number of applications to be covered.

ISO*SORP* LP-flow

In this example LP stands for low pressure, flow expresses the type of gas dosing. A flow and function diagram of this ISO*SORP* LP-flow is illustrated. Special features of the gas dosing are the connection possibilities for several gases whereby a defined gas flow can be fed through the measuring cell by means of the gas flow controllers. The pressure in the measuring cell is monitored by a reaction pressure control. This pressure regulator provides in addition a precise pressure measurement. The temperature in the measuring cell is recorded with a Pt 100 probe.

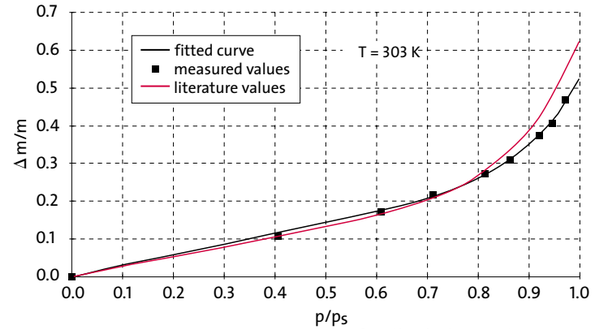
Technical data and measuring ranges of the ISO*SORP* LP-flow are given in the following table.



measuring / control device	characteristic data	
balance		
resolution	0.01 mg	1 µg
reproducibility	± 0.02 mg	± 2 µg
sample load	⩽ 30 g / 80 g	⩽ 5 g / 10 g
pressure	UHV up to 3 bar	
pressure measuring	± 0.5 %	
temperature	up to 520 K	
temperature measuring	± 0.02 %	
gas dosing		
control range	2 x 300 ml/min	
reaction pressure regulator	UHV up to 3 bar	

The pressure range can be extended in this system configuration up to 150 bar. Higher temperatures are also attainable using other thermostating components. Temperature and pressure measuring accuracy can also be improved by using higher resolution devices.

The sorption of toluol in polyurethane at 303 K as a function of relative vapor pressure is shown in the figure on the right.

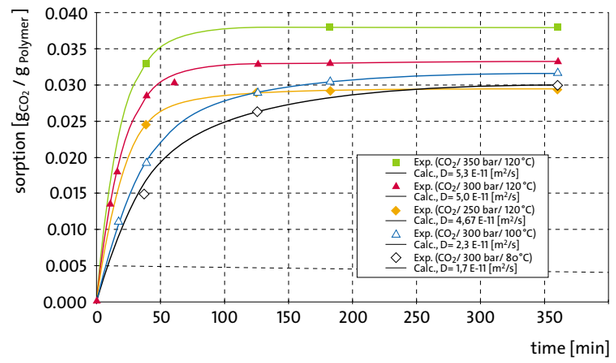


Sorption isotherm of toluol in polyurethane – Bayer AG, Leverkusen

ISO*SORP* MP-static

The ISO*SORP* MP-static is suitable for measuring sorption processes in a medium pressure range in static atmospheres. The gas dosing is such that certain pressure values of utmost accuracy can be set automatically. The measuring range of the ISO*SORP* MP-static is given in the following table.

An electrical heating unit for the measuring cell is available. This allows sample temperatures up to 720 K to be reached for activation. An adsorption measurement of CO_2 in PET is illustrated in the figure adjacent.



Measurement of sorption kinetics of CO_2 at PET, Chair of Process Engineering II at the Technical University of Hamburg-Harburg

measuring / control device	characteristic data	
balance		
resolution	0.01 mg	1 µg
reproducibility	± 0.02 mg	± 2 µg
sample load	⩽ 30 g / 80 g	⩽ 5 g /10 g
pressure	UHV up to 150 bar	
pressure measuring	Δp/p ⩽ 0.01 % FS	
temperature	up to 520 K	
temperature measuring	± 0.02 %	
sample preparation	up to 720 K (sample temperature)	
gas dosing		
control range	up to 210 bar	
accuracy	< ± 0.04 % FS	

