The new draft Eurachem/CITAC Guide: a summary of structure and changes

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Uncertainty evolution

Pre-1978 Random/systematic error; Error propagation in chemistry (Eckschlager 1961); Collaborative study

1980 • BIPM INC-1 (1980)
– Type A / Type B
– Combine as variances

1982

1986

1993 • **ISO Guide**

1995 • EURACHEM Guide 1st ed

2000 • EURACHEM Guide 2nd ed (QUAM:2000

2010 • GUM Supplement 1 (MCS)

- AOAC Stats manual *(Development/validation)*
- ISO 5725:1986 *(Collab trial)*
- ISO 5725:1994 *(Adds trueness)*
- *ISO 21748 – Uncertainty from collab study data*

2011 • Draft 3rd Edition EURACHEM/CITAC guide

Principles of the Revision

- Remember translators
  - Do not revise ‘cosmetically’ – keep existing text wherever possible
- Refer to existing guidance where possible
  - Do not re-state other guides
- Add new sections rather than intersperse modifications
- Adopt any known corrections

A note on Terminology

- VIM 3 is published
  - Why not update to VIM 3 terminology throughout?
- QUAM:2011 Implements the GUM
  - needs to use GUM terms
- It is for Chemists
- VIM 3 definitions are often quite different in wording and need explanation
- There’s a detailed Eurachem guide to VIM 3
- Decision:
  - Use GUM terminology with discussion of VIM terms where different

QUAM:2011 - Contents

- Foreword to the third edition
- 1. Scope and field of application
- 2. Uncertainty
- 3. Analytical measurement and uncertainty
- 4. The process of measurement uncertainty estimation
- 5. – 8. Detailed guidance on steps in measurement uncertainty estimation
- 9. Reporting uncertainty

Appendices

A. Examples
B. Definitions
C. Uncertainties in analytical processes
D. Analysing uncertainty sources
E. Useful statistical procedures
F. Measurement uncertainty at the limit of detection/limit of determination
G. Common sources and values of uncertainty
H. Bibliography

Scope

- Evaluation of ... uncertainty ... for a single method implemented as a defined measurement procedure in a single laboratory.
- [Use of] Information from method development and validation.
- [Use of] Results from defined internal quality control procedures in a single laboratory.
- [Use of] Results from collaborative studies
- [Use of] Results from proficiency test schemes used to assess the analytical competency of laboratories.

Ch 2: Uncertainty

- Definition and discussion of the concept of measurement uncertainty
- Comparison of ‘error’ and ‘uncertainty’
- The VIM 3 definition of uncertainty

Ch. 3. Analytical Measurement and Uncertainty

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- Relationship of uncertainty and validation
- Conduct of experimental studies
- Traceability

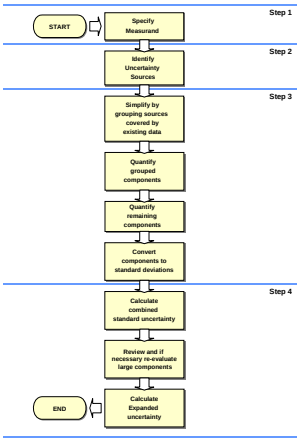
- Traceability section reduced and referring to Eurachem/CITAC Traceability guide

Ch. 4. The Process of Measurement Uncertainty Estimation

Setting standards in analytical science

- Outline of the process
  - Specify measurand
  - Identify Sources
  - Group and quantify
  - Combine

- Unchanged in 2011



Ch 5: Specification of the Measurand

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- Improved statement of ‘specification’
  - a statement of
    a. the particular kind of quantity to be measured, usually the concentration or mass fraction of an analyte.
    b. the item or material to be analysed and, if necessary, additional information on the location within the test item.
    c. where necessary, the basis for calculation of the quantity reporting results.
- Taken from the Eurachem Guide to the VIM

Ch 6: Identifying Uncertainty Sources

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- A list of likely sources of uncertainty
- Unchanged in 2011

Ch 7: Quantifying Uncertainty

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- Introduction and procedure
- Evaluating uncertainty by quantification of individual components
- Closely matched certified reference materials
- Uncertainty estimation using prior collaborative method development and validation study data
- Uncertainty estimation using in-house development and validation studies
- Data from proficiency testing
- Empirical and ad-hoc methods

