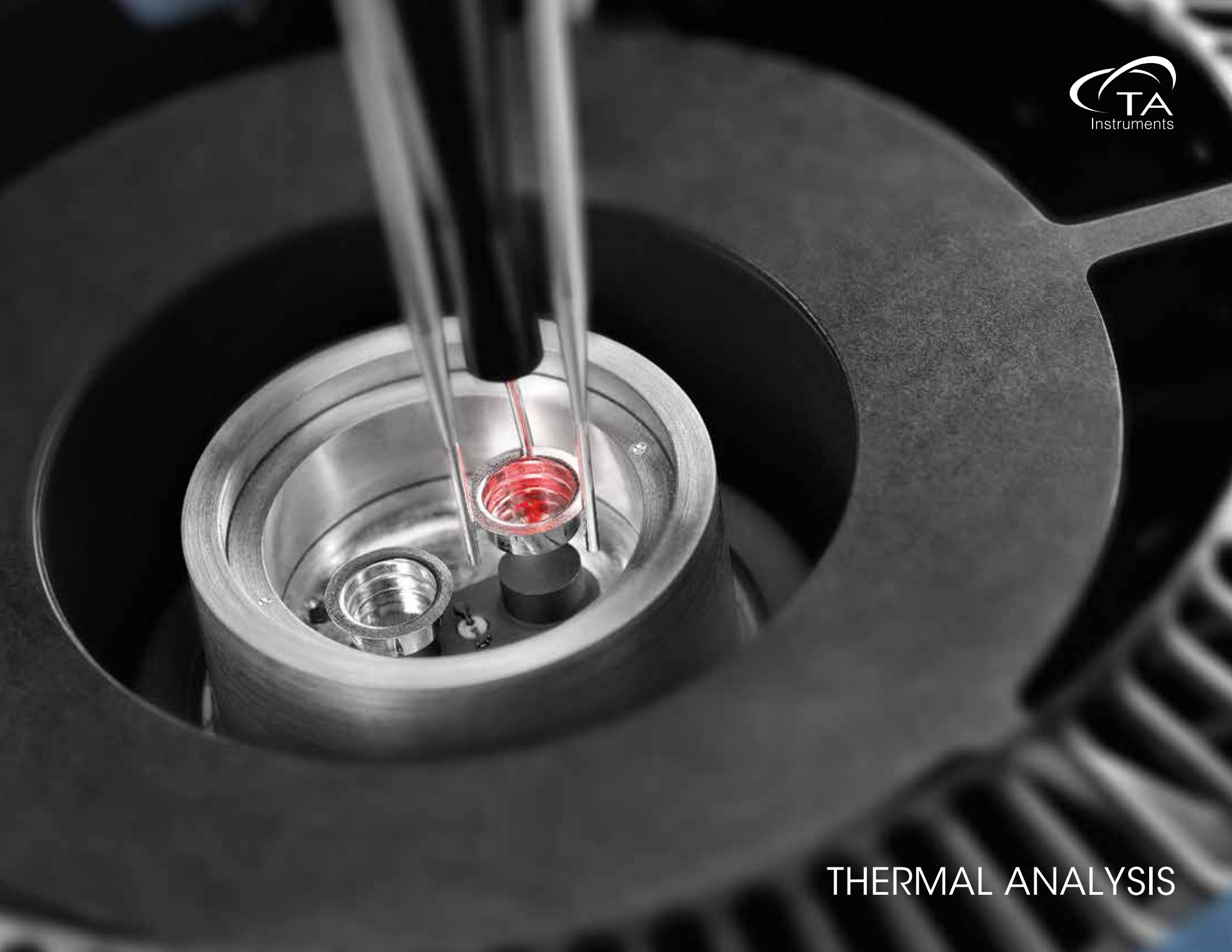
TA
Instruments
THERMAL ANALYSIS

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.

Thermogravimetric Analysis

A TGA designed to meet the most demanding research applications

Q500

THERMOGRAVIMETRIC ANALYSIS

The Q500 is the world's best-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.

Sensitivity	0.1 µg
Weighing Precision	± 0.01%
Baseline Dynamic Drift*	< 50 µg
Maximum Sample Weight	1 g
Dynamic Weighing Range	1g
Furnace Heating	Resistance Wound
Temperature Range	Ambient to 1 000°C
Isothermal Temp Accuracy	± 1°C
Isothermal Temp Precision	± 1°C
Controlled Heating Rate	0.01 to 100°C/min
Furnace Cooling (forced air/N2)	1000 to 50°C < 12 min
Temperature Calibration	Curie Point, ASTM E1582
Evolved Gas Analysis Furnace (EGA)	○
16 Position Autosampler	○
Hi-Res TGA™	○
Auto Stepwise TGA	●
Modulated TGA™	○
TGA/MS Operation	○
TGA/FTIR Operation	○
Platinum™ Software	●
Sample Pans	Platinum 50, 100 µL
	Ceramic 100, 250, 500 µL
	Aluminum 100 µL

* From 50 to 1000°C at 20°C/min using empty platinum pans, no baseline/blank subtraction.

● Included

○ Optional

Sensitivity	0.1 µg
Weighing Precision	± 0.01%
Baseline Dynamic Drift*	< 50 µg
Maximum Sample Weight	1 g
Dynamic Weighing Range	1g
Furnace Heating	Resistance Wound
Temperature Range	Ambient to 1 000°C
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Temperature Calibration	Curie Point, ASTM E1582
EGA Furnace	○
Auto-Loader	●
Auto Stepwise TGA	●
TGA/MS Operation	○
TGA/FTIR Operation	○
Platinum™ Software	●
Sample Pans	Platinum 50, 100 µL
	Ceramic 100, 250, 500 µL
	Aluminum 100 µL

* From 50 to 1000°C at 20°C/min using empty platinum pans, no baseline/blank subtraction.

