U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 4, SCIENCE and ECOSYSTEM SUPPORT DIVISION
ATHENS, GEORGIA 30605-2720

4SES-EI

MEMORANDUM

SUBJECT: Determination of Volatile Organic Compounds in Soil

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I. INTRODUCTION

On June 13, 1997, SW846 Update II was revised; this changes the soil collection and analysis procedures for volatile organic compounds (VOCs). The updated procedures are contained in SW846 (Update III) Method 5030B and Method 5035.

Method 5030B incorporates the following:

- the analysis of aqueous samples, soils and other solid samples with a high VOC concentration (greater than 200 μg/kg), or
- a high concentration oily waste sample (greater than 200 μg/kg).

Method 5035 describes the following:

- the collection and analysis of low level VOC solid samples (soils, sediments, and solid waste with VOC concentrations in the range of 0.5 to 200 μg/kg). The analysis consists of a closed-system purge-and-trap method
- procedures for collecting and preparing solid samples containing high concentrations of VOC's and for oily wastes.

The revised RCRA methods require different sampling and analysis procedures for samples having high concentrations of VOC's versus low concentrations of VOC's. Update III sample collection techniques are more complicated and tedious for volatile organic analysis than those of Update II; however, **the accuracy of the Update III soil collection techniques warrant their**

immediate use versus traditional methods. Previous methodology has been shown to report significantly lower concentration of VOC's in soil. SESD is in the process of updating the *Environmental investigations Standard Operating Procedures and Quality Assurance Manual,* dated May, 1996, to incorporate these new procedures.

II. RECOMMENDATIONS

1. Starting January 1, 1998, all RCRA Corrective Actions and Underground Storage Tank (UST) activities should determine VOCs in soil using sample collection procedures consistent with Methods 5021 or 5035 of Update III to SW-846, "Test Methods for Evaluating Solid Waste" as published in Federal Register of June 13, 1997, Vol. 62, No. 114, pp. 32452-463.

2. For Work Plan/Quality Assurance Project Plans (QAPPs) approved prior to January 1, 1998 with traditional "low-concentration volatiles in soil" sampling during the first quarter of 1998 (Jan. 1-March 31, 1998), we encourage modifying the sampling plan to reflect the use of Update III techniques for soil/solids. For any sampling done after March 31, 1998, regardless of when the work plan was approved, sampling techniques consistent with Update III must be utilized.

3. Although Update III to SW-846 was effective June 13, 1997, EPA's Office of Solid Waste, in a policy memorandum, recommended Update III changes be cautiously implemented to allow laboratory and sampling organizations time to purchase new instrumentation/equipment. A six (6) month delay in implementing Update III was suggested, and this is equivalent to the above January 1, 1998 date.

4. The need and use of the low concentration option versus the high concentration option from Method 5021 or Method 5035 will be determined for each sampling activity based on Data Quality Objectives, risk, project needs, intended data use, etc.

5. It is relatively easy to implement the methanol extraction for sample collection/laboratory analysis. Soil VOCs determination, using methanol, is done using the same instrumentation currently in place for waters. *Note: If the methanol extraction procedure is used for low VOCs in soil samples, data quality objectives may not be met due to the higher detection limits of the analytical procedure.*

6. U.S. EPA contractor support (e.g., oversight activities) for RCRA Corrective Action or UST activities, should collect and analyze VOCs in soil/solids using Update III procedures.

7. Soil VOC samples which are collected and analyzed by SESD (RCRA, Superfund and Water) will be consistent with Update III. SESE initiated the use of this soil method in October, 1997.

