FoodScreener

- Wine-Profiling Module

User Manual

Version 001

Innovation with Integrity

NMR

Document Number: 10000057289

P/N: H161720

Contents

Contents

1 Introduction

With the Wine-Profiling™ module for the FoodScreener™ platform, Bruker provides a standardized analysis method for authenticity and quality control of wine samples. Standardized Operating Procedures (SOPs) for sample preparation and measurement guarantee highly reliable results. After the acquisition, data analysis is performed by Bruker's data analysis server applying the most recent version of wine profiling analysis.

This manual includes:

- Recommended procedures for quality control.
- Instructions on how to measure a single wine sample.
- Information on software services and programs.

Introduction

2 Recommended Quality Control Procedures

It is recommended to validate the overall performance of the Wine-Profiling method on a regular basis. This includes the performance of the NMR-spectrometer (e.g. temperature, shimming, solvent suppression), the preparation and the measurement.

2.1 Regular Quality Control of the FoodScreener System

In this chapter, the regular quality control of the FoodScreener system is described. It is recommended to carry out these validations at least once per week.

2.1.1 Sucrose Sample

The sucrose sample is used to access information about the performance of the water suppression, the resolution, the signal to noise (S/N) ratio, the half width of the DSS signal, (second experiment: SUC_ZGPR_8) and the probe (first experiment: PROF_PROFIL).

For the sucrose measurement, SampleTrack™ provides a work step template with the analysis experiment Sucrose. For the acquisition, the PROF_PROFIL parameter set and the SUC_ZGPR_8 parameter set will be used. To create an order for the sucrose sample, select in the Sample Orders view: **File** | **New** | **Profiler Setup Form**. This starts the general guide for creating samples:

- Select **Enter Samples in Batch Mode**.
- Enter a Project Name, e.g. References.
- Select **Do Not Use Source Container Information**.
- Enter a Sample-ID at position 1, e.g. Sucrose-[DATE].
- Select **Use super method for all further steps**, then **Sucrose (NMR-SPECT)**.

These steps will setup the following Sample Order:

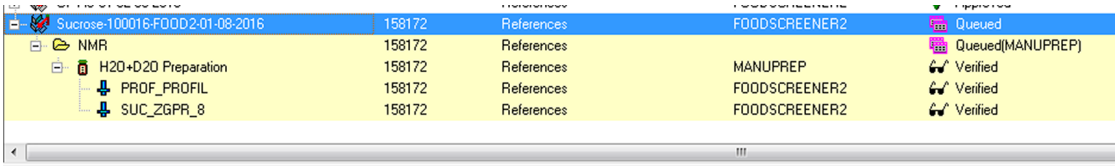


Figure 2.1: The SampleTrack Sample Order

During the manual preparation step, you have to enter the pre-existing Tube-ID with which the sucrose sample is labeled (this Tube-ID will stay the same for each measurement of this sample).

The first experiment PROF_PROFIL demonstrates whether the probe is okay or not.

If there is no *dip* inside the profile, the probe and z-gradient are okay:

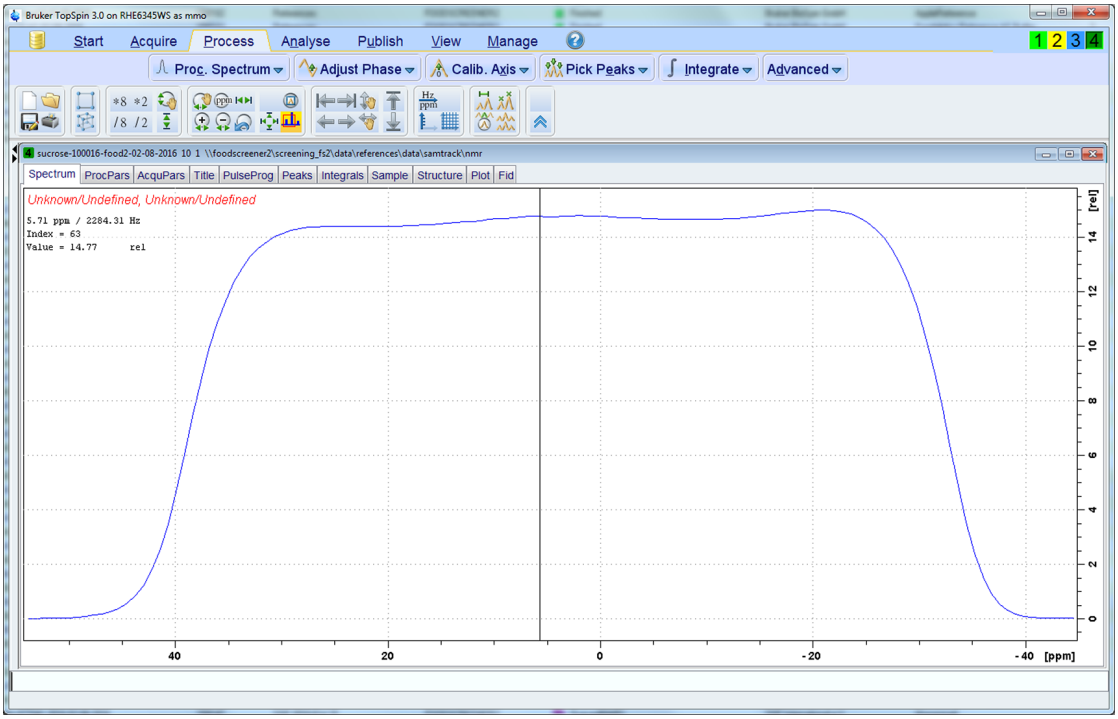


Figure 2.2: Profile Indicating the Probe and Z-Gradient are Okay.

A *dip* in the profile indicates problems, e.g. dust particles in the probe:

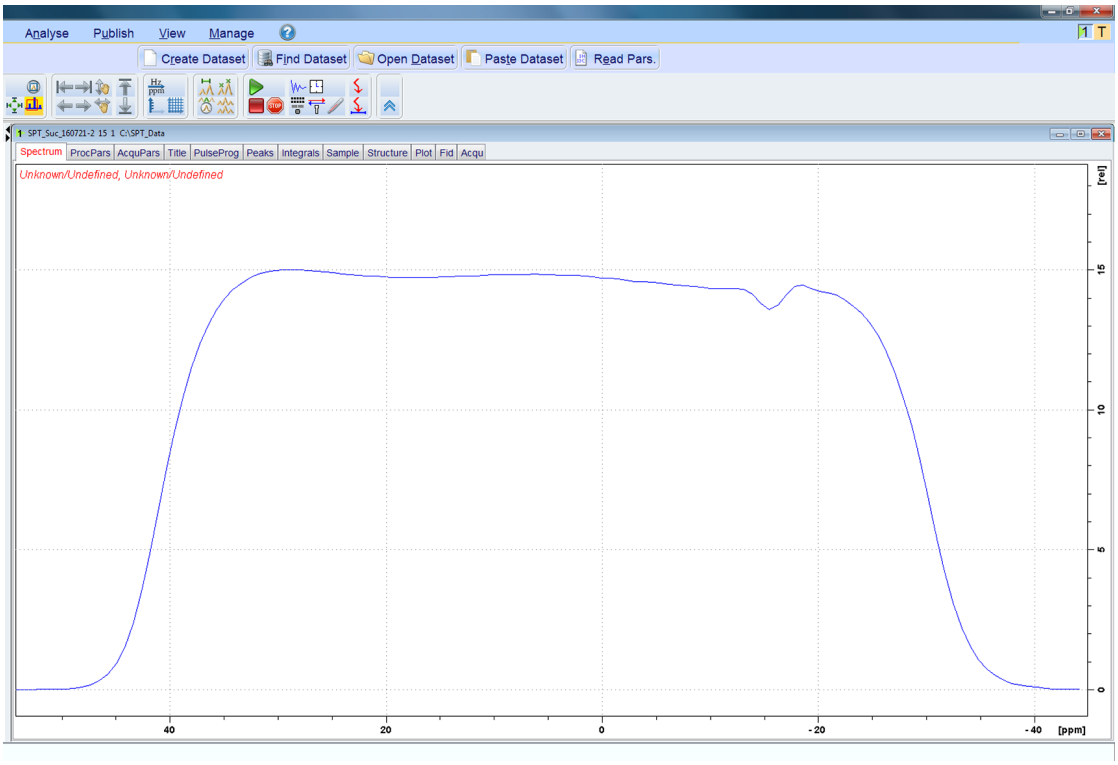


Figure 2.3: Profile Indicating There is a Problem with the Probe or Z-Gradient.

