TA Instruments

Thermal Analysis

TA Instruments

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TA Instruments, Worldwide

More worldwide customers choose TA Instruments as their preferred thermal analysis supplier. We earn this distinction by best meeting customer needs and expectations for high technology products, quality manufacturing, timely deliveries, excellent training, and superior after-sales support.

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TGA

Sensitivity · Precision · Automation

Thermogravimetric Analysis

TA Instruments Q5000, Q500 and Q50 are the instruments best equipped to meet the needs of the thermal analyst for high performance TGA operation in a wide variety of applications. The unique design features of the remarkable Q5000 confirms our commitment to continuously improve the capabilities of traditional products (Q5000 IR) and to provide innovative new ways to characterize materials (Q5000 SA). All Q Series TGA instruments deliver superb value and are backed by the industry's leading support team.

TA INNOVATIONS

Vertical balance & Horizontal gas purge

Hi-Res TGA™

Modulated TGA™

IR Heating

High heating rate TGA

Autosampler with patented pan punching mechanism

Humidity Chamber

Integrated Curie Point temperature calibration

Q5000IR

The highly automated Q5000 IR is the TGA best suited to meet the most demanding research applications. It outperforms all competitors in baseline flatness, sensitivity to low-level weight changes, and flexibility in both standard and high heating rate operation. Other powerful features include a 25-position integrated autosampler with contamination-free, sealed pan-punching capability, an internal electromagnet for easy Curie Point temperature calibration, and Platinum™ software for user convenience in scheduling automatic calibration, verification and diagnostic tests to keep the Q5000 IR constantly in top operating condition.

Temperature Controlled Thermobalance	Included
Weight Range	100 mg
Weighing Accuracy	+/- 0.1%
Weighing Precision	+/- 0.01%
Sensitivity	< 0.1 µg
Baseline Dynamic Drift*	< 10 µg
Signal Resolution	0.01 µg
Furnace Heating	Infrared
Temperature Range	Ambient to 1200 ˚C
Isothermal Temp Accuracy	+/- 1 ˚C
Linear Heating Rate (˚C/min)	0.1 to 500
Furnace Cooling (Forced air / N_2)	1200 to 35 ˚C < 10 min
Vacuum	10^{-2} torr
Temperature Calibration	Electromagnetic Coil/Curie Point Stds
Autosampler – 25 sample	Included
Hi-Res TGA™	Included
Auto Stepwise TGA	Included
Modulated TGA™	Included
TGA/MS Operation	Option
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 µL
	Ceramic 100, 250 µL
	Aluminum 80 µL
	Aluminum Sealed Pan 20 µL

* From 50 to 1000 ˚C at 20 ˚C/min using empty platinum pans

Q5000SA

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. The patented Q5000 SA delivers the performance and reliability required in a leading sorption analyzer and in a compact, user-friendly design.

Temperature Controlled Thermobalance	Included
Weight Range	100 mg
Weighing Accuracy	+/- 0.1%
Weighing precision	+/- 0.01%
Sensitivity	< 0.1 µg
Baseline Dynamic 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**	Included
Platinum™ Software	Included
Sample Pans	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

Q500

The Q500 is the world's #1 selling, research-grade thermogravimetric analyzer. Its field-proven performance arises from a responsive low-mass furnace, ultra-sensitive thermobalance, and efficient horizontal purge gas system (with mass flow control). Its convenience, expandability and powerful, results-oriented software make the Q500 ideal for the multi-user laboratory where a wide variety of TGA applications are conducted, and where future expansion of analytical work is anticipated.

Temperature Compensated Thermobalance	Included
Maximum Sample Weight	1 g
Weighing Precision	+/- 0.01%
Sensitivity	0.1 µg
Baseline Dynamic Drift*	< 50 µg
Furnace Heating	Resistance Wound
Evolved Gas Analysis Furnace (EGA)	Optional
Temperature Range	Ambient to1000 ˚C
Isothermal Temp Accuracy	+/- 1 ˚C
Isothermal Temp Precision	+/- 0.1 ˚C
Controlled Heating Rate (˚C/min)	0.01 to 100
Furnace Cooling (forced air / N_2)	1000 to 50 ˚C < 12 min
Temperature Calibration	Curie Point
Autosampler-16 sample	Optional
Hi-Res TGA™	Optional
Auto Stepwise TGA	Included
Modulated TGA™	Optional
TGA/MS Operation	Optional
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 µL Ceramic 100, 250, 500 µL Aluminum 100 µL

* From 50 to 1,000 ˚C at 20 ˚C/min using empty platinum pans

Q50

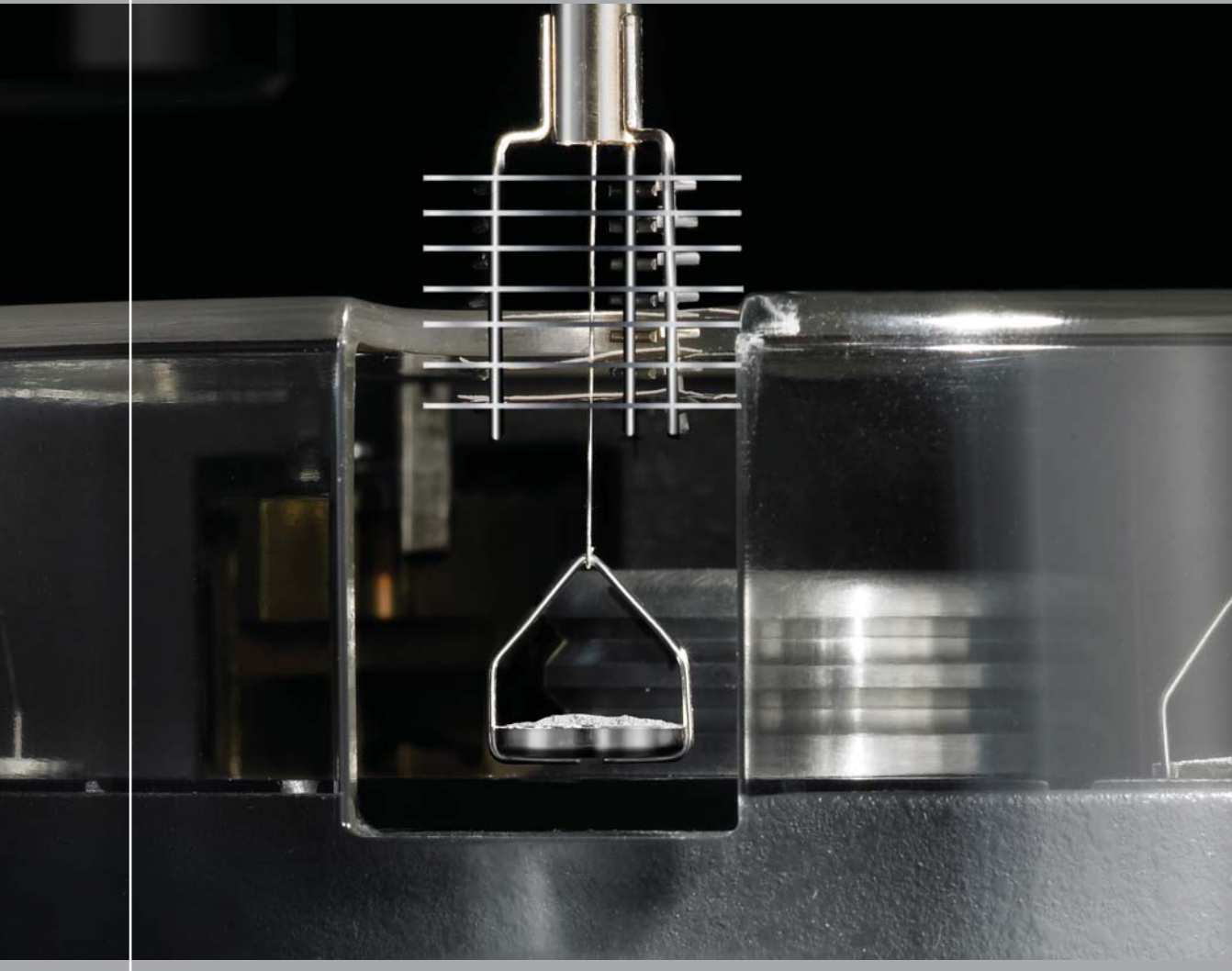
The rugged, reliable, and cost-effective Q50 TGA, with many features of the Q500, offers exceptional value as a compact, general-purpose thermogravimetric analyzer that typically outperforms competitive research-grade models. Its integral mass flow control, gas switching capability, superb software, and ease-of-use make the Q50 ideal in basic research, teaching, or in industrial laboratories that need quality results at a modest cost.

