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CITAC
Cooperation on International Traceability in Analytical Chemistry

## Workshop

# Quality in Analytical Measurements

## Uncertainty Evaluation and Results Interpretation

# Book of Abstracts


**Lisbon, 11 and 12 May 2026**

https://eurachem2026.events.chemistry.pt/

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**Quality in Analytical Measurements, Uncertainty Evaluation and Results Interpretation**
**Book of Abstracts**
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# Welcome Message

Eurachem-Portugal and the Analytical Chemistry Division of the Portuguese Society of Chemistry welcome you to the Eurachem/CITAC Workshop on Quality in Analytical Measurements: Uncertainty Evaluation and Results Interpretation. This event follows two successful Eurachem workshops organised in Lisbon in 2011, where the third edition of the guide "Quantifying Uncertainty in Analytical Measurement" was presented, and in 2014, where the 25th Anniversary of Eurachem was celebrated. This year, the presentation of the first edition of the Eurachem/CITAC guide on "Evaluation of measurement uncertainty from in-house precision and recovery data" can be highlighted. The document provides detailed guidance on how to use in-house method validation and/or test quality control data to evaluate measurement uncertainty. The guide complements existing international guidance by providing evaluation approaches that guarantee the accuracy of the measured value, pragmatic modelling of how measurement uncertainty varies with the measured value, and plenty of solutions to optimise the magnitude of the measurement uncertainty.
The two-days workshop dedicated to technical aspects of the quality of analytical work will be held on 11 and 12 May, following a training course on the new Eurachem/CITAC guide for the evaluation of measurement uncertainty. More than 70 participants from Europe, Africa, Asia, America and Oceania will join the training. The workshop will be attended by more than 110 experts from the five continents, demonstrating the worldwide impact of the Eurachem/CITAC documents. The programme will cover the evaluation of measurement uncertainty, the assessment of conformity of tested items, the reliability of qualitative analysis and sampling uncertainty. After invited lectures by experts on the session topic, participants will have the chance to discuss relevant aspects of chemical analysis reliability in lively breakout sessions moderated by two facilitators. Posters and Short Communications on the topics of the event will also be presented.
The workshop dinner will be held at the Maritime Museum, celebrating the Portuguese Discoveries that pioneered globalisation and multiculturalism, as illustrated by the geographical distribution of workshop participants. The social event will be an opportunity to discuss how relevant it is to promote the quality of chemical analysis in a more relaxed setting.
The organising committee of the event decided to take advantage of the opportunities offered by the event to convey key messages about the quality of chemical analysis, such as the need to report results' uncertainty. We hope the event will inspire you to pursue and disseminate the best analytical practices!

*Ricardo Bettencourt da Silva*
President of the Analytical Chemistry Division of the Portuguese Society of Chemistry

# Program - 11 May 2026 (Monday)

| | | |
|---|---|---|
| 8:00 | Registration | |
| 9:00 | Opening (Eurachem \| CITAC \| SPQ \| FCUL ) | |
| | **Session 1: Evaluation of the measurement uncertainty** | |
| 9:20 | Uncertainty in the measurement process, *Steve Ellison* | 12 |
| 9:50 | Modelling uncertainty across the working range, *Ricardo Bettencourt da Silva* | 13 |
| 10:15 | Trueness-related uncertainty, *Vicki Barwick* | 14 |
| 10:40 | **Coffee break & poster** | |
| | **Breakout sessions 1 and 2** - Parallel sessions | |
| 11:00 | Breakout session 1<br>How can measurement trueness be tested when various reference materials are available? | |
| 11:00 | Breakout session 2<br>How can the variation of the measurement uncertainty with concentration be modelled? | |
| 12:00 | Breakout sessions reports | |
| 12:30 | **Lunch** | |
| | **Session 2: Conformity assessment** | |
| 13:30 | Consumer and producers' risks in compliance assessment, *Bertil Magnusson* | 15 |
| 14:00 | Conformity with multiple parameters, *Felipe Lourenço* | 16 |
| 14:20 | Conformity assessment in the oil and gas industry, *Élcio Oliveira* | 17 |
| 14:40 | **Coffee break & posters** | |
| | **Breakout sessions 3 and 4** - Parallel sessions | |
| 15:00 | Breakout session 3<br>Conformity with multiple parameters | |
| 15:00 | Breakout session 4<br>Decision rule selection | |
| 16:00 | **Posters** | |
| 16:30 | Breakout session reporting | |
| 17:00 | Closing the Day | |
| 20:00 | Workshop Dinner - Maritime Museum, Belém, Lisboa | |

# Program - 12 May 2026 (Tuesday)

| | | |
|---|---|---|
| 8:00 | Registration | |
| 9:00 | Opening (RELACRE \| ForMEQ \| SPMet ) | |
| | **Session 3: Qualitative Analysis** | |
| 9:20 | Assessing the performance of qualitative analysis, *Ricardo Bettencourt da Silva* | 18 |
| 9:50 | Validation and quality control of qualitative analysis, *Paulo Pereira* | 19 |
| 10:15 | Validation of the identification of microplastics by micro-FTIR and micro-Raman, *Carla Palma* | 20 |
| 10:40 | **Coffee break & posters** | |
| | **Breakout sessions 5 and 6** - Parallel sessions | |
| 11:00 | Breakout session 5<br>How detailed should qualitative analysis validation be? | |
| 11:00 | Breakout session 6<br>How useful is the reporting of qualitative analysis uncertainty? | |
| 12:00 | Breakout sessions reports | |
| 12:30 | **Lunch** | |
| | **Short communications 01** - Parallel sessions | |
| 13:30 | Updates of Handbooks from Nordtest, *Bertil Magnusson* | 24 |
| 13:45 | The use of collaborative trial in sampling for the validation of measurement procedures, *Peter D. Rostron* | 25 |
| 14:00 | The Fit for Purpose of Methods in Marine Matrices: The Portuguese Hydrographic Institute approach, *Carlos Borges* | 26 |
| | **Short communications 02** - Parallel sessions | |
| 13:30 | Assignment of Self-Certified Reference Values for PhotonAssay using ISO 33405:2024, *Makeen Buckley* | 27 |
| 13:45 | Measurement Uncertainty in Chemical and Microbiological Analyses: Implications for Conformity Assessment of Live Bivalves, *Helena Maria Lourenço* | 28 |
| 14:00 | Metrological Sound Assessment of the Risk of Food Exposure to Nitrate from Consumers of a Public Canteen, *Cristina M. Oliveira* | 29 |
| | **Session 4: Uncertainty from sampling** | |
| 14:20 | Review what has been achieved in estimation and applications of uncertainty from sampling, *Nathalie Guigues* | 21 |
| 14:40 | Identifying the new challenges for Uncertainty from Sampling, *Mike Ramsey* | 22 |

15:00 **Coffee break & posters**

15:20 **Breakout sessions 7 and 8** - Parallel sessions

15:20 Breakout session 7
Improving approaches to UfS estimation & VaMPIS*

15:20 Breakout session 8
Helping labs work with samplers on VaMPIS & QC

16:20 Drafting the report

16:30 Breakout session reporting

17:00 Best poster award (Award coordinator: Mike Ramsey)

17:10 **Closing Ceremony**

# Poster Communications

# Invited Oral Communications

# IC01

## Uncertainty in the measurement process

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Measurement uncertainty (MU) is a well established topic in chemical measurement. This presentation briefly introduces the main ideas underpinning measurement uncertainty and its evaluation, and its use in conformity assessment.

No measurement is perfectly exact; MU is an inevitable feature of all measurements. Many factors can contribute to MU; for example, uncertainties on certified values used in calibration, sampling, matrix effects, environmental conditions, and ever-present random variation. Evaluation of uncertainty is important for at least two reasons: First, to inform decisions made on the basis of measurement results; second, to identify and minimize important sources of uncertainty.

Historically, there have been different approaches to evaluation of MU. Since the publication of the 1993 ISO Guide (available as JCGM100 [1]), however, evaluation of MU for most 17025-accredited laboratories has followed harmonised principles and guidelines. These have been interpreted for chemical measurement by Eurachem Guide [2]. JCGM100 [1] combines uncertainties in input quantities using error propagation theory; this approach is sometimes referred to as a "bottom-up" approach. This is particularly well suited to measurements in which the uncertainty is dominated by uncertainties in measured input quantities. Where the major sources of uncertainty are associated with less well-understood effects, it is often more useful to consider the observed performance of the measurement method in practice, using (for example) reproducibility data and information from method validation; this is often referred to as a "top-down" strategy. The current Eurachem Guide [2] covers both approaches, and a new Eurachem guide [3] provides considerable additional detail on the use of in-house validation data.

MU contributions can be combined in a number of ways, including algebraic and numerical approaches. JCGM100 ("the GUM") uses an algebraic approach, often reduced to simple rules for common circumstances. Numerical approaches include various applications of finite difference methods to obtain the necessary gradient terms. More recently, Monte Carlo methods have become important; a JCGM document provides extensive detail on "propagation of distributions" via Monte Carlo methods [4]. Most recently, Bayesian methods have started to be used. An introduction to these approaches will be presented.

Conformity assessment is, perhaps, where knowledge of MU is most critical. In addition to guidance on evaluation of MU, Eurachem has published guidance on use of MU in conformity assessment [5]; ILAC provide more general guidance. A brief introduction to the principles will be provided.

**References:**

[1] BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP, and OIML. Evaluation of measurement data — Guide to the expression of uncertainty in measurement. Joint Committee for Guides in Metrology, JCGM 100:2008. doi:10.59161/JCGM100-2008E..
[1] S. L. R. Ellison, A Williams (Eds), Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, Eurachem, 2012.
[3] R Bettencourt da Silva (ed.) Eurachem/CITAC Guide: Evaluation of measurement uncertainty from in-house precision and recovery data. First Edition, Eurachem (2026). ISBN 978-972-9348-30-3. DOI: https://doi.org/10.56526/2026.0005. Available from http://www.eurachem.org.
[4] BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP, and OIML. Evaluation of measurement data — Supplement 1 to the "Guide to the expression of uncertainty in measurement" — Propagation of distributions using a Monte Carlo method. Joint Committee for Guides in Metrology, JCGM 101:2008. doi:10.59161/JCGM101-2008
[5] A. Williams and B. Magnusson (eds.) Eurachem/CITAC Guide: Use of uncertainty information in compliance assessment (2nd ed. 2021). ISBN 978-0-948926-38-9. Available from www.eurachem.org.
[6] ILAC G8:09/2019 Guidelines on Decision Rules and Statements of Conformity. ILAC, 2019. Available at https://ilac.org/publications-and-resources/ilac-guidance-series/

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# IC02

## Modelling uncertainty across the working range

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Many chemical analysis methods[1] are applicable to wide concentration[2] intervals requiring estimating their performance for the whole interval. Ideally, performance should be assessed from experimental data collected at various concentration levels, but frequently laboratory cannot afford the costs or time required for the assessment of performance at many concentration levels. Therefore, methodologies for optimising resources for measurement performance evaluation are required.
Typically, measurement precision varies with the concentration level, and systematic effects affecting measurement can be kept rather constant over that interval. In instrumental method of analysis, if adequate validation of their calibration, for instance through a linear regression model, is achieved, systematic effects do not vary significantly within the calibration interval. For classical methods of analysis and sample preparations prior instrumental methods of analysis, if chemical constraints such as reagent restraints or solubility limits are not achieved, systematic effects affecting measurement do not vary within the analytical interval. Therefore, in those cases, the modelling of measurement uncertainty over the analytical interval involves modelling measurement precision in the interval.
For evaluations of the measurement uncertainty based on in-house method validation or tests quality control data, in most cases, it is relevant to evaluate the measurement intermediate precision (i.e., between-days precision). The measurement repeatability relevant for the management of replicates in the same conditions is also useful for cases where sample results are the mean of replicate results obtained under these conditions.
It is known that regardless of the concentration value studied, in most cases, the absolute intermediate precision standard deviation reduces with the concentration, while the relative intermediate precision standard deviation reduces as the concentration increases. These almost universal trends can be used to simplify the modelling of the variation of measurement precision and consequently the measurement uncertainty with the concentration. When laboratories have precision estimates at various concentration levels, more complex precision models can be used.
This communication describes the approach proposed in the new Eurachem/CITAC guide on "Evaluation of measurement uncertainty from in-house precision and recovery data" for developing and optimising models of the variation of the measurement uncertainty with the concentration.

