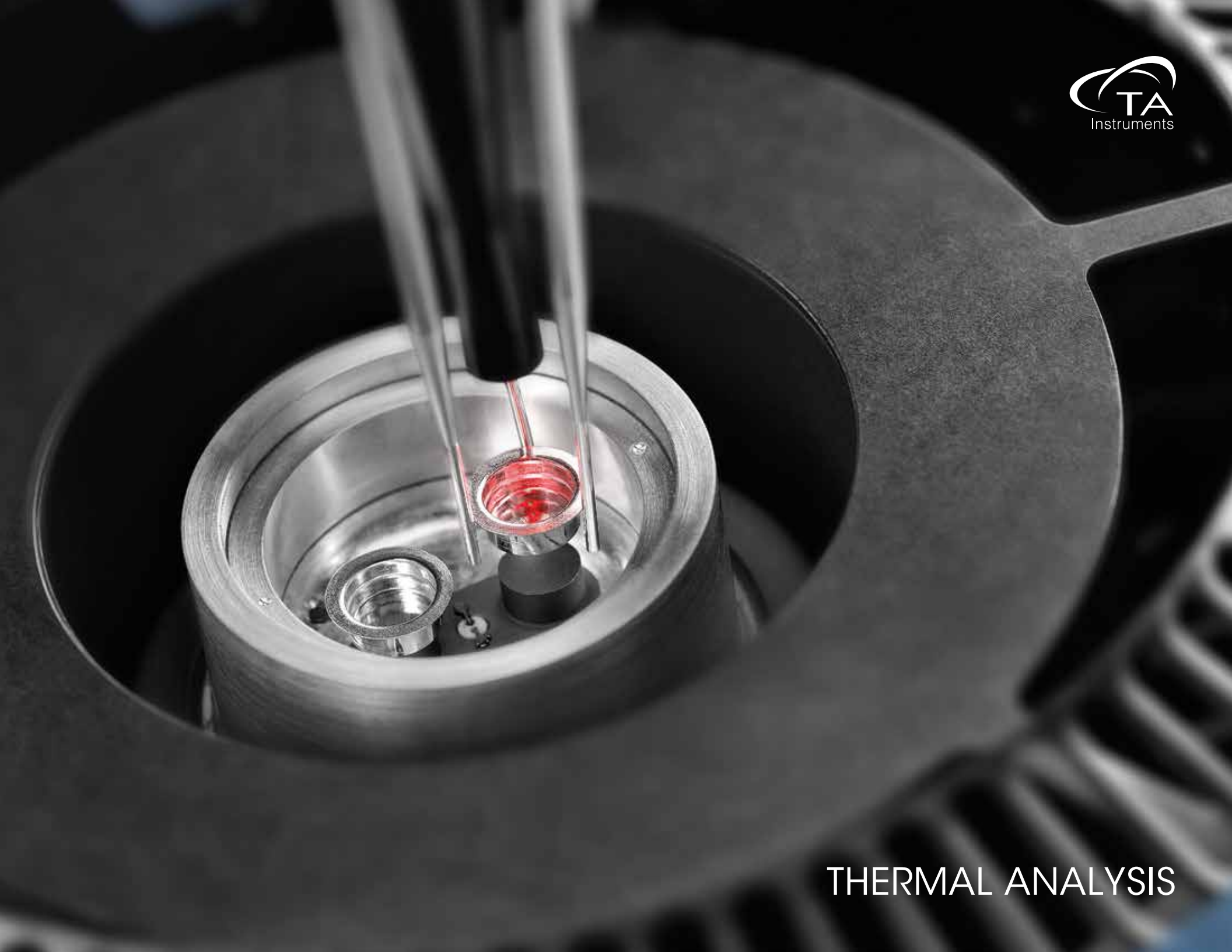

TA
Instruments
THERMAL ANALYSIS

New Castle, DE USA

Lindon, UT USA

Hüllhorst, Germany

Shanghai, China

Beijing, China

Tokyo, Japan

Seoul, South Korea

Taipei, Taiwan

Bangalore, India

Sydney, Australia

Guangzhou, China

Eschborn, Germany

Wetzlar, Germany

Brussels, Belgium

Etten-Leur, Netherlands

Paris, France

Elstree, United Kingdom

Barcelona, Spain

Milano, Italy

Warsaw, Poland

Prague, Czech Republic

Sollentuna, Sweden

Copenhagen, Denmark

Chicago, IL USA

São Paulo, Brazil

Mexico City, Mexico

Montreal, Canada



Thermal Analysis

Thermal Analysis is important to a wide variety of industries, including polymers, composites, pharmaceuticals, foods, petroleum, inorganic and organic chemicals, and many others. These instruments typically measure heat flow, weight loss, dimension change, or mechanical properties as a function of temperature. Properties characterized include melting, crystallization, glass transitions, cross-linking, oxidation, decomposition, volatilization, coefficient of thermal expansion, and modulus. These experiments allow the user to examine end-use performance, composition, processing, stability, and molecular structure and mobility.

