Implementing ISO11929 at our laboratories

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Why ISO 11929?

Refreshing decision threshold and detection limit (Currie approach)

What is ISO 11929 all about?
  - Characteristic limits
  - Limits of the confidence interval

How should we report our results according to ISO 11929?

How will we implement this in our laboratories
Why implementing ISO11929?

- Many ISO norms involving radioactivity measurements (in use in our labs) are referring to ISO11929 (see list in following slides).
- Authorities (i.e. follow European legislation and guidelines → referring also to ISO11929 so they expect us to work according to this norm).
- Some fundamental reasons:
  - MDA value according to Currie is generally underestimating.
  - Measurements close to zero with large uncertainty include negative activity values (which is physically not possible).
ISO norms related to ISO11929

**RADIOLOGICAL PROTECTION** (not up to date)

ISO norms related to ISO11929

ISO TC 85 SC 3 (NUCLEAR FUEL CYCLE) (not up to date)

• ISO 11483:2005 (Nuclear fuel technology – Preparation of plutonium sources and determination of $^{238}\text{Pu}/^{239}\text{Pu}$ isotope ratio by alpha spectrometry).
ISO norms related tot ISO11929

ISO TC147 SC3 (RADIOACTIVITY) (not up to date)

- ISO 13160:2012 (Water quality – Strontium 90 and strontium 89 – Test methods using liquid scintillation counting or proportional counting).
Decision threshold and detection limit
Currie approach

null distribution
standard deviation: $\sigma_0$

signal distribution for a sample whose content equals the MDA
standard deviation: $\sigma_D$

$0 \quad S_C \quad S_D$

critical value
minimum detectable net signal

$k_\alpha \sigma_0 \quad k_\beta \sigma_D$
• The detection limit $L_{D}$ is the smallest value of the measurand above the decision level for which the wrong assumption that the physical effect is absent does not exceed the specified probability $\beta$

• The quantity $L_{D}$ is used to find out whether a measurement procedure is suitable for the measurement purpose e.g. in comparing with a specified reference value $L_{R}$
A count exceeding $S_C$ is detected
A count below $S_C$ is not detected
But there is a probability $\alpha$ of a false decision that a signal has been detected while there was no signal
FALSE POSITIVE
A count equal to $S_D$ has a probability $\beta$ of deciding that there is no signal while there was:
FALSE NEGATIVE
“The detection limit $S_D$ is the smallest value of the measurand above the decision level for which the wrong assumption that the physical effect is absent does not exceed the specified probability $\beta$”
Decision threshold and detection limit
Currie approach

- General case: $k_{1-\alpha} = 1.645 (\alpha = 0.05)$

$$L_C = 1.645 \sigma_0$$

- Simple counting

$$L_C = 1.645 \sqrt{2B}$$

- Simple peak analysis (gamma spectrometry, $n$ and $m$ number of channels in ROIs)

$$L_C = 1.645 \sqrt{B \left[ 1 + \frac{n}{2m} \right]}$$
Decision threshold and detection limit
Currie approach

\[ \sigma^2(N) = \sigma^2(G) + \sigma^2(B) \]
\[ \sigma^2(N) = \sigma^2(N + B) + \sigma^2(B) \]
\[ \sigma^2(0) = \sigma^2(0 + B) + \sigma^2(B) \]
\[ \sigma^2(0) = 2\sigma^2(B) \]
\[ \sigma(0) = \sqrt{2B} \]
Decision threshold and detection limit
Currie approach

- **general case:**
  \[ L_D = 2.71 + 3.29 \sigma_0 \]

- **Simple counting**
  \[ L_D = 2.71 + 3.29 \sqrt{2B} \]

- **Peak integration**
  \[ L_D = 2.71 + 3.29 \sqrt{B \left[ 1 + \frac{n}{2m} \right]} \]

Lloyd A. Currie: “Limits for Qualitative Detection and Quantitative Determination”
Decision threshold and detection limit
Currie approach

\[ L_D = 2.71 + 3.29 \sigma_0 \]

