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# New Developments for Temperature Control of Rotational Rheometers

Jörg Läuger, Klaus Wollny, Monika Bernzen, Gerhard Raffer

Physica Messtechnik GmbH, Vor dem Lauch 6, D-70567 Stuttgart, Germany
phone: +49-711-72091-0, fax: +49-711-72091-30, e-mail: laeuger@physica.anton-paar.de
http://www.physica.de.

## ABSTRACT

Three temperature control units for different temperature ranges and applications based on Peltier, electrical resistance and convection heating, respectively, are described. These temperature chambers have been developed for having the correct absolute temperature, minimal temperature gradients and no temperature overshoots or undershoots during the temperature setting.

## INTRODUCTION

Since for almost all samples the temperature has a great influence on the rheological behavior, controlling the temperature with a high precision is crucial to receive reliable rheological data. However, in practical measurements inaccurate temperature control is still responsible for a large number of measurement uncertainties and errors. In 1994 Macosko[1] wrote:

*‘’ It can be as important to control the temperature, pressure, or humidity of a sample as it is to control the shear stress. Yet these factors, especially temperature, seems to be less exciting to the engineers who design rheometers. At least there are often large temperature gradients even in popular commercial instruments.’’*

Although this statement might still hold for some commercial rheometers on the market, it is the aim of this paper to show that at least some engineers who design rheometers are eager to solve these problems. As will be shown it is possible to build various temperature control systems based on different principles which fulfill the requirements of an accurate temperature control in all respects and are still commercially affordable.

Rheological measurements require an accurate and precise control of the sample temperature. Possible sources of errors are:

1. Difference in the absolute temperature,
2. Non-uniform temperature distribution throughout the sample, i.e. temperature gradients,
3. Variations in the temperature history, for example temperature overshoots or undershoots which might change or destroy the sample,
4. Fluctuations in the temperature, i.e. insufficient temperature stability

Designing a temperature control unit has to fulfill the main tasks of to set and measure the right absolute temperature, to maintain a constant temperature throughout the sample without any temperature gradients, and to produce no significant temperature overshoots during the control process.

Three different temperature control units for different temperature ranges and applications were developed which fulfill the above mentioned requirements:

1. A Peltier temperature device with an actively Peltier controlled bottom plate

and an actively Peltier controlled hood (PTD 150) (pat. pent.),

2. A direct electrical heated system with resistance heated bottom plate and resistance heated hood (ETD 400),
3. A convection temperature device based on electrical resistance heating and forced convection (CTD 600)

Whereas the Peltier system PTD 150 and the direct electrical heated system are based on a combination of conduction and free gas convection the convection temperature device CTD 600 uses a forced gas convection to control the temperature.

For all units special certified calibration sensors are available which in combination with an automatic temperature calibration software module can be used by the individual user to assure that the sample has the correct absolute temperature.

For development purposes, special tools were designed to determine and minimize the horizontal and vertical temperature gradients. Results of temperature gradient measurements are shown for all three units.

## EXPERIMENT AND METHODS

### Instrument

All temperature chambers have been developed for the rheometers of the Physica MCR series. All measurements have been performed on a Physica MCR 300 rheometer equipped with an electronically commutated synchronous motor (EC-Motor)[2,3].

### Temperature sensor tools

To measure the temperature distribution three temperature sensor tools have been developed. The sensor tools consist of a disc or bar made of a plastic material in which a number of thermo couples are embedded.

Figure 1a shows schematic drawings of the sensor tools and the location of the temperature probes for parallel-plate geometries. Both disc tools have a thickness of 2 mm and diameters of 25 mm or 50 mm, respectively. The smaller tool has one temperature probe located at the top and one at the bottom of the disc, respectively, allowing to evaluate the temperature gradient in vertical direction. In the tool with 50 mm diameter four temperature probes are embedded which enable the determination of temperature gradients in vertical and horizontal direction.

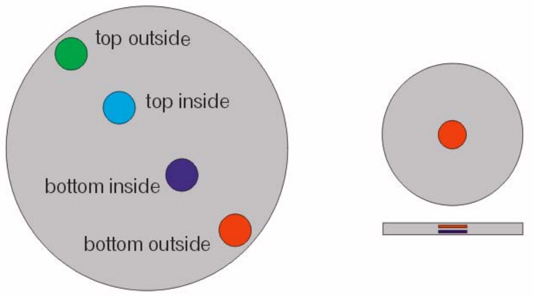

Figure 1a: Sensor tools for parallel-plate geometries (see text)

In Figure 1b a tool for testing the temperature behavior for solid-bar geometries is shown. It consists of four temperature probes at four different positions. Before using the sensor tools all temperature probes have been calibrated with respect to a certified high precision temperature probe.