ISO*SORP* 2000 instrumentation examples

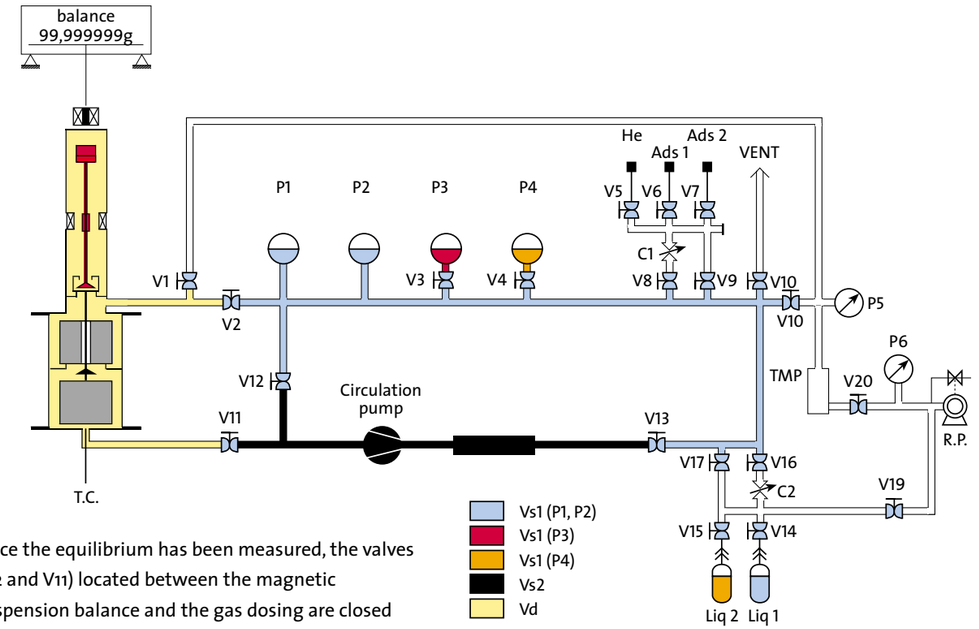
ISO*SORP* BG – gravimetric & volumetric

In order to take automatic measurements of adsorption equilibria of binary gas or vapor mixtures our partner company BEL Japan Inc. has developed a volumetric gas dosing system for the ISO*SORP*. A defined gas or vapor mixture for the sorption is made available with this gas dosing whereby the pure components are filled into two calibrated volumes (marked black and blue on the flow scheme). From the pressure (P1 to P4) and temperature measuring and with the help of the thermal equation of state, the quantity of each component and therefore the composition of the binary mixture can be determined. After the circulating pump has mixed both components these are brought into contact with the adsorbent by opening two valves (V2, V11). The sorption which takes place is measured with the magnetic suspension balance. Once the sorption equilibrium has been reached (no change over time of the balance reading nor the pressure), then the density of the gas mixture is determined with the magnetic suspension balance. This occurs according to the sorption and density measuring process described on page 10. If the components of the binary mixtures have different molar masses then this density is a function of the composition of the gas mixture. This means that the density measured with the magnetic suspension balance serves here to determine the concentration.

As a result of the measured data, the concentration of the gas mixture before and after sorption, as well as knowledge of the gas quantities available, the selectivity of the sorption and therefore the adsorbed mass of every component of the gas mixture can be ascertained without requiring additional analytical devices.

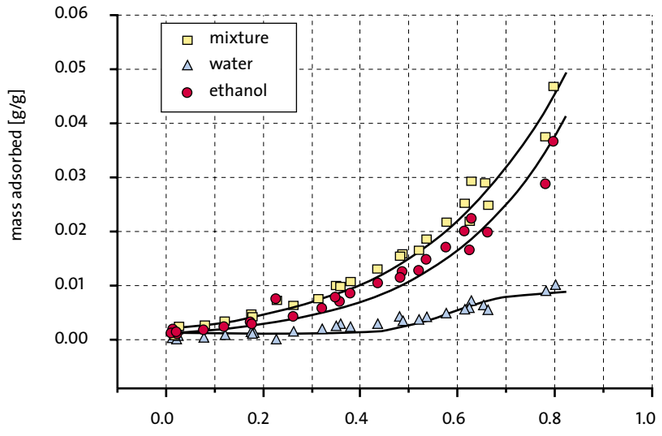
BEL Japan Inc.

