



DISCOVERY HYBRID RHEOMETERS
TEMPERATURE SYSTEMS AND ACCESSORIES



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discover

THE HIGHEST PERFORMING RHEOMETER TEMPERATURE SYSTEMS AND ACCESSORIES

The Discovery Hybrid Rheometers are the most powerful and versatile single-head rheometers from the world leader in rheological measurements.

All DHR temperature systems and accessories are designed with superior performance and ease-of-use in mind. Only TA Instruments' DHR offers the convenience and versatility of Smart Swap™ geometries, temperature systems, and accessories. Smart Swap™ technologies provide fast and easy interchanging of accessories and automatic detection and configuration of the rheometer for operation.

advanced peltier plate

TEMPERATURE SYSTEMS

New Advanced Peltier Plate

The new Advanced Peltier Plate combines ultimate flexibility with exceptional temperature performance in a single Peltier Plate temperature system designed to cover the widest range of applications. The unique Quick Change Plate system provides the ability to easily attach lower plates of different materials and surface finishes, disposable plates for testing curing materials, and an Immersion Cup for characterizing materials in a fluid environment.

Features and Benefits

- Unique Smart Swap™ technology
- Wide temperature range: -40 °C to 200 °C
- Accurate temperature control: ± 0.1 °C
- Plates and cones up to 50 mm in diameter
- Robust hardened chrome finish
- Quick Change Plates provide flexibility for modifying lower geometry surface
 - Stainless steel, Anodized Aluminum and Titanium plates
 - Smooth, Sandblasted and Crosshatched plates
 - Disposable plates available for curing materials
- Fully accessorized
 - Extremely efficient Solvent Trap with built-in Purge Cover
 - Immersion Cup
 - Camera Viewer option
- Open platform to easily adapt custom-made lower geometries





Stepped Plate



Sandblasted Plate



Disposable Plate

Quick Change Plates

The Advanced Peltier Plate's simple bayonet-style locking ring facilitates the effortless attachment of a wide range of different lower plate covers including hard anodized aluminum, titanium and stainless steel plates with either a smooth, sandblasted or crosshatched surface finishes. Standard disposable Aluminum plates expand the capabilities of the system to test curing materials using a single Peltier Plate.



8 mm Stainless Steel



20 mm Disposable
Aluminum



25 mm Crosshatched



40 mm Anodized
Aluminum



40 mm Sandblasted



50 mm Titanium

Immersion Cup

The Advanced Peltier Plate's Immersion Cup adds the capability to characterize material properties when completely immersed in a fluid. It is easily attached to the top of the Advanced Peltier Plate through the bayonet fixture and gives access to the sample for loading, trimming, and subsequent sealing and filling – a rubber ring provides the fluid seal. The Immersion Cup is ideally suited to investigating hydrogel materials and can accommodate plates or cones up to 40 mm in diameter.



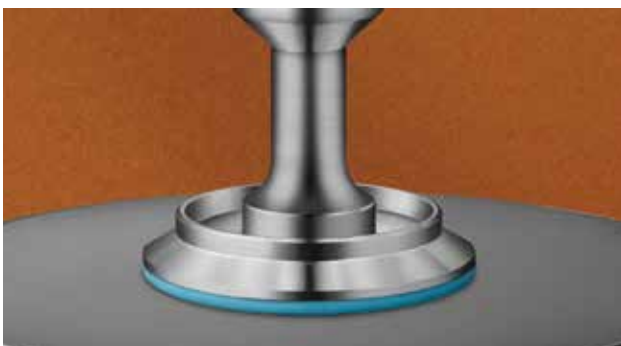
peltier plate

TEMPERATURE SYSTEMS

Complete Peltier Plate Temperature Systems

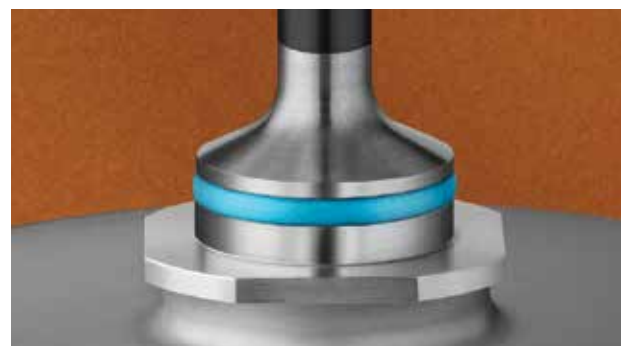
Over 20 years ago, TA Instruments first introduced Peltier Plate temperature control to rheometers. Since then, this core technology has been continuously developed and adapted to meet the expanding needs of our customers. With superior technology designed into four convenient Smart Swap™ models, we offer the highest performing, most versatile, and best accessorized Peltier Plate Temperature Systems available.





Standard Peltier Plate

The Standard Peltier Plate is the most common selection, offering an 80 mm diameter hardened chrome surface to accommodate up to 60 mm upper plates for maximum sensitivity.



Stepped Peltier Plate

The Stepped Peltier Plate provides the convenience of interchanging plate diameters and surfaces up to 25 mm in diameter or for remote sample preparation. Stainless steel and titanium plates are available in flat, sandblasted, and crosshatched finishes.



Stepped Disposable Peltier Plate

The Stepped Disposable Peltier Plate is ideal for thermoset curing or other single-use applications and is compatible with standard disposable plates.



Dual Stage Peltier Plate

The new Dual Stage Peltier Plate is the perfect choice for applications requiring sub-ambient temperature control. The unique design uses a stacked Peltier element approach, enabling fast and easy temperature control down to an unprecedented -45°C , without the use of expensive circulators.

peltier plate

TECHNOLOGY

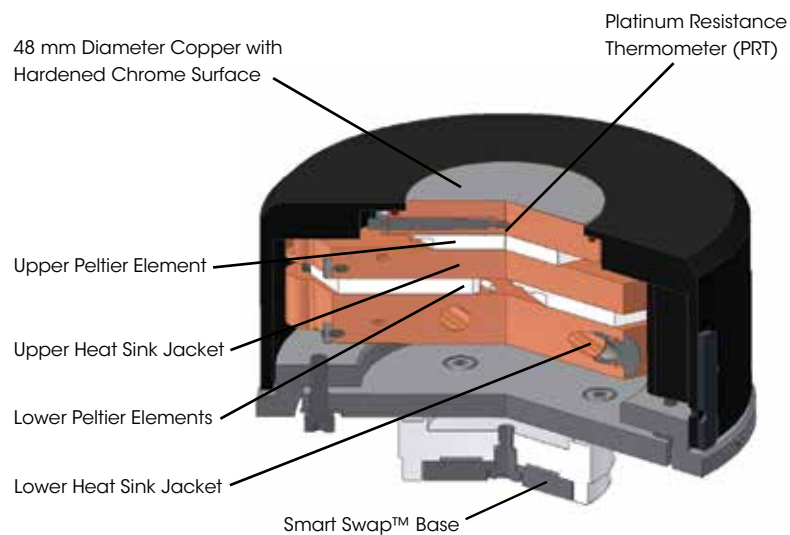
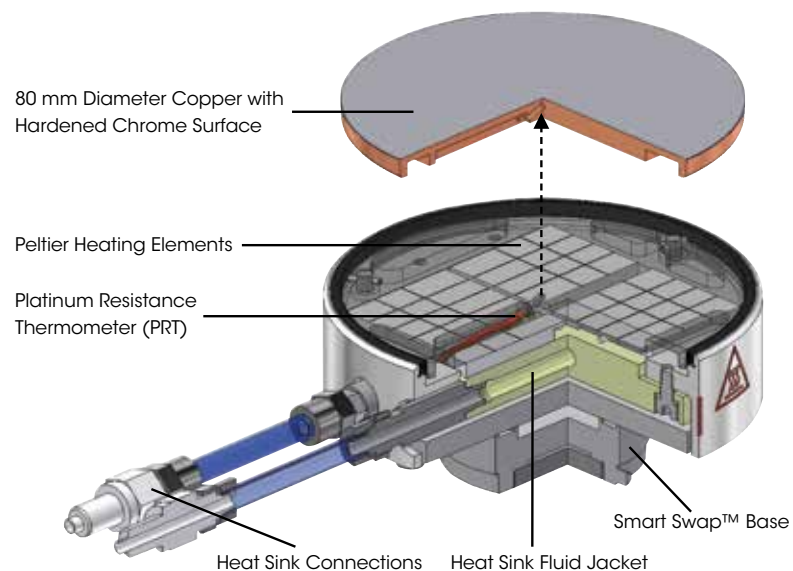
Peltier Technology

Standard and Stepped Peltier Plates offer a temperature range of $-40\text{ }^{\circ}\text{C}^*$ to $200\text{ }^{\circ}\text{C}$, heating rates up to $50\text{ }^{\circ}\text{C}/\text{min}$, and temperature accuracy of $0.1\text{ }^{\circ}\text{C}$. Four Peltier heating elements are placed directly in contact with a thin, 80 mm diameter, copper disc with an extremely rugged, hardened chrome surface. A platinum resistance thermometer (PRT) is placed at the exact center, ensuring accurate temperature measurement and control. The unique design provides for rapid, precise, and uniform temperature control over the entire 80 mm diameter surface. This allows for accurate testing with standard geometries up to 60 mm in diameter.

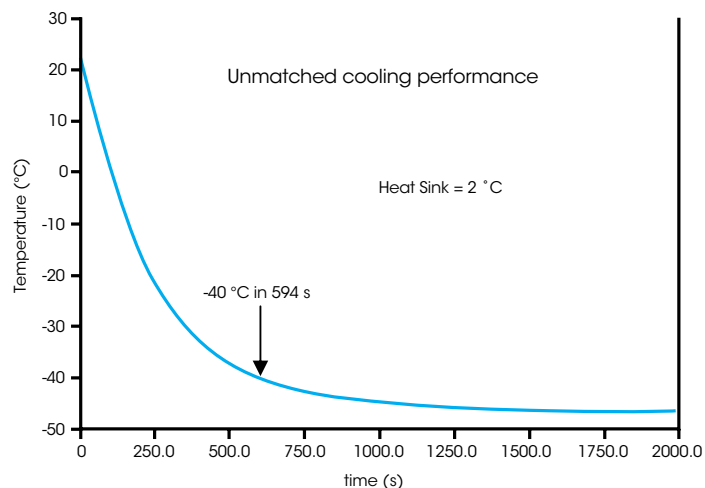
* with appropriate counter cooling

Dual Stage Peltier Plate Design

The new Dual Stage Peltier Plate uses a unique stacked Peltier element design that offers a temperature range of $-45\text{ }^{\circ}\text{C}$ to $200\text{ }^{\circ}\text{C}$, with standard counter-cooling options. It integrates a unique stacked Peltier element configuration that provides enhanced low temperature responsiveness and continuous temperature control over the entire operating range with a single heat sink temperature of $2\text{ }^{\circ}\text{C}$. This eliminates the need to have expensive powerful circulators to obtain temperatures down to this range.



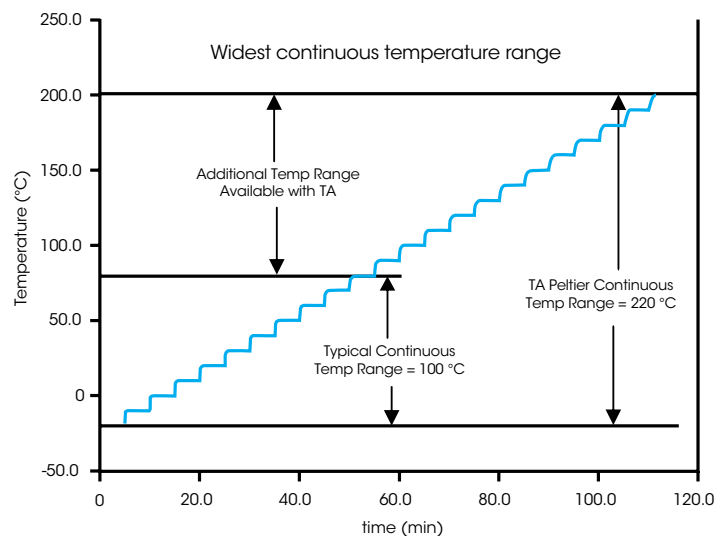
Dual Stage Peltier Plate Cooling Test



Performance

Peltier temperature control devices require that they be connected to a heat sink, typically a circulating fluid medium such as water. Most Peltier systems have a continuous temperature range of approximately 100 °C for a single heat sink temperature. The unique design of the Standard and Stepped Peltier Plate systems from TA Instruments extends the continuous range to 220 °C, as seen in data in the figure to the left. The benefit of this wide range is that it more than doubles the actual useable temperature range during any single test. The new Dual Stage Peltier Plate extends the low temperature limit and dramatically improves cooling performance. The figure to the left shows that this device can reach -40 °C from room temperature in under 10 minutes with a heat sink set at 2 °C.

Peltier Plate Temperature Steps over 220 °C Range



Features and Benefits

- Unique Smart Swap™ technology
- Wide Temperature Range: -45 °C to 200 °C
- Widest Continuous Temperature Range
- Accurate Temperature Control: ± 0.1 °C
- Hardened Chrome Surface
- Standard, Stepped, and Dual Stage Models
- Plates and Cones up to 60 mm in Diameter
- Disposable Plates
- Large Variety of Geometry Materials and Types
- Fully Accessorized
 - Extremely Efficient Solvent Trap
 - Smooth, Crosshatched, and Sandblasted Covers
 - Purge Gas Cover
 - Insulating Thermal Cover
 - Camera Viewer Option
 - Immersion Cell

peltier plate

GEOMETRIES

Smart Swap™ Peltier Plate Geometries

An extensive range of TA Instruments unique Smart Swap™ geometries⁽¹⁾, with automatic recognition are available for use with Peltier Plates. Cones and plates come standard in a variety of sizes, cone angles and material types. Custom geometries of non-standard sizes, materials, and surface finishes (such as sandblasted or Teflon® coated) are available upon request.

(1) Patent # 6,952,950

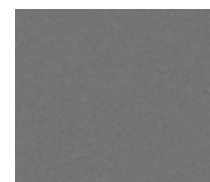


Standard Geometry Dimensions

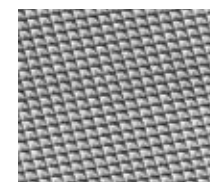
Peltier Plate geometries are available in 8 mm, 20 mm, 25 mm, 40 mm, 50 mm, and 60 mm diameters. Upper Cone geometries are readily available in 0.5°, 1°, 2°, and 4° cone angles. Non-standard diameters and cone angles are available upon special request. By changing diameter and cone angle, the measurable range of stress and strain or shear rate can be varied to capture the widest range of material properties.



Smooth



Sandblasted



Crosshatched

Materials of Construction and Surfaces

Peltier Plate geometries come standard in the following materials:

- **Stainless Steel:** Rugged, very good chemical resistance for highly basic or acidic materials
- **Stainless Steel with Composite Heat Break:** Same properties as stainless steel with added benefit of composite heat break, which insulates upper geometry when controlling temperatures away from ambient
- **Hard Anodized Aluminum:** Excellent thermal conductivity, low mass, fair chemical resistance
- **Titanium:** Low mass, excellent chemical resistance

Geometries are available in multiple surface finishes, including smooth, sandblasted, and crosshatched.



No Solvent Trap



With Solvent Trap



With Solvent Trap and
Composite heat break

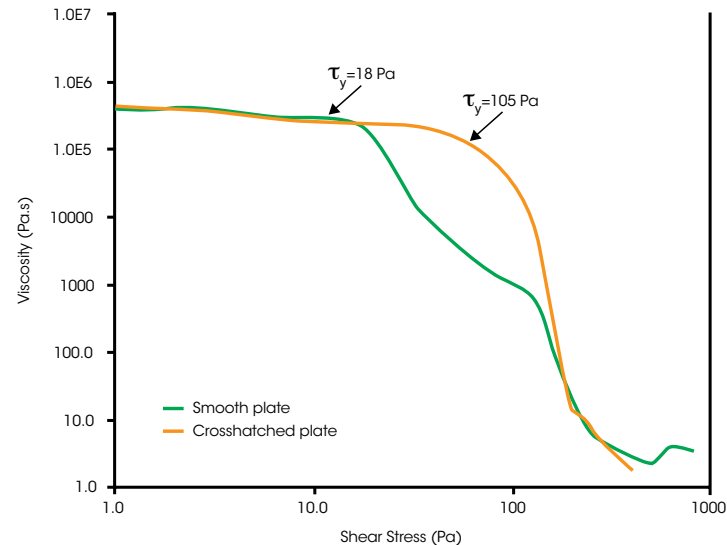
Peltier Plate Standard Geometry Types

Peltier Cone and Peltier Plate geometries are available in three basic types. They include geometries without solvent trap, geometries with insulating composite heat break, and geometries with solvent trap. Heat break geometries are available in stainless steel only. Solvent trap geometries are designed for use with the solvent trap system discussed separately. The figure below shows a comparison of stainless steel 40 mm geometry types.

