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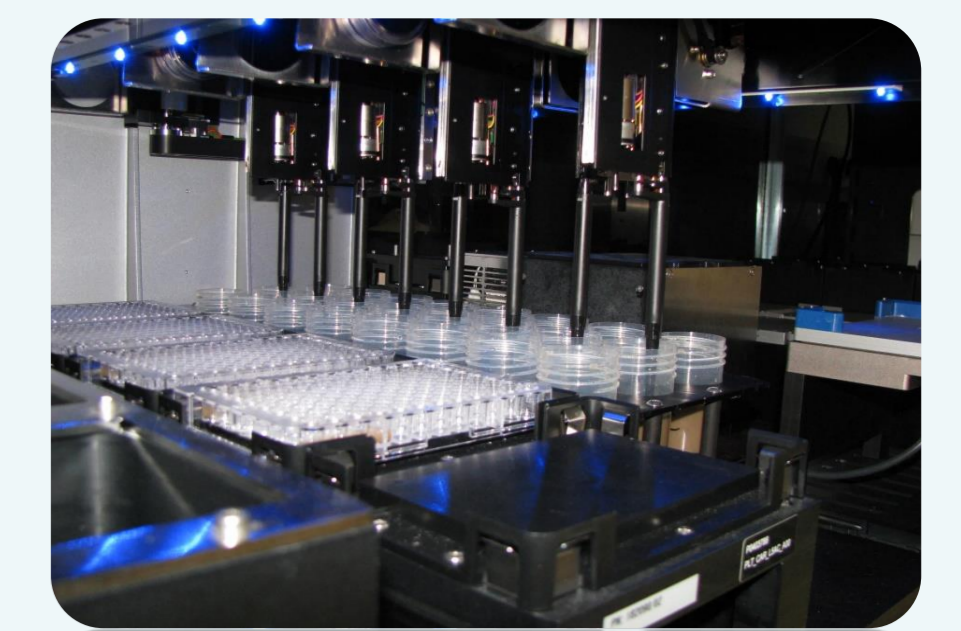
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THE VALIDATION OF ANALYTICAL METHODS AS AN EVALUATION TOOL OF RESEARCH RESULTS RELIABILITY

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Context

The platform Biochem-Env:

- Was created in 2012 by INRA (French National Institute for Agricultural Research) with the support of the ANR program "Investissements d'avenir" as a service of the infrastructure ANAEE-France,
- For the **biochemical characterization of natural environments** (soils and sediments) and **associated macrofauna** in research projects,
- By developing and validating methods in order to **provide traceable analytical data with high level of confidence**.

For intra-laboratory validation of quantitative analytical methods, the INRA's Quality Guidelines for research and experimental units (2013) recommends "the accuracy profile" method according to the NF V03-110:2010 standard.

Could we use a same internally developed method to quantify proteins in various biological models ?

