

0170.000      **IMPLEMENTATION OF EPA METHOD 5035 - SOIL PREPARATION FOR EPA  
METHODS 8015B, 8021B AND 8260B.**

**LEVEL TWO**                      **Arizona Department of Environmental Quality**

**Originator:**                      David Esposito, Director  
   ADEQ Waste Programs Division

**Contact:**                              Kenyon C. Carlson, Manager  
  
   ADEQ QA/QC Unit

**Issue Date:**                      April 19, 2000

**Next Scheduled Review Date:**                      2 years from issuance

**Authority:**                              Arizona Revised Statutes (A.R.S.) §49-104 (A)

**I.      PURPOSE**

The EPA Office of Solid waste promulgated Method 5035, *Closed-System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples* (Attachment 1), in June 1997 in SW-846, Update III. The Arizona Department of Health Services (ADHS) Office of Laboratory Licensure, Certification and Training adopted Method 5035 in May 1998 and Method 5035 became enforceable on March 1, 1999 in Arizona. The collection and analytical procedures for the approved method are flexible and, without further guidance, could result in multiple interpretations.

This policy establishes the sampling options and the preservation holding time requirements for individual programs within the ADEQ's Waste Programs Division. This policy is necessary to provide an understanding of the options set forth by the method and the limitations imposed on specific field sampling requirements. This policy does not eliminate the need to read and understand EPA Method 5035. The method, in conjunction with this policy, will provide a technically defensible and consistent approach to sampling for Volatile Organic Compounds (VOCs) in soils.

II. DEFINITIONS: (FOR PURPOSES OF EPA METHOD 5035 ONLY).-

1. Sample Preservation: The addition of methanol or sodium bisulfate to an unpreserved sample in the field or in the laboratory.
2. Sample Extraction: The addition of methanol to an unpreserved sample in the laboratory. After extraction, the methanol is transferred to a vial and can be stored at 4°C(± 2°C) until analysis.
3. Hermetically Sealed: For the purposes of this policy a hermetically sealed container shall be defined as a sample storage device that consistently shows less than 10% loss from volatilization over the intended storage holding time(usually 14 days)or a minimum of 48 hours for the compounds of concern at a given site.
4. Sample Freezing: A preservation technique in which the sample is frozen and stored at 0°C (32°F), or lower upon receipt at the laboratory. Blue ice is unacceptable.
5. Calcareous Soil: A soil whose content of carbonate is sufficient to cause effervescence when tested with hydrochloric acid.  
(Reference: Bates R. L. and Jackson J. A.. (1987). Glossary of Geology. (3rd ed. )Alexandria: American Geological Institute.)

### III. POLICY

Method 5035 is structured as a 2-tier approach for low and high concentration sampling<sup>1</sup>. Preservation is recommended for both low and high contaminant concentrations as stated in the Method. Based upon program requirements, preservation can be conducted in the field or subsampled in an EnCore™ Sampler and the sample preserved in accordance with sample handling.

#### **A. Sample collection options for low reporting limits (<200 Ag/kg)**

##### **I. Methanol Preservation-**

EPA has permitted the use of methanol preservation for low level analysis if the target analyte(s) can be quantitated below 200 µg/kg. As a result, laboratories must demonstrate their ability to detect below 200 µg/kg to the client and ADHS. Samples preserved in the field with methanol using a 40 mL glass VOA vial with a plastic screw cap and a Teflon septa must be analyzed within 14 days from the time of sample collection.

##### **ii. EnCore™ Sampler-**

The sample can be collected using either a 5-gram or 25-gram EnCore™ Sampler. The sample must be stored at 4°C (±2°C) and preserved or extracted within 48 hours if not preserved. Approved preservatives include either methanol or sodium bisulfate. Once preserved, the sample must be analyzed within 14 days from the time of sample collection. The EnCore™ Sampler 48-hour preservation hold time as required in the method applies only to the EnCore™ Sampler option and is based on manufacturers' studies. Freezing the unpreserved sample in the EnCore™ Sampling device can extend the

<sup>1</sup>Refer to EPA Method 5035 (Attachment 1) and Regional Interim Policy for Determination of Volatile Organic Compound (VOC) Concentrations in Soil and Solid Matrices, June 23, 1999 (Attachment 2).

holding time up to seven days (e.g., 48 hours unfrozen and 5 days frozen.)

iii. Sodium Bisulfate Preservation-

Samples preserved in the field with sodium bisulfate must be analyzed 14 days from the time of sample collection. This technique should be used if detection limits in the range of 2 - 5 µg/kg are desired. Calcareous samples, however, may effervesce upon contact with the sodium bisulfate preservative solution (thereby liberating the volatile gases) and compromise the integrity of the sample. In these instances, sodium bisulfate preservative solution cannot be utilized to attain the lower reporting levels and one of three alternative sample collection methods must be employed.

