

# TA Instruments THERMAL ANALYSIS

New Castle, DE USA

Lindon, UT USA

Crawley, United Kingdom

Shanghai, China

Beijing, China

Taipei, Taiwan

Tokyo, Japan

Seoul, Korea

Bangalore, India

Paris, France

Eschborn, Germany

Brussels, Belgium

Etten-Leur, Netherlands

Sollentuna, Sweder

Milano, Ita

Barcelona, Spair

Melbourne, Australia

Mexico City, Mexico





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, Hi-Res™ TGA, Modulated TGA™, 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
Dynamic 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
Ballistic Heating	>2000 °C/min
Linear Heating Rate	0.1 to 500 °C/min
Furnace Cooling (Forced air / N <sub>2</sub> )	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
TGA/FTIR Operation	Option
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 μL, Platinum-HT, 100 μL, Ceramic 100, 250 μL, Aluminum 80 μL, Aluminum Sealed Pan 20 μL

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



The Q500 is the world's #1 selling, research-grade thermogravimetric analyzer. Its field-proven performance arises from a responsive low-mass furnace, ultrasensitive 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 to 1000 °C
Isothermal Temp Accuracy	+/- 1 °C
Isothermal Temp Precision	+/- 0.1 °C
Controlled Heating Rate	0.01 to 100 °C/min
Furnace Cooling (forced air / N <sub>2</sub> )	1000 to 50 °C < 12 min
Temperature Calibration	Curie Point
16 Position Autosampler	Optional
Hi-Res TGA™	Optional
Auto Stepwise TGA	Included
Modulated TGA™	Optional
TGA/MS Operation	Optional
TGA/FTIR Operation	Optional
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 µL
	Ceramic 100, 250, 500 µL
	Aluminum 100 µL



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 μς
Baseline Dynamic Drift*	< 50 μς
Furnace Heating	Resistance Wound
EGA Furnace	Optiona
Temperature Range	Ambient to 1000 °C
Isothermal Temp Accuracy	+/- 1 °C
Isothermal Temp Precision	+/- 0.1 °C
Controlled Heating Rate	0.1 to 100 °C/min
Furnace Cooling (forced air / N <sub>2</sub> )	1000 to 50 °C <12 mir
Temperature Calibration	Curie Poin
Auto-Loader	Included
Auto Stepwise TGA	Included
TGA/MS Operation	Optiono
TGA/FTIR Operation	Optiono
Platinum™ Software	Included
Sample Pans	Platinum 50, 100 µ
	Ceramic 100, 250, 500 µ
	Aluminum 100 μ

<sup>\*</sup> From 50 to 1,000 °C at 20 °C/min using empty platinum pans, no baseline / blank subtraction.

# Q5000 IR TECHNOLOGY

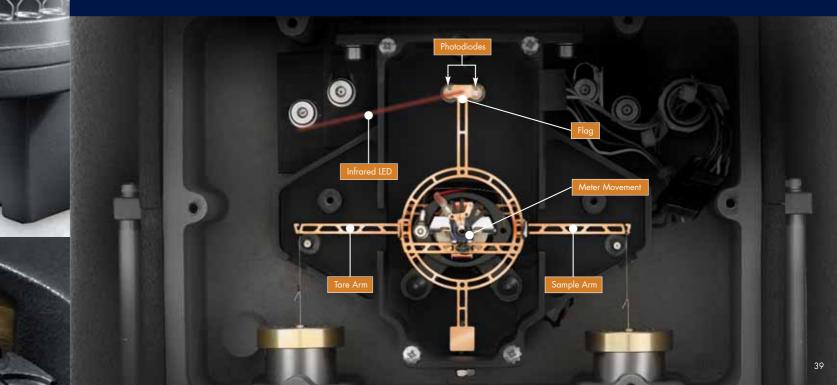
The highly automated Q5000 IR is the clear choice for complex TGA applications, for low-level detection of impurities, kinetic studies, off-gas analysis, and for high heating rate operation. Its design integrates a thermobalance engineered for maximum baseline flatness and high sensitivity, with the 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.

# Autosampler

The integrated Q5000 IR Autosampler features a programmable, 25-position 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. 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 auto-sampler feature is the patented pan-punching mechanism designed to reliably open sealed aluminum pans used to protect atmosphere sensitive samples.

