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Mnova qNMR 2.0 – the tool for all skill levels

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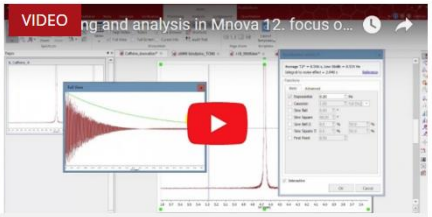
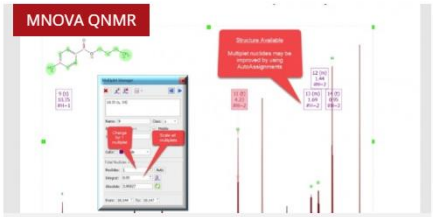


BY ENRIQUE SÁNCHEZ – 14 FEBRUARY, 2018 0

Processing and Analysis in Mnova 12. Focus on requirements for qNMR

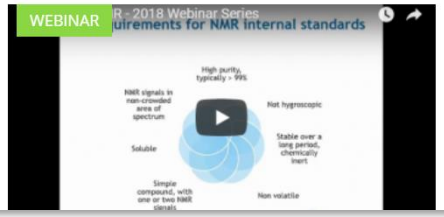
In this video Dr. Mike Bernstein focuses on requirements for qNMR analysis using Mnova 12....

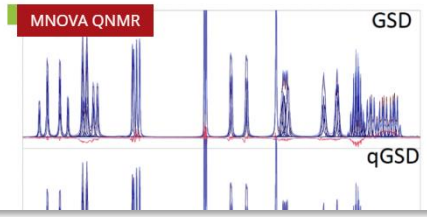


BY ENRIQUE SÁNCHEZ – 14 FEBRUARY, 2018 0

Processing and Analysis in Mnova 12. Focus on requirements for qNMR (pdf)

Title: Processing and Analysis in Mnova 12. Focus on requirements for qNMR Author: Mike Bernstein...





