

Assessing limits of detection

S Ellison

/Eurachem-UK/1



/Eurachem-UK/2

Concepts



- **Critical value**
 - instrument response used to trigger action
- **Detection limit**
 - amount of substance leading to action
- **Quantitation limit**
 - lowest level at which uncertainty is acceptable

/Eurachem-UK/4

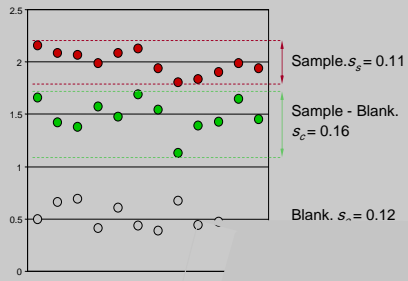


Typical experiments

- Standard deviation of blank response

J/Eurachem-UK'10

Independent corrections



/Eurachem-UK/13

Quantitation Limit (LoQ)



“Level at which the uncertainty becomes unacceptable”

- Common assumptions
 - acceptable uncertainty is 10%
 - leads to $10s_0$
- Other levels used:
 - $5s_0$, $6s_0$
- Recommendation
 - use $10s_0$ unless otherwise required

/Eurachem-UK/16

Alternative procedures



- Instrument response
- ISO 11843-2
 - From calibration data
- SANCO
 - From multi-level, longer term precision data
 - Uses ISO 11843 procedure

Usually based
on repeatability

/Eurachem-UK/17

Setting limits from instrument response



J/Eurachem-UK/19

Current recommendations



- "Less than LOD" does NOT mean "invalid result"
 - Report the raw result and its uncertainty if you can
- Not all systems provide results below thresholds
 - A case for a different approach?
 - Maximum likelihood estimation...?

/Eurachem-UK/22

Which standard deviation?



- Instrument noise?
 - If a signal is visible, there must be some analyte present!
- Repeatability?
 - Takes into account extraction/preparation
- In-house reproducibility?
 - Adds longer-term effects
- Suggestion: Smallest SD for critical value; largest for "LOD" addition.


/Eurachem-UK/23

Conclusions



- Detection limits are based on statistical reasoning
- Detection limits determined during validation are indicative
 - for typical in-house validations, approximate values are usually adequate – e.g. $3s$ for "LOD"
 - decision limits on which action depends should be rigorously checked and monitored regularly
- Report raw values if you can
 - Investigate 'censored data' methods if you can't
- Some more work is needed on which standard deviation to use for critical decisions

/Eurachem-UK/24



“A detection limit is something to stay well away from”

/Eurachem-UK/25
