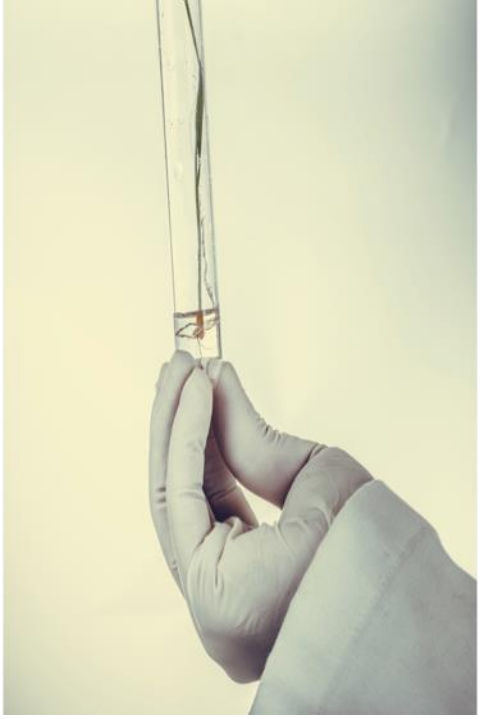
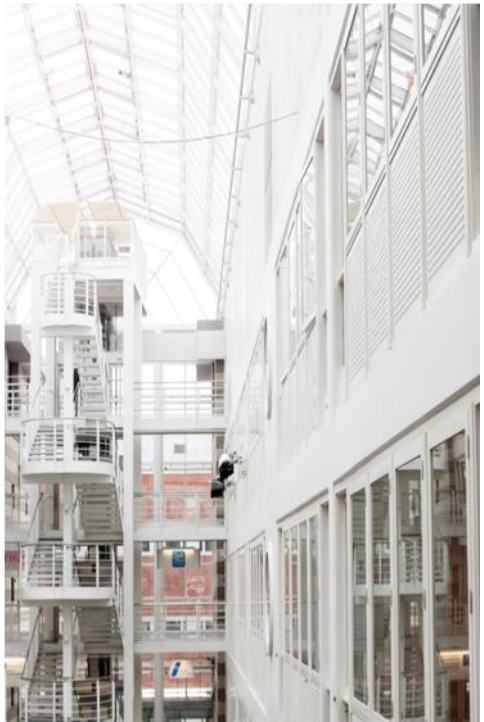
BIOECONOMY
BUILT ENVIRONMENT
ICT
BIOSCIENCE AND MATERIALS
SAFETY & TRANSPORT
CERTIFICATION





Research Institutes of Sweden

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

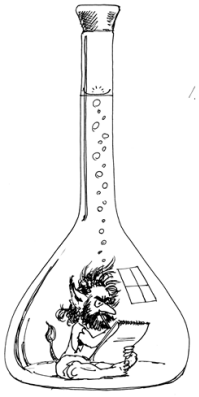
Standard solution & Gas

Digestion
SYNLAB & Sandviken
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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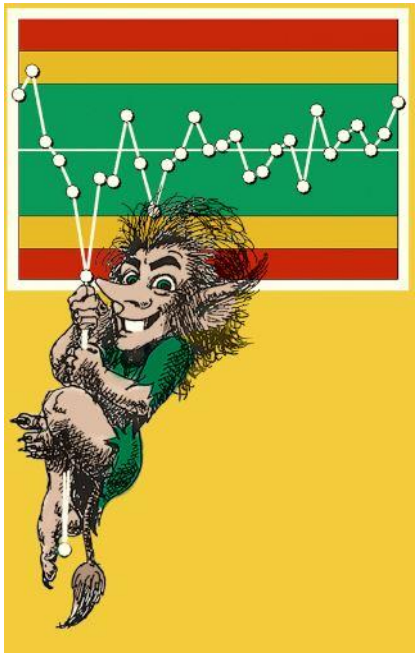
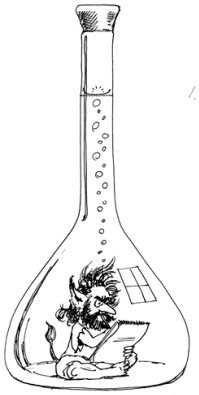
Quality Control in our Laboratories

