Florida Department of
Environmental Protection

Memorandum

To: Petroleum Cleanup Program Staff
District Program Coordinators
Environmental Consultants
Laboratories
Interested Parties

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Subject: **SPLP Procedures for Petroleum-Contaminated Sites**

The Leachability-based Soil Cleanup Target Levels (CTLs) are based on average soil properties, and therefore it is possible for those CTLs to be exceeded in a sample even though in nature the contaminants will not leach from the soil at concentrations that exceed Groundwater CTLs even if sufficient moisture is present. Unless the contamination is derived from used oil or similar petroleum products, true leaching potential of contaminants from soil when only Leachability-based Soil CTLs are exceeded and the contaminant concentrations are relatively low may be evaluated through extraction of a soil sample using the Synthetic Precipitation Leaching Procedure (SPLP, EPA Method 1312) followed by the analysis of the leachate (1312 extract). Depending on which Soil CTLs were exceeded in soil samples collected during the site assessment, a supplemental site assessment proposal (and Work Orders and Task Assignments for funded sites) may scope SPLP extraction and analysis for BTEX/MTBE, PAHs, or both (and occasionally TRPHs). Preservatives must not be added to the soil samples prior to SPLP extraction. In most cases, sub-samples for total analysis also are collected and therefore SPLP extraction should be delayed until it is confirmed by total analysis of the corresponding sub-sample that at least one of the Leachability-based Soil CTLs is exceeded in the sample but no Direct Exposure Residential Soil CTLs are exceeded.

1) BTEX/MTBE and other volatile organic compounds (VOCs): When EPA Method 5035 became effective, establishing soil sampling procedures for VOCs and a 48-hour holding time for the closed-system analyses of low level (low concentration) samples, EPA Method 1312 was not updated to reduce the holding time for VOCs from 14 days to 48 hours. Sub-subparagraph 62-770.680(1)(c)2.c., Florida Administrative Code, allows the use of EPA Method 1312 to establish alternative Leachability-based Soil CTLs to close sites without institutional controls, which justifies adjusting EPA Method 1312 holding times for VOCs to match those of EPA Method 5035. Since EPA Method 1312 requires 25 grams of soil for VOCs in order to prevent headspace in the Zero Headspace Extraction (ZHE) Vessel used, the volume of soil (5 grams) obtained using EPA Method 5035 procedures is insufficient; therefore, when SPLP extraction has been proposed, the samples usually have been collected in a bulk jar, with corresponding loss of VOCs. Freezing, which is an option for EPA Method 5035 samples, is not appropriate if the sample is collected in a bulk jar. Based on these considerations, although more costly, the most efficient (and effective) procedure to minimize loss of VOCs during collection and handling of soil samples for SPLP extraction and VOC analysis is the following:

a) The samples must be obtained using an appropriate coring device (such as a 25-gram EnCore® sampler) that is used for collection, transport and storage of the samples with minimal loss of VOCs (if only volatile extraction will be performed, a sub-sample for moisture content should be collected in a bulk jar). Sub-samples for total analysis are placed in 40 mL VOC vials.

b) The samples must be transported on wet ice at 4°C, must be received at the laboratory within 48 hours of collection, must be frozen on arrival at the laboratory to extend the holding time to 14 days from the date of collection, and must be maintained at -10°C. If sub-samples for total analysis are not collected, freezing is not necessary if the SPLP extraction is performed within 48 hours of sample collection. Neither the samples for SPLP extraction nor the samples for total analysis may be frozen prior to delivery to the laboratory in order to meet the 48-hour holding time.

c) Unless sub-samples for total analysis are not collected because it was decided in advance that SPLP extraction would be performed, SPLP extraction should be performed only on those samples where results of the analyses of the corresponding sub-samples showed that one or more Leachability-based Soil CTLs are exceeded but no Direct Exposure Residential Soil CTLs are exceeded.

d) Once a sample is thawed (to 4°C), the SPLP extraction must be performed immediately. After the SPLP extraction has been completed, the holding time for

analysis of the leachate is seven days if unpreserved and 14 days if acid preserved (sample preparation and analysis of the leachate should be performed by the laboratory performing the SPLP extraction).

