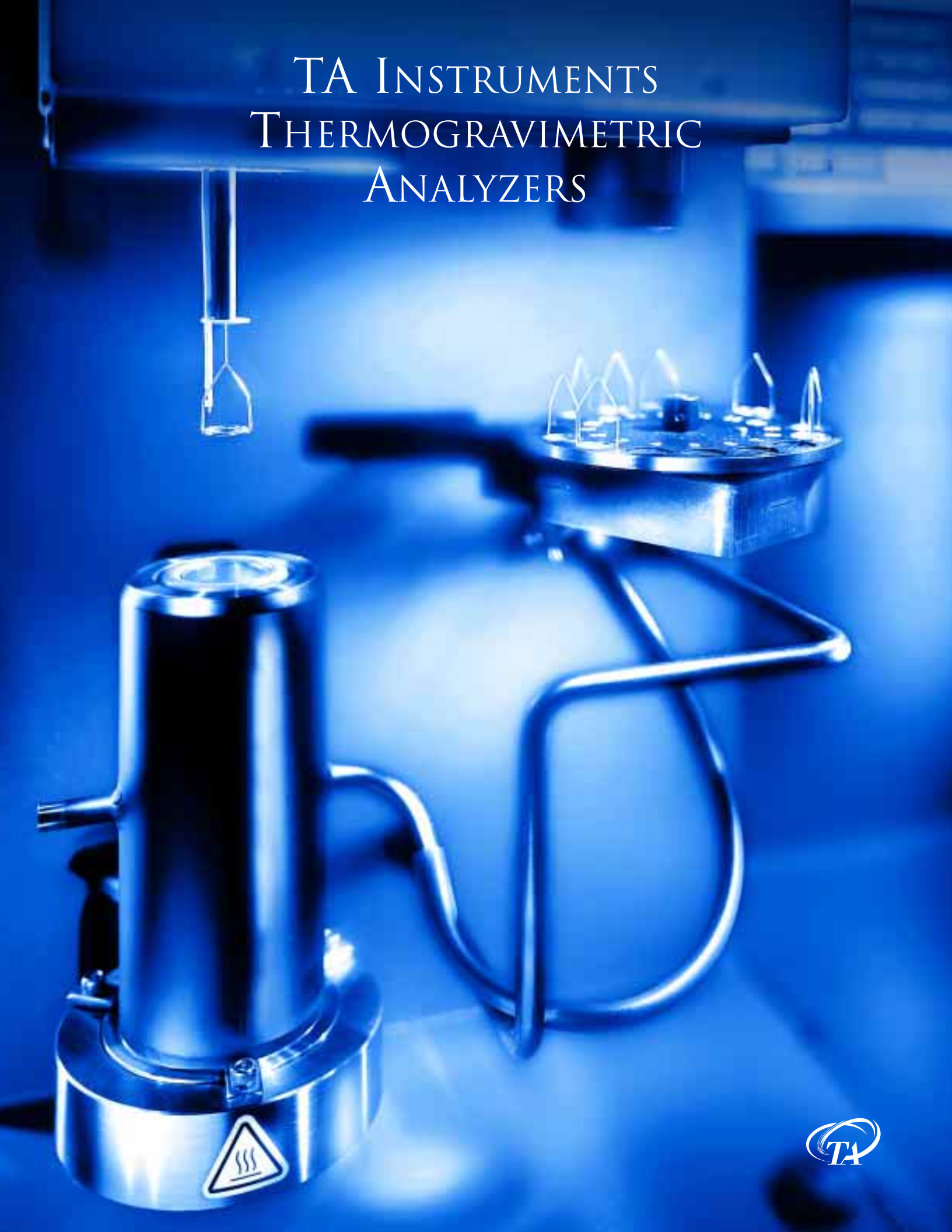
TA Instruments Thermogravimetric Analyzers



TA Instruments Q Series™ Thermogravimetric Analyzers

Sensitive, Precise, Rugged and Automated are words that describe a TA Instruments Thermogravimetric Analyzer. The Q500 and Q50 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.

