Setting Data Requirements

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Outline

1. Collection and Processing of data without manual transcription
2. Setting Data Requirements
3. Data required to estimate and report the measurement uncertainty
4. Example of setting and managing data requirements
5. Structuring data
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1. Collection and Processing of data without manual transcription

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- Manual correction of the data can also affect validity of the data
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Note	**The use of commercial software can make the data management fully automatic but is expensive, makes laboratory dependent of paid services and, some times, requires the laboratory to change their practice to fulfil the structure of data collection and control.**

2. Setting Data Requirements

- The data collected (obviously) must be sufficient to meet the intended purpose of the measurement, but needs to be defined before starting the measurement
- As an example, we consider the data required to meet the specified target uncertainty

3. Data required to estimate and report the measurement uncertainty

- The bottom-up evaluations of the measurement uncertainty (MU) require more data during the validation of the measurement procedure and during the routine analysis
- The top-down evaluations of the MU require less data but the analytical range is frequently divided in various intervals of the studied quantity associated with different MU models (e.g. Interval I: $< 2q_{LOQ}$; Interval II: $\geq 2q_{LOQ}$)
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- The number of replicate measurements is relevant for the evaluation of the MU
- If replicates do not meet the quality control criteria, that information must be reported (ISO 5725-6)
- If measurements are close to the admissible limit, the measurement uncertainty must be reported (ISO/IEC 17025)

3. Data required to estimate and report the measurement uncertainty

- (…)

»	**Before reporting the result, it should be checked if the MU is fit for the intended use; i.e. if it is smaller than a target MU.**

Note 1	**Measurement results are only fit for the intended use if:** **- Are traceable to an adequate reference** **- MU is small enough of the intended use**
Note 2	**A measurement procedure is valid if:** **- It is applicable to an adequate scope (i.e. list of matrices and interval of quantities)** **- Frequently produces results with a fit for the intended use MU**

4. Example of setting and managing data requirements

Determination of nitrate in drinking waters by ion chromatography	Bottom-up evaluation of the MU

Measurand: mass concentration of nitrate in a water sample

Measurement traceability: Results are traceable to the value of the reference used to prepare the calibrators of the ion chromatograph

Target expanded MU: 7.5 mg L^{-1} (latest amendment to Council Directive 98/83/EC)

Measurement model:

$$\gamma = \gamma_{\mathrm{d}} F_{\mathrm{d}} f_{\mathrm{Std}}$$

γ – mass concentration of nitrate in the sample
γ_{d} – mass concentration of the diluted sample
F_{d} – dilution factor
f_{Std} – unitary factor for calibrators

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Measurement uncertainty model:

$$U(\gamma) = ku(\gamma) = k\,\gamma\sqrt{\left(\frac{\frac{s_y}{b}\sqrt{\frac{1}{m}+\frac{1}{n}+\frac{(\bar{I}_{\mathrm{d}}-\bar{I})^2}{b^2\,\Sigma(\gamma_i-\bar{\gamma})^2}}}{\gamma_{\mathrm{d}}}\right)^2 + \left(\frac{u(f_{\mathrm{std}})}{f_{\mathrm{std}}}\right)^2 + \left(\frac{u(F_{\mathrm{d}})}{F_{\mathrm{d}}}\right)^2}$$

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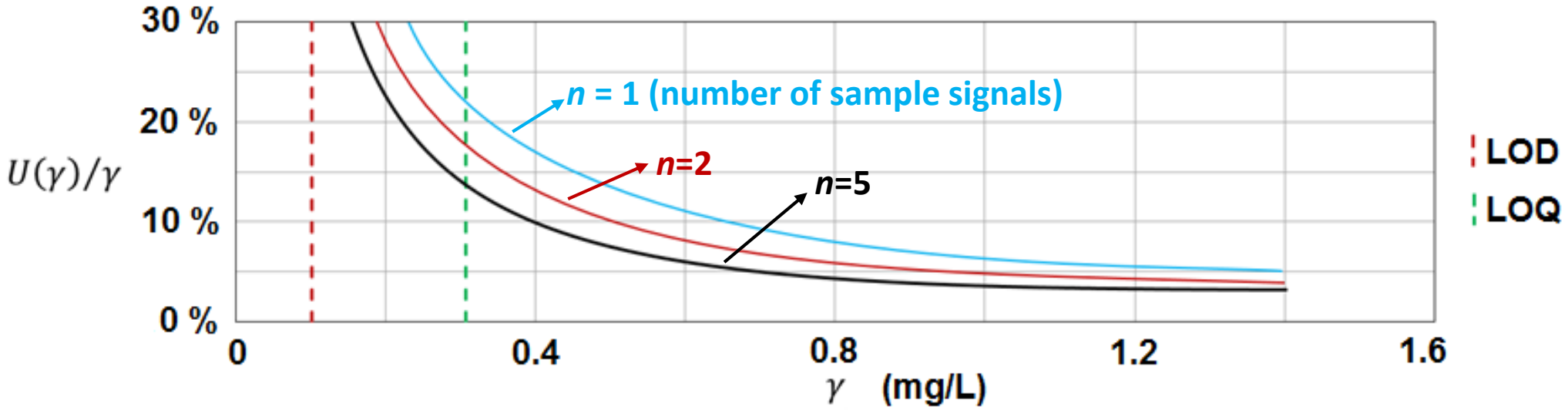

4. Example of setting and managing data requirements

Determination of iron in drinking waters by ICP-OES	Top-down evaluation of the MU

Measurand: mass concentration of total iron in a water sample

Measurement traceability: Results are traceable to the value of the reference material used in trueness (recovery) tests.

