Eurachem
A Focus for Analytical Chemistry in Europe

From Customer Requests to Interpreting Results

How Eurachem Guidance Supports the Measurement Cycle

Vicki Barwick
Chair, Education and Training Working Group

1

Eurachem
A Focus for Analytical Chemistry in Europe

Overview

- The measurement cycle
- Types of guidance
- Understanding the measurement cycle
  - Eurachem guidance

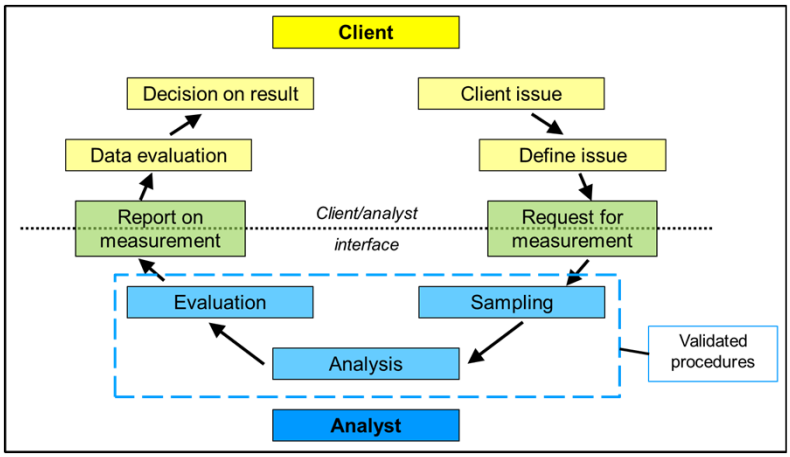
2

Eurachem
A Focus for Analytical Chemistry in Europe

The measurement cycle

Eurachem's focus is measurement quality...

...from client issue to decision on results



Eurachem
A Focus for Analytical Chemistry in Europe

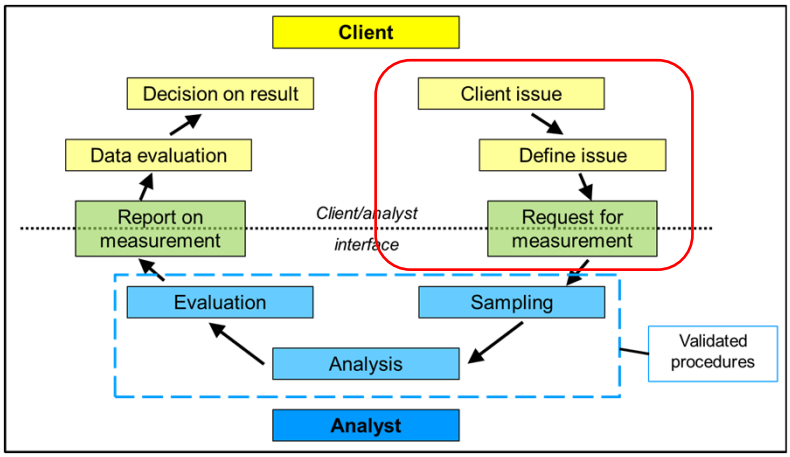
Guidance documents

Available at www.eurachem.org

- Eurachem guides
  - recommendations on good practice
- Supplementary guidance
  - recommendations on good practice illustrating/amplifying Guide topics
- Information leaflets
  - 2-page document, introducing a Eurachem Guide or providing information on a topic of interest
- Reading list
  - to provide references to a selection of websites, standards, guides and books

