Understand(s) your compounds

qNMR: setting up a CCF

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Intro

- You will need a spectrum of the concentration reference sample recorded on the spectrometer you will usually use
- The probe should be tuned and matched
- Use your typical acquisition conditions
  - the pulse repetition rate should not too rapid

Processing

- Zero fill once
- Window function
  - Exponential multipication ca. 0.3 Hz
- Baseline correction (if using “sum” integrals)
  - Bernstein polynomials, Order 3
- Phase: best possible if “sum” integrals

- Auto Multiplets or Auto Assignments done

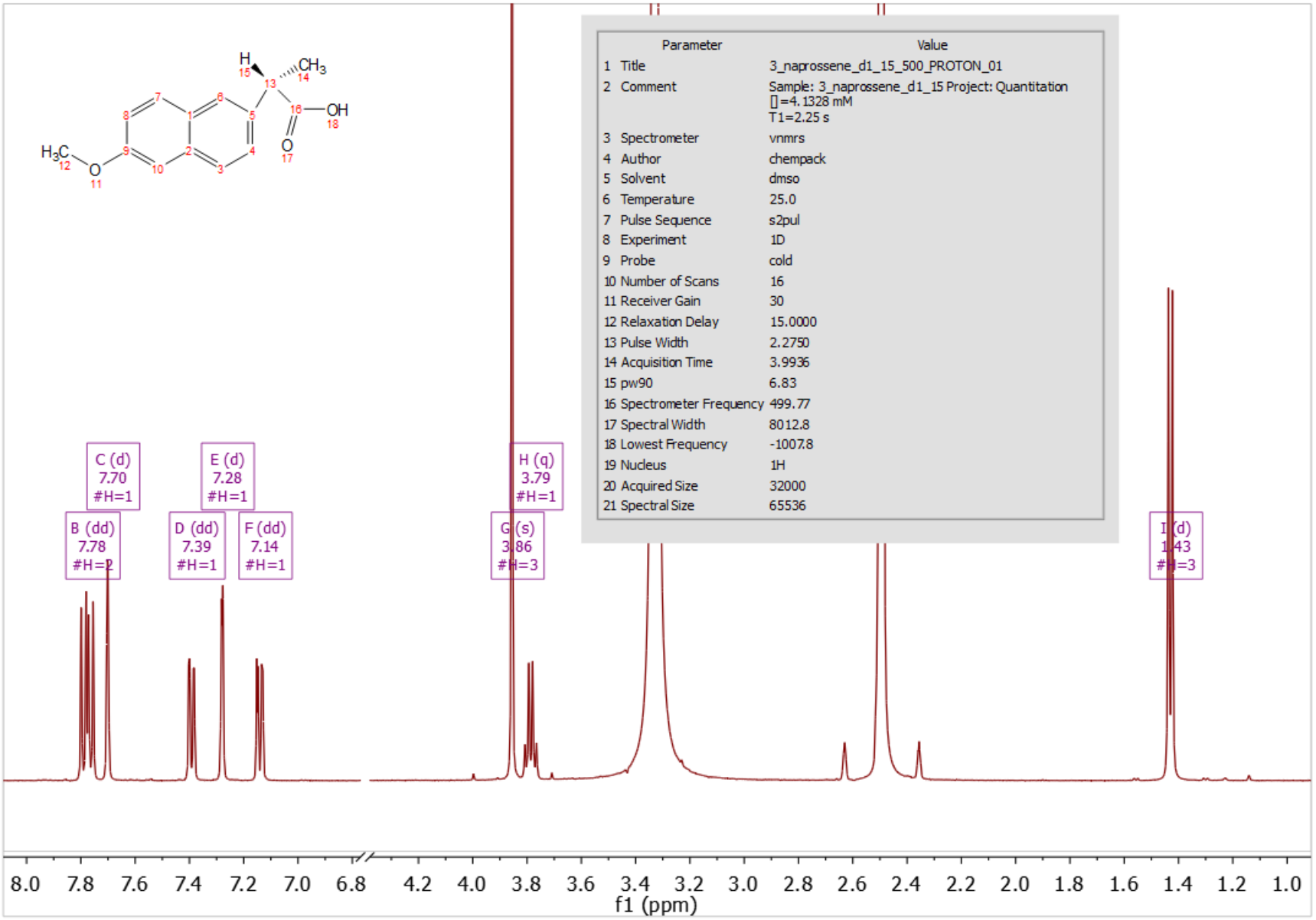
Integration method

Determine a CCF for the method you use

- "Sum": for simpler molecules and when the most accurate results are needed
- GSD: for most cases, assuming shimming is good, etc.
- Line fitting: can improve GSD, but not good for crowded spectra