Eliminating Wall Slip on Toothpaste with Crosshatched Plates

Wall slip phenomena can have large effects on steady shear rheological measurements. To mitigate such issues, a roughened surface finish is typically used. The figure to the right shows the steady state flow testing results on toothpaste with smooth and crosshatched plate geometries. With this type of material, standard smooth surface plates slip at the interface and lead to a false measured yield stress on the order of about 18 Pa. However, with crosshatched geometries, slip is eliminated and an accurate yield stress of 105 Pa, which is more than 5 times higher, is measured.

Yield Stress Measurements on Toothpaste



peltier plate

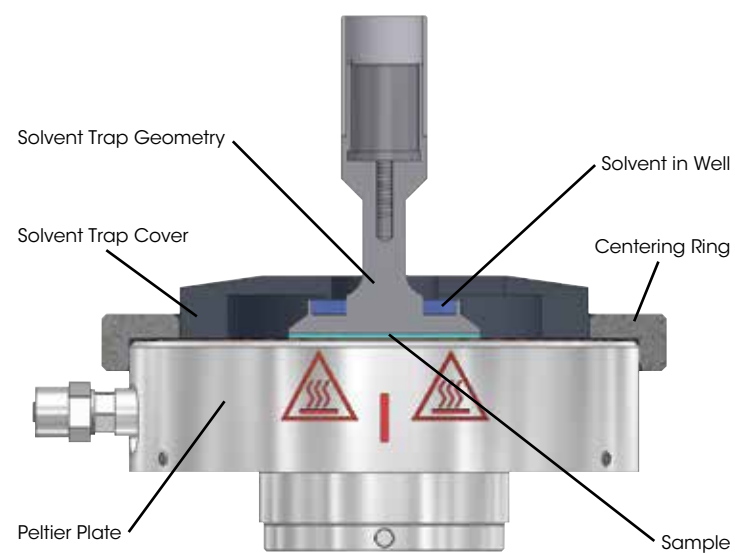
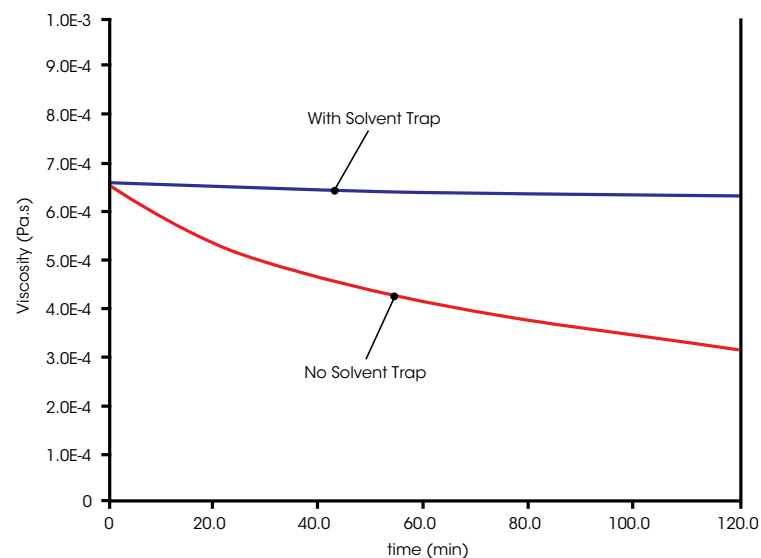
ACCESSORIES

Peltier Solvent Trap and Evaporation Blocker

The Solvent Trap cover and Solvent Trap geometry work in concert to create a thermally stable vapor barrier, virtually eliminating any solvent loss during the experiment as shown in data for water at 40 °C to the right. The geometry includes a well that contains very low viscosity oil, or even the volatile solvent present in the sample. The Solvent Trap cover includes a blade that is placed into the solvent contained in the well without touching any other part of the upper geometry. The Solvent Trap sits directly on top of the Peltier Plate surface and an insulating, centering ring insures perfect placement for quick and easy sample loading. The solvent trap is also available in an insulated model. See Insulating Thermal Covers section for details.



Water at 40 °C with and without Solvent Trap



Purge Gas Cover

The Purge Gas Environmental Cover is a hard-anodized aluminum two-piece split cover with 6 mm diameter compression fittings. An insulating location ring insures precise and easy location of the cover. This cover is ideal for purging the sample area with nitrogen to prevent condensation during experiments performed below room temperature or with a humidified purge to keep a sample from drying.



Insulating Thermal Covers

Thermal Insulation Covers are constructed of an anodized Aluminum core surrounded by an insulating exterior. The aluminum core conducts heat to the upper geometry, providing uniform temperature throughout the sample. The cover is available in standard and solvent trap models. The standard cover accommodates up to 25 mm plates and can be used with all Peltier plate models. It is recommended for use over a temperature range of -10 °C to 90 °C, with samples not susceptible to drying such as oils, caulk, epoxy, and asphalt binder. The Insulated Solvent Trap Cover is compatible with the Standard Peltier Plate and geometries up to 60 mm in diameter. It is recommended for testing of low viscosity materials over the same temperature range above and offers the added benefit of evaporation prevention. Heat break geometries are recommended for use with both covers.



Peltier Immersion Ring

The Peltier Plate Immersion Ring allows samples to be measured while fully immersed in a fluid. The immersion ring is compatible with all Peltier Plate models and is easily attached to the top of the Peltier Plate. A rubber ring provides the fluid seal. This option is ideal for studying the properties of hydrogels.



Peltier Plate Covers

A variety of Peltier Plate Covers are available for applications that can harm the chromium surface of the plate or for samples that exhibit slip during testing. They are available in stainless steel, hard-anodized aluminum and titanium. Crosshatched and sandblasted Peltier covers are used to eliminate sample slippage effects. Covers are compatible with solvent trap.



Peltier Plate Camera Viewer

The camera viewer is used in conjunction with streaming video and image capture software. Real-time images can be displayed in the software and an image can be stored with each data point for subsequent viewing during data analysis. The camera viewer is perfect for long experiments with unattended operation for visual inspections of data integrity.



upper heated plate

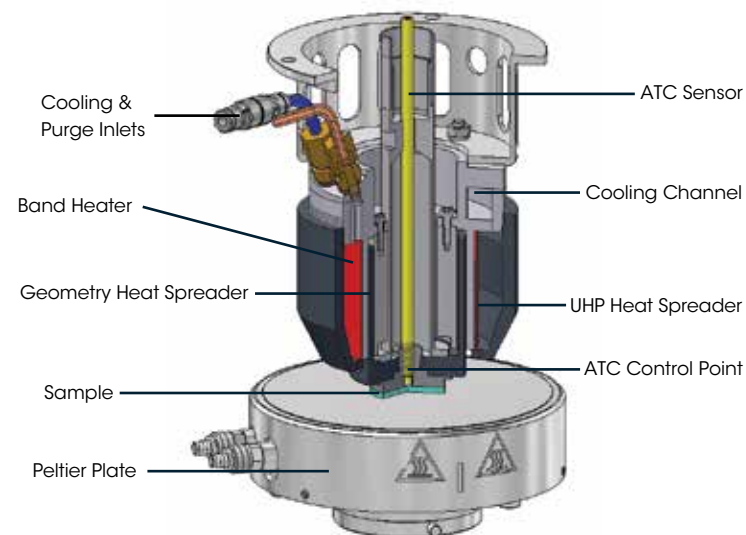
TEMPERATURE SYSTEM

Upper Heated Plate For Peltier Plate (UHP)

The UHP is a temperature option designed for use with Peltier plates to eliminate vertical temperature gradients in samples. These thermal gradients can become significant above 50 °C and lead to errors in absolute rheological data. The UHP is the most advanced non-contact heating system available, using patented heat spreader technology⁽¹⁾ to deliver maximum heat transfer efficiency and patented active temperature control⁽²⁾ for direct measurement and control of the upper plate temperature. The UHP has a maximum operating temperature of 150 °C and the lower temperature can be extended using liquid or gas cooling options. (Note: To extend the upper heated temperature range to 200 °C, see electrically heated plates option).

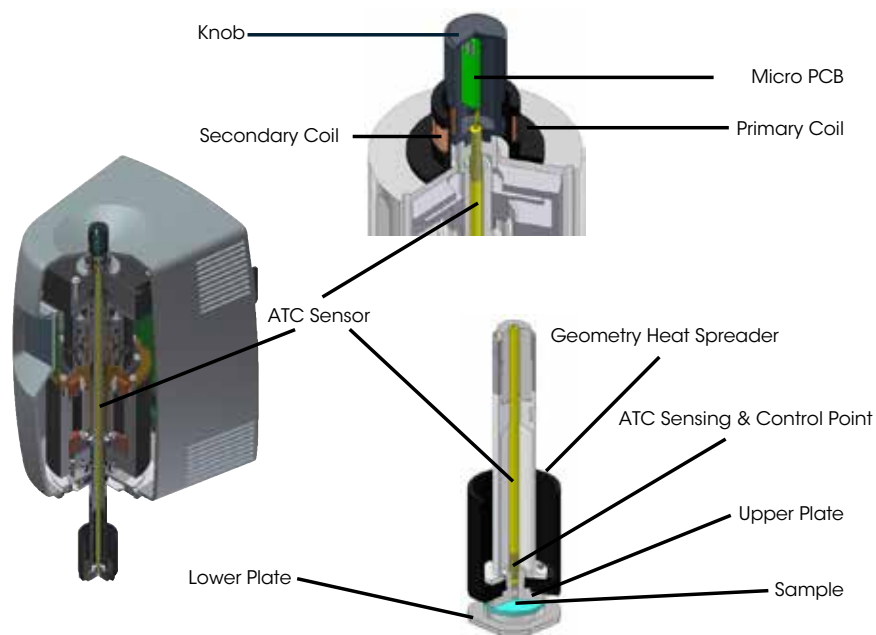
(1) Patent # 7,168,299

(2) Patent # 6,931,915



Technology

The UHP sets a new standard in non-contact heating with patented technologies that deliver the most accurate and reliable temperature control. A cylindrical heat transfer unit, with an integrated electric heater and a liquid/gas cooling channel, surrounds the cylindrical heat spreader geometry. These two components are in very close proximity, but do not contact, enabling efficient heat transfer and unimpeded torque measurement. Unlike competitive designs, the heat transfer unit and the geometry heat spreader remain in constant spatial relation to one another regardless of the test gap, keeping heat transfer uniform at all times. A unique calibration permits the system to match upper and lower plate temperatures at all heating rates, ensuring uniform sample heating on both sides, virtually eliminating the need for thermal equilibrium time and enabling true temperature ramp experiments. Conventional, non-contact upper heaters require an offset calibration between the directly measured heater temperature and the indirectly calibrated plate temperature. Patented Active Temperature Control (ATC) eliminates the need for a heater-to-plate offset table by directly measuring the upper plate temperature at all times. See ATC technology section for more details.



Features and Benefits

- Unique Smart Swap™ technology
- Wide temperature range: -30 °C* to 150 °C
- Eliminates vertical temperature gradients
- Patented heat spreader technology for optimum heat transfer
- Substantially reduces thermal equilibrium time
- Compatible with all Peltier Plate models
- Patented Active Temperature Control** for significant temperature control advantages:
 - Only active temperature measurement and control of non-contact heating system available
 - Temperature known rather than inferred
 - No need for offset calibrations and tables
 - True temperature ramp capability up to 15 °C per minute
 - Faster temperature response compared to traditional non-contact systems
- Includes sample trimming and plate removal tools
- Gas purge port and environment cover
- Disposable Plate system available for reactive systems
- Compatible with Peltier liquid cooling, circulator, or gas cooling options

*With appropriate cooling device

**Not available with disposable plates

Active Temperature Control Technology (ATC)

Patented Active Temperature Control (ATC) provides non-contact temperature sensing for active measurement and control of the upper testing surface. A Platinum Resistance Thermometer (PRT) is housed within the special ATC draw rod. This PRT is positioned in intimate contact with the center of the upper measurement surface. The temperature signal is transmitted to a micro PCB in the knob, from which the temperature reading is transmitted through a non-contact mechanism to the rheometer head assembly. This temperature reading enables direct control of the actual upper plate temperature. Because the upper plate temperature is measured and controlled directly, the system has many advantages over traditional systems. The advantages of ATC include: more responsive temperature control, no vertical temperature gradients, and no need for inferring actual temperature from complex calibration procedures and offset tables. Together with the PRT in the lower plate, real-time control of both plates allows temperature to be changed at both surfaces at the same rate for true temperature ramp profiles.

NOTE: Active Temperature Control (ATC) is available for Upper Heated Plate (UHP) and Electrically Heated Plate (EHP) temperature systems.

UHP and ATC

TEMPERATURE PERFORMANCE

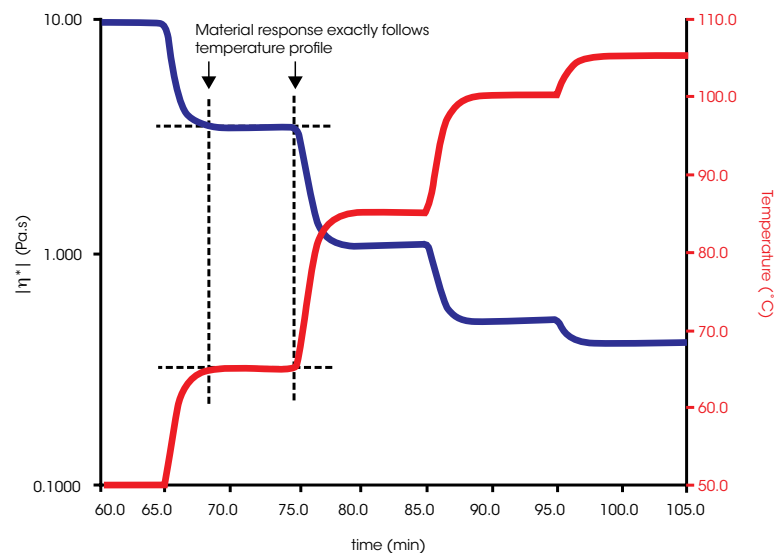
UHP and ATC Temperature Performance

The true test of any rheometer temperature control system is to compare sample response to the reported system temperature. This evaluation is especially important for a combined lower Peltier device with intimate contact heating and an upper non-contact heating device. In this situation, the top plate heats more slowly than the bottom plate. Because the top and bottom plates heat at different rates, the sample temperature differs greatly from the reported system temperature. In competitive systems, the sample response lags the temperature profile, requiring the user to determine system-based thermal equilibration times and program long delays. This issue is eliminated with TA's unique UHP and ATC technologies, by continuously controlling and matching the upper and lower plate temperatures. This accurate response is demonstrated in the following figures with simple oscillation time sweep measurements.

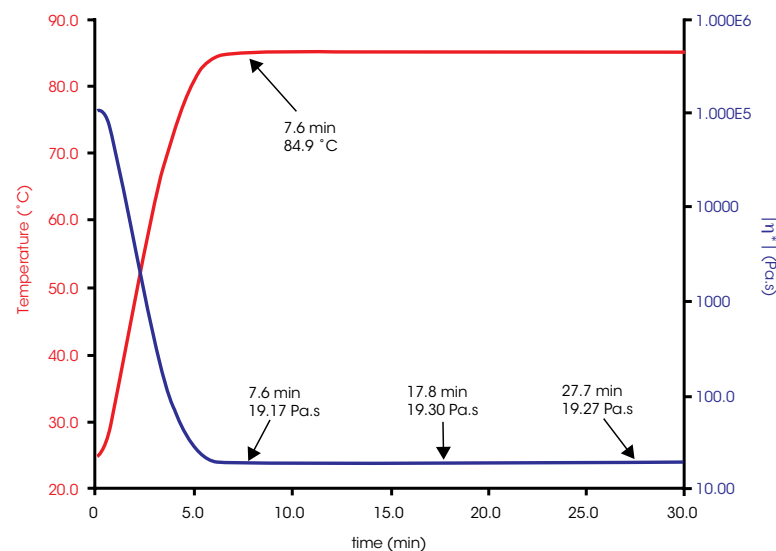
The Silicone Oil Viscosity Standard in the top figure shows that the complex viscosity response exactly follows the reported temperature profile, proving that there is no lag between set-point and real sample temperature. A similar test is shown for a sample of Asphalt binder which, according to federal standards, cannot be tested until temperature is fully equilibrated to within 0.1 °C of the set point. The asphalt data show that as soon as the temperature is within 0.1 °C, the viscosity of the asphalt binder is fully equilibrated and no change is seen in the viscosity even after 20 additional minutes.