All TA Instruments thermal analysis instruments are manufactured to exacting standards and with the latest technology and processes for the most accurate, reliable, and reproducible data available. Multiple models are available based on needs; suitable for high sensitivity R&D as well as high throughput quality assurance. Available automation allows for maximum unattended laboratory productivity in all test environments.

As the world leader in Thermal Analysis for over 50 years, TA Instruments brings technical expertise in thermal analysis measurements and provides a world-renowned global support network that is specialized in thermal analysis.

Vapor Sorption Analysis

Sensitive Measurements, Precise RH Control

Q5000 SA

SORPTION ANALYSIS

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 latest 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.

specifications

Q5000 SA

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 50, 100 µL
	Aluminum Sealed Pan 20 µL

* 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

● Included

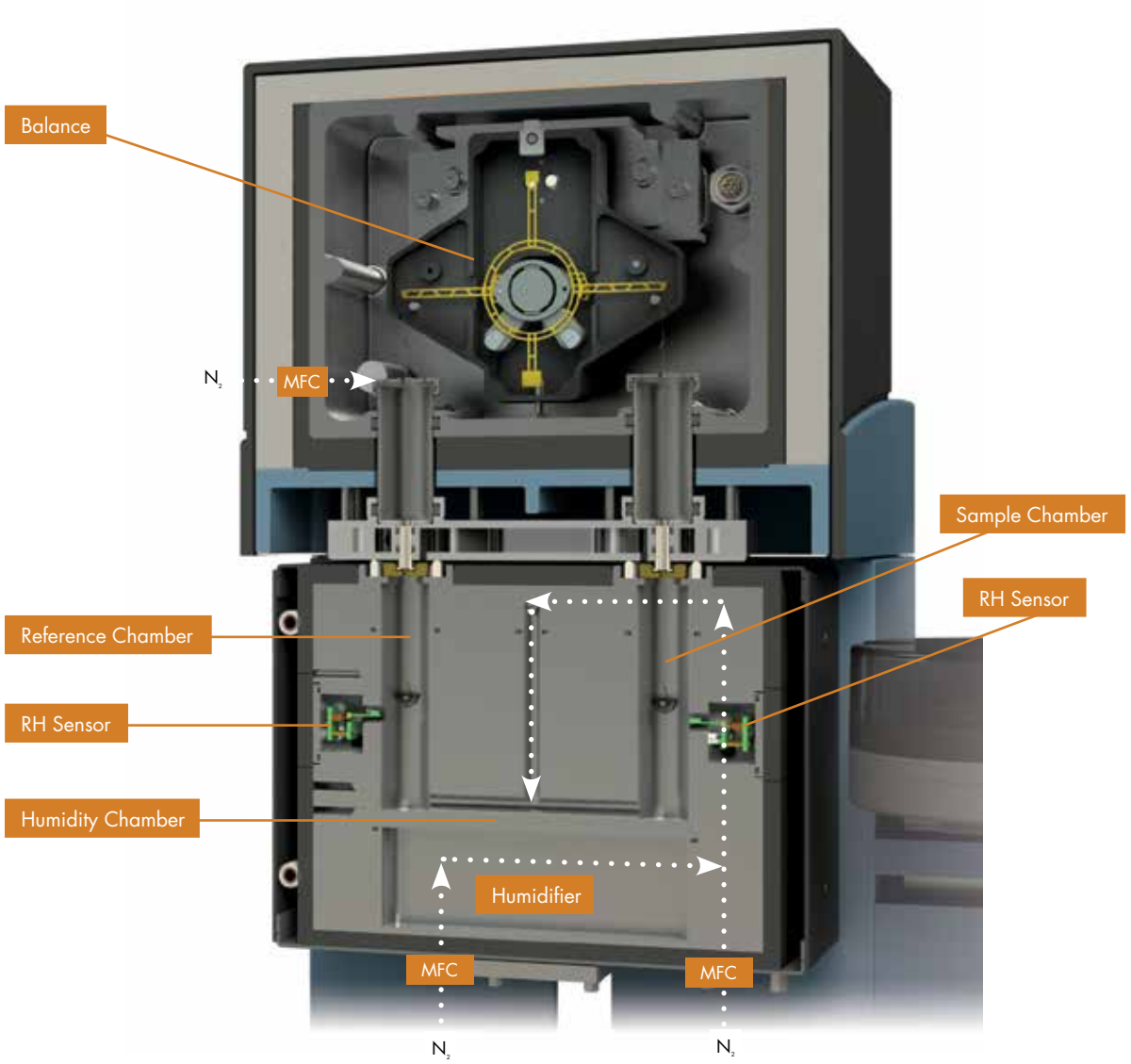
technology

Q5000 SA

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 (50 and 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 latest 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.

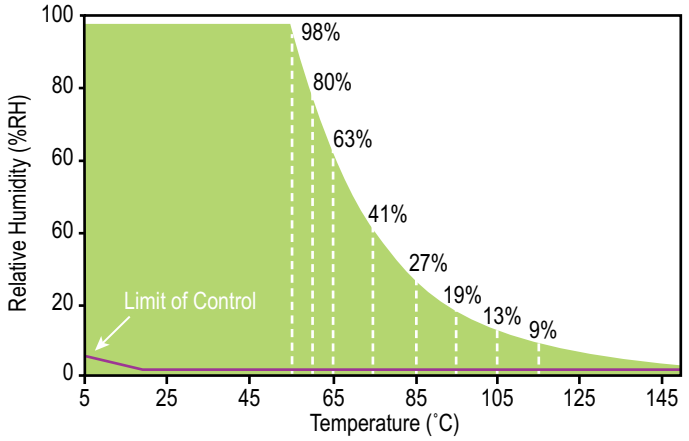
VTI-SA+

VAPOR SORPTION ANALYSIS

The VTI-SA+ Vapor Sorption Analyzer is a continuous vapor flow sorption instrument for obtaining precision water and organic vapor isotherms at temperatures ranging from 5°C to 150°C at ambient pressure. The VTI-SA+ combines the features of VTI's original SGA design with almost two decades of field-proven performance: the isothermal aluminum block construction, the three isolated thermal zones and chilled-mirror dew point analyzer for primary humidity measurements with the field-proven TA Instruments thermobalance technology… all to provide precise and accurate gravimetric measurements with excellent temperature and RH stability.

Maximum Sample Weight	750 mg/5 g
Dynamic Range	100 mg/500 mg
Weighing Accuracy	± 0.1%
Weighing Precision	± 0.01%
Sensitivity	0.1 µg/0.5 µg