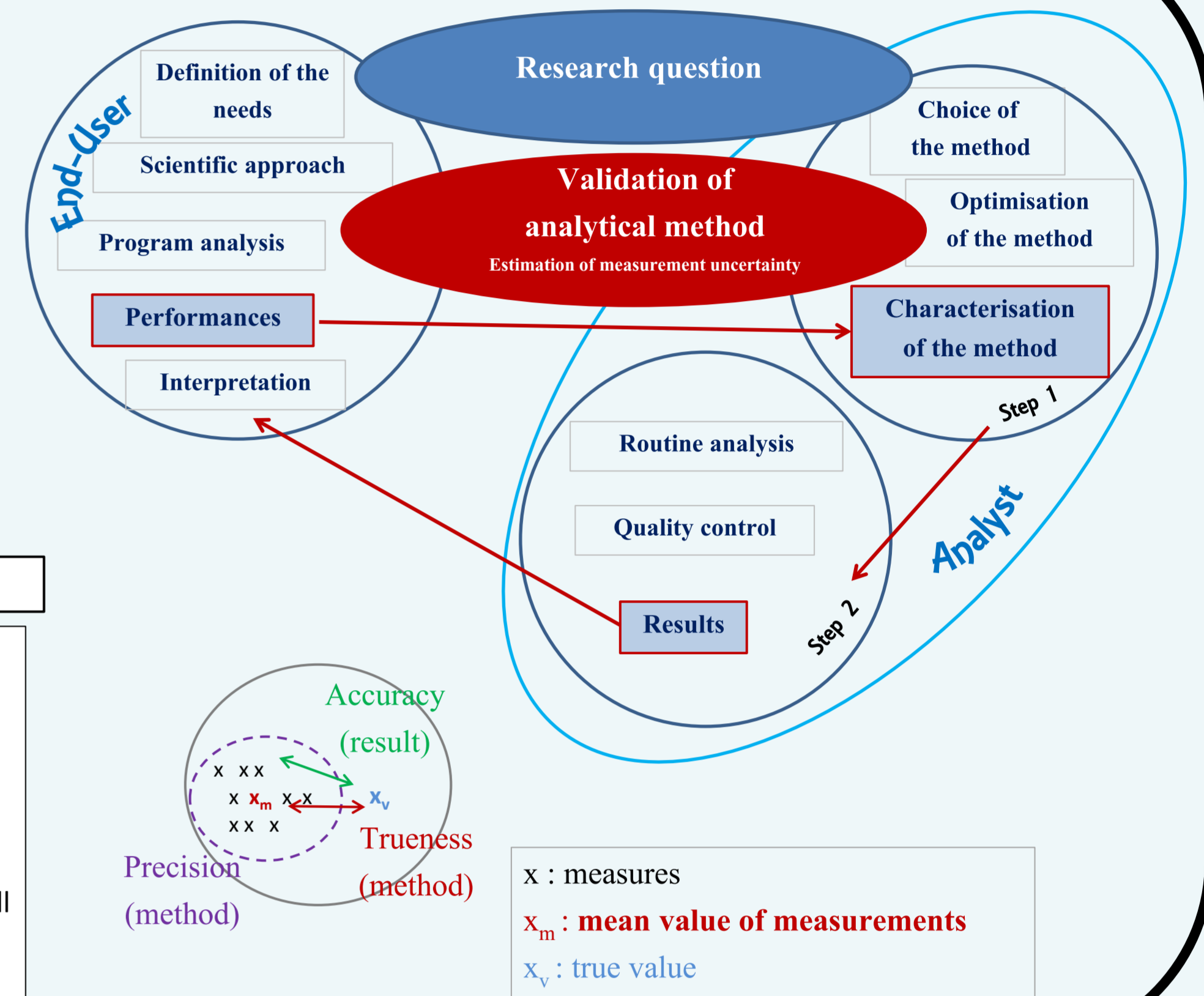
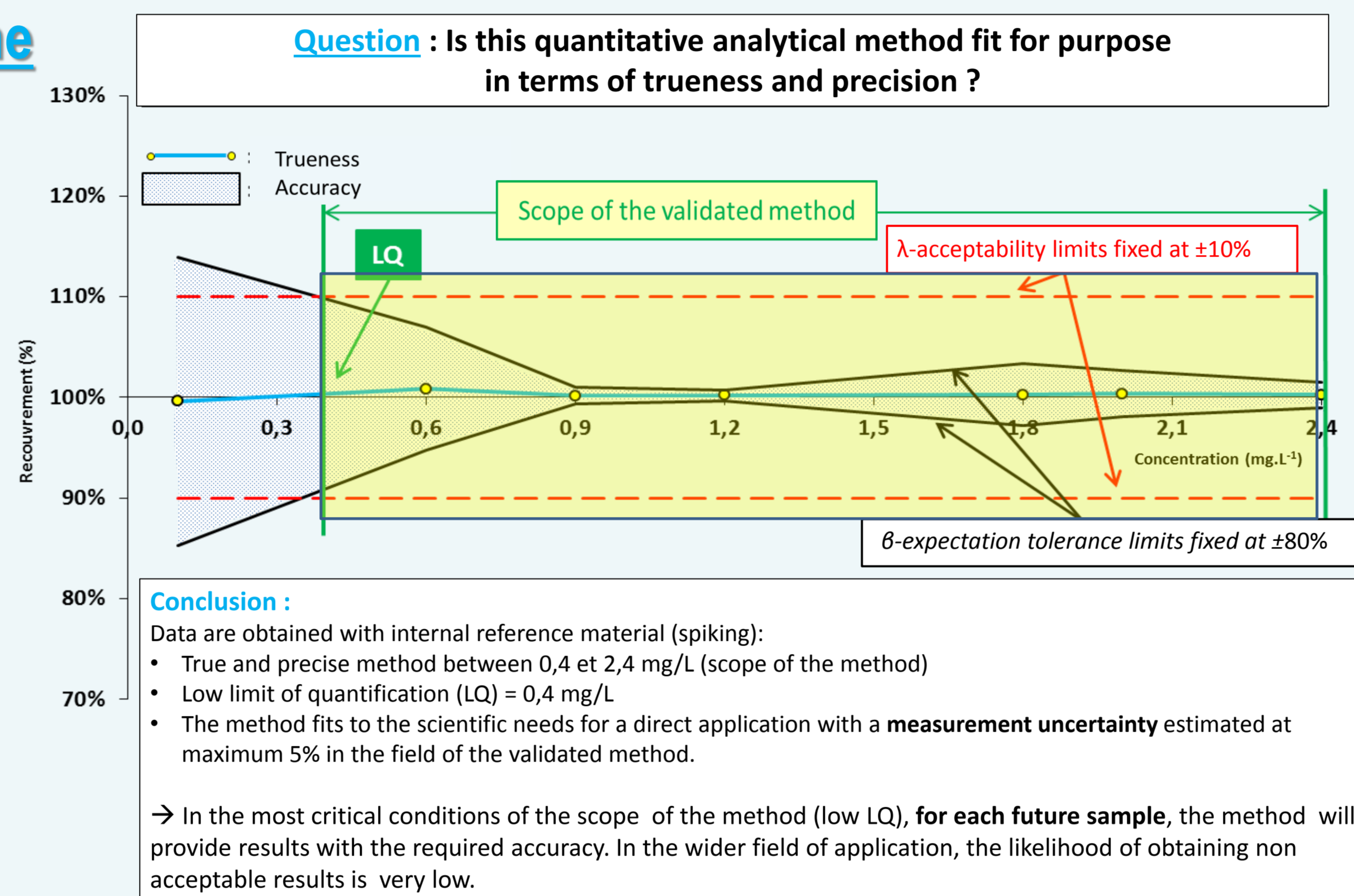
Validation of analytical methods by the accuracy profile approach

