

## No Need to PANIC



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### Overview of the Newsletter

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- ValidNMR Workshop (March, 2018)  
Summary and Feedback
- Validation Flowchart
- Kim Colson: Goodbye to Bruker &  
ValidNMR
- qNMR Summit in Würzburg (Oct, 2018)



# ValidNMR Workshop - Summary (March 8, 2018)

On March 8, 2018, approximately 70 people convened at the Validation Workshop to discuss an overall NMR Validation process as well as requirements according to USP, FDA and GxP, recapped the progress of the qNMR wiki and learned from various presentations.

The Validation Workshop takes place following the PANIC Conference each year. This year, we were very excited to have a turnout of about 70 people, which represents an increase from the previous year. We began the day with opening statements from both Kim Colson (Bruker BioSpin) and Torsten Schönberger (Bundeskriminalamt).

Elina Zailer (University of Würzburg, Spectral Service) was presented as the second recipient of the Bruker Fellowship for Excellence in NMR Validation, presented by PANIC annually. Elina presented the basics of improving qNMR results. Carlos Amezcua presented qNMR Method Validation Following the USP 1210 Chapter, Statistical Tools for Procedure Validation. Informative presentations on qNMR were given by Bernd Diehl (Spectral Service), Dan Sorensen (Eurofins|Alphora Research Inc.) and Ian Burton (NRC). The results of the  $^1\text{H}$ ,  $^{19}\text{F}$  and  $^{31}\text{P}$  NMR proficiency test was presented by Markus Obkircher (Merck KGaA). The presentation slides will be provided soon on our homepage [www.validnmr.com](http://www.validnmr.com).

Within the workshop we broke three times into two teams to address important topics include: validation wiki, validation parameters, data integrity, education, uncertainty analysis and reference materials. We learned how to use the qNMR wiki and statistics, we created a flowchart for the validation process, we thought about how to educate new and advanced NMR people and how to manage data and reference materials.

**We would like to thank all attendees for your engagement, your ideas and your innovations!**



The participants of the workshop "Statistical Analysis" by Michael Maiwald (BAM)



The participants of the workshop "Reference Standards" by Kevin Millis (Isotope)



Time and space for discussions with qNMR experts is very important for the Validation group

# ValidNMR Workshop - Feedback (March 8, 2018)

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## What have we learned?

- Our group consists of 9% beginners, 77% intermediate and 14% experts (our dinosaurs ☺)
- Validation workshop is important for exchanging the knowledge and experience in the qNMR field
- NEW: one scan for quantification by using the solvent as the internal standard ( $^2\text{H}$ )
- Experiment: use at least 8 scans to minimize noise
- Statistical analysis:  $R^2$  does not confirm linearity! Other methods such as residues are required to determine the linearity
- We as a group will prepare flowcharts (instrument and method validation, data integrity, certifying reference standards) which will be presented to industry, authorities, universities, etc.
- To get more input, we will publicize the NMR wiki by publishing articles about it in scientific journals and other platforms such as the ValidNMR homepage (validnmr.com)
- Please provide collecting materials and further information to Elina (elina@validnmr.com). We will find a way to provide these documents to the Validation workshop group, without making it public to everyone!

## What is required? What should we change?

- Even smaller tutorial groups
- Task for the workshop should be more clearly defined
- Statistical analysis is very important for evaluating huge qNMR data sets, however knowledge and experience are required, but not present → where can we get it?
- Demand of flowcharts (instrument and method validation, data integrity, certifying reference standards)
- Demand of better education:
  - live spectra evaluation at PANIC booth (Bruker, ACD, Mestrelab) → competition and prize
  - Providing workshop “step-by-step spectra evaluation for beginners” → who would like to teach? Where?
  - Proficiency / Interlaboratory tests for spectra evaluation (data should be available online to download → validnmr.com as platform?)
  - Statistics & Chemometrics
- Make clear which requirements are important for the company we work for such as GxP, ISO, etc.
- Defining NMR language (<http://www.validnmr.com/w/index.php?title=Nomenclature>)
- More explanations required for working with the NMR Wiki → Kristie Adams
- We should work more closely with developers and vendors of NMR spectra evaluation software to demonstrate our need and challenge as NMR experts
- Required: “private” platform for important NMR documents and presentations which are available only for the validation group