**References:**

[1] JCGM, International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (3rd ed.) (JCGM 200), BIPM, 2012.
[2] R. Bettencourt da Silva (ed.), Eurachem/CITAC Guide: Evaluation of measurement uncertainty from in-house precision and recovery data, First Edition, Eurachem, 2026. ISBN 978-972-9348-30-3.

[1] According to the International Vocabulary of Metrology [1] the term "measurement procedures" should be used in this context.
[2] In this text, the term "concentration" is used in a general sense to mention any other types of quantities, such as concentration, mass concentration, mass fraction, etc.

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# IC03

## Trueness-related uncertainty

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In addition to considering precision (random effects) the 'top-down' approach to uncertainty evaluation requires an estimate of trueness-related components. The trueness component reflects the impact of systematic effects on measurements. When the uncertainty is being evaluated from overall in-house method performance data, the trueness-component will typically be obtained using data from method validation studies of bias or recovery.

Evaluating trueness requires measurement results to be compared with a suitable reference value. This value can be provided by certified reference materials, test samples spiked in the laboratory, test samples characterised using an alternative method, or (under certain circumstances) materials from proficiency testing schemes. The materials used to evaluate trueness should be representative of routine test samples in terms of the analyte and analyte level, and sample matrix.

When evaluating the trueness component of uncertainty there are two issues that need to be considered: the estimate of the bias (or recovery) and the uncertainty associated with that estimate. This presentation will give an overview of approaches for evaluating bias/recovery and the associated uncertainty, focusing on the procedures outlined in the recently published Eurachem/CITAC guide on Evaluation of measurement uncertainty from in-house precision and recovery data [1]

**References:**

[1] R Bettencourt da Silva (ed.) Eurachem/CITAC Guide: Evaluation of measurement uncertainty from in-house precision and recovery data. First Edition, Eurachem (2026). ISBN 978-972-9348-30-3. Available from https://www.eurachem.org

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# IC04

## Consumer and producer risks in compliance assessment

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To decide whether a result indicates compliance or non-compliance with a specification or legal limit, it is necessary to use decision rules. These rules prescribe how to determine compliance or non-compliance with a limit, considering the acceptable level of the probability of making a wrong decision i.e. the associated risk.

Based on a decision rule, an “acceptance zone” and a “rejection zone” can be determined, so that if the measurement result lies in the acceptance zone the item is declared compliant (approved) and if in the rejection zone it is declared noncompliant (rejected). The decision rule can either focus on low producer risk, or low consumer risk, or shared risk.

**Consumer risk**– probability, P, that the value of the measurand lies within the limit focusing on high achieving high confidence in correct acceptance and a low probability of false acceptance.

**Producer risk** - probability, P, that the value of the measurand lies outside the limit focusing on achieving high confidence of correct rejection and a low probability of false rejection.

The presentation will introduce compliance assessment and give examples of specific risks with

- simple acceptance,
- focus on correct acceptance – low consumer risk – type II error
- focus on correct rejection – low producer risk – type I error

and discuss global risk for a producer where we need to know both the measurement uncertainty and the process distribution.

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# ICO5

## Conformity with multiple parameters

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Conformity assessment in analytical chemistry is traditionally based on comparing measured values with specification limits, and measurement uncertainty information may be used to assess the risk of false decisions. In many practical situations, however, conformity must be demonstrated simultaneously for multiple parameters rather than for a single parameter. In these contexts, decisions based only on the particular risk values of individual parameters may be insufficient, requiring the estimation of total risk through a multivariate assessment that accounts for the combined effect of multiple parameters and their associated uncertainties.

This work presents and discusses computational and statistical tools for assessing conformity with multiple parameters. In conformity assessment, particular risk refers to the probability of a false decision (consumer's or producer's risk) associated with a single measured parameter. In contrast, total risk represents the probability of a false decision when all parameters are considered simultaneously and may be significantly higher than the corresponding particular risk values. In addition, correlation between variables plays a critical role in conformity assessment involving multiple parameters, as it can either increase or decrease the total risk. Therefore, total risk estimation must explicitly consider correlation between variables to provide reliable conformity assessments.

Conventional decision rules based on guard-bands adjust specification limits using measurement uncertainty to control particular risk, typically ensuring a predefined maximum probability (e.g., 5%) of false decisions. However, these univariate guard-band approaches are insufficient for multiparametric decisions, as they do not guarantee control of total risk. To address this limitation, multivariate guard-band decision rules can be used to define acceptance limits that simultaneously ensure acceptable particular and total risk values. These multivariate acceptance limits are generally more restrictive than univariate ones.

Bayesian methods offer a powerful approach for conformity assessment by incorporating prior knowledge into the conformity assessment process. In this context, particular and total global risks can be estimated by combining prior information (e.g., manufacturing process data, historical data, or physicochemical constraints) with analytical. Furthermore, multivariate acceptance limits may be also defined using Bayesian approach, which takes into account both prior information and analytical data to control the probability of false decisions at a predefined level.

Multiparametric conformity assessment is essential in several application areas, such as pharmaceutical quality control, food analysis, environmental monitoring, and fuel testing, where decisions depend on multiple parameters evaluated simultaneously.

**References:**

[1] R. L. N. Maranho, F. R. Lourenço, Development of a Python-based tool for evaluation of multivariate decision risk considering measurement uncertainty and correlation effects, Microchem. J. 224 (2026) 117824. https://doi.org/10.1016/j.microc.2026.117824
[2] R. J. N. Bettencourt da Silva, F. R. Lourenço, D. B. Hibbert, Setting multivariate and correlated acceptance limits for assessing the conformity of items, Anal. Lett. (2022). https://doi.org/10.1080/00032719.2022.2042549
[3] R. J. N. Bettencourt da Silva, F. R. Lourenço, F. R. Pennecchi, D. B. Hibbert, I. Kuselman, Spreadsheet for evaluation of global risks in conformity assessment of a multicomponent material or object, Chemom. Intell. Lab. Syst. 188 (2019) 1–5. https://doi.org/10.1016/j.chemolab.2019.02.010
[4] C. M. da Silva, F. R. Lourenço, Definition of multivariate acceptance limits (guard-bands) applied to pharmaceutical equivalence assessment, J. Pharm. Biomed. Anal. 222 (2023) 115080. https://doi.org/10.1016/j.jpba.2022.115080

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# IC06

## Conformity assessment in the oil and gas industry

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Every measurement, including oil and gas sector, truly begins with sampling, a stage frequently performed outside the laboratory and often overlooked in uncertainty budgets. Despite its critical role, sampling uncertainty is rarely given the same attention as analytical uncertainty, even though international standards such as ISO / IEC 17025 require its consideration during accreditation.

This workshop seeks to demonstrate that uncertainty from sampling is an indispensable part of overall measurement uncertainty and can decisively influence conformity assessments. Through several illustrative case studies – covering measurands like manganese in produced water, sulfur in diesel, ethane in natural gas, phosphorus in biodiesel, and water in crude oil – it becomes evident that decisions based solely on analytical uncertainty may wrongly accept non-conforming products or reject batches that meet specifications. Incorporating sampling uncertainty frequently narrows the safety margin, occasionally reversing previously acceptable outcomes.

Ultimately, controlling the sampling process with the same rigor applied to laboratory analyses is essential. By combining both sampling and analytical uncertainties in risk analyses, laboratories and industry stakeholders achieve a more realistic appraisal of compliance, thereby better protecting consumers, producers, and regulatory integrity.

**References:**

[1] A. Williams, Principles of the EURACHEM/CITAC guide “Use of uncertainty information in compliance assessment”, Accred. Qual. Assur. 13 (2028) 633 – 638.
[2] L. Magnusson, S. L. R. Ellison, A. Williams (Eds.), Eurachem/CITAC Guide: Use of uncertainty information in compliance assessment, 2nd ed., Eurachem, 2021.
[3] JCGM, 2012. Evaluation of measurement data: the role of measurement uncertainty in conformity. Assessment 106, 2012.
[4] R.M. Moreira, C.L. Biazon, E.C. de Oliveira, Measurement uncertainty and compliance evaluation applied to natural gas moisture. Appl. Sci. 15 (2025) 2482. https://doi.org/10.3390/app15052482.
[5] J.D. Hernandez-Vásquez, E.C. de Oliveira, The Importance of Measurement Uncertainty Arising from the Sampling Process in Conformity Assessment: The Case of Fuel Quality. Metrology 5 (2025) 7. https://doi.org/10.3390/metrology5010007.

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# IC07

## Assessing the performance of qualitative analysis

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Many chemical analyses with relevant individual or socioeconomic impact are exclusively qualitative or involve a demanding qualitative analysis stage prior to the quantification. According to the latest version of the International Vocabulary of Metrology [1], the terms "examination" and "measurement" are used for qualitative and quantitative analysis, respectively.
Examination results are typically binary, such as "evidence" or "no evidence" of the presence of cocaine in a urine sample or of the compositional equivalence of oil patterns from an oil spill and suspected source of the spill. These binary results are frequently designated as "positive" or "negative" for presence/equivalence or no evidence of presence/equivalence, respectively.
The Eurachem/CITAC guide on "Assessment of performance and uncertainty in qualitative chemical analysis" [2] discusses the relevance of assessing the performance of examination procedures and presents some methodologies and metrics to perform these evaluations. Difficulties and alternatives to evaluation procedures and performance metrics are presented to make these evaluations feasible.
The determination of the posterior probability of a specific case given their evidence (e.g. the probability of cocaine presence in an urine sample considering evidences from the presence from the examination procedure based on GC-MS/MS analysis), deduced from the Bayes Theorem, requires using reliable information of the prevalence of the studied cases in the studied population (e.g. the percentage of athletes from the cycling competition that consumes cocaine). Prevalence information initially available (i.e. prior information) is updated with analytical evidence of cocaine presence driving to the posterior probability. Since frequently prior information is difficult to obtain and has a major impact on the posterior probability, some authors use non-informative prior information, i.e. assume the prevalence of the cases is 50%.
Another challenge laboratories have to face is the impossibility of quantifying the performance, typically the true positive and true negatives rates (i.e. the probability of an examination method producing correct evidence of positive or negative cases) from expensive and not widely used selective examination methods. For instance, for the reliable determination of a rate of false positive results of 0.01% from a GC-MS/MS identification above the Limit of Quantification of quantitative analysis, half a million negative cases must be analysed, making the experimental determination of this value impractical. However, simple signal modelling implemented in a user-friendly MS-Excel file can be used to quantify examination performance in these cases [3].
This communication discusses the difficulties of quantifying and reporting the quality of selective chemical examinations based on the instrumental method of analysis and presents tools that laboratories can use to understand if the identification criteria considered are adequate for producing adequately reliable examination results.

