

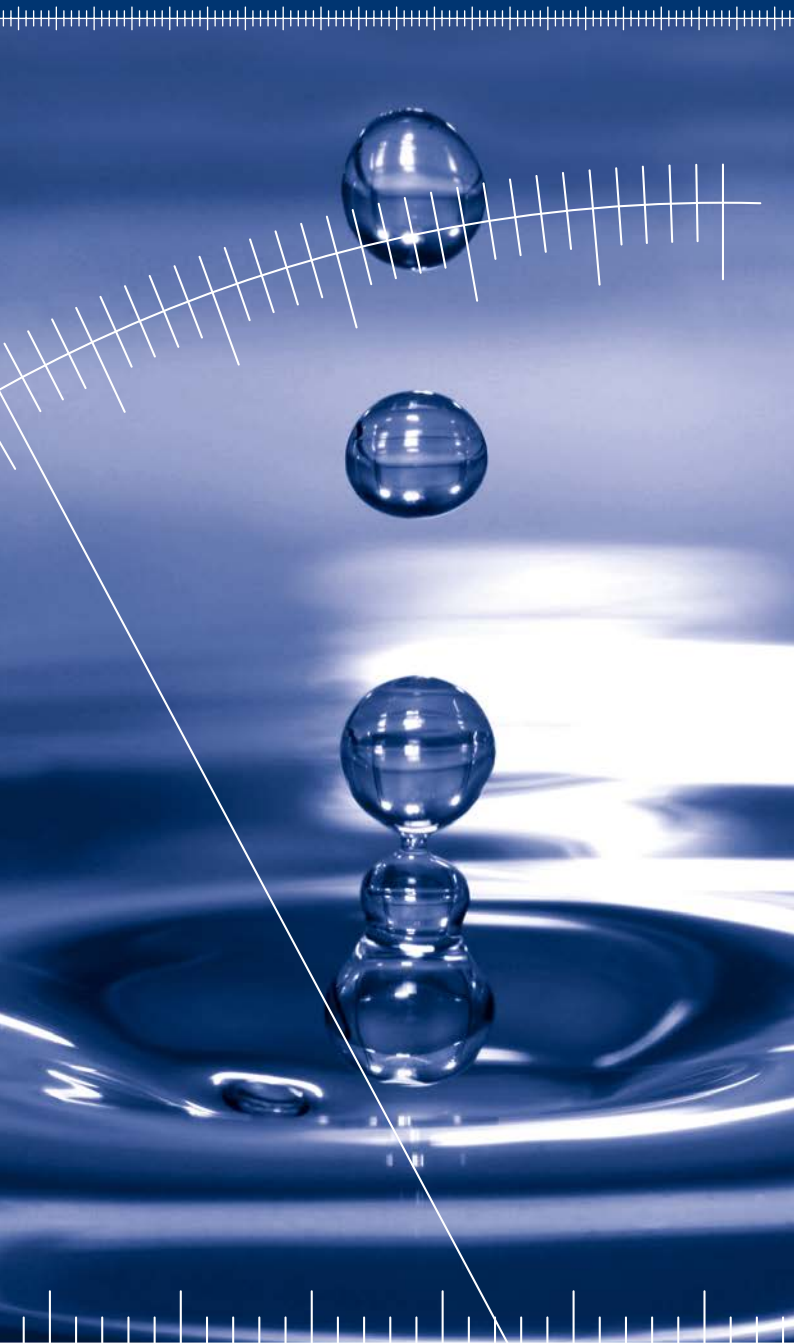
SPS - 水蒸气吸附分析仪

SPS - Moisture Sorption Analysis





水分 Moisture



几乎所有材料和周围空气中的水蒸气都有着或多或少的相互作用。水分引起的物料性质变化是影响产品生产、包装、存储、保质期的关键因素。

Virtually all materials interact more or less strongly with humidity. The knowledge of moisture induced changes of the material properties is a key issue in decisions related to processing, packaging, storage and shelf life of a product.

