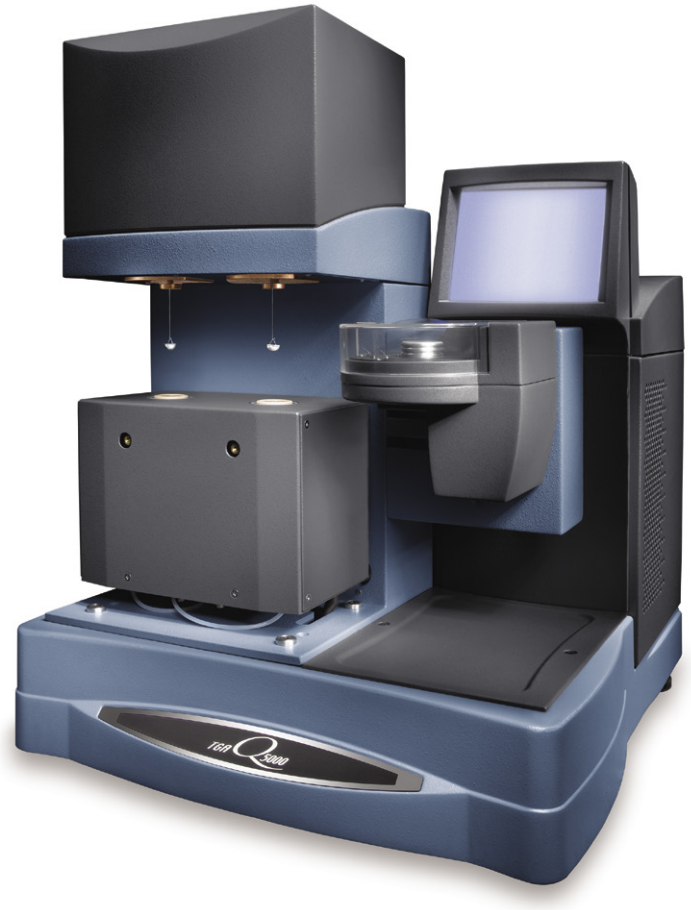
TA Instruments

VAPOR SORPTION ANALYSIS

Q5000 SA

Sensitive Measurements, Precise RH Control

The patented Q5000 SA delivers the performance and reliability required in a leading sorption analyzer in a compact, user-friendly design. The Q5000 SA is designed for manual or automated sorption analysis of materials under controlled conditions of temperature and relative humidity (RH). Its design integrates our high-sensitivity, temperature-controlled thermobalance with an innovative humidity generation system, multi-position autosampler, and powerful Advantage™ software with technique-specific programs and Platinum™ features.

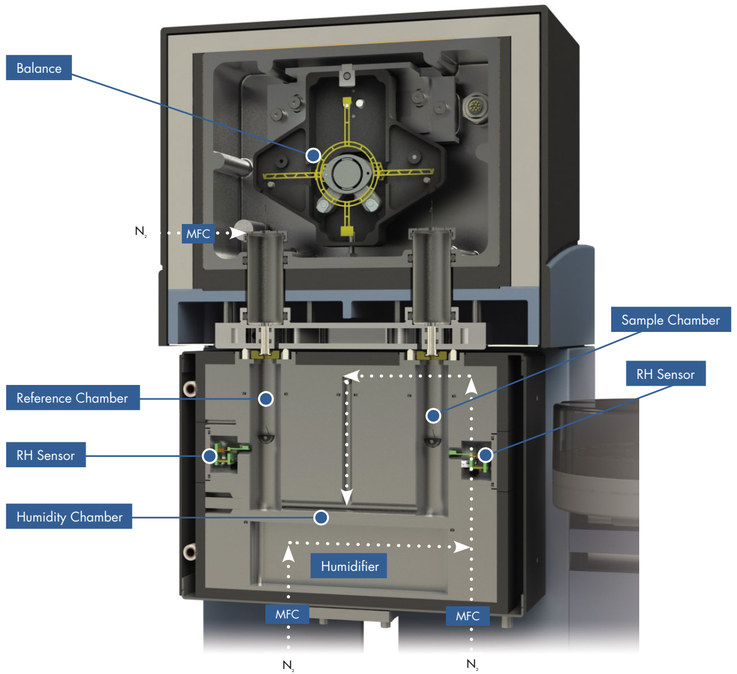


Q5000 SA | TECHNOLOGY

The Q5000 SA is a compact, benchtop instrument that delivers the performance and reliability required in a leading sorption analyzer designed for the study of materials under controlled conditions of temperature and relative humidity. Its modern, user-friendly design features a high sensitivity, temperature-controlled thermobalance, an innovative humidity generation system, a 10-position autosampler, and our latest Advantage™ software with Platinum™ features.

