Dreefs 3080/2

Lighted Rocker Switch

TGA 2950

Thermogravimetric

Analyzer

Instruments
Thermal Analysis & Rheology
A Subsidiary of Waters Corporation

Thermogravimetric Analysis:

The Technique

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. Measurements are used primarily to determine the composition of materials and to predict their thermal stability at temperatures up to 1000°C. The technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration. It is especially useful for studying polymeric materials, including composites, but also is widely applicable to other organic materials, such as lubricants and edible oils, and to a wide range of ceramics, metals and other inorganics.

What TGA can tell you

TGA experiments provide important information that can be used to select materials, predict product performance, and improve quality. The technique is particularly useful for determining:

- Composition of multicomponent systems
- Thermal stability of materials
- Oxidative stability of materials
- Estimated lifetime of a product
- Decomposition kinetics of materials
- The effect of reactive or corrosive atmospheres on materials
- Moisture and volatiles content of materials

Uses of TGA

Research and Development

- Theoretical research on new materials and processes
- Materials selection
- Formulation optimization
- Applications development
- End-use performance prediction
- Competitive product evaluation

Quality Control/Assurance

- "Fingerprinting"
- Vendor certification
- Incoming/outgoing material consistency
- Process optimization
- Finished product performance
- Troubleshooting

TA Instruments TGA 2950 Thermogravimetric Analyzer

The TA Instruments TGA 2950 brings new levels of sensitivity, automation, ease-of-use, and flexibility to the technique of thermogravimetric analysis. The design and versatility of this plug-in module for the TA Instruments thermal analysis systems make the TGA 2950 a valuable, easy-to-use tool for the selection of materials and the prediction of a product's performance and lifetime. Ultra-high sensitivity (0.1μg) and the ability to simultaneously generate a calibrated derivative assure accurate measurements and make possible the detection of even minor components in a compound. The addition of optional high resolution capability further enhances the analyzer's quantitative accuracy and precision.

Improvements over previous generations of TA Instruments and competitive thermogravimetric analyzers include mechanical and electronic automation features that will appeal to novices as well as to experienced scientists and laboratories in which high throughput is important. The instrument's one button operation benefits both. Once an experiment has been programmed and the sample has been positioned and tared, a single keystroke initiates the complete sequence of operating functions: loading the sample pan, raising the furnace, starting and running the method, collecting and storing the data, unloading the sample pan, and rapidly cooling the furnace. Changes in purge gas can be automated, if the system is equipped with an optional Gas Switching Accessory.

Automated sample handling is a functional advantage as well as a convenience, considering the inherent delicacy of the technique, small sample size, and ultra-sensitivity of the balance mechanism. The need for operator dexterity is limited to filling the sample pan and placing it in the well-defined, adjustable receptacle on the sample loading arm.

The vertical configuration of the balance assembly contributes significantly to the instrument's sensitivity and high capacity. The horizontal flow of purge gas assures envelopment of the sample in the desired atmosphere while minimizing buoyancy and chimney effects. In addition, low-volume inert gas flows from the balance chamber into the furnace to prevent back diffusion of purge gas and any gases that evolve from the sample. This prevents possible contamination or corrosion of the balance.

Further analysis of evolved gases is possible by connecting the TGA 2950 (equipped with optional EGA Furnace) to an FTIR, mass spectrometer, or other gas analyzer. This is done by installing an interconnection from the purge gas outflow port.

High Resolution TGA

The Hi-Res™ TGA option improves the ability to separate closely occurring events. This option includes three control algorithms, (constant reaction rate, automated stepwise isothermal and Dynamic Rate) the most powerful of which is the TA Instruments patented* Dynamic Rate method.

Modulated TGA™

This optional patent pending technique is the result of TA Instruments' continuing research and development in the use of variable heating rate thermal analysis techniques. Similar to TA Instruments widely acclaimed MDSC® technology, MTGA™ exposes the sample to a linear heating rate which has a superimposed sinusoidal temperature oscillation (modulation) resulting is a cyclic heating profile. The data generated in MTGA experiments provides the ability to generate complete decomposition kinetic parameters in a single experiment, including the activation energy and pre-exponential factor.