If we may be on any assistance concerning the above information, please contact Diane A. Guthrie, P. E. of the Hazardous Waste Section staff at (706)355-8622 or email *guthrie.diane@epamail.epa.gov.*

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SOIL SAMPLING FOR VOLATILE ORGANIC CONSTITUENTS

12.4 Special Techniques and Considerations

12.4.1 Collection of Soil Samples for Volatile Organic Compounds (VOC) Analysis

The following sampling protocol is recommended for site investigators assessing the extent of volatile organic compound (VOC) soil contamination in vadose and groundwater zones. This procedure is designed to establish more representative VOC concentrations in soils, sediments and solid waste samples than the "low concentration volatiles in soil" techniques which are described in Update II SW-846, or earlier editions. Update III SW-846 Method 5035 ad Method 5021, promugated in June, 1997, soil collection techniques are more complicated and tedious for volatile samples; however, the accuracy of these new techniques warrants their immediate use versus traditional methods. Previous methodology has been shown to significantly under-report the presence o f volatiles in soils. These new procedures require careful coordination between field

12.4.1.1 *Testing for the presence of carbonates in waste and soil samples using an effervescence test.*

Moisten approximately 1 gram of material that has been place on a watch glass or similar surface with water, then stir to remove any trapped air; add drop-wise a cold solution of 4 N HCI while observing for effervescence, using a hand lens. Or, collect a test sample, and to a pre-preserved vial, and check for effervescence. If effervescence (rapid formation of bubbles) is observed then preservation by acidification is not appropriate. If effervescence is observed, add 5 mils of reagent-free water and approximately 5 grams of material to a 40-ml glass vial. Subsampling should be conducted as noted in section 12.4.1.3. VOA soil samples which are not preserved must be shipped to the laboratory the same day the sample is colleted. If effervescence is not observed, then proceed with the appropriate preservation methods outlined in Sections 12.4.1.4.

12.4.1.2 *On-site Estimation of Total VOC Concentration at Sampling Locations*

The paramount concern when using Method 5035 are that the sample preparation procedures are based upon whether the soil samples have a low or high concentration of total volatile organics. Therefore, screening results can be used to establish which samples have a; low concentration of total VOCs, I.e. having an estimated concentration of less than 200; micrograms/kilogram (μg/kg) or having a high concentration of total VOCs equal to or greater than 200 μg/kg. The following procedure establishes the total VOC concentration at a given sampling location requiring high or low sample preparation procedures for the laboratory analysis. The decision criteria which is based upon the response of a hand held PID to a 200 μg VOC/kg site-specific working standard established on-site during sampling activity. *(Note: Screening activities can be avoided if the sampler collects two vials for low concentration analysis and 1 fro a high concentration analysis. The disadvantages associated with this methodology are (1) the methanol is hazardous and appropriate health and safety protocol must be followed and (2) the generation of a sample which will be treated as a hazardous waste, which may not be necessary.)*

1. Preparation of Working Standard Stock Solution: The stock standard should consist of the principal VOC of site interest in polypropylene glycol (PPG). The stock standard should be prepared based on the density of the analyte of interest so that a 1 to 2 microliter volume of neat reagent transferred to

2.5 mL of PPG, would be appropriate for many common chlorinated and aromatic compounds. For example:

Stock standard: 1.24* g/mL x 0.0020 mL / 2.5 mL = 1.1 mg TCE/mL.
Working Standard: 1.1 mg TCE/mL x 0.0018 mL / 10 g matrix = 0.2 μg TCE/g.
* density of TCE

2. Working Standard and Sample VOA Preparation: Modifications to VOA vials for both working standards and samples entails punching a 5 to 6 mm hole through the center of the Teflon faced silicon septum, followed by covering the glass rim of each VOA vial with a 3 x 3 cm square of light gauge aluminum foil (see Figure 1).