Humidity Control Chamber

The patented design features a pair of mass flow controllers that accurately meter and proportion gas to a symmetrical, well-insulated, aluminum block. The block contains a humidifier, gas transmission and mixing lines, plus easily accessible, identically arranged, sample and reference measurement chambers. Temperature regulation of the block interior from 5°C to 85°C is performed by four thermoelectric (Peltier) devices in conjunction with a thermistor in a closed-loop system. The mass flow controllers adjust the amounts of wet (saturated) and dry gas to obtain humidities from 0 to 98 %RH. Identical sensors are located adjacent to the sample and reference crucibles, and provide a continuous indication of humidity. Benefits of the design include precise temperature control and highly consistent atmosphere within the sample and reference chambers.

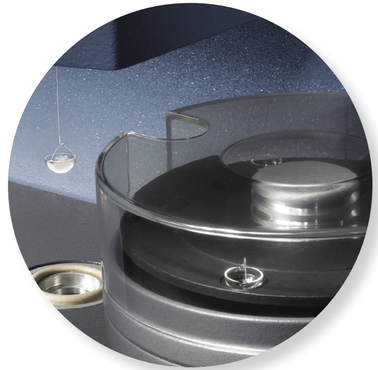


Sample Crucibles

Semispherical quartz, metal-coated quartz (180 µL) and optional platinum (100 µL) TGA pans are available for use with the Q5000 SA. The former are commonly used in sorption analysis because of their anti-static capabilities, chemical inertness and ease of cleaning, while Platinum pans are generic for TGA analysis of most materials. Sealed aluminum pans are also an option for ensuring the integrity of materials which readily adsorb moisture or lose volatiles.

Thermobalance

The heart of the Q5000 SA is our high performance thermobalance maintained at a constant temperature (±0.01 °C) by three symmetrically arranged heaters in a well-insulated, gas-purged chamber. Isolated from the furnace by a water-cooled plate, the sensitive null-balance design features the latest in precision weighing technology. A key feature of the design for sorption analysis operation is the perfect symmetry of the balance assembly. Customer benefits of the patented design include sensitive, reliable operation with superior baseline flatness and exceptional accuracy and precision in weight-change detection. These factors are critical for proper gravimetric sorption-analysis performance and results that are totally free from error caused by vapor condensation or electrostatic forces.



Autosampler

The integral Q5000 SA Autosampler features a programmable multi-position sample carousel that permits automated analysis of up to 10 samples using semispherical quartz (or metal-coated quartz) crucibles, and 25 samples using the optional Discovery TGA tray and platinum or sealed aluminum pans. The design provides smooth and efficient loading and unloading of the sample pan without disturbing the balance. All aspects of sample testing are automated and software controlled including pan taring and loading, sample weighing, autosampler movement, furnace movement, pan unloading, and furnace cooling. Autosampler productivity is maximized by our Advantage™ software which provides pre-programmed analysis, comparison, and presentation of results.