The to be revised
"Troll book" TR569

www.nordtest.info





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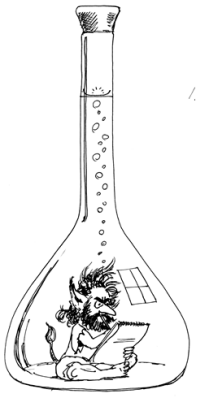
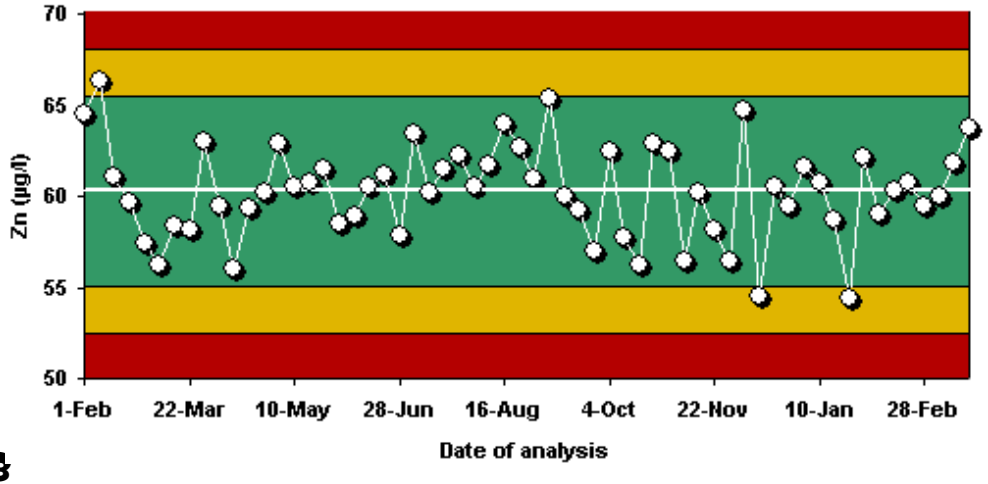
Purpose of internal QC

- Identify whether results can be released to the customer
- Identify trends
- Monitor method performance over time





There are different charts for monitoring different aspects of method performance

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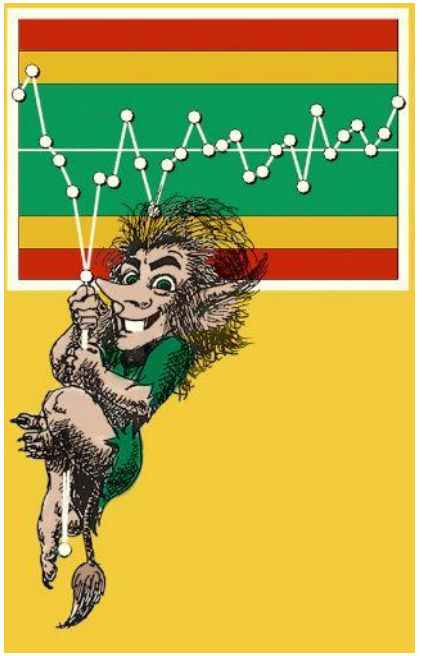
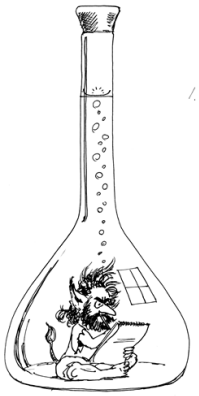
Nordtest TR 569 **Handbook for**
Internal Quality conTROLL

Based on experience gained by many laboratories using QC since 1980s in the Nordic countries.





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Some general recommendations in QC

Control limits

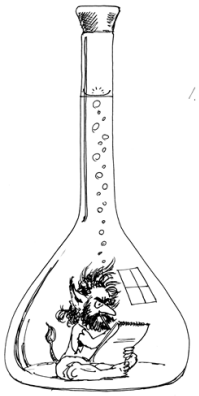
- Use fixed limit
- Use target limits if possible
  - Multielement analyses – target limit!