Working Standard VOA Vial Preparation: Working standards are prepared by adding microliter amounts (0.001 to 0.002 mL) of the PPG based stock standard containing the principal analyte of interest to VOA vials containing 10 mL of reagent water and 10 g of the uncontaminated site specific matrix, such that a concentration of .02 mg total VOC/kt is achieved. Immediately after spiking, these vials are covered with a single sheet of aluminum foil which is tightly held in position with a modified septum (hole punched in middle) and screw cap. The contents of the working standards should be thoroughly mixed y hand shaking, then transported to location of the sampling activity, stored out of direct sunlight, and allowed to equilibrate for 1 hour prior to use. Working standards should be prepared daily. The sample weight, and correspondingly the volume of working standard can be altered depending on the response of PID, the principle analyte of site interest in the matrix of concern. The PID response to the working standard should be at least 10x greater than its response to a blank (reagent water, contamination free matrix, and appropriate volume of PPG). Note: For analytes with high vapor pressures and/or low octanol water partition coefficients, and sample matrices with low organic carbon contents, it may not be necessary to include the sample matrix in the working standards. This should established on a site by site basis, by comparing the mean of triplicate working standards with and without the sample matrix. As a general rule, if the means differ by more than 20%, then it is recommended that the sample matrix be included in the working standards.

Sample Screening Vial Preparation: Prior to the field sampling episode 10 mL of reagent water is added to the modifies VOA vials. Once prepared the VOA vials for screening samples should be transported to the sampling location and stored our of direct sunlight until they are used.

3. Sample Collection: The native structure of the material being sampled for screening should be kept intact, therefore experiencing as little disaggregation as possible during the collection and transfer process. This can often be accomplished with a coring tool designed to obtain a discrete sample. For example, a modified 10 mL syringe is a practical tool for obtaining a 5 g sample. This devise is transparent and comes with gradient markings thus the volume/weight relationship for a given material can easily be established with a portable balance. The location of samples taken for both screening purposes and laboratory analysis should be as close as possible to each other (generally within 10 cm radius), and from the same stratum. Prior to preparing (or exposing) a fresh sampling surface, for instance, opening a split spoon or scrapping away the top layer of a material, the cap and aluminum foil should be removed from the screening VOA vial. To obtain a sample, the corer, should be inserted perpendicular to the surface presented (unless the depth is inadequate or if the material is very dry, then a lateral insertion could be used). After retrieving a discrete sample the core barrel should be inserted into the mouth of the screening VOA vial and the sample extruded. When extruding the sample plug from the corer, precautions should be taken to avoid contacting the reagent grade water. Once the sample has been extruded the aluminum foil and cap should be returned to the vial. This collection and transfer process should take less than 10 seconds, and the sample weight only has to

approximate (10 plus or minus 2 g). If samples larger than 5 g are required, more than one corer can be used, or several transfers with a single corer can be made.

4. Analysis of Working Standards and Samples: Before the analysis of a working standard or sample, the VOA vial should be hand shaken for 10 to 15 seconds. The vials are then visually checked for both the complete dispersion of the sample matrix and for particles adhering to the aluminum foil cap liner (knock large particles off the aluminum foil if present). Then the inlet tube of the PID is pushed through the foil liner, to a set position about 3 cm below the rim. A maximum response for each sample screened and for the analysis of each working standard, should be recorded. Cohesive materials, such as silts and clays, do not break apart rapidly upon shaking and may require more than 15 seconds for complete dispersion.

5. Operational Procedure and Annotations: The PID should be initially calibrated with a cylinder of standard gas (for example, 100 ppm isobutylene) at the beginning of each day. This task can be performed prior to going to the sampling location. However, the analysis of both site specific working standards and the screening of a sampling location should be performed under the same conditions, thereby, normalizing meteorological influences, on the PID's performance. Site specific working standards should be prepared daily, and in sufficient quantity to satisfy the studies objectives. At a minimum, one working standard should be analyzed for every hour of site activity. Collection of samples for VOC analysis should always be the first operation performed after a surface to be samples has been exposed to the atmosphere. This includes both samples for screening and for laboratory analysis. To establish how to handle and prepare the discrete sample for laboratory analysis (low or high level procedure), a total VOC screening analysis should be performed at each sampling location. Therefore, before opening a split spoon, scrapping a fresh surface on a pit wall, removing surface vegetation and the appropriate amount of top soil for a surface grid location, or removing the first several inches of some other type of waste material, the PID of choice should be operating. Furthermore, if a working standard is being utilized to verify performance of the PID for the sampling surface. Once a fresh surface has been exposed, a sample should be quickly obtained, transferred to a screening VOA vial, dispersed, and analyzed. If the maximum response is greater than the working standard (or the running average), the sample or samples taken for laboratory analysis should be prepared using the high-level procedure (i.e., MeOH extraction). If the maximum response is below the working standard, the laboratory sample(s) should be prepared using a low-level procedure (i.e., direct PT/GC or GC/MWS analysis). The total elapsed time between exposing a fresh surface, screening a sample and obtaining and samples for laboratory analysis, should be less than 2 minutes. Samples taken for laboratory analysis should follow the procedures provided in Method 5035. As a precaution against false positive and false negative screening estimates relative to the decision point, locations where screening results are between 0.5 and 2x of the working standard response, could have samples prepared by both high and low level procedures.