By matching upper and lower temperature and heating rates, and actively controlling the upper plate with patented technology, TA provides the most advanced and accurate non-contact heating technology.

Silicone Oil Viscosity Standard Response to Temperature changes in UHP

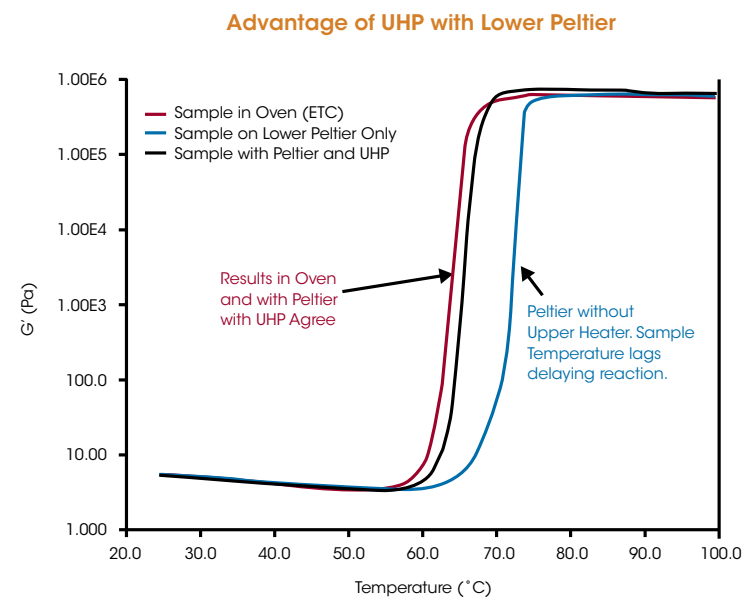


Asphalt Binder Response to Temperature Change in UHP



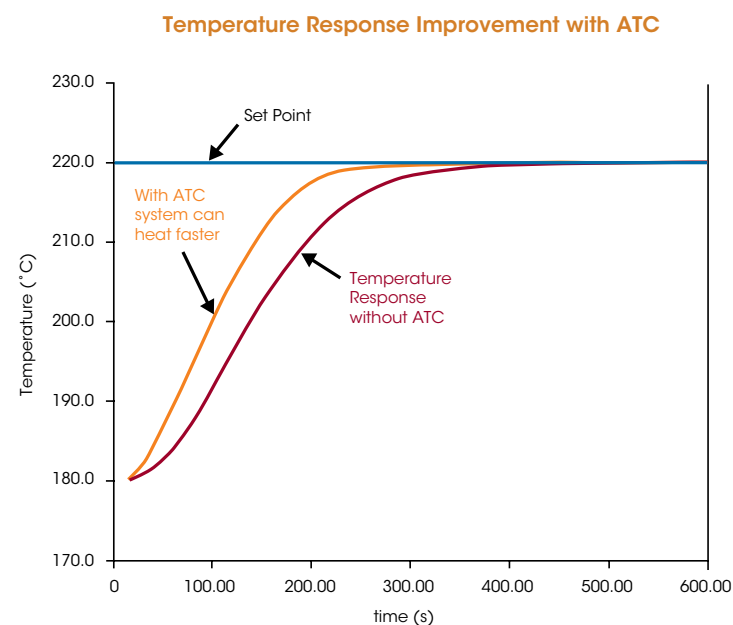
Plastisol Sample Evaluation with Upper Heated Plate

A plastisol is a mixture of resin and plasticizer that can be molded, cast, or formed into a continuous film by applying heat. Plastisols are used in applications such as screen-printing on fabrics and ink printing, where the material is hardened at moderately low temperatures. The graph shows the value of the Upper Heated Plate (UHP) for eliminating sample temperature gradients. An oscillatory time sweep was conducted on a plastisol using three temperature system configurations, including a combined convection-radiation oven, ETC, a lower Peltier Plate only, and a lower Peltier Plate with UHP. The figure shows when the sample is surrounded by heat from the top and bottom, as in the ETC and UHP, the hardening temperature, observed as a sharp increase in G' , occurs at approximately 60 °C. However, when heating the sample using only the lower Peltier Plate, the sample temperature lags behind the heating profile due to the vertical gradient, making the hardening point appear to be approximately 70 °C.



ATC Performance Advantage

Active Temperature Control (ATC) is patented technology that enables active measurement and control of non-contact upper heating systems. The benefit of this technology is that it provides optimized temperature control when compared to traditional passive non-contact heating systems that require offset calibrations. The improved responsiveness of ATC is demonstrated on a polymer melt and shown in the figure to the right. A temperature step was performed on the Electrically Heated Plates (EHP) from 180 °C to 220 °C with and without ATC. The data with ATC exhibit a faster heating rate as observed by the steeper slope, as well as a more rapid arrival to the final temperature of 220 °C.

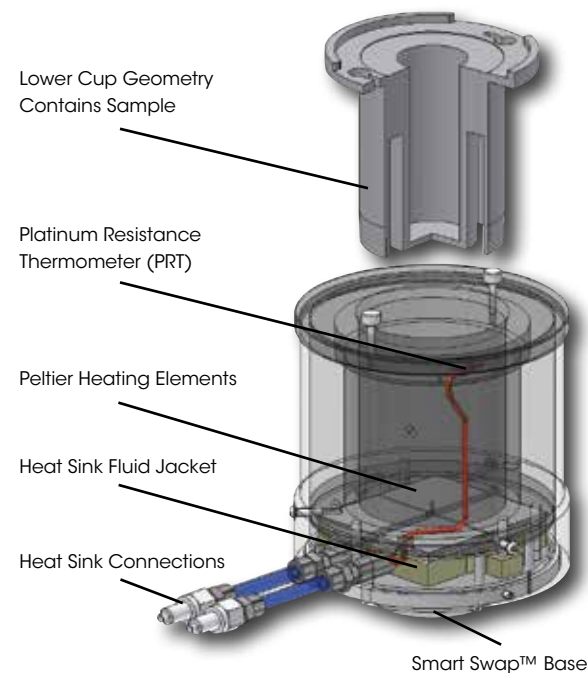


peltier concentric cylinder

TEMPERATURE SYSTEM

Peltier Concentric Cylinder Temperature System

The Peltier Concentric Cylinder Temperature System combines the convenience of Smart Swap™ and Peltier heating technology with a wide variety of cup and rotor geometries. Concentric Cylinder geometries are commonly used for testing low viscosity fluids, dispersions or any liquids that are pourable into a cup. Examples of materials suitable for Concentric Cylinder include low concentration polymer solutions, solvents, oils, drilling mud, paint, varnish, inkjet ink, ceramic slurries, pharmaceutical suspensions such as cough medicine and baby formula, foams, food products such as juices, thickeners, dairy products such as milk and sour cream, salad dressings, and pasta sauce.



Technology

The Peltier Concentric Cylinder system offers a temperature range of -20 °C to 150 °C, with a maximum heating rate up to 13 °C/min. Four Peltier heating elements are placed in intimate contact with a lower cup geometry held in place by an insulated jacket. The unique patented design⁽¹⁾ of the lower geometry provides fast and efficient heat transfer up the walls of the cup. A platinum resistance thermometer (PRT) is placed close to the top of the cup ensuring accurate temperature measurement and control. The maximum controllable heating rate will depend on heat sink fluid temperature, circulator flow rate and cooling/heating capacity, and viscosity of heat sink fluid.

(1) Patent # 6,588,254



Cup and Rotor Geometries

The standard Peltier Concentric Cylinder geometries include a cup radius of 15 mm, configured with either a Recessed End or DIN Rotor. Both rotors have a radius of 14 mm and height of 42 mm. The double gap concentric cylinder has an additional shearing surface over single gap providing lower stress and higher sensitivity for extremely low viscosity solutions.

Special Cups and Rotors

Specialty geometries include various vanes, helical, and starch pasting impeller rotors, as well as large diameter and grooved cups. These special concentric cylinder geometries are very valuable for characterizing dispersions with limited stability, preventing error from slip at the material/geometry interface, and for bulk materials with larger particulates. Vane geometries are available in both a 14 mm and 7.5 mm radius. The large diameter cup has a radius of 22 mm. The helical and impeller rotor and cup keep a sample mixed or particles suspended during shearing.

Features and Benefits

- Unique Smart Swap™ technology
- Wide temperature range: -20 °C to 150 °C
- Peltier temperature control for fast heating and cooling
- Popular DIN standard, Recessed End and Double Gap Options
- Geometries available in Stainless Steel and Hard Anodized Aluminum
- Wide variety of cup diameters
- Impeller and Vane geometries for preventing settling and slipping, and handling of large particles
- Pressure Cell
- Torsion Immersion
- Special geometries available upon request



Concentric Cylinder Cup and Rotor Compatibility Chart

Cup/Rotor	DIN	Recessed End	Starch Impeller	Vane	Wide Gap Vane	Double Gap	Helical Rotor
Standard (rad= 15 mm)	●	●		●	●		
Large Diameter (rad= 22 mm)	●	●	●	●	●		●
Starch (rad= 18.5 mm)	●	●	●	●	●		●
Grooved				●	●		
Double Gap						●	
Helical (rad= 17 mm)							●

concentric cylinder

ACCESSORIES AND APPLICATIONS



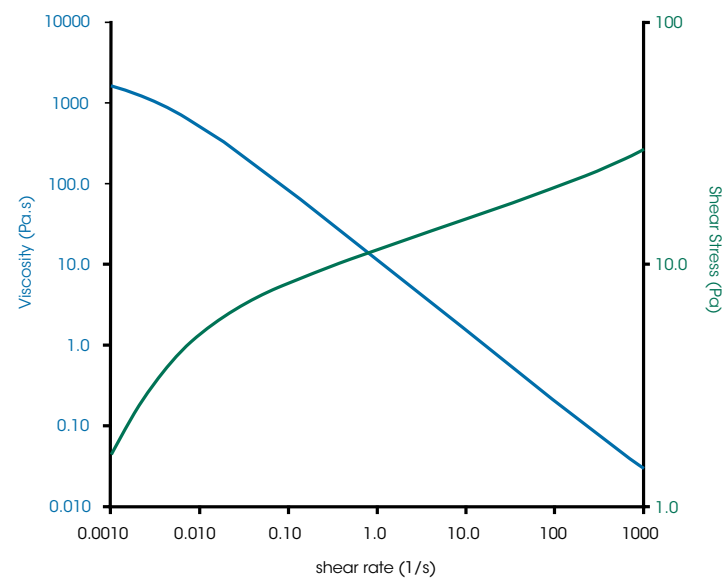
Generic Container Holder

The Generic Container Holder is a Smart Swap™ option that can hold any container with an outer diameter of up to 80 mm for characterizing materials with rotors. This allows for quick off-the-shelf evaluation of materials, such as paints and varnishes, creams, pasta sauce, etc., without creating large shearing from sample loading. It also is an excellent platform for beakers or jacketed beakers.

Flow Curve on Xanthan Gum

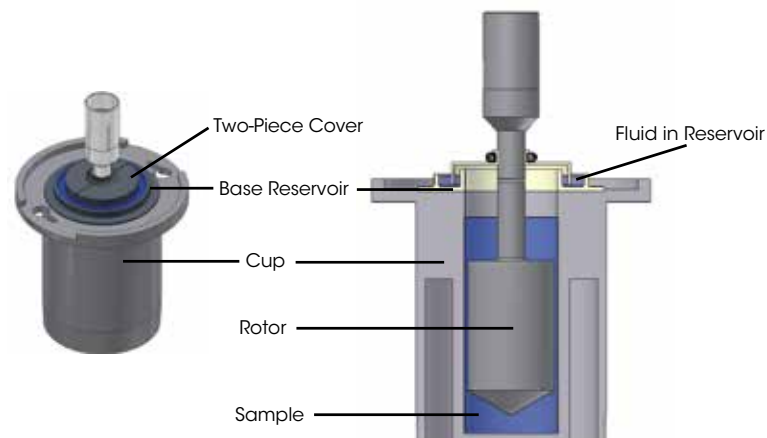
Concentric Cylinder geometries are useful for gathering viscosity flow curve information over a wide range of shear rates. An example is shown in the figure for a Xanthan Gum solution. Five decades of viscosity are easily obtained over six decades of shear rate. This system is also a powerful alternative to parallel plate or cone and plate geometries for materials with limited stability or prone to edge failure or rapid solvent evaporation.

Xanthan Gum Solution in Concentric Cylinder



Concentric Cylinder Solvent Trap Cover

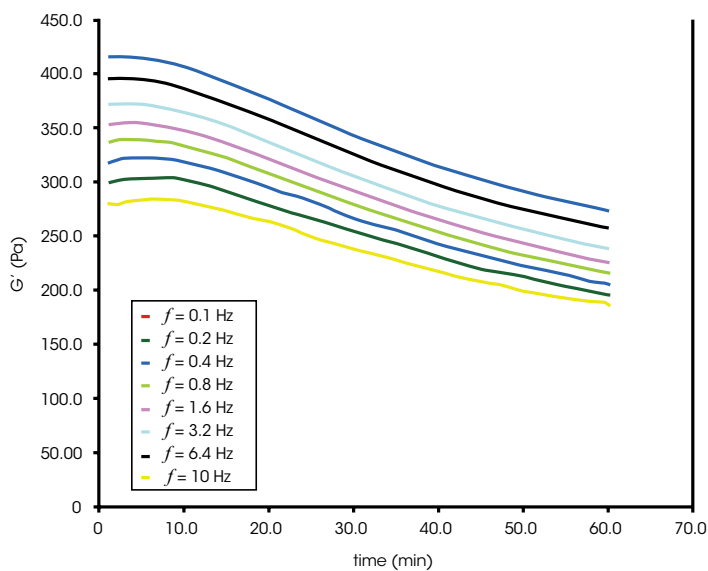
A Solvent Trap is available for the Peltier Concentric Cylinder. It includes a base reservoir and a two-piece cover that is mounted to the shaft of the rotor. The Solvent Trap provides a vapor barrier to seal the environment inside the cup and prevents solvent evaporation.



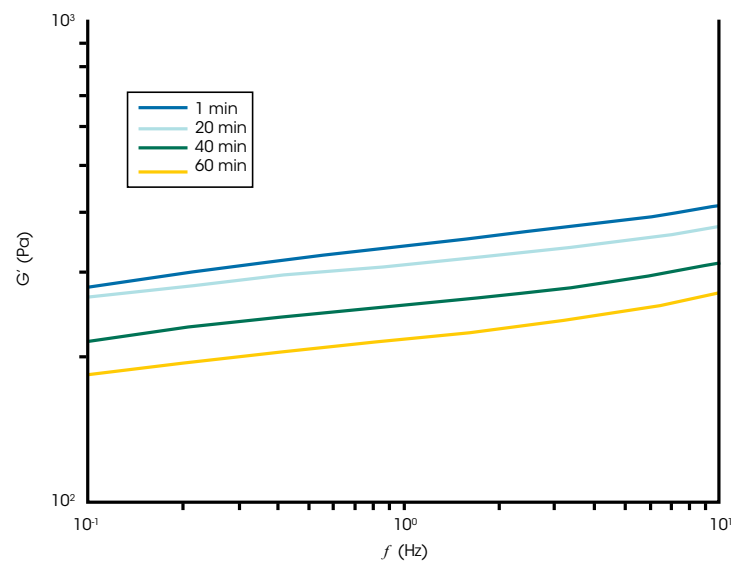
Characterization of Foam with Vane Rotor

The figures below show an example of the time and frequency dependent response of a foam shaving cream characterized using a standard cup and vane geometry. The structure of shaving foam has a limited lifetime, or limited stability. The vane geometry minimizes shearing stress that occurs during loading in the gap with standard rotor, keeping the delicate foam structure intact for testing. A wide range of structural information can be captured very quickly using multiwave characterization on the DHR. The figure to the left shows a decay in storage modulus G' as the structure of the foam breaks down with increasing time. Using the multiwave, the data are simultaneously collected over a wide range of frequencies. The data can be plotted as frequency sweeps at increasing time, as shown on the right. These data show the time dependent viscoelastic response of the shaving foam.