Central line

- Use fixed central line
- Use reference value if possible

Daily interpretation of QC

- All control values in green area are OK



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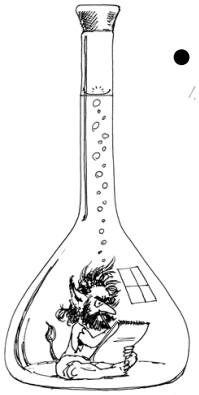
Terminology in internal QC

- Control sample:
  - Sample material whose analytical results are used to construct control charts, e.g. standard solutions, test samples, blank samples
- Control value:
  - Value entered on the control chart
- Measurement result:
  - Value reported as defined in the method. It is derived from the response by application of the calibration function
- Control limits:
  - Limits in a control chart (warning and action)



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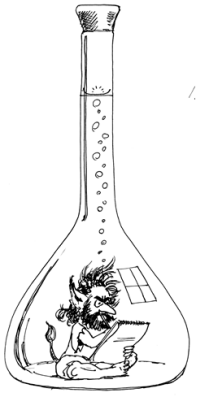
Planning QC

- Type of QC
  - Standard solutions, stable test samples, blank samples…
- Number of control sample
- Number of measurements made on control sample
- Frequency of QC
- Type of control chart
- Setting control limits
- Interpretation of charts
- Procedure for long-term review of charts



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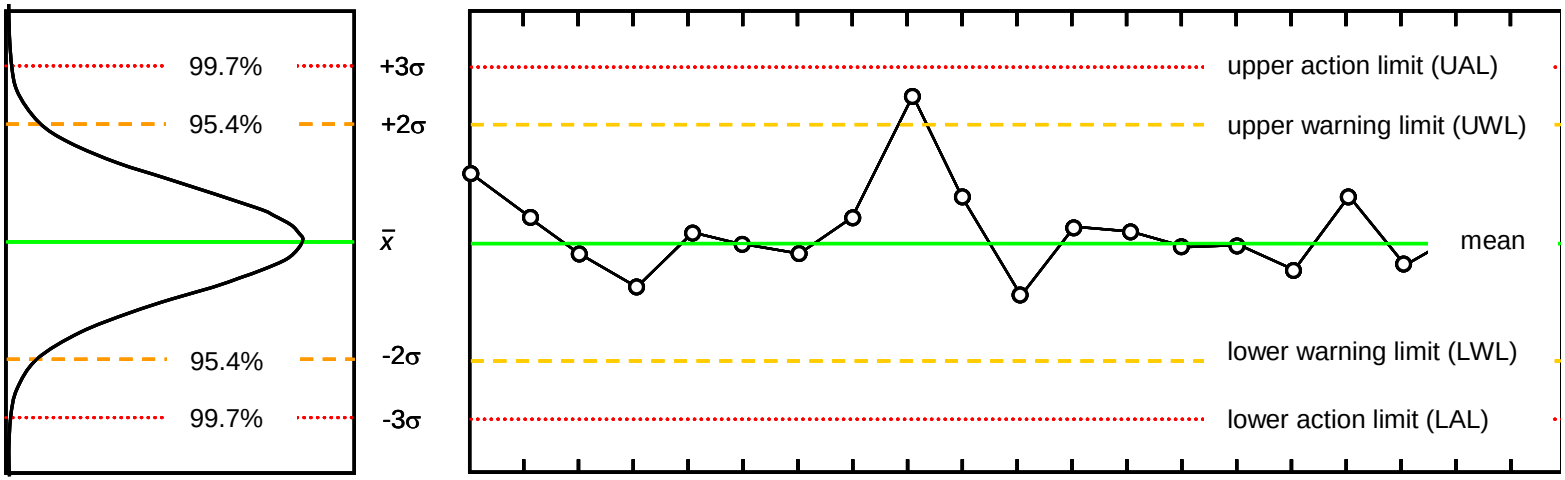
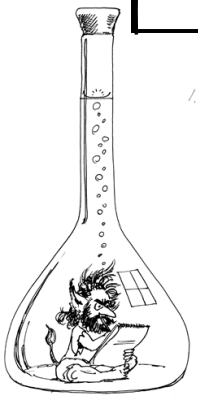
X-chart

- Used to monitor bias and precision
- Individual control values plotted in time ordered sequence



- Key features
  - central line
  - upper and lower warning limits
  - upper and lower action limits



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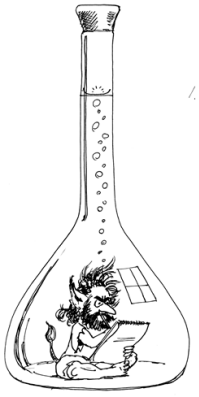
X-chart

