



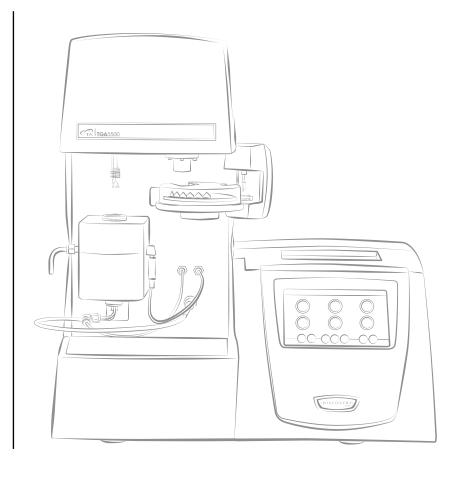
The WORLD'S FINEST line of THERMOGRAVIMETRIC ANALYZERS

# HIGH PERFORMANCE TGA SYSTEMS that deliver the

**Most Accuracy** 

**Highest Sensitivity** 

**Greatest Reliability** 



#### Thermogravimetric Analysis

TA Instruments invites you to experience the world's finest line of Thermogravimetric Analyzers, the Discovery TGA 55, TGA 550, and TGA 5500. Discover the advanced engineering and attention to detail that provides enhancements in every aspect of TGA technology and a new level of user experience. From the most cost-effective and flexible TGA with industry-leading performance, to the most advanced TGA available, there is a Discovery TGA to meet your needs and exceed your expectations.

**TGA** 55

Premium Performing TGA



The TGA 55 is specifically designed for those who want a rugged, reliable, and cost-effective TGA, and are not willing to compromise on performance. Utilizing TA's proprietary Tru-Mass™ Balance as the core of the measurement, the TGA 55 will outperform competitive research-grade models. Its sensitivity, accuracy, and ease-of-use make this TGA an ideal instrument for basic research in academic or industrial labs that need quality results.

TGA 550

Premium performance with advanced options and configuration flexibility



The TGA 550 will not only outperform competitive top-tier systems, but will also give users the flexibility to add advanced features like Hi-Res™ TGA, MTGA™, DTA signal, and our new 25-position autosampler. The performance, flexibility, and ease-of-use make this an excellent TGA for research and multi-user laboratories where a wide variety of TGA experiments are conducted and future expansion of analytical work is anticipated.

#### **TGA** 5500

#### Ultimate performance with every option to meet the requirements of the most demanding applications

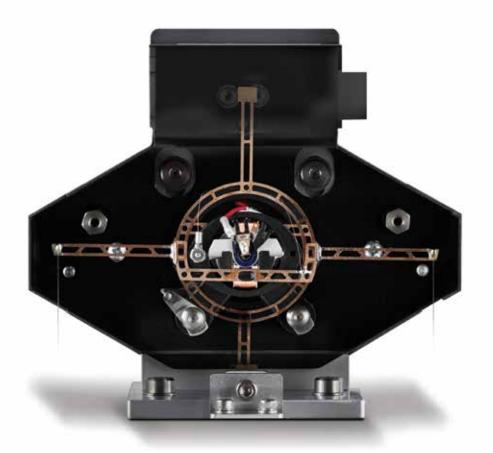
The TGA 5500 is designed for the researcher who requires the highest level of performance and features in one package. Built to maximize temperature control and minimize signal drift, the TGA 5500 has less drift than any competitive TGA – even those using post-test data manipulation! The TA patented IR furnace\* delivers the fastest heating and cooling rates available. The all-new 25-position autosampler takes productivity to the highest level, while also featuring the pan punch mechanism for automated sequencing of materials where the sample environment must be controlled prior to testing.



\* U.S. Patent no. 7,416,328 and 7,566,167



## No one makes a MORE SENSITIVE and ACCURATE THERMOBALANCE



At the core of every new Discovery TGA is the proprietary Tru-Mass™ Balance. The Tru-Mass Balance system is Thermally isolated for high sensitivity in every laboratory environment, delivers the highest Resolution to separate components of the most challenging TGA samples, and has Ultra-low drift (Tru-Mass) for weight accuracy. Unlike competitive designs, the Discovery TGA delivers optimum performance without requiring baseline subtractions and other post-test manipulation required by competitors. The result is an innovative TGA with unrivaled performance in weight drift and sensitivity.

#### **Balance Features and Benefits:**

- Ultra-low drift balance design ensures accurate detection of even the smallest weight changes
- · High capacity (1 g) Tru-Mass balance with auto-ranging capability to ensure the best sensitivity no matter the size of the sample
- Free-hanging sample eliminates the heat sink prevalent in top-loading designs, for the most efficient heat transfer and gas flow around the sample
- Thermally isolated balance with low drift and high sensitivity to deliver the most accurate real-time data

The proprietary Tru-Mass™ balance delivers pure real-time weight data.

Low DRIFT
High CAPACITY
Most ACCURATE DATA

## WIDEST RANGE of HEATING & COOLING RATES



every furnace on EVERY system is designed and manufactured by TA specifically for high performance TGA measurements. From the economical high-performing wire wound and EGA furnaces to the patented IR furnace\* with industry-leading heating rates, there is a TGA furnace to meet your needs.

#### IR Furnace

The TGA 5500 is the only system offering patented infrared heating technology.

- Ambient to 1200°C
- Linear controlled heating rates of 0.1 to 500°C/min
- Ballistic heating rates >1500°C/min for the highest efficiency available
- Fastest cooling for improved sample throughput
- Low volume, vacuum tight, and quartz lined with heated outlet option for best evolved gas results
- Quartz liner makes furnace easy to clean
- Integrated electromagnet for automated verification and calibration using Curie point standards



Wire Wound (Pt/Rh) Furnace

#### Standard furnace for the TGA 55 and TGA 550.

- Ambient to 1000°C
- Linear controlled heating rates of 0.1 to 100°C/min
- Ballistic heating rates >600°C/min
- Low mass furnace allows fast cooling for quick and efficient turnaround between runs



**EGA Furnace** 

Optional Evolved Gas Analysis (EGA) furnace for the TGA 55 and TGA 550.

- Ambient to 1000°C
- Linear controlled heating rates of 0.1 to 50°C/min
- Low volume, vacuum tight, and quartz lined for excellent evolved gas results
- Quartz liner makes furnace easy to clean

All TA furnaces are built to be rugged and reliable and are covered by the industry's ONLY 5-YEAR WARRANTY

#### BEST SAMPLE-ATMOSPHERE INTERACTION

All Discovery TGA models are designed with superior atmosphere control to meet the most demanding applications. Whether maintaining an inert atmosphere, switching to an oxidative purge, or maintaining a high vacuum, the Discovery TGA is up to the task.

#### Atmosphere Control Features and Benefits:

- Innovative Gas-Delivery Manifold (GDM) design eliminates potential leak points from tubing and hardware connections ensuring the most consistent, repeatable atmosphere
- Integrated software-controlled gas switching for experiments requiring dynamic or reactive atmospheres
- An optional Blending Gas Delivery Module features in-line mixing of binary gases and advanced atmosphere control where the concentration ratio of the gases may be held constant, incremented, or ramped
- Horizontal gas purge for optimal sample-atmosphere interaction
- Vacuum tight to ensure inert, oxygen-free atmospheres
- Sealed pan option to maintain the atmosphere of the sample until the experiment starts



The Discovery TGA features a 25-position autosampler designed to be the most rugged and reliable system ever developed.

