More worldwide customers choose TA Instruments than any competitor as their preferred thermal analysis or rheology supplier. We earn this distinction by best meeting customer needs and expectations for high technology products, quality manufacturing, timely deliveries, excellent training, and superior after-sales support.

SALES AND SERVICE
We pride ourselves in the technical competence and professionalism of our sales force, whose only business is rheology and thermal analysis. TA Instruments is recognized worldwide for its prompt, courteous, and knowledgeable service staff. Their specialized knowledge and experience are major reasons why current customers increasingly endorse our company and products to their worldwide colleagues.

INNOVATIVE ENGINEERING
TA Instruments is the recognized leader for supplying innovative technology, investing twice the industry average in research and development. Our new Q Series™ Thermal Analysis modules are the industry standard. Patented innovations like Modulated DSC®, Tzero™ technology, and Hi-Res™ TGA are available only from TA.

QUALITY PRODUCTS
All thermal analyzers and rheometers are manufactured according to ISO 9001:2000 procedures in our New Castle, DE (USA) or our Crawley, UK facilities. Innovative flow manufacturing procedures and a motivated, highly-skilled work force ensure high quality products with industry-leading delivery times.

TECHNICAL SUPPORT
Customers prefer TA Instruments because of our reputation for after-sales support. Our worldwide technical support staff is the largest and most experienced in the industry. They are accessible daily by telephone, email, or via our website. Multiple training opportunities are available including on-site training, seminars in our application labs around the world, and convenient web-based courses.
Technology, performance, versatility, and reliability are words that describe a TA Instruments Q Series™ Differential Scanning Calorimeter (DSC). The Q1000, Q100, Q10, and Q10P are fifth-generation products from the world leader in differential scanning calorimetry. Each represents an unparalleled investment because it delivers cutting-edge technology, is designed with the customer in mind, and is backed by superior support that is the hallmark of our company.
TA INNOVATIONS

Differential Thermal Analysis
Heat Flux DSC
Pressure DSC
First Microprocessor Controlled Calorimeters
First Automated DSC
Dual Sample DSC
Differential Photocalorimetry
Modulated DSC®
Tzero™ DSC Technology
DIFFERENTIAL SCANNING CALORIMETERS

Q1000

The Q1000 is TA Instruments’ top-of-the-line, research-grade DSC, with unmatched performance in baseline stability, sensitivity, and resolution. It contains Advanced Tzero™ technology, the most powerful DSC technology commercially available. Its industry leading features include Modulated DSC™, a 50-position intelligent autosampler, and digital mass flow controllers. Photocalorimetry and pressure DSC accessories are also available, making the Q1000 the best-equipped analyzer to meet the needs of the most demanding researcher.

Q100

The Q100 is a versatile, research-grade DSC with our patented Tzero™ technology. With many Q1000 performance features, the Q100 easily outperforms competitive research models. It is an expandable module, to which MDSC®, a 50-position autosampler, or a photocalorimeter can be added. Innovative technology, performance, upgradability, and ease-of-use make the Q100 a superb addition to any laboratory.

Q10

The Q10 is a cost-effective, easy-to-use, general-purpose DSC with basic performance features equivalent to many competitive research-grade models. It is ideal for research, teaching, and quality control applications that require a rugged, reliable, basic DSC.
# Technical Specifications

<table>
<thead>
<tr>
<th>Feature</th>
<th>Q1000</th>
<th>Q100</th>
<th>Q10</th>
<th>Q10P</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tzero™ Technology</strong></td>
<td>Advanced</td>
<td>Basic</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>MDSC®</strong></td>
<td>Included</td>
<td>Optional</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Direct Cp Measurement</strong></td>
<td>Yes</td>
<td>Not Available</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Touch Screen</strong></td>
<td>Included</td>
<td>Included</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>User-Replaceable Cell</strong></td>
<td>Yes</td>
<td>Not Available</td>
<td>Not Available</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Pressure DSC Cell</strong></td>
<td>Optional</td>
<td>Not Available</td>
<td>Not Available</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Photocalorimeter</strong></td>
<td>Optional</td>
<td>Optional</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Digital Mass Flow Controller</strong></td>
<td>Included</td>
<td>Included</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>50-Position Autosampler</strong></td>
<td>Included</td>
<td>Optional</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Auto Lid</strong></td>
<td>Included</td>
<td>Included</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Temperature Accuracy</strong></td>
<td>± 0.1 °C</td>
<td>± 0.1 °C</td>
<td>± 0.1 °C</td>
<td>± 0.1 °C</td>
</tr>
<tr>
<td><strong>Temperature Precision</strong></td>
<td>± 0.01 °C</td>
<td>± 0.05 °C</td>
<td>± 0.05 °C</td>
<td>± 0.05 °C</td>
</tr>
<tr>
<td><strong>Temperature Range (with cooling accessory)</strong></td>
<td>-180 to 725 °C</td>
<td>-180 to 725 °C</td>
<td>-180 to 725 °C</td>
<td>-130 to 725 °C</td>
</tr>
<tr>
<td><strong>Calorimetric Precision (metal standards)</strong></td>
<td>± 0.05%</td>
<td>± 0.05%</td>
<td>± 1%</td>
<td>± 1%</td>
</tr>
<tr>
<td><strong>Sensitivity</strong></td>
<td>0.2 µW</td>
<td>0.2 µW</td>
<td>1.0 µW</td>
<td>1.0 µW</td>
</tr>
<tr>
<td><strong>Baseline Curvature with Tzero (-50 to 300 °C)</strong></td>
<td>10 µW</td>
<td>10 µW</td>
<td>Not Available</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Baseline Reproducibility with Tzero</strong></td>
<td>±10 µW</td>
<td>±10 µW</td>
<td>&lt;0.04 mW</td>
<td>Not Available</td>
</tr>
<tr>
<td><strong>Relative Resolution</strong></td>
<td>2.9</td>
<td>2.1</td>
<td>1.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>
**Tzero Cell Design**

The Tzero™ cell is designed for excellence in both heating and cooling operation. Its many innovations include a new sensor with raised sample and reference platforms. The sensor is machined for symmetry from a single piece of durable, thin wall, high response constantan and brazed to the silver heating block. **Benefits:** Provides faster signal response, flat baselines, superior sensitivity and resolution, plus improved data precision.

A new chromel/constantan Tzero thermocouple is located midway between the sample and reference sensor platforms. **Benefits:** It provides for independently measured sample and reference heat flows that produce superior DSC and MDSC results. It simultaneously acts as a control sensor to assure precise isothermal furnace operation.

Matched chromel area thermocouples are welded to the underside of each sensor platform and provide superior performance to other thermocouple designs. **Benefits:** High sensitivity detection of any temperature transition that results from a physical change in the sample.

**Auto Lid**

The Q1000 and Q100 have a new and improved auto lid assembly that consists of dual silver lids and a dome-shaped heat shield. The auto lid automatically covers and uncovers the cell as necessary. The Q10 lid assembly is manually operated. **Benefits:** More accurate measurements result from improved thermal isolation of the cell.
COOLING RODS & RING
The innovative design features an array of 54 symmetrically arranged, high conductivity, nickel cooling rods that connect the silver furnace with the cooling ring. **Benefits:** This provides superior cooling performance over a wide temperature range. High cooling rates and instantaneous turnaround from heating to cooling are now achievable. Lower subambient temperatures and unmatched baseline performance can now be obtained with our range of cooling accessories in isothermal, programmed cooling, and MDSC® experiments. Turnaround time between experiments is dramatically reduced.

FURNACE
The sample and reference platforms are surrounded by a high thermal conductivity, silver furnace, that uses rugged, long-life Platinel™ windings. Purge gases are accurately and precisely metered by mass flow controllers and uniformly heated to cell temperature, prior to introduction to the sample chamber. **Benefits:** The design provides a highly uniform thermal environment for the sample and reference. Precise temperature control algorithms deliver accurate isothermal temperatures, linear heating rates, rapid temperature response and the ability to heat at rates up to 200 °C / min. The rugged heater windings ensure long furnace life. Superior data quality results from the uniform purge gas flow.

Platinel™ is a trademark of Englehard Industries
AUTOSAMPLER

The patented DSC autosampler is a powerful performance and productivity enhancer for the Q Series™ DSC modules. It provides reliable, unattended operation of the Q1000 or Q100 DSC, even when using the RCS or LNCS cooling accessories. Its 50-sample, 5-reference pan carousel tray, enables research and analytical laboratories to analyze samples “around-the-clock.”

The sample arm controls loading and unloading of sample and reference pans in sequential or random order. An optical sensor guides the sample arm, ensuring precise pan placement and automatic calibration of the system. Maximum productivity from the DSC autosampler is achieved when paired with our intelligent Advantage™ software, that permits pre-programmed analysis, comparison, and presentation of results.

*D. Patent No. 6,644,136; 6,652,015; 6,760, 679; 6,823,278

DSC SAMPLE PANS

TA Instruments offers a wide selection of sample pans to meet multiple standard and specialized applications. These include aluminum, alodined aluminum, copper, gold, platinum, graphite and stainless steel. They can be used under a variety of temperature and pressure conditions. Samples can be run in the standard DSC mode in open pans, crimped or hermetically sealed pans / lids or in pressure capsules. Samples in open pans can also be run at controlled pressures using the PDSC Cell. All aluminum standard pans have the same temperature and pressure rating. General details of the pans are as follows:

<table>
<thead>
<tr>
<th>Standard</th>
<th>Aluminum Temperature (°C)</th>
<th>Copper Temperature (°C)</th>
<th>Platinum Temperature (°C)</th>
<th>Gold Temperature (°C)</th>
<th>Graphite Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>-180 to 600</td>
<td>-180 to 725</td>
<td>-180 to 725</td>
<td>-180 to 725</td>
<td>-180 to 725</td>
</tr>
<tr>
<td>Pressure</td>
<td>100 kPa</td>
<td>100 kPa</td>
<td>100 kPa</td>
<td>100 kPa</td>
<td>100 kPa</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Hermetic</th>
<th>Aluminum Temperature (°C)</th>
<th>Alodined Aluminum Temperature (°C)</th>
<th>Gold Temperature (°C)</th>
<th>Hi Volume Temperature (°C)</th>
<th>Pressure (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>-180 to 600</td>
<td>-180 to 600</td>
<td>-180 to 725</td>
<td>-100 to 250</td>
<td>3.7 MPa</td>
</tr>
<tr>
<td>Pressure</td>
<td>300 kPa</td>
<td>300 kPa</td>
<td>600 kPa</td>
<td>10 MPa</td>
<td>10 MPa</td>
</tr>
</tbody>
</table>
**Q10P Pressure DSC Module**

The Q10P is a dedicated pressure DSC system that provides heat flow measurements on pressure sensitive materials from -130 to 725 °C, at pressures from 1 Pa (0.01 torr) to 7 MPa (1,000 psi). The cell employs standard heat flux DSC technology and incorporates pressure control valves, a pressure gauge, and over-pressure protection. The pressure DSC cell is also an accessory for the Q1000 DSC and can be used as a standard cell from -180 to 725 °C.

**Photocalorimeter**

The Photocalorimeter Accessory (PCA), for the Q1000 and Q100 DSC, permits characterization of photocuring materials between -50 and 80 °C. UV/Visible light (250-600 nm) from a high pressure mercury source is transmitted to the sample chamber via an extended range, dual-liquid light guide with neutral density or band pass filters. Tzero™ technology permits direct measurement of light intensity. It also provides for simultaneous measurement of two samples. The PCA can also be equipped with a dual quartz light guide for operation up to 250 °C.

**Mass Flow Controllers**

High quality DSC experiments require precise purge gas flow rates, especially when using high conductivity gases, such as helium. Mass flow controllers, along with integrated gas switching, provide flexible control as part of individual methods. Purge gas flow rates are settable from 0-240 mL/min in increments of 1 mL/min. The system is precalibrated for helium, nitrogen, air and oxygen and suitable calibration factors may be entered for other gases.
**TEMPERATURE CONTROL OPTIONS**

---

**REFRIGERATED COOLING SYSTEM**

The Refrigerated Cooling System (RCS) is frequently selected as the cooling device of choice for trouble-free, unattended DSC and MDSC™ operation. It operates from -90 °C to 550 °C using a two-stage, closed, evaporative refrigerator system. Because it is a sealed system requiring only electrical power, the RCS is frequently preferred for operation in areas where other refrigerants, such as liquid nitrogen, are difficult or expensive to obtain. The RCS is compatible with Q1000, Q100, and Q10 DSC modules.