吸附等温线

The Sorption Isotherm

温度恒定时，样品在不同相对湿度情况下的平衡含水率组成了水蒸气吸附等温线。

吸附等温线可以用来判断基础物料的性质，为物料的处理提供有关水分的参考信息。

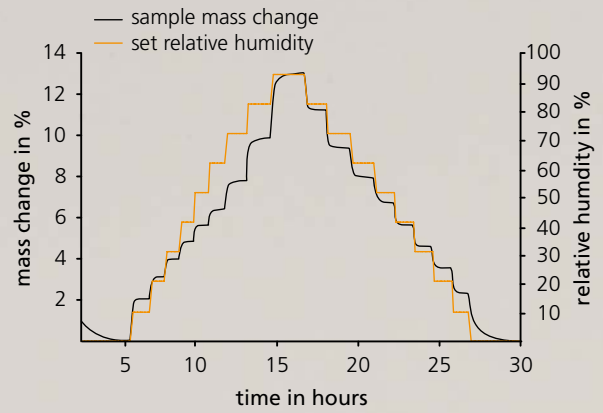
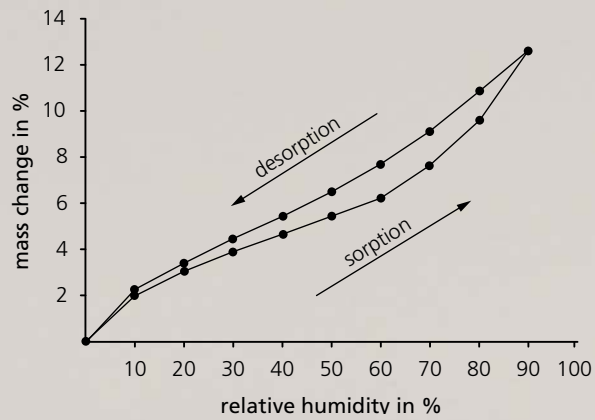
通过物料吸附的动力学曲线，可以判断出水分引起的结构变化，这些变化常常伴随着突然或暂时的含水率变化。

The equilibrium water content of a sample plotted as a function of the relative humidity, at constant temperature, is called the water vapour sorption isotherm.

The sorption isotherm enables to draw conclusions on fundamental material properties and provides information on material handling with respect to moisture.

From the sorption kinetics curve of a material, moisture induced structural changes are determined that are often accompanied by sudden, temporary changes in moisture content.





微晶纤维素粉末的吸附等温线和吸附动力学
Sorption isotherm and sorption kinetics of microcrystalline cellulose powder



样品高通量 High Sample Throughput

多样品分析仪

SPS吸附分析仪最多可同步检测23个样品，和单样品检测仪器相比，该仪器提供了非常大的样品通量。

物料放在样品托盘上的一圈实验皿里，通过托盘的持续旋转和升降，每隔一个固定的时间，实验皿下降到天平上方进行称重。

Multi-sample instrument

The SPS sorption test systems enable the measurement of up to 23 samples simultaneously. This gives a very high analysis throughput compared with a single-sample instrument.

Sample material in small dishes is placed in a circular arrangement on a sample tray. By continuously turning and lowering of the tray, the dishes are placed onto the balance and weighed at fixed time intervals.





样品大小和称重范围

Sample Size and Weight Range

样品大小变化多:

仪器配备了不同尺寸的实验皿，最大直径为50mm的样品。大实验皿提供了大的样品吸附表面，从而缩短检测周期。

重量动态范围广:

SPS吸附分析仪可以检测重量从几毫克到上百克的样品，在检测过程中，无论样品初始的重量是多少，称重范围都能达到天平量程的最大限。

Variable sample size

Sample dishes of different size enable the handling of samples up to 50 mm in diameter. A large dish gives a large sample surface area for sorption to take place, resulting in shorter measurement cycles.

High dynamic weight range

Sample weights from a few milligrams up to several grams are handled by the SPS system. In a test run, the full weighing range up to the maximum load is available for all samples independent from the sample weight.





样品大小和称重范围

Sample Size and Weight Range

对于水分含量非常低的物料，将大量的样品呈薄层状平铺于大的实验皿中才能得到更准确的结果。

各种不同种类的样品，从精细粉末、颗粒、片剂、胶囊、纸张、薄片到棉花、纤维、较重的或是块状的样品，如木料和建筑材料等都可以用SPS进行分析。

For materials with very low water uptake, high accuracy is achieved by using a larger volume distributed in a thin layer using a large dish.

All different kinds of samples from fine powders, granules, tablets and capsules, papers and foils, even cotton and fibers and also heavy and bulky samples like wood and construction materials are handled by the SPS instruments.





