16th Nordic User meeting for AAS, ICP-AES and ICP-MS

Day 2 – Sample prep., quality control and more….

Analytical method

REQUIREMENT

METHOD TO LAB

INTERNAL QUALITY CONTROL (QC)

INSTRUCTION (SOP)

VALIDATION

PRELIMINARY UNCERTAINTY

ACCREDITATION

METHOD IN USE

PROFICIENCY TESTING (PT)

REVIEW

UNCERTAINTY (U)

ANNUAL QC REVIEW

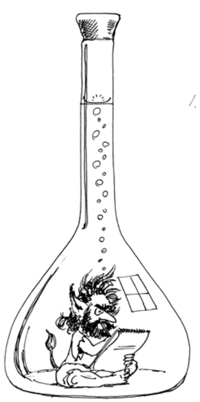
Standard solution & Gas

Digestion
SYNLAB & Sandviken&**SYKE**
Anton Paar & CEM & Milestone

QC – the Trollbook

Blanks & LOD

Measurement uncertainty, *U*
U from QC and validation data
U for a new matrix/analyte



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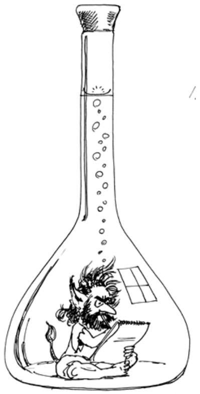
Measurement Uncertainty from validation and QC data

NORDTEST NT TR 537 edition 4 2017:11

The new revised Nordtest handbook



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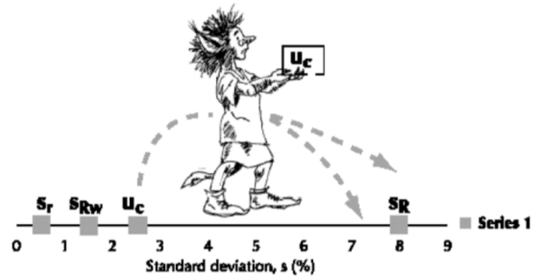
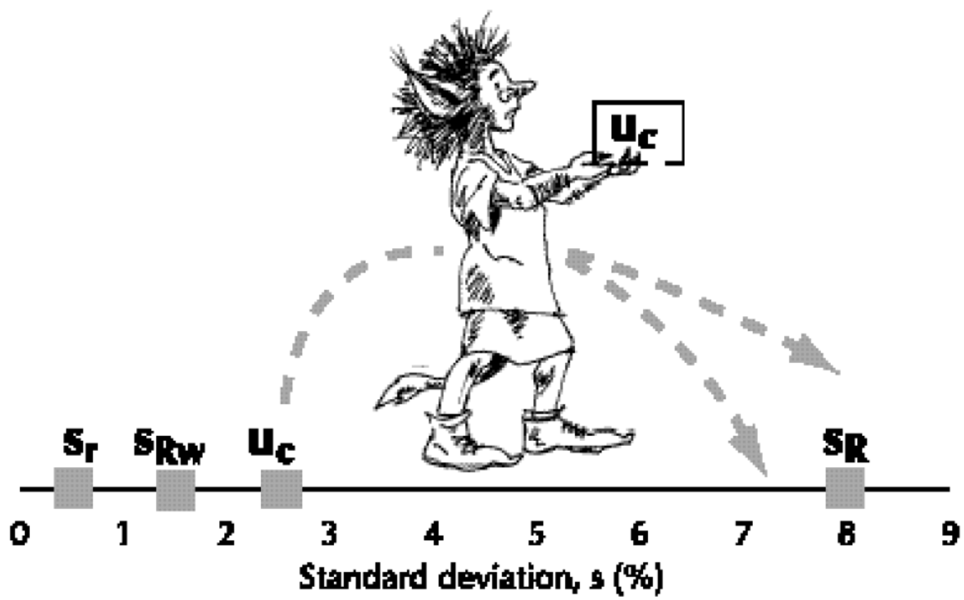
Handbook
for
Calculation of
Measurement Uncertainty
in
Environmental Laboratories



Nordtest TR 537 **Handbook for**
Calculation of Measurement Uncertainty in Environmental Laboratories

Based on experience gained by many laboratories using this approach to estimate measurement uncertainty a new edition 4 is published 2017



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Nordtest TR 537 – major updates 2017

