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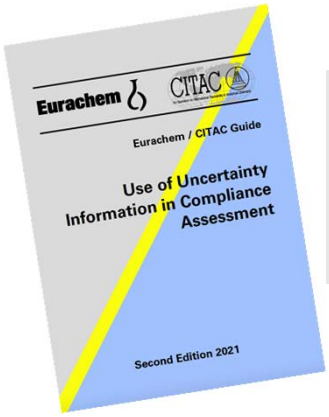
Eurachem Guidance on compliance assessment

B. Magnusson and A. Williams



Workshop 2021
Trends and Challenges in Ensuring Quality in Analytical Measurements

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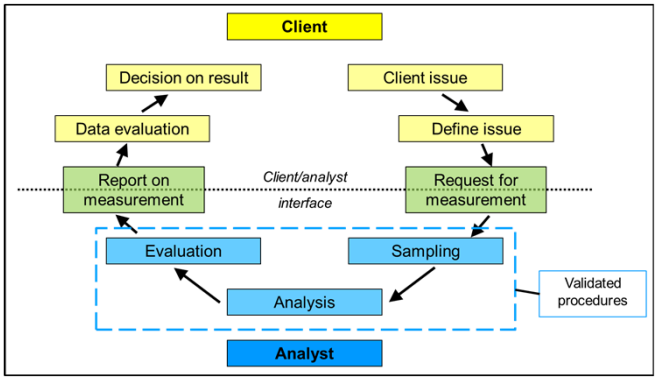
The measurement cycle

Eurachem's Focus is measurement quality...

... from client issue to decision on results



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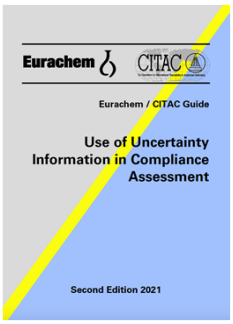
Use of uncertainty information in compliance assessment

Second edition (2021)

The issue here is whether a **measurement result complies with limits** e.g. specifications, regulatory or legal limits.



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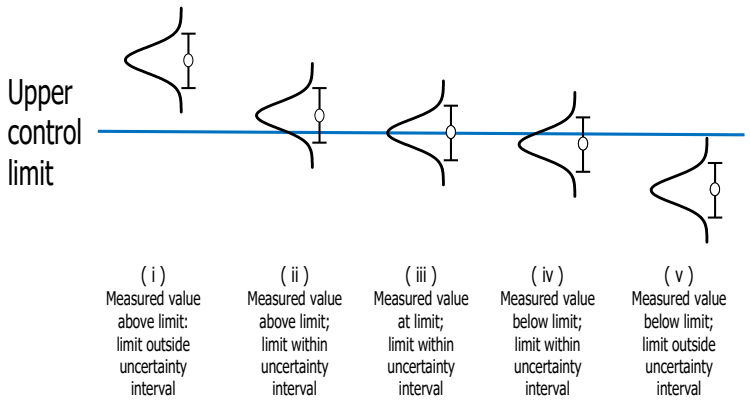
Five differens cases – compliant?



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Conformity or compliance?

ISO uses “conformity assessment”; This can, however, include a broad range of activities, from product testing to inspection and licensing.

This Guide is concerned with whether a **measurement result complies with limits**, e.g. specifications, regulatory or legal limits – term used is **“compliance assessment”.**

In ISO/IEC 17025, compliance of a measurement result with stated limits is often used as the basis for a “statement of conformity”.

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Compliance assessment(1)

- **Consumer products** – can this toy be used by children below 3 years of age?
- **Industry** – in the batch of stainless steel delivered, is the mass fraction of nickel between 16 and 18 %?

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Compliance assessment (2)

- **Forensic** – the driver has a measured alcohol level of 0.052 mg/100 ml; can one be confident that the alcohol content in the blood is over the limit of 0.05 mg/100 ml?
- **Food** – are there are any pesticides present above the maximum residue limit in this batch of shrimps?

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Compliance in production (3)

Case not covered by the Guide

- **Manufacturing** – do the QC results obtained today show that the production process is under control?

In this case the uncertainty is NOT used!

What shall we use instead of uncertainty here to be able to answer the question if the results are within stated limits?

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Terminology

***decision rule*: rule that describes how measurement uncertainty is accounted for when stating compliance**

***specification limit (tolerance limit)*: upper or lower bound of *permissible values* of a property**

acceptance limit: specified upper or lower bound of *permissible measured values*

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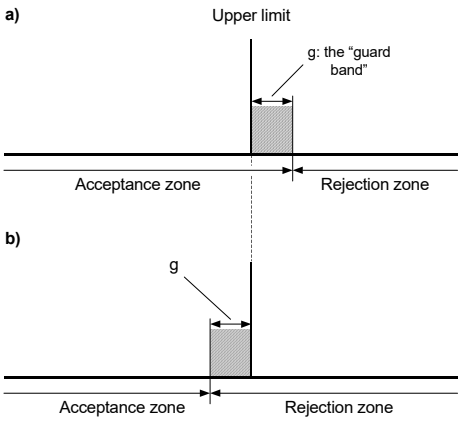
Terminology

guard band: interval between a specification limit and a corresponding acceptance limit