Figure 1b: Sensor tool for solid-bar geometries for torsional DMTA measurements (see text)

### Temperature Calibration Sensors

Special calibration sensor were developed which in combination with an automatic temperature calibration software module can be used to assure that the sample has the correct absolute temperature. A calibrated Pt100 sensor is inserted in a disc which can be placed between the upper and lower plate of any temperature control unit like a normal sample as shown in Figure 2. The sensor is then connected directly to the rheometer.

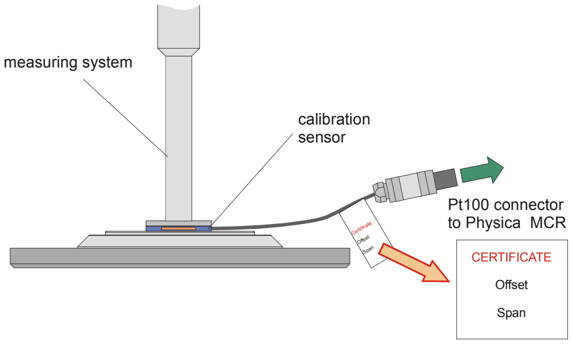

Figure 2: Schematic diagram of the temperature calibration sensor.

The calibration procedure itself is carried out fully automatically by a temperature calibration module of the Physica US 200 software. The software checks the temperature at user selected temperature values, the calibration data are calculated and can be stored in the software. Two types of calibration sensors covering different temperature ranges are available. Each sensor is delivered with a certificate.

## TEMPERATURE UNITS AND RESULTS

### Advanced Peltier System PTD150 (pat. pent.)

The Advanced Peltier System PTD 150 consists of a Peltier controlled bottom plate and an actively Peltier controlled hood (pat. pent.). This technology together with a small gas flow in the chamber (either dry air or inert gas) results in an extremely small temperature gradient across the sample.

a) b) c)

Figure 3: Different setups of a Peltier system: a) open system, b) system with insulating cover, c) PTD 150 with an actively controlled hood (pat. pent.)

Figure 3 sketches the differences between the various setups. Figure 3a represents a system with controlled bottom plate and no protection, i.e. an open system which exhibits large temperature gradients. Figure 3b shows the setup with controlled bottom plate and a passive insulation cover. This setup still leads to large temperature gradients in the sample. Whereas in Figure 3c the principle of the PTD 150 with the actively controlled bottom plate and the actively controlled hood is displayed, which reduces gradients to insignificant levels.

Figure 4a: Photo of the Advanced Peltier System PTD 150 (pat. pent.)

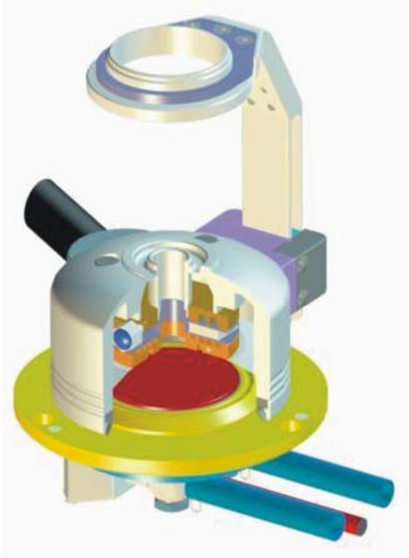

Figure 4b: Drawing of the Advanced Peltier System PTD 150 (pat. pent.)

Figure 5 shows the test results obtained with the 25 mm sensor tool in the temperature range –30°C to +120°C using a conventional Peltier system with a passive insulation cover. A standard stainless steel parallel-plate geometry with a diameter of 25 mm was used as the upper tool.

The design of the PTD 150 is shown in Figure 4a and 4b. The actively controlled hood can be moved up and down on a guiding rail thus giving easy access for sample loading and trimming.

As can be seen large temperature gradients occur. A difference between the set temperature and the sample temperature might be adjusted by a temperature calibration, but the temperature gradients can not be corrected. At –30°C and +120°C temperature gradients as large as 12 K and 16 K occur, respectively. Even at intermediate temperatures like 60°C gradients of about 6 K are existing. A simple redesign of the measurement geometry will not improve the situation substantially, since the geometry used is already designed for having a small heat flow through the shaft. Even though the 2 mm thick plastic material as a sample represents the worst case there still will be a rather large and significant temperature gradient across the sample in most practical applications.