SEARCH

qNMR Search

UPDATES

2018

MAR 23 What's new in Mnova 12.0.2

JAN 09 What's new in Mnova 12.0.1

2017

OCT 11 What's new in SMA 2.0

NEWSLETTER SUBSCRIPTIONS

qNMR resources

YouTube ➔ Mestrelab channel

RESOURCES

http://www.nmrvalidation.org/

qnmr.org

Agenda

qNMR basics

Results expectations

Do's and don't of processing

Integration options

qNMR Purity plug-in - *DEMO*

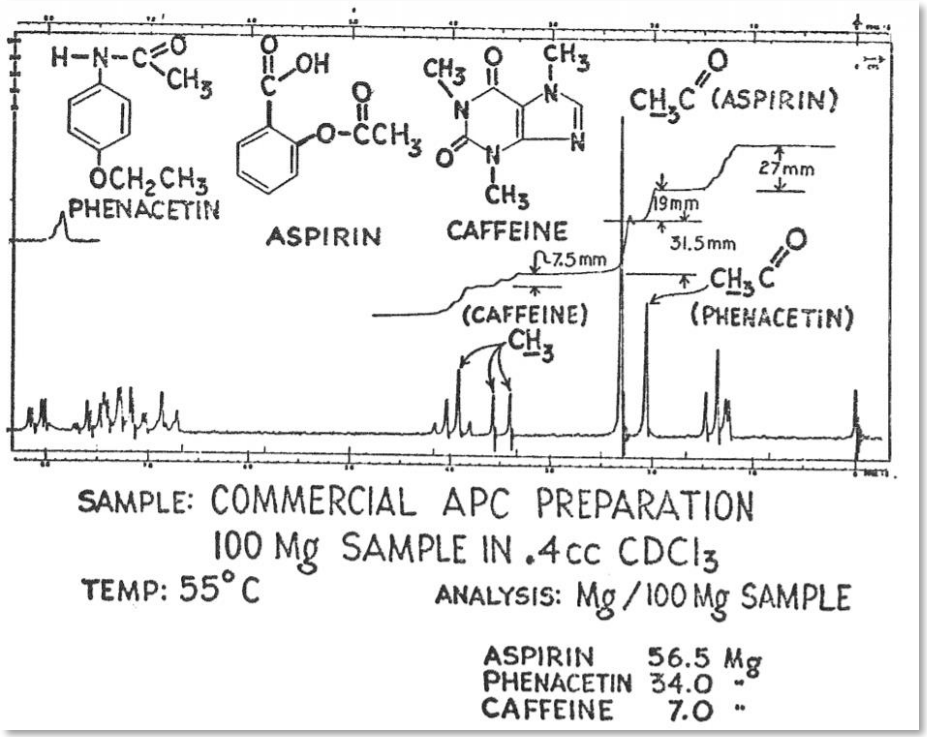
SMA to perform purity – *DEMO*

Automated Purity – *DEMO*

Questions

The fundamentals are well known

J Shoolery, "A Basic Guide to NMR", 1972



Internal reference method

Test sample known compound(s)' weight(s) known
→ purity to be determined

Quantitation Reference Material

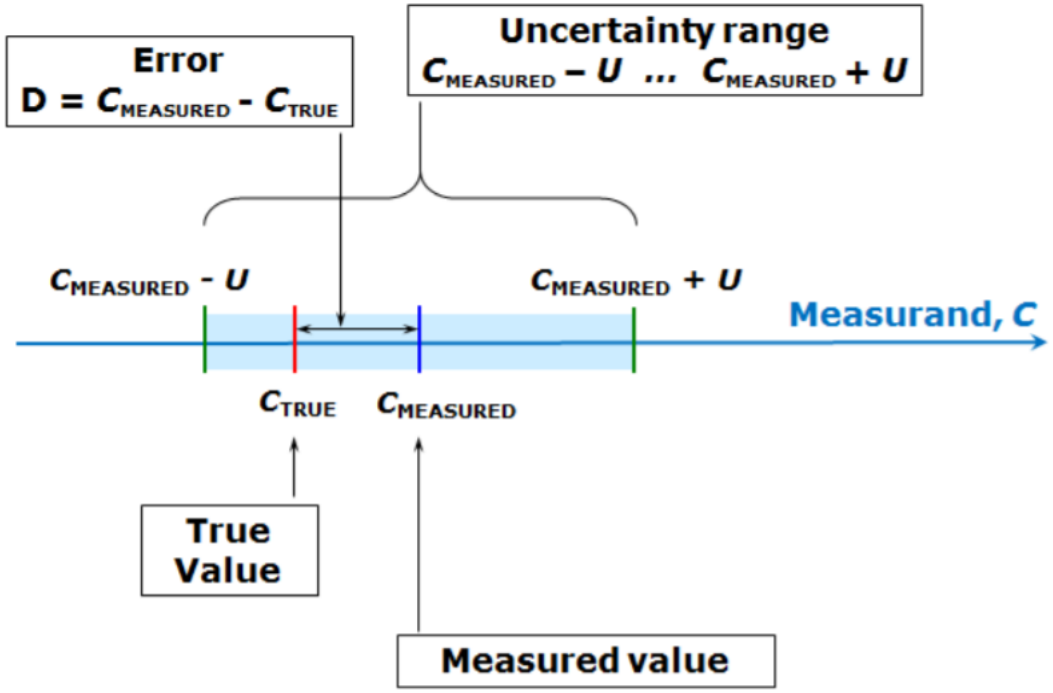
known purity

- no overlapping signals in a given matrix
- precisely weighable (non-hygroscopic / non-volatile)
- chemically inert with respect to the solvents used

and the matrix analyzed

Weber, M.; Hellriegel, C.; Rueck, A.; Wuethrich, J.; Jenks, P. *J. Pharm. Biomed. Anal.* **2014**, *93*, 102–110.
YouTube: "Basics for qNMR - 2018 Webinar Series", John Gauvin, DSM

Are you certain about uncertainty?

