Thermal Analysis Option

TGA-Sorption System

Innovation Expands Flexibility

The new TGA-Sorption System is designed to analyze materials under defined conditions of temperature and relative humidity (RH). The design concept combines the performance and versatility of the large furnace TGA/DSC 1 and TGA/SDTA85x^e thermoanalyzers with a unique interface approach that allows the humidity generator to be added within a few minutes.

The TGA-Sorption System is available in two versions. Each consists of a thermoanalyzer, a humidity generator, and an interface. In the simpler version, the humidified gas produced in the generator is transferred to the thermoanalyzer via a heated transfer line. The more sophisticated version produces the humidified gas in a mixing chamber close to the interface.

Measurements under conditions of controlled relative humidity provide information that is crucial for understanding the effects that moisture content can have on the properties of a wide range of materials:

- **Processing** – spray-dried powder that becomes moist can block production equipment
- **Shelf life of products** – may be reduced due to moisture uptake caused by inadequate packaging
- **Structural properties** – moisture content can affect bioavailability and influence the therapeutic effect

Features and benefits

- **Unique flexibility** – a highly sensitive thermoanalyzer combined with full sorption analysis capability
- **Defined environment** – the effect of moisture and temperature on material properties can be easily investigated
- **Preconditioning versatility** – methods allow the use of elevated preconditioning temperatures (up to 150 °C)
- **DSC signal** – simultaneous measurement of sorption enthalpies
- **Modularity** – an existing large furnace TGA/DSC 1 or TGA/SDTA85x^e thermoanalyzer can be quickly expanded to perform humidity experiments

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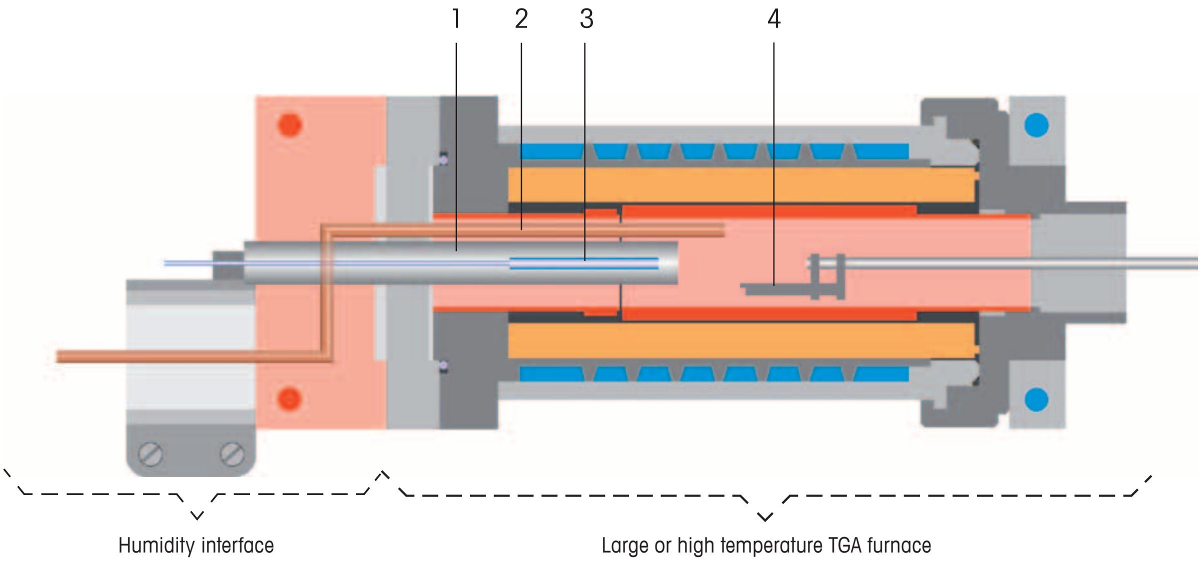
The Humidity Interface
for Precise Control of Relative Humidity

The TGA-Sorption System allows you to precondition samples at temperatures up to 150 °C and to increase or decrease the relative humidity continuously or in steps. You can also simultaneously measure sorption enthalpies and analyze the sorption results. This makes the TGA-Sorption System one of the most versatile instruments for sorption analysis on the market.