---

**LIQUID NITROGEN COOLING SYSTEM**

The Liquid Nitrogen Cooling System (LNCS) provides the highest performance and greatest flexibility in cooling. It has the lowest operational temperature (to -180 °C), greatest cooling rate capacity (to 140 °C/min), and an upper temperature limit of 550 °C. It is ideal for isothermal crystallization studies. The LNCS uses liquid nitrogen efficiently, thus reducing operating costs. Its autofill capability allows the LNCS to be automatically refilled from a larger liquid nitrogen source for continuous DSC operation. The LNCS is available for the Q1000, Q100, and Q10 DSC modules.
Finned Air Cooling System

The Finned Air Cooling System (FACS) is an innovative cooling accessory for the Q1000, Q100, and Q10 DSC modules that offers a cost-effective alternative to the refrigerated and liquid nitrogen cooling systems. The FACS can be used for controlled cooling experiments, thermal cycling studies, and to improve sample turnaround time. It uses compressed air to cool the DSC cell. Stable baselines and linear heating and cooling rates can be achieved between ambient and 725 °C. A special version of the Quench Cooling Accessory is available for use with the FACS to speed cooling of the DSC cell to ambient temperatures.

Quench Cooling Accessory

The Quench Cooling Accessory (QCA) is a manually operated cooling accessory that is a cost-effective alternative to the automated RCS or LNCS. Its primary use is with the Q10 DSC to quench cool a sample to a subambient temperature prior to heating to an upper limit. Since active cooling is not present in QCA experiments, the T1 signal is the measured entity. The recommended temperature of operation of the QCA is from –180 to 400 °C. The QCA reservoir is easily filled with ice water, liquid nitrogen, dry ice, or other cooling mixtures.
Tzero™ Technology Provides:

- Essentially flat baselines with better than an order of magnitude improvement on other designs, especially in the subambient temperature range
- Superior sensitivity due to flatter baselines and better signal-to-noise ratio
- Best available resolution (even over power compensation devices)
- Faster MDSC® experiments
- Direct measurement of heat capacity (Q1000)

Tzero® technology produces the truest available representation of heat flowing to and from a sample, by removing instrumental thermal effects that degrade baseline flatness, sensitivity, and resolution in other designs. The Tzero cell’s unique internal reference temperature sensor and electronic circuitry measure the resistive and capacitive imbalances that cause these effects. An advanced four-term, heat flow expression accounts for them, and also for known heating rate differences at the sample and reference that occur during major thermal events (e.g., melting). Tzero technology is available on the Q1000 and the Q100. Advanced Tzero (available only on the Q1000) compensates for pan contact resistance, thereby further improving resolution and allowing direct heat capacity (Cp) measurements.

*U.S. Patent No. 6,431,747; 6,488,406; 6,523,998
**Baseline Stability (Flatness)**

Figure 1 shows a comparison of a Q1000 empty cell baseline with that from a high performance, non-Tzero, heat flux DSC. The data shows that the Q1000 baseline is superior in every way. The start-up offset is much smaller, the baseline is dramatically straighter, and the slope is greatly reduced. Notice the heat flow scale, and that the signal is almost zero throughout the -80 to 400 °C temperature range. This also contrasts markedly with results from other DSC designs, where a baseline bow of 1 mW over the same temperature range is often considered acceptable.

**Sensitivity**

Figure 2 shows a Q1000 high sensitivity glass transition (Tg) measurement, as a function of heating rate, for a very small (1 mg) sample of polypropylene, whose Tg is not easily measured by DSC, even with a larger sample, due to its highly crystalline nature. The data shows that the Tg is easily detected even at a slow 5 °C/min heating rate. The excellent Q1000 baseline is the essential key for accurate measurements of glass transitions and heat capacity from materials that exhibit weak and broad transitions.

**Resolution**

Figure 3 shows a comparative resolution plot of indium performed on the Q Series DSC’s (Q1000, Q100, and Q10) under identical conditions. The Q10 data is typical of a good performing DSC system without Tzero™ technology. The improvements seen in the Q1000 and Q100 are impressive displays of the power of Tzero™ technology in the steeper leading edge trace and in the subsequent faster return to baseline. This is especially true in the Q1000, which outperforms power-compensated DSC models in recognized resolution tests.
MDSC® Technology Provides:

- Separation of complex transitions into more easily interpreted components
- Increased sensitivity for detecting weak transitions and melts
- Increased resolution without loss of sensitivity
- Direct measurement of heat capacity
- More accurate measurement of crystallinity

In MDSC*, a sinusoidal temperature oscillation is overlaid on the traditional linear ramp. The net effect is that heat flow can be measured simultaneously with changes in heat capacity. Using Fourier transformation, the heat flow generated is separated in real time into a heat capacity component and a kinetic component. In MDSC, the DSC heat flow is called the Total Heat Flow, the heat capacity component is the Reversing Heat Flow, and the kinetic component is the Nonreversing Heat Flow. The Total Heat Flow signal contains the sum of all thermal transitions, just as in standard DSC. The Reversing signal contains glass transition and melting transitions, while the Nonreversing Heat Flow contains kinetic events like curing, volatilization, melting, and decomposition. The Q1000 uniquely permits increased MDSC productivity of high quality data by its ability to operate at standard DSC heating rates (e.g., 10 °C / min.).

*U.S. Patent Nos. 5,224,775; 5,248,199; 5,346,306

\[
\frac{dH}{dt} = C_p \frac{dT}{dt} + f(t, T)
\]

Heat Flow = Heat Capacity Component + Kinetic Component
Total Heat Flow = Reversing Heat Flow + Nonreversing Heat Flow
**MDSC® APPLICATIONS**

**Separation of Complex Transitions**

Figure 4 shows MDSC® results for a copolymer of polyethylene terephthalate and acrylonitrile/butadiene/styrene (PET/ABS) when analyzed over the temperature range from ambient to 170 °C. The MDSC total heat flow signal shows only the PET glass transition and cold crystallization, with no evidence of the ABS. The reversing heat flow clearly identifies glass transitions for both PET and ABS. The non-reversing trace indicates the cold crystallization peak for PET, plus an enthalpic relaxation resulting from the sample’s previous history.

**Improved Signal Sensitivity**

Figure 5 shows the improved sensitivity of MDSC for measuring very broad and weak transitions, such as glass transitions in highly crystalline polymers or where the Tg is hidden beneath a second overlapping thermal event. This data was generated using a very small (2.2 mg) sample of a polymer coating. The total heat flow shows no transitions in the region where a Tg would be expected, though the large endotherm around 40 °C indicates solvent loss. The Reversing Heat Flow does indicate a very weak (8.5 µW) Tg around 109 °C, illustrating the sensitivity of the MDSC technique.

**Improved Data Interpretation**

Figure 6 shows an application of interest in studies of foods or pharmaceuticals, in which the MDSC total heat flow signal and its reversing and non-reversing components are displayed for a 40% aqueous sucrose sample. While the former is not easy to interpret, the reversing signal clearly indicates a Tg for sucrose between -43.6 and -39.4 °C. The exothermic non-reversing signal (peak max -36 °C; heat of crystallization 5.7 J/g) relates to crystallization of free water that could not crystallize during quench cooling of the sample due to a significant increase in mobility and diffusion of the material at the glass transition.
**Transition Temperatures**

DSC provides rapid and precise determinations of transition temperatures using minimum amounts of a sample. Common temperature measurements include the following:

- Melting
- Glass Transition
- Thermal Stability
- Oxidation Onset
- Cure Onset
- Crystallization
- Polymorphic Transition
- Liquid Crystal
- Protein Denaturation
- Solid-Solid Transition

*Figure 7* shows typical shapes for the main transitions observed in DSC.

**Heat Flow**

The DSC heat flow signal is commonly used to measure the following:

- Specific Heat Capacity
- Hazard Potential
- Lifetime Estimation
- Glass Transition
- Cure Rates
- Kinetics

*Figure 8* shows a single analysis measurement of total heat and heat capacity (Cp) by advanced DSC technology (see page 12). The heat capacity increases as the sample passes through its glass transition, cold crystallization, and melting events.

**Enthalpy**

Heat flow signal integration gives quantitative enthalpy information about the transition. For example:

- Heat of Fusion
- Percent Crystallinity
- Heat of Crystallization
- Explosion Potential
- Degree of Cure
- Heat of Reaction

*Figure 9* The DSC plot of a thermosetting resin allows determination of the heat of reaction and degree of cure. Specialty kinetics software can also provide the reaction order, activation energy, and reaction rates.
Photoncalorimeter Accessory (PCA) provides a convenient tool to assess reactions initiated with UV/Visible light. **Figure 12** compares two different acrylic formulations under the same conditions. The data shows that formulation A cures rapidly upon exposure to UV radiation, while formulation B reacts slower, and has both a longer time-to-peak and lower energy. In all PCA experiments, the peak shapes and transition energies are affected by the formulation chemistry, additives, initiators, and the purge gas used.

**Time**

Kinetics is the study of the effects of time and temperature on a reaction. Common ASTM test methods include reaction induction time (E2046), oxidation induction time (OIT; D3895), and constant temperature stability (E487). Comparative OIT tests permit relative ranking of the effectiveness of different anti-oxidant packages in a given polymer. As seen in **Figure 10**, the analyses are rapid, and with relative performance quickly established, the antioxidant selection can be made on needs, processing conditions, and relative cost.

**Pressure (and Time)**

Pressure DSC accelerates OIT analyses and "sharpens" the onset of the oxidation process. **Figure 11** shows a comparative study of a series of two component polymer dispersions containing different levels of the same antioxidant. Clear performance differences are readily seen. The tests provided the same answer in under two days that took up to two months of traditional "field exposure" to obtain. Other common PDSC applications include a) thermoset resin cures, b) catalyst studies, and c) micro-scale simulations of chemical reactions.

**Photocuring**

The Photocalorimeter Accessory (PCA) provides a convenient tool to assess reactions initiated with UV/Visible light. **Figure 12** compares two different acrylic formulations under the same conditions. The data shows that formulation A cures rapidly upon exposure to UV radiation, while formulation B reacts slower, and has both a longer time-to-peak and lower energy. In all PCA experiments, the peak shapes and transition energies are affected by the formulation chemistry, additives, initiators, and the purge gas used.
THERMOGRAVIMETRIC ANALYZERS

TA Instruments Q500 and Q50 Thermogravimetric Analyzers are recognized for their sensitivity, precision, reliability and automation, features that are amplified in our latest product, the remarkable Q5000. Its design confirms our commitment to continuously improve the capabilities of traditional products (Q5000 IR) and to provide innovative new ways to characterize materials (Q5000 SA). All Q TGAs deliver superb value and are backed by the industry’s leading support team.
TA INNOVATIONS

Vertical Balance & Horizontal Gas Purge

Hi-Res TGA™

Modulated TGA™

IR Heating

High Heating Rate TGA

Autosampler with patented pan punching mechanism

Humidity Chamber

Integrated Curie Point temperature calibration
**Q5000 IR**

The new, highly automated Q5000 IR is designed to be the TGA best suited to meet the most demanding research applications. It delivers superior baseline flatness, high sensitivity, accurate temperature measurement, and flexibility in both standard and high heating rate operation. New features include an infrared furnace, vacuum operation, greatly simplified Curie Point temperature calibration, powerful new Advantage software with automatic calibration, verification and diagnostic capability, and a contamination free multi-position autosampler with a patented contamination free, sealed pan-punching mechanism.