- a) The sample can be collected in a VOA vial containing 10 mL of reagent grade water, sealed with a plastic screw cap containing a Teflon septa and stored at 4°C (± 2°C) This sample must be analyzed within 48 hours from the time of sampling using a closed system purge and trap.
- b) The sample can be collected in a dry VOA vial, sealed with a plastic screw cap containing a Teflon septa and stored at 4°C (± 2°C) Once at the lab, water must be introduced through the septa and analyzed by closed purge and trap within 48 hours from the time of sample collection. Freezing the unpreserved sample can extend the holding time an additional 5 days for a total of 7 days from the time of sample collection.
- c) The sample can be collected in an EnCore™ Sampler, stored at 4°C (± 2°C) and analyzed within 48 hours from the time of sample collection. Freezing the unpreserved sample can extend the holding time up to seven days.

iv. Bulk Sampling-

The rationale for the collection of bulk samples must be clearly documented and approved by the appropriate program in a work or sampling plan or other written

communication with ADEQ. If samples are not preserved in the field, the reasons for not preserving must be clearly documented and approved by the relevant program.

ADHS rules require laboratories to flag data generated from samples that have not been preserved in the field or have not been collected in recommended containers if the reporting levels are below 200 µg/kg.

**B. Sample collection options for high reporting limits (>200 µg/kg):**

I. Methanol Preservation-

This technique may be used if the reporting limits are above 200 µg/kg. Samples preserved in the field with methanol using a 40 ml glass VOA vial with a plastic screw cap and a Teflon septa must be analyzed within 14 days from the time of sample collection.

ii. EnCore™ Sampler-

The sample can be collected using an EnCore™ Sampler. Methanol must be added within the 48-hour period immediately following sample collection. The EnCore™ Sampler 48-hour preservation hold time as required in the method is applicable specifically only to the EnCore™ subcoring device and is based on the manufacturers' studies. After collection the sample must be stored on ice at 4°C (±2°C) until analyzed. Freezing the unpreserved sample in the EnCore™ sampling device can extend the holding time up to seven days (e.g., 48 hours unfrozen and 5 days frozen.) Once the sample is preserved, it must be analyzed within 14 days from the time of sample collection.

iii. Bulk Sampling-

The rationale for collection of bulk samples must be clearly documented and approved by the

appropriate program in a work or sampling plan or other written communication with ADEQ. If samples are not preserved in the field or subsampled in EnCore™ Samplers, the reasons for not preserving must be clearly documented and approved by the relevant program.

Significant volatile loss occurs when samples are collected in glass jars and transported to a laboratory for analysis<sup>2</sup>. Therefore, **glass jars with Teflon<sup>®</sup> - lined lids containing no preservative ARE NOT ACCEPTABLE** for the collection of soil for VOC analysis, unless otherwise specified in this policy (Program Specific Requirements) or prior approval has been received from the appropriate program.

### III. Program Specific Requirements<sup>3</sup>:

#### 1. *WQARF, Hazardous Waste Compliance, Solid Waste Programs*

When utilizing the field preservation option of the 5035 method, samples must be preserved immediately after collection with minimal handling to be considered reliable compliance samples. Samples may be collected and held on ice at 4°C (±2°C) for a maximum of 2 hours before preserving or analyzing the sample. This option of holding samples on ice for up to 2 hours is accepted, but not encouraged, due to the known volatile loss over time.

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<sup>2</sup>Siegrist, R.L., and P.D. Jennsen, 1990. Evaluation of Sampling Method Effects of Volatile Organic Compound Measurements in Contaminated Soil, *Environmental Science and Technology*, Vol.24, pp. 1387-1392.

<sup>3</sup>For specific programs, a sample collected in a brass/steel sleeve is acceptable under the conditions noted in Section IV. The brass or steel sleeves must have each end covered with a sheet of Teflon, aluminum foil(aluminum is optional, but preferred) and sealed with a plastic cap. The plastic caps must be secured and the capped sleeve should be placed in a plastic ziplock bag which is then taped to ensure the caps are secure. The use of tape to bind the cap to the end of the sleeve is discouraged. The length of time a sample can be held in this container is finite and subject to specific program requirements set forth in Section V.