The Q500 is our top-of-the-line research-grade thermogravimetric analyzer. Its efficient low mass furnace, ultra-reliable thermobalance, unique purge gas system (with mass flow control), and advanced automation provide for superior TGA performance. The Q500 is an expandable system that is well equipped to handle TGA applications, from the routine to the most demanding.

The Q50 is a cost-effective, easy-to-use, general-purpose thermogravimetric analyzer with many of the basic features of the Q500. It offers performance superior to most research grade models. The Q50 is ideal for laboratories that need a high quality TGA for standard applications.

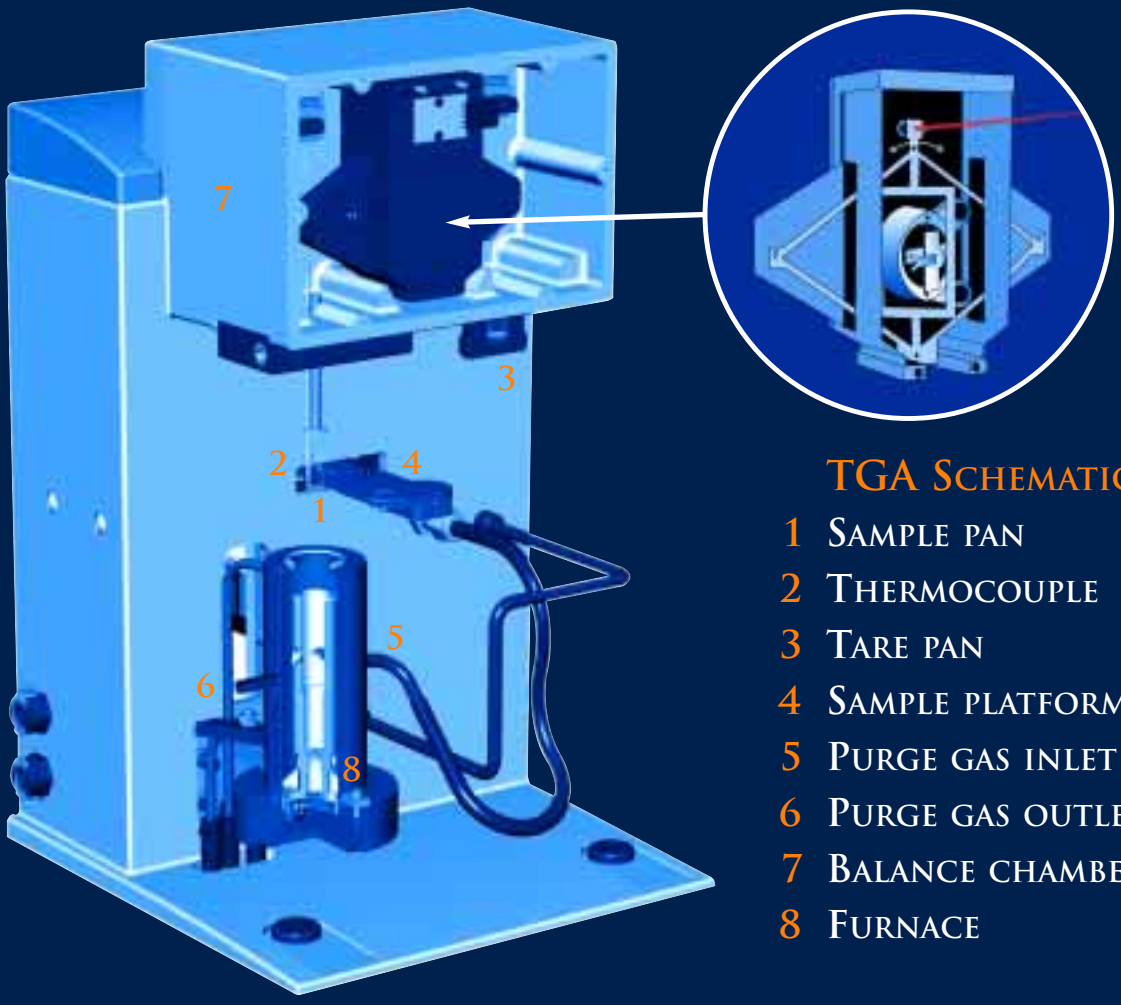
Technical Specifications

	TGA Q500	TGA Q50
Furnace - Low Mass	Standard	Standard
Furnace - EGA	Optional	Optional
Touch-Screen Display	Standard	Not Available
Mass Flow Controller with automatic gas switching	Standard	Optional
Autosampler	Optional	Not Available
Hi-Res TGA™	Optional	Not Available
Modulated TGA™	Optional	Not Available
TGA / MS operation	Optional	Optional
