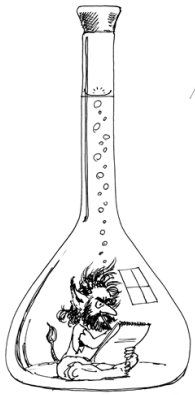# Internal quality control using the Nordic Trollbook yesterday, today and tomorrow
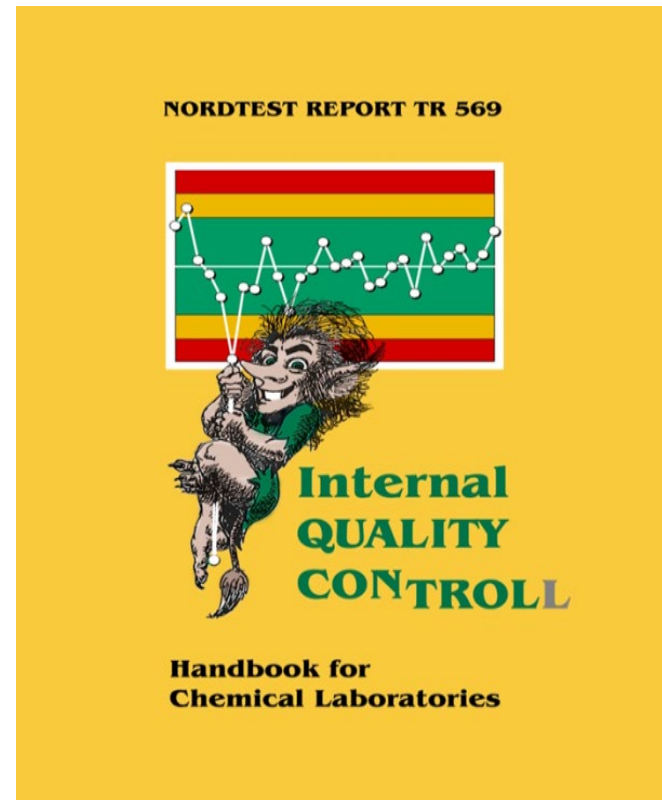
Nordtest Trollbook 569

www.nordtest.info

Bertil Magnusson

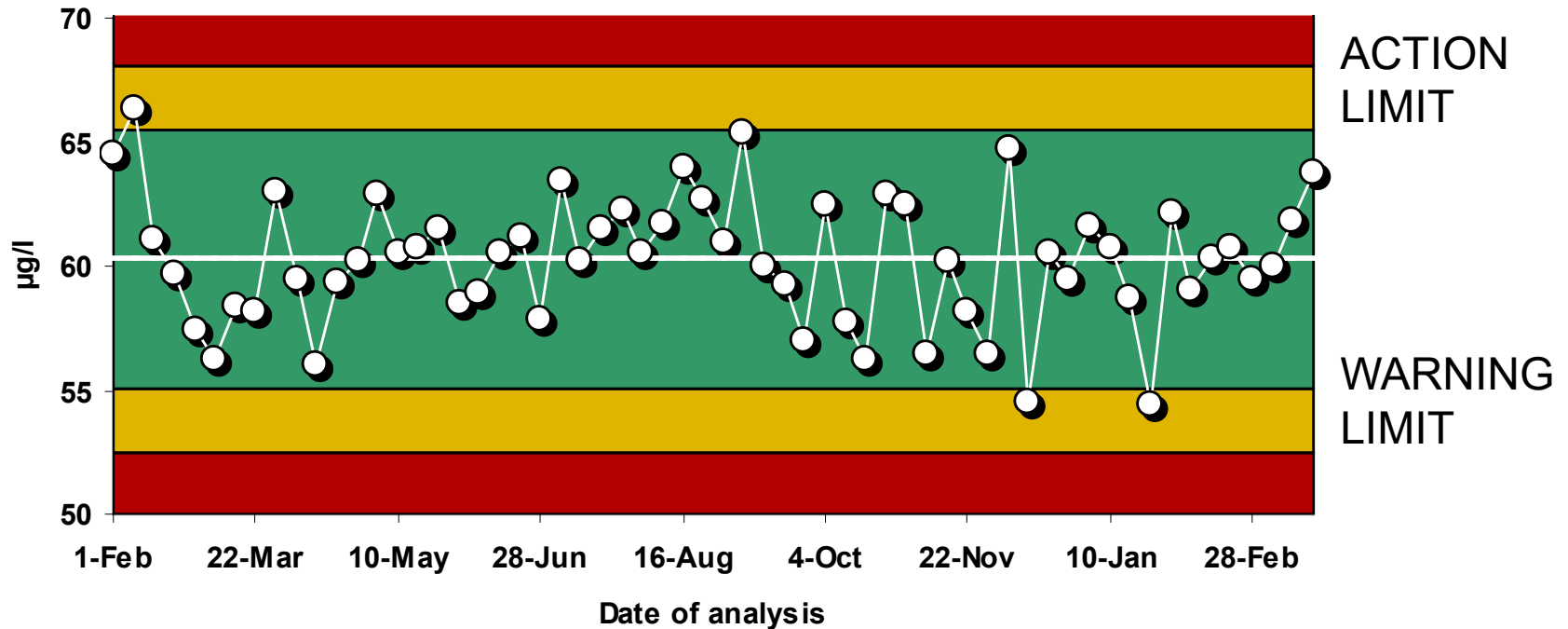TROLLBOKEN AB

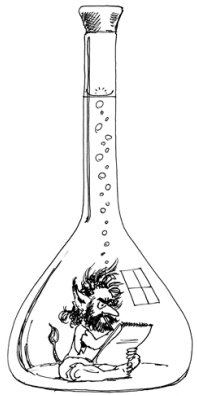NORDTEST REPORT TR 569

Internal QUALITY CONTROLL

Handbook for Chemical Laboratories

QC chart – control limit at 2 and 3 *s*

X-Chart: Zn

The QC (internal quality control) is important for a laboratory to know and **show** the quality of the results.
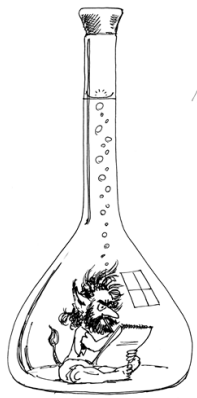
Summary

