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A Focus for Analytical Chemistry in Europe

Workshop
Method Validation in Analytical Sciences
Current practices and future challenges

Gent, 9-10 May 2016

Report from WG 4



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**Challenges in validation of
multiparameter methods**

- *Securing that a method is valid for a wide range of analytes*

- Moderator:
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Questions

- In what fields are multiparameter methods applied?
- Which approaches are applied for their validation?
- How to deal with requirements for sums of parameters (e.g. for LOD/LOQ)?
- How do you decide about the extent of validation needed?
- What are the documents / protocols available for guidance?
- What are the challenges experienced in different areas?



In what fields are multiparameter methods applied?

- Pesticides, biocides
- Food analysis (“unknown” matrices)
- Environmental analysis (contaminants)
- Feed additives, amino acids, organic acids
- Drug residues
- Water analysis
- Microbiology
- Multivariate calibration in pharmaceutical analysis



Which approaches are applied for their validation?

	Pesti- cides, biocides	Pharma- ceutical analysis	Food & environmen- tal analysis	Feed additives, amino acids, organic acids	Drug residues	Water analysis
Identification test	x					
Quantitative test for impurity		x	x			x
Limit for impurity			x			x
Quantificatio n of main components		x	x	x		x
Quantificatio n of minor components	x	x	x	x	x	x



How to deal with requirements for sums of parameters (e.g. for LOD/LOQ)?

- Guidelines for summing parameters are given in different legislation e.g. water directive, pesticides, vet drug residues.
- Challenges: level at which sum is considered non-compliant may need to be calculated each time a positive sample is found.
- MU may also need to be calculated each time.



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How do you decide about the extent of validation needed?

Performance characteristics	Pesticides, biocides	Pharmaceutical analysis	Food & environmental analysis	Feed additives, amino acids, organic acids	Drug residues	Water analysis
Selectivity						
LOD						
LOQ						
Working range incl. linearity						
Trueness/bias						
Precision (repeatability and intermediate precision)						



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What are the documents/protocols available for guidance?

- NEN7777 (In Dutch, Method validation - practical guide; NEN7779: How to calculate the MU)
- ISO, SANCO, 2002/657EC, Water Directive, ICH, IUPAC, Eurachem guide



What are the challenges experienced in different areas?

- Lack of certified reference materials
- Lack of proficiency tests
- Validation in multiple matrices
- Stability of reference materials, calibration standard solutions, samples and sample extracts.
- Different permitted limits in different species.
- Cost and time