Eurachem
A Focus for Analytical Chemistry in Europe

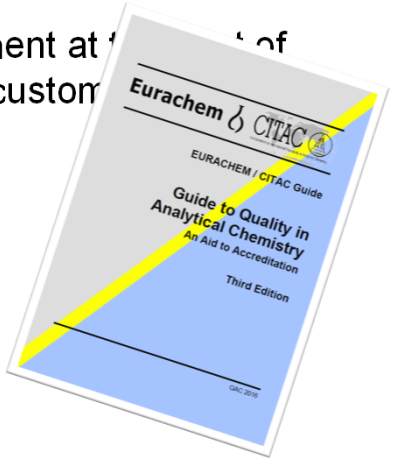
Understanding the client's issue



Eurachem
A Focus for Analytical Chemistry in Europe

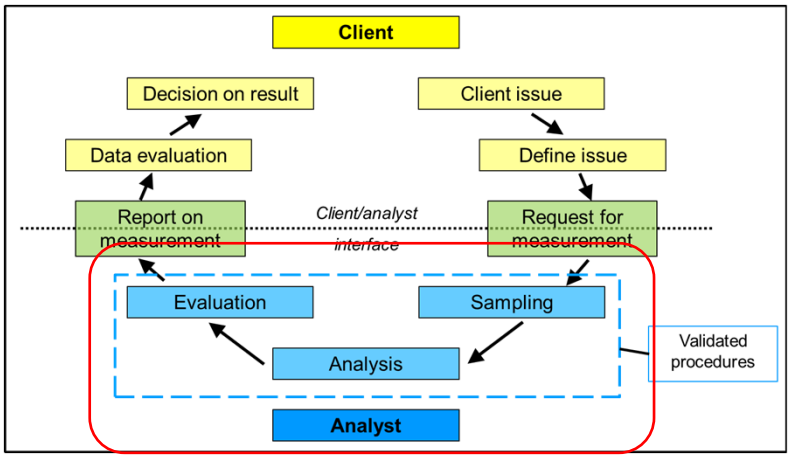
Understanding the client's issue

- Understanding the client/customer's requirement at t… of the measurement cycle is critical to meeting custom…
  - what is being measured and why?
    - what decisions will be made on the basis of the results?
  - what are the samples?
  - target measurement uncertainty?
  - cost and time constraints?
  - reporting requirements?
    - opinion/interpretation required