Top GSD tip: Use "High resolution" and 5 fitting cycles (Peak Picking > Options)

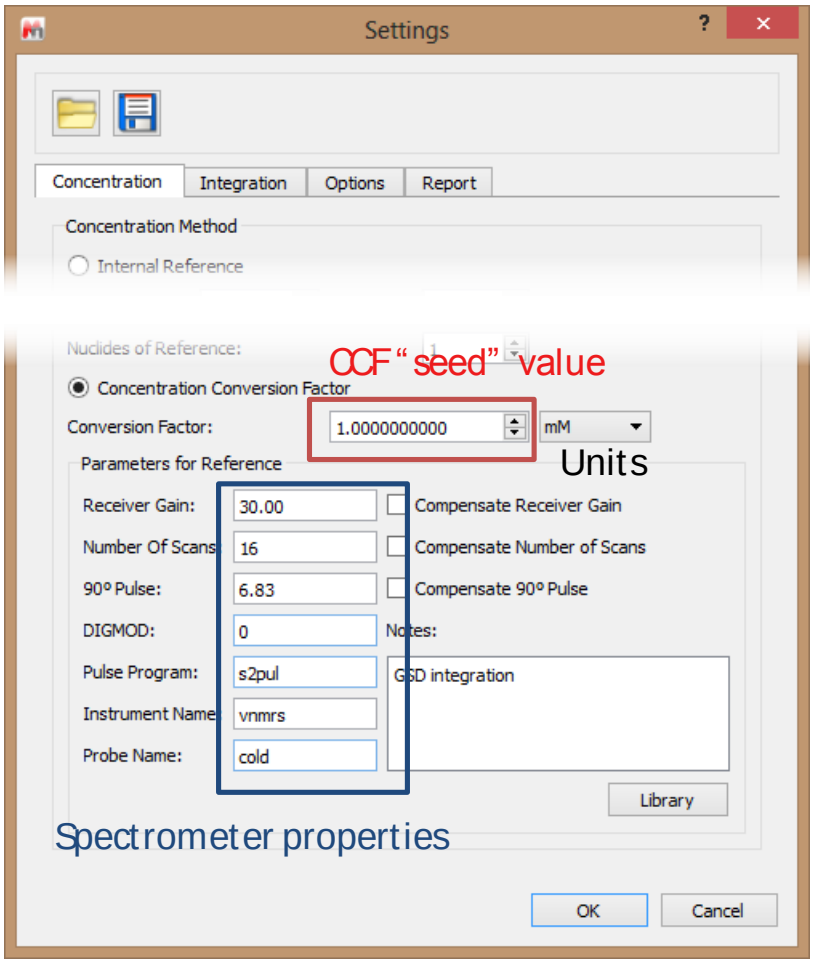
4.13 mM naproxen in DMSO-d6



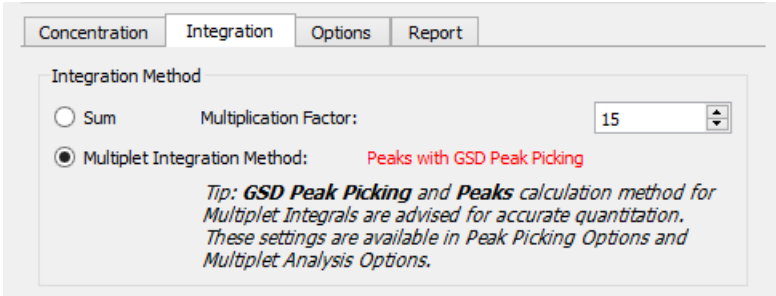
	Parameter	Value
1	Title	3_naprossene_d1_15_500_PROTON_01
2	Comment	Sample: 3_naprossene_d1_15 Project: Quantitation []=4.1328 mM T1=2.25 s
3	Spectrometer	vnmrs
4	Author	chempack
5	Solvent	dmso
6	Temperature	25.0
7	Pulse Sequence	s2pul
8	Experiment	1D
9	Probe	cold
10	Number of Scans	16
11	Receiver Gain	30
12	Relaxation Delay	15.0000
13	Pulse Width	2.2750
14	Acquisition Time	3.9936
15	pw90	6.83
16	Spectrometer Frequency	499.77
17	Spectral Width	8012.8
18	Lowest Frequency	-1007.8
19	Nucleus	1H
20	Acquired Size	32000
21	Spectral Size	65536