SPS版本与技术参数

SPS Versions and Technical Data

| | High versatility with the SPSx-1 μ series | |
|----------------------|--|---|
| SPS instrument | SPSx-1 μ Advance | SPSx-1 μ High Load |
| | High resolution and reproducibility for applications from pharmaceutical research up to material testing | High resolution and very high load range for applications in the food and chemical industry, including permeability tests |
| Number of samples | 11/23 | |
| Sample size, max | Ø 33 mm/Ø 50 mm | |
| Load range, min/max | < 10 mg - 22 g | < 30 mg - 22 g/220 g (dual range) |
| Resolution | 1 μ g | 1 μ g/10 μ g |
| Repeatability | $\pm 2 \mu$ g | $\pm 5 \mu$ g/ $\pm 20 \mu$ g |
| Temperature | 5 °C to 60 °C | |
| Accuracy | ± 0.1 K time/ ± 0.5 K uniformity | |
| Relative Humidity | 0 % RH to 98 % RH* | |
| Accuracy | ± 0.6 % RH (0 ... 98 % RH) at 23 ± 5 °C | |
| Hardware accessories | CCD video camera Raman spectroscopy | CCD video camera Raman spectroscopy Permeability Kit |
| Software option | CFR21 part 11 software package | |

* 注：在温度略高于室温的情况下才能达到全湿度范围

* Note: The full humidity range is only available at chamber temperatures slightly above room temperature.

| | Solutions for high demanding applications | |
|----------------------|---|--|
| SPS instrument | SPS23-100n | SPS11-10 μ |
| | Highest resolution and sensitivity for applications in pharmaceutical research and preformulation | High load range for applications in building physics, quality control and for permeability tests |
| Number of samples | 23 | 11 |
| Sample size, max | \varnothing 18 mm | \varnothing 50 mm |
| Load range, min/max | < 10 mg - 2000 mg | < 50 mg - 111 g/220 g (dual range) |
| Resolution | 0.1 μ g | 10 μ g/100 μ g |
| Repeatability | \pm 1.5 μ g | \pm 20 μ g/ \pm 100 μ g |
| Temperature | 5 °C to 40 °C | 5 °C to 60 °C |
| Accuracy | \pm 0.1 K time/ \pm 0.5 K uniformity | |
| Relative Humidity | 0 % RH to 98 % RH* | |
| Accuracy | \pm 0.6 % RH (0 ... 98 % RH) at 23 \pm 5 °C | |
| Hardware accessories | CCD video camera Raman spectroscopy | CCD video camera Raman spectroscopy Permeability Kit |
| Software option | CFR21 part 11 software package | |

* 注：在温度略高于室温的情况下才能达到全湿度范围

* Note: The full humidity range is only available at chamber temperatures slightly above room temperature.

SPS的硬件配件

SPS Hardware Accessories

CCD摄像机

通过联用高分辨率CCD摄像机现场记录吸附试验过程中由水分引起的视觉变化。

渗透率检测组件

使用SPS水分吸附仪检测薄片或薄膜的渗透率请参考ENISO7783-1，水蒸气渗透率检测的第一部分“游离膜的碟皿检测法”。

拉曼光谱

水分吸附仪和拉曼光谱的联用为固体材料的分析带来了更广阔的前景。

CCD video camera

In-situ documentation of moisture induced visual changes during a sorption measurement with an adapted high-resolution CCD camera.

Permeability Kit

Determination of the permeability of foils and films with the SPS moisture sorption instruments according the EN ISO 7783-1 for the determination of water-vapor transmission rate part 1: "Dish method for free films".

Raman spectroscopy

Sensor fusion combining water vapour sorption and Raman spectroscopy opens new and exciting perspectives for the analysis of solid materials.



拉曼光谱仪
Raman spectroscopy



Permeability



SPS仪器使用体验

Working with the SPS instruments

样品制备

只需将样品放进实验皿中，再简单的放置在样品托盘上。符合人体工程学的结构设计和非常容易打开的实验腔使得准备样品成为非常轻松的过程。

Sample preparation

Samples are simply filled into dishes that are easily placed on the sample tray. The sound construction and easy to access measurement chamber render sample preparation a comfortable procedure.