In this case:

- Take the probe out of the magnet.
- Turn it around.
- Put it back in.
- Recheck the profile.

When it is okay you can start with the measurement, if not the probe has to be send to Bruker for repair.

The results of the second experiment are displayed in the title of the spectrum:

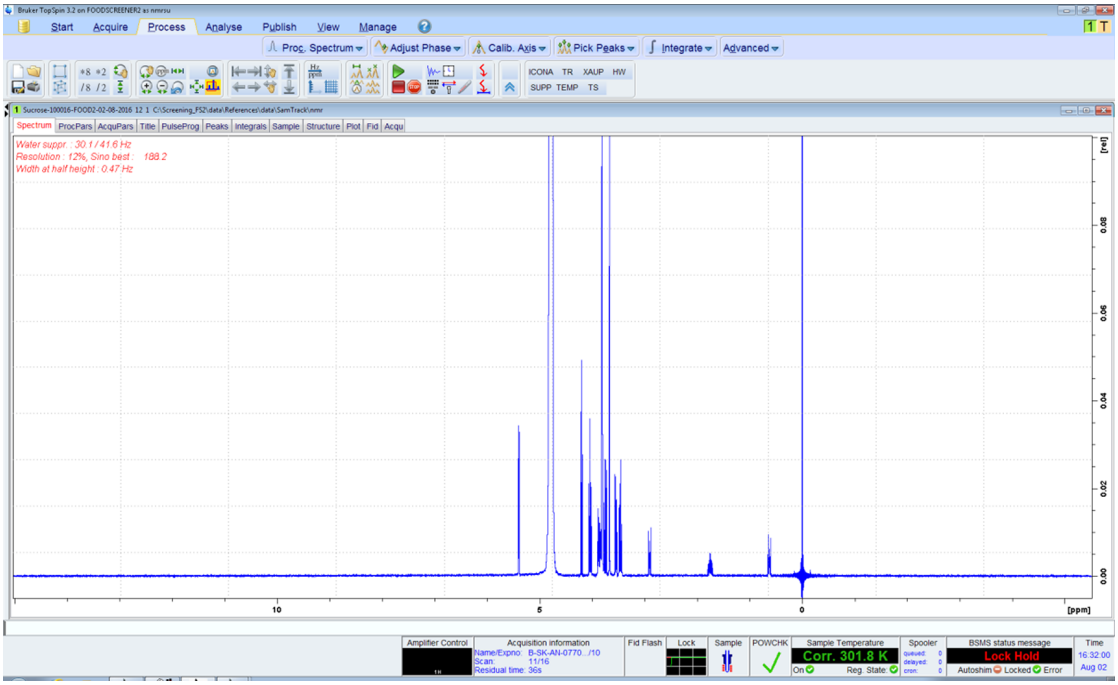


Figure 2.4: Results after the Second Experiment

The values which are reached during the installation are used as reference values. Tolerance criteria should be defined (typically 10%-20% of the reference values).

The sucrose sample is also used for 3D shimming. Refer to the TopSpin User and IconNMR User Manuals for detailed information.

Note: Never use a wine sample for 3D shimming.

2.1.2 MeOD Sample

The temperature is validated with the MeOD (99,8% methanol-d4) sample.

For this measurement, SampleTrack provides a work step template with the analysis experiment *Temp300*. For the acquisition, the PROF_WINE_TEMP parameter set is used. To create an order for the methanol-d4 sample, select in the Sample Orders view: **File** | **New** | **Profiler Setup Form**. This starts the general guide for creating samples:

- Select **Enter Samples in Batch Mode**.
- Enter a Project Name, e.g. References.
- Select **Do Not Use Source Container Information**.
- Enter a Sample-ID at position 1, e.g. MeOD-[DATE].
- Select **Use super method for all further steps**, then **Temp300 (NMR-SPECT)**.

These steps will setup the following Sample Order:

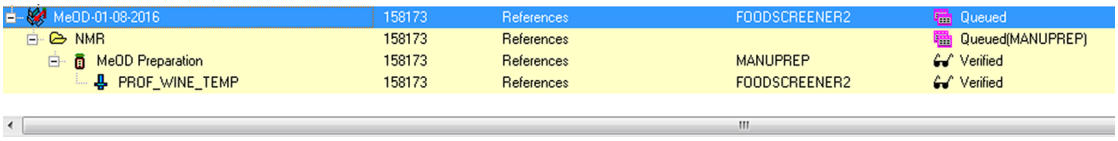


Figure 2.5: MeOD Sample Setup

During the manual preparation step, you have to enter the pre-existing Tube ID, with which the Methanol-d4-Sample is labeled (this Tube ID will stay the same for each measurement of this sample).

The temperature that is checked is the one which is used for the wine screening: 300 K. The temperature should be in the range of ± 0.1 K. The result is displayed in the title of the spectrum.

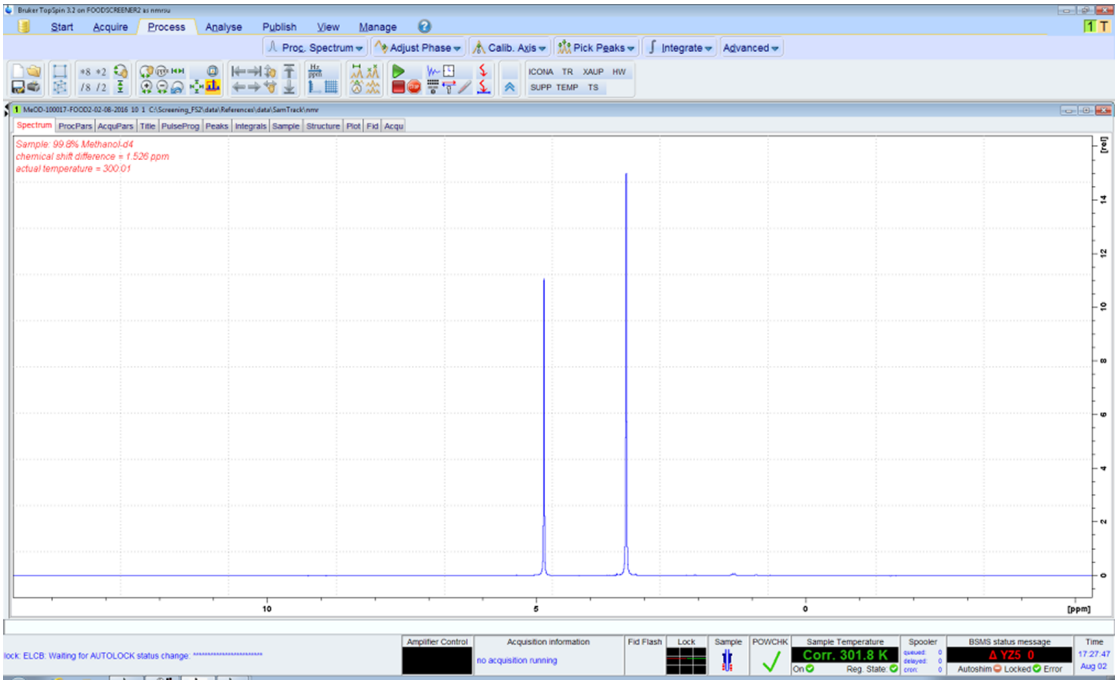


Figure 2.6: MeOD Sample Experiment Results

If the result is out of range, the temperature correction has to be changed.