### Thermobalance

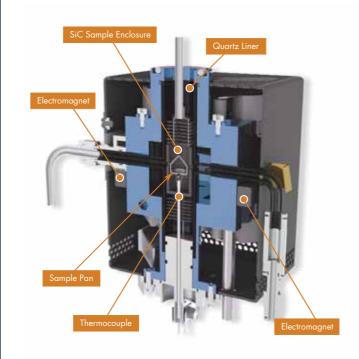
The heart of the Q5000 IR is a high performance thermobalance maintained at a constant 40.00 °C by three symmetrically arranged heaters in a well-insulated, gas-purged chamber. Isolated from the furnace by a water-cooled plate, the sensitive, null-balance design features the latest in precision weighing technology. Design benefits include sensitive, 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.

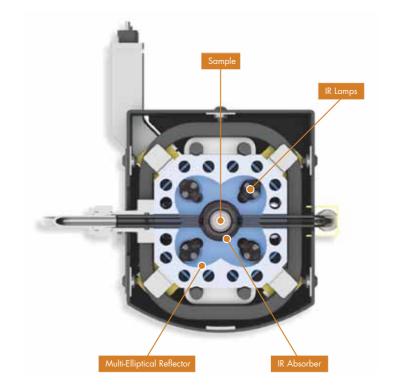


# Q5000 IR TECHNOLOGY

### 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 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 include rapid dynamic response and excellent performance and flexibility in standard, advanced (Hi-Res™ TGA; Modulated TGA™), and high heating rate experiments. 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 IR benefit.





# 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 thermo-balance / 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.

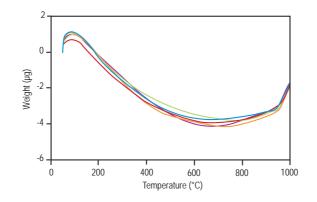
# **PERFORMANCE**

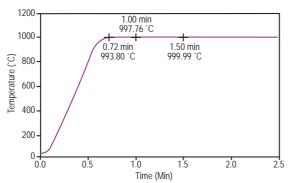
# Dynamic Baseline Stability

This figure shows a set of replicate Q5000 IR empty pan baseline runs from 50 °C to 1000 °C. Baseline drift in all cases was considerably below 10 µg, while maintaining impressive reproducibility. 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.

# Heating Rate Performance

The IR-heating furnace of the Q5000 IR TGA provides the widest range of heating rates available on the market. Ballistic heating from ambient to 1000 °C is accomplished in less than 1 minute, at instantaneous rates of over 2000 °C/min. Linear heating rates up to 500 °C/min are easily achieved, providing better simulation of rapid-heating processes and dramatically improving productivity and sample turnaround.



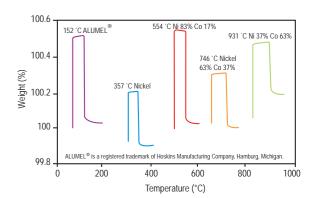


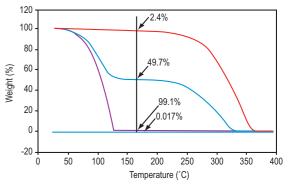
# Curie Point Calibration - Electromagnet

This figure shows an overlay plot of a series of ICTAC certified, NIST traceable Curie Point reference materials used to calibrate a TGA. 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. The figure to the right shows a plot of the decomposition profiles of 2-ethoxyethylacetate at all three concentrations. This data demonstrates the accuracy of the measurements and confirms that there is no interference from the polyol in the determination.





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# Q500 / Q50 TECHNOLOGY

Sensitive, precise, rugged, and automated all describe the 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 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<sup>™</sup> TGA and Modulated TGA<sup>™</sup>. 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.

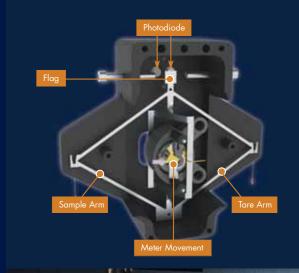


### Thermobalance

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 Q50 / 500 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.

