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VALIDATION OF ANALYTICAL METHODS AS AN EVALUATION TOOL FOR RESEARCH DATA 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

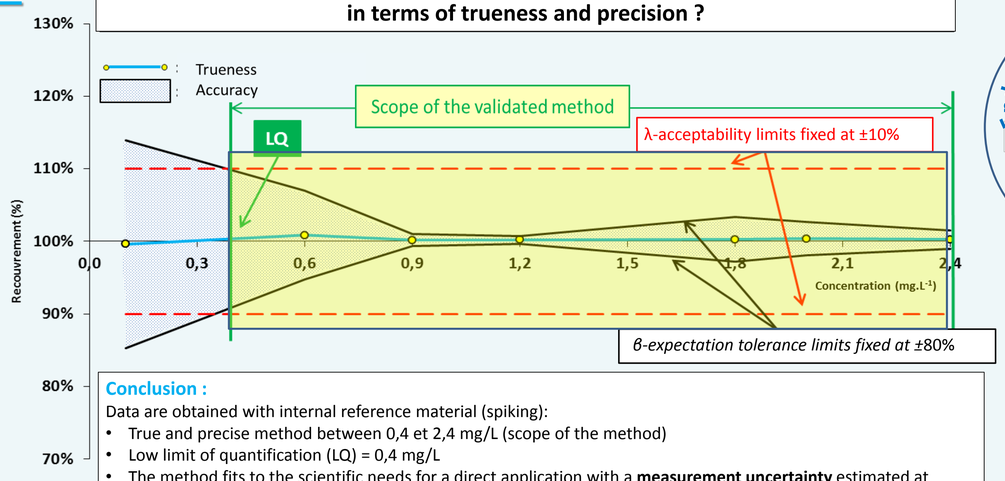
Purpose:

- 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

Question : Is this quantitative analytical method fit for purpose in terms of trueness and precision ?

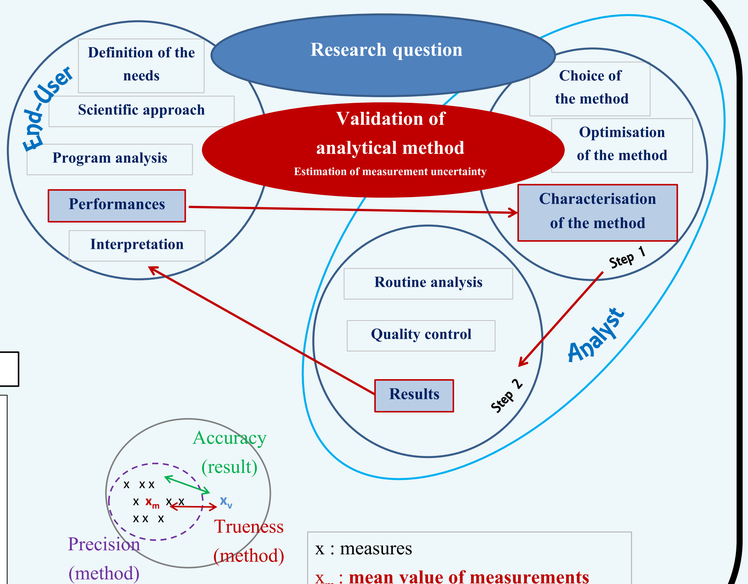


Conclusion :

Data are obtained with internal reference material (spiking):

- True and precise method between 0,4 et 2,4 mg/L (scope of the method)
- Low limit of quantification (LQ) = 0,4 mg/L
- The method fits to the scientific needs for a direct application with a **measurement uncertainty** estimated at maximum 5% in the field of the validated method.

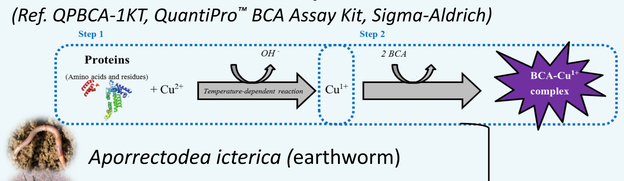
→ In the most critical conditions of the scope of the method (low LQ), **for each future sample**, the method will provide results with the required accuracy. In the wider field of application, the likelihood of obtaining non acceptable results is very low.