6. Method Limitations: For this method of sampling location screening to work, the VOC(s) of site interest must be identified and detectable by photoionization. If more than one analyte is of site interest, and there are large discrepancies (greater than a factor of 2) in photoionization potential, then the range around the decision point where samples are prepared by both high and low level procedures should be increased, proportionally. That is, if the response factor to the VOCs of site interest differ by a factor of 3x, and the analyte with the highest response is used to make the working standard, then laboratory samples from locations where screening results are only 0.3 of the working standard, should be prepared by both procedures. This often will not be a problem for sites contaminated with common chlorinated and aromatic compounds because they have similar photoionization potentials. Another case where this approach may not be effective is for sample matrices that are not readily dispersed in water.

12.4.1.3 *General Subsampling Guidance*

After a fresh surface is exposed to the atmosphere, the subsample collection process should be completed in a minimal amount of time. Removing a subsample from a material should be done with the least amount of disruption (disaggregation) as possible. Additionally, rough trimming of the sampling location's surface layers should be considered if the material may have already lost VOCs (been exposed for more than a couple of minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Removal of surface layers can be accomplished by scraping the surface using a clean spatula, scoop, knife, or shovel.

Subsampling of Cohesive Granular but Uncemented Materials Using Devices Designed to Obtain a Sample Appropriate for Analysis. Subsamples of the appropriate size for analysis should be collected using a metal or rigid plastic coring tool (Fig. 2). For example, coring tools for the purpose of transferring a subsample can be made from disposable plastic syringes by cutting off the tapered front end and removing the rubber cap from the plunger. These smaller coring devices help maintain the sample structure during collection and transfer to the VOA vial or a larger bottle, as do their larger counterparts used to retrieve subsurface materials. When inserting a clean coring tool into a fresh surface for sample collection, air should not be trapped behind the sample. If air is trapped, it could either pass through the sampled material causing VOCs to be lost or cause the sample to be pushed prematurely from the coring tool. For greater ease in pushing into the solid matrix, the front edge of these tools can e sharpened. The optimum diameter of the coring tool depends on the following: size of the opening on the collection vial or bottle (tool should fit inside mouth), dimensions of the original sample, particle size of the solid materials (e.g., gravel-size particles would require large samplers), and volume of sample required for analysis. For example when a 5 g subsample of soil is specified, only a single 3 cm^3 volume of soil has to be collected (assuming the soil has density of 1.7 g/cm^3). Larger subsample masses or more subsample increments are preferred as the heterogeneity of the material increases. After an undisturbed sample has been obtained by pushing the barrel of the coring tool into a freshly exposed surface and then removing the corer once filled, the exterior of the barrel should be quickly wiped with a clean disposable towel. The next step varies, depending on whether the coring device is used for sample storage and transfer or solely for transfer. If the coring tool is used as a storage container, cap the open end after ensuring that the sealing surfaces are cleaned (see Section 12.4.1.4). If the device is solely used for collection and not storage, immediately extrude the sample into a tared VOA vial or bottle by gently pushing the plunger. The volume of material collected should not cause excessive stress on the boring tool during intrusion into the material, or be so large that the sample easily falls apart during extrusion. Obtaining and transferring a sample should be done rapidly (<10 seconds) to reduce volatilization losses. If the vial or bottle contains methanol or another liquid, it should be held at an angle when extruding the sample into the container to minimize splashing. Just before capping, a visual inspection of the lip and threads of the sample vessel should be made, and any foreign debris should be removed with a clean towel, allowing an airtight seal to form.