GRAVIMETRIC VAPOR SORPTION ANALYSIS | GENERAL PRACTICE

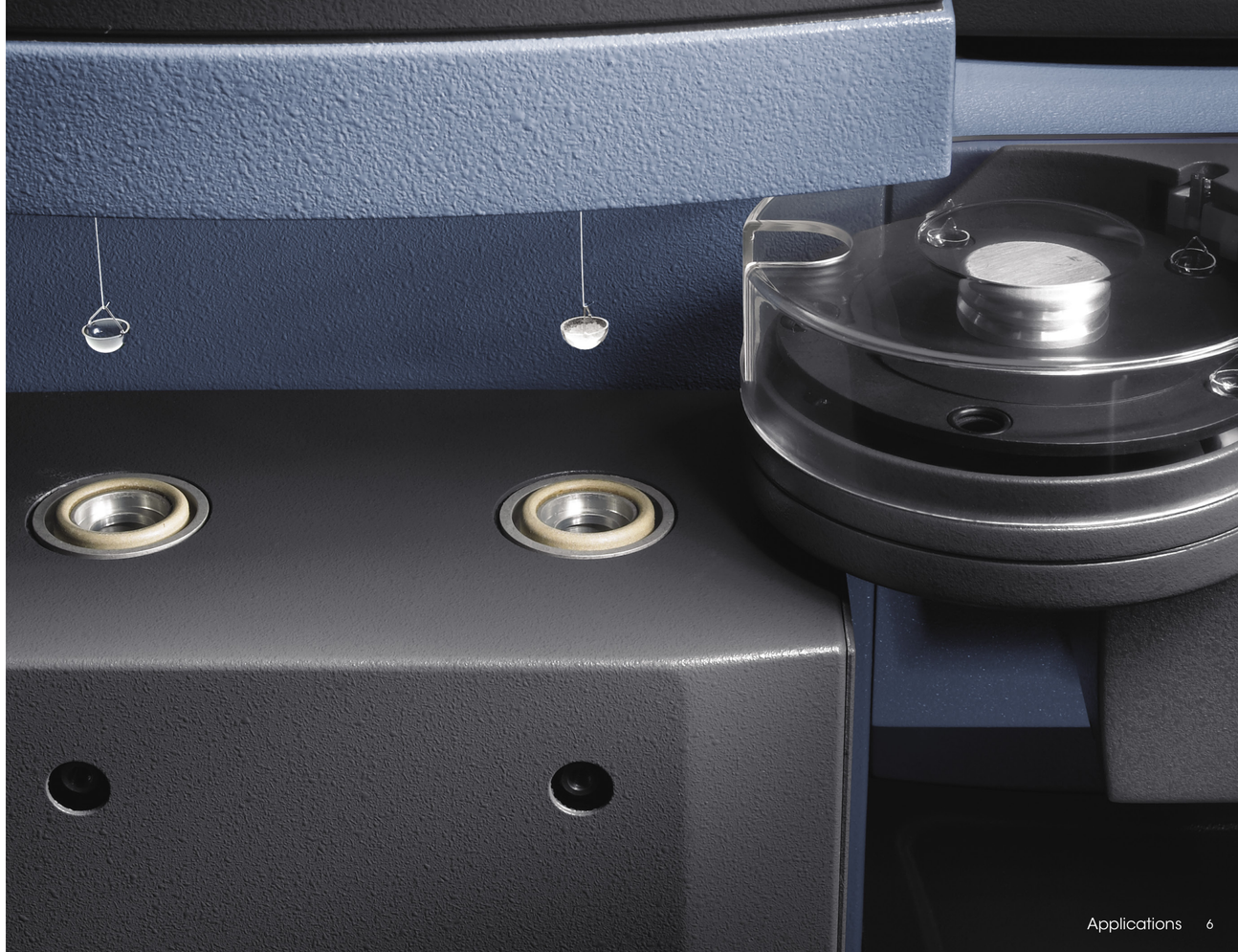
Vapor Sorption analysis is an established technique for determining the effect on materials of exposure to controlled conditions of temperature and humidity. Isotherm and Isohume™ experiments are the most commonly performed analyses.

All TA Instruments sorption analyzers perform a range of essential sorption experiments such as time-courses, isotherms (constant temperature, variable RH), and isohumidity (Isohume™) experiments (constant RH, variable temperature). Complex protocols with step changes in temperature and RH can be defined and saved for later use. Also, multiple experiments can be run sequentially without further operator assistance.

In isothermal experiments, a weighed sample is "dried" externally, or preferably in the instrument, and exposed to a series of humidity step changes at constant temperature. The sample is staged at each humidity level until no further weight change is detected or a set time has elapsed. A data point is recorded, the humidity is changed in 5 or 10% controlled RH steps, and the process repeated in an increasing or decreasing procedure. Isohume experiments involve a series of temperature step changes at constant humidity and result in similar plots. They are used to determine how sample exposure to a given humidity results in a physiochemical change, such as a change in the sample's hydration state. The curve shape provides useful information to this end.

TA Instruments analysis software offers Sorption Analysis, BET Analysis, and GAB programs. In addition, the full power and flexibility of our renowned Universal Analysis software provides for easy data manipulation, advanced reporting, plotting, and file exporting capabilities. Analysis options include:

- Kinetic analysis for the determination of rate constant of adsorption
- Isosteric heat of adsorption using the Clausius-Clapeyron equation
- Surface area calculation using the BET equation for water vapors



