LAB QUALITY INTERNATIONAL

Eurachem
A focus for analytical chemistry in Europe

Is the sampling method as valid as the analytical method?

Presentation at

Eurachem Workshop

Trends and challenges in ensuring quality in analytical measurements

17 - 19 May 2021 in Prague, Czech Republic (Online event)

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”Trends and challenges…”

- Issues related to quality of samples and the validity of the sampling processes
  - Certainly a **challenge**
  - But not a **trend** – it has been there all the time
- Eurachem guide issued in 2007:

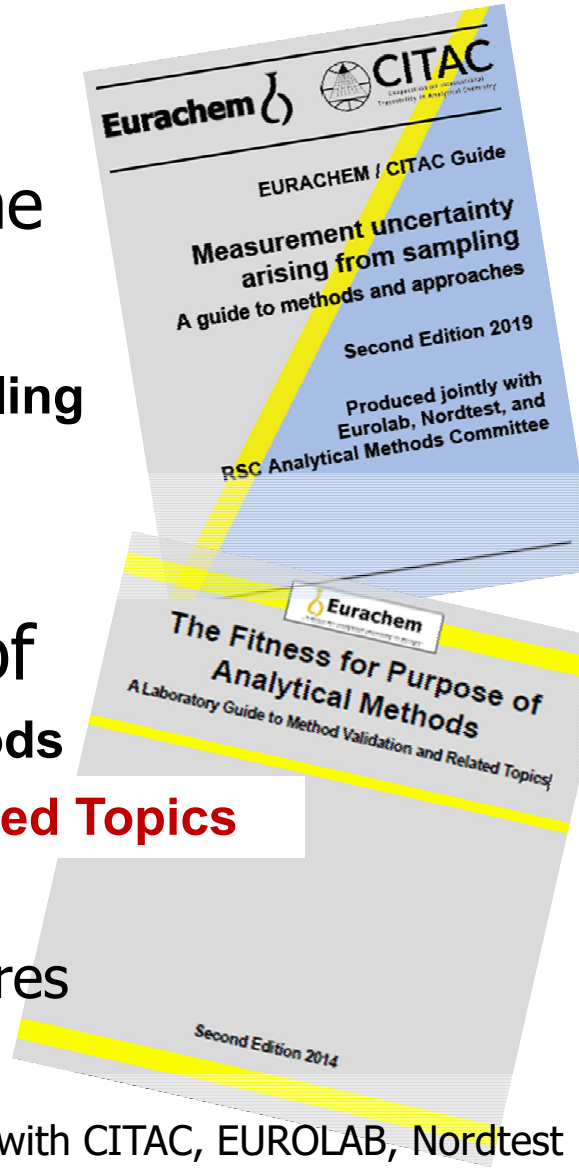
  Measurement uncertainty arising from sampling
  A guide to methods and approaches

  - 2nd edition issued in 2019 *)
- Working on a new sub-section to 3rd edition of

  The Fitness for Purpose of Analytical Methods

  A Laboratory Guide to Method Validation and Related Topics

  - 5.7 ”Sampling and sample handling in relation to MV”
  - Not (yet) a guideline on validation of sampling procedures

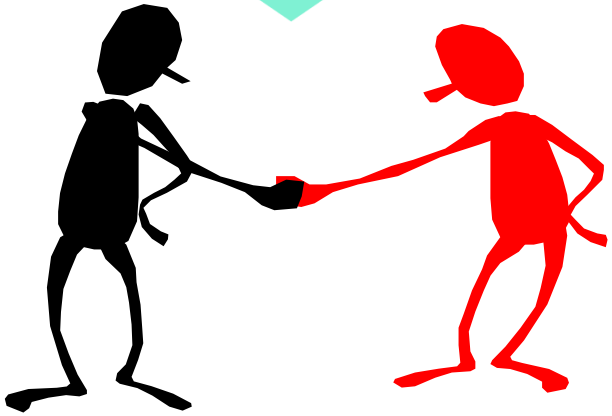


DISCLAIMER!
None of the statements in this presentation can be seen as a final Eurachem position on the subject

*) Produced jointly with CITAC, EUROLAB, Nordtest & RSC Analytical Methods Committee

