AutoCalibrate

User Manual

Version 003

Innovation with Integrity

NMR

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1 Overview

At the time of installation, a qualified Bruker engineer measured or calibrated many parameters that were used to test the NMR spectrometer. Over time these parameters deviate from their optimal values, due mostly to natural system drift. AutoCalibrate is designed to automatically test the main parameters which drift and contribute to poor data quality. AutoCalibrate is configured and controlled through IconNMR automation software package which uses TopSpin to acquire and evaluate data. Upon completion of the AutoCalibrate tests, a PDF report is generated summarizing the results obtained. Though there are some alerts which will be sent through to IconNMR, it is recommended to view the AutoCalibrate Report at the completion of each run.

AutoCalibrate will check the 1H p90 pulse length, 13C p90 pulse length, temperature calibration and 3D shims. AutoCalibrate will perform these tests on a single optimal sample, the AutoCalibrate Sample. In using the same sample fixed in a spinner for these tests, AutoCalibrate can test not only for the parameters listed above, but also include integral integrity/concentration calibration, which ensures the entire system is running optimally.

2 AutoCalibrate-Configuration

AutoCalibrate is designed to automatically run several tests on a regular (daily) basis to establish each system's optimal performance level and to catch early deviations from this optimal performance level. AutoCalibrate has no user set parameters of its own, so configuration entails only scheduling and priority optimization. AutoCalibrate requires that the same sample always be used. In the configuration screen, users can identify the position of this sample as well as the barcode of the sample, thus ensuring consistent results.

AutoCalibrate scheduling is accessed through the IconNMR Configuration window (command **iconc** from the TopSpin command line). In the pane to the left, select **AutoCalibrate** from the options under Automation. From this window (see figure below), the NMRSuperUser is able to (1) activate, suspend, or deactivate AutoCalibrate, (2) schedule AutoCalibrate and (3) set priority for AutoCalibrate.

AutoCalibrate must finish before any other samples queued in IconNMR can be acquired. The entire program runs in approximately 1 hour, depending on the field strength and probe-type of the system. The user has some control over the IconNMR AutoCalibrate start time when samples are queued and actively being acquired.

It is possible to queue the AutoCalibrate at any time as NMRSuperUser. Once AutoCalibrate starts, it can be stopped through the Tools pull-down menu, however there are only a few positions in which it can stop. As a result there may be a long lag between a request to stop and the actual stop.

AutoCalibrate requires certain settings be possible, or it will fail with error:

- AutoCalibrate requires a special sample that needs to be ordered directly from Bruker, along with the license. This sample has a barcode that will need to be entered into IconNMR configuration. Part number for this sample is Z171901. In case the sample breaks or in any other way is no longer usable, this is the part number for replacement. It does not include the license. Bundle of license + sample is AH1510/AH1510A.
  - Barcode (found on sample CAP) must be entered or AutoCalibrate will Error.
- Temperature set to 298K if possible. If not, higher temperature values are selected until 308K is reached. If system cannot stabilize to 308K or lower, AutoCalibrate will fail.
- Pulses must be within range of those entered in the prosol table
  - 1H cannot deviate by more than 10% or AutoCalibrate will Error
  - 13C cannot deviate by more than 20% or AutoCalibrate will Error
  - Quantification cannot deviate by more than 25% or AutoCalibrate will Error

