

Thermal Analysis Excellence



**DMA/SDTA 1+**

**STAR<sup>®</sup> System**

Innovative Technology

Versatile Modularity

Swiss Quality



## Dynamic Mechanical Analysis Sets New Standards

**METTLER TOLEDO**

# Precise Measurement Technology for Highest Performance

**Dynamic mechanical analysis (DMA) is used to measure the mechanical and viscoelastic properties of a material as a function of temperature, time and frequency while it is subjected to an oscillating stress.**

## Features and benefits of the METTLER TOLEDO DMA/SDTA 1+:

- **Unique measurement of both displacement and force**  
results in a very accurate determination of moduli
- **Wide force range from 1 mN to 40 N**  
allows you to measure very soft and very stiff samples
- **Broad frequency range from 0.001 to 1000 Hz**  
means you can perform measurements under real conditions  
or more rapidly at higher frequencies
- **DMA Experiment Wizard**  
sets up the perfect experiment for the best results
- **Patented SDTA technique**  
enables you to calibrate sample temperature and accurately  
measure thermal effects
- **Extremely wide stiffness range**  
means you can measure a sample from the glassy to the  
liquid state in one single run
- **Ergonomic design and touch-screen control**  
ensures faster setup and optimization of experiments





Due to its revolutionary technology, the DMA/SDTA 1+ provides unattained performance and offers time-saving external sample clamping.



# Unsurpassed Measurement Results Thanks to Innovative Solutions

The measurement principle of the DMA/SDTA 1+ ensures high accuracy because it measures both displacement and force. This yields very accurate modulus values. It was designed to measure material behavior at high frequencies to match real-life conditions. This is enabled by its very stiff stand resulting in an intrinsic resonance frequency of about 1500 Hz – well above the measurement frequencies used (up to 1000 Hz).

## Force measurement using a piezoelectric crystal



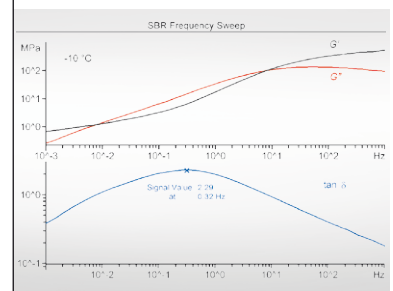
Force is measured directly by means of a piezoelectric crystal and is not set using a force-current graph as in conventional DMA instruments. The force measured is that which is actually applied to the sample. Compensation for frictional losses, membrane force and inertia is no longer necessary.

## Even more accurate displacement measurement



A special temperature-resistant LVDT allows measurements to be performed over a large measurement range with nanometer resolution. The LVDT is located close to the sample so that only the deformation of the sample is measured. This eliminates any effect due to deformation of the stand and also improves the accuracy of measurement of the phase shift. The reproducibility of the displacement measurement is improved by measuring the temperature of the LVDT sensor and correcting for the deviation.

## Wide frequency range from 0.001 to 1000 Hz



The frequency range has been extended to the kHz region for the first time ever in a DMA instrument. In the shear mode, six decades are available. The region above 1 Hz is particularly interesting because it means that measuring times can be kept to a minimum.



The stiffness range is given by the force and displacement ranges. With the DMA/SDTA 1+, more than six decades are available. This means that it is now possible to measure samples from the glassy to the viscoelastic state in one experiment without having to change the sample geometry or the deformation mode.

# Simple Operation

## Clever Sample Holder Design

**Sample preparation and sample clamping are critical steps for accurate DMA measurements. This is particularly the case for shear and tension samples which are very often small and thin. The sample holder system therefore offers important advantages.**

### Simplified sample holder system



The METTLER TOLEDO sample holder system is a completely new design that helps save valuable instrument operating time. The samples are prepared and mounted externally in the sample holder. This can then be quickly installed in the instrument. The concept also allows you to change from one deformation mode to another without performing an adjustment. For example, you can prepare a bending experiment while a measurement in the tension mode is in progress.

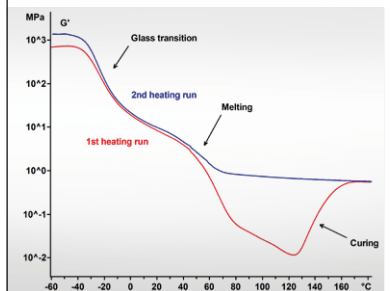
### Touchscreen terminal – for displacement and force calibration



The touch-sensitive color terminal of the DMA/SDTA 1+ presents clear and precise information and is easily seen from a distance.

- All force and length calibration routines are controlled via the terminal. This ensures that these operations are performed reliably and easily.
- The 4-axis alignment adjustment is also carried out via the terminal; the resulting values are transferred to the STAR<sup>®</sup> software. This eliminates any possible transfer errors.

### Automatic switch mode



Force measurement also allows the instrument to be operated in a way not possible with conventional DMAs: The instrument can be operated under either force or displacement control and an intelligent automatic switch mode is also possible.



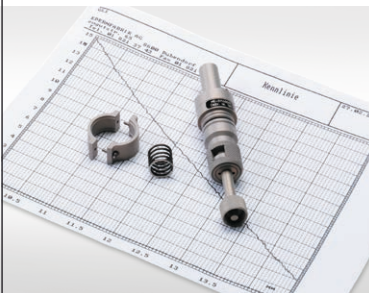


The thermoelement can be placed close to the sample and allows thermal events to be measured.

