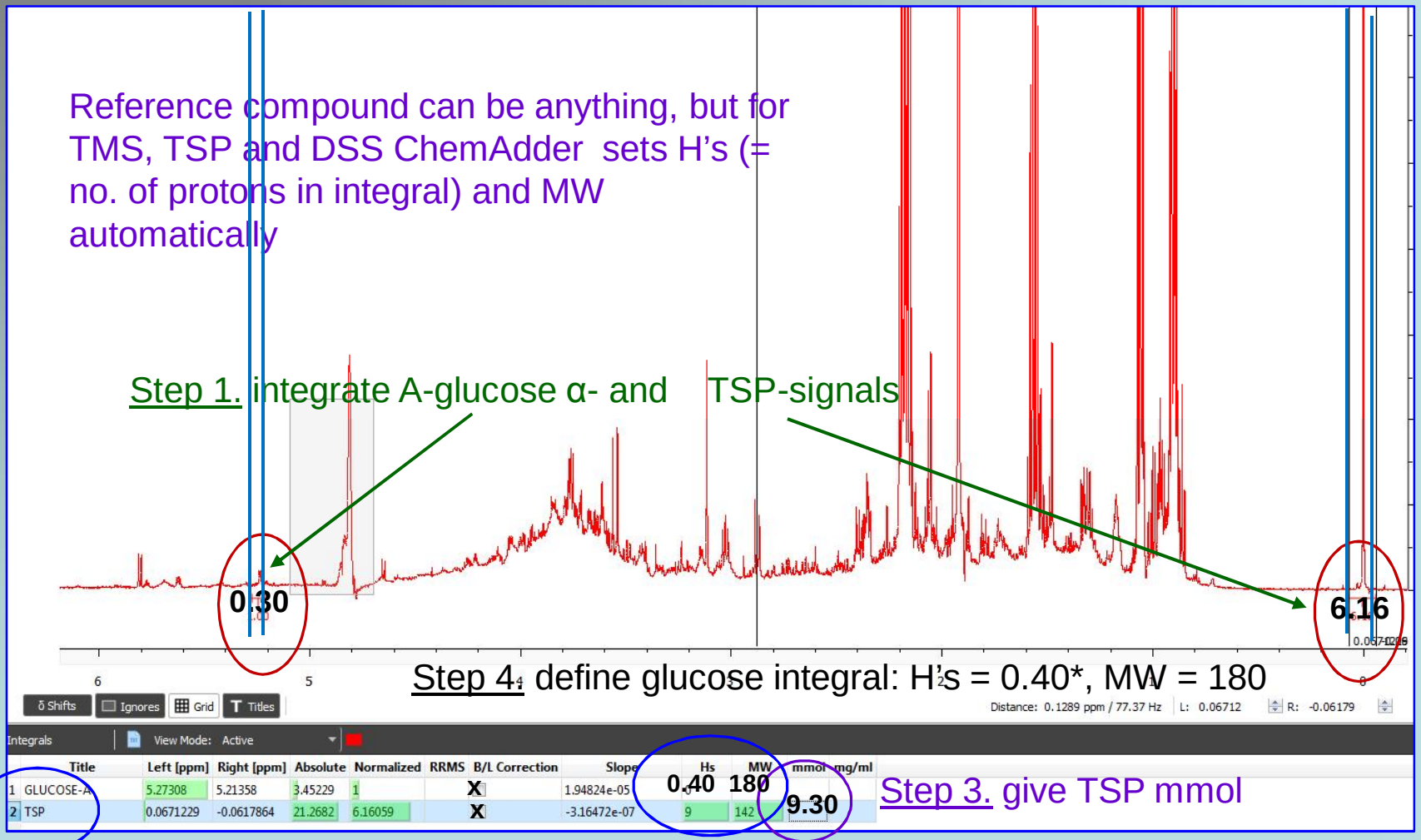

QUANTITATIVE NMR
Integration, deconvolution or qQMSA ?
Reino Laatikainen and Pekka Laatikainen
SpinDiscoveries Ltd.
Examples by Hannu Maaheimo (VTT) and Yrjö Hiltunen (UEF)

Basic integration

*When there is quantitative reference (TMS, TSP, DSS or REFi) and its concentration are given (in PMR-file), any spectral integrals (area) can be transformed into **mmol** when the number of protons in the integral area are defined. When the corresponding molecular weight (**MW**) is known, also **mg/ml** is obtained.*