Always communicate to client the control chart

Use target control limits for setting up QC

Use simple rules for evaluating the QC

Uncertainty estimation - precision

Tell client results is ± 2 s_{Rw}

TROLLBOKEN AB

Communicate to client the control chart two scenarios

1) the client trusts **all** the figures reported in the result

2) the client is questioning the results when a very different result from normal is reported or not in line with theory

Let's start with client questioning the result

TROLLBOKEN AB
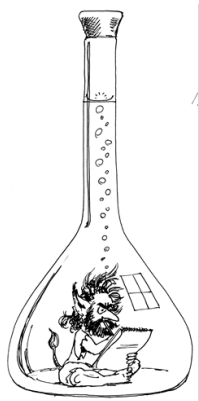
Once upon a time there was a laboratory who always quarrelled with the catalyst department…

…but one day during coffee break,
one of my colleagues said:

*There are no extra samples from the catalyst department any more.
They always used to send a second or a third sample because they did
not believe the first results*

- I was a new analytical chemist at this analytical laboratory of a chemical company, later AKzoNobel
- I wanted to start with QC in the lab but
  - My colleagues did not like me proposing MORE work

TROLLBOKEN AB
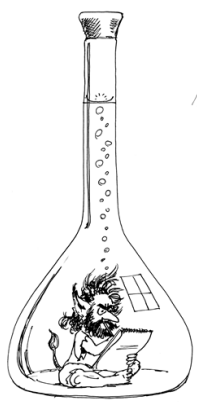
The catalyst department

- Produced fluid cracking catalysts, FCC, to crack crude oil into lighter fractions

- The catalyst is poisoned by metals in the crude oil, e.g. Ni and V – maximum 1000 mg/kg

- Each month the refinery took a sample of the catalyst and send to the laboratory
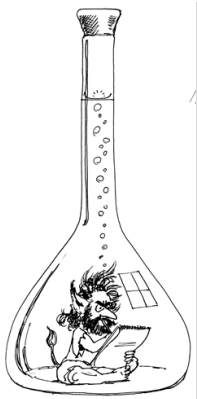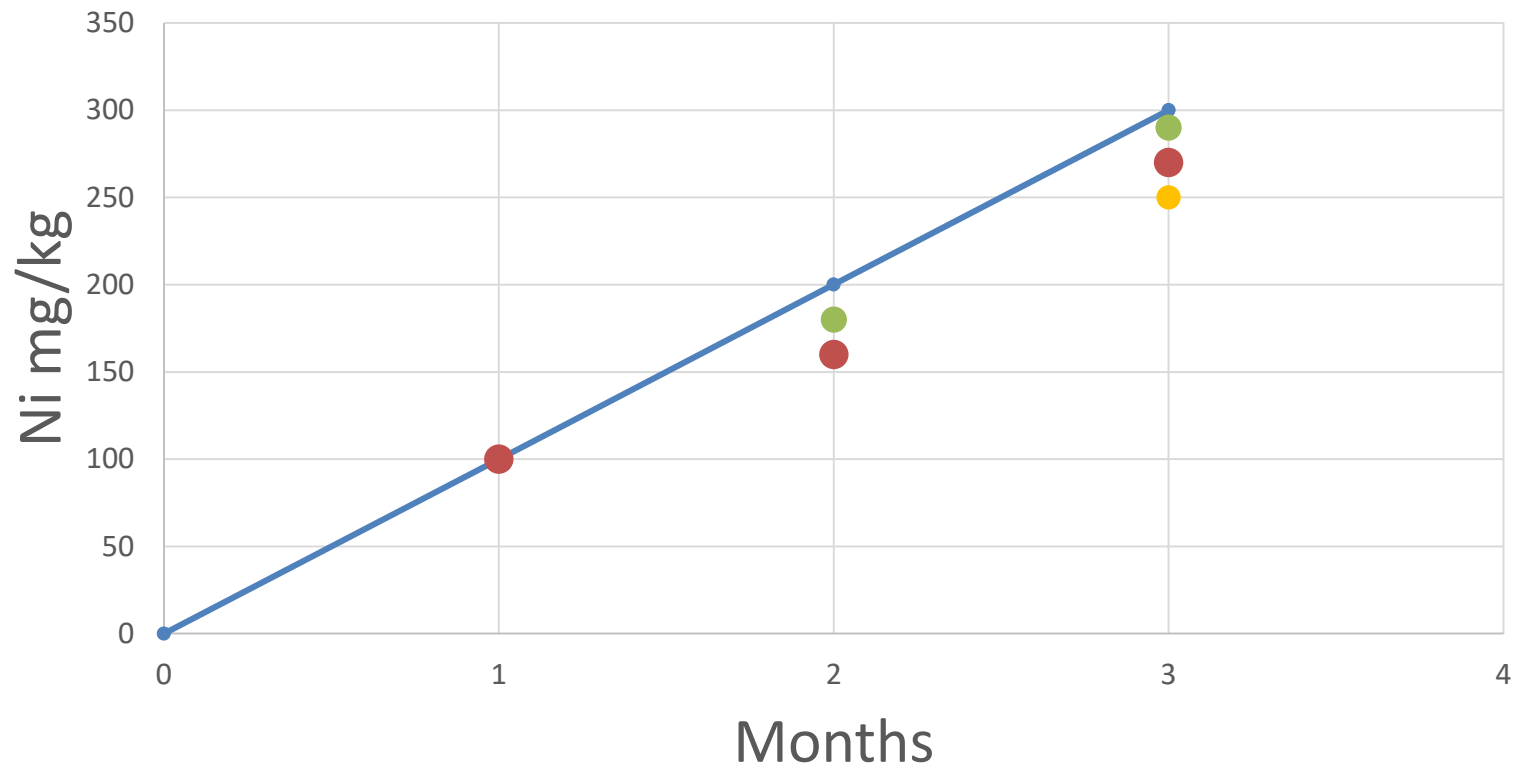
- The laboratory analyse Ni, V ….