The huge improvement offered by the System PTD 150 is shown in Figure 6. The measurement was performed with the 50 mm diameter sensor tool in the temperature range from –40°C to +150°C. The use of the actively Peltier controlled hood ensures that the absolute sample temperature matches the set temperature and it eliminates virtually any temperature gradients. Figure 7 shows a similar measurement for different temperatures.

A closer examination of data like in Figures 6 and 7 gives an average sample temperature which only has a maximum deviation of ± 0.2 K from the set temperature. The temperature gradients in both direction are lower than ± 0.3 K (deviation from the average temperature) across the sample in the entire temperature range from –40°C to +150°C even for the plastic tools with 2 mm thickness

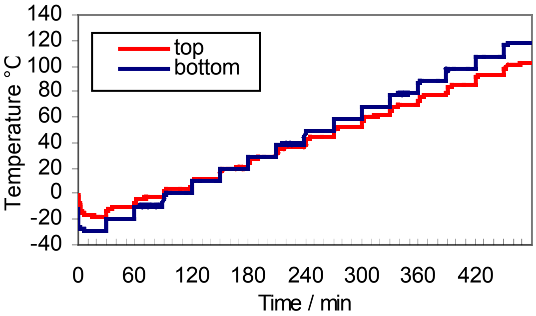

Figure 5: Temperature gradients with a conventional Peltier system.

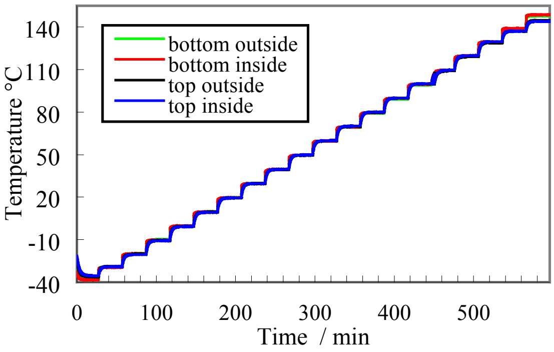

Figure 6: Temperature gradients for thr PTD 150.

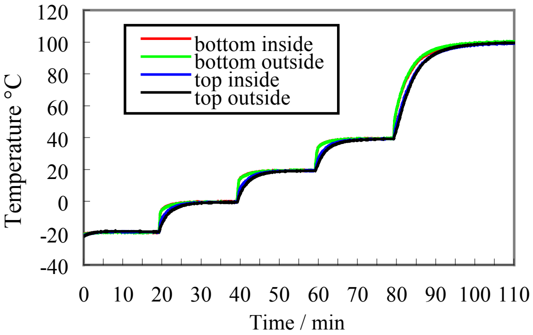

Figure 7: Temperature gradients with the 50 mm sensor tool for the PTD 150.

Typical applications for the PTD 150 are measurements on samples which require exact temperature control in the range from –40°C to +150°C. Examples are tests on lubrication grease at low temperatures, food samples such as ice cream at low temperatures, or chocolate at +40°C, asphalt, surfactants, the exact temperature dependence and stability of cosmetics or pharmaceuticals, the gelling behavior of PVC pastes, and many more. The exact temperature throughout the sample allows investigations of crystallization processes or phase transitions of complex fluids.

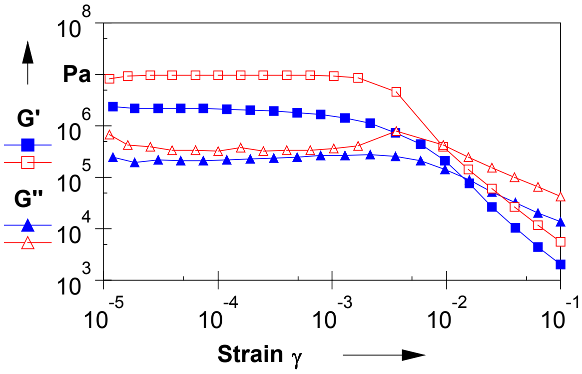

Figure 8. Strain sweep ($\omega = 10s^{-1}$) on a lubrication grease at –30°C (filled symbols) and at – 40°C (open symbols)

Figure 8 shows results for a typical application of the PTD 150. An strain sweep has been performed on a lubricating grease at –30 and –40°C. As expected the storage (G') and the loss (G'') modulus are at higher values for –40°C compared to the measurement at –30°C. These results have been compared to results measured with a gas convection temperature device and found to be in good agreement. The main advantage of the PTD 150 is that these temperatures are reached by the Peltier system and no nitrogen supply is needed.

