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 French National Institute for Agricultural Research (INRA) with the support of the ANR program "Investissements d'avenir" (ANR-11-INBS-0001) as a service of the infrastructure ANAEE-France. Missions of Biochem-Env include providing facilities for biochemical study in natural environments (enzyme activities in soils and sediments) and associated meso- and macro-fauna (enzyme activities and biomarkers in terrestrial and benthic organisms) and, as a research partner, guaranteeing the output of traceable and high-level confidence analytical data.

Questions. If research laboratories work with within-laboratory developed, non-standardized analytical methods, or when the methods are outside the scope of application of related standard, INRA's Quality Policy requests the validation of such methods [1]. For within-laboratory validation of quantitative analytical methods, the INRA Guidelines for research and experimental units recommends "the accuracy profile" method (AP) according to the NF V03-110:2010 standard [1;2].

Methodology. The AP consists in an overall statistical method combining trueness and precision by a simple graphic interpretation of analytical data and allowing a rapid decision to accept or reject the analytical method [3]. It defines the quantification limits (lower and upper limits) and hence, the scope of application. It indicates the need of applying a correction factor when systematic accuracy biases are revealed (i.e. matrix effect). It also provides an estimation of the uncertainty measurement associated with future results [4]. In fine, the process of analytical method validation allows establishing fitness for purpose by the interpretation of criteria in relation to the scientific objectives [5;6].

Application. In the present work, we applied the AP method to assess the performance of a within-laboratory developed analytical method used at Biochem-Env. We focused on the final compound (β-naphthylamine) produced by the arylamidase enzyme, an indicator of the biogeochemical nitrogen cycle. The analytical method was tested for samples from four different soil environments : humic, silty-clay, clay and sandy soils. The AP method informed about matrix effects and how improving steps for sample preparation and analysis. We validated the within-laboratory analytical method within range of 0.2 to 3.0 mmol.L^{-1} of β-naphthylamine, representing currently observed levels of arylamidase activity in soils, with a constant uncertainty measurement of 10% (P=95%).

Keywords
Method validation; accuracy profile; correction factor; measurement uncertainty; soil enzymes; N-cycle

Related references

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