Temperature Compensated Thermobalance	Included
Maximum Sample Weight	1 g
Weighing Precision	+/- 0.01%
Sensitivity	0.1 µg
Baseline Dynamic Drift*	< 50 µg
Furnace Heating	Resistance Wound
EGA Furnace	Optional
Temperature Range	Ambient to1000 ˚C
Isothermal Temp Accuracy	+/- 1 ˚C
Isothermal Temp Precision	+/- 0.1 ˚C
Controlled Heating Rate (˚C/min)	0.1 to 100
Furnace Cooling (forced air / N_2)	1000 to 50 ˚C <12 min
Temperature Calibration	Curie Point
Autosampler-16 sample	Not Available
Hi-Res TGA™	Not Available
Auto Stepwise TGA	Included
Modulated TGA™	Not Available
TGA/MS Operation	Optional
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 µL Ceramic 100, 250, 500 µL Aluminum 100 µL

* From 50 to 1,000 ˚C at 20 ˚C/min using empty platinum pans

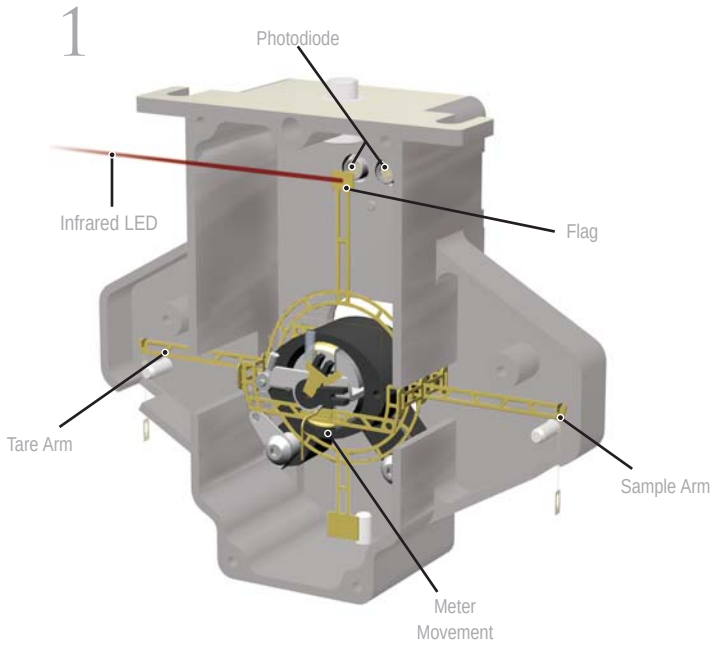
Q5000 IR Technology

The highly automated Q5000 IR is the clear choice for complex TGA applications, for low-level detection of impurities, kinetic studies, off-gas analysis, and for high heating rate operation. Its design integrates a thermobalance engineered for maximum baseline flatness and high sensitivity, with the power and flexibility of an infrared furnace, and a proven horizontal purge gas system. Its many user convenience features include the 25-position autosampler, the integral electromagnet, and Platinum™ software for scheduling automatic calibration, verification and diagnostic tests.



1 Thermobalance

The heart of the Q5000 IR is our latest high performance thermobalance maintained at a constant 40.00 °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. Design benefits include smooth, reliable operation over the entire temperature range (ambient to 1200 °C), unmatched dynamic baseline flatness, and exceptional accuracy and precision in weight change detection, essentially free from any vapor condensation or electrostatic forces.



2 Autosampler

The integrated Q5000 IR Autosampler features a programmable, 25-sample carousel and provides a new level of performance, flexibility and reliability in TGA sample analysis. 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 delivers pre-programmed analysis, comparison, and presentation of results. The autosampler design provides smooth loading and unloading of the sample pan without disturbing the balance. The carousel accommodates platinum, ceramic, and sealed aluminum pans. A special autosampler feature is the patented pan punching mechanism designed to reliably open sealed aluminum pans used to protect atmosphere sensitive samples. The mechanism is force controlled, reliable, and contamination free. A special detection circuit prevents an "unpunched" pan from being placed into the furnace.

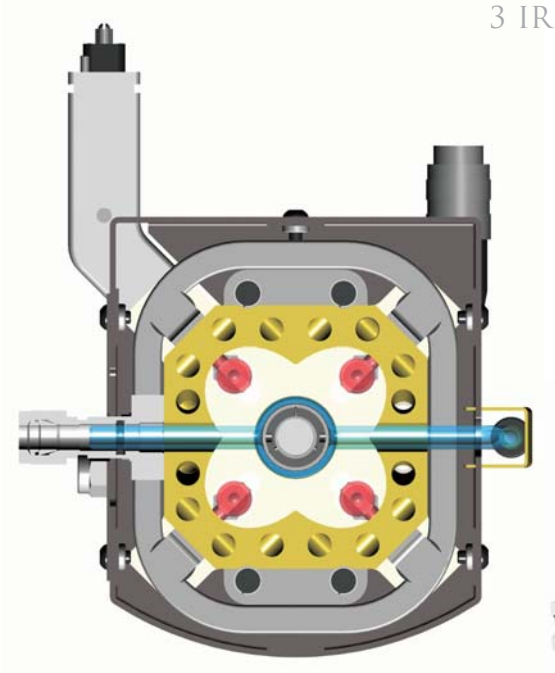
3 Furnace Design and Temperature Measurement

