



# FLORIDA DEPARTMENT OF Environmental Protection

Bob Martinez Center  
2600 Blair Stone Road  
Tallahassee, FL 32399-2400


Ron DeSantis  
Governor

Jeanette Nuñez  
Lt. Governor

Shawn Hamilton  
Secretary

## Memorandum

**TO:** Waste Site Cleanup Section (WSCS)

**FROM:** Jennifer Farrell, P.G., Program Administrator   
Waste Cleanup Program

**SUBJECT:** Variances and Clarifications from Sampling Standard Operating Procedures  
Relative to the Collection/Analysis of Quality Control Samples for WSCS Sites

**DATE:** March 2, 2022

---

### History

The Quality Assurance Rule, Chapter 62-160, Florida Administrative Code (F.A.C.), resulted in the Florida Department of Environmental Protection (FDEP) generating Standard Operating Procedures (SOPs) that are cited in the QA Rule. The Field Procedures SOPs were last updated in January 2017 (DEP-SOP-001/01) and became effective on April 16, 2018 (<https://floridadep.gov/dear/quality-assurance/content/dep-sops>). SOP FQ 1000 (Quality Control) describes the purpose and procedures for the collection/analysis of several quality control samples, including equipment, field, and trip blanks, and duplicate samples. FQ 1000 goes on to specify mandatory field quality control (QC) samples, stating that blanks are to be collected “at a minimum of 5% of each reported test result/matrix combination for the life of the project.”

A Division of Waste Management memorandum (signed by both the Division Director and the Quality Assurance Section Administrator) dated July 15, 1998, “New Soil Sampling Procedures and Recommended EPA Analytical Methods and other Quality Assurance Issues for the Division of Waste Management” (attached), states that equipment and trip blanks are not required for petroleum cleanup program sites (now known as the Petroleum Restoration Program or PRP) “unless the results of a sampling event are considered to be questionable (in which case all QA/QC procedures will be followed during the next sampling event) or they are going to be used for site rehabilitation completion.” The memo also includes a statement that “Duplicate samples are no longer part of QA/QC requirements and therefore are no longer warranted under any circumstance.” This memorandum cited direction from the legislature to manage sites in a cost-effective manner and one way to reduce costs was to reduce the number of samples collected for QA/QC purposes.

Variations and Clarifications from Sampling Standard Operating Procedures Relative to the Collection/Analysis of Quality Control Samples for WSCS Sites  
March 2, 2022

A subsequent PRP-specific SOP (PCS-005) entitled “Groundwater Sampling SOPs Variations and Clarifications for Bureau of Petroleum Storage Systems (now known as the PRP) Sites” (<https://floridadep.gov/waste/petroleum-restoration/documents/sop-pcs-005-gw-sampling-sop-variations-and-clarifications>) reiterated the above procedures from the 1998 memorandum.

The above procedures were adopted “based on the review of results of QC samples from several thousand sampling events” in which “the presence of contaminants in equipment blanks and trip blanks was rare, and when it occurred, the chemicals were present at low concentrations.”

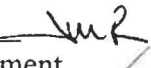
**Waste Site Cleanup Section (WSCS) Adoption of QC Sample Variance from FDEP SOPs**


The WSCS has reviewed the results of QC samples from thousands of sampling events and has come to the same conclusion that the PRP did, namely that the costs of QC samples is high and QC samples have provided little value to the Drycleaning Solvent Cleanup Program and State-Lead sites managed by the WSCS. There are scenarios (e.g. contaminants of emerging concern, contaminants with very low detection limits and cleanup target levels, specific enforcement cases) in which the collection of blanks may be advisable (e.g. Per- and Poly-fluoroalkyl substances or determining responsible parties for unknown sources) or even required (e.g. some EPA approved analytical methods for *drinking water* require trip blanks) and these should be considered on a site-specific basis, but QC samples including blanks and duplicates should no longer be collected on a routine basis for most sites. Decisions on the need for QC samples should be made by Waste Cleanup Program staff.