**Q5000 SA**

The new automated Q5000 SA is designed for high performance sorption analysis of materials under controlled conditions of temperature and humidity. Its design integrates our latest high sensitivity, temperature controlled thermo-balance with an innovative humidity generation system, advanced multi-position autosampler, and powerful Advantage software with technique specific programs. The Q5000 SA delivers the performance and reliability required in a leading sorption analyzer and in a compact, user-friendly design.
## Technical Specifications

### Q5000IR

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermobalance temperature</td>
<td>Included</td>
</tr>
<tr>
<td>controlled</td>
<td></td>
</tr>
<tr>
<td>Weight Range</td>
<td>0.1 g</td>
</tr>
<tr>
<td>Weighing Accuracy</td>
<td>+/- 0.1%</td>
</tr>
<tr>
<td>Weighing precision</td>
<td>+/- 0.01%</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>&lt; 0.1 µg</td>
</tr>
<tr>
<td>Baseline dynamic drift*</td>
<td>&lt; 10 µg</td>
</tr>
<tr>
<td>Signal Resolution</td>
<td>0.01 µg</td>
</tr>
<tr>
<td>Furnace heating</td>
<td>Infrared</td>
</tr>
<tr>
<td>Temperature range</td>
<td>Ambient to 1200 ºC</td>
</tr>
<tr>
<td>Isothermal Temp Accuracy</td>
<td>+/- 1 ºC</td>
</tr>
<tr>
<td>Linear Heating Rate (*C/min)</td>
<td>0.1 to 500</td>
</tr>
<tr>
<td>Furnace Cooling (forced air/N2)</td>
<td>1200 to 35 ºC &lt; 10 min</td>
</tr>
<tr>
<td>Vacuum</td>
<td>10⁻¹ torr</td>
</tr>
<tr>
<td>Temperature calibration</td>
<td>Electromagnetic Coil/Curie Point Stds</td>
</tr>
<tr>
<td>Autosampler – 25 sample</td>
<td>Included</td>
</tr>
<tr>
<td>Hi-Res TGA™</td>
<td>Included</td>
</tr>
<tr>
<td>Auto Stepwise</td>
<td>Included</td>
</tr>
<tr>
<td>Modulated TGA™</td>
<td>Included</td>
</tr>
<tr>
<td>TGA/MS Operation</td>
<td>Option</td>
</tr>
<tr>
<td>Sample Pans</td>
<td>Platinum 50, 100 µL</td>
</tr>
<tr>
<td></td>
<td>Ceramic 100, 250 µL</td>
</tr>
<tr>
<td></td>
<td>Aluminum 80 µL</td>
</tr>
<tr>
<td></td>
<td>Aluminum Sealed Pan 20 µL</td>
</tr>
</tbody>
</table>

* From 50 to 1000 ºC at 20 ºC/min using empty platinum pans

### Q5000SA

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermobalance temperature</td>
<td>Included</td>
</tr>
<tr>
<td>controlled</td>
<td></td>
</tr>
<tr>
<td>Weight Range</td>
<td>0.1 g (nom)</td>
</tr>
<tr>
<td>Weighing Accuracy</td>
<td>+/- 0.1%</td>
</tr>
<tr>
<td>Weighing precision</td>
<td>+/- 0.01%</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>&lt; 0.1 µg</td>
</tr>
<tr>
<td>Baseline**</td>
<td>&lt; 5 µg</td>
</tr>
<tr>
<td>Signal Resolution</td>
<td>0.01 µg</td>
</tr>
<tr>
<td>Temperature Control</td>
<td>Peltier Elements</td>
</tr>
<tr>
<td>Temperature Range</td>
<td>5 to 85 ºC</td>
</tr>
<tr>
<td>Isothermal Stability</td>
<td>+/- 0.1 ºC</td>
</tr>
<tr>
<td>Relative Humidity Control Range</td>
<td>0 to 98%RH</td>
</tr>
<tr>
<td>Accuracy</td>
<td>+/- 1% RH</td>
</tr>
<tr>
<td>Autosampler – 10 samples***</td>
<td>Included</td>
</tr>
<tr>
<td>Sample Pans</td>
<td>Quartz 180 µL</td>
</tr>
<tr>
<td></td>
<td>Platinum 50, 100 µL</td>
</tr>
<tr>
<td></td>
<td>Aluminum Sealed Pan 20 µL</td>
</tr>
</tbody>
</table>

** Over 24 hours at 25º C and 20% RH with empty quartz pans

*** Optional tray accommodates 25 samples for use with platinum and sealed aluminum pans
THERMOGRAVIMETRIC ANALYZERS

Q500

The Q500 is a research grade thermogravimetric analyzer, whose leading performance arises from a responsive low-mass furnace; sensitive thermobalance, and efficient horizontal purge gas system (with mass flow control). Its convenience, expandability and powerful, results-oriented software make the Q500 perfect for the multi-user laboratory where a wide variety of TGA applications are conducted and where future expansion of analytical work is anticipated.

Q50

The easy-to-use Q50 is a rugged, reliable, cost-effective TGA with many features of the Q500. It offers outstanding value as a compact, general-purpose thermogravimetric analyzer that frequently outperforms competitive research-grade models. Its integral mass flow control, gas switching capability, and superb software make the Q50 ideal for academic teaching facilities or in industrial laboratories that need quality results at a modest cost.
### Technical Specifications

<table>
<thead>
<tr>
<th></th>
<th>Q500</th>
<th>Q50</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermobalance-</td>
<td>Included</td>
<td>Included</td>
</tr>
<tr>
<td>Temperature Compensated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Sample Weight</td>
<td>1 g</td>
<td>1 g</td>
</tr>
<tr>
<td>Weighing Precision</td>
<td>+/- 0.01%</td>
<td>+/- 0.01%</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>0.1 µg</td>
<td>0.1 µg</td>
</tr>
<tr>
<td>Baseline Dynamic Drift*</td>
<td>&lt; 50 µg</td>
<td>&lt; 50 µg</td>
</tr>
<tr>
<td>Furnace Heating</td>
<td>Resistance</td>
<td>Resistance</td>
</tr>
<tr>
<td>EGA Furnace</td>
<td>Optional</td>
<td>Optional</td>
</tr>
<tr>
<td>Temperature range</td>
<td>Ambient to 1000 ºC</td>
<td>Ambient to 1000 ºC</td>
</tr>
<tr>
<td>Isothermal Temp Accuracy</td>
<td>+/- 1 ºC</td>
<td>+/- 1 ºC</td>
</tr>
<tr>
<td>Isothermal Temp Precision</td>
<td>+/- 0.1 ºC</td>
<td>+/- 0.1 ºC</td>
</tr>
<tr>
<td>Controlled Heating Rate (ºC/min)</td>
<td>0.01 to 100</td>
<td>0.1 to 100</td>
</tr>
<tr>
<td>Furnace Cooling (forced air / N2)</td>
<td>1000 to 50 ºC &lt; 12 min</td>
<td>1000 to 50 ºC &lt; 12 min</td>
</tr>
<tr>
<td>Temperature calibration</td>
<td>Curie Point</td>
<td>Curie Point</td>
</tr>
<tr>
<td>Autosampler-16 sample</td>
<td>Optional</td>
<td>Not Available</td>
</tr>
<tr>
<td>Hi-Res TGA™</td>
<td>Optional</td>
<td>Not Available</td>
</tr>
<tr>
<td>Auto Stepwise TGA</td>
<td>Included</td>
<td>Included</td>
</tr>
<tr>
<td>Modulated TGA™</td>
<td>Optional</td>
<td>Not Available</td>
</tr>
<tr>
<td>TGA/MS Operation</td>
<td>Optional</td>
<td>Optional</td>
</tr>
<tr>
<td>Sample Pans</td>
<td>Platinum 50, 100 µL</td>
<td>Ceramic 100, 250, 500 µL</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Aluminum 100 µL</td>
</tr>
</tbody>
</table>

* From 50 to 1000 ºC at 20 ºC/min using empty platinum pans
Q5000IR Technology

The highly automated Q5000 IR offers unmatched baseline flatness, high sensitivity, accurate temperature measurement, and precise control in both standard and high heating rate operation. Other great features include vacuum operation, automated Curie Point temperature calibration, powerful new software and a 25-position, smart autosampler with a unique contamination-free, sealed pan punching mechanism. It is clearly the best analyzer for complex TGA applications.
1 THERMOBALANCE

The heart of the Q5000 IR is our latest 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 an optically active servo loop that maintains the balance arm in the horizontal reference (null) position by regulating current in a transducer coil. An infrared LED source and a matched photodiode pair detect any weight change as a beam imbalance. The net photodiode output feeds a control program that electrically “nulls” or “re-zeros” the balance with the current required being directly proportional to the weight change. The electrically grounded meter movement is connected to a rigid, lightweight metallic frame for increased stability.

Benefits: The patented design provides smooth, reliable operation over the entire ambient to 1200 °C temperature range, with unmatched dynamic baseline flatness and exceptional accuracy and precision in weight change detection, free from any vapor condensation or electrostatic forces.

2 AUTOSAMPLER

The integrated Q5000 IR Autosampler features a programmable, 25-sample 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, including pan taring and loading, sample weighing, autosampler movement, furnace movement, pan unloading and furnace cooling. Autosampler productivity is maximized by our Advantage™ software, which delivers pre-programmed analysis, comparison, and presentation of results.

The patented autosampler design provides smooth and efficient pick-up and subsequent release of the sample pan without disturbing the balance. The carousel accommodates platinum, ceramic, and aluminum pans. A special autosampler feature is the pan punching mechanism designed to reliably open sealed aluminum pans used to protect atmosphere sensitive samples. This patented punching mechanism is force controlled, reliable, and contamination free. A special detection circuit prevents a sealed pan from being placed into the furnace.
3 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. Radiation from four symmetrically placed lamps is reflected by the gold plated, multi-elliptical, internal furnace surfaces and focused on a silicon carbide sample enclosure. The cylindrical enclosure ensures uniform heating of the sample and pan. The furnace interior features a quartz liner, upper and lower heat shields, and a unique control and measurement area thermocouple assembly. Other new features include an integrated electromagnetic coil, forced gas furnace cooling, and vacuum operation.

Benefits: The dynamic furnace response offers outstanding performance and flexibility in standard, advanced (Hi-Res™ TGA; Modulated TGA™), and high heating rate experiments. The integral autosampler, rapid heating and swift cooling significantly enhance productivity. The quartz liner’s chemical inertness negates the need for special furnaces. It and the heat shields are easily cleaned. Vacuum operation improves resolution of closely related events. The thermocouple ensures tight furnace temperature control and provides accurate transition temperature measurement, while a second “safety” thermocouple disables the furnace if a temperature differential exceeds a set value. The ease of Curie Point temperature calibration is a unique Q5000 benefit.

4 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 thermobalance / furnace design. A regulated portion of the gas is also directed through the balance chamber and the combined gases plus any sample effluent exit the system by a side arm that can be readily connected to a MS or FTIR. Digital mass flow controllers 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.

Benefits: The design optimizes TGA performance and provides efficient removal of decomposition products from the sample area. Automatic buoyancy corrections provide more accurate weight change data, while the mass flow controllers improve data quality. Gas flow rates are stored data file signals.
The new Q5000 SA permits sorption / desorption analysis of materials under controlled conditions of temperature and humidity. Its design integrates our latest high sensitivity, temperature controlled thermobalance with an innovative humidity generation system, advanced autosampler, and excellent software. The Q5000 SA delivers the performance and reliability required in a leading sorption analyzer and in a compact, modern, user-friendly design.
1 **THERMOBALANCE**

The heart of the Q5000 SA is our latest high performance thermobalance maintained at a constant 35.00 °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 employs an optically active servo loop that maintains the balance arm in the horizontal reference (null) position by regulating current in a transducer coil. An infrared LED source and a matched photodiode pair detect any weight change as a beam imbalance. The net photodiode output feeds a control program that electrically “nulls” or “re-zeros” the balance with the current required being directly proportional to the weight change. The electrically grounded meter movement is neatly integrated into a rigid, lightweight metallic frame for increased stability. A key feature of the design for sorption analysis operation is the perfect symmetry of the balance assembly.

**Benefits:** The design provides smooth, reliable operation with superior dynamic baseline flatness, exceptional accuracy and precision in weight change detection, factors that are critical for proper gravimetric sorption analysis performance and are totally free from any vapor condensation or electrostatic forces.