Target expanded MU: 60 µg L^{-1} (latest amendment to Council Directive 98/83/EC)

Measurement model:

$$\gamma = \frac{\gamma_{\mathrm{d}} F_{\mathrm{d}}}{\bar{R}}$$

γ – mass concentration of iron in the sample
γ_{d} – mass concentration of the diluted sample
F_{d} – dilution factor
$\bar{R}$ – mean recovery

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$\bar{R}$ – mean recovery

Measurement uncertainty model:

Interval II: ≥ $2\gamma_{\mathrm{LOQ}} = 20$ µg L^{-1}

$$U(\gamma) = 2\gamma u'(\gamma) = 2\gamma \sqrt{s'^2_{\mathrm{I}} - s'^2_{\mathrm{r}} \left(1 - \frac{1}{n}\right) + \sum_{i=1}^{N} R_i^2 \left[\left(s'_{\mathrm{I}}(\gamma_i)\right)^2 + \left(u'(\gamma_{\mathrm{Refi}})\right)^2\right] \Big/ \bar{R}^2 N}$$

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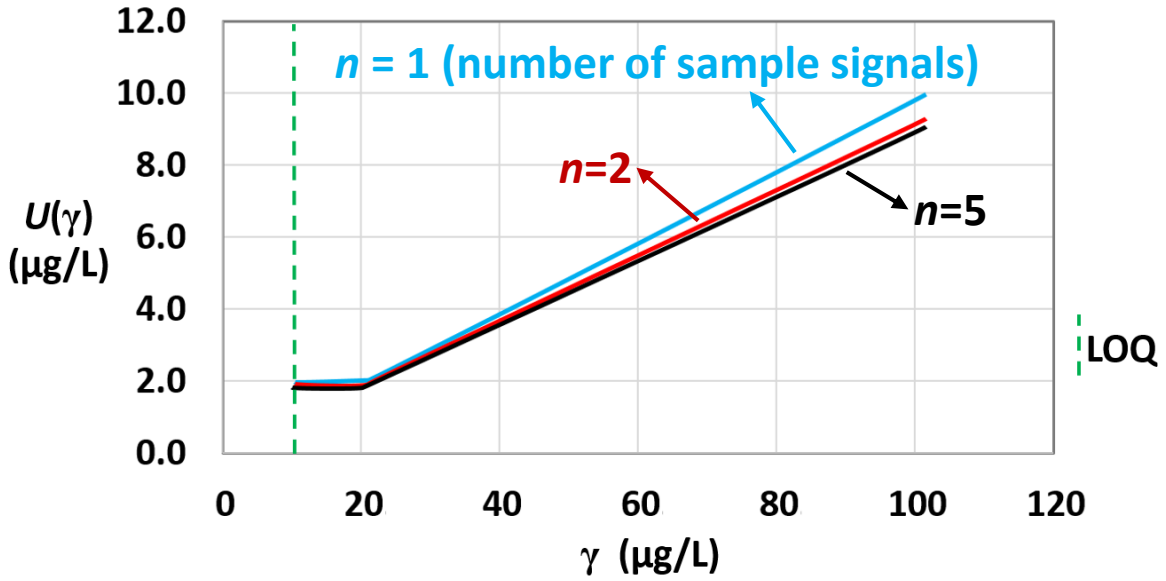
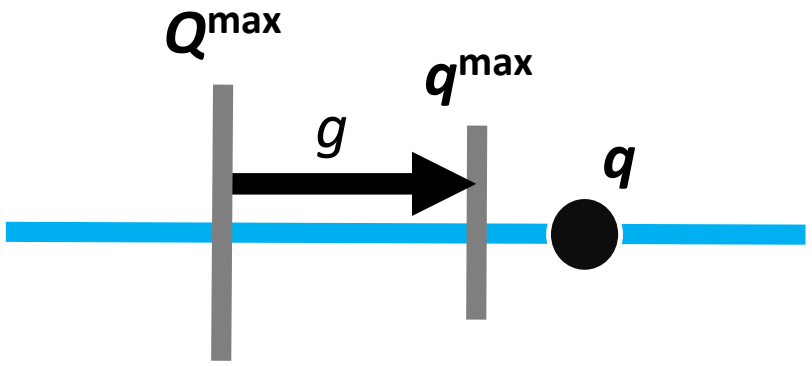


5. Structuring data| 5.1. Data from previous measurements to assess the need to revise measurement requirements

- If the reported MU frequently produces risks of wrong compliance decisions close to the maximum admissible value of the risk (e.g. 5 %), it can be decided to reduce the target MU.

