Tempspin: A new TopSpin tool to automatically compensate frictional heating

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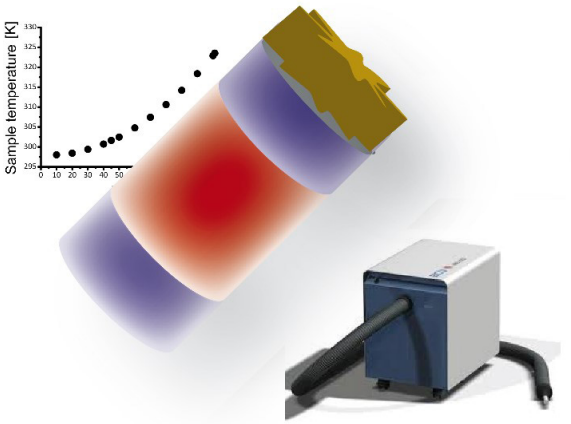
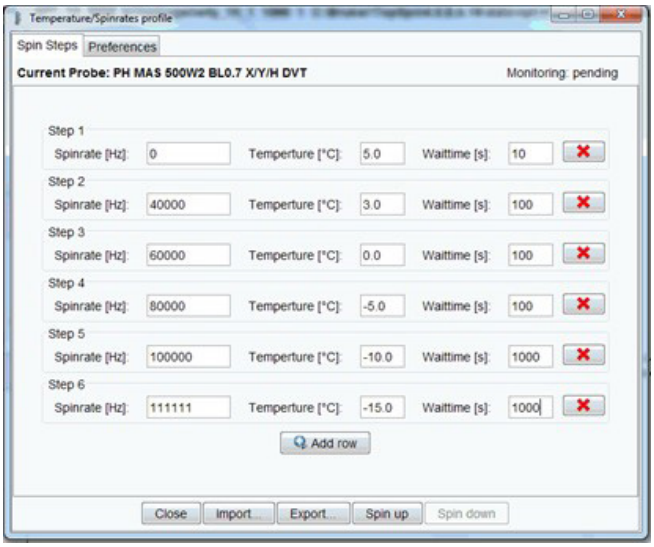


When Magic Angle Sample spinning is employed, the friction between the rotor-wall and the surrounding air causes heating of the NMR-Sample. A variety of samples must be kept within a defined temperature range. Biological samples, for instance, must be kept between 0° C and 37° C, mostly. Therefore, the frictional heating must be compensated. Furthermore, this compensation cannot be performed in a single step because this would undercool the sample. By contrast, the temperature of the VT (variable temperature) gas-stream must be adjusted in a stepwise procedure while changing the spinning speed. This can be a lengthy and inconvenient procedure.

In order to automate this process, we provide a TopSpin tool (from TopSpin 4.1.0 on) called "tempspin". By using this tool, a stepwise change of VT gas temperature according to the spinning speed can be predefined for an unlimited number of spinning speed steps, as shown in Figure 1. The resulting table acts like a temperature profile, adjusting the VT gas temperature for optimal heat compensation.

In order to generate the values for this temperature profile, a calibration using a sample acting as NMR-thermometer is recommended. For the 0.7mm standard and wide-bore probes, a detailed calibration is described in the application note "Quantification of frictional heating at ultra-fast magic angle spinning".

Figure 1



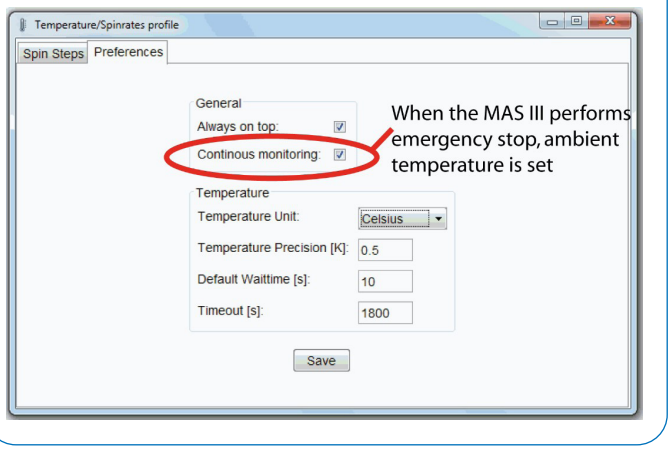
Editable temperature profile. In the left column the spin rate is set, the middle column sets the VT-gas temperature and in the right column a waiting time for temperature calibration is editable. The programmed temperature profile can be saved using the "Export" button and can be reimported by using the "Import" button.

According to the programmed profile (Figure 1), the temperature is adjusted prior to the spinning rate while spinning up and is adjusted after the spin rate while spinning down. Therefore, the sample will never be overheated.

Besides using "tempspin" right from the start, the tool can also be used when the sample is already spinning. In this case, when starting the "Spin up" process, the next higher spin-rate step will be taken. Hereby, first the VT gas temperature of this step will be set and after temperature equilibrium is reached, the spinning speed will be raised.

Vice versa, if the "Spin down" process is started, the next lower spinning speed is set and after this is reached safely, the VT gas temperature is adjusted. If the process of spinning up or down should be stopped for any reason, this can be done by pressing the "cancel" button right under the progress bar. The process stops immediately, no new commands are submitted to the MAS III or the BSMS. However, the last command submitted to the BSMS or the MAS III, before cancelling, will still be executed by these devices.

Figure 2



Options for running the program. The unit of temperature can be adjusted to Celsius, Kelvin or Fahrenheit. A Temperature precision can be set. The process in only continued, if the probe-temperature is within limits of this precision. The timeout defines the duration of trying to reach the specified parameters after which the process is set on halt. Continuous monitoring provides the safety feature, that after a stop is executed, the VT-gas temperature is automatically set to the value corresponding to 0 Hz spin rate (Figure 1).

Further, "tempspin" contains the safety feature "Continuous monitoring" (Figure 2) which automatically sets the VT-gas temperature to the value given at 0 Hz spinning speed (see Figure 1, in this case 5° C) if a stop is performed. This function is intended to prevent the sample from being undercooled, i.e., preventing water containing samples from freezing, which could destroy the sample and the rotor. An emergency stop (error stop) is performed by the MAS unit to protect the probe-head in case of any problem. These kinds of stops are often induces in absence of the spectroscopist.

Detailed procedure performed by the tool while "spin up" or "spin down".

After filling the table shown in Figure 1 and starting the "spin up" process, first the "probe temperature" value in TopSpin is set to the value corresponding to the next spin rate step. When this value is reached within the precision, as set in the preferences tab (Figure 2), a default waiting time of 10 seconds starts. During this time, the probe temperature must not violate the temperature precision, otherwise the 10 seconds will start again. After the default waiting time, the equilibration time starts, as shown in Figure 1, right column. After the equilibration time ended, the MAS III sets the corresponding spin rate value. This procedure is repeated until the last spin rate step. If the process of "spin down" is started, the spin rate is set first, thereafter the corresponding VT-gas temperature is set, including waiting time for equilibration. The waiting time for equilibration can be edited for every spin rate step individually as shown in Figure 1, right. The programmed temperature profile can be saved by using the "Export" button shown in Figure 1. A previously saved profile can be reimported by using the "Import" button.

Short summary:

The TopSpin tool "tempspin" automatically adjusts the sample temperature to the spinning speed keeping the sample in the desired temperature range. An additional safety feature sets the VT gas to ambient temperature if an emergency stop happens.

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