TROLLBOKEN AB
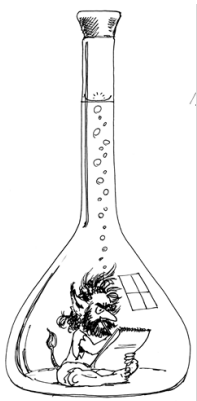
Prediction based on results month 1.
Lab results month 2 does not fit with predicted!

Ni in FCC catalyst from refinery X

TROLLBOKEN AB
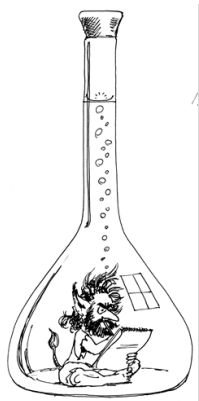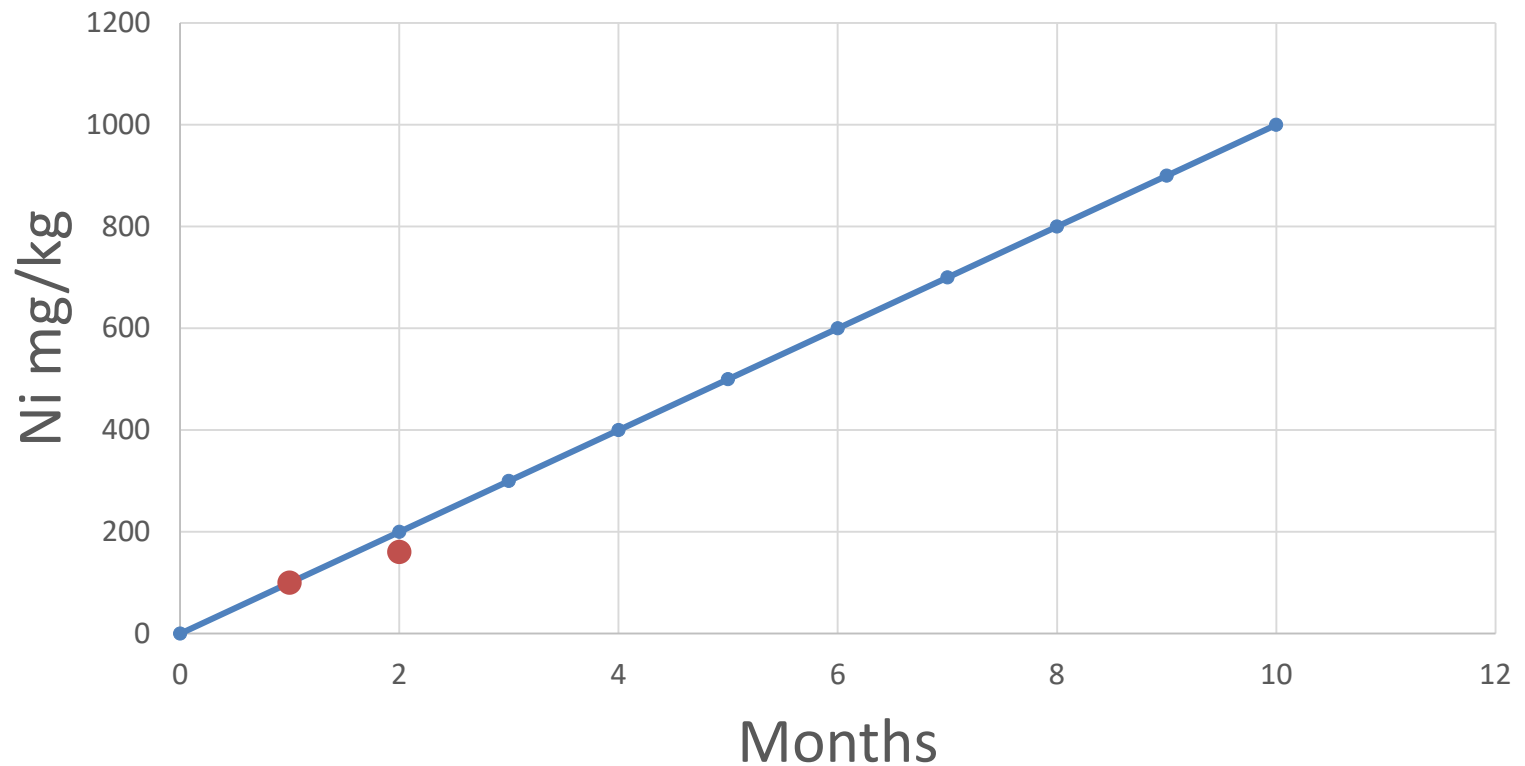
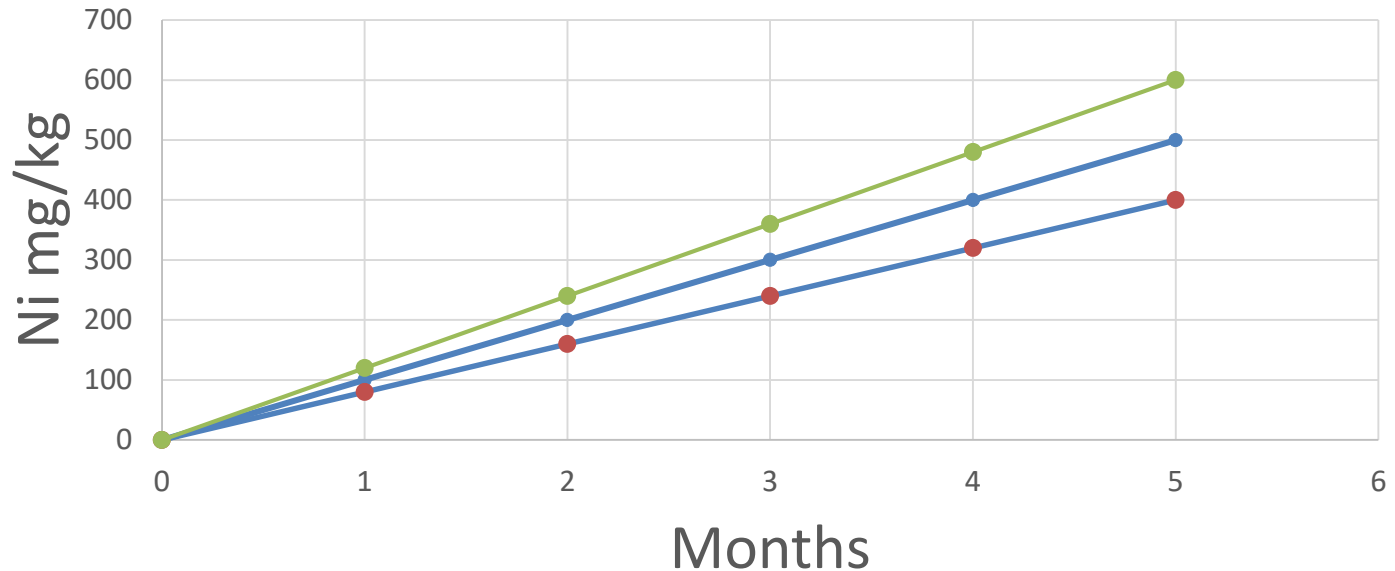# Lab results month 2 and 3 does not fit with predicted – each month extra analysis