# Calibration and Adjustment for Maximum Accuracy

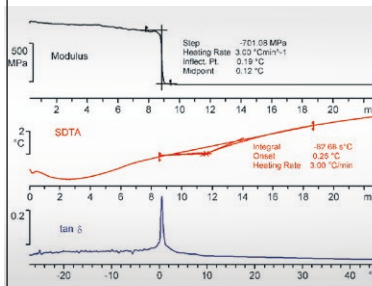
**Calibration determines how well measurement equipment performs. The standard DMA/SDTA 1+ calibration and adjustment procedures allow the user to achieve compliance and avoid potential costs resulting from inaccurate measurements. A Standard Operational Procedure is available and describes the calibration of the individual quantities (force, length, temperature).**

## Adjustment using reference standards



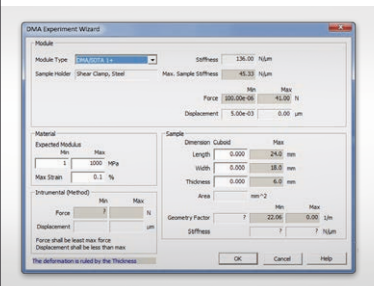
- The adjustment of force, displacement and temperature is based on defined reference standards.
- Force is adjusted using a certified spring and displacement using gauge blocks.
- Temperature measurement close to the sample allows calibration using the melting points of pure substances.

## Sample temperature measurement and calibration/adjustment



Temperature calibration and adjustment is particularly important. An additional temperature sensor is located close to the sample. The sensor also allows thermal effects to be simultaneously measured by SDTA (Single DTA).

## DMA Experiment Wizard



The DMA Experiment Wizard, with its built-in expert knowledge, is designed to help users establish optimal sample dimensions, necessary for achieving accurate results. The software is also able to determine ideal force and displacement amplitudes.





### Matching accessories

For mounting clamp assemblies, sample holders, and for performing calibrations, we supply an accessories box with all the materials needed for calibration (temperature and mechanical) and for installing samples (optional). This ensures that you quickly obtain precise measurement results.

### Small clamping assembly

Deformation mode	Clamps	Dimensions
Tension	Small tension clamp 9 mm	Length: 9 mm
	Small shear clamp	Diameter: $\leq 15.0$ mm Thickness: $\leq 6.5$ mm
Shear	Small shear clamp (for liquids)	
	Small shear clamp (no surface structure)	

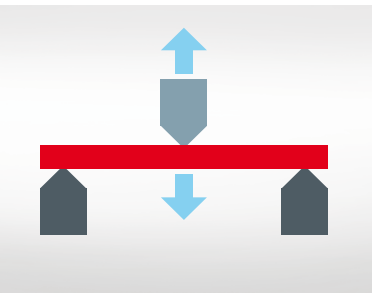
### Large clamping assembly

Deformation mode	Clamps	Dimensions
Bending	Bending clamp for 3-point bending	Free length: 30 to 90 mm, max. length: 100 mm
	Single cantilever bending clamp	Free length: 10 to 40 mm, max. length: 100 mm
	Dual cantilever bending clamp	Free length: 20 to 80 mm, max. length: 100 mm
Tension	Tension clamp 5.5 mm	Length: 5.5 mm
	Tension clamp 10.5 mm	Length: 10.5 mm
	Tension clamp 19.5 mm	Length: 19.5 mm
Compression	Large compression clamp	Diameter: $\leq 19.0$ mm, thickness: $\leq 9.0$ mm

# Sample Holders

## Simple, Ingenious and Timesaving

### 3-point bending

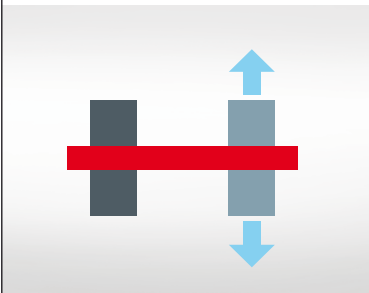


This bending mode is ideal for measuring extremely stiff samples, such as composite materials or thermosets, particularly below the glass transition temperature.



In the 3-point bending mode, the ends of the sample rest on two knife edges and an oscillatory force is applied to the middle of the sample by a moving knife edge.

### Single cantilever bending

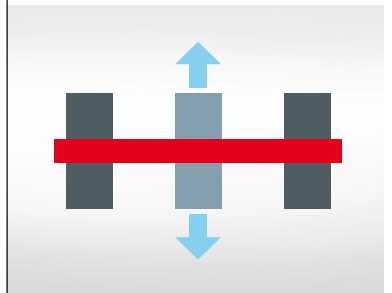


This bending mode is suitable for samples that expand or contract in the longitudinal direction during the measurement. This applies primarily to thermoplastic samples.



The single cantilever mode is very similar to the dual cantilever mode except that only one end of the sample is fixed while the other end is connected.

### Dual cantilever bending

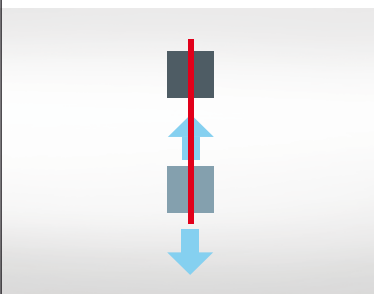


Dual cantilever is the perfect mode for samples that would otherwise bend excessively under strong pretension. These are usually thermoplastics or thermosets.



In the dual cantilever mode, the ends of the sample are fixed and the middle is clamped to the moving part providing the oscillatory force.

### Tension

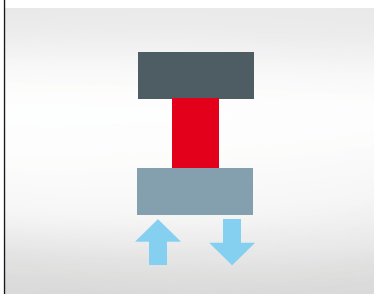


Tension is the mode most suitable for films, fibers and thin bars and rods. The advantage is that sample clamping hardly affects the deformation.



In the tension mode, one end of the sample is fixed and the other is subjected to an oscillatory force. The sample must be prestressed to prevent it from buckling during the oscillatory movement.

### Compression

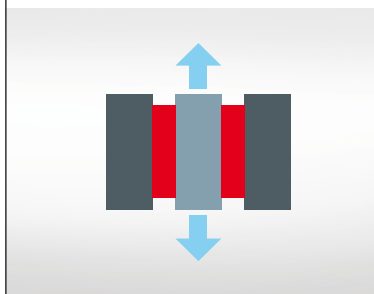


This type of measurement is less suitable for the determination of absolute values of the modulus; valuable relative information can however be obtained when comparing soft materials like elastomers, pastes or foams.