SPS仪器使用体验

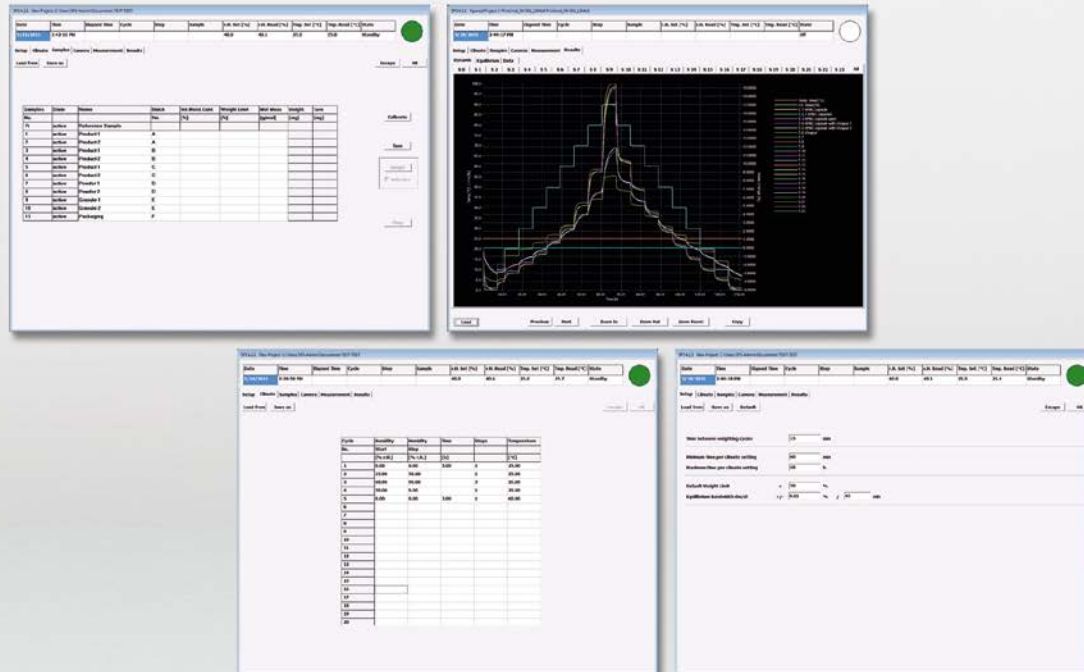
Working with the SPS instruments

操作流程

- 表格式输入使得检测周期的设置非常快捷，即使是非常复杂的水分吸附研究也是如此；
- 可以设计的吸附/解吸循环；
- 可选择在检测开始之前干燥样品或是平衡样品；
- 可使用范本来规范标准检测流程；
- 软件界面友好，具备用户指导功能，避免操作错误。

Step-by-step procedure

- A tabular input enables quick and easy setup of measuring cycles even for very complex water sorption studies
- Programmable sorption / desorption cycles
- Optional initial sample drying, or equilibration before starting the actual measurement
- Templates are available to specify standard measurement procedures
- User-friendly and intuitive interface with active user guidance prevent operating errors





SPS 4.2.0 New Project: 20130307_8CK302

| Date | Time | Elapsed Time | Cycle | Step | Sample | r.H. Set [%] | r.H. Read [%] | Temp. Set [°C] | Temp. Read [°C] | State |
|----------|------------|--------------|-------|------|--------|--------------|---------------|----------------|-----------------|---------|
| 3/7/2015 | 5:39:40 PM | 00:00:14 | 1 | 0 | | 0.0 | 2.9 | 25.0 | 25.0 | Running |

Setup | Climate | Samples | Measurement | Results

SPS

Logout

Dr. Petra Expert Lab Group Leader

Close

- Water
- Door
- Autosampler
- Peltier
- Column

On

Off

Climate

On

Off

Nominal Actual

Temperature 25.0 25.0

Humidity 0.0 2.9

Set

Measurement

Project

New

Open

Data

- Climate
- Tare
- Weight

Measure

Start

Next Step

Stop

Pause

On

Next Sample

First Setting

Last Setting

Other

°C

% r.H.