**References:**

[1] JCGM, International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (3rd ed.) (JCGM 200), BIPM, 2012.
[2] R. Bettencourt da Silva and S. L. R. Ellison (eds.), Eurachem/CITAC Guide: Assessment of performance and uncertainty in qualitative chemical analysis, First Edition, Eurachem, 2021. ISBN 978-0-948926-39-6. Available from www.eurachem.org.
[3] R. Bettencourt da Silva, P. Salgueiro, "Statistically sound identification of compounds by low-resolution GC-MS: Identification of tear agents in tear gas sprays", Talanta 282 (2025) 127061.

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# IC08

## Validation and Quality Control of Qualitative Analysis

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Qualitative examinations are widely used in medical laboratories for the classification of clinical states such as reactive/non-reactive, positive/negative, or present/absent. However, the metrological challenge in qualitative analysis is not the validation of a verbal label itself, but the validation of the probability that the assigned class is correct for its intended use. This presentation discusses a scientifically grounded framework for the validation and routine quality control of qualitative analytical methods, with emphasis on binary medical laboratory examinations.
The central validation model is the 2×2 contingency table, from which clinical sensitivity, clinical specificity, false-positive rate, and false-negative rate are derived. Particular attention is given to the importance of confidence intervals, since point estimates alone may substantially overstate the strength of the evidence, especially in high-stakes screening settings. The presentation also addresses the interpretive value of Bayesian reasoning, showing how disease prevalence influences posterior probability and therefore the clinical meaning of a reported positive result.
A practical anti-HCV antibody examination is used to illustrate how validation data can be translated into fitness-for-purpose claims and how routine quality control should be designed to protect the validated decision rule, particularly near the cutoff or equivocal zone. The overall message is that validation and quality control of qualitative analysis should be focused on classification risk, uncertainty, and decision reliability in real use.

**References:**

[1] R. Bettencourt da Silva, S. L. R. Ellison (Eds), Eurachem/CITAC Guide: Assessment of performance and uncertainty in qualitative chemical analysis, First edition, Eurachem, 2021.
[2] Clinical and Laboratory Standards Institute, CLSI EP12: Evaluation of Qualitative, Binary Output Examination Performance, Third edition, Clinical and Laboratory Standards Institute, 2023.
[3] P. Pereira, Quality Control of Qualitative Tests for Medical Laboratories, Lisbon: Author-edition, 2019.
[4] P. Pereira, Quality control perspectives: Weak signal detection to prevent clinical sensitivity failures in donor screening, Transfus. Apher. Sci. 64 (3) (2025) 104152.
[5] P. Pereira, J. Seghatchian, Spotlights on the trends in performance assessment of qualitative in vitro diagnostic medical devices in transfusion medicine, Transfus. Apher. Sci. 63 (2) (2024) 103887.
[6] P. Pereira, B. Magnusson, E. Theodorsson, J. O. Westgard, P. Encarnação, Measurement uncertainty as a tool for evaluating the 'grey zone' to reduce the false negatives in immunochemical screening of blood donors for infectious diseases, Accred. Qual. Assur. 21 (1) (2016) 25–32.

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# IC09

## Validation of the identification of microplastics by micro-FTIR and micro-Raman

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The properties of plastic materials that drive their commercial success—such as low cost and high chemical and physical resistance—combined with their improper disposal, have led to the widespread presence of persistent plastic particles in the environment. The environmental impact of plastics depends on their type, size, and shape, with smaller particles capable of entering living organisms, where their effects remain largely unknown. Based on size, plastic particles are classified as macroplastics (> 25 mm), mesoplastics (5 mm to 25 mm), microplastics (1 µm to 5 mm), and nanoplastics (< 1 µm). Alongside assessing the toxicological effects of these particles, it is also essential to evaluate their levels, trends, and primary sources. This information is crucial for guiding and monitoring policies aimed at reducing the impact of plastics on the environment and human health.

There is a lack of standardised methodologies for monitoring plastic contamination in food products and environmental samples. Furthermore, approaches for assessing method performance and quantifying results uncertainty remain underdeveloped, highlighting the need for further methodological advancement [1- 3].

Before counting plastic particles in a food or environmental sample, it is necessary to identify them. Micro-FTIR and micro-Raman are the most popular tools for plastic particle identification before characterising their size and shape. Reference and particle spectra are compared manually or automatically. Manual identifications is time-consuming and must be performed by qualified analysts, whereas automatic identification is fast and does not require expert knowledge. Given the number of plastic particles observed in some samples, automatic identification is recommended.

The automatic identification of particles requires quantifying the correlation or match between reference and particle spectra and defining a minimum match above which identification achieves an adequately high positive result rate and a low false positive result rate. The match threshold can be defined for reference and particle spectra collected using the same or different equipment, with the diversity of equipment representing an additional challenge for analysts.

This study presents the development and validation of a method for the automated identification of polyethylene terephthalate (PET) microplastics using micro-Raman spectroscopy or micro-FTIR analysis, using reference and sample spectra obtained from different instruments. Identification results are considered valid when they achieve a true positive rate of at least 95% and a false positive rate no greater than 5%.

**References:**

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# IC10

## Review: What Has Been Achieved in Estimation and Applications of Uncertainty from Sampling (UfS)

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Since the publication in 2007 [1] of the first edition, then in 2019 [2] of the second edition of the Eurachem/CITAC guidance on "Measurement uncertainty arising from sampling" - UfS, more than 200 articles, conference papers and book chapter have been published which include the contribution of sampling to the measurement uncertainty.
Around 25% of the articles only underline the importance of taking into account sampling in the measurement process and subsequently, but do not attempt to estimate its contribution to the measurement uncertainty.

Looking at the different approaches put in place to estimate the measurement uncertainty arising from sampling, the top-down (mainly the duplicate) method has been largely used in more than 40 % of the papers. Some more general concepts have been also discussed, e.g. the introduction of the uncertainty factor, or the calculation of confidence intervals on robust uncertainty estimates, or the implications for accredited laboratories. Technical Briefs from the Analytical Method Committee of the Royal Society of Chemistry fall in this category. Interlaboratory Comparison, simulation (e.g. using Monte Carlo or Bayes theory) and the bottom-up approach following Pierre Gy's sampling theory are also used, however less often than the duplicate method.

The main application area is environmental monitoring (representing 46% of articles), with soil (for agriculture or remediation) for 41% and water for 37% (surface water, groundwater, seawater, drinking water and wastewaters) of the environmental articles. Sediments and plants / moss, used to monitor bioaccumulation, are also investigating the uncertainty arising from sampling.
Another important application of the UfS is food and animal feed with 26% of articles, followed by geochemical and mining, industrial applications and human biomonitoring.

Only a couple of articles have cited the newly published Eurachem Guide on Validation of Measurement Procedures that Include Sampling (VaMPIS) [3], as would be expected since its recent publication in 2024.

Examples from a selection of articles will be present to illustrate new advances and applications in measuring uncertainty arising from sampling.

**References:**

[1] Ramsey M. H.and Ellison S. L. R. (eds.) Eurachem/EUROLAB/ CITAC/Nordtest/ AMC Guide: Measurement uncertainty arising from sampling: a guide to methods and approaches (1st ed. 2007) 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] 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.

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# IC11

## Identifying the new challenges for Uncertainty from Sampling (UfS)

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The first publications on recognising sampling as part of the measurement process, and consequently the measurement uncertainty that arises from sampling (UfS), were around thirty years ago. Since then, UfS has become recognised as an important and often dominant part of measurement uncertainty (MU), with a Eurachem Guide on UfS estimation [1], and many hundreds of papers reporting its application and consequences for the interpretation of measurement results [2]. This paper aims to identify the new and ongoing challenges for UfS in the future in terms of:-

1. Improving how UfS (& MU) is estimated to include:-
    a. systematic effects (e.g. bias) from both analysis, and sampling (where possible)
    b. confidence interval of UfS estimates reported, if required
    c. ongoing QC to monitor possible changes in UfS & MU after validation
2. Applying UfS estimation to a wider range of:-
    a. different materials (with spatial analyte heterogeneity and/or temporal variability)
    b. application sectors (e.g. environmental, food and feed, industrial processes, medical)
    c. measurement types (e.g. *ex situ, in situ* and on site)
3. Improving the take-up of UfS (as part of MU) estimation using:-
    a. Accreditation requirements (e.g. ISO 17025)
    b. Reduced cost (e.g. using unbalanced and/or simplified experimental designs)
    c. Enhanced communication and collaboration between samplers and lab staff
4. Recognising the benefits of knowing UfS within MU in terms of:-
    a. Judging the fitness for purpose, and therefore more inclusive validation, of the whole measurement procedure that includes sampling (VaMPIS) [3]
    b. More reliable compliance/conformity decisions, due to more realistic estimates of MU

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# Oral Communications

# OC01

## Updates of Handbooks from Nordtest

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Nordtest is a trademark of conformity assessment. The emphasis of Nordtest is to develop, promote and innovate Nordic test methods and to publish guidance documents. The ultimate is to remove technical barriers to trade and promote the concept: "Approved once, accepted everywhere". All documents are available at www.nordtest.info. Here the guides for analytical chemists will be presented

Currently there are four handbooks from Nordtest:

- **Nordtest 537**
  Handbook for calculation of measurement uncertainty in environmental laboratories
- **Nordtest 569**
  Internal Quality Control – Handbook for Chemical laboratories
- **Nordtest 604**
  Uncertainty from sampling – A Nordtest Handbook
- **Nordtest 626**
  In-house calibration and control of piston operated pipettes

A revised version of the Nordtest 569 6$^{th}$ edition was recently published.
The main updates for this 6th edition are:

- now only two rules recommended for the daily evaluation of the quality control;
- now discussing the frequency and how to reduce the false out-of-control situations;
- more focus on target control limits, when possible, based on uncertainty requirements;
- introducing control charts with only action limit.

The former editions were published in many languages. Nordtest welcomes volunteers for the translation.