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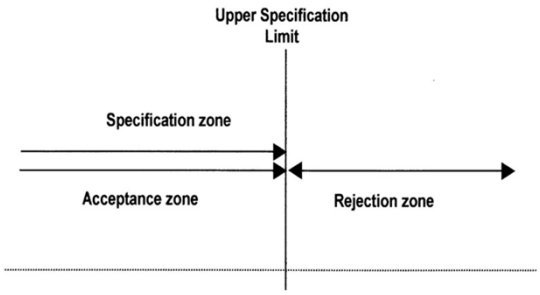
Terminology

simple acceptance: a decision rule in which the acceptance limit is the same as the specification limit



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Example simple acceptance batch of steel (1)

- The specification zone is from 16.0 % Ni to 18.0 % Ni.
- The measured value is 16.1 % Ni
- Simple acceptance – the risk is shared
  Batch is compliant
  (but with another decision rule …)

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Information needed to make an assessment of compliance

- a measurand clearly specified;
- a measurement uncertainty for a measured value at the limit(s).
- a permitted upper or lower limit or both;
- a decision rule;
- a measured value:

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Example – batch of steel (2)

Measurand	Mass fraction of nickel, Ni in a batch of steel delivered to a customer.
Uncertainty	The expanded uncertainty (analysis and sampling), U, is 0.2 % Ni, k = 2 (95 %). Standard uncertainty, u = 0.1 % Ni.
Specification	The specification zone is from 16.0 % Ni to 18.0 % Ni.
Decision rule *High confidence of correct acceptance*	*The acceptance zone is the mass fraction interval where it can be decided with a confidence level of not less than 95 % (α=0.05) that the batch has a mass fraction above the lower limit and below the upper limit.*
Distribution	The distribution is assumed to be Normal.
⇩	
Guard band	Guard band is calculated as $1.64u \approx 0.17$ % with k value 1.64 from the one-tailed 95 % upper quantile for the normal distribution.
Acceptance zone	16.2 % Ni to 17.8 % Ni, after rounding to one decimal place.
Measured value	16.1 % Ni

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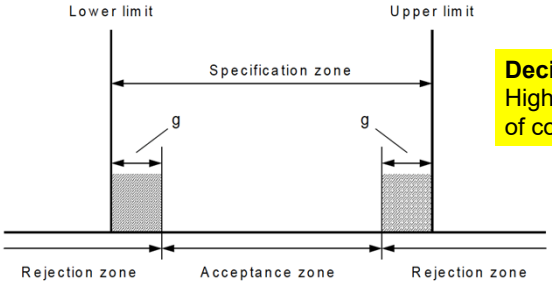
Example – batch of steel (3)



Decision rule
High confidence (>95%) of correct acceptance

Guard bands (g), and acceptance and a rejection zones based on a lower and upper limit and a decision rule stating high confidence of correct acceptance

The measured value, 16.1 % Ni is below the lower acceptance limit of 16.2 %, i.e. it is in the rejection zone.
The batch is non-compliant.

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Decision rule

The key to the assessment of compliance is the concept of “Decision rule”.

A decision rule should have a well-documented method of determining the location of acceptance and rejection zones, ideally including acceptable levels of probability, *P*, that the value of the measurand either

1) lies within the specification limit (correct acceptance)
   or
2) lies outside the specification limit (correct rejection)

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Differerent decision rules

In chapter 4 of the Guide the following decision rules are presented

Decision rules with:

- pass/fail using simple acceptance
- pass/fail using guard band
- conditional or inconclusive results
- specifying a two stage procedure

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Decision rule may also give (1)

- the maximum allowed uncertainty at the limit;
- an assumed distribution, e.g. normal or lognormal
- rules for rounding before assessing compliance;
- the required number of replicate measurements (if any) and the procedure for using replicate results including (for example) whether results should comply individually, or should be averaged before comparison with limits;
- procedures for dealing with outliers;

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Decision rule may also give (2)

- procedures for further action, for a non-binary decision rule, when the decision is conditional (pass/fail);
  - e.g requiring additional measurements;
- recommendations on how to report compliance/non-compliance, e.g. pass/fail, within tolerance/out of tolerance, within specification/out of specification;
- recommendations on how to state the decision rule used in the statement of compliance.

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Use of uncertainty factor (1)

- At very very high uncertainty we get an asymmetric distribution that often can be approximated with a lognormal.
- This results in a non-symmetric acceptance zone e.g.