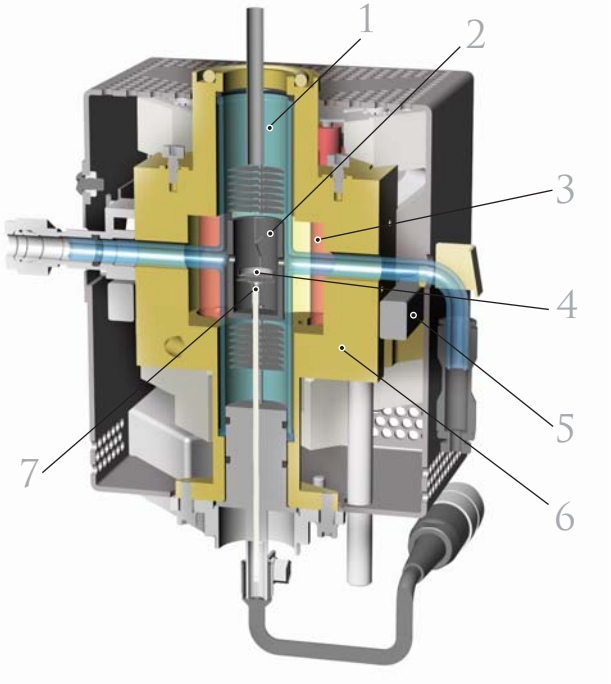
The Q5000 IR surpasses other models with its new infrared furnace that offers the widest range of linear (0.1 to 500 ˚C/min) and ballistic (>2000 ˚C/min) heating rates from ambient to 1200 ˚C. The innovative design employs four symmetrically-placed IR lamps, and a silicon carbide IR-absorbent enclosure. The cylindrical enclosure ensures uniform heating of the sample and pan. The quartz-lined furnace contains upper and lower heat shields and a unique control and measurement area thermocouple assembly. Other features include an integrated electromagnetic coil, forced gas furnace cooling, and vacuum operation. The design benefits are many and include rapid dynamic response and excellent performance and flexibility in standard, advanced (Hi-Res™ TGA; Modulated TGA™), and high heating rate experiments. The integral autosampler, rapid heating and swift cooling significantly enhance productivity. The quartz liner's chemical inertness negates the need for special furnaces and is easily cleaned. Vacuum operation improves resolution of closely related events. The primary thermocouple ensures precise temperature control and provides accurate transition temperature measurement, while a redundant "safety" thermocouple disables the furnace if a temperature differential exceeds a set value. The ease of automated Curie Point temperature calibration is a unique Q5000 benefit.

4 Purge Gas System

An efficient horizontal purge gas system that allows accurately metered gas to flow directly across the sample is integrated into the vertical thermobalance / furnace design. A regulated portion of the gas is also directed through the balance chamber and the combined gases plus any sample effluent readily exit the system. An optional heated side arm is available which provides the highest level of sensitivity for off-gas (MS or FTIR) analysis. Digital mass flow controllers are used to provide accurate and precise metering and proportioning of the purge gases. The automatic low volume, high-speed switching valves deliver instantaneous change of purge gas that is critical when converting between inert and oxidizing atmospheres. Automatic buoyancy corrections provide more accurate weight change data, while the mass flow controllers improve data quality. Gas flow rates are available as stored data file signals.

1 Quartz Liner 2 SiC Sample Enclosure
3 IR Lamps 4 Sample Pan 5 Electromagnet
6 Multi-elliptical Reflector
7 Thermocouple





Q5000 SA Technology

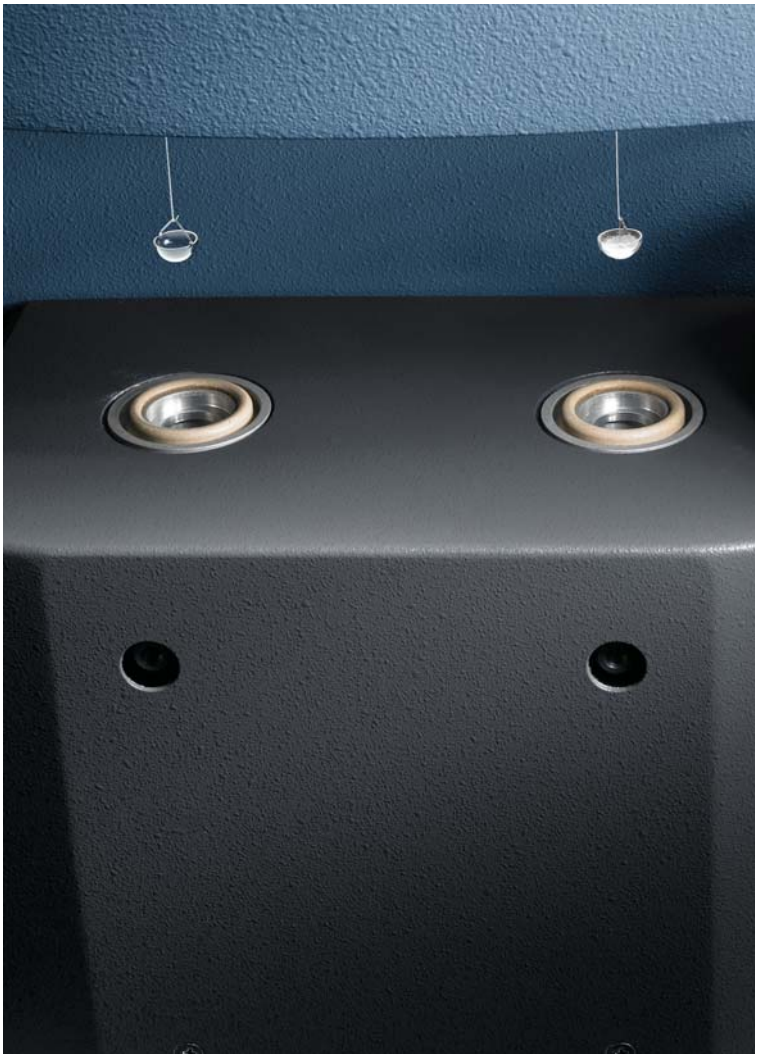
The Q5000 SA is a compact, benchtop instrument that delivers the performance and reliability required in a leading sorption analyzer designed for the study of materials under controlled conditions of temperature and relative humidity. Its modern, user-friendly design features a high sensitivity, temperature-controlled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

1 Thermobalance

The heart of the Q5000 SA is our latest high performance thermobalance maintained at a constant 35.00 °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 smooth, reliable operation with superior dynamic baseline flatness and exceptional accuracy and precision in weight change detection; factors that are critical for proper gravimetric sorption analysis performance and are totally free from any vapor condensation or electrostatic forces.