Multiwave time sweep



Frequency Sweeps on Foam at Different Time Intervals



concentric cylinder

ACCESSORIES

Torsion Immersion Cell

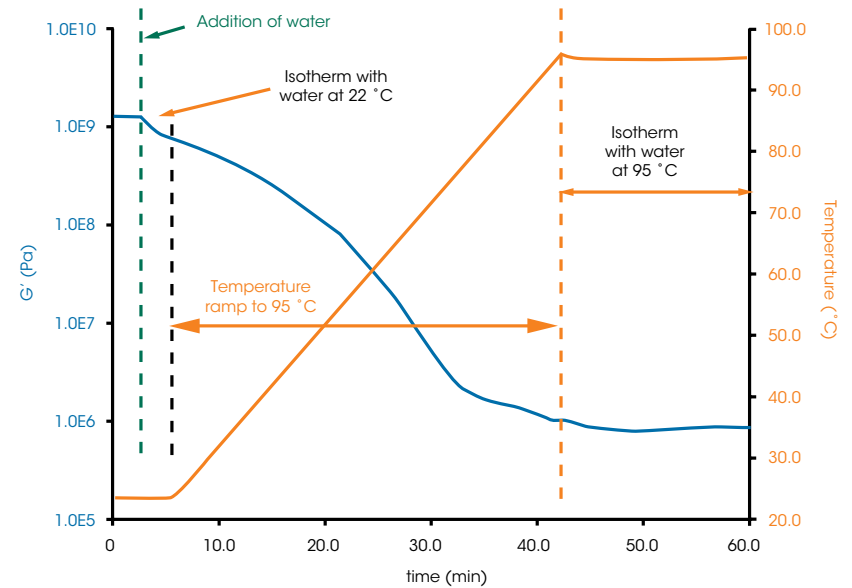
The Torsion Immersion Cell allows rectangular bar-shaped samples to be clamped and characterized while immersed in a temperature controlled fluid. The resulting change in mechanical properties, caused by swelling or plasticizing, can be analyzed in oscillatory experiments. This option provides a way to better understand materials under real-world conditions, such as body implants in saline or rubber seals in contact with oils and solvents.

Rheology of Pasta During Cooking

The Torsion Immersion Cell can be used for various food applications such as cooking of pasta. In this example, a piece of fettuccini pasta was tested using an oscillatory time sweep test at a frequency of 6.28 rad/s and temperature of 22 °C. Data were collected on the dry sample for 2.5 min to establish a baseline storage modulus G' . Water was added after 2.5 min and the effect of the moisture is seen immediately as a decrease in the G' . At 5 min, G' was monitored as temperature was ramped to 95 °C and held isothermally. As the pasta cooks the modulus drops about three decades and then levels out when cooking is complete.



Rheology of Pasta During Cooking



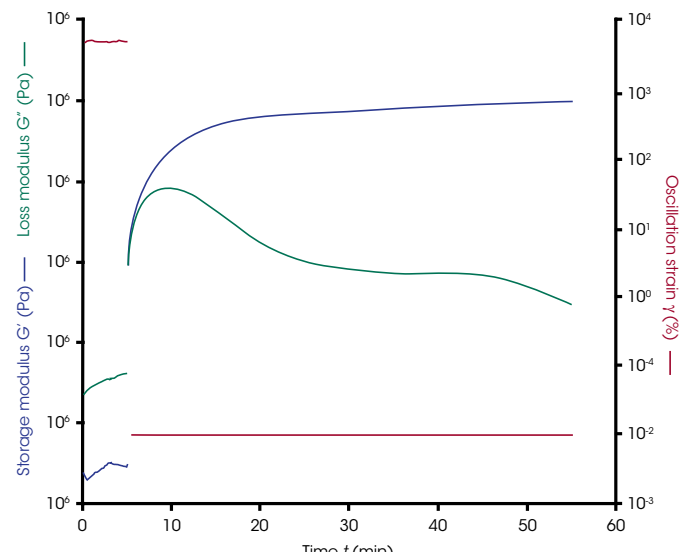


DHR Building Materials Cell

The Building Materials Cell is a specially designed, abrasion-resistant and durable concentric cylinder cup and rotor for testing samples with large particles such as concrete slurries and mixes. The paddle type rotor, slotted cage, and the large diameter cup promote adequate sample mixing while preventing sample slip at both the cup and rotor surfaces. The removable slotted cage permits easy sample cleaning after the test while the concentric cylinder Peltier jacket provides accurate temperature control. Together with the existing array of specialty rotors and cups, the new Building Materials Cell provides the ultimate flexibility for testing a diverse range of samples with large particles including construction materials and food products.

The data below follows the structural recovery of a concrete mixture tested at 25 °C using the Building Materials Cell. The concrete sample was initially subject to large deformation to mimic processing conditions encountered during pumping. A subsequent small strain fast oscillation test simulated the development of the sample's moduli following flow cessation. The results reveal a rapid increase in the material's storage modulus within 10 minutes, before ultimately reaching a plateau value.

Concrete Recovery Following Pumping



Paddle Rotor



Slotted Cage



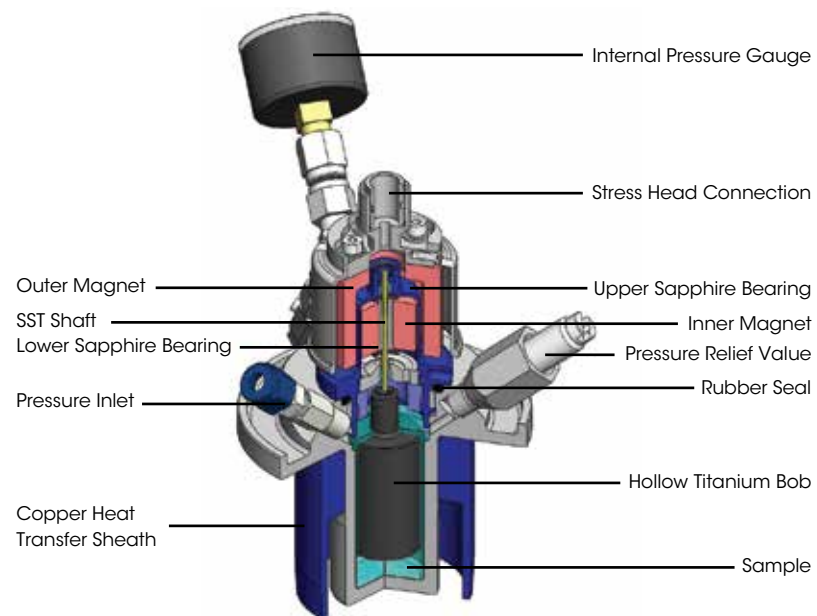
Large Diameter Cup

pressure cell

ACCESSORY

Pressure Cell Accessory

The Pressure Cell is a sealed vessel that can be pressurized up to 138 bar (2,000 psi), over a temperature range of -10 °C to 150 °C. It can be used either in self-pressurizing mode, in which the pressure is produced by the volatility of the sample or by externally applying the pressurization, typically with a high pressure tank of air or nitrogen gas. Comes standard with 26 mm conical rotor and optional vane rotor is available. All necessary plumbing and gauges are included as a manifold assembly. The Pressure Cell is ideal for studying the effect of pressure on rheological properties, as well as studying the materials that volatilize under atmospheric pressure.



Technology

The pressure cell accessory is used with the Peltier Concentric Cylinder jacket. The sealed and pressurized volume is contained within a stainless steel cup to withstand high pressures and is surrounded by a copper sheath for optimal heat transfer. Also connected to the cup are the pressure inlet, internal pressure gauge, and pressure relief valve. An outer magnet assembly is attached to the rheometer drive shaft and houses strong rare earth magnets. The rotor assembly houses the inner magnet which couples to, and is levitated by, the outer magnet assembly and drives the hollow titanium cylindrical rotor. The rotor shaft is made from low friction Titanium nitride-coated stainless steel and is supported above and below by precision sapphire bearings. This innovative high-powered magnetic coupling and low-friction bearing design allows for both steady shear and dynamic measurements.



Pressure Cell Rotors

The Pressure Cell Accessory is compatible with any of three rotor designs. The standard rotor is the Conical Rotor, which is ideal for the quantitative measurement of liquids. Suspensions and slurries may be more well-suited to testing using the Starch Rotor, which prevents settling, or the Vane Rotor that is tolerant to larger particles and inhibits slip.



Conical Rotor

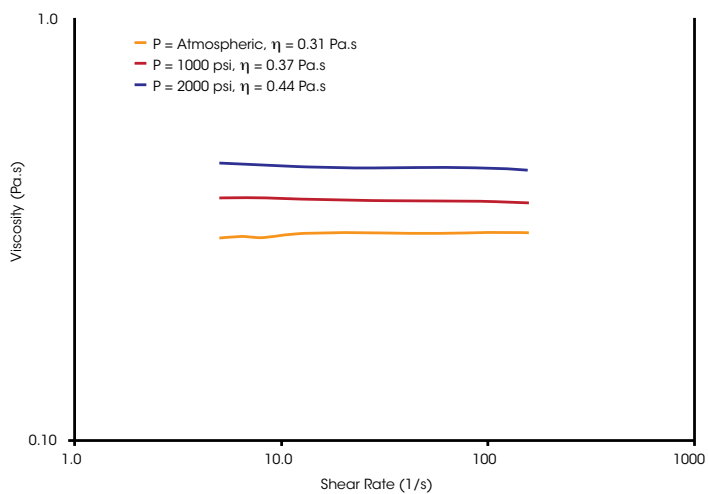


Starch Rotor



Vane Rotor

Effect of Pressure on Viscosity of Motor Oil



Effect of Pressure on Motor Oil

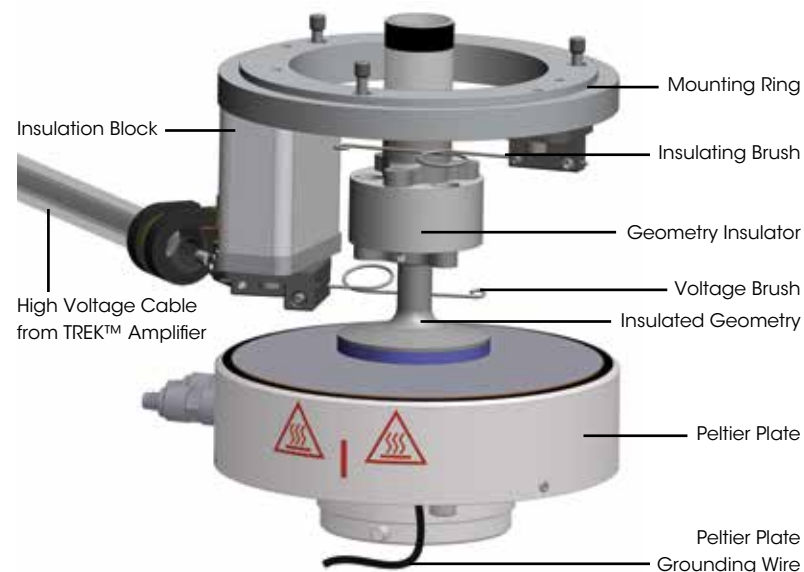
To understand the ability of motor oil to provide necessary lubrication under different environmental conditions, it is critical to know its viscosity over a range of temperatures and pressures. The figure to the left shows results of steady state flow tests conducted on automotive motor oil. The temperature was held constant at 20 °C and tests were run at atmospheric pressure, 1,000 psi (69 bar), and 2,000 psi (138 bar) of pressure. The results show that the pressure acts to increase internal friction, as observed by the increase in viscosity.

electrorheology

ACCESSORY

Electrorheology Accessory (ER)

Electrorheological, or "ER", fluids are suspensions of extremely fine non-conducting particles in an electrically insulating fluid. These materials show dramatic and reversible rheological changes when the electric field is applied. The Discovery Hybrid Rheometer ER accessory provides the ability to characterize ER fluids up to 4,000 volts using either parallel plate or concentric cylinder geometries. The accessory is available for all DHR models and compatible temperature systems include the popular Peltier Plate (-40 °C to 200 °C) and Peltier Concentric Cylinder (-20 °C to 150 °C). A custom waveform and function generator enables the user to program a wide range of voltage profiles directly in TRIOS Software. Voltage Profiles include: constant voltage, step voltage, ramp voltage, sine wave voltage function, triangle wave voltage function, and wave functions with DC offsets. There are no limitations to the type or rheological experiments that can be performed with this accessory. A protective polycarbonate shield with trigger interlocks is also included with the accessory to provide safety from electrical shocks.



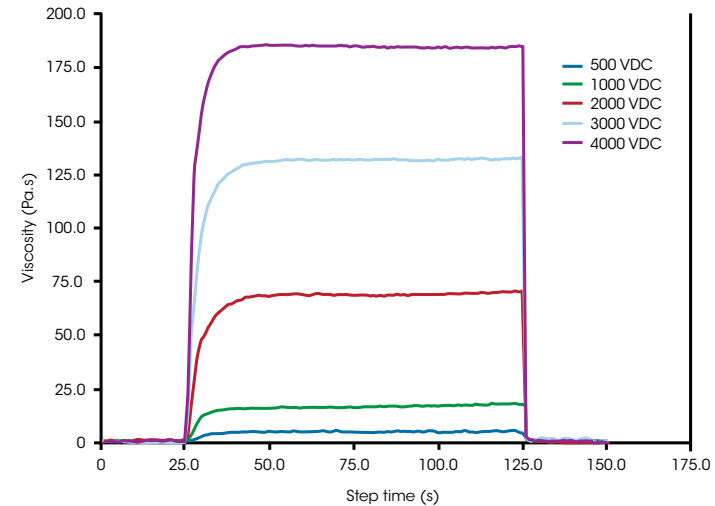
Features and Benefits

- Smart Swap™ technology
- Easy installation and removal
- Compatible with Peltier Plate and Peltier Concentric Cylinder
- 25 mm and 40 mm diameter plates with ceramic insulation
- 28 mm diameter DIN concentric cylinder rotor
- Wide voltage range: 4000 VDC, 4000 VAC (8000 V peak to peak)
- Compatible temperature systems
 - Peltier Plate: -40 °C to 200 °C
 - Peltier Concentric Cylinder: -20 °C to 150 °C
- Fully programmable from TRIOS Software
- Flexible voltage profile programming including:
 - constant voltage
 - step voltage, ramp voltage
 - sine wave voltage function
 - triangle wave voltage function
 - wave functions with DC offsets

Step Voltage on Starch Suspension under Steady Shear

A 10% starch solution in silicon oil demonstrates dramatic and reversible changes in structure under the application of high voltage. The figure shows time dependent viscosity with varying DC voltage, from 500 to 4000 V, applied for 100 s. The underlying rheological test is a constant rate at 1 s^{-1} , which minimizes the disturbance to the structuring process. When an electrical field is applied, polarization of the starch particles in the non-conducting silicone oil leads to stringing of the starch particles, which align between the electrode plates. This orientation is responsible for the strong viscosity increase. The time to align the particles depends on the viscosity of the suspending fluid and the strength of the electrical field. Because under the applied shear rate, deformation of the structuring process is not completely eliminated, a maximum viscosity is observed when the dynamic equilibrium between forming and breaking of strings of aligned particles is achieved.

