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June 17, 2015

Mr. Paul Cho, P.G.  
Water Resources Control Engineer  
California Regional Water Quality Control Board, Site Cleanup Unit IV  
Los Angeles Region  
320 West 4<sup>th</sup> Street, Suite 200  
Los Angeles, CA 90013

Subject: Soil Field Preservation and Laboratory Handling – EPA Method 5035  
Defense Fuel Support Point Norwalk  
15306 Norwalk Boulevard, Norwalk, California  
(SCP NO. 0286A, Site ID NO. 16638)

Dear Mr. Cho:

On June 15, 2015, The Source Group, Inc. (SGI), on behalf of the Defense Logistics Agency – Energy (DLA –Energy), submitted the *Revised Field Sampling and Analysis Plan* (Plan). Subsequent to the submittal of that document the RWQCB requested additional details regarding field methods used to collect soil samples via the TerraCore™ method. The RWQCB also requested a description of the handling of TerraCore™-preserved soil samples upon delivery to the laboratory.

The TerraCore™ soil samplers are designed for single use at a soil sampling location. After a soil sample is collected in the field, the TerraCore™ sampling device is used to collect sub-cores of the soil sample for placement into 40 milliliter VOA containers. As part of the sampling protocol to be used at DFSP Norwalk, the TerraCore™ sampler will be used to place three soil cores into each of three VOA containers (one VOA container is supplied by the laboratory with a methanol preservative, the other two VOA containers are supplied by the laboratory with a sodium bisulfate preservative). Instructions for use of the TerraCore™ sampler are attached.

Per RWQCB request, SGI contacted American Analytics Laboratory of Chatsworth, California. American Analytics will provide analysis of soil samples collected during the DFSP Norwalk project. The laboratory director prepared a description of the process used by the laboratory upon receipt of soil samples preserved in accordance with the EPA Method 5035. The summary of the handling of soil samples and the SOP provided by the laboratory is attached.

Sincerely,

Neil F. Irish, P.G.  
Project Manager  
The Source Group, Inc

Ec: Mr. Nick Carros, DLA Energy  
Mr. Kenneth Wall, SGI  
File: DFSP Norwalk – 04-NDLA-007

Attachments  
Field Method Description  
E-mail from American Analytics  
Laboratory Standard Operating Procedure – EPA 5035

1962 Freeman Avenue  
Signal Hill, California 90755

Telephone: (562) 597-1055  
Facsimile: (562) 597-1070

# Recommended Use Of The Terra Core®



**NOTE:** *The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.*



## Step 1

Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.



## Step 2

Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.



## Step 3

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. **Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.

**Subject:** Processing of Samples Collected in VOA Vials in Accordance with EPA Method 5035 Upon Arrival at the Laboratory

**Date:** Tuesday, June 16, 2015 at 4:37:00 PM Pacific Daylight Time

**From:** George Havalias

**To:** Neil Irish

Hi Neil,

As per our earlier telephone conversation, the following procedure describes the processing of the sample VOA vials collected in accordance with the requirements of EPA method 5035 upon arrival at the laboratory. These procedures are specific to the field preservation of soil samples in pre-tared VOA vials containing sodium bisulfate and methanol preservatives.

- The samples arrive at the laboratory with Chain of Custody in coolers appropriately preserved on ice at approximately 4 deg C.
- The samples are logged into the Laboratory Information Management System (LIMS) and are appropriately labeled with the laboratory assigned unique sample identification numbers.
- The samples are then visible on the departmental chemical analysis schedulers and are loaded onto the auto-sampler for analysis by the responsible chemist providing there is adequate analytical capacity. The samples up to the time of analysis are maintained at 4 deg C and in this state must be analyzed within forty eight (48) hours from the time of collection.
- Alternatively, if the samples cannot be promptly analyzed, in accordance with the requirements of EPA method 5035, the samples are frozen until analyzed. In this state, the sample holding time in accordance with EPA method 5035 is extended to fourteen (14) days from the time of sample collection.
- In accordance with EPA method 5035 requirements, the sodium bisulfate preserved samples are used for low level VOC analysis and the methanol preserved samples are used for high level VOC analysis by dilution of an aliquot of the methanol in water prior to sample analysis.