2 **AUTOSAMPLER**

The integral Q5000 SA Autosampler features a programmable multi-position sample carousel that permits automated analysis of up to 10 samples using hemi-spherical quartz crucibles, and 25 samples using the optional Q5000 IR tray and platinum or sealed aluminum pans. The design provides smooth and efficient pick-up and subsequent release of the sample pan without disturbing the balance. All aspects of sample testing are automated and software controlled, including pan taring and loading, sample weighing, autosampler movement, furnace movement, pan unloading and furnace cooling. Autosampler productivity is software maximized by our Advantage™ software, which provides pre-programmed analysis, comparison, and presentation of results.
3 Humidity Control Chamber

The design features a pair of mass flow controllers (MFCs) that accurately meter and proportion gas to a symmetrical, well-insulated, aluminum block. This contains a humidifier, gas transmission and mixing lines, plus easily accessed, identically arranged, sample and reference measurement chambers. Temperature regulation of the block interior from 5 to 85 °C is performed by four thermo-electric (Peltier) devices in conjunction with a thermistor in a closed-loop system. A pair of precise, calibrated mass flow controllers (MFC’s) adjust the relative amounts of wet (saturated) and dry gas to obtain humidities from 0 to 98% RH. Identical sensors, pre-calibrated against NIST traceable equipment, are located adjacent to the sample and reference crucibles, and provide a continuous indication of humidity.
Benefits: The design provides precise temperature control and a highly consistent atmosphere within the sample and reference chambers. Peltier temperature control is more efficient than an external temperature controlled fluid bath. MFCs deliver continuous, precise gas stream metering and proportioning. The calibrated sensors negate the need for an internal chilled mirror device. The design simplifies sample loading and the autosampler maximizes productivity.
SENSITIVE, PRECISE, RUGGED AND AUTOMATED ARE WORDS THAT DESCRIBE A TA INSTRUMENTS THERMOGRAVIMETRIC ANALYZER (TGA). 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.
1 **Furnace**

Our custom-designed furnace is a key element of a Q Series 500/50 TGA. It features low mass, rugged heater windings, and proprietary heater control technology. **Benefits:** Include rapid, accurate, and precise temperature programming over a wide range, plus optimized use in the Q500 of advanced techniques such as Hi-Res™ TGA and Modulated TGA™. Our reliable, long-life furnaces also increase the value of your investment.

2 **Thermobalance**

The heart of a Q500/Q50 TGA is the accurate, and reliable, vertical thermobalance housed in a temperature controlled environment. It uses the field proven, null-balance principle, where an optically active servo loop maintains the balance arm in the horizontal reference (null) position by current regulation in a transducer coil. An infrared LED source and a matched photodiode pair detect beam movement, while a flag attached to the balance arm controls the amount of light reaching each photodiode. A sample weight change unbalances the beam, and the net photodiode output feeds a control program, which electrically “nulls” the balance. The current required is directly proportional to the weight change. The design permits automatic switching between the dual 0-200 mg/0-1 g weight ranges. **Benefits:** The design provides the best accuracy and precision in weight change detection from ambient to 1000 °C, low baseline drift, and smooth, reliable operation over the entire weight range.

**Purge Gas System**

An efficient horizontal purge gas system, that allows accurately metered purge gas to flow directly across the sample, is expertly integrated into the vertical thermobalance / furnace design. A regulated portion of the gas is also directed through the balance chamber and the combined gases plus any sample effluent exit the system by a side arm. **Benefits:** The design minimizes buoyancy effects, and optimizes removal of decomposition products from the sample area. The side arm exit port design makes for easy connection to a MS or FTIR. The digital mass flow controllers improve data quality.

**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 in the same sleeve 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 serves to automatically disable the furnace should the temperature difference between the thermocouples exceed a set value.
**AUTOSAMPLER**

The Q500 Autosampler accessory is a programmable, multi-position sample carousel that allows automated analysis of up to 16 samples. All aspects of sample testing are automated and software controlled, including pan taring and loading, sample weighing, furnace movement, pan unloading, and furnace cooling. The autosampler has the flexibility to meet the needs of both research and QC laboratories. Autosampler productivity is maximized by our Advantage™ software, which permits pre-programmed analysis, comparison, and presentation of results.

**EVPOLVED GAS ANALYSIS (EGA) FURNACE**

The rugged and reliable EGA is an optional, quartz-lined furnace for the Q500 or Q50. The liner is essentially chemically inert to products produced from decomposition of the sample, 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.

TA Instruments offers a 300 amu bench-top, triple-filter, quadrupole mass spectrometer, and TGA module-specific interface kits. The decomposed gaseous products exit the TGA and pass down a heated quartz-lined stainless steel capitary to the Mass Spectrometer. A searchable MS database is available. A variety of FTIR suppliers provide gas cells and interfaces for use with all our TGA modules.
**MASS FLOW CONTROL (WITH AUTOMATIC GAS SWITCHING)**

The digital mass flow controllers provide accurate and precise purge gas metering that consistently exceeds conventional flow control devices for superior data quality. The automatic low volume, high-speed switching valves deliver instantaneous change of purge gas that is critical when converting between inert and oxidizing atmospheres. The gas flow rates are stored as a data file signal.

**TEMPERATURE CALIBRATION AND WEIGHT LOSS VERIFICATION**

TA Instruments offers the widest range of ICTAC certified and NIST traceable Curie Point reference materials that permit temperature calibration of the Q Series TGA modules over the range from 150 to 1120 °C. The advanced Q5000 IR has an internal electromagnet coil that permits temperature calibration with unprecedented convenience (see page 39).

TA Instruments also offers certified Mass Loss Reference Materials for validation of instrument performance (see page 44).
Advanced TGA Techniques

TA Instruments has been the main commercial 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 good 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 tight 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 optionally available for the Q500. Each offers specific advantages in resolution, but the dynamic rate technique is simpler to use, requires less operator expertise, and generates high quality results faster than the other methods. This capability is particularly valuable in analytical methods development. Auto-stepwise isothermal is a third high resolution technique, and is supplied with all the Q Series TGA models.

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 No. 6,113,261 and 6,336,741
**Q5000IR**

Platinum (50 and 100 µL), ceramic (100 and 250 µL), and aluminum open (80 µL) and sealed (20 µL) pans are available for the Q5000 TGA. Platinum is recommended for most applications (ambient to 1,000 °C) for its inertness and ease of cleaning. For operation to 1200 °C the larger ceramic pans (with ceramic bale) are recommended. The larger pan is best for higher volume / low density samples such as foams. The ceramic pans are also advised for samples that react with or form amalgams with platinum. The aluminum pans are cost-effective substitute pans but cannot be used above 600 °C. These new pans are designed exclusively for the Q5000 IR.

**Q5000SA**

Hemispherical metallicized quartz crucibles (180 µL) and optional platinum (50 and 100 µL) TGA pans are available for use with the Q5000 SA. Quartz crucibles are commonly used in sorption analysis because of their chemical inertness and ease of cleaning, while Platinum pans are generic for both SA and TGA analyses. Sealed aluminum pans are also an option for ensuring the integrity of materials which readily adsorb moisture or lose volatiles.

**Q500 / Q50**

Platinum (50 and 100 µL), and new style ceramic (100, 250, and 500 µL) pans are available for use with the Q500 and Q50 TGA modules from ambient to 1,000 °C. Platinum is 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 amalgams with platinum. The aluminum (100 µL) pans are cost-effective substitute pans but cannot be used above 600 °C.
**APPLICATIONS**

**HIGH SENSITIVITY VOLATILES ANALYSIS**

Unwanted water or other volatiles in a formulation can wreak havoc during product processing. The improved sensitivity of the Q5000 IR allows even small amounts of these undesirable components to be quantified. Special new aluminum hatch-top pans allow moisture sensitive samples to be sealed and queued in the autosampler until moments before analysis. **Figure 1** shows the volatiles analysis for a small (2.4 mg) sample of polyethylene terephthalate (PET) bottle stock. The 0.2% weight change reflects an absolute weight loss of only 5.2 micrograms!

**CHARACTERIZATION OF HYDRATES**

Pharmaceutical scientists routinely characterize the thermodynamic parameters of drug candidates. **Figure 2** shows the determination of the hydrate decomposition temperature for lactose monohydrate, an excipient material. The sample was sealed in a capsule having a 20 micron hole to suppress weight loss until the equilibrium vapor pressure is reached. Use of radiation heating in the Q5000 IR results in improved temperature response while maintaining excellent resolution.

**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. **Figure 3** 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!
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. Figure 4 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.

Flame Retardant Test

The addition of flame retardant additives to a product is a common requirement for materials produced or imported into most developed countries today. In practice, the flame retardant additive functions to moderate oxidative decomposition of materials by evolving a non-flammable component, which blankets the material as it decomposes. Use of a fast scanning rate capability of the Q5000 IR better simulates the conditions of a fire. Figure 5 shows TGA of polypropylene with, and without, a flame retardant additive run in the Q5000 TGA at 500 °C/min in air. The degree of effectiveness of the flame retardant is seen as the decomposed material effectively “smothers” the unreacted sample and prevents further oxidation.

Simplified Curie Point Temperature Calibration

The electromagnetic coil integrated into the Q5000 IR furnace greatly simplifies Curie Point calibration measurements. This is a selectable method segment that can be changed in the course of an experiment so that Curie Temp materials of differing magnetic properties can be characterized in the same experiment.
Gravimetric Moisture Sorption Analysis – General Practice

Moisture Sorption Analysis is an established technique for determining the effect on materials of exposure to controlled conditions of temperature and humidity. Isotherm and Isohum experiments are the most common analyses.

In isotherm experiments, a weighed sample is “dried” externally, or preferably in the instrument, and exposed to a series of humidity step changes at constant temperature. The sample is staged at each humidity level until no further weight change is detected or a set time has elapsed. A data point is recorded, the humidity is changed in 5 or 10% RH steps and the process repeated in an upward and/or downward procedure. Isohum experiments involve a series of temperature step changes at constant humidity and result in similar plots. They are used to determine how sample exposure to a given humidity results in a physiochemical change, such as a change in the sample’s hydration state. The curve shape provides useful information to this end.

The Q5000 SA analysis software offers Sorption Analysis, BET Analysis, and GAB programs. In addition, the full power and flexibility of our renowned Universal Analysis provides easy data manipulation, advanced reporting, plotting and file exporting capabilities, plus several new user convenience features such as system diagnostics, e-mail results notification and web-mail update notification of new software features.

The Q5000 SA is factory calibrated for weight, temperature, and humidity. Deliquescent salts (e.g., NaCl) are widely accepted as standards for periodic verification of system performance. TA Instruments Platinum Software with scheduling and autoanalysis features simplifies the verification process.

Deliquescence

Deliquescence is a common behavior encountered in sorption analysis. The humidity at which deliquescence occurs can be determined several ways including ramping the humidity, plotting the rate of change in mass with respect to rate of the change in humidity (\(\frac{dm}{d\%RH}\)), and detecting where that curve crosses zero (i.e., the %RH where the sample is in equilibrium with its environment) while decreasing humidity from above the deliquescence point. Determination of deliquescence using ramped humidity takes less than 3 hours and yields excellent results even for materials like potassium sulfate which deliquesces near 100% RH at 25°C.
**Evaluation of Small Samples**

When evaluating pharmaceuticals particularly in the active ingredient screening & preformulation stage, it is common for only small amounts of material to be available for conducting multiple analytical tests including sorption analysis. Hence, the ability to work with small samples is beneficial. The low baseline drift of the Q5000SA means that good results can be obtained on even 10-20 milligrams of crystalline drugs such as prednisone which adsorb <1% moisture over a broad humidity range. The sorption results shown in Figure 2 represent about 15 micrograms of weight change full-scale.

**Optimizing Drying Conditions**

Sorption analysis results can also be used to determine processing (drying) conditions for materials. In this case, the objective is to determine the lowest humidity which can be used at a specific drying temperature without changing the structure of the material. The results are obtained by stepping humidity from a high starting humidity to lower humidities (right to left in Figure 3). The humidity where a significant weight change occurs indicates loss of the water of hydration. At 25 °C drying temperature, humidities as low as 10% RH can be used. At 40 °C and above, the humidity during drying must be at least 15%RH to avoid issues. The impact of sample form (milled versus unmilled) has little effect on the drying properties.

**Evaluation of Hygroscopic Materials**

Polyvinyl Pyrrolidone (PVP) is a water soluble polymer commonly used as a performance validation tool for sorption analyzers. Figure 4 shows a comparison of results obtained on a dried PVP sample after exposure to lab conditions for 8 and 24 hours versus a dried sample sealed for 24 hours in a special Q5000SA pan that is automatically opened (punched) just prior to analysis. Because PVP is hygroscopic, only the “sealed” sample yields the expected weight gain (42 ± 2 wt%).
**HIGH RESOLUTION™ TGA**

Figure 5 shows comparative decomposition profiles of a polyvinyl acetate performed by standard, stepwise isothermal (SWI), and dynamic TGA techniques. The superior resolution provided by the latter pair is obvious. While the SWI method provided the separation with highest resolution for this sample, the dynamic technique produced comparable results in a fraction of the time needed to develop the SWI method, and also provided the high-resolution analysis much faster than the standard TGA method.

