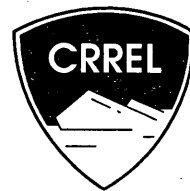


**SPECIAL REPORT**

**94-2**



# **Vapor-Fortified QA/QC Samples for the Analysis of Volatile Organic Compounds**

Alan D. Hewitt

February 1994

**Abstract**

Vapor fortification is a precise treatment method for preparing soil subsamples for the analysis of volatile organic compounds (VOCs). This method of spiking is unique in that the fortification level is soil-type specific and the treatment mechanism is analogous to how soil in the vadose zone becomes contaminated by vapors from liquid VOC point sources. Soil subsamples treated by vapor fortification and sealed in glass ampoules until analysis have shown both good precision within batch and among batches, and analyte concentration stability for a month or longer. Vapor-fortified quality assurance/quality control (QA/QC) soils are more environmentally representative than soils fortified by liquid spiking procedures for the analysis of VOCs in vadose zone samples.

For conversion of SI metric units to U.S./British customary units of measurement consult ASTM Standard E380-89a, *Standard Practice for Use of the International System of Units*, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

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# Special Report 94-2



**US Army Corps  
of Engineers**

Cold Regions Research &  
Engineering Laboratory

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Alan D. Hewitt

February 1994

Prepared for  
U.S. ARMY ENVIRONMENTAL CENTER  
SFIM-AEC-TS-CR-94030

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## **PREFACE**

This report was prepared by Alan D. Hewitt, Research Physical Scientist, Geological Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory, Hanover, New Hampshire.

Funding for this work was provided by the U.S. Army Environmental Center (formerly the U.S. Army Toxic and Hazardous Materials Agency), Martin H. Stutz, Project Monitor. The author thanks Dr. C.L. Grant and Dr. T.F. Jenkins for critical review of the text and Chad Pidgeon and Lawrence Perry for analyzing some of the samples.

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# Vapor-Fortified QA/QC Samples for the Analysis of Volatile Organic Compounds

ALAN D. HEWITT

## INTRODUCTION

The validity of laboratory estimates of analyte concentrations in environmental samples is initially dependent on analytical calibration. Thereafter, validity is monitored during routine analysis of real samples by reference to the results of accompanying quality assurance (QA) and quality control (QC) samples. For this system to work effectively, QC and QA reference samples must be available in a stable form and with accurately known concentrations. In the case of volatile organic compounds (VOCs) in soils, preparation of such reference materials is very difficult.

As defined in "A Rationale for the Assessment of Errors in the Sampling of Soils" (EPA 1989), QA is usually intended to aid in data judgment by an independent party, whereas QC provides a means to qualify data during the analysis. For this reason, QA samples are usually analyzed "blind" and sometimes "double blind" to ensure that the analyst is unaware of the analytes present and their concentrations. In contrast, analyte concentrations in QC samples are known, so the analyst can detect problems and take appropriate measures before generating large quantities of erroneous data. These data qualifiers play important roles in the investigation and remediation of hazardous waste sites and ultimately control the allocation of millions of dollars annually.

The wide use and subsequent improper disposal of petroleum products and chlorinated solvents has made the group of chemical compounds referred to as VOCs our most ubiquitous environmental hazardous waste problem (Plumb and Pitchford 1985, Zarrabi et al. 1991). When dealing with the analysis of VOCs, QC practices include instrument tuning and calibration, and QA can include within-laboratory analysis of duplicate samples, independent laboratory split-sample analysis, and spike and re-

covery tests. Due to the difficulty in preventing losses of VOCs from soils during sampling and storage, the analysis of both sample duplicates and splits is suspect (Hewitt, in press-a). Moreover, despite the large number of vadose zone soil samples routinely characterized for VOCs, no performance evaluation materials presently exist for this matrix (Zarrabi et al. 1991).

As a result of these problems and others, the most commonly practiced QA/QC procedure has been sample spike and recovery tests (Maskarinec et al. 1989). Typically this is performed by adding the analytes of interest dissolved in a carrier solvent (i.e., methanol) to 5 mL of water, which is then transferred to a purge vial that contains VOC-blank soil. Due to complications associated with volatilization losses, sample spiking and analysis typically are performed in a continuous sequence, while the purge tube containing VOC-blank soil remains attached to the purge-and-trap manifold. This spike and recovery procedure has only a very short analyte/soil contact period (minutes), so it limits analyte distribution and sorption and fails to address the effects of solvents used to carry the analytes. Thus, this procedure tests mainly the instrumental determination step and largely ignores analyte extraction from soil. In addition, since this procedure is usually performed by the analyst, the analytes present and their concentrations are known.

Performance evaluation materials for the analysis of VOCs in soil, prepared by vapor fortification, offer several advantages to solution spike and recovery tests (Hewitt 1993). In this method soil subsamples are fortified with a group of volatile organic analytes by exposing them in a closed desiccator to vapors from an organic solution that contains the analytes of interest. During exposure, each soil subsample is contained in an open glass ampoule that can be heat-sealed after a treatment period of

several days. Impregnating soil samples with VOCs in this manner is similar to how vadose zone soils are contaminated by vapors generated from a liquid point source (Hewitt, in press-b) and requires an extraction step prior to instrumental analysis.