The modular concept of the large furnace TGA/DSC 1 and TGA/SDTA85x[e] thermoanalyzers allows the humidity generator to be quickly attached via the unique humidity interface. The TGA-Sorption System extends the application possibilities of both thermoanalyzers. With one TGA system, you can now perform:

- Traditional weight-change experiments up to 1100 °C or 1600 °C
- Sorption studies under conditions of defined relative humidity and temperature

Schematic diagram of part of the TGA-Sorption System showing the humidity interface with the large TGA furnace or the high temperature TGA furnace:



1 Furnace outlet
2 Humidity supply capillary positioned close to the sample
3 Optional RH sensor
4 Sample holder attached to the thermostated TGA microbalance

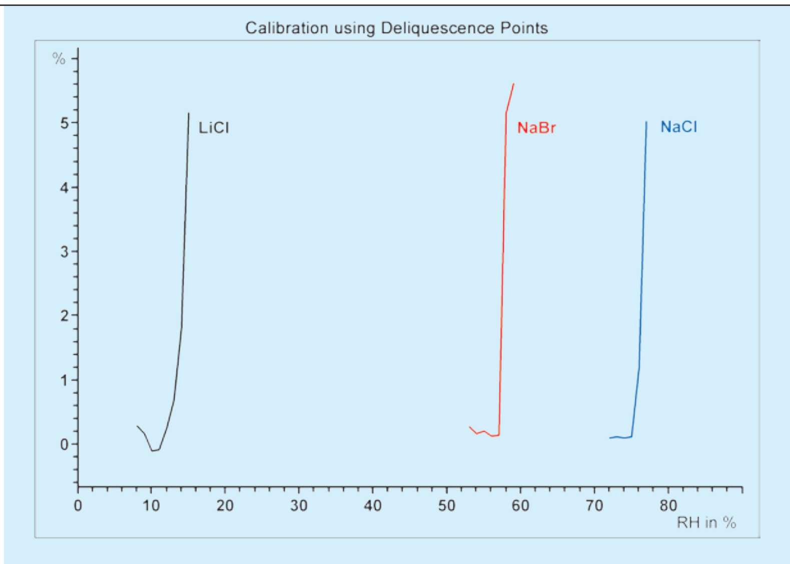
The influence of humidity

The influence of humidity depends on the material or the constituents of the formulation or product. In general, the moisture absorbed by materials is classified either as free water or as bound water:

Free water	Bound water
• is water absorbed from the environment, often as surface moisture	• is water chemically bound as water of crystallization (water of hydration)
• is not chemically bound to the material	• forms part of the crystal structure
• is usually only weakly bound and more easily desorbed than bound water	• affects the physical appearance and material properties

Industry	Applications
Common for all industrial segments	• Storage and stability • Moisture migration • Surface adsorption and thermal desorption • Processability of raw materials
Pharmaceutical and personal care	• Activity of ingredients • Effect of fillers • Phase transformations • Loss on drying • Development of spray drying technology • Moisture sorption and hydrophilic characterization
Food flavorings and ingredients	• Aging of food products • Analysis of dehydrated structures • Oxidation • Non-enzymatic browning • Enzymatic changes • Material flow and crystallization • Plasticization and swelling characteristics
Plastics (elastomers, thermosets, thermoplastics), films and fibers	• Plasticization influence on material and product performance
Catalyst and porous support materials	• Variations in surface activity
Construction materials and minerals	• Stability of cement • Wood, metal and rust investigation • Impact on explosives
Chemicals	• Fertilizers and surface activity

Calibration and Performance Qualification

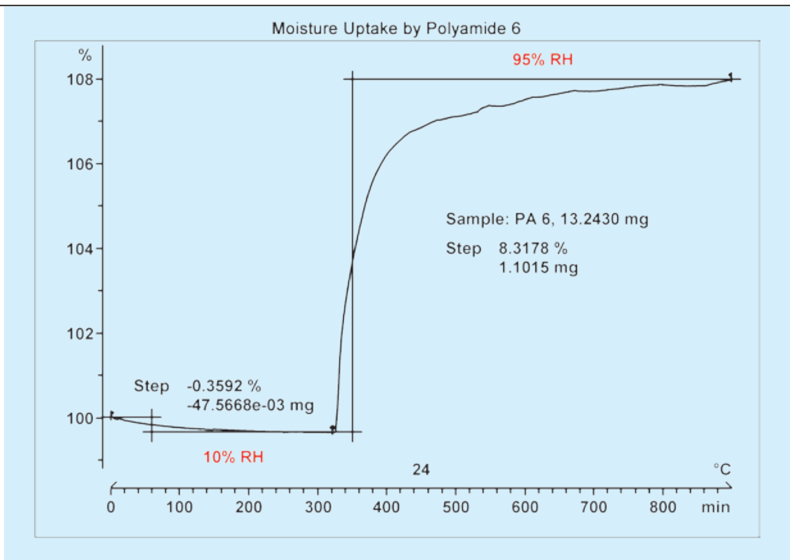


Calibration

The calibration of the TGA-Sorption System makes use of the phenomenon of deliquescence, the process by which a substance absorbs water vapor from the air and forms a solution. This occurs when the vapor pressure of the solution that is formed is less than the partial vapor pressure of the water vapor in the air (i.e. the RH). The response is an increase in weight. The relative humidity (RH) at which deliquescence begins is called the deliquescence point. The examples shown in the plot at 25 °C cover a wide range of RH values:

- lithium chloride (11.3%)
- sodium bromide (57.6%)
- sodium chloride (75.3%)

Application Examples

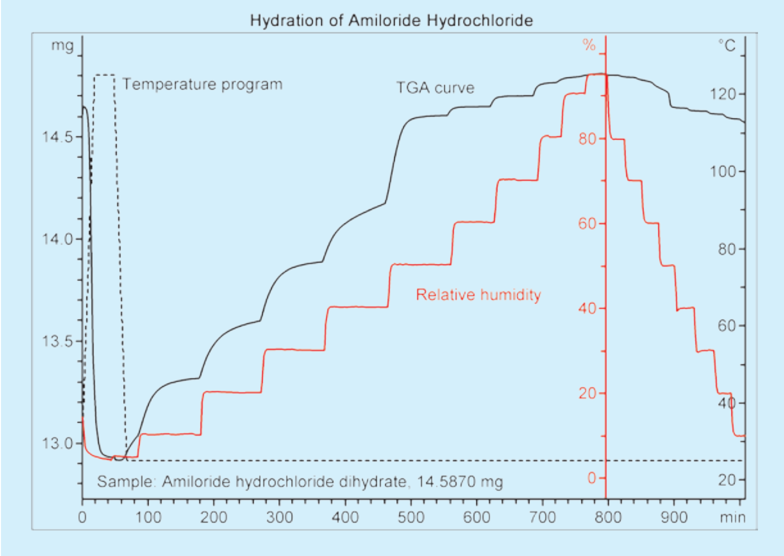


Plasticization

The water content of polymers can drastically change their physical and chemical properties. The main effects are:

- a reduction in mechanical strength through plasticization
- a shift in the glass transition to lower temperatures due to increased free volume and swelling.

Polyamides absorb appreciably more moisture than most other engineering plastics. The example shows the measurement of a sample of polyamide 6 in which the relative humidity was changed from 10 to 95% RH at 24 °C. Under these conditions, the weight increased by about 8.3% after 10 hours.

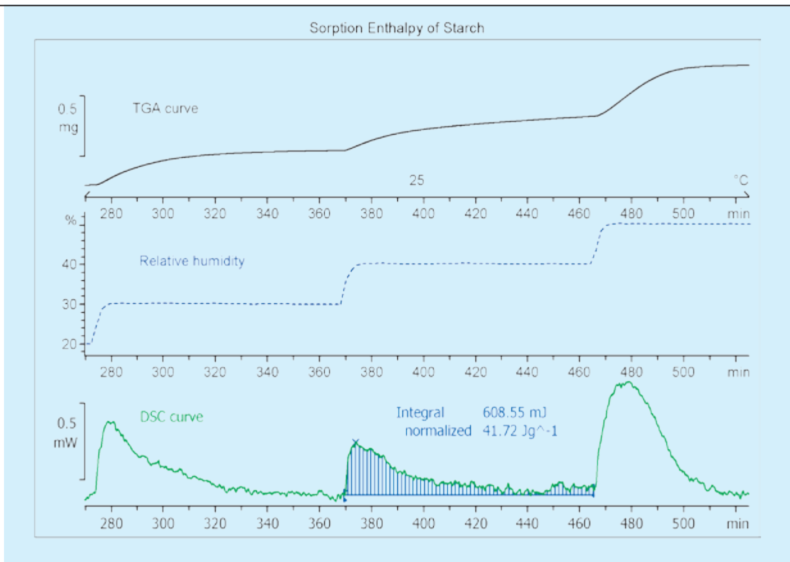


Dynamic sorption curve

The TGA curve shows the uptake and release of moisture by a sample of amiloride hydrochloride dihydrate as a function of RH. The stages of the analysis include:

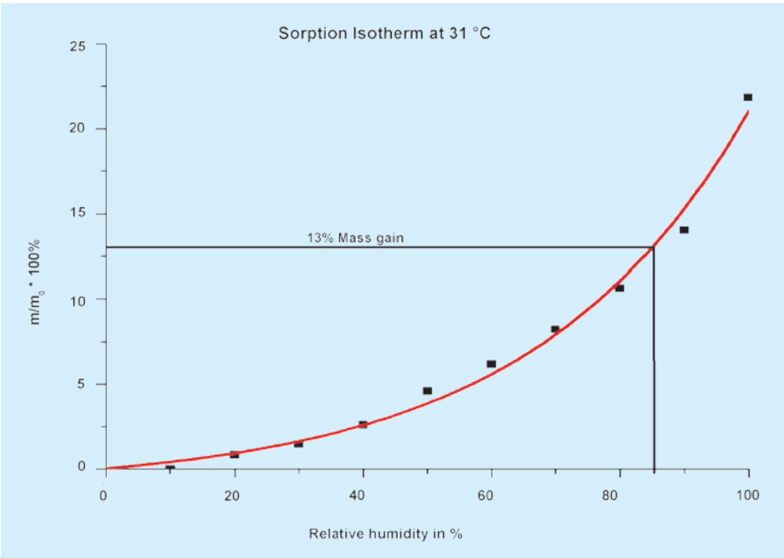
- temperature program with a preconditioning segment (dehydration) at 125 °C (dotted line)
- increase of RH in steps of 5% with equilibration (red curve)
- resulting weight changes for each 5% change in RH (black curve)