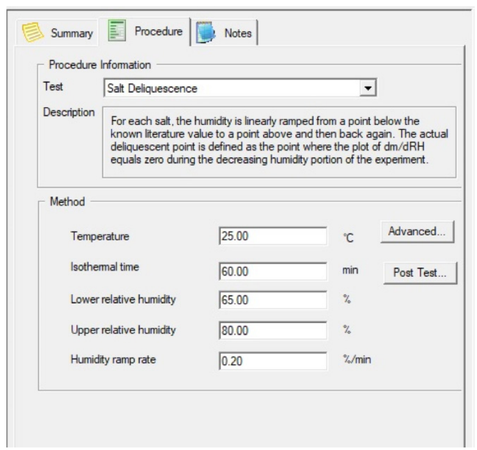
Q5000 SA | APPLICATIONS HUMIDITY VERIFICATION

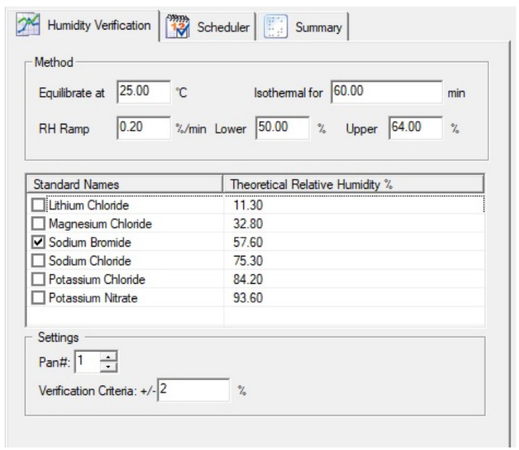
Applications Humidity Verification

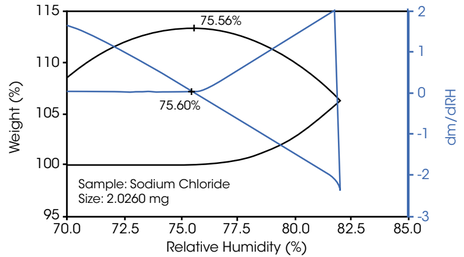
The Q5000 SA Thermal Advantage Platinum software has a built-in humidity verification feature and deliquescence method that allows the user to determine the humidity level in the purge gas at a fixed temperature. The method conforms to ASTM E2551.

Deliquescent salts are used to verify the humidity of the environment. Deliquescent materials absorb very little water as the humidity is raised until the percent relative humidity (%RH) reaches a critical level or deliquescence point. At a given temperature and that specific %RH, the hygroscopic material suddenly starts absorbing any available moisture resulting in rapid increase of weight until the salt dissolves.

- The Q5000 SA has a built-in humidity verification routine for selected salts
- User specified humidity verification routines can be scheduled to run daily, weekly, or as needed
- Prior to testing a sample or running an autosampler queue, the Q5000 SA humidity control can be verified using deliquescent salts such as Bromide, Soldium Chloride and Potassium Nitrate.

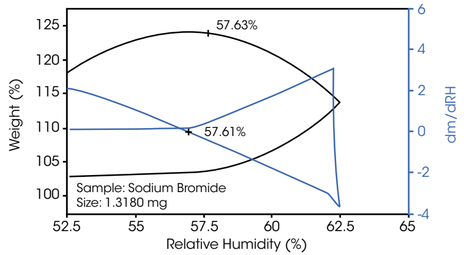






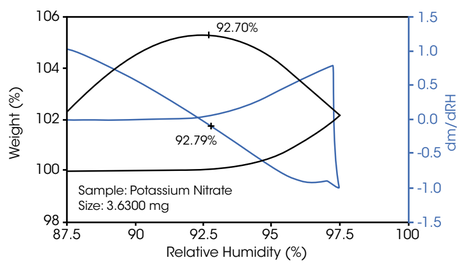
Deliquescence of Sodium Chloride

Humidity Verification: 75.60%. Expected Sodium Chloride Deliquescence Point 75.30% ± 1.00% the Deliquescence point is within the specified tolerance. Verification successful.



Deliquescence of Sodium Bromide

Humidity Verification: 57.61%. Expected Sodium Bromide Deliquescence Point 57.60% ± 1.00% the Deliquescence point is within the specified tolerance. Verification successful.

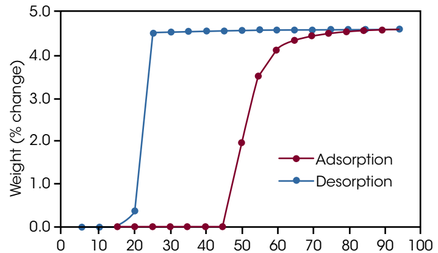


Deliquescence of Potassium Nitrate

Humidity Verification: 92.79%. Expected Sodium Bromide Deliquescence Point 93.60% ± 1.00% the Deliquescence point is within the specified tolerance. Verification successful.