Counts

\[ MDA = \frac{2.71 + 3.29 \sigma_0}{\varepsilon I t} \]

Activity

But these parameters are not free of uncertainty

Lloyd A. Currie: “Limits for Qualitative Detection and Quantitative Determination”
What is ISO 11929 all about? decision threshold & detection limit

- A procedure for the estimation of characteristic limits:
  - **Decision threshold** $y^*$ and **detection limit** $y^#$

- **Basis(1)** *ISO/IEC Guide 98-3, GUM*
  - $y$ primary measurement result ($y < 0$ no problem)
  - $u(y)$ primary standard uncertainty
  - Model of measurand $Y = G(X_1, X_2, X_3, X_4, ..., X_m)$

- **Basis(2) ** *Bayesian statistics*
  - $\tilde{y}$ true value of measurand ($y \geq 0$; non-negative measurand)
  - $\hat{y}$ best estimate (non negative)
  - $\tilde{u}(\tilde{y})$ standard uncertainty of the true value
  - $u(\hat{y})$ standard uncertainty associated with $\hat{y}$
Decision threshold:

\[ y^* = k_{1-\alpha} \tilde{u}(0) \]

Detection limit:

\[ y^# = y^* + k_{1-\beta} \tilde{u}(y^#) \]

Implicit equation

Standard uncertainty when zero activity

Standard uncertainty when activity = \( y^# \)
What is ISO 11929 all about? decision threshold & detection limit

Bayesian conditional distributions

null distribution

standard deviation: $\sigma_0$

$\tilde{u}(0)$

$k_\alpha \sigma_0$

$k_\beta \sigma_D$

signal distribution for a sample whose content equals the MDA

standard deviation: $\sigma_D$

$\tilde{u}(y^\#)$

0 $S_C$ $S_D$

critical value minimum detectable net signal

Decision threshold Detection Limit
How does this relates to the Currie formula?

- Currie formula (counts) is obtained considering a simplified model (linear model -\( \tilde{u}^2(\tilde{y}) = c_0 + c_1\tilde{y} \))
- Currie formula (activity, MDA) is obtained by assuming uncertainty for the conversion factor \( w \), \( u(w) \).

\[
A = Nw
\]

\[
u(A) = Nw \sqrt{\frac{u(N)^2}{N^2} + \frac{u(w)^2}{w^2}}
\]

This is detection efficiency and some other conversion parameters (mass, volume...).
How does this relates to the Currie formula?

- Decision level \((activity \ or \ activity \ concentration)\)
  \[
  L_C = kw \sqrt{\frac{b}{t_s} + \frac{b}{t_0}}
  \]

- Detection limit \((activity \ or \ activity \ concentration)\)
  \[
  L_d = \frac{2L_C + \frac{k^2w}{t_s}}{1 - k^2u(w)_{rel}^2}
  \]

This is the difference

These simplified formulae are the result of the selection \(k_{(1-\alpha)} = k_{(1-\beta)} = k\) 
\(u^2(y) = c + y\)
How does this relates to the Currie formula?

For small relative uncertainty of $w$ the correction factor is almost equal to 1 (but for large values may be negative!, no detection limit can be computed.)
How does this relate to the Currie formula?

- MDA is not obtained by taking DL (counts) and scale this to activity
  - Uncertainty on the scaling factor is involved, but when this factor is small, MDA is almost DL (activity)

- Pitfall: simplified models following ISO 11929 may still fail to produce acceptable results
  - in low count rate applications (alpha spec ?)
  - $\sqrt{n} + 1$ is to be considered for small $n$
  - If background counts do not follow poisson statistics (e.g. due to other variations involved)
How does this relate to the Currie formula?