Temperature Range	Ambient to 1000°C	Ambient to 1000°C
Isothermal Temperature Accuracy	+/- 1°C	+/- 1°C
Isothermal Temperature Precision	+/- 0.1°C	+/- 0.1°C
Weighing Capacity	1.0 gm	1.0 gm
Sensitivity	0.1µg	0.1µg
Weighing Precision	+/- 0.01%	+/- 0.01%
Temperature Calibration	Curie Point / metal stds (1 to 5 points)	
Heating Rate - Low Mass	0.1 to 100°C / min in 0.01°C / min increments	
Heating Rate - EGA	0.1 to 50°C / min in 0.01°C / min increments	
Furnace Cooling	Forced air 1000°C to 50°C <12 min	
Sample Pans	Platinum: 50 µL, 100 µL Ceramic: 100 µL, 250 µL, 500 µL Aluminum: 100 µL	

Instrument Design Features and Benefits

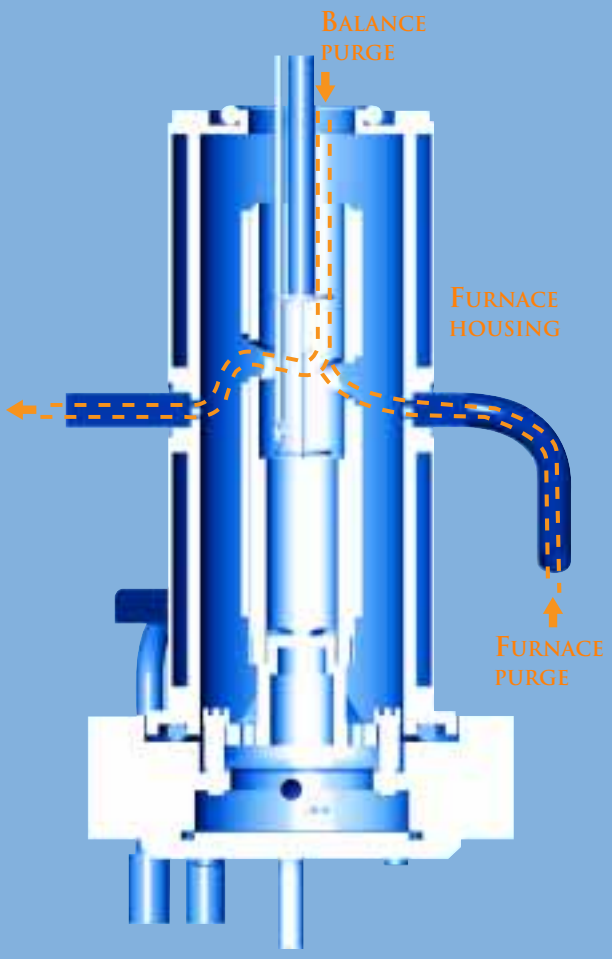
A TGA is only as good as its ability to accurately and reproducibly measure mass and temperature changes. Experience is a vital part of TGA design, and the knowledge gained by our engineers in thirty five years of integrating customized thermobalances, furnaces and purge gas systems to produce thermogravimetric analyzers, has resulted in the unparalled performance and reliability of the Q Series™.