- Used to detect changes in the mean (bias)…
  - if the mean shifts due to presence of bias, results will be nearer to one set of warning limits so probability of an 'out of control' situation increases
- …and changes in standard deviation (precision)
  - if standard deviation increases, scatter of results will increase so probability of an 'out of control' situation increases
- Variations on the simple *x*-chart
  - plot mean of *n* results
  - plot %recovery – central line is target of 100% recovery



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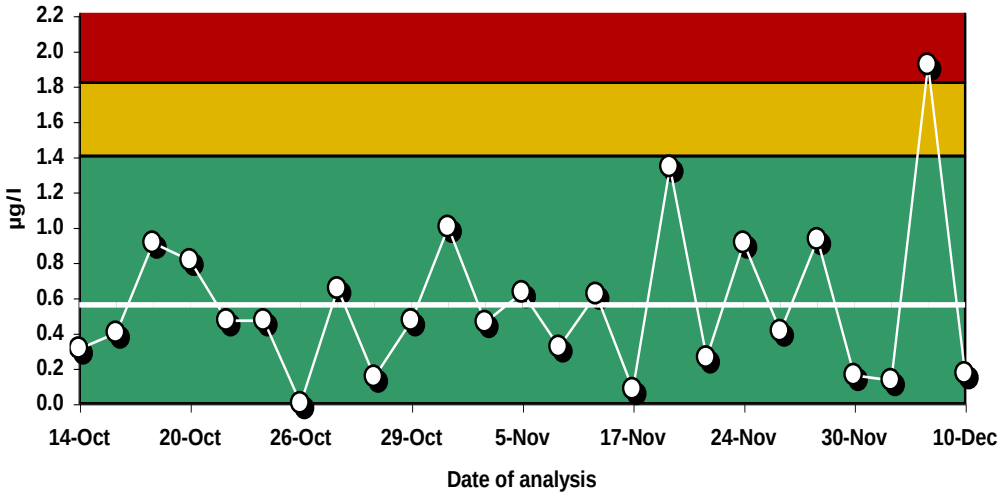
Range chart – R or r%

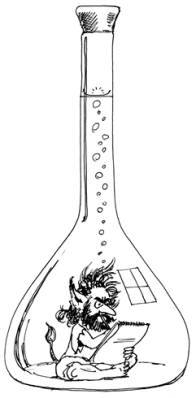
- Used specifically to monitor repeatability (within-run precision)
  - control value is range of each set of *n* results ($R = x_{max} - x_{min}$)
  - typically *n*=2 or 3, for larger *n* the standard deviation can be plotted
  - can also plot % range (*r*%) or CV%



Key features:

Central line

Upper warning and action limits



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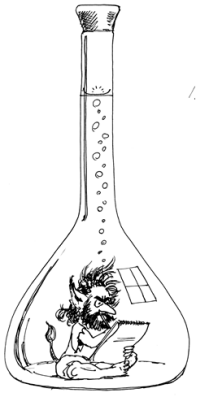
Uncertainty over the measurement range

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



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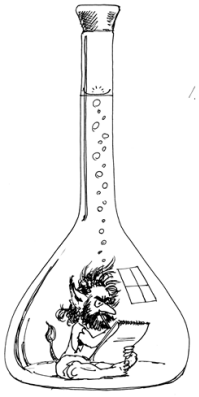
Daily interpretation