Table 2 — Values $k_{1-\alpha}$, $k_{1-\beta}$, $k_{1-(\gamma/2)}$ as a function of the error probabilities $\alpha$ and $\beta$ and of the confidence level $1-\gamma$ (quantiles of normal distribution)

<table>
<thead>
<tr>
<th>Error of probability</th>
<th>Confidence level</th>
<th>$k_{1-\alpha}$</th>
<th>$k_{1-\beta}$</th>
<th>$k_{1-(\gamma/2)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$ or $\beta$</td>
<td>$1-\gamma$</td>
<td>1.000</td>
<td>1.282</td>
<td>1.645</td>
</tr>
<tr>
<td>0.1586</td>
<td>0.682</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1000</td>
<td>0.800</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0500</td>
<td>0.900</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0250</td>
<td>0.950</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0228</td>
<td>0.955</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0100</td>
<td>0.980</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0050</td>
<td>0.990</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0014</td>
<td>0.997</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0010</td>
<td>0.998</td>
<td></td>
<td></td>
<td>3.090</td>
</tr>
</tbody>
</table>
The limits of the confidence interval take into account the fact that the measurand is non-negative although $y$ can be negative.

The limits of the confidence interval $y^\triangledown$ and $y^\triangleright$ define a confidence interval containing the true value $\tilde{y}$ of the measurand with a specified probability taken as $1 - \gamma$. 
What is ISO 11929 all about?

**Best estimate**

Limits of the confidence interval

\[ y \triangleleft = y - k_p u(y) \quad p = \omega \cdot (1 - \frac{\gamma}{2}) \]

\[ y \triangleright = y + k_q u(y) \quad q = 1 - \frac{\omega \gamma}{2} \]

\[ \omega = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \frac{y}{u(y)} \exp \left( - \frac{v^2}{2} \right) dv \]

\[
\frac{y}{u(y)} > 4 \rightarrow \omega \approx 1
\]
What is ISO 11929 all about?

Best estimate

Limits of the confidence interval

Generally the limits of the confidence interval are not symmetrically around $

\gamma$ or $\tilde{y}$

\[
\tilde{y} = y + \frac{u(y) \exp\left(-\frac{y^2}{2 u^2(y)}\right)}{\omega \sqrt{2\pi}}
\]
Examples of confidence limits and best estimate conversion for different u(y)/y values

These are easily computed using predefined functions in Excel
What is ISO 11929 all about?

Best estimate

Limits of the confidence interval

<table>
<thead>
<tr>
<th>γ</th>
<th>Risk of exceeding quoted confidence limits</th>
<th>None</th>
<th>Set by user</th>
</tr>
</thead>
<tbody>
<tr>
<td>ω</td>
<td>Required to calculate p and q</td>
<td>$\phi \left( \frac{\gamma}{u(\gamma)} \right)$</td>
<td>Use norm. s. dist $\left( \frac{\gamma}{u(\gamma)}, \text{true} \right)$</td>
</tr>
<tr>
<td>p</td>
<td>Required to calculate $k_p$</td>
<td>$\omega \left( 1 - \frac{\gamma}{2} \right)$</td>
<td></td>
</tr>
<tr>
<td>q</td>
<td>Required to calculate $k_q$</td>
<td>$1 - \left( \frac{\omega \cdot \gamma}{2} \right)$</td>
<td></td>
</tr>
<tr>
<td>$k_p$</td>
<td>Coverage factor for lower confidence limit</td>
<td>Complex</td>
<td>Use norm. s. inv$(p)$</td>
</tr>
<tr>
<td>$k_q$</td>
<td>Coverage factor for lower confidence limit</td>
<td>Complex</td>
<td>Use norm. s. inv$(q)$</td>
</tr>
<tr>
<td>$\gamma^{\downarrow}$</td>
<td>Lower confidence limit</td>
<td>$k_p \cdot u(\gamma)$</td>
<td></td>
</tr>
<tr>
<td>$\gamma^{\uparrow}$</td>
<td>Upper confidence limit</td>
<td>$k_q \cdot u(\gamma)$</td>
<td></td>
</tr>
<tr>
<td>$\hat{\gamma}$</td>
<td>Best estimate of $\gamma$ when $\frac{\gamma}{u(\gamma)} &lt; 4$</td>
<td>$\gamma + u(\gamma) \cdot \frac{\left( \frac{-\gamma^2}{2(u(\gamma))^2} \right)}{\omega \cdot \sqrt{2 \pi}}$</td>
<td></td>
</tr>
<tr>
<td>$u(\hat{\gamma})$</td>
<td>Best estimate of $u(\hat{\gamma})$ when $\frac{\gamma}{u(\gamma)} &lt; 4$</td>
<td>$\sqrt{u^2(\gamma) - (\gamma - y) \cdot \hat{\gamma}}$</td>
<td></td>
</tr>
</tbody>
</table>
What is wrong with my old expanded uncertainty?