The Nordtest 537 is now under review. Issues that are discussed in the working group are the following:

- Change title – new title *Handbook for evaluation of measurement uncertainty in testing laboratories*. The Handbook was developed mainly for environmental laboratories but have been used by laboratories from many different sectors. Therefore, *environmental* was changed to *testing.* Since uncertainty is difficult to calculate exactly the word *calculation* was changed to *evaluatio*n.
- New approach for revaluation of bias from proficiency testing results.
- Include reference *Measurement uncertainty in clinical chemistry*, ISO 20914
- Update to follow the new *Uncertainty for water analysis*, ISO 11352 2025 –This includes estimate of within-lab reproducibility for control charts with only action limits.
- Include reference *Reference materials – Requirements and recommendations for use*, ISO 33403 (2024) which in detail discuss how to handle bias from several CRM.
- Setting LOQ – A possibility to use different factors for LOQ dependent on the expanded uncertainty in order to be able to use relative uncertainty in the whole measurement range.
- Discuss equation for uncertainty over the concentration ranges.

The current state. Of Nordtest 537 will be presented.

For Nordtest 604 and 626 no revision is planned – they are from year 2020 and 2025 respectively.

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# OC02

## The use of collaborative trial in sampling for the validation of measurement procedures

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A new guidance document published by Eurachem in December 2024 describes approaches for validating measurement procedures that include primary sampling (VaMPIS) [1]. A methodology proposed for demonstrating fitness for purpose of the complete measurement procedure is the estimation of measurement uncertainty (MU), followed by comparison with a target MU. The approach taken to this comparison is referred to as the Optimised Uncertainty (OU) approach. The Guide also recommends estimating MU using the Duplicate Method [2]. Worked examples are provided for two scenarios: (1) *ex situ* laboratory analysis, where the analytical method has already been validated; and (2) an *in situ* method using portable X-ray fluorescence, where both sampling and analysis were validated simultaneously.

An alternative way of estimating MU is the use of a Collaborative Trial in Sampling (CTS). This has the advantage of incorporating between-sampler bias (expressed as a variance component) into the MU estimate, thereby providing a more realistic estimate. However, no worked example of this approach was included in the Guide. To address this gap, a recent submission to *Accreditation and Quality Assurance* presents a worked example of applying a CTS to the OU approach within the VaMPIS framework [3].

The data for this study were derived from a previously published sampling proficiency test (SPT) in which each participant used the same sampling procedure[4], and analysed using two pieces of software developed by the Royal Society of Chemistry Analytical Methods Committee [5]. Minor modifications to the normal use of the software were required to accommodate the specific characteristics of the SPT dataset. Application of the OU approach to the CTS data indicated that the measurement procedure was fit for purpose. However, when the same data were averaged to simulate results from the Duplicate Method (i.e. a single sampler), the procedure appeared overly conservative. This (erroneously) suggests that, in this case, there may be scope for cost savings through acceptance of a higher MU.

These findings highlight the value of CTS-based approaches to validation studies, where a more realistic estimate of MU can be achieved. Practical aspects of implementing the OU approach will also be discussed briefly, including the selection of the cost of misclassification and the analyte concentration at which optimisation is performed.

**References:**

[1] M.H. Ramsey, P.D. Rostron, F.C Raposo (eds.) Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide: Validation of Measurement Procedures that Include Sampling, Eurachem, 2024. https://www.eurachem.org/index.php/publications/guides/vampis
[2] M.H. Ramsey, S. L. R. Ellison, P. D. Rostron (eds.) Eurachem/EUROLAB/ CITAC/Nordtest/ AMC Guide: Measurement uncertainty arising from sampling: a guide to methods and approach, Second Edition, Eurachem, 2019. http://www.eurachem.org/index.php/publications/guides/musamp
[3] M.H. Ramsey, P.D. Rostron "Collaborative Trial in Sampling for Validation of Measurement Procedures that Include Sampling" Under review by Accred. Qual. Assur, submitted April 2026.
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[5] RANOVA and OptiMU software, freely available from AMC (RSC UK) https://members.rsc.org/site/content/Community/SubjectCommunity/Analytical_Science_Community/AMCSoftware.aspx

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# OC03

## The Fit for Purpose of Methods in Marine Matrices: The Portuguese Hydrographic Institute approach

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The adequacy of methods to be applied to marine matrices is a very complex and challenging issue. This complexity arises mainly from two factors: the effect of salinity, which usually causes interferences that need to be minimized, and the typical concentration range of some constituents and contaminants, which can differ significantly from those of non-marine matrices, such as fresh waters or terrestrial soils. As a result, international standards are not, often, adequate to be used directly in the analysis of such matrices. Thus, they need to be adapted and, consequently, validated, in order to prove that they are fit for the intended use [1].
The Portuguese Hydrographic Institute's laboratories are accredited according to ISO/IEC 10725 for the analysis of several chemical parameters in saline waters and marine sediments. As part of its efforts to ensure the robustness of the produced results, it developed internal procedures to determine a method's fitness for purpose and define quality control criteria to be used in routine. The selected approach, based on several standards and guidelines (e.g., from Eurachem/CITAC, Relacre, Nordtest and ISO), uses internal and external (when available) data and allows the selection of different strategies to evaluate the method's performance characteristics [1-5].
The approach was implemented on an Excel® spreadsheet that allows the selection of the strategy to be used for each performance characteristic, calculates its value and verifies if each one of these characteristics is compliant with the *a priori* requirements defined by the laboratory. In the end, a summary of the obtained results is produced, which allows to determine the method's fitness for purpose and to define quality control criteria to be used in routine.

**References:**

[1] B. Magnusson, U. Örnemark (eds.), Eurachem Guide: The fitness for purpose of analytical methods: A laboratory guide to method validation and related topics, 2nd edition, Eurachem, 2014.
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[3] ISO 11352:2012. Water quality – Estimation of measurement uncertainty based on validation and quality control data. International Organization for Standardization.
[4] S. L. R. Ellison, A Williams (eds.), Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, 3rd edition, Eurachem, 2012.
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# OC04

## Assignment of Self-Certified Reference Values for PhotonAssay using ISO 33405:2024

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ISO 33405:2024 [1] (previously ISO Guide 35:2017) has formalised statistical requirements for certified reference material (CRM) characterisation and value assignment. This study presents a method-specific value assignment of commercially available gold and silver CRMs using PhotonAssay™ as an alternative to existing measurement methods such as fire assay or acid digestion.

Multi-instrument PhotonAssay data were evaluated using a hierarchical random-effects model to estimate between-instrument and within-instrument variance components. Variance estimation was performed using both Analysis of Variance (ANOVA) and Restricted Maximum Likelihood (REML) mixed-effects modelling to assess robustness. Assigned values were determined from unweighted instrument means, with combined uncertainty incorporating both repeatability and between-instrument contributions. Expanded uncertainties were calculated using Welch–Satterthwaite effective degrees of freedom.

Comparison of ANOVA and REML approaches showed minimal differences in assigned concentration (<0.1%) and small differences in expanded uncertainty (2–5%), confirming model stability. This study proposed the above as a compliant framework for method-specific CRM assignment through ISO 33405, supporting the accredited implementation of PhotonAssay™ as an analytical method using existing reference materials.

**References:**

[1] International Organization for Standardization, Reference materials — Approaches for characterization and assessment of homogeneity and stability (ISO 33405:2024), https://www.iso.org/standard/84226.html (accessed 13 February 2026)

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# OC05

## Measurement Uncertainty in Chemical and Microbiological Analyses: Implications for Conformity Assessment of Live Bivalves

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Evaluation of measurement uncertainty (MU) is a requirement for laboratories operating under ISO/IEC 17025 accreditation systems, including those performing chemical and microbiological quantitative analyses in foodstuffs. Measurement uncertainty can be defined as a parameter associated with the result of a measurement that characterizes the dispersion of values that could reasonably be attributed to the measurand. The main sources of uncertainty must be identified, and these are usually associated with the key stages of the methodologies.

The estimation of measurement uncertainty (MU) for chemical methods applied to seafood is generally based on the Eurachem Guide (2012) [1], whereas for microbiological methods within the food chain, including bivalves, it is calculated according to ISO 19036 (2019) [2]. When conducting official controls of live bivalve molluscs, including the monitoring of production areas, there is currently no guidance on the application of measurement uncertainty in the conformity assessment of primary production and/or live bivalves placed on the market [3].

In this study, several scenarios are presented involving results obtained from chemical methods used to quantify Cd, Hg, and Pb contaminants in bivalves. Additionally, various scenarios are provided for assessing faecal contamination levels in live bivalves through the quantification of E. coli using the most probable number (MPN) technique.

The results indicate the need to establish a decision rule for these parameters, particularly for chemical contaminants when their concentrations are close to EU regulatory limits. Regarding E. coli levels, the distributional uncertainty associated with an MPN value of 230 may reach 700. This latter value corresponds to the maximum permitted in 20% of samples or sample units collected from production areas with the highest sanitary status (Class A) or from live products placed on the market [3].

Consequently, applying measurement uncertainty (MU) in the conformity assessment of bivalves from primary production can significantly affect the management of production areas, including their prohibition or reclassification, with important implications for aquaculture and fishing. Therefore, a harmonized European decision rule is needed to define how MU is considered when assessing compliance with specified requirements.

Funding: This work was supported by the national project SNMB-Monit 2026 (MAR2030).

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[1] S. L. R. Ellison, A Williams (Eds), Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, Eurachem, 2012.
[2] ISO 19036, Microbiology of the food chain — Estimation of measurement uncertainty for quantitative determinations, 38 pages, 2019.
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# OC06

## Metrological Sound Assessment of the Risk of Food Exposure to Nitrate from Consumers of a Public Canteen

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The management of food safety involves determining safe toxicological limits for relevant food components and monitoring the risk of these limits being exceeded by food consumption, considering component levels in foods, consumed quantities and consumer body weight. This can be assessed through specific consumption scenarios or by developing more general probabilistic models that account for the variability and uncertainty of the input variables. When determining the levels of food components, estimated values are often correlated because they use the same instrumental calibration, potentially affecting the consumption risk estimate [1]. The growing use of nitrate in food and agriculture as a preservative and fertiliser constitutes a potential food risk due to its role as a precursor to potentially carcinogenic molecules such as *N*-nitrosamines [2]. This work describes a Monte Carlo simulation [3] of the probability that students at the University of Lisbon ingest nitrate from lunch and dinner at a public canteen at the University, exceeding the acceptable daily intake of nitrate (ADI), which is 3.7 mg/kg of body weight [4]. Lognormal distributions of student weights, derived from available statistics, are considered. Fifty meals collected from the canteen were characterised for total mass and nitrate mass fraction by ion chromatography (IC) with UV/Vis detection after extraction with water at 50 °C. Since diluted extracts from various meals were quantified using weekly produced IC calibration curves, the impact of this correlation on risk determinations was assessed. Risk simulations considering correlated and independent mass concentrations from extracts were equivalent, proving that the analytical procedure simplification did not affect the determination. Considering the entire student population or only lighter female students, the risk of ingestion, quantified as the probability that a student consumes nitrate above the ADI, is 0.43 % and 1.2 %, respectively. Since the public canteen uses generally available food products and follows regular Portuguese culinary practices, the risk assessment can be extrapolated to a wider population of young adults. The estimated intake risk is considered adequate (≤ 5%), although it does not include any other meals or beverages consumed over the day. The developed tool is implemented in a user-friendly Excel file that can be adapted to other ingestion scenarios.

