

## Validation of sampling procedures

Breakout session 2.2  
at Eurachem Workshop

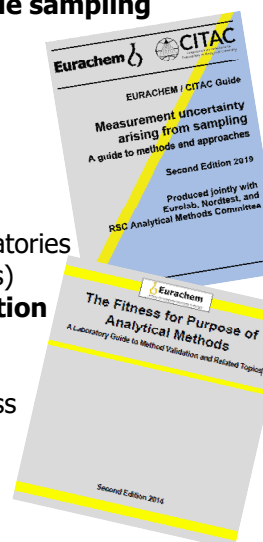
**Quality Assurance Challenges of Measurements  
from Field to Laboratory  
with a Focus on ISO/IEC 17025:2017 Requirements**

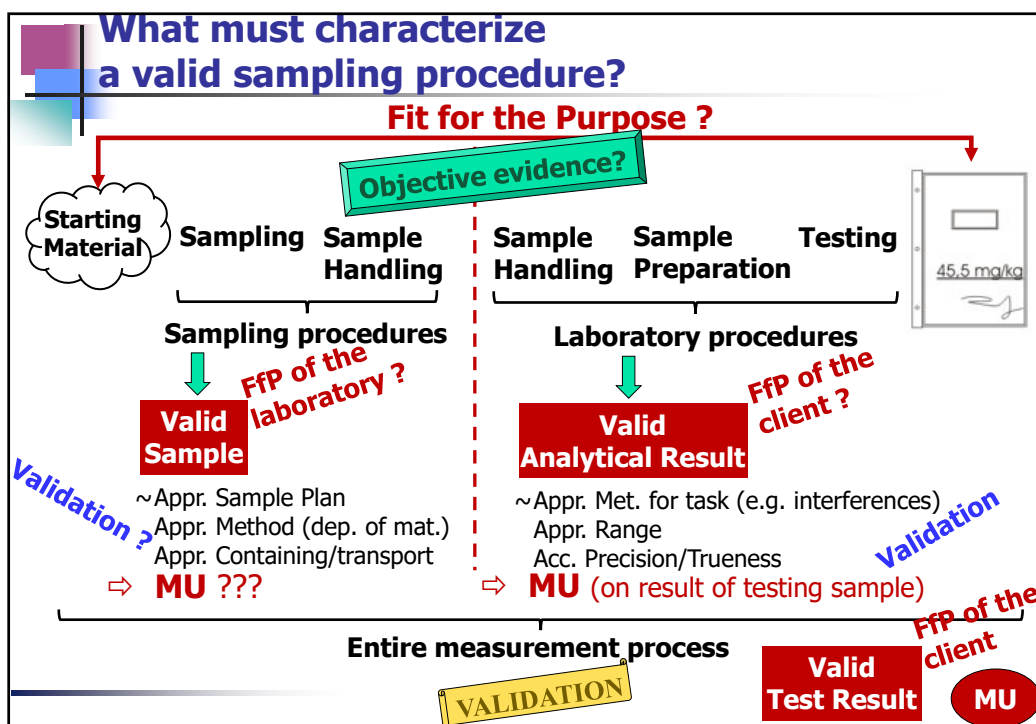
**16 - 18 May 2022 in Tbilisi, Georgia  
(Online event)**

Chaired by  
Mike Ramsey, UK (Chair of Eurachem UFSWG)  
& Lorens P. Sibbesen, Denmark (Chair of Eurachem MWWG)

### Introduction (by/ Lorens Sibbesen)

- ✓ Establishment of new Eurachem/EUROLAB Joint Task Group JTG
  - **Validation of measurement processes that include sampling**
  - Inviting members of EUROLAB to join
- ✓ Based on the activities in two Eurachem WGs:
  - Eurachem WG on Uncertainty from Sampling
  - Eurachem WG on Method validation
- ✓ The JTG has been established with the purpose of...
  - Establishing some **practical guidelines** for test laboratories and professional samplers (e.g., from inspection bodies) on **best possible practices for validation/verification of sampling procedures** (including sample handling) used either for **stand-alone** sampling activities or as an **integrated** part of a full measurement process (from sampling to issuing of final measurement result; including in situ measurements).

