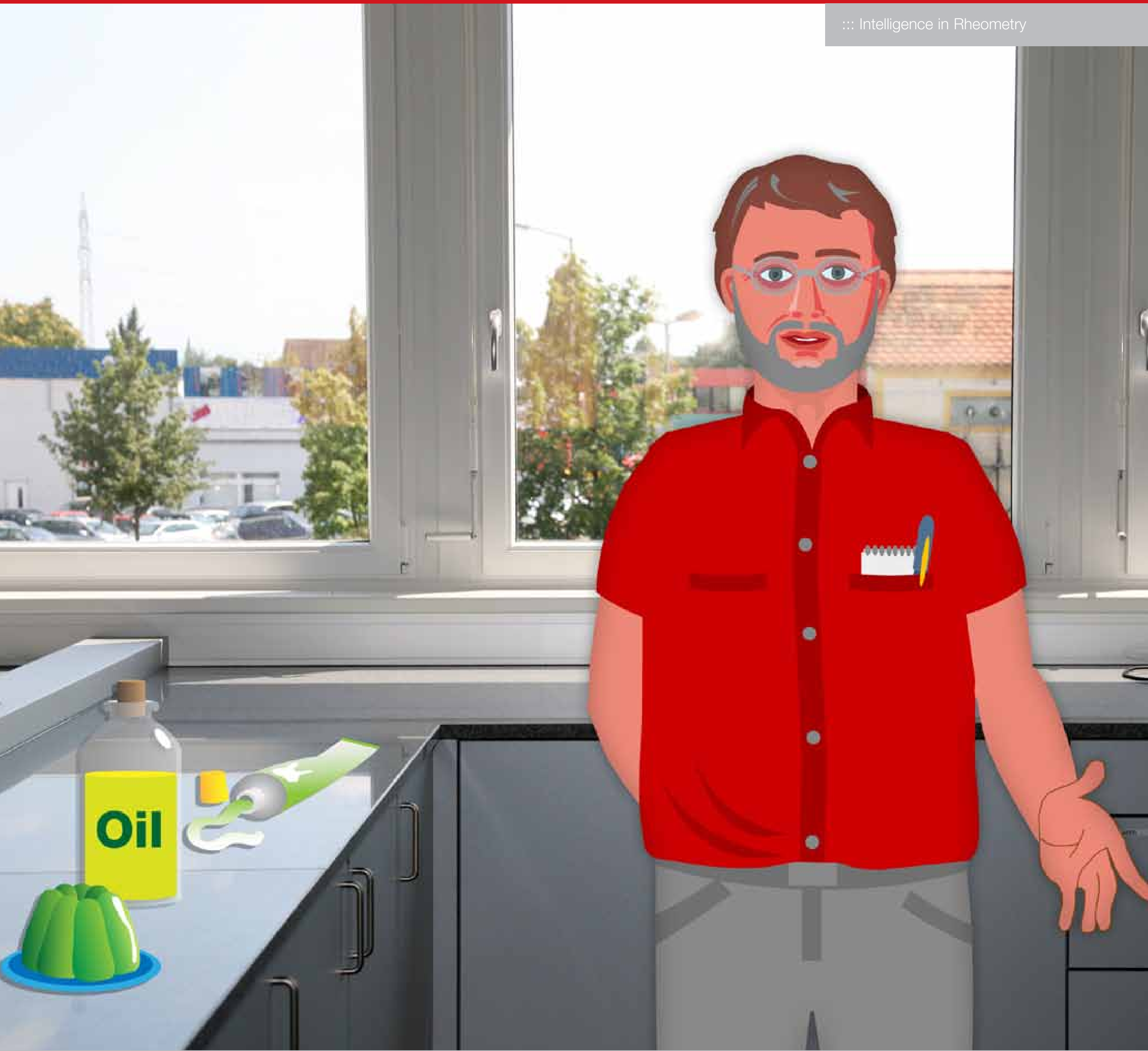
Anton Paar

::: Intelligence in Rheometry



Tips and Tricks from Joe Flow

Temperature Control: Heating Up and Cooling Down

"I would like to discuss the importance of temperature control in rheology. To see how much influence the temperature has on the rheological behavior of samples, you only need to read off the viscosity values of a calibration standard at different temperatures. A difference of just one degree centigrade between two measurements leads to a 7 % deviation in the viscosity value. It is therefore extremely important to control the temperature accurately and precisely."