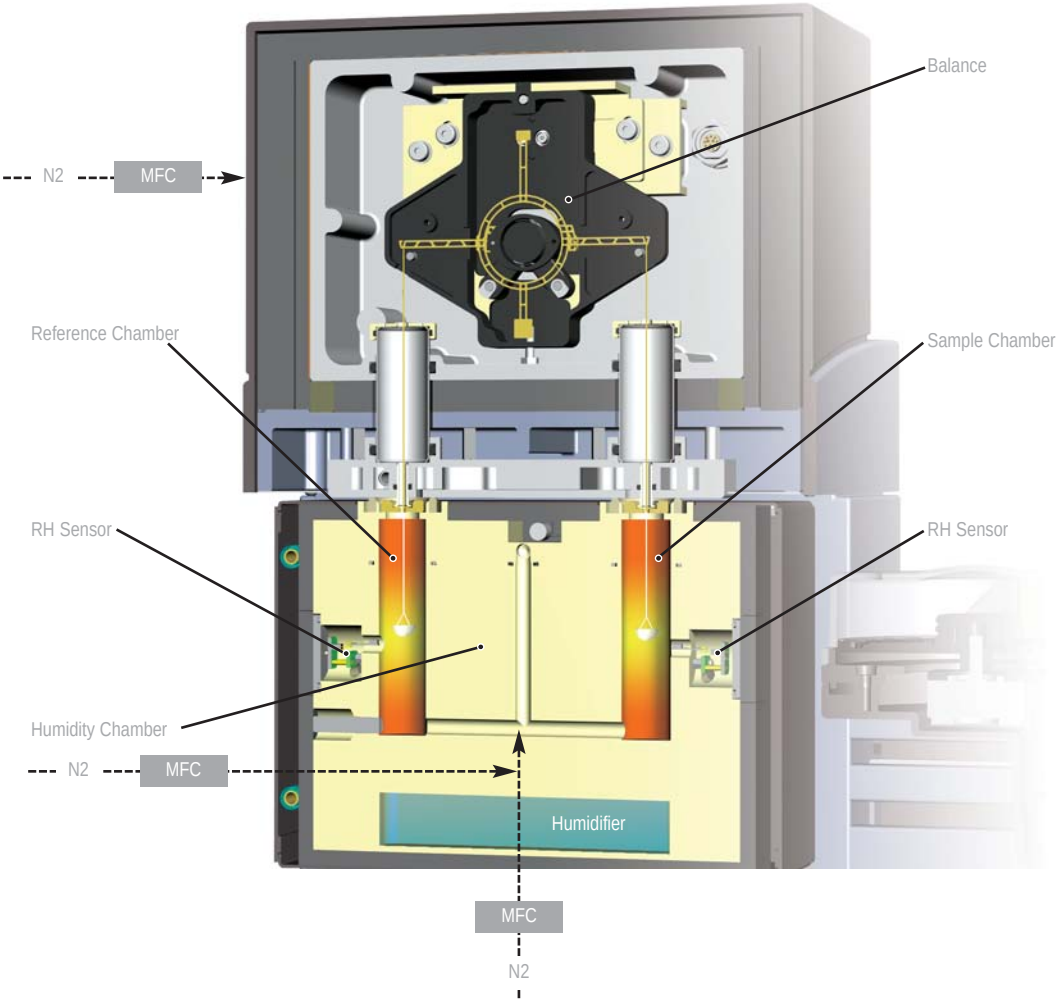
2 Autosampler

The integral Q5000 SA Autosampler features a programmable multi-position sample carousel that permits automated analysis of up to 10 samples using semi-spherical quartz (or metal-coated quartz) crucibles, and 25 samples using the optional Q5000 IR 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 software maximized by our Advantage™ software, which provides pre-programmed analysis, comparison, and presentation of results.



3 Humidity Control Chamber

The patented design features a pair of mass flow controllers (MFCs) 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 10 to 85 °C is performed by four thermoelectric (Peltier) devices in conjunction with a thermistor in a closed-loop system. (Peltier temperature control is more efficient than an external temperature controlled fluid bath.) The mass flow controllers adjust the amounts of wet (saturated) and dry gas to obtain humidities from 0 to 98 %RH. Identical sensors, pre-calibrated against NIST traceable equipment, 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. MFCs deliver continuous, precise gas stream metering and proportioning. The calibrated sensors negate the need for an internal chilled mirror device. The design simplifies pan loading and the autosampler maximizes productivity.


Balance
N2
MFC
Reference Chamber
Sample Chamber
RH Sensor
RH Sensor
Humidity Chamber
N2
MFC
Humidifier
MFC
N2

Q500 / Q50 Technology

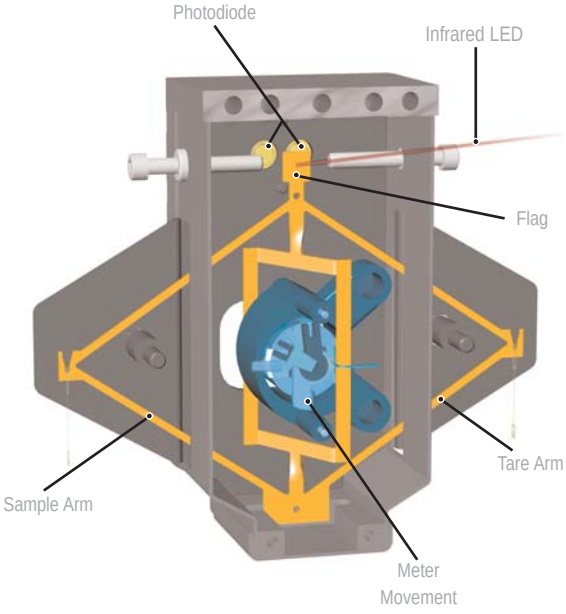
Sensitive, precise, rugged and automated are terms that describe a TA Instruments Q500 and Q50 Thermogravimetric Analyzer (TGA). These are fourth generation products from the world leader in thermogravimetric analysis. Each represents an unparalleled investment because it delivers outstanding performance, is designed with the customer in mind, and is backed by superior support that is the hallmark of our company.

Furnace

Our custom-designed furnace is a key element of a Q Series 500 / 50 TGA. It features low mass, rugged heater windings, and proprietary heater control technology. User benefits include rapid, accurate, and precise temperature programming over a wide range, plus optimized use in the Q500 of advanced techniques such as Hi-Res™ TGA and Modulated TGA™. Our reliable, long-life furnaces also increase the value of your investment.

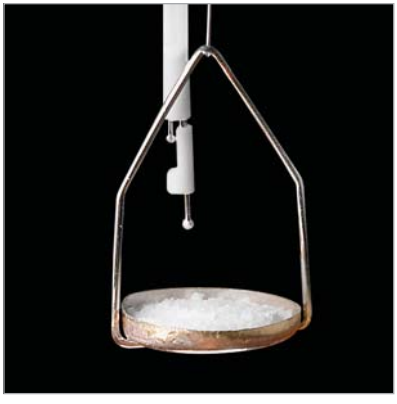
Thermobalance

The heart of a Q500 / Q50 TGA is the accurate and reliable vertical thermobalance housed in a temperature-compensated environment. Unlike competitive instruments, no expensive circulator is required for optimal performance. It uses the field proven and industry-standard null-balance principle, which is free from the complications also inherent in competitive designs. The Q50/500 balance provides the best accuracy and precision in weight change detection from ambient to 1000 °C, low baseline drift, and smooth, reliable operation over the entire weight range.



Temperature Control and Measurement

Our unique, custom-designed system features a single control / sample thermocouple positioned immediately adjacent to the sample. A second thermocouple is located in the same sleeve slightly above the principal one. The design ensures that simultaneous heating rate control and sample temperature measurement are accurately and precisely accomplished. This innovative "control and feedback" design enables the system controller to program and maintain the temperature environment and heating rate selected by the operator. The second thermocouple also serves to automatically disable the furnace should the temperature difference between the thermocouples exceed a set value.