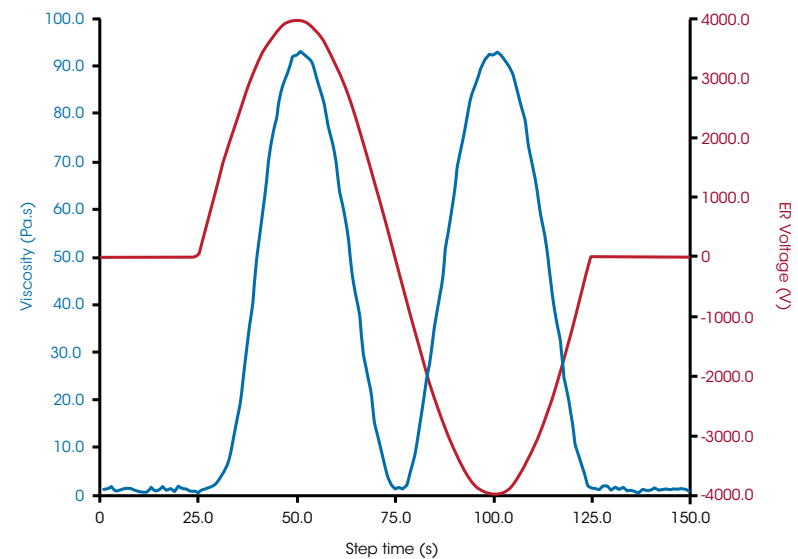
DC Voltage Response of a Starch in Oil Suspension



Sinusoidal Voltage Oscillation under Steady Shear

Electrorheological materials demonstrate interesting responses to AC voltage profiles. The figure to the right shows the viscosity response at a constant shear rate when an AC voltage with a peak maximum of 4000 V and a frequency of 0.01 Hz is applied to a starch in oil suspension. Under this applied electrical profile, it can be seen that the viscosity changes with twice the frequency of the voltage, or put another way, is in phase with the absolute value of the voltage. This behavior occurs because the viscosity is independent of the sign of the voltage. When the electrical field has zero voltage, the viscosity has the lowest value.

AC Voltage Response of a Starch in Oil Suspension

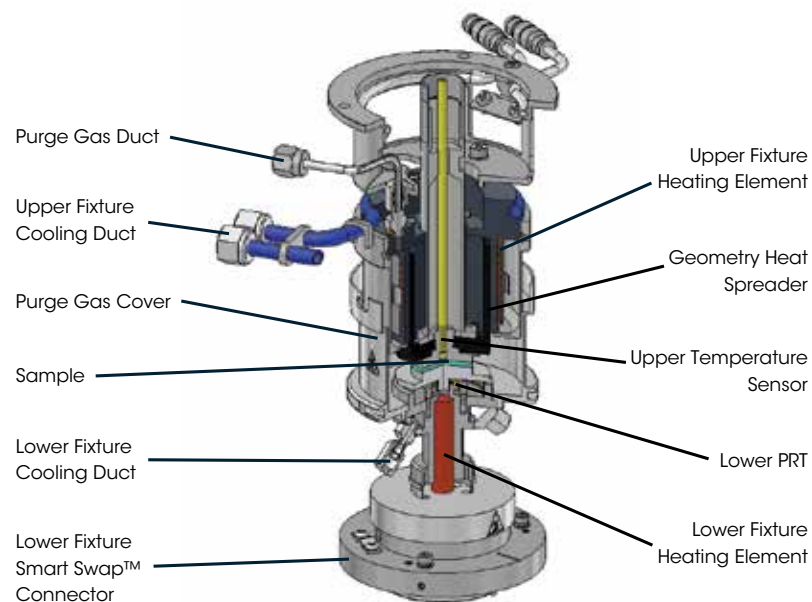


electrically heated plate

TEMPERATURE SYSTEM

Electrically Heated Plate (EHP) Temperature System

The EHP provides active heating and cooling of parallel plate and cone and plate geometries. With standard and disposable systems it's ideal for rheological characterization of polymer melts and thermosetting materials up to a maximum temperature of 400 °C. The optional Gas Cooling Accessory extends the minimum temperature to -70 °C. Standard features include 25 mm diameter parallel plate geometry, environmental cover, and heated purge gas. An optional clear cover is available for sample viewing and for use with the Camera Viewer option. The EHP offers Active Temperature Control (ATC) making it the only EHP system capable of direct temperature control of the upper and lower plates (See ATC Section for more details on this exciting technology). The Upper EHP can be used with lower Peltier Plates for temperature control to 200 °C and as temperature control to 150 °C for UV curing options.



Technology

The EHP lower assembly incorporates a cartridge-heater and cooling channel directly below the lower plate. A Platinum Resistance Thermometer (PRT) is positioned at the center and is in contact with the opposite face of the lower plate, providing intimate measurement and control of the sample temperature. In the upper assembly, a cylindrical heat transfer unit, with an integrated electric heater and a liquid/gas cooling channel, surrounds the cylindrical heat spreader geometry. These two components are in very close proximity, but do not contact, enabling efficient heat transfer and unimpeded torque measurement. Unlike competitive designs, the heat transfer unit and the geometry heat spreader remain in constant spatial relation to one another regardless of the test gap, keeping heat transfer uniform at all times. A unique calibration matches upper and lower plate temperatures at all heating rates, ensuring uniform sample heating at both sides – virtually eliminating the need for thermal equilibrium time and enabling true temperature ramp programming. Patented Active Temperature Control (ATC) eliminates the need for upper heater offset calibrations by providing actual measurement and control of the upper plate temperature. See ATC technology section for more details. A heated purge and cover create an oxygen-free environment around the sample to inhibit sample degradation.





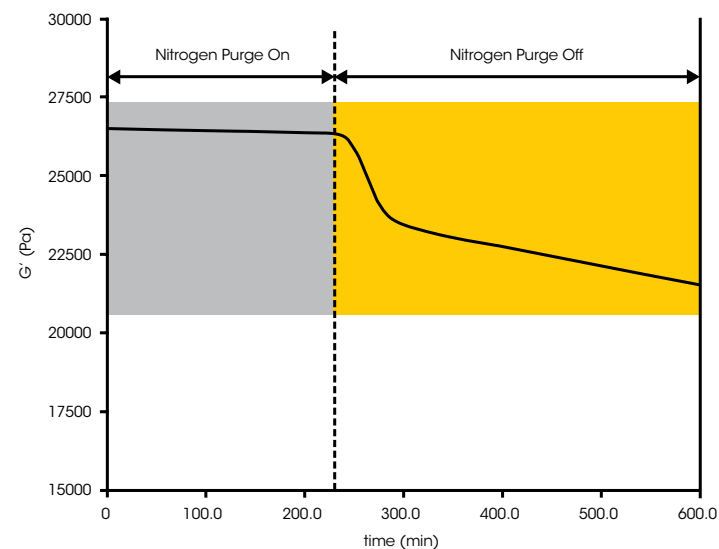
Controlling Polymer Degradation During Testing

Viscoelastic properties of polymer melts can be affected by thermal and oxidative degradation at elevated temperatures. It is important to measure rheological properties in the absence of degradation, as well as evaluating the effectiveness of stabilizing additives such as antioxidants. This figure shows how effectively the EHP controls the environment for commercial polystyrene melt during a 10-hour time sweep experiment at 200 °C. The storage modulus, G' , can be seen to be very stable during the early stages of the test when the sample is purged with nitrogen. The data demonstrate the environment in the EHP is virtually oxygen-free. After about 4 hours, the inert gas is shut off and the effect of presence of oxygen on the viscoelastic response is seen immediately. The polystyrene degrades as evidenced by the sharp decrease in G' .

Features and Benefits

- Unique Smart Swap™ technology
- ATC with patented Non-Contact Upper Temperature Sensor
- Patented Smart Swap™ geometries
- Maximum temperature of 400 °C
- Optional low temperature cooling to -70 °C
- Maximum heating rate of up to 30 °C/min
- Controllable heating rates of 10 °C/min
- Environmental cover and heated purge gas
- Modeled for optimum heat transfer with minimum sample thermal equilibration time
- Heat transfer to sample independent of gap setting
- Sample trimming and plate removal tools
- Disposable Plate System
- Optional glass cover for sample viewing and for use with camera
- Ideal for QC testing or R&D
- Upper heater compatible with all Peltier Plate systems and UV Curing accessories

Controlling Polymer Degradation During Testing



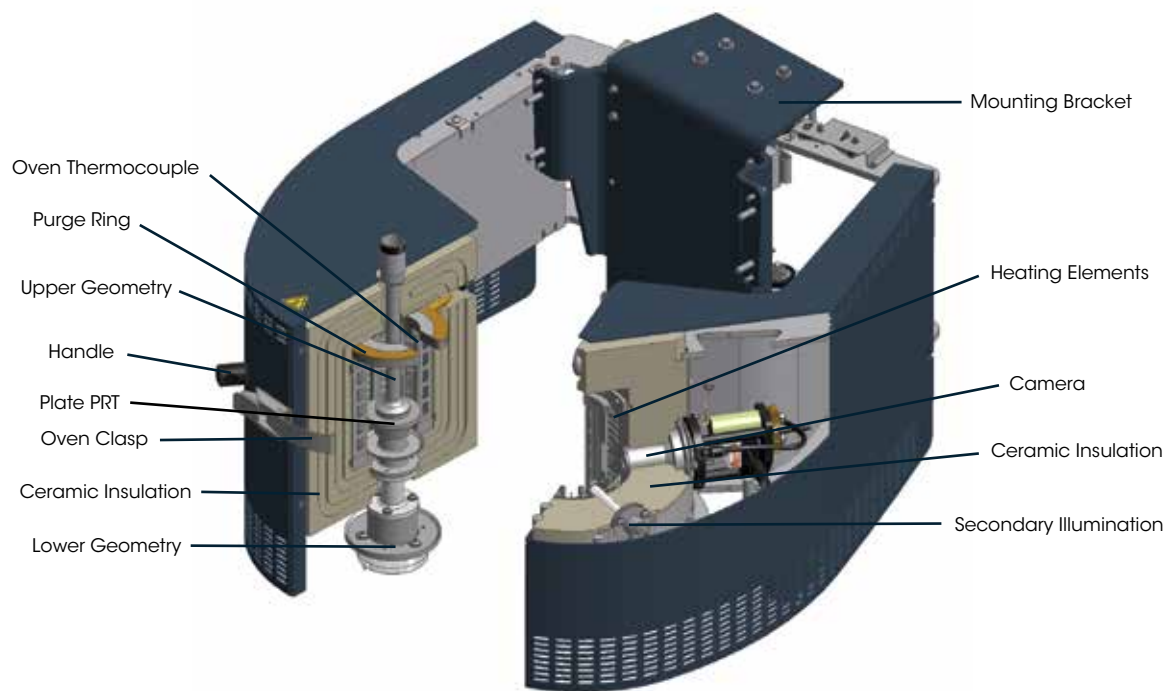
ETC oven

ENVIRONMENTAL TEST CHAMBER

Environmental Test Chamber (ETC)

The ETC is a high temperature Smart Swap™ accessory that employs a combination of radiant and convective heating and has a temperature range of -160 °C to 600 °C with heating rates up to 60 °C/min. This hybrid temperature control design provides fast response and temperature stability over a continuous range of 760 °C. The ETC is a very popular option for polymer applications and can be used with parallel plate, cone and plate, disposable plate, rectangular torsion clamps for solids, and the SER2 for extensional viscosity measurements. Typical materials that can be tested include thermoplastics, thermosets, elastomers, caulks and adhesives, solid polymers, asphalt binder, and oils and greases.





Features and Benefits

- Unique Smart Swap™ technology
- No extra electronic boxes
- Combined convection and radiant heating design
- Wide temperature range: -160 °C to 600 °C
- Maximum heating rate of 60 °C/min
- Liquid nitrogen option connects directly to bulk source
- Wide variety of stainless steel plates, cones, crosshatched, and disposable geometries
- Optional built-in camera viewer
- Melt rings for thermoplastic pellet samples
- Die punch for molded plaques
- Sample cleaning and trimming tools
- Extensional rheology measurements with SER2 Universal Testing Platform

ETC Technology

The Smart Swap™ ETC is a “clam-shell” design. Each half of the oven contains an electric radiant heating element surrounded by a ceramic insulation block. Air or nitrogen gas is introduced through a purge ring with the gas flow directed over the heated coils, providing heat transfer by convection. For subambient testing, both gas and liquid nitrogen are fed through the purge ring. Liquid nitrogen usage is kept to a minimum by switching from gas to liquid nitrogen only when cooling is required. Temperature sensors are used both in the oven and below the lower plates for temperature measurement and control. The ETC also features a camera viewer option installed through the chamber’s right-side ceramic block. The camera option features multiple light sources, remote focusing, and can be used over the entire temperature range. Used in conjunction with the streaming video and image capture software, real-time images can be displayed in the software and an image is stored with each data point for subsequent viewing. The ETC camera viewer is an ideal tool for data validation.

ETC Geometry Accessory Kits

The ETC features five standard geometry accessory kits configured for thermoplastics and rubber, thermosetting and other curing systems, solid polymers, pressure-sensitive adhesives, asphalt binder, and extensional viscosity with the SER2 Universal Testing Platform. In addition, a wide variety of stainless steel geometries of various diameters and cone angles, and a wide variety of disposable plates are available to fully accessorize the temperature system. The kits feature patented Smart Swap™ geometries that are automatically recognized and configured for use.



Parallel Plate



Disposable Plate



Torsion Rectangular



Torsion Cylindrical



SER2 Extensional

Air Chiller System (ACS-3)

The new Air Chiller System, ACS-3, is a unique gas flow cooling system that enables temperature control of the Environmental Test Chamber to temperatures as low as -85 °C. Equipped with a three-stage cascading compressor design, the ACS-3 allows for low temperature environmental control without the use of liquid nitrogen, instead utilizing compressed air (7 bar, 200 L/min) as the cooling medium. The ACS-3 can help eliminate or reduce liquid nitrogen usage and associated hazards from any laboratory and offers an incredible return on investment.

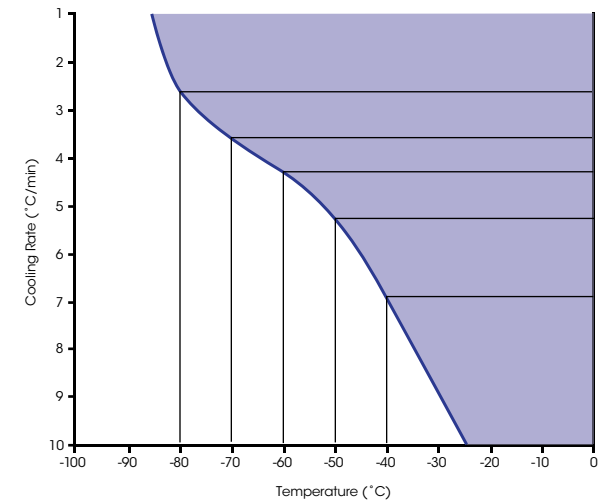


Features and Benefits

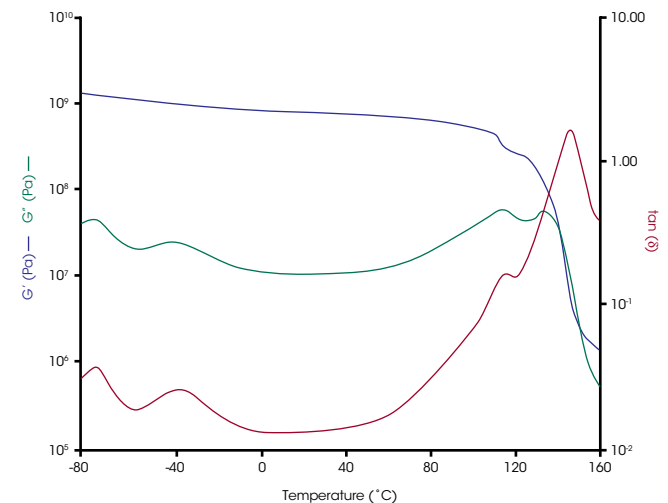
- Safe: eliminates the need for liquid nitrogen or other refrigerated gases
- Convenient: never change, refill, or order another tank of liquid nitrogen. The ACS-3 is ready to run whenever you are.
- Small: occupies less space than equivalent liquid nitrogen cooling systems.
- Quiet: operating noise level of 55 dB
- Affordable: provides considerable cost savings over recurring gas deliveries.

Low Temperature Polymer Transitions

Polymers are often blended to produce a desirable combination of toughness, modulus, and processing characteristics. One such combination is a blend of polycarbonate (PC) with acrylonitrile butadiene styrene (ABS). The ACS-3 provides a sufficient range of temperature control to characterize the multiple low and high temperature transitions of this multi-component sample. The data in the figure were collected during a temperature ramp with a rectangular specimen in torsion.