Signal Resolution	0.01 µg/0.05 µg
Temperature Control	Peltier Elements, Resistance Heaters
Experimental Temperature Range	5 to 150°C
Isothermal Stability	± 0.1°C
Relative Humidity Control Range	See Figure Below
Accuracy	± 1% RH
Humidity Control	Closed Loop, Dew Point Analyzer
Organic Solvent Capability	O
Camera/2.5x Microscope Accessory	O
Raman Probe Accessory	O

O Optional



*Performance may vary slightly, depending on laboratory conditions

Symmetrical Microbalance Design

The VTI-SA+ Analyzer is a symmetrical vapor sorption instrument where both the sample and reference chambers are exposed to the same conditions of temperature and humidity. In this symmetrical design, any water or organic vapor sorption onto the hangdown wires and sample holder is differentially eliminated and the resultant data represents the uptake by the sample alone. This eliminates the need for background subtraction experiments and associated uncertainty typical in competitive, asymmetrical systems.

Resolution and Stability of the Microbalance

The standard VTI-SA+ boasts a microbalance designed and manufactured by TA Instruments with 0.1 microgram sensitivity optimized for pharmaceutical applications. A higher mass capacity version (5 g, 0.5 microgram sensitivity) is also available. To ensure effective work in pharmaceutical studies, the design provides an enhanced stability by maintaining the balance compartment at a constant temperature, independent of the sample temperature. Because the balance is maintained at constant temperature, the user has the option of drying the sample at temperatures other than the experimental temperature or to run different temperature and RH profiles without removing the sample.

technology

VTI-SA+

Precision Humidity Measurements

As part of our standard design, the VTI-SA+ employs a chilled mirror dew point analyzer (a NIST-traceable standard for humidity) to determine the absolute relative humidity at the sample. In applications where RH control is critical (as in most pharmaceutical studies), chilled-mirror dew point analyzers are the preferred method, because of the absence of drift and long term stability.