2) PAHs: Unless sub-samples for total analysis are not collected because it was decided in advance that SPLP extraction would be performed, SPLP extraction should be performed only on those samples where results of the analyses of the corresponding sub-samples showed that one of more Leachability-based Soil CTLs are exceeded but no Direct Exposure Residential Soil CTLs are exceeded [if any of the seven carcinogenic PAHs is detected during total analysis it is necessary to perform the conversion to Benzo(a)pyrene equivalents]. The sub-samples for total analysis and SPLP extraction are collected in one bulk jar with a Teflon®-lined lid, transported on wet ice at 4°C, and maintained at 4°C until the SPLP extraction is performed (the holding time for SPLP extraction is 14 days from the date of collection). After the SPLP extraction has been completed, the holding time for sample preparation is seven days and the holding time for analysis of the leachate is 40 days (sample preparation and analysis of the leachate should be performed by the laboratory performing the SPLP extraction).

3) TRPHs: It is appropriate to perform SPLP extraction either when the Leachability-based Soil CTL for TRPHs is exceeded during total analysis of the corresponding sub-sample but the Direct Exposure Residential Soil CTL was not exceeded (i.e., the TRPH concentration is > 340 mg/kg but ≤ 460 mg/kg), or when fractionation results show that none of the Direct Exposure Residential Soil CTLs of the fractions were exceeded but one or more of the Leachability-based Soil CTLs of the fractions were exceeded. The sub-samples for total analysis and SPLP extraction are collected in one bulk jar with a Teflon®-lined lid, transported on wet ice at 4°C, and maintained at 4°C until the SPLP extraction is performed (the holding time for SPLP extraction is 14 days from the date of collection). After the SPLP extraction has been completed, the holding time for sample preparation is seven days and the holding time for analysis of the leachate is 40 days (sample preparation and analysis of the leachate should be performed by the laboratory performing the SPLP extraction).

Valuable recommendations were received from Michael Blizzard from the Standards and Assessment Section of the Bureau of Assessment and Restoration Support, and from Kerry Tate from the Chemistry Section and Andy Tintle from the Laboratory Support Section of the Bureau of Laboratories.

Analyte(s)	SPLP Sample Container	Field and Transport Preservation	Maximum Holding Times for SPLP Samples		
			For Sample Transport to the Laboratory	For SPLP Extraction (EPA Method 1312)	For Sample Preparation Following SPLP Extraction and for Analysis of the Leachate
BTEX/MTBE and other VOCs	Coring device appropriate for the collection, transport and storage of a 25-gram sample, such as a 25-gram EnCore® sampler	Cool to 4°C with wet ice (preservatives must not be added)	48 hours from sample collection	*Sample frozen by the laboratory on arrival, and maintained at a temperature of -10°C for up to 14 days from the date of collection	**7 days total after the date of SPLP extraction if not adjusted to pH 2, and 14 days total after the date of SPLP extraction if acidified
PAHs and TRPHs	Glass bulk jar (8-ounce or larger) with Teflon®-lined lid	Cool to 4°C with wet ice (preservatives must not be added)	14 days from date of sample collection	Sample maintained at a temperature of 4°C for up to 14 days from the date of collection	**Sample preparation within 7 days of SPLP extraction and analysis within 40 days of sample preparation

* If the sample is not frozen, the SPLP extraction must be performed within 48 hours of sample collection; if the sample is frozen, the SPLP extraction must be performed immediately once the sample is thawed to 4°C. Note: Neither the samples for SPLP extraction nor the samples for total analysis may be frozen prior to delivery to the laboratory in order to meet the 48-hour holding time.

** Must be performed by the laboratory that performed the SPLP extraction.