qNMR Concentration: 1st settings

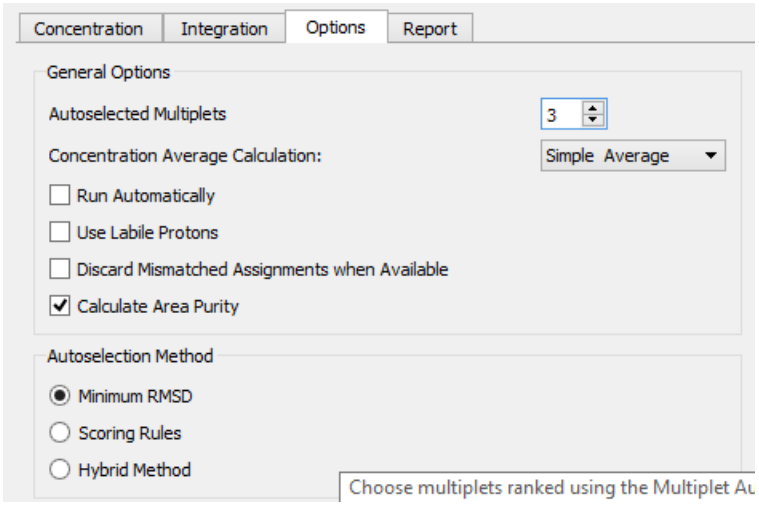
NB The CCF value at this stage is a total guess