● Included
○ Optional

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.

technology

Q500/Q50

Sensitive, precise, rugged, and automated all describe the TA Instruments Q500 and Q50 Thermogravimetric Analyzers (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

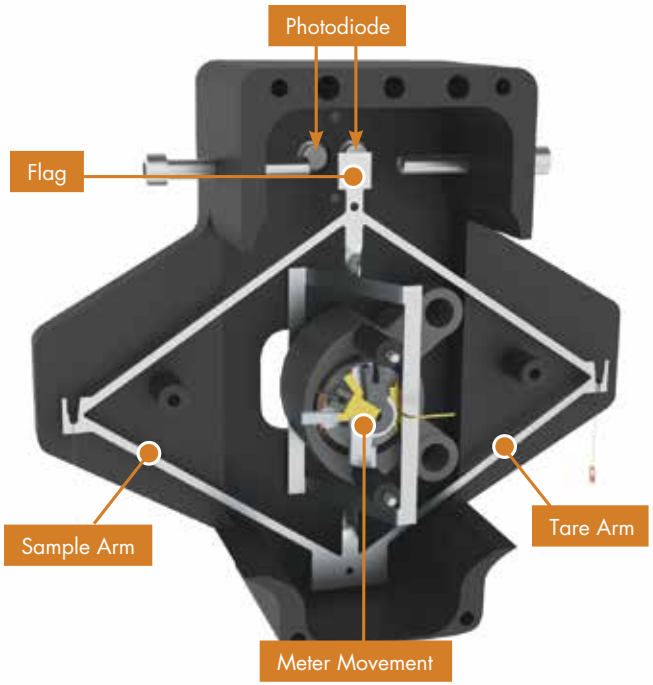
Our custom-designed furnace is a key element of a Q500/Q50 TGA. It features low mass, rugged heater windings, and proprietary heater control technology. User benefits include rapid, accurate, and precise temperature and rate programming, plus optimized use in the Q500 of advanced techniques such as Hi-Res™ TGA and Modulated TGA™. Our reliable, long-life furnaces also increase the value of your investment.

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 slightly above in the same sleeve. 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 as a safeguard to automatically disable the furnace should the temperature difference between the thermocouples exceed a set value.

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. 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.



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 baseline complications also inherent in competitive designs. The Q500/Q50 balance provides the best accuracy and precision in weight change detection from ambient to 1000˚C, low baseline drift, and sensitive, reliable operation over the entire weight range.

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.

accessories & options

Evolved Gas Analysis (EGA) Furnace

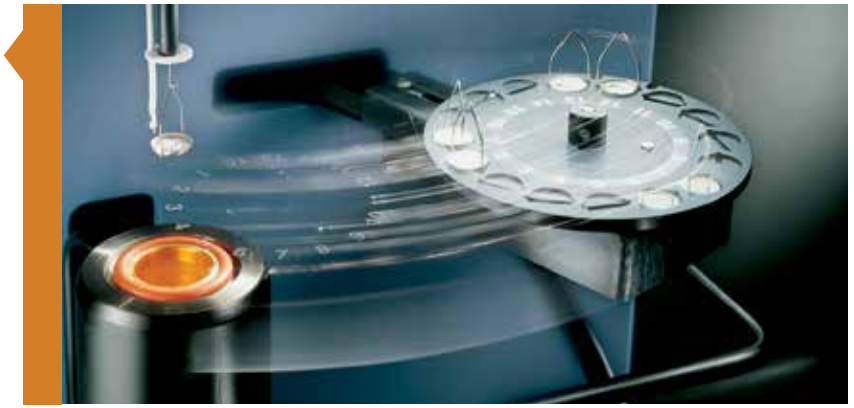
The rugged and reliable EGA is an optional, quartz-lined furnace for the Q500 or Q50. The liner is chemically inert to products produced from decomposition of the sample, resistant to adsorption of offgas products, and its reduced internal volume ensures rapid exit of these materials from the sample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

Sample Pans

Platinum (50 and 100 µL), and 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.

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.



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 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 Discovery TGA and the Q500 designs are ideal for this purpose, with rapid response furnaces for 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 Discovery TGA and are available for the Q500. Auto-stepwise isothermal is a third high resolution technique, and is supplied with all the TA Instruments TGA models.

*U.S. Patent No. 5,165,792
Canadian Patent No. 2,051,578
European Patent No. 0494492

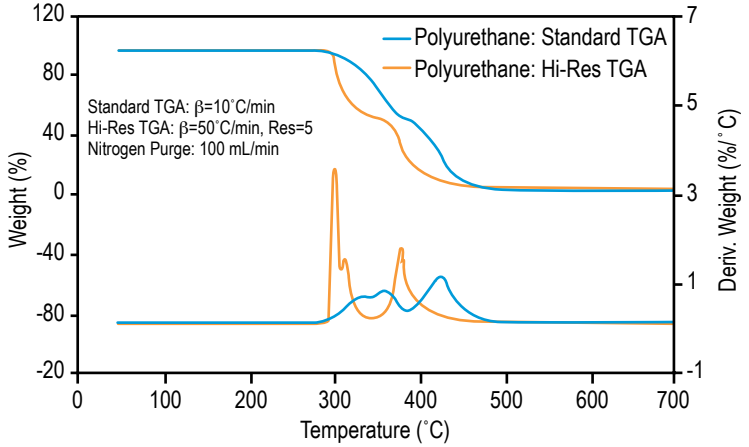
Modulated TGA™ (MTGA™)

MTGA** is another TA Instruments innovation that offers advantages for material decomposition studies. 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 Nos. 6,113,261 and 6,336,741