• ***Thermobalance*** The heart of a Q Series™ TGA is the accurate and ultra-reliable vertical thermobalance, which is housed in a temperature controlled environment. It operates under the field proven null-balance principle, where an optically active servo loop maintains the balance arm in the horizontal reference (null) position by regulation of current flowing through a transducer coil. An infrared LED source and a pair of matched photodiodes detect beam movement, while a flag attached to the balance arm controls the amount of light reaching each photodiode. As sample weight changes, the beam becomes unbalanced, and the net photodiode output is fed to a control program, which electrically "nulls" the balance. The current required is directly proportional to the sample weight change. The design provides for automatic switching between the dual weight ranges of 0 to 200 milligrams and 0 to 1 gram. ***Benefits:*** High accuracy and precision in detection of minute weight changes from ambient to 1000°C, low baseline drift, plus smooth, continuous, reliable operation over the entire weight range.



TGA Schematic

1 Sample pan
2 Thermocouple
3 Tare pan
4 Sample platform
5 Purge gas inlet
6 Purge gas outlet
7 Balance chamber
8 Furnace



•***Furnace*** Our custom designed furnace is the second key element of a Q Series™ TGA. It features low mass, rugged heater windings, and proprietary heater control technology. ***Benefits:*** Rapid, accurate and precise temperature programming over a wide range, plus optimized use of advanced techniques such as Hi-Res™ TGA and Modulated TGA™. Our reliable, long-life furnaces also increase the value of your investment

•***Purge Gas System*** A distinguishing feature of a Q Series™ TGA is the successful integration of a sensitive vertical thermobalance, a high performance furnace and a unique horizontal purge gas system. In this design, purge gas enters the furnace through a side arm and flows directly across the sample located in an open pan suspended from the vertical balance. A low volume of inert purge gas is also directed through the balance chamber to prevent any back diffusion of the primary purge gas (if different) or decomposition products into the balance chamber. The combined gases leave the sample chamber by a second side arm that can be readily connected to a second identification instrument (e.g. MS or FTIR). ***Benefits***: This design minimizes buoyancy effects and has been proven superior to other vertical purge gas designs in sweeping away decomposition products from the sample area. The metering of purge gas to the furnace and balance housing is accurately and precisely controlled by the mass flow controllers, which are a standard feature on the Q500, and optionally available for the Q50 (see Accessory section for further details).

•***Temperature Control and Measurement*** Our unique, custom designed system features a single control / sample thermocouple positioned immediately adjacent to the sample. The value of this innovative design is enhanced by a second thermocouple located in the same sleeve but slightly above the principal one. ***Benefits:*** 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 functions as a furnace protection mechanism. The controller monitors the temperature difference between both thermocouples, and if it exceeds a set point the furnace is automatically shutdown.

Q500 Accessories & Options

Autosampler

The Q500 Autosampler accessory is a programmable, multi-position sample carousel that allows overnight or "round-the-clock" automated analyses, in a random or sequential fashion, of up to 16 samples. All aspects of sample testing are totally automated and controlled by the Q500 software, including pan taring, pan loading, sample weighing, furnace movement, pan unloading and furnace cooling.

The autosampler has the flexibility to satisfy the needs of both analytical and quality control laboratories. Maximum productivity from the Q500 Autosampler is achieved when paired with the intelligent Thermal Advantage Autoanalysis software, which permits pre-programmed analysis, comparison and presentation of results.