At a RH of 50%, the substance has regained its original water of crystallization. Further increase of RH results in the uptake of free surface water. This is liberated when the RH is reduced.



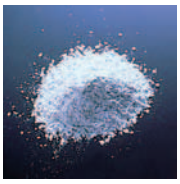
Sorption enthalpy

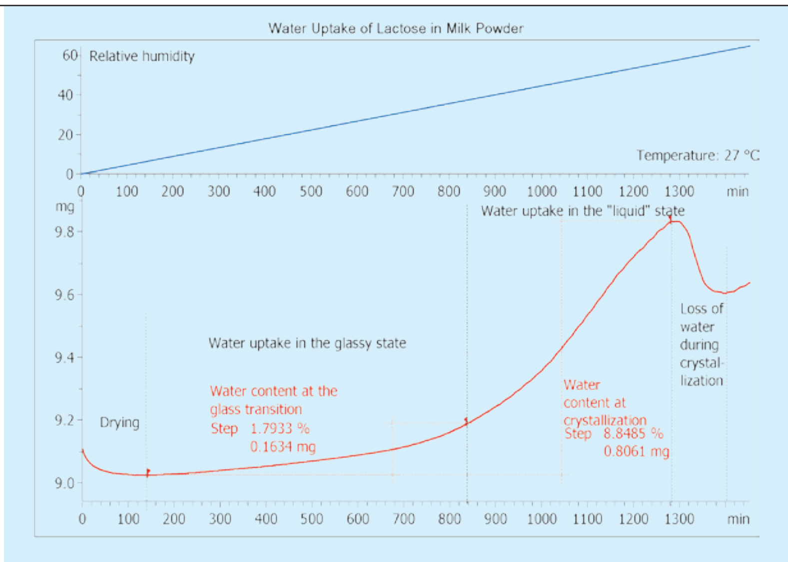
The TGA-Sorption System also allows you to measure sorption enthalpies. The figure summarizes the results obtained when a modified starch sample was subjected to a dynamic sorption program. The DSC curve shows exothermic peaks that slowly tail off after each change in RH. The integral of the area under the middle peak yields a value of about 42 J/g, which is typical for starch products. The correlation between sorption enthalpies and moisture content allows the temperature dependence of water sorption to be described at any RH. This is often very important for materials characterization.



Storage conditions and shelf life

Many food products are sensitive to moisture. This affects storage conditions and shelf life. Rice presents special problems during transportation because it is not dry when shipped. If the intrinsic moisture content of rice is too high, there is a risk of mold formation, mustiness and fermentation, as well as self-heating and agglomeration. The example summarizes measurements performed on basmati rice, which has a water content of more than 15% when freshly harvested. The sorption isotherm of the pre-dried rice displays the relative gain in mass as a function of RH. Clearly, the RH should not exceed about 85% during transport and storage. This analysis has proven useful for studying rice grown in regions where dry and rainy seasons occur and where shipment deadlines are critical for the quality of the product.





Moisture-induced crystallization of lactose in milk powder

Low-fat milk powder consists of about 50% lactose. This is present in an amorphous state due to the production process. In the amorphous form, the powder is very hygroscopic and binds water very efficiently. The diagram shows the effects of increasing relative humidity on the lactose in milk powder at 27 °C. Uptake of water causes the glass transition temperature to decrease to below room temperature. The amorphous lactose starts to melt. In general, a liquid takes up more water than the solid amorphous form. This excess water increases the mobility of the lactose molecules and allows them to rearrange to form crystals. Crystalline lactose is however less hygroscopic than amorphous lactose. The crystallization process is therefore accompanied by the release of water. This excess water evaporates and is observed as a weight loss in the TGA curve. The peak maximum corresponds to the water content in the lactose at which crystallization occurs.

References

- L. Greenspan, "Humidity Fixed Points of Binary Saturated Aqueous Solutions", Journal of Research of the National Bureau of Standards, A. Physics and Chemistry, 81A (1), p. 89–96.
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