2.2 Quantitative Calibration and Validation of the NMR Spectrometer

For screening, each NMR system has to be calibrated to obtain quantitative results. The intensity (integrals) of the NMR signals is proportional to the respective concentrations and the instrumental factor of this relationship (e.g. integral of 1 mol/L of one proton) has to be obtained.

This calibration has to be done after each significant change/modification of the instrument, including e.g. change of probe or amplifiers.

A validation of this calibration should be done in a reasonable timeframe, e.g. once a day. Bruker provides a Reference-Sample which will be used for both, the calibration and the validation of the system (name: QuantRefA).

The software for the management of ERETIC calibrations is provided by Bruker and is located on the SampleTrack server (Bruker ERETIC Manager, C:\Bruker\ERETICDB\).

2.2.1 ERETIC Calibration

For the calibration of the system, SampleTrack provides a work step template with the analysis experiment Quant Calibration. For the acquisition, the QUANTREF_A parameter set is used. To create an order for the calibration sample, select in the Sample Orders view: **File** | **New** | **Profiler Setup Form**. This starts the general guide for creating samples:

- Select **Enter Samples in Batch Mode**.
- Enter a Project Name, e.g. References.
- Select **Do Not Use Source Container Information**.
- Enter a Sample-ID at position 1, e.g. QuantRefA-[DATE].
- Select **Use super method for all further steps**, then **Quant Calibration (NMR-SPECT)**.

These steps will setup the following sample order:

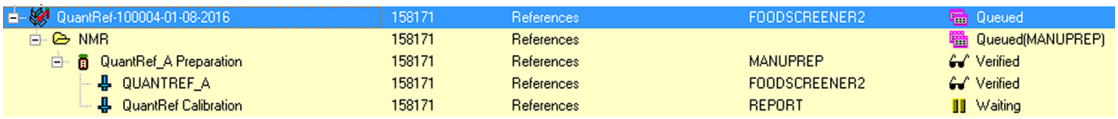
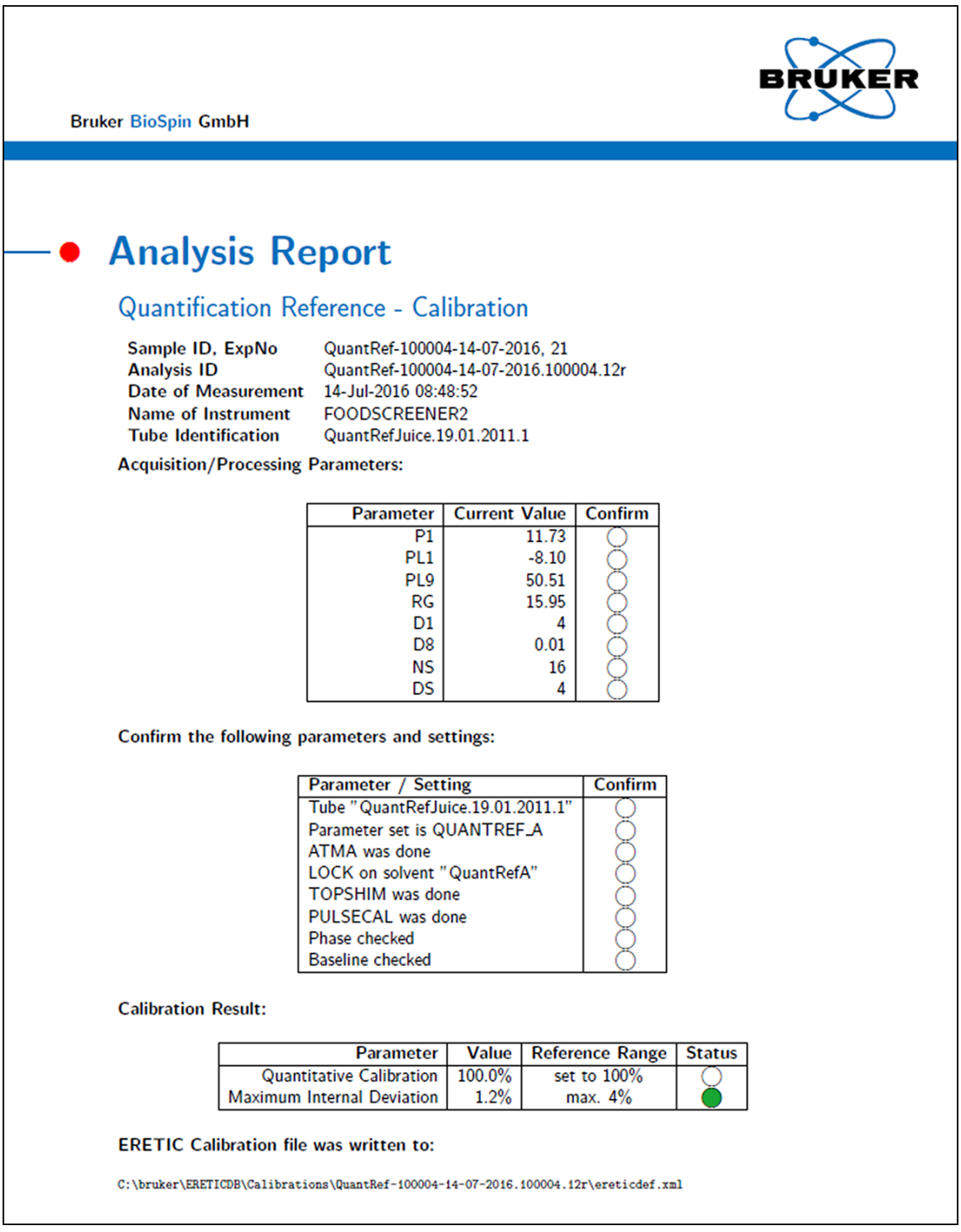


Figure 2.7: ERETIC Calibration Setup

During the manual preparation step, you have to enter the pre-existing Tube ID with which the QuantRefA sample is labeled (this Tube ID will stay the same for each measurement of this sample).

The analysis is done on the SampleTrack server and will create the calibration file *ERETICdef.xml* and a PDF document which can be used for documentation and SOP (Standard Operation Procedures) processes.