#### **Autosampler Features and Benefits:**

- Compatible with all pan types and sizes for ultimate flexibility
- Sealed pan\* and pan punch option for effective isolation of air-sensitive or volatile samples
- Scheduling of unattended calibrations and verifications give scientists more time for research
- Integrated electromagnet allows for unattended Curie point calibrations<sup>1</sup>
- New TRIOS software makes it easier than ever to manage and run a large and diverse sample queue. The Design view and Running queue allow for quick and efficient autosampler programming

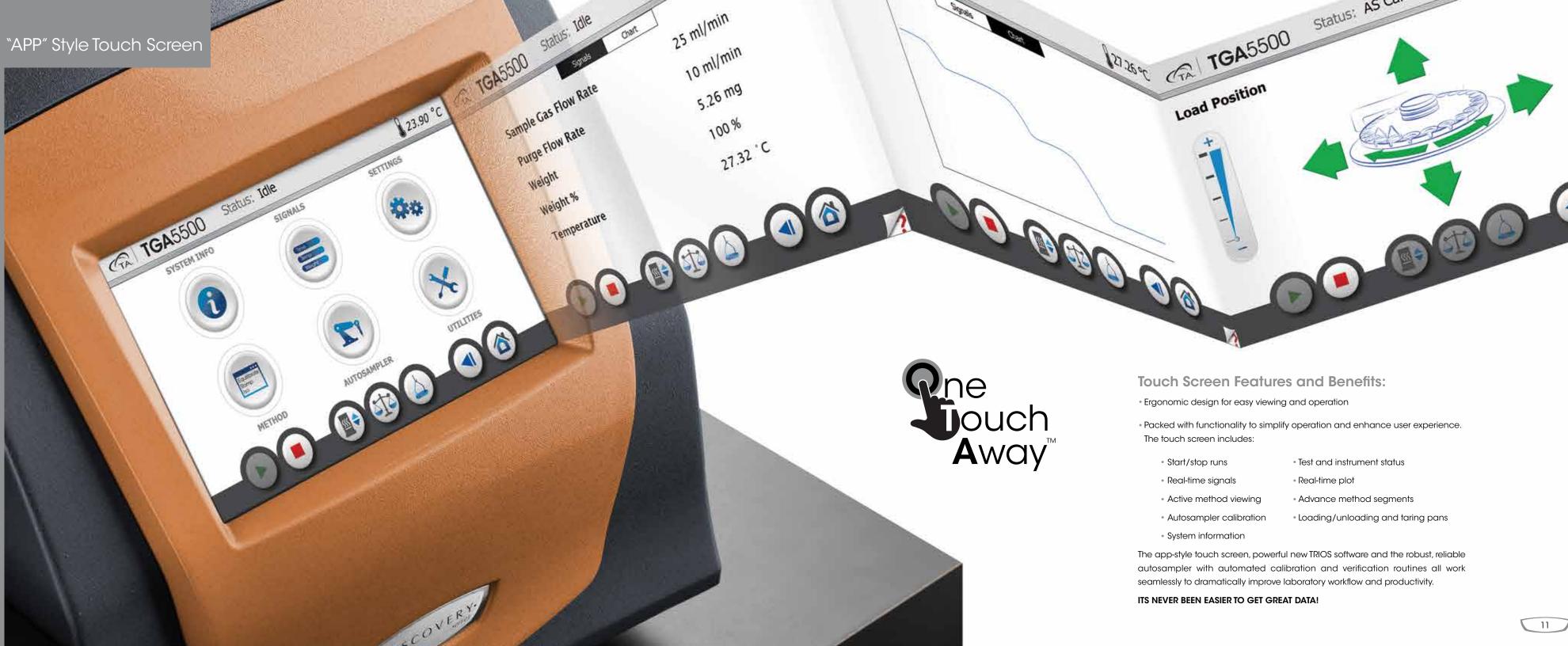


<sup>1</sup> TGA 5500 only

\* U.S. Patent no. 6,840,668

### FLEXIBLE DESIGN for ENHANCED PRODUCTIVITY





#### TRIOS Software

Discover powerful TRIOS software that delivers exceptional user experience in a combined package for instrument control, data analysis, and reporting for thermal analysis and rheology. New features such as multiple calibration sets, real-time test method editing, and inter-laboratory data and test method sharing provide unmatched flexibility, while one-click analysis and custom reporting raise productivity to new levels.



#### **TRIOS Features:**

- Control multiple instruments with a single PC and software package
- Overlay and compare results across techniques including DSC,TGA, DMA, SDT and rheometers
- One-click repeated analysis for increased productivity

#### Ease of Use

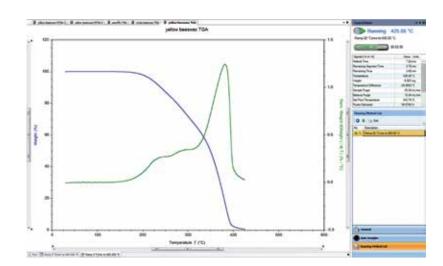
TRIOS software makes calibration and operation of the entire line of Thermogravimetric Analyzers simple. Users can easily generate multiple calibration data sets under varying experimental conditions (e.g. different heating rates or gas selections) and seamlessly switch between them to match the experimental conditions used for sample testing. Real-time signals and the progress of running experiments is readily available, with the added capability of modifying a running method on the fly.TRIOS software offers a level of flexibility that is unmatched in the industry.



- Automated custom report generation including: experimental details, data plots and tables, and analysis results
- Convenient data export to plain-text, CSV, XML, Excel®, Word®, PowerPoint®, and image formats
- Optional TRIOS Guardian with electronic signatures for audit trail and data integrity

#### **Complete Data Record**

The advanced data collection system automatically saves all relevant signals, active calibrations, and system settings. This comprehensive set of information is invaluable for method development, procedure deployment and data validation.



### The Most VERSATILE CONTROL and ANALYSIS SOFTWARE!

#### **Complete Data Analysis Capabilities**

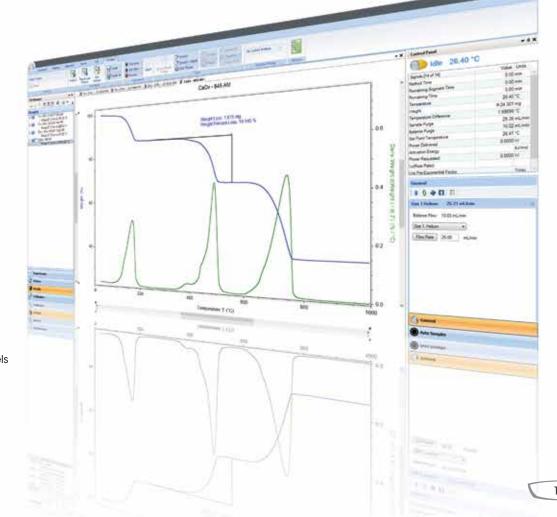
A comprehensive set of relevant tools are available for real-time data analysis, even during experiments. Gain actionable insights into your material's behavior through a powerful and versatile set of features seamlessly integrated into TRIOS.