## Memorandum

# Florida Department of Environmental Protection

TO: Interested Parties

FROM: John M. Ruddell, Director   
Division of Waste Management

FROM: Sylvia S. Labie, Administrator   
Quality Assurance Section

DATE: July 15, 1998

SUBJECT: New Soil Sampling Procedures and Recommended EPA Analytical Methods  
(per changes to USEPA SW-846) and other Quality Assurance Issues for the  
Division of Waste Management

### APPLICABILITY

The revised procedures for soil sampling and analysis described in this memorandum apply to the cleanup of sites contaminated with volatile organic compounds (VOCs) and pertain solely to programs within the Division of Waste Management. However, in cases other than cleanup of contaminated sites, there may be certain wastes which have a very low likelihood of significant VOC contamination due to process handling or waste treatment prior to sampling. Since the data quality objectives of these applications may be different from those associated with the cleanup of contaminated sites, it may be acceptable to use more general sampling methods which screen for VOCs rather than the specific procedures contained in this memorandum. For these special cases, alternative sampling protocols may be approved by a Department Program through Standard Operating Procedures written for the specific need. These alternative sampling protocols may not be used without prior approval from the Department.

### USEPA SW-846 OPTIONS FOR SOIL SAMPLING AND ANALYSIS:

The USEPA determined that a significant percentage of volatile organic compounds (VOCs) may be lost (mainly by volatilization) from solid (soil and sediment) samples during traditional sampling and analysis procedures. This situation could have a significant impact on decisions affecting sites with VOC contamination. Therefore, the USEPA included in the June 13, 1997 update to SW 846 (Update III) changes in both sampling and analytical procedures, intended to minimize VOC losses during those processes.

The VOC concentration (Low Level or High Level) in each sample determines the analytical procedure that must be used, where the USEPA defines concentrations of VOCs of less than 200  $\mu\text{g}/\text{kg}$  as Low Level, and concentrations of VOCs greater than 200  $\mu\text{g}/\text{kg}$  as High Level. For sample collection and preparation, and for analyses of Low Level samples, the USEPA replaced Method 5030 with Method 5035, which requires analyses using a closed-system, purge-and-trap equipped with a gas chromatograph or gas chromatograph/mass spectrometer. EPA Method 5030 is still used for analyses of the solvent extracts from Method 5035 for High Level solid samples and for aqueous samples. Samples for High Level analyses must be extracted with purge-and-trap grade methanol or polyethylene glycol. These samples may need to be diluted

*"Protect, Conserve and Manage Florida's Environment and Natural Resources"*

*Printed on recycled paper.*

MEMORANDUM

July 15, 1998

Page 2

prior to analysis and therefore the method detection limits (MDLs) may not meet some cleanup target levels.

Update III also includes Method 5021 (a headspace method) for Low Level analyses. However, EPA Region IV does not recommend its use..

The most significant change in 5035 is the inclusion of specific sample collection procedures. The method presents several options for collecting solid samples.

Two of the options are not recommended because they involve the use of methanol as a preservative in the field. Methanol is difficult to handle in the field because of its high volatility and toxicity and is considered to be a hazardous material. Its use may generate a hazardous waste because the sample may fail the ignitability characteristic, thus adding to the shipping requirements and costs. These methods are not recommended by the FDEP except when a mobile laboratory is utilized to perform the analyses on-site.

The other options can be implemented in the field without much difficulty. The method discusses several options for both high level and low level collection. The options for Low Level analyses provide reliable samples, however, one of these options specifies the collection of an additional bulk sample for High Level analyses. Since high VOC loss may occur during collection and analysis of bulk samples, collection of a bulk sample is not appropriate if cleanup target levels for the Chemicals of Concern (COCs) exceed 200 µg/kg. Since most of the COCs have cleanup target levels higher than 200 µg/kg, the collection of a bulk sample is not acceptable. Because of the Department's concern with samples containing high level VOCs, another option is proposed (see high level analysis below).

For any option, extreme care must be taken to minimize volatilization by quickly getting the sample into the device used for transporting it to the laboratory. The samples must then be placed immediately on wet ice to minimize biodegradation, and unpreserved samples must be shipped to the laboratory on the same day of collection because they have a holding time of 48 hours. On arrival at the laboratory, Low Level samples preserved only with ice may be frozen in the vial to -10°C to extend the holding time to 14 days from the time of collection. During freezing, the vials must be stored on their side or at a slanted angle to maximize surface area and minimize the possibility of breakage when the liquid expands during freezing.

Laboratories may require a minimum of one additional (triplicate) Low Level sample (usually one for every 10 to 20 sampling locations), to be used for their internal Quality Assurance/ Quality Control (QA/QC) procedures.