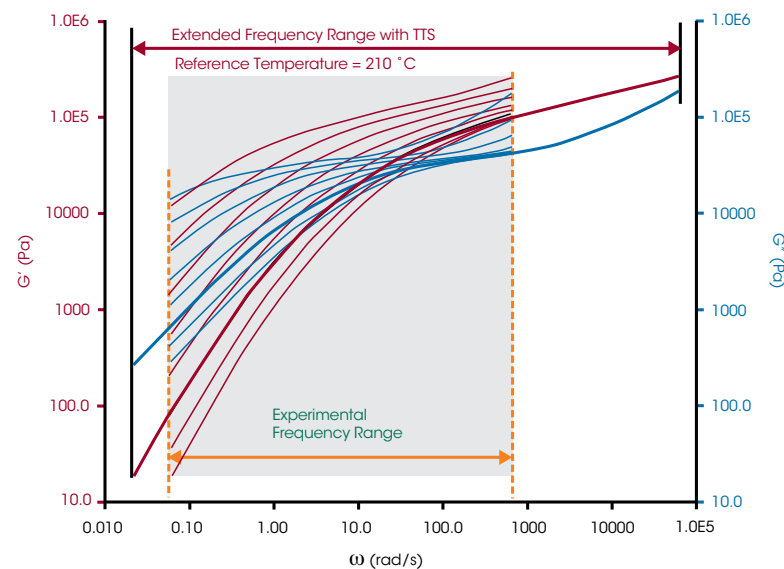
ABS/PC Blend Temperature Ramp



Thermoplastic Polymer Rheology Using Parallel Plate

Parallel Plate geometries are most popular for testing of thermoplastic polymer melts. An example of polystyrene characterized over a temperature range of 160 °C to 220 °C is shown in the figure to the right. Frequency sweeps were run at multiple temperatures over an experimental range of 0.06 to 628 rad/s. The magnitude of the viscoelastic properties, storage modulus (G') and loss modulus (G''), for the individual sweeps can be seen to decrease with increasing temperature over this frequency range. Since polymer melts are viscoelastic, their mechanical response will be time dependent, so low frequency corresponds to long time behavior. Time Temperature Superposition (TTS) is used to widen the range of data to higher and lower frequencies and generate a Master curve at a reference temperature pertinent to the application. The polymer's molecular structure dictates the magnitude and shape of the G' and G'' curves.

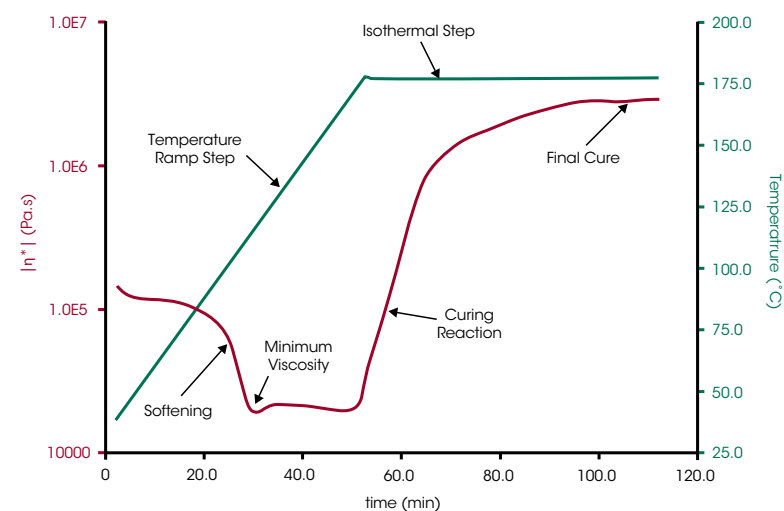
Polystyrene Frequency Sweeps from 160 °C to 220 °C



Thermoset Cure Using Disposable Plate Kit

Changes in the viscoelastic properties of reactive systems provide valuable information about their processing and end-use properties. Often these materials can start in the form of a low viscosity liquid, a paste, or even a powder, and after reacting end as a high modulus solid. If the material hardens and adheres to the testing surface, low-cost disposable parallel plates are required for testing. The figure to the right shows the complex viscosity from a typical oscillatory temperature ramp and hold curing test conducted on a B-stage prepreg using 25 mm diameter disposable plates. The resin is impregnated on a woven glass matrix, which becomes part of the permanent high strength composite structure once the resin is cured. Upon heating, the resin softens until reaching a minimum viscosity, which is a very important processing parameter. If the viscosity is too high or too low, the resin will not flow or coat the matrix uniformly leaving voids, creating flaws in the composite. Eventually the viscosity starts building and the curing reaction takes off dramatically. The temperature is held constant at the processing temperature and the viscosity is monitored until the viscosity reaches a plateau indicating the completion of the reaction.

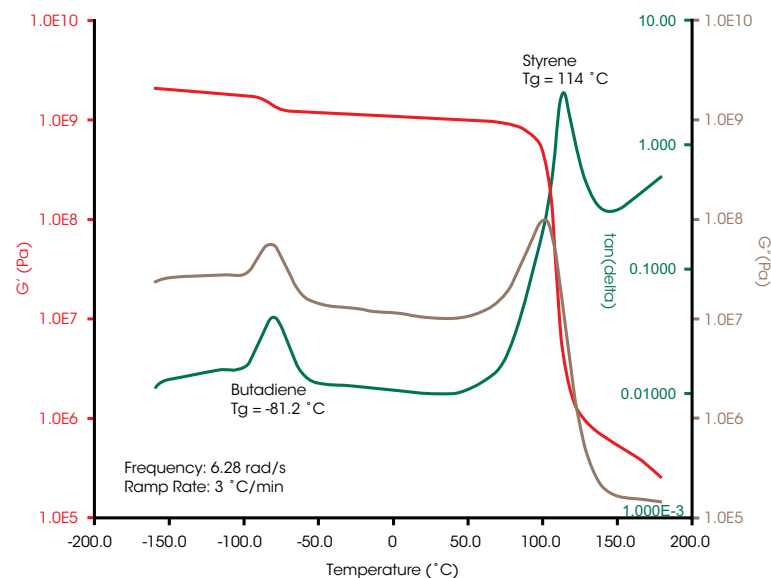
B-Stage Prepreg Temperature Ramp and Hold Cure



Solid Polymer Rheology Using Torsion Rectangular Clamps

The ETC oven has the ability to characterize the viscoelastic properties of rectangular solid samples up to 5 mm thick, 13 mm wide, and 50 mm long. The figure to the right shows an example of an oscillatory temperature scan on a solid ABS specimen run at 3 °C/min from -160 °C to 200 °C. The ABS is a copolymer that exhibits two glass transition temperatures: one at -81 °C associated with the Butadiene and one at 114 °C associated with Styrene. Transitions, or relaxations of molecular segments, are observed as step changes in the storage modulus (G') and as peaks in the loss modulus (G'') and damping ($\tan\delta$). The magnitude and shape of these parameters will depend on chemical composition as well as physical characteristics (such as crystallinity, orientation, fillers, and degree of cross-linking).

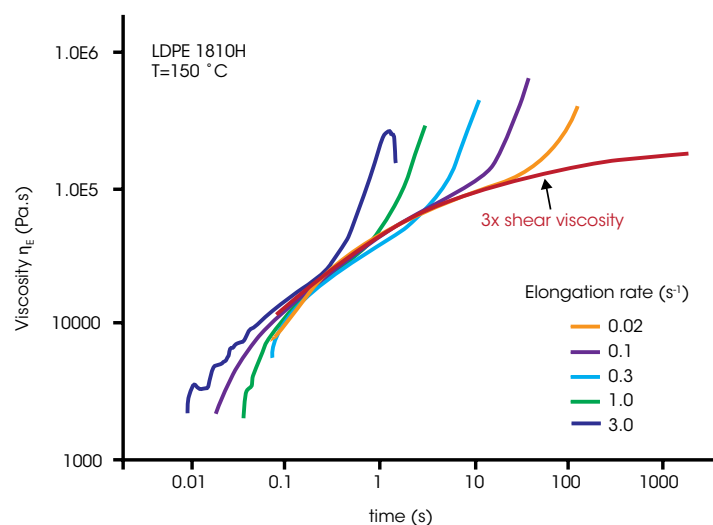
ABS Oscillation Temperature Ramp in Torsion Rectangular



Extensional Viscosity measurements with the SER2 attachment

The ETC is designed to accommodate the SER2 (Sentmanat Extension Rheometer) for extensional viscosity measurements of polymer melts. In the figure to the right, data are plotted for extensional viscosity measurements of standard LDPE 1810H at 150 °C at extensional rates from 0.02 to 3 s⁻¹ using the SER2. These results are compared to three times the corresponding low shear rate viscosity, which agrees well with the zero rate extensional viscosity prior to the onset of extensional thickening at different extension rates. In addition to extensional viscosity, the SER2 can also be used for solids tensile testing, tear testing, peel testing, friction testing, as well as high-rate fracture testing.

SER2 compared to shear viscosity data

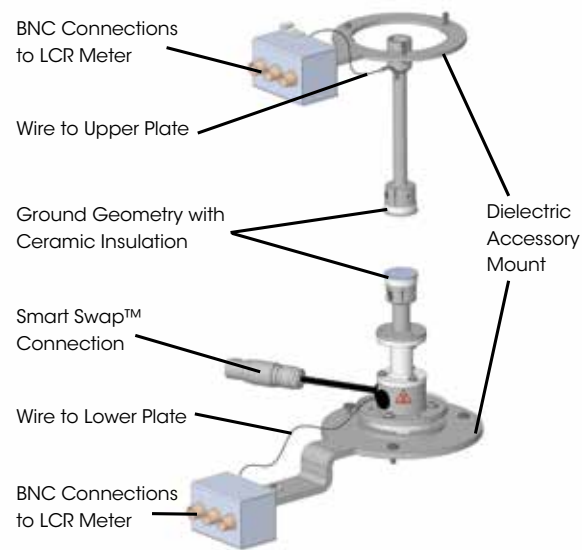


dielectric measurement

ACCESSORY

DIELECTRIC ACCESSORY

The Dielectric Accessory, available for all DHR models, extends material characterization capabilities by providing an additional technique similar to dynamic mechanical. In dielectric analysis, an oscillation electrical field (AC Field) is used as opposed to mechanical force (stress) and the oscillating strain is a stored charge (Q) in the sample. The technique measures the degree to which the sample is storing a charge (capacitance) or passing a charge (conductance) through its bulk. The DHR provides a flexible platform for easy test setup and calibration, and data accuracy through standard features such as the Environmental Test Chamber, axial force control, and gap temperature compensation routines. Dielectric analysis is a very powerful technique for characterizing polar materials such as PVC, PVDF, PMMA, and PVA, for phase separating systems, and for monitoring curing kinetics of materials such as epoxy and urethane systems. Dielectric analysis extends the measurable frequency range over traditional dynamic mechanical analysis which is typically limited to 100 Hz.

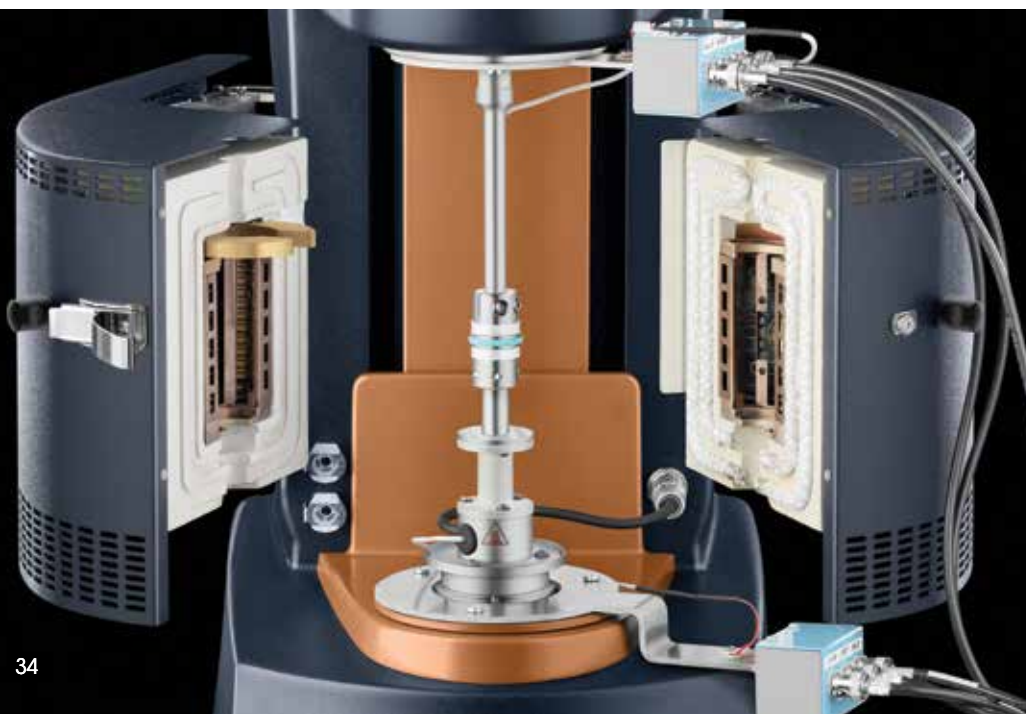


Technology

The Dielectric accessory consists of a special set of 25 mm parallel plates that are fitted with wiring and hardware for interfacing with a dielectric LCR meter (Agilent Model E4980A LCR) that imposes a signal at a certain voltage and frequency/ies. The voltage range available is between 0.005 to 20 V with a frequency range of 20 Hz to 2 MHz. The Environmental Test Chamber (see page 24) provides temperature control at a range of -160 °C to 350 °C. The accessory allows for the simultaneous collection of rheological and dielectric information, or dielectric measurements can be run independently.

Features and Benefits

- Smart Swap™ technology
- 25 mm diameter plates with ceramic insulation
- Disposable plates for curing system
- Stand-alone dielectric measurements
- Combined rheological and dielectric measurements
- Fully programmable from TRIOS Software
- Time-Temperature Superposition
- Master curve generation
- Wide dielectric frequency range: 20 Hz to 2 MHz
- Easy installation and removal
- Compatible with ETC over a temperature range of -160 °C to 350 °C
- USB Interface



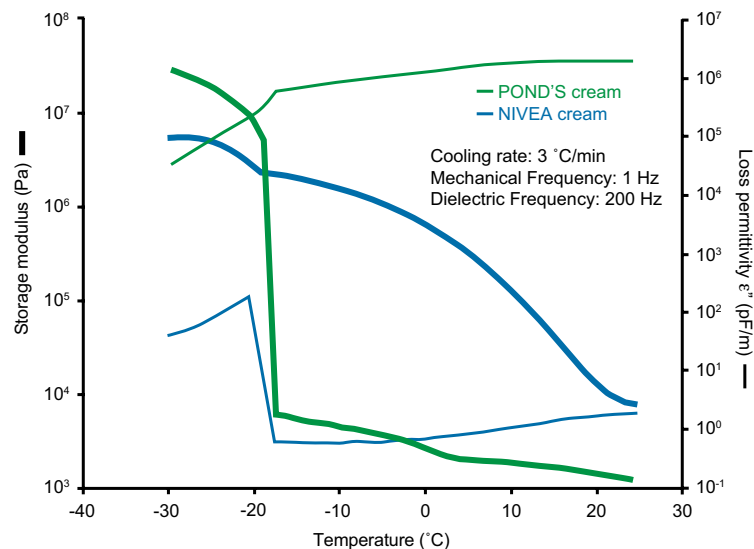
Phase Separation in Cosmetic Creams

Temperature stability of materials such as food and cosmetics is very important for product performance in storage and transportation. Rheological testing is widely used for stability evaluations; however, the ability to simultaneously measure dielectric properties can provide more valuable insight for complex formulations. An example is shown in the figure to the right for two water-based cosmetic creams tested by cooling from 25 °C down to -30 °C. In comparing only storage modulus, G' , data of the two materials, the POND'S® cream shows little increase followed by a three decade jump at -18 °C, but NIVEA cream exhibits a more continuous change in the modulus over the entire temperature range. One may conclude from the mechanical response alone that the large jump in G' of POND'S at -18 °C is associated with instability. However, having the simultaneous measurement of the loss permittivity, ϵ'' , provides information relating to the change in ion mobility; primarily of the water phase in these samples. In the ϵ'' the NIVEA shows a two decade jump compared to very little change in the ϵ'' of the POND'S. The large increase in ϵ'' is due to increased ion mobility in the material as the water separates. In the final analysis, phase separation occurs in the NIVEA, not the POND'S. During the cooling process, as phase separation gradually occurs, the water phase grows changing the morphology. As the morphology gradually changes, so too does the G' . The large change in G' of the POND'S is the result of a transition of a more stable and uniform morphology.