In the compression mode, the sample is clamped between a fixed part and the moving part providing the oscillatory force. The sample is compressed statically and subjected to an alternating load.

### Shear



The great advantage of the shear mode is that everything from viscous to very hard samples can be measured. This mode is therefore ideal for elastomers, thermoplastics, and thermosets.



In the shear mode, two identical samples are clamped symmetrically between two fixed outer parts and a central moving part. The shear clamp guarantees a homogeneous temperature distribution.

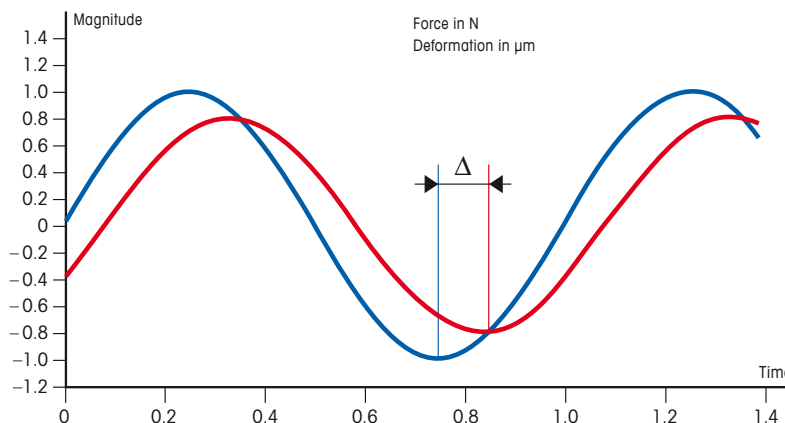


# DMA Theory

## Force, Displacement and Phase Angle

The different moduli can be calculated from the raw data namely the measured force and displacement amplitudes,  $F_0$  and  $L_0$ , and the phase shift  $\delta$ :

- Complex modulus  $M^*$ , elastic modulus  $E^*$  for bending, tension and compression or the shear modulus,  $G^*$ , for shear deformation.
- Storage modulus  $M'$  (is reversible and proportional to the energy stored elastically).
- Loss modulus  $M''$  (irreversible and proportional to the energy transformed into heat).
- Loss factor  $\tan \delta$ . Completely elastic materials exhibit no phase shift  $\delta$  while purely viscous materials exhibit a  $90^\circ$  phase shift. The loss factor of viscoelastic materials is between 0 and infinity ( $\delta = 90^\circ$ ).



Force and displacement at a frequency  $f$  of 1 Hz. The time shift  $\Delta$  results in the phase shift  $\delta: \delta = 2\pi f\Delta$ .

The value of  $\tan \delta$  corresponds to the ratio of  $M''$  to  $M'$ . The moduli are calculated from the measured stiffness according to the following equations:

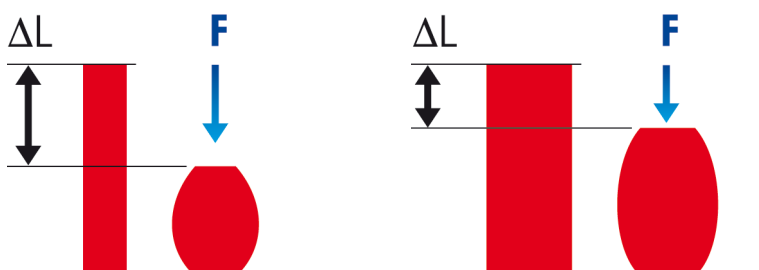
$$|M^*| = S * g = \frac{F_0}{L_0} * g \quad S = \frac{F_0}{L_0}$$

where  $g$  is the geometry factor calculated from the sample dimensions.  $S$  is the stiffness of the sample (the actual measured quantity).

The stiffness of the sample can thus be influenced by changing the sample geometry.

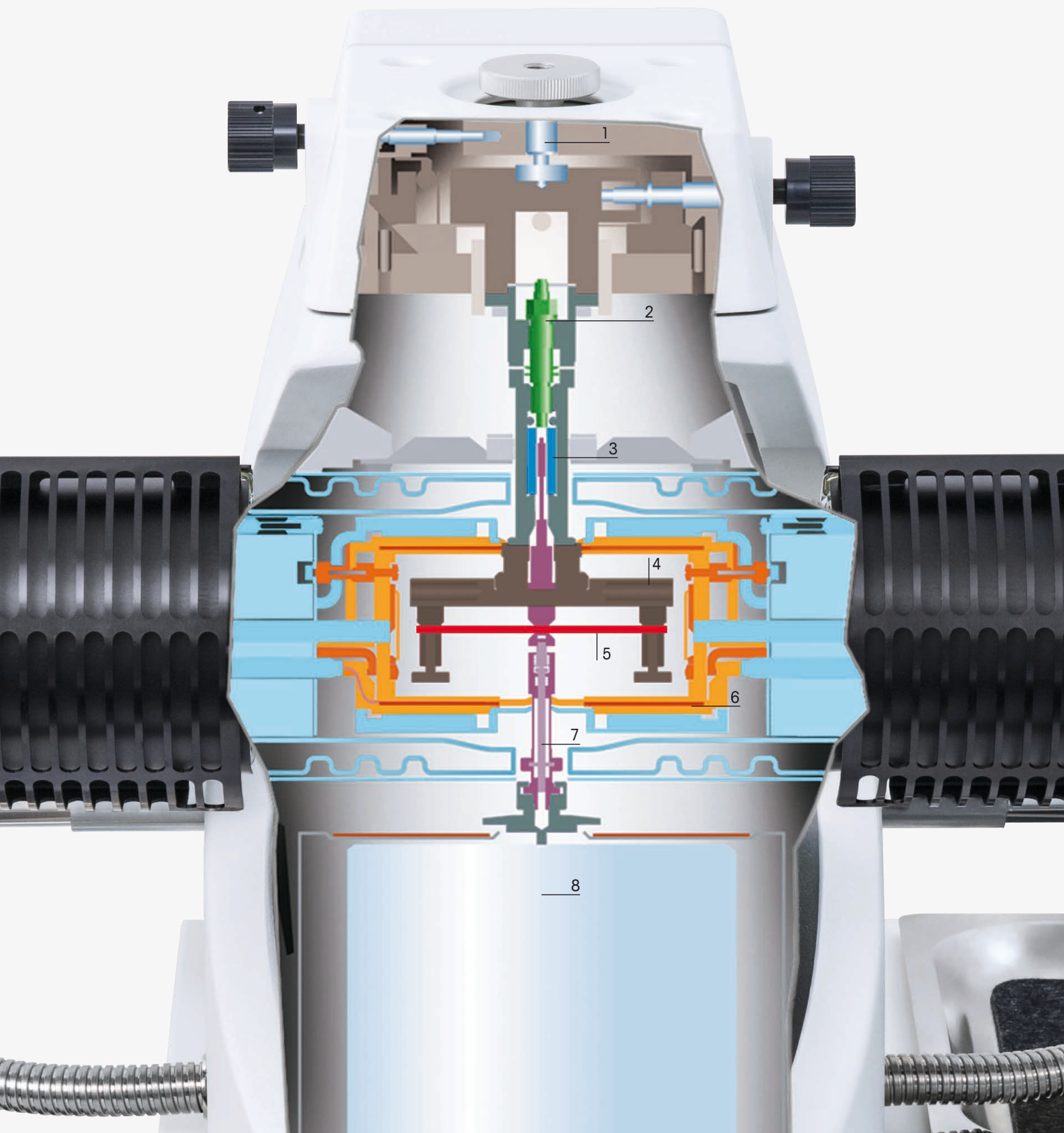
$$M' = |M^*| \cos \delta \quad M'' = |M^*| \sin \delta$$