Devices that Can be Used for Subsampling a Cemented Material: The material requiring sampling may be so hard that even metal coring tools cannot penetrate it. Subsamples of such materials can be collected by fragmenting a larger portion of the material using a clean chisel to generate aggregate(s) of a size that can be placed into a VOA vial or bottle. When transferring the aggregate(s), precautions must be taken to prevent compromising the sealing surfaces and threads of the container. Losses of VOCs by using this procedure are dependent on the location of the contaminant relative to the surface of the material being sampled. Therefore, caution should be taken in the interpretation of the data obtained from materials that fit this description. As a last resort when this task can not be performed onsite, a large sample can be collected in a vapor-tight container and transported to the laboratory for subsampling. Collecting, fragmenting, and adding the sample to a container should be accomplished as quickly as possible.

Devices that Can be Used for Subsampling a Noncohesive Granular Material: As a last resort, gravel, or a mixture of gravel and fines, that can not be easily obtained or transferred using coring tools, can be quickly sampled using a stainless steel spatula or scoop. Typically the collection vial or bottle contains methanol or an aqueous solution, therefore, samples should be dislodged with minimal splashing and without the spatula or scoop contacting the liquid contents. For some solids, a wide-bottom funnel or similar channeling device may be necessary to facilitate transfer to the container and prevent compromising of the sealing surfaces of the container. Caution should be taken in the interpretation of the data obtained from materials that fit this description. Losses of VOCs are likely because of the nature of the sampling method and the noncohesive nature of the material exposes more surface area to the atmosphere than other types of samples. During the sampling process, noncohesive materials also allow for the separation of coarser materials from fines, which can skew the concentration date if the different particle sized, which have different surface areas, are not properly represented in the sample.

12.4.1.4 *Sample Preparation and Preservation*

Low Concentration Samples - VOC Concentrations of 0.5 to 200 μg/kg.

Preservation in the Laboratory: A soil sample may be collected in a hermetically-sealed sample vial such as an EnCore™ sampler and shipped directly to the laboratory for analysis or preservation within 48 hours. Three aliquots per location will be required for analysis. To determine percent moisture collect a soil sample per location in one 2-oz. Glass jar or plastic jar with lid. This is required only if VOCs are the only parameter analyzed; otherwise, moisture content may be determined from soil collected for the determination of semivolatiles and inorganics. *Note: Field personnel will ship VOC samples on the day the sample is collected.*

Preservation in the Field: Collect sample according to procedures outlined in the sampling plan taking care to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial, such as the EnCore™ sampler, the Purge-and-Trap Sampler™, or a; cut plastic syringe with a barrel smaller than the neck of the VOC vial (a disposable syringe is needed for each aliquot collected). Using an appropriate sample collection device, collect approximately 5 g or 3 mL of sample as soon as possible after the surface of the soil or solid material has been exposed to the atmosphere. Wipe the exterior of the collection device with a clean cloth or towel. Always wear gloves whenever handling the tared sample vials. Add the 5 g of soil to the sample vial containing the preservative solution, brush any soil off the vial threads and seal immediately with the Teflon-lined septum screw cap. Using a portable balance which is calibrated in the field using an appropriate weight for the sample containers employed, weigh the sealed vial containing the sample t ensure that 5.0 ± 0.5 g (4.5 to 5.5 grams) of sample were added. Record the weight of the sealed vial containing the sample to the nearest 0.01 g. Collect three aliquots; they should be collected from the same soil stratum of the same section of soil or solid material being sampled, and within close proximity to the location from which the original sample is colleted In addition, if only VOC's are being sampled, and with close proximity to the location from which the sample is collected. In addition, if only VOC's are being analyzed, a fourth aliquot which is collected in the same manner and is not preserved (i.e., no sodium bisulfate) must be obtained for a dry weight determination. Samples should be clearly labeled as to whether they contain sodium bisulfate preservative so that the laboratory will use the closed-system purge-and-trap equipment. Store samples at 4°C.