Assumed distribution	Relative standard uncertainty, u_{rel}	Upper limit	Acceptance limits	
			Correct acceptance	Correct rejection
Normal	0.3	100	51	149
Lognormal	0.3	100	**61**	**164**

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Use of uncertainty factor (2)

This non-symmetric acceptance zone can be calculated using an uncertainty factor.

$$^{F}U \approx \exp(ku_{\mathrm{rel}})$$

k is the coverage factor & u_{rel} is the standard uncertainty

For correct rejection with a standard uncertainty of 0.3 (30 %) and an upper limit of L = 100 the **upper** acceptance limit is then:

$$L \times {}^{F}U = 100 \times 1.64 = 164$$

For the **lower** you divide by the uncertainty factor

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Example with very very high U (1)

Measurand	Mass fraction of a banned substance in a sample.
Uncertainty	The relative standard uncertainty u_{rel} is 35 %.
Specification	Upper permitted limit L_u is 2 ng/g.
Decision rule *High confidence of correct rejection*	*The concentration of the banned substance will be deemed to be above the limit if the probability of the concentration being greater than the limit is ≥ 95 %.*
Distribution	The distribution is assumed to be lognormal.
⇩	
Guard band	*k* is 1.64, giving $^{F}U \approx \exp(1.64u_{\mathrm{rel}}) = 1.78$. $g = L_u \times {}^{F}U - L_u = 1.6$ ng/g.
Acceptance limit	3.6 ng/g
Measured value	3.3 ng/g

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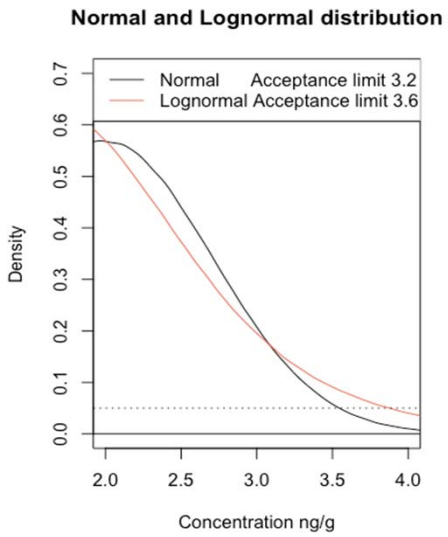
Example with very very high U (2)

Measured value 3.3 ng/g.
Result compliant assuming a lognormal distribution

Figure to the right shows:
Right side of probability distributions for a limit value of 2 ng/g with a relative standard uncertainty of 35 %.
Horizontal line at density 0.05 to aid visual comparison.



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Other issues in the Guide

- Producer and consumer risks
- Decision rules that provide for conditional or inconclusive results are introduced
  (sometimes called “non-binary” decision rules);
- Specific or Global Risk

References to current guidance from e.g. ILAC and JCGM

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Summary (1) – key is decision rule

***decision rule*: rule that describes how measurement uncertainty is accounted for when stating compliance**

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Summary (2) – Use of guard band

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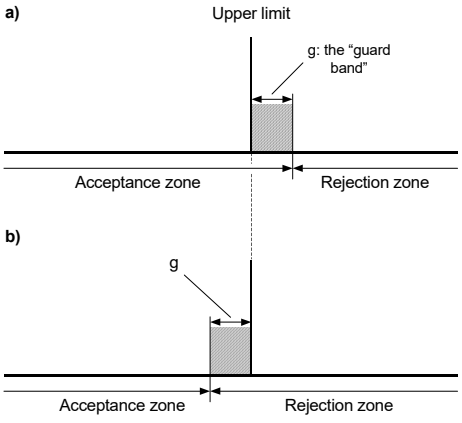

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Summary (3) – Differerent Decision rules

In chapter 4 of the Guide the following decision rules are presented

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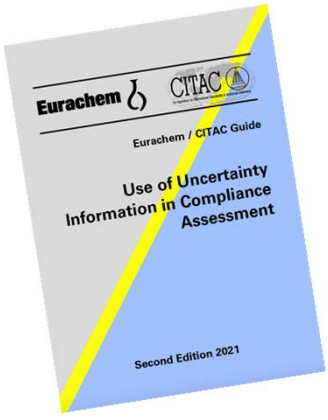

THANKS FOR LISTENING

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Breakout session – compliance assessment

- We discussed several examples
  - Drinking water, animal feed, - used simple acceptance and a max *U*
  - Petrochemical products – used a 2 stage if first non compliant. If second within limits – product compliant
  - Pharmacopea – also simple acceptance but here specification are set using guard band base on a “normal” U in the labs
  - QC – using decision rules similar to Nordtest 569 (QC)ased on
  - Calibration spec based on precision and bias(distanct from target value) – also used simple acceptance
  - PT provider assessing compliance statement of the lab
  - Medicine – assement of compliance us
  - CODEX acceptance sampling

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Example of

- Compliance of the QC sample
- Piotr – assessing compliance statement in PT
- Ref stand – prod of ref standard –used for compliance assessment for quality of medicine; target U, assigned value with low U
- Calibration certificate verify conformity with a spec on precison and trueness (inaccuracy)
- Food classifaction – CODEX metod of compliance

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Example of

- Drinking water quality ubio EU dire***
- Animal feed sampling – trace elements
  - Legislation U 25 % EU ***
- Petrochemical use 2 approaches if not compliant make a new measurement
- Pharm – the uncertainty “is” included in the speciations using “normal” uncertainty
- Limit is 20
  - Value of 19.92 non C
  - V alue of 19.97 is rounded 20.0