**HIGH RESOLUTION™ TGA**

Figure 6 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 integral and first derivative 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™**

Figure 7 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 optionally available with the Q500.
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

**Thermal Stability**

TGA is often used to determine sample thermal stability and to reveal weight-loss decomposition profiles. Figure 8 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.

**Composition 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. Figure 9 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.
**Applications**

**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 (Figure 10) 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 moisture at the salt surface or held to it by weak attractive forces.

**EFFECT OF ADDITIVES**

Figure 11 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 retardent material is to inhibit flame propagation.

**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 verification of instrument performance. These are 2 %, 50 % and 98 % solutions of 2-ethoxyethylacetate (b.p.150 °C) in a stable higher boiling polyol. Figure 12 shows a plot of the decomposition profiles of 2-ethoxyethylacetate at all three concentrations. The data shows that there is no interference from the polyol in the determination.
**Dynamic Baseline Stability**

Figure 13 shows a set of replicate Q5000 IR empty pan baseline runs from 50 °C to 950 °C. Baseline drift in all cases was considerably below 10 µg, and the reproducibility at this level of performance was excellent! 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.

**Curie Point Calibration - Electromagnet**

Figure 14 shows an overlay plot of a series of ICTAC certified, NIST traceable Curie Point reference materials used to calibrate a TGA apparatus. The data was developed 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.

**Isothermal Baseline Stability**

Figure 15 demonstrates the Q5000IR’s excellent isothermal baseline stability. Four experiments on separate days, over 18 hours yielded an isothermal drift of less than one microgram.
SENSITIVE, RUGGED AND RELIABLE ARE WORDS THAT DESCRIBE THE TA INSTRUMENTS SDT Q600, A SECOND GENERATION SIMULTANEOUS TGA / DSC THAT PROVIDES RESEARCH QUALITY RESULTS ON A BROAD RANGE OF SAMPLES OVER A WIDE TEMPERATURE RANGE. ITS STRONG PERFORMANCE, EASE-OF-USE, AND OUR UNMATCHED SUPPORT MAKE THE Q600 AN EXCELLENT INVESTMENT FOR ANY LABORATORY.
TA INNOVATIONS

True simultaneous TGA / DTA

TGA / DSC measurement

Horizontal, gas purge

Unique dual balance design

Automatic compensation for beam growth

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

<table>
<thead>
<tr>
<th>System Design</th>
<th>Horizontal Balance &amp; Furnace</th>
</tr>
</thead>
<tbody>
<tr>
<td>Balance Design</td>
<td>Dual Beam</td>
</tr>
<tr>
<td>Sample Capacity</td>
<td>200 mg (350 mg including sample holder)</td>
</tr>
<tr>
<td>Balance Sensitivity</td>
<td>0.1 µg</td>
</tr>
<tr>
<td>Furnace Type</td>
<td>Bifilar Wound</td>
</tr>
<tr>
<td>Temperature Range</td>
<td>Ambient to 1500 °C</td>
</tr>
<tr>
<td>Heating rate – Ambient to 1000 °C</td>
<td>0.1 to 100 °C/ min</td>
</tr>
<tr>
<td>Heating rate – Ambient to 1500 °C</td>
<td>0.1 to 25 °C/ min</td>
</tr>
<tr>
<td>Furnace Cooling</td>
<td>Forced Air (1500 to 50 °C in &lt; 30 min)</td>
</tr>
<tr>
<td>Thermocouples</td>
<td>Platinum/Platinum-Rhodium (Type R)</td>
</tr>
<tr>
<td>Temperature Calibration</td>
<td>Metal Standards (1 to 5 Points) or Curie Materials</td>
</tr>
<tr>
<td>DTA Sensitivity</td>
<td>0.001 °C</td>
</tr>
<tr>
<td>Calorimetric Accuracy/Precision</td>
<td>± 2% (based on metal standards)</td>
</tr>
<tr>
<td>Mass Flow Controller with Automatic Gas Switching</td>
<td>Included</td>
</tr>
<tr>
<td>Vacuum</td>
<td>to 7 Pa (0.05 torr)</td>
</tr>
<tr>
<td>Reactive Gas Capability</td>
<td>Included – separate gas tube</td>
</tr>
<tr>
<td>Dual Sample TGA</td>
<td>Included</td>
</tr>
<tr>
<td>Auto-Stepwise TGA</td>
<td>Included</td>
</tr>
<tr>
<td>Sample Pans</td>
<td>Platinum: 40 µL, 110 µL</td>
</tr>
<tr>
<td></td>
<td>Alumina: 40 µL, 90 µL</td>
</tr>
</tbody>
</table>
**Thermobalance**

The Q600 has an accurate, and reliable, horizontal dual-balance mechanism that supports both DSC and TGA measurements. The sample balance monitors actual sample weight, while the reference balance is used to correct the TGA measurement for beam growth. **Benefits:** Design provides high sensitivity, accuracy, and precision in detecting very small weight changes (0.1µg). The dual-beam design results in less drift compared to single-beam designs, improving accuracy and precision. It also uniquely permits independent TGA measurements on two samples simultaneously.

**Temperature Control and Measurement**

A matched Platinum / Platinum-Rhodium thermocouple pair embedded in the ceramic beams provides direct sample, reference, and differential temperature from ambient to 1,500 °C. Single or multiple point temperature calibration can be done with Curie Point materials or high-purity metal standards. Sapphire is used to calibrate the Q600 for DSC operation. **Benefits:** Accurate and reproducible (to 0.001 °C) sample and reference temperature measurements ensure the most sensitive detection of thermal events. The dual-beam/dual-thermocouple design provides superior ΔT accuracy compared to single-beam designs that locate the reference thermocouple in the furnace. Calibration of the DSC signal using sapphire results in DSC baseline performance superior to competitive SDT instruments.
**Furnace**

A Q600 feature is the reliable operation of its rugged, bifilar-wound furnace, which moves horizontally on a motor-driven screw assembly. An air-cooling feature provides for automatic post-test cooling of the furnace. **Benefits:** Design allows for accurate and precise temperature programming over a wide range, with smooth automatic furnace opening/closing, easy sample loading, and rapid post-experiment furnace cool-down for increased analysis productivity.

**Purge Gas System**

The Q600 uses a field-proven, horizontal purge gas system with digital mass flow controllers and gas switching capability. Accurately metered purge gas flows horizontally through the furnace and directly across the sample and reference pans prior to exiting the analyzer. A separate Inconel® tube allows the use of reactive gases. The exit port can be directly interfaced to a MS or FTIR. **Benefits:** Design provides better baselines, minimizes buoyancy effects, prevents back diffusion, and efficiently removes decomposition products from the sample area. The Inconel tube permits special experiments using oxidizing or reactive gases.

© Inconel is a registered trademark of INCO
MASS FLOW CONTROLLER  
(WITH AUTOMATIC GAS SWITCHING)

The digital mass flow controller provides accurate and precise purge gas metering that exceeds conventional flow control devices for superior data quality. The low volume, high-speed switching valves deliver instantaneous change of purge gas that is critical when converting between inert and oxidizing atmospheres. The purge gas flow rates are stored data file signals.

HIGH RESOLUTION SDT

If separation of closely related weight losses cannot be obtained by standard conditions (linear heating rate programming between a starting and final temperature), the Q600 offers an automated version of Stepwise Isothermal (SWI), the classical technique for improved TGA resolution. It involves first studying the total decomposition profile under standard conditions, and using the information to develop a series of isothermal steps where heating stops once an operator defined weight loss rate is exceeded, then restarts after this rate falls below a second defined value. The result is optimum weight loss resolution.

Q600 SAMPLE PANS

Platinum pans (40 and 110 µL), and ceramic cups (40 and 90 µL) are available for use with the Q600. The platinum cups are recommended for operation to 1000 ºC, and for their general inertness and ease of cleaning. The ceramic cups are advised for operation to 1,500 ºC, and for samples that react with platinum.

TEMPERATURE CALIBRATION AND WEIGHT LOSS VERIFICATION

TA Instruments offers the widest range of ICTAC certified and NIST traceable Curie Point reference materials that permit SDT apparatus temperature calibration over the range from 150 to 1,120 ºC. TA Instruments also offers certified Mass Loss Reference Materials for validation of SDT instrument performance.
**IMPROVED DSC DATA**

In SDT experiments, better accuracy in DSC data is obtained when the instantaneous weight rather than the initial sample weight is used in heat flow calculations. Figure 1 shows data for sodium chloride cycled through its melt four times, and the heat of fusion (J/g) determined using the instantaneous weight. The table below shows a comparison of this data versus an identical experiment where the initial sample weight was used for calculating the DSC data in each cycle. The differences are very apparent.

<table>
<thead>
<tr>
<th>Cycle</th>
<th>Heat of Fusion Initial Weight (J/g)</th>
<th>Heat of Fusion Instantaneous Weight (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cycle 1</td>
<td>468.7</td>
<td>468.7</td>
</tr>
<tr>
<td>Cycle 2</td>
<td>384.1</td>
<td>459.1</td>
</tr>
<tr>
<td>Cycle 3</td>
<td>324.7</td>
<td>459.2</td>
</tr>
<tr>
<td>Cycle 4</td>
<td>266.9</td>
<td>464.4</td>
</tr>
</tbody>
</table>

**HIGH SENSITIVITY**

Figure 2 shows a high sensitivity application of Q600 in which a small (3 mg) sample of Sodium Tungstate is analyzed at 10 °C/min from ambient to 800 °C. The integral and derivative TGA signals quantitatively record the dehydration step. The DSC trace quantitatively shows the loss of water and higher temperature solid state and melting transitions respectively. The latter pair are events where no weight loss occurs.

**SIMULTANEOUS DSC/TGA**

Figure 3 shows simultaneous DSC and TGA data to 1300 °C for a soda ash sample. The TGA curve quantitatively tracks the loss of water and the onset of a higher temperature decomposition. The DSC signal quantitatively reveals transitions associated with the dehydration, a polymorphic phase transition and the high temperature melt. The inset shows details about the phase transition. In the Q600, heat flow integrations are automatically normalized using the dynamic weight at the start of each transition.
TA Instruments, the world's leading supplier of DMA's is uniquely positioned to provide the best solution for any application. The Q800 DMA, based on a combined motor and transducer design, uses advanced, non-contact, linear motors to control stress and measure strain with a highly sensitive optical encoder. Its open architecture makes it appropriate for a wide range of applications. The RSA III is based on a separate motor and transducer design. Strain is applied via a high performance, direct-drive motor and the resultant stress is measured using patented transducer technology. Its design offers high force, and an upper frequency range independent of sample stiffness.
TA INNOVATIONS

Q800
First DMA to incorporate optical encoder
Unique air bearing design
Low mass, high stiffness clamps
Automated furnace
Non-contact magnetic drive motor

RSA III
Patented force rebalance transducer™
Simultaneous DMA and DEA analysis
High 35 N force capability
Real-time wave form display
The Q800 is the world’s best-selling DMA, for very good reasons. It utilizes state-of-the-art, non-contact, linear drive technology to provide precise control of stress, and air bearings for low friction support. Strain is measured using optical encoder technology that provides unmatched sensitivity and resolution. With its unique design, the Q800 easily outperforms competitive instruments, and is ideal for high-stiffness applications including composites.

