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

Nordtest Trollbook 569

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The QC (internal quality control) is important for a laboratory to know and show the quality of the results.
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 \( \pm 2 \, s_{Rw} \)
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
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
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 ....
Prediction based on results month 1. Lab results month 2 does not fit with predicted!

Ni in FCC catalyst from refinery X

- Ni mg/kg vs Months
- Graph shows linear increase over months.
Lab results month 2 and 3 does not fit with predicted – each month extra analysis

Ni in FCC catalyst from refinery X

![Ni concentration over months graph](image-url)
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...
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**
Show the client the control chart

X-Chart: Zn

Client trust all figures: Solution
A reported value $x$ could be in the range

$$x \pm 2s_{R_w}$$
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 $\pm 2 \ s_{RW}$

Based on requirements
Setting target control limits

The standard deviation needed for the control chart, $s_{\text{target}}$, is estimated from the requirement on $s_{Rw}$ (the within-laboratory Reproducibility)

- 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.
Target control limits based on requirements

Examples

• LOQ
• Measurement System Analysis – MSA
• EU directives
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
Target control limit
Target s from tolerance

MSA – Measurement System Analysis

- $s_{Rw} < 1.7\%$ of tolerance/specification
- $s_{Rw} < 5\%$ is acceptable
Target control limits
Target s from directives

• EC drinking water directive 98/83/EC
  at a specific concentration
• Within-laboratory reproducibility, $2 \cdot s_{Rw} < 10\%$

• 2009/90/EC pointing to the
Water Framework Directive 2000/60/EC
at a specific concentration
• LOQ < 30 %
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 $\pm 2 s_{Rw}$

Based on requirements

Propose 3s and 2s rules and yearly review

Use the control limits NOT the actual $s$ measured
Trollboken AB

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 previous* control values is also between warning and action limits
Is this analytical method in control?

Yes – all values within warning limits but we need to do a yearly review of the chart.
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 $\pm 2 \, s_{Rw}$

Based on requirements

Propose 3s and 2s rules and yearly review

Use the control limits NOT the actual $s$ measured
Ladder of errors

$u_{Rw}$

Reproducibility within laboratory

$u(bias)$

Bias

$u_c$

Measurement Uncertainty
Control chart limits can give $s_{Rw} = u_{Rw}$ for samples covering the whole analytical process.

The control limits can be set wider - target control limits. Important to use the actual control limits for $s_{Rw}$.
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
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 $\pm 2 s_{Rw}$.
- Based on requirements.
- Propose 3s and 2s rules and yearly review.
- Use the control limits NOT the actual $s$ measured.
Thanks for listening – more info
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Analytical method

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 \( s(R_w) \)
- Trueness - expressed as a requirement on maximum bias
- Ruggedness
- Measurement uncertainty