Green area (within warning limits)
→ the test sample results are reported

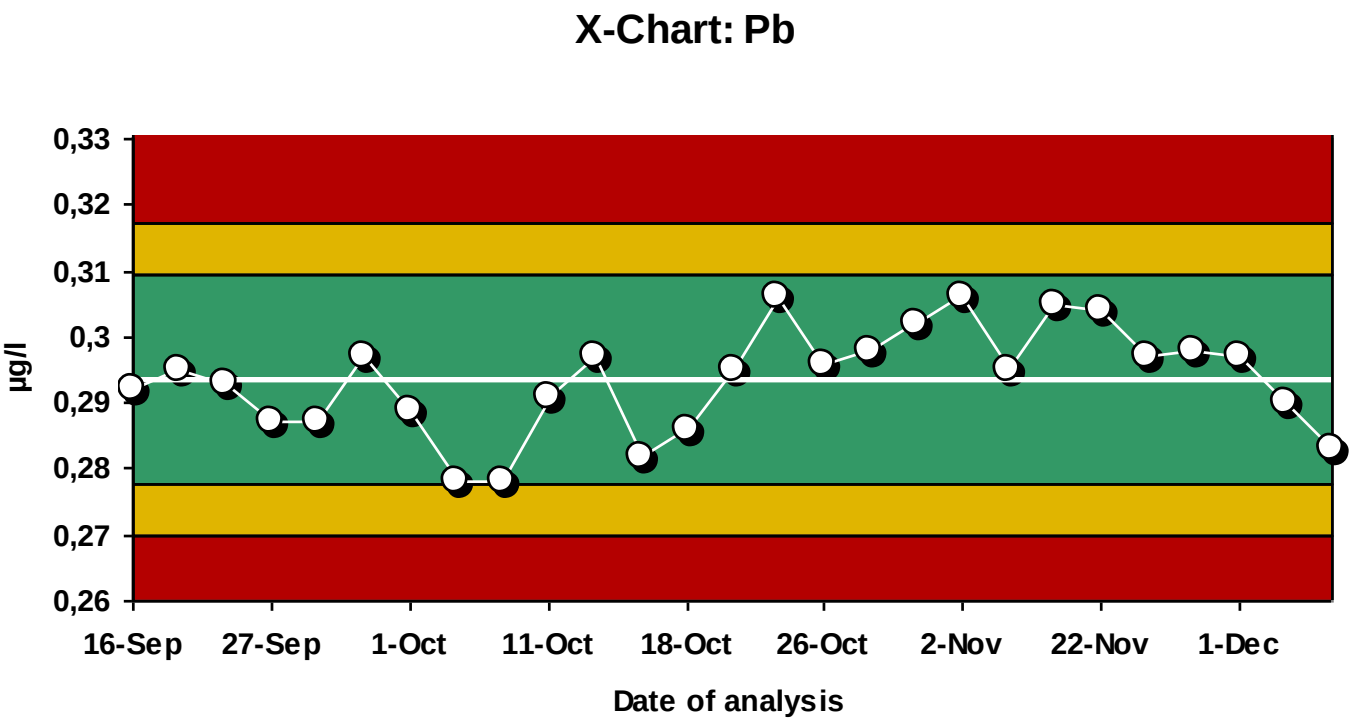
Red area (outside the action limits)
→ no test sample results are reported

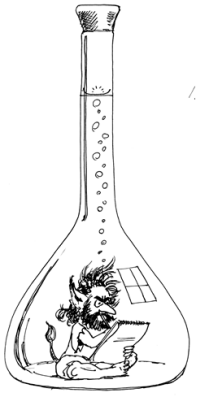
Yellow area (between warning and action limits)
→ 2 out of 3 in yellow area – no test samples are reported



TROLLBOKEN AB

Daily interpretation





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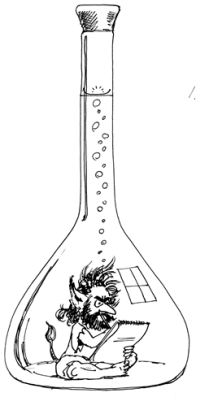
Rules that we do NOT recommend

In ISO 8258 Shewhart control charts (1991) there are more rules e.g.

- Nine points in a row on one side of the central line
- Six points increasing or decreasing
- Fourteen points in a row alternating up and down

In the Westgard rules (www.westgard.com) there are **also** more rules e.g.

- Four points in a row between the action and warning limit



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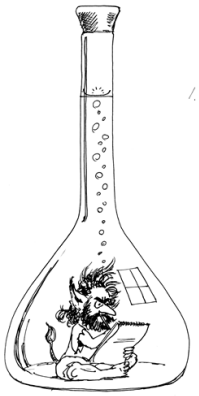

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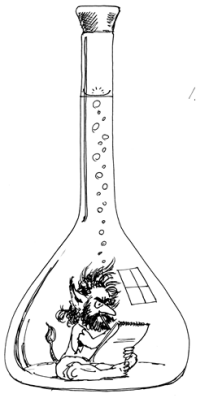
Target control limits

Target control limits based on customers requirement

The standard deviation needed for the control chart, s_{target} is estimated from the requirement on $\boldsymbol{s_{Rw}}$ (the **w**ithin laboratory **R**eproducibility) from

- ❑ Client/Production
- ❑ Directive – e.g. EU
- ❑ Regulations
- ❑ Information in standard method