#### All Standard TGA Analyses:

- Weight change (absolute and as a percentage)
- Residue content
- 1st and 2nd derivatives
- Weight at a specified time or temperature
- Weight loss at a specified time or temperature
- Peak height and area
- Temperature at peak maximum
- Onset and endset analyses
- Step transition analysis
- Easily import and export TGA data with TRIOS

#### **Advanced Analysis Capabilities:**

- Activation energy determination with Modulated TGA™
- Decomposition kinetics as obtained from constant or dynamic heating rate, and constant reaction rate experiments
- DTA signal for endothermic and exothermic thermal events such as melting, crystallization, cure reactions and decomposition
- Advanced custom analysis with user-defined variables and models



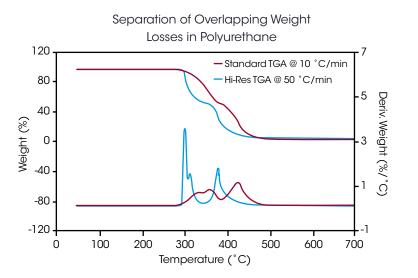
In Hi-Res™ TGA (only available from TA Instruments), the heating rate is controlled by the decomposition rate of the sample. The Discovery TGA 5500 and 550 designs are ideal for these measurements, featuring rapid response furnaces for precise temperature control and sensitive thermobalances designed to quickly detect small weight changes.

#### Benefits of Hi-Res TGA include:

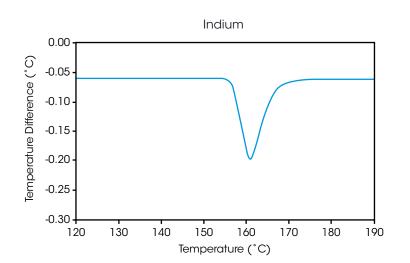
- Separation of broad and overlapping weight losses
- Increased productivity with better resolution
- Rapid survey over wide temperature range with excellent resolution
- Simple method setup

#### **DTA Signal**

The DTA signal is a qualitative measurement of endothermic and exothermic reactions occurring in the TGA. This signal can also be used for temperature calibration by using melting point standards.

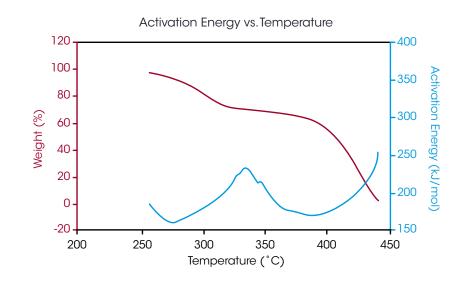


The figure above shows the Hi-Res TGA results for a polyurethane material by standard and Hi-Res TGA. The superior resolution provided by the Hi-Res technique is clearly evident in both the TGA weight loss and the first derivative (DTG) signals. The latter signal is especially useful in defining the onset and the endset of the individual weight loss segments, as well as indicating subtle events that provide a "fingerprint" of the sample.



### DISCOVER more abo

### more about your MATERIALS



TA's patented MTGA™\* is another TA Instruments innovation that offers advantages for material decomposition studies. Developed from the proprietary heater control technology utilized by Hi-Res™ TGA and MDSC®, MTGA produces model-free kinetic data. Activation energy can be continuously calculated during the test and studied as a function of time, temperature, and conversion.

#### **Benefits of MTGA include:**

- Increased productivity for studying kinetics
- Model-free kinetic data
- Can be combined with Hi-ResTGA for better separation of overlapping weight losses
- Direct determination of activation energy

The figure to the left shows the MTGA plot from a 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 can infer the mechanism involved.

<sup>\*</sup> U.S. Patent no. 6,113,261 and 6,336,741

#### 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 benchtop, quadrupole mass spectrometer with a heated capillary interface, and TGA module-specific interface kits for the Discovery TGA. A variety of FTIR suppliers provide gas cells and interfaces.

The Discovery TGA is the ideal platform for evolved gas analysis studies. A horizontal purge stream over the sample and a short path to the exit port eliminates dead volume in the furnace, thereby reducing product dilution and optimizing EGA sensitivity. Heated EGA adapters are designed to interface directly with the MS or FTIR transfer line to ensure continuous heating of the offgas stream through the furnace wall, dramatically reducing offgas condensation and improving EGA sensitivity.

TA Instruments' TRIOS 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 Features and Benefits:**

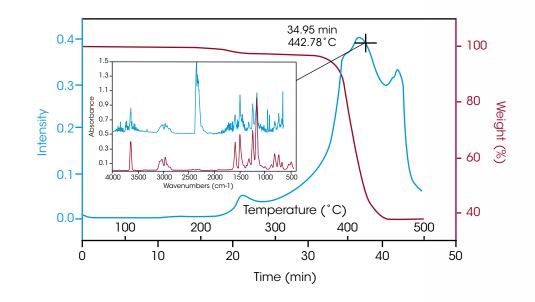
- Identification of decomposition products
- Additional information for the interpretation of the reactions during TGA scans
- Exact control of the furnace atmosphere before and during experiments

#### Design Features and Benefits of the Discovery TGA for EGA Analysis:

- Horizontal purge stream over the sample for optimal sensitivity
- Low volume furnace reduces dilution by eliminating dead volume
- Heated EGA adaptor eliminates cold spots and condensation
- Powerful TRIOS software allows importation of MS or FTIR data for improved data interpretation

#### **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.





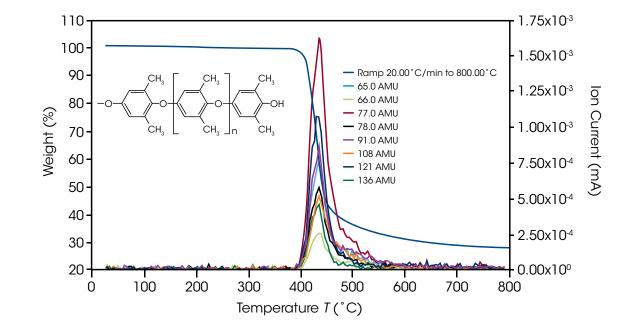
#### Discovery Mass Spectrometer (DMS)

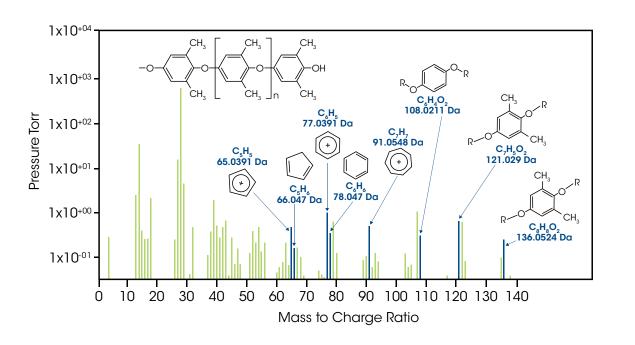
The Discovery MS is a benchtop quadrupole mass spectrometer, designed and optimized for evolved gas analysis. It features industry-standard technology configured for the efficient transfer and rapid detection of offgas from the TGA furnace. Parts per billion (ppb) sensitivity is ensured with our state-of-the-art 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	1-300 amu		
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	Bar graph and Peak Jump		
Scanning Speed			
Bar graph 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		