### Electrical temperature device ETD 400

The electrical resistance heated system ETD 400 which is shown in Figure 9 uses the same principle of an actively controlled bottom plate and an actively controlled hood as the Peltier system PTD 150. Whereas the PTD 150 uses Peltier elements to control the bottom plate and the hood, in the ETD 400 the bottom plate and the hood are controlled by electrical resistance heating. Together with a small gas flow in the chamber (either dry air or inert gas), which leads to a free gas convection, the control by electrical resistance heating results in an extremely small temperature gradient across the sample. The ETD has a working range from ambient temperatures up to 400°C.

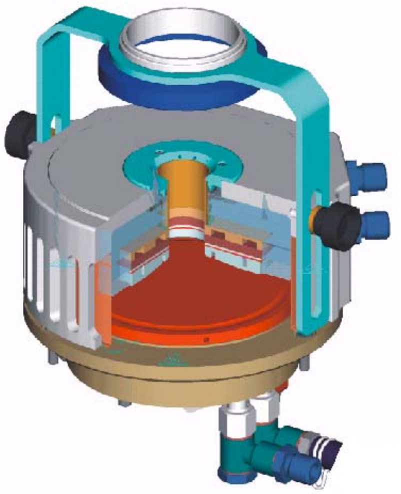

Figure 9: Design of the ETD 400

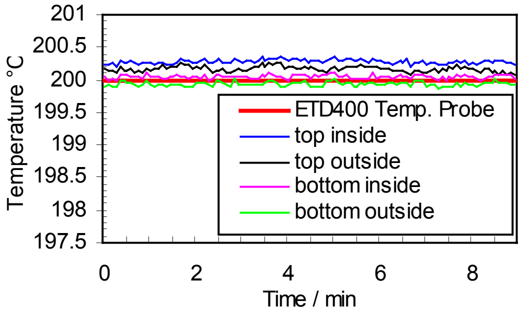

Figure 10: Results for the ETD 400 temperature probe and the four probes using the 50 mm tool at 200°C.

In Figure 10 an example for a measurement of the temperature gradients in the ETD 400 is shown. The test was performed at a constant temperature of 200°C with the 50 mm diameter sensor tool. As can be seen the temperature gradients in horizontal and vertical direction are lower than ± 0.3 K even for the 2 mm thick sensor tool. This behavior was verified over the whole temperature range of the chamber.

Convection Temperature Device CTD 600

The CTD 600 shown in Figure 11 is based on a combination of electrical and convection heating and has a fully symmetrical set up. It consists of two half shells which can be independently moved on a rail for an easy opening and closing of the chamber giving good sample access for trimming and cleaning. Glass windows allow the observation of the sample even while the chamber is closed.

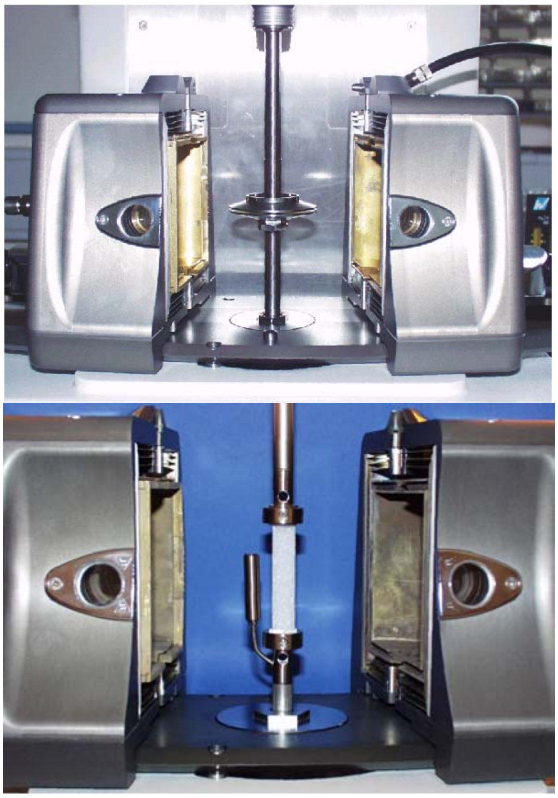

Figure 11: CTD 600 with parallel-plate and solid-bar (DMTA) geometry

The CTD 600 can be operated with air from ambient temperature up to + 600°C. By the use of a liquid nitrogen source and an evaporation unit the temperature range is extended from –150°C to +600°C. Different measurement geometries like parallel-plate, cone-and-plate, and fixtures for solid-bars and films can be used.