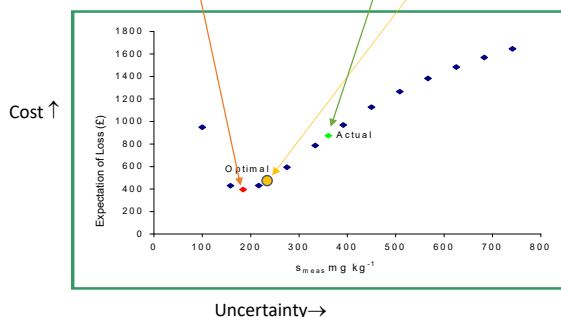




## Introduction (by/ Mike Ramsey)

### UFS estimation enables judgement on FFP for Validation, and identifies what to change to achieve FFP

- From previous talk: MU shown to be not FFP – method not validated (MU too high x2) , but..
- Shows that increasing number of lettuce heads (increments) from 10 to 40 lettuce heads...
- Reduces MU ( $s_{meas}$ ) by factor of 1.8 from 360 to 244  $mg\ kg^{-1}$  ( $U'$  from 16.4 % to 11.1%)
- Close to near optimal value much reduced Cost (~£500, down from £800 per target)
- Achieves Fitness-for-Purpose (FFP) = MU that minimises to overall financial loss



## Summary of Quantitative Approach to VaMPIS

### Validation of Measurement Procedures that Include Sampling

- To judge FFP, and hence validate the whole measurement process, need to:-
  - Including sampling within the measurement process
  - Use uncertainty of measurement values (MU - including UfS) as key metric to judge FFP
  - Identify what to improve to achieve FFP (e.g. sampling, sample prep, analysis?)
  - If analytical procedure already validated – can re-assess its FFP for particular application (e.g. when MU dominated by UfS)
- UfS (and hence MU) can be estimated with the Duplicate Method
  - At Validation take ~10% of field samples in duplicate
  - Use ANOVA (e.g. RANOVA3) to calculate MU and its components (e.g. UfS)
  - Also analyse reference materials (CRMs) to estimate analytical bias – add to MU
  - Ongoing QC to monitor MU using duplication of 10 % of routine samples
  - Applicable to any sampling medium: soil, sediment, herbage, waters, gases etc.
  - Also applicable to *in situ* measurements (no physical sample removed, e.g. PXRF)
- Collaborative Trial in Sampling results can be used to include between-sampler bias within UfS & MU estimate, and for Validation = *even better option*

## Breakout Group 2.2

✓ Number of participants: 43

### ✓ Occupation

- Laboratory: 7
  - engaged in sampling: 5
- Sampler: 5
- Other: 1 from AB
- ...
- ...

## DISCUSSIONS

### Subjects/Questions for discussion #1 & 2

1. Sampling of material for subsequent chemical analysis is a common activity carried out routinely in many different fields (food, environment, forensics etc.). Sampling and analysis are in principle two parts of the same measurement process, but can either be the responsibility of the same group of people (integrated process) OR of two groups of people (the sampler(s) and the laboratory; the "stand-alone" approach).  
**Q:** Which of the two possible approaches do you see as the most feasible one for ensuring reliable results of the measurement process?  
What are the pro's and con's ?
2. The sampling for subsequent chemical analysis can be done by various people involved in the measurement process from primary sampling to issuing of the final test result: The clients / Professional Samplers / Inspection Bodies / Laboratory staff  
**Q:** What should be the minimum level of training in sampling for people with that responsibility?  
Do you think all people involved in sampling have the full awareness of the validity of the sampling process - and its impact on the final test result?

## Subjects/Questions for discussion #3 & 4

3. Like for the analysis in the laboratory, the procedures followed should be valid – i.e. fit for the purpose.  
Validation in the laboratory is normally based on experiments, providing evidence that certain requirements for application of that particular method are fulfilled.  
**Q:** How can validation of sampling procedures be carried out?  
Which requirements – or criteria for validity – should be fulfilled?
4. Sampling may be done in accordance with a written procedure (e.g. a standard, a regulation, a documented tradition in the actual field of business).  
**Q:** Can you assume that, following a given “standard procedure” for sampling will ensure the validity of a sample for subsequent testing? I.e., ensuring the sample being fit for the purpose of analysing it in the lab., giving valid test results?  
Have those developing such “standard procedures” been validating them?

## NOTES FROM DISCUSSIONS

**Disclaimer:**

Please note, that the wording in the following notes are taken directly from discussions (incl. chat comments) and that the notes not necessarily express the opinion of Eurachem

## Discussion #1

### 1. Poll question

#### Question 1 - შეკითხვა 1

Poll | 1 question | 23 of 42 (54%) participated

1. Do you mostly see sampling as a process integrated with the final laboratory analysis (i.e., as a combined measurement process—from sampling to final reporting of test result)—or as a “stand-alone” process separated from the subsequent analysis in the lab? (Single Choice) \*

23/23 (100%) answered

Integrated/ინტეგრირებული	(16/23) 70%
Stand-alone/განცალკევებული-დამოუკიდებელი	(7/23) 30%

## Discussion #1

### 1. Sampling of material for subsequent chemical analysis is a common activity carried out routinely in many different fields (food, environment, forensics etc.). Sampling and analysis are in principle two parts of the same measurement process, but can either be the responsibility of the same group of people (integrated process) OR of two groups of people (the sampler(s) and the laboratory; the “stand-alone” approach).