1. Uncertainty over the measurement range
2. Standard deviation from duplicates – new method
3. Control chart limits can give within-lab reproducibility
4. Harmonisation with ISO 11352 *Water quality — Estimation of measurement*
5. Recommended
   - software
   - on-line course

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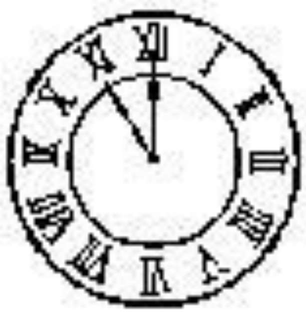
What do we get with measurement quality in place

comparability: property of measurement results enabling them to be compared because they are metrologically traceable to the same stated metrological reference; independent of:



Time

Place

Laboratory/operator/procedure

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Uncertainty approaches*

GUM principles

Definition of the measurand
List of uncertainty components

Intralaboratory

Interlaboratory

based on …

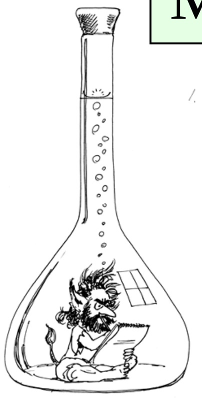
Modelling

Single laboratory validation

Interlaboratory validation

Proficiency testing

Experimental approaches



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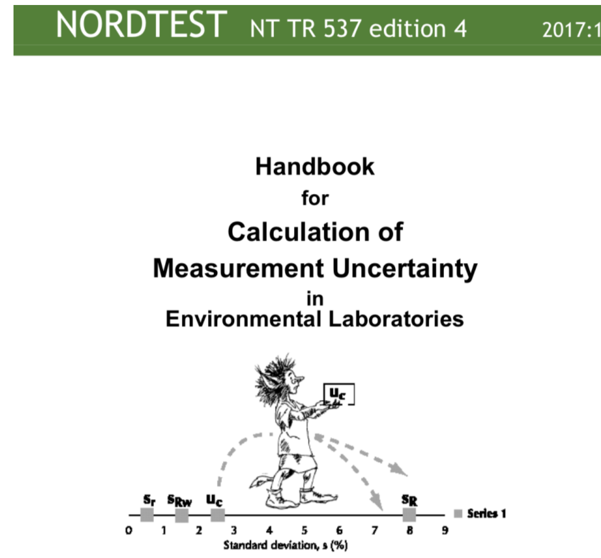
*Uncertainty revisited, Euroolab report 2007, www.eurolab.org

History of uncertainty – some documents

Year	Document
1993	GUM
1995	1st Eurachem
2000	1st ISO/IEC 17025
2003	Eur. Acc EA 04/16
2003	1st Nordtest 537
2007	Eurolab – uncertainty revisited
2012	ISO 11352 Water quality – Uncertainty from validation and QC
2017	4th Nordtest 537

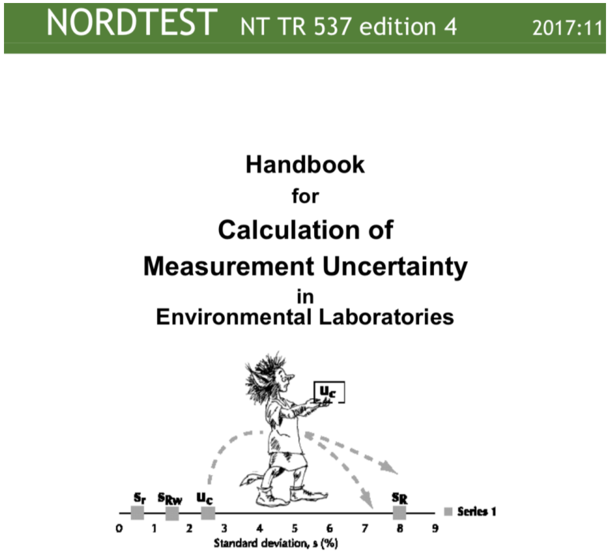


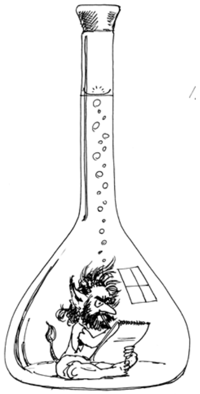
Now the QC and validation approach most used in analysis/testing

Standard uncertainty, *u*

The standard uncertainty can be obtained from

- Measurement $\boxed{\rightarrow}$ $s = u$
- Other sources $\boxed{\rightarrow}$ u