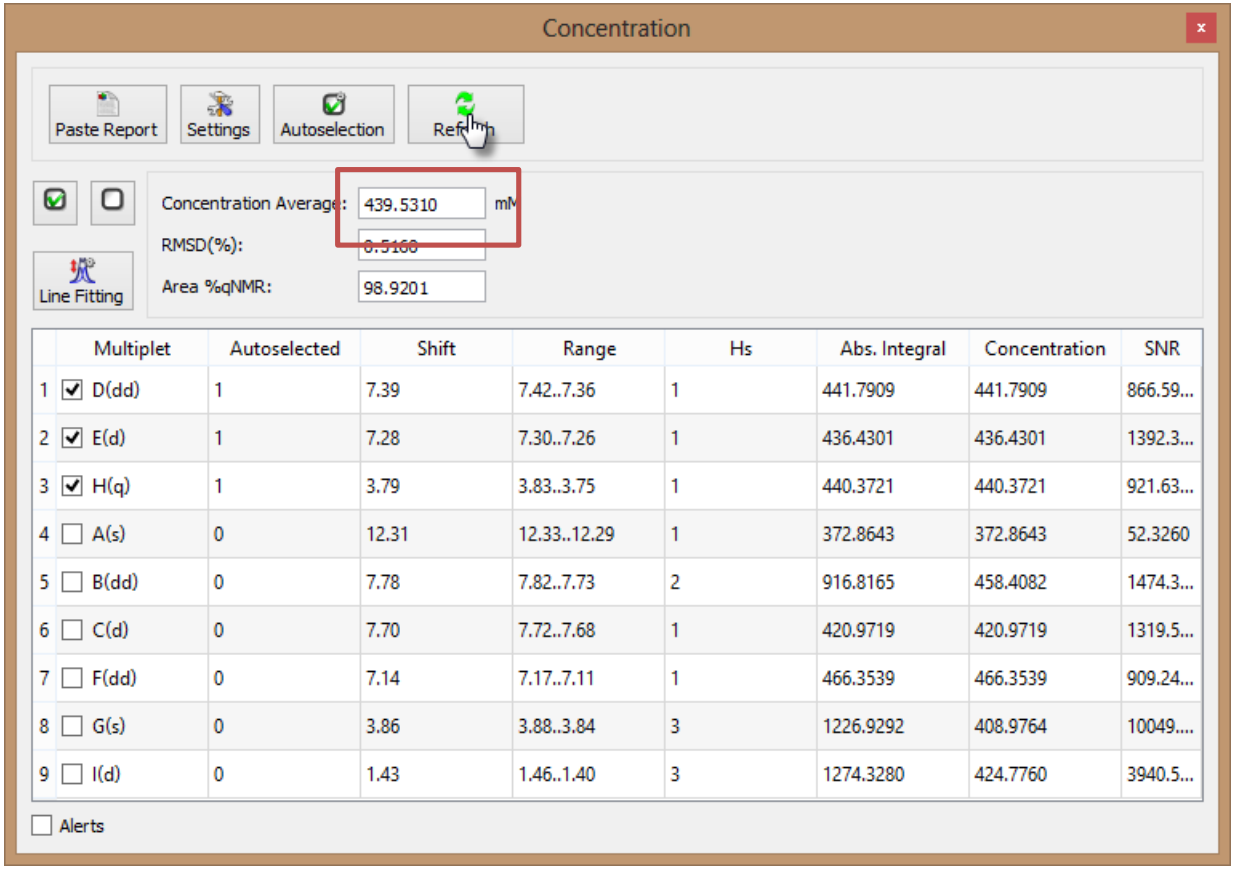
For this example



Simply…



Calculating the real CCF



	Multiplet	Autoselected	Shift	Range	Hs	Abs. Integral	Concentration	SNR
1	D(dd)	1	7.39	7.42..7.36	1	441.7909	441.7909	866.59...
2	E(d)	1	7.28	7.30..7.26	1	436.4301	436.4301	1392.3...
3	H(q)	1	3.79	3.83..3.75	1	440.3721	440.3721	921.63...
4	A(s)	0	12.31	12.33..12.29	1	372.8643	372.8643	52.3260
5	B(dd)	0	7.78	7.82..7.73	2	916.8165	458.4082	1474.3...
6	C(d)	0	7.70	7.72..7.68	1	420.9719	420.9719	1319.5...
7	F(dd)	0	7.14	7.17..7.11	1	466.3539	466.3539	909.24...
8	G(s)	0	3.86	3.88..3.84	3	1226.9292	408.9764	10049....
9	I(d)	0	1.43	1.46..1.40	3	1274.3280	424.7760	3940.5...