#### Decomposition of an Engineered Plastic

Poly(phenylene oxide), or PPO, is a high-performance, engineered thermoplastic with desirable specifications for heat resistance and dimensional stability. With a glass transition temperature as high as 215°C, the processing temperature of PPO would need to be high and will result in a cumbersome and costly manufacturing process. In many cases, other polymers such as polystyrene (HIPS) are blended with PPO to both aid in processing and improve the ductility over PPO alone. In this example, neat PPO is heated through decomposition in an inert nitrogen atmosphere with the offgas collected by the attached MS. The TGA-MS hyphenated technique allows for the detection and identification of the resultant decomposition products. The data is displayed as an overlay of ion current and weight loss with respect to temperature.TGA shows a monotonic weight loss step; however, the mass spectroscopy data presents the detection of several decomposition entities that range in mass to charge ratios of 65 to 136 amu, which is the molecular ion of the repeating unit. Proposed possible decomposition products, based on the structure of the polyether, are also shown.

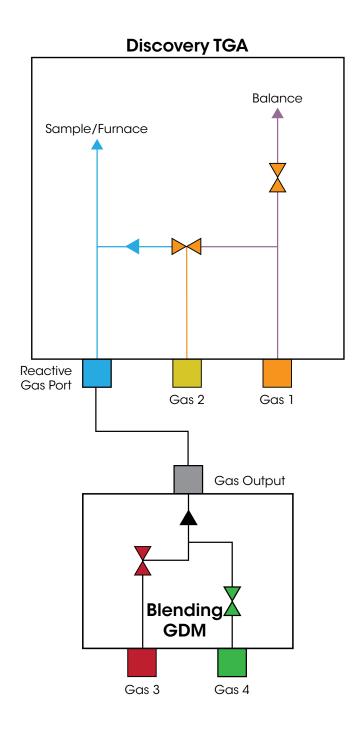
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#### Blending Gas Delivery Module

#### **Blending Gas Delivery Module**

The Blending Gas Delivery Module (Blending GDM) delivers flexibility in gas handling on the Discovery TGA 5500, TGA 550, and SDT 650. The Blending GDM is an external accessory with two gas inlet ports that, when connected to the reactive gas port on the TGA or SDT, gives the user a total of four gases to control. The software-controlled accessory allows for automated switching between the four gas ports as well as blending of binary mixtures of gases. The added blending capability allows for TGA experiments to be carried out in an atmosphere where the concentration ratio between gases may be fixed, stepped incrementally or ramped at a controlled rate. The Blending GDM is compatible with Nitrogen, Argon, Helium, Air, Oxygen, Carbon Dioxide, Carbon Monoxide and Forming Gas and can be used to study sorption of gases onto a material, redox reactions, and thermal stability of materials in a controlled atmosphere.

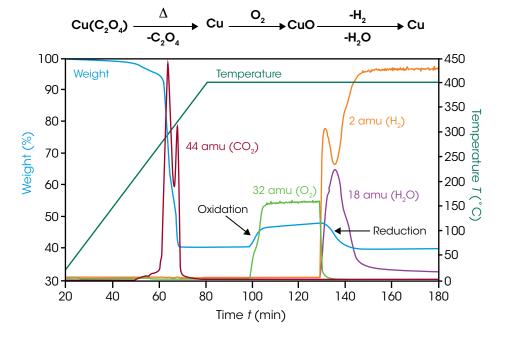




Gas Port	Gases Supported	Blend Options		
1 Instrument, also used as balance purge	N <sub>2</sub> , He, Ar	3 or 4		
2 Instrument	N <sub>2</sub> , O <sub>2</sub> , Air, He, Ar	3 or 4		
3 Blending GDM	N <sub>2</sub> , O <sub>2</sub> , Air, He, Ar, Forming Gas, CO, CO <sub>2</sub>	1, 2 or 4		
4 Blending GDM	N <sub>2</sub> , O <sub>2</sub> , Air, He, Ar, Forming Gas, CO, CO <sub>2</sub>	1, 2 or 3		

#### Redox Reaction of Copper Oxalate

Copper oxalate ( $CuC_2O_4$ ) is a salt that decomposes to elemental copper upon heating in an inert atmosphere. It is often used to measure the inertness of the TGA atmosphere, as the high surface area of copper is readily oxidized at high temperatures. In this example an oxidation-reduction (redox) reaction experiment was accomplished using the Blending GDM, Discovery MS and the Discovery TGA. After decomposition of the oxalate during the initial temperature ramp, oxygen was introduced into the TGA sample chamber resulting in the formation of copper (II) oxide. Subsequent reduction of the copper oxide was achieved through the introduction of small amounts of hydrogen gas. Forming gas was safely used as the hydrogen source in the TGA.

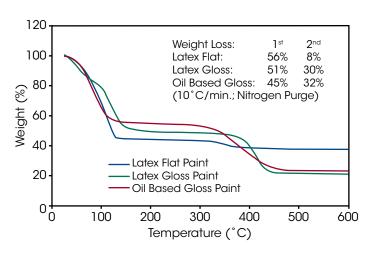


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#### **Applications**

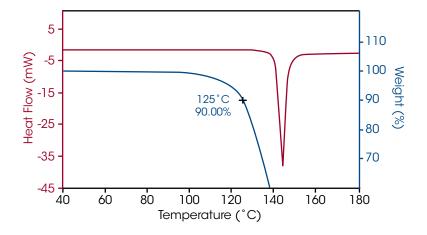
#### **Compositional Analysis**

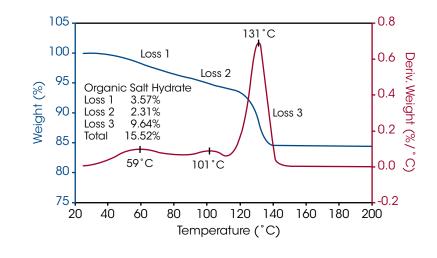
TGA is used to determine sample composition by measuring the weight of each component as it volatilizes or decomposes under controlled conditions of temperature, time, and atmosphere. This figure shows quantitative differences in type, amount, and decomposition mechanism of the main polymers in three paint samples. A 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.



#### **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.



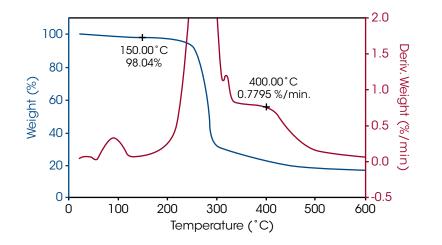


#### **Volatiles Analysis**

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.

