



**HAL**  
open science

## The validation of analytical methods as an evaluation tool of research results reliability

Richard Vilte, Anne Jaulin, Virginie Grondin, Nathalie Cheviron

### ► To cite this version:

Richard Vilte, 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

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - ShareAlike 4.0 International License



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

Richard VILTE, Anne JAULIN\*, Virginie GRONDIN, Nathalie CHEVIRON

UMR ECOSYS, Platform Biochem-Env, INRA, AgroParisTech, Université Paris-Saclay, 78026, Versailles, FRANCE



## 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

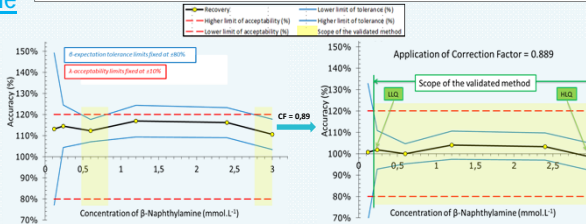
#### Purpose:

- > 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:

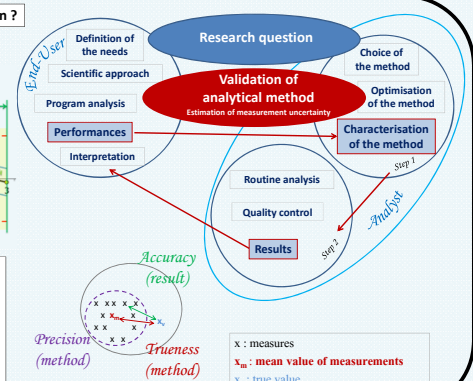
- > An overall statistical method combining trueness and precision
- > A standardized approach based on NF V03-110:2010
- > A simple, pragmatic and graphical interpretation for a rapid decision
- > The determination of quantification limits and the scope of the method
- > The use of a Correction Factor 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 ?



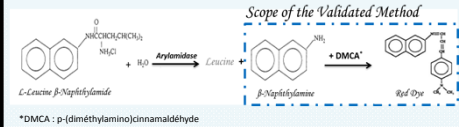
#### Conclusion :

- Data are obtained with internal reference material (spiking):
  - True and precise method with LLQ = 0.19 and HLQ = 3.0 mmol.L<sup>-1</sup> (scope of the validated method)
  - The method fits to the scientific needs for a direct application with a measurement uncertainty estimated at maximum 10% in the field of the validated method.
- In the most critical conditions of the scope of the method (LLQ) and for each future sample, the method will provide results with the required accuracy. In a wider field of application, the likelihood of obtaining non acceptable results is very low.



## Material and methods

Analytical method for  $\beta$ -Naphthylamine quantification in soils by UV-Visible spectrophotometry ( $\lambda = 540$  nm) for the measurement of Arylamidase activity :



Validation method according to the 'accuracy profile' approach :

#### Selection of validation samples:

- 4 spiked soils : Humic Soil, Silty Clay Soil, Clay Soil, Sandy Soil
- 6 levels of  $\beta$ -Naphthylamine spiking : 0.12 ; 0.24 ; 0.6 ; 1.2 ; 2.4 ; 3.0 mmol.L<sup>-1</sup>
- 3 replicates (repeatability)
- 5 days of analysis or series (intermediate precision)

#### Calibration (indirect quantitative analysis):

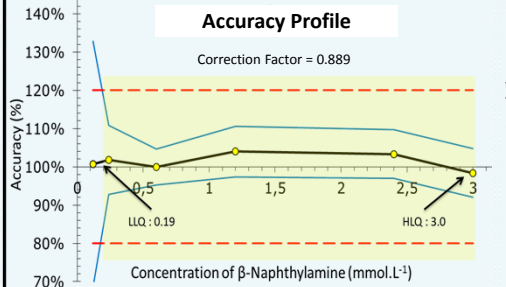
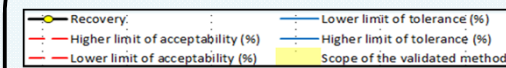
- 6 levels of  $\beta$ -Naphthylamine (0 ; 0.12 ; 0.24 ; 0.6 ; 1.2 ; 2.4 mmol.L<sup>-1</sup>)
- Regression model : linear

#### Fixed criteria :

- $\beta = 80\%$  (tolerance limits)
- $\lambda = \pm 20\%$  (acceptability limits defined by the end-user)

## Results

### Validation of $\beta$ -Naphthylamine quantification in 4 soils for the measurement of Arylamidase activity



#### Performance of the analytical method ( $\beta = 80\%$ and $\lambda = \pm 20\%$ )

- Scope of the validated method : 0.20 mmol.L<sup>-1</sup> (LLQ) to 3.0 mmol.L<sup>-1</sup> (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

## Contacts

Christian Mouglin  
christian.mouglin@inra.fr

Nathalie Cheviron  
nathalie.cheviron@inra.fr

Anne Jaulin (\* corresponding author)  
anne.jaulin@inra.fr

Richard Vilte  
richard.vilte@inra.fr

Virginie Grondin  
virginie.grondin@inra.fr

## 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. LaboStat : 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 Crosic (2012).
6. INRA. Validation des méthodes d'analyse quantitative par le profil d'exactitude. Numéro spécial du Cahier des Techniques de l'INRA (2010).