Logging

Sample 1: Batch changed to CRM 0459
 Sample 7: Batch changed to CRM 0459
 Sample 1: Initial Moisture Content changed to 1.23 %
 Sample 7: Initial Moisture Content changed to 1.23 %
 Sat Mar 07 16:28:34 2015: Dr. Petra Expert Lab Group Leader
 Door opened
 Sat Mar 07 16:29:47 2015: Dr. Petra Expert Lab Group Leader
 Door closed
 Sat Mar 07 16:30:34 2015: Dr. Petra Expert Lab Group Leader
 Tare
 Sat Mar 07 16:36:29 2015: Dr. Petra Expert Lab Group Leader
 Confirmed sample changes:
 Sample 0: Tare changed to 1151.193 mg
 Sample 1: Tare changed to 1171.741 mg
 Sample 7: Tare changed to 1176.672 mg
 Sat Mar 07 16:37:29 2015: Dr. Petra Expert Lab Group Leader
 Climate set. Nominal 25.0°C 2.0 %r.H. Actual 25.0°C 20.0 %r.H.
 Sat Mar 07 16:37:45 2015: Dr. Petra Expert Lab Group Leader
 Door opened
 Sat Mar 07 16:38:38 2015: Dr. Petra Expert Lab Group Leader
 Door closed
 Sat Mar 07 17:07:30 2015:
 Logged out automatically
 Sat Mar 07 17:32:41 2015: Dr. Petra Expert Lab Group Leader
 Checked in
 Sat Mar 07 17:32:41 2015: Dr. Petra Expert Lab Group Leader
 Weight
 Sat Mar 07 17:38:39 2015: Dr. Petra Expert Lab Group Leader
 Confirmed sample changes:
 Sample 1: Wet Weight changed to 145.392 mg
 Sample 7: Wet Weight changed to 211.240 mg
 Sat Mar 07 17:39:26 2015: Dr. Petra Expert Lab Group Leader
 Measurement start

选配了CFR21的SPS软件
 SPS Software with CFR21 option

检测流程

Measuring Process

完全自动化的检测过程：

一旦按下“start”按钮，仪器自动开始检测并采集数据，整个过程无需人为干涉。实验过程中可以透过仪器的透明上盖直接观察样品。

优化的气流：

持续的气流通过样品表面带来了恒定的气氛循环，精确的湿度控制使得样品在此环境下能够快速到达平衡，缩短试验周期。

天平的稳定性：

内标实验皿完美的保证了漂移补偿和基线的稳定，且天平具备全自动校准功能。

Fully automated process

As soon as the “Start” button is pressed, experiments run unattended by automated measurement and data acquisition. The transparent glass cover enables the direct observation of the samples during the test.

Optimized gas flow

Permanent circulation of the atmosphere with an efficient gas flow along the sample surface and precise humidity regulation enable a fast equilibration of the samples to the respective climatic conditions, resulting in shorter measurement cycles.

Balance stability

An internal reference ensures an excellent drift compensation and baseline stability. Calibration of the balance is fully automated.





检测结果

Measurement Results

数据格式:

采集到的数据保存为电子安全数据格式。可以将数据输入Excel模板中，用这种标准的office工具实现所有统计和可视化功能。

Excel模板中图表和表格包括:

- 作为温湿度环境和时间的函数的样品重量动态变化
- 不同相对湿度下，平衡状态下的样品含水率

Excel中所有的图表都可以进行用户配置

数据安全:

标准的SPS软件可以升级成为SPS.CFR21软件包，来满足CFR21.part.11中的关于数据记录安全的要求。

Data format

Acquired data is saved in an electronically secured data format. Data import into an Excel® template offers all the statistical and visual capabilities of this standard office tool.

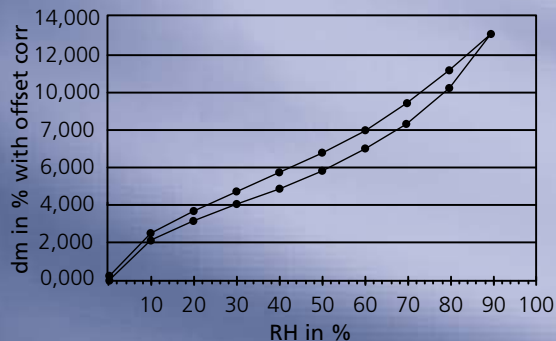
The graphics and tables in the Excel® template include:

- Dynamic change of the sample mass as a function of time and climatic conditions
- Equilibrium water uptake at different relative humidity levels