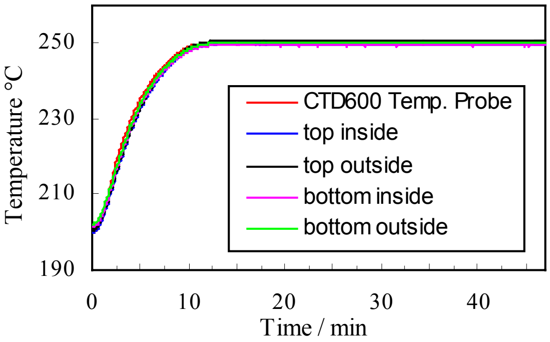

Figure 12: Ramp from 200°C to 250°C. Results for the CTD 600 temperature probe and the four probes using the 50 mm tool

The gas is heated by electrical resistance in the two half shells and flows into the sample room through a number of small holes close to the shafts of the lower and the upper tools. The gas flow in the sample area is totally symmetric leading to a uniform temperature distribution. By the use of liquid nitrogen temperature ramps can be conducted from –150°C up to +300°C without any switching from nitrogen to air. The liquid nitrogen consumption is automatically adjusted according to the measuring temperature. This leads to a low overall nitrogen consumption.

For the parallel-plate and cone-and-plate setup the temperature sensor is located in the shaft of the lower tool directly below the sample. In the case of the solid-bar geometry the temperature sensor is mounted close to the sample bar.

As an example in Figure 12 and 13 results of a temperature jump from 200°C to 250°C measured with the 50 mm diameter sensor tool are shown. From Figure 12 and 13 it can be seen that the CTD 600 shows only a modest temperature overshoot and exhibits only very small and insignificant temperature gradients throughout the sample.

Moreover, the results in Figures 12 and 13 prove that the reading of the temperature sensor of the CTD 600 represents exactly

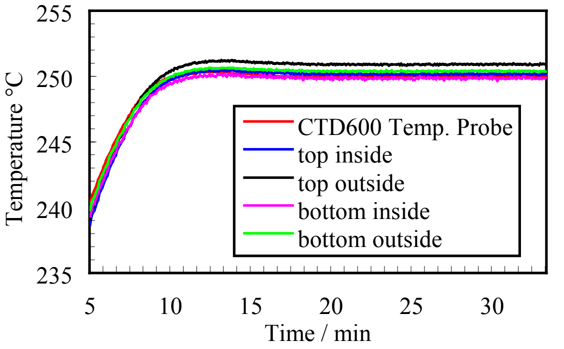

Figure 13: Same data as in Figure 12 plotted on a wider scale

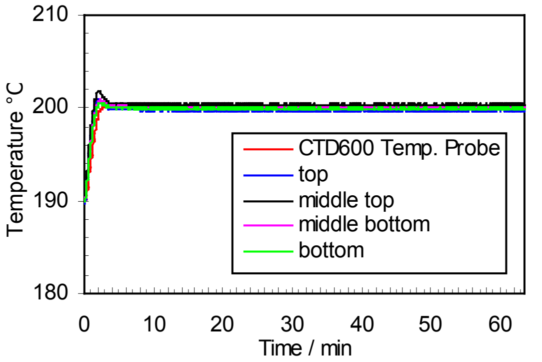

Figure 14: Ramp from 150°C to 200°C. Results for the CTD 600 temperature probe and the four probes of the solid-bar sensor tool (DMTA-measurements).