measuring / control device	**characteristic data**	
balance		
resolution	0.01 mg	1 µg
reproducibility	± 0.02 mg	2 µg
sample load	up to 20 g	up to 5 g
density measurement with Ti-sinker	$\Delta\rho \leq 0.002$ kg/m³	$\Delta\rho \leq 0.001$ kg/m³
pressure	UHV up to 135 bar	
pressure measuring	up to 4 sensors, $\Delta p/p \leq 0{,}25\%$ MV	
temperature	up to 520 K sample temperature up to 350 K gas dosing temperature	
gas circulation	magnetically coupled circulation pump (up to 350 K and 150 bar)	
gas dosing connections	2 x	
vapor dosing	2 vapor pressure cells	
vacuum system	rotary pump (turbomolecular pump)	



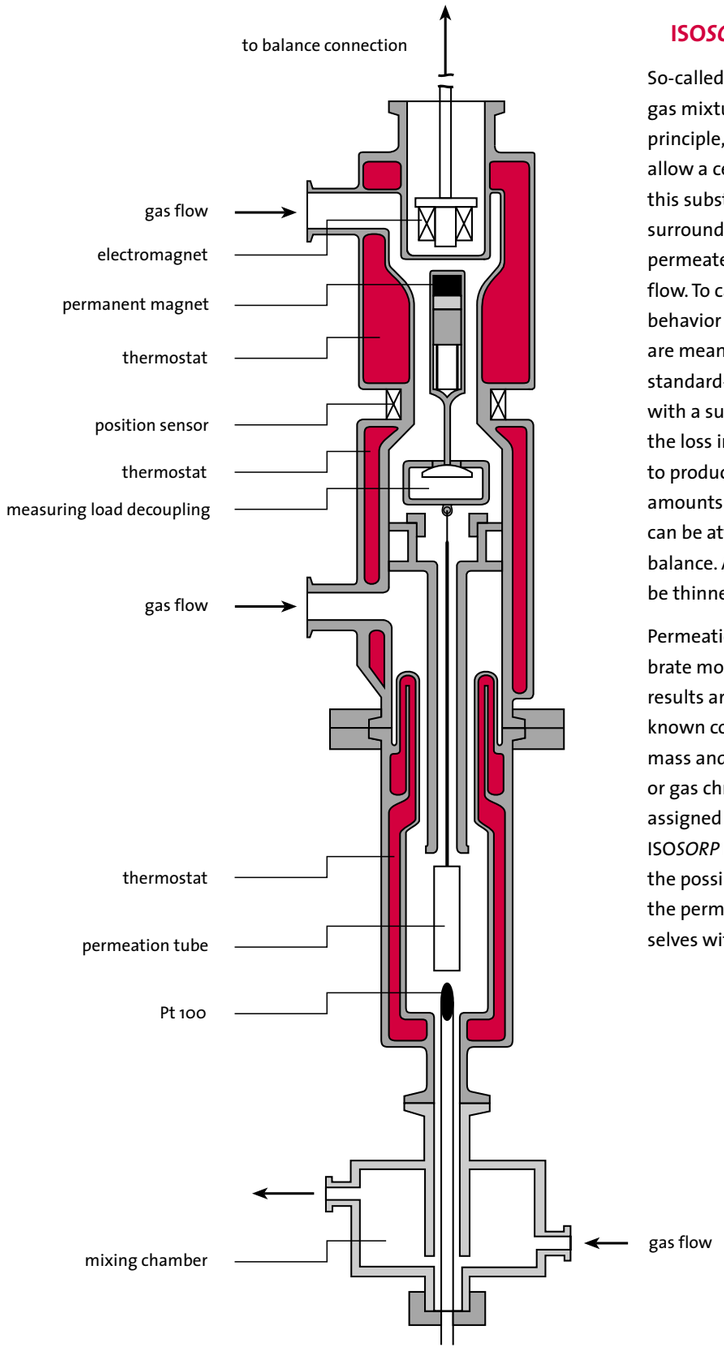
Once the equilibrium has been measured, the valves (V2 and V11) located between the magnetic suspension balance and the gas dosing are closed and the volume of the gas dosing is evacuated using the vacuum pump. The measuring process is then continued by drawing up a new binary gas mixture under higher pressure. This mixture is then also brought into contact with the sorbent until it has reached the equilibrium point. This procedure is continued until all the co-adsorption measuring points which are supposed to be measured have been dealt with. This entire process of gas dosing and measuring is carried out automatically. Up to 10 measuring points in a series can be taken before the sorbent has to be activated.