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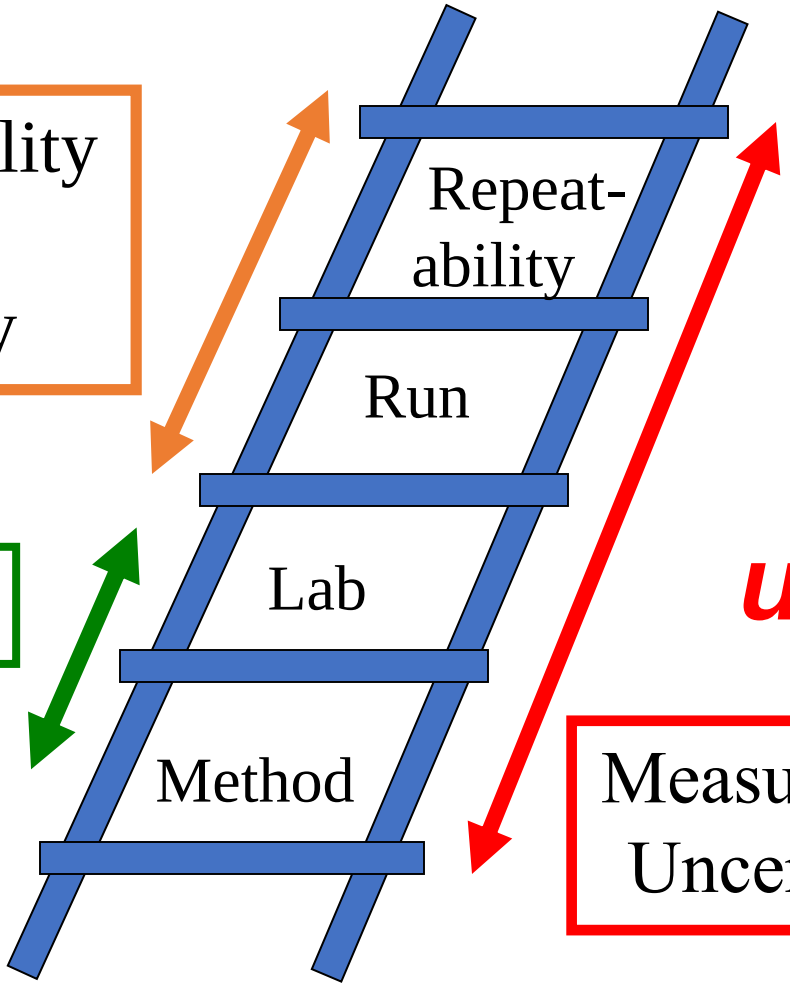
Ladder of Errors

u_{Rw}

Reproducibility within laboratory

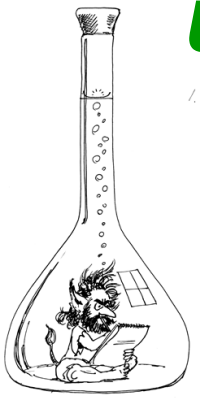


u_c

Measurement Uncertainty

u(bias)

Bias



Target control limits – what does the Troll say?

From a target uncertainty, U

$s_{Rw} = U/4$

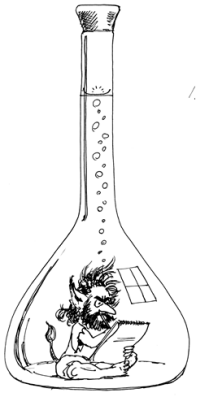
When test and control samples are similar and bias is low*