Mass Flow Controller (with automatic gas switching)

The mass flow controllers supplied with the Q500, and available for the Q50, permit accurate, reproducible purge gas metering to the TGA sample and balance chambers. The benefit is consistently superior data quality over conventional flow control devices. The automatic gas switching capability employs low volume, high-speed valves to provide instantaneous changeover of purge gases as programmed in the test method. This is especially useful when rapid changes from inert to oxidizing atmospheres are required. Purge gas flow rates are stored as a signal in the data file.

Q500 / Q50 Sample Pans

TA Instruments offers a series of sample analysis pans in various sizes and in platinum, aluminum or ceramic. In general, platinum is preferred because of its inertness and post-experiment ease of cleaning. Ceramic pans are used for samples that react with platinum, and where larger capacity is required. Aluminum pans are a low cost alternative for applications below 600°C.

High Resolution TGA™ (Hi-Res™ TGA)

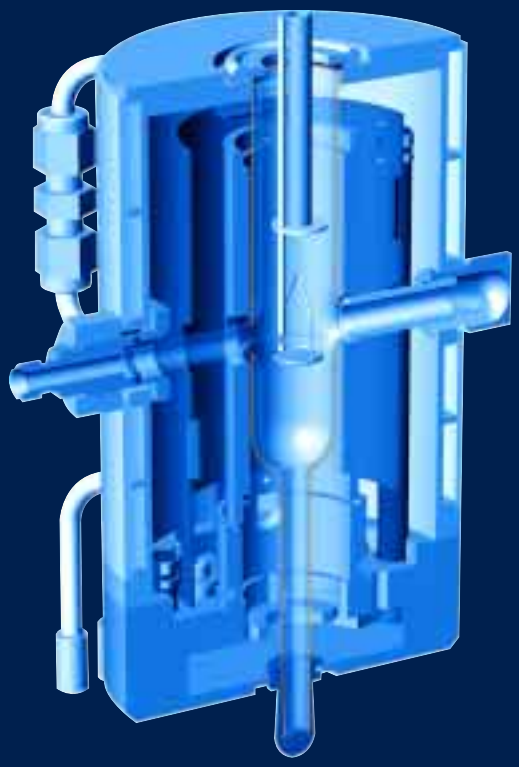
Hi-Res™ TGA is a patented TA Instruments furnace control technology that improves the ability to separate closely occurring decomposition events. The intelligent design of the Q500 makes it ideally suited for such experiments, where it is critical to precisely and rapidly control sample temperature and monitor small weight changes. This Q500 option includes three control algorithms (constant reaction rate, stepwise isothermal, and dynamic rate) that consistently improve weight change resolution over results from linear heating rate TGA. Each technique has specific advantages, but the dynamic method is the most generally applicable since it is the simplest to use, minimizes the need for operator expertise, and generates comparable results in a fraction of the time required for the other methods.

Modulated TGA™

Modulated TGA (MTGA) is another TA Instruments innovation for the study of material decomposition by TGA. It produces continuous, model-free kinetic data, equivalent in quality to that from the traditional TGA decomposition method (ASTM Standard E1640), but in a fraction of the time. Desired parameters, such as activation energy and the pre-exponential factor can be calculated on a continuous basis, and studied as a function of time, temperature and conversion. MTGA is easy-to-use, and provides the kinetic information needed to follow and improve productivity in many industrial processes. This Q500 accessory takes advantage of the proprietary heater control technology developed for Hi-Res™ TGA and also for the widely accepted Modulated DSC® (MDSC®) technique - another first from TA Instruments.