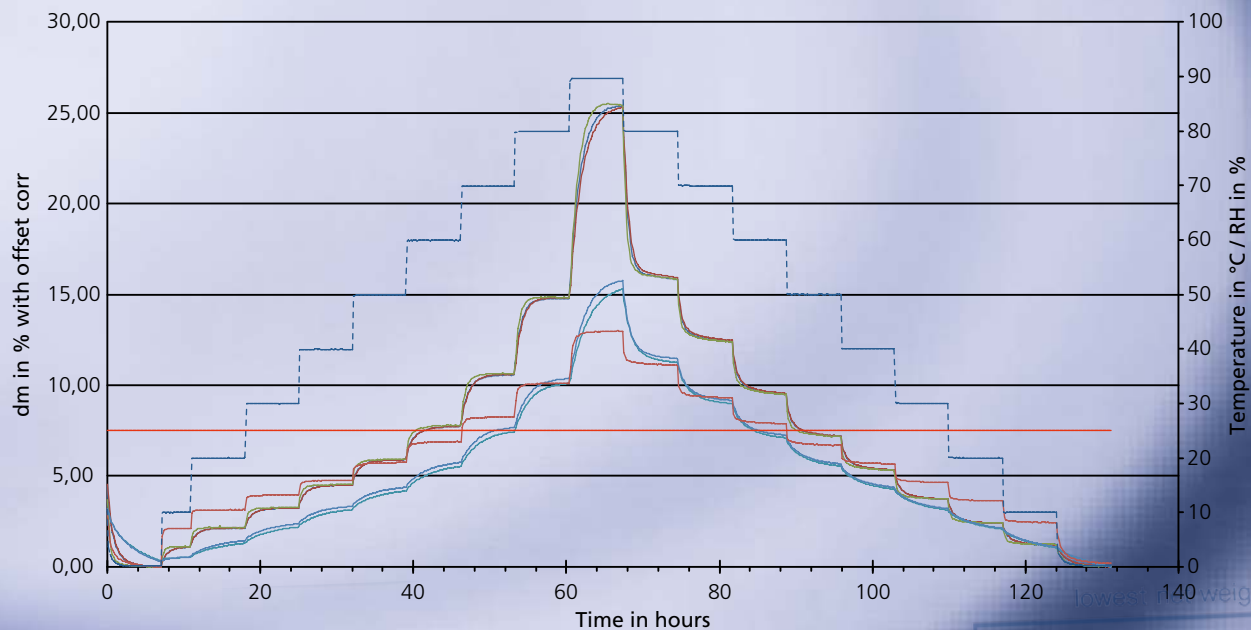
All graphics and calculations in Excel® are fully user configurable.

Data security

The standard SPS software is upgradable with an optional SPS CFR 21 package in order to meet the data security requirements for electronic records in accordance with CFR21 part 11.



| No. | Time [h] | Total Weight [mg] | Net Weight [mg] (Sample Tara) |
|-----|----------|-------------------|-------------------------------|
| 1 | 0,05 | 1115,448 | 136,824 |
| 2 | 0,19 | 1115,350 | 136,770 |
| 3 | 0,29 | 1115,330 | 136,754 |
| 4 | 0,41 | 1115,387 | 136,741 |
| 5 | 0,53 | 1115,399 | 136,745 |
| 6 | 0,64 | 1115,396 | 136,738 |
| 7 | 0,76 | 1115,387 | 136,728 |
| 8 | 0,88 | 1115,385 | 136,726 |
| 9 | 0,99 | 1115,377 | 136,718 |
| 10 | 1,11 | 1115,378 | 136,710 |



lowest weight 136,617
Data for Equilibrium Curve

| | RH Set [%] | Temp. Set [°C] | Temp. Read [°C] | Equilibrium (1) | Mol ratio | net weight at Equilibrium | |
|--|------------|----------------|-----------------|-----------------|-----------|---------------------------|---------|
| | 0,0 | 11,5 | 25,0 | 24,7 | 0 | -0,05847 | 136,617 |
| | 0,0 | 4,9 | 25,0 | 25,1 | 0 | -0,09792 | 136,62 |
| | 0,0 | 3,6 | 25,0 | 24,9 | 0 | -0,10958 | 136,61 |
| | 0,0 | 2,5 | 25,0 | 25,0 | 0 | -0,11907 | 136,62 |
| | 0,0 | 2,1 | 25,0 | 24,9 | 0 | -0,11611 | 136,63 |
| | 0,0 | 1,7 | 25,0 | 25,0 | 0 | -0,12125 | 136,62 |
| | 0,0 | 1,5 | 25,0 | 25,0 | 0 | -0,12857 | 136,62 |
| | 0,0 | 1,3 | 25,0 | 24,9 | 0 | -0,13005 | 136,62 |
| | 0,0 | 1,2 | 25,0 | 25,0 | 0 | -0,13688 | 136,63 |
| | 0,0 | 1,1 | 25,0 | 25,0 | 0 | -0,14171 | 136,64 |
| | 0,0 | 1,1 | 25,0 | 25,0 | 0 | -0,14785 | 136,64 |
| | 0,0 | 1,0 | 25,0 | 25,0 | 0 | -0,15399 | 136,63 |