$$\tan \delta = \frac{M''}{M'}$$



F: Static force  
 $\Delta L$ : Deformation F

A thick sample is stiffer than a thin sample.



- |  |                 |
|--|-----------------|
| 1. 4-axis alignment ( $x/y$ , $\alpha/\beta$ ) | 5. Sample       |
| 2. Force sensor                                | 6. Furnace      |
| 3. LVDT (displacement sensor)                  | 7. Drive shaft  |
| 4. Clamp                                       | 8. Linear motor |

# Characterization of Materials for a Wide Range of Applications

**The DMA/SDTA 1+ is the perfect solution when maximum accuracy is required over a wide range of stiffness or frequency. Thanks to its wide dynamic force and displacement ranges and the large variety of sample sizes and geometry, the DMA/SDTA 1+ can be used to analyze practically all solid materials as well as medium and high viscosity fluids.**

Materials are subjected to a wide range of different stresses in practical daily use. The most important factors are the frequency and intensity of the stress, the temperature, and the environment in which the load or stress is applied.

## **Effects and properties that can be characterized by the DMA/SDTA 1+:**

- Viscoelastic behavior
- Relaxation behavior
- Glass transition
- Mechanical modulus
- Damping behavior
- Softening
- Viscous material flow
- Crystallization and melting
- Gelation
- Phase transitions
- Composition of blends
- Curing and polymerization reactions
- Material defects
- Effects caused by filler materials

### **Application fields**



Stability testing, damage and failure assessment, usability tests, material characterization.

### **Materials tested by DMA**

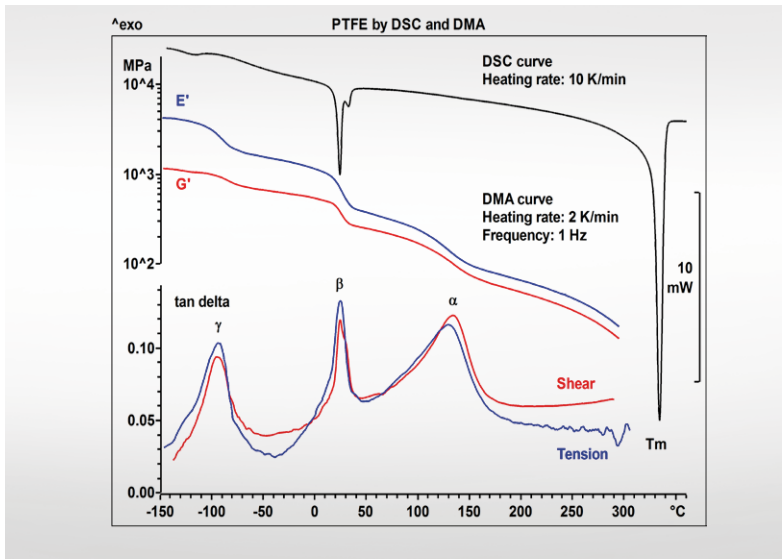


Thermoplastics, thermosets, elastomers, adhesives, metals, composites, paints, films and fibers, construction materials, pharmaceuticals, and foodstuff.



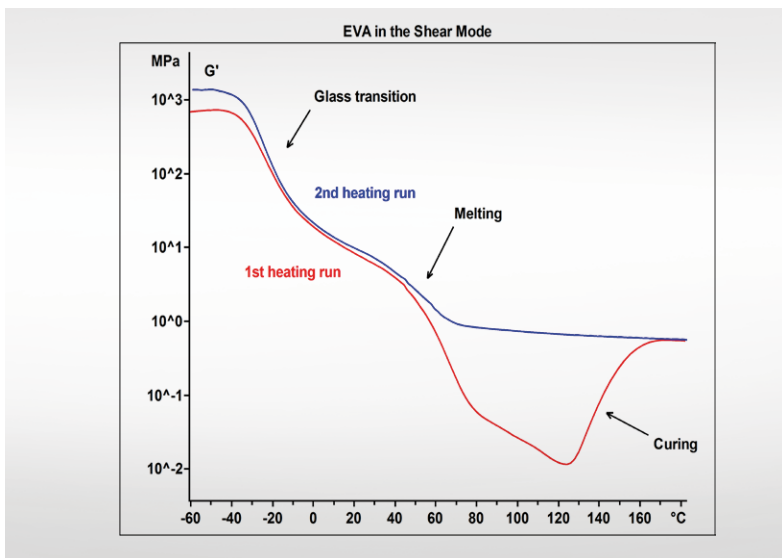


Today's composites are often less expensive compared to traditional materials, and highly sought after in many different industries where high strength to weight ratios are paramount. DMA is ideal for the characterization of composite materials with a wide range of moduli.