Sorption Testing Using an Organic Vapor

The VTI-SA+ can also be configured for organic vapor sorption. In the VTI-SA+, the concentration of the organic vapor in the gas stream reaching the sample is determined by the fraction of gas going through the organic solvent evaporator and the fraction of dry gas.

In competitive systems, assumptions are made that the evaporator is 100% efficient and that the temperature of the evaporator is constant from low to high concentrations. The VTI-SA+ system measures the temperature of the organic solvent in the evaporator and uses this information together with the Wagner equation to control the organic vapor concentration in the gas phase. This method solves the issue of adiabatic cooling of the solvent, a major source of error in competitive systems.

The solvent containers/evaporators are easily removed and exchanged so there is no need for decontamination or cleaning of the system when changing organic solvents or reverting to water sorption experiments. For safety, the evaporator compartment is purged with dry nitrogen and fitted with a combustible gas sensor with an audible alarm that, when triggered, shuts down the power to the analyzer.

Simultaneous Microscope Camera or Raman Measurement

The VTI-SA+ is fully compatible with simultaneous optical measurements, including a high-resolution CCD camera / 2.5x microscope or a dedicated Raman Probe.* These optional accessories are field installable, providing the highest level of flexibility for your measurements.

Sample Chamber Design

In the VTI-SA+ Analyzer, the sample and reference chambers are located within an aluminum block maintained at constant temperature (within ±0.1°C) by precise Peltier control elements. Our unique aluminum block design has two distinct advantages. First, due to the high thermal conductivity of the aluminum, thermal gradients within the chambers are minimal. Second, because the chamber is a metal block, errors associated with static electricity are eliminated. This feature is especially useful when analyzing finely divided powders, as is often the case with pharmaceuticals. The sample temperature is measured with a highly accurate, calibrated platinum resistance thermometer. When higher temperatures are required, the block can be heated to 150 °C using installed resistance cartridge heaters.

*Raman Spectrometer Required

gravimetric vapor sorption analysis

GENERAL PRACTICE

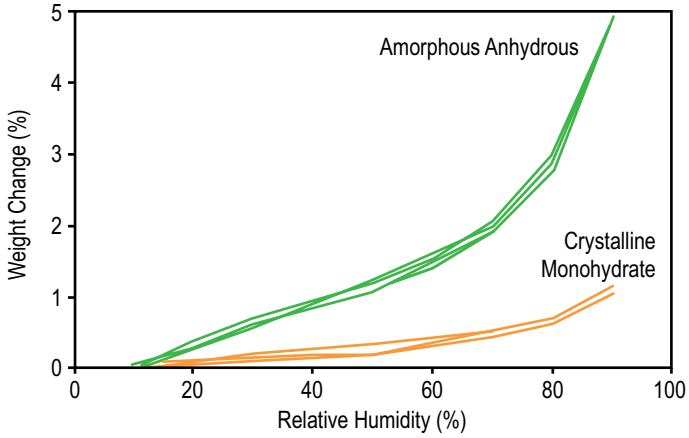
Vapor Sorption analysis is an established technique for determining the effect on materials of exposure to controlled conditions of temperature and humidity. Isotherm and Isohume™ experiments are the most commonly performed analyses.

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.

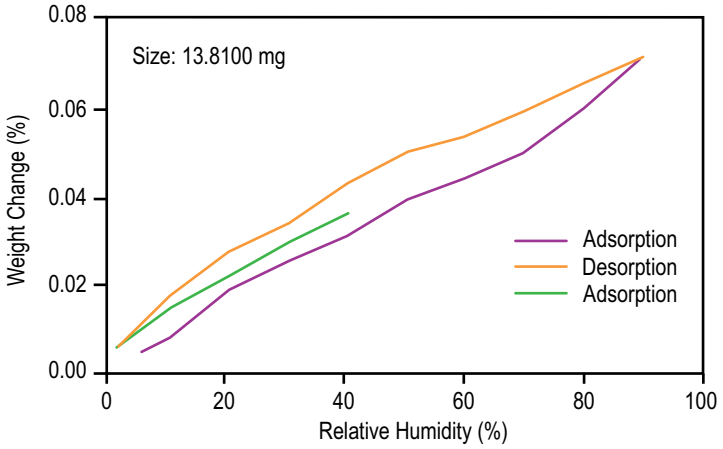
TA Instruments analysis software offers Sorption Analysis, BET Analysis, and GAB programs. In addition, the full power and flexibility of our renowned Universal Analysis software provides for easy data manipulation, advanced reporting, plotting, and file exporting capabilities. In addition, advanced data reduction of VTI-SA+ data can be performed using custom-designed data analysis packages. 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 either water or organic vapors



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.