Example:



Q^{max} – Maximum admissible quantity;
g – guard band for 5 % risk of wrong rejections;
$q^{max} = Q^{max} + g$: Limit value for the measured quantity, q.

The q indicates the product is not compliance with a risk of wrong rejections of 4.3 %.

5. Structuring data| 5.2. Data from different laboratories to conclude about the feasibility of achieving a specific performance

- If duplicate results obtained under intermediate precision conditions (i.e. in different days of the same laboratory) and proficiency test results of various laboratories are available, it is possible to pool that information and assess if the currently used procedures can achieve a defined target MU.

Lab.	Rep.1 (Day X)	Rep.2 (Day. Y)
1	155.1	164.4
2	179.0	180.6
3	149.8	142.8
4	143.5	148.9
5	158.8	135.4
6	122.8	119.1
7	148.8	124.3
8	124.0	128.7
9	172.5	184.9
10	150.2	144.6
11	159.6	168.2
12	147.5	149.1
13	178.2	178.7
14	143.5	139.6
	s'_I(Polled):	6.6%

Proficiency test results:			
Lab.	Q	$u(Q)$	q
A	170.88	3.42	178.4
B	160.84	3.22	185.0
C	125.93	2.52	97.20
D	133.74	2.67	138.7
E	186.16	3.72	187.0
F	125.92	2.52	118.8
G	176.88	3.54	166.3
H	121.95	2.44	108.6
I	128.62	2.57	128.5
J	131.96	2.64	126.2
K	168.42	3.37	179.9

u'_I(Pooled):	2.2%	
R(mean):	98.2%	(R(mean) equivalent to 100 %)

U':	14%

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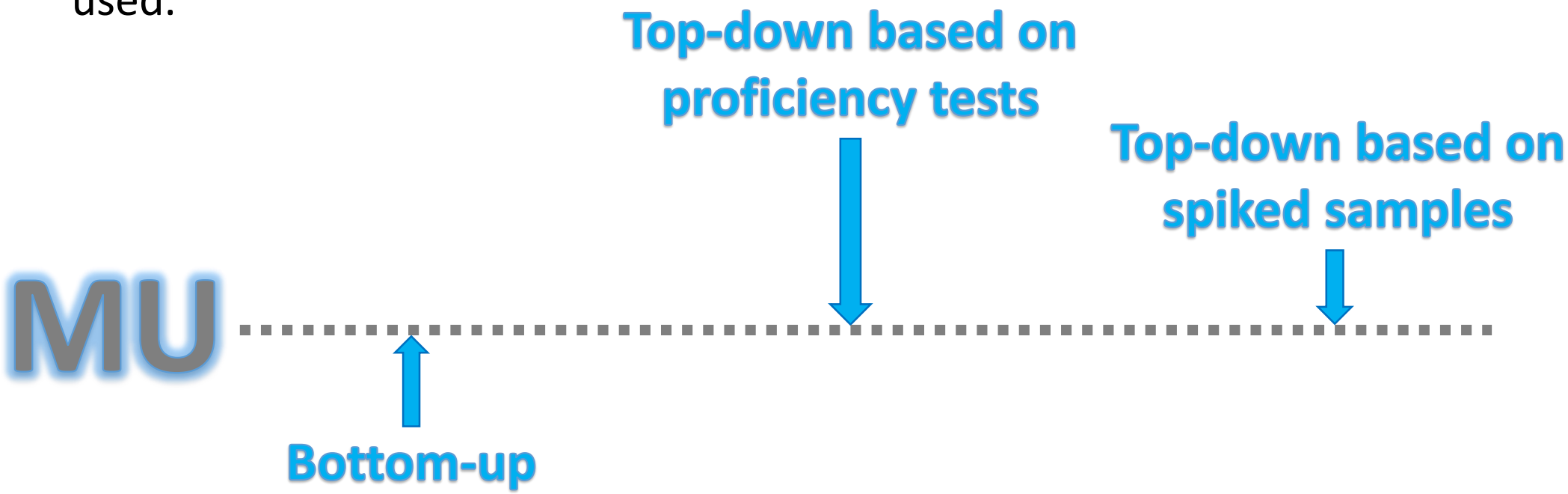
U': 14%

Example:

If the legislated permissible limits and/or target performance parameters are to be changed, it is useful to understand if the community has the technical capacity to achieve the required performance.

5. Structuring data| 5.3. Data from different approaches of the evaluation of the MU

- If models or estimates of the MU using different approaches are made available, it can be used to decide which strategy for the evaluation of the MU should be used.



Final Remarks

- The automation of data processing reduces the risk of being reported inaccurate results but the flexibility of the automation is crucial
- The data that needs to be daily managed in routine analysis depends on the complexity of the measurement and used measurement uncertainty model
- Measurements uncertainty must be smaller than a target value
- The structuring of data produced within or between laboratories can be used to update measurement requirements.