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



### Choose the **BEST TGA** for YOUR NEEDS

Instrument Features	TGA 55	TGA 550	TGA 5500
Low Mass IR Furnace	_	_	•
Hi-Res TGA™	_	0	•
Modulated TGA™	_	0	•
Auto-Stepwise TGA	•	•	•
DTA Signal	_	0	•
Auto-loader	•	•	_
25-Position Autosampler	_	0	•
Sealed Pan Punch	_	0	•
Color App-Style Touch Screen	•	•	•
Long-Life Wire Wound (Pt/Rh) Furnace	•	•	_
EGA Furnace Capable	0	0	•
Dual Input Gas-Delivery Manifold	•	•	•
Integrated Electromagnet	_	_	•
Temperature Calibration Curie Point (ASTM E1582)	•	•	•
Temperature Calibration Melting Point Standards	_	0	•
Blending Gas Delivery Module	_	0	0
Heated EGA Furnace Adapter	_	_	0
TGA/MS Operation	0	0	0
TGA/FTIR Operation	0	0	0

Instrument Specifications	TGA 55	TGA 550	TGA 5500	
Temperature Range	Ambient to 1000°C	Ambient to 1000°C	Ambient to 1200°C	
Temperature Accuracy	±1°C	±1°C	±1°C	
Temperature Precision	±0.1°C	±0.1°C	±0.1°C	
Heating Rate (Linear)	0.1 to 100 °C/min	0.1 to 100°C/min	0.1 to 500°C/min	
Heating Rate (Ballistic)	>600°C/min	>600°C/min >600°C/min		
Furnace Cooling (Forced air/N2)	1000°C to 50°C in <12 min	00°C to 50°C in <12 min 1000°C to 50°C in <12 min		
Sample Weight Capacity	1000 mg	1000 mg	1000 mg	
Dynamic Weighing Range	1000 mg	1000 mg	1000 mg	
Weighing Precision	±0.01 %	±0.01 %	±0.01 %	
Resolution	0.1 μg	0.1 µg	<0.1 µg	
Weight Baseline Driff[1] (Ambient to 1000 °C)	<25 µg	<25 µg	<10 µg	
Vacuum	50 μTorr (EGA furnace)	50 μTorr (EGA furnace)	50 μTorr	

<sup>[1]</sup> Without baseline subtraction

All TA Instruments furnaces are covered by the industry's only 5 YEAR WARRANTY

#### Pan Specifications

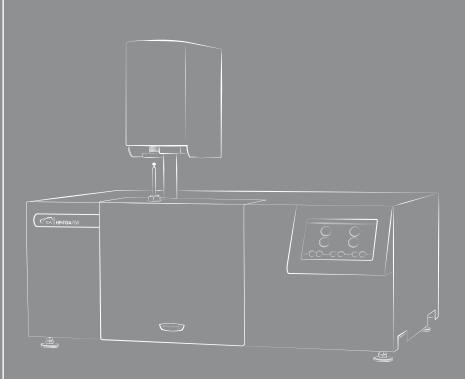
Material	Size	Temperature Range	Notes	
Platinum	50 μL 100 μL	Ambient to 1000°C	Robust, high performance, reusable pans	
Ceramic	100 μL 250 μL	Ambient to 1200°C	Reusable pans for higher temperatures	
Aluminum	80 µL	Ambient to 600°C	One-time use, can be sealed to prevent volatilization before experiment	

HIGH PRESSURE
TGA SYSTEMS
for MEASUREMENTS
UNDER EXTREME
CONDITIONS

**High Pressure** 

High Temperature

**Corrosion Resistant** 



#### High Pressure TGA

TA Instruments invites you to experience two new high pressure thermogravimetric (HP-TGA) instruments, the Discovery HP-TGA 75 and HP-TGA 750. From the world leader in Magnetic Suspension Balance (MSB) technology for over 20 years comes an ingeniously designed, user-friendly top-loading microbalance with unprecedented performance. In addition, the Discovery HP-TGA's are the first available in a convenient benchtop design and feature on-board gas dosing and blending systems, temperature control to 1100°C, One-Touch-Away™ functionality, and TA's powerful TRIOS software. Getting great high pressure data has never been so easy!

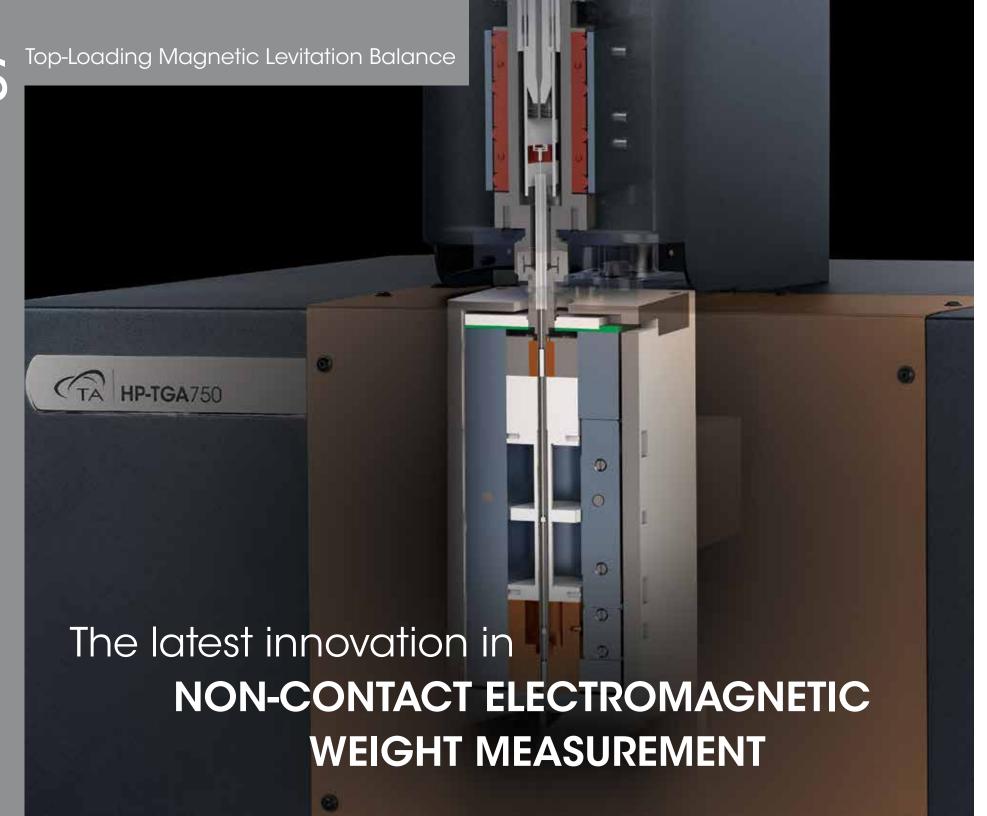
#### Features and Benefits:

- Patented\* top-loading magnetic levitation balance enables sealed environment for thermogravimetric testing in reactive atmospheres under high pressure (up to 80 bar) and high temperatures (up to 1100°C)
- Unrivaled 0.1 µg balance resolution provides the most accurate measurements of small samples or materials with rapid reaction kinetics
- Top-loading balance design ensures superior weight stability at high temperature and pressure, and provides convenient access to sample for easy loading/ unloading
- Integrated gas dosing & pressure control eliminates need for a separate system and enables a compact footprint
- Highly accurate balance temperature control for optimized baseline stability
- Non-porous isolation material in contact with the reaction gas within the furnace eliminates potential retention of gases ("memory" effect) and enables rapid attainment of vacuum
- Curie-point calibration eliminates the effects of the reaction gas type and pressure on the temperature measurement
- Compact design puts high pressure TGA on the benchtop, minimizing valuable lab space requirements and enabling installation in a fume hood to easily manage ventilation when working with toxic gases
- High heating and cooling rates (~250 K/min\*\*), even under high pressure, reduces potential for unwanted side reactions and improves sample throughput
- Small internal volume allows for rapid gas changes and quick pressurization, low gas consumption, and safe operational conditions due to the small quantity of compressed gas
- \* European Patent: 1958323, U.S. Patent: 2009/0,160,279 AI, German Patent: DE 10 2015 116 767.0
- \*\* Cooling rate at ~250 K/min possible when at T  $\geq$  300°C

## The world's first BENCHTOP HIGH PRESSURE TGA featuring patented

TOP LOADING MAGNETIC
SUSPENSION BALANCE





At the core of every Discovery HP-TGA is the new top-loading magnetically-levitated (MagLev) balance. Multiple patented\* technologies are combined to bring to life a highly sensitive and compact balance that can operate under high pressure and high temperature.