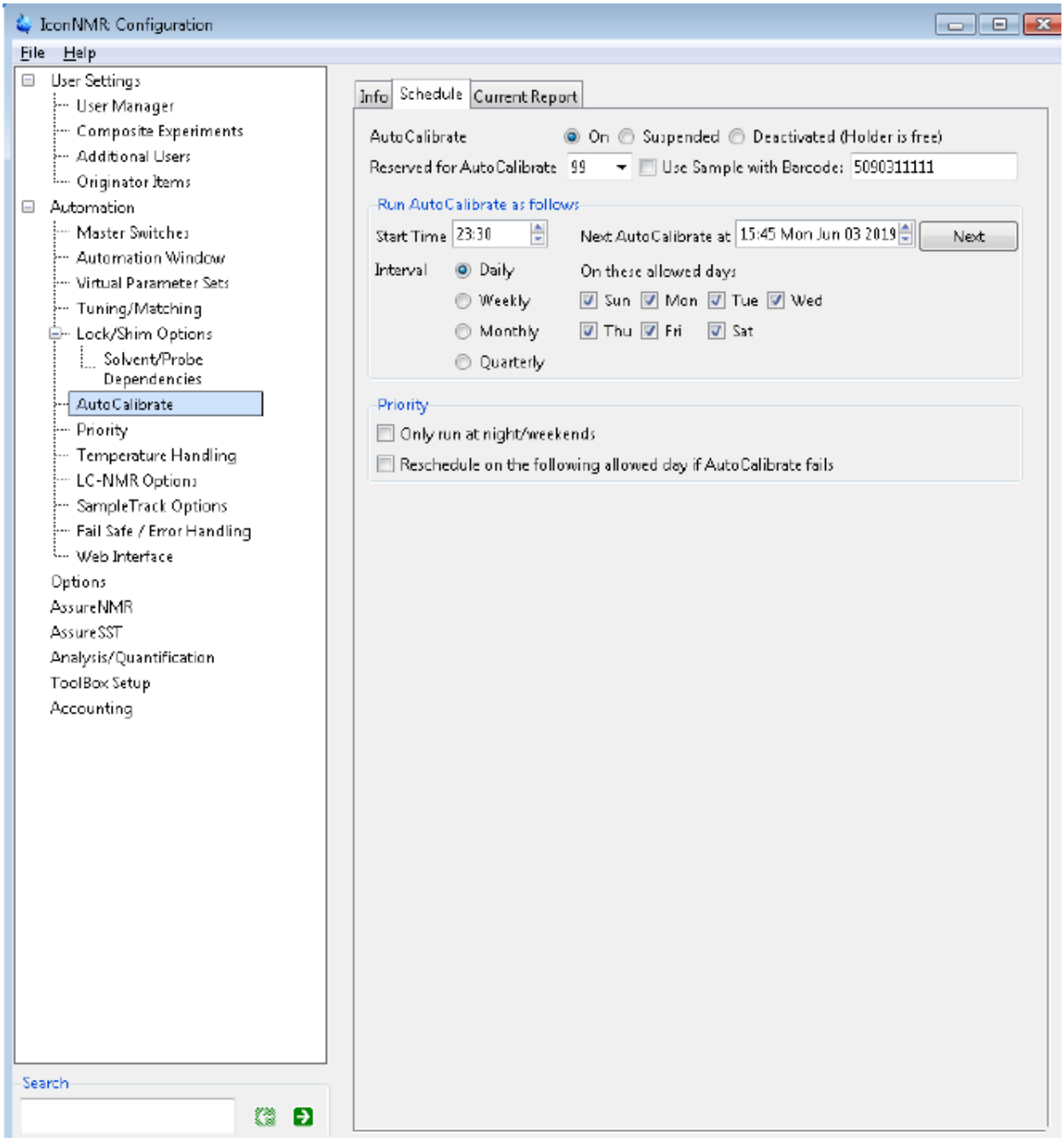


Figure 2.1: The AutoCalibrate Schedule Tab in the Calibration section of Automation in the IconNMR Configuration Window.

To activate AutoCalibrate, click the **On** radial button of the Schedule tab, select a holder position for the AutoCalibrate Samples, and enter the barcode of the specific sample that will be used. As mentioned previously, AutoCalibrate requires that the same sample always be used. If the sample is broken, please contact Bruker for replacement. The sample barcode can be found on the sample cap. It is a 10 digit number plus 1 digit checksum for the changer.

AutoCalibrate is automatically queued for acquisition as required to meet the time set in the entry for Start Time. Note the times use a 24-hour format. If other samples are queued at the time when AutoCalibrate is scheduled to run, then AutoCalibrate will delay until there is time available.