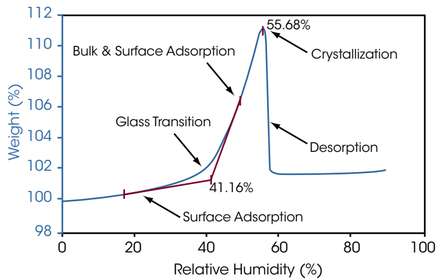
Hydrate Formation

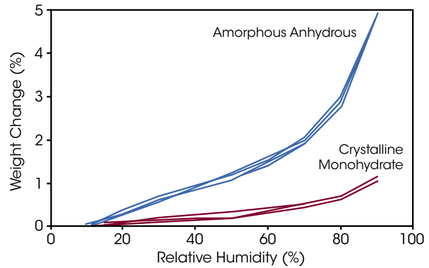
The figure to the right contains the experimental results demonstrating the formation of a hydrate. The hydrate formation is characterized by a plateau in the desorption branch of the isotherm. In this example the hydrate is formed at around 45% RH. The sample adsorbs about 4.5% by weight water and does not lose the water of hydration until the RH is lowered below 25%. This hydrate would be considered as a labile or unstable hydrate.



Characterization of Morphological Stability

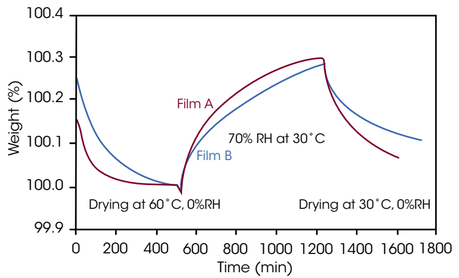
Exposure to elevated humidity can initiate morphological changes in some pharmaceutical materials, particularly in amorphous sugars. As the humidity is increased, the adsorbed water plasticizes the material and lowers the glass transition. When the glass transition temperature decreases to the experimental temperature, crystallization will typically occur. The data in the figure shows the behavior of amorphous lactose at 25°C under a constant increase in humidity. Note how the character in the measured weight signal is indicative of a variety of morphological changes including the glass transition and subsequent crystallization of the amorphous phase.





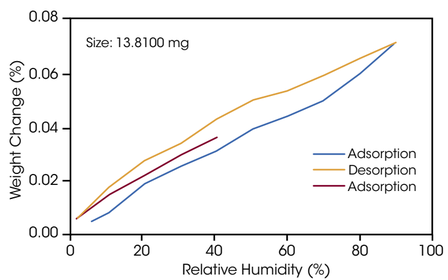
Evaluation of Amorphous Structure

Pharmaceutical scientists are often interested in determining the amount of amorphous material in a drug formulation. As the amorphous and crystalline forms are chemically identical, classical analysis techniques are often insensitive to amorphous content. The figure shows the moisture sorption analysis of a generic drug in its amorphous and crystalline forms. As the amorphous form absorbs significantly more water, the Q5000 SA can be used to quantify relative amorphous content in drug mixtures.



Packaging Film Analysis

In addition to evaluation of the actual pharmaceutical formulations, sorption analysis can also be valuable in comparing the polymeric films which are being considered for packaging the drugs and other materials. The figure to the right shows comparative profiles for two different packaging materials undergoing temperature and relative humidity cycling. Film A adsorbs and desorbs moisture at a more rapid rate than the other film evaluated which suggests it may not be suitable for packaging moisture sensitive compounds.