Eurachem
A Focus for Analytical Chemistry in Europe

Making measurements

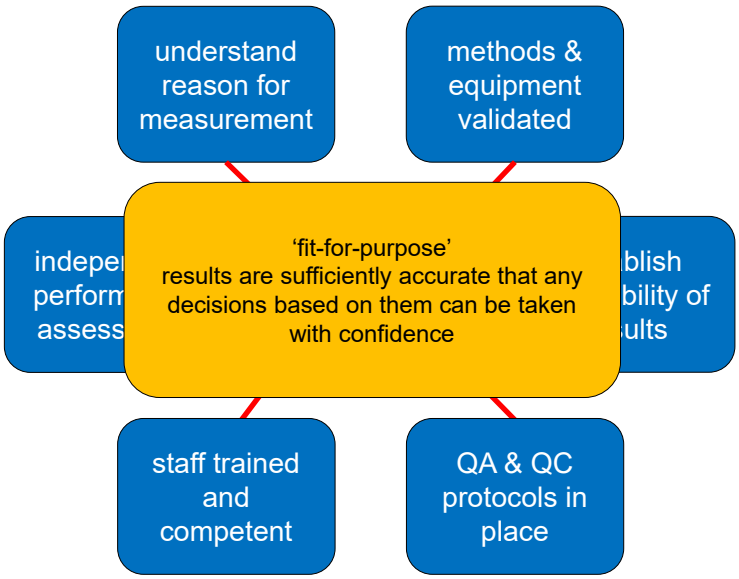


7

7

Eurachem
A Focus for Analytical Chemistry in Europe

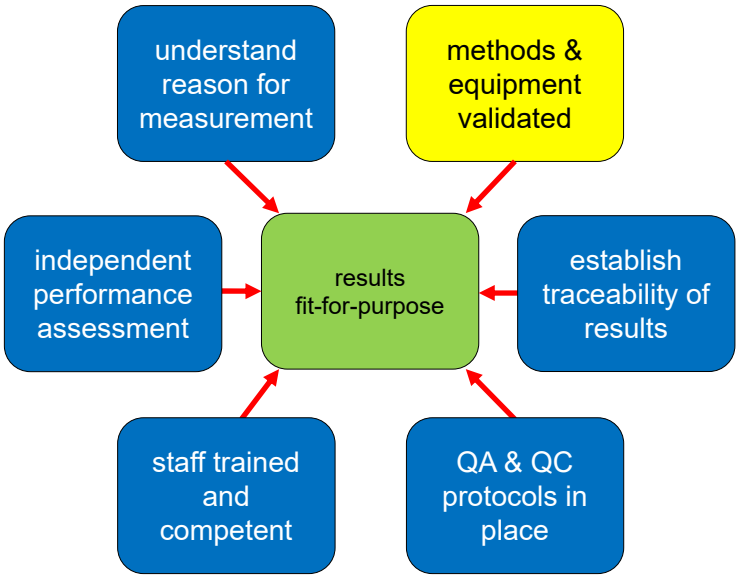
Principles of valid analytical measurement



8

Eurachem
A Focus for Analytical Chemistry in Europe

Principles of valid analytical measurement



9

Eurachem
A Focus for Analytical Chemistry in Europe

Concept of method validation

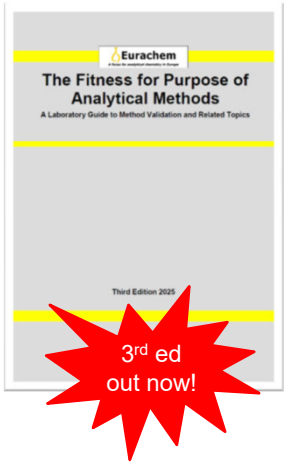
- Establish required performance of the method
  - what do I need to measure?
  - what are the sample types?
  - what performance is acceptable (precision, bias, limit of detection…)
    - may be specified in legislation
- Determine actual performance of the method
  - experimental studies
- Confirm whether the actual performance matches the required performance

10

Eurachem
A Focus for Analytical Chemistry in Europe

The fitness for purpose of analytical methods

- Definition of validation
  - validation vs verification
- Importance of method validation
- Extent of validation
- Planning and reporting validation studies
- Method performance characteristics
  - selectivity, limits of detection and quantification, working range, trueness, precision, ruggedness
- Documentation of validated methods



11

Eurachem
A Focus for Analytical Chemistry in Europe

Supplementary guidance

- To be effective, validation should be a planned activity
  - a template for planning validation
    - which performance characteristics? materials and experiments? data analysis? assessment of fitness-for purpose?
- Blanks – an important tool in the determination of many performance characteristics
  - types and uses of blanks in method validation
    - calibration blank, procedural blank, reagent blank, solvent blank, sample blank
    - approaches if no suitable blank available

12

Eurachem
A Focus for Analytical Chemistry in Europe

Validation of sampling procedures

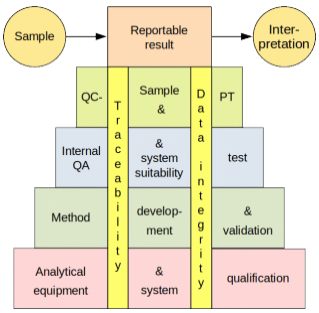
- Measurement process starts when primary sample is taken from sampling target
- Validation process should also begin at this point
- New guidance describes an extension to the established validation methodologies
  - gives an integrated approach for the overall measurement process (including sampling steps)

13

Eurachem
A Focus for Analytical Chemistry in Europe

The fitness for intended use of analytical equipment and systems

- The Fitness for Purpose of Analytical Equipment
  - guidance under development
- Lifecycle of laboratory equipment
  - Design Qualification
  - Installation Qualification
  - Operation Qualification
  - Performance Qualification



14

Eurachem
A Focus for Analytical Chemistry in Europe

Evaluating measurement uncertainty

Step 1
Be clear about what is being measured

Write down equation used to calculate result.

Step 2
Identify the sources of uncertainty

Parameters appearing in the equation will contribute to the uncertainty. What other factors will influence the result?

Step 3
Quantify uncertainty components

Estimate the size of each uncertainty component (the effect it will have on the result). Convert all estimates to the same form (standard uncertainty, u).