- Expanded uncertainty for activity can yield a confidence interval including negative values
  - This is corrected by ISO 11929
  - But it requires at least 3 numbers to be specified (limits of confidence and best estimate)
- When relative uncertainty is small
  - $\tilde{y} \approx y$
  - $u(\tilde{y}) \approx u(y)$
<table>
<thead>
<tr>
<th>Condition</th>
<th>Report</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>$y &lt; y^*$</td>
<td>$&lt; y^*$</td>
<td>The effect is not detected. Qualify this information with: 'This is the decision threshold for $^m$A in this analysis; $^m$A has not been detected in this analysis.'</td>
</tr>
<tr>
<td>$y^* &lt; y &lt; y^#$</td>
<td>$&lt; y^#$</td>
<td>The effect is detected, but not quantifiable. Qualify this information with: 'This is the detection limit for $^m$A in this analysis, and is approximately twice the decision threshold; it is possible that $^m$A has been detected, but is not quantifiable in this analysis.'</td>
</tr>
<tr>
<td>$y^# &lt; y &lt; 4. u(y)$</td>
<td>$\hat{y} \pm k. u(\hat{y})$</td>
<td>A best estimate of the result may be reported. This information may be qualified with: 'A has been identified and quantified in this analysis, although the result is close to the detection limit, $y^#$, which is reflected in the relatively large uncertainty.'</td>
</tr>
<tr>
<td>$4. u(y) &lt; y$</td>
<td>$y \pm k. u(y)$</td>
<td>The result may be unambiguously reported and no additional qualification is needed. It may be instructive for the user if this statement is made: 'A has been unambiguously identified and quantified in this analysis, where the detection limit for this analysis is $y^#$.'</td>
</tr>
</tbody>
</table>
ISO 11929 allows also to deal with the special situations

Pitfalls can be associated with the validity of the models used

Currie approach is a simplified model compatible with ISO11929 if uncertainty on conversion factor (to go from counts to activity) is also considered

- Breaks down at low count rates (as before)

- When not detected: report detection limit (as proof of what the method can measure)

- Reporting according to ISO11929: in case of negative values in confidence interval or important relative uncertainty:
  - Report limits of confidence interval (2 numbers)
  - Best estimate
  - Primary results?
Some further comments

For large values of relative systematic uncertainty we find that the ISO 11929 method tends to overestimate the MDA, and for very large values it fails to return a finite or physically meaningful value at all. Furthermore, for small values of the systematic uncertainty, the correction provided by the ISO 11929 MDA compared to the traditional Currie MDA is very small, and for applications in this regime adoption of the ISO approach may not provide significant benefits.

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Minimum detectable activity, systematic uncertainties, and the ISO 11929 standard

J. M. Kirkpatrick · R. Venkataraman · B. M. Young
How to implement ISO11929 in our labs

- Spreadsheet reporting → change formulae
- Commercial software
  - Genie 2K gammaspec: → ISO11929 is included (CAMparameters)
- Home made softwares
  - Change formulae
- At very low counts -> choise of any alpha & beta not free!
- Reporting according to ISO11929: in case of negative values in confidence interval or important relative uncertainty:
  - Many reporting tools are not ready yet for this situation
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