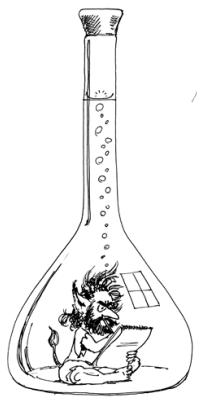## Ni in FCC catalyst from refinery X

TROLLBOKEN AB

Solution – Communicate to client value month 1 could be 80 or 120 (100 ± 20 mg/kg)

New predictions
Ni in FCC catalyst from refinery X

Note: ± is **not** expanded uncertainty it is 2 s_{Rw}
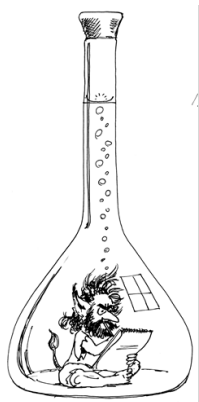s_{Rw} is within-laboratory reproducibility (intermediate precision

…and they lived happily all after

and no more extra samples from the catalyst department

and

my colleagues started to ask me

Could you help me with setting-up this control charts that they use for the FCC catalyst…

First scenario

The QC (internal quality control) is important for a laboratory to know and show the quality of the results.

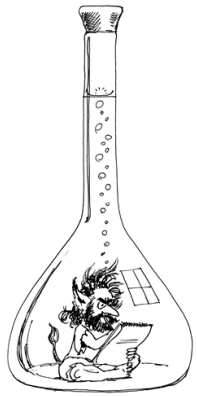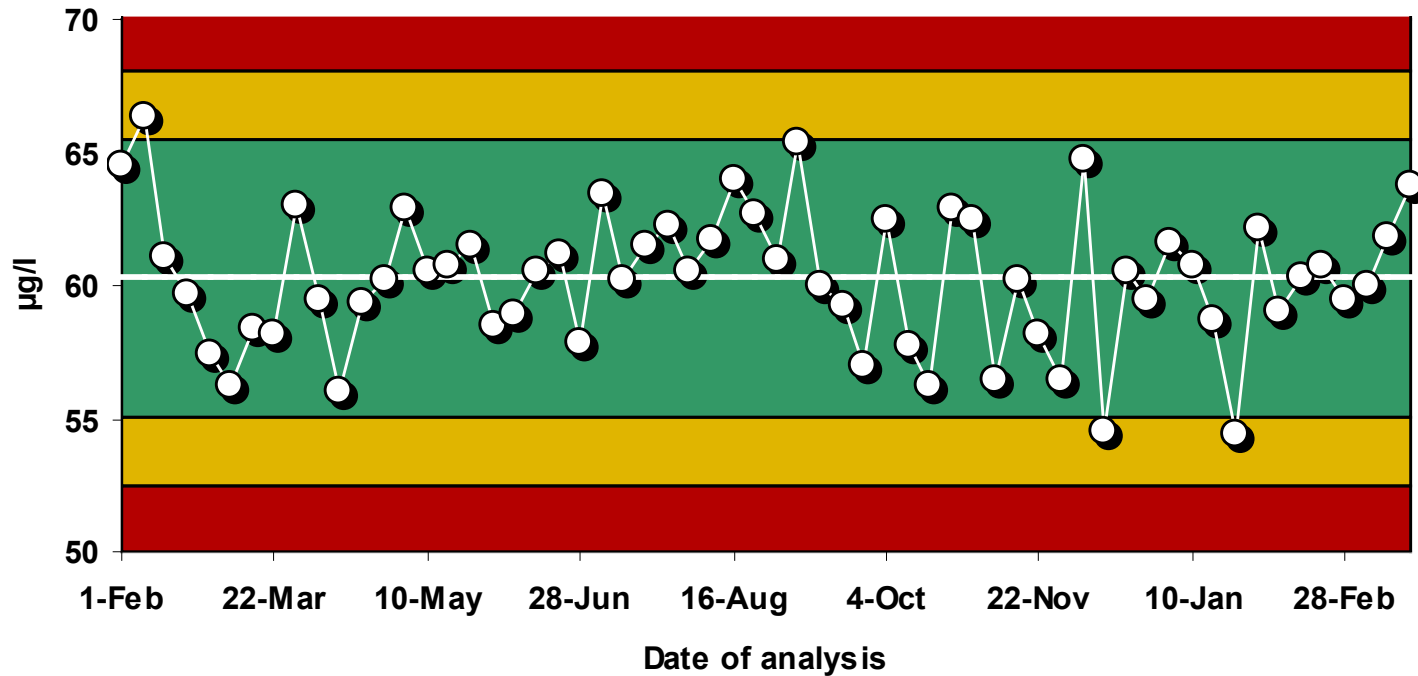We will start with two common scenarios for a production laboratory