Example 1

Quantification of *total* glucose in IQUQ

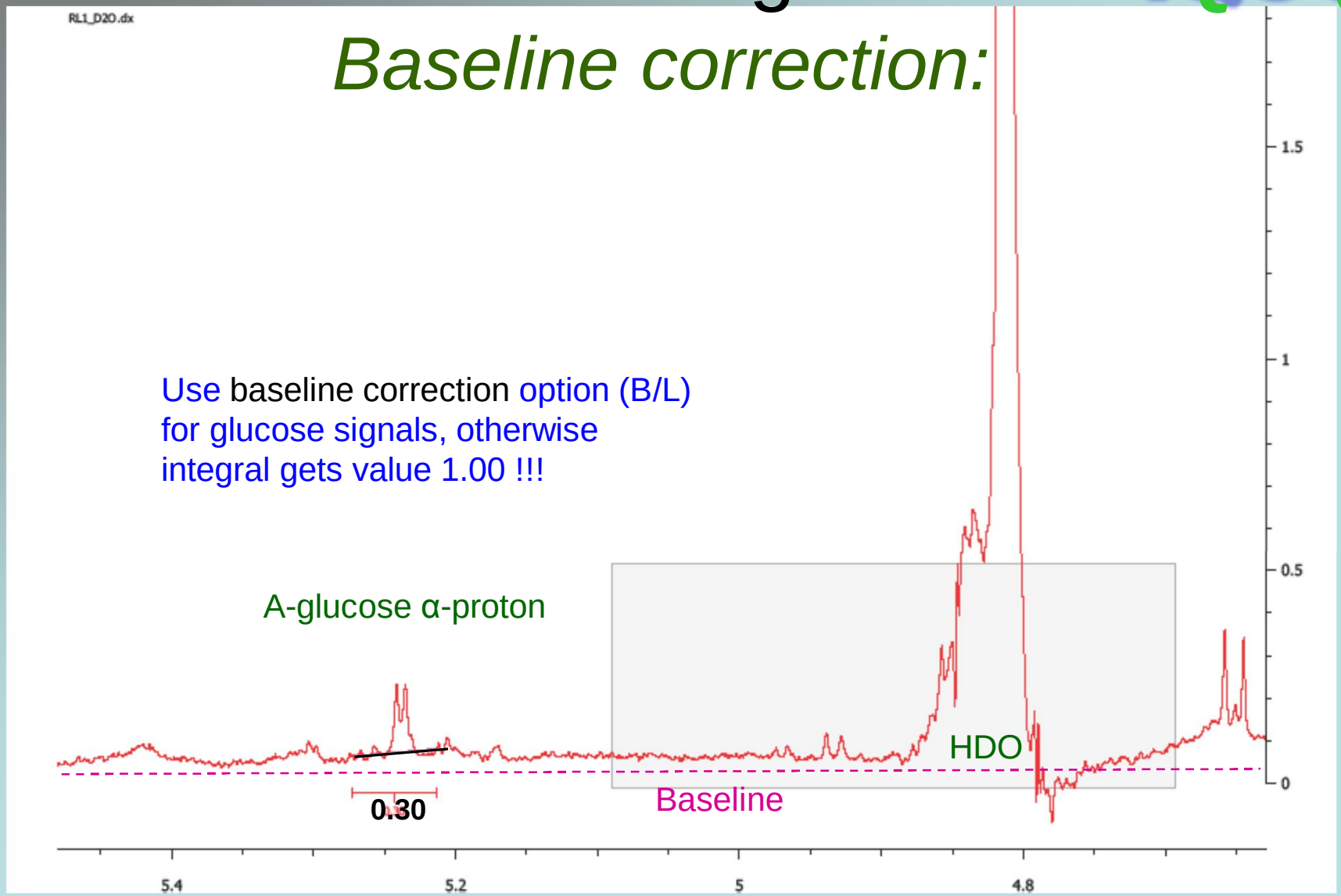


Step 2. Name integrals, use 'TSP'

*Only 40% of glucose is A-glucose

Quantification of *total* glucose in IQUQ

Baseline correction:

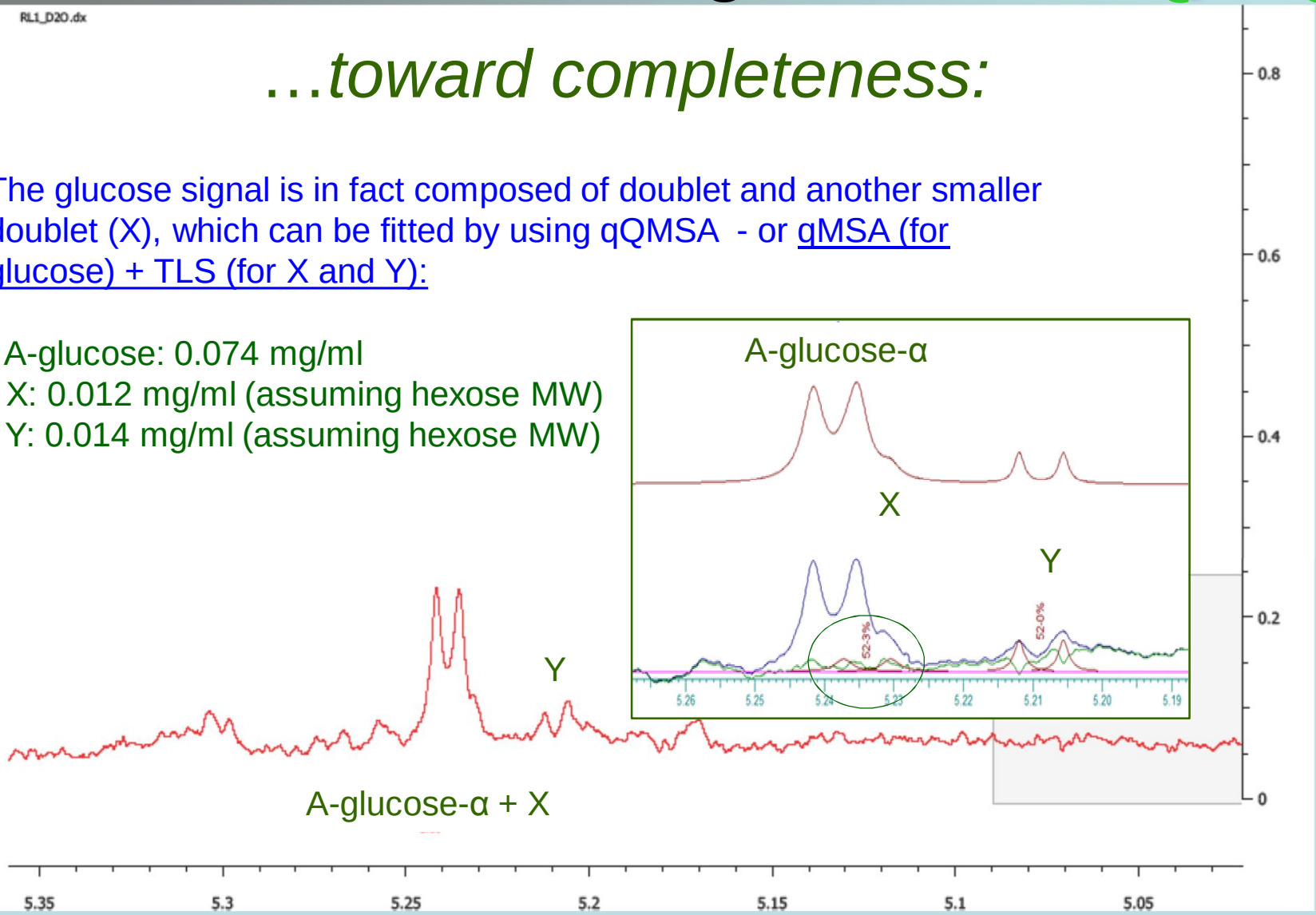


Quantification of *total* glucose in IQUQ

…*toward completeness:*

The glucose signal is in fact composed of doublet and another smaller doublet (X), which can be fitted by using qQMSA - or gMSA (for glucose) + TLS (for X and Y):

A-glucose: 0.074 mg/ml
X: 0.012 mg/ml (assuming hexose MW)
Y: 0.014 mg/ml (assuming hexose MW)