Bruker BioSpin GmbH

Analysis Report

Quantification Reference - Calibration

Sample ID, ExpNo	QuantRef-100004-14-07-2016, 21
Analysis ID	QuantRef-100004-14-07-2016.100004.12r
Date of Measurement	14-Jul-2016 08:48:52
Name of Instrument	FOODSCREENER2
Tube Identification	QuantRefJuice.19.01.2011.1

Acquisition/Processing Parameters:

Parameter	Current Value	Confirm
P1	11.73	○
PL1	-8.10	○
PL9	50.51	○
RG	15.95	○
D1	4	○
D8	0.01	○
NS	16	○
DS	4	○

Confirm the following parameters and settings:

Parameter / Setting	Confirm
Tube "QuantRefJuice.19.01.2011.1"	○
Parameter set is QUANTREF_A	○
ATMA was done	○
LOCK on solvent "QuantRefA"	○
TOPSHIM was done	○
PULSECAL was done	○
Phase checked	○
Baseline checked	○

Calibration Result:

Parameter	Value	Reference Range	Status
Quantitative Calibration	100.0%	set to 100%	○
Maximum Internal Deviation	1.2%	max. 4%	●

ERETIC Calibration file was written to:

C:\bruker\ERETICDB\Calibrations\QuantRef-100004-14-07-2016.100004.12r\ereticdef.xml

Figure 2.8: Analysis Report

Both files will be copied to the calibration directory, C:\Bruker\ERETICDB\Calibrations, with the corresponding Analysis ID (Sample ID).

Once the operator has acknowledged the protocol for sample calibration, the calibration can be added with the ERETIC Manager for further usage:

- Open the ERETIC Manager from the SampleTrack server.
- Use **Browse** to select the calibration file, than press **Add Calibration** to add the calibration file to the ERETIC Manager.

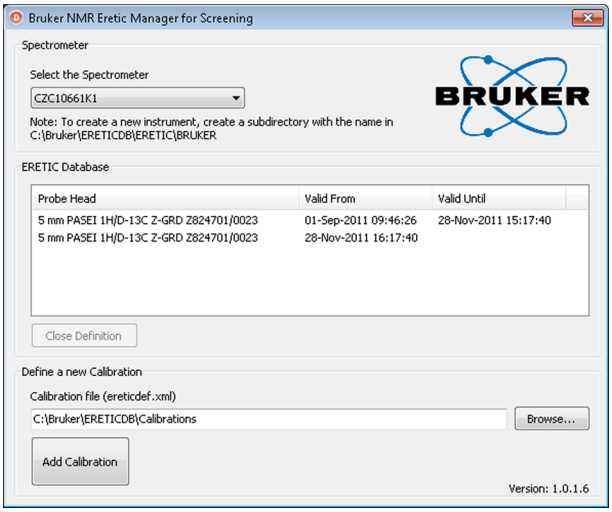


Figure 2.9: NMR ERETIC Manager for Screening

2.2.2 ERETIC Validation

Once the calibration is done the sample is used to validate the quantification. SampleTrack provides a work step template with the analysis experiment Quant Validation, which is also used for the validation of the system. The setup is the same as for the calibration.

To create an order for the validation sample, select **File** | **New** | **Profiler Setup Form** in the Sample Orders view. This starts the general guide for creating samples:

- Select **Enter Samples in Batch Mode**.
- Enter a Project Name, e.g. References.
- Select **Do Not Use Source Container Information**.
- Enter a Sample-ID at position 1, e.g. QuantReference-date.
- Select **Use super method for all further steps**, then **Quant Validation (NMR-SPECT)**.

These steps will setup the following Sample Order:

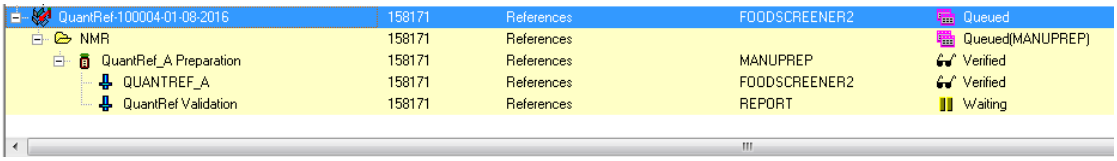
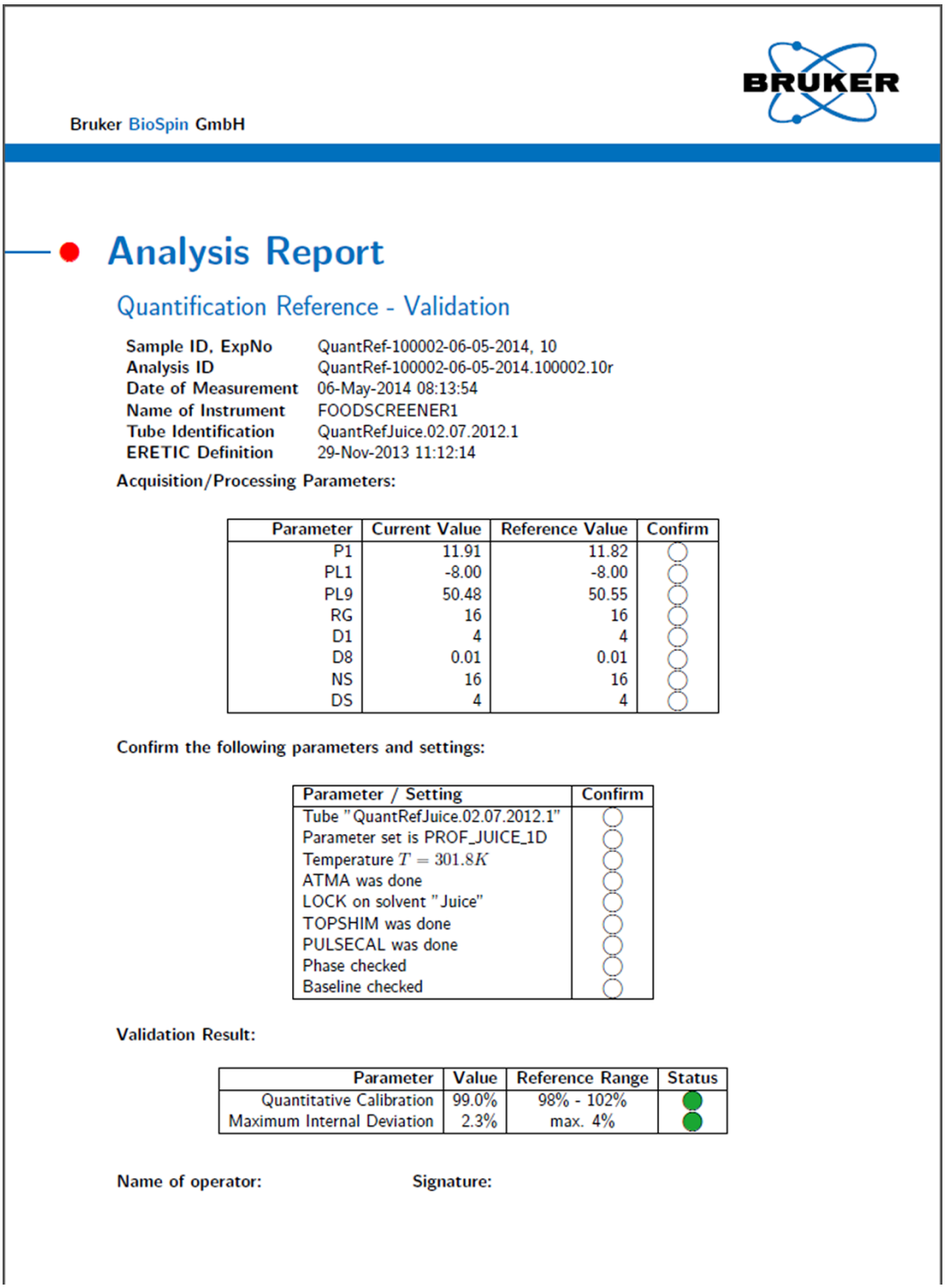


Figure 2.10: Sample Setup for ERETIC Validation

The analysis is done on the SampleTrack server and will create a PDF document which can be used for documentation.

