C9 Why Current Detection and Reporting Limits are Useless in Low Level Data Evaluation

Michele W. Sanders, BS, and Jack R. Hall, BS, Materials and Chemistry Laboratory, Inc., East Tennessee Technology Park, Building K-1006, 2010 Highway 58, Suite 1000, Oak Ridge, TN 37830*

After attending this presentation, attendees will have a better understanding of the basis of laboratory detection and reporting limits, and how those limits fail to provide the information necessary to evaluate low level data with a high degree of confidence.

This presentation will impact the forensic science community by providing data users reasons for the inadequacies of the current complex series of terms used for detection and reporting limits.

Currently, the environmental field is plagued with a number of acronyms for both detection limits (DL) and reporting limits (RL). For example, method detection limit (MDL), minimum quantitation limit (ML), level of detection (LD), limit of quantitation (LQ), estimated detection limit (EDL), practical quantitation limit (PQL), contract required quantitation limit (CRQL), etc. These are all derived either statistically, by contract, or from regulatory limits. None of the terms used are based on results from real world samples. The environmental laboratory industry is being required to report results down to the DL without justification or proof; thus reporting non-defendable data which the user may misinterpret. Laboratories have even been judged based on their MDLs (labs with lower DLs are better!). DLs and RLs are not the same and the terms should not be used interchangeably. The DL should be the concentration at which one can state with a known confidence level that an analyte is present. The RL should be the lowest concentration at which the analyte is determined within a known precision and accuracy range. Unfortunately, RLs in most cases are calculated as a factor times the DL, with no requirement to confirm for a specific sample. For example, a statistical approach (MDL) was originally developed by the USEPA to demonstrate that the methods being promulgated for regulated analytes had detection limits below the regulatory levels in discharge permits of Publicly Owned Waste Treatment facilities (POWTs) and receiving water bodies. The formula is MDL = 3.143 times the standard deviation of the measured analyte concentration of seven replicate spiked laboratory water solutions. The USEPA minimum level of quantification (ML) was originally stated as ten times the standard deviation used to calculate the MDL in the described way. The ML is also commonly calculated as 3.1 times the MDL.

The original intent of the USEPA was to use the MDL procedure to determine detection limits of analyte(s) in the method user's samples. Instead, the MDL requirement has been interpreted such that the environmental laboratory industry is required to determine MDLS on all analytes, general matrix types, and instruments used, but not on specific samples.

This approach has led to unbelievably low DLs and RLs in laboratory reports and subsequently in project databases. How does one defend a beryllium in soil result of <0.01mg/kg when the associated matrix spike (MS) and matrix spike duplicate (MSD) data has 15% and 12% recovery with spiking at the 1mg/kg level? The laboratory flagged the low recovery MS and MSD data as a matrix effect. This implies that the sample itself interferes with recovery of beryllium. Using the reporting limit of <0.01mg/kg would be in error and if the regulatory limit was 0.05mg/kg, one would be reporting non-defendable data that truly is wrong. The data show that one can only state with any certainty a reporting limit of 1 mg/kg.

Now, if the same data in the above example were reported for the analysis of hexavalent chromium by Ion Chromatography in soil, then the results would be understandable and defendable. Hexavalent chromium is converted to trivalent chromium by this sample matrix and thus gives low recoveries for MS and MSD samples. This can be proven by having acceptable recovery of a post digestion spike sample.

Other examples of the pitfalls of DLs and RLs and their application will be presented. Recommendations will be made on a more realistic approach to a reporting limit and its confirmation, using appropriate quality control samples, what information should be provided to the laboratory to achieve useful data and most importantly, what questions to ask the laboratory to better understand the data.

Detection Limits, Reporting Limits, Quantitation Limits

 ** Presenting Author*