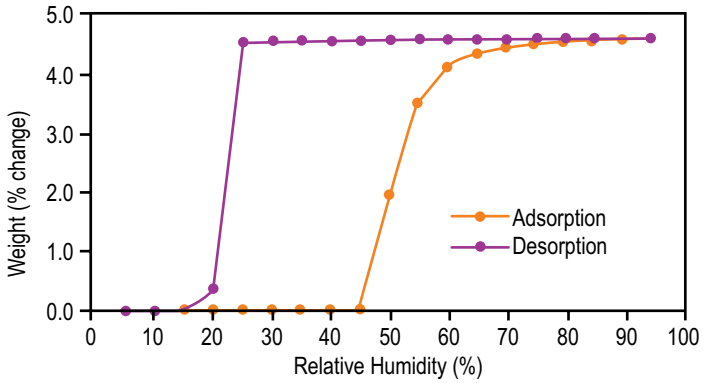


Analyzing Small Amounts of Pharmaceuticals

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.

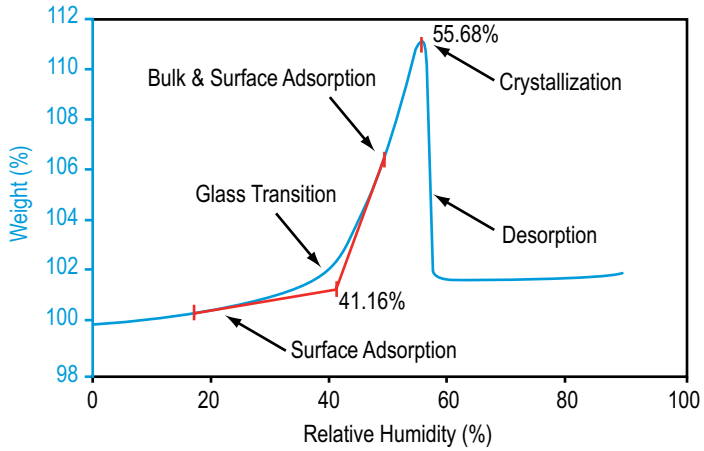
Hydrate Formation

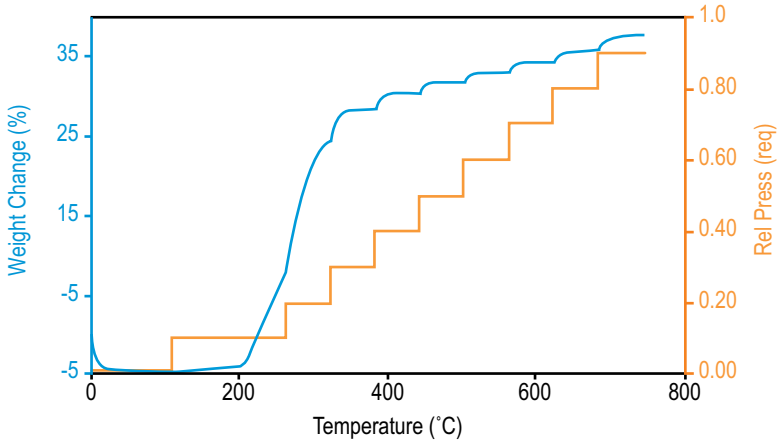
The figure to the right contains the experimental results demonstrating the formation of a hydrate. The hydrate formation is characterized by a plateau in the desorption branch of the isotherm. In this example the hydrate is formed at around 45% RH. The sample adsorbs about 4.5% by weight water and does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

