2025 IMEKO Joint Conference
TC8 - TC11 - TC25
Torino, Italy, September 14-17, 2025

Validation of Measurement Procedures that Include Sampling (VaMPIS) – a new Eurachem Guide

Michael H. Ramsey[1]

[1] *Co-Chair of Eurachem Joint Working Group on VaMPIS; School of Life Sciences, University of Sussex, UK*

***Abstract* – The validation of measurement procedures has traditionally focused on the analytical procedures applied to extracted samples within the laboratory (i.e. *ex situ*). However, it has been widely accepted that the measurement procedure actually begins at the moment that a sample is taken from the sampling target. The validation process therefore needs to be expanded to include the primary sampling procedure, and how to do this is explained in the new Eurachem Guide on Validation of Measurement Procedures that Include Sampling (VaMPIS). It is applicable to measurement procedures whether they are applied *ex situ*, or *in situ* when no physical sample is extracted. It can also be applied either simultaneously to the whole measurement procedure, or sequentially when a previously validated analytical procedure is used. Worked examples are given in this new Guide for each of these situations.**

I. THE NEED FOR VAMPIS

Measurement procedures (often called 'analytical methods or procedures') have traditionally been considered to be only the activities occurring in a laboratory that are applied to a 'laboratory sample' delivered for *ex situ* measurement in that laboratory. The well-established approach to validation of that analytical procedure has therefore focused on whether that procedure and its measurement results are fit for purpose in that context [1]. However, in the last thirty years there has been an increasing realization that a measurement procedure actually begins much earlier, at the time that the primary sample is taken from the sampling target [2]. (The sampling target is defined as the 'portion of material, at a particular time, that the sample is intended to represent' [2]). This realization required a new approach to the validation of the whole measurement procedure that includes the primary sampling, and any treatment of that sample prior to analysis or testing. This broader view of the measurement process has been recognized in ISO/IEC 17025 [3].

There is also a need to be able to validate measurement procedures that are applied *in situ*, without the need to remove a physical sample from the sampling target, or send it to a laboratory.

II. PERFORMANCE CHARACTERISTICS AND FITNESS FOR PURPOSE

In the traditional validation of analytical procedures, the following eight performance characteristics are recognized [1]: selectivity, limit of detection (or quantification), working range, analytical sensitivity, trueness (as bias or recovery), precision (analytical, repeatability, intermediate precision and reproducibility), ruggedness (robustness) and measurement uncertainty [4]. In the wider approach to the measurement procedure that includes sampling, most of these characteristics are not applicable to the sampling component. Measurement uncertainty (MU) is, however, the one characteristic that includes sampling, summarises the effects on data quality of all of the other characteristics, and is used in compliance decisions that are based upon the measurement result. MU is therefore the primary characteristic that is used to judge the fitness for purpose of the whole measurement process in VaMPIS.

III. APROACHES TO VAMPIS

There are two ways in which VaMPIS can be applied**:** In **simultaneous** validation, both the sampling procedure (SP) and the analytical procedures (AP) are validated at the same time as part of a unified measurement procedure (MP). In **sequential** validation the sampling procedure is validated using an analytical procedure that has previously been validated for the required purpose in isolation. Worked example are given of both of these approaches, discussed below (Section V, [5]).

IV. FLOW CHART FOR VAMPIS

The application of VaMPIS can be summarized using a flow chart showing its 11 steps (Fig. 1). There are three initial planning steps (in blue boxes) followed by the implementation steps (in grey). The measurement uncertainty (MU) and its component arising from sampling (UfS) are usually estimated using the Duplicate Method followed by Analysis of Variance (ANOVA)

(Step 7, described in more detail in [2]). The decision on whether the measurement procedure (MP) is fit for purpose (FFP) is based upon comparing the actual MU against the Target MU (Fig. 1, in orange). The value of the Target MU, including the contribution from sampling (UfS), should ideally be set by a regulator. If this value is not available, it can be calculated (Step 8b) using a technique such as the Optimized Uncertainty method [3]. Specially prepared software (OptiMU) is available to make these calculations [6]).

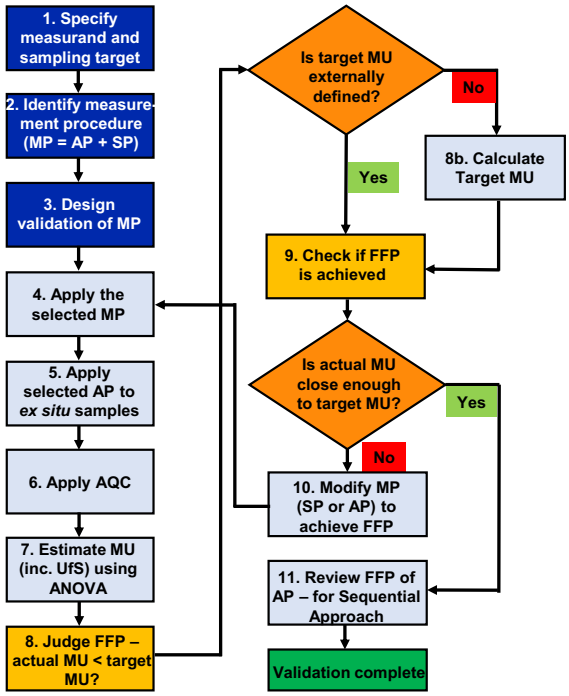

Fig. 1. Flow chart for application of VaMPIS (for explanation and abbreviations, see Sections III & IV). Taken from [7].

