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► To cite this version:

Richard Vilté, Anne Jaulin, Virginie Grondin, Nathalie Cheviron. The validation of analytical methods as an evaluation tool of research results reliability. Functional Ecology Conference AnaEE France, Mar 2017, La Grande Motte, France. 2017. hal-01602702

HAL Id: hal-01602702

<https://hal.science/hal-01602702>

Submitted on 2 Jun 2020

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Method validation in research : a relevant tool for assessing the performance of analytical methods

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Context

The analytical platform for environmental biochemistry 'Biochem-Env' :

- was created in 2012 by the 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 (ANR-11-INNB-0001),
- is mainly involved in research projects for the **biochemical characterization of natural environments** : soils, sediments and associated meso- and macro-fauna,
- **develops and validates methods** in order to **provide traceable analytical data with high-level of confidence**.

For within-laboratory validation of quantitative analytical methods, the INRA's Quality Guidelines for Research and Experimental Units recommends "the accuracy profile" method according to the NF V03-110:2010 standard.

Aim of the study Validate and assess the performance of a within-laboratory developed method for the quantification of enzymatic activities in different types of soil.

Method validation by the accuracy profile approach

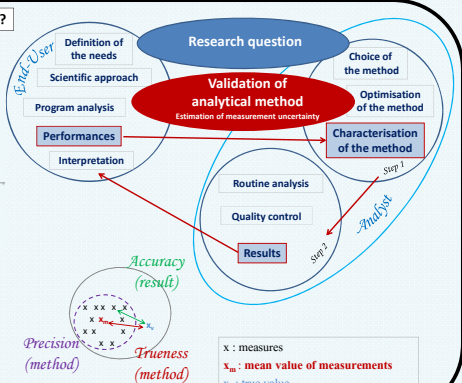
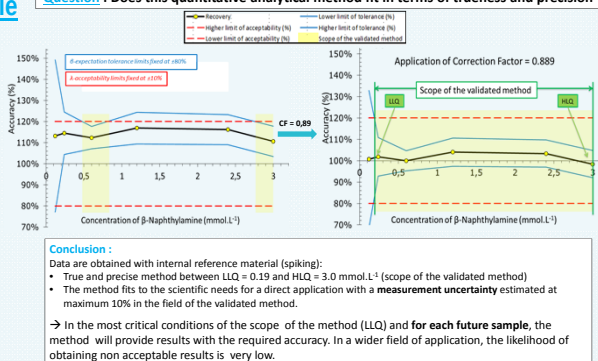
Purpose:

- 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:

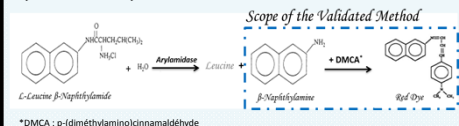
- An overall statistical method combining **trueness** and **precision**
- A standardized approach based on NF V03-110:2010
- A **simple, pragmatic and graphical interpretation** for a rapid decision
- The determination of quantification limits and the **scope of the method**
- The use of a **Correction Factor CF** when a **systematic bias** of trueness is brought out
- Estimation of **measurement uncertainty** for the future results
- No limit in the choice of the calibration model → large scope range

Question : Does this quantitative analytical method fit in terms of trueness and precision ?



Material and methods

Analytical method for β-Naphthylamine quantification in soils by UV-Visible spectrophotometry (λ = 540 nm) for the measurement of Arylamidase activity :



Validation method according to the 'accuracy profile' approach :

➤ **Selection of validation samples:**

➤ 4 spiked soils :



- 6 levels of β-Naphthylamine spiking : 0.12 ; 0.24 ; 0.6 ; 1.2 ; 2.4 ; 3.0 mmol.L⁻¹
- 3 replicates (*repeatability*)
- 5 days of analysis or series (*intermediate precision*)

➤ **Calibration (indirect quantitative analysis):**

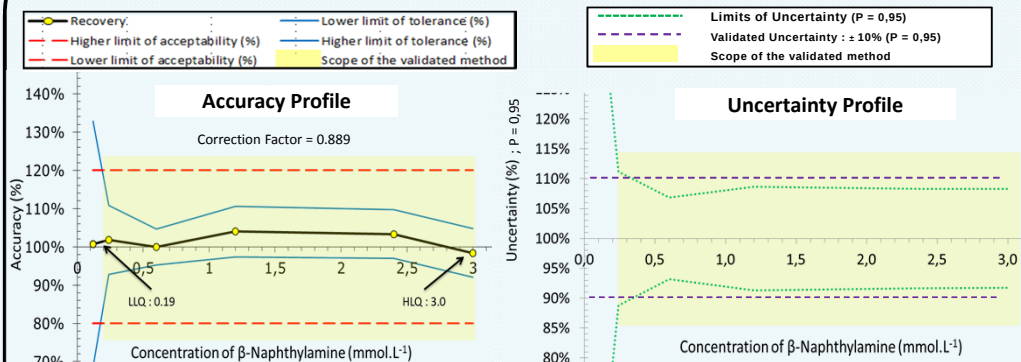
- 6 levels of β-Naphthylamine (0 ; 0.12 ; 0.24 ; 0.6 ; 1.2 ; 2.4 mmol.L⁻¹)
- Regression model : linear

➤ **Fixed criteria :**

- β = 80 % (tolerance limits)
- λ = ± 20 % (acceptability limits defined by the end-user)

Results

Validation of β-Naphthylamine quantification in 4 soils for the measurement of Arylamidase activity



Performance of the analytical method (β = 80 % and λ = ± 20 %)

- **Scope of the validated method** : 0.20 mmol.L⁻¹ (LLQ) to 3.0 mmol.L⁻¹ (HLQ)
- **Correction Factor CF** assessed at 0.889 to include in the calculation of the expression of results
- **Relative Measurement Uncertainty** (Probability = 95 %) assessed at 10 % and constant over the scope

Conclusions

The validation of this analytical method using the Accuracy Profile method helped to :

- determine the performance of the method : limits of quantification and scope of application, under fixed criteria,
- estimate the related measurement uncertainty for future results,
- increase the scope of the method with a validated correction factor CF (matrix effect systematically taken into account)
- improve the steps for sample preparation and analysis (i.e. necessity in buffering all soil solutions)
- manage risks and guarantees for both end-users and laboratories

A single analytical method for the measurement of arylamidase activity in different types of soils was validated considering scientific specifications and needs.

- **Clear & good interconnexion between method performance and intended use was aimed.**

For the future Practicing and using routine control boards

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References

1. INRA Quality Policy and Quality Guidelines for the research and experimental units (2013).
2. NF V 03-110 Standard (2010). Protocole de caractérisation en vue de la validation d'une méthode d'analyse quantitative par construction du profil d'exactitude.
3. Labo-Stat : Guide de validation des méthodes d'analyse, Feinberg M. Retirage (2012).
4. Feinberg M. et Laurentie M. A global approach to method validation and measurement uncertainty. Accred. Qual. Assu. 11, 3-9 (2006).
5. Jaulin A. et Deschamps M. La validation de méthodes d'analyse quantitative, c'est l'affaire de tous ! Journées de la Mesure et de la Métrologie de l'INRA, 8-11/10/2012, Le Croisic (2012).
6. INRA. Validation des méthodes d'analyse quantitative par le profil d'exactitude. Numéro spécial du Cahier des Techniques de l'INRA (2010).