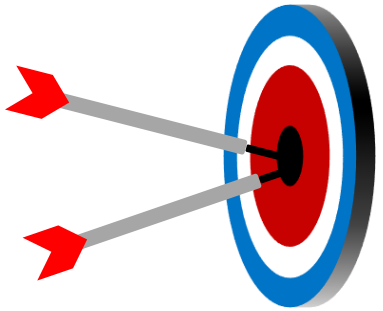


Scheme 1.1. Interrelations between the concepts true value, measured value, error and uncertainty.

I. Leito et al., Estimation of measurement uncertainty in chemical analysis (analytical chemistry) course

Complete uncertainty analysis:

- Trueness
- Precision
- Selectivity
- Limit of detection
- Limit of quantification
- Linear range
- Working range
- Robustness
- Measurement uncertainty



Malz, F., & Jancke, H. (2005). Validation of quantitative NMR. **J Pharm Biomed Anal**, *38*(5), 813–823.

How accurate and precise do you need your qNMR result?

“High performance qNMR” ($u < 0.5$)

Accurate purity ($u < 2.0$)

Standard purity ($u < 5.0$)

“Quantitative” NMR spectrum

Full peak integrals

Good peak-shape

Signal-noise-ratio (SNR)

Time between excitation pulses (“relaxation delay”)

Temperature control of the sample

Shimming

Total data acquisition time (number of scans)

http://resources.mestrelab.com/qnmr-purity-recipe-book-ii/

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Spectrum processing

Apodisation/Line-broadening

improves peak shape at base (“wiggles”)

Data points for FT (zero filling)