7.11ff: Quantification of individual components

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- Experimental estimation of individual uncertainty contributions
- Estimation based on other results or data
- Modelling from theoretical principles
- Estimation based on judgement
- Significance of bias
  - updated to reflect recent research

CH 8: Combining uncertainties

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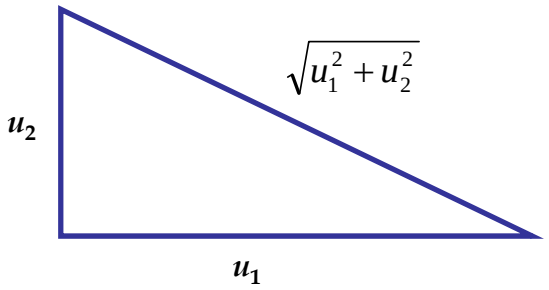
- The basic GUM theory



- Adds cross-reference to simulation methods

Ch 9: Reporting uncertainties

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- Expanded uncertainty
- Interpretation against limits
  - Now references Eurachem Guide on “Use of uncertainty information in compliance assessment”

Appendix A: Examples

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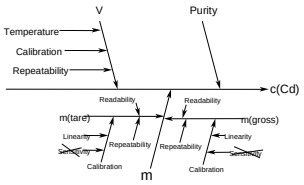
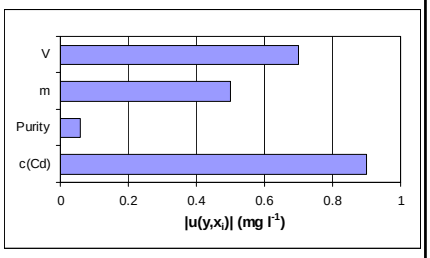
- Detailed worked Examples
- Summary provided for each
- All illustrate spreadsheet methods

- Some examples amended in revision





Appendices B-D

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A. Definitions
   - Reduced to simplify and refer to other guides

B. Uncertainties in analytical processes
   - List of uncertainties

C. Analysing uncertainty sources
   - Cause-and-effect analysis
   - Minor change to clarify

Appendix E: Useful Statistical Procedures

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- Distribution functions
- Spreadsheet method for uncertainty calculation
- Evaluation of uncertainty using Monte Carlo simulation *New section*
- Uncertainties from linear least squares calibration
- Documenting uncertainty dependent on analyte level

Appendix F. Measurement uncertainty at the limit of detection/limit of determination

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- Introduction
  - Observations and estimates
  - Validity of observations below zero
  - Reporting 'less than'/'greater than'
- Measurement uncertainty near zero
  - Expanded uncertainty intervals near zero: Classical approach
  - Expanded uncertainty intervals near zero: Bayesian approach *Substantial revision*

Uncertainty near limits

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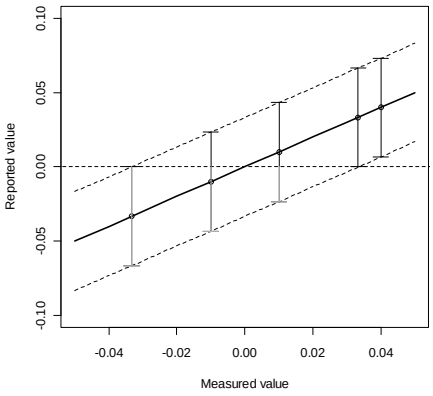
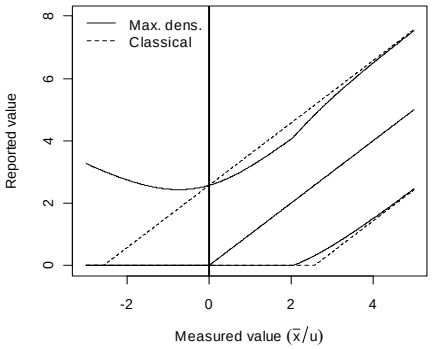
Classical



Bayesian



Summary

Setting standards in analytical science

- A broad guide to measurement uncertainty estimation in analytical chemistry
- Covers basic GUM but also covers validation approaches thoroughly
- New sections on
  - Assessing compliance with limits
  - Uncertainty near detection limits
  - Monte Carlo methods

Appendices G, H

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- G: Common sources and values of uncertainty
  - Reference list to assist initial estimation
- H: Bibliography
  - Updated and with additional references