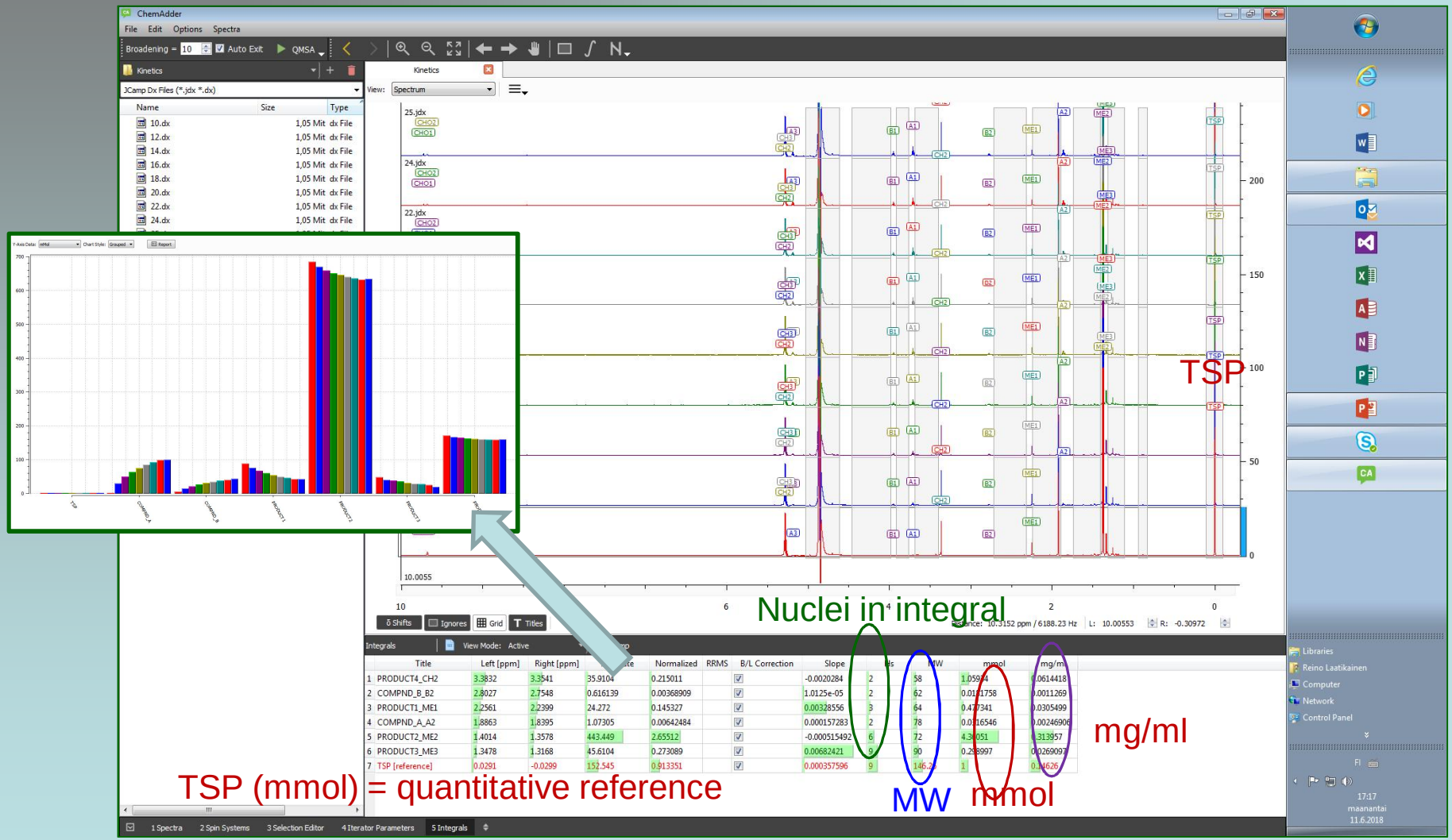
Example 2: kinetics by novel traditional integration

*Step 1: normalize spectra so that the volume of **reference** (here TSP) is the same in all the spectra.*
*Step 2: integrate **reference** signal and (if not TMS, TSP or DSS), name it, give no. of protons (9 inTSP) and molarity of it (MMOL).*
Step 2: select the signals to be integrated, name them, give no. of protons in each signal (for example, 3 for CH_3) and the MW of the corresponding compound.
Step 4: Integrate all the spectra in stacked mode.
Step 5: Show concentrations by ChemAdder tool, or transfer to EXCEL.

Easy, works well if signals well separated.

ChemAdder integration tool allows (spline polynomial) baseline fitting and therefore also small signals sitting on large broad signal can be safely integrated.

Integrate or fit (QMSA), then drag-and-drop to EXCEL or use the tools of ChemAdder – all data with same click!



