TA Instruments - Temperature Calibration: Span and Offset using a Standard Oil

November 1, 2000

Necessary Steps:

1. Standard oil (Covering the temperature range of user's interest, along with **c0**, **c1**, **c2** and Vn parameters from the oil test certificate, including viscosity-temperature coefficients[1])
2. Geometry (Parallel Plate – 40mm, used here for demonstration)
3. Procedure Settings (Shown below)
4. Span and Offset Calculation (Described below)

After obtaining the proper standard oil, including a Test Report along with the viscosity-temperature coefficients, the geometry must be attached, calibrated and the geometry gap zeroed. The values of the Span and Offset should be set to 1 and 0, respectively, before the following calibration procedure begins. This is found in the 'Options', 'Instrument' window under 'Miscellaneous' tab. If the values are other than previously stated, the values then must be changed in the 'Temperature Calibration' section in the appropriate row for the specified Temperature System (Figure 1).

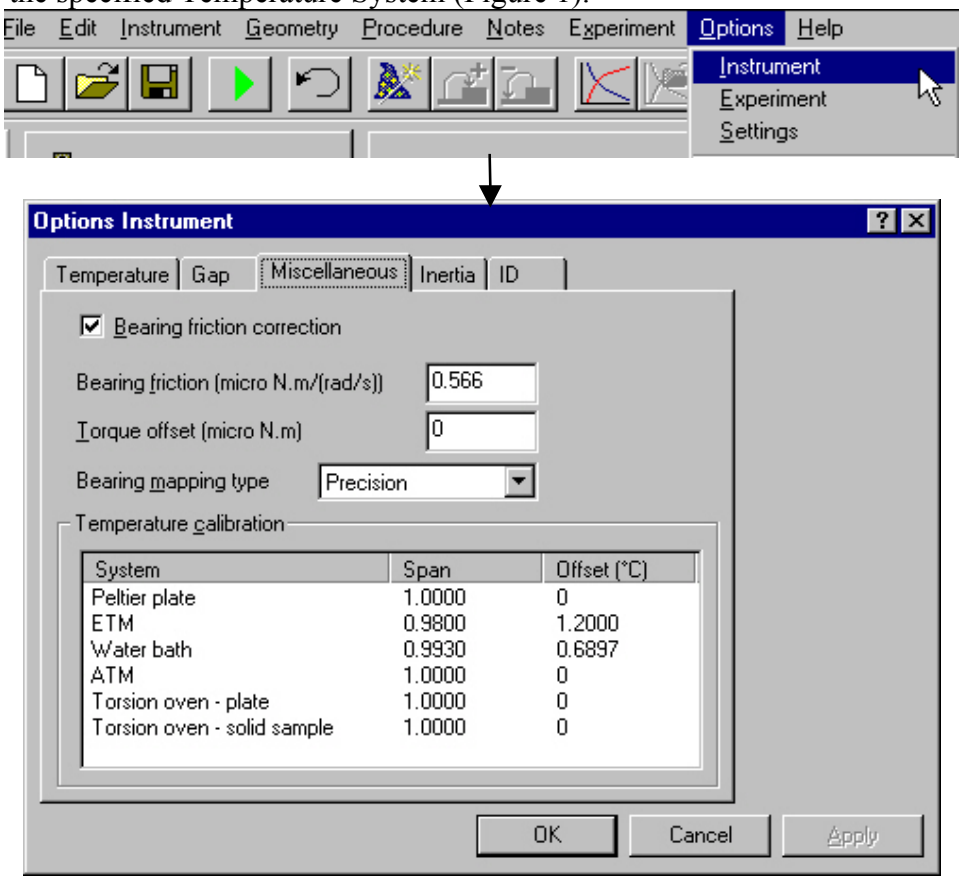

Figure 1

Procedure Settings

The procedure is another method in determining the Span and Offset values used for calibrating the temperature when using different bottom assemblies on the AR Series Rheometers. The method for calculating these values will use the Flow Mode, Peak Hold step. In order to set up this test parameter, first choose 'Procedure' from the Main Toolbar, then select 'New', then 'Flow' (Figure 2).