RECOMMENDED SOIL SAMPLING PROCEDURES:

The laboratories should continue to prepare the field sampling kits, which will determine the number and type of sampling devices and sample vials that will be needed at each sampling location. Both a Low Level and High Level sample are required per sampling location (they need

MEMORANDUM

July 15, 1998

Page 3

to be collected as soon as possible after the surface of the soil has been exposed to the atmosphere to minimize volatilization):

- For Low Level analysis:

Collect two samples (approximately 5 g), using a disposable plastic syringe with a barrel diameter that is smaller than the neck of the VOC vial, and whose tapered front end is cut off and the rubber cap from the plunger removed. Since approximately 5 g (or 3 cm<sup>3</sup>) of soil or sediment are needed, it is recommended that field personnel have the ability to make a good estimate of this volume when disposable syringes are used to collect the samples. The sample in the syringe must be transferred immediately (within 10 seconds) into the VOC vial, which was prepared at the laboratory with approximately 5 ml of organic-free reagent-grade water and a magnetic stirring bar (unless a different means of stirring the sample is employed by the purge-and-trap device). A portable balance may be used to confirm that  $5.0 \pm 0.5$  g of sample were added to the vial. Because the VOC-vials must be weighed (tared) at the laboratory prior to shipping to the field personnel, lids must not be interchanged in the field and labels other than those affixed by the laboratory must not be added. In addition, it is imperative to avoid spilling any of the water during transfer of the sample from the syringe to the vial. Splashing can be minimized by holding the vial at an angle. Other precautions include making sure that the vial is not chipped; completing the collection process with the least amount of disaggregation possible; wiping the outside of the syringe with an unused, lint-free, VOC-free wipe (that must be kept in a sealed container until ready to use) immediately after sample collection; and making sure that the lip and threads of the VOC vial are free of debris before sealing it with the Teflon<sup>TM</sup>-lined septum screw cap. Preservation with approximately 1 g of sodium bisulfate is acceptable, but not recommended for Florida because a significant proportion of soils in Florida are calcareous in nature and calcareous soils will react with the preservative (denoted by effervescence). If a preservative is not added, shipping is simpler because sodium bisulfate is considered to be a corrosive material according to FDOT regulations. If field preservation is desired, the sodium bisulfate must be added to the vials by the laboratory before the initial tare weight is taken.

OR

- Collect two 5 g samples, using EnCore<sup>TM</sup> samplers (or equivalent). Each sampling device is sealed after sample collection and is used to transport the sample to the laboratory.
- For High Level analysis. collect one 5 g or 25 g sample using an EnCore<sup>TM</sup> sampler (or equivalent). The sample size is specified by the laboratory performing the analyses. The sampler is sealed after sample collection and is used to transport the sample to the laboratory. Although the 25 g EnCore<sup>TM</sup> sample generates more waste than the 5 g sample, it allows the laboratory the opportunity to increase the volume used for the analysis when dealing with low dry weight samples.
- For determination of the moisture content. collect one bulk sample in a two or four ounce glass container with Teflon<sup>TM</sup>-lined septum lid, filling the container as much as possible

MEMORANDUM

July 15, 1998

Page 4

without compromising the seal. If other analyses are to be performed on the soil sample (e.g., metals, PAHs, etc.), a separate bulk sample for moisture content may not be necessary, as long as all analyses are performed by the same laboratory.

- For laboratory screening, one sample (approximately 1 g), collected in a headspace vial, filling the vial as much as possible without compromising the seal. This sample is not required if screening is performed in the field using a 200 µg VOC/kg site-specific standard, and some laboratories may not need this additional vial if the sample for moisture content is collected in a four ounce jar or if they prefer to subdivide the large EnCore™ (or equivalent) sample.

The following two field options are acceptable to conduct soil evaluations in Florida:

Options	Low Level	High Level	Moisture Content	Screening
1	(2) EnCore™ sampler (or equivalent)	(1) EnCore™ sampler (or equivalent)	(1) Bulk sample <sup>1</sup>	(1) Headspace vial <sup>1</sup>
2	(2) Syringe + VOC vial	(1) EnCore™ sampler (or equivalent)	(1) Bulk sample <sup>1</sup>	(1) Headspace vial <sup>1</sup>

<sup>1</sup> Optional, the need for a bulk sample may not be required if the laboratory is performing other analyses (e.g. metals) from the same sampling location..