Samples collected and preserved or analyzed after 2 hours will be considered bulk samples and not suitable for compliance purposes. Data generated from samples collected and transported to a laboratory in this manner has limited compliance value and may not be accepted by the above referenced programs.

**2. Hazardous Waste Inspections and Emergency Response Programs**

For planned field sampling events, samples must be preserved immediately after collection, with minimal handling, to be considered compliance samples. The sample may be held on ice at 4°C ( $\pm 2^\circ\text{C}$ ) for a maximum of 2 hours before preserving or analyzing the sample.

For unanticipated sampling events, where significant difficulties exist for preserving samples onsite, bulk soil samples may be collected and stored at 4°C ( $\pm 2^\circ\text{C}$ ) but must be preserved within 72 hours with the approval of the program.

**3. UST Program**

When site-specific sampling conditions prevent the use of appropriate sample collection and preservation techniques as defined in Section I or Section II, samples may be submitted in properly sealed brass sleeve containers maintained at 4°C ( $\pm 2^\circ\text{C}$ ) for laboratory analysis of VOCs. The laboratory must document sample holding time and flag the associated analytical results if sample preservation or extraction exceeds 48 hours, regardless of the reporting limit. Reasons for lack of field preservation within the 48 hour period and submittal of bulk samples for laboratory analysis must be clearly documented.

**IV. Quality Control for unpreserved samples:**

Unpreserved samples submitted to the laboratory should have matrix spikes and surrogates added directly to an aliquot of the sample before extraction. The laboratory should be requested to provide a narrative describing

the procedures for sample spiking and flag all data in which the matrix was not directly spiked prior to extraction.

**V. Example of Holding Time Calculations for Frozen Samples:**

Example 1 Sample is placed in a vial without chemical preservative in the field and stored at 4°C (±2°C).

The sample must be analyzed within 48 hours of collection.

Example 2 The sample is collected in a hermetically sealed subcoring and storage device in the field, stored at 4°C (±2°C) and transferred into a vial without chemical preservative in the laboratory.

The sample must be analyzed within 48 hours of collection.

Example 3 The sample is collected in a hermetically sealed subcoring and storage device, transported/stored at 4°C (±2°C), frozen at the laboratory 18 hours after collection, thawed (at ambient temperature) after 4 days and transferred into a vial without a chemical preservative in the laboratory.

The sample must be analyzed within 30 hours from the time the sample is defrosted to 4°C (±2°C) .

48 hours allowed before analysis - 18 hours before freezing = 30 hours allowed from thawing (at ambient temperature) to analysis.

Freezing can only extend the holding times for unpreserved samples. Freezing is an alternative to preserving samples in the field. Freezing can never extend the holding times of samples beyond the analytical methods required holding time. (Ex. Freezing cannot extend the holding time from 14 days to 19 days).

## VI. RESPONSIBILITY

All staff in the respective Waste Programs Division programs are responsible for knowledge and implementation of this policy. Supervisors are responsible for ensuring that the information contained in this policy is consistently and equitably applied by all staff. It is the responsibility of the sampler to inform the laboratory receiving personnel which program requirements are appropriate for the sample.

0000.000 IMPLEMENTATION OF EPA METHOD 5035 - SOIL PREPARATION

LEVEL TWO: Waste Programs Division

**Originator:** David Esposito, Director  
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**Contact:** Kenyon C. Carlson, Manager ADEQ  
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**Issue Date:**

**Next Scheduled  
Review Date:**

APPROVED BY

David Esposito, Date 4/20/00  
Director  
Waste Programs Division

Mark Santana Date 4/21/00  
Administrative Counsel  
Office of Administrative Counsel

The Policy Review Committee has posted, reviewed and accepted this policy by motion as of April 19, 2000.

Juanita Guidry Copeland, Date 4/21/00  
Acting Policy Coordinator

# Attachment 1

## EPA Method 5035

See SW-846

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION IX  
75 Hawthorne Street  
San Francisco, CA 94105-3901

June 23, 1999

MEMORANDUM

SUBJECT: Regional Interim Policy for Determination of Volatile Organic Compound (VOC) Concentrations in Soil and Solid Matrices.