3 AutoCalibrate-Priority

As AutoCalibrate is a scheduled maintenance action, it needs to have some priority to run on a regular basis. The default operation mode for AutoCalibrate is to run as close to the start time as possible. If the start time is reached and another sample is running, the AutoCalibrate program will wait for the current sample to finish the current experiment. It will then insert itself into the queue to run. AutoCalibrate takes approximately 1 hour to complete and there are only limited possibilities to stop the routine. Once AutoCalibrate is finished, the samples waiting in the queue will run.

The run-time interface was designed with user flexibility in mind. Users can set to run Daily, on the days selected. Note that the NextAutoCalibrate field will update as you change the preferences to reflect current settings. The options to run weekly, monthly, or quarterly also require a day to be selected. The run will start on the next selected day if the week, month or quarter and will only run once per week, month, or quarter.

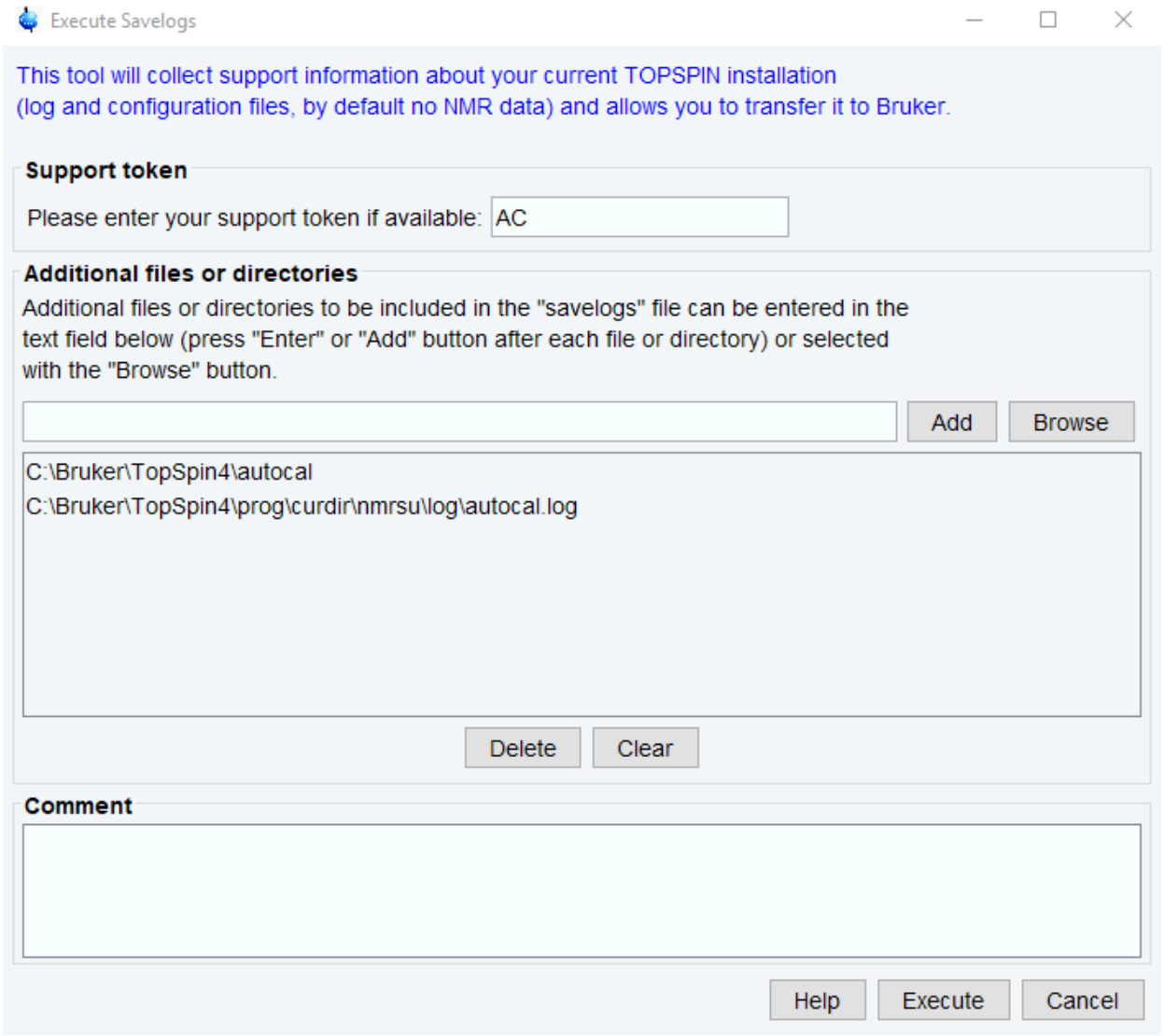
If the option to only run at night/weekends is selected, then the AutoCalibrate start time will reflect a time that is as close to the desired start time as possible, while also falling within the night/weekends time set up in the Priority section of the IconNMR Configuration manual. If the sample currently running will take the entire night/weekend period, the AutoCalibrate start time will begin on the next scheduled day.