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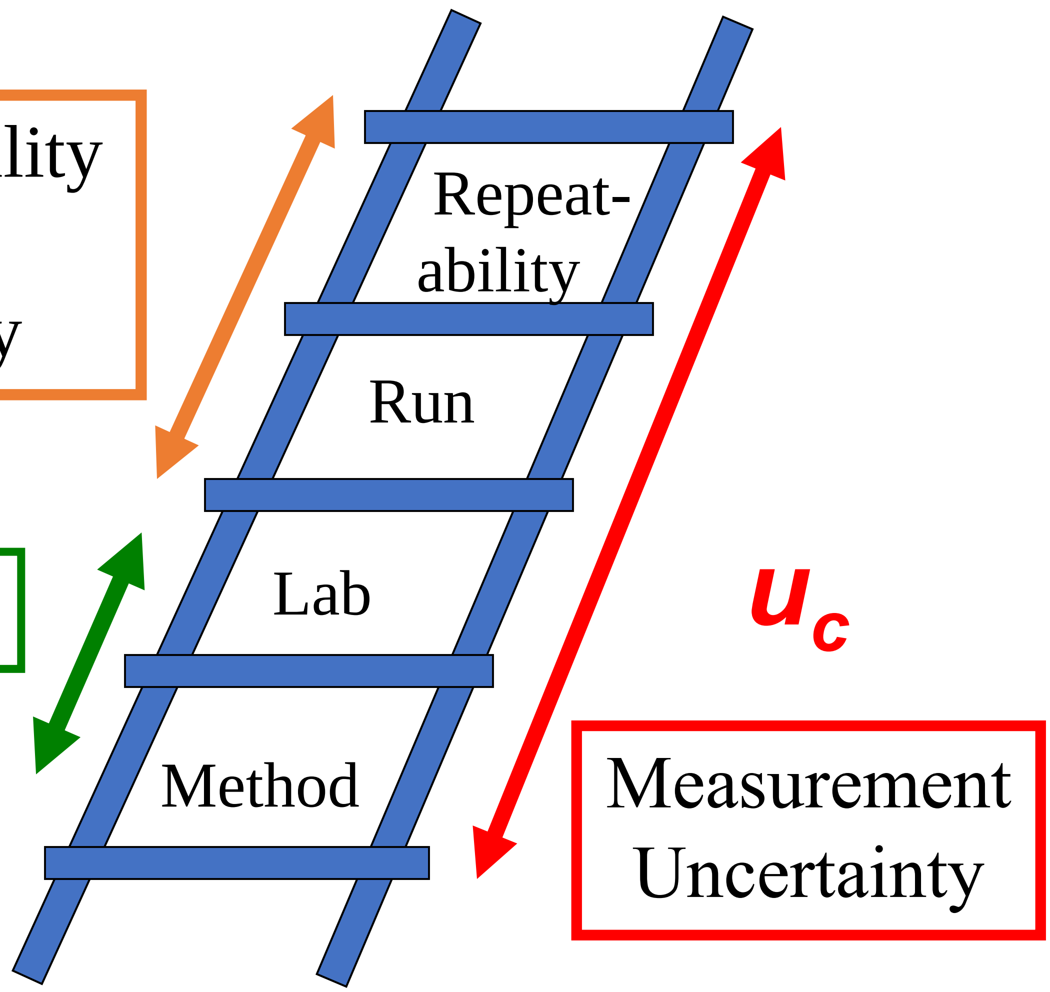
Ladder of Errors

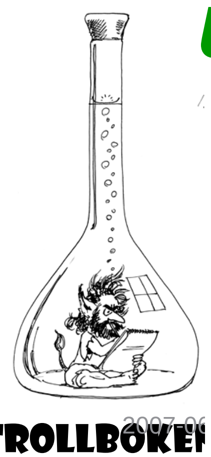
u_{Rw}

Reproducibility within laboratory

$u(bias)$

Bias





Uncertainty from validation and QC

NORDTEST NT TR 537 edition 4 2017:11

Handbook
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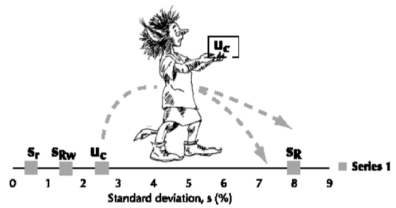
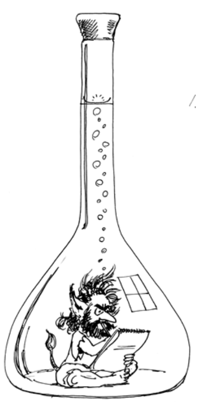