This large number of sequential measuring points can only be achieved by using a magnetic suspension balance and the ability to measure the density of sorptive gas mixtures. Conventional measuring procedures such as pure volumetry would, through the accumulation of measuring mistakes, lead to results of little value.



Coadsorption measurement of a water / ethanol mixture on activated carbon fibres, BEL Japan Inc.

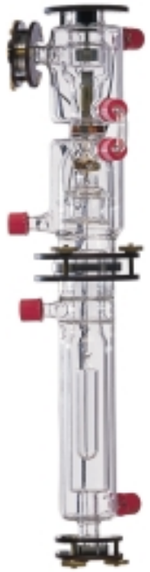
ISO*SORP* 2000 instrumentation examples



ISO*SORP* Permeation

So-called permeation tubes are used to produce gas mixtures with clearly defined composition. In principle, they are closed containers whose walls allow a certain substance to permeate through. If this substance is contained inside and the tube is surrounded by a gas flow, then the substance permeates its way through the walls to the gas flow. To calibrate and control this permeation behavior glass versions of our suspension balance are meanwhile being used at several European standard-institutes. The permeation tube is filled with a substance, connected to the balance, and the loss in mass is measured continuously. In order to produce gas mixtures with particularly small amounts of certain components a mixing chamber can be attached to the magnetic suspension balance. Any gas mixture leaving the balance can be thinned down to a factor between 10 and 100.

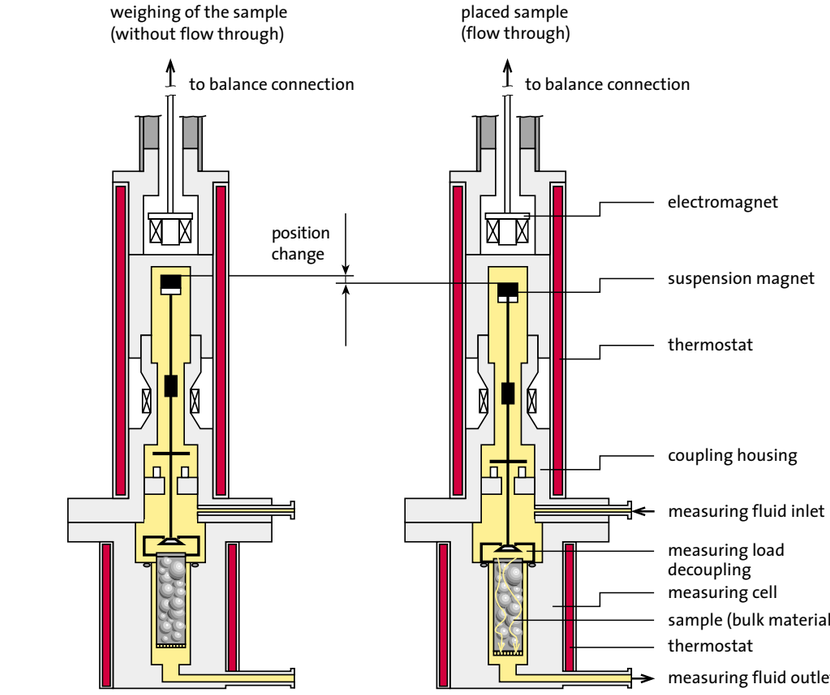
Permeation tubes are definitely needed to calibrate modern gas analysers. Only if the measuring results are compared with a similar gas mixture of known concentration can the results gained with mass and infra-red spectrometers or gas chromatographs be assigned quantitatively. The ISO*SORP* Permeation offers the possibility of calibrating the permeation tubes themselves with utmost precision.