**References:**

[1] F. R. Lourenço, R. Bettencourt da Silva, “Risk of false conformity decisions of multicomponent items controlled by correlated measurement results due to the sharing of analytical steps”, Talanta 196 (2019) 174-181.
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[4] Commission Regulation (EU) 2023/2108 of 6 October 2023 amending Annex II to Regulation (EC) No 1333/2008 of the European Parliament and of the Council and the Annex to Commission Regulation (EU) No 231/2012 as regards food additives nitrites (E 249-250) and nitrates (E 251-252)

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# Poster Communications

# P01

## Assignment of Self-Certified Reference Values for PhotonAssay using ISO 33405:2024

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ISO 33405:2024 [1] (previously ISO Guide 35:2017) has formalised statistical requirements for certified reference material (CRM) characterisation and value assignment. This study presents a method-specific value assignment of commercially available gold and silver CRMs using PhotonAssay™ as an alternative to existing measurement methods such as fire assay or acid digestion.

Multi-instrument PhotonAssay data were evaluated using a hierarchical random-effects model to estimate between-instrument and within-instrument variance components. Variance estimation was performed using both Analysis of Variance (ANOVA) and Restricted Maximum Likelihood (REML) mixed-effects modelling to assess robustness. Assigned values were determined from unweighted instrument means, with combined uncertainty incorporating both repeatability and between-instrument contributions. Expanded uncertainties were calculated using Welch–Satterthwaite effective degrees of freedom.

Comparison of ANOVA and REML approaches showed minimal differences in assigned concentration (<0.1%) and small differences in expanded uncertainty (2–5%), confirming model stability. This study proposed the above as a compliant framework for method-specific CRM assignment through ISO 33405, supporting the accredited implementation of PhotonAssay™ as an analytical method using existing reference materials.

**References:**

[1] International Organization for Standardization, Reference materials — Approaches for characterization and assessment of homogeneity and stability (ISO 33405:2024), https://www.iso.org/standard/84226.html (accessed 13 February 2026)

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# P02

## Development of chemical metrology tools for standardization of the Brazilian hops analysis

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The incorporation of Quality Management Systems in analytical laboratories has become fundamental in the face of increasing market demands and regulatory agencies, with the ISO/IEC 17025 [1] standard widely recognized as the main international reference for demonstrating technical competence. Among its requirements, the use of Certified Reference Materials (CRMs) and participation in Proficiency Testing (PT) stand out as fundamental instruments for evaluating the reliability, accuracy, and metrological traceability of analytical results. In this context, this work aims to develop a Certified Reference Material containing the main chemical compounds responsible for the sensory properties of Brazilian hops, as well as its application in a proficiency test using four distinct varieties of Brazilian hops representative of national production.

Hops (Humulus lupulus L.) are one of the most important ingredients in the brewing industry, often called the "soul of beer" due to their direct influence on aroma, flavor, bitterness, and foam stability. α, β, and γ resins and essential oils are primarily responsible for the product's sensory characteristics. Currently, analytical reference methods for hop characterization are mostly established by the American Society of Brewing Chemists (ASBC) [2]. However, in the Brazilian context, there is a lack of a specific national technical standard, as well as an absence of consolidated metrological studies and proposals for Certified Reference Materials aimed at quality control of national hops. As a preliminary step in this study, a quantity of national hops was subjected to supercritical fluid extraction using carbon dioxide ($CO_2$), aiming to produce a homogeneous material containing the compounds of interest to be certified in the material to be produced. The results obtained were promising, showing adequate recovery of the target compounds (initially was evaluated the α resin compounds by spectrophotometry) and preservation of relevant chemical characteristics, indicating the viability of the technique to produce a reference material. The production of the Reference Material will follow the guidelines of ISO Guide 33405 [3], including studies of homogeneity, short- and long-term stability, characterization by liquid chromatography with UV detection and mass spectrometer detector, as well as the estimation of the expanded combined uncertainty, contributing to the metrological traceability, analytical reliability, and competitiveness of Brazilian hops.

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[1] ISO/IEC 17025:2017 – *General requirements for the competence of testing and calibration laboratories*. Geneva: ISO, 2017.
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# P03

## Long-Term Analytical Performance and Stability of Silver Alloy Reference Materials

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Silver alloy reference materials with compositions representative of those commonly used in jewellery production are generally considered chemically stable. However, a well-known property of silver alloys is their tendency to tarnish when exposed to air, primarily through reaction with hydrogen sulfide, forming silver sulfide ($Ag_2S$). Other environmental factors, including relative humidity and the presence of oxidizing species, may also influence the tarnishing process. This study examines the long-term analytical behaviour of silver alloy reference materials through years of monitoring and evaluates their stability under designed extreme exposure conditions.

Given the Institute of Metrology of Bosnia and Herzegovina extensive experience in silver alloy analysis, formal documentation of stability is necessary to support the production and shipment of reference materials, where environmental conditions may influence reference values. Furthermore, the recent rise in silver prices and its growing role as an investment asset have increased demand for accurate silver alloy testing. The objective is therefore to provide stable, well-characterized reference materials that ensure reliable and traceable analytical results.

In this paper two silver alloy reference materials were assesed: MA/Ag.10 (925 mg/g Ag) and MA/Ag.06 (950 mg/g Ag), with copper as balance. The first was monitored over five years and included in a multilateral proficiency testing scheme, while the second has been monitored for the past six years with an ongoing observation. Reference values were evaluated using ISO 13756 and confirmed either through proficiency testing results or recharacterization using additional method - ISO 11427. No statistically significant changes in reference values were observed.

An additional stability study was conducted according to ISO 33405 using an isochronous design to simulate extreme conditions that may occur during transport and handling. Samples were exposed for one month to ambient laboratory and outdoor atmospheric conditions in Sarajevo, with environmental parameters recorded using a thermohygrometer and by data from the Federal Hydrometeorological Institute of Bosnia and Herzegovina. Samples were subsequently analyzed.

The results obtained so far show no deviation from the reference value. The stability monitoring experiment remains ongoing to further evaluate long-term behavior.

**Keywords:** reference materials, silver alloys, stability study, isochronus design, surface tarnishing, precious metals, analytical metrology, long-term monitoring.

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# P04

## Silver Telluride Nanocrystals as SEIRA-active Amplifiers for the Detection of Pharmaceuticals

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The detection and quantification of molecular species at trace levels remain a critical challenge across chemistry, environmental science, and biomedicine. One pressing issue is monitoring organic microcontaminants, such as pharmaceuticals, in aquatic environments, where continuous discharges from wastewater, industrial, and agricultural sources lead to their persistence and bioaccumulation. These compounds are associated with ecological disruption and potential health risks, including the spread of antimicrobial resistance. Despite these implications, practical and efficient monitoring strategies are still limited. Conventional chromatographic methods (HPLC-MS, GC–MS) provide excellent sensitivity and selectivity but rely on costly instrumentation, specialized operators, and labor-intensive sample preparation, making them unsuitable for routine or large-scale water analysis [1]. These constraints have encouraged the development of simpler, faster, and more sustainable approaches.

Surface-enhanced infrared absorption (SEIRA) spectroscopy has emerged as a promising tool for sensitive and portable detection of microcontaminants. As a Green Analytical Chemistry (GAC) technique, SEIRA requires minimal sample handling, enables *in situ* measurements, and generates minimal waste [2]. Infrared (IR) spectroscopy is inherently powerful for molecular identification through the analysis of vibrational signatures, but its utility diminishes when dealing with compounds that exhibit weak IR absorption signals or occur at low concentrations. To address this, nanomaterials have been employed as enhancement substrates, facilitating molecular detection at trace levels.

Silver chalcogenide quantum dots (QDs) represent a new class of SEIRA-active materials offering favorable optical properties in the near-infrared region while remaining free of toxic metals [3,4]. Our group has developed three different aqueous synthesis routes for silver telluride ($Ag_2Te$) QDs using different stabilizing agents: mercaptosuccinic acid (MSA), cysteine (Cys), and glutathione (GSH), to investigate their performance in detecting the pharmaceutical venlafaxine (VEN). All $Ag_2Te$ QDs exhibited SEIRA activity, with enhancement factors (EF) varying according to the stabilizer. Preliminary results showed an EF of up to 17× at the 1611 $cm^{-1}$ band of VEN, demonstrating substantial signal amplification. These findings highlight the stabilizing agent's key role in modulating analyte–substrate interactions and confirm the potential of $Ag_2Te$ QDs as efficient, sustainable SEIRA-active materials for sensitive pharmaceutical detection in water.

**Acknowledgments:** The authors acknowledge financial support for the project NanoSEIRA (COMPETE2030-FEDER-00866600; doi.org/10.54499/2023.16977.ICDT), and FCT – Fundação para a Ciência e Tecnologia I.P, under the project CESAM-Centro de Estudos do Ambiente e do Mar (UID/50017/2025, doi.org/10.54499/UID/50017/2025; and LA/P/0094/2020, doi.org/10.54499/LA/P/0094/2020).

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# P05

## Bioanalytical Method Validation: XVAL 1.0 – A Practical Tool for Analysts

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In the various fields of analytical toxicology (forensic, clinical, occupational health, environmental, addiction, doping control, and alternative matrices), reliable and interpretable results can only be obtained through fully validated analytical methods.

Because toxicological analyses are frequently performed in biological matrices such as plasma, blood, and urine, they involve specific constraints compared with conventional chemical analyses. Consequently, both method validation and routine application in bioanalysis require dedicated approaches.

Several guidance documents have been published to support bioanalytical method validation, including the European Medicines Agency (EMA) guideline [1] which remains a major reference in this field.

The purpose of this work was to provide analysts with a practical tool for implementing these recommendations. XVAL 1.0 is a spreadsheet-based utility developed entirely in Microsoft Excel®. It enables users to perform the statistical calculations required for bioanalytical method validation according to the general validation scheme and methodology proposed by EMA experts.

Designed to be free, accessible, and practical, this tool aims to support analysts in applying current bioanalytical validation recommendations in a simple and operational manner.