for effective integration

Phase correction

for effective integration

Baseline correction

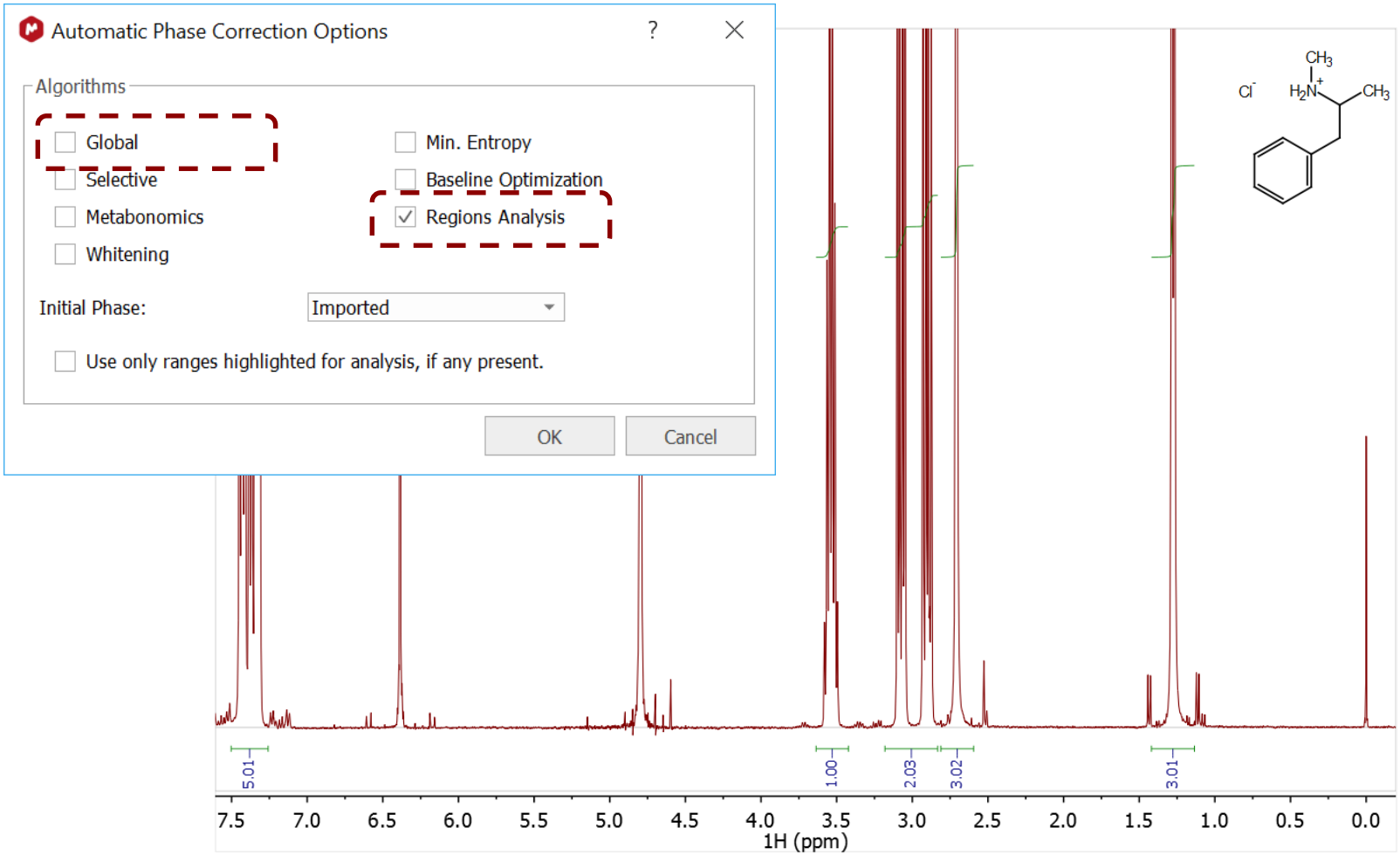
for effective integration