$$u_c = \sqrt{u(Rw)^2 + u(bias)^2}$$

$$Expanded\ U = 2 \cdot u_c$$



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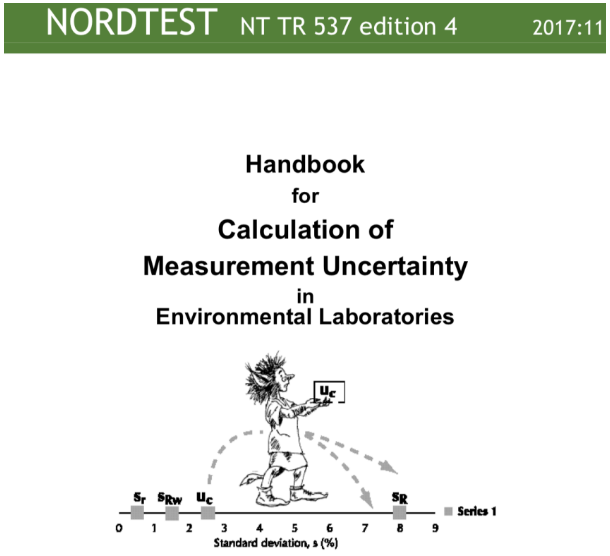
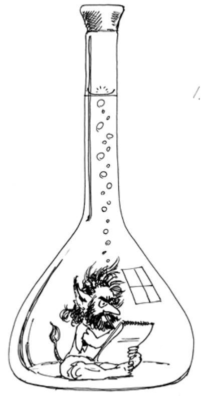
Uncertainty from validation and QC

Important difference in Nordtest from other guidance

- Bias – can take into account
  - bias variation
  - uncorrected bias
- Stresses U over measurement range





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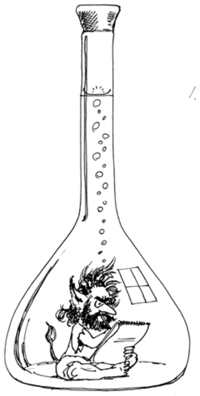
Uncertainty using MUkit

Estimated measurement uncertainties

Concentration range (µg/l)	Reproducibility method	u (Rw)	Bias method	u (bias)	Combined uncertainty	Expanded uncertainty
50-500	Control sample covering the whole analytical process	1.67 %	Interlaboratory comparisons / Proficiency tests	2.73 %	3.20 %	6.4 %



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Free software here
http://www.syke.fi/en
Search: *MUkit*

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Uncertainty over the measurement range

Measured value	Measurement Uncertainty, *U*(95%)	
	Absolute	Relative
3 - 20 µg/L	2 µg/L	
20 µg/L - …		10 %