If the actual MU is judged as being not FFP, the decision on whether to improve the sampling procedure (SP) or the analytical procedure (AP) depends on their respective contributions to both the MU and to the measurement cost. In the sequential approach to VaMPIS, the FFP of the AP, that has been previously validated in isolation, is also checked to see if it is still FFP within this whole MP (Step 11). If the MU from the analytical procedure needs to be improved, the most appropriate of the seven other traditional performance characteristics (Section II, [1]) need to be modified to that end.

V. WORKED EXAMPLES

The application of VaMPIS is explained further using two worked examples.

*Example A1: Nitrate in glasshouse grown lettuce - **Sequential** approach to VaMPIS for an **ex situ** measurement procedure*

This is an example of the sequential application of VaMPIS, to an *ex situ* measurement procedure, where the analytical procedure had previously been validated for this purpose (in this case using an interlaboratory collaborative trial). The actual MU value, estimated across eight typical sampling targets, proved to be approximately twice the target MU (calculated using the Optimised Uncertainty method). The main souce of MU is shown to be the sampling. Reducing the MU down to the Target MU was, therefore, best acheived by reducing the sampling component of the MU (i.e. UfS). This was acheived in practice, and FFP effectively achieved, by increasing the number of increments in the primary composite sample by a factor of four.

*Example A2: In situ measurement of total lead in topsoil - **Simultaneous** approach applied to an **in situ** measurement procedure.*

This example judges FFP using estimates of the MU for measurements of lead in topsoil by portable x-ray fluorescence (pXRF) applied *in situ* to the surface of the soil. The primary sampling is an integral part of the measurement procedure when the pXRF is positioned on the soil surface. During validation, the pXRF was positioned twice independently at each of the 24 sampling targets to generate duplicate measurements. The analytical component of the MU was estimated using duplicate analyses within measurements (in this case using pXRF on extracted *ex situ* samples). The systematic component of the MU was estimated by comparing the *in situ* pXRF measurements against ICP-AES measurement on *ex situ* samples from the same locations. The actual MU was found to be large, and dominated by the sampling component. The Target MU (including sampling) was again calculated using the Optimum Uncertainty method, but for two different potential purposes. The *in situ* pXRF was found to be not FFP for the classification of the soil for lead contamination against a regulatory limit at this test site. This was cause by the high risk of misclassification due to the high levels of MU. By contrast, this *in situ* pXRF measurement procedure was found to be FFP for the mapping of the lead concentration across this test site to identify areas of higher and lower concentrations.

VI. FOLLOW UP AFTER VALIDATION

The second section of the Guide [5] considers the actions required after the initial validation. The use of ongoing quality control (QC) also needs to be extended to include the sampling procedures, to give Integrated Measurement Quality Control (IMQC). This includes internal QC procedures such as the taking of duplicated samples in addition to the more usual duplicated chemical analyses.

External QC procedure such as Proficiency Testing also need to be extended to include primary sampling. This can be by participation in Sampling Proficiency Tests (SPTs), or even better in Measurement Proficiency Test (MPTs) that include both sampling and analysis within an integrated whole measurement procedure.

VII. MANAGEMENT ISSUES

The last main section of the Guide [5] discusses the considerable management issues involved in the implementation of VaMPIS. For many decades there has usually been a large organizational divide between the activities of primary sampling and chemical analysis (or testing). These two activities have usually been undertaken by different organisations using different approaches to ensure suitable data quality. Successful implementation of VaMPIS requires that these previous barriers be overcome and that an integrated approach be taken to the whole measurement procedure. This requires better effective communication, and also increased cooperation, between the personnel involved in both the sampling and the chemical analysis. Moreover, one person needs to be ultimately responsible for the quality of the reported measurement results, and hence for the quality of both the sampling and the chemical analysis (or testing).

REFERENCES

[1] Cantwell H. (ed.) Eurachem Guide: The Fitness for Purpose of Analytical Methods – A Laboratory Guide to Method Validation and Related Topics, Eurachem (3rd ed. 2025). http://www.eurachem.org.

[2] Ramsey M. H., Ellison S. L. R. and Rostron P., (eds.) Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide: *Measurement Uncertainty Arising from Sampling: a Guide to Methods and Approaches*. Eurachem (2nd ed. 2019) http://www.eurachem.org.

[3] ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories. ISO Switzerland. https://www.iso.org

[4] Raposo, F.C., Ramsey, M.H. (2024) The role of measurement uncertainty in the validation of a measurement procedure. Accreditation and Quality Assurance. https://doi.org/10.1007/s00769-024-01593-y

[5] Ramsey M.H., Rostron P.D., and Raposo F.C. (eds.) Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide: *Validation of Measurement Procedures that Include Sampling,* Eurachem (2024). http://www.eurachem.org.

[6] AMC Software https://members.rsc.org/site/content/Community/SubjectCommunity/Analytical_Science_Community/AMCSoftware.aspx

[7] VaMPIS - Validation of Measurement Procedures that Include Sampling, Eurachem Leaflet. http://www.eurachem.org.