Potentially large error source

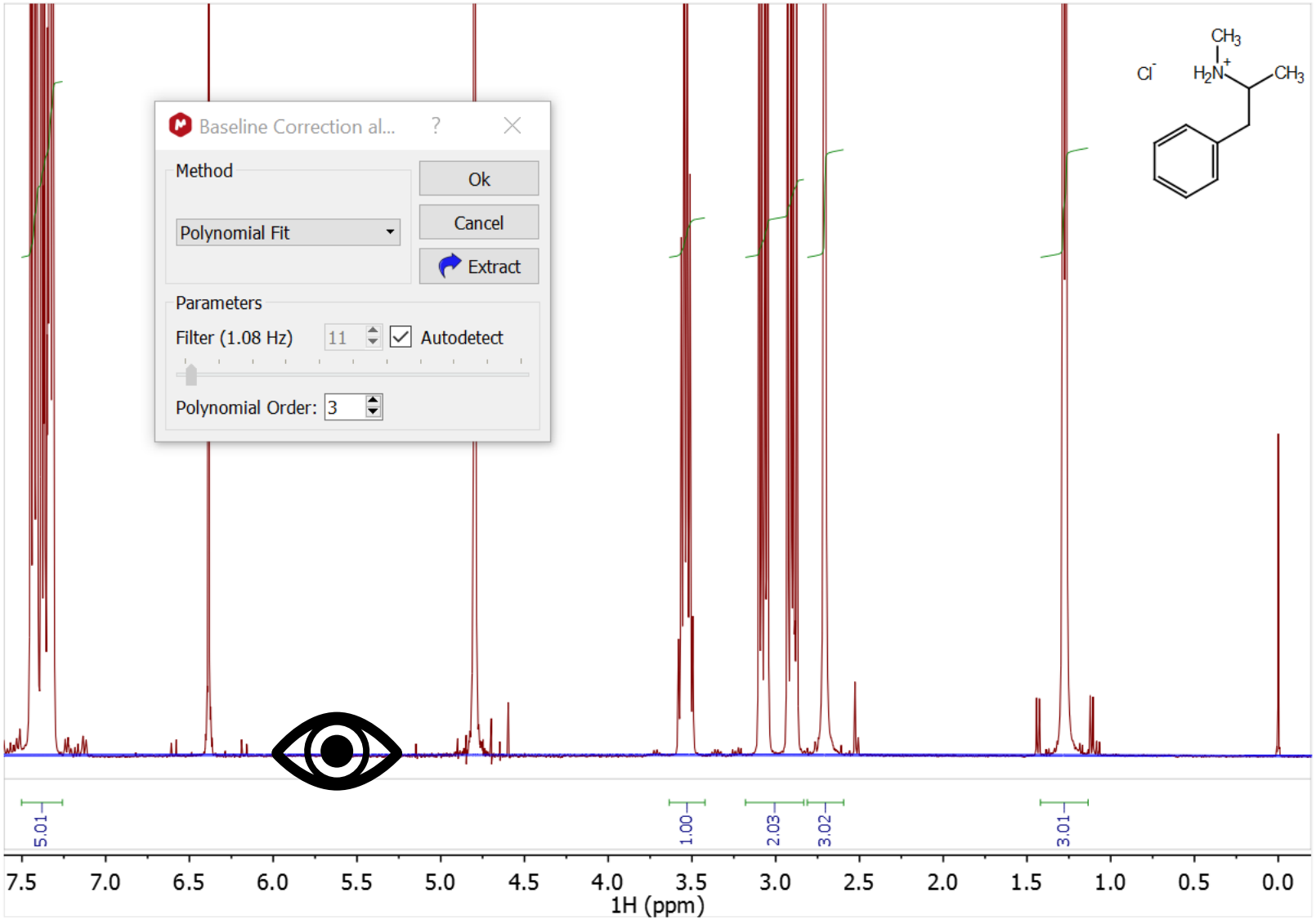
“Processing and analysis in Mnova 12. focus on requirements for qNMR”
https://www.youtube.com/watch?v=cctl2I2XjwQ