Evolved Gas Analysis (EGA) Furnace

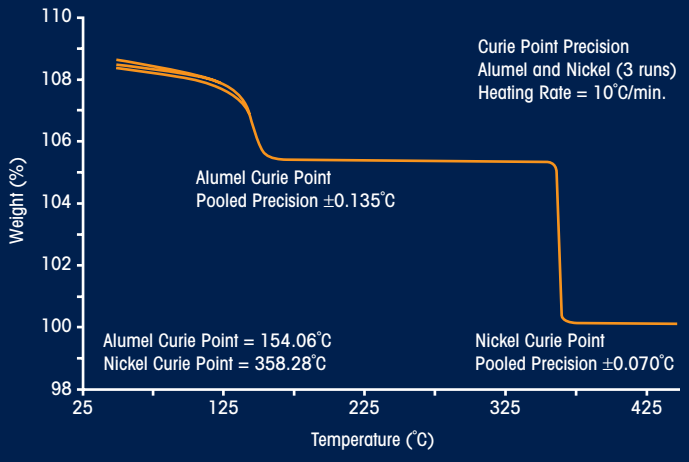
The EGA is a larger optional furnace for a Q Series™ TGA that uses a similar rugged design but incorporates a quartz liner between the heating element and the sample measurement area. This arrangement results in a small internal volume, which further reduces the possibility for back diffusion of sample off-gases into the balance chamber by assuring rapid transfer of the gases out of the furnace exit port. The quartz liner provides an essentially inert surface to sample decomposition products. This furnace is ideal for coupled use with a MS or FTIR, instruments that can uniquely identify the decomposition products from the TGA analysis. The results from these coupled techniques are qualitative and quantitative information on the temperature stability of the sample, its composition, method of decomposition, and identification of the products evolved.



Calibration Standards

The Thermal Advantage software supplied with the Q Series™ TGA supports the use, for temperature calibration purposes, of either Curie Point or melting point standards (1 to 5 materials). User-friendly software "wizards" simplify the process, which follows the ASTM Temperature Standard (E1582) for TGA calibration. Each Q Series™ TGA is supplied with temperature calibration materials in the accessory kit. However, traceable calibration standards are also available for those customers, whose experimental protocols necessitate their use.

The TGA Curie Point method records for each standard, an apparent sharp weight change at a well defined temperature, which corresponds to a known transformation in the standard's ferromagnetic properties at that temperature. This technique is also termed thermomagnetometry. This Figure shows the relative temperature precision obtained from three replicate calibration runs using alumel and nickel Curie Point standards.



Thermal Advantage Software

A quality Thermal Analyzer requires flexible, intelligent software to power it. No one believes this more than our software engineers, who have pioneered most of the features commonly seen in today's modern thermal analyzers. 32-bit Thermal Advantage™ for Q Series software is Microsoft Windows™ based, and expandable to meet growing customer needs.

Thermal Advantage – Instrument Control

- ***Multitasking*** – conduct experiment and simultaneously analyze data
- ***Multimodule*** – can operate up to 8 modules simultaneously
- ***Wizards*** – guides and prompts in setting up experiments
- ***Preference Choices*** – customize the "look and feel" of the experiment
- ***Flexible Method Editor*** – design & save the correct method for your material
- ***Real-time plot*** – provides a real-time picture of the progress of the experiment
- ***Autoqueing*** – permits pre-programmed set-up of future experiments
- ***"How To" feature*** – provides extensive, context sensitive operator assistance

Universal Analysis 2000 – Data Analysis

- ***Single Package*** – analyzes data from all TA Instruments thermal modules
- ***Picture-in-a-picture*** – provides easy one plot analysis of large and small events
- ***Real-Time data analysis*** – ability to analyze data "as it arrives"
- ***Interchangeable graphics / spreadsheet view of data***
- ***Custom report generation*** – within UA 2000 using Microsoft Word™ & Excel™ templates
- ***Autoanalysis*** – for pre-programmed analysis, comparison & presentation of results
- ***Saved Analyses*** – for quick retrieval of previously analyzed data files
- ***Library of Specialty Data Analysis*** – specialty analysis programs (e.g., TGA Decomposition Kinetics Software, ASTM D1640).