Kinetics by qQMSA

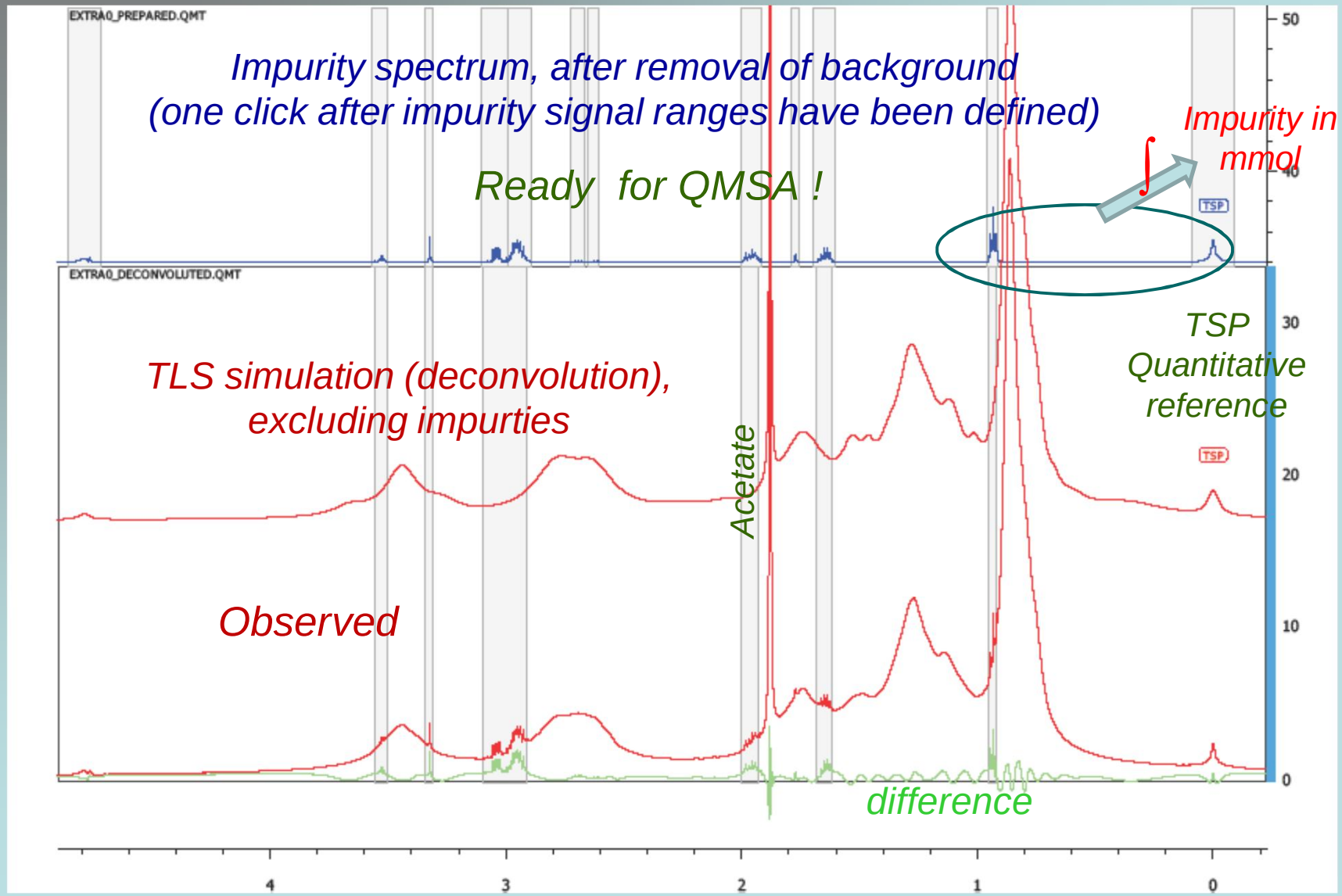
Step 1: analyze one representetative spectrum to make QMSA model for the system, use ignore for extra the signals.

Step 2: batch analyze the spectra with QMSA.

Step 3: show the concentrations by ChemAdder tool or transfer to EXCEL.

Spectral parameters needed but works also if signals not separated.