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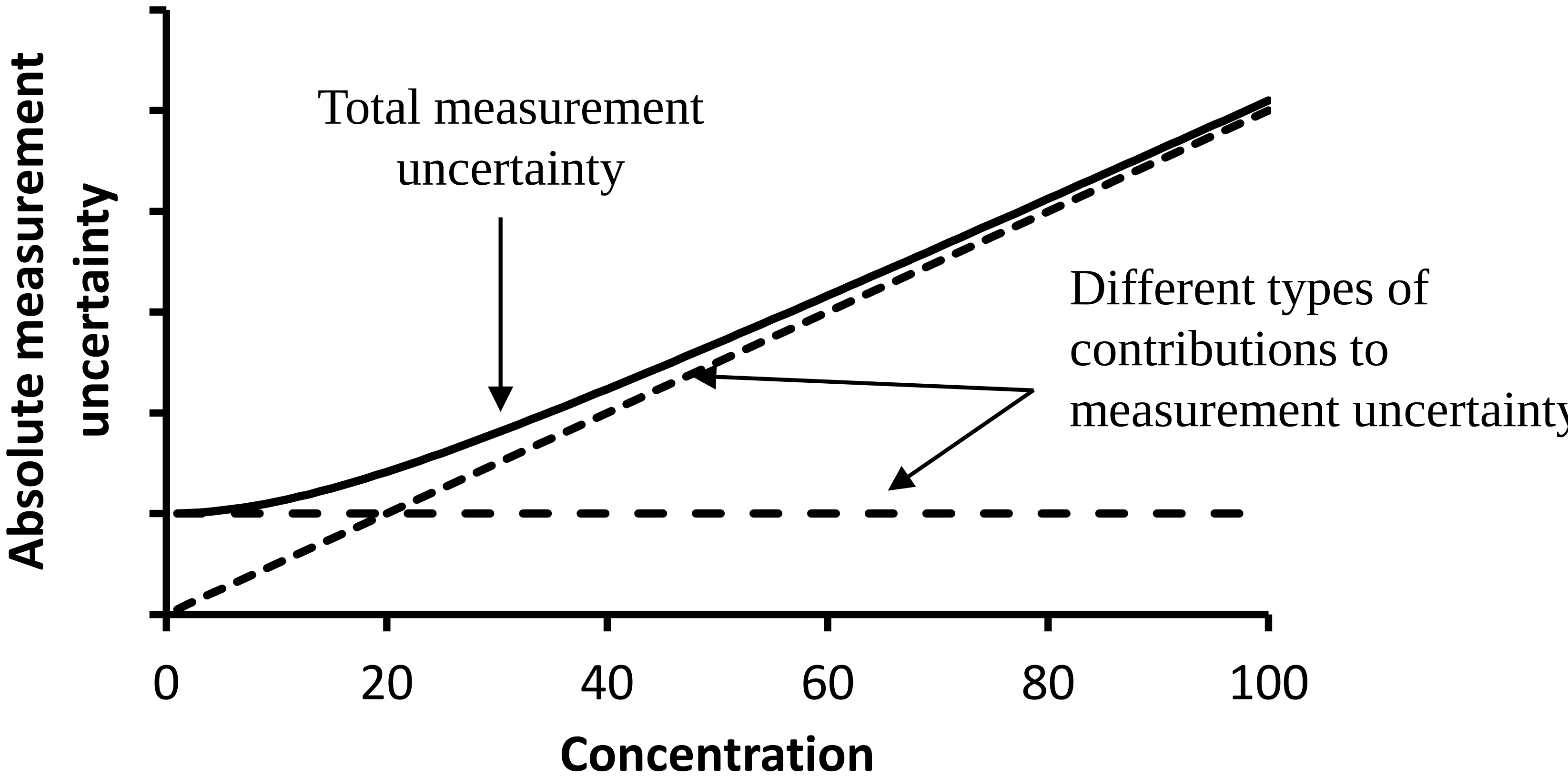
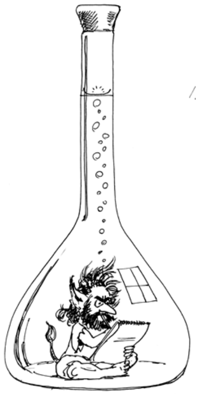
Uncertainty over the measurement range