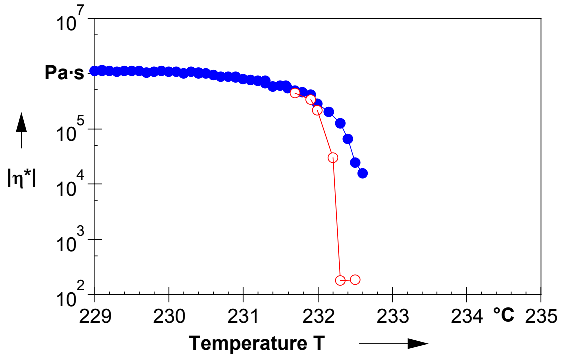

Figure 15 - Temperature ramp on a tin powder. Filled Symbols: 0.5C/min; Open Symbols: 1°C/min. Specified: 231.97°C.

the sample temperature. This is an important feature which distinguishes the CTD 600 from the Peltier system PTD 150 and the electric system ETD 400. The Peltier and the electric system are based on conduction in combination with a free convection. In these units the temperature sensor is located directly below the sample but also in direct contact with the actively controlled bottom plate. Therefore the temperature sensors in these units react faster to a change in the set temperature as the sample. The sample needs a certain time for temperature equilibration and only after the equilibrium is reached the measured temperature represents the sample temperature. In the CTD 600 the sample temperature is controlled by a forced gas convection. The temperature sensor of this unit is located in such a way that the temperature measured by this sensor represents exactly the sample temperature not only after equilibration but also during the heating process itself. The user always knows the absolute sample temperature even during heating or cooling.

In Figure 14 an example of a temperature gradient measurement for the solid-bar geometry is shown. The gradients were measured during a temperature jump from 150°C to 200°C with the sensor tool described in Figure 1b. The solid-bar geometry allows the testing of solids in torsional oscillation (DMTA). As for the parallel-plate geometry the CTD 600 exhibits only a very small temperature overshoot and insignificant temperature gradients across the sample for the solid-bar geometry. Again the temperature measured by the temperature probe in the CTD 600 represents the temperature of the sample even during the heating process. This excellent temperature behavior was verified for the whole temperature range of the CTD 600 for both the parallel-plate and the solid-bar geometries.

A simple test for confirming the absolute temperature of a temperature control unit is the measurement of the melting point of known material. In Figure 15 the complex viscosity $|\eta^*|$ as obtained in an oscillatory test at a fixed amplitude and a fixed frequency for a tin powder sample is shown. Since the measurement was done by applying a certain normal force the viscosity $|\eta^*|$ is a relative quantity and only the change in the complex viscosity is relevant. As can be seen the viscosity drop which represents the melting point of the tin sample occurs at 232°C ± 0.1°C which is in close agreement with the melting point of tin powder (231.97°C) as found in literature. This result and similar tests confirmed that the CTD 600 indeed gives the correct absolute sample temperature.

An example where the temperature accuracy is tested indirectly is shown in Figure 16. A polystyrene melt supplied by the University of Freiburg / Germany as a calibration standard (PS 140)[4] was measured in a frequency sweep at 170°C. The resulting zero-shear viscosity is $\eta_0$ = 46900 Pas. This represents a difference of about 2% to the value of $\eta_0$ = 48000 Pas provided by the University of Freiburg. However, in such a test not only the temperature has an influence on the result but also other parameters like the accuracy of the rheometer itself, the sample preparation, and the filling of the gap.

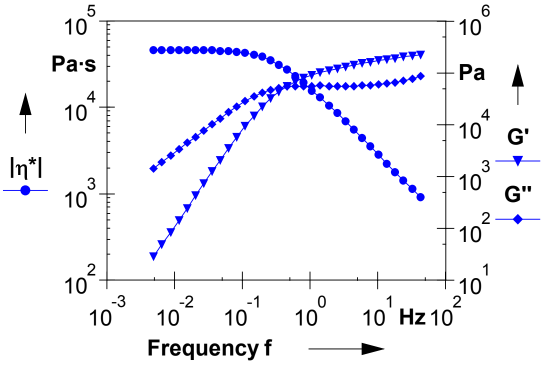

Figure 16: Frequency Sweep on the sample PS140 at 170°C. Slight overfilling (Trim position at 1.05 mm, Gap 1 mm).

## CONCLUSIONS

Three different temperature devices based on the Peltier effect (PTD 150), electrical resistance (ETD 400) and forced gas convection (CTD 600), respectively, were presented. Measurements of absolute temperatures and temperature gradients across the sample show the excellent performance of all three described devices. All units provide a sample temperature control with the correct absolute temperature, insignificant temperature gradients across the sample, good temperature stability with time and almost no temperature overshoots or undershoots during the heating or cooling. The temperature measured by the temperature sensor of the forced gas confection device represents exactly the sample temperature even during heating and cooling processes. Calibration sensors covering the whole range of the presented temperature devices are available for automatic temperature calibration by the user himself.

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