* US Patent Numbers 5,368,391; 5,165,792

Data Analysis Software

Universal Analysis

A versatile "general purpose" data analysis program is an integral part of the *Thermal Solutions* Software. This program analyzes files from all the core thermal analysis modules (DSC, DTA, TGA, SDT, TMA, DEA, and DMA) and provides the following analysis capabilities and features:

TGA Standard Analysis

- Temperatures of transitions
- Step transitions
- Weight loss or gain
- Weight % at temperature
- Weight loss at temperature
- Moisture-free/ash-free calculations
- Residue analysis

Generic Analysis Functionality

- Peak integration
- Partial areas
- Onset temperature
- Step transition
- Running area integral plots
- Data point value
- Tabular data report
- Results report
- ASCII file export
- PCX and HPGL file export
- Curve rotation
- File addition and subtraction
- Generic equation calculations
- X and Y linear transformation
- Curve overlay
- Saved analysis
- Saved session

Specialty Programs

In addition to Universal Analysis, TGA Decomposition Kinetics software is available. This program calculates Kinetic parameters (e.g., activation energy) from which a nomographic plot can be produced automatically. This is an easy-to-read plot showing estimated lifetime at various end-use temperatures.

Figure 2
TGA 2950 Schematic

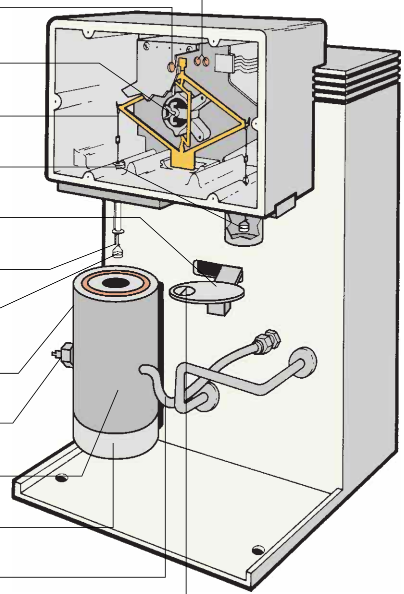


Figure 3
Sample Pans

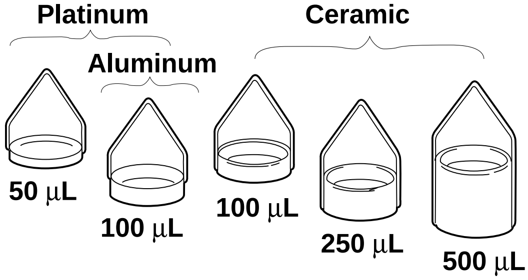


Figure 4
Placing sample pan on platform

Principles of Operation

Electromechanical components of the TGA 2950 Thermogravimetric Analyzer are shown in Figure 2. The system operates on a null-balance principle, using a highly sensitive transducer coupled to a taut-band suspension system to detect minute changes in the mass of a sample. An optically actuated servo loop maintains the balance arm in the horizontal reference (null) position by regulating the amount of current flowing through the transducer coil. An infrared LED light source and a pair of photosensitive diodes detect movement of the beam. A flag at the top of the balance arm controls the amount of light reaching each photosensor. As sample weight is lost or gained, the beam becomes unbalanced, causing the light to strike the photodiodes unequally. The unbalanced signal is fed into the control program, where it is nulled. This changes the amount of current supplied to the meter movement, causing the balance to rotate back to its null position. The amount of current required is directly proportional to the change in the mass of the sample.

The TGA 2950 operates in either of two weight ranges: 0 to 100 milligrams, or 0 to 1 gram. Since each is continuous throughout its weight-loss range, the entire range can be viewed without any loss of information. Range selection is automatic.

Heating rate and sample temperature are measured by the thermocouple located immediately adjacent to the sample (ideally, 2 mm above). This positioning enables the controller to program and maintain the temperature environment and heating rate selected by the operator. It also helps assure accurate measurement of sample temperature.

Sample pans are available in several sizes and in either platinum, aluminum or ceramic (Figure 3). Platinum is preferred because it is easy to clean and does not react with most materials. Ceramic is used for samples that might amalgamate or react with platinum, and when large capacity is required. Aluminum is used when disposability is desired up to 600°C.