High Concentration Samples - VOC Concentrations greater than 200 μg/kg

Preservation in the Laboratory: A soil sample may be collected in a hermetically-sealed sample vial such as an EnCore™ sampler and shipped directly to the laboratory for analysis or preservation within 48 hours. Two aliquots per location will be required for analysis. To determine percent moisture collect a soil sample per location in on 2-oz. glass jar or plastic jar with lid. This is required only if VOCs are the only parameter analyzed; otherwise, moisture content may be determined from soil colleted for the determination of semivolatiles and inorganics. With high concentrations only, a bulk sample may be collected, filling the sample container as full as practical in order to minimize the headspace. The collection of a bulk sample by using a spatula-type device to completely fill a storage and transportation bottle fail to control surface area exposure, for both cohesive and noncemetitious materials. Moreover, in the process of filling a bulk sample bottle to capacity, the sealing surfaces often become compromised (dirty), preventing a vapor-tight seal during storage. If a bulk sample is collected, usually less than 10 percent of the in-situ contamination will be determined by the analytical procedure. Note: Since this procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling. Store samples on ice at 4°C. *Note: Field personnel will ship VOC samples on the day the sample is collected.*

Preservation in the Field: using an appropriate sample collection device, collect approximately 5 g of sample as soon as the surface of the soil or other solid material has been exposed to the atmosphere. Carefully wipe the exterior of the sample collection device with a clean cloth. Using the sample collection device, add about 5 g of soil to the vial containing 5 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Using a portable balance which is calibrated in the field using an appropriate weight for the sample containers employed, weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. Record the weight of the sealed vial containing the sample to the nearest 0.01 g. Collect two aliquots; they should be collected from the same soil stratum of the same section of soil or solid material being sampled, and within close proximity to the location from which the original sample is collected. In addition, if only VOAs are being analyzed, a third aliquot which is unpreserved (i.e., no methanol) and collected in the same manner must be collected for a dry weight determination. Samples should be clearly labeled as to whether they contain methanol so that the laboratory will use the aqueous surge-and-trap procedure in Method 5030. Store samples on ice at 4°C.

Oily Waste Samples

When oily waste samples are known to be soluble in methanol or polyethylene glycol (PEG), the sample may be collected in a vial containing such a solvent, using procedures similar to those described for the high concentrated soil sample. When the solubility of the oily waste is not known, the sample should either be collected in a vial without preservative, as described for the high concentrated sample, or a visual evaluation of the solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described for the high concentrated sample preserved in the field. Otherwise, collect an unpreserved sample as described for the high concentrated sample preserved in the laboratory. All samples for volatiles analysis should be cooled to approximately 4°C.

12.4.1.5 *Sample Shipping Procedures*

Methanol is considered a hazardous material therefore shipping of the sample containers is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment set in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulation must be followed when shipping methanol between the laboratory and the field. Consult the above documents or the shipping company for additional information.

The shipment of the quantity of methanol used for the sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

(1) The maximum volume of methanol in a sample container is limited to thirty (30) mls.

(2) The sample container must not be full of methanol.

(3) The sample container must be stored upright and have the lid held securely in place. The mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container.

(4) Sample containers must be packed in absorbent material capable of absorbing spills from leaks or breakage of the sample containers.

(5) The maximum sample shuttle weight must not exceed 64 pounds.

(6) The maximum volume of methanol per shipping container is 500 mls.

(7) The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.

(8) The package must not be opened or altered until no longer in commerce.

12.4.1.6 *Safety*

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should always be stored in a cooler with ice at all times.