To disable AutoCalibrate temporarily, select the 2nd radial button at the top of the Schedule tab, labeled **Suspended**. This will stop any further AutoCalibrate runs from being queued (if there is already an AutoCalibrate running, it will continue until finished.) This feature is meant as a temporary stop. If the user plans to discontinue AutoCalibrate for a significant time period, the **Deactivated** (Holder is free) option should be used.

To stop the AutoCalibrate routine, from the Tools pull-down menu of IconNMR select Sop AutoCalibrate. There are only a few positions in which AutoCalibrate will be able to get the stop command from IconNMR. If these are missed, the routine will have to complete.

4 AutoCalibrate-log files

Data can be found in the *<TSHome>\autocal\db* folder. A running log is kept of autocalibrate. This can be found in *<TSHome>\ prog\curdir\<user>\log\autocal.log*. To send data to Bruker for support, please compress the */db* directory into a zip file, add the *autcal.log* file and send when requested. An easy method of doing this is through the **savelogs** command. Please type **savelogs** at the TopSpin command line, then fill in savelogs GUI as shown below. Please make sure to add the 2 additioanl selections by clicking on Browse and finding first the log file located in *<TSHome>/prog/curdir/<user>/log/autocal.log* and the autocal directory found at *<TSHome>/autocal*. After each is located, click the tab **Add** to populate the lower window. Once the form is completed, click on **Execute** at the bottom of the window. It is important that at the very end you make sure to click **Send** to initiate the actual transfer of the file.