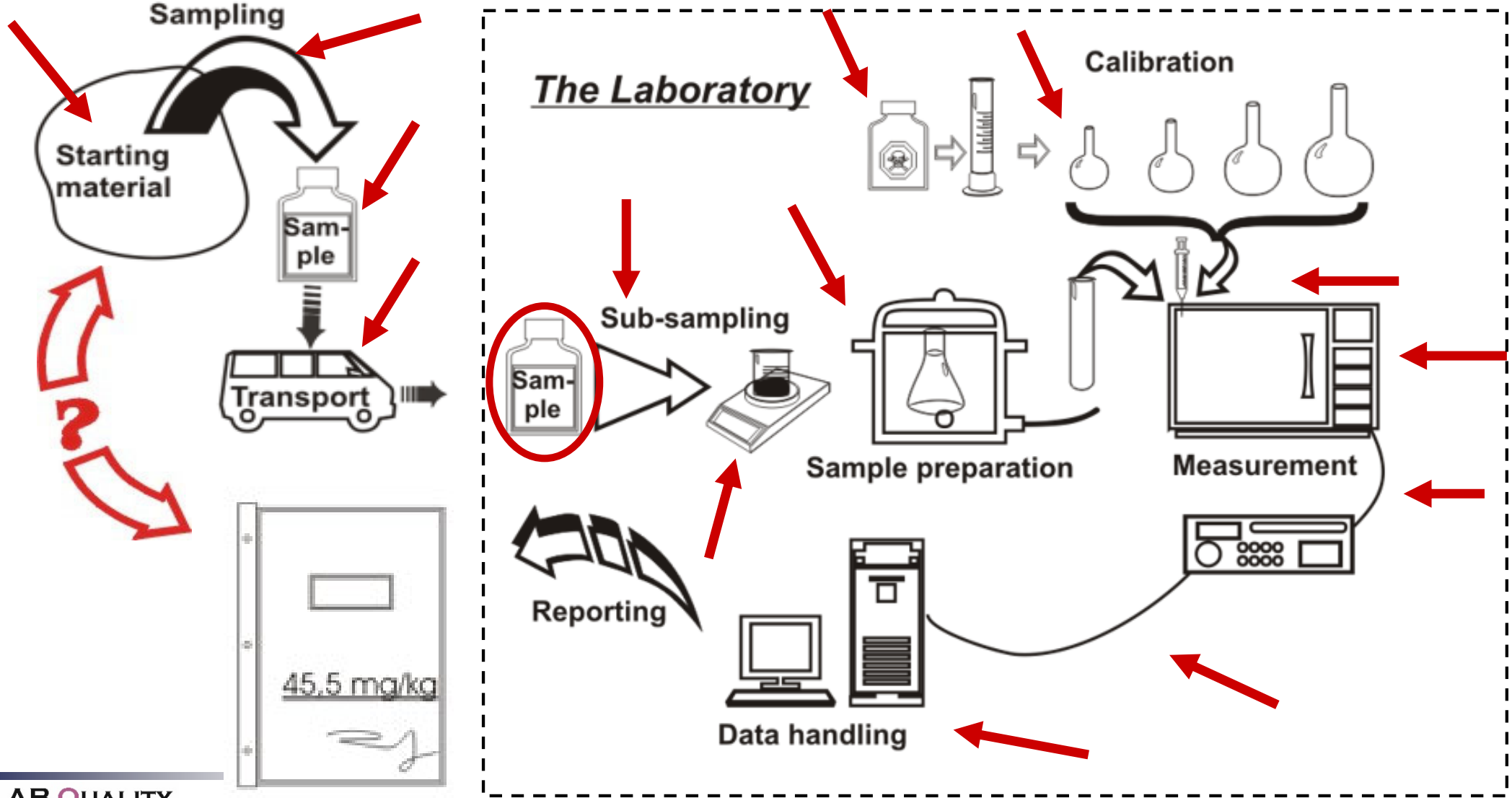
Method Validation

- ✓ ….. the process of proving that an analytical method is acceptable for its intended purpose
- ✓ …and gives results which are valid for decision making

