

**VALIDATION  
TRACEABILITY  
MEASUREMENT UNCERTAINTY  
CHALLENGES FOR THE 21<sup>ST</sup> CENTURY'S ANALYSTS**

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Workshop group 1.1:  
**Which guidance is needed for planning and performing a good Method Validation Study?**

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## WG 1.1 questions

- Which of the existing guidelines on Method Validation has been used mostly?
- Are any of these guidelines sector specific (e.g. food analysis, environmental analysis etc.) – or are they mainly generic in their approach?
- Which phase in a method validation process is found most difficult to handle and accomplish?
- Is the extent and details of a method validation study always clear before commencement of the practical work in the laboratory?
- Are there any specific needs regarding establishing the protocol for a method validation study?
- Is establishment of Traceability and estimation of Measurement Uncertainty seen as an intrinsic part of a Method Validation study?
- Are examples of method validations studies (described in details) seen as useful – or as a limitation (e.g. because the actual example cannot be used directly)?
- Which specific topics could be relevant to include in a revised Eurachem Guide on Method validation?



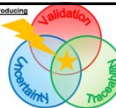
### a) Which of the existing guidelines on Method Validation has been used mostly?

- NB: Distinction between validation and verification!
- Eurachem guideline
- IUPAC guideline
- Basis: ISO 5725
- Air: Eurachem was used, now looking at best method. Question: include sampling? → broader issue!
- In-house validation procedures (comparable with Eurachem guideline)
- Handbook for validation for Nordic countries (NMKL)
- EA 04/16, chapter 6 (on MU estimation based on validation data)
- ISO water standard



**a) ...cont.**

- SANCO/12495/2011 (Met. Val. & QC for pesticides in food and feed)
- Various EU Regulations and Commission decisions (give criteria, no specification of procedure) – e.g.:
  - CD 2002/657/EC (general on met. performance and interpr. of results)
  - 401/2006 (Mycotoxines) (+ Ammendm. 178/2010)
  - 333/2007 (Metals + MCPD and Benzo(a)pyrene in foodstuffs)
- National documents – e.g....
  - Ukraine, Russia (recommendations)
  - Guidelines Dutch accreditation Body (RvA)
  - NEN (Dutch standards: NEN 7777, 7779)
- Books:
  - J.O. Westgard
  - Kromidas (German)
  - J. Klaessens



**b) Are any of these guidelines sector specific (e.g. food analysis, environmental analysis etc.) – or are they mainly generic in their approach?**

- [see answers to question a]

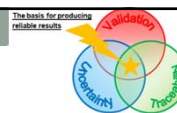
### c) Which phase in a method validation process is found most difficult to handle and accomplish?

- Recovery; especially organic compounds (especially related to correcting for recovery of internal standards)
- Kind of matrix (Example: synthetical fuels compared with mineral fuels, aflatoxine testing in 1 type of nuts usable for all nuts?).
- Difference between an official standard method and an in-house method
- 1 (one!) procedure for establishing LOD, LOQ etc.
- Where to find the criteria for the results of the validation study?
  - (annex of ISO e.g.. Are they useable?)



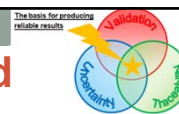
### c) ...cont.

- Test under repeatability of reproducibility conditions?
- Number of "operators" required for establishing reproducibility (min. 3?)
- Number of tests to be performed in the validation study
- Calibration study, validation on several levels over the whole range of concentrations
- Definition of "range of the test" (minimum, maximum)
  - Can the maximum be overcome by dilution?



**d) Is the extend and details of a method validation study always clear before commencement of the practical work in the laboratory?**

- [partly answered under the previous questions!]



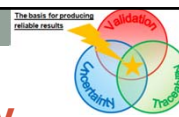
**e) Are there any specific needs regarding establishing the protocol for a method validation study?**

- An in-house protocol is required, can be a general procedure in case of routine testing
- Examples of good protocols



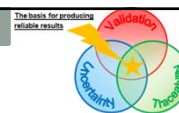
**f) Is establishment of Traceability and estimation of Measurement Uncertainty seen as an intrinsic part of a Method Validation study?**

- "Model equation" versus "use of validation data" for calculating measurement uncertainty



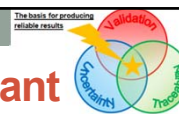
**g) Are examples of method validation studies (described in details) seen as useful – or as a limitation (e.g. because the actual example cannot be used directly)?**

- Yes!



## h) Which specific topics could be relevant to include in a revised Eurachem Guide on Method validation?

- Harmonization of ISO, EN guidelines and Eurachem, IUPAC guidelines
- Software for Method Validation: make reference to in new Eurachem guideline.
- Guideline must answer the question: when is re-validation required?
- Outcome of validation: best way to perform quality control
- Initial validation and ongoing validation (QA/QC)



## Additional subjects discussed

- [Nothing in addition to subjects under previous sections]