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# P06

## Screening, Confirmation and Quantification of Cocaine by GC–MS/MS in Samples of Drug Checking Service

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Cocaine is the second most used substance of abuse in Europe, with 4.5 million users (approximately 1.6% of European adults). There is a high availability of this substance on the illicit market, as evidenced by the increased number of cocaine seizures. In Europe, cocaine is more commonly found in its salt form (powder) than in its freebase form (crack), its use is associated with urban environments. The cocaine purity has increased in Europe, while still showing wide variability (17 - 96%) [1]. This variability is partly explained by the presence of cutting agents, which are added to increase volume (e.g., talc or sugar) or to mask the purity of cocaine (e.g., caffeine or lidocaine) [2]. The objective of this study was to develop and validate a gas chromatography–tandem mass spectrometry (GC–MS/MS) method for the identification and quantification of cocaine and tested in samples submitted to a drug checking service (DCS). The sample preparation involved extraction with methanol and, in some cases, filtration of the resulting solution. An initial screening of the samples was performed in SCAN mode, allowing the detection and tentative identification of cocaine and potential cutting agents. Subsequently, cocaine was confirmed, and its quantification was carried out using multiple reaction monitoring (MRM) mode, with two transitions (*m/z* 182 > 93 and *m/z* 182 > 122) monitored to ensure both selectivity and sensitivity. The method demonstrated limits of detection and quantification of 0.05 and 0.1 mg/L, respectively, good linearity ($r^2 \geq$ 0.99), and high precision (< 15%). Furthermore, it was observed presence of cocaine with purity above 50% in samples, and the detected adulterants were phenacetin, caffeine, and levamisole, while common impurities include ecgonidine methyl ester, tropacocaine, and norcocaine. In conclusion, the proposed method provides a sensitive and selective approach for the detection and quantification of cocaine, supporting reliable analytical workflows in DCS.

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# P07

# Implementation and Validation of a Bioanalytical Method for Multidrug Detection in Routine Laboratory Practice

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The consumption of alcohol and other substances of abuse poses significant risks, including dependency, health complications, and amplified dangers from polysubstance use [1,2]. Consequently, it is important to reinforce the need for robust analytical strategies capable of comprehensive polysubstance detection in biological matrices [3]. This work presents two complementary, fully validated methodologies that combine high-throughput liquid chromatography–tandem mass spectrometry (LC–MS/MS), and a novel sorbent-based microextraction approach for multi-analyte determination in whole blood and urine, respectively.

The LC–MS/MS method was developed for the simultaneous quantification of phosphatidylethanol (PEth) homologues (PEth 16:0/18:1, 16:0/18:2, 18:0/18:1), cocaine, its metabolites, and additional drugs in whole blood [4]. Sample preparation was performed via liquid–liquid extraction, followed by chromatographic separation on a C18 column under basic buffer-free conditions. The method demonstrated strong validation performance, with inter-assay precision and accuracy within ± 16 % across multiple concentration levels. Recovery ranged from 42–79 % for most analytes, with acceptable matrix effects (± 25 %) for most analytes. The internal standards corrected matrix effects for each compound. This approach enables sensitive and reliable detection of combined alcohol and drug exposure, addressing a critical need in forensic and clinical toxicology.

The Sorbent Strip Microextraction (SSµE) technique coupled with HPLC-DAD, introduces an innovative, eco-friendly device that simplifies sample preparation while reducing solvent consumption and waste [5]. Method validation showed excellent analytical performance, including recoveries of 78–108 %, trueness within ± 7 %, and precision below 13 %. The method exhibited strong linearity ($r^2 \geq 0.9922$) over relevant concentration ranges and was successfully applied to real samples, confirming its reliability for routine screening.

These validated methodologies, developed and assessed in accordance with established bioanalytical method validation guidelines, demonstrate robustness, accuracy, and reliability for multidrug detection in biological matrices. By combining the high sensitivity and selectivity of LC–MS/MS with the simplicity and sustainability of SSµE/HPLC-DAD, they provide complementary and fit-for-purpose solutions that support the effective implementation of validated analytical methods in routine laboratory practice, with direct applicability to clinical and forensic settings, and public health surveillance.

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# P08

## The Fit for Purpose of Methods in Marine Matrices: Validation of a Method for Determining TPHs in Water Samples Using an Internal Procedure Developed in an Excel® Spreadsheet

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Methods for analysing marine matrices are difficult to apply considering salinity interferences and concentration ranges that often differ greatly from non-marine environments. As a result, international standards usually require adaptation and validation to ensure they are fit for their intended use [1].
The Portuguese Hydrographic Institute's laboratories are accredited in accordance with ISO/IEC 17025 for the analysis of several chemical parameters in marine environment matrices. In order to ensure the robustness of the results produced, internal procedures were developed to determine the fit for purpose of a certain method, thus defining quality control criteria to be used in routine.
The approach, based on several national and international standards and guidelines, implemented in an Excel® spreadsheet, allows the selection of the strategy to be used for each method performance characteristics, the calculation of its value, and the verification of whether each of these characteristics complies with the requirements previously defined by the laboratory [1-5].
Finally, a summary of the results obtained makes the assessment possible whether a method is fit for purpose and which criteria regarding quality control is defined and applied in routine analysis. The use of the spreadsheet to validate the method for determining total petroleum hydrocarbons in water samples is demonstrated.

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# P09

## Development, DoE Optimization and Validation of a Chromatographic Method for the Determination of Synthetic Opioids

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The global unregulated drug market is increasingly threatened by extremely potent new synthetic opioids, namely fentanyls and the more recent class of nitazenes, both associated with very high overdose rates worldwide. Many of these substances are reported to be hundreds to thousands of times more potent than morphine or even heroin. Due to their high potency, even minimal consumption doses can lead to severe health effects or even fatal overdoses, making them a public health concern. Their growing prevalence is driven by ease of synthesis, low production costs, and the ability to circumvent existing drug control frameworks. The detection of these compounds presents some analytical challenges, as conventional toxicological screening methods often fail to identify these compounds and their typically low concentrations in biological matrices require highly sensitive analytical techniques [1-3].
The present work describes a simple, fast, and successful method for monitoring synthetic opioids (fentanyls and nitazenes) in whole blood samples.
The sample preparation step consisted in 50 µL of whole blood protein precipitation with refrigerated acetonitrile containing formic acid and was optimized using Full Factorial Design to achieve the best conditions for compounds extraction from matrix. Following centrifugation, the supernatant extract is directly injected into an ultra-high-performance liquid chromatograph coupled to a triple quadrupole linear ion trap mass spectrometer (Sciex UHPLC-QTRAP-MS® 6500+) and analyzed in a 5-minute run in Multiple Reaction Monitoring mode with 2 transitions for each compound. The developed analytical methodology was fully validated according to the guiding principles of the ANSI/ASB Standard 036.
The methodology linearity was assessed between 1 and 20 ng/mL. The precision and accuracy were satisfactory, with values <15% and within ±15% (20% at the Limit of Quantification), respectively. The limits of detection were between 0.1 and 1 ng/mL, depending on the compound. Dilution ratios were also successfully evaluated. Selectivity was confirmed by analyzing spiked samples containing several therapeutic drugs and other drugs of abuse.
This method provides a robust and sensitive approach for the detection of fentanyls and nitazenes and contributes to improving analytical capabilities in forensic and toxicological investigations involving emerging synthetic opioids.

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# P10

# Validation of an Analytical Method for Drug Research in Hair in the Context of Drug Facilitated Sexual Assault

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In the investigation of drug-facilitated sexual assault (DFSA), hair has emerged as a primary alternative matrix due to its extended detection window. This is critical given the frequent delays in forensic reporting often triggered by drug-induced amnesia which typically exhausts the viability of traditional fluids like blood or urine [1]. Benzodiazepines, being globally prevalent in clinical prescriptions, remain the most frequently identified substances in these incidents alongside ethanol [2]. Consequently, establishing robust, high-sensitivity analytical strategies is vital for capturing the trace levels resulting from single-dose exposures. The aim of this work was the development and validation of a simple method for the detection of substances related to DFSA cases, with particular emphasis on benzodiazepines and their metabolites, allowing its implementation in routine toxicological analysis.

Hair samples (20 mg) were washed and dried before being transferred to 2 mL Precellys® tubes for the pulverisation stage. Within these same tubes, the samples were fortified with target analytes and internal standards. The scope of the study included 30 substances, with linear ranges defined from 1–250 pg/mg or 5–250 pg/mg, depending on the analyte. The extraction procedure consisted of direct extraction with 500 µL of methanol. An ultra-high-performance liquid chromatography coupled with triple quadrupole linear ion trap tandem mass spectrometry (UHPLC-QTRAP-MS/MS) was used for analyte determination, operating in MRM mode with two transitions monitored for each analyte.

The method was validated following ANSI/ASB Standard 036 criteria, encompassing selectivity, linearity, limits of detection (LOD), lower limits of quantification (LLOQ), precision, bias, matrix influence, processed sample stability, and carry-over. LOD values were successfully reached at 1 and 5 pg/mg, depending on the analyte. This validated multi-compound method allows for the simultaneous detection of additional substances, such as drugs of abuse and other pharmaceuticals. The method's practical utility was confirmed through its successful application to authentic routine cases, involving both clinical and post-mortem hair specimens.

This analytical method is characterised by its simplicity, faster sample preparation and reduced environmental impact. It offers a robust and sustainable alternative to traditional extraction techniques, proving highly effective for high-throughput forensic applications.

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# P11

## Metrological Challenges and Measurement Infrastructure for Ammonia ($NH_3$) as a Renewable Hydrogen Carrier

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Ammonia ($NH_3$) is emerging as a key energy carrier in the transition toward a low-carbon economy, particularly as a carrier and storage medium for renewable hydrogen.
The use of ammonia as a hydrogen vector requires its subsequent thermochemical decomposition (cracking), generating a gaseous mixture primarily composed of hydrogen and nitrogen, with possible residual ammonia and trace impurities. For downstream applications such as fuel cells or mobility systems, hydrogen purity requirements are extremely stringent. Impurities at the level of parts per million (ppm) or parts per billion (ppb), including $NH_3$, CO, $CO_2$, $H_2S$, $NO_x$, and $H_2O$, can poison catalysts, degrade membranes, and compromise system durability. Accurate quantification of these species is therefore essential, placing metrology at the core of ammonia-based energy systems, as reliable measurements underpin both technical validation and market acceptance.
Significant measurement challenges arise throughout the ammonia value chain, including production, storage, transport, cracking, and emission monitoring. Ammonia is a reactive and adsorptive molecule, prone to interactions with cylinder walls and sampling systems, particularly at low concentration levels. The preparation of stable certified reference gas mixtures containing $NH_3$ and relevant impurities requires strict gravimetric procedures, high-purity materials, controlled cylinder treatments, and rigorous uncertainty evaluation. The stability of low-level ammonia mixtures over time is also critical for calibration reliability.
The European project MetNH3Energy [1] addresses these gaps by developing a comprehensive metrological framework to support ammonia use in emerging applications, including the production of primary reference materials [2], establishment of traceable analytical methods, validation of calibration procedures [3], and development of emission monitoring techniques. Particular emphasis is placed on traceability to the International System of Units (SI), interlaboratory comparability, and uncertainty evaluation [4].
Within this framework, the Portuguese Institute for Quality (IPQ), through its Reference Gas Laboratory, plays a key role in the validation of analytical methods for $NH_3$ determination and in the rigorous evaluation of measurement uncertainties, considering contributions from preparation, gas purity, calibration, stability, and sampling. IPQ is also actively involved in the characterisation of critical impurities in ammonia matrices, particularly nitrogen dioxide ($NO_2$) and sulfur dioxide ($SO_2$), assessing their stability and analytical impact, thereby strengthening confidence in trace-level measurements required for process control and emission monitoring.
In summary, ammonia represents a promising hydrogen carrier capable of facilitating large-scale renewable energy integration. However, its viability depends critically on high-accuracy gas metrology, certified reference materials, validated analytical methods, reliable uncertainty evaluation, and harmonised measurement standards, enabling safe industrial adoption and long-term sustainability within the ammonia-based energy economy.