Step 4
Combine the uncertainties

Combine using rules for combination of variances.

$$u_c = \sqrt{u_1^2 + u_2^2 + u_3^2 + ...}$$

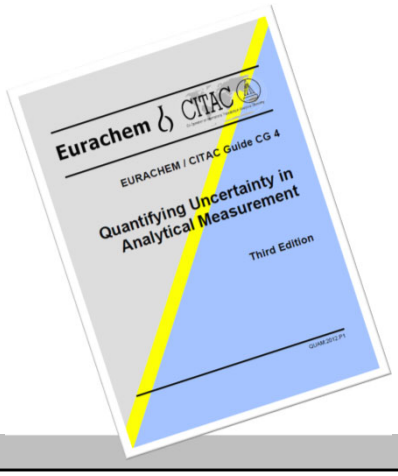
Step 5
Expand combined uncertainty

Multiply the combined uncertainty by a coverage factor to obtain an expanded uncertainty. $U = k.u_c$

15

Eurachem
A Focus for Analytical Chemistry in Europe

Quantifying uncertainty in analytical measurement

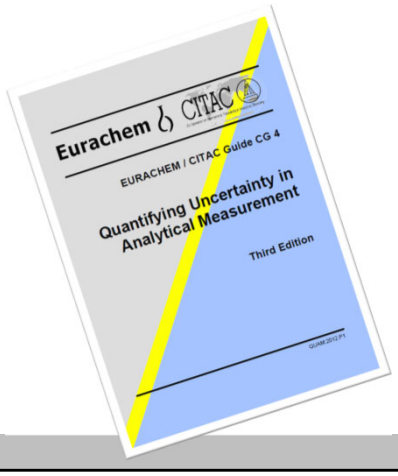


- Definition of uncertainty
- Sources of uncertainty
- Link with traceability and validation
- Detailed procedure for evaluating uncertainty
- Worked examples

16

Eurachem
A Focus for Analytical Chemistry in Europe

Quantifying uncertainty in analytical measurement

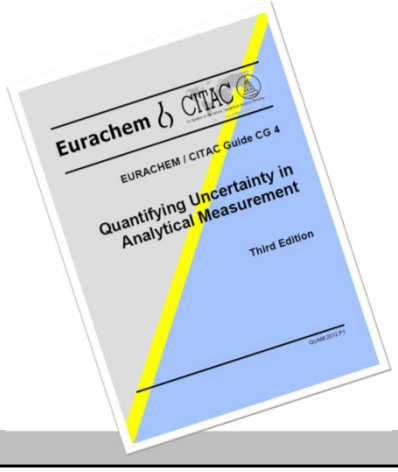


- Definition of uncertainty
- Sources of uncertainty
- Link with traceability and validation
- Detailed procedure for evaluating uncertainty
- Worked examples

17

Eurachem
A Focus for Analytical Chemistry in Europe

Quantifying uncertainty in analytical measurement



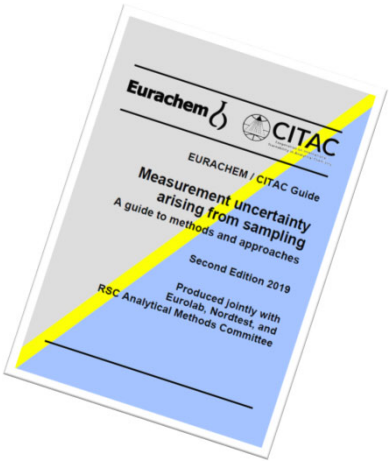
- Definition of uncertainty
- Sources of uncertainty
- Link with traceability and validation
- Detailed procedure for evaluating uncertainty
- Worked examples
- Guide under development
  - evaluation of measurement uncertainty from in-house precision and trueness tests

18

Eurachem
A Focus for Analytical Chemistry in Europe

Measurement uncertainty arising from sampling

- Sampling in the measurement process
- Conducting studies for evaluating sampling uncertainties
- Statistical approaches
- Quality control of sampling
- Reporting and interpreting uncertainty