$s_{Rw} = U/3$

U = *expanded uncertainty*

s_{Rw} = within-lab reproducibility

*Will be in next version of TR569



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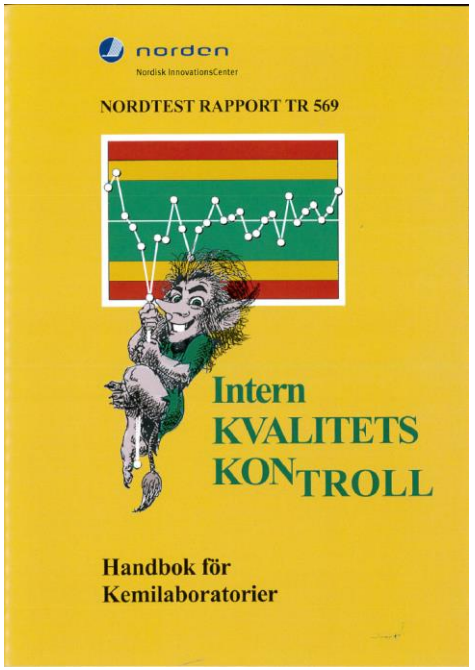
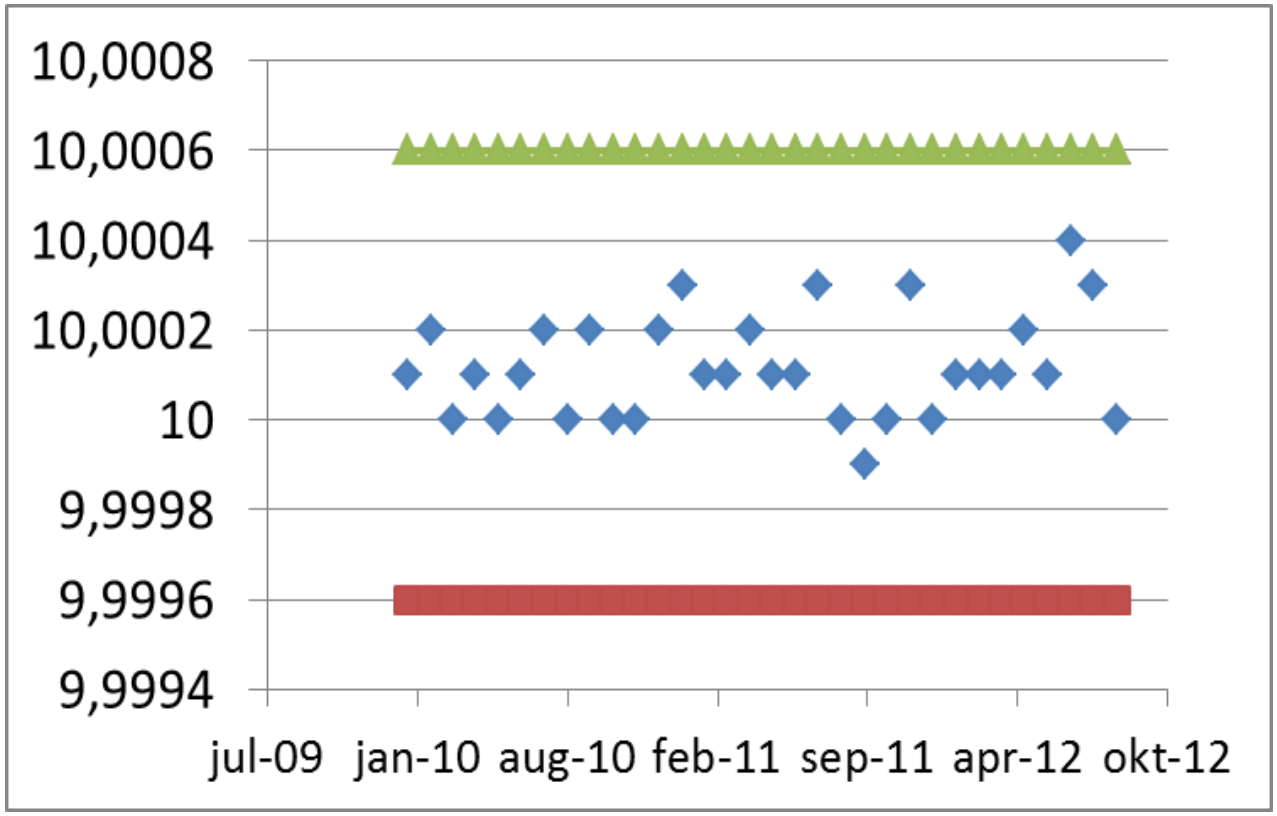
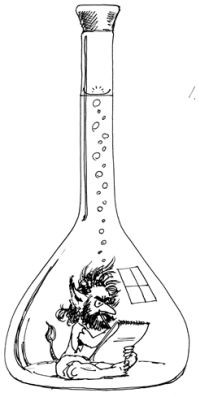


www.nordtest.info

Target limits for a balance

Use 5 digits as action limits





- 4 decimal Mettler AX204
  Nominal weight 10.0001 g.
  Action limits 10,0006 and 9.9996 g.