"Regions" autophase

Automatic phasing can provide consistency, and often outperform manual phasing

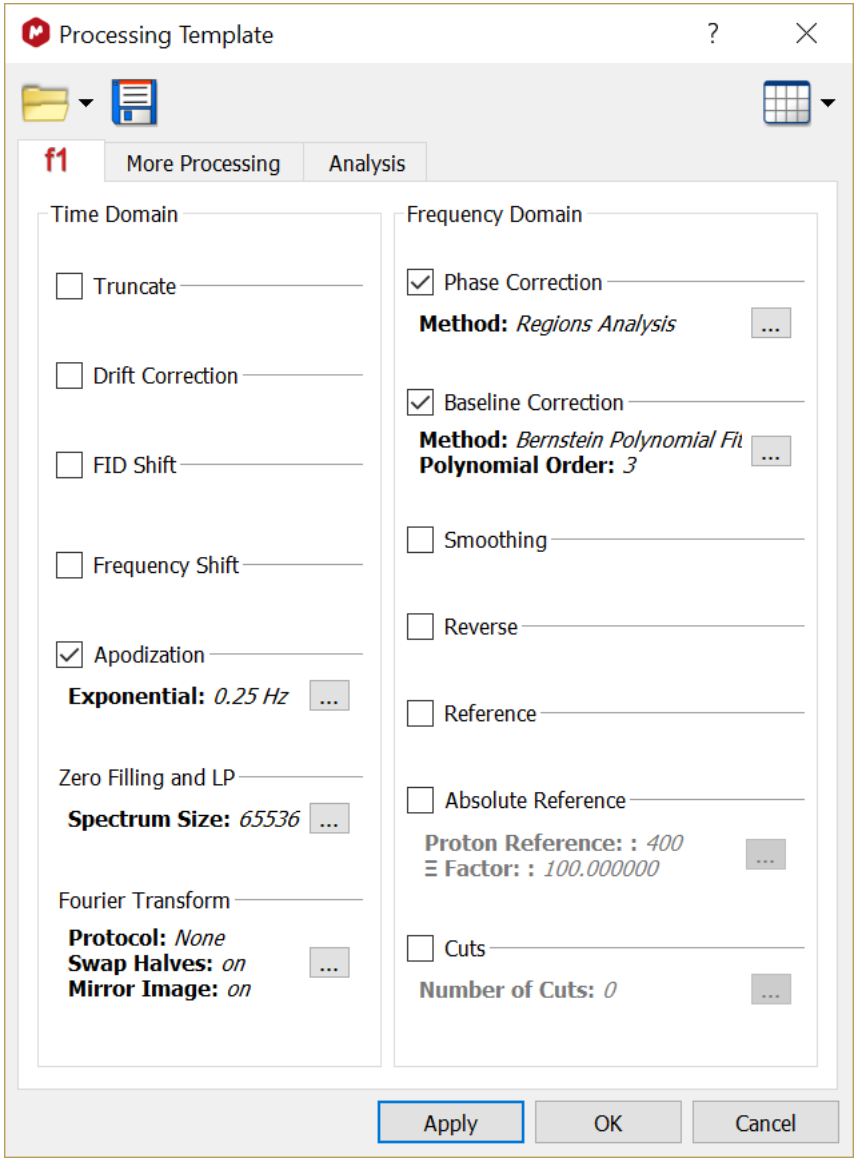


Polynomial baseline correction



Processing templates

Use this for *consistency*



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Integration method – IMPORTANT!

- **GSD** – good when overlap or close multiplets, but precision/accuracy is worse
- **Sum** – this is the "standard" method
- **qGSD** (≥Ver 12.0) – the accuracy of Sum, and handles close or overlapping peaks
- **Edited sum** – improved Sum

Mnova qNMR uses:

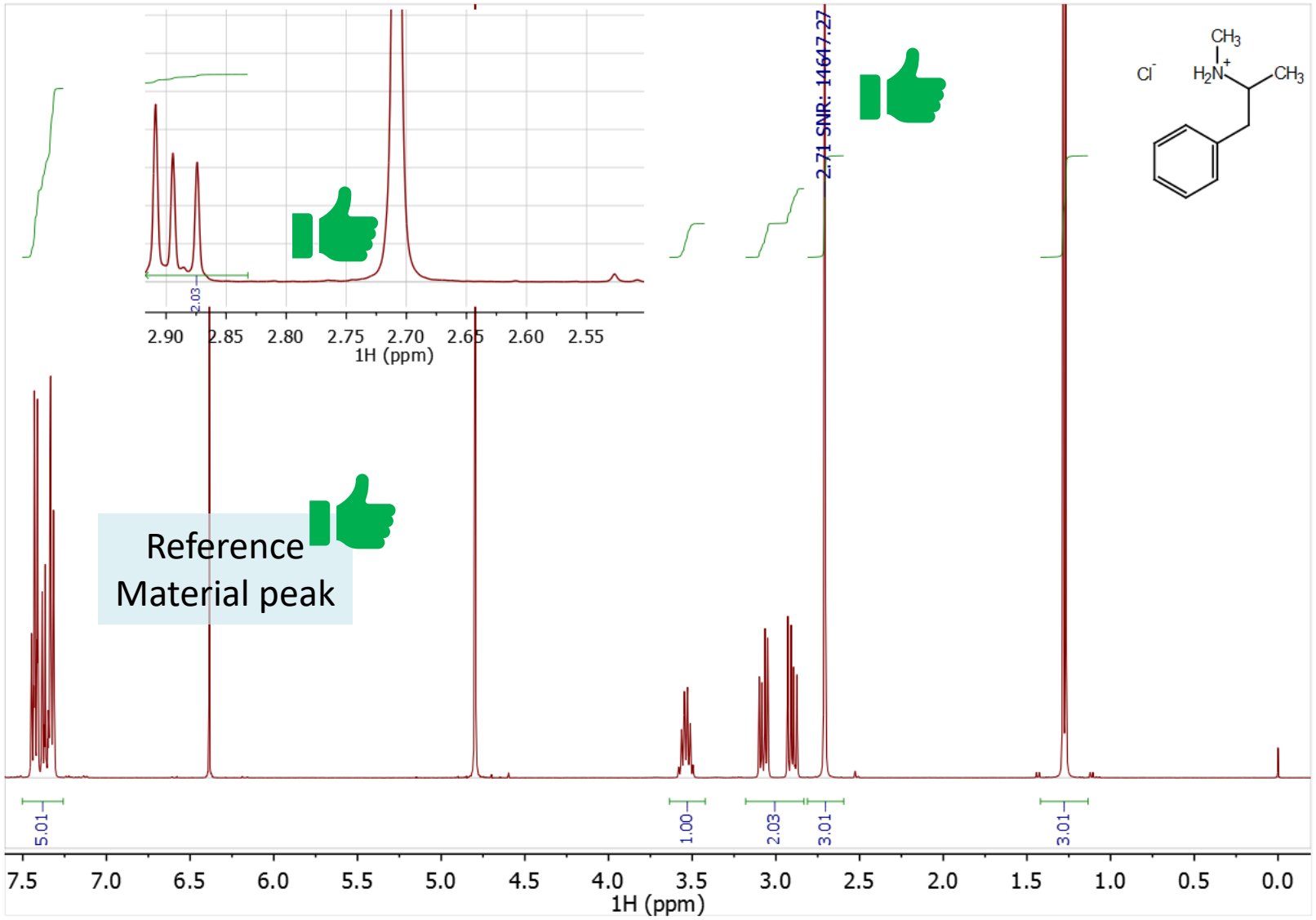
Standard integration → for the reference peak(s)