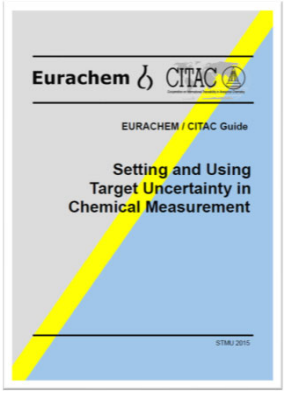


19

Eurachem
A Focus for Analytical Chemistry in Europe

Assessing fitness-for-purpose – target uncertainty

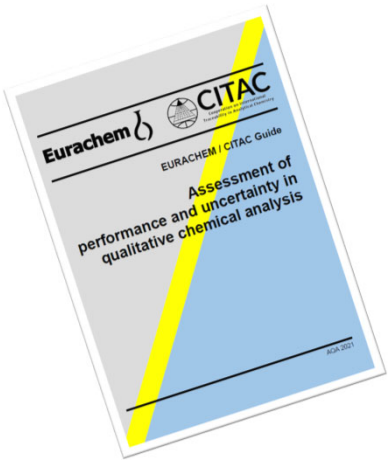
- All measurements made for a reason
  - compliance with a specification or regulatory limit; medical diagnosis, treatment and monitoring; environmental monitoring; supporting a prosecution (forensics)…
- To assess fitness-for-purpose of a measurement procedure
  - specify performance characteristics such as precision and bias
  - set a target value for the measurement uncertainty



20

Eurachem
A Focus for Analytical Chemistry in Europe

Specific guidance for qualitative analysis

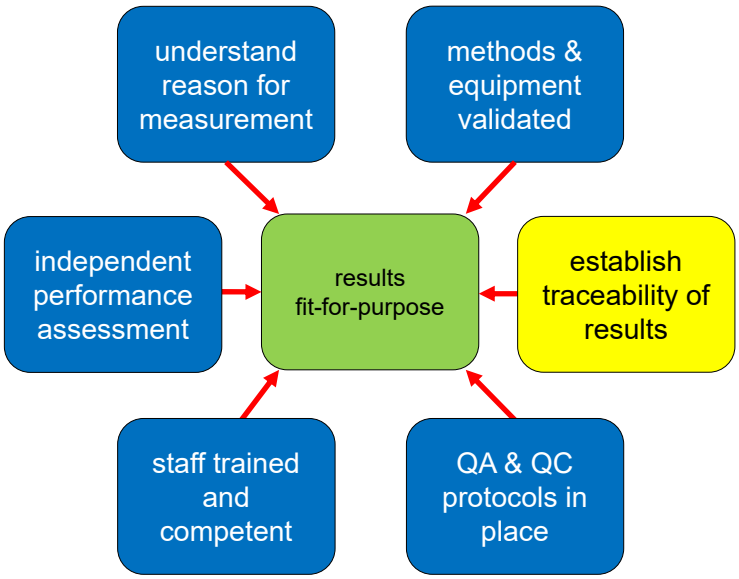


- Evaluation of uncertainty in qualitative analysis has received much less focus than quantitative analysis
- Laboratories need to be aware of reliability of qualitative results
- Guide covers
  - performance assessment for qualitative analysis
  - expressions of confidence in qualitative analysis
  - reporting the qualitative analytical result
  - worked examples

21

Eurachem
A Focus for Analytical Chemistry in Europe

Principles of valid analytical measurement



22

Eurachem
A Focus for Analytical Chemistry in Europe

Concept of traceability

- *"Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty."*
- Results must be traceable to recognised references
  - definition of a measurement unit (SI)
  - measurement procedure
  - measurement standard (reference material)
- Traceability to internationally agreed references allows results to be compared
  - different locations, different times