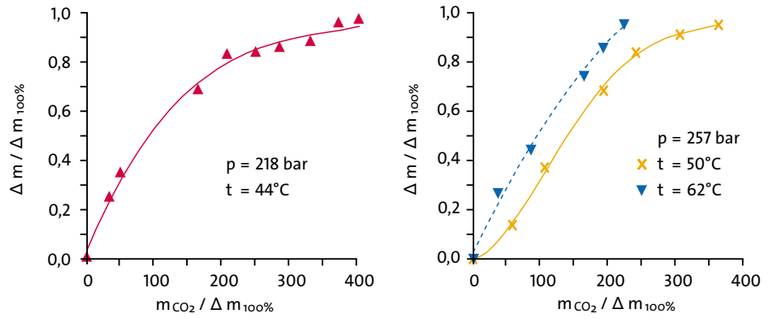
ISO*SORP* Bulk

A special version of the ISO*SORP* was developed to investigate mass transfer processes in forced flow through bulk material beds. This apparatus is used to investigate for example supercritical fluid extraction as close to the real process as possible. Therefore the bulk material container is placed on a sealed plate and large gas volumes flow through it without by-pass flows occurring. The measurement of its weight change due to the mass transfer to the gas phase occurs in brief time intervals. Then the flushing is stopped for a short time and the container is lifted to a freely suspended state and weighed.

Sample container for measurements in forced flow through



Magnetic suspension balance for measurement of mass transfer in forced flow through bulk material beds (ISO*SORP* Bulk)



Delubrication of bleaching earth contaminated with rape oil by means of supercritical CO_2 in a forced flow through bulk material bed, Chair of Process Engineering II, Technical University of Hamburg-Harburg

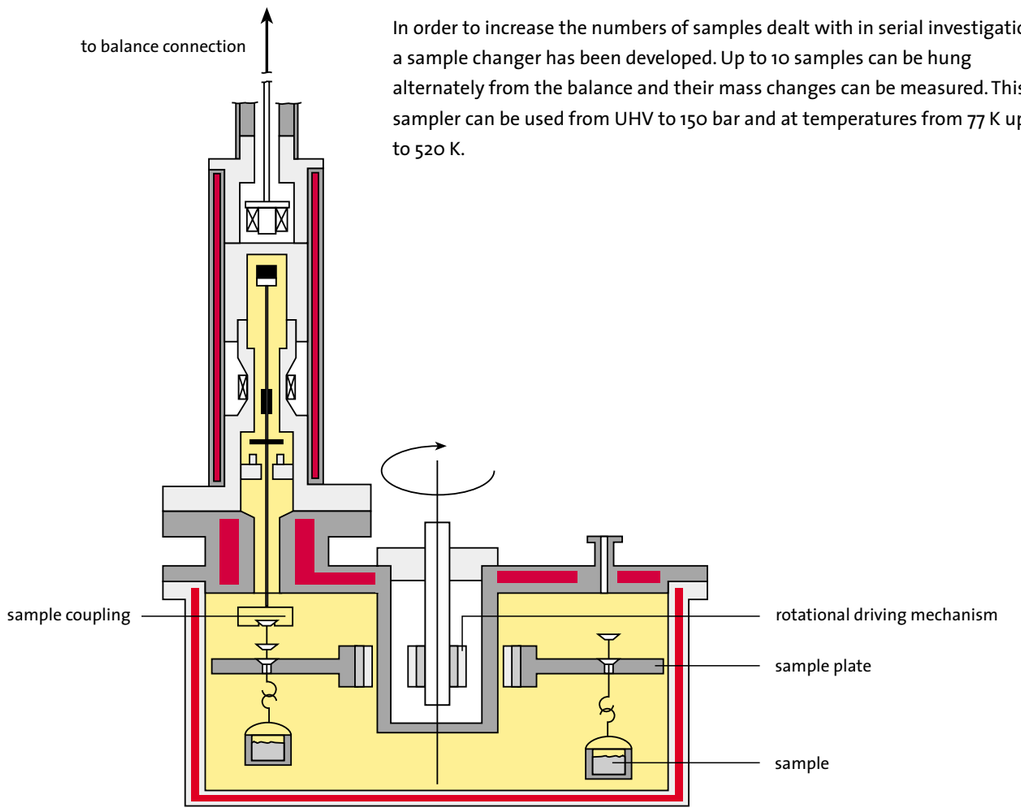
ISO*SORP* 2000 instrumentation examples

ISO*SORP* Visible

An additional optical observation of the sample to be weighed often provides further important information. For example, the swelling of polymers during the sorption process can be photographed and evaluated. Therefore, the ISO*SORP* can be supplied with a measuring cell with two viewing windows through which the samples can be illuminated and observed. The ISO*SORP* can also be fitted with a turning mechanism using the suspension coupling so that a three-dimensional observation can be made. The sample is turned 90° without contact and enables observation on two planes using only one optical system.

