ISO/TS 21748: A new guide to uncertainty estimation using interlaboratory study data

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Overview

- Reproducibility and uncertainty estimation
- Introduction to ISO TS 21748

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What is Measurement Uncertainty?

"A parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand"

(ISO Guide)

The number after the ±

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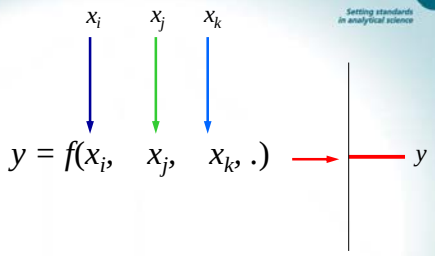
Models and Uncertainty



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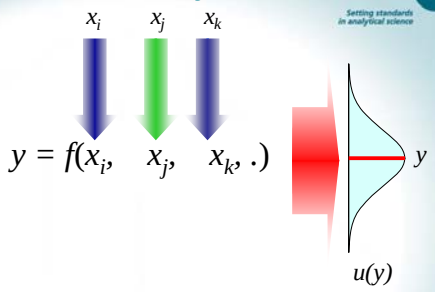
Models and Uncertainty



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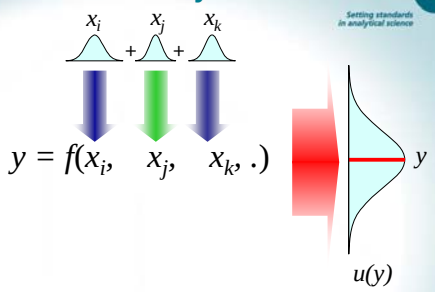
Models and Uncertainty



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ISO Guide approach

- Specify the measurand
  - including complete equation
- Quantify significant uncertainties in all parameters
  - A: from statistics of repeated experiment
  - B: by any other means (theory, certificates, judgement...)
- Express as standard deviation
- Combine according to stated principles

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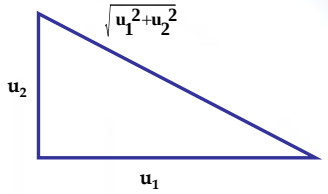
Combining uncertainties (ISO)



- *Standard deviations*
- *Established error propagation theory*

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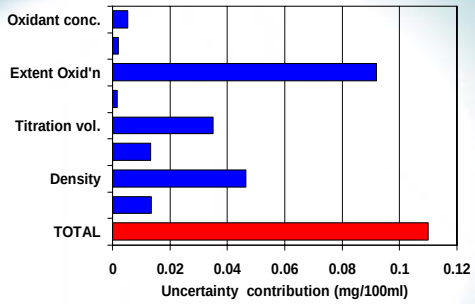
Example: Forensic alcohol standard titration



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Implementing ISO in Chemistry
Building models

- Every determinand is unique
  - Every element, every molecule, every formulation
- Every 'matrix' is unique
  - Different interactions with substrate
- Interactions with environment and substrate rarely understood
- **Models are difficult to build!**

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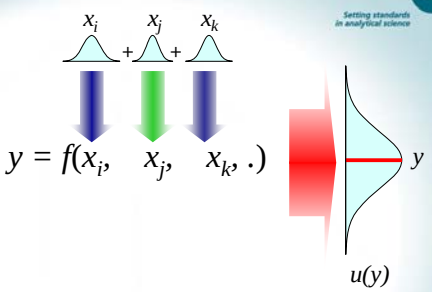
Two views of influence factors



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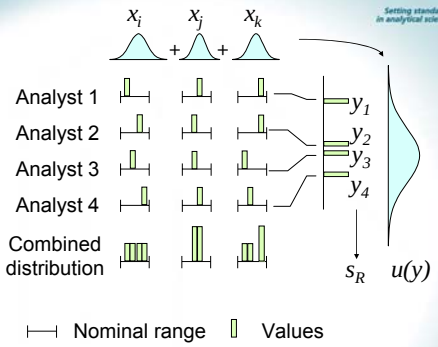
Two views of influence factors



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A simpler model

- The best available estimate of precision
  - *An effect varied representatively during a precision experiment requires no further study*
- The best available estimate of bias *and its uncertainty*
- Other significant effects evaluated
  - By experiment, or from standing data

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Collaboratively trialled methods

- Collaboratively trialled method
  - bias and precision verified in-house
- Assessed against collaborative study estimates
  - ISO/TS 21748
  - demonstrate method bias and repeatability are under control