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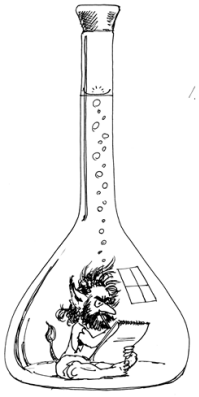
Target limits – demand in environmental sector

- Drinking water directive 98/83/EC
- An environmental directive 09/90/EC
- German demands from LAWA* AQS Merkblatt A6/1 2004
- Danish demands in law BEK nr 1146 24/10 2017
  https://www.retsinformation.dk/Forms/R0710.aspx?id=194194

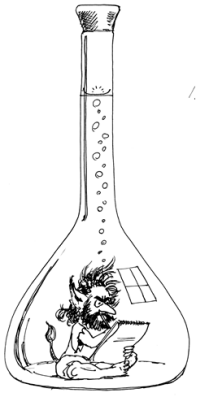


*The LAWA is the German Working Group on water issues of the Federal States and the Federal Government represented by the Federal Environment Ministry

Target limits – demand from MSA

Measurement System Analysis MSA sets demand from specification (tolerance)

- Bias ≈ 0
- Acceptable s_{Rw} < 5 % of specification
- Good s_{Rw} < 1.7 % of specification



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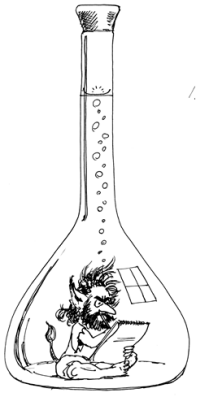
Target limits – EU drinking water directive

At the level of interest

- Bias < 10 %
- Acceptable s_{Rw} < 5 % of specification



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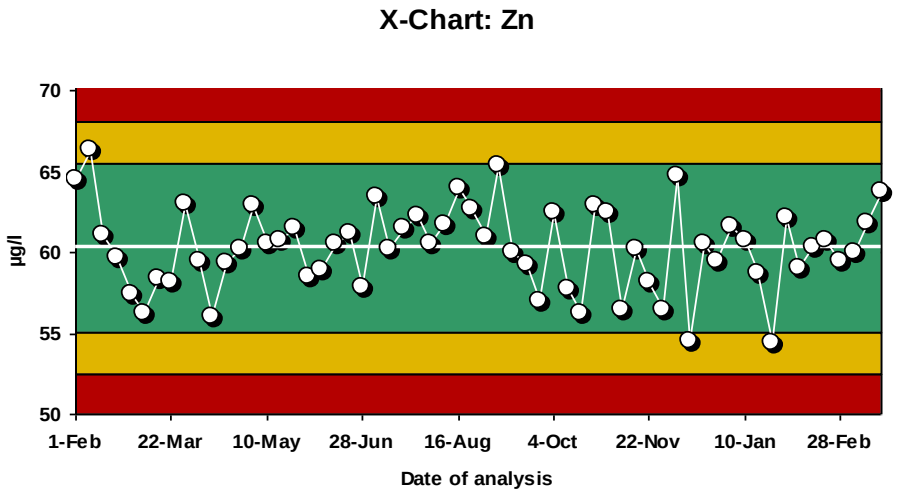
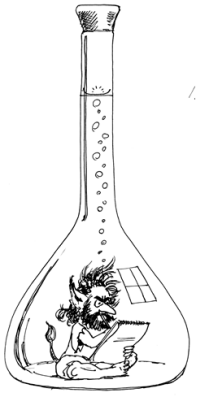
Uncertainty estimation - Control limits give s_{Rw}





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*.

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Some general recommendations in QC

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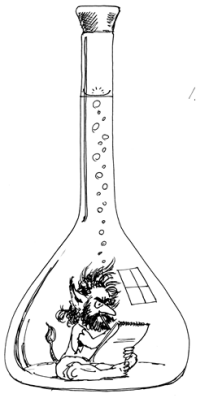

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Thanks for listening – more info www.trollboken.se

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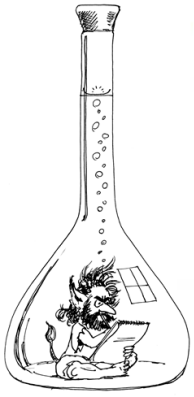
UNCERTAINTY (U)

ANNUAL QC REVIEW

SVENSKA

Set up the internal quality control by choosing suitable control samples and set-up control charts with preliminary control limits (preferably *target control limits*) - see Nordtest TR 569 "Trollbok" for further guidance, www.nordtest.info



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