#### **How it Works**

Within the Mag-Lev Balance, a small-diameter high pressure-resistant steel alloy tube encloses a suspension shaft and crucible setup. A dual Anti-Helmholtz coil array, LVDT electronic sensor coils and the quadrupole magnet assembly are outside of the tube. The Anti-Helmholtz coils generate an extremely uniform electromagnetic field which levitates a permanent magnet attached to the suspension shaft. A platform at the top of the shaft holds the sample crucible. The suspension shaft is centered horizontally inside the tube by patented 2D magnet quadrupole bearing rings located at the top and bottom of the shaft. The vertical location of the permanent magnet is held constant via a control feedback loop between the Anti-Helmholtz coils and an LVDT position sensor with sub-micron resolution located on the shaft below the magnet. The amount of current that is delivered to the coils in order to maintain a constant position of the magnet is proportional to the weight of the shaft, magnet, and crucible. This weight is set to zero through a balance tare. When a sample is added to the crucible, the current required to maintain the balance position is now proportional to the sample weight.

In this configuration, the components contained within the small-volume tube are completely sealed off from the outside. The electronmagnetic coils and other sensitive parts are located outside of the tube and operate under normal atmospheric conditions to generate the electromagnetic levitation force through the pressure-resistant tube. Only the sample crucible and other components within the small-volume tube need to be pressurized and can be exposed to a variety of gases or gas mixtures. This complete separation of the balance electronics from the reaction atmosphere enables TGA measurements to be performed from vacuum to high pressures using toxic, corrosive and explosive reaction atmospheres.

<sup>-</sup>Furnace Tube Sample Crucible -Heating Element Cell Closure Upper Horizontal Quadrupole Bearing Suspension Shaft Anti-Helmholtz Solenoid\* Permanent Magnet Anti-Helmholtz Solenoid - High-pressure Resistant Steel Alloy Tube Position Sensor Lower Horizontal Quadrupole Bearing

<sup>\*</sup> Solenoid = Coil wound in a straight hollow helix.

High Pressure Furnace & Integrated Gas Dosing



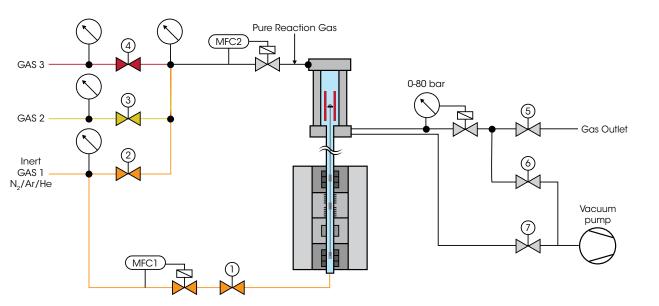
An innovative high-pressure reaction furnace for the most accurate and responsive temperature control under ALL pressure and gas flow conditions.

At the core of the Discovery HP-TGA furnace is a robust corrosion-resistant ceramic tube with an embedded platinum heating element capable of temperature control to 1100°C\*. Sample temperature is measured by a thermocouple within the heater tube directly adjacent to the sample. The compact, low-mass design is highly responsive and capable of heating/cooling rates of up to 250°C/min. The ceramic heater tube is contained within a pressure vessel which enables characterizing samples to 80 bar. Testing can be performed in corrosive reaction atmospheres and, as no porous material is in contact with the atmosphere, changing of the reaction gas is clean, fast, and without any memory effects. The Discovery HP-TGA is the only high pressure TGA to feature Curie-point temperature calibration at any pressure and with any reaction gas. HP-TGA temperature calibration has never been so easy.

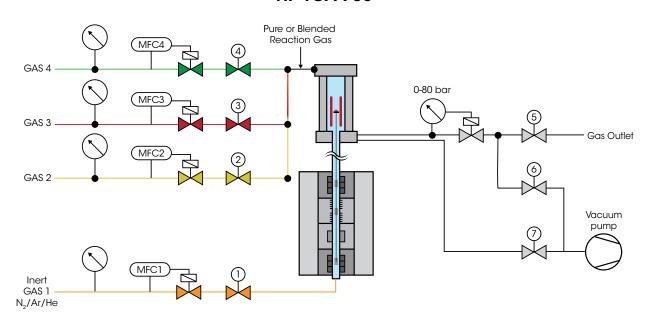
 Maximum temperature obtained with N<sub>2</sub> and other reaction gases with similar heat conductivity