# New Validation Flowchart

## Instrument Validation

A qualified instrument is a prerequisite for the whole procedure

Documents provided by vendors (SQ, IQ, OQ)

Customer specific requirements (PQ)

The validation workshop made clear that there is a need to discuss the "validation" process in order to define a standard procedure for a qNMR validation. Within the workshop a first flowchart was created by the attendees and especially **Stefan Garms (Lonza)**. This flowchart will be the basis for the standard validation procedure which will be further developed by the validation group.

**You want to be part of the discussion round?**

Then, we can offer you two opportunities:

1) The flowchart will be also published on the wiki. There, you can give your opinion on the discussion platform.

2) Send your opinion and your ideas to Elina (elina@validnmr.com). Elina will forward your mail to the validation core group.

## Method Validation

**Independent purpose of use?**  
1) Analytical task  
2) Regulated environment

**Method Development**

**Method Validation**  
according to required standards (GxP, ISO, ...)

**Sample preparation**  
Internal / external standard, amount, solvents, ...

**Instrument parameters**  
Type of experiment, number of scans, D1, AQ, TD, ...

**Validation parameters**  
according to ICH / USP  
e.g. LOD, LOQ, precision,...

**Additional validation parameters (if required)**  
e.g. robustness after extraction

**Statistical Tools**

see next page

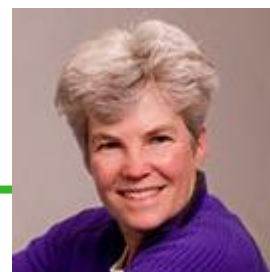
# Validation parameters



Type of Method	Limit Test	Identity
Repeatability		
Intermediate Precision		
Specificity		
Accuracy		
LOD/LOQ		
Stability		



## Goodbye (by Kim Colson)



Dear ValidNMR Community,

After 20 years at Bruker, it is time for me to move on. I will leave Bruker on May 18th, 2018 and step down from my role in ValidNMR.

Through my experience at Bruker, I've had the opportunity to be part of product, application and business development for both software and hardware products. With these roles came the privilege of working with a tremendous number of highly talented and enlightening people throughout the world. I will treasure this forever. My path headed me towards the highly motivated and energized community that we named the ValidNMR. It's been a pleasure to be part of this community and help it establish and move towards its goals. I am certain ValidNMR will succeed at its mission of NMR acceptance and commonality in regulatory environments. This will have significant impact reaching far beyond the NMR community. Thank you for the opportunity to co-lead the ValidNMR community. It has been a highlight of my career that I hold dear. The ValidNMR committee will continue to lead the efforts that we started and continue its progress. I wish all of you the very best in your efforts moving forward.

Sincerely,

Kim Colson



# SUMMIT 2018

Julius-Maximilians-  
**UNIVERSITÄT  
WÜRZBURG**

**We invite you!**

Be part of the next qNMR summit in Würzburg (10-11 Oct 2018)  
and register here:

<https://www.uni-wuerzburg.de/index.php?id=203750>

## Preliminary Program

### First Day

- Registration
- Plenary Lecture on Regulatory Affairs in qNMR
- Poster Sessions and Workshops (e.g. Software Presentations, Software News)
- Sessions on Regulatory Affairs (GMP in qNMR, qNMR at EDQM and in Pharmaceutical Companies, Panel Discussion)

### Second Day

- Plenary Lecture on Pure Shift NMR
- Flash Presentations (Protocols for Measurement and Processing)
- Open Space Discussion (e.g. on Round Robin Test)
- Final Discussion

### Location

Institut für Pharmazie und Lebensmittelchemie  
Am Hubland  
97074 Würzburg

**Want to be featured in the next ValidNMR newsletter?**

The deadline for submissions and contributions to the next newsletter is June 15, 2018.  
Please contact us at [committee\(at\)validnmr.com](mailto:committee(at)validnmr.com)!



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