Once the pan is filled, it is placed in the receptacle on the sample platform (Figure 4). Its orientation can be adjusted to assure proper positioning for automatic pickup. Then the operator presses the “start” key to begin an automatic sequence of events: rotation of the sample platform, automatic sample pickup, raising of the furnace, and initiation of the previously programmed experimental conditions and procedures. An optional auto sampling accessory (Figure 6) is available for increased throughput and maximum efficiency.

The progress of an experiment can be monitored by the status display screen on the module and by real-time plotting or operator-initiated display of status on the controller.

Data is automatically collected and stored and can be analyzed and prepared for presentation while experiments are in process.

End-of-run routines, including sample unloading and rapid furnace cooling, can be programmed or controlled by the operator.

Features and Benefits

The TGA 2950 is designed with the operator in mind. It offers new levels of convenience, automation, and measurement sensitivity, while retaining all of the proven attributes of its predecessors. Many of the benefits are inherent in its operation as a component of a Thermal Analyst controller/module system. Key features of the TGA 2950 and the benefits they offer include:

Modular Design, with electronics and operating software contained within the module for reliability and convenient connection to the central controller.

Exceptional Weight Sensitivity, permitting detection and measurement of very small weight gains or losses (less than 1μg), and use of small quantities of sample.

Five-Point Temperature Calibration, for maximum temperature accuracy. The operator can choose from one to five calibration standards. Temperature calibration is with either well characterized curie temperature materials, or high-purity metals with well documented melting points.

Wide Dynamic Range, permitting measurement of sample weight losses of up to 1 gram.

Controlled Atmosphere, with horizontal flow of gas across the sample (Figure 5). The indirect flow pattern helps assure good sample/atmosphere interaction while minimizing the buoyancy and chimney effects. Purge gases can be inert or reactive. In addition, a positive flow of inert gas from the balance chamber into the furnace protects delicate components against back diffusion of furnace purge gases or sample effluents.

High Resolution Capability, improves the ability to resolve (separate) successive overlapping weight losses for more accurate, reproducible weight changes.

Modulated TGA™, provides the ability to generate complete decomposition kinetic parameters in a single experiment.

Local Control at the Module, including experiment start/stop, heater on/off, furnace positioning, taring of the empty pan, and real-time display of sample temperature and experiment status.

Methods Versatility, with ability to store an unlimited number of methods containing up to 60 segments each selected from 18 available functions, including control of heating (0.1 to 100°C/min), environment, and data handling.

Unattended Operation. Once an experiment has been set up and programmed, all functions are performed automatically. Automatic change of atmosphere requires use of the optional Gas Switching Accessory. Multiple samples can be run unattended using an optional autosampler accessory (Figure 6).]

Ease of Use. Operator-oriented features include:

- Automated weight calibration
- Automated taring and determination of sample weight
- Automated pickup of the loaded sample pan.
- Automated collection, storage, and display of data.
- One-button operation. Once an experiment has been programmed, the sample positioned, and the balance tared, a single keystroke initiates the complete sequence of operating functions - raising the furnace, starting and running the method, collecting and storing the data, unloading the sample pan, and rapidly cooling the furnace.

Rapid Turnaround. Programmable end-of-run conditions include accelerated furnace cooling using forced air purge.

Compatibility with other Techniques. The TGA 2950 is one of many TA Instruments thermal analysis and rheology techniques that complement each other. These modules, together with TA Instruments controllers and software, constitute the most complete, versatile and cost effective systems available for characterizing materials.

Figure 5
Purge gas system

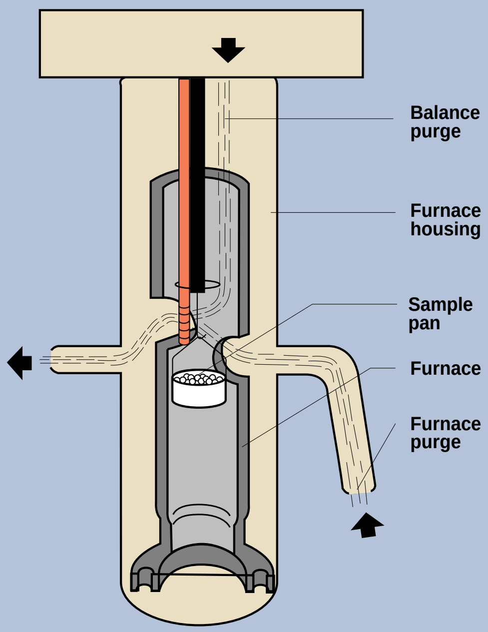
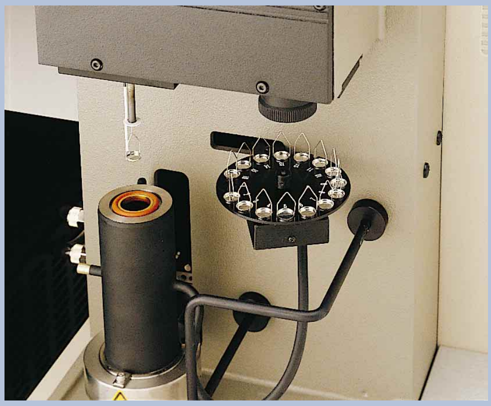