应用

Applications

SPS吸附仪专门为多种工业和科学应用设计，适用于不同学科：制药领域、食品科学、化妆品、塑料工业、化学、纺织工业、生物学、矿物学等等。

动态水蒸气吸附仪适用于检测多种固体物料，如粉末、颗粒、小球、片剂、薄膜和带包装物品的水分吸附特性：

- 水蒸气吸附/解吸的质量和动力学
- 平衡含水率

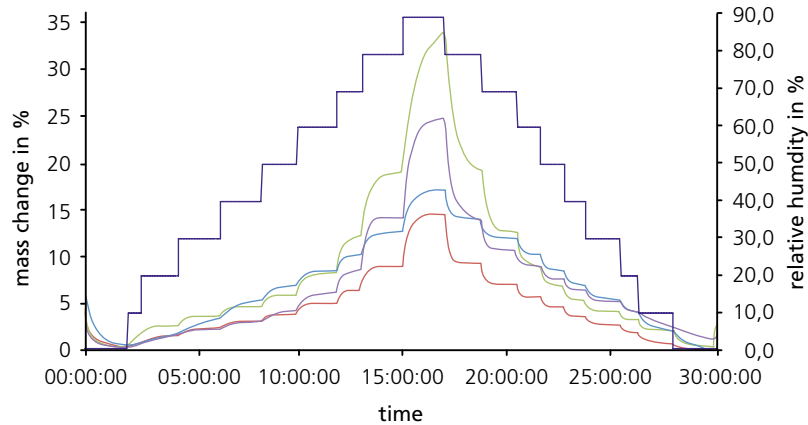
The SPS instruments are designed for a multitude of industrial and scientific applications from different disciplines: pharmaceuticals, food science, cosmetics, plastics industry, chemistry, textile industry, biology, mineralogy, etc.

Dynamic vapour sorption is applied to measure

- Quantity and kinetics of water vapour sorption/desorption
- Equilibrium moisture content

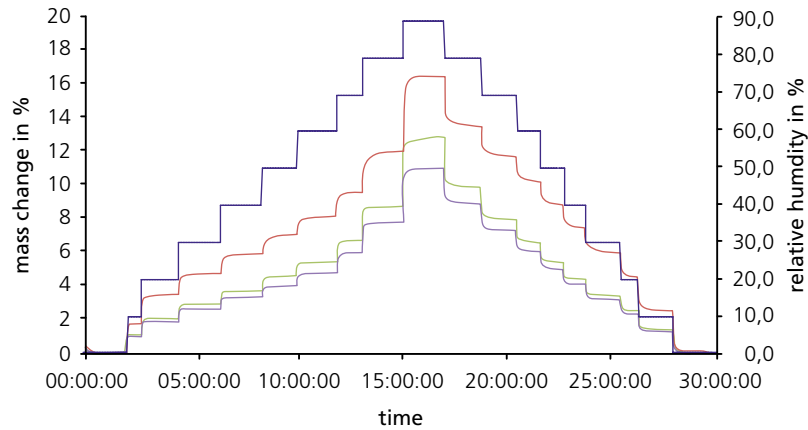
of a variety of solid materials like powders, granules, pellets, tablets, films or packages.





食品原料的水蒸气吸附
Water vapour sorption of food materials

- Marshmallows
- Potato chips
- Basmati rice
- Linseed
- Set relative humidity



纺织材料的吸附动力学
Sorption kinetics of textile materials

- Sheep wool, raw
- Cotton, raw
- Cotton, white purified
- Set relative humidity

应用 Applications

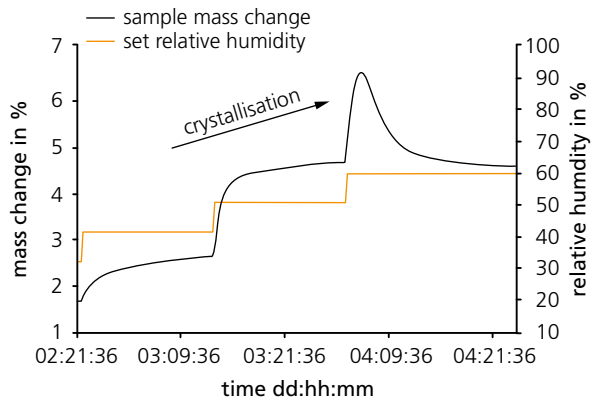
具体应用于:

- 水合物的形成和水分引起的结构变化 (结晶)
- 物料的热力学参数, 如吸附/解吸热
- 不同孔隙率的测定
- 扩散率和渗透率的研究 (薄膜和包装)
- 质量保证: 检测同一产品不同批次的细微差别