Purpose:

- To provide **guarantees on analytical results**, for the analyst and the end-user
- To demonstrate **analytical method fitting with the scientific objectives**
- To allow **laboratory recognition**
- To **improve analysts working practices**

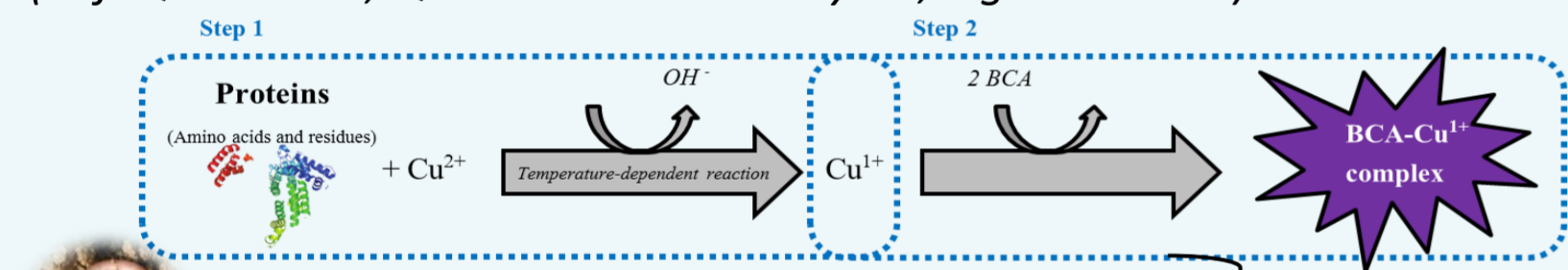
Benefits of the accuracy profile approach:

- An **overall statistical method** combining **trueness and precision**
- A **standardized approach** : NF V03-110:2010
- A **simple and graphic interpretation** for a **rapid decision**
- The **determination of the scope of the method**
- The **determination of quantification limits**
- An **estimation of measurement uncertainty**



Material and methods

Protein determination method by the Bicinchoninic acid (Ref. QPBCA-1KT, QuantiPro™ BCA Assay Kit, Sigma-Aldrich)