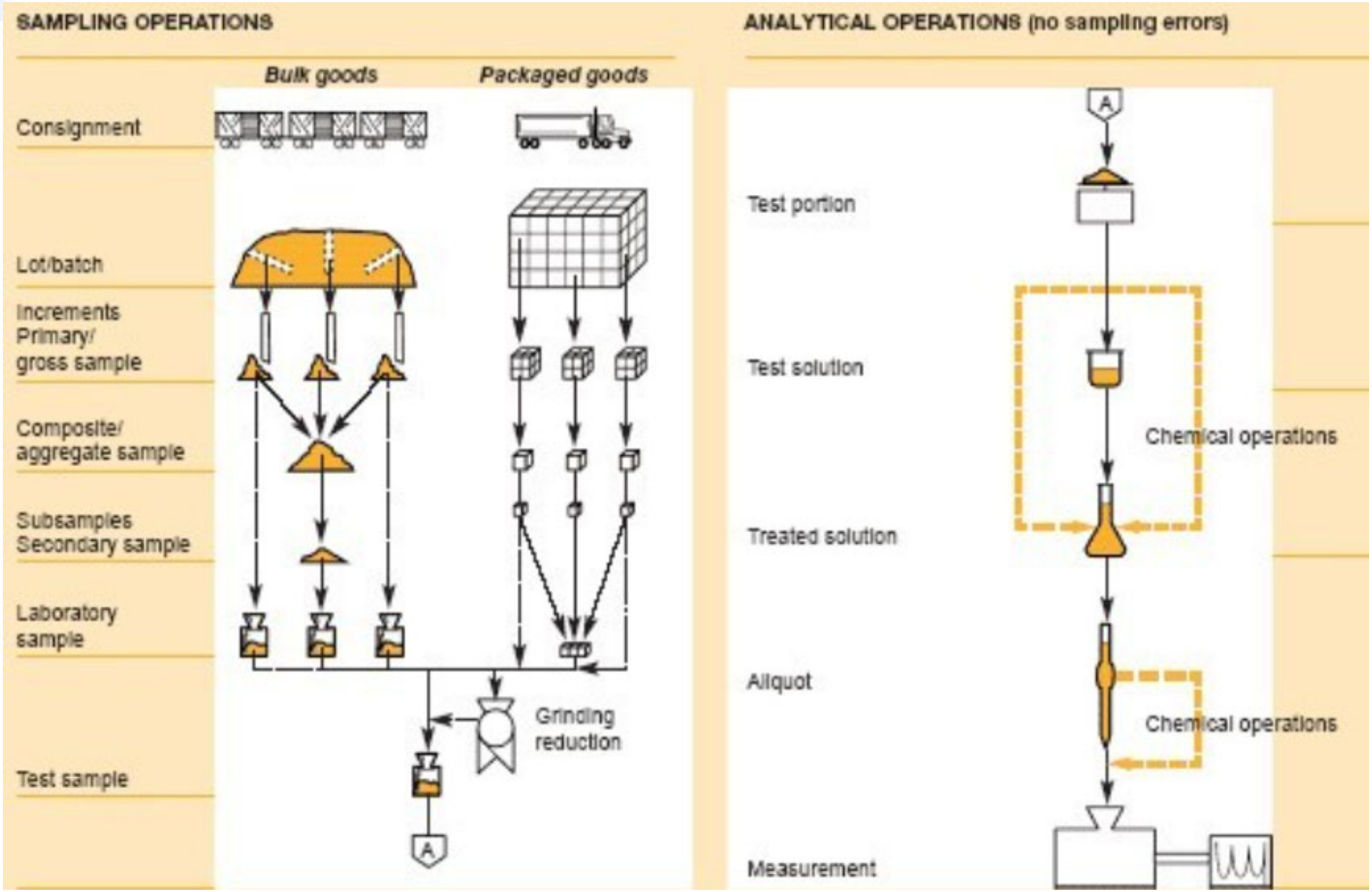


Method validation/verification in the lab.

- Focusing on the analytical process IN the lab.
  - ....and any critical points in that proces



Old issue !



Re.: IUPAC recommendation from Horwitz, 1990

Valid methods vs. Valid test results

- **Valid methods** are expected to give **valid results**
  - Ensured through validation and verification
- I.e. results being fit for the purpose of making reliable decisions
- Not only depending on the validity of the test method
- But also (in most cases) on the **validity of the sampling and sample handling** preceding the analytical work.
- **Uncertainty from sampling** is often (mostly) much bigger than the uncertainty contribution from the analytical method
  - Increased focus on uncertainty stemming from sampling in the revised version of the ISO/IEC 17025 standard
  - And: "Validation can include procedures for sampling, handling and transportation of test or calibration items". (Note in 7.2.2.1)

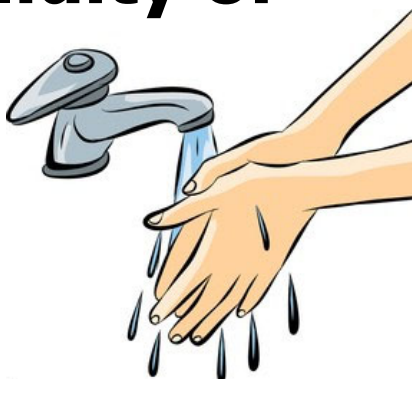
Valid sampling procedures

- NOT only related to the contribution to the final MU on the test result but also to the fitness for the purpose of ensuring representative samples
  - In relation to
    - possible inhomogeneity of sample origin
    - conditions at sampling spot – and at time for sampling
    - use of devices for the sampling
    - the performance of the sampling

- The sampling process must lead to **valid samples**
  - I.e. **fit for testing – giving valid results**
- The problem with the validity/quality of the sampling/samples is that it cannot be evaluated before after the testing of the samples has been done
  - and then valuable time/resouces may have been wasted.