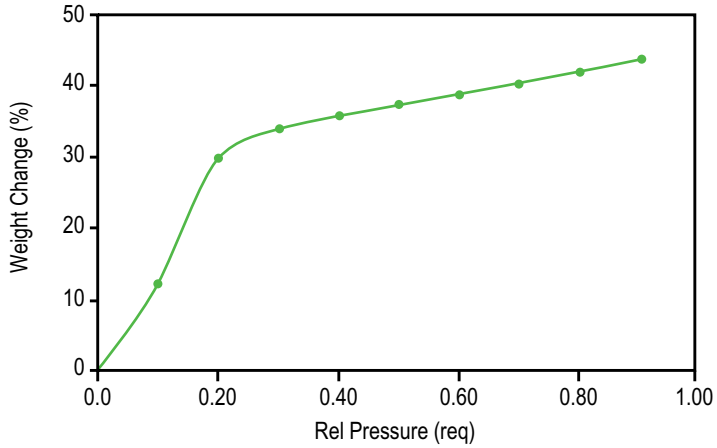


Characterization of Morphological Stability

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.







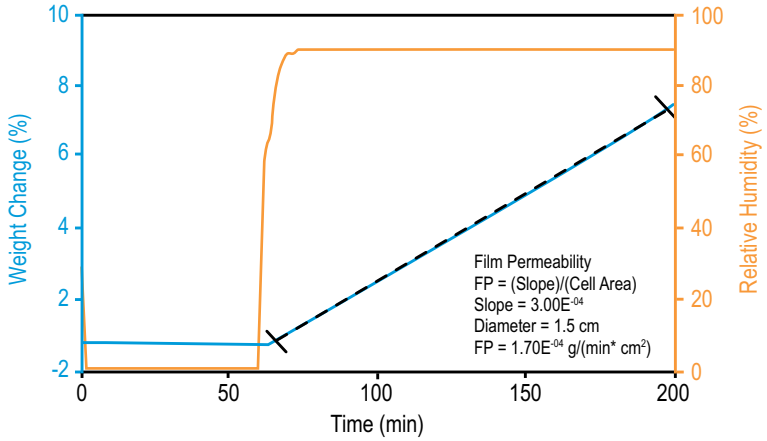
Organic Vapor Sorption (VTI-SA+)

With the organic vapor sorption capability, the VTI-SA+ can obtain not only water sorption isotherms, but can also be used to measure organic vapor isotherms. The use of organic vapor increases the sensitivity of the sorption measurement for many pharmaceutical and polymer materials, and provides information on the specificity of solvent adsorption for many materials. In the first figure, the time course data for the adsorption of ethanol on activated carbon is shown. The sample is initially dried at 0% RH, then the relative pressure of the ethanol is stepped in 0.10 increments.

This second figure shows the sorption isotherm plot for the carbon/ethanol experiment, excluding the initial drying step. The sample exhibits a significant adsorption at low solvent concentrations. This is typical of the particle and internal pore-size distribution of activated carbon which is designed to allow for rapid gas-phase adsorption with low pressure drop.

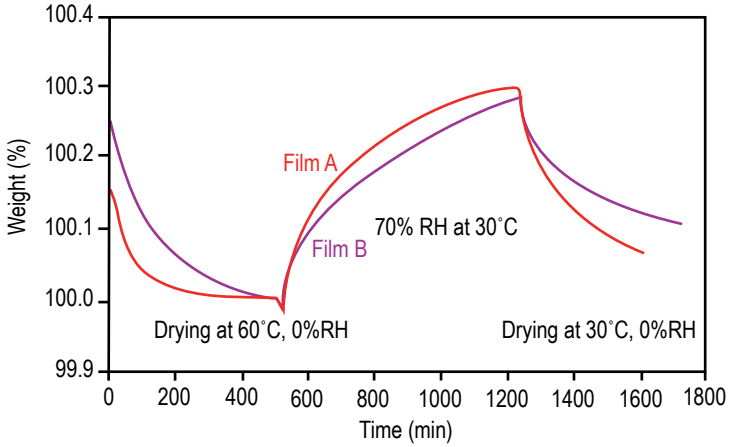
Rate of Diffusion

The VTI-SA+ can be equipped with a diffusion cell which allows for the direct measurement of the permeability of a film or membrane for a particular solvent vapor. The cell consists of a cavity that is filled either with a desiccant or absorber, a gasketed lid for attaching the film to be tested, and a wire stirrup to hang the assembled cell on the hang-down wire of the balance. Any vapor permeating through the film gets absorbed immediately and the weight of the cell will increase until steady-state conditions are reached. The normalized rate of permeation is obtained from the slope of this line (weight per unit time) and the diameter of the permeating film.



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.



NOTES

L90020.001