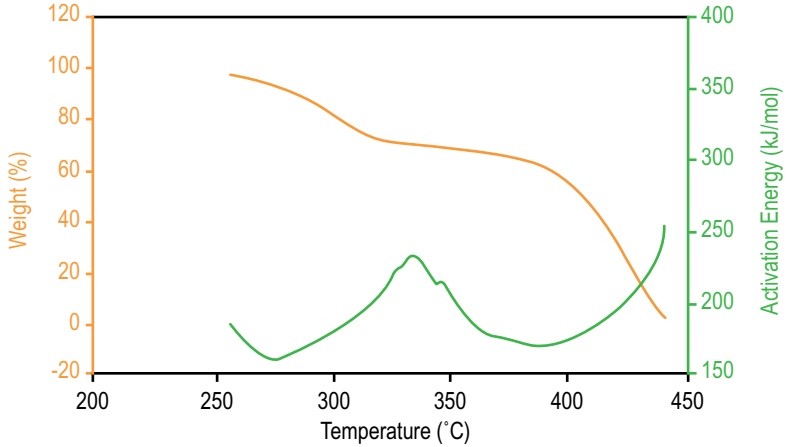
High Resolution™ TGA

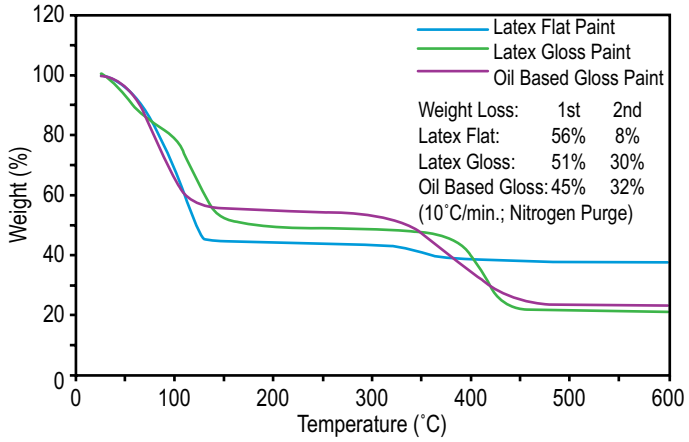
This figure compares the decomposition profile plots of a polyurethane material by standard and by Hi-Res™ TGA. The resolution superiority of the Hi-Res technique is clearly evident in both the TGA and first derivative (DTG) signals. The latter signal is especially useful in defining the onset and endset 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.

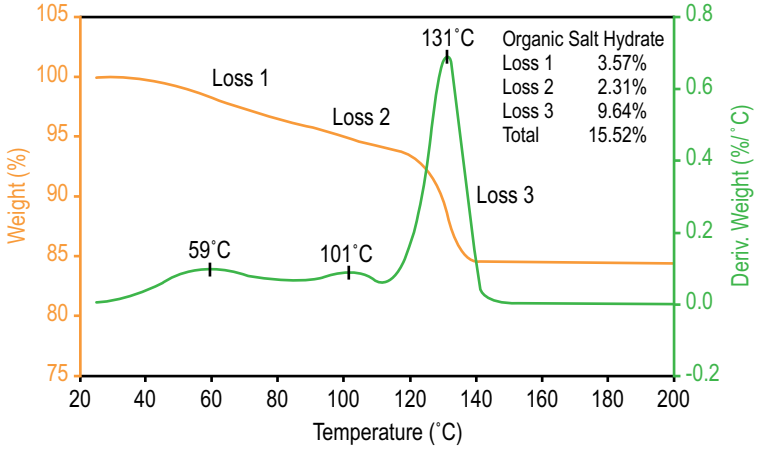


Modulated TGA™

The figure to the left shows data from a MTGA™ kinetic study of the effect of temperature on the decomposition of 60% ethylene vinyl acetate (EVA) in a single experiment. 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 available for the Q500.







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. This figure shows quantitative 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.

Volatiles Analysis

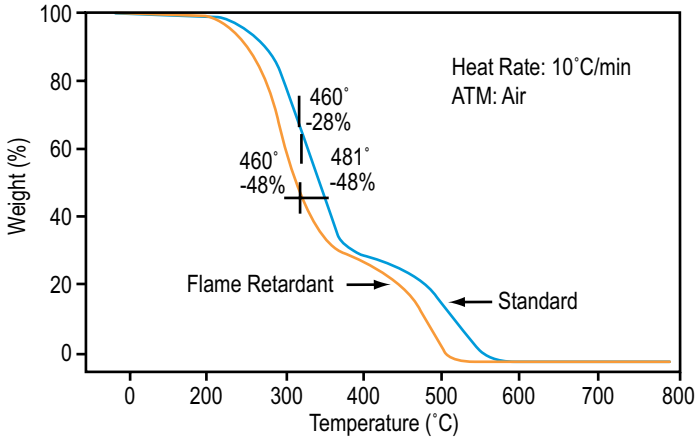
TGA determinations of absorbed, bound, or occluded moisture, and organic volatiles are important analyses for product performance and environmental acceptance. Analysis of an organic salt hydrate in nitrogen atmosphere shows a bound-water content of 9.6%, and two lower temperature weight losses of 3.6%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