1) the client trusts **all** the figures reported in the result

2) the client is questioning the results when a very different result from normal is reported or not in line with theory

TROLLBOKEN AB
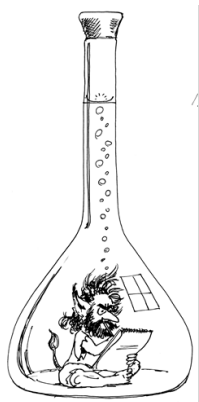
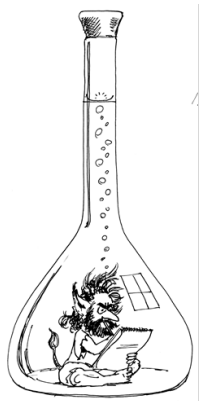# Client trust all figures: Solution
Show the client the control chart

X-Chart: Zn

TROLLBOKEN AB

Client trust all figures: Solution
Tell the client $\pm 2s_{Rw}$

A reported value x could
be in the range
$x \pm 2s_{Rw}$

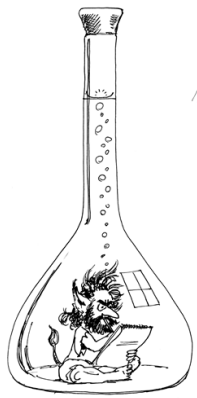TROLLBOKEN AB

Summary

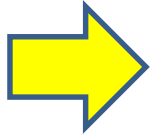**Always communicate to client the control chart**

Tell client results is ± 2 s_{Rw}

➡️ **Use target control limits for setting up QC**

Based on requirements

Use simple rules for evaluating the QC

**Uncertainty estimation - precision**

Setting target control limits

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

- Warning limits will be $\pm\ 2\ s_{\text{target}}$
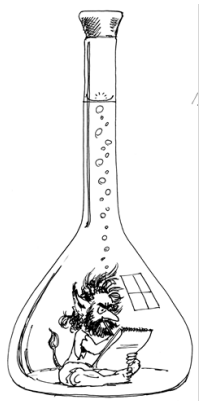- Action limits will be $\pm\ 3\ s_{\text{target}}$

In multi-analyte analyses target control limits could be used for those analytes/parameters that are less important

TROLLBOKEN AB
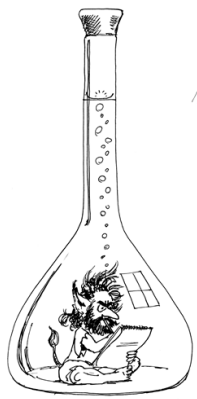
Target control limits based on requirements

Examples

- LOQ

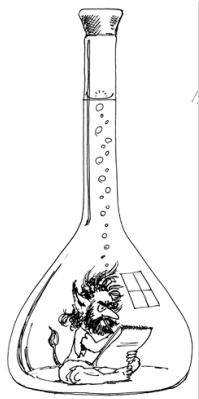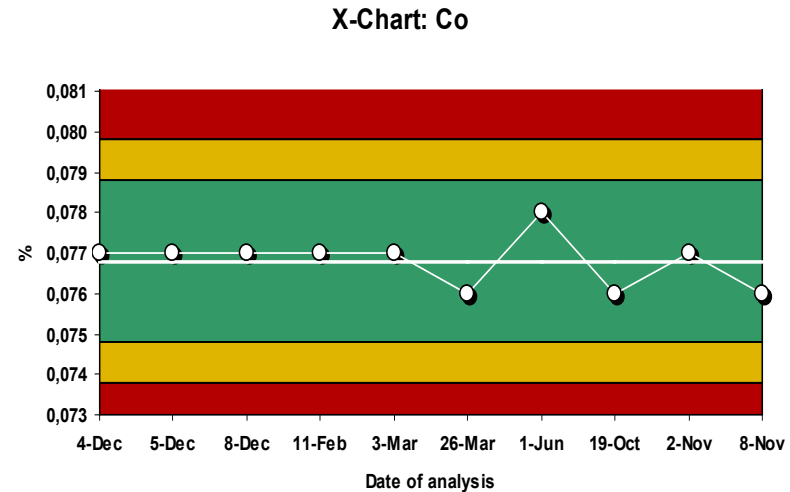- Measurement System Analysis – MSA

- EU directives

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Target control limits
Target *s* from LOQ

The requirement for LOQ;
Limit Of Quantification

- LOQ is 0,01 % cobalt.

