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RESULT CALCULATIONS FOR MULTI-PHASIC SAMPLES

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHEMISTRY PROGRAM

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RESULT CALCULATIONS FOR MULTI-PHASIC SAMPLES

1. SCOPE AND APPLICATION

This SOP provides calculation instructions for averaging and reporting analytical results from multiple-phase samples, based on separate analytical determinations for each phase. Such samples include multiple-phase Toxicity Characteristic Leaching Procedure (TCLP) extracts, Synthetic Precipitation Leaching Procedure (SPLP) extracts, and total recoverable analysis for waste samples.

2. SUMMARY OF METHOD

When analyzing complex multi-phasic samples, it is not unusual for each phase to require a separate preparation or even a different preparation method. Results from the analysis of each phase must then be converted to the same units, if necessary. In the case of TCLP and SPLP extracts, results are proportionally averaged, based upon the relative **volume** of each phase of the extract. For total recoverable analyses on waste samples, the proportional average is based upon the relative **weight** of each phase of the sample. A multi-phase sample should be identified as such in the test comment.

3. CALCULATION PROCEDURE

There are two calculation categories, one where all phases are qualified the same and one where one or more of the phases are qualified differently. The approach to each calculation scenario is slightly different as you will find in the examples provided below. The TCLP preparation test will be used as an example throughout, but the calculations may be applied to any analyte and method. Example test comments have also been provided to help address the numerous complicated scenarios that can occur. Nevertheless, supervisors are encouraged to explain significant results in additional comments, where deemed necessary.

Category 1:

The results of all phases are qualified similarly for an analyte, i.e. they are all above the Practical Quantitation Limit (PQL), all below the PQL, or all below the Method Detection Limit (MDL). In this circumstance the reported result, MDL, and PQL are all determined by mathematically combining the values based upon the relative proportion of each phase.

Example:

A TCLP extract consisting of two phases yields results that are above the PQL(s) for the method(s). Phase 1 is 90 % of the extract by volume.

Phase 1 Result (P1) = 5.0 mg/L (MDL = 1.0 mg/L; PQL = 4.0 mg/L)

Phase 2 Result (P2) = 50 mg/L (MDL = 10 mg/L; PQL = 40 mg/L)

Reported Result = [P1 × (Phase 1 Fraction)] + [P2 × (Phase 2 Fraction)]

$$= (5.0 \times 0.90) + (50 \times 0.10) = 9.5 \text{ mg/L}$$

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Reported MDL = (1.0 × 0.90) + (10 × 0.10) = 1.9 mg/L

Reported PQL = (4.0 × 0.90) + (40 × 0.10) = 7.6 mg/L

Test Comment: The TCLP extract of the sample is comprised of (e.g., two) incompatible phases: Phase 1 (90 %) is an aqueous phase, Phase 2 (10 %) is an organic phase. The results and detection/quantitation limits were calculated and reported in accordance with the protocol outlined in DEP SOP CM-027.

Category 2:

The results of contributing phases are qualified differently e.g. one is U qualified and another is I qualified (Example 1) or one is above the PQL and another is I qualified (Example 2). In such a case, the volume or weight corrected contribution of the phase that is best quantified is reported along with the MDL and PQL for that phase corrected for amount of contribution. The best quantified phase is defined to be the one with a result above the PQL if there is one, or if not, the phase with a result greater than the MDL. It is recognized that there may be a significant positive contribution from the other phase(s), but this contribution is ignored in the calculation since it cannot be quantified as well. This protocol ensures that the reported amount is a conservative estimate of the true value.

Example 1:

A TCLP extract consisting of two phases yields one result that is below detection and one that is detected but below the quantitation limit. Phase 1 is 90 % of the extract by volume.

Phase 1 Result (P1) = 3.0 I mg/L (MDL = 1.0 mg/L; PQL = 4.0 mg/L)

Phase 2 Result (P2) = 10 U mg/L (MDL = 10 mg/L; PQL = 40 mg/L)

Reported Result = (Result from best quantified phase) × (that phase's fraction)

= (3.0 × 0.90) = 2.7 I mg/L

Reported MDL = (1.0 × 0.90) = 0.90 mg/L

Reported PQL = (4.0 × 0.90) = 3.6 mg/L

Test Comment: The TCLP extract of the sample is comprised of (e.g., two) incompatible phases: Phase 1 (90 %) is an aqueous phase, Phase 2 (10 %) is an organic phase. The results and detection/quantitation limits were calculated and reported in accordance with the protocol outlined in DEP SOP CM-027.

Example 2:

A TCLP extract consisting of two phases yields one result that is above the PQL and one that is above detection but below quantitation. Phase 1 is 90 % of the extract by volume.

Phase 1 Result (P1) = 5.0 mg/L (MDL = 1.0 mg/L; PQL = 4.0 mg/L)

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Phase 2 Result (P2) = 39 I mg/L (MDL = 10 mg/L; PQL = 40 mg/L)

Reported Result = (Result from best quantified phase) × (that phase's fraction)

= (5.0 × 0.90) = 4.5 mg/L

Reported MDL = (1.0 × 0.90) = 0.90 mg/L

Reported PQL = (4.0 × 0.90) = 3.6 mg/L

Test Comment: The TCLP extract of the sample is comprised of (two) incompatible phases: Phase 1 (90 %) is an aqueous phase; Phase 2 (10 %) is an organic phase. The results and detection/quantitation limits were calculated and reported in accordance with the protocol outlined in DEP SOP **CM-027**.