5 AutoCalibrate Values and Ranges

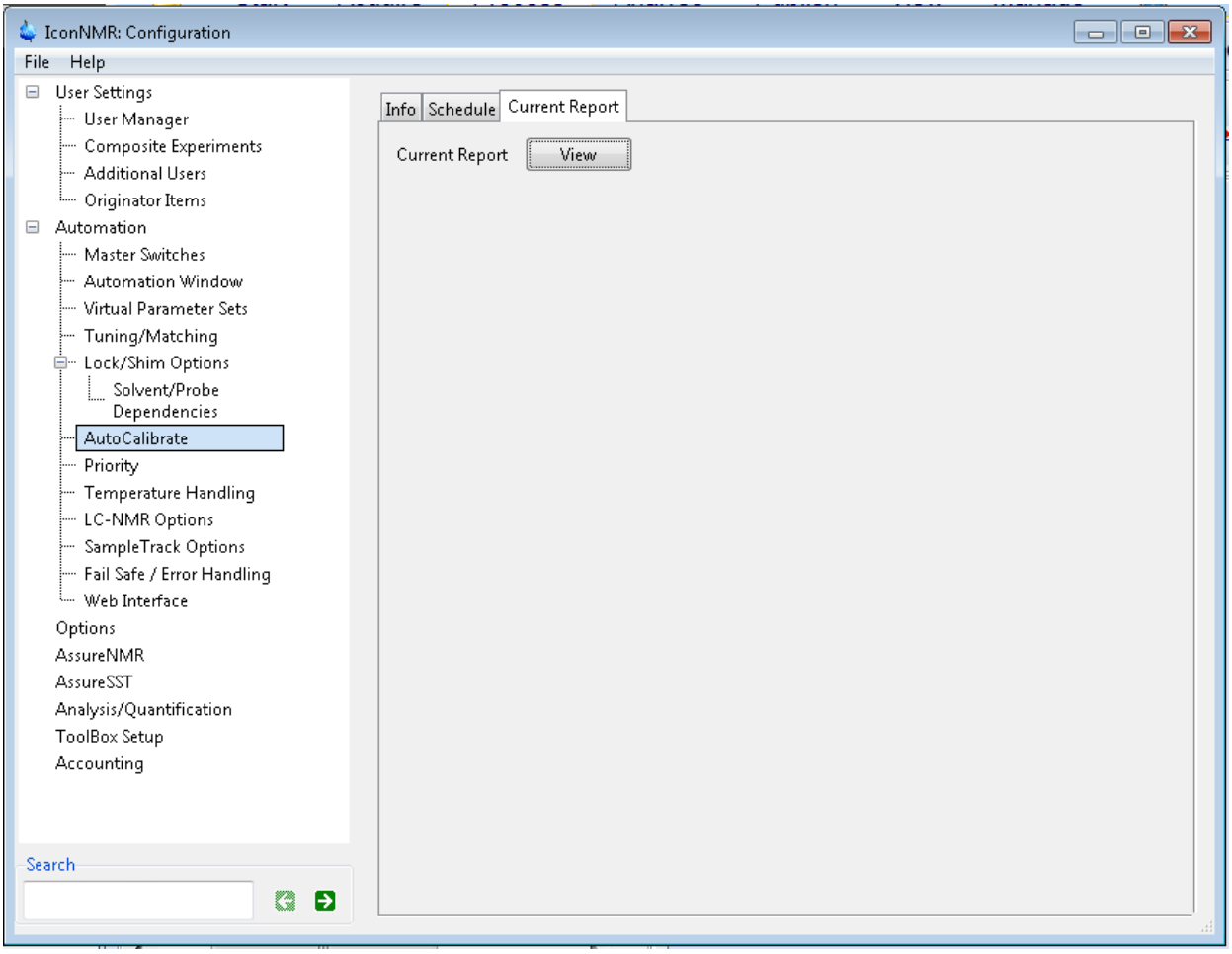
AutoCalibrate is running many tests behind the scenes as it completes its run. AutoCalibrate is designed to look at current values for each test and run a diagnostic test based on allowed deviation from the current setting. These limits for deviation are hard coded into the program and cannot be changed. They were selected based on knowledge of 'normal' deviation from natural spectrometer drift as well as understanding of the impact large deviations have on good data. As such the following decisions are made by AutoCalibrate:

Test	Deviation	Action
1H pulse	5% - 10%	Update prosol table with the new pulse value
	>10%	Error
13C pulse	>20%	Warning - please check pulse with appropriate 13C sample.

