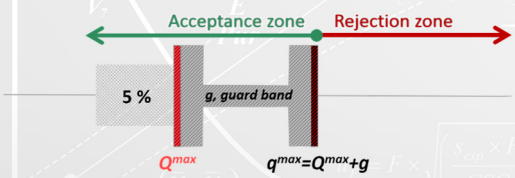
Critical Issues of the Accreditation Standards
Nicosia, 21-22 February 2019

The use of a decision rule



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22 Feb. 2019

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Outline

1. **Measurement goal**
2. **Compliance assessment**
3. **Measurement uncertainty concept**
4. **Compliance assessment rule**
5. **Sampling uncertainty in compliance assessment**
6. **Setting and using the guard band**
7. **Examples**
8. **References**

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1. Measurement goal

Most measurements in chemistry are performed to assess the compliance of products with a legislation or specification, or in fundamental and applied research.

Measurement uncertainty is relevant for measurement interpretation:

» Compliance with a specification or legislation limit or interval;

» Assessment of trends or differences in studies items.

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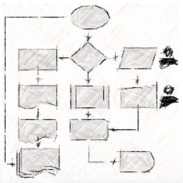
2. Compliance assessment

Most socioeconomic relevant compliance assessments are performed by accredited laboratories.

In some regulations, rules for how to deal with measurement uncertainty in compliance assessment are stablished: **The Decision Rule**.



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2. Compliance assessment

The Decision Rule.

Case A: Medicines control using Pharmacopeia reference substances and measurement procedures [1] – Comparison of the measured quantity value with the specification interval does not takes measurement uncertainty into account;

1. EDQM, European Pharmacopoeia, 9th edition, 2018.

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2. Compliance assessment

Case B: European monitoring of pesticide residues in foodstuffs: Only if the measured quantity value, q, minus the expanded uncertainty, U, is above the Maximum Residues Level, Q^{max} (q-$U > Q^{max}$), product is not compliant [2]. (…)

E12 (..) Thus, the sample is considered non-compliant if x-U > MRL. (..)

2. SANTE/11813/2017, Method Validation & Quality Control Procedures for Pesticide Residues Analysis in Food & Feed, 2017.

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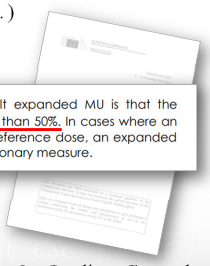
However: $(U/q) \leq 50$ %:

E10 (..) A prerequisite for the use of the 50% default expanded MU is that the laboratory must demonstrate that its own expanded MU is less than 50%. In cases where an exceedance of an MRL is also an exceedance of the acute reference dose, an expanded MU with a lower confidence level can be applied as a precautionary measure.



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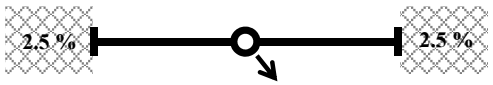
3. Measurement uncertainty concept

If measurement expanded uncertainty is accurately estimated:



Measured quantity value, q

(U – expanded uncertainty for 95 % confidence level)

According to the estimated result, the true value of the measurand should be within ($q \pm U$) for 95 % confidence level.

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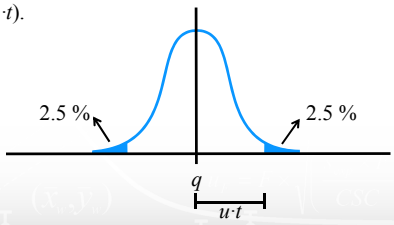
3. Measurement uncertainty concept

(…)

$(q \pm U)$

The expanded uncertainty, U, is estimated by taking the standard uncertainty, u, and the two tailed Student-t, t, for 95 % confidence level ($U = u \cdot t$).



t = T.INV.2T(P,d.f.) = T.INV.2T(0.05,50)

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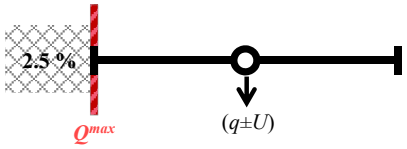
3. Measurement uncertainty concept

If the lowest limit of result interval (q - U) is equivalent to the maximum permissible value (Q^{max}) ($q-U = Q^{max}$):



The probability of the "true" value of the measurand being below or above the Q^{max} is 2.5 % and 97.5 %, respectively.