### Phase transitions of PTFE by DSC and DMA

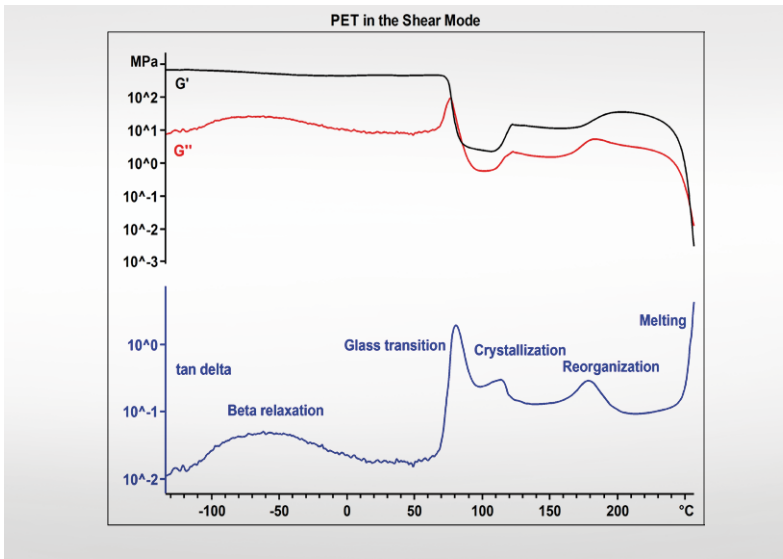
The DSC curve of PTFE shows the glass transition of the mobile amorphous fraction ( $\gamma$ ) at about  $-100\text{ }^{\circ}\text{C}$  followed by two crystal-crystal transitions ( $\beta$ ) at  $30\text{ }^{\circ}\text{C}$ . The crystallites melt at  $330\text{ }^{\circ}\text{C}$ . These transitions can also be measured by DMA. The technique shows in addition the glass transition of the rigid amorphous fraction ( $\alpha$ ) at  $130\text{ }^{\circ}\text{C}$ . Glass transitions are easier to detect by DMA than by DSC. From Poisson's ratio, it follows that  $E'$  is in principle always greater than  $G'$ .



### Vulcanization of ethyl-vinyl acetate (EVA)

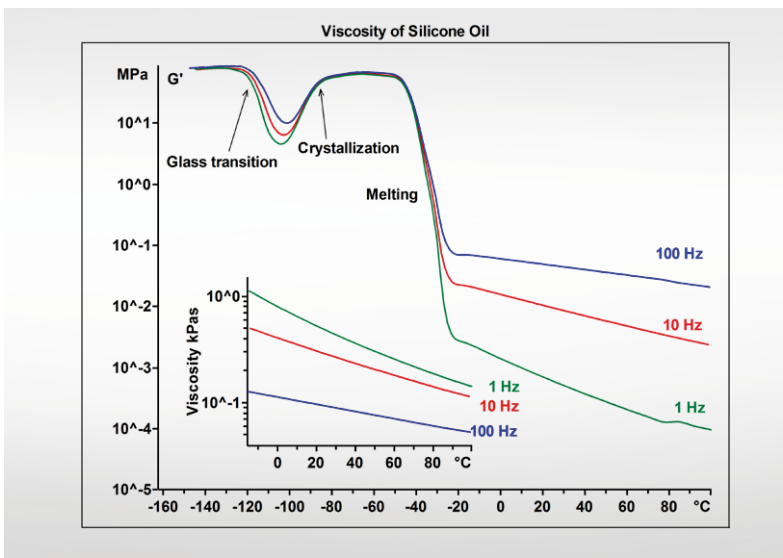
The vulcanization of EVA was investigated in the shear mode. The diagram displays the storage modulus of two consecutive heating runs. The first heating run shows a glass transition at about  $-30\text{ }^{\circ}\text{C}$  followed by melting of the crystalline ethylene segments above about  $50\text{ }^{\circ}\text{C}$ . The increase of the modulus from  $120\text{ }^{\circ}\text{C}$  onward is a result of the vulcanization or curing reaction. The second heating run shows the glass transition and melting of the fully vulcanized EVA.





### Characterization of polyethylene terephthalate (PET)

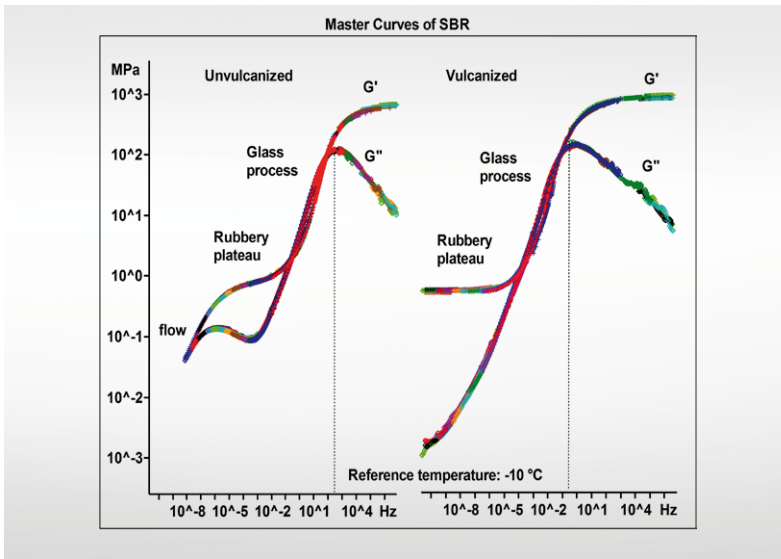
The mechanical behavior of amorphous PET (polyethylene terephthalate) was investigated between  $-130$  and  $260$  °C in the shear mode at a frequency of 1 Hz. The broad peak at  $-60$  °C in the tan delta curve is due to secondary relaxation (beta relaxation). The glass transition occurs at about  $80$  °C. The storage modulus increases at about  $110$  °C as a result of cold crystallization. Reorganization processes then occur followed by melting of the crystallites.