The VOA vials used for field sampling are provided by American Analytics. These vials are purchased from a reputable vendor and they are pre-cleaned, pre-labeled, and pre-tared for sample weight determination and contain VOC free sodium bisulfate and methanol preservatives. In addition, the VOA vials with the sodium bisulfate contain a stir bar for automated mixing of the sample when on the auto-sampler as specified by EPA method 5035.

Each production batch of VOA vials received by American Analytics from our vendor is batch certified by testing a random VOA vial from the batch for the presence of VOCs. The VOA vials are provided to our client for sample collection only if the test results are below the Method Detection Limit (MDL) for all target compounds.

After the samples are dispensed into the VOA vials in the field using dedicated Terracores and sealed with Teflon lined septa and caps, the vials are not opened again throughout the analytical procedure ensuring that VOCs are not lost due to volatilization. The sodium bisulfate reduces the sample pH eliminating bio-attenuation processes.

If you have questions please contact us at American Analytics.

Sincerely,

George Havalias

George Havalias

*Vice President & Laboratory Director*

**American Analytics, Inc.**

9765 Eton Ave

Chatsworth, CA

(818) 998-5547 Ext. 318



American Analytics, Inc.  
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Title: EPA 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

Filename: 6115v3.doc

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<u>Rev. No.</u>	<u>Effective Date</u>
3	06-17-2015

Prepared By

Date

Technical Review

Date

*Allen A*

06-17-2015

*George Havalias*

06-17-2015

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Name/Title: Allen Aminian, Quality Manager

George Havalias, Laboratory Director

QA Approval/Date:

*Allen A*

06-17-2015

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Allen Aminian, Quality Manager

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TABLE OF CONTENTS

**1.0 SCOPE AND APPLICATION ..... 4**

**2.0 METHOD SUMMARY ..... 4**

**3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE ..... 5**

**4.0 INTERFERENCES AND POTENTIAL PROBLEMS ..... 6**

**5.0 EQUIPMENT & APPARATUS ..... 6**

**6.0 SAMPLING PROCEDURES ..... 7**

**7.0 HEALTH AND SAFETY ..... 8**

**8.0 REFERENCES ..... 8**



## 1.0 SCOPE AND APPLICATION

- 1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.
- 1.2 The low-concentration soil method utilizes a hermetically-sealed VOA sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low-concentration soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.
- 1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.
- 1.4 Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.
- 1.5 Method 5035, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the light end petroleum hydrocarbon fuels (e.g., gasoline). For the aromatic fraction (BTEX), use Method 5035 and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

## 2.0 METHOD SUMMARY

- 2.1 Low concentration soil method - generally applicable to soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg:
  - 2.1.1 Volatile organic compounds (VOCs) are determined by collecting an approximately 5 g sample and placing it in a pre-weighed vial with a septum-sealed screw-cap that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to the laboratory. The entire vial is then placed, unopened, into the instrument autosampler. Immediately before analysis, organic-free reagent water, surrogates, and internal standards are automatically added through the septum without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with stirring of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap

is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg:

2.2.1 The sample introduction technique in Sec. 2.1.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/PID, etc.). In such instances, collect an approximately 5 g sample in a pre-weighed vial with a septum-sealed screw-cap that contains 5 mL of a water-miscible organic solvent (e.g., methanol). At the time of analysis, a measured aliquot of the methanol extract is added to a pre-measured volume of water in a VOA vial, followed by the addition of the surrogate and internal standard(s). The VOA vial containing the diluted sample is then purged using Method 5030 and analyzed by an appropriate determinative method.

### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 There are four EPA-approved container options for collecting samples in the field. The four container options are:

3.1.1 A 40 mL VOA vial preserved with sodium bisulfate, which reduces the sample pH thereby eliminating bio-attenuation processes, and containing a magnetic stir bar (for low-level VOC analysis)

3.1.2 A 40 mL VOA vial containing a magnetic stir bar (also called an “empty vial”) (for low-level VOC analysis)

3.1.3 An EnCore™ sampler (for low-level VOC analysis)

3.1.4 A 40 mL VOA vial containing 10 mL of methanol (for high-level VOC analysis)

3.2 Each of the VOA vial container options employs the use of the Terra Core™ sampler in order to collect a ~5 g aliquot of soil for insertion into the VOA vial. Sample collection for low-level VOC analysis which use VOA vial containers are filled in the field and never opened during the analytical process. Sample collection for low-level VOC analysis which use EnCore™ containers are filled in the field and then transferred into a VOA vial by a chemist back in the laboratory.