The RSA III provides a powerful platform for high-performance DMA measurements. The RSAIII uses an advanced direct-drive linear motor to apply the strain and the patented Force Rebalance Transducer™ to measure force. Low friction air bearings ensure optimal sensitivity. The RSA III is particularly well-suited for compression testing of soft materials, such as gels and elastomers, and for low stiffness, high frequency measurements on films and fibers.
## Technical Specifications

### Q800 vs RSA III

<table>
<thead>
<tr>
<th>Specification</th>
<th>Q800</th>
<th>RSA III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Force</td>
<td>18 N</td>
<td>35 N</td>
</tr>
<tr>
<td>Minimum Force</td>
<td>0.0001 N</td>
<td>0.001 N</td>
</tr>
<tr>
<td>Force Resolution</td>
<td>0.0001 N</td>
<td>0.001 N</td>
</tr>
<tr>
<td>Strain Resolution</td>
<td>1 nanometer</td>
<td>1 nanometer</td>
</tr>
<tr>
<td>Modulus Range</td>
<td>10^9 to 3x10^12 Pa</td>
<td>10^9 to 3x10^12 Pa</td>
</tr>
<tr>
<td>Modulus Precision</td>
<td>± 1%</td>
<td>± 1%</td>
</tr>
<tr>
<td>Tan δ Sensitivity</td>
<td>0.0001</td>
<td>0.0001</td>
</tr>
<tr>
<td>Tan δ Resolution</td>
<td>0.00001</td>
<td>0.00001</td>
</tr>
<tr>
<td>Frequency Range</td>
<td>0.01 to 200 Hz</td>
<td>2x10^4 to 80 Hz</td>
</tr>
<tr>
<td>Dynamic Sample Deformation Range</td>
<td>± 0.5 to 10,000 μm</td>
<td>± 0.5 to 1,500 μm</td>
</tr>
<tr>
<td>Temperature Range</td>
<td>-150 to 600 °C</td>
<td>-150 to 600 °C</td>
</tr>
<tr>
<td>Heating Rate</td>
<td>0.1 to 20 °C/min</td>
<td>0.1 to 60 °C/min</td>
</tr>
<tr>
<td>Cooling Rate</td>
<td>0.1 to 10 °C/min</td>
<td>0.1 to 60 °C/min</td>
</tr>
<tr>
<td>Isothermal Stability</td>
<td>± 0.1 °C</td>
<td>± 0.1 °C</td>
</tr>
<tr>
<td>Time/Temperature Superposition</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>

### Output Values

<table>
<thead>
<tr>
<th>Property</th>
<th>Q800</th>
<th>RSA III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Storage Modulus</td>
<td>Complex/Dynamic Viscosity</td>
<td>Time</td>
</tr>
<tr>
<td>Loss Modulus</td>
<td>Creep Compliance</td>
<td>Stress/Strain</td>
</tr>
<tr>
<td>Storage/Loss Compliance</td>
<td>Relaxation Modulus</td>
<td>Frequency</td>
</tr>
<tr>
<td>Tan Delta (δ)</td>
<td>Static/Dynamic Force</td>
<td>Sample Stiffness</td>
</tr>
<tr>
<td>Complex Modulus</td>
<td>Temperature</td>
<td>Displacement</td>
</tr>
</tbody>
</table>

### Deformation Modes & Sample Size

<table>
<thead>
<tr>
<th>Instrument/Clamp</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dual/Single Cantilever</td>
<td>Sample Size</td>
</tr>
<tr>
<td>RSA III (Dual Only)</td>
<td>30 or 48* mm (L), Up to 12.5 mm (W) and 6 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>8/4 ** mm (L), Up to 15 mm (W) and 5 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>20/10 ** mm (L), Up to 15 mm (W) and 5 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>35/17.5 ** mm (L), Up to 15 mm (W) and 5 mm (T)</td>
</tr>
<tr>
<td>3-Point Bend</td>
<td>Sample Size</td>
</tr>
<tr>
<td>RSA III</td>
<td>30, 40 or 50 mm (L), Up to 12.5 mm (W) and 5 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>5, 10, or 15 mm (L), Up to 15 mm (W) and 7 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>20 mm (L), Up to 15 mm (W) and 7 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>50 mm (L), Up to 15 mm (W) and 7 mm (T)</td>
</tr>
<tr>
<td>Tension</td>
<td>Sample Size</td>
</tr>
<tr>
<td>RSA III (Film/Fiber)</td>
<td>Up to 35 mm (L), Up to 12.5 mm (W), and 1.5 mm (T)</td>
</tr>
<tr>
<td>Q800 (Film/Fiber)</td>
<td>5 to 30 mm (L), Up to 8 mm (W) and 2 mm (T)</td>
</tr>
<tr>
<td>Q800 (Fiber)</td>
<td>5 to 30 mm (L), 5 denier (0.57 tex) to 0.8 mm diameter</td>
</tr>
<tr>
<td>Shear</td>
<td>Sample Size</td>
</tr>
<tr>
<td>RSA III</td>
<td>15 mm square, 0.5, 1.0 and 1.5 mm (T)</td>
</tr>
<tr>
<td>Q800</td>
<td>10 mm square, Up to 4 mm (T)</td>
</tr>
<tr>
<td>Compression</td>
<td>Sample Size</td>
</tr>
<tr>
<td>RSA III</td>
<td>8, 15, and 25 mm diameter</td>
</tr>
<tr>
<td>Q800</td>
<td>15 and 40 mm diameter, Up to 10 mm (T)</td>
</tr>
<tr>
<td>Submersion (Q800)</td>
<td>Sample Size</td>
</tr>
<tr>
<td>Tension</td>
<td>5 to 30 mm (L), Up to 8 mm (W) and 2 mm (T)</td>
</tr>
<tr>
<td>Compression</td>
<td>15 and 40 mm diameter, Up to 10 mm (T)</td>
</tr>
</tbody>
</table>

*Dual cantilever only   **Lengths are for dual/single cantilever
The Q800 uses a non-contact, direct drive motor to provide the oscillatory or static force required. The motor is constructed of high performance composites that ensure low compliance and is thermostated to eliminate heat build-up even when using large oscillation amplitudes and high deformation forces. Sophisticated electronics enable the motor current to be rapidly adjusted in small increments. **Benefits:**

The motor can deliver reproducible forces over a wide range and the force can be changed rapidly, enabling a broad spectrum of material properties to be measured.
2 **Air Bearings**

The non-contact drive motor transmits force directly to a rectangular air bearing slide. The slide is guided by eight porous carbon air bearings grouped into two sets of four near the top and bottom of the slide. Pressurized air or nitrogen flows to the bearings forming a frictionless surface that permits the slide to "float". The slide, which connects to the drive shaft and sample clamp, can move vertically 25 mm and its rectangular shape eliminates any twisting of the sample. **Benefits:** Very weak materials like films and fibers can be characterized with ease.

3 **Optical Encoder**

A high-resolution linear optical encoder is used to measure displacement on the Q800 DMA. Based on diffraction patterns of light through gratings (one moveable and one stationary), optical encoders provide exceptional resolution compared to typical LVDT technology. **Benefits:** Due to the excellent 1 nanometer resolution of the optical encoder, very small amplitudes can be measured precisely. This combined with the non-contact drive motor and air bearing technology provides excellent modulus precision and high Tan δ sensitivity, allowing the Q800 DMA to characterize a broad range of materials.

4 **Low Mass, High Stiffness Sample Clamps**

The Q800 features a variety of sample clamps that provide for multiple modes of deformation. The clamps were designed using "finite element analysis" to provide high stiffness, with low mass, and attach to the drive shaft with a dovetail connection. The clamps are simple to use and adjust, and each is individually calibrated to insure data accuracy. **Benefits:** A broad range of samples can be analyzed. The high stiffness minimizes clamp compliance, and the low mass ensures rapid temperature equilibration. The simple, yet elegant designs reduce the time necessary to change clamps and load samples.

5 **Furnace**

The Q800 features a bifilar wound furnace that automatically opens and closes. **Benefits:** The furnace design combined with the Gas Cooling Accessory provides for efficient and precise temperature control over the entire temperature range, both in heating, cooling, and isothermal operation. The automatic furnace movement simplifies experimental setup.

6 **Rigid Aluminum Casting**

The Q800 drive motor, air bearing slide assembly with optical encoder and air bearings are all mounted within a rigid aluminum casting that is temperature controlled. **Benefits:** The rigid aluminum housing minimizes system compliance and the temperature-controlled housing ensures precise data.
1 **Drive Motor**
Strain is applied to the sample via a high-performance linear motor. The motor is a direct-drive, DC servo actuator that controls strain, strain rate, and frequency. Temperature compensated, rare-earth magnets provide high force. **Benefits:** Accurate measurements of viscoelastic properties are possible, and the fast response ensures exceptional performance during transient tests such as stress relaxation. High force expands the range of applications.

2 **Frame Assembly and Linear Slide**
All the components of the RSA III DMA are housed in a rigid, cast steel frame with low compliance. The transducer (upper head) is attached to the frame via a linear slide. A motor drives the precision slide via a preloaded spindle to prevent backlash. **Benefits:** Low compliance, stiff housing and linear slide ensures that all measurements made are accurate and precise.

2 **Air Bearings**
The high performance motor and the Force Rebalance Transducer™ incorporate air bearings that provide a very stiff, yet low friction means of supporting linear motion. **Benefits:** Air bearings reduce friction and enhance the sensitivity of measurements, especially for weak samples like films and fibers.
3 Force Transducer

The RSA III patented* Force Rebalance Transducer™ (FRT) measures the force generated by the sample when the motor applies the deformation. In an FRT, a position sensor (3A) detects movement and a linear motor (3B) measures the reaction force required to drive the clamp back to the original position. **Benefits:**

- The FRT provides a wide force range with high force sensitivity and negligible inertia. Since the motor inertia is decoupled from the force measurement, the FRT can make accurate and precise measurements over the complete frequency range, independent of sample stiffness.

*U.S. Patent 4601195

Furnace

The Forced Convection Oven (FCO) is an air convection oven with dual-element heaters and counter-rotating airflow for unmatched temperature stability. Optional cooling devices include a liquid nitrogen cooling device for subambient operation to -150 °C. The FCO includes a sample sight glass. **Benefits:** Combined with either of the two cooling devices, the FCO oven provides accurate and precise temperature control, and fast heating rates, for a broad range of applications.

The sight glass allows samples to be viewed during the experiment.

Simultaneous Measurements

The RSA III is available with optional simultaneous measuring techniques. These include dielectric measurements and exposing a sample to a UV light source. **Benefits:** Simultaneous measurements extend the range of applications.
1 **Dual/Single Cantilever**

In this mode, the sample is clamped at both ends and either flexed in the middle (dual cantilever) or at one end (single cantilever). Cantilever bending is a good general-purpose mode for evaluating thermoplastics and highly damped materials (e.g., elastomers). Dual cantilever mode is ideal for studying the cure of supported thermosets.

2 **3-Point Bend**

In this mode, the sample is supported at both ends and force is applied in the middle. 3-point bend is considered a “pure” mode of deformation since clamping effects are eliminated. The 50 and 20 mm clamps on the Q800 utilize unique low-friction, roller bearing supports that improve accuracy.

3 **Shear Sandwich**

In this mode, two equal-size pieces of the same material are sheared between a fixed and moveable plate. This mode is ideal for gels, adhesives, high viscosity resins, and other highly damped materials. The RSA III also offers a quartz shear clamp for exposing a sample to a UV light source.
4 Compression
In this mode, the sample is placed on a fixed flat surface and an oscillating plate applies force. Compression is suitable for low to moderate modulus materials (e.g., foams and elastomers). This mode can also be used to make measurements of expansion or contraction, and tack testing for adhesives.

5 Tension
In this mode, the sample is placed in tension between a fixed and moveable clamp. In oscillation experiments, the instruments use a variety of methods for applying a static load to prevent buckling and unnecessary creep. The clamps are suitable for both films and fibers.

6 Submersible Clamps
Both film tension and compression clamps are available in submersible configurations for the Q800. A tension submersion clamp is available for the RSAIII. These clamps allow samples to be analyzed in a fluid environment up to 80 °C.
**Q800 Subambient Operation**

The Gas Cooling Accessory (GCA) extends the operating range of the Q800 to -150 °C. The GCA uses cold nitrogen gas generated from controlled heating of liquid nitrogen. Automated filling of the GCA tank can be programmed to occur either after the scan is complete or during a run. The ability to automatically refill during the middle of a run is particularly useful during long DMA experiments typically encountered when generating data for Time/Temperature Superposition (TTS).

**Q800 Air Compressor Operation**

The Q800 uses air bearings for support of the drive shaft. The air bearings use a clean, dry compressed air or nitrogen supply that typically comes from a centralized supply. In cases where this is not possible, the Air Compressor Accessory (ACA) is available. This self-contained air compressor provides an air supply required for the air bearings (appropriate filters required).