Figure 6
TGA Autosampler Accessory



Applications

The extended capabilities of the TA Instruments TGA 2950 as a valuable tool for materials characterization and quality control are illustrated by these applications. The examples are chosen to represent both the broad range of information that can be developed by TGA experiments and the variety of materials that can be evaluated.

Multipoint Temperature Calibration

The TGA 2950 uses a single thermocouple to both control the furnace and monitor sample temperature. This simplified design is particularly beneficial to the temperature calibration process, and contributes significantly to the precision and accuracy of the temperature measurements. Because the thermocouple is located adjacent to the sample pan, highly exothermic or endothermic reactions within the sample cause minor fluctuations in the programmed heating rate. While these fluctuations are not of sufficient magnitude to affect the results of an experiment, they do create an easily detected peak in the heating rate derivative. Hence, the melting of commonly used metal standards offers a rapid, convenient way to calibrate the instrument. Figure 7 shows the results of a multipoint calibration procedure using indium, tin and zinc. When more than two standards are used, the TGA 2950 calibration software employs a cubic-spline method to calculate a third-order polynomial equation (French curve) to determine precise temperature corrections anywhere on the curve.

When an additional standard, lead, was run to verify the calibration, the melting response was within 0.5°C of theoretical.

Temperature calibration can also be achieved using Curie point standards where the magnetic transition of ferromagnetic materials is characterized as shown in Figure 8.

Composition Analysis Using High Resolution TGA

The high balance sensitivity, single thermocouple design, and horizontal purge gas flow of the TGA 2950 make it ideally suited for Hi-Res™ experiments where the ability to precisely and rapidly control sample temperature and monitor small weight changes is critical. This option includes three control algorithms, (constant reaction rate, automated stepwise isothermal and Dynamic Rate) the most powerful of which is the TA Instruments patented* Dynamic Rate method. In a Dynamic Rate Hi-Res experiment, the heating rate is controlled in response to the measured rate of weight loss. When the weight is stable, the sample heats rapidly. As the rate of weight loss increases, the heating rate decreases. The result is that each weight loss step occurs over a narrower temperature range, hence improving the ability to separate, or resolve, subsequent weight loss steps. Because the patented Dynamic Rate method varies the heating rate smoothly and continuously, it is the fastest and most reliable of the various techniques. Figures 9 and 10 illustrate the benefits of high resolution TGA.

Ethylene vinyl acetate (EVA) is a common copolymer which illustrates the ability of TGA to determine the relative percentages of the polymer present based on the degradation profile. In nitrogen, EVA degrades in a two step process, where the first weight loss corresponds to acetic acid. Using a weight ratio, which accounts for the vinyl acetate/acetic acid stoichiometry, it is possible to determine the % vinyl acetate in the copolymer. Figure 9 shows the weight loss curves for several different EVA fomulations by conventional TGA. Although the formulations are obviously differentiated, the two steps are not well resolved and therefore compositional analysis will be based on operator judgment. Figure 10, on the other hand, illustrates the results of the same EVA samples using the patented Hi-Res™ Dynamic Rate method. Resolution of the two weight loss steps is clear facilitating compositional analysis.