applications

TGA

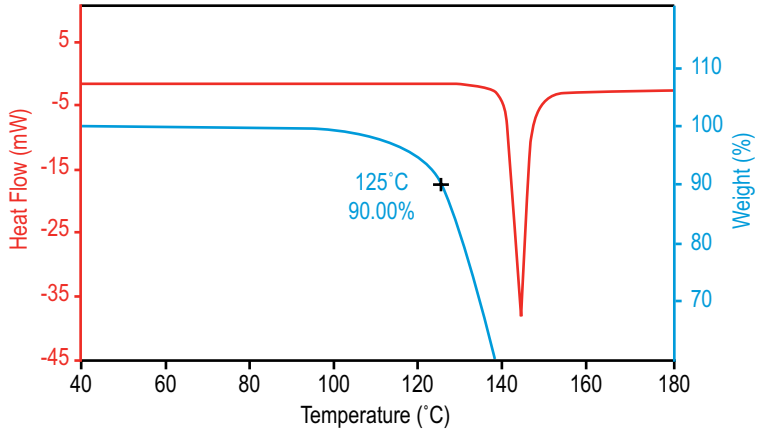
Effect of Additives

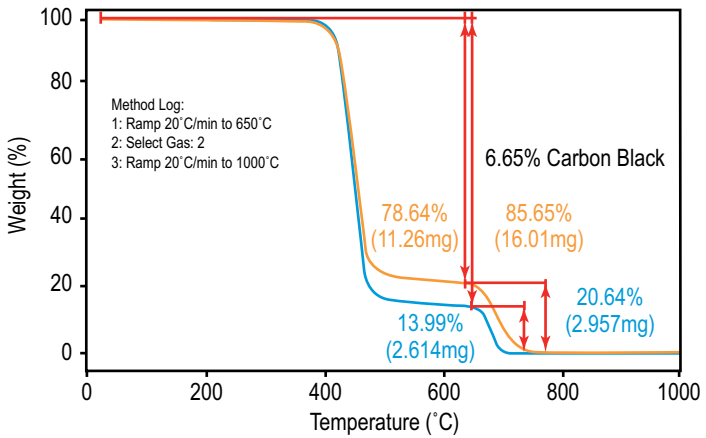
This figure compares the decomposition profiles of a poly-carbonate material with and without an added flame retardant. The flame-retarded material consistently decomposed at a temperature about 20-25°C lower than that of the unmodified sample. The former material also lost a greater percentage of weight 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 retardant material is to inhibit flame propagation.



Verification of Thermal Events

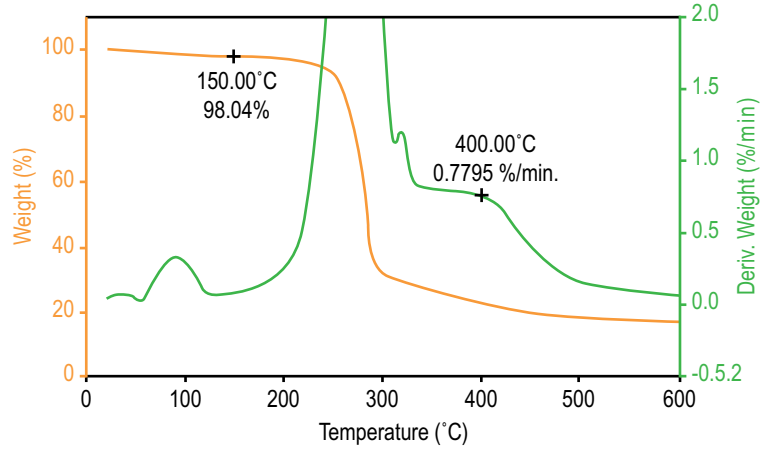
TGA is very useful in conjunction with other thermal analysis techniques such as DSC and is often critical to understanding the true nature of thermal events. In this data, a pharmaceutical material undergoes an endothermic transition above 125°C which was previously thought to be melting. TGA analysis demonstrates considerable weight loss below 125°C, which suggests that the endotherm is actually decomposition. DSC analysis at multiple rates exposes rate-dependence of this transition which confirms decomposition.





Quantification of Filler Content

TGA is a sensitive technique for analyzing and quantifying the filler content of polymeric composites. This figure contains a comparison of the TGA results for a virgin and filled PET sample. The virgin material is first analyzed for comparison. By quantifying the weight loss of the initial lower-temperature decomposition, and comparing it to the oxidative decomposition in the second weight loss, the filler content of the composite material is accurately quantified.



Moisture Content & Thermal Stability of a Pharmaceutical Material

TGA is a useful technique for determining the absolute and relative thermal stability of pharmaceutical compounds, as well as the moisture content. In this example, an active pharmaceutical ingredient (API) is analyzed by TGA at a heating rate of 10°C/min. The data show a small (~2%) weight loss below 150°C, which is typical for adsorbed water. The material is relatively stable up to 200°C, after which a large, multi-step weight loss is indicative of thermal decomposition.

evolved gas analysis

Evolved gas analysis involves the qualitative investigation of the evolved gas products from a TGA experiment. These products are generally the result of decomposition, but can also evolve from desorption, evaporation or chemical reactions. Evolved gas analysis is typically performed by interfacing a mass spectrometer (MS) or Fourier transform infrared spectrometer (FTIR) to the exit port of the TGA furnace. Through the use of a heated transfer line, the evolved gas stream is delivered to the MS or FTIR instrument, and the compositional analysis is performed in real time. TA Instruments offers a 300 amu bench-top, quadrapole mass spectrometer with a heated capillary interface, and TGA module-specific interface kits for its Discovery TGA, Q500 and Q50 modules. A variety of FTIR suppliers provide gas cells and interfaces for use with all our TGA modules.

TA Instruments Thermogravimetric Analyzers are the ideal platform for evolved gas analysis studies. Each TA Instruments TGA features a horizontal purge stream over the sample and a short path to the exit port. This eliminates dead volume in the furnace thereby reducing product dilution and optimizing EGA sensitivity. The Q500 and Q50 can be equipped with the quartz-lined evolved gas analysis (EGA) furnace which minimizes adsorption of effluent gases onto the furnace. The Discovery TGA features heated EGA adapters designed to interface directly with the MS or FTIR transfer line. These adapters ensure continuous heating of the offgas stream through the furnace wall, dramatically reducing offgas condensation and improving EGA sensitivity.

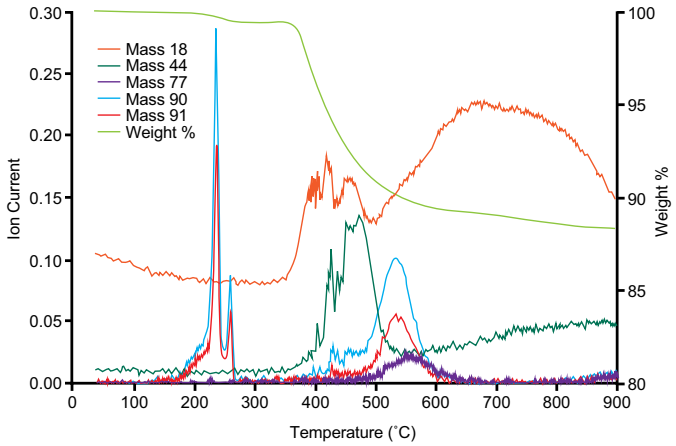
TA Instruments Universal Analysis software supports the importation of MS (trend analysis) and FTIR data (Gram-Schmidt and Chemigram reconstructions), allowing TGA and EGA data to be displayed on a common axis of temperature and/or time.

discovery mass spec

The Discovery MS is a benchtop quadrupole mass spectrometer, designed and optimized for evolved gas analysis. It is compatible with all thermogravimetric instruments in the TA Instruments product line, including the Discovery TGA, Q-Series TGAs, and Q600 SDT.