If both quality indicators (quantitative calibration and maximum internal deviation) are inside the tolerance range, the quantitative calibration is still valid.



Bruker BioSpin GmbH

BRUKER

Analysis Report

Quantification Reference - Validation

Sample ID, ExpNo	QuantRef-100002-06-05-2014, 10
Analysis ID	QuantRef-100002-06-05-2014.100002.10r
Date of Measurement	06-May-2014 08:13:54
Name of Instrument	FOODSCREENER1
Tube Identification	QuantRefJuice.02.07.2012.1
ERETIC Definition	29-Nov-2013 11:12:14

Acquisition/Processing Parameters:

Parameter	Current Value	Reference Value	Confirm
P1	11.91	11.82	○
PL1	-8.00	-8.00	○
PL9	50.48	50.55	○
RG	16	16	○
D1	4	4	○
D8	0.01	0.01	○
NS	16	16	○
DS	4	4	○

Confirm the following parameters and settings:

Parameter / Setting	Confirm
Tube "QuantRefJuice.02.07.2012.1"	○
Parameter set is PROF_JUICE_1D	○
Temperature $T = 301.8K$	○
ATMA was done	○
LOCK on solvent "Juice"	○
TOPSHIM was done	○
PULSECAL was done	○
Phase checked	○
Baseline checked	○

Validation Result:

Parameter	Value	Reference Range	Status
Quantitative Calibration	99.0%	98% - 102%	●
Maximum Internal Deviation	2.3%	max. 4%	●

Name of operator: **Signature:**

If one of the quality indicators is not inside the tolerance range, please first perform a new 3D-shim on the sucrose sample and measure the QuantRef sample again. If it is still outside the tolerance range, add a new calibration.

Bruker BioSpin GmbH

Analysis Report

Quantification Reference - Validation

Sample ID, ExpNo	QuantRef-100004-14-07-2016, 10
Analysis ID	QuantRef-100004-14-07-2016.100004.10r
Date of Measurement	14-Jul-2016 07:55:41
Name of Instrument	FOODSCREENER2
Tube Identification	QuantRefJuice.19.01.2011.1
ERETIC Definition	23-Jun-2016 09:41:36

Acquisition/Processing Parameters:

Parameter	Current Value	Reference Value	Confirm
P1	11.72	11.82	○
PL1	-8.10	-8.10	○
PL9	50.52	50.45	○
RG	15.95	15.95	○
D1	4	4	○
D8	0.01	0.01	○
NS	16	16	○
DS	4	4	○

Confirm the following parameters and settings:

Parameter / Setting	Confirm
Tube "QuantRefJuice.19.01.2011.1"	○
Parameter set is PROF_JUICE_1D	○
Temperature $T = 301.8K$	○
ATMA was done	○
LOCK on solvent "Juice"	○
TOPSHIM was done	○
PULSECAL was done	○
Phase checked	○
Baseline checked	○

Validation Result:

Parameter	Value	Reference Range	Status
Quantitative Calibration	103.3%	98% - 102%	●
Maximum Internal Deviation	1.2%	max. 4%	●

Name of operator: **Signature:**

2.3 Daily Quality Control using a Wine Sample

In addition to the standard quality control measurements using Sucrose, MeOD and the QuantRef-sample, it is recommended to monitor the quality of the complete process using a real wine sample. For this, select any wine and create several aliquots of it. On each day that the Wine-Profiling method is used, add one of the aliquots to the measurements.

Select a representative list of results from the quantification and statistical analyses and create a continuous quality control sheet.

3 How to Setup and Run a Single Wine Sample

In order to run the fully automated Wine-Profiling method under SampleTrack, the following software components need to be started:

- TopSpin and IconNMR at the spectrometer PC. IconNMR needs to be in **automation** with mode **SampleTrack**.
- The SampleTrack server PC, SampleTrack Communicator and SampleTrack Web service need to be running (symbols on desktop, if running on task-bar).
- For automated retrieving of the reports, the Bruker Analysis Instrument also has to be started (symbol on desktop).

Start the SampleTrack client and setup with a new order.

In the window, click on the empty page icon and select **Wine Easy Dialog**:

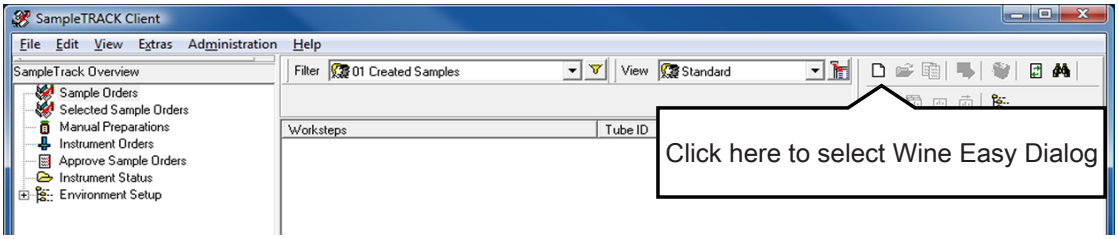


Figure 3.1: Selecting Wine Easy Dialog in SampleTrack

The Wine Easy Dialog window will open:

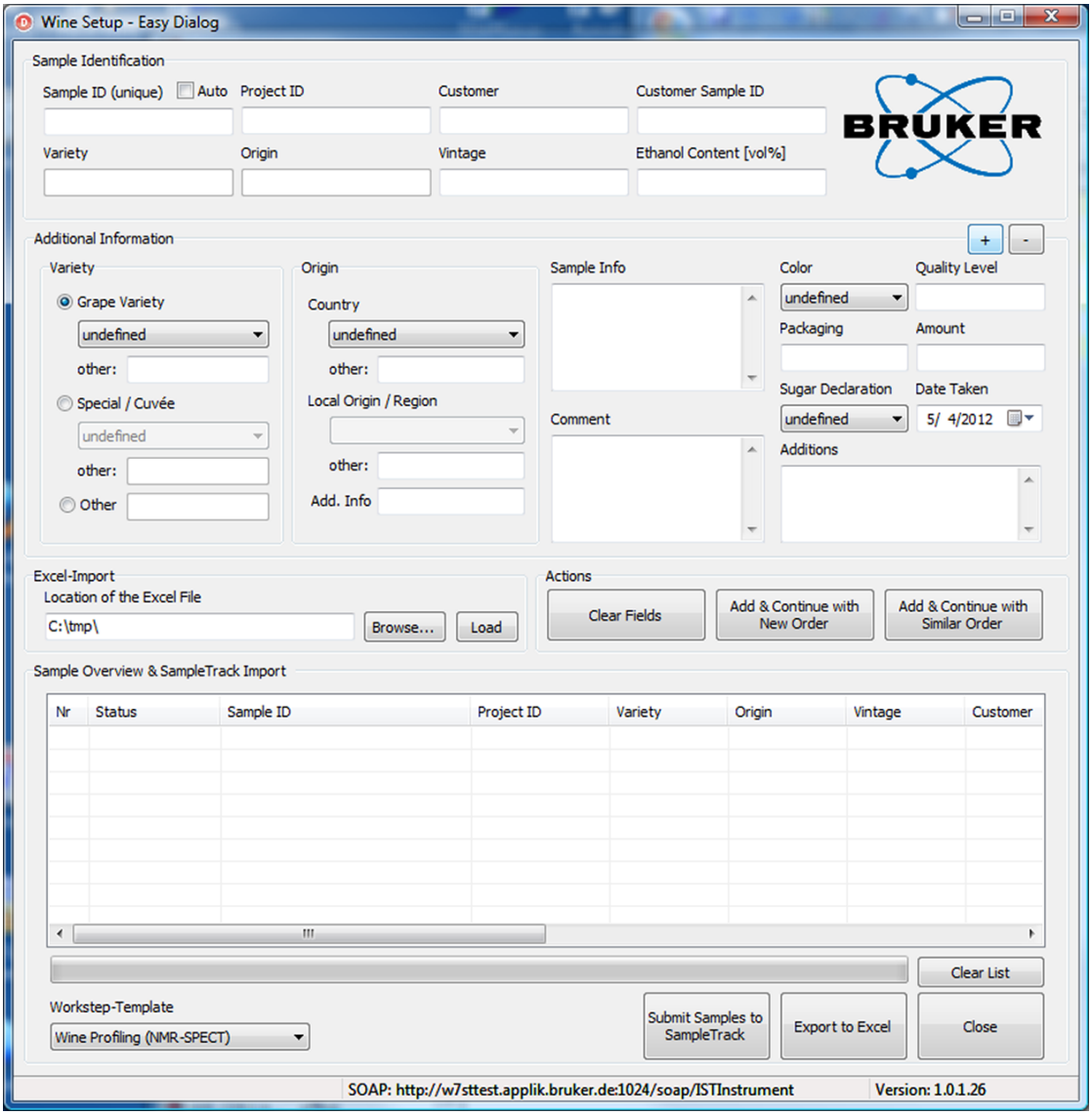


Figure 3.2: Wine Setup – Easy Dialog

The minimum information needed for a setup of a sample is a unique sample ID. As an option, unique Sample IDs can be generated by activating the box **Auto**. To enable variety dependent **verification** models, the grape variety must be defined as well (e.g. Riesling).

As an example, the following figures and steps demonstrate how to setup a wine sample with Sample ID *120504005* (the numbers in the brackets **[ ]** correspond to the figures):

- Select the **Grape Variety** (e.g. *Riesling*), **Origin** (e.g. *Germany - Rheinhessen*), and the **Vintage** (e.g. *2011*) **[1]**.

  You can collapse or expand the additional information with the "**+**" and "**-**" buttons.
- Add the new sample to the Sample Overview & SampleTrack Import table by selecting **Add & Continue with New Order [2]**.
- Select the new sample from the table with your mouse **[3]**.
- Submit the sample to SampleTrack by selecting **Submit Samples to SampleTrack** [4].

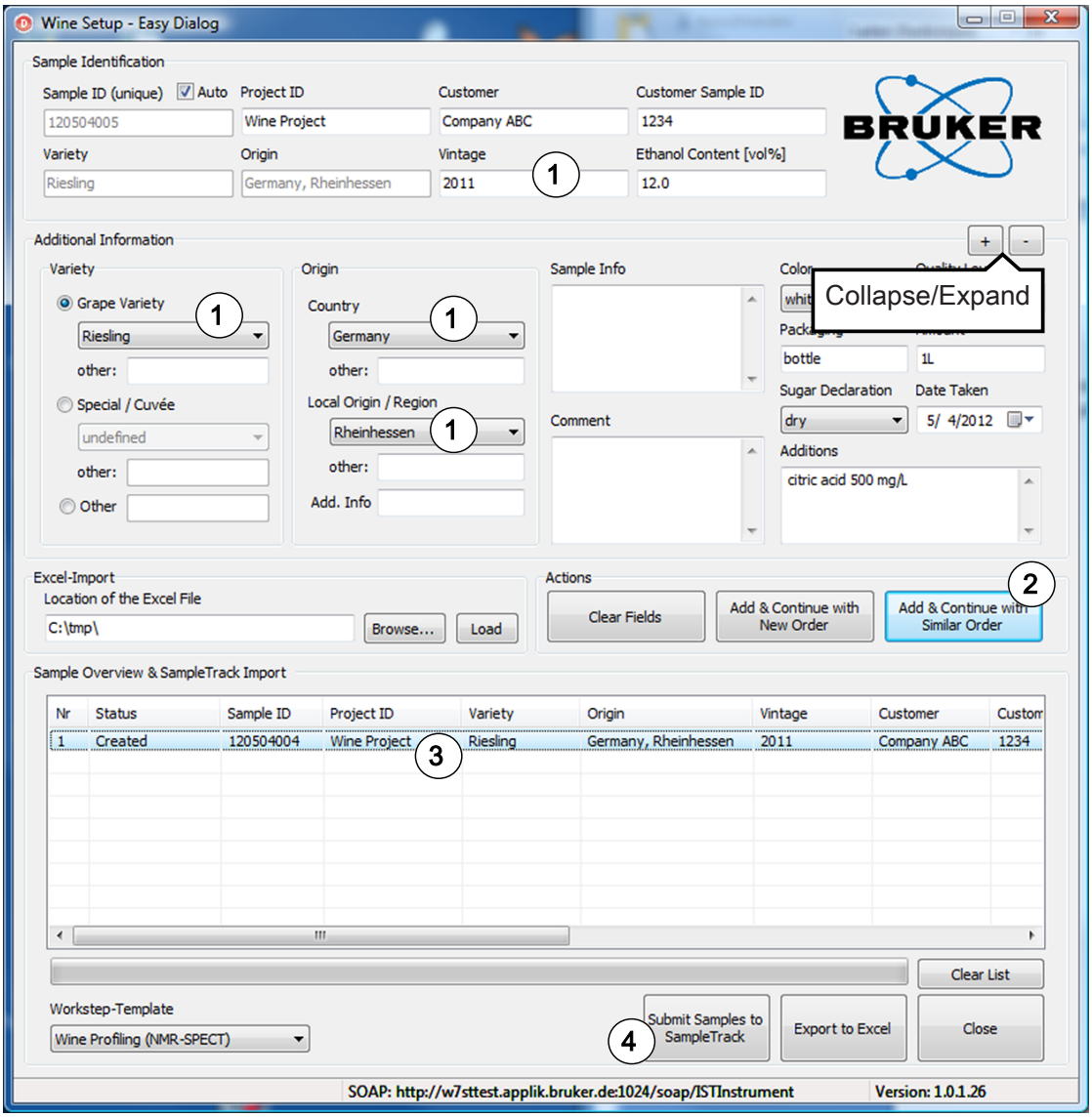


Figure 3.3: Wine Setup - Easy Dialog

After this step, the sample is registered in SampleTrack in the Sample Orders Overview **[5]**. You can use filters to select the samples of interest (refer to the SampleTrack manual for further information). The sample order now contains the method NMR, the preparation Wine Preparation and the NMR-Experiments WINE_PREP, WINE_NOESY, WINE_JRES and WINE_QUANT. In addition, there is now a report-work step Wine Analysis which is responsible for the data transfer to Bruker, and finally for retrieving the analysis report.

