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Calibration Guidelines for the Bruker NMR instrument

The ChemisTwin™ portal can perform quantitative analysis by using a digital external quantification approach. This approach uses the absolute integral provided by the instrument. The absolute integral is instrument dependent and to perform digital external quantification, a calibration of the instrument must be done to be able to compare the signal provided by the different instruments (admin vs user).

There are many parameters that can affect the absolute integral such as temperature, type of NMR tubes used, the number of scans, D1, etc. The first version of the portal will support the digital quantification using Bruker NMR spectrometers. Here, we provide a detailed guideline to calibrate the NMR instrument in the portal. The calibration measurements can't be older than a week prior to the upload of the samples into the portal.

Before calibrating the NMR instrument, it is necessary to set and maintain the NMR spectrometer in a stable temperature (e.g. 20 – 30 °C). The standard and the NMR tubes suitable for the calibration are:

PN: **42350** / Bruker quantitative PQ Certified Reference Material (**https://www.sigmaaldrich.com/CH/en/product/supelco/42350**)

Schott® NMR sample tubes economic | Sigma-Aldrich (**sigmaaldrich.com**)

Sample preparation:

An ampoule of the standard is opened, and the standard solution is transferred to an NMR tube. It is then placed into the NMR instrument and six independent ^{1}H NMR measurements are to be performed on the same NMR tube using the following steps:

- Set a ^{1}H measurement using qNMR set up (pulse of 90°)
- Set Number of Scans to 16 (NS = 16)
- Set relaxation time to 140 s (D1 = 140 s)
- Set acquisition time to 10 s (AQ = 10 s)
- Set 6 different Receiver Gain (RG) (one RG per measurement) in the following range: 1st RG: 5 – 15, 2nd RG: 12 – 23, 3rd RG: 28.9 – 38.9, 4th RG: 58 – 78, 5th RG: 125 – 145, 6th RG: 165 – 185.
- Stable temperature (e.g., 20 – 30 °C) – recommended is 25 °C

Save spectra from Bruker in JDX format

The six measurements are converted into jdx files using topspin:

- Open one of the ^{1}H NMR measurement in Topspin software
- Save the spectra in .jdx file (JCAMP format, **Figure 1**) following the corresponding parameters (**Figure 2**)
- Upload the six jdx files into the portal and perform the calibration.

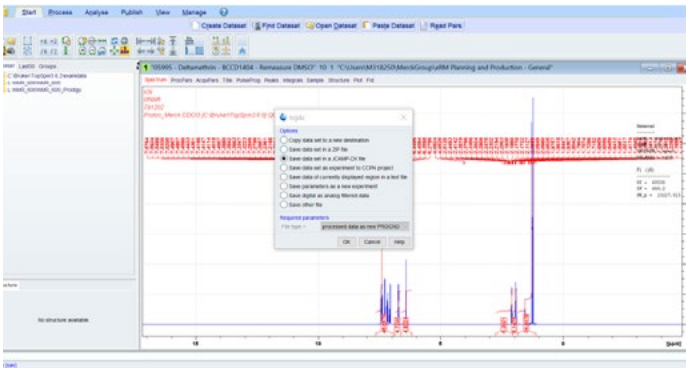

Figure 1. Save spectra in jdx file format in TopSpin

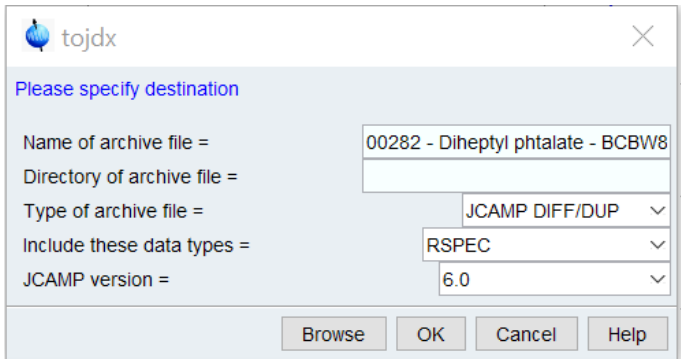

Figure 2. Parameters of the jdx file (type of archive file and data types)

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MilliporeSigma
400 Summit Drive
Burlington, MA 01803

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