Decision rule:
if $(q-U) > Q^{max}$ → product is not compliant;
if $(q-U) \leq Q^{max}$ → product is compliant.

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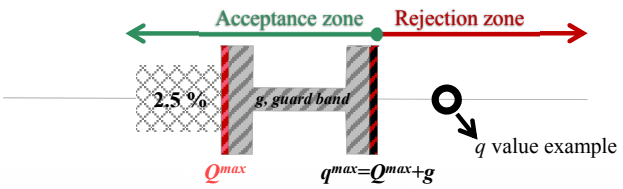
4. Compliance assessment rule

If the 97.5 % confidence level is considered adequate, it can be defined a 'guard band', g, that if added to Q^{max} stablishes a limit value, q^{max}, for the measured quantity value, q, :



In this case, g is the measurement standard uncertainty, u, times the two tailed Student's t for 95 % confidence level, t ($g = t \cdot u$).

In this example, there is a 2.5 % chance of rejecting compliant items.

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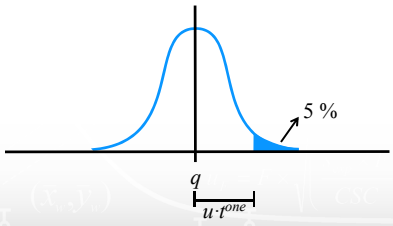
4. Compliance assessment rule

If the reference probability for compliance assessment is 95 %, the guard band should be smaller:

$$g = u \cdot t^{one}$$

where t^{one} is the one-tailed Student-t for 95 % confidence level.



t^{one} = (-1)*T.INV(P, d.f.) = (-1)*T.INV(0.05*2, 50)

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4. Compliance assessment rule

Student's t distribution values:
t – two tailed Student's t distribution values;
t^{one} – one tailed Student's t distribution values.

Conf. Value	P (Type I error)	t (T.INV.2T(P,50)) §	t^{one} (-1)*(T.INV(P,50)) §
90%	0.1	1.676	1.299
95%	0.05	2.009	1.676
97.5%	0.025	2.311	2.009
99%	0.01	2.678	2.403
99.5%	0.005	2.937	2.678

§ - MS-Excel formula

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5. Sampling uncertainty in compliance assessment

- Usually, compliance with a legislation does not take sampling uncertainty into account, but sampling should be performed following a regulated procedure;
- In the industry, the sampling uncertainty can be considered in compliance assessment to guarantee that any portion of the lot is compliant. The determination of sampling uncertainty involves assessing population heterogeneity.

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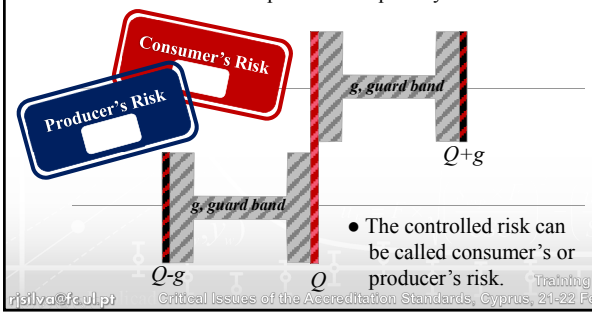
6. Setting and using the guard band

● Depending of the interest to be first protected and the type of permissible quantity value (Maximum, Q^{max}, minimum, Q^{min}, or an interval, [Q^{min} , Q^{max}], of permissible quantities), the guard band is added or subtracted to the permissible quantity.



● The controlled risk can be called consumer's or producer's risk.

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7. Examples

EXAMPLE 1:

Gold mass fraction in a gold alloy must be larger than 800 ‰, the probability of accepting alloys with a value below 800 ‰ should not be larger than 5 % and measurements standard uncertainty is 2.3 ‰.

Which decision rule should be adopted?

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7. Examples

EXAMPLE 2:

The mass concentration of procimidone in green wine imported in the USA must be below 0.50 mg L^{-1}, the probability of accepting a product not meeting specification should not be larger than 1 % and measurement expanded uncertainty§ is 0.053 mg L^{-1} for a coverage factor of 2.01.

