Limit of Detection (LOD)

The limit of detection is the lowest level of a substance that can be detected by a method.

In general terms the Limit of Detection (LOD) of an analyte in a sample is that concentration which gives an analytical signal (e.g. a peak height or a titre), which is significantly different, at a stated level of confidence, from that of a blank sample or the background signal. The operational definition needs to be expanded around this statement to account for analysis over time, the analytical procedure and the level of confidence associated with the result.

There are a number of phrases and acronyms which are used interchangeably to denote 'Limit of Detection' ('LOD'). However, they do not represent the same thing and should not be confused.

The following is a list of search terms:

- Reporting Limits (RL)
- Minimum Reporting Level (MRL)
- Method Detection Limit (MDL)
- Lower Limit of Detection (LLD)
- Limit of Detection (LOD)
- Instrument Detection Limit (IDL)

The definitions of these terms can vary upon use. It is therefore essential to understand the hierarchy of these terms in relation to the levels of confidence they bestow on analytical results.

When analysis is undertaken, the recipient of the data needs to be reasonably confident that if a very low level of analyte is reported, then it is really present. Conversely, when no value, i.e. a less than value of an analyte is reported, the recipient again needs to be reasonably confident that no detectable level of analyte is present. In analytical terminology, this avoids the occurrence of Type 1 and Type 2 errors, false detection and false non-detection respectively. Lower reported levels should ideally have a confidence level of around 95%.

Two other terms used are Lower Level of Detection (LLD) and Method Detection Limit (MDL). Like IDL they are statistically based but with a increased level of confidence.

The MDL can be calculated from the analysis of a specified number of samples, blank samples and standards which are processed through the complete analytical method, not just through the final quantification stage.

Even with MDL, different multipliers can be used with the standard deviation of the results to give the lower reporting value.

In order to arrive at the Reporting Limit (RL) there is an additional rounding of the Method Detection Limits, which will vary dependent on either the laboratory policy or regulatory body preference.

In order to harmonise protocols for determining Reporting Limits within ALS Environmental, three key criteria are met:

1. The performance of the whole test method is assessed, not just the instrument performance.
2. The performance characteristics, precision, bias and LOD are arrived at using a defined protocol which involves the analysis of blank samples, low value and higher value standards, real samples and spiked samples eleven times in duplicate over a period of time from 6 days to 3 months. For soils and waters validation testing, up to three types of samples (e.g. clay, silt and loam for a soil) will be used, along with the supplementary validation. This protocol is designed to test the robustness of the whole analytical system, equipment, materials and analysts over a period of time.
3. The data from the testing in (2) is treated in a statistical manner, using an Analysis of Variance Programme (ANOVA) to establish the sources of variability within batch and between batch analyses. The aim for the calculated LOD is to reduce the risks of false positive and false negative LOD results to a low probability (95% confidence).

It is only by completing this full process and then applying a regulatory body recommendation for rounding to Reporting Levels, that a laboratory can be sure that its method is producing a sound estimate of analyte concentrations, especially at low levels. For example, reliance solely upon; Instrument Levels of Detection would not give the same level of assurance.

The key to ensuring a quality data set is the accuracy and repeatability/reproducibility in a statistically valid process. An IDL, for example, can often be very low due to only part of the method being employed.

The assessment is not as robust as the ANOVA protocol. A MDL will normally be higher then the corresponding IDL because the whole of the method is being evaluated. The reporting limit can be the LOD or can be a higher value in some instances to reduce even further the possibilities of a false positive result.

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