Applications

Thermogravimetric Analysis (TGA) 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 commonly used in academic, industrial and government research, as well as in quality control laboratories. TGA is particularly useful for the following material characterization determinations:

- Composition of multi-component materials
- Thermal stability of materials
- Oxidative stability of materials
- Decomposition kinetics of materials
- Estimated lifetime of materials
- Moisture and volatile contents of materials

Thermal Stability

A main use of TGA is to determine the thermal stability of materials and to reveal their weight loss decomposition profiles. ***Figure 1*** shows a series of thermal curves for some common polymers (PVC, PMMA, HDPE, PTFE) plus a highly filled polyimide. The information allows design engineers to select materials suitable for end uses where heat stability at specified temperatures is required.

FIGURE 1

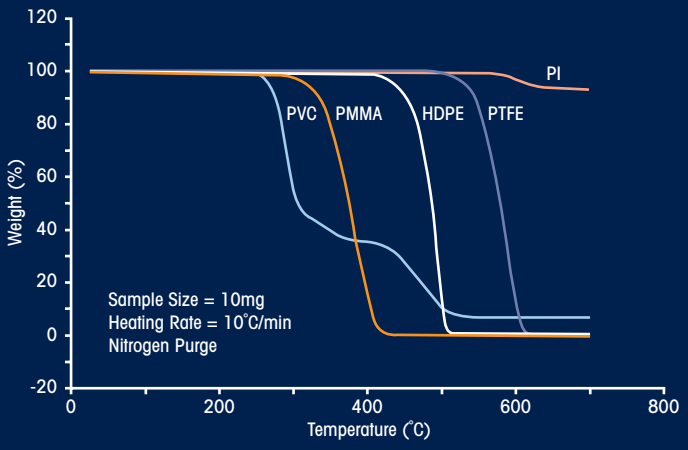
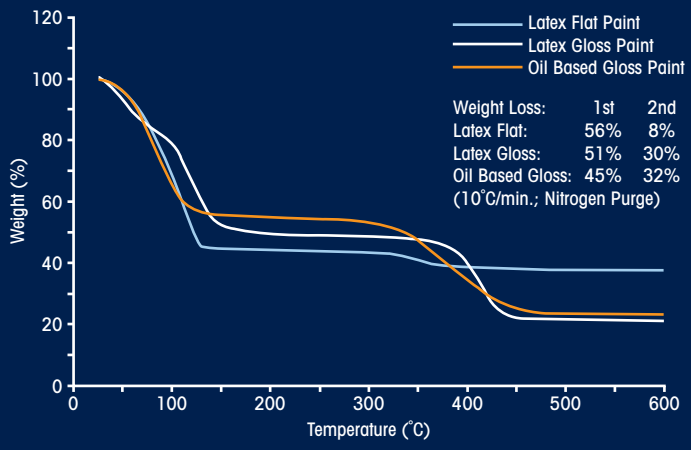


FIGURE 2



Composition Analysis

The other main use of TGA is to determine the composition of a material by measuring the weight (mg or wt %) of each component as it decomposes in response to the experimental conditions of temperature, time and atmosphere. ***Figure 2*** shows the quantitative decomposition profile of three paints. Differences in the type, amount, and the decomposition mechanism of the main copolymers used are readily observed. Examination of the low temperature region (<150°C) would reveal further information on the amount and possible nature of the carrier solvent (aqueous or oil) used in each paint.

High Resolution TGA

Figure 3 compares the results from conventional and Hi-Res™ TGA in the analysis of a polyurethane. In both analyses, the sample is stable up to almost 300°C, where it undergoes a multi-step decomposition process. The superior resolution of the Hi-Res™ TGA results are apparent, especially in the derivative signal. The Hi-Res™ method also reached the decomposition temperature in one fifth of the time taken by the standard method.