The Discovery MS features industry-standard technology configured for the efficient transfer, and rapid detection of offgas from the TGA furnace. Parts per billion (ppb) sensitivity is ensured with our state-of-the- art quadrupole detection system, including a closed ion source, a triple mass filter and a dual (Faraday and Secondary Electron Multiplier) detector system. This analyzer configuration is selected to optimize sensitivity and long term stability performance.

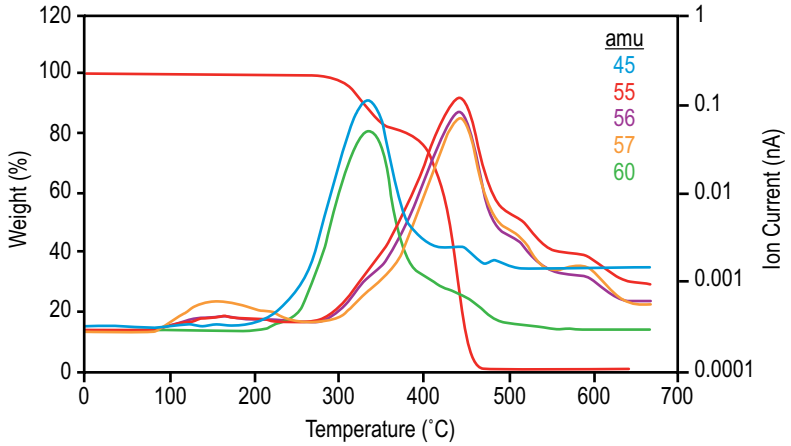
Control of the experimental parameters and analysis of the mass spectral data is achieved through a user-friendly, recipe-driven software interface. Data collection can be triggered directly from the TGA software, and the resulting MS data can be combined with the corresponding TGA results for direct overlaying and comparison.



Parameter	Performance
Mass range (amu)	1-300
Mass Resolution	>0.5 amu
Sensitivity	< 100 ppb (gas-dependent)
Ionization Source	Electron Ionization
Detector System	Dual (Faraday and Second Electron Multiplier)
Sample Pressure	1 atm (nominal)
Data Collection Modes	Bargraph and Peak Jump
Scanning Speed	
Bargraph Mode	>50 amu/s
Peak Jump Mode	>64 channels/s
Transfer line Temperature	300°C (fixed)
Transfer line	1.8 meters, flexible
Filaments	Dual, customer changeable
Capillary	Stainless Steel, changeable
Capillary size	I.D. = 0.22 mm
Inputs	Data collection controlled by TGA Trigger

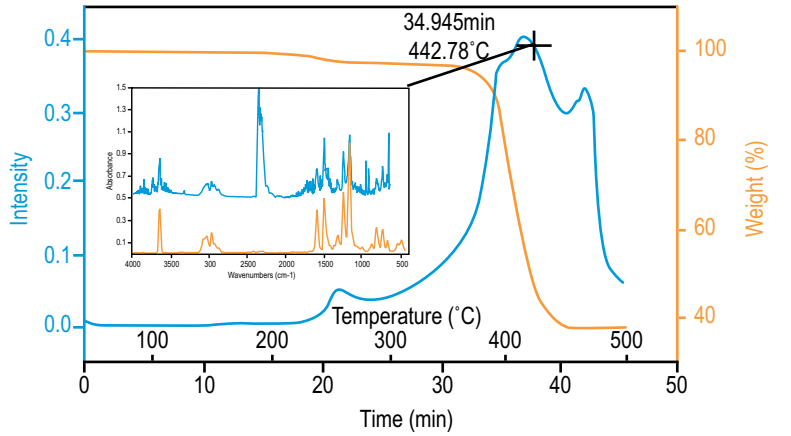
TGA-MS: Polymer Analysis

This data shows the TGA-MS results for the decomposition of ethylene vinyl acetate copolymer. The first step involves the decomposition of the vinyl acetate phase, resulting in the production of acetic acid. By monitoring signals typical of acetic acid, the production of the offgas compound is readily detected. The second step involves the thermal decomposition of the polyethylene phase, and its unique decomposition products are easily identified and recorded.



TGA-FTIR: Phenolic Resin Decomposition

This figure contains the TGA-FTIR results for the thermal decomposition of a phenolic resin adhesive. A Gram-Schmidt reconstruction of the time-resolved FTIR spectra is compared to the weight loss signal as a function of time and temperature. The inset image contains the FTIR spectrum of the offgas composition at 34.95 minutes, near the point of the maximum rate of decomposition. The FTIR spectrum corresponding to this temperature indicates that the offgas products are primarily composed of phenols, including bisphenol A, which is included as a comparison spectrum. This level of chemical specificity is useful in comparing similar products, quality control, and fingerprint analysis.