Which temperature device is suitable for which temperature range?

Depending on the temperature range and type of sample there are a number of temperature devices available.

Today, for standard temperature devices **Peltier elements** are usually used. With appropriate counter-cooling, these elements can be operated from -40 °C to +200 °C.

Temperature range Peltier element	-40 °C to +110 °C	-15 °C to +150 °C	+35 °C to +200 °C
Counter-cooling	-15 °C	+20 °C	+70 °C
Cooling agents	Water + ethanol or isopropanol	Water	Water

Table 1: Suggestions for counter-cooling temperatures for different temperature ranges

The suggested temperatures in Table 1 are valid for isothermal measurements. If parts of the temperature ramp are outside the suggested ranges, you can continuously adjust the counter-cooling. If the thermostat/cryostat is connected to the rheometer, this is easy to do via the RheoPlus software. Good counter-cooling extends the working life of Peltier elements.

With a heating rate of up to 60 °C per minute, Peltier elements allow a quick and accurate heating and cooling of the sample using the thermoelectric effect. Rather than focusing on a high heating rate, make sure that the sample can reach the temperature in the specified time.

Another option for temperature control is to use **liquid-temperature-controlled systems**. These are particularly suitable for measurements at room temperature, although they can also achieve temperatures from -20 °C to +180 °C, depending on the liquid and thermostat used. The maximum heating performance for a temperature ramp is approx. 1 °C/min.

The following systems are available for samples which should be measured at higher temperatures:

- **Electrically temperature-controlled plates in combination with an electrically heated hood,** which reach high heating rates and temperatures up to 400 °C.
- **Convection heating systems**, which achieve even higher temperatures, i.e. up to 1000 °C. (see Fig. 1)

Both systems enable cooling down to -150 °C with liquid nitrogen and therefore allow determination of the glass transition temperature (Tg) of various plastics and other samples.

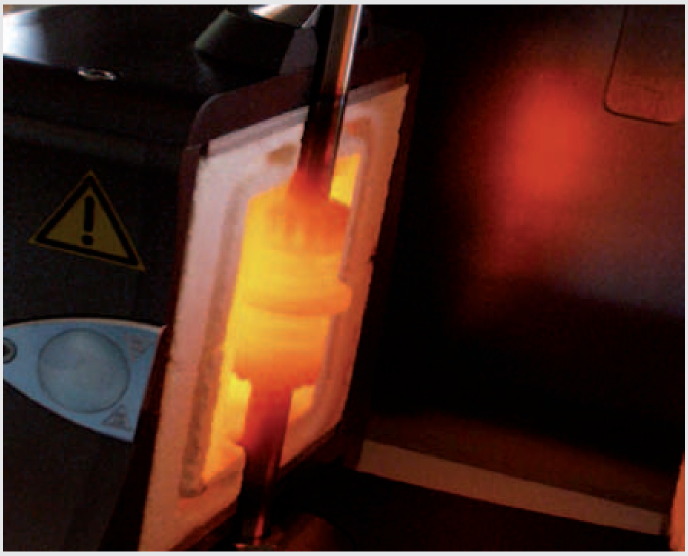


Fig. 1: Red-hot CTD 1000, opened at 1000 °C

Convection heating systems are closed systems which control the temperature via a hot or cold gas volume flow. The outside temperature-isolated jacket of the system is cooled with water and can still be touched with bare hands, despite the very high measuring temperatures inside.

The new rheometer system FRS 1600 even permits the measurement of glass melts or metal melts at temperatures up to 1600 °C.

What do I need to consider when setting the temperature?

Many products are very dependent on the temperature. This behavior can be determined in a **temperature ramp**. A temperature ramp is measured in oscillation within the linear-viscoelastic range of the sample. It should be performed at a constant heating rate of **0.5 to 2 °C/min**. At this heating rate most samples have enough time to adjust to the temperature change.