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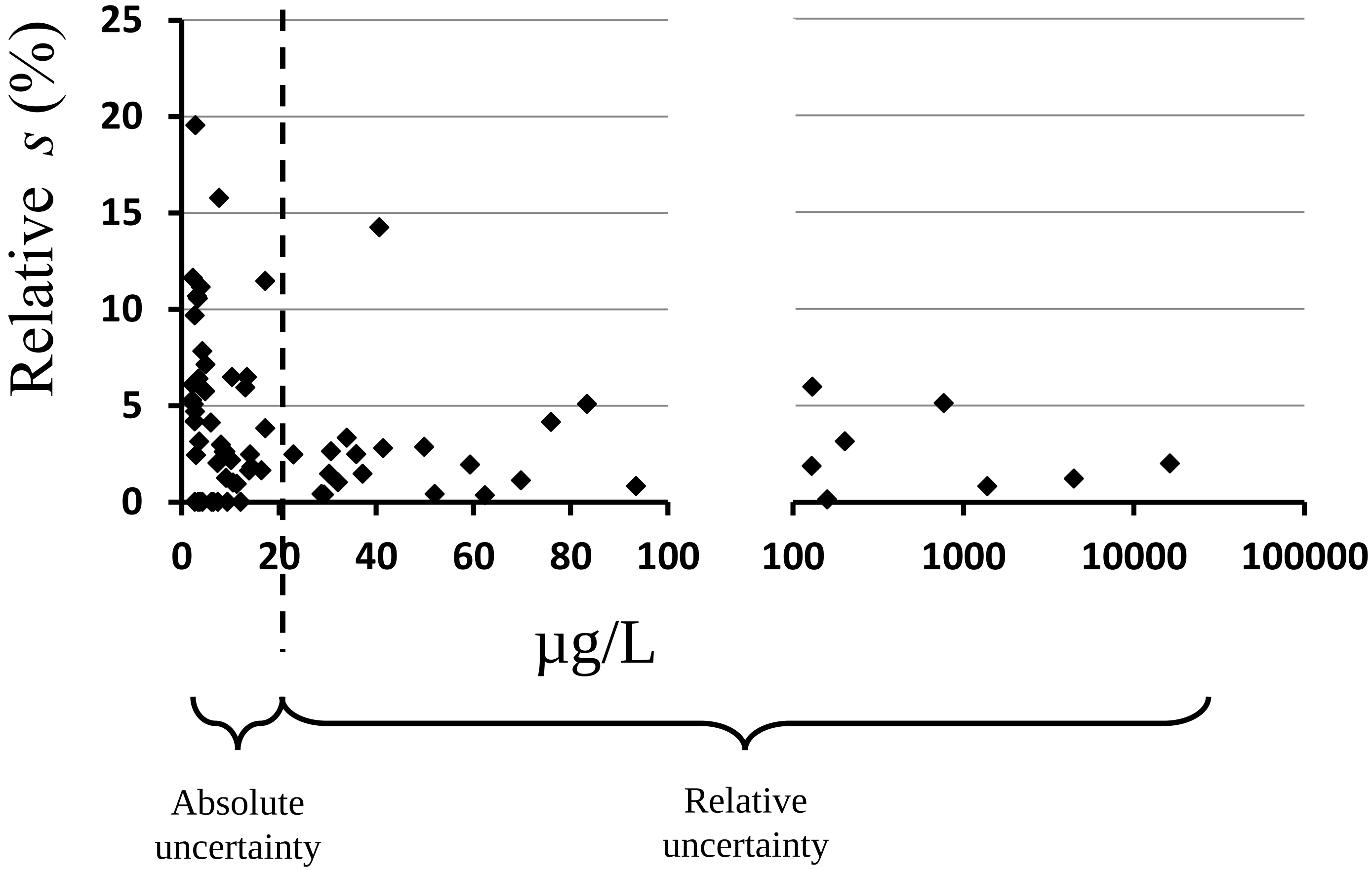
Relative uncertainty from duplicates NH_4-N



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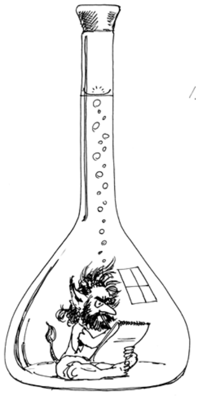
Standard deviation from duplicates - repeatability

1st result µg/L	2nd result µg/L	CV
3.60	3.10	10.6
7.46	7.25	2.0
31.9	32.36	1.0



More correct than range calculations used before

More versatile – works also with > 2 results

For each range a pooled CV (or *s*) is calculated

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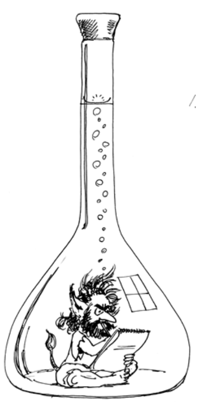

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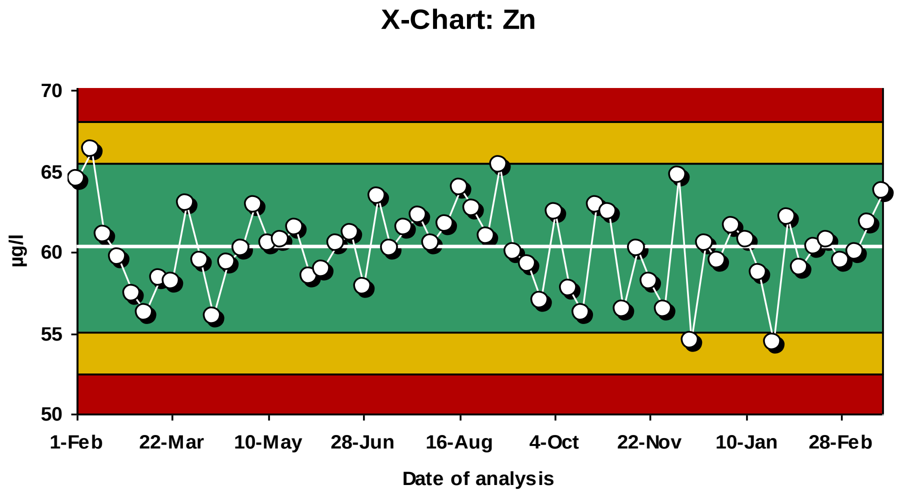
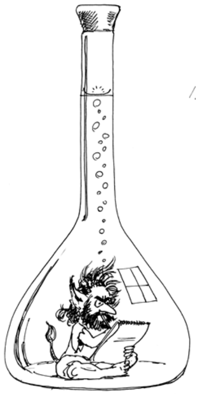
Control chart limits can give s_{Rw}