### Viscosity of silicone oil

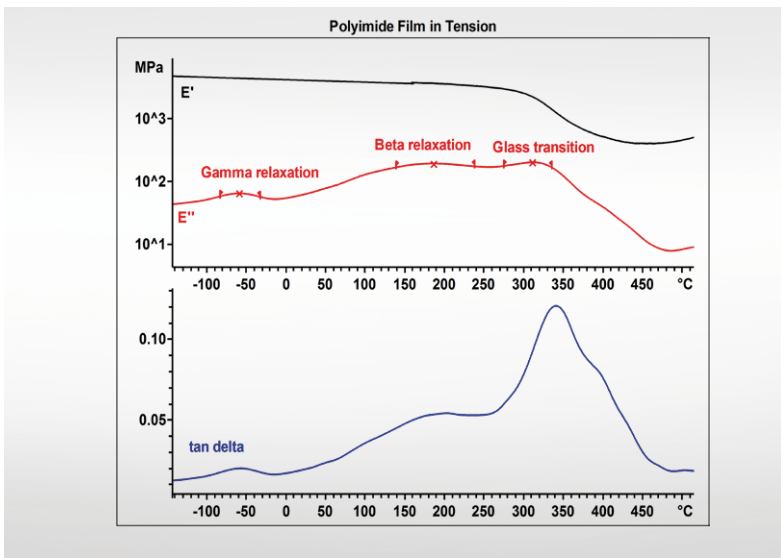
When silicone oil is rapidly cooled, crystallization is suppressed and the oil vitrifies. If a sample is then heated, a glass transition (frequency dependent) is observed at about  $-110$  °C (at 1 Hz) followed by crystallization (frequency independent) at about  $-85$  °C and melting (frequency independent) from about  $-45$  °C onward. The viscosity and its frequency dependence (inset diagram) can then be calculated from the modulus curves in the liquid state (i.e. above  $-10$  °C).





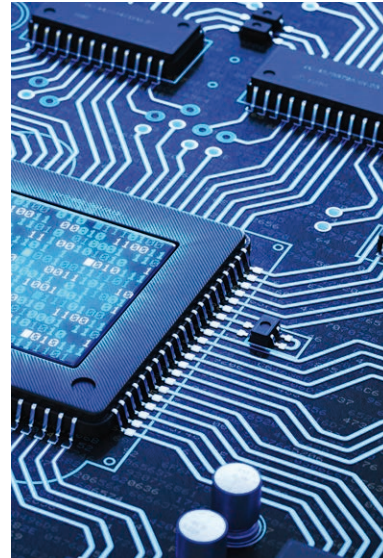
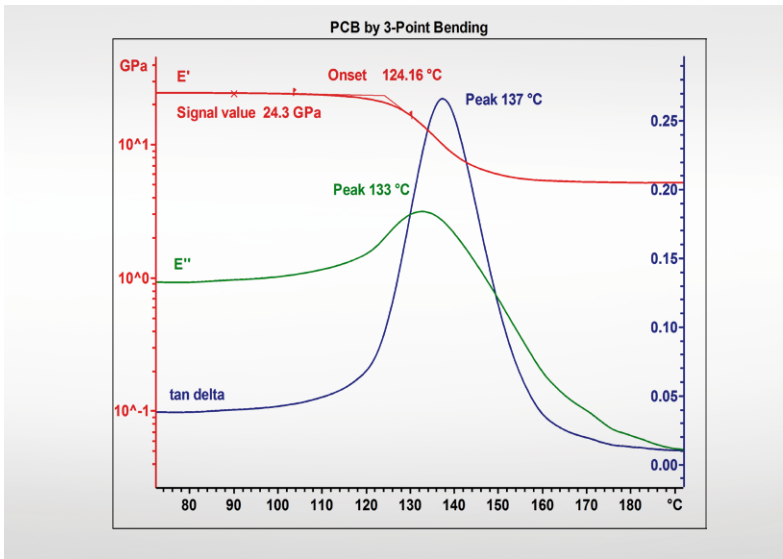
### Master curves of styrene butadiene elastomers

Master curves over a wide frequency range provide detailed information about material properties. The excellent temperature stability and accuracy together with the possibility of performing measurements at high frequencies allow master curves to be quickly constructed. The figure displays master curves of unvulcanized and vulcanized SBR. The curves not only describe dynamic behavior but also provide information about the molecular structure and network.



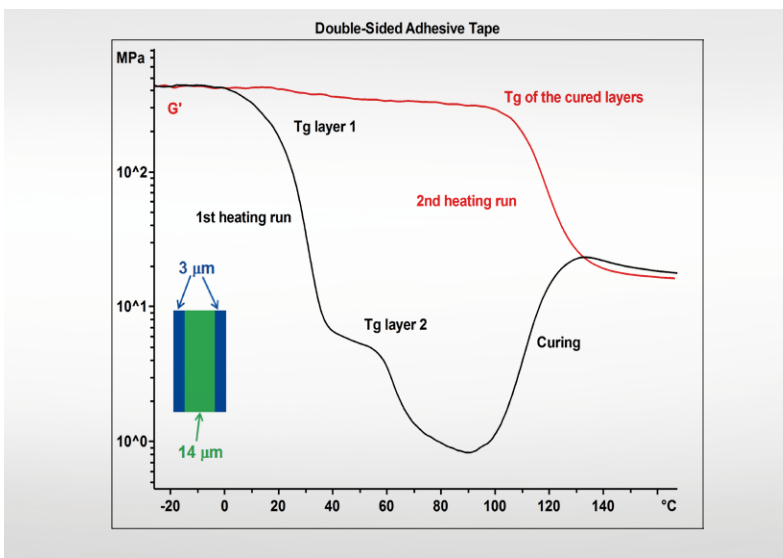
### Polyimide film measured in tension

Polyimides are high performance polymers that are not brittle at low temperatures and that can be exposed to temperatures of up to 400 °C for a short time without losing their mechanical stability. The loss modulus curve (E'') of a 20- $\mu$ m thick polyimide film measured in the tension mode at 1 Hz shows relaxation processes at about -59 °C, 186 °C and 311 °C (maxima in E''). The process at 311 °C is the glass transition. The material begins to decompose at about 450 °C.