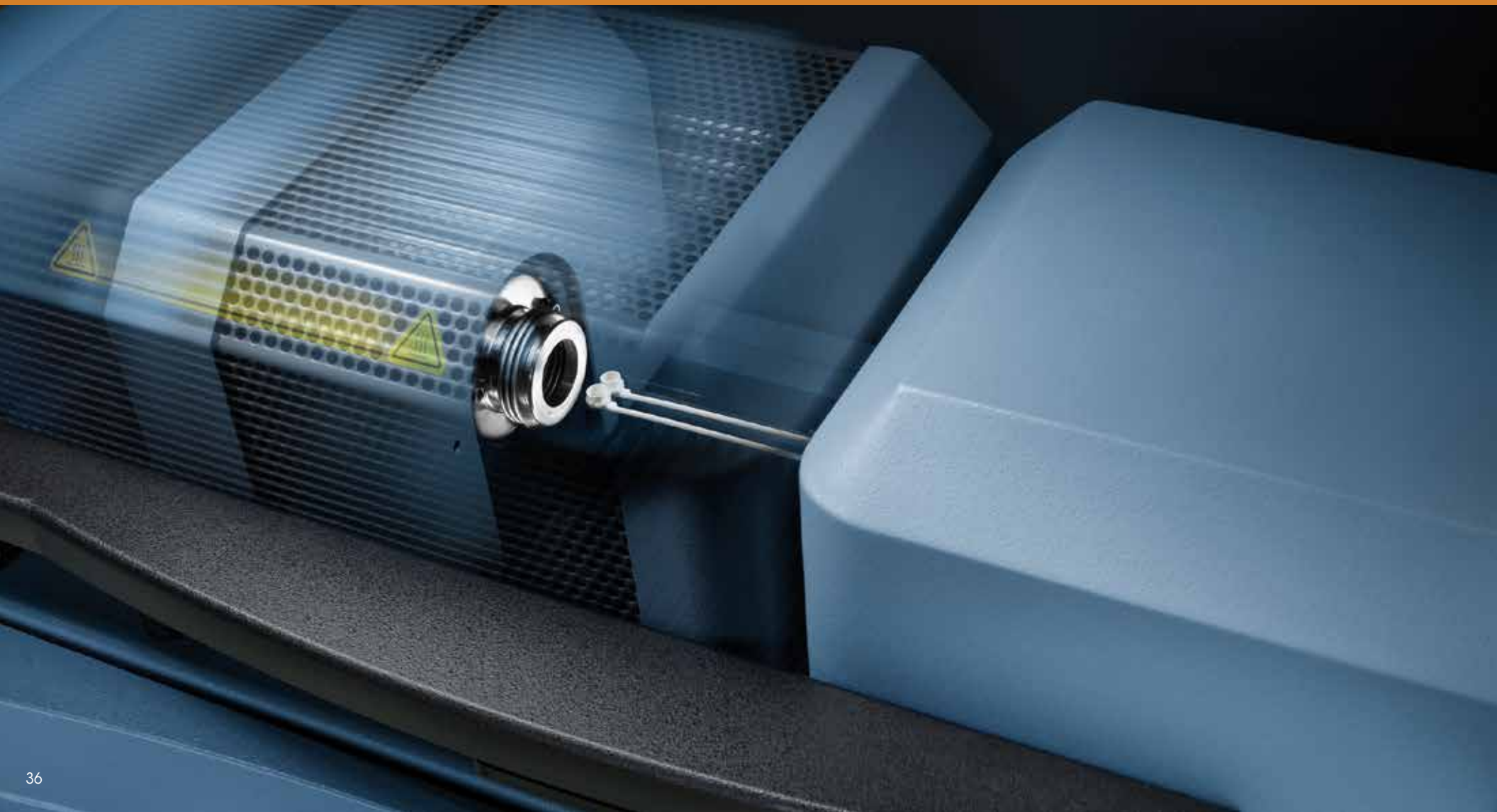
Simultaneous TGA/DSC

Providing high-quality, truly differential simultaneous measurements

Q600

SIMULTANEOUS TGA/DSC

The Q600 provides simultaneous measurement of weight change (TGA) and true differential heat flow (DSC) on the same sample from ambient to 1 500˚C. It features a field-proven horizontal dual beam design with automatic beam growth compensation, and the ability to analyze two TGA samples simultaneously. DSC heat flow data is dynamically normalized using the instantaneous sample weight at any given temperature.



specifications

Q600

Balance Sensitivity	0.1 µg
Baseline Dynamic Drift: RT to 1000˚C	< 50 µg
Baseline Dynamic Drift: 1000˚C to 1500˚C	< 50 µg
Sample Capacity	200 mg (350 mg including sample holder)
Temperature Range	Ambient to 1 500°C
Heating Rate – Ambient to 1 000°C	0.1 to 100°C/min
Heating Rate – 1000˚C to 1500°C	0.1 to 50°C/min
Furnace Cooling	Forced Air (1 500 to 50˚C in < 30 min, 1 000˚C to 50˚C in < 20 min)
Calorimetric Accuracy/Precision	± 2% (based on metal standards)
DTA Sensitivity	0.001°C
System Design	Horizontal Balance & Furnace
Balance Design	Dual Beam
Thermocouples	Platinum/Platinum-Rhodium (Type R)
Temperature Calibration	Curie Point or Metal Standards (1 to 5 Points)
Mass Flow Controller with Automatic Gas Switching	●
Vacuum	to 7 Pa (0.05 torr)
Reactive Gas Capability	● – separate gas tube
Dual Sample TGA	●
Auto-Stepwise TGA	●
Sample Pans	Platinum: 40 µL, 110 µL Alumina: 40 µL, 90 µL

● Included

technology

Q600

Thermobalance

The Q600 features a highly reliable horizontal dual-balance mechanism that supports precise TGA and DSC measurements. It delivers superiority in weight signal measurements (sensitivity, accuracy and precision) over what is available from single beam devices, since the dual beam design virtually eliminates beam growth and buoyancy contributions to the underlying signal. It also uniquely permits independent TGA measurements on two samples simultaneously.

Temperature Control and Measurement

A matched Platinum/Platinum-Rhodium thermocouple pair within the ceramic beams provides direct sample, reference, and differential temperature measurements from ambient to 1 500°C. This results in the best available sensitivity in detection of thermal events. Curie Point or pure metal standards can be used for single or multi-point temperature calibration. Calibration of the DSC signal with sapphire standards results in a differential heat flow (DSC) signal that is intrinsically superior to that from single beam devices.