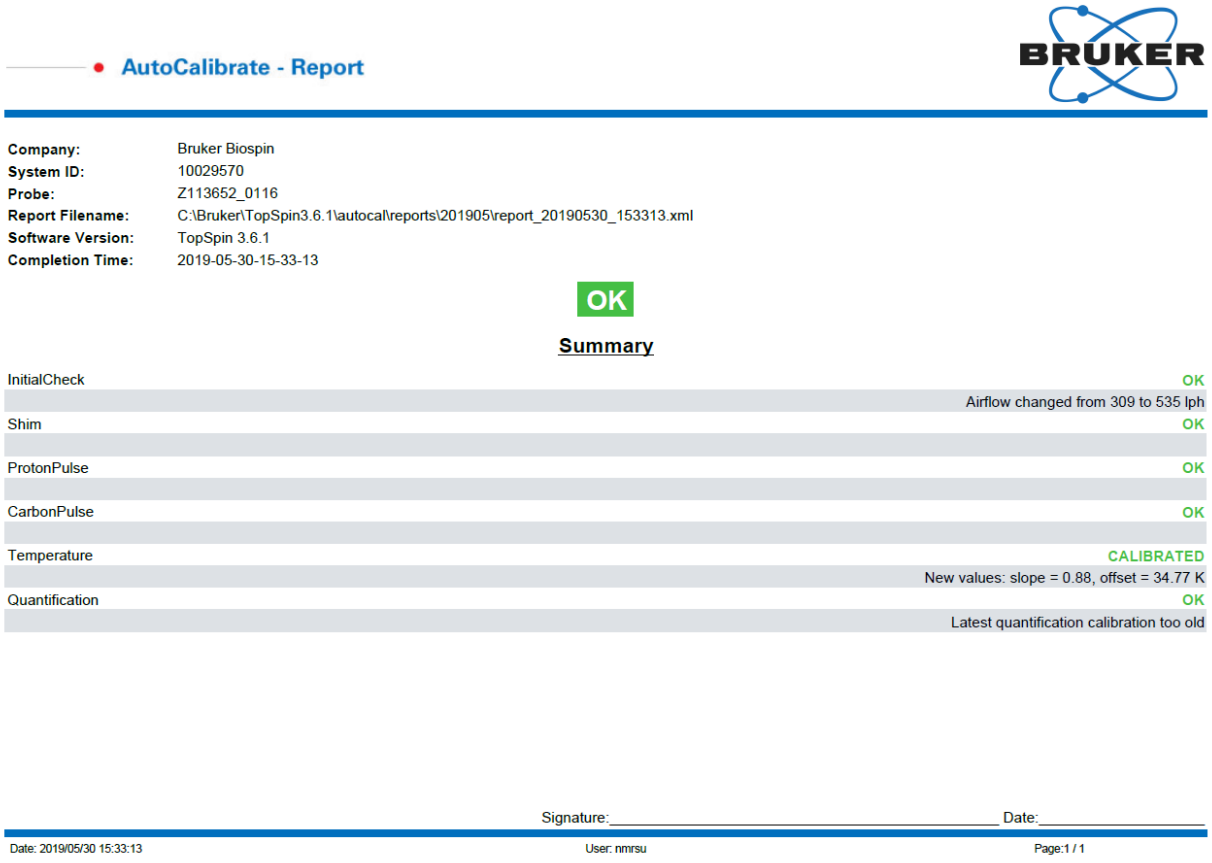
ERETIC	>25%	Warning - ERETIC2 value should be checked and updated.
Temperature	>308K	Error
Shim	W ½ >2Hz	Error

6 AutoCalibrate Reports

Repots for AutoCalibrate are created at the end of each run. They are stored with the other AutoCalibrate data in *<TSHome>/autocal/reports* directory. It is important to note that the only errors shown in the IconNMR Remarks field in the Automation window are errors resulting form an IconNMR failure, not the failure of a test with in AutoCalibrate. The only way to see if a test passes or fails is to view the report. The most current reports can be viewed from the IconNMR Configuration window, under **AutoCalibrate** in the Current Report tab (see figure below).



An example of a report is shown below in figure.



AutoCalibrate - Report

Company:	Bruker Biospin
System ID:	10029570
Probe:	Z113652_0116
Report Filename:	C:\Bruker\TopSpin3.6.1\autocal\reports\201905\report_20190530_153313.xml
Software Version:	TopSpin 3.6.1
Completion Time:	2019-05-30-15-33-13

OK

Summary

InitialCheck	OK
	Airflow changed from 309 to 535 lph
Shim	OK
ProtonPulse	OK
CarbonPulse	OK
Temperature	CALIBRATED
	New values: slope = 0.88, offset = 34.77 K
Quantification	OK
	Latest quantification calibration too old

Signature:_____ Date:_____

Date: 2019/05/30 15:33:13 User: nmrsu Page:1 / 1

7 Contact

Manufacturer

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Germany

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http://www.bruker.com
WEEE DE43181702

Bruker BioSpin Hotlines

Contact our Bruker BioSpin service centers.

Bruker BioSpin provides dedicated hotlines and service centers, so that our specialists can respond as quickly as possible to all your service requests, applications questions, software or technical needs.

Please select the service center or hotline you wish to contact from our list available at:

https://www.bruker.com/service/information-communication/helpdesk.html

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