Which decision rule should be adopted?

§ - Measurement uncertainty takes sampling component into account.

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7. Examples

EXAMPLE 3:

The mass concentration of procimidone in green wine imported in the USA ~~must~~ should be below 0.50 mg L^{-1}, the probability of ~~accepting~~ rejecting a product ~~not~~ meeting specification should not be larger than 1 % and measurement expanded uncertainty§ is 0.053 mg L^{-1} for a coverage factor of 2.01.

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7. Examples

EXAMPLE 4:

The mass fraction of copper in coins should be between 65 % -75 % with a 5 % probability of accepting coins outside this specification. Measurements of copper in coins are associated with a standard uncertainty of 0.22 %.

Which decision rule should be adopted?

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7. Examples

EXAMPLE 5:

The mass concentration of procimidone in a formulation is 1 g L^{-1}, formulation specification is 0.9 g L^{-1} - 1.1 g L^{-1}, the probability of rejecting a complaint formulation should not be larger than 5 %, and measurement standard uncertainty is 0.012 g L^{-1}.

Which decision rule should be adopted?

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8. References

1. S. Ellison, A. Williams (Eds), Eurachem/CITAC Guide - Use of uncertainty information in compliance assessment, Eurachem, 2007 (www.eurachem.org).
2. JCGM 106:2012, Evaluation of measurement data – The role of measurement uncertainty in conformity assessment, BIPM, 2012 (www.bipm.org).

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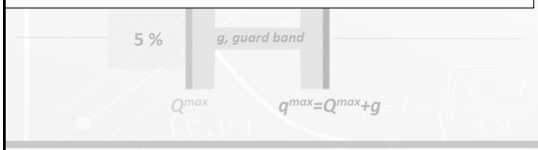
Solutions of the Examples



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7. Examples

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Gold mass fraction in a gold alloy must be larger than 800 ‰, the probability of accepting alloys with a value below 800 ‰ should not be larger than 5 % and measurements standard uncertainty is 2.3 ‰.

Which decision rule should be adopted?

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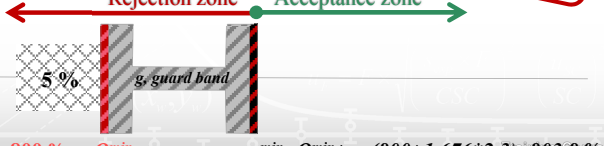
Decision Rule:

Consumer's Risk 5 %



800 ‰ = Q^{min}

$q^{min}=Q^{min}+g=(800+1.676*2.3)=803.8$ ‰

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7. Examples

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§ - Measurement uncertainty takes sampling component into account.

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Which decision rule should be adopted?

Consumer's Risk 1 %

Decision rule: Acceptance zone | Rejection zone

q^{max} = $= Q^{max}-g$ = $= (0.5-2.403*(0.053/2.01))$ = $= 0.437$ mg L^{-1}

$Q^{max}-g=0.437$ mg $L^{-1}= q^{max}$ $Q^{max}=0.5$ mg L^{-1}

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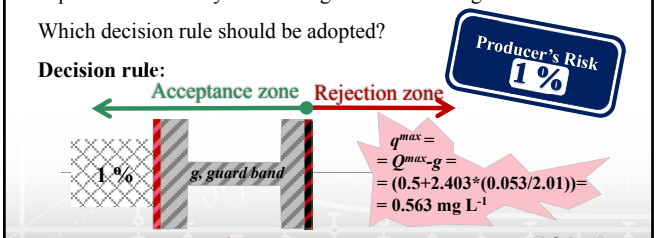

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Dec. Rule:

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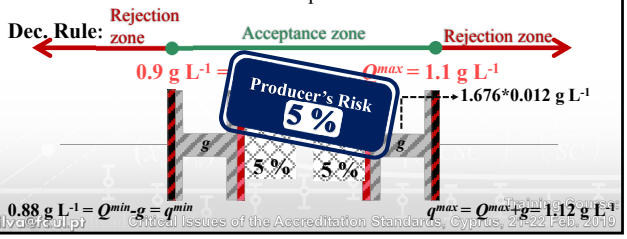
Which decision rule should be adopted?



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