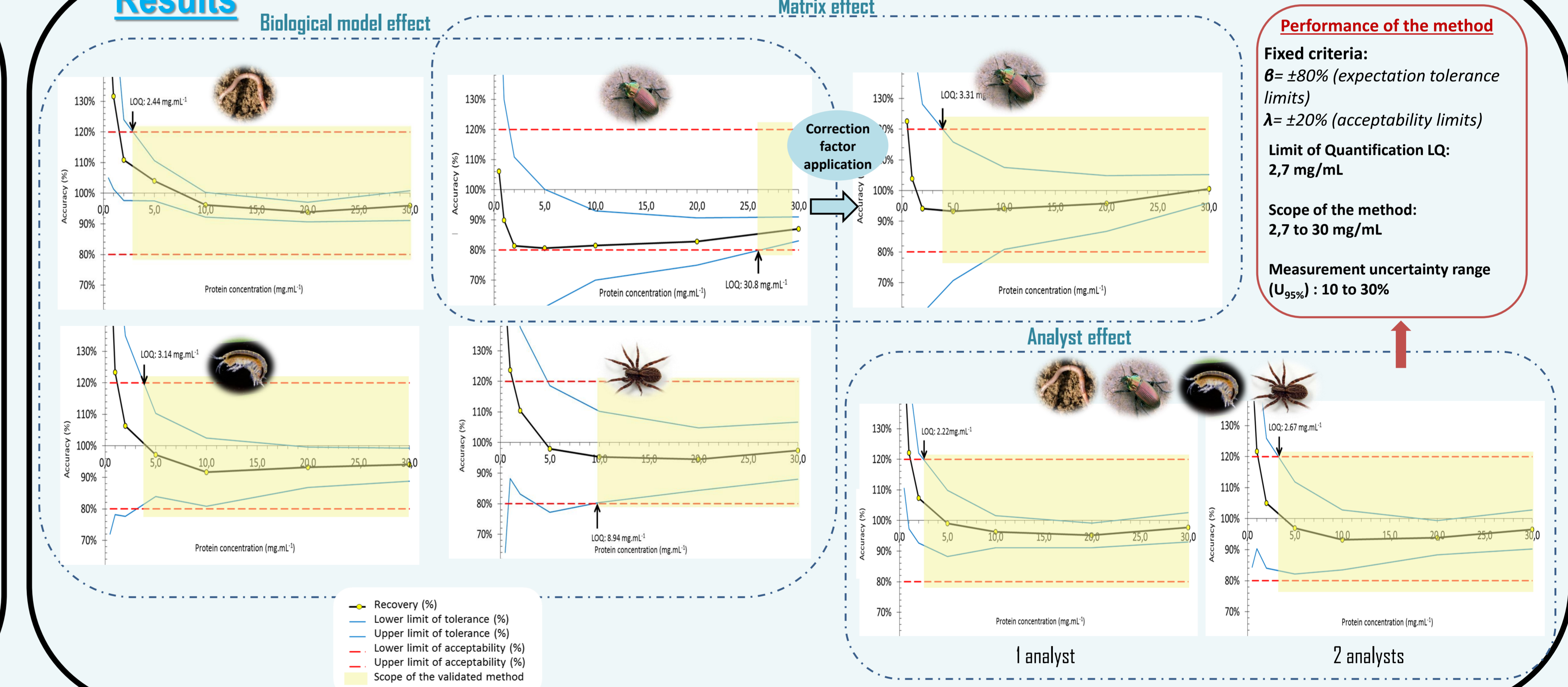


- 4 biological models:
- Aporrectodea icterica* (earthworm)
 - Poecilus cupreus* (beetle)
 - Gammarus pulex* (gammarid)
 - Alopecosa pulverulenta* (spider)

Validation method according to the « accuracy profile » approach (NF V03-110:2010 standard):

- Selection of validation samples:**
 - Spiked reference material: Bovine Serum Albumine
 - 7 levels (0.5 ; 1 ; 2 ; 5 ; 10 ; 20 ; 30 mg.mL⁻¹)
 - 3 replicates (repeatability)
 - 6 days of analysis (*intermediate precision*)
 - 2 analysts (*intermediate precision*)
- Calibration (indirect quantitative analysis):**
 - 6 levels (0 ; 2 ; 4 ; 8 ; 16 ; 20 ; 30 ; 40 mg.mL⁻¹)
 - Regression model : polynomial

Results



Conclusions

Accuracy profile approach:

- Global statistical combination of trueness and precision** and pragmatic
- Simple graphic interpretation**, allowing a clear and easy comparison between method performance and intended use and, a **rapid decision**
- No limit in the choice of the calibration model => **large scope range**
- Methods with very low variability can be validated (not rejected by a H0)
- Diagnostic tool**, matrix effect taken into account
- Risks and guarantees managed for both end-users and laboratories
- Estimation of measurement uncertainty**

The validation of this analytical method helped us to:

- Determine the performance of the method
- Improve the steps for sample preparation and analysis
- Assess the matrix effect
- Pointed out the importance of an experimented analyst

Decision on the fitness for purpose

- Analytical method for protein quantification in 4 biological models was validated with a good accuracy considering scientific specification and needs.**

Perspectives :

- Adapt λ -acceptability values according to the concentration range
- Extend the method to other biological models and biomarkers (Lipid, glycogen...)

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