Challenges in validation of multiparameter methods

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

- Moderator:
  - Elin Gjengedal, Norwegian University of Life Sciences, Norway
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?

<table>
<thead>
<tr>
<th>Identification test</th>
<th>Pesticides, biocides</th>
<th>Pharmaceutical analysis</th>
<th>Food &amp; environmental analysis</th>
<th>Feed additives, amino acids, organic acids</th>
<th>Drug residues</th>
<th>Water analysis</th>
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</thead>
<tbody>
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<td>x</td>
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<tr>
<td>Quantitative test for impurity</td>
<td>x</td>
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<tr>
<td>Limit for impurity</td>
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<td>x</td>
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<tr>
<td>Quantification of main components</td>
<td>x</td>
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<tr>
<td>Quantification of minor components</td>
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</table>

### 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.
How do you decide about the extent of validation needed?

<table>
<thead>
<tr>
<th>Performance characteristics</th>
<th>Pesticides, biocides</th>
<th>Pharmaceutical analysis</th>
<th>Food &amp; environmental analysis</th>
<th>Feed additives, amino acids, organic acids</th>
<th>Drug residues</th>
<th>Water analysis</th>
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<tbody>
<tr>
<td>Selectivity</td>
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<td>LOD</td>
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<td>LOQ</td>
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<td>Working range incl. linearity</td>
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<tr>
<td>Trueness/bias</td>
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<td>Precision (repeatability and intermediate precision)</td>
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</tbody>
</table>

What are the documents/protocols available for guidance?

- NEN7777 (In Dutch, Method validation - practical guide; NEN7779: How to calculate the MU)
What are the challenges experienced in different areas?

- Lack of certified reference materials
- Lack of proficiency tests
- Validation in multiple matrices
- Different permitted limits in different species.
- Cost and time