23

Eurachem
A Focus for Analytical Chemistry in Europe

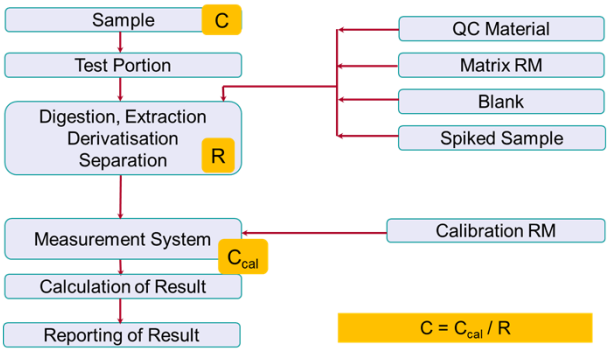
Metrological traceability in chemical measurement

- Principles of metrological traceability
- International definition of traceability
- A practical approach to establishing traceability
- Choosing appropriate references
- Worked examples

24

Eurachem
A Focus for Analytical Chemistry in Europe

Selection and use of reference materials

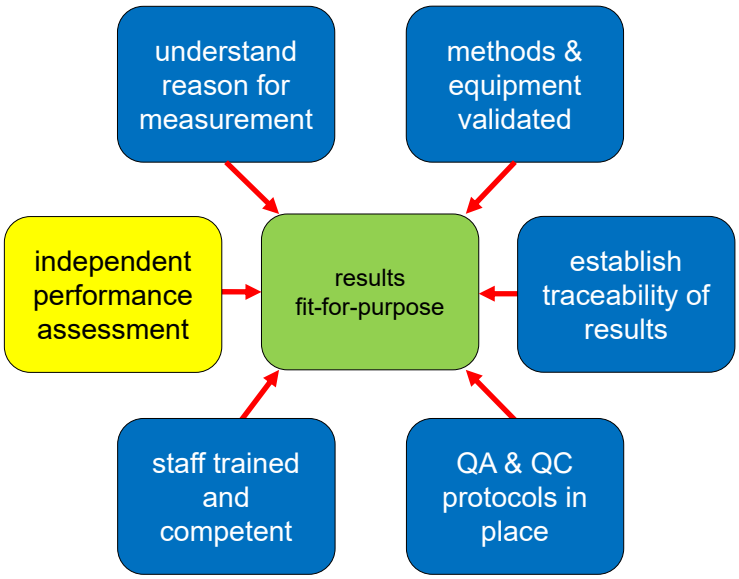


- Reference materials play a crucial rule at many stages in the measurement process
- Guidance on selection and use of RMs under revision

25

Eurachem
A Focus for Analytical Chemistry in Europe

Principles of valid analytical measurement

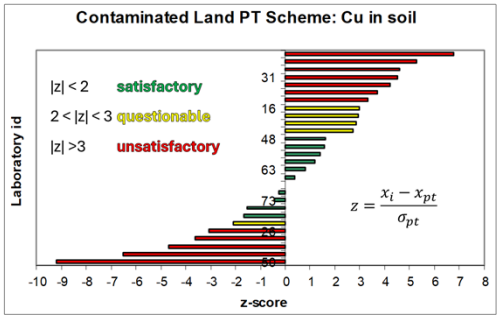


26

Eurachem
A Focus for Analytical Chemistry in Europe

Independent assessment of performance

- Regular independent assessment of the technical performance of a laboratory
  - necessary to assure the validity of results
  - should be part of an overall quality strategy
- Common approach
  - participate in proficiency testing/external quality assessment schemes