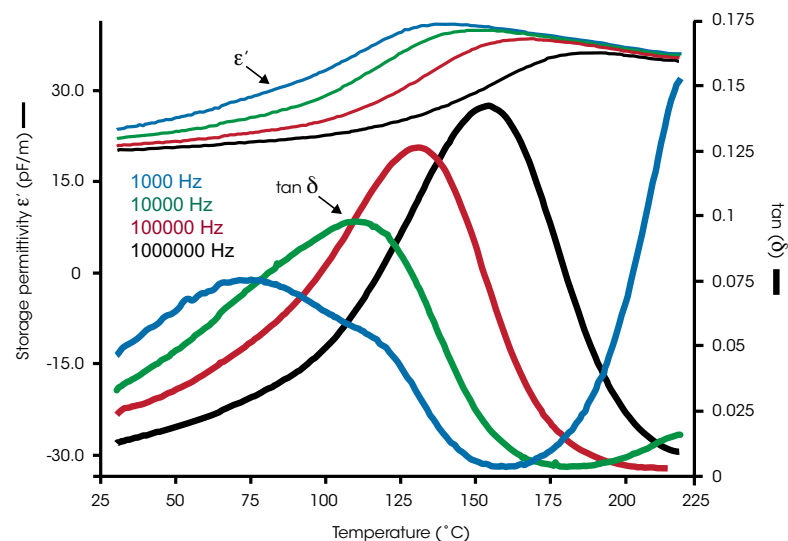
Dielectric Temperature Ramp at Multiple Frequencies

The figure to the right shows a temperature ramp on a Poly (methyl methacrylate), PMMA, sample at four different dielectric frequencies ranging from 1,000 Hz to 1,000,000 Hz. It can be seen here that the magnitude of ϵ' decreases with increasing frequency through the transition region and the peak of the transition in $\tan \delta$ moves to higher temperatures with increasing frequency.

Simultaneous Dielectric and Rheology of Hand Creams on Freezing



Dielectric Temperature Ramp on PMMA

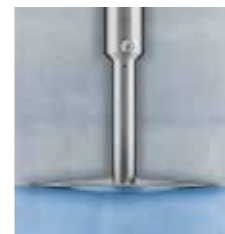


interfacial rheology

ACCESSORIES

Interfacial Accessories

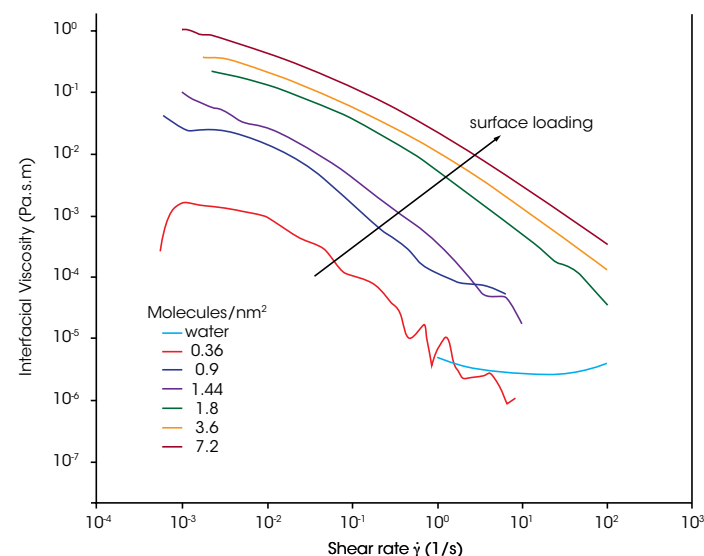
Rheometers are typically used for measuring bulk or three-dimensional properties of materials. In many materials, such as pharmaceuticals, foods, personal care products and coatings, there is a two-dimensional liquid/liquid or gas/liquid interface with distinct rheological properties. Only TA Instruments offers three separate devices for the most flexibility and widest range of quantitative measurements for the study of interfacial rheology. The options include a patented Double Wall Ring (DWR) system for quantitative viscosity and viscoelastic information over the widest measurement ranges, a Double Wall Du Noüy Ring (DDR) for samples available in limited volumes, and a traditional Bicone for interfacial viscosity measurements.



Bicone



Double Wall Du Noüy Ring (DDR)



Application

In this series of tests, the surfactant SPAN65 was spread evenly at the water-air interface using a solution of SPAN in chloroform. After the evaporation of the chloroform, the SPAN65 film deposited on the water was measured using the Double Wall Ring Interfacial accessory. Different loadings of surfactant were tested from 0 (just water, no surfactant layer) to 7.2 molecules per nm². Continuous shear experiments were conducted and the interfacial viscosity was measured as a function of shear rate and interfacial concentration. As expected, the surfactant layer shows significant shear thinning. At high rates, the sub-phase contributions dominate for the loadings less than 1.8 molecules/nm². Sub-phase correction becomes important below an interfacial viscosity of 10⁻⁵ Pa.s.m and the well-defined geometry of the DWR makes these quantitative sub-phase corrections possible. At higher interfacial viscosities, sub-phase contributions are negligible and the correction is unnecessary.



Technology

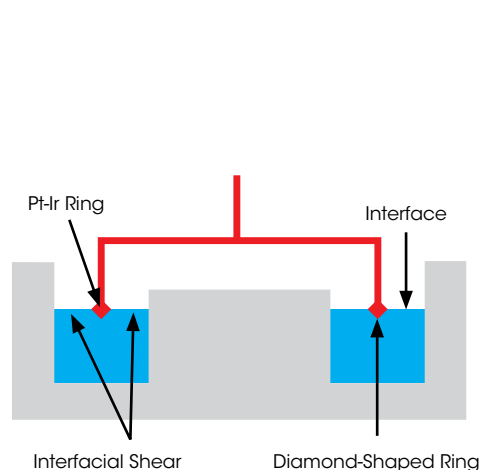
In all TA Instruments interfacial rheology systems, the sample is contained in a Delrin® trough complete with fluid level monitoring windows and injection ports. The measuring rings of the Double Wall Ring (DWR) and Double Wall Du Noüy Ring (DDR) geometries are made of platinum-iridium. These materials are selected for their inert chemistry and ease of cleaning. TA Instruments is the only supplier offering patented double wall geometry configurations that provide interfacial shear planes on both sides of the geometry surface for higher sensitivity to the monolayer viscoelastic response.

Only the DWR is capable of truly quantitative viscoelastic parameters because the interface is “pinned” to the diamond-shaped cross-section of the geometry ring. This patented ultra-low inertia ring⁽¹⁾ has a diameter of 60 mm and was designed for ease-of-use and maximum sensitivity. Surface viscosity measurements can be conducted on surface viscosities as low as 10^{-5} Pa.s.m without complicated sub-phase corrections. And, oscillation measurements are possible over the widest frequency range of any interfacial system.

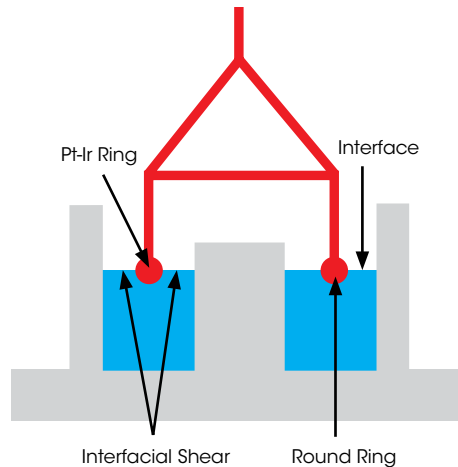
The du Noüy ring geometry of the DDR is an industry standard device used for surface tension measurements. The round cross-section allows for meniscus formation between the interface and geometry, creating a slight error in the absolute data. With a much smaller diameter of 20 mm, this system is ideal for testing interfacial properties of samples that are available in very limited quantities, such as biological or pharmaceutical materials.

The Bicone is a double conical stainless steel geometry with a sharp edge that reproducibly pins the interface. Because of the large drag created by the surface of the cone submerged within the sub-phase, large corrections are required to obtain quantitative parameters. The geometry's large moment of inertia limits measurement capability to interfacial viscosity in steady shear mode, precluding valuable measurements of quiescent structure and elasticity.

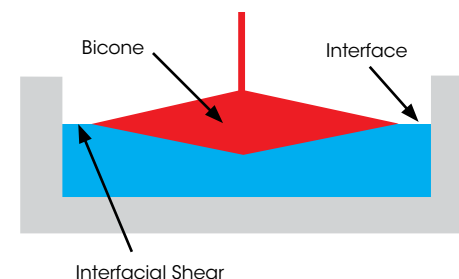
(1) Patent # 7,926,326



Double Wall Ring (DWR) Cross-Section



Double Wall Du Noüy Ring (DDR) Cross-Section



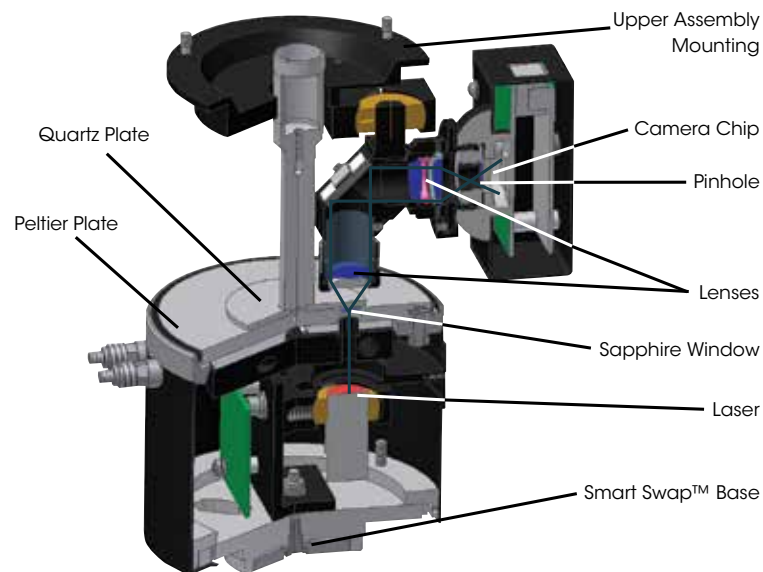
Bicone Cross-Section

small angle light scattering

ACCESSORY

Small Angle Light Scattering (SALS)

The Small Angle Light Scattering (SALS) System is an option for simultaneously obtaining rheological and structural information, such as particle size, shape, orientation and spatial distribution. It is available for the DHR-3 and DHR-2 Rheometers. The option incorporates TA Instruments' Smart Swap™ technology, bringing a new level of speed and simplicity for making simultaneous rheology and SALS measurements. The system can be installed, aligned, and ready for measurements in as little as five minutes. It features patented Peltier Plate temperature control⁽¹⁾ and the scattering angle (θ) range over which measurements can be made is $\sim 6^\circ$ to 26.8° . The scattering vector range (q) is $1.38 \mu\text{m}^{-1}$ to $6.11 \mu\text{m}^{-1}$ and the length scale range is about $1.0 \mu\text{m}$ to $\sim 4.6 \mu\text{m}$.



Technology

The SALS accessory consists of upper and lower assemblies and quartz plate geometry. The lower assembly includes an integrated Class 2 laser (with 0.95 mW diode and wavelength $\lambda = 635 \text{ nm}$) situated below a patented⁽¹⁾ Peltier Plate with a 5 mm diameter quartz window. The Peltier Plate surface is stainless steel with a temperature range of 5°C to 95°C . The upper assembly consists of a set of lenses and a camera. The scattered light is focused through a lens pair mounted within a height-adjustable cap to focus at varying sample depths. The light is then focused through a second lens and sent through an adjustable polarizer for both polarized and depolarized measurements. Finally, the scattering is collected through a pinhole and recorded by the camera. The upper geometry is a 50 mm diameter, 2 mm thick optical quartz disk. To comply with the single-point correction for the parallel plates, the laser is set at 0.76 times the plate radius which is 19 mm from the axis of rotation of the plate. This arrangement keeps the SALS system compact, while allowing for quick and reproducible positioning and focusing. A set of neutral density filters is available as an option to reduce laser intensity.

⁽¹⁾ Patent # 7,500,385

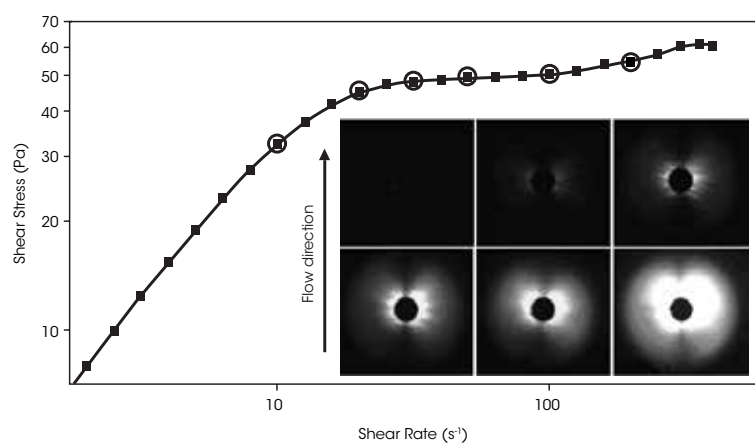




Features and Benefits

- Smart Swap™ technology
- q vector range $\sim 1.38 \mu\text{m}^{-1}$ to $6.11 \mu\text{m}^{-1}$
- Objects length scale range $\sim 1 \mu\text{m}$ to $4.6 \mu\text{m}$
- Scattering angle $\sim 6^\circ$ to 26.8°
- Wavelength 635 nm
- Compact upper assembly requiring minimal adjustment
- Smart Swap™ lower assembly with factory aligned laser
- Class 2 laser - No safety issues
- Adjustable laser intensity with optional neutral density filters
- Variable depth focus to adjust for different geometry gaps
- Adjustable polarizer for scattering in parallel or perpendicular to the incident light
- Image focused directly onto camera chip – Does not require screen or darkened room
- Quantitative measurements possible by calibration with monodisperse Polystyrene beads.
- Optional Analysis Software
- Patented Peltier Plate temperature control

Scattering Images Captured with Rheometry Data



Shear-Induced Phase Separation of Micellar Solutions

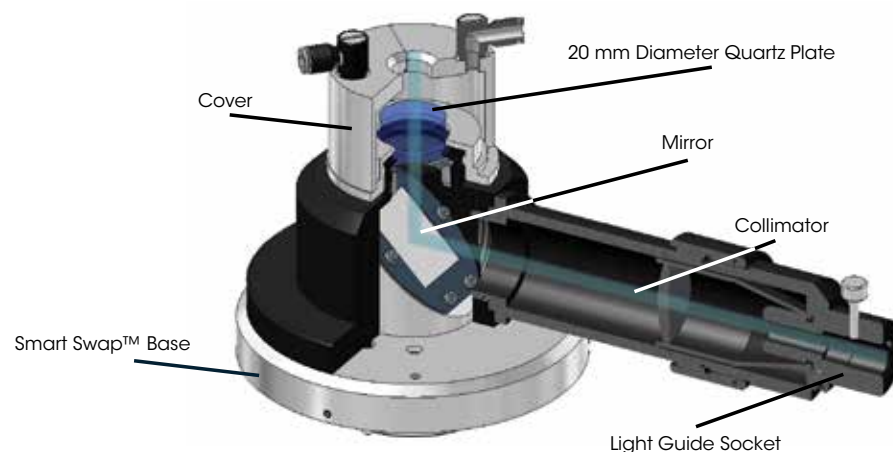
Self-assembled surfactant micelles show a variety of shear-induced microstructural transformations that are important for material formulation and function for a wide array of applications. Simultaneous measurements of rheology and surfactant microstructure, using SALS under shear, provides a valuable tool in examining shear-induced transitions in such fluids. The data in the figure show scattering images captured synchronously with rheometry data on a surfactant system. At low shear rates below the stress plateau, no measurable scattering is obtained from the sample, suggesting no large-scale structuring of the fluid. However, in the stress plateau, a strong anisotropic scattering pattern develops with increasing shear rate. This "butterfly" pattern results from phase separation, where the interface between the two phases generates a strong scattering contrast.