Both options are identical except for the procedure used to collect the samples for Low Level analyses.

There is an advantage to the use of the EnCore™ samplers (or equivalent) in that they are used to transport the samples to the laboratory, eliminating further handling in the field. This method assures the lowest loss of volatiles even though the current version of the device might allow some humidity (and therefore some VOCs) to escape. The disadvantages are its unit cost and the fact that the "T-handle" (the device used to help push the sampler into the soil) must be decontaminated in the field. Independent of these considerations, the EnCore™ sampler is the only available method to sample sediment located under water.

The main advantage of the plastic syringes is their lower cost and the fact that field decontamination is not necessary because the syringes must be discarded after a single use. The main disadvantage of this option is that it involves a transfer step in the field is required, and subject to a somewhat higher risk of loss of volatiles.

If the number of samples to be collected justifies the cost of utilizing a mobile laboratory, samples for High Level analyses may be collected using the disposable syringe technique and transferring the contents immediately into tared VOC vials containing methanol.

MEMORANDUM

July 15, 1998

Page 5

RECOMMENDED EPA ANALYTICAL METHODS:

Several methods were deleted by the USEPA in Update III of SW-846. For the purposes of most cleanup programs in the Division of Waste Management, the most important of these are EPA Methods 8010 and 8020, and to a lesser extent EPA Methods 8240 and 8250. Based on these deletions, the options available for VOC analyses are as follows:

Solid samples:

Volatile Organic Aromatics (BTEX plus MTBE) and Volatile Organic Halocarbons: EPA Methods 8021 or 8260.

BTEX plus MTBE: EPA Methods 8021 or 8260 (in part).

Liquid samples:

Volatile Organic Aromatics (BTEX plus MTBE) and Volatile Organic Halocarbons: EPA Methods 601/602, 624, 8021 or 8260.

BTEX plus MTBE: EPA Method 602 or EPA Methods 624, 8021 or 8260 (in part).

PREAPPROVAL/STATE CLEANUP DEVIATION FROM QA/QC REQUIREMENTS:

The FDEP was directed by the legislature to manage sites in a cost-effective manner. One way to reduce cleanup costs is to minimize the number of samples that need to be collected for QA/QC purposes. Effective immediately for preapproval and state cleanup sites in the petroleum cleanup program, the following QA/QC requirements are waived:

Solid samples:

Equipment blanks, trip blanks and duplicate samples are not required for any sampling event.

Liquid samples:

Equipment blanks and trip blanks are not required unless the results of a sampling event are considered to be questionable (in which case all QA/QC procedures will be followed during the next sampling event) or they are going to be used for site rehabilitation completion.

This decision was based on the review of results of QC samples from several thousand sampling events. The presence of contaminants in equipment blanks and trip blanks was rare, and when it occurred, the chemicals were present at low concentrations. Also, the presence of contamination in the equipment and/or trip blank only means that the degree of groundwater contamination may have been overestimated, which is not a significant problem in the petroleum cleanup program because the sites are confirmed to have contamination.

For non-program and voluntary cleanup sites in the petroleum cleanup program, it may be possible to reduce the number and frequency of QC samples if requested by the responsible party or consultant and agreed to by the FDEP or local program technical reviewer.

MEMORANDUM

July 15, 1998

Page 6

METHOD DETECTION LIMITS (MDLs):

In accordance with Chapters 62-770 and 62-785, FAC, the MDLs must meet the specified cleanup target levels (in the case of soil samples, both the direct exposure cleanup target levels for a residential scenario and the leachability-based cleanup target levels for protection of the groundwater apply). This requirement must be met if the intent of the analysis is to determine whether a chemical or group of chemicals is present, or to confirm that site rehabilitation completion is appropriate (unless the MDL using the most sensitive and currently available technology is higher than a specified criterion, in which case the practical quantitation limit must be used). If it has been confirmed that the site is contaminated exclusively with products from the Gasoline Analytical Group, a maximum MDL of 1 ppb is acceptable for those Polycyclic Aromatic Hydrocarbons (PAHs) with cleanup target levels lower than 1 ppb in groundwater samples. If the contamination is affecting or may potentially affect a surface water body, the surface water criteria must be met. Some PAHs have very low surface water criteria; therefore, the groundwater samples must be analyzed using the most sensitive and currently available technology, such as EPA Method 8310.

JMR/SSL/giw