- LOQ is 10 s_{Rw}

The target standard deviation *s*

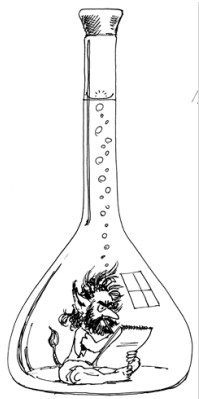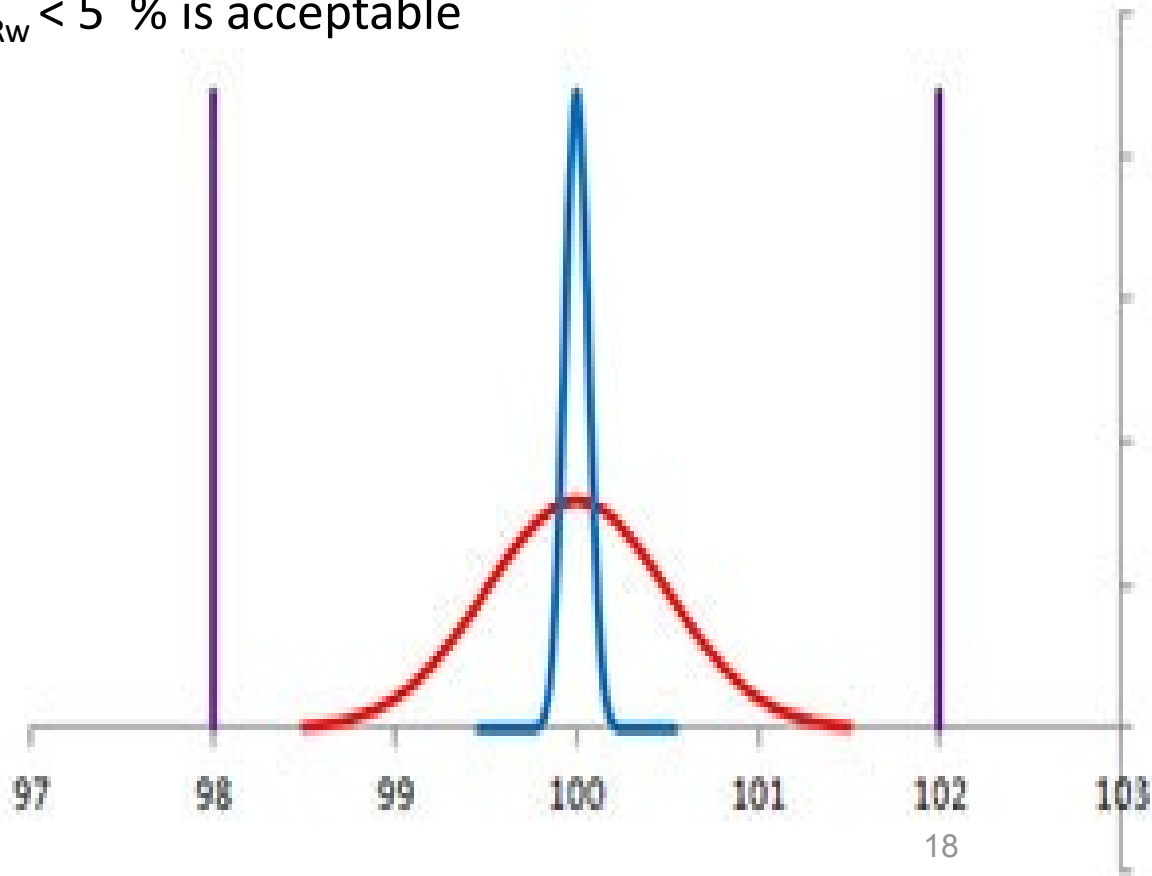for setting the control limits is then 0,001 % cobalt

X-Chart: Co

MSA – Measurement System Analysis

- $s_{Rw} < 1,7$ % of tolerance/specification
- $s_{Rw} < 5$ % is acceptable

97   98   99   100   101   102   103

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Target control limits
Target s from directives

- **EC drinking water directive 98/83/EC**
at a specific concentration

- Within-laboratory reproducibility, $2\, s_{Rw} < 10\ \%$

- **2009/90/EC pointing to the**
Water Framework Directive 2000/60/EC
at a specific concentration

- LOQ $< 30\ \%$
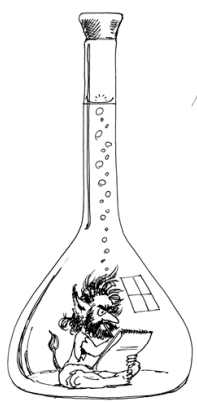
TROLLBOKEN AB

Summary

Always communicate to client the control chart	Tell client results is ± 2 s_{Rw}
Use target control limits for setting up QC	Based on requirements
Use simple rules for evaluating the QC	Propose 3s and 2s rules and yearly review
Uncertainty estimation - precision	Use the control limits NOT the actual s measured
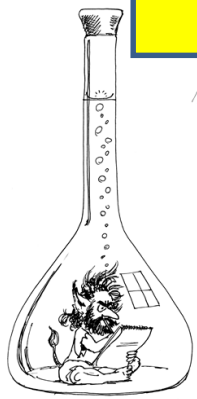
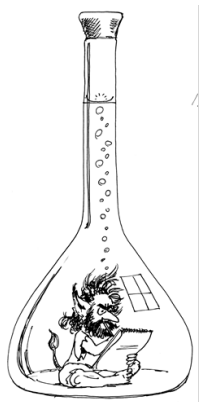# Simple and clear rules for QC according to the Trollbook