UV curing

ACCESSORIES

UV Curing Accessories

UV-curable materials are widely used for coatings, adhesives, and inks. When these materials are exposed to UV radiation, a fast cross-linking reaction occurs, typically within less than a second to a few minutes. Two Smart Swap™ accessories for rheological characterization of these materials are available for the DHR-3 and DHR-2 rheometers. One accessory uses a light guide and reflecting mirror assembly to transfer UV radiation from a high-pressure mercury light source. The second accessory uses self-contained light emitting diodes (LED) arrays to deliver light to the sample. Accessories include 20 mm quartz plate, UV light shield, and nitrogen purge cover. Optional temperature control to a maximum of 150 °C is available using the Electrically Heated Plates (EHP) option. Disposable plates are available for hard UV coatings, which cannot be removed from the plates once cured.



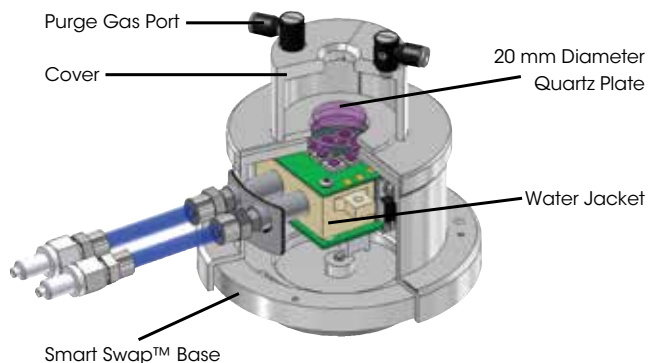
UV Light Guide Accessory Technology

The UV Light Guide accessory includes a lower Smart Swap™ assembly with quartz plate, light source mount, collimator, 5 mm diameter light guide, and UV mercury lamp source (Exfo Omnicure S2000). It provides a broad wavelength spectrum from 250 nm to 600 nm, with a primary peak at 365 nm. The maximum output intensity is greater than 300 mW/cm². External filter holder and filters are available for the light source.

Features and Benefits

- Smart Swap™ technology
- Convenient compact
- Collimated light and mirror assembly insure uniform irradiance across plate diameter
- Maximum intensity at plate 300 mW/cm²
- Broad range spectrum with main peak at 365 nm
- One system with specific wavelengths accessible through filtering options
- Cover with nitrogen purge ports
- Optional disposable acrylic plates
- Optional temperature control to 150 °C
- Software programmable trigger time and intensity





UV LED Accessory Technology

The UV LED accessories use arrays of light emitting diodes that provide single peak wavelength light sources. The LED array is mounted on a PCB and is fixed to a water jacket that cools the LED's during use. Like the UV light guide system, LED's are pre-aligned to insure uniform irradiance across the surface of the plate. There are two LED accessories available at wavelengths of 365 nm and 455 nm. The maximum output UV intensity is 150 mW/cm² and 350 mW/cm², respectively. They are fully integrated with the rheometer through a Smart Swap™ option. Trigger time and intensity are conveniently programmed through the software.

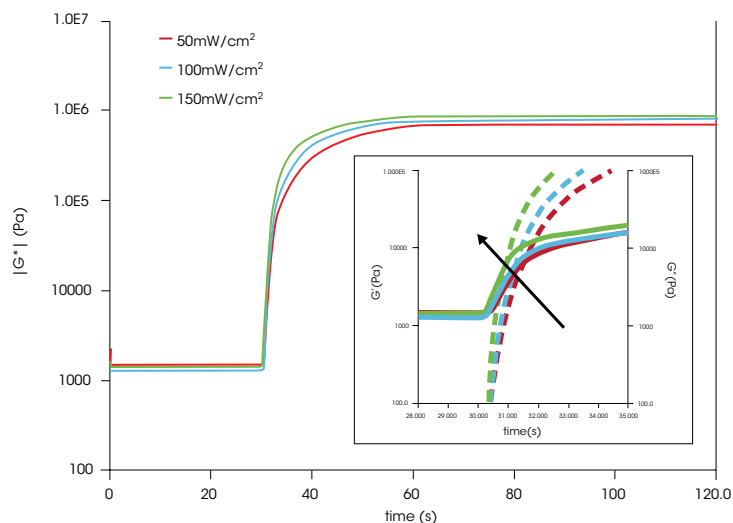
Features and Benefits

- Smart Swap™ technology
- New technology replaces mercury bulb systems
- 365 nm wavelength with peak intensity of 150 mW/cm²
- 455 nm wavelength with peak intensity of 350 mW/cm²
- No intensity degradation over time
- Even intensity across plate diameter, LED positioned directly below plate
- Compact and fully integrated design including power, intensity settings and trigger
- Cover with Nitrogen purge ports
- Optional disposable Acrylic plates
- Optional temperature control to 150 °C

UV Accessory Application

These accessories allow the study of UV curing reactions by monitoring the elastic (G') and viscous (G'') moduli. The example on the left shows results of a pressure sensitive adhesive, (PSA), characterized with the UV Light Guide Accessory. The PSA was held at an isothermal temperature of 25 °C and the curing profile was measured at radiation intensities from 50 mW/cm² to 150 mW/cm². The sample is measured for 30 seconds before the light is turned on. The data show faster reaction kinetics with increasing intensity, as evidenced by the shorter time for crossover of G' and G'' . Similar results can be obtained with controlled temperature, where the reaction is seen to occur more quickly at higher temperatures. The curing reaction happens in less than two seconds. The fast data acquisition of the Discovery Hybrid Rheometers (up to 50 pts/sec) enables clear identification of the liquid to solid transition. Note that changing the intensity and temperature by small amounts shifts the crossover point by a fraction of a second. This information is important for understanding adhesive control parameters for high-speed UV curing processes, as well as for understanding differences in initiators when formulating materials.

Effect of UV Curing Intensity

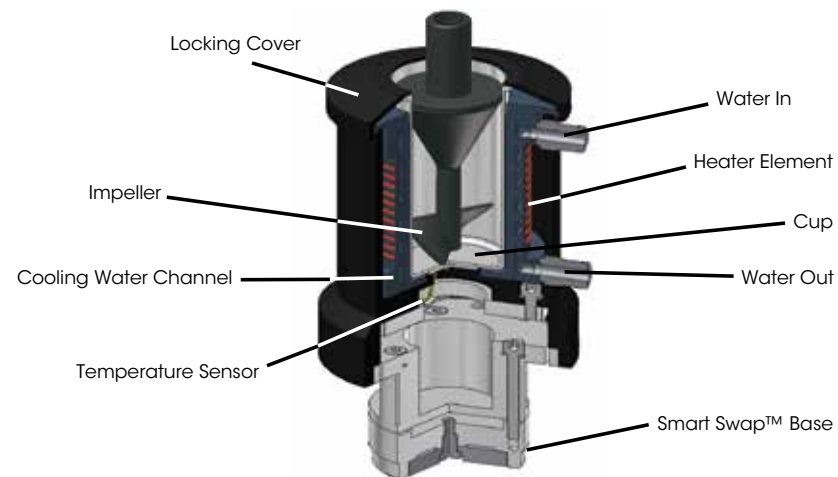


starch pasting cell

ACCESSORY

Starch Pasting Cell (SPC)

The Starch Pasting Cell (SPC) provides a more accurate and powerful tool to characterize the gelatinization of raw and modified starch products, as well as the properties of the starch gels. It can also be used for characterizing many other highly unstable materials. It uses an innovative impeller design for mixing, reduction of water loss, and control of sedimentation during testing. The actual sample temperature is measured and controlled in a temperature chamber with heating/cooling rates up to 30 °C/min.



Technology

The SPC consists of the cell jacket, an impeller, and aluminum cup with locking cover. The cell jacket houses a heating coil and liquid cooling channel, which surrounds the Aluminum cup for fast heating and cooling. A Platinum Resistance Thermometer (PRT) is located in intimate contact with the bottom of the cup for precise and accurate sample temperature control. The impeller is designed with blades at the bottom for sample mixing. Solvent loss is minimized via a conical ring at the top of the rotor, which acts to condense water (or other solvents) that vaporizes during heating, and return it to the bulk sample.

Features and Benefits

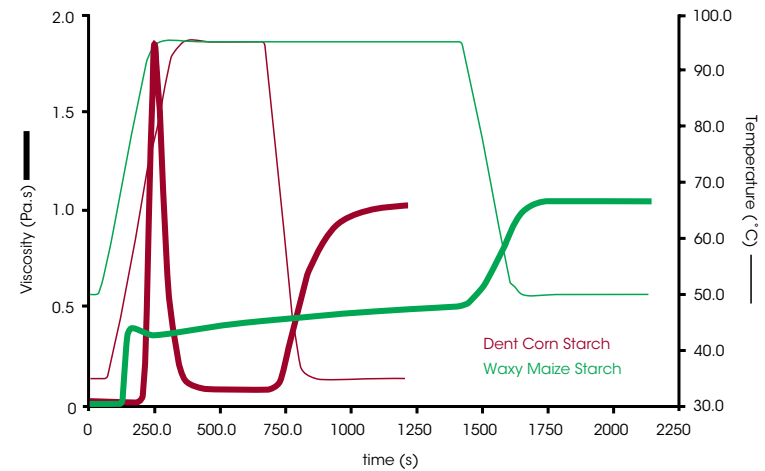
- Smart Swap™ technology
- Heating/Cooling rates up to 30 °C/min
- Higher accuracy for greater reproducibility
- Robust Cup and Impeller
- Impeller keeps unstable particles suspended in liquid phase during measurements
- Impeller design minimizes loss of water or other solvents
- Sample temperature measured directly
- All rheometer test modes available for advanced measurements on gelled starches and other materials
- Optional conical rotor for traditional rheological measurements



Gelatinization of Starch Products

Starch is not only a food product; functionally modified starches are widely used in the industry including adhesives, paper, coatings, wood, packaging, pharmaceutical, and many others. When starch is heated above a critical temperature, the starch granules undergo an irreversible process, known as gelatinization. The properties of the starch gels depend on the origin of the raw starch (crop, potatoes, etc.), the environmental conditions (seasons) or the modification. The viscosity curve, referred to as pasting curve, produced by heating and cooling starches generally has a similar characteristic shape. The figure to the right shows two scans each of both a Dent Corn and Waxy Maize starch. The benefit of the DHR starch cell design can easily be observed in the unprecedented reproducibility of the pasting curves for these two starch products.

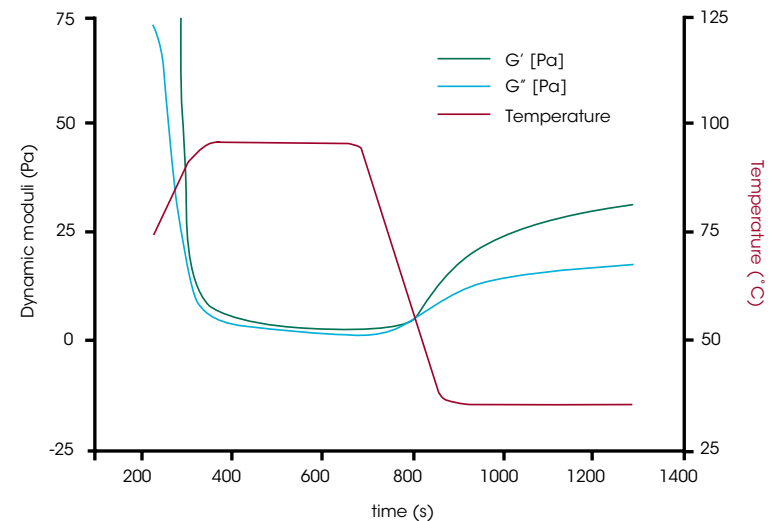
Two Scans each of Dent Corn and Waxy Maize Starch



Advanced Starch Rheology

In addition to measuring the characteristic pasting curve of starch products, the starch cell brings new testing capabilities for measuring properties of the starch gels. The figure to the right shows additional data obtained on Dent Corn starch using an oscillation test to monitor the gelation process of the starch under negligible shearing. In this test, the sample is sheared while ramping temperature to keep starch particles suspended. At 75 °C, when the viscosity is high enough to inhibit particle settling, the steady shear was stopped and testing was continued at a small oscillating stress. The figure shows storage modulus, G' , and the loss modulus, G'' , which provide extremely sensitive information about the structural characteristics of the starch gelation and final gel. This enables the development of valuable structure-property relationships. The ability to make these sensitive measurements is not possible on traditional starch characterization instrumentation.

Dent Corn Starch Gelatinization



tribo-rheometry

ACCESSORY

Tribology

Tribology is defined as the study of interacting surfaces undergoing relative motion. The new Tribo-Rheometry Accessory, available for all DHR models, enables the capability to make coefficient of friction measurements between two solid surfaces under dry or lubricated conditions. The unique self-aligning design ensures uniform solid-solid contact and axial force distribution under all conditions. A modular set of standard and novel geometries offers a choice of different contact profiles and direct simulation of end-use conditions. Accurate and precise control of axial force, rotational speed, and temperature inherent to TA Instrument rheometers provides for the best and widest range of friction measurements.

Features and Benefits

- Compatible with Stepped Disposable Peltier Plate and Environmental Test chamber
- Characterize materials wear and coefficient of friction under dry or lubricated conditions
- Unique self-aligning design for best solid-solid contact
- Modular set of geometries offers choice of contact profiles
- Interchangeable parts for customized substrates
- Automatic software calculation of relevant friction parameters
- Easy installation and removal

The advanced TRIOS software offers easy setup and control of tribo-rheometry tests and contains a complete set of variables required for data analysis including the coefficient of friction (μ), load force (F_L), friction force (F_f) and Gumbel number (Gu). These may be used to construct Stribeck curves, static friction measurements, or explore specific combinations of temperature, contact force, and motion.





Ring on Plate



Ball on three plates



Ball on Three Balls

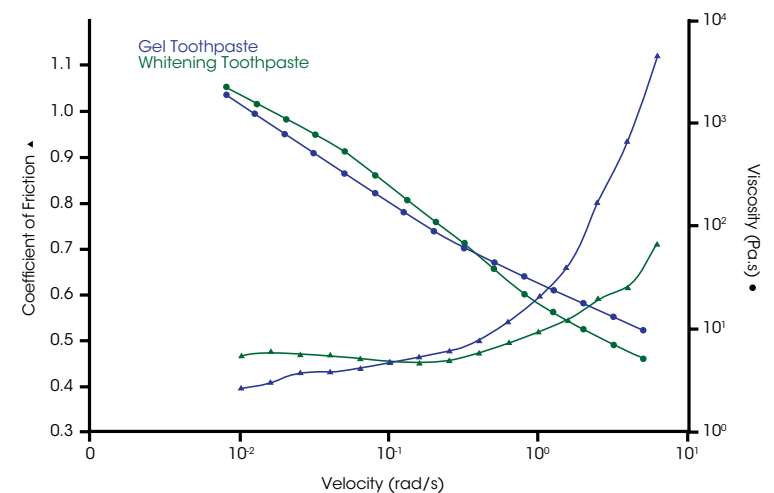


Three Balls on Plate

The Tribo-Rheometry Accessory is compatible with both the Stepped Disposable Peltier Plate and the Environmental Test Chamber (ETC) for accurate and stable temperature control for all test geometries. The choice of four standard geometries – Ring on Plate, Ball on Three Plates, Three Balls on Plate, and Ball on Three Balls – meets the diverse requirements of tribology applications and offers a variety of contact profiles. The ring on plate geometry may also be configured as a partitioned ring, which permits the replenishment of lubricant between the two solid surfaces. The accessory's versatile configurations and easily interchangeable substrates are ideal for studying the effect of friction and long-term wear on materials ranging from automotive components and greases, lubrication in prosthetic devices, and the performance of personal care creams and lotions.



The accompanying figure shows the coefficient of friction profiles of two commercially available toothpastes tested between textured PMMA plates (acting as tooth substitutes) using the ring on plate configuration. The whitening toothpaste, with abrasive particles, has higher friction at low speeds, but the gel toothpaste's friction profile shows a rapid increase at higher speeds. This behavior can be explained by comparing the flow curves of the two toothpastes – although both materials are shear thinning, the viscosity of whitening toothpaste decreases more rapidly than the gel toothpaste. This results in increased hydrodynamic drag and greater friction at higher rotation speeds.



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