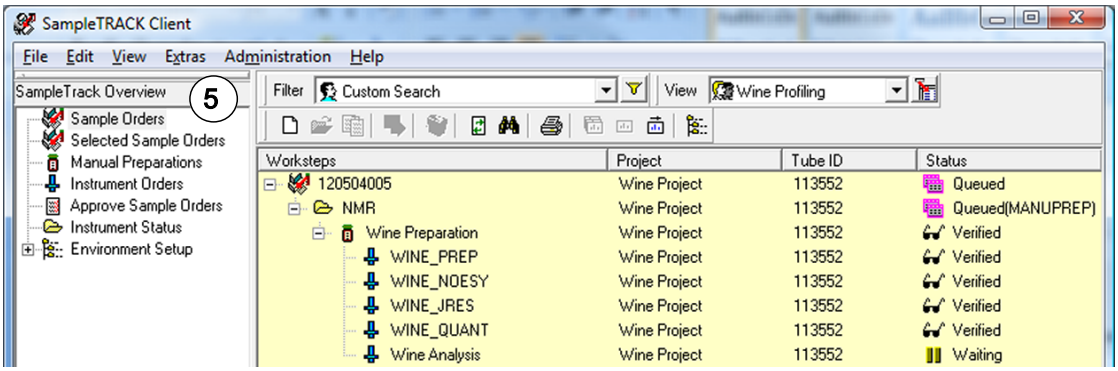


Figure 3.4: SampleTrack Sample Order Overview

The next step is the preparation of the sample.

- In the view Manual Preparations **[6]** all samples which are waiting for preparation are listed **[7]**. Select the sample and click **Preparation Guide [8]**.

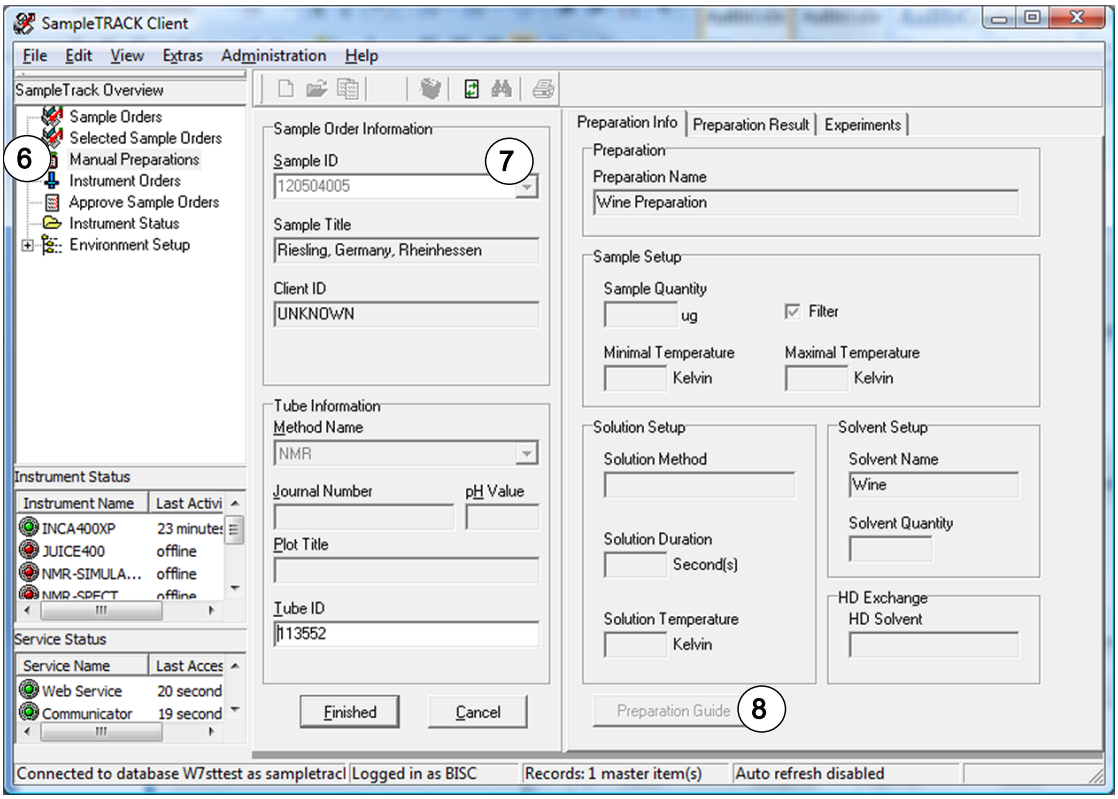


Figure 3.5: Sample Preparation in SampleTrack

You can now start the preparation of the wine sample (refer to the Standard Operating Procedure for wine).

- If available, enter parameters obtained by preparation (e.g. density, added volumes of acid and base) into the form of preparation guide.
- Click **Finish** to print a barcode for this tube **[9]**.

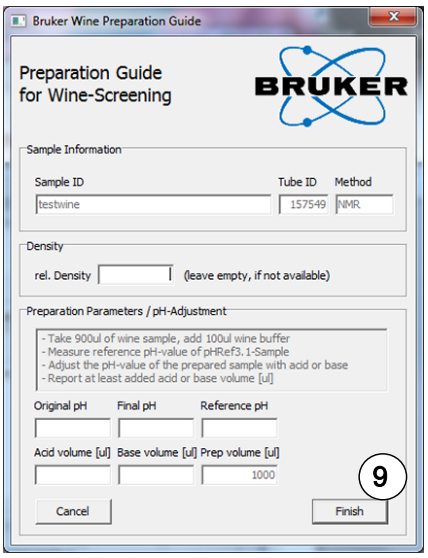


Figure 3.6: Wine Preparation Guide

- After this step, put the barcode on the NMR tube and insert this tube into the sample changer (ensure that the upper end of the barcode-label is 15 mm below the top of the tube).

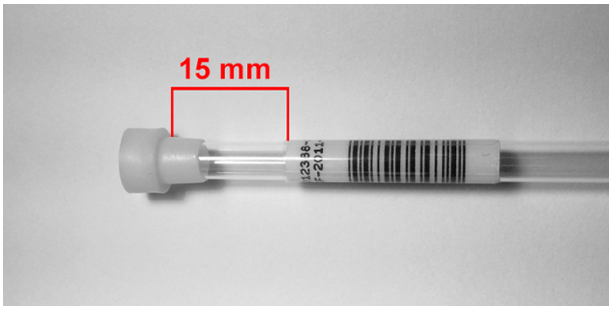


Figure 3.7: Placing the Barcode on the NMR Tube

3.1 Accessing a Sample Status, Spectra and Report

In SampleTrack, the status of the sample can be checked in the SampleTrack client window, click on **Sample Order** and look at the status field.

The overall order and the NMR method are registered as *Queued*, the wine preparation is listed as *Finished*, and as long as the tube has not been taken into the magnet, the NMR experiments are listed as *Verified*.

The wine analysis has a status *Waiting* until data is available for the sample.

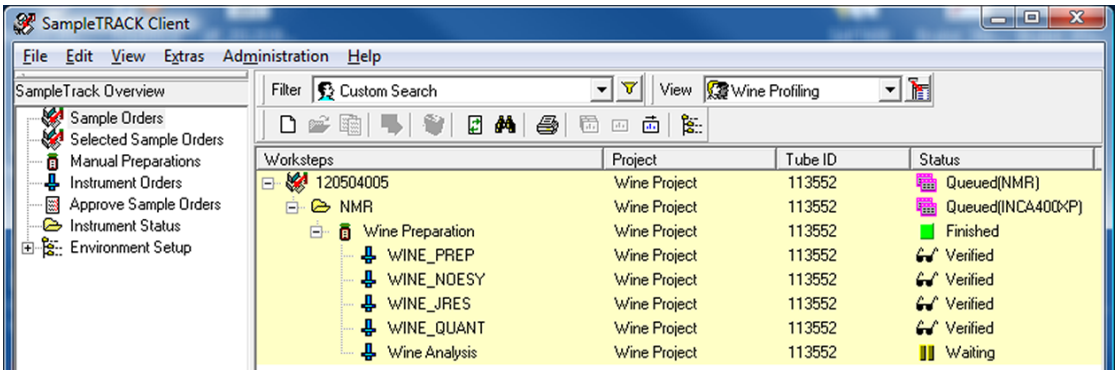


Figure 3.8: Sample Status in SampleTrack

Once, the wine screening is completed, all the status items switch to *Finished*. Now, spectra can be displayed by:

1. Right-clicking with the mouse on the respective experiment (blue in the figure below).
2. Selecting **View Spectrum**.
3. Selecting the viewer, i.e. either **AMIX** or **TopSpin**.