#### HP-TGA 75

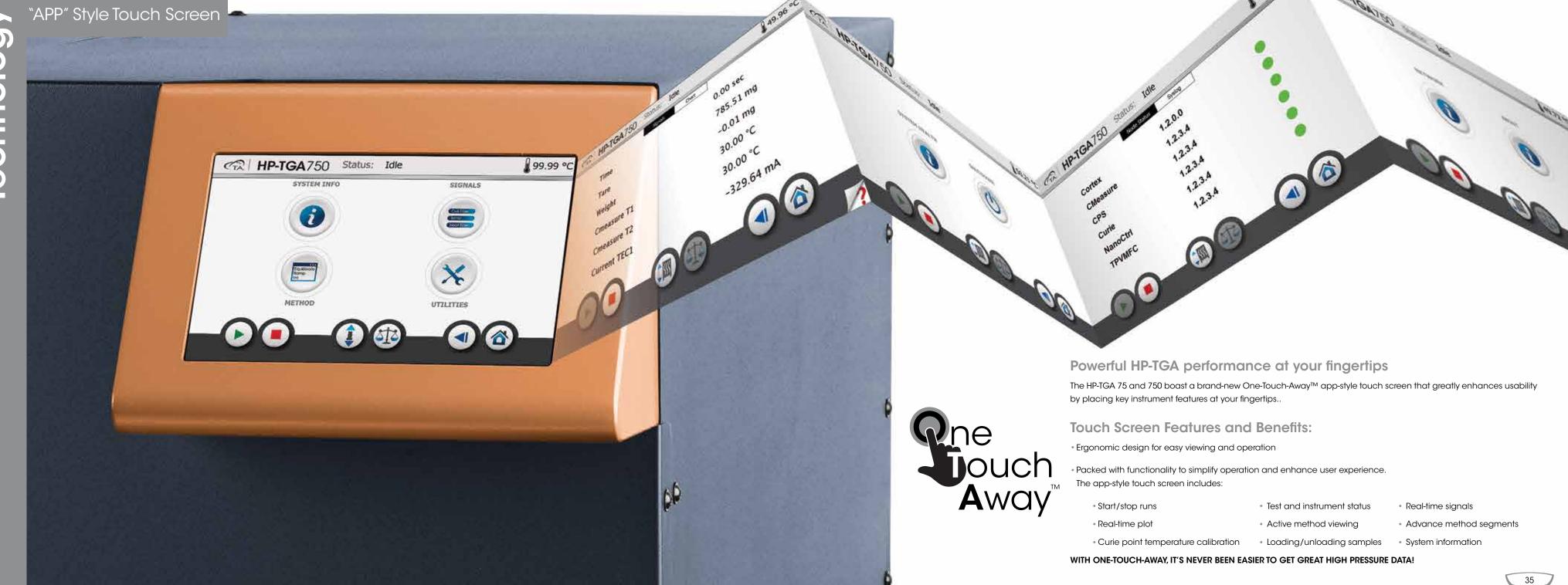


#### **HP-TGA 750**



### Integrated Gas Dosing and Blending Systems with Pressure Controllers

The accuracy of TGA measurements depends on reliable control of the pressure and composition of the reaction atmosphere. All Discovery HP-TGA models feature integrated gas dosing and blending systems with pressure controllers, which ensure the highest data quality while providing flexibility to address the widest range of applications. The pressure can be controlled in the range from 200 mbar to 80 bar or complete evacuation to ultimate vacuum. Both the HP-TGA 75 and 750 instruments include a mass flow controller connected to an inert gas for the balance purge. The Discovery HP-TGA 75 is equipped with a single reaction gas mass flow controller and three gas connections. One reaction gas can be selected from the three connected. During a measurement the reaction gas can be switched. The Discovery HP-TGA 750 is equipped with three reaction gas connections and three independent reaction gas mass flow controllers, which enables the reaction gas to be a pure gas or a blend of up to three gases.



#### TRIOS Software

Discover powerful TRIOS software that delivers exceptional user experience in a combined package for instrument control, data analysis and reporting for thermal analysis and rheology. New features such as multiple calibration sets, real-time test method editing, and inter-laboratory data and test method sharing provide unmatched flexibility, while one-click analysis and custom reporting raise productivity to new levels.



#### **TRIOS Features:**

- Control multiple instruments with a single PC and software package
- Overlay and compare results across techniques including DSC,TGA, DMA, SDT and rheometry
- One-click repeated analysis for increased productivity

#### Ease of Use

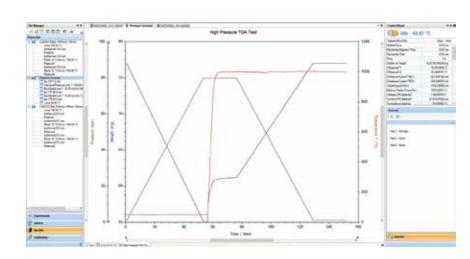
TRIOS software makes calibration and operation of the entire line of Thermogravimetric Analyzers simple. Users can easily generate Curie-point temperature calibration data sets under varying experimental conditions (e.g. different pressures or gas selections) which are automatically applied to match the experimental conditions used for sample testing. Real-time signals and the progress of running experiments is readily available with the added capability of modifying a running method on the fly.TRIOS software offers a level of flexibility that is unmatched in the industry.



- Automated custom report generation including: experimental details, data plots and tables, and analysis results
- Convenient data export to plain-text, CSV, XML, Excel®, Word®, PowerPoint®, and image formats
- Optional TRIOS Guardian with electronic signatures for audit trail and data integrity

#### **Complete Data Record**

The advanced data collection system automatically saves all relevant signals, active calibrations, and system settings. This comprehensive set of information is invaluable for method development, procedure deployment, and data validation.



### The Most VERSATILE CONTROL and ANALYSIS SOFTWARE!

#### **Complete Data Analysis Capabilities**

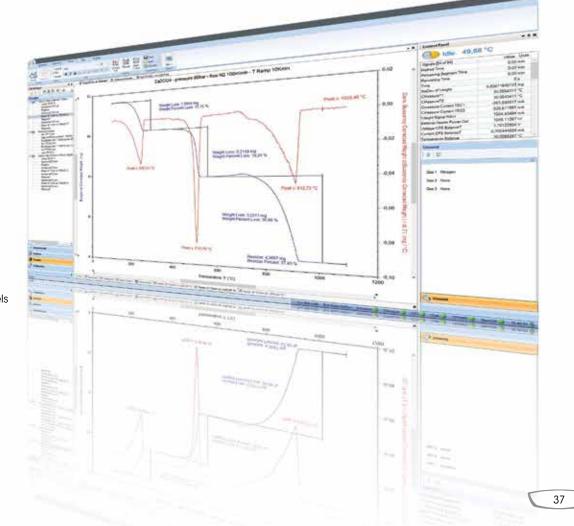
A comprehensive set of relevant tools are available for real-time data analysis, even during experiments. Gain actionable insights into material behavior through a powerful and versatile set of features seamlessly integrated into TRIOS.

#### All Standard TGA Analyses:

- Weight change (absolute and as a percentage)
- Residue content
- 1st and 2nd derivatives
- Weight at a specified time or temperature
- Weight loss at a specified time or temperature
- Peak height and area
- Temperature at peak maximum
- Onset and endset analyses
- Step transition analysis
- Easily import and export TGA data with TRIOS

#### **Advanced Analysis Capabilities:**

- Decomposition kinetics
- Advanced custom analysis with user-defined variables and models



## The **DISCOVERY HP-TGA**is Uniquely Suited for Challenging Thermogravimetric Applications



- High Temperature Corrosion
- Coal & Biomass Gasification
- CO<sub>2</sub> and CH<sub>4</sub> Getter Materials
- Pyrolysis Processes
- Catalyst Materials (TPx, Sulphidation, Coking)
- CVD Coating Processes
- Decomposition & Degradation Reactions





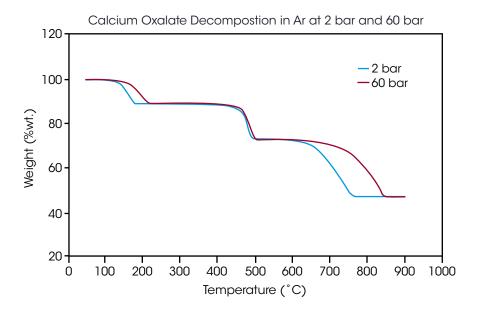


#### Weight Loss of Calcium Oxalate under Varying Conditions

Calcium Oxalate is a widely characterized material with very well-known and understood weight loss behavior. It undergoes three discrete decomposition events, each with a pronounced step change in weight. The onset of the weight change associated with each decomposition is affected by sample mass, heating rate, and pressure. The magnitude of the weight changes, as a percent of total starting weight, should not change with these variables.

In standard thermogravimetry measurements (TGA), the onset of decomposition can be studied under variable mass and heating rate. However, with the TA Instruments high pressure TGA (HP-TGA), measurements can be conducted as a function of all three variables (mass, heating rate, and pressure).