Figure 7
Multipoint temperature calibration

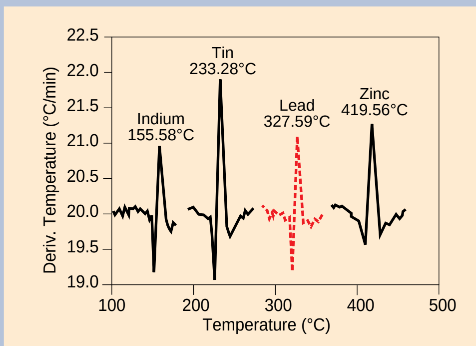


Figure 8
TGA Curie Point Temperature Calibration (Nickel)

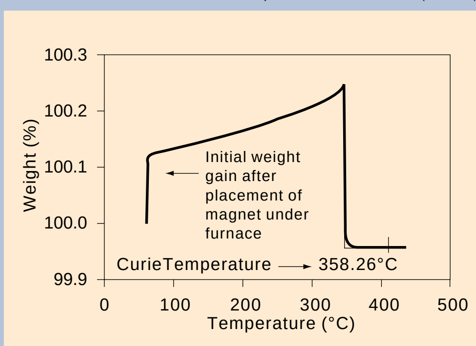


Figure 9
Ethylene Vinyl Acetate - Conventional TGA

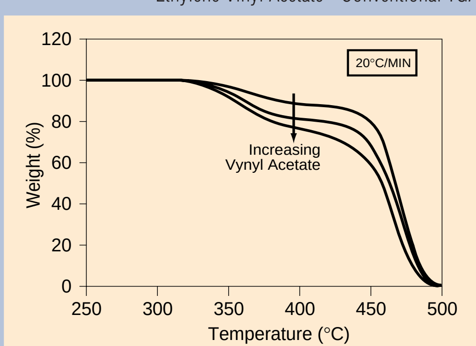
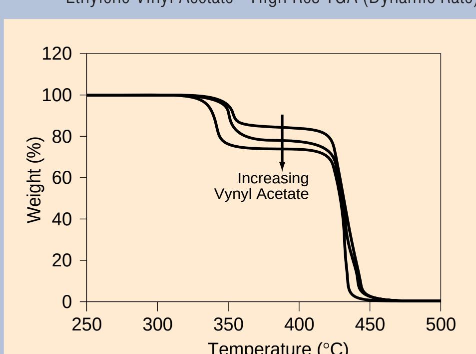


Figure 10
Ethylene Vinyl Acetate - High Res TGA (Dynamic Rate)



Oxidative Stability of Edible Oils

A critical quality parameter for edible oils is the resistance to oxidation. One widely used technique for predicting this resistance to oxidation (rancidity) involves heating the oil in an oven and periodically testing it for weight gain due to oxygen uptake. TGA allows the same measurement to be made continuously, as shown in Figure 11. The oil is heated to the temperature of interest in an inert atmosphere, then the purge gas is changed to oxygen. The time from oxygen introduction to the onset of weight gain is the oxidative stability. Excellent isothermal temperature stability and the ability to detect small weight changes makes the TGA 2950 ideal for obtaining reproducible results.

Estimating Polymer Lifetime

Estimating the lifetime of a polymer requires some form of accelerated aging. Traditional techniques, such as oven aging, are time consuming, requiring weeks or months of exposure. TGA decomposition kinetics offers a rapid alternative that can provide definitive results within minutes or hours. Figure 12 shows the TGA decomposition curves for a wire insulation subjected to several heating rates. A specific percent-decomposition (or "conversion") rate usually is used to compare the data. In this case, 5% decomposition was chosen as the best indication of lifetime since it was high enough to assure that the decomposition was attributable to failure of the polymer rather than evolution of volatiles. The kinetics software then calculates specific values (e.g., activation energy), from which a nomographic plot (Figure 13) can be produced automatically. This plot is an easy-to read depiction of the lifetime of the material at various end-use temperatures.

Evaluation of Superconductors

High temperature superconductors are an exciting new class of ceramic-metal oxides which typically are synthesized by pyrolysis of stoichiometric mixtures of metal salts under controlled conditions. Many of the materials currently being developed as candidate superconductors have a specific stoichiometry (particular oxygen content) that is critical to obtaining super-conductive behavior. The high sensitivity and good atmosphere control of the TGA 2950 make this instrument ideal for determining the temperature processing window, inorganic-to-oxygen ratio, and post-sintering cooling rate required to obtain optimum superconductor products. Figure 14 shows the oxygen uptake of a lanthanum-copper oxide (La_2CuO_x) material. On first heating, La_2CuO_4, which is not superconductive, shows a weight loss at about 875°C, which corresponds to oxygen loss (decomposition). Upon cooling, however, the sample gains more oxygen than it had at the start of the TGA experiment, becoming $La_2CuO_{4.14}$. When the $La_2CuO_{4.14}$ is subjected to a reheating cycle, the 4.14 oxygen stoichiometry does not change. This indicates that $La_2CuO_{4.14}$ is a stable form of the ceramic material. $La_2CuO_{4.14}$ is still being tested for superconductivity, but $La_2CuO_{4.03}$ is known to be a superconductor.