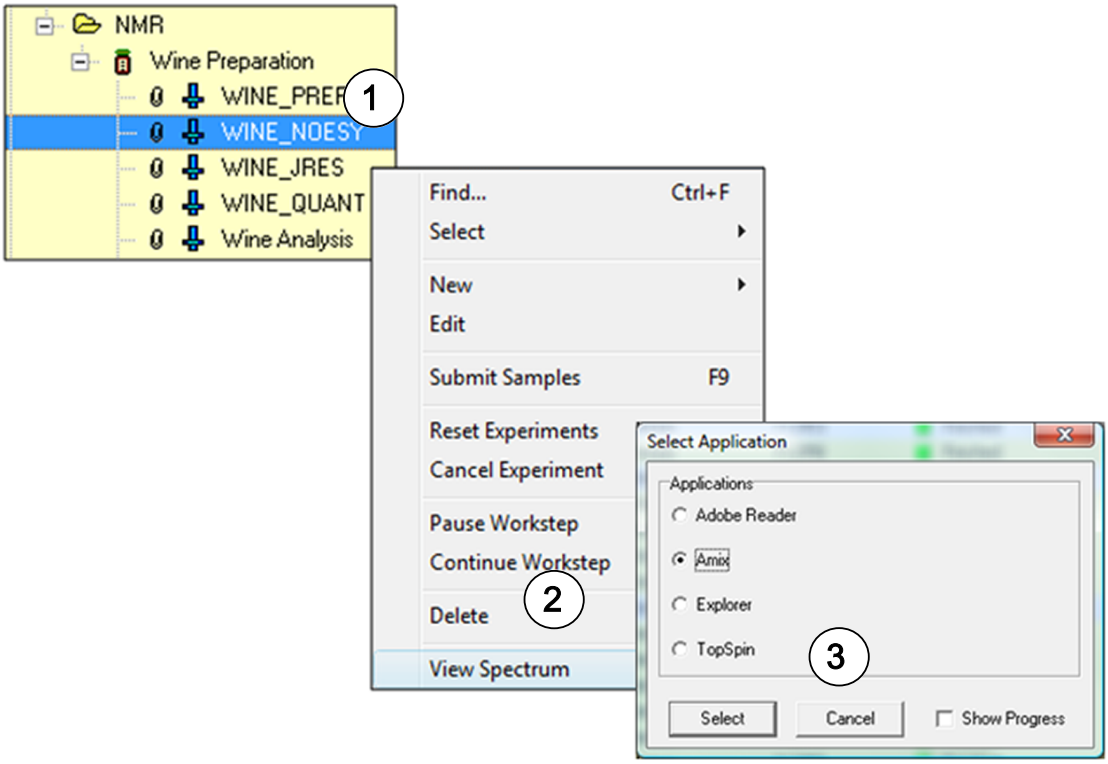


Figure 3.9: Accessing the Sample Status, Spectra and Report

The spectrum is then displayed in the respective program.

Using the same steps, a PDF report summarizing the wine analysis results can be displayed:

- Select **Wine Analysis** under step 1.
- Select the **Adobe Reader** under step 3.

4 Steps for Re-Measurement, Re-Preparation or Re-Analysis

In the case that a sample needs to be re-measured or re-prepared and re-measured, respective work steps can be used in SampleTrack.

4.1 Re-Measurement (overwriting existing experiments)

To re-measure a sample and overwrite the existing data:

- In the Sample Orders select **View the corresponding experiments** (PROF_WINE_PREP, PROF_WINE_NOESY, PROF_WINE_JRES and PROF_WINE_QUANT).
- Right click on the sample order and select **Reset Experiments**.

  This will reset the experiments and will set the status to *Created*.
- To verify this process, select the sample order, then **Submit Sample** (e.g. with a right mouse click or **Submit Icon** on the Icon bar).

4.2 Re-Measurement (adding of experiments)

To re-measure sample without overwriting existing data:

- Select the corresponding sample order in the Sample Orders View.
- Right click on the sample order and select **Add Work steps**.

  This will open a dialog for selecting the work step template.
- Select the work steps **PROF_WINE_PREP**, **PROF_WINE_NOESY**, **PROF_WINE_JRES** and **PROF_WINE_QUANT** and **Wine Analysis** for the template wine profiling (NMR-SPECT) and commit with **OK**.

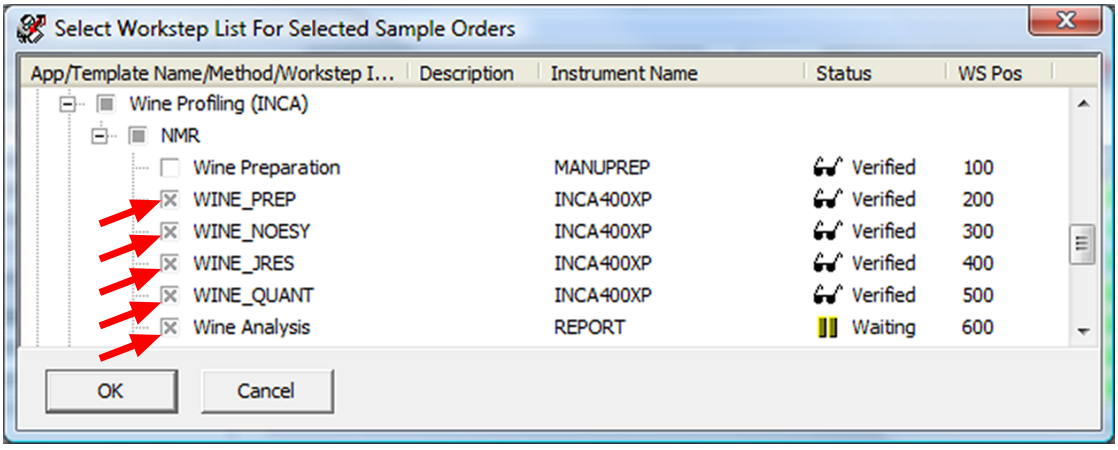


Figure 4.1: Selecting the Work step List for Selected Sample Orders

Now, there will be five additional work steps listed for this sample, whereas you can perform these steps as often as you want.

4.3 Re-Analysis (without Re-measurement)

For a new generation of the PDF report, it is not necessary to re-measure the sample. You just have to reset the wine analysis-work step to re-submit the sample (both are done in the context-menu by performing a right mouse-click on the **Juice Analysis** work step).

5 Contact

Manufacturer

Bruker BioSpin GmbH
Silberstreifen 4
D-76287 Rheinstetten
Germany
http://www.bruker.com

WEEE DE43181702

NMR Hotlines

Contact our NMR service centers.

Bruker BioSpin NMR 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 NMR service center or hotline you wish to contact from our list available at:

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

Phone: +49 721-5161-6155

E-mail: *nmr-support@bruker.com*

Contact

List of Figures

List of Figures

Bruker Corporation

info@bruker.com
www.bruker.com

Order No: H161720