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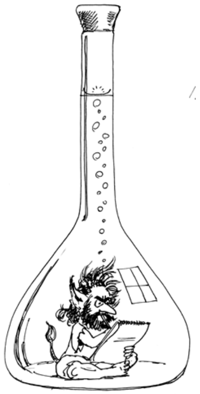
The control limits can be set wider - target control limits
Important to the use the actual control limits for s_{Rw}

NOTE if test samples are more difficult one needs to add a repeatability *s*.

Harmonisation with ISO 11352 *Water quality — Estimation of measurement and the software MUkit*

The Mukit software was developed from Nordtest handbook
The ISO standard was also develop from Nordtest TR 537.

Some changes were made…
The version 4 of the Nordtest handbook is now harmonised with the ISO standard and the software.



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On-line course – University of Tartu – Ivo Leitp

ESTIMATION OF MEASUREMENT UNCERTAINTY IN CHEMICAL ANALYSIS

Search

- Course introduction
- 1. The concept of measurement uncertainty (MU)
- 2. The origin of measurement uncertainty
- 3. The basic concepts and tools
- 4. The first uncertainty quantification
- 5. Principles of measurement uncertainty estimation
- 6. Random and systematic effects revisited
- 7. Precision, trueness, accuracy
- 8. Overview of measurement uncertainty estimation approaches
- 9. The ISO GUM Modeling approach
- 10. The single-lab validation approach
- 11. Comparison of the approaches
- 12. Comparing measurement results
- 13. Additional materials and case studies
- 14. Tests and Exercises

This course will be offered as online course during March 27 – May 7, 2018.
Registration is available here.

The course runs in Moodle environment . The students who have successfully passed the course will be issued a certificate of completion by the University of Tartu. A digital certificate of completion is free of charge. A certificate of completion on paper can be requested for a fee of 60 euros.

Course introduction

This is an introductory course on estimation of measurement uncertainty, specifically related to chemical analysis (analytical chemistry). The course gives the main concepts and mathematical apparatus of measurement uncertainty estimation and introduces two principal approaches to measurement uncertainty estimation – the ISO GUM modeling approach (the "bottom-up" or modeling approach) and the single-lab validation approach as implemented by Nordtest (the "top-down" or Nordtest approach). The course contains lectures, practical exercises and numerous tests for self-testing.

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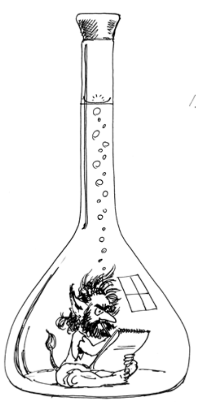

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Thanks for listening – more info www.trollboken.se

HOME ABOUT ANALYTICAL METHOD RESOURCES CASE STUDIES CONTACT SEA

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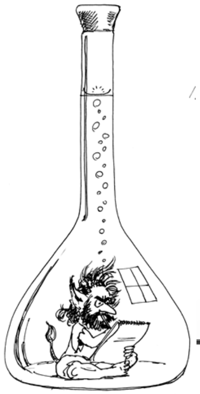
SVENSKA

Faced with a particular customer problem, the laboratory must first set the analytical requirement which defines a method that solves that problem. The main requirements are the following

- Scope - determination of a parameter (concentration of an analyte) in matrices,,,
- Measuring interval - also called range
- Precision - we often focus on a requirement on within-lab reproducibility *s(Rw)*
- Trueness - expressed as a requirement on maximum bias
- Ruggedness
- Measurement uncertainty



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