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# P12

## Metrological Sound Assessment of the Risk of Food Exposure to Nitrate from Consumers of a Public Canteen

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The management of food safety involves determining safe toxicological limits for relevant food components and monitoring the risk of these limits being exceeded by food consumption, considering component levels in foods, consumed quantities and consumer body weight. This can be assessed through specific consumption scenarios or by developing more general probabilistic models that account for the variability and uncertainty of the input variables. When determining the levels of food components, estimated values are often correlated because they use the same instrumental calibration, potentially affecting the consumption risk estimate [1]. The growing use of nitrate in food and agriculture as a preservative and fertiliser constitutes a potential food risk due to its role as a precursor to potentially carcinogenic molecules such as *N*-nitrosamines [2]. This work describes a Monte Carlo simulation [3] of the probability that students at the University of Lisbon ingest nitrate from lunch and dinner at a public canteen at the University, exceeding the acceptable daily intake of nitrate (ADI), which is 3.7 mg/kg of body weight [4]. Lognormal distributions of student weights, derived from available statistics, are considered. Fifty meals collected from the canteen were characterised for total mass and nitrate mass fraction by ion chromatography (IC) with UV/Vis detection after extraction with water at 50 °C. Since diluted extracts from various meals were quantified using weekly produced IC calibration curves, the impact of this correlation on risk determinations was assessed. Risk simulations considering correlated and independent mass concentrations from extracts were equivalent, proving that the analytical procedure simplification did not affect the determination. Considering the entire student population or only lighter female students, the risk of ingestion, quantified as the probability that a student consumes nitrate above the ADI, is 0.43 % and 1.2 %, respectively. Since the public canteen uses generally available food products and follows regular Portuguese culinary practices, the risk assessment can be extrapolated to a wider population of young adults. The estimated intake risk is considered adequate (≤ 5%), although it does not include any other meals or beverages consumed over the day. The developed tool is implemented in a user-friendly Excel file that can be adapted to other ingestion scenarios.

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# P13

## Uncertainty estimation in the determination of lithium concentration in mining soil samples

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Lithium plays an essential role in the ongoing energy transition, particularly in the fields of electric mobility and energy storage, as a key chemical element in the development of batteries for hybrid and electric vehicles, as well as other applications [1]. In fact, lithium-ion batteries contribute to decrease the reliance on combustion-based transport, thereby contributing to lower carbon dioxide emissions and thus mitigating the intensification of the greenhouse effect and the associated climate changes observed in recent decades. Within this context, Portugal stands out as the European Union country with the largest identified lithium resources and is among the top ten globally. Although, presently, there are no active lithium mining operations, ongoing exploration activities are increasing the known deposits data base. The main lithium projects in the country, which are intended to operate as open-pit and hybrid mining operations, are presently in a licensing stage process. Despite its strategic importance, lithium extraction is associated with considerable environmental challenges, particularly in the case of open-pit mining methods and therefore it is of most crucial importance to determine lithium concentration, as well as other heavy metals, in soil samples of future mining activity sites, thus contributing to the evaluating of environmental impact associated with ore extraction and processing activities and implementation of sustainable mining practices. Early-stage determination of lithium concentrations, prior to the onset of mining activities, must be carried out with the highest possible level of analytical rigor. This requires not only the use of robust and validated methodologies, but also a thorough assessment of the uncertainty associated with the measured concentration values [2], supporting the adequate and effective implementation of strategies for the prevention and mitigation of the environmental impacts associated with lithium mining activities.
In this context, our research group carried out an early-stage determination of lithium concentrations in a set of soil samples collected from Romano mine, located in the Montalegre district. Given the relatively high lithium content observed in these samples, quantification was achieved using flame photometry, an atomic emission spectrophotometric analytical technique [3]. This method is characterized by its operational simplicity, rapid response and relatively low cost associated. Furthermore, the compact and portable nature of the instrumentation allows for its transport and implementation in proximity laboratories or temporary facilities, such as those found in lithium mining activity sites. In addition to the concentration values obtained, the associated uncertainties sources were also identified, allowing for the estimation of the respective combined and expanded uncertainty, ensuring the reliability and robustness of the analytical results.

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# P14

## Dynamic Partitioning of Mechanical Transients in Potentiometric Measurements for GUM-Compliant Uncertainty Evaluation in Sustainable Monitoring Systems

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Potentiometric ion-selective electrodes encounter environmental disturbances that create non-Gaussian artifacts [1]. These transients complicate the objective evaluation of measurement uncertainty. Standard metrological frameworks often struggle to isolate these effects. This limitation leads to the significant overestimation of Type A components. Such errors compromise high-reliability sensing required for national green development research. Inaccurate uncertainty budgets increase the risk of incorrect conformity decisions. This study proposes a framework for the dynamic evaluation of electrochemical uncertainty.

We propose automated transient partitioning to preserve the integrity of precision estimates. Windowed robust regression removes 60 Hz mains interference. We implemented iteratively reweighted least squares with Huber weighting. This approach prevents transient outliers from biasing correction parameters. Synchronized hysteresis-based algorithms isolated mechanical events. This logic distinguishes sharp transients from slow chemical drift phenomena. Quiet measurement intervals establish the non-linear baseline for drift estimation.

A four-component uncertainty budget follows the JCGM 100:2008 framework [2]. It incorporates random noise. Mains residual was also included. Electrode drift and transient artifacts were quantified separately. We calculated standard uncertainties for each component across six distinct datasets. Three datasets provided undisturbed reference signals for baseline characterization. The remaining datasets contained deliberate mechanical transients generated by piezoelectric ignitions. Combined uncertainty calculations followed the law of propagation of uncertainty [2]. Validation utilized JCGM 101:2008 Monte Carlo simulations using fifty thousand samples [3].

Undisturbed reference signals established a stable expanded uncertainty floor ($U$, $k = 2$) of 0.0008 mV. Disturbances induced substantial inflation in the total uncertainty budget. The sodium electrode exhibited a 77.1-fold increase in expanded uncertainty during disturbance intervals. This value reached a maximum of 0.058 mV. The pH electrode showed a lower but significant 14.8-fold inflation factor. Component analysis revealed a clear shift in budget dominance. Transient artifacts dominated the uncertainty budget in most disturbed datasets.

Monte Carlo validation demonstrated agreement with JCGM 100:2008. The maximum relative deviation between the methods was 0.48% ($4.8 \times 10^{-3}$). This agreement confirms that linear propagation remains adequate for the modeled conditions. Numerical tolerance checks confirmed the convergence of simulation results. Dynamic partitioning ensures that transient effects are correctly classified without inflating reported precision. This methodology facilitates accurate conformity assessment and decision rules in high-precision sensing. High rigor supports data quality for industrial research and environmental monitoring.

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# P15

## Validation of a test method for the determination of metals in natural fresh waters and drinking water by TQ-ICP-MS

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This work aims to validate a test method for determining cadmium, lead, mercury, and nickel in natural freshwater and drinking water by triple quadrupole inductively coupled plasma mass spectrometry (TQ-ICP-MS).
Water, as a resource essential to life, must be free of elements toxic to human health, and their monitoring is important in supporting the management policies of this natural resource [1].
This inductively coupled plasma (ICP) spectroscopy technique is used to identify and measure elements based on the ionisation of these elements within the sample. The Mass Spectrometer (MS) separates the ions by their mass-to-charge ratio after they pass through the ICP. Finally, the detector contains the number of ions selected per second, allowing the instrument to determine the concentration of each chosen element. In this technique, it is possible to present the concentration value of a given metal through a mass/charge ratio (m/z) of an isotope [2]. Given that the abundances of isotopes are constant in nature, with exceptions, such as lead, where the isotopic ratios from the combustion of coal or gasoline are different from those from the smelting of ores, it is therefore necessary to read the sum of the 3 most abundant isotopes [3].
In the development of this work, some isotopes were tested in different reading modes, with the aim of choosing the most suitable isotope and reading mode for the determination of these metals. For this purpose, several parameters were also evaluated, such as linearity, analytical limits, interference validation, internal standard validation, intermediate precision, repeatability, analyte recovery and measurement uncertainty.
In the uncertainty calculation, this was carried out using ISO 11352:2025 standard [4], and the new Eurachem/CITAC guide “Evaluation of measurement uncertainty from internal precision and recovery data” [5]. Both approaches will be discussed in this work.

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# P16

## Impact of the Calibration Curve on Uncertainty Estimation and Reproducibility: A Theoretical Study

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The paper published by Monya Baker [1] intensified the discussion of a possible reproducibility crisis across several scientific fields. In the reported survey, chemistry appeared among the fields in which respondents reported greater difficulty to reproduce the published results from different papers. Reproducibility should be understood as the attainment of results for a given sample, analysed independently at different locations, that yield consistent results, rather than exact numerical coincidence, since strict equality is inconsistent with the inherent variability of measurements and the presence of random error [2]. In metrological terms, this interpretation is aligned with the JCGM [3] through adoption of the concept of metrological compatibility, i.e., comparison between results based on the difference between measured quantity values in relation to the uncertainty of that difference.

In a study published by Ferreira and co-workers [4], it was observed that the lack of reproducibility in analytical method development may be explained by the high uncertainty estimates obtained. The results indicated that 28% of the studies assessed in that article (covering papers from different journals) reported uncertainty estimates greater than 100% at the first points of the calibration curve; only 19% performed validation studies according to established protocols; and in only 17% of the validations did all performance characteristics meet the acceptance criteria. These findings support, with considerable probability, the presence of weaknesses associated with inappropriate validation practices and insufficient treatment of measurement uncertainty, with potential impact on reproducibility. Among the factors that most contribute to uncertainty, the calibration curve deserves particular emphasis. The equation used to estimate uncertainty associated with the calibration curve is already well established in references such as Eurachem [5], Skoog et al. [6], and Miller & Miller [7]; however, it is often neglected in practice.

A theoretical analysis of this equation makes it possible to highlight five main factors that may provide a significant contribution: (1) calibration range; (2) slope of the calibration line; (3) regression residuals; (4) number of calibration points; and (5) number of replicate measurements of the sample. This work aims to present, in a theoretical and straightforward manner, how these factors affect uncertainty estimation, in order to properly guide analytical chemists in producing fit-for-purpose calibration curves, thereby positively impacting the reproducibility of results.