For temperature ramps it is necessary to use an additional **temperature-controlled hood** in combination with the bottom plate. This guarantees a virtually **gradient-free temperature distribution** in the sample. It also has a constant volume flow of dry air which prevents ice forming at temperatures below 0 °C.

If you do not use a hood for a measurement, significant **temperature gradients** occur in the sample at temperatures from 10 °C above or below room temperature (see Fig. 2).

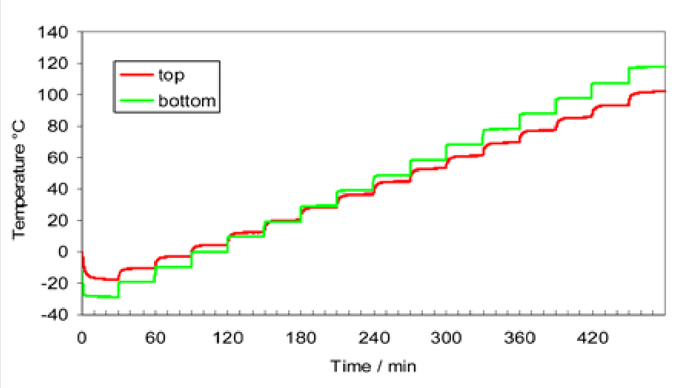


Fig. 2: Temperature progression of a Peltier plate without a hood from -40 °C to +120 °C

Fig. 3 shows the temperature progression of the sample when using an actively heated hood.

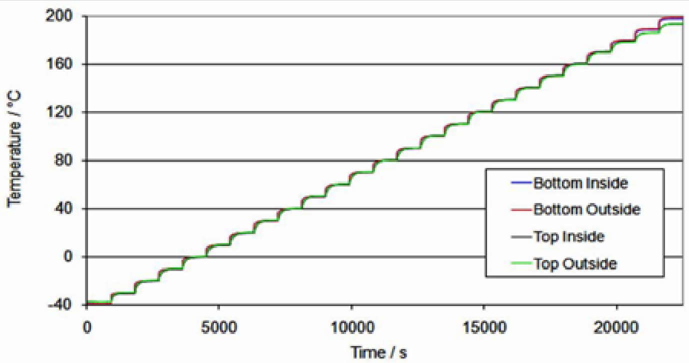


Fig. 3: Temperature progression of a Peltier plate with a hood from -40 °C to +200 °C

As the measuring bob (cone, plate) and the measuring plate both expand during temperature ramps, **the zero gap should always be set in the middle of the ramp**. This prevents the gap deviating too much from the setting at the end of the test.

Measurements with **TruGap™** have clear benefits. During the measurement the gap is constantly determined via magnetic induction and exactly adjusted. For samples which react to temperature changes by expanding or shrinking considerably, an additional normal force setting of 0 N is required. In this case the rheometer constantly adjusts the gap as the sample expands or shrinks and **TruGap™** continues to measure the gap with high accuracy.

How correct is the temperature which is displayed?

You can perform a calibration to check the precision of your temperature device and adjust if required. For a calibration of cylinder measuring systems you need a calibrated and certified temperature probe; for plate measuring systems you need a temperature sensor in the shape of a sample.

The calibration runs automatically with the **RheoPlus software**. The newly determined calibration constants can then be stored for the temperature device.

The calibration sensor for parallel plate measurements should be coated with a thermally conductive material. The measuring gap should be set via the normal force limit. The thickness of the measuring sensor determines the measuring gap.

If you want to calibrate a cylinder measuring system, use a suitable calibration standard and place the temperature sensor in the sample.

The exact procedure for calibration is described in detail, step by step, in the instruction manual for the software.

How long should I wait for tempering my sample?

To make sure the sample has reached the required measuring temperature, you should wait **approx. three minutes** after beginning temperature control, if possible. This holds for both parallel plate and cone/plate measurements.

If you are using a cylinder measuring system, you should usually give the sample five minutes **- ten minutes is better -** to reach the set temperature. Due to its large volume the sample needs more time to reach the set temperature. The measuring system also needs time to reach the measuring temperature.

If the sample is reactive it may be better not to wait after beginning temperature control, in order to measure the reaction. It may also be better not to wait if the sample contains solvents which dry out very quickly.