Multiplets integration → for the solute multiplets

M Bernstein, "Processing and analysis in Mnova 12. Focus on requirements for qNMR"

Spectrum acquisition

Good



Desirable but not totally necessary

Chemical structure of analyte

Better nuclides determination (AutoAssign)

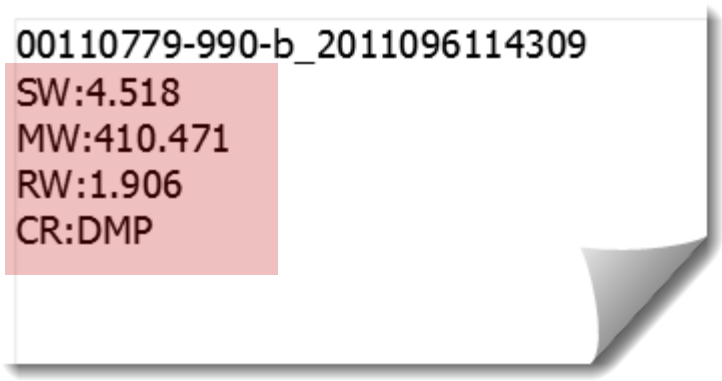
Better peak “types” (impurity, etc.)

Sample metadata in the title

Good laboratory practice

Transcriptional errors ↓

Metadata co-exists with the data – for ever



How does Mnova do Purity qNMR (1-3: Purity plug-in)?

Reference materials' essential data are stored (and can be shared)

Sample spectrum processing templates

"One off" new sample analysis **(1),** or fixed SOP "experiment" **(2)**

Replicates and repeats **(3)**

Mnova will select the best multiplets for quantitation

- How many to select and average
- Selection "rules"

Multiplet integration limits are automatically set

Nuclides *per* multiplet are determined (→"Normalized" integrals)

Automatic selections can be manually changed

- Nuclides
- Integral range

DEMO

How does Mnova do Purity qNMR (4: SMA)?

A flexible, quantitation tool that you can tailor to your needs

1D and 2D regions integration

Automatic equation generation, including qNMR Purity

DEMO

How does Mnova do *Automatic* Purity qNMR (5: Batch qNMR)?

Based on conventional Mnova qNMR Purity plugin

Mgears* automation-

- Input – batch, listener, DB, command line interface
- Processing file
- Analysis, e.g. Purity
- Output format
- Output file(s) to be written

DEMO

*Mnova Ver 12.0.2 and higher

Conclusions

Mnova has strong, “basic” processing and analysis capabities

Purity analysis can be easily and reliably performed

- single sample
- repeats or replicate
- prescribed analysis

More complex quantitation is performed using SMA

Full-automated operation can be performed

- e.g., JEOL resonance “qNMR Seamless”