Example 3: Analysis of a macromolecular material with impurities (example by Yrjö Hiltunen)

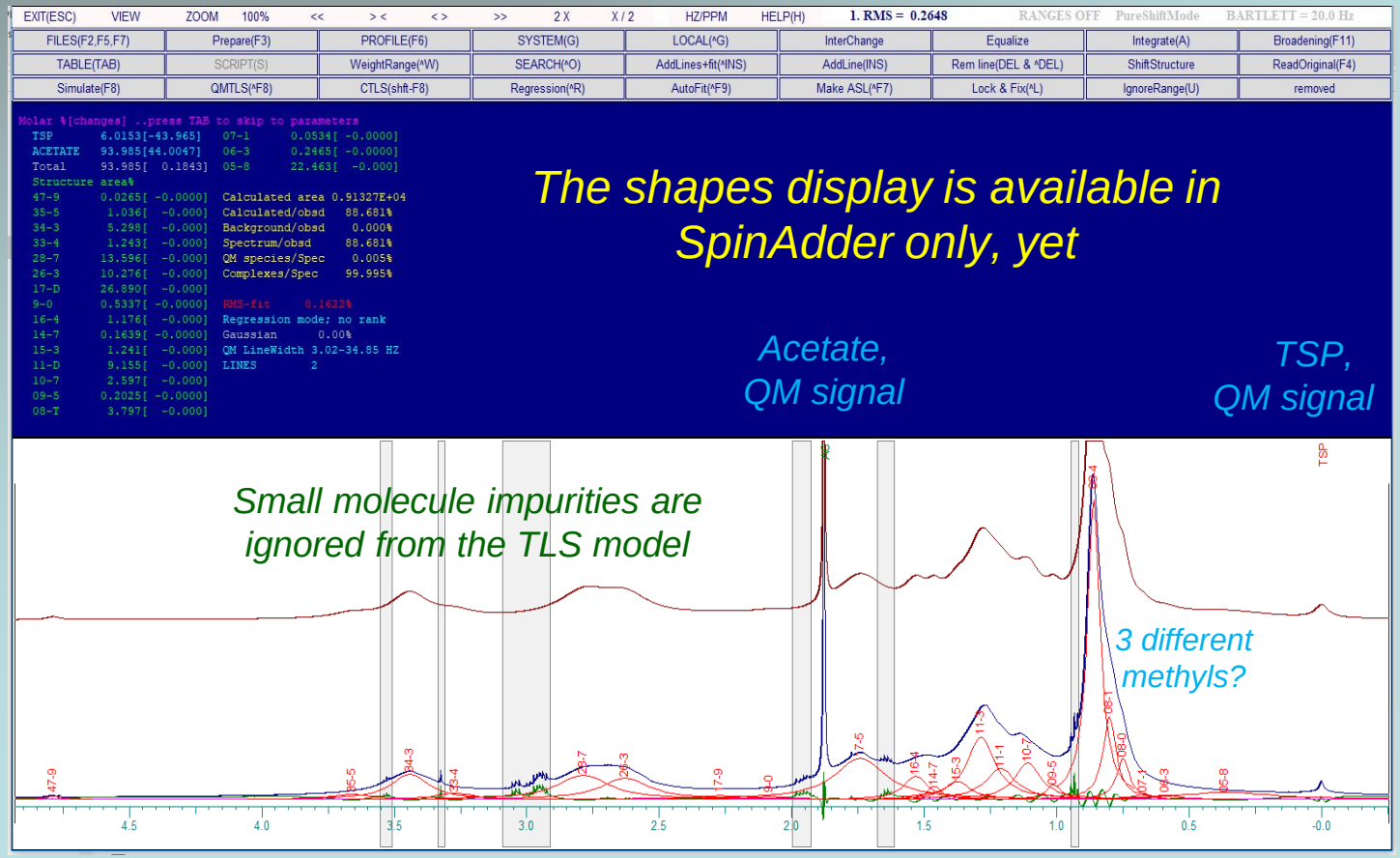


Deconvolution (TLS) analysis

Can be used to model signals which are not described by QM models – together with QMSA signals

*The spectrum (not explained by QM model) can be composed from **extra** lines or regular **multiplets** (doublets, triplets, etc.), which can be also named and grouped.*

If one sets a signal range and the number of lines in it, SpinAdder creates a model assuming equidistant lines with same line-width – the positions and line-widths can then be optimized.



HOLISTIC qQMSA

Combines qQMSA, TLS, integration, pattern search and forms mathematical model for correlations of metabolite chemical shifts

See Presentation: ChemAdder_HOLISTICS


IQUQ NMR
No problem with amount of sample!