A CCF value of 1.0 results in a concentration of 439.5 mM

Correct CCF =

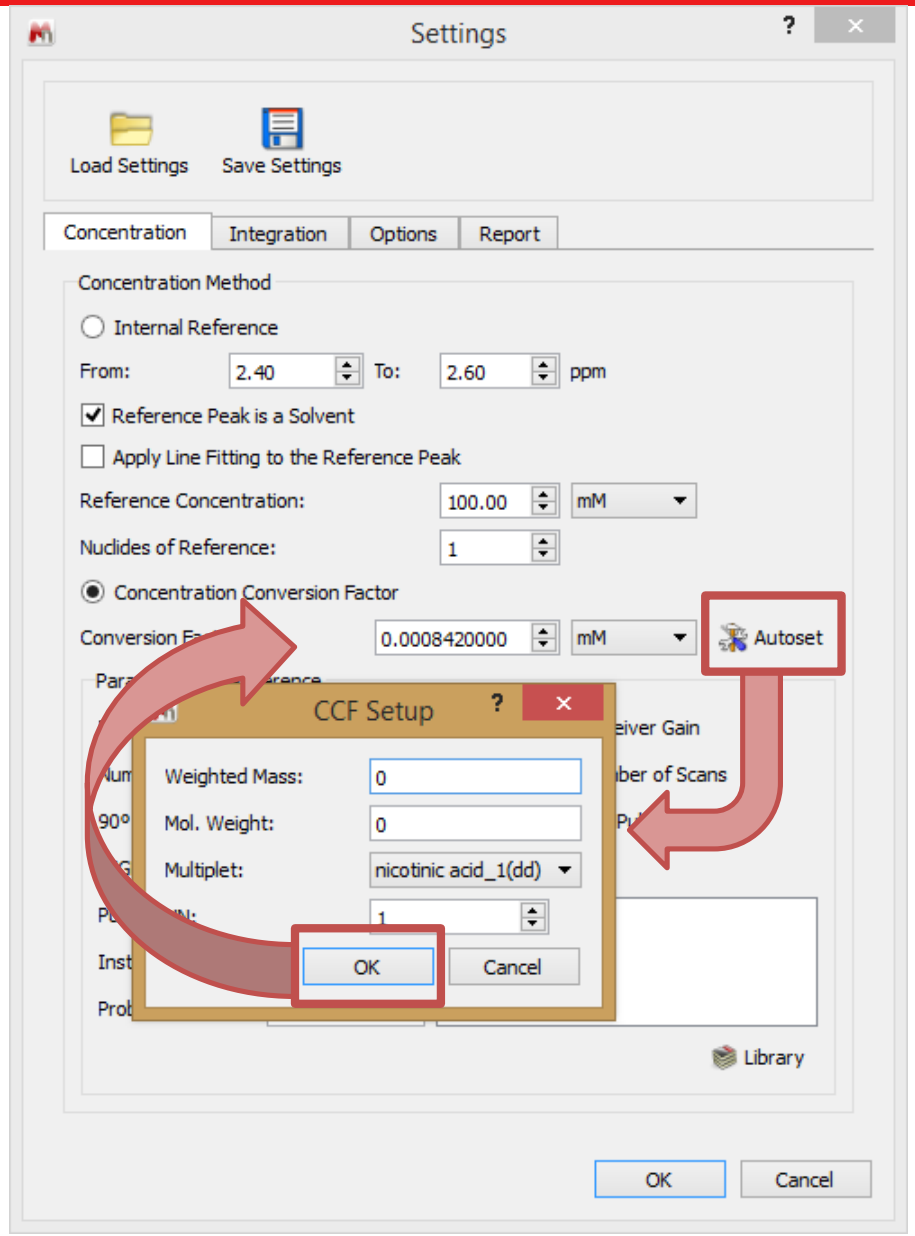
[real] / [apparent] * "seed value" =

4.13 / 439.53 = **0.0093964007**

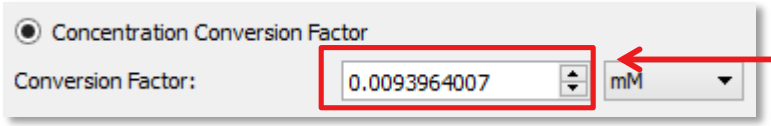
This value is inserted in the settings "Conversion Factor"

“Autoset” to calculate CCF

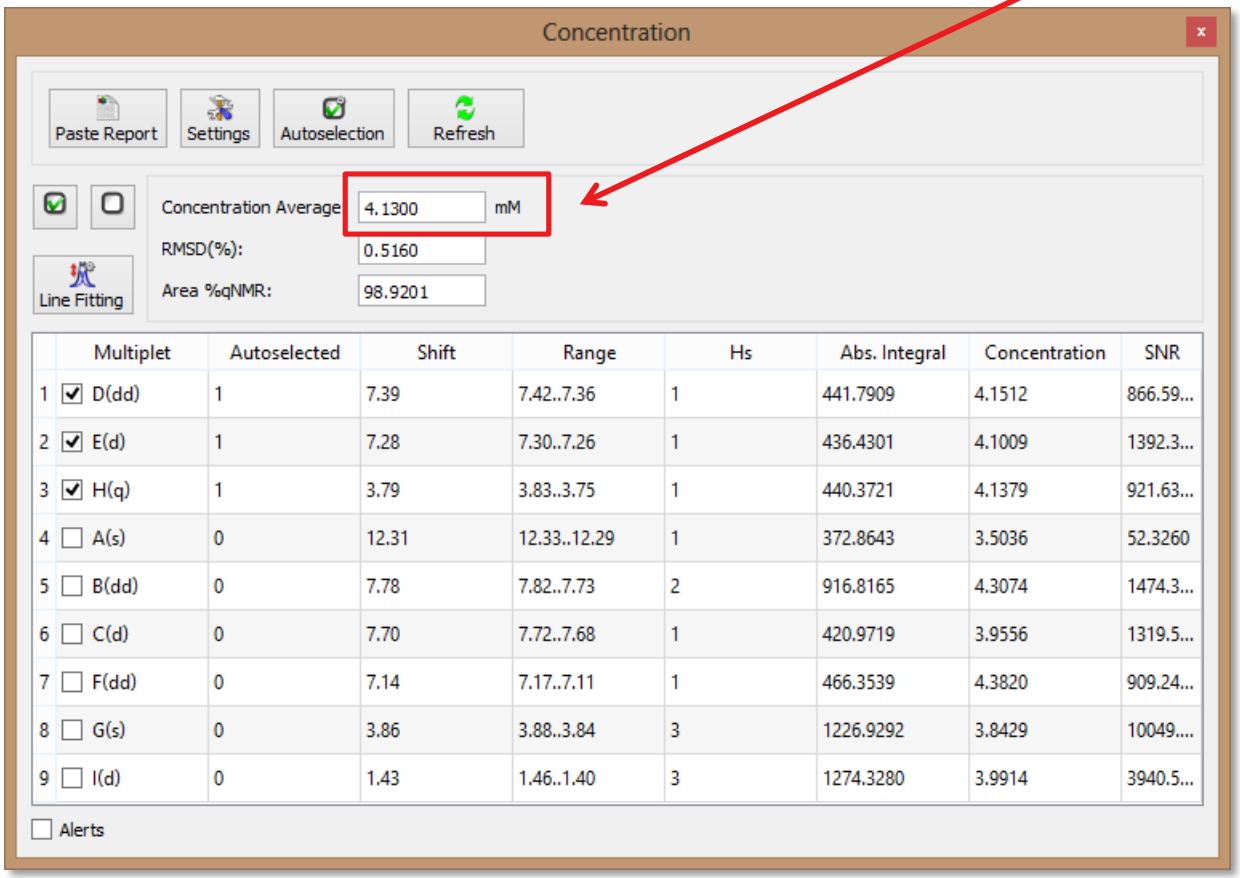
Using the spectrum of your concentration reference…