Valid test results vs. Valid samples

- In ISO/IEC 17025:2017 it is required (in relation to reporting):
  - "..reference to the sampling plan and sampling method used by the laboratory or other bodies where these are relevant to the validity or application of the results".
  - Maybe a sampling plan/method stated by regulation?
    - or another "standard" method?
- BUT - was it a valid method?
  - and did it provide samples, which were valid for testing?
- **What can be done to ensure/improve the validity of the sampling procedures and the samples – and who is responsible for doing it?**



Three scenarios

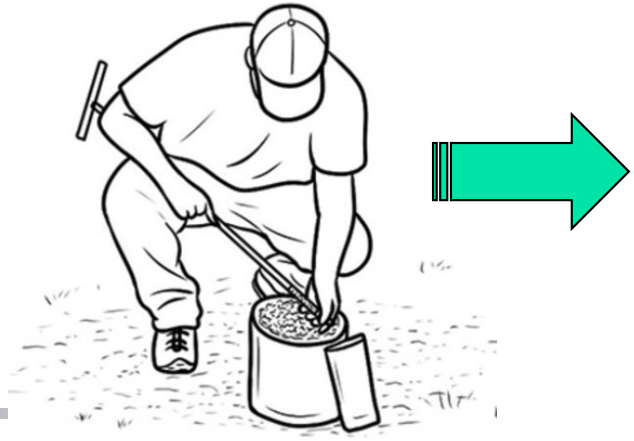
1. **Laboratory not responsible for the sampling process**
   - The primary sampling often done by the client (or by an independent body specializing in sampling)
   - Test report should include "…a statement to the effect that the results relate only to the items tested" (Re. ISO/IEC 17025)
2. **Laboratory responsible for sampling as part of method performance**
   - The primary sampling by the laboratory itself before testing the samples
3. **Field-testing**
   - The laboratory is (partly) responsible for bringing test equipment to the place of the original material from which samples are to be taken and the analytical method is carried out in the field - either by direct measurement (in situ) or at the spot (at site)

1. Laboratory not responsible for the sampling

- Direct validation of the sampling procedure not possible
  - Some **verification** of the validity of the samples being brought to the laboratory should be done (see later slide)
- However, the laboratory should **take the responsibility of carefully informing the sampler** (client or professional) about issues, which may influence the validity of the laboratory sample and its suitability for being tested (giving reliable results).
  - Communication with the client/sampler
    - highlighting any "critical points" in the sampling process (and sample transportation)
    - maybe "reaching out" by preparing sample containers etc.
    - recommend some kind of validation to be done & documented.

2. Laboratory responsible for sampling

- A proper **validation of the entire process** should be done.
  - Or a **verification**, if well-described (and validated) sampling procedures (protocols) are used.
- As the laboratory is handling both sampling and testing procedure, the validation/verification can conveniently be a **combined process**
  - Giving the possibility of **evaluating the outcome of the sampling process** and its impact on the final result of the testing the samples.



Field-testing

- For analytical method carried out outside the laboratory premises, a **thorough validation/verification** should be carried out.
  - Take into consideration all **special** (relevant) **conditions** in the sampling/testing situation, which may impact the validity of the method (and as such, of the final results)
- The validation/verification of such methods should preferably be done through **comparison** with in-house ('ex situ') performance of the method (or comparable methods) in the laboratory.
  - Especially for in-situ measurement devices it is important to verify the documentation provided by the producer
    (As such the validation/verification becomes more like an **instrument qualification**)

Sample receipt and handling

- The **validity of the laboratory sample** (i.e. whether it is fit for the testing and will give (sufficiently) representative results) **must be evaluated** by the laboratory upon receipt of the sample
  - Such evaluation must be based on the immediate appearance of the sample, any sample packaging and on how, the sample was brought to the laboratory (e.g. kept below a certain temp.).