Material and methods

Protein determination method by the Bicinchoninic acid

(Ref. QPBCA-1KT, QuantiPro™ BCA Assay Kit, Sigma-Aldrich)



Aporrectodea icterica (earthworm)


Poecilus cupreus (beetle)


Gammarus pulex (gammarid)


Alopecosa pulverulenta (spider)

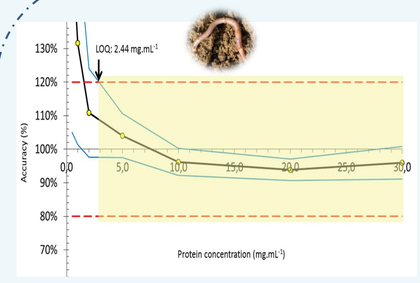
4 biological models

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

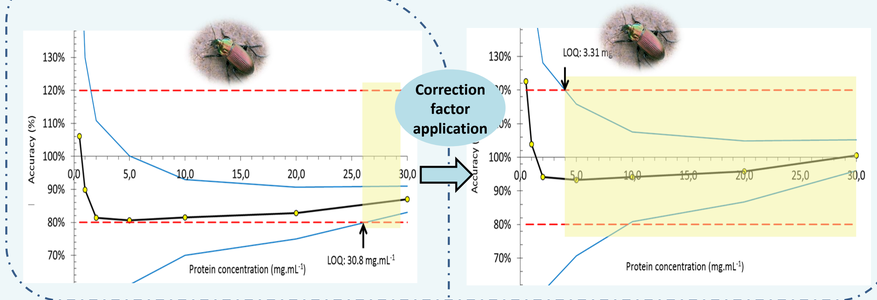
- **Selection of validation samples:**
  - Spiked reference material: Bovine Serum Albumine
  - 7 levels (0.5 ; 1 ; 2 ; 5 ; 10 ; 20 ; 30 mg.mL^{-1})
  - 3 replicates (repeatability)
  - 6 days of analysis *(intermediate precision)*
  - 2 analysts *(intermediate precision)*
- **Calibration (indirect quantitative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

Results

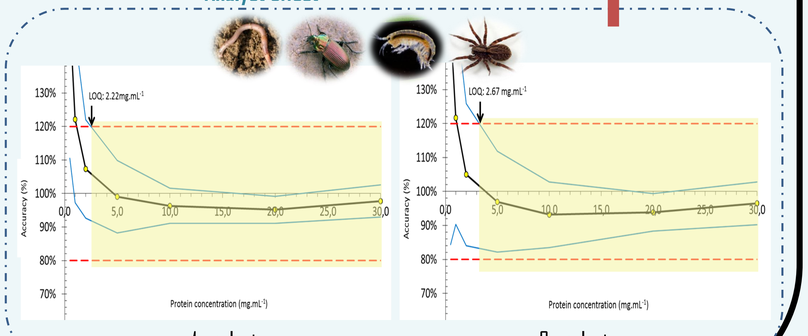
Biological model effect



Matrix effect



Analyst effect



Recovery (%)
Lower limit of tolerance (%)
Upper limit of tolerance (%)
Lower limit of acceptability (%)
Upper limit of acceptability (%)
Scope of the validated method

Performance of the method

Fixed criteria:

β *= ±80% (expectation tolerance limits)*

λ *= ±20% (acceptability limits)*

Limit of Quantification LQ: 2,7 mg/mL

Scope of the method: 2,7 to 30 mg/mL

Measurement uncertainty range ($U_{95\%}$) : 10 to 30%

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:

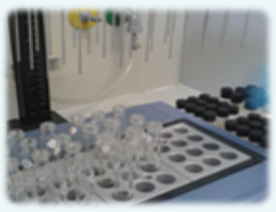
- Determine the performance of the method
- Improve the steps for sample preparation and analysis
- Assess the matrix effect
- Pointed out the importance of an experimented analyst

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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