Checking it works



The corrected CCF gives the correct concentration



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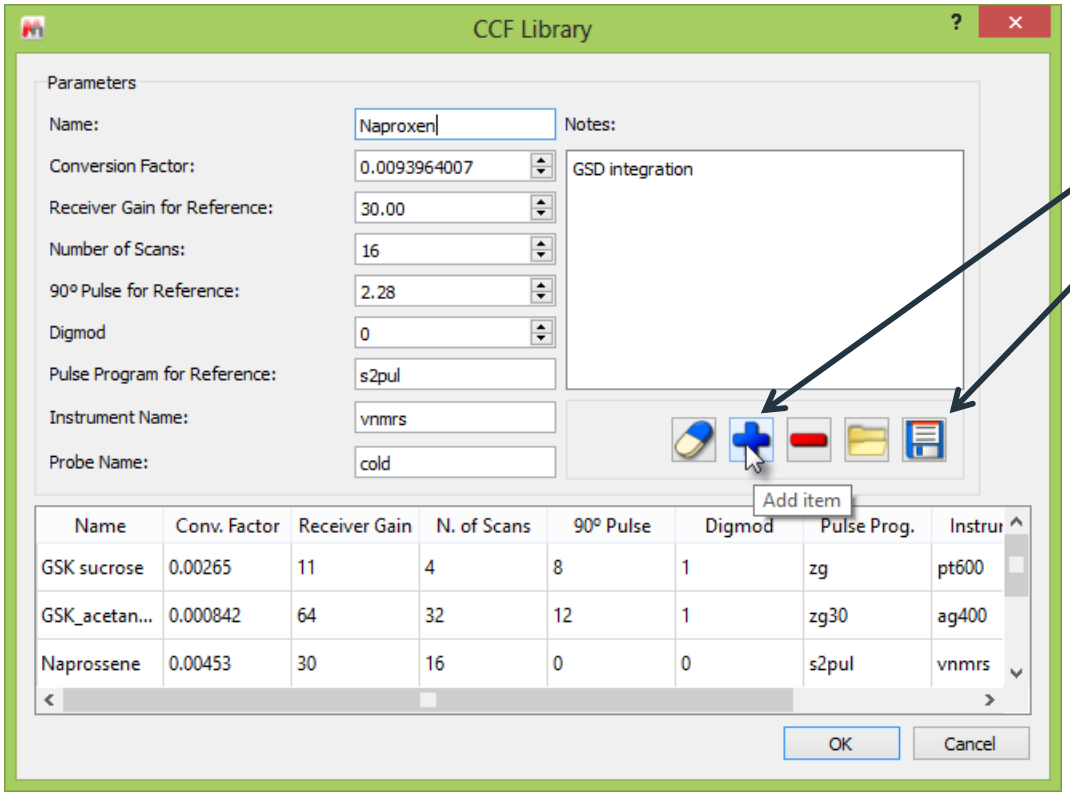
6	C(d)	0	7.70	7.72..7.68	1	420.9719	3.9556	1319.5...
7	F(dd)	0	7.14	7.17..7.11	1	466.3539	4.3820	909.24...
8	G(s)	0	3.86	3.88..3.84	3	1226.9292	3.8429	10049....
9	I(d)	0	1.43	1.46..1.40	3	1274.3280	3.9914	3940.5...

Saving the correct values to the Library



1. Click to add to the table
2. Save the updated table

Last considerations

- You may want to repeat this "calibration" for your spectrometer from time-to-time
- Some may prefer to make 3 or more concentration standards to get a more accurate CCF, and to test the values
- Samples and spectra can be measured in multiples for higher accuracy and precision