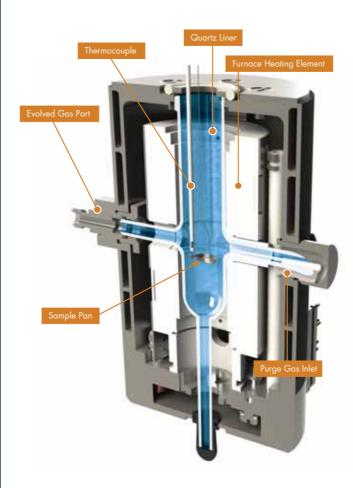




# TGA ACCESSORIES & OPTIONS

# Evolved Gas Analysis (EGA) Furnace

The rugged and reliable EGA is an optional, quartz-lined furnace for the Q500 or Q50. The liner is 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.



# 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<sup>TM</sup> software, which permits pre-programmed analysis, comparison, and presentation of results.



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### **EVOLVED GAS ANALYSIS**

Evolved gas analysis involves the qualitative investigation of the evolved gas products from a TGA experiment. These products are typically the result of decomposition, but can also evolve from desorption, evaporation or chemical reactions. Evolved gas analysis is typically performed by interfacing a quadrapole 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, quadrupole mass spectrometer with a heated capillary interface, and TGA module-specific interface kits for its Q5000 IR, 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 Q5000IR 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.



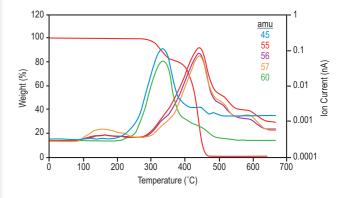
# **EGA APPLICATIONS**

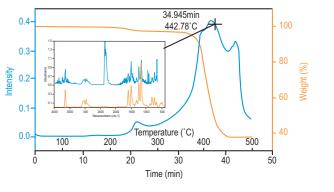
### TGA-MS: Polymer Analysis

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.





# **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 Q5000 IR 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 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.

\*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. 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 Nos. 6.113.261 and 6.336.741







### Q5000 IR

Platinum (50 and 100  $\mu$ L), high temperature platinum-HT (100  $\mu$ L), ceramic (100 and 250  $\mu$ L) and aluminum open (80  $\mu$ L) and sealed (20  $\mu$ L) 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 bail) are recommended. The larger pan is best for higher volume / low density samples such as foams. 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.

### Q500 / Q50

Platinum (50 and 100  $\mu$ L), and new style ceramic (100, 250, and 500  $\mu$ L) 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  $\mu$ L) pans are cost-effective substitute pans but cannot be used above 600 °C.

# **APPLICATIONS**

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:

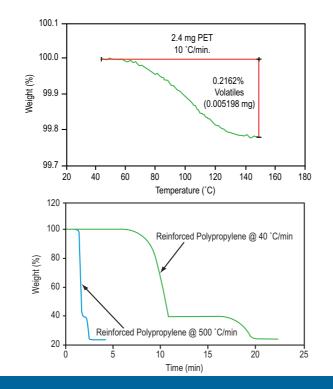
• Thermal stability • Decomposition kinetics • Composition • Estimated lifetime • Oxidative stability • Moisture and volatile contents

# 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. The volatiles analysis for a small (2.4 mg) sample of polyethylene terephthalate (PET) bottle stock is shown here. The 0.2% weight change reflects an absolute weight loss of only 5.2 micrograms!

# **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. This figure 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.

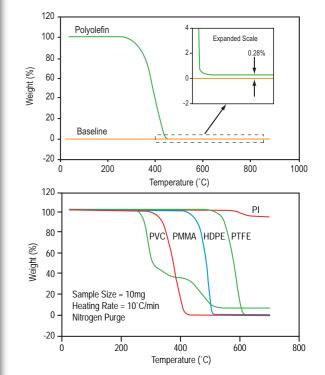


### Accurate Residuals

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. This figure 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!

# Thermal Stability

TGA is often used to determine sample thermal stability and to reveal weight-loss decomposition profiles. The figure to the right 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.



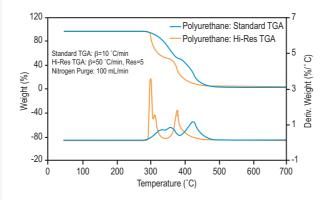
# **APPLICATIONS**

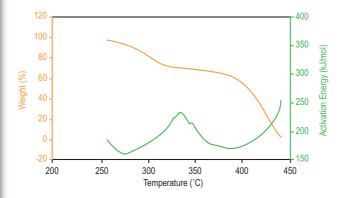
# High Resolution™ TGA

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 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.