As will be seen, analyte concentration attained using vapor fortification is precise both within batch and among treatment batches, is soil-specific in terms of both treatment concentration and solvent extraction efficiency, and sorbed analyte concentrations remain stable for extended periods (30 days or more).

## MATERIALS AND METHODS

### Materials

Four soil matrices were used in this study. Two of the soils serve as reference matrices for the U.S. Army Environmental Center (formerly the U.S. Army Toxic and Hazardous Materials Agency):

- A marine sediment from Tampa Bay, Florida (TB), and
- A composite soil from the Rocky Mountain Arsenal (RMA) in Denver, Colorado.

The other two were site-specific soils:

- A soil from Point Barrow, Alaska (PBA), and
- A soil from the Cold Regions Research and Engineering Laboratory in Hanover, New Hampshire (CRREL).

Table 1 shows the percentage of organic carbon and clay in these soils.

Table 1. Soil characteristics.

Characteristic	TB	RMA	PBA	CRREL
% organic carbon	0.311	0.053	6.69	0.344
% clay	<5	NA	20.1	<5

The VOC analytes selected for soil treatment were trans-1,2-dichloroethylene (TDCE), trichloroethylene (TCE), benzene (Ben), and toluene (Tol). A stock solution for both instrument calibration and soil matrix fortification containing these four analytes was prepared fresh on a monthly basis by adding the following analyte quantities to a 100-mL volumetric flask and diluting to volume with methanol (MeOH): 0.60 g Tol, 0.59 g TCE, 0.50 g TDCE, and 0.35 g Ben. A mixture of 25 mL of this MeOH stock solution and 25 mL of tetraethylene glycol dimethyl ether (tetraglyme) was used when

preparing the vapor fortification solution. This fortification solution mixture resulted in treatment levels between 5 and 70  $\mu\text{g/g}$  for all of the analytes of interest with these four different soils. This range of concentration was desirable so that both aqueous and MeOH solvent extractions could be analyzed by headspace gas chromatography (HS/GC). All of the chemicals were reagent-grade quality or better.

Laboratory supplies consisted of a large glass desiccator (5.6 L), a desiccant (anhydrous  $\text{CaSO}_4$ ), 1.0-mL glass ampoules (Wheaton), a ring stand with metal tension clamp, a 60-mL glass bottle, plastic funnel, stainless steel spatula, top loading balance, 5-mm-diameter glass beads, and a plumber's propane torch.

### Soil subsample fortification and handling

All test soils were air-dried, and in almost every case they were sieved through a 30-mesh screen and mixed thoroughly before subsampling. Only the PBA soil used for the soil-specific vapor fortification study (Table 2) was not sieved prior to subsampling. A stainless steel spatula and plastic funnel were used to transfer soil subsamples into the 1.0-mL glass ampoules. The mass of PBA and CRREL soil subsamples were weighed to  $1.25 \pm 0.01$  g, while the RMA soil and TB sediment subsamples were weighed to  $2.00 \pm 0.01$  g. These masses of soil filled the glass ampoules to a point just below the marked (colored), prescored line on the vial's neck.

As many as 15 soil-filled ampoules were placed in a 5.6-L desiccator with a dish of anhydrous  $\text{CaSO}_4$ . After two days of desiccation the  $\text{CaSO}_4$  was replaced with an open 60-mL glass bottle containing 50 mL of fortification solution prepared by combining 25 mL of the MeOH stock solution with 25 mL of tetraglyme. After seven days of equilibration, the desiccator was opened and 5-mm-diameter glass beads were rapidly placed on top of each of the ampoules, forming temporary caps. Then, as quickly as possible, each ampoule was put in a metal tension clamp. Holding the ampoule just below the glass bead, the neck was heat-sealed using a plumber's propane torch. A sharp-pointed tip was created when sealing the ampoules to facilitate ease of breaking when preparing for analysis. Details on the development of this procedure and its performance have been documented elsewhere (Hewitt 1993, in press-b).

### Analysis

Prior to analysis an ampoule was placed with its tip pointing downward into a 40-mL volatile or-

ganic compound analysis (VOA) vial equipped with an open-faced cap and Teflon-lined silicone rubber septum. The VOA vial into which the ampoule was placed contained either 20 mL of MeOH or 30 mL of water as extraction solvents. The ampoule was then broken inside the closed VOA vial and the soil completely dispersed by vigorous shaking by hand for approximately 2 minutes. The VOA vials containing water were analyzed by aqueous extraction HS/GC, and those containing MeOH were analyzed either by HS/GC or purge-and-trap/gas chromatography/mass spectrometry (PT/GC/MS) following Method 8240 in SW-846 (EPA 1986). Only small quantities (100  $\mu$ L or less) of the MeOH extractant were transferred for analysis by either of these two analytical methods. The analytical calibration standards for HS/GC analysis were prepared from the same stock standard used to make the vapor fortification treatment solution. Working standards were prepared by diluting the stock standard with MeOH and then transferring small ( $\mu$ L) volumes to VOA vials that contained 30 mL of water (Hewitt et al. 1992). Analytical standards for PT/GC/MS analysis were independently prepared following the guidelines in Method 8240 (EPA 1986). All of the values determined were reported with two significant figures due to the limitations of manual strip-chart peak height measurements of the HS/GC analyses.

## RESULTS AND DISCUSSION

### Soil-specific