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METHOD VALIDATION IN ANALYTICAL SCIENCE
Current Practices & Future Challenges
WORKSHOP - 9&10 MAY 2016 - GENT - BELGIUM

WG 2 day 2

Working range – from LOD/LOQ to upper limit of the measuring range

Convenor:

Lorens Sibbesen

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METHOD VALIDATION IN ANALYTICAL SCIENCE
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- How do you define the “working range” of the method?
  - Is there a difference between “method working range” and “instrument working range”?
- What are the different approaches applied in different fields to establish the “working range”?
- When can dilution of high concentration samples be applied to justify an expanded method working range?
- Is linearity of a method working range crucial for the validity of the method?
- What are the documents available for guidance?
- What are the challenges experienced in different areas?

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METHOD VALIDATION IN ANALYTICAL SCIENCE
Current Practices & Future Challenges
WORKSHOP - 9&10 MAY 2016 - GENT - BELGIUM

- Participants in WG (28)
- Countries
  - UK
  - Austria
  - Ireland
  - Belgium
  - Czech Republic
  - Turkey
  - Iceland
  - Spain
  - Ireland
  - Iran
  - Germany
  - Norway
  - Sweden

METHOD VALIDATION IN ANALYTICAL SCIENCE
Current Practices & Future Challenges
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- Participants in WG
- Fields of activity
  - Petrochemical
  - Training on QA related topics
  - X-ray fluorescence
  - Water analysis
  - Research
  - Agrochemistry
  - Accreditatipon
  - Marine analysis
  - Forensics (drugs/alcohol)
  - Beverage testing
  - Medical labs.
  - Feed analysis
  - Product testing (consumer protection)
  - Human toxicology (pathology)
  - Commonwealth Ph. methods
  - Nuclear research
  - Consultancy
  - Mycotoxins
  - Pharmaceuticals
  - PU tests
  - Spectrochemical analysis

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Output from discussions 1

- **How do you define the "working range" of the method?**
  - Is there a difference between "method working range" and "instrument working range"?
- From LOQ to upper end of linear range
- Working range defined in standard methods
- Region where we expect to do reliable measurements
  - i.e. with acceptable recovery and precision
- From LOQ to "what is needed" (high levels not needed)
- Depends on specification/limit values for toxicology
  - depending on the toxicity of the actual component
- For residues in water both low and high end is important.

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Output from discussions 2

- **What are the different approaches applied in different fields to establish the "working range"?**
- Problems in covering a very wide range of levels for micronutrients in fertilizers (dilution after extraction)
  Must be covered in the validation study
- Issue of one dominant metal component (Gd) in samples where metal impurities are also of interest
  - dealt with in two steps (for the preparation)
- Some parameters in a sample may not be measurable after dilution (done for the sake of other parameters in the sample)
  - need for splitting up samples
- Woking with different calibration ranges
- Regulatory limit(s) must be included in the working range
  - maybe not possible!
- Forced to "dilute" to get enough sample material for several measurements
  - May be a problem for low level analytes

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Output from discussions 3

- **When can dilution of high concentration samples be applied to justify an expanded method working range?**
- Use of internal standards to point out any problems
  Expected to react in the same way as the analyte
  Used as an indication of any problems!
- Different MU for low and high range values
  Diffent values of MU for non-diluted and diluted samples
- Important to include any dilution steps in the validation
- Calibration of glassware may be crucial in some fields

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Output from discussions 4

- **Is linearity of a method working range crucial for the validity of the method?**
- Linear or just well characterized?
- Uncertainty documented over the whole range is important
- Examine the working range by 1 – 2 reference materials (+ spikings) to document the uncertainty on results within the whole range
- Not necessarily any direct relation between results on low level and on high level

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Output from discussions 5

- **What are the documents available for guidance?**
- [none!]

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Output from discussions 6

- **What are the challenges experienced in different areas?**
- [Dealt with under question 2]