**RSA III Subambient Options**

The RSA III is available with a liquid nitrogen cooling system for operation from -150 to 600 °C. This accessory connects directly to a bulk liquid nitrogen dewar and provides rapid cooling capability.
**DMA Theory**

Dynamic Mechanical Analysis (DMA) is a technique used to measure the mechanical properties of a wide range of materials. Many materials, including polymers, behave both like an elastic solid and a viscous fluid, thus the term viscoelastic. DMA differs from other mechanical testing devices in two important ways. First, typical tensile test devices focus only on the elastic component. In many applications, the inelastic, or viscous component, is critical. It is the viscous component that determines properties such as impact resistance. Second, tensile test devices work primarily outside the linear viscoelastic range. DMA works primarily in the linear viscoelastic range and is therefore more sensitive to structure.

DMA measures the viscoelastic properties using either transient or dynamic oscillatory tests. Transient tests include creep and stress relaxation. In creep, a stress is applied to the sample and held constant while deformation is measured vs. time. After some time, the stress is removed and the recovery is measured. In stress relaxation, a deformation is applied to the sample and held constant, and the degradation of the stress required to maintain the deformation is measured versus time.

The most common test is the dynamic oscillatory test, where a sinusoidal stress (or strain) is applied to the material and a resultant sinusoidal strain (or stress) is measured (Figure 1). Also measured is the phase difference, \( \delta \), between the two sine waves. The phase lag will be zero degrees for purely elastic materials and 90 degrees for purely viscous materials. Viscoelastic materials (e.g., polymers) will exhibit an intermediate phase difference.

Since modulus is stress/strain, the complex modulus, \( E^* \), can be calculated. From \( E^* \) and the measurement of \( \delta \), the storage modulus, \( E' \), and loss modulus, \( E'' \), can be calculated as illustrated in Figure 2. \( E' \), the storage modulus, is the elastic component and related to the sample’s stiffness. \( E'' \), the loss modulus, is the viscous component and is related to the sample’s ability to dissipate mechanical energy through molecular motion. The tangent of phase difference, or \( \tan \delta \), is another common parameter that provides information on the relationship between the elastic and inelastic components. All of these parameters can be calculated as a function of time, temperature, frequency, or amplitude (stress or strain) depending on the application.

---

**Range of Material Behavior**

<table>
<thead>
<tr>
<th>Solid Like</th>
<th>Liquid Like</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ideal Solid</td>
<td>Most Materials</td>
</tr>
<tr>
<td>Purely Elastic</td>
<td>Viscoelastic</td>
</tr>
</tbody>
</table>

**Viscoelasticity:**

Having both viscous and elastic properties

---

**Figure 1**

![Diagram showing the relationship between stress and strain in 100% Elastic and 100% Viscous Behavior.]

**Figure 2**

![Diagram illustrating the calculation of storage modulus, \( E' \), and loss modulus, \( E'' \), using the complex modulus, \( E^* \), and the phase difference, \( \delta \).]
1 **Multi-Frequency Mode**

The multi-frequency mode can assess viscoelastic properties as a function of frequency, while oscillation amplitude is held constant. These tests can be run at single or multiple frequencies, in time sweep, temperature ramp, or temperature step/hold experiments. The RSA III has the additional capability of multiwave analysis during an isothermal or temperature ramp.

2 **Multi-Stress/Strain Mode**

In this mode, frequency and temperature are held constant, and the viscoelastic properties are monitored as strain or stress is varied. This mode is primarily used to identify the Linear Viscoelastic Range (LVR).

3 **Creep/Stress Relaxation Mode**

With creep, the stress is held constant and deformation is monitored as a function of time. In stress relaxation, the strain is held constant and the stress is monitored vs. time.

4 **Controlled Force/Strain Rate Mode**

In this mode, the temperature is held constant while stress or strain is ramped at a constant rate. This mode is used to generate stress/strain plots to obtain Young’s Modulus. Alternatively, stress can be held constant with a temperature ramp while strain is monitored.

5 **Isostrain Mode**

In isostrain mode, available on the Q800, strain is held constant during a temperature ramp. Isostrain can be used to assess shrinkage force in films and fibers.
**Measurement of Tg of Polymeric Materials**

A common measurement on polymers is the glass transition temperature, Tg. It can be measured with various techniques, but DMA is by far the most sensitive. Figure 1 shows a scan of a pressure sensitive adhesive run in the tension clamps at a frequency of 1 Hz. Tg can be measured by the E’ onset point, by the E’ peak, or the peak of Tan δ. In addition to the Tg, the absolute value of the various viscoelastic parameters is also useful.

**Frequency Effect on Modulus and Glass Transition of Polyethylene Terephthalate (PET)**

Because the Tg has a kinetic component, it is strongly influenced by the frequency (rate) of deformation. As the frequency of the test increases, the molecular relaxations can only occur at higher temperatures and, as a consequence, the Tg will increase with increasing frequency as illustrated in Figure 2. In addition, the shape and intensity of the Tan δ peak as well as the slope of the storage modulus in the transition region will be affected. Based on end-use conditions, it is important to understand the temperature and frequency dependence of transitions.

**The Measurement of Secondary Transitions in Vinyl Ester**

DMA is one of the few techniques that can measure β and γ secondary transitions. Secondary transitions arise from side group motion with some cooperative vibrations from the main chain as well as internal rotation within a side group. The transitions, as shown in Figure 3, are below the Tg and typically subambient. They are very important as they affect impact resistance and other end-use properties. This data was generated using 3-point bending and also illustrates the ability to run stiff composites.
MEASURING EFFECT OF ADHESIVE COATINGS ON FILMS

Figure 4 shows a comparison among three PET samples in tension on the RSA III: one with a uniform adhesive layer that performs well; one with a non-uniform layer that performs poorly; and one that is uncoated. A transition peak due to the adhesive is seen in Tan δ around 40 °C in the “good” sample, whereas the “poor” sample shows a much smaller peak. Knowing the characteristics of good and poor samples enables quality control of the coating process and the finished product.

CHARACTERIZING PRINTED CIRCUIT BOARDS

Printed Circuit Boards (PCB) are typically comprised of fiberglass braid impregnated with a thermosetting resin. Characterizing the Tg of PCB’s is often difficult due to the very low amount of resin used. Figure 5 shows a typical PCB run in single cantilever bending. The Tg is clearly discernible and the difference between the sample “as received” and “post baked” clearly shows the effect that further crosslinking has on both the Tg and the absolute value of modulus.

EFFECT OF CARBON BLACK IN ELASTOMERS

Another very common application is the effect of fillers and additives on viscoelastic properties. Figure 6 illustrates the effect on Storage Modulus (E) and Tan δ when adding carbon black to an SBR rubber. This test, performed in dual cantilever on the RSA III, shows that adding carbon black increases the absolute value of the Storage Modulus and significantly increases the Tg temperature. Understanding how fillers and additives affect material properties is crucial in many industrial applications.
Characterizing Packaging Films Using Creep

In a thermoforming process, a film is pulled down into a heated mold to form a desired shape. The ability to produce a stable product can be predicted by using a creep-recovery experiment. Figure 7 illustrates data on a packaging film using the tension mode. In the recovery phase, the equilibrium recoverable compliance, \( J_{er} \) can be calculated. If the sample compliance is too high, as observed by a high \( J_{er} \), then the elasticity may be too low at the forming temperature to maintain the desired shape.

Tack Testing of a Pressure Sensitive Adhesive (PSA)

Figure 8 shows how the RSA III can be used to evaluate the tack properties of a PSA using the compression clamp. The fixture is brought into contact with the sample at a fixed stress, and then removed at a constant rate. The force required to pull the probe away from the sample is recorded. Although Sample A requires almost twice the maximum stress compared to Sample B, the strain is nearly 20 times less.

Predicting Material Performance using Time/Temperature Superpositioning (TTS)

The TTS technique, well grounded in theory, is used to predict material performance at frequencies or time scales outside the range of the instrument. Data is usually generated by scanning multiple frequencies during a series of isothermal step-hold experiments over a temperature range. A reference temperature is selected and the data shifted. A shift factor plot is generated and fit to either a William-Landel-Ferry (WLF) or Arrhenius model. Finally, a master curve at a specific temperature is generated as illustrated in Figure 9 for a PET film sample. Using this technique, properties at very high frequencies (short time scales) or very low frequencies (long time scales) can be assessed.
The Q400 is a sixth-generation product from the world leader in thermal analysis. Its performance, ease-of-use, and reliability aptly demonstrate our long experience in designing novel instruments for high sensitivity mechanical measurements over a wide temperature range.
TA INNOVATIONS

FIRST COMMERCIAL TMA

AUTOMATED PROBE CALIBRATION

MODULATED TMA™

ELECTRONIC APPLICATION OF PROGRAMMABLE FORCE IN TMA

STRESS / STRAIN, CREEP, STRESS RELAXATION, AND DYNAMIC MODES OF OPERATION
The Q400EM is a high-performance, research-grade thermomechanical analyzer (TMA), with unmatched flexibility in operating modes, test probes, fixtures, and available signals. For standard TMA applications, the Q400 delivers the same performance and reliability. It is ideal for research, teaching, and quality control applications, with performance equivalent to competitive research models.
## Technical Specifications

### Q400EM vs. Q400

<table>
<thead>
<tr>
<th>Specification</th>
<th>Q400EM</th>
<th>Q400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature Range (max)</td>
<td>-150 to 1,000 °C</td>
<td>-150 to 1,000 °C</td>
</tr>
<tr>
<td>Temperature Precision</td>
<td>± 1 °C</td>
<td>± 1 °C</td>
</tr>
<tr>
<td>Furnace Cool Down Time</td>
<td>&lt;10 min from 600 °C to 50 °C</td>
<td>&lt;10 min from 600 °C to 50 °C</td>
</tr>
<tr>
<td>(air cooling)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Sample Size - solid</td>
<td>26 mm (L) x 10 mm (D)</td>
<td>26 mm (L) x 10 mm (D)</td>
</tr>
<tr>
<td>Maximum Sample Size - film/fiber</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Static Operation</td>
<td>26 mm (L) x 1.0 mm (T) x 4.7 mm (W)</td>
<td>26 mm (L) x 1.0 mm (T) x 4.7 mm (W)</td>
</tr>
<tr>
<td>Dynamic Operation</td>
<td>26 mm (L) x .35 mm (T) x 4.7 mm (W)</td>
<td>___</td>
</tr>
<tr>
<td>Measurement Precision</td>
<td>± 0.1 %</td>
<td>± 0.1 %</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>15 nm</td>
<td>15 nm</td>
</tr>
<tr>
<td>Dynamic Baseline Drift</td>
<td>&lt;1 μm (-100 to 500 °C)</td>
<td>&lt;1 μm (-100 to 500 °C)</td>
</tr>
<tr>
<td>Force Range</td>
<td>0.001 to 1 N</td>
<td>0.001 to 1 N</td>
</tr>
<tr>
<td>Force Resolution</td>
<td>0.001 N</td>
<td>0.001 N</td>
</tr>
<tr>
<td>Frequency</td>
<td>0.01 to 2 Hz</td>
<td>Not Available</td>
</tr>
<tr>
<td>Mass Flow Control</td>
<td>Included</td>
<td>Included</td>
</tr>
<tr>
<td>Atmosphere (static or controlled flow)</td>
<td>Inert, Oxidizing, or Reactive Gases</td>
<td>Inert, Oxidizing, or Reactive Gases</td>
</tr>
<tr>
<td>Standard</td>
<td>Included</td>
<td>Included</td>
</tr>
<tr>
<td>Stress/Strain</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Creep</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Stress Relaxation</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Dynamic TMA (DTMA)</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Modulated TMA™ (MTMA™)</td>
<td>Included</td>
<td>Not Available</td>
</tr>
</tbody>
</table>

### Operational Modes

<table>
<thead>
<tr>
<th>Mode</th>
<th>Q400EM</th>
<th>Q400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard</td>
<td>Included</td>
<td>Included</td>
</tr>
<tr>
<td>Stress/Strain</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Creep</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Stress Relaxation</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Dynamic TMA (DTMA)</td>
<td>Included</td>
<td>Not Available</td>
</tr>
<tr>
<td>Modulated TMA™ (MTMA™)</td>
<td>Included</td>
<td>Not Available</td>
</tr>
</tbody>
</table>

Note: The Q400 can be field upgraded to the Q400EM.
A thermomechanical analyzer measures sample dimensional changes under conditions of controlled temperature, time, force, and atmosphere. Our engineering experience in design and integration of critical furnace, temperature, dimension measurement, and atmosphere control components meld with powerful, flexible software to optimize the many tests that the Q Series™ TMA can perform.