### Modulated TGA™

The Figure to the right 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.



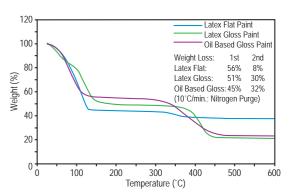


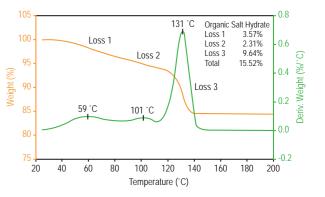
# Compositional Analysis

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 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.

# 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.





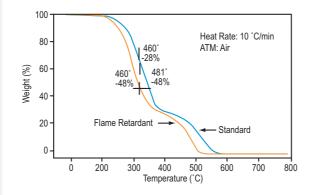
# **APPLICATIONS**

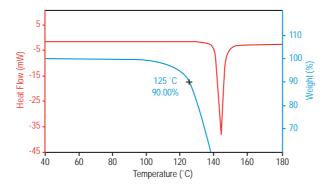
### Effect of Additives

This figure 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 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.



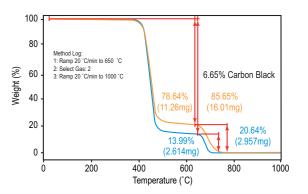


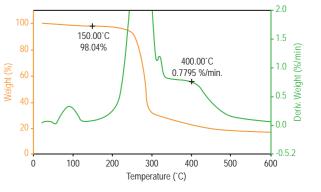
### Quantification of Filler Content

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  $10^{\circ}\text{C/min}$ . The data show a small (~2%) weight loss below  $150^{\circ}\text{C}$ , which is typical for adsorbed water. The material is relatively stable up to  $200^{\circ}\text{C}$ , after which a large, multi-step weight loss is indicative of thermal decomposition.





# TGA-HP

The TA Instruments TGA-HP Series instruments are specialty gravimetric analyzers designed to provide unique capabilities for High-Pressure, Ultra-High Vacuum, and High-Temperature under static or dynamic reactive atmospheres.

The TGA-HP50 is our standard system, employing a high-sensitivity balance in a robust design. The TGA-HP50 can accommodate samples up to 5 g in mass, with a sensitivity of 0.5 microgram. The TGA-HP50 is the instrument of choice for high-pressure studies (up to 50 Bar) at measurement temperatures up to 800°C, and can accommodate a variety of gas compositions under high-pressure static or dynamic flow. Our optional Ultra High Vacuum (UHV) accessory can provide atmospheres down to 1 x 10° torr.

When higher pressures or corrosive gas atmospheres are required, the TGA-HP150 is recommended. This top-of-the line model provides static pressures up to 150 Bar and utilizes an advanced Rubotherm Magnetic Suspension balance with a 25 g capacity. This allows the reaction chamber to be completely sealed, allowing for aggressive gas chemistry while isolating the microbalance assembly. The TGA-HP150 can be operated up to 750°C at the maximum pressure. The optional UHV accessory is also available for studies under atmospheres down to 1 x 10° torr.

The TGA-HPs is a specialized instrument designed for high temperature and pressure combinations. The maximum temperature of 1200°C is achieved safely in a unique double-wall reactor. The TGA-HPs is equipped with a steam generator which makes it ideal for coal gasification studies and includes 5 gas inlets for the maximum flexibility in dynamic reactive atmospheres.

All TGA-HP models can be interfaced for evolved gas analysis as described on page 48.



