

Quality Assurance – Basic Dilution Principles
Supplement to the BPSS Memorandum
“Quality Assurance and Related Issues” dated May 14, 2007

Since the April 17, 2005 revision of Chapter 62-770, Florida Administrative Code (F.A.C.), Practical Quantitation Limits (PQLs) may not exceed the Cleanup Target Levels (CTLs) during routine laboratory operating conditions unless the CTLs are below the capability of the instrument being used for the analysis. This is the case for four carcinogenic PAHs in groundwater [Benzo(a)anthracene, Benzo(b)fluoranthene, Dibenz(a,h)anthracene, and Indeno(1,2,3-cd)pyrene] and Target PQLs (alternative CTLs) have been published for them, as explained in the BPSS Memorandum “Quality Assurance and Related Issues” dated May 14, 2007.

Typically, this is not a problem unless a dilution is necessary prior to sample analysis. In this instance, the Method Detection Limits (MDLs) and the PQLs are raised since they are multiplied by the dilution factor that was used. Lately, numerous laboratory reports showed that excessive or unnecessary dilutions were performed, perhaps due to the fact that contaminant concentrations in the samples were greatly overestimated during laboratory screening. Depending on the dilution factor, the post-dilution PQLs may exceed the respective CTLs, rendering non-detects (in most instances) useless for rehabilitation and enforcement purposes. Therefore, it is mandatory for laboratories to adhere to the following basic dilution principles:

- 1) Analytical samples shall be diluted only when:
 - a) The concentration(s) of one or more analytes in the undiluted sample exceed(s) the upper limit of the calibration curve; or
 - b) Matrix interference is present, or the presence of high concentrations of non-target analytes is likely based on the review of prior (recent) results or substantiated after review of undiluted analytical results.
 - Unless dilution may be absolutely justified, no dilution must be performed if the post-dilution PQLs of the target analytes are expected to exceed the CTLs. Due to the variable nature of environmental samples, it is the laboratory's responsibility to take every action needed to overcome the interferences without compromising the quality of the data.
- 2) If a dilution is performed, a minimal dilution factor must be applied to ensure that the highest detected analyte is in the upper 60% of the calibration curve.
- 3) The reason for a dilution must be documented in a case narrative. Example: “A dilution of 1:2 was performed to prevent damage to the instrumentation or saturation of the mass spectrometer due to the high concentrations ({{state value(s)}}) of non-target analyte(s) ({{state name(s)}}) reported on {Date}. The after-dilution PQLs of non-detect target analytes did not exceed the corresponding CTLs.”

- 4) The Laboratory Control Sample (LCS) as part of the Quality Control (QC) for each batch of samples must include:
 - a) Actual LCS analytical results in addition to calculated percent (%) recoveries, and the information must be included in the laboratory report,

and / or
 - b) The calibration and/or continuous calibration data (clearly defining a calibration range for each target analyte).
- 5) If the sample is analyzed both undiluted and diluted, the results for both sets of results must be provided, as well as the MDLs and PQLs for each run (adjusted for the diluted run), and the estimated analytical results for analytes with concentrations that exceed the linear range or highest calibration standard must be appropriately qualified.
- 6) If the dilution was based on the most recent historical undiluted data, the supporting analytical results must be included in the deliverable.

Note: If the results of the first run on a sample show that an excessive dilution was used, the laboratory is expected to run the sample again, either undiluted or at a lower dilution in order to provide meaningful data.

Reminder of one of the General Reporting Requirements:

Subsection 62-160.340(6), F.A.C. states that “When data are provided to the Department in a document that is a summary, a re-published format or in a reduced form (e.g., report, table, report form), the document shall not change the original data, or delete any data qualifiers reported by the originating laboratory unless specified by Department contract, order, permit, or Title 62 rule. Copies of the original laboratory report(s) shall be submitted with all such reports unless directed to do otherwise by the Department.”

Valuable recommendations were received from Andy Tintle from the Laboratory Support Section of the Bureau of Laboratories.