GUIDANCE for Example 2: If the volume or weight-corrected contribution of the phase that is "least well" quantified (the one quantified with an "I" qualified result) is greater than the calculated reported result, the supervisor must be notified and a decision on how to report the result will be made on a case-by-case basis.

In case of relevant findings (values exceeding some critical or regulatory level), reporting estimated results in the comment must be considered the last resort after the supervisor made efforts to resolve uncertainty by all appropriate means.

Total Recoverable Analyses: Waste samples for total recoverable analysis that are analyzed in multiple phases are calculated in exactly the same way with the exceptions that the units are different, and the fractional contribution is calculated by weight instead of volume. SPLP extracts are calculated just as are TCLP, except the units are sometimes different (e.g., for arsenic, SPLP units are µg/L instead of mg/L).

4. QUALITY CONTROL

4.1 For TCLP and SPLP samples when one of the phases is treated as a waste sample and digested according to EPA 3050, use the TCLP (or SPLP) matrix and test ID in QC Manager. The weight used and the density of the non-aqueous portion of the sample are accounted for in the dilution factor when using the MT-MULTIPHASE Form located on the Division's intranet page.

4.2 Instructions and Forms for the preparation, analysis, and reporting of volatile TCLP samples are provided in SOPs **VO-005** and **VO-001**.

4.3 The results for quality control samples should be uploaded from the most representative phase. In the example where phase 1 is 90 % of the extract by volume the method blank, LFB and matrix spikes should be uploaded from the preparation batch containing phase 1. In cases when only one phase is reported due to quantitation (Category 2, Example 1), the spike recoveries and precision should be reported from the best quantitated phase (same phase as reported result).

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APPENDIX 1. FORM FOR MULTIPHASIC SAMPLES

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TCLP Multiphasic Sample Result Calculation

MT-MULTIPHASE-1.0

Enter information into blue cells

Volume of Phase 1 (aqueous layer), mL:	1310	% of Sample from Phase 1	0.987934
Volume of Phase 2 (organic layer), mL:	16	% of Sample from Phase 2	0.012066
Volume of Total TCLP Extract, mL:	1326		1

This information is found on the TCLP extraction logbook extract finalization section.

Weight of Sample (g):	0.589	(Digestate volume (mL))/((Sample mass (g))/(Density))
Density of Phase 2 (g/mL):	0.71	Dilution Factor = 60.27165
Final Volume of Digestate (mL):	50	

This information can be found on the prep worksheet for the waste digestion of the sample. The dilution factor should be entered into QCM instead of the sample weights from prep worksheet manager.

	As	Ba	Cd	Cr	Pb	Se	Ag
Phase 1 (prepped as TCLP), mg/L:	0.1	50	0.01	0.2	5.31	0.1	0.005
Qualifier:	U	U	U	U		U	U
Phase 2 (prepped as Waste), mg/L:	3	200	0.3	13.2	289	0.4	0.02
Qualifier:		U				U	U
SOP Category/Example:	2	1	2	2	1	1	1
Combined TCLP Extract, mg/L:	0.036	51.810	3.62E-03	0.159	8.733	0.104	0.005
Qualifier	I	U				U	U

Formula for Combined TCLP Extract (If one phase is better quantitated, SOP category 2) = (Phase X, mg/L) * (% Sample Phase X)
Formula for Combined TCLP Extract (If both phases quantitated equally, SOP category 1) = (Phase 1, mg/L) * (% Sample Phase 1) + (Phase 2, mg/L) * (% Sample Phase 2)

MDL/ PQL Calculations: same equations as Combined TCLP Extract, but use MDL in mg/L instead of Phase X mg/L

	As	Ba	Cd	Cr	Pb	Se	Ag
Phase 1 MDL (prepped as TCLP), mg/L:	0.005	50	0.01	0.2	0.04	0.1	0.005
Phase 2 MDL (prepped as Waste), mg/L:	0.81	200	0.04	0.81	8.1	0.4	0.02
Calculated MDL (mg/L)	0.010	51.810	0.000	0.010	0.137	0.104	0.005
Calculated PQL (mg/L)	0.039	207.240	0.002	0.039	0.549	0.414	0.021

The dilution factor should be applied in LIMS to the 3050 waste digestion samples (do not enter weights into LIMS).
The Test ID and Matrix for the waste digestion should be set to TCLP, this will calculate results in mg/L.

Note to analysts:

1: The batch QC for the analytes comes from the more representative phase (higher %) in cases where the qualification is equal. Otherwise the QC for the analyte comes from the batch with the better qualifier.
2: The results should be changed in QC manager to reflect the accurate concentration, MDL and PQL, then uploaded with the appropriate QC.
3: Comment in LIMS that "The TCLP extract of the sample is comprised of incompatable phases and the results have been mathematically combined."

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_MT-MULTIPHASE-1.0 Example

APPENDIX OF SIGNIFICANT CHANGES

02/21/2008 All sections – Edited format. Edited Section 3calculation examples: changed units to mg/L.

02/25/2009 Section 3 added some guidance for special case samples.

03/29/2010 Added Quality Control Section; Appendix 1 added.

02/16/2016 Added a cover page and other aesthetic updates. Updated form in the appendix.

02/17/2017 Updated referenced SOPs and reworded awkward sentences.

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