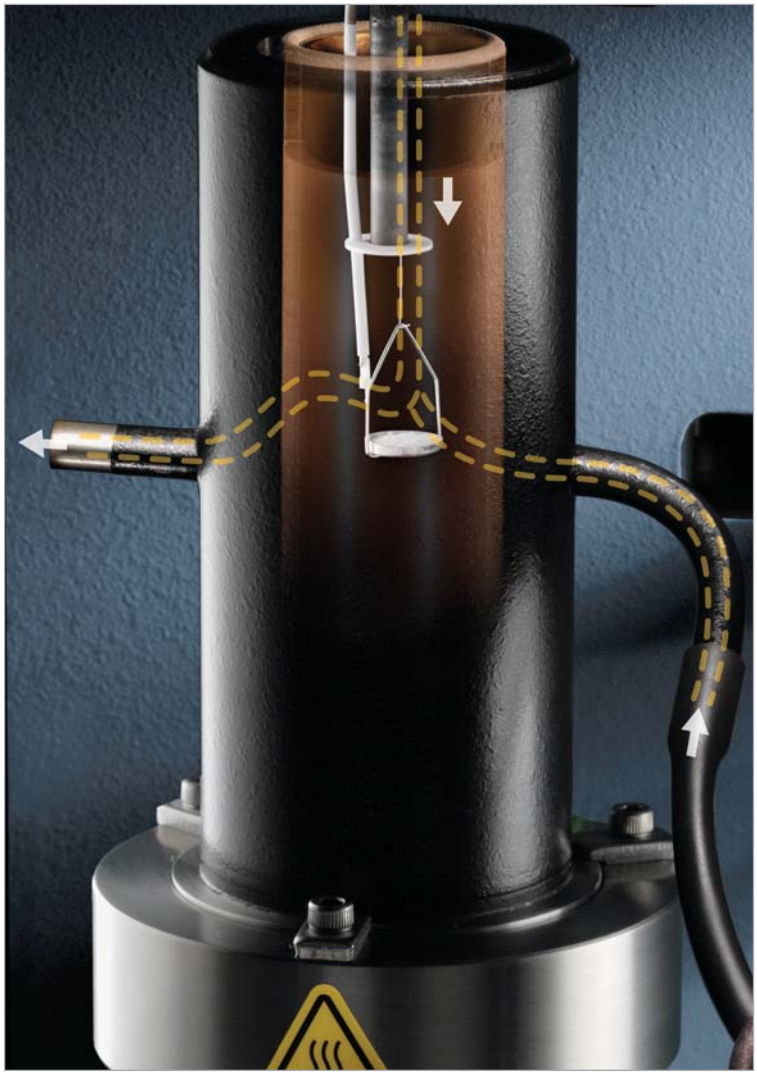


Purge Gas System

An efficient horizontal purge gas system allows accurately metered purge gas to flow directly across the sample, and is expertly integrated into the vertical thermobalance / furnace design. A regulated portion of the gas is also directed through the balance chamber to eliminate backflow, and the combined gases plus any sample effluent exit the system by a side arm. The design minimizes buoyancy effects, and optimizes removal of decomposition products from the sample area. The digital mass flow controllers improve data quality.

Mass Flow Control (with automatic gas switching)

Dual digital mass flow controllers (standard on all TA Instruments TGAs) provide accurate and precise purge gas metering that consistently exceeds conventional analog flow control devices for superior data quality. The automatic low volume, high-speed switching valves deliver instantaneous change of purge gas that is critical when converting between inert and oxidizing atmospheres. Gas flow rates are available as stored data file signals.



TGA Accessories & Options

Evolved Gas Analysis (EGA) Furnace

The rugged and reliable EGA is an optional, quartz-lined furnace for the Q500 or Q50. The liner is essentially chemically inert to products produced from decomposition of the sample, resistant to adsorption of off-gas products, and its reduced internal volume ensures rapid exit of these materials from the sample chamber. These features make the EGA an ideal furnace for use in combined TGA/MS or TGA/FTIR studies.

TA Instruments offers a 300 amu bench-top, triple-filter, quadrupole mass spectrometer with a heated capillary interface, and TGA module-specific interface kits for its Q5000 IR, Q500 and Q50 modules. A mass spectrum database is also available. A variety of FTIR suppliers provide gas cells and interfaces for use with all our TGA modules.

Autosampler

The Q500 Autosampler accessory is a programmable, multi-position sample carousel that allows fully automated analysis of up to 64 samples (16 samples per tray). All aspects of sample testing are automated and software controlled, including pan taring and loading, sample weighing, furnace movement, pan unloading, and furnace cooling. The autosampler has the flexibility to meet the needs of both research and QC laboratories. Autosampler productivity is maximized by our Advantage™ software, which permits pre-programmed analysis, comparison, and presentation of results.

Temperature Calibration And Weight Loss Verification

TA Instruments offers the widest range of ICTAC certified and NIST traceable Curie Point reference materials that permit full temperature calibration of the Q Series TGA modules over the range from 150 to 1120 °C. The advanced Q5000 IR has an internal electromagnet coil that permits temperature calibration with unprecedented convenience (see page 60).

TA Instruments also offers certified Mass Loss Reference Materials for validation of instrument performance (see page 69).

Advanced TGA Techniques

TA Instruments has been the pioneer in advancing the science of improved resolution TGA techniques, and in providing powerful but easily used software to accelerate material decomposition kinetic studies while preserving good data quality.

High Resolution TGA™ (Hi-Res™ TGA)

Hi-Res TGA* is a patented furnace control technology that produces significant improvements over standard linear heating rate TGA in the separation of closely occurring decomposition events. Both the Q5000 IR and the Q500 designs are ideal for this purpose, with rapid response furnaces for tight precise temperature control and sensitive thermobalances designed to quickly detect small weight changes. Specific control algorithms (constant reaction rate and dynamic rate) are supplied with the Q5000 IR and are available for the Q500. Auto-stepwise isothermal is a third high resolution technique, and is supplied with all the Q Series™ TGA models. Each offers specific advantages in resolution, but the dynamic rate technique is simpler to use, requires less operator expertise, and generates high quality results faster than the other methods. This capability is particularly valuable in analytical methods development.

Modulated TGA™ (MTGA™)

MTGA** is another TA Instruments innovation that offers advantages for material decomposition studies. It is supplied with the Q5000 IR and as an option with the Q500 TGA. Its development arose from the proprietary heater control technology developed for Hi-Res TGA and MDSC®*. MTGA produces model-free kinetic data, from which activation energy can be calculated and studied as a function of time, temperature, and conversion. It is easy-to-use and produces in a single run, the kinetic data needed to improve industrial process productivity.

*U.S. Patent No. 5,165,792
Canadian Patent No. 2,051,578
European Patent No. 0494492
**U.S. Patent No. 6,113,261 and 6,336,741

Sample pans

Q5000IR

Platinum (50 and 100 µL), ceramic (100 and 250 µL), and aluminum open (80 µL) and sealed (20 µL) pans are available for the Q5000 IR TGA. Platinum pans are recommended for most applications (ambient to 700 °C) for inertness and ease of cleaning. For operation to 1200 °C the ceramic pans (with ceramic bale) are recommended. The larger pan is best for higher volume / low density samples such as foams. The ceramic pans are also advised for samples that react with or form alloys with platinum. The aluminum pans are cost-effective substitute pans but cannot be used above 600 °C. The aluminum pans are also used in conjunction with aluminum lids to provide the sealed pan system. These new pans are designed exclusively for the Q5000 IR.

Q5000SA

Semispherical metal-coated quartz crucibles (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.

Q500 / Q50

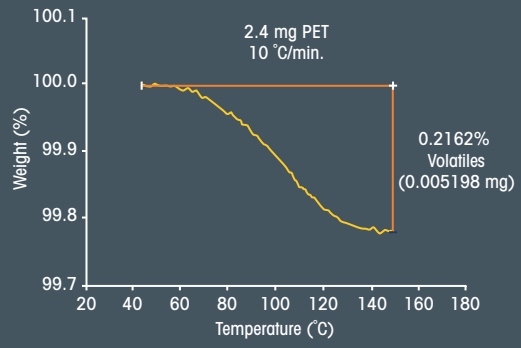
Platinum (50 and 100 µL), and new style ceramic (100, 250, and 500 µL) pans are available for use with the Q500 and Q50 TGA modules from ambient to 1,000 °C. Platinum pans are recommended in most cases due to its inertness and ease of cleaning. The larger ceramic pans are best for analysis of higher volume / low density samples such as foams. They are also advised for use with samples that react with or form alloys with platinum. The aluminum (100 µL) pans are cost-effective substitute pans but cannot be used above 600 °C.

Applications

High Sensitivity Volatiles Analysis

Unwanted water or other volatiles in a formulation are often detrimental during product processing. The improved sensitivity of the Q5000 IR allows even small amounts of these undesirable components to be quantified. Sealed aluminum pans allow moisture sensitive samples to be isolated and queued in the autosampler until moments before analysis. Figure 1 shows the volatiles analysis for a small (2.4 mg) sample of polyethylene terephthalate (PET) bottle stock. The 0.2% weight change reflects an absolute weight loss of only 5.2 micrograms!