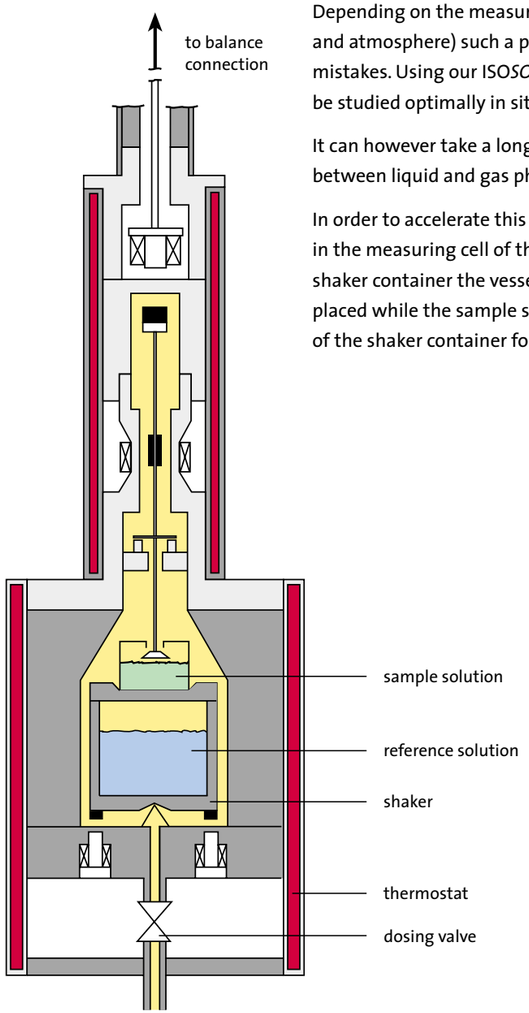
ISO*SORP* Multisample

In order to increase the numbers of samples dealt with in serial investigations, a sample changer has been developed. Up to 10 samples can be hung alternately from the balance and their mass changes can be measured. This sampler can be used from UHV to 150 bar and at temperatures from 77 K up to 520 K.



ISO*SORP* Isopiestic

The aim of isopiestic measurements is to evaluate solvent activities for example in electrolyte solutions. As a result a reference solution of known activity is brought into an equilibrium with the sample solution under isothermal conditions. Therefore, the solvent exchange takes place via the common vapor phase. After establishing the thermodynamic equilibrium in conventional apparatus the sample containers are taken out of the autoclaves and weighed under atmospheric conditions.



Depending on the measuring conditions (pressure, temperature, and atmosphere) such a procedure causes large measuring mistakes. Using our ISO*SORP* Isopiestic the weight change can be studied optimally in situ under process conditions.

It can however take a long time to establish equilibrium between liquid and gas phase.

In order to accelerate this process a shaker has been integrated in the measuring cell of the ISO*SORP*. Inside the copper made shaker container the vessel with the reference solutions is placed while the sample solution is set down on the lid (copper) of the shaker container for an optimal thermal contact.

- **Sorption**
  Adsorption
  Absorption
  Diffusion
  Extraction
  Polymerization
  Coating

- **Thermogravimetry**
  Corrosion
  Decomposition
  Pyrolysis

- **Thermophysical properties**
  Density
  Viscosity
  Surface Tension

RUBOTHERM
PRÄZISIONSMESSTECHNIK GMBH

UNIVERSITÄTSSTRASSE 142
D - 44799 BOCHUM
PHONE: 00 49 - 2 34 - 70 99 6-0
FAX: 00 49 - 2 34 - 70 99 6- 22

WWW.RUBOTHERM.DE