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::: Intelligence in Rheometry

Tips and Tricks from Joe Flow

Limits of the measuring range: This far and no further!

No matter which rheometer you use for your investigations, each instrument has measuring limits which you should respect.

Which limits are there?

Every rheometer has its limits. Depending on whether it is an air-bearing or ball-bearing rheometer, the measuring limits for the raw data are different. Raw data are torque, speed and angle of deflection. From the raw data, rheological properties such as shear stress, shear rate and deformation are calculated using conversion factors.

Table 1 shows the measuring ranges for raw values from different rheometers.

Limits of raw data/ instrument	Torque range	Speed range	Angle of deflection setting	Angle reso-lution
RheolabQC ball bearing rheometer	250 µNm to 75 mNm	0.01 to 1500 1/min	-	2 µrad
MCR 51 ball bearing rheometer	250 µNm to 125 mNm	10^{-5} to 3000 1/min	1 to ∞	0.01 µrad**
MCR 101 air bearing rheometer	0.1 µNm to 150 mNm	10^{-6} to 3000 1/min	1 to ∞	0.01 µrad**
MCR 301 air bearing rheometer	0.05 µNm to 200 mNm	10^{-7} to 3000 1/min	0.1 to ∞	0.01 µrad**
MCR 501 air bearing rheometer	0.05 µNm to 230 mNm	10^{-7} to 3000 1/min	0.1 to ∞	0.01 µrad**
MCR 501s air bearing rheometer	0.05 µNm to 300 mNm	10^{-7} * to 3000 1/min	0.1 to ∞	0.01 µrad**

* at a speed of 10^{-7} 1/min one rotation would take 6944 days
** the resolution corresponds to 7 cm along the 42000 km long equator

Table 1 Limits of raw data for RheolabQC, MCR 51, MCR 101, MCR 301 and MCR 501

How do I recognize measuring limits?

In most cases you will recognize measurements which are at the limit or outside of the measuring range by looking at the measuring curves. They do not have a constant slope, show fluctuations in the measuring points or have a bend which can be particularly pronounced.

I recommend always displaying the status messages in the measuring value table in the RheoPlus software. Status messages such as M- or M+ show results which are outside the upper or lower torque range.

Can I influence the limits of the measuring range?

Some measuring ranges can be influenced by changing the diameter of the measuring system, the cone angle or the gap between the plates. However, many measuring limits depend on the sample. In this case you can adapt the measuring profile or change the test type (rotation or oscillation) to optimize the measurement results.

Depending on the rheometer you use, you can set it to graphically display the maximum measuring range for each measuring system you use.

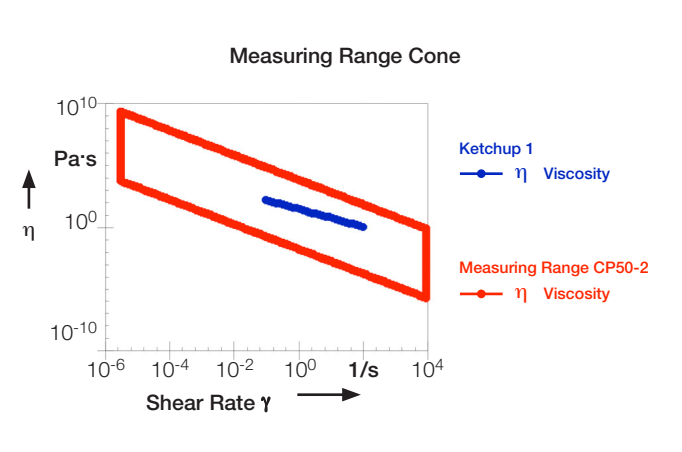

Fig. 1: Diagram showing the limits of the measuring range for cone CP50-2 (red) and the results of a rheological measurement (blue)

This function is available as an analysis method in the RheoPlus software. In the analysis window, enter the analysis method "Generate Measuring System Envelope", select the measuring system and rheometer type and start the analysis. The diagram shows the measuring range of your measuring system.

Checking this diagram together with the result tells you immediately whether the measurement is in the ideal measuring range or not. If the measurement results are near or outside the limit, I recommend using another measuring system.

Tips for measurements in a high shear rate range

High shear rates are produced best using a small cone angle (e.g. 0.5°) or small gap between the plates (e.g. 0.25 mm).

If you are investigating a sample for the first time with the rheometer, visually check the sample in the gap during the measurement. Is the gap still completely filled with sample or does sample come out of the gap?

Figure 2 shows a measurement in which some of the sample escapes from the gap at a shear rate of approx. 5 1/s. You cannot always detect this occurrence in the viscosity curve. Nor is it marked in the table with a warning. The decreasing shear stress curve shows an incorrect measurement, however.

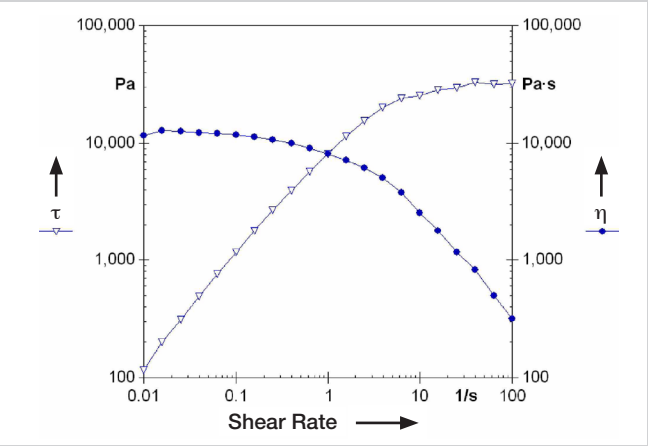

Fig. 2: The gap empties due to high shear speed

In such cases the shear rate is too high or the measuring point interval at high shear rates is too long. To solve this problem, you can change the measuring profile and shorten the measuring point interval at high shear rates (e.g. 10 ms with the MCR air-bearing rheometers). This means the test duration is so short that the measurement is over before the sample escapes from the gap.

Tips for measurements in a low shear rate range

For material characterization at low load or, for example, to determine the yield point, it is sometimes necessary to exploit the low speed range as much as possible. To produce the smallest shear rates you can use a larger cone angle or a larger gap between the plates. At low shear rates it is important to follow the rule of thumb for the measuring point interval: The measuring point interval must be at least 1/shear rate.

Tips for measurements on reactive samples

With curing reactions the sample may change strongly during the measurement. Often the sample begins as a thin fluid and ends the reaction as hard as a rock. Oscillatory tests have shown to be very useful in these cases as the sample is put under less stress by the very small oscillation movements compared to the case of rotational tests. For this reason, the gap is less likely to empty in an oscillatory test than in a rotational test.

To investigate the whole reaction time it is often advisable to set a linear or logarithmic deformation ramp instead of constant set values. This allows you to put the sample under larger strain when it is a thin fluid. With increasing curing time the strain is continually decreased. The measurement therefore hardly disturbs or influences the curing. The measuring values for each measuring point are within the limits of the measuring range.

Tips for measurements in a large frequency range

The frequency range of the rheometer is limited to max. 100 Hz. The principle of the time/temperature shift tells us that time and temperature have the same effect on rheological properties. Cooling a substance has the same effect as putting it under a short period of stress (in other words under a high frequency). The result: the substance becomes stiff. However, this is only true for thermo-rheologically simple substances, i.e. for substances which do not fundamentally change their structural character within the observed temperature range.

If you want to evaluate the behavior of your sample at higher frequencies, carry out frequency sweeps at low temperatures. You can then use the results to calculate values at a reference temperature. This gives you a master curve which has a considerably wider frequency range.