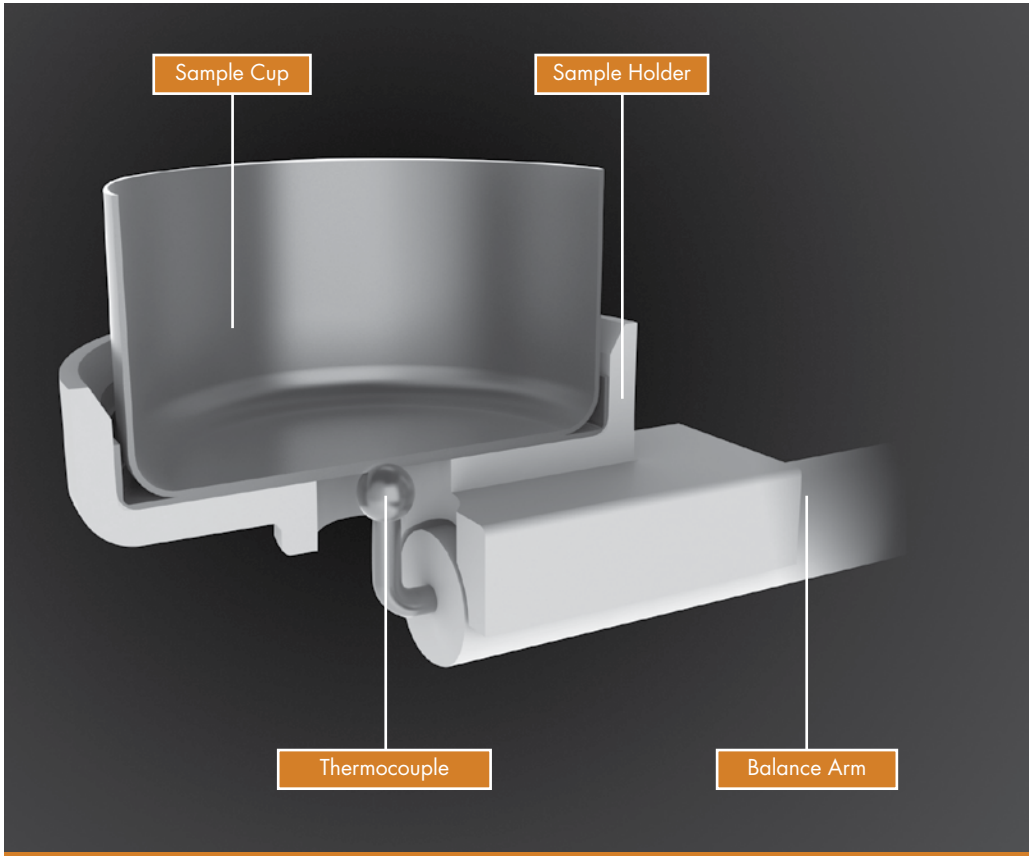
Furnace

The Q600 features a rugged, reliable, horizontal furnace encased in a perforated stainless steel enclosure. The design ensures accurate and precise delivery of programmed and isothermal operation over the full temperature range from ambient to 1 500°C. The design also provides for operator ease-of-use due to its automatic furnace opening/closing, easy sample loading, and rapid post-experiment furnace cool-down.

Purge Gas System

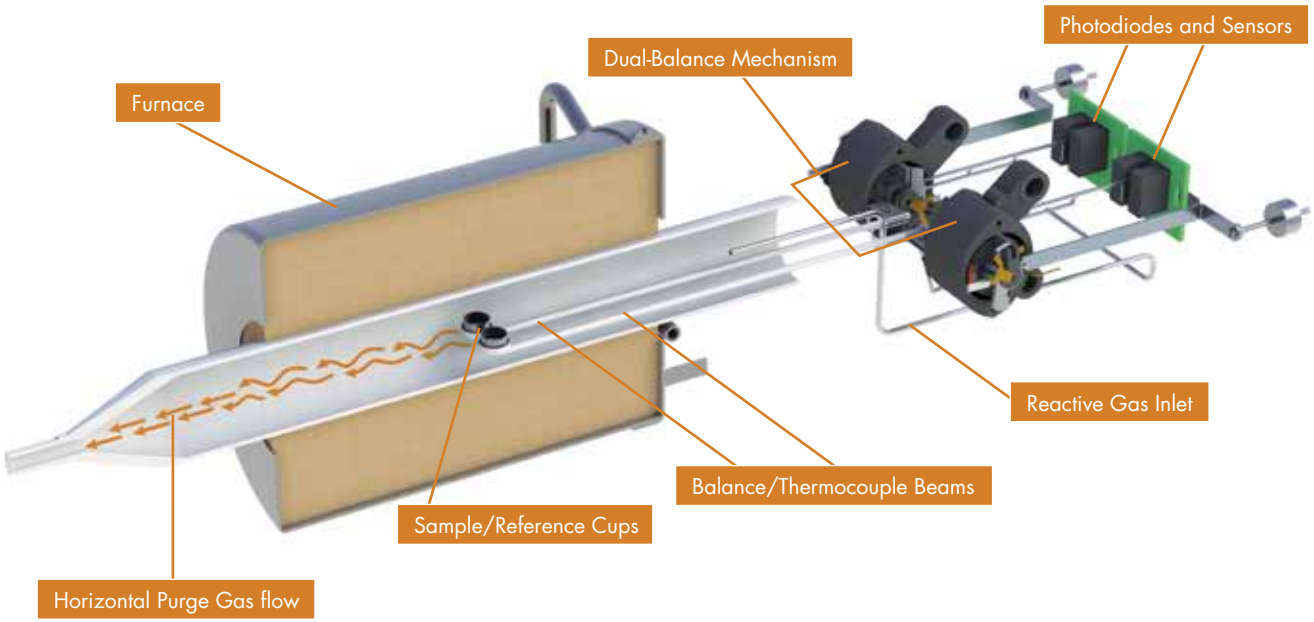
A horizontal purge gas system with digital mass flow control and integral gas switching capability provides for precise metering of purge gas to the sample and reference pans. The design produces better baselines, prevents back diffusion, and efficiently removes decomposition products from the sample area. A separate Inconel® gas inlet tube efficiently delivers reactive gas to the sample. The Q600 exhaust gas port can be readily connected to a MS or FTIR for component identification purposes.

