Measurement Uncertainty in Chemistry and Microbiological Analyses: Implications for Conformity Assessment of Live Bivalves

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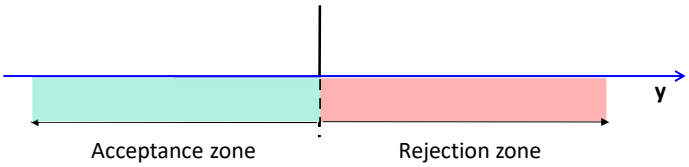
Conformity assessment

it is necessary to define:

- **Acceptance Zone**– A set of values of a characteristic, for a specific process and decision rule, that determine the conformity/acceptance of the product when the measurement result falls within this zone.
- **Rejection zone –** a set of values of a characteristic, for a specific process and decision rule, that determine the non-conformity/rejection of the product when the measurement result falls within this zone.



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Decision Rule

"It is the documented rule that describes how measurement uncertainty is considered when declaring conformity to a specified requirement.."

it is necessary to define:

- Measurand (Y);
- Analytical result (y);
- Uncertainty; for an expanded uncertainty (U(y)), present factor k and corresponding confidence level (e.g., k = 2, for 95% confidence);
- Specification that establishes the limit(s) (e.g., upper limit T_U)

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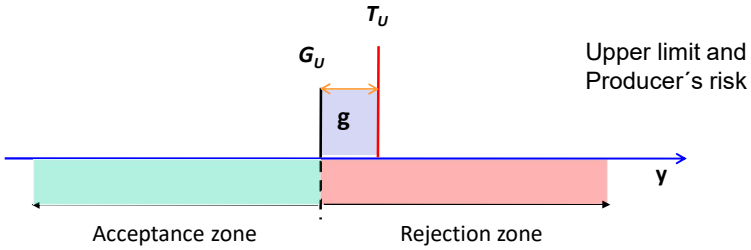
Measurement uncertainty and acceptance limits

Guard band or safety band (g): the interval between the tolerance/specification limit (T_U) and the acceptance limit (G_U). Using this interval can reduce the likelihood of an incorrect decision when we need to verify whether or not the item conforms to the requirements.



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Our laboratory – Metals in seafood

- ✓Determinations of Cd, Hg_T and Pb;
- ✓Accredited by ISO 17025:2017;
- ✓ Regulation with upper limits;
- ✓Measurement uncertainty;
- ✓Reports with [Metal] $\pm U_{exp}$
- ✓ To date, we have not included conformity assessment in the reports.

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Upper Limits - Commission Regulations:

(EU) 2023/915 and (EU) 2025/1891

Element	Maximum limit level for bivalves molluscs (mg/kg)
Inorganic arsenic (iAs)	0.50
Mercury (Hg)	0.50
Lead (Pb)	1.50
Cadmium (Cd)	1.0




Decision rule – (Concentration – U_{exp}) > Upper limit

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Quantification of uncertainty

Metals – atomic absorption spectrometry – in house validation

Precision component	Recovery component
Use of duplicate metal readings in multiple bivalve samples	Use of CRM (DORM5, DOLT5, SQID1 – NRC Canada)
$S'_{precision} = \frac{\bar{A}'}{1.128}$	$u(\bar{R}_m) = \bar{R}_m \times \sqrt{\left(\frac{s^2_{obs}}{n \times C^2_{obs}}\right) + \left(\frac{u(C_{MRC})}{C_{MRC}}\right)^2}$
$u_c = \sqrt{u^2_{precision} + u^2_{Rm}}$	
$U_{exp} = ku_c$ (k = 2; 95%)	

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Microbiological criteria

Regulation (EC) 2073/2004

Live bivalve molluscs – *Escherichia coli (E. coli)*

Parameter	Limit for direct human consumption (tested by most probable number – MPN – technique, expressed per 100g)
E. coli	230 ≈ 2.36 (log10)

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Estimation of measurement uncertainty for Quantitative Food Microbiology (ISO 19036, 2019)

Applicable to all quantitative microbiological analyses of the food chain, including MPN based methods

Considers 3 types of uncertainty components: i) technical uncertainty, ii) matrix uncertainty, and iii) distributional uncertainty

- A component is negligible if its standard uncertainty is equal or below 1/5 of the magnitude of the largest component standard uncertainty

With measurand: *E. coli (by* MPN)

MPN methods: $u_c(y) = \sqrt{u_{tech}^2 + u_{matrix}^2 + u_{MPN}^2}$

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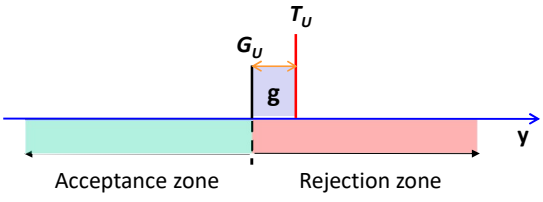
Cadmium

The legal maximum limit (T_U) of Cd allowed in bivalves is 1.0 mg/kg. The expanded uncertainty (U) of this measurement is 10 % (0.1 mg/kg). What is the upper acceptance limit (G_U), considering a maximum risk of false rejection of 5%?



Knowing that :

$g = t \times u_c$ (t = 1.64; 95%)

$u_c = U/k$ (k = 2; 95%) *u_c: Combined uncertainty, t: t-student*

Then:

$g = 1.64 \times u_c = 1.64 \times 0.05 = 0.082$

$G_U = T_U - g = 1.0 - 0.082 =$ 0.9 mg/kg

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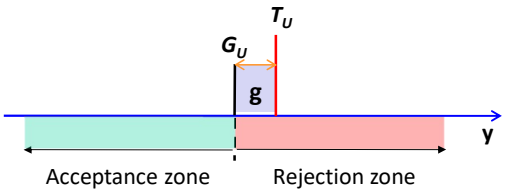
Escherichia coli

The legal maximum limit (T_U) of E. coli allowed in a sample unit of bivalves is 230 MPN/100g ≈ 2.36 ($\log_{10}$) MPN/100g. The expanded uncertainty (U) of this measurement is 0.9 ($\log_{10}$) in our laboratory. What is the upper acceptance limit (G_U), considering a maximum risk of false rejection of 5%?



Knowing that :

$g = t \times u_c$ (t = 1.64; 95%)

$u_c = U/k$ (k = 2; 95%)

u_c: Combined uncertainty, t: t-student

Then:

$g = 1.64 \times u_c = 1.64 \times 0.434 \log_{10} = 0.711 \log_{10}$

$G_U = T_U - g = 2.36 \log_{10} - 0.711 \log_{10} = 1.649 \log_{10} = 10^{1.649} = 45$ NMP/100g

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Final Considerations

- ✓Measurement uncertainty must be explicitly considered in conformity assessment to support transparent and scientifically sound compliance decisions.
- ✓The application of decision rules with guard bands reduces the probability of false rejection and improves confidence in analytical result interpretation.
- ✓The impact of measurement uncertainty is particularly relevant in microbiological analyses, where higher uncertainty may significantly reduce the acceptance zone.
- ✓Clear definition of decision rules, including risk level and acceptance limits, is essential for harmonised interpretation of regulatory limits.

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