### TGA-HP **SPECIFICATIONS**

	TGA-HP50	TGA-HP150	TGA-HPs
Balance	Standard Balance	Rubotherm Magnetic Suspension	Rubotherm Magnetic Suspension
Balance Capacity	5 g	25/10g	25/10g
Dynamic Range	500 mg	25/10g	25/10g
Weighing Accuracy	+/- 0.1%	± 0.002%	± 0.002%
Weighing precision	+/- 0.01%	±30/± 4µg	±30/± 4µg
Sensitivity	0.5 µg	10/1 µg	10/1 µg
Reactor Type	Single Block Inconel 625®	Single Block René 41®	Double-Wall Alumina/Inconel 625
Upper Temperature Limit			
(Ambient Pressure)	1000 °C	1000 °C	1200 °C
(Full Pressure)	800 °C	750 °C	1200 °C
Pressure Limit	50 bar (725 PSI)	150 bar (2175 PSI)	50 bar (725 PSI)
Gas Inlets	3	3	5
Dynamic Flow-Dosing	Available	Available	Standard
Steam Generator	N/A	N/A	Standard
Optional High Vacuum	1 x 10 <sup>-6</sup> torr	1 x 10 <sup>6</sup> torr	N/A
Corrosive Gas Compatibility	N/A	Recommended	Recommended

Inconel 625 is a registered trademark of Special Metals Corporation. René 41 is a registered trademark of Teledyne Industries, Inc.

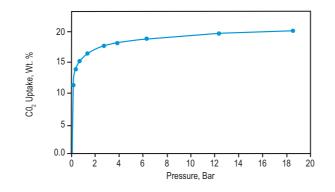
# TGA-HP APPLICATIONS

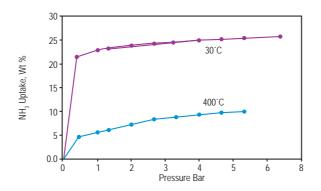
# Carbon Dioxide Sequestration

Zeolite and other inorganic molecular sieve membranes have shown potential for gas sequestration based on molecular size and shape because of their small pore size, typically less than 1 nm, and their narrow pore size distribution. The size of these channels controls the size of the molecules or ions and therefore a zeolite like chabazite can act as a chemical sieve, allowing some ions to pass through while blocking others. This figure contains the TGA-HP data for the adsorption of CO<sub>2</sub> into raw chabazite at 60 °C and at pressures up to 20 Bar. Note how the quantitative gravimetric sorption is easily determined using the TGA-HP technology.

# Adsorption of NH<sub>3</sub> on Zeolite Catalyst

The data in this figure shows the TGA-HP data of the adsorption of ammonia gas onto a zeolite matrix at two discreet temperatures, 30 °C and 400 °C, at pressures up to 7 Bar (ca. 100 psi). The TGA-HP is used to measure the quantitative capacity of the zeolite to adsorb the ammonia, thus allowing for the effective design of the exhaust stream catalyst technology.



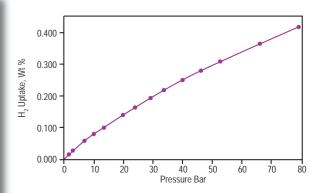


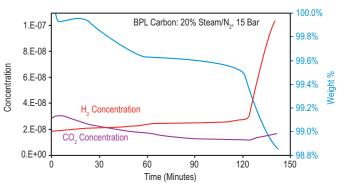
# Adsorption of H<sub>2</sub> on Carbon

Recent research has focused on the development of transportable reversible systems for hydrogen storage with a high capacity. These systems are critical to the large-scale application of hydrogen fuel cells, in particular for mobile applications such as automotive use. A higher-energy efficiency is attainable with systems in which hydrogen is concentrated by physical adsorption onto a matrix material above 70 K. The TGA-HP is the ideal tool for measuring the adsorbent efficiency for hydrogen sorption. This data shows the adsorption of H<sub>2</sub> gas onto an activated carbon matrix at 25 °C (273 K). Note how the adsorption is quantified over the wide pressure range from sub-atmospheric to nearly 80 Bar (ca. 1150 PSI).

### Steam Gasification of Carbon

Gasification refers to the process of conversion of carbonaceous materials into gas, typically under conditions of high pressure and temperature, and usually in the presence of steam. There is wide interest in gasification because it produces lower quantities of air pollutants and the resulting combustible products burn cleaner than the original material. This figure shows the result of the gasification of carbon performed in the TGA-HPs, at 800 °C under a 15 bar atmosphere of 20% steam / N<sub>2</sub>. The weight signal clearly shows the onset of gasification at about 120 minutes. Simultaneous mass spectral analysis of the offgas detects a high production rate of H<sub>2</sub>, the primary product of interest, along with a relatively low concentration of the less desirable CO<sub>2</sub>.







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