The method is out of control if
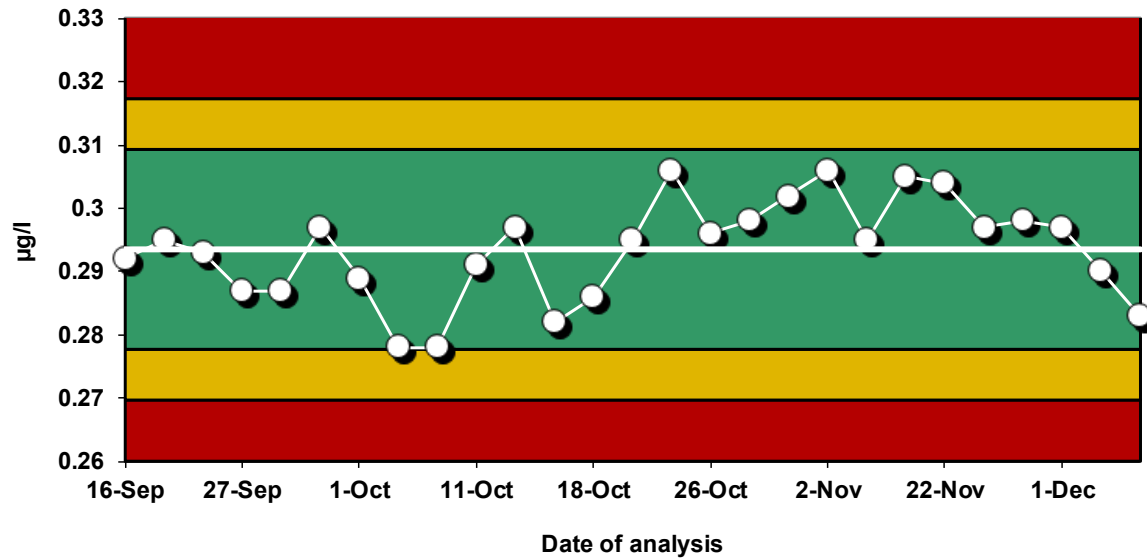
- control value is outside action limits,

- control value is between warning and action limits and ***at least one of the two previou*s** control values is also between warning and action limits
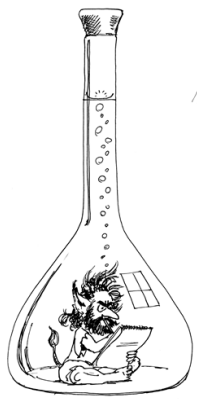
TROLLBOKEN AB

Is this analytical method in control?

X-Chart: Pb

Yes – all values within warning limits **but** we need to do a yearly review of the chart