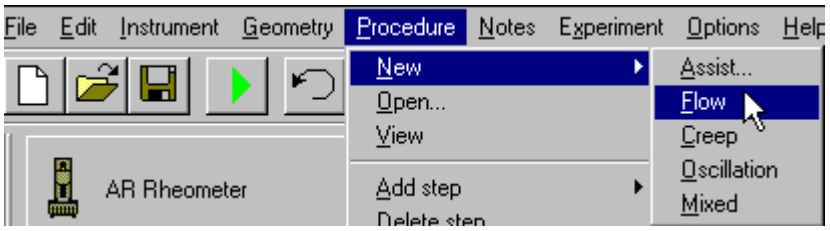

Figure 2

In the 'Conditioning Step: Settings', type in the temperature value of interest when running the standard oil in the 'Initial Temperature' and check the box 'Wait for correct temperature'. Then check the box under 'Equilibration: Perform equilibration' for a duration of 2 minutes. Uncheck all other settings (Figure 3).

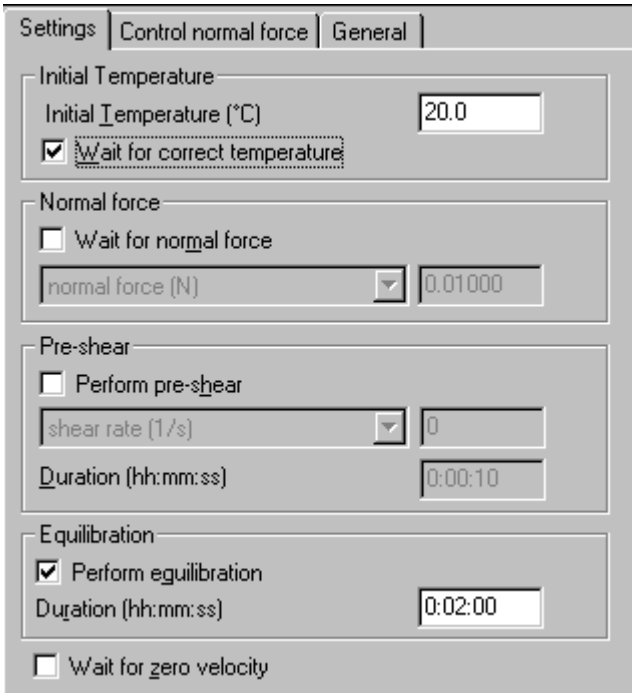

Figure 3

Now highlight the 'Steady State Flow step', which is the default step in a Flow Procedure and choose the 'Peak hold' in the Test Type section, using the dropdown window (Figure 4).

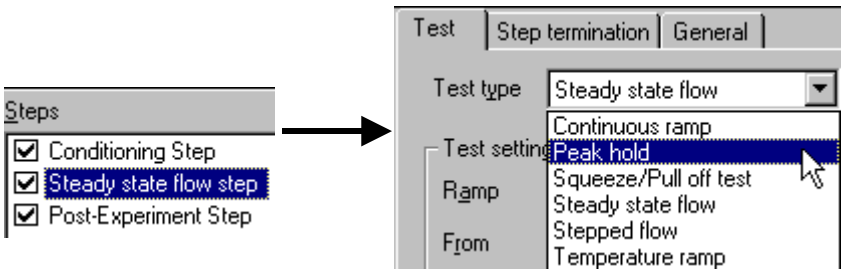

Figure 4

Change the 'Test settings' in the new 'Peak hold step' window as follows (Figure 5): make the 'Hold' control variable 'shear rate', set 'at' to a value of 0.10 1/s for medium viscosity fluids (this value should be suitable, but may be changed accordingly to measure a higher or lower viscosity value at the temperature of interest) and set 'for' to a time of 1 minute. The 'Sampling' should be set to 'Sampling points' of 10. The 'Other settings: Temperature' should be set to the temperature of interest, same as the temperature used in the 'Conditioning step' (20^{o}C is used as an example). NOTE: The 'Initial Temperature' in the 'Conditioning Step' should be same value used in the 'Peak hold step'.