FIGURE 3

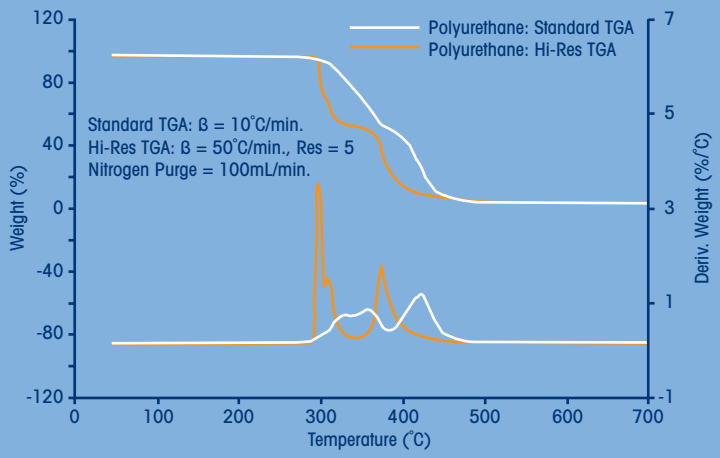
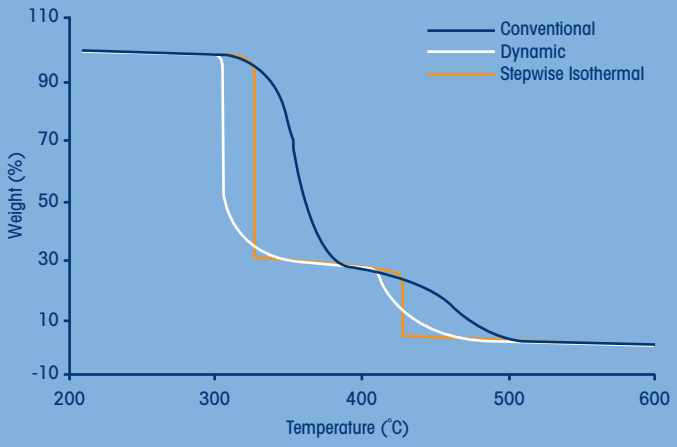


FIGURE 4



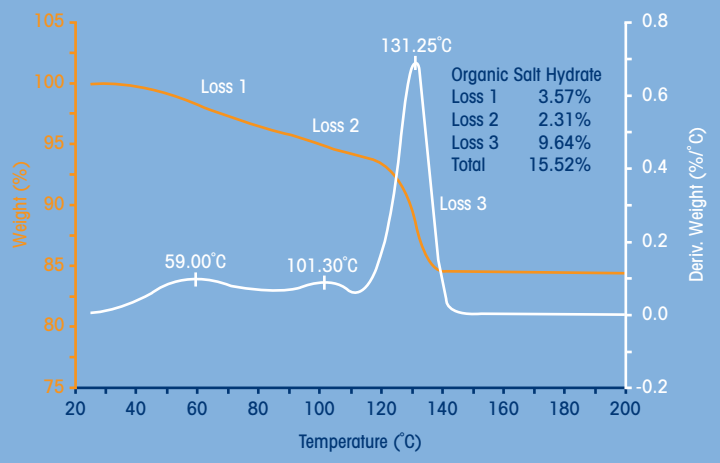
High Resolution TGA

Similar results are shown in ***Figure 4***, which compares the analysis of a polyvinyl acetate polymer by conventional TGA, the stepwise isothermal and dynamic Hi-Res™ techniques. The superiority in resolution of the latter two is clearly evident. While the stepwise isothermal method produces the sharpest weight losses, the dynamic method shows comparable resolution but in a shorter experiment time.

Moisture and Volatiles Analysis

Determination of adsorbed, bound or included moisture, as well as organic volatiles, is an important TGA application in a wide variety of industries. Product performance is often dependent on this parameter. Environmental concerns are increasingly important in the case of volatile organic compounds (VOC's). TGA analysis of an organic salt hydrate in a nitrogen atmosphere ***(Figure 5)***, shows a bound water content of 9.6%, with two lower temperature weight losses of 3.6% and 2.3 % respectively. These lower temperature losses are likely due to moisture either at the surface of the salt or held to it by weak attractive forces.

FIGURE 5



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