® Inconel is a registered trademark of Special Metals Corporation



High Resolution SDT

If separation of closely related weight losses is required, the Q600 offers an automated version of StepWise Isothermal (SWI), the classical technique for improved TGA resolution. The stepwise isothermal approach consists of heating at a constant rate until a weight change begins (as determined by an operator-chosen rate or amount of weight loss) and then holding isothermally until the weight change is complete. This sequence of heating and isothermal steps is repeated for each weight change encountered. The result is optimum weight loss resolution.



Temperature Calibration and Weight Loss Verification

TA Instruments offers the widest range of ICTAC certified and NIST traceable Curie Point reference materials that provide SDT apparatus temperature calibration over the range from 150 to 1120°C. TA Instruments also offers certified Mass Loss Reference Materials for validation of SDT instrument performance.

Q600 Sample Pans

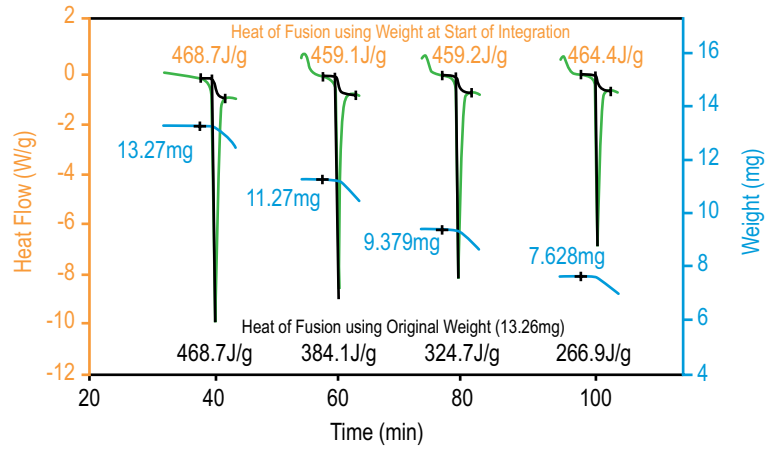
Platinum pans (40 and 110 µL) and ceramic cups (40 and 90 µL) are available for use with the Q600. The platinum cups are recommended for operation to 1 000°C, and for their general inertness and ease of cleaning. The ceramic cups are advised for operation to 1 500°C, and for samples that react with platinum.

applications

Q600

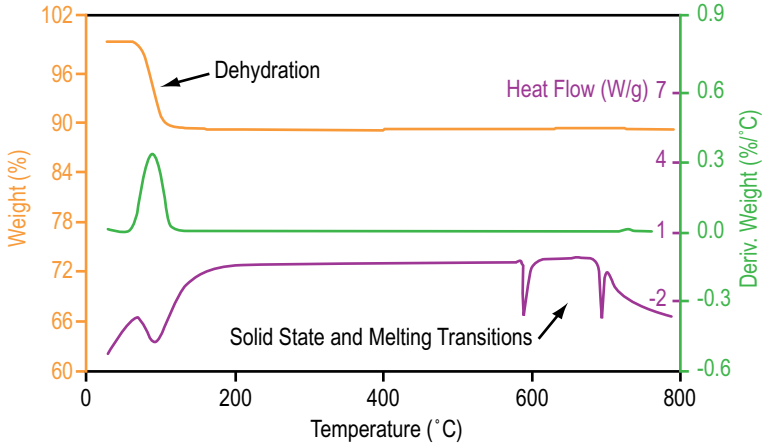
Improved DSC Data

In SDT experiments, superior accuracy in DSC data is obtained when the instantaneous weight (rather than the initial sample weight) is used in heat flow calculations. This figure shows data for sodium chloride (which loses weight on heating) cycled through its melt four times, and the heat of fusion (J/g) determined using the instantaneous weight. At the bottom of the figure are the determinations of heat of fusion using the original sample weight, and shows that the heat of fusion for the fourth melt is wrong by almost 60% compared to using instantaneous weight.



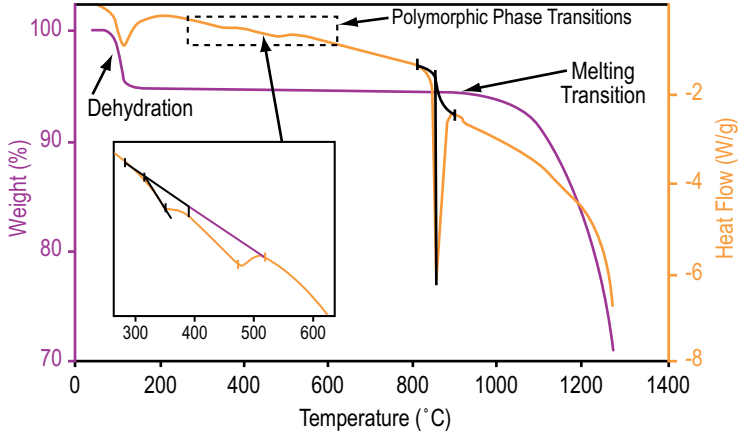
High Sensitivity

This figure shows a high sensitivity application of the Q600 in which a small (3 mg) sample of sodium tungstate is analyzed at 10°C/min from ambient to 800°C. The TGA and derivative TGA (DTG) signals quantitatively record the dehydration step. The DSC trace quantitatively shows the loss of water plus higher temperature solid state phase and melting transitions respectively. The latter pair are thermal events where no weight loss occurs.



Simultaneous DSC/TGA

This figure contains simultaneous DSC and TGA data to 1 300°C for a soda ash sample. The TGA signal measures the dehydration and the onset of a higher temperature decomposition. The DSC signal reveals transitions associated with the dehydration, a polymorphic phase transition and the high temperature melt. The inset shows details about the phase transition. In the Q600, heat flow integrations are automatically normalized using the dynamic weight at the start of each transition.



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