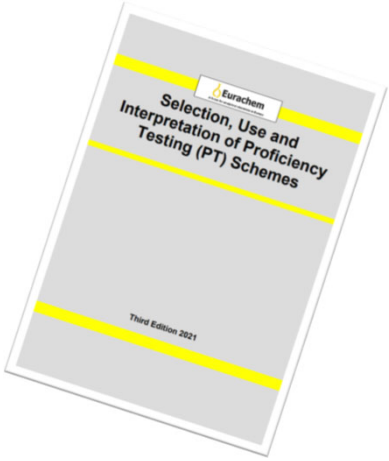


27

Eurachem
A Focus for Analytical Chemistry in Europe

Guidance on selection, use and interpretation of PT schemes

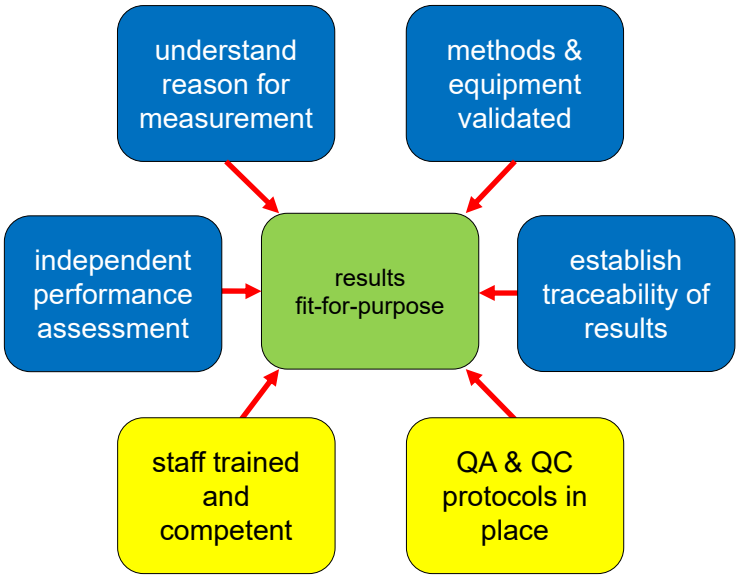
- Introduction to PT
- Selection of appropriate PT schemes
- Use of PT in laboratories
- Evaluation of laboratory performance
- Laboratory interpretation of PT results
- Investigation of unsatisfactory or questionable PT results



28

Eurachem
A Focus for Analytical Chemistry in Europe

Principles of valid analytical measurement



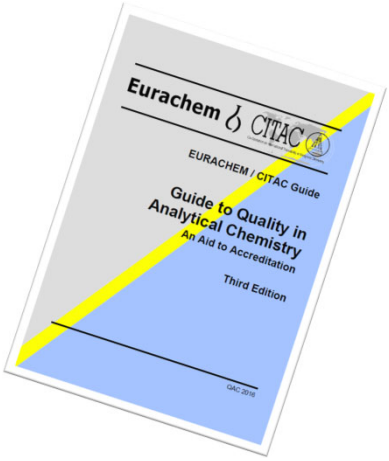
29

Eurachem
A Focus for Analytical Chemistry in Europe

Guidance on quality assurance and accreditation

Guide to quality in analytical chemistry

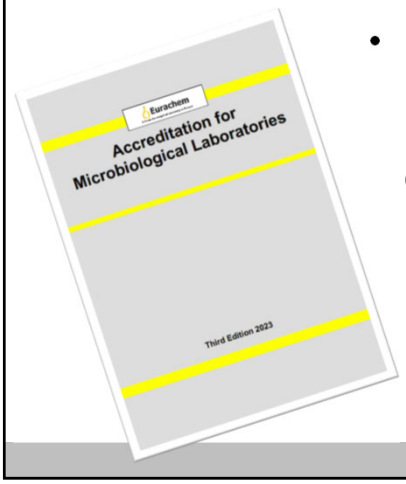
- Provides laboratories with guidance on best practice for analytical operations
- Helps laboratories implement a quality management system
  - in particular to meet requirements of ISO/IEC 17025
- Focuses on technical aspects of quality management