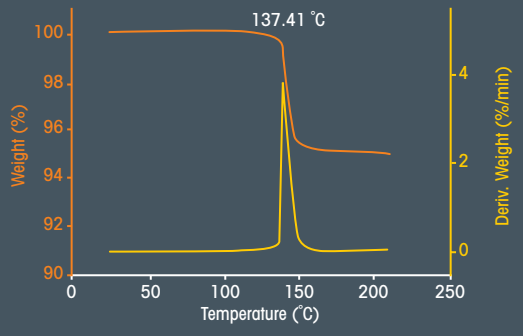
FIGURE 1



Characterization of Hydrates

Pharmaceutical scientists routinely characterize the thermodynamic parameters of drug candidates. Figure 2 shows the determination of the hydrate decomposition temperature for lactose monohydrate, an excipient material. The sample was sealed in a capsule having a 20-micron hole (Knudsen cell) to suppress weight loss until the equilibrium vapor pressure is reached. Use of radiation heating in the Q5000 IR results in improved temperature response while maintaining excellent resolution.

FIGURE 2



Accurate Residuals

A common TGA analysis is the determination of the amount of inorganic filler or pigment in an organic matrix. A key element in the analysis is residue accuracy, which depends on baseline quality and tare reproducibility, two aspects which have been improved by an order of magnitude in the Q5000 IR. Figure 3 shows the decomposition of a 15 milligram sample of a polyolefin fruit juice package, in which the 0.28% residue has been measured to hundredths of a percent!

Faster Separations

For routine separation of organic and inorganic components, the agile response of the infrared furnace in the Q5000 IR sharply reduces sample analysis time by fast heating and quick cool-down. Figure 4 shows the determination of carbonate filler in a polypropylene matrix at 40 °C/min and at 500 °C/min respectively. The Q5000 IR allowed the analysis test time to be reduced by a factor of at least six (6), while preserving excellent data quantitation. Multiple sample analysis using the integrated 25-position autosampler would be a significant productivity enhancer in analyses of this type.

FIGURE 3

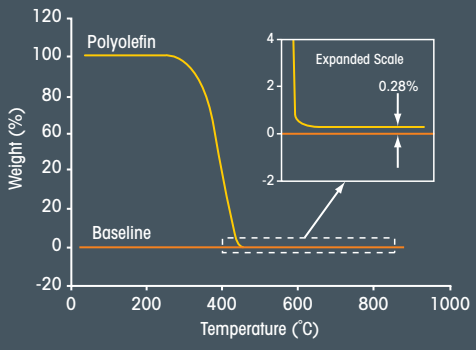
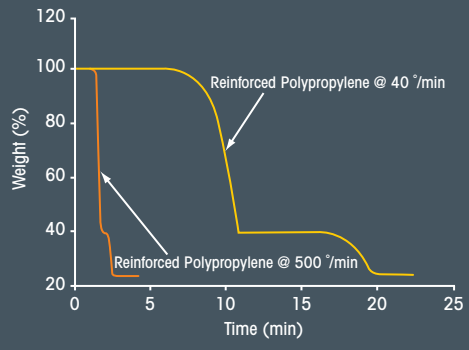


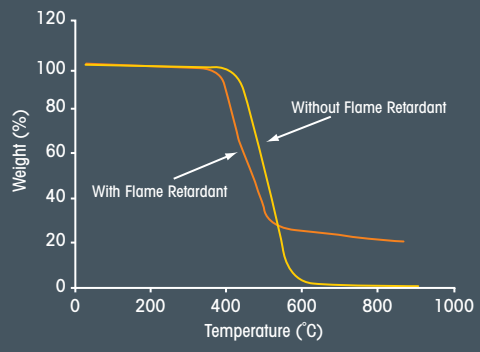
FIGURE 4



Flame Retardant Test

The addition of flame retardant additives to a product is a requirement for many materials produced or imported into most developed countries today. In practice, the flame retardant additive functions to moderate oxidative decomposition of materials by evolving a non-flammable component, which blankets the material as it decomposes. Use of the fast scanning rate capability of the Q5000 IR better simulates the conditions of a fire. Figure 5 shows TGA of polypropylene with, and without, a flame retardant additive run in the Q5000 IR TGA at 500 °C/min in air. The degree of effectiveness of the flame retardant is seen as the decomposed material effectively "smothers" the unreacted sample and prevents further oxidation.

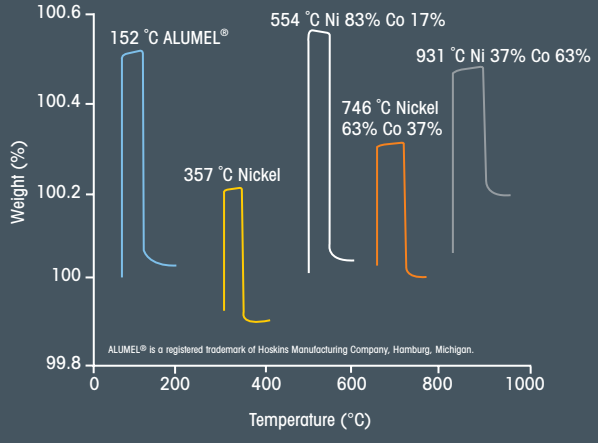
FIGURE 5



Simplified Curie Point Temperature Calibration

The electromagnetic coil integrated into the Q5000 IR furnace greatly simplifies Curie Point calibration measurements. This is a selectable method segment that can be changed in the course of an experiment so that Curie Temp materials of differing magnetic properties can be characterized in the same experiment.

FIGURE 6



Gravimetric Moisture Sorption Analysis – General Practice

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

In isotherm 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 controlled RH steps and the process repeated in an increasing or decreasing procedure. Isohum 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.

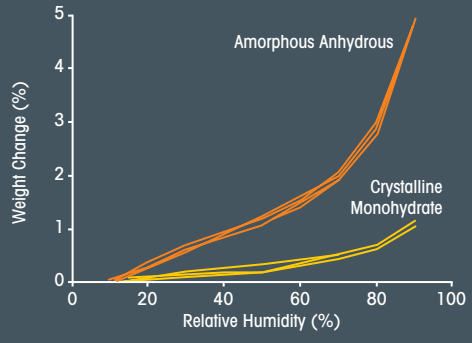
The Q5000 SA analysis software offers Sorption Analysis, BET Analysis, and GAB programs. In addition, the full power and flexibility of our renowned Universal Analysis software provides for easy data manipulation, advanced reporting, plotting and file exporting capabilities. Platinum™ control software provides for several new user convenience features such as system diagnostics, e-mail results notification and web-mail update notification of new software features.

The Q5000 SA is factory calibrated for temperature and humidity. Certified deliquescence salts (e.g., NaCl, NaBr) are widely accepted as standards for periodic verification of system performance. TA Instruments Platinum™ Software with scheduling and autoanalysis features simplifies the verification process.

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. Figure 1 shows the moisture sorption analysis of a generic drug in its amorphous and crystalline forms. As the amorphous form absorbs significantly more water, the Q5000SA can be used to quantify relative amorphous content in drug mixtures.

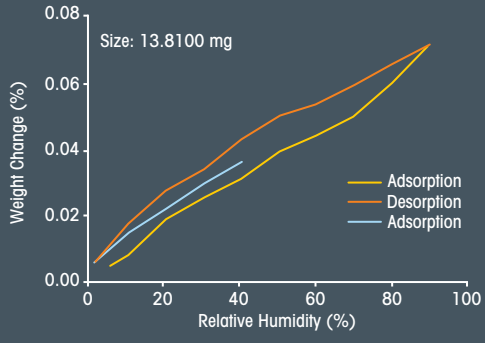
FIGURE 1



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 crystalline drugs such as prednisone, which adsorbs <0.1% moisture over a broad humidity range. The sorption results shown in Figure 2 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.