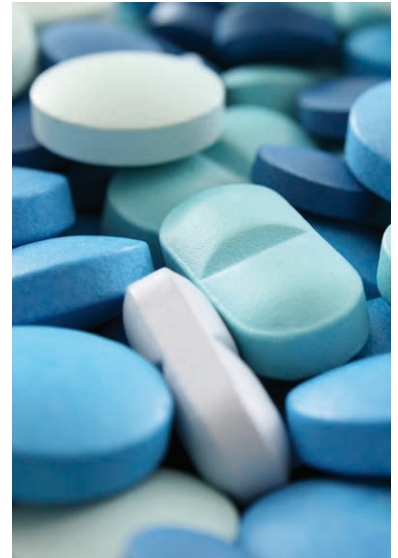
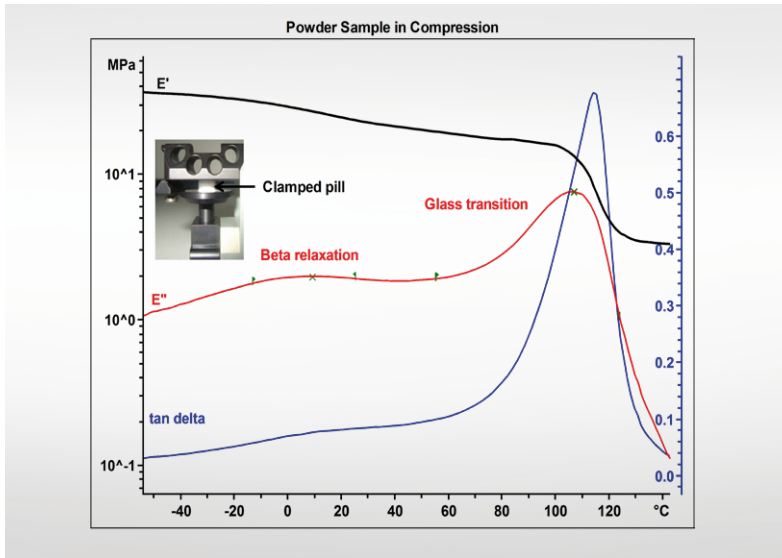
### Determination of the modulus of a composite material

Printed circuit boards are made of fiber-reinforced plastics. Their maximum working temperature must be known and is usually about 10 °C below the glass transition temperature. Another important factor is the modulus. This and the glass transition can be easily determined by means of a DMA measurement in the 3-point bending mode. In this example, the maximum operating temperature should not exceed 114 °C. Up to this temperature, the modulus remains practically constant (24 GPa).



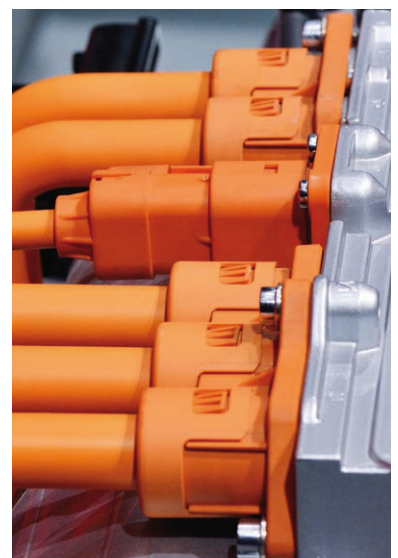
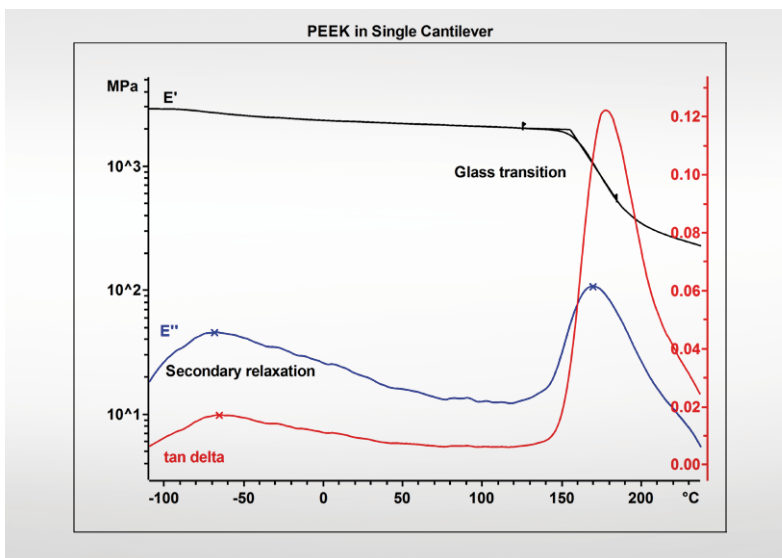
### Shear measurement of an adhesive tape

The sample was a 20-μm thick double-sided adhesive tape. The adhesive layer (3 μm) on one side was slightly stickier than on the other side (3 μm). The figure shows the storage modulus of the tape. In the first heating run, the modulus decreases in two steps. The steps correspond to the glass transitions (T<sub>g</sub>) of the two adhesive layers (the T<sub>g</sub> of the stickier layer is lower). The layers cure in the range 100 to 140 °C. The second heating run shows the glass transition of the fully cured material.