* US Patent Numbers 5,368,391; 5,165,792

Figure 11
Oxidative stability of edible oil

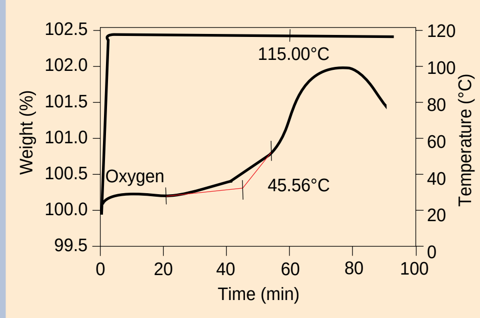


Figure 12
Decomposition kinetics

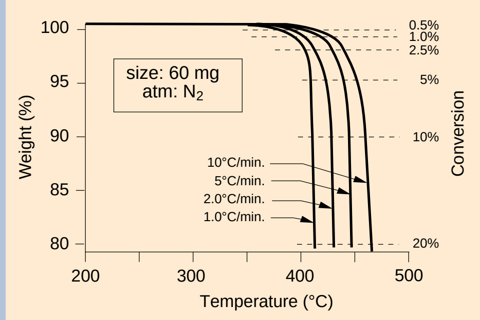


Figure 13
Estimating polymer lifetime by TGA kinetics

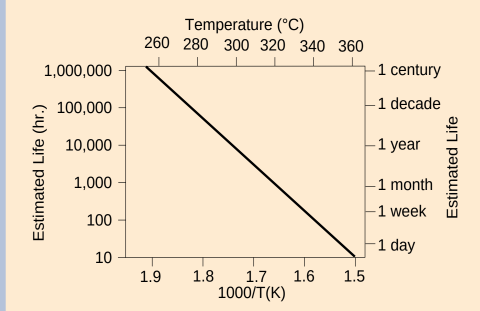
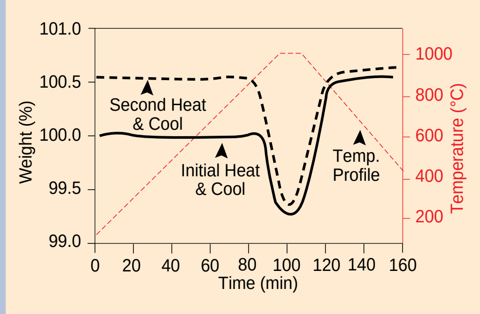


Figure 14
Evaluating La_2CuO_x superconductor



Specifications

Temperature Range:	Ambient to 1000°C
Sample Capacity (max):	1.5 gm (2.5 gm including sample pan system)
Weighing Capacity (max):	1.0 gm
Sensitivity:	0.1µg
Balance Accuracy:	±0.1%
Purge Gas Rate:	Furnace 60 mL/min; balance 40mL/min
Temperature Calibration:	1 to 5 points, based on metal or Curie Point standards
Heating Rate:	0.1 to 100°C/min in 0.01°C/min increments
Furnace Cooling:	Forced air
Sample Pans:	Platinum: 50µL, 100µL
	Alumina: 100µL, 250µL, 500µL
	Aluminum: 100µL
Thermocouple:	Platinel II
Data Collection Rate:	0.5 to 1,000 sec/point

Specifications are subject to change.

TA Instruments Commitment

The TGA 2950 is designed and engineered to assure easy, reliable, trouble free operation. It is supported by a full range of services, including an applications laboratory, publications, training courses, technical seminars, applications CD's, an internet website, and a telephone Hotline for customer consultation. Highly qualified service personnel specializing in thermal analyzer/rheometer maintenance and service are available throughout the world. All of these items reflect TA Instruments commitment to providing thermal analysis & rheology products and related support services that deliver maximum value for your investment.

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