1 **Furnace**

The Q400 features a rugged and reliable furnace. Its customized electronics provide excellent heating rate control and rapid response over a wide temperature range. Furnace raising and lowering is software-controlled. **Benefits:** The design ensures long life and performance consistency. Excellent heating rate control provides for superior baseline stability and improved sensitivity, while the rapid response permits Modulated TMA™ operation. Furnace movement provides operational convenience, and easy access to the sample chamber.

2 **Sample Chamber**

Located in the furnace core, the easily accessed chamber provides complete temperature and atmosphere control for sample analysis. Purge gas furnace routing is optimized and regulated by a digital mass flow controller. **Benefits:** Enhanced flexibility, data quality, ease-of-use, and productivity. Heating rate control and purge gas routing provide optimized performance in both standard and temperature modulated modes of operation. The open design simplifies installation of available probes (See Modes of Deformation), sample mounting, and thermocouple placement. Data precision is enhanced by mass flow control of the purge gas.
3 Force Motor

A non-contact motor provides a precisely controlled, friction-free, calibrated force to the sample via the measurement probe or fixture. The force is programmable from 0.001 to 1 N, and can be increased to 2 N by addition of weights to a special tray. A precision sine wave generator provides a set of ten individual frequencies for use in dynamic experiments. **Benefits:** The motor smoothly generates the accurate and precise static, ramped, or oscillatory dynamic force necessary for quality measurements in all modes of operation. The choice of frequencies allows optimization of dynamic TMA (DTMA) experiments in compression, 3-point bending, or tension modes of deformation.

4 Linear Variable Differential Transducer

The heart of the Q400 TMA sample measurement system is the precision, moveable-core, linear variable differential transducer (LVDT). **Benefits:** It generates an accurate output signal that is directly proportional to a sample dimension change. Its precise and reliable response over a wide temperature range (-150 to 1,000 °C) makes for reproducible TMA results. Its location below the furnace protects it from unwanted temperature effects and ensures stable baseline performance.
The Q400 offers all the major TMA deformation modes necessary to characterize solids, foams, films, and fibers. These include compression, tension, and 3-point bending.

**Compression**
In this mode, the sample is subjected to either a static, linear ramp, or dynamic oscillatory force, while under a defined temperature program, and atmosphere. Sample displacement (strain) is recorded by either expansion/penetration experiments used to measure intrinsic material properties, or by dynamic tests and used to determine viscoelastic parameters (DTMA), detect thermal events, and separate overlapping transitions (MTMA™).

**Expansion**
Expansion measurements determine a material’s coefficient of thermal expansion (CTE), glass transition temperature (Tg), and compression modulus. A flat-tipped standard expansion probe (Figure 1) is placed on the sample (a small static force may be applied), and the sample is subjected to a temperature program. Probe movement records sample expansion or contraction. This mode is used with most solid samples. The larger surface area of the macro-expansion probe (Figure 2) facilitates analysis of soft or irregular samples, powders, and films.

**Penetration**
Penetration measurements use an extended tip probe (Figure 3) to focus the drive force on a small area of the sample surface. This provides precise measurement of Tg, softening, and melting behavior. It is valuable for characterizing coatings without their removal from a substrate. The probe operates like the expansion probe, but under a larger applied force. The hemispherical probe (Figure 4) is an alternate penetration probe for softening point measurements in solids.
3-Point Bending

In this bending deformation (also known as flexure), the sample is supported at both ends on a two-point, quartz anvil atop the stage (Figure 5). A fixed static force is applied vertically to the sample at its center, via a wedge-shaped, quartz probe. Material properties are determined from the force and the measured probe deflection. This mode is considered to represent “pure” deformation, since clamping effects are eliminated. It is primarily used to determine bending properties of stiff materials (e.g., composites), and for distortion temperature measurements. Dynamic (DTMA) measurements are also available with the Q400EM, where a special, low-friction, metallic anvil replaces the quartz version.

Tension

Tension studies of the stress/strain properties of films and fibers are performed using a film/fiber probe assembly (Figure 6). An alignment fixture (Figure 7) permits secure, and reproducible, sample positioning in the clamps. The clamped sample is placed in tension between the fixed and moveable sections of the probe assembly. Application of a fixed force is used to generate stress/strain and modulus information. Additional measurements include Tg, softening temperatures, cure, and cross-link density. Dynamic tests (e.g. DTMA, MTMA™) in tension can be performed to determine viscoelastic parameters (e.g., E', E'', tan δ), and to separate overlapping transitions.

Specialty Probe/Fixture Kits

Additional sample measurement probes and fixtures are available for use with both the Q400 and Q400EM in specialty TMA applications. These include the following:

**Dilatometer Probe Kit** – for use in volume expansion coefficient measurements

**Parallel Plate Rheometer** – for the measurement of low shear viscosity of materials (10 to 10⁷ Pa.s range) under a fixed static force

The expansion, macro-expansion, and penetration probes are supplied with the Q400. These probes, plus the flexure probe, and the low-friction bending fixture, are included with the Q400EM module. Data analysis programs relevant to each of the measurements described are provided in our Advantage™ software.
TMA measures material deformation changes under controlled conditions of force, atmosphere, time and temperature. Force can be applied in compression, flexure, or tension modes of deformation using specially designed probes described in pages 76-77. TMA measures intrinsic material properties (e.g., expansion coefficient, glass transition, Young’s modulus), plus processing / product performance parameters (e.g., softening points). These measurements have wide applicability, and can be performed by either the Q400 or the Q400EM.

The Q400 and Q400EM operating modes permit multiple material property measurements. The Q400 features the Standard mode, while the Q400EM additionally offers Stress / Strain, Creep, Stress Relaxation, Dynamic TMA and Modulated™ TMA modes as described below.

1 & 2 Standard Mode (Q400/Q400EM)

Temperature Ramp: Force is held constant, and displacement is monitored under a linear temperature ramp to provide intrinsic property measurements.

Isostrain: Strain is held constant, and the force required to maintain the strain is monitored under a temperature ramp. This permits assessment of shrinkage forces in materials such as films / fibers.

Force Ramp: Force is ramped, and the resulting strain is measured at constant temperature to generate force / displacement plots and modulus assessment.

3 Stress/Strain Mode (Q400EM)

Stress or strain is ramped, and the resulting strain or stress is measured at constant temperature. Using customer entered sample geometry factors, the data provides both stress / strain plots and related modulus information. In addition, calculated modulus can be displayed as a function of stress, strain, temperature, or time.
4 Creep and Stress Relaxation

TMA can also measure viscoelastic properties using transient (e.g., creep or stress relaxation) tests. These require the Q400EM module. In Creep, input stress is held constant, and resulting strain is monitored usually as a function of time. In Stress Relaxation, input strain is held constant, and stress decay is measured as a function of time. Both are transient tests used to assess material deformation and recovery properties of materials. The data can also be displayed in units of compliance (creep mode) and stress relaxation modulus (stress relaxation mode).

5 & 6 Dynamic TMA Mode (Q400EM)

In Dynamic TMA (DTMA), a known sinusoidal stress and linear temperature ramp are applied to the sample (Figure 5), and the resulting sinusoidal strain, and sine wave phase difference (δ) are measured (Figure 6a). From this data, storage modulus (E'), loss modulus (E''), and tan δ (E'' / E') are calculated as functions of temperature, time, or stress (Figure 6b). This technique can be useful in the analysis of thin polymer films.

7 Modulated TMA™ (MTMA™; Q400EM)

In Modulated TMA™ (MTMA™), the sample experiences the combined effects of a linear temperature ramp and a sinusoidal temperature of selected amplitude and period (Figure 7). The output signals, after Fourier transformation of the raw data, are total displacement and the change in thermal expansion coefficient. Both can be resolved into their reversing and non-reversing component signals. The reversing signal contains events attributable to dimension changes, and is useful in detecting related events (e.g., Tg). The non-reversing signal contains events that relate to time dependent kinetic processes (e.g., stress relaxation). This technique is unique to the Q400EM.
**Intrinsic and Product Property Measurements**

Figure 1 shows expansion and penetration probe measurements of Tg, and softening point, of a synthetic rubber using a temperature ramp at constant force. The large CTE changes in the expansion plot indicate the transition temperatures. In penetration, they may be detected by the sharp movement of the loaded probe into the changing material structure.

**Accurate Coefficient of Thermal Expansion Measurements**

Figure 2 demonstrates the use of the expansion probe to accurately measure small CTE changes in an aluminum sample over a 200 °C temperature range. Advantage™ software permits analysis of the curve slope using an “at point,” “straight line,” or “best fit” method to compute the CTE at a selected temperature, or over a range.

**Material Performance and Selection**

Figure 3 is an example of a 3-point bending mode (flexure probe) experiment on a polyvinyl chloride (PVC) sample, using the ASTM International Test Method E2092 to determine the distortion temperature. This test specifies the temperature at which a sample of defined dimensions produces a certain deflection under a given force. It has long been used for predicting material performance.
**Multilayer Film Analysis**

Figure 4 shows a compression mode analysis, using a penetration probe, of a double layer PE/PET film sample, supported on a metal substrate. The sample temperature was linearly ramped from ambient to 275 °C at 5 °C/min. The plot shows probe penetrations of the PE layer (93.2 µm) at 103 °C, and the PET layer (14.8 µm) at 258 °C respectively.

**Film Property Testing**

Figure 5 illustrates a classic isostrain experiment, in the tension mode, on a food wrapping film. The film was strained to 20% at room temperature for 5 minutes, cooled to -50 °C and held for 5 more minutes, then heated at 5 °C/min to 40 °C. The plot shows the force variation required to maintain a set strain in the film. The test simulates film use from the freezer to the microwave.

**Film Tensile Testing**

Figure 6 displays a strain ramp experiment, at a constant temperature, on a proprietary film in tension. The plot shows an extensive region where stress and strain are linearly related, and over which a tensile modulus can be directly determined. Quantitative modulus data can also be plotted as a function of stress, strain, time, or temperature. The results show the ability of the Q400EM to function as a mini tensile tester for films and fibers.
**Fiber Stress/Strain Measurements**

Stress/strain measurements are widely used to assess and compare materials. **Figure 7** shows the different regions of stress/strain behavior in a polyamide fiber (25 µm) in tension, when subjected to a force ramp at a constant temperature. The fiber undergoes instantaneous deformation, retardation, linear stress/strain response, and yield elongation. Other parameters (e.g., yield stress, Young’s modulus) can be determined.

**Thermal Stress Analysis of Fibers**

**Figure 8** displays a tension mode experiment, using a temperature ramp at a constant strain (1%), to perform a stress analysis on a polyolefin fiber, as received, and after cold drawing. The plot shows the forces needed to maintain the set strain as a function of temperature. The data has been correlated with key fiber industry processing parameters, such as shrink force, draw temperature, draw ratio, elongation at break, and knot strength.

**Creep Analysis**

Creep tests help in materials selection for end uses where stress changes are anticipated. **Figure 9** illustrates an ambient temperature creep study on a polyethylene film in tension. It reveals the instantaneous deformation, retardation, and linear regions of strain response to the set stress, plus its recovery, with time, on stress removal. The data can also be plotted as compliance, and recoverable compliance, versus time.
**Stress Relaxation Analysis**

Figure 10 shows a stress relaxation test in tension on the same polyolefin film used for the creep study in Figure 9. A known strain is applied to the film, and maintained, while its change in stress is monitored. The plot shows a typical decay in the stress relaxation modulus. Such tests also help engineers design materials for end uses where changes in deformation can be expected.

**Viscoelastic Property Determination - Dynamic TMA**

Figure 11 illustrates a dynamic test, in which a semi-crystalline polyethylene terephthalate (PET) film in tension is subjected to a fixed sinusoidal stress during a linear temperature ramp. The resulting strain and phase data are used to calculate the material’s viscoelastic properties (e.g., $E'$, $E''$, and $\tan \delta$). The plotted data shows dramatic modulus changes as the film is heated through its glass transition temperature.

**Separating Overlapping Transitions - Modulated™ TMA**

Figure 12 shows an MTMA™ study to determine the Tg of a printed circuit board (PCB). The signals plotted are the total dimension change, plus its reversing and non-reversing components. The total signal is identical to that from standard TMA, but does not uniquely define the Tg. The component signals, however, clearly separate the actual Tg from the stress relaxation event induced by non-optimum processing of the PCB.