### DMA analysis of powders

Powder samples can be analyzed by DMA using a so-called powder pocket. This consists of a thin folded steel sheet which is filled with powder and measured in the single cantilever mode. Alternatively, powders can be compressed to a pellet and measured in compression. This allows relaxation processes to be detected with better sensitivity. The diagram shows DMA curves of a compressed active pharmaceutical ingredient. The sample exhibits  $\beta$ -relaxation at about 9 °C and a glass transition at about 107 °C.

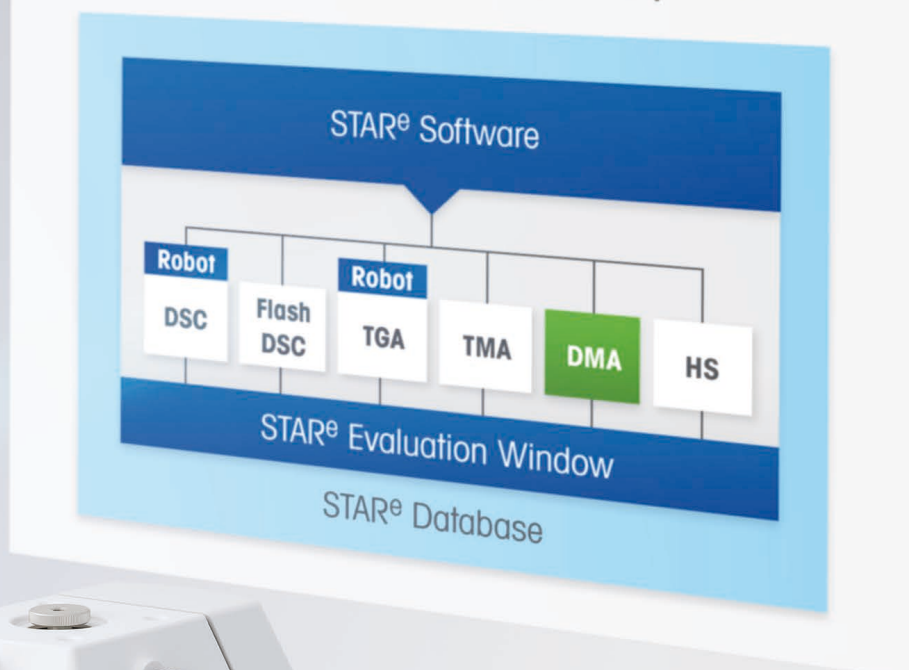


### Analysis of PEEK in single cantilever mode

Polyether ether ketone (PEEK) is a high-temperature resistant plastic with a melting point of 335 °C. It is, for example, used in the automobile industry for thermally stressed components. To characterize the sample by means of DMA, a thin plate (17 x 5 x 2 mm) was measured in the single cantilever mode between -120 and 240 °C. Two effects were detected: the secondary relaxation at -65 °C and the glass transition at 155 °C.



# Complete Thermal Analysis System



## Complete thermal analysis system

A complete thermal analysis system consists of four basic measuring techniques. Each technique characterizes the sample in its own specific way. The full picture that simplifies interpretation is only obtained when all the different results are combined. The techniques measure the mechanical modulus (DMA), the heat flow (DSC, Flash DSC), the weight change (TGA), and the change in length (TMA). All these measurement variables change with temperature.

# World-Class Service and Support Provide Results You Can Trust

**METTLER TOLEDO's portfolio of services is designed to ensure the continuous performance and reliability of your thermal analysis systems. Factory-trained in Switzerland, our worldwide teams bring the professional expertise and know-how needed to provide you with the highest level of after-sales support as well as the experience necessary to optimize services for your own particular needs.**

## Interesting and comprehensive application material



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## DMA/SDTA 1+ Specifications

### Temperature

Range	-150 to 500 °C
Technical resolution	0.003 K
Accuracy	0.5 K

### Force

Range	0.001 to 40 N
Technical resolution	0.15 mN (0 to 5 N), 1.5 mN (5 to 50 N)
Sensitivity	1 mN

### Displacement

Range	±1.6 mm
Technical resolution	0.6 nm
Sensitivity	5 nm

### Stiffness

Range	10 to 10 <sup>8</sup> N/m
Precision	0.2%

### Tan delta

Range	0.0001 to 5000
Technical resolution	0.00001
Sensitivity	0.0001

### Frequency

Range	0.001 to 1000 Hz (*)
Technical resolution	0.00001
Frequency increments ( $\Delta f$ )	0.0001
Frequency modes	<ul style="list-style-type: none"> <li>• Linear or logarithmic</li> <li>• Frequency series</li> <li>• Multi-frequency</li> </ul>

### Deformation modes

3-point bending	Length: 30 to 90 mm, length: 20 to 80 mm
Dual cantilever	Width: <15 mm, thickness: <5 mm
Single cantilever	Max. Sample length: 100 mm
Bending stiffness range	1 to 10 <sup>8</sup> N/m
Shear	Diameter: ≤15 mm, thickness: ≤6.5 mm
Shear stiffness range	1 to 10 <sup>8</sup> N/m
Tension	Length: 19.5 mm, 10.5 mm, 9.0 mm, 5.5 mm Width: ≤7 mm, thickness: ≤3 mm
Tensile stiffness range	1 to 10 <sup>7</sup> N/m
Compression	Diameter: ≤19 mm, thickness: ≤9 mm
Compressive stiffness range	1 to 10 <sup>7</sup> N/m

### Approvals

IEC/EN 61010-1, IEC/EN61010-2-010 and IEC/EN61010-2-081  
 CAN/CSA C22.2 No. 61010-1, No. 61010-2-010 and No. 61010-2-081  
 UL Std. No. 61010-1  
 IEC/EN61326-1 (class B)  
 IEC/EN61326-1 (industrial requirements)  
 FCC, Part 15, class A  
 AS/NZS CISPR 11, AS/NZS 61000.4.3

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