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# P17

# A Practical and Metrological Approach to the Development of Qualitative Proficiency Test (Pt) in the Assessment of Clinical Diagnostic Capacity

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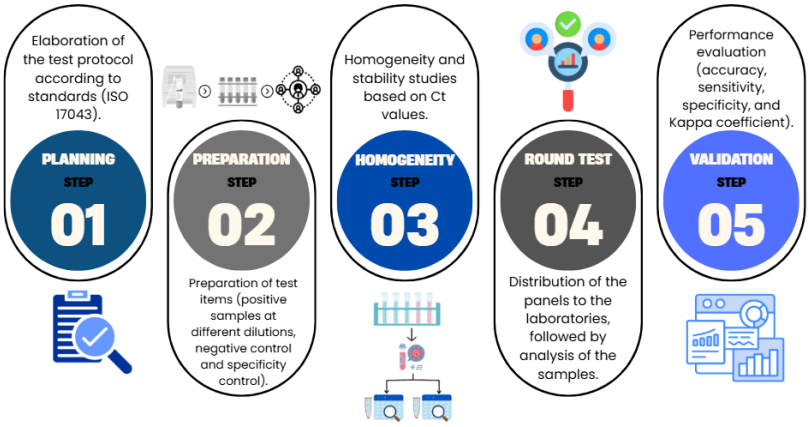
Qualitative analysis refers to analytical methods designed to determine the presence or absence of a substance based on its biological or physicochemical properties, generally providing a binary response ("yes" or "no") relative to a predefined cut-off concentration of the analyte [1,2]. These methods are widely used for screening purposes, particularly in the context of clinical diagnosis and epidemiological surveillance [2].

Diagnostic tests play a fundamental role in detecting infectious agents and supporting clinical and public health decision-making [3]. The reliability of these results depends on the implementation of robust quality assurance practices, including analytical method validation, quality control, and participation in proficiency testing [4,5]. Proficiency testing (PT) represents an essential metrological tool for evaluating laboratory technical competence and ensuring interlaboratory comparability of results [4,6].

In this context, the present study describes the development of a qualitative proficiency testing scheme to evaluate the diagnostic capability for SARS-CoV-2 detection using RT-qPCR. The scheme was designed in accordance with the requirements of the ISO/IEC 17043 and ISO 13528 standards, using an interlaboratory comparison model. The test panel consisted of five items, including three positive samples at different dilution levels, one negative control, and one specificity control containing inactivated Newcastle Disease Virus. The validation of the scheme included diagnostic performance indicators and interlaboratory agreement analysis, demonstrating the applicability of PT as a tool for monitoring and assuring quality in molecular diagnostic assays.

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# P18

## Development of a web-based tool for assessing false conformity decision risks in multiparametric analyses of pharmaceutical products

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In pharmaceutical quality control, analytical results support conformity assessment of several products and, in general, evaluation is based on multiple quality parameters for each product. A correct decision on the conformity depends on confidence in these results and must consider the measurement uncertainty associated with each parameter, as well as possible correlation effects among them, in order to maintain the risks of approving non-conforming products at acceptable levels. The objective of this study was to develop a tool to assess the risks of false conformity decisions in pharmaceutical product analyses, estimating both particular risk and total consumer's risk values, while accounting for measurement uncertainties and possible correlations among tested parameters. A Monte Carlo simulation algorithm was implemented using the Python programming language, employing the NumPy package for numerical computation, Matplotlib for graphical output, and Streamlit for the web-based interface. The resulting interface allows users to input the number of parameters, number of simulations, measured values and their uncertainties, specification limits, and correlation matrices. The output screen presents a summary table with the calculated risks, along with graphical representations of data distributions relative to specification limits and the effects of parameter correlation. The algorithm was further applied to quality control test results of 20 batches of seven different pharmaceutical products analyzed in our laboratory. The evaluated scenarios included up to six parameters, unilateral and bilateral specification intervals, and parameters with or without correlation, and showed consumer's risks ranging from negligible (<1% for acyclovir) up to more than 40% (for acetaminophen and ciprofloxacin). Higher total risks were obtained when measured values were close or above specification limits. The results highlight the critical role of measurement uncertainty and correlation effects in pharmaceutical quality assessment, particularly in multiparametric analyses, as their direction and strength can either amplify or reduce the overall probability of false acceptance. Therefore, the developed tool provides an efficient and flexible framework for estimating false conformity decision risks in routine analytical laboratories, facilitating practical implementation of risk-based decision-making aligned with metrological guidance, such as the recommendations of JCGM 106 and Eurachem/CITAC.

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# P19

## Metrological traceability for medical measuring devices using optical absorbance liquid filters through the degree of equivalence

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There is a lack of metrological traceability for the measurement results of optical absorbance obtained with the optical absorbance liquid filters (OALFs) which are currently used to calibrate medical measuring devices (MMDs). A European project, the "23RPT02 ETraceAbs - Establishing European traceability for medical measuring devices through optical absorbance liquid filters", was launched, in 2024, to bridge the gap among the different National Metrology Institutes or Designated Institutes concerning the manufacturing, the characterization and the use of the OALFs [1].

In the first stage of the ETraceAbs project [2], based on existing OALFs used in in vitro diagnostic medical devices, solutions containing potassium dichromate, nickel, cobalt, copper and chromium ions were optimized in terms of stability to be used as certified reference materials that meet the needs of characteristic wavelengths and absorbance levels. These solutions are then used in an intercomparison exercise that consists of a diagnostic intercomparison and a consolidation comparison. As they correspond to the usual operating conditions for spectrophotometric measurements of MMDs, the spectrophotometers' spectral interval ranges from 220 nm to 780 nm, for optical absorbance values from 0.001 to 3.000, corresponding to 99.77 % to 0.10 % transmittance values respectively. Measurements are performed at atmospheric pressure, with relative humidity between 30 % and 70 % and temperature between 15 °C and 40 °C, targeting relative absorbance measurement uncertainty from 0.3 % to 1.0 %. for different absorbance levels. The objective of the diagnostic measurement comparison is to demonstrate and correct unexpected issues in the optical absorbance measurements by the participants, to ensure suitable results of the consolidation comparison. It is performed in the "round robin" scheme involving four groups of laboratories, with three different chemical composition solutions, each prepared at five different concentrations. Each participant receives five samples with a given nominal optical absorbance.

This communication presents the absorbance measurement data processing for the measurement comparisons. Spectrophotometers are operated in absorbance mode, with the measurements corrected for the absorbance of the solvent, if obtained against air. The spectrophotometers measurement parameters include a 1 s integration time, in scan mode, with a 1 nm step and a bandwidth between 1 nm to 2 nm. The determination of the measurement uncertainty components follows the corresponding well-established procedure of the literature [3], taking into account that the "chemical" uncertainty source is well controlled, due to the work done in the first step of the project [2]. As a matter of fact, rigorously estimating the measurement uncertainty is crucial not only to estimate a level of confidence associated with the measurement result, but also to check the consistency of the data. Indeed, the knowledge of the measurement uncertainty enables us to compute the unilateral degree of equivalence (DoE) and the bilateral DoE [4], and the DoEs associated uncertainty, for all the absorbance measured values at all the characteristic wavelengths, for the participants. Such procedure is planned to be used for the consolidation comparison for the participants now well prepared, complying with one of the main objectives of the project. Potential improvements to the comparison procedure are analyzed, in the light of the available communications [5].

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# P20

## From uncertainty to decision: quantifying false-result risk in sterility testing using likelihood ratios

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Sterility testing remains a cornerstone of pharmaceutical quality control, yet its interpretation is still largely based on a binary pass/fail paradigm that neglects the probabilistic nature of microbial detection. This limitation becomes critical under low contamination prevalence, where false-negative results pose significant risks to patient safety and false-positive outcomes may lead to unnecessary batch rejection. Despite extensive regulatory frameworks, a quantitative approach to uncertainty and decision risk in sterility testing is still lacking.
In this study, we introduce a probabilistic framework that shifts sterility testing from a deterministic classification to a risk-based decision model. This approach explicitly links experimental conditions to the likelihood of false decisions, providing a quantitative foundation for result interpretation.
The model demonstrated high discriminative performance (AUC ≈ 0.91), confirming its robustness across experimental conditions. More importantly, predictive outputs were reformulated into Likelihood Hazard Ratios (LHR), enabling direct quantification of evidential strength. When combining results from Tryptic Soy Broth and Thioglycolate media, the overall likelihood ratio (LHR*) reached values between 96.0 and 354.7, far exceeding the minimum decision threshold, thus providing strong confirmatory power for sterility assessment.
A key finding is that uncertainty is strongly time-dependent: incubation periods shorter than 10 days were associated with elevated false-negative risk, whereas extended incubation significantly improved decision reliability. These results provide quantitative evidence supporting, and refining, current pharmacopeial practices.
Unlike conventional approaches, the proposed framework enables sterility results to be interpreted as probabilities and strength-of-evidence metrics rather than binary outcomes. This represents a paradigm shift in qualitative microbiological analysis, aligning sterility testing with modern metrological principles and risk-based regulatory expectations.
The integration of probabilistic modelling and likelihood-based decision metrics provides a powerful tool for uncertainty evaluation in qualitative assays. This approach not only enhances the reliability of sterility testing but also establishes a generalizable framework for decision-making in analytical chemistry, with potential applications across regulated environments where false decisions carry critical consequences.

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# P21

# Impact of Source Variability on Measurement Uncertainty and Decision-Making in Apple Pomace Characterisation

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**Abstract**
Apple pomace is increasingly valorised as a secondary raw material in food, feed, and biorefinery applications due to its high carbohydrate content and continuous availability as an agro-industrial by-product. However, its heterogeneous origin ranging from small-scale household processing to industrial juice, cider, and dried feed production introduces substantial compositional variability. Factors such as apple cultivar, processing conditions, pressing efficiency, enzymatic treatment, and drying technology influence residual sugar levels and overall matrix composition. Although analytical measurement uncertainty is routinely estimated in laboratory practice, the contribution of source-related material variability to overall result dispersion is often overlooked. This study investigates the relative contributions of analytical uncertainty and production-source variability to the total variability observed in the determination of total sugars in apple pomace.

Total sugars were quantified spectrophotometrically using a validated calibration model operating within a defined analytical range and verified performance parameters. For each production source kitchen pomace, juice industry pomace, cider industry pomace, and dried feed pomace replicate analyses were conducted under repeatability conditions to isolate within-laboratory precision. Analytical uncertainty was estimated from precision data and calibration statistics and expressed as expanded uncertainty (U, k = 2) [1]. To distinguish analytical variability from intrinsic material heterogeneity, between-source and within-source variance components were evaluated using analysis of variance (ANOVA). This structured variance-component approach enabled quantitative comparison of compositional differences relative to the estimated analytical uncertainty.

The results demonstrate that variability attributable to production source substantially exceeds the calculated analytical uncertainty. The between-source variance component represents the dominant contribution to total variability, while repeatability-related uncertainty remains comparatively minor. This disparity has important implications for conformity assessment and industrial decision-making when apple pomace is treated as a uniform raw material [2]. Observed statistical differences between production streams may reflect inherent material heterogeneity rather than limitations of the analytical method.
The findings highlight the importance of clearly separating analytical measurement uncertainty from material variability in heterogeneous biomass matrices. A variance-component framework is proposed to support fitness-for-purpose evaluation and to reduce decision risk when comparing analytical results across diverse production sources.

**Keywords:** measurement uncertainty, source variability, apple pomace, total sugars, analytical quality, variance components, decision risk

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