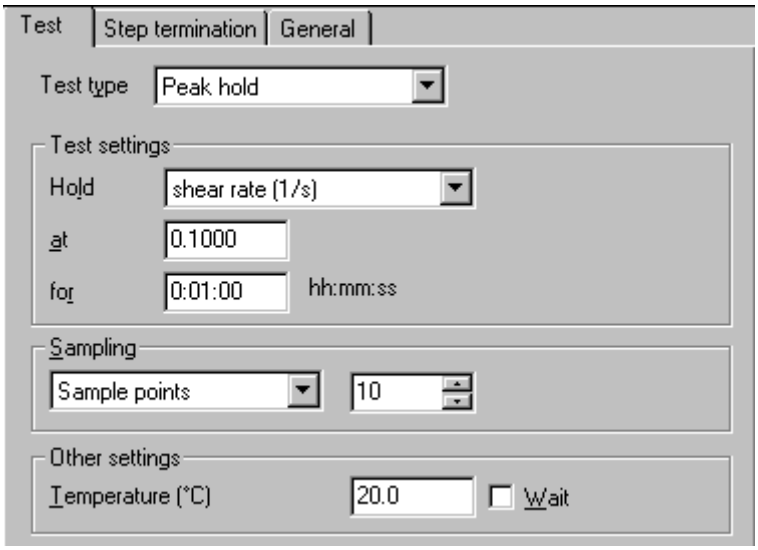

Figure 5

The 'Post-Experiment' step does not need to be adjusted. After creating the proper filename in the 'Notes' page and finish loading the sample to 1000 µm, the test can be started.

Viscosity Values

After collecting the viscosity information from the prior tests (two different viscosities values at their different temperatures are sufficient, but more values at the different temperatures are recommended), the information must be entered into a spreadsheet for

Span and ***Offset*** determination. The next section will describe how to calculate these values.

Span and Offset Method Using Viscosity

Using the Certified Values from the standard oil used[1], the following viscosity-temperature equation is used to interpolate the viscosity data in cP or mPa·s at temperatures (T), in °C, in order to calculate the span and offset parameters for the instrument:

$$\ln \ln (\text{viscosity}+0.7) = \mathbf{c0} + \mathbf{c1}*\ln(T+273) + \mathbf{c2}*[\ln[T+273]]^2 \quad \text{.....(eq.1)}$$

By manipulation of this equation and the viscosity information at the tested temperature, the use of the equation 2, the calculated Temperature, compared to the set temperature during the experiment, can be plotted to generate Figure 6. The values needed for calculation for the Span and Offset from the Temperature Calibration generated figure is the slope and the intercept. These calculations are found in equations 3 and 4.

$$Temp_{calculated} = \left\langle \exp \frac{\left(-c1\right) - \left(\left(c1^2\right) - 4(c2)\left((c0) - \left(\ln\left(\ln\left(vis\cos ity_{measured} + 0.7\right)\right)\right)\right)^{0.5}\right)}{2(c2)} \right\rangle - 273 \quad \text{.....(eq.2)}$$

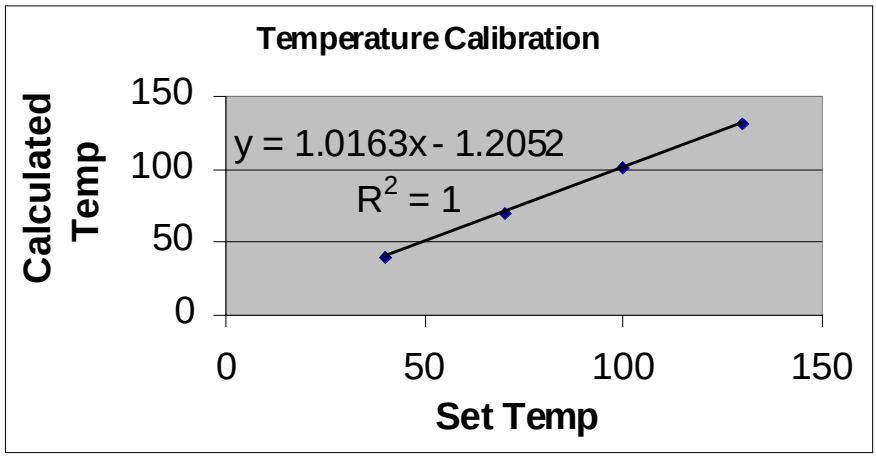

Figure 6

From the slope and the y-intercept, the span and offset values are calculated as:

$$\text{Span} = 1/\text{slope} \quad \text{.....(eq.3)}$$

$$\text{Offset} = -\ \text{intercept/slope} \quad \text{.....(eq.4)}$$

These values then must be added into the Rheology Instrument Software described in Figure 1.

[1] Cannon Instrument Company, Cannon Certified Viscosity Standard - N450000, Test report