**Q:** Which of the two possible approaches do you see as the most feasible one for ensuring reliable results of the measurement process?  
What are the pro’s and con’s ?

Comments from group

- Depends on type and role of a lab. (private, public, competent authority etc.)
- Usually the lab has no idea about how the sampling was done
- Integrated approach not the most common as of now
- Laboratories (accr.) only report result referring to sample as received
- Are sampling and analysis really two separate processes? What if the laboratory sample is such that sample preparation is actually sampling, even if the laboratory would never admit this

## Discussion #2

### 2 | Poll Question

#### Question 2- შეკითხვა 2

Poll | 1 question | 26 of 41 (63%) participated

1. Are people doing sampling always aware of whether the sampling procedure they are following is valid? / ნიმუშის აღება პირებმა ყოველთვის იციან თუ არა, რომ ნიმუშების აღების პროცედურა რომლითაც ისინი ხელმძღვანელობენ ვალიდურია? (Single Choice) \*

26/26 (100%) answered

YES/დიახ	(12/26) 46%
NO/არა	(14/26) 54%

## Discussion #2

### 2 | The sampling for subsequent chemical analysis can be done by various people involved in the measurement process from primary sampling to issuing of the final test result: The clients / Professional Samplers / Inspection Bodies / Laboratory staff

**Q:** What should be the minimum qualifications for people doing sampling? Do you think all people involved in sampling have the full awareness of the validity of the sampling process - and its impact on the final test result?

Comments from group

- No, this is not the case in practice
- The lab should inform the client about the sampling
- They should be certified or the sampling method accredited – but this is difficult to achieve in all fields
- Samplers are (often) not competent for each type of sample and method to be followed
- Necessary to have a special training for the operators. The sampling process should also reflect the appropriate regulation (e.g. for pesticides)

(cont.)

## Discussion #2 (cont'd)

- It is important to envisage the specifics of the laboratory and type of sample to be tested
- Developing the correct procedures for sampling depending on the specifics.
- Accredited Sampling bodies will employ samplers competent for each category of products/methods
- Samplers are often poorly paid, but this is false economy
- Unfortunately, very often sampling is performed by less qualified personnel
- The lab. has the responsibility to give instructions on how to sample, but it is difficult to know whether they were followed by the client.
- The integrated scheme is most probable for QA throughout the whole procedure; not many samplers are accredited.

## Discussion #3

### 3. Poll question

#### Question 3 - შეკითხვა 3

Poll | 1 question | 27 of 40 (67%) participated

1. Must the validity of a sampling procedure always be based on quantitative evidence? / უნდა იყოს თუ არა ნიმუშის აღების პროცედურის ვალიდურობა ყოველთვის დადგენიებული რაოდენობრივ მკვიდრობაზე? (Single Choice) \*

27/27 (100%) answered

YES/დიახ	(17/27) 63%
NO/არა	(10/27) 37%



## Discussion #3

4. Like for the analysis in the laboratory, the procedures followed should be valid – i.e. fit for the purpose.  
Validation in the laboratory is normally based on experiments, providing evidence that certain requirements for application of that particular method are fulfilled.

**Q:** How can validation of sampling procedures be carried out?  
Which requirements – or criteria for validity – should be fulfilled?


### Comments from group

- Working in laboratory, having nothing to do with the sampling.  
Need to reach out to the inspectors doing the sampling.  
Otherwise it can have serious consequences  
Also depending on the conditions under which the samples are taken  
Develop SOPs  
Conditions for having a valid sampling
- Contribution to MU from sampling is difficult to establish. A final target value would be useful, but based on experimental information (pilot study)
- Also validation based on repeated analysis (if feasible)

(cont.)

## Discussion #3 (cont'd)

- Problem with harmonization of approach among everybody working in the analytical branch
  - Also conducting validation based on the results of repeatable analyses
  - Dublicate/repetitive sampling is one of the problems in the case of medical examinatis (but then the quoted MU excludes sampling)
  - Adequate knowledge of the target sample. Furthermore adequate information on the specific conditions at the time of sampling
  - Sampling is agreement in a convention, in practice it is impossible to determine whether my sample is actually representative of the "total sample" to be examined.
  - Knowledge of the analytical method to be used (it will have an impact on the conservation and transportation of the sample)
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**Discussion #4**

(Unfortunately the time frame didn't allow for a discussion on this subject)

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