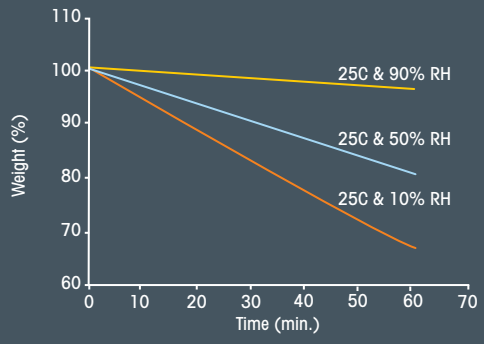
FIGURE 2



Optimizing Drying Conditions: Latex Paint

Drying of paints is a process that is strongly affected by the temperature and humidity of the environment where the paint is being used. The Q5000 SA with sealed/punched pans is an excellent way to study and compare different situations on a given paint formulation. Figure 3 contains the data from the drying of latex paint at 25°C, and 10%, 50% or 90% relative humidity. From the data in Figure 3, it is clear that the relative humidity dramatically influences the rate of drying. After 60 minutes, the sample at 10% RH has lost nearly 33% of it is initial mass, whereas the sample at 90% RH has lost only about 4%. Obviously, the sample at 90% RH would still be considered "Wet Paint" after one hour.

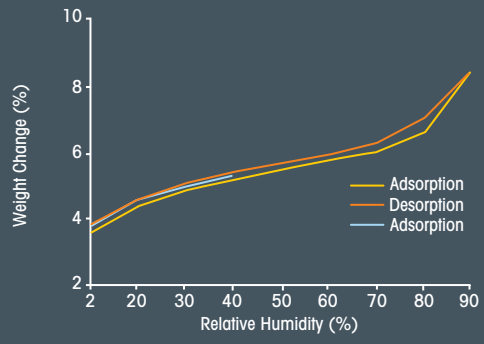
FIGURE 3



Evaulation of Hygroscopic Materials: Zeolite Catalyst

Porous materials such as zeolites tend to pick up water through both an "absorption" (molecules taken up by the volume) and "adsorption" (molecules taken up by the surface). The difference in sorption mechanism is often a function of the pore size. Figure 4 contains the sorption analysis of a zeolite material. The absence of hysteresis on increasing and decreasing the % RH, as well as the relatively small amount of water taken up suggests that this particular zeolite is undergoing adsorption. This could be indicative of the relative pore size distribution.

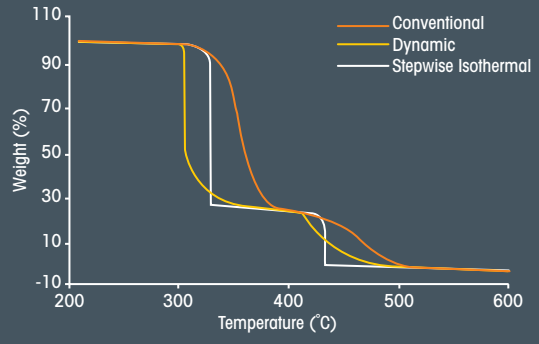
FIGURE 4



High Resolution™ TGA

Figure 5 shows comparative decomposition profiles of a polyvinyl acetate performed by standard, stepwise isothermal (SWI), and dynamic (Hi-Res) TGA techniques. The superior resolution provided by the latter pair is obvious. While the SWI method provided the separation with highest resolution for this sample, the Hi-Res technique produced comparable results in a fraction of the time needed to develop the SWI method, and also provided the high-resolution analysis much faster than the standard TGA method.

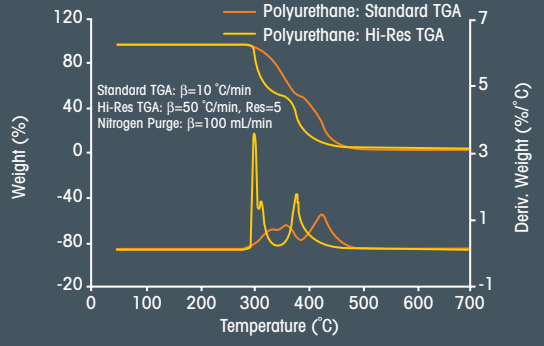
FIGURE 5



High Resolution™ TGA

Figure 6 compares the decomposition profile plots of a polyurethane material by standard and by Hi-Res TGA. The resolution superiority of the Hi-Res technique is clearly evident in both the TGA and first derivative (DTG) signals. The latter signal is especially useful in defining the onset and end set of the individual weight loss segments, as well as indicating subtle events that help to provide a "fingerprint" of the sample under the analysis conditions.

FIGURE 6



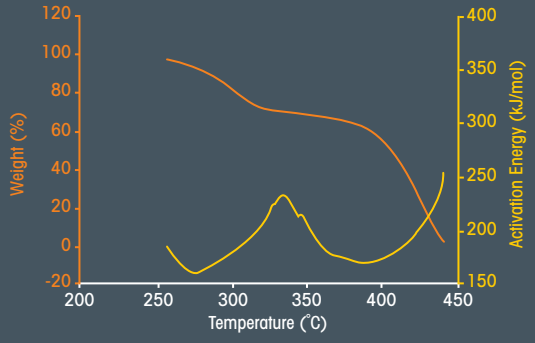
Modulated TGA™

Figure 7 shows data from a MTGA kinetic study of the effect of temperature on the decomposition of 60 % ethylene vinyl acetate (EVA) in a single analysis. The plot quantitatively shows the EVA decomposition profile and changes in activation energy as functions of temperature. The data supports a dual-step decomposition mechanism. MTGA can also monitor activation energy as a function of conversion, which indicates the mechanism involved. MTGA is supplied with the Q5000 IR and is available for the Q500.

Thermogravimetric Analysis measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. It is widely used in both research and quality control laboratories. TGA is particularly useful for the following measurements:

- Thermal stability
- Composition
- Oxidative stability
- Decomposition kinetics
- Estimated lifetime
- Moisture and volatile contents

FIGURE 7



Thermal Stability

TGA is often used to determine sample thermal stability and to reveal weight-loss decomposition profiles. Figure 8 shows typical thermal profiles for some common polymers (PVC, PMMA, HDPE, PTFE, and PI). The information allows materials selection for end uses where stability at specific temperatures is required.

Compositional Analysis

TGA is used to determine sample composition by measuring the weight of each component as it volatilizes or decomposes under controlled conditions of temperature, time, and atmosphere. Figure 9 shows quantitatively the differences in type, amount, and decomposition mechanism of the main polymers in three paint samples. More detailed examination of the profiles below 150 ˚C may reveal further information on the amount and possible nature of the carrier solvent (aqueous or oil) used in each paint.

FIGURE 8

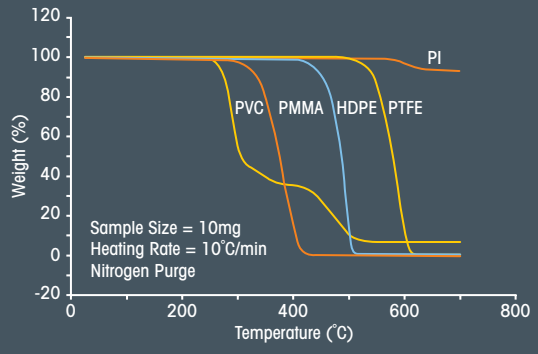
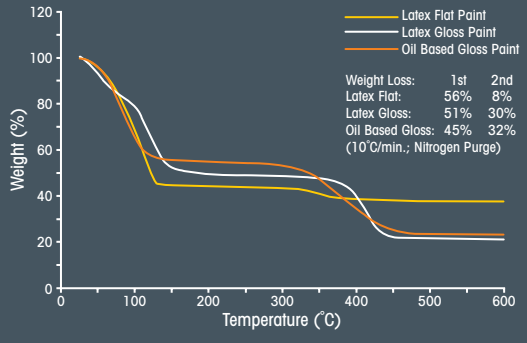


FIGURE 9



Volatiles Analysis

TGA determinations of absorbed, bound, or occluded moisture, and organic volatiles are important analyses for product performance and environmental acceptance. Analysis of an organic salt hydrate in nitrogen atmosphere (Figure 10) shows a bound-water content of 9.6%, and two lower temperature weight losses of 3.6% and 2.3% respectively. These losses are likely due to adsorbed moisture at the salt surface or held to it by weak attractive forces.