3.3 The VOA vials used for field sampling are provided by American Analytics. These vials are purchased from a reputable vendor and they are pre-cleaned, pre-labeled, and pre-  
tared for sample weight determination. VOA vials used for low-level VOC analysis either contain VOC-free sodium bisulfate preservatives or are empty. VOA vials for high-level VOC analysis contain methanol preservatives. In addition, the VOA vials with the exception of the methanol-preserved vials contain a stir bar for automated mixing of the sample when on the autosampler as specified by EPA method 5035.

3.4 All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice.

### 3.5 Holding Times

3.5.1 Published holding times should be followed, unless performance data can be produced to support longer time periods.

3.5.2 In accordance with the EPA requirements, the holding time for unpreserved samples maintained at  $4 \pm 2^\circ\text{C}$  is 48 hours between sample collection and either analysis or preservation of VOC soil aliquots in VOA vials.

3.5.3 Implementing a 48-hour holding time can be difficult when transporting VOC soil samples (via overnight air carrier) from the field to an off-site support laboratory. All interested parties (i.e., field and laboratory personnel) need to be cognizant that the 48 hour holding time begins from the time of sample collection. If the VOC analysis cannot be completed prior to the expiration of the initial 48 hour period, other preservation measures (i.e., freezing, chemical preservation, and methanol extraction) are required in order to extend the analysis holding time to 14 days from the time of sample collection.

## 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 Acidification of certain soils with sodium bisulfate may produce a false positive acetone artifact of 100 – 200 ppb, or more.

4.2 If a perfect seal is not achieved during EnCore™ sampling in the field due to soil particulates on the o-rings, VOCs may be lost during transport. This problem is magnified as a function of time.

## 5.0 EQUIPMENT & APPARATUS

5.1 Terra Core™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent

5.2 EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent

5.3 40 mL pre-cleaned, pre-labeled, pre-tared VOA vial with sodium bisulfate preservative and magnetic stir bar – (Scientific Specialties Service, Inc., 7201 Standard Dr, Hanover, MD 21076), or equivalent

5.4 40mL pre-cleaned, pre-labeled, pre-tared VOA vial with magnetic stir bar – (Scientific Specialties Service, Inc., 7201 Standard Dr, Hanover, MD 21076), or equivalent

5.5 40 mL pre-cleaned, pre-labeled, pre-tared VOA vial with methanol preservative – (Scientific Specialties Service, Inc., 7201 Standard Dr, Hanover, MD 21076), or equivalent

- 5.6 GC/MS Auto-sampler: Manufactured by Varian, Model Archon, closed system auto-sampler, fully compliant with the requirements of EPA method 5035 for the analysis of soil samples in hermetically-sealed VOA vials. Capable of automated internal standard addition, and purging of the VOCs from the sample matrix while concurrently stirring and heating the contents of the VOA vial. The VOA vial remains hermetically sealed with no VOC losses throughout the entire transfer of the VOCs to the concentrator and subsequently the Gas Chromatography/Mass Spectrometer (GC/MS) chemical analysis system.

## 6.0 SAMPLING PROCEDURES

- 6.1 Collect the sample according to the procedures outlined below. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to collect the samples, including devices such as the EnCore™ sampler and the Terra Core™ sampler. Always wear gloves whenever handling the pre-tared sample vials.
- 6.2 For Terra Core™ sampling:
- 6.2.1 Using the Terra Core™ sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere, generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.
- 6.2.2 Using the Terra Core™ sample collection device, transfer the soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.
- 6.2.3 NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analytes, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution.
- 6.2.4 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the

same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

### 6.3 For EnCore™ sampling:

- 6.3.1 Using the EnCore™ sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere, generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel and immediately seal the EnCore™ sampling device with its cap and turn the rod to lock the EnCore™ sampling device shut.
- 6.3.2 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.
- 6.3.3 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

## 7.0 HEALTH AND SAFETY

- 7.1 When working with potentially hazardous materials, follow EPA, Occupational Safety and Health (OSHA) and laboratory health and safety practices. More specifically refer to AA SOP 1015, Health and Safety Plan.

## 8.0 REFERENCES

- 8.1 U.S. EPA SW-846 Methods 5035 and 5035A, *Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples* (U.S. EPA 1996 and 2002).