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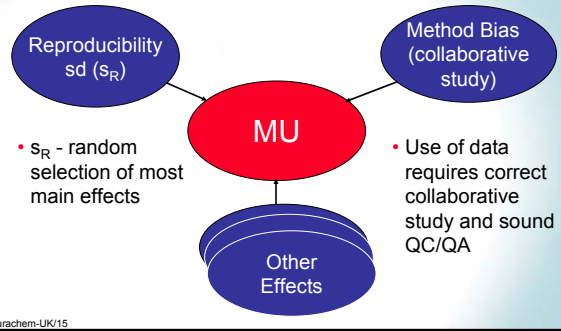
Collaborative trial data



- s_R - random selection of most main effects
- Use of data requires correct collaborative study and sound QC/QA

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Process for evaluating uncertainty according to ISO/TS 21748

- Obtain repeatability, reproducibility and bias estimates from collaborative study
- Establish whether laboratory bias is within that expected on the basis of the collaborative study
- Establish whether laboratory precision is within that expected on the basis of the collaborative study
- Where laboratory bias and precision are under control, combine effects appropriately to form a combined uncertainty estimate

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TS21748 uncertainty budget

$$u_c^2(y) = \hat{s}_R^2 + u^2(\hat{\delta}) + \sum_{i=k,n} c_i^2 u^2(x_i)$$

- Combined uncertainty
- Adjusted reproducibility estimate
- Uncertainty associated with method bias
- Effects not covered, e.g. sampling uncertainty

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Demonstrating acceptable laboratory bias

- Study of CRMs
  - Simple test for significant bias
  - Ideally with small bias check uncertainty
- Comparison with known test method
  - Typically paired tests on several materials
- Comparison with other laboratories
  - Proficiency testing or other collaborative study

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Including trueness data in an uncertainty estimate

$$u_c^2(y) = \hat{s}_R^2 + u^2(\hat{\delta}) + \sum_{i=k,n} c_i^2 u^2(x_i)$$

- Combined uncertainty
- Adjusted reproducibility estimate
- **Uncertainty associated with method bias**
- Effects not covered, e.g. sampling uncertainty

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Demonstrating acceptable laboratory precision

- Requires a check on repeatability standard deviation
  - F-test at appropriate level.
- If within-laboratory precision differs from Study s_r
  - Replace study s_r with in-house repeatability SD
- Otherwise, continue using published s_R

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Adjusting for changes in test item

- Allow for inhomogeneity and sample prep via additional uncertainty contributions
- Allow for changes in precision with analyte level
  - Linear model most common
  - Often sufficient to use %RSD

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Including reproducibility data

$$u_c^2(y) = \hat{s}_R^2 + u^2(\hat{\delta}) + \sum_{i=k,n} c_i^2 u^2(x_i)$$

- Combined uncertainty
- **Adjusted reproducibility estimate**
- Uncertainty associated with method bias
- Effects not covered, e.g. sampling uncertainty

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Other effects

- Simple “worst case” calculations can often show an effect is negligible
- Formal uncertainty calculations often show negligible components
- Typical criteria:
  - $u < u_{max}/3$ (<6% effect on combined uncertainty)
  - $u < u_{max}/5$ (<2% effect on combined uncertainty)
  - ISO/TS 21748 effects $< 0.2 S_R$

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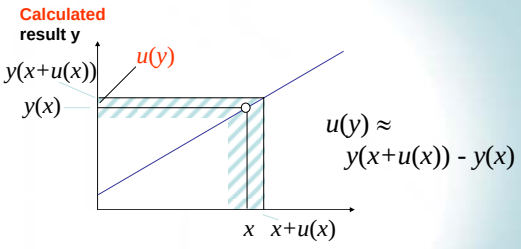
Numerical calculation



$$u(y) \approx y(x+u(x)) - y(x)$$

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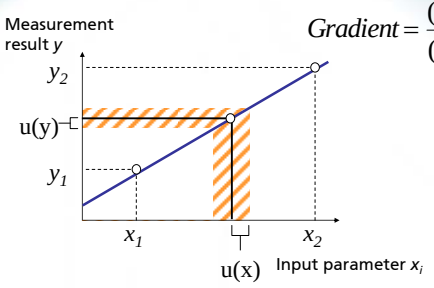
Other effects: A simple uncertainty experiment



$$Gradient = \frac{(y_2 - y_1)}{(x_2 - x_1)}$$

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"Other effects"

$$u_c^2(y) = \hat{s}_R^2 + u^2(\hat{\delta}) + \sum_{i=k,n} c_i^2 u^2(x_i)$$

- Combined uncertainty
- Adjusted reproducibility estimate
- Uncertainty associated with method bias
- **Effects not covered, e.g. sampling uncertainty**

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Conclusions

- ISO TS 21748 provides a recipe for using collaborative study data
- Requires checks for consistency with study performance
- Allows for changes in the test item type
- Often reduces to a simple reproducibility SD

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