Analyzing Small Amounts of Pharmaceuticals

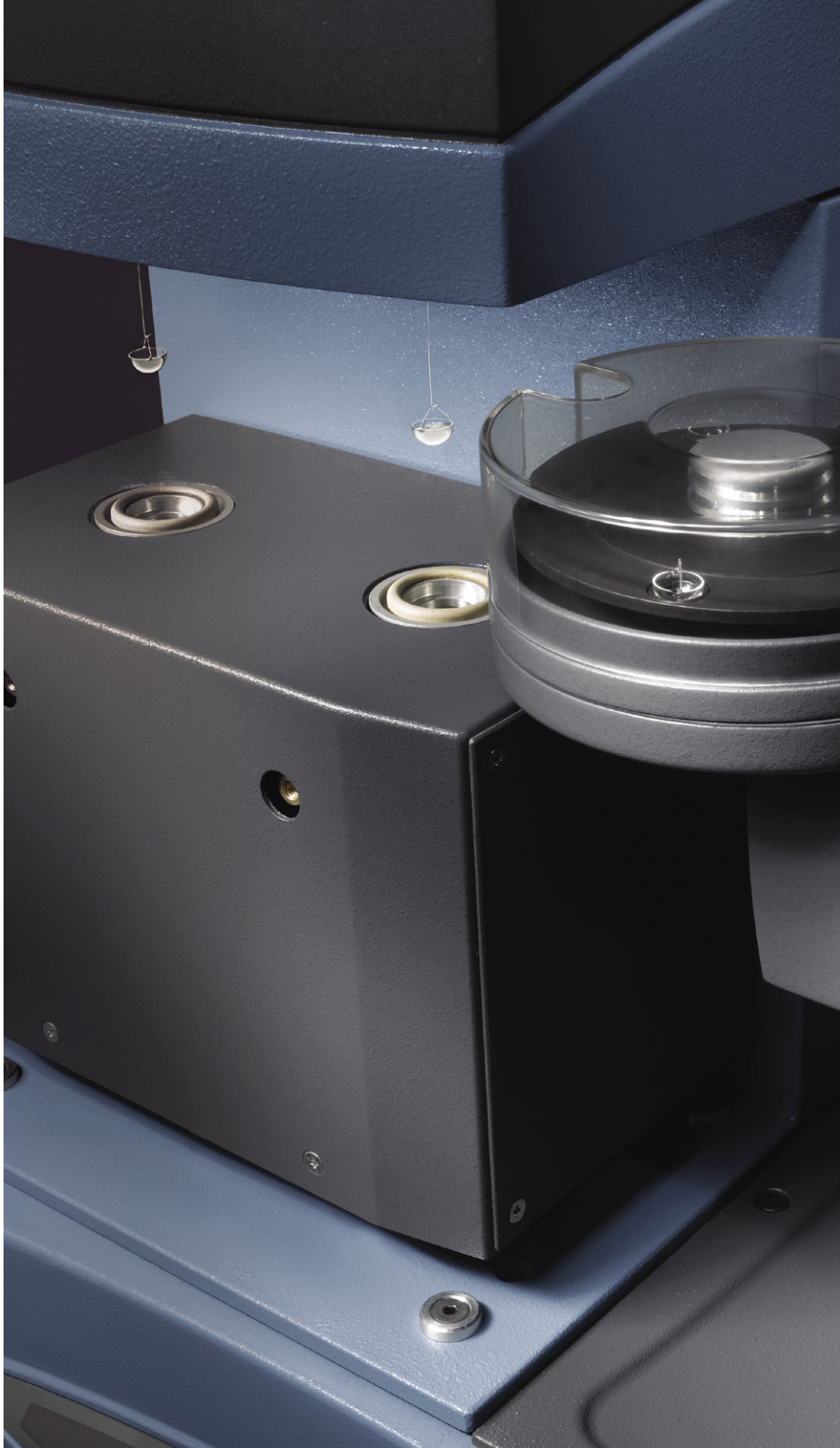
When evaluating pharmaceuticals it is common for only small amounts of material to be available for conducting multiple analytical tests. Hence, the ability to work with small samples is critical. The low baseline drift of the Q5000 SA means that good results can be obtained on even 10-20 milligrams of a crystalline drug, such as prednisone, which adsorbs <0.1% moisture over a broad humidity range. The sorption results shown below represent about 15 micrograms of weight change full-scale. The reversibility (lack of hysteresis) in the sorption/desorption profile for prednisone (as well as the low level of moisture adsorbed) indicates that the moisture picked up by the material is adsorbed on the surface of the material rather than being absorbed into its structure.

PERFORMANCE SPECIFICATIONS

Temperature Controlled Thermobalance	●
Dynamic Range	100 mg
Weighing Accuracy	± 0.1%
Weighing Precision	± 0.01%
Sensitivity	< 0.1 µg
Baseline Drift*	< 5 µg
Signal Resolution	0.01 µg
Temperature Control	Peltier Elements
Temperature Range	5 to 85°C
Isothermal Stability	± 0.1°C
Relative Humidity Control Range	0 to 98% RH
Accuracy	± 1% RH
Autosampler – 10 samples**	●
Platinum™ Software	●
Sample Pans	Quartz or Metal-Coated Quartz 180 µL
	Platinum 100 µL
	Aluminum Sealed Pan 20 µL

● Included

* Over 24 hours at 25°C and 20 % RH with empty metal coated quartz pans
** Optional tray accommodates 25 samples for use with platinum and sealed aluminum pans



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