FROM: Nora McGee, Assistant Regional Administrator  
USEPA Region 9

TO: USEPA Region 9 Personnel and Parties Collecting Environmental Measurements Under Regional Programs.

Purpose

Appropriate methodologies to minimize volatilization and biodegradation losses in solid matrices have not been consistently implemented throughout Region 9. This memorandum articulates the Region's policy on the adoption of sampling and laboratory methodologies for the collection of volatile organic compound (VOC) data from soil or solid matrices. USEPA SW846, Update III, Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," incorporating procedures to minimize VOC losses was finalized by USEPA in June 1997. This Region 9 policy requires the use of Method 5035, or an equally or more effective method, for the collection of representative and precise data for VOCs in soil and solid matrices. Additionally, this policy was developed to be consistent with the Agency's Data Quality Objectives (DQO) Process (outlined in "Guidance for the Data Quality Objectives Process," USEPA QA/G-4, September 1994) by allowing for a graded approach through the collection of representative data that meets project data quality needs.

Policy

Scope and Applicability

Environmental data collection activities performed under USEPA Region 9 programs for the determination of VOC concentrations in soil and solid matrices.

This policy is applicable to data collection activities conducted by USEPA staff and contractors, USEPA grantees, Federal Facilities, entities complying with USEPA regulatory requirements and/or other entities producing data for USEPA decision making. This includes data being collected under ongoing quality assurance plans and sampling plans.

### Time Frame for Implementation

This policy should be adopted quickly and to the maximum practicable extent. Cases where it is not practicable to implement this policy should be brought to the attention of the USEPA Region 9 QA Office. This is being put forth as an interim policy, as USEPA is still evaluating technical information to further refine procedures for minimization of VOC losses. Please note, an amendment to this policy may be required.

### Statement of Policy

Methods for the collection and analysis of VOCs in soil or other solid matrices must minimize volatile losses. Because USEPA SW-846 Method 5035 does not rigorously dictate specifics of field sample collection and laboratory sample handling protocols, project specific procedures to minimize volatile losses must be developed and be included in the site/program quality assurance project plan (QA-PP) or sampling and analysis plan (SAP). USEPA SW-846 Method 5021 "Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis," also incorporates procedures to minimize volatile losses. However, Method 5021 should be used with caution, as it can be reasonably interpreted and performed in a way which does not prevent loss of VOCs. USEPA Region 9 considers the following practices as minimum requirements to reduce volatile losses in soil samples:

1. Samples are handled as intact<sup>2</sup> soil cores in the field and laboratory.
2. Samples are stored in containers which can be reliably sealed to prevent volatilization losses<sup>3</sup> over the project specified analytical holding time.
3. Samples are analyzed or chemically, acid or methanol, preserved within 48 hours of collection, if any contaminant may undergo biodegradation.

Exposure of the sample core to the atmosphere in the field and laboratory should be minimized<sup>4</sup>.

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ASTM Method D4547-98 "Standard Guide for Sampling Waste and Soils for VOCs," is a good reference for VOC sampling protocols.

- <sup>2</sup> Soils should always be collected and transferred using a coring device, such as a metal sleeve or cut off syringe. Use of transfer devices, such as spatulas, is not acceptable either in the field or laboratory.
- <sup>1</sup> Volatilization losses from sampling/storage containers must be less than what would be expected from a volatile organic analysis vial with a Teflon/silicon septa stored for 14 days, unless project DQOs require more stringent requirements.
- <sup>4</sup> Field sub-cores should be taken immediately upon exposing the soil core to ambient conditions. Sub samples should be directly extruded into the analysis containers. Total exposure of samples to ambient conditions should not be more than 15 seconds.

USEPA Region 9 will consider exceptions to this policy on a case-by-case basis. All deviations from procedures outlined in Method 5035 should be documented in a QA-PP or a SAP which must be submitted to, and approved by, the Region 9 QA Office. Additionally, the party responsible for data collection must demonstrate that the methodologies proposed will result in data that meet project/program data quality objectives (DQOs).

#### Additional Considerations

**Field Laboratories:** The use of field laboratories, that analyze samples within several hours of collection, is an excellent choice to prevent loss of volatiles in transit and storage. However, the sample collection and analysis procedures used must prevent volatilization losses and comply with requirements 1 and 4 articulated in the Statement of Policy. Additionally, the quality control criteria and quality assurance system used by a field laboratory must be adequate for generation of data which will meet project DQOs.