In the figure to the right, two Calcium Oxalate decomposition measurements in Ar at 2 bar and 60 bar pressure are compared. While the decomposition temperatures of the three steps are all shifted to higher temperatures at high pressures, the weight changes in each decomposition step are identical. This illustrates the kinetic nature of decomposition. Varying the pressure, heating rate, or initial sample mass will impact the temperature at which a material decomposes.



#### Pyrolysis and Gasification

Coal, biomass, waste and other organic materials are gasified for energy utilization or as alternative feedstock. Such processes can be measured under application-relevant conditions in the Discovery HP-TGA. The first step in a gasification process is pyrolysis of the raw material, where, while heating the organic material in an inert atmosphere (eg. N2 or Ar), volatile components (water, hydrocarbons, tar) are evaporated and char is generated. Gasifying this carbon-rich char as a second reaction step requires a gasifying agent.

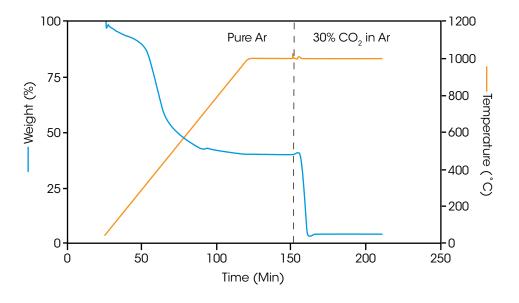
The gasification agent carbon dioxide and the carbon char generate carbon monoxide gas according to the following main reaction:

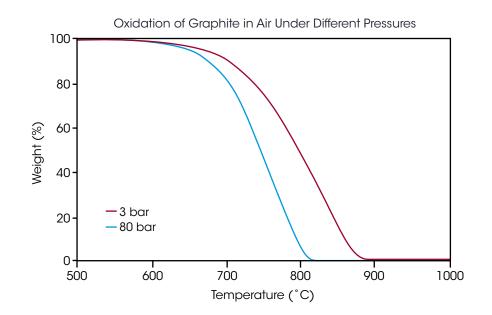
#### CO, + C → 2CO

Additional gases can be products of further or incomplete conversions and side reactions.

Because the process reaction kinetics depend on the reaction conditions and the raw material, the composition and pressure of the gases generated will vary. Discovery HP-TGA instruments allow optimization of operating conditions for a given raw material. In addition, they can be equipped with a mass spectrometer for evolved gas analysis.

In the figure to the right, the pyrolysis and gasification process of a lignite at 30 bar measured with the Discovery HP-TGA is shown. During heating to  $1000^{\circ}$ C at a heating rate of  $10^{\circ}$ C/min, Ar is dosed as the reaction gas. The resulting weight loss of approximately 60% is due to charring and pyrolysis of the lignite. When constant weight is reached, 30% CO<sub>2</sub> is blended into the Ar to start the gasification process. Gasification leads to another weight loss of 35%.





#### **Graphite Oxidation**

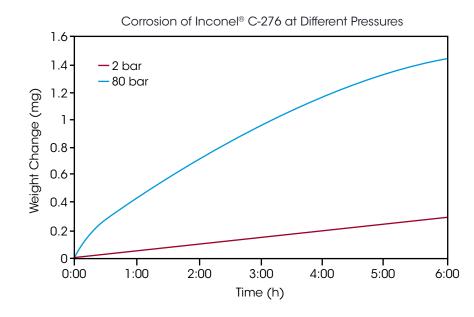
Burning of solid or liquid fuels is an oxidation process. The oxidation temperature and the reaction kinetics depends on the pressure and the oxygen content of the reaction gas. With the Discovery HP-TGA the influence of pressure and oxygen concentration on the oxidation can be studied. In this example, graphite was oxidized in air at 3 bar and at 80 bar. The data in the figure to the left show that at the higher pressure of 80 bar, the reaction comes to completion at a much lower temperature compared to the experiment at lower pressure. The ability to complete a reaction with lower energy input can save significant cost in manufacturing processes. Some relevant applications include pressurized fluidized bed power plant design and underground coal gasification.

## PRESSURE can change the REACTION KINETICS

## PRESSURE DEPENDENCE of your MATERIALS

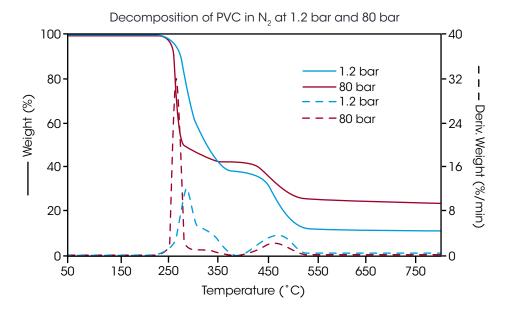
#### **High Temperature Corrosion**

Understanding the corrosion resistance of a material can be critical for improving technical processes and increasing efficiency. For example, the efficiency of gas or steam turbines and jet engines is directly related to their maximum operation temperature. The maximum temperature is limited by the high-temperature corrosion of the materials used. The mass change of a metal or other material caused by corrosion is generally very small. Additionally, even high temperature corrosion is usually a slow process. The Discovery HP-TGA is ideally suited for such measurements because the exceptional high resolution and accuracy allows measurement of small changes in sample mass over a comparably short period of time. The figure to the right compares the mass increase of a Inconel®\* C-276 alloy in air at 1000°C at 3 bar and at 80 bar. The observed weight gain is caused by oxidation of the alloy's surface. The total mass change here is about 287 µg at 3 bar and 1444 µg at 80 bar. As expected, the pressure of the corrosive atmosphere has an influence on the kinetics of, and amount of, corrosion.



#### **PVC** Decomposition

Thermal decomposition of polymeric materials is a routine thermogravimetric test. The new Discovery HP-TGA enables the added dimension of understanding the influence of pressure on decomposition temperatures and kinetics. This information is critical to define the limits of operation which should not be exceeded during manufacturing or applications. Polymer materials can be tested under real-world conditions using the actual pressures and reaction gases of interest. In the figure to the right, the decomposition of PVC-P in nitrogen gas is compared at pressures of 1.2 and 80 bar. The decomposition is a multistage process. Typically HCL, aliphatic and aromatic hydrocarbons are the decomposition products. At higher pressure, the kinetics of the first step of decomposition is much faster compared to the measurement at ambient pressure. The following decomposition steps are more clearly separated than at low pressure. Decomposition temperature is not significantly changed by the higher pressure. At 80 bar however, a residue of ca. 23%wt is remaining after the decomposition while at ambient pressure only 10% of the PVC is not decomposed.



<sup>\*</sup> INCONEL® is a trademark of Huntington Alloys Corporation, Huntington, WV 25705, United States of America

Model	Max. Sample Temperature	Max. Heating / Cooling Rate	Maximum Pressure [1]	Weighing Resolution	Mass Range	Reaction Atmosphere
HP-TGA 75		250°C/min 250°C/min	80 bar	0.1 µg	500 mg	Pure Gas (select 1 of 3)
HP-TGA 750	1100°C*	(at T > 300°C)				Pure Gas & Gas Blends (of 3 Gases)

<sup>\*</sup> Maximum temperature obtained with N, and other reaction gases with similar heat conductivity





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