30

Eurachem
A Focus for Analytical Chemistry in Europe

Specific guidance for microbiological laboratories and R&D/non-routine analysis

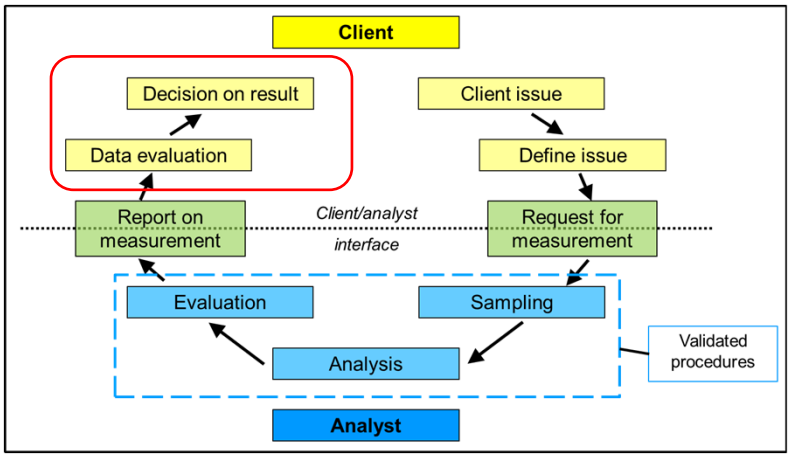


- Provides laboratories carrying out microbiological testing with appropriate information on how to fulfil the requirements of ISO/IEC 17025
  - Guidance on quality assurance for research and development and non-routine analysis under review

31

Eurachem
A Focus for Analytical Chemistry in Europe

Making decisions



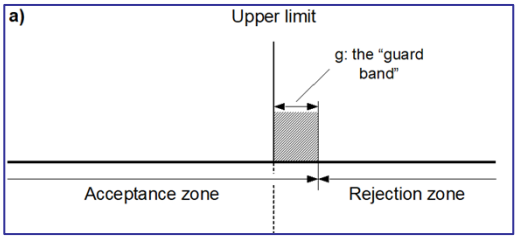
32

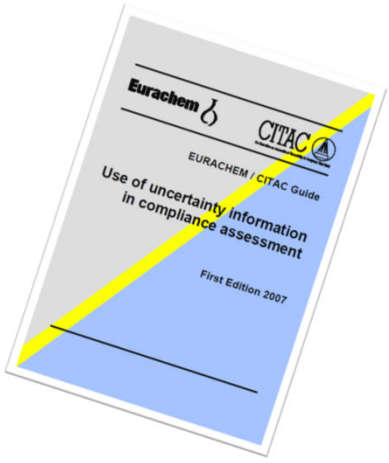
32

Eurachem
A Focus for Analytical Chemistry in Europe

Use of uncertainty information in compliance assessment

- Decision rules and acceptance zones
- Choosing acceptance and rejection zone limits
- Specifying an acceptable uncertainty



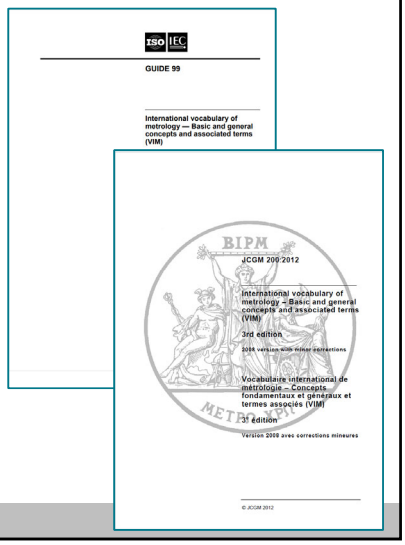


33

Eurachem
A Focus for Analytical Chemistry in Europe

Importance of a common language of measurement

- Basic principles of measurement are the same across disciplines
  - increase in interdisciplinary activities
- Clear definitions
  - improve communication
  - reduce disputes/misunderstanding
  - allow consistent interpretation
    - e.g. between regulatory/assessment bodies and laboratories
  - facilitate translations



34

Eurachem
A Focus for Analytical Chemistry in Europe

Guide to terminology in analytical measurement

- Applicable to several sectors
  - chemical analysis
  - biological measurements
  - clinical chemistry
- Important concepts identified
- Terms requiring special attention identified
- Link to terms used in ISO standards and Guides
  - current terminology in the workplace

35

Eurachem
A Focus for Analytical Chemistry in Europe

Accessing the guides

- All available free of charge from the Eurachem website
  - https://www.eurachem.org/index.php/publications/guides
- Translations available

Scan to access current guides

36