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Summary

Always communicate to client the control chart

Tell client results is $\pm$ 2 s_{Rw}

Use target control limits for setting up QC

Based on requirements

Use simple rules for evaluating the QC

Propose 3s and 2s rules and yearly review

Uncertainty estimation - precision

Use the control limits NOT the actual *s* measured
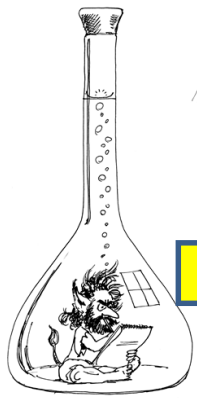
Ladder of errors
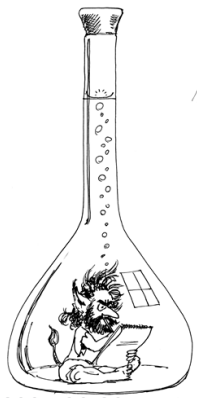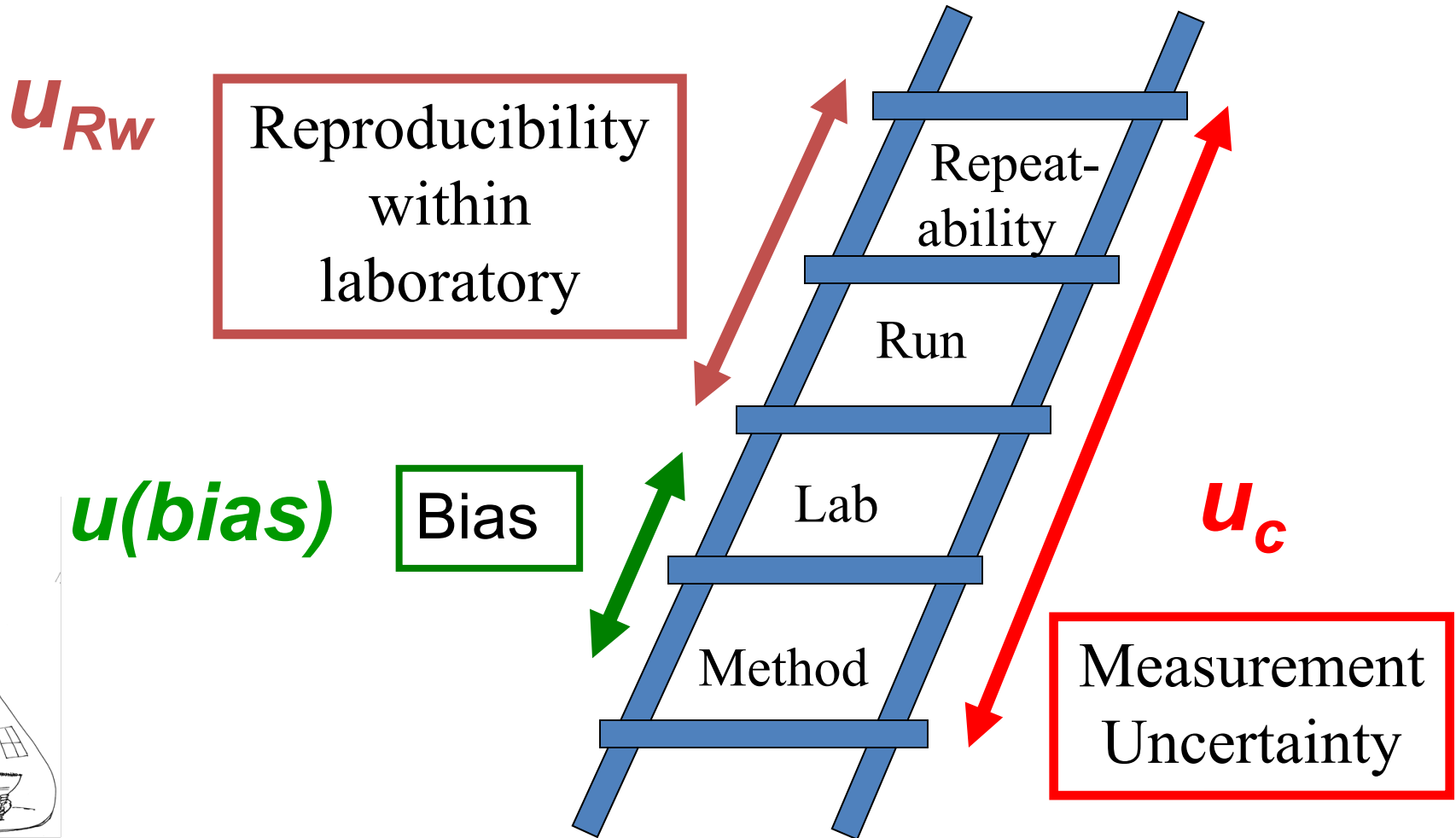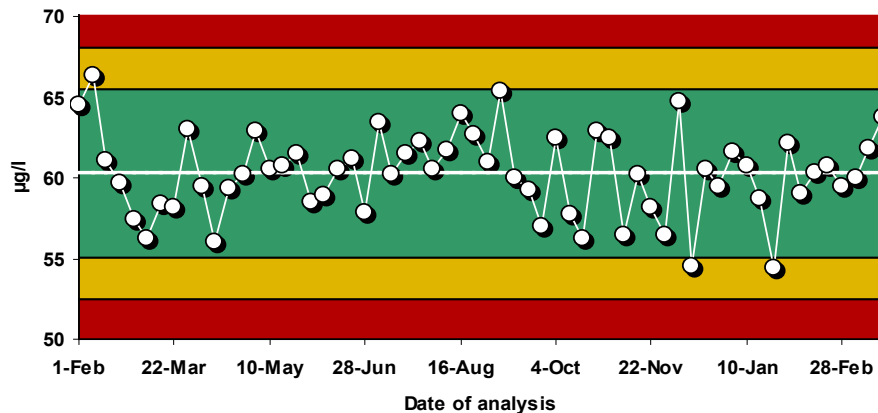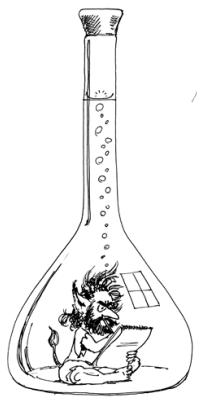
Control chart limits can give $s_{Rw} = u_{Rw}$ for samples covering the whole analytical process

X-Chart: Zn

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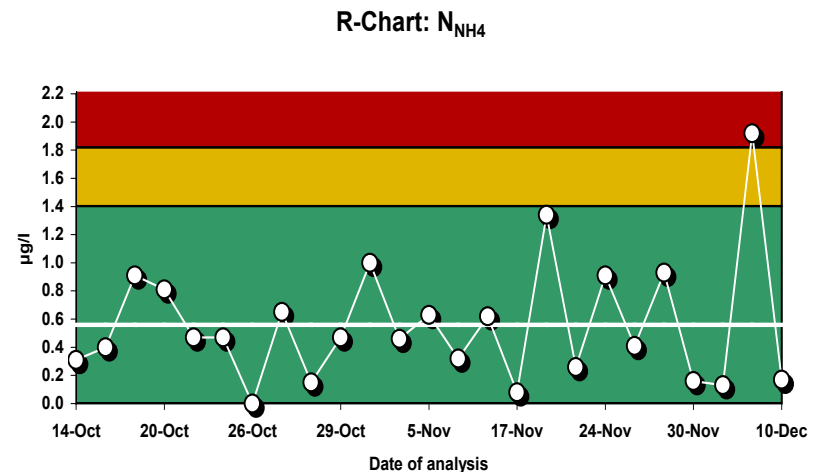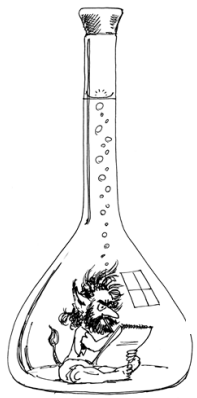
TROLLBOKEN AB

Why the Troll

Once upon a time there were a lot of Trolls in the Nordic countries. They were sometimes pestering us so we have a saying something like

The Troll is up to mischief meaning

there are some odd things going on which we do not understand like a control value in the red area in the control chart

R-Chart: N_{NH4}

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Summary

Always communicate to client the control chart

Tell client results is ± 2 s_{Rw}

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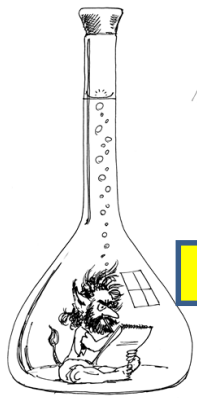
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Thanks for listening – more info
www.trollboken.se

HOME ABOUT ANALYTICAL METHOD RESOURCES CASE STUDIES CONTACT SEA

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

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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