Effect of Additives

Figure 11 compares the decomposition profiles of a polycarbonate material with and without an added flame retardant. The flame retarded material consistently decomposed at a temperature about 20-25 ˚C lower than that of the unmodified sample. The former material also lost a greater percentage of weight than the standard material (e.g., 48% vs. 28%) at a given temperature (e.g., 460 ˚C) during the decomposition step. This indicates that flame-retardant additives accelerate the polycarbonate decomposition. The purpose of the retardent material is to inhibit flame propagation.

FIGURE 10

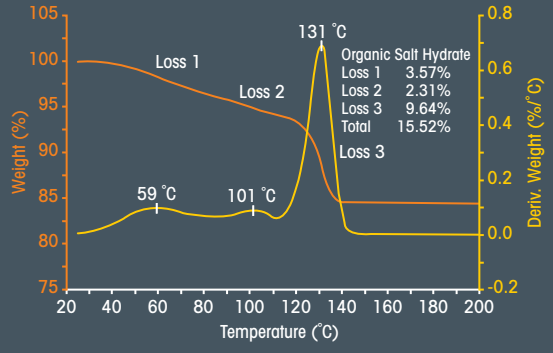
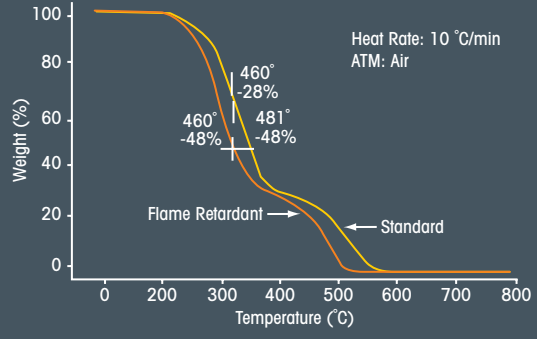


FIGURE 11



Dynamic Baseline Stability

Figure 12 shows a set of replicate Q5000 IR empty pan baseline runs from 50 ˚C to 950 ˚C. Baseline drift in all cases was considerably below 10 µg, and the reproducibility at this level of performance was excellent! This makes the Q5000 IR the ideal instrument for high sensitivity detection of low level of components in a matrix, such as volatiles in a polymer, food, or pharmaceutical product.

Isothermal Baseline Stability

Figure 13 shows an empty crucible, baseline drift experiment on the Q5000 SA, where a series of adsorption / desorption isotherms were programmed over a 1000 minute period at 25 ˚C over the relative humidity range from 5% to 95 % respectively. The total baseline drift was less than 1 microgram. This demonstrates the impressive level of temperature control in the Q5000 SA balance and in the humidity generation chamber. It confirms the instruments ability to perform high quality moisture sorption analysis.

FIGURE 12

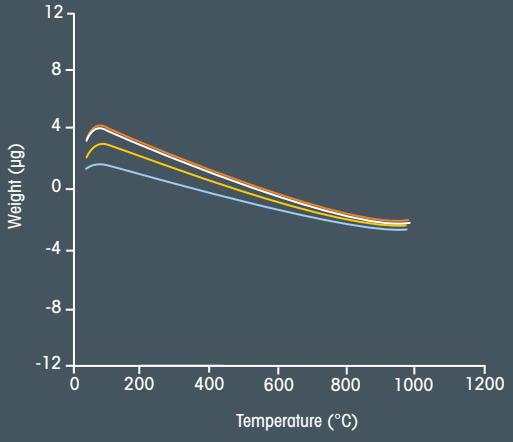
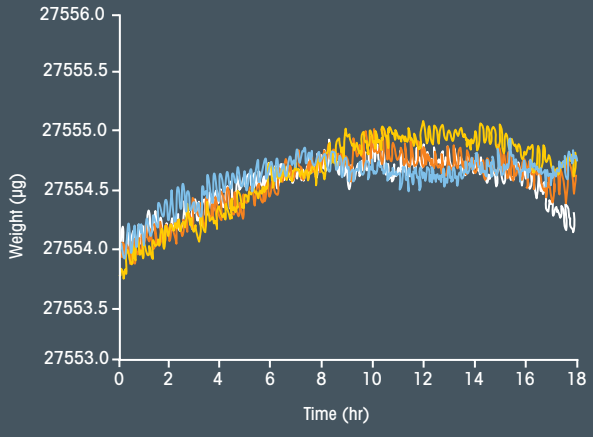


FIGURE 13



Curie Point Calibration - Electromagnet

Figure 14 shows an overlay plot of a series of ICTAC certified, NIST traceable Curie Point reference materials used to calibrate a TGA apparatus. The data was collected on the Q5000 IR and agrees very well with the published values. The Q5000 IR has an integrated electromagnetic coil that greatly simplifies the work involved in performing Curie Point determinations, and provides for automated temperature calibration.

Performance Verification

There is increasing interest in a means to verify the accuracy of measured weight changes (losses) by TGA. TA Instruments now offers certified Mass Loss Reference Materials for validation of instrument performance. These are 2 %, 50 % and 98 % solutions of 2-ethoxyethylacetate (b.p.150 °C) in a stable higher boiling polyol. Figure 15 shows a plot of the decomposition profiles of 2-ethoxyethylacetate at all three concentrations. The data confirms that there is no interference from the polyol in the determination.

FIGURE 14

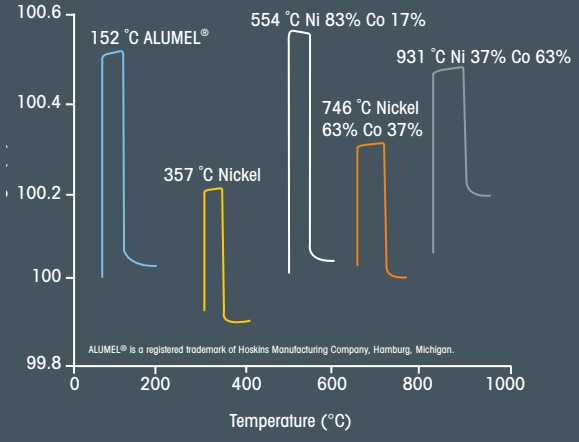
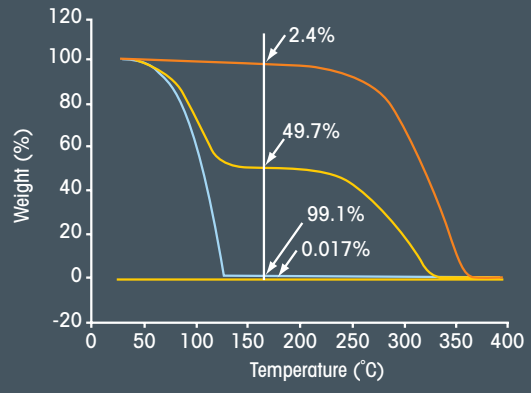


FIGURE 15



Notes