Specific applications are:

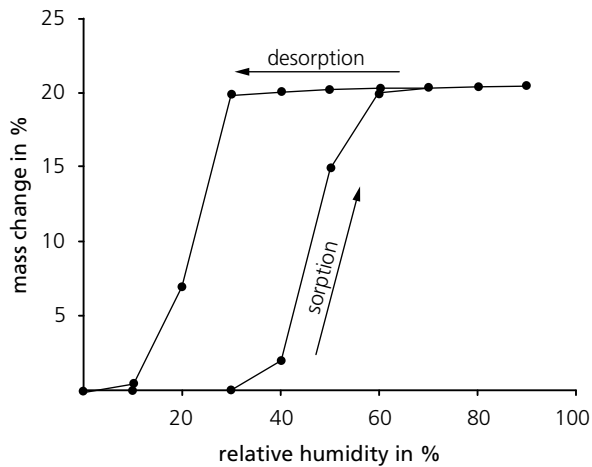
- Hydrate formation and moisture induced structural transformations (crystallisation)
- Thermodynamic parameters of materials such as heat of sorption and desorption
- Determination of porosity differences
- Diffusion and permeability studies (films and packages)
- Quality assurance: determination of subtle differences between different batches of a product





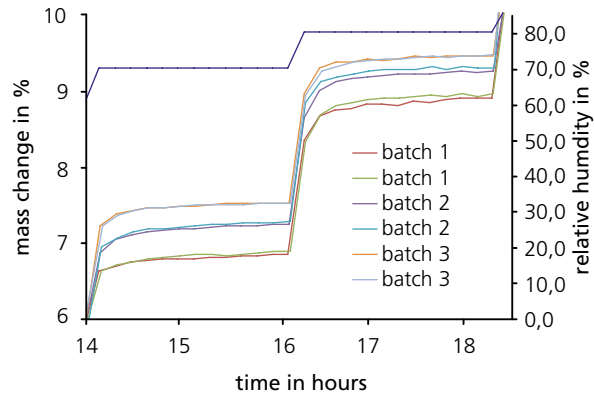
脱脂奶粉中无定型乳糖的结晶过程

Crystallisation of amorphous lactose in skimmed milk powder



氨基酸水合物的吸附等温线

Sorption isotherm of a hydrate forming amino acid



三个批次的纤维素粉末在70%和80%RH条件下的水分吸收情况
同批次样品具有非常好的重复性
也可以准确的检测到批次间细微差距

Water uptake of three batches of cellulose powder at 70 % and 80 % RH, net weight: 100 mg

Perfect repeatability of results within the same batch
Precise determination of subtle differences between batches



硬糖在湿度条件下的视觉变化记录

Visual documentation of the crystallisation of hard candy at high relative humidity

SPS概述

The SPS in a nutshell

高通量:

23个样品处于同一个温湿度环境下同步检测，保证了样品之间可以直接进行比较。

重量动态范围广:

样品重不超过2g时天平分辨率为 $0.1\mu\text{g}$ (SPS23-100n)，样品大于100g时，天平分辨率为 $10\mu\text{g}$ (SPS11-10 μ)，保证了微量样品检测的准确度，同时在样品量大的情况下也能够准确测量出其微小的重量变化。

基线校准:

内部参比称重保证在整个称重过程中的漂移得到补偿和基线的稳定。

多功能性:

可选择与摄像机、拉曼光谱和检测薄膜或包装材料的渗透率组件联用。

可持续性设计:

公司1999年销售的第一台商业用途的仪器仍在正常运行，可以使用最新的SPS软件版本，而且可以与最新的硬件兼容。

High sample throughput

Measurement of up to 23 samples simultaneously at the same climatic conditions enables the direct comparison of the samples.

High dynamic weight range

Balance resolution of $0.1\mu\text{g}$ for samples up to 2 g (SPS23-100n) and $10\mu\text{g}$ for samples $> 100\text{g}$ (SPS11-10 μ) enables the analysis of small samples as well as the detection of small changes within large samples.

Base line correction

An internal reference weight ensures an excellent drift compensation over the whole weighing process.

Versatility

Optional equipment like video camera, Raman spectroscopy and permeability measurement of foils and packages is available for the SPS instruments.

Sustainable design

Even the first commercial available instruments from 1999 run with the latest SPS software version and are compatible with the newest hardware.