- But whether the results of the testing are also valid for the analytes in the sampling target (i.e. the material that the sample is intended to represent), **depends on the validity of the sampling** (and sample transportation) procedure used

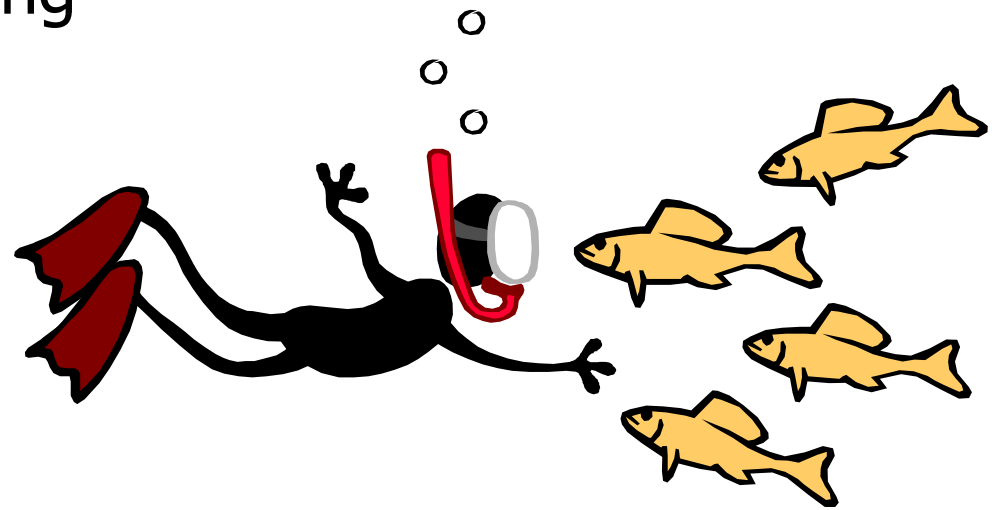
Some conclusion

- Validation of a sampling procedure can be seen as having estimation (and if necessary reduction) of the uncertainty from sampling as its primary purpose, and the sampling procedure can be seen as being valid, if the uncertainty stemming from sampling enables the final measurement to be fit for its intended purpose *).
- In-house handling and preparation of samples, should be validated/verified as part of the full method performance according to normal principles, but validation or verification of the primary (or field) sampling procedures (or protocols) is an issue for which guidance is still needed.
  - So far, the laboratory should just take responsibility for ensuring that a valid sampling method has been in use.

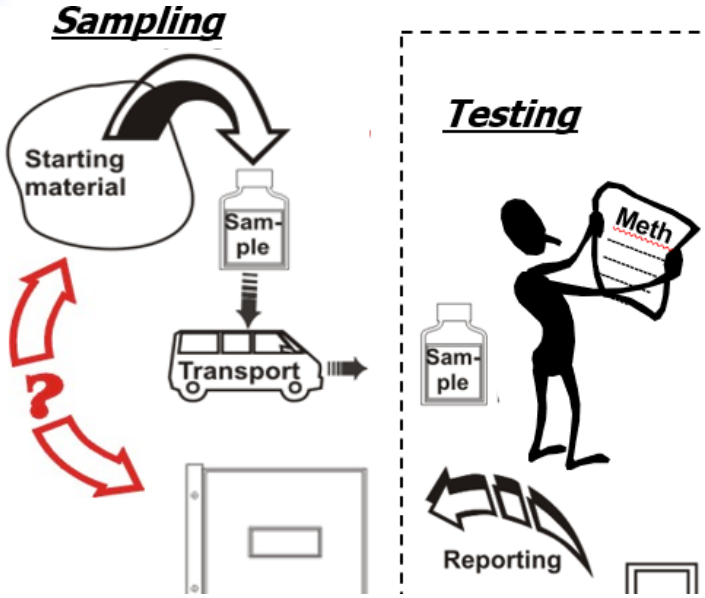
*) Eurachem Guide, "Measurement uncertainty arising from sampling. A guide to methods and approaches"

What can (will) we do?

- ✓ Study, follow and monitor the sampling processes
  - PT schemes including sampling
- ✓ Only apply regulated (validated?) procedures?
  - Should still be verified though
- ✓ Establish principles for validation/verification of sampling procedures
  - Eurachem WG on Method Validation starts cooperation with WG on Uncertainty from Sampling



Valid sampling and test method



- ✓ ….. ensuring that the entire testing process is acceptable for its intended purpose
- ✓ …and gives results which are valid for decision making