
**Cosmetics — Analytical methods —
Development of a global approach
for validation of quantitative
analytical methods**

*Cosmétiques — Méthodes analytiques — Développement d'une
approche globale pour la validation des méthodes analytiques
quantitatives*

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Foreword

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This document was prepared by Technical Committee ISO/TC 217, *Cosmetics*.

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Introduction

The purpose of this document is to propose a characterization protocol for the validation of a quantitative analysis method in the cosmetic field and thus responds to the requirements of ISO/IEC 17025, i.e. using the performance goals as a basis. The theoretical principles of this approach can be found in Reference [1]. This document is based on the French Standard NF V 03-110[2].

Analytical methods for analyses of cosmetics need to be validated. Validation has been long considered as a process consisting in individually verifying several different criteria, i.e. selectivity, repeatability, linearity, trueness, etc. The global approach, as proposed since 2003[1], is based on the total error concept and the term “global” means that only a single criterion should be checked to validate a method: the agreement between a future experimental result and the true value. This approach has already been applied in the domains of pharmacy[1],[9], agricultural chemistry[2], and is in agreement with quality assurance guidelines such as GLP or ISO/IEC 17025. This validation process applies generally to already developed methods and includes evaluations of the following criteria: specificity/selectivity, precision, trueness, linearity range, LOD/LOQ, stability, ruggedness.

The large number of cosmetic products and the variety of matrices present a challenge for an analytical laboratory requiring that standardized methods to be adapted for each type of samples. Additional difficulties are linked to the very low concentrations to be measured, generally of the order of the mg/kg (ppm) or µg/kg (ppb). In such context, criteria such as accuracy and uncertainty of measurement of the analytical results are of utmost importance.

When the concentration of a substance is determined by an analytical laboratory, it is important to evaluate the gap between the measured value and the known true value. This difference indicates the trueness of the analysis. If cosmetic samples are analysed several times in different conditions (laboratory, instrument, operator), the individual results will present a dispersal around the average value which represents the precision of the measurement. As for the individual measurement, it represents an error with the average value and an inaccuracy with regard to the reference value (i.e. the true value).

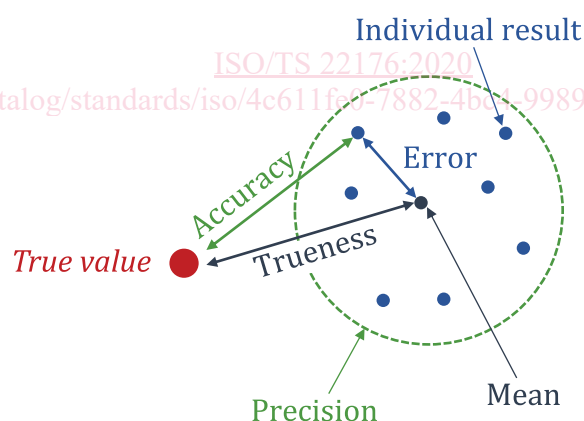


Figure 1 — Illustration of the concepts of accuracy, precision and trueness

When a laboratory measures the concentration of a given substance in a cosmetic product sample, the value which is obtained is thus characterized by a given accuracy which includes at the same time the notion of trueness and precision (see Figure 1). It can also be considered as total error. The insurance that the accuracy of a result is below acceptable limits, is thus one of the ways to make sure of the validity of a measurement.

The accuracy profile (plot of accuracy versus concentration), such as it is developed in numerous domains[3] to [9], is thus the way to know the accuracy on a result obtained with a given method applied to a type of sample in the environment of a given laboratory.

To reach this accuracy profile, it is necessary to undergo a specific assay allowing to demonstrate the validity of the analytical method, as well as the accuracy of the measurement for a given substance. In this approach, it is necessary to determine a tolerance interval^[10] which contains a given proportion (β) of future measured values inside (in average). If this tolerance interval is located inside a limit of acceptability defined a priori, taking into consideration several parameters such as the type and concentration of analyte, type of matrix, of analysis and conditions of the experiments, in this case, the method will be considered as valid, and if it goes outside this limit of acceptability, the method will be considered as non-valid (see Figure 2).

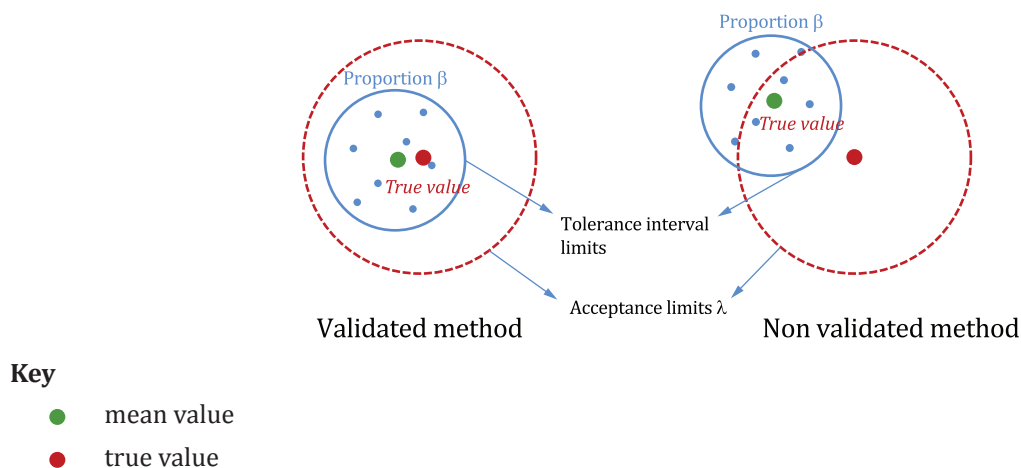


Figure 2 — Illustration of the validation principle

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