**Addition of Surrogates and Matrix Spiking Compounds in the Field:** The most appropriate time for addition of analytical surrogate and matrix spiking compounds into soils is prior to sample extraction, by water or a solvent. Method 5035 does not incorporate the addition of the compounds prior to extraction in the field. Because this is an important control check on the analytical process, which begins at extraction, for some project/program DQOs it may be appropriate to incorporate a procedure which adds surrogate and/or matrix spiking compounds prior to extraction.

**Holding Times:** The holding time for preserved soil samples should be interpreted as 14 days from the time of sample collection(stored at  $4\pm 2^{\circ}\text{C}$ ). Due to potential biodegradation losses, samples stored in sealed containers, but not chemically preserved, should not be stored for more than 48 hours. On a project/program specific basis, USEPA Region 9 will consider other alternatives to extend the holding time of soils that have not been chemically preserved (see Attachment A). Holding time will be considered as cumulative (see Attachment B for holding time examples). Exceptions should be documented in a QA-PP or a SAP submitted to and approved by the Region 9 QA Office.

**Unconsolidated Solid Matrices:** Solid Matrices that are not amenable to the use of a coring technique should be collected in such a way as to preserve the integrity of the sample matrix. Transferring of these soils with spatulas or similar devices into sampling containers is discouraged as this disrupts the sample pore spaces and greatly increases the sample surface area available for volatilization. For soil piles, fresh soil at an adequate depth should be sampled.

**Calcareous Soils:** Method 5035 notes that, "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." Calcareous soils that effervesce on contact with the low-level preservative solution should be collected using an alternative preservation technique (see Attachment A).

**Soil Gas:** This policy is not intended to address the role of soil gas in the environmental decision making process. The Region recognizes that soil gas data is used extensively, in USEPA Region 9, for site decision making and in some cases soil gas is the preferred tool for gathering data on subsurface conditions. However, there are also scenarios where soil gas data are unacceptable for agency decision making (e.g., in excavated soils and when determining disposal options).

**Drilling Techniques:** This policy does not address the impact of drilling techniques on the collection of a representative VOC sample. Site/program QAPPs and SAPs should address the impact of all collection techniques on sample integrity and select those appropriate for the DQOs. Potential VOC losses due to drilling techniques include, but are not limited to: sample compression and loss of pore space; air introduction into the sample matrix; heat introduced in the drilling process; and volatilization from prolonged periods in a non-hermetically sealed sampling apparatus.

## Background

Traditional practices for the sampling and analysis of volatile organic compounds (VOCs) in soil have been shown to have a significantly low bias of inconsistent magnitude (Grant, 1996) from volatilization (Hewitt, 1996) and biodegradation (Hewitt, 1994). Based on this and other research, the USEPA modified the methodology in SW846 for collection and analysis of volatiles in soil. Soil was deleted as an option from Method 5030 and Method 5035 and Method 5021 were added. These methods provide for handling of samples as intact soil cores, chemical preservation techniques, storage of samples in hermetically sealed containers and minimization of analyte losses due to direct volatilization (both in the field and the laboratory) and biodegradation.

"Traditional" collection techniques, such as transferring soils to a glass jar with minimal head space and collecting samples directly into a brass sleeve (e.g., CA Split Spoon) do not yield accurate or consistent results. It has been specifically demonstrated that capped brass sleeves show significant losses. Hewitt and Lukash (Hewitt, 1996) demonstrated capped sleeves can show substantial losses in less than one day. Hewitt and Lukash also demonstrated volatile losses in uncapped core liners of up to 90% in less than 40 minutes for trichloroethene (TCE). Because other analytes and matrix types can have higher mobility than those tested, substantial losses may occur in an even shorter period of time. Grant, Jenkins and Mudambi (Grant, 1996) examined split sampling results from a cross section of laboratories. For VOCs in soil they noted that, "The magnitude of this scatter [for a typical data comparison] is so large that it is impossible to recommend effective limits of acceptability. Instead, we believe that steps are urgently needed to improve data quality." Hewitt noted (Hewitt, 1994) that biodegradation of Benzene and Toluene in soil samples stored in sealed glass ampules at 4 C for 14 days could be substantial, demonstrating a need for chemical preservatives. Turriff and Reitmeyer (Turriff, 1998) demonstrated that a variety of soil matrices could be held for 48 hours at 4 C, in sealed zero headspace containers, without substantial VOC losses. Additionally, Turriff

and Reitmeyer demonstrated that freezing was an option to extend holding times of En Core™1 sampling devices. Because volatile losses have been linked to disturbance of the soil matrix and exposure to the atmosphere, samples should be handled in intact soil cores and stored in hermetically sealed vessels in both the field and the laboratory.

This USEPA Region 9 policy is based on the best scientific information available at this time and is subject to further clarifications and additions as other research becomes available. If you have any questions please call Vance Fong at 415 744-1492 or Mathew Plate at 415 744-1493.

## **References**

**Hewitt, A.D.** (1994) Concentration Stability of Four Volatile Organic Compounds in Soil Subsamples. US Army Cold Regions Research and Engineering Laboratory, Special Report 946.

**Grant, C.L., T.F. Jenkins and A.R. Mudambi** (1996) Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories, Corps of Engineers Archived Data. US Army Cold Regions Research and Engineering Laboratory, Special Report 96-9.

**Hewitt, A.D. and J.E. Lukash** (1996) Obtaining and Transferring Soils for In-Vial Analysis of Volatile Organic Compounds. US Army Cold Regions Research and Engineering Laboratory, Special Report 96-5.

**Turriff, D. Ph.D. and C. Reitmeyer** (1998) Validation of Holding Times for the EnCore™ Sampler. En Novative Technologies, Inc.

## Attachment A

**Preservation Alternatives:** The following are preservation alternatives that may be appropriate for some projects/programs and are subject to project/program specific approval by the USEPA Region 9 QA Office.

**Freezing of unpreserved samples:** It has been shown in several studies that freezing of unpreserved soils is an effective means of slowing the biodegradation process. At this time, USEPA Region 9 will accept freezing of unpreserved soils as a method to extend holding times up to seven days on a project specific basis. While there is some evidence that freezing for longer periods may also be acceptable for some data needs, USEPA Region 9 does not believe that the current scientific evidence supports a longer holding time for frozen samples in most cases. Samples should be frozen in containers that have an air tight seal and can maintain this seal while frozen. Because water expands in the freezing process, VOA vials with water or samples with extremely high moisture contents may rupture the storage container.

Preservatives: Acids other than sodium bisulfate may be used to preserve low level samples. The choice of an alternative acid should be made in consultation with the USEPA Region 9 QA Office. In all cases the preserved sample pH should be 2.

**Sampling Containers:** Currently the Region recognizes three sample collection/storage alternatives which can be used (other than acid/water or methanol, as specified in Method 5035).

1. A VOA vial with 5 mL of water without preservative and approximately 5 g of sample. Which must be analyzed within 48 hours of collection by closed system purge and trap.
2. A VOA vial with approximately 5 g of sample. Water must be introduced through the septa at time of analysis by closed system purge and trap. Sample must be analyzed within 48, hours of collection if stored at  $4\pm 2^{\circ}\text{C}$  or 7 days if frozen. (This alternative must be approved on a project specific basis.)
3. An En Core<sup>TM</sup> I sampler which is analyzed or preserved within 48 hours of collection if stored at  $4\pm 2^{\circ}\text{C}$  or analyzed within 7 days if frozen. (Freezing of En Core<sup>TM</sup> samplers must be approved on a project specific basis.)

If requested, USEPA Region 9 QA Office will consider the applicability of other sampling containers/devices that have been demonstrated, with appropriate supporting documentation, to be adequate for collection and storage of VOCs.

## Attachment B Examples of Holding Time Policy

- Example 1      Sample is placed into a vial without chemical preservative in the field (due to effervescence) and stored at  $4\pm 2^{\circ}\text{C}$ .
- Sample must be analyzed within 48 hours of collection.
- Example 2      Sample is collected into a hermetically sealed sub-coring and storage device in the field, stored at  $4\pm 2^{\circ}\text{C}$  and transferred into a vial without chemical preservative in the laboratory.
- Sample must be analyzed within 48 hours of collection.
- Example 3      Sample is collected into a hermetically sealed sub-coring and storage device, transported/stored at  $4\pm 2^{\circ}\text{C}$ , frozen at the laboratory 28 hours after collection, defrosted after 2 days and transferred into a vial without chemical preservative in the laboratory.
- Sample must be analyzed within 20 hours from the time the sample is defrosted to  $4\pm 2^{\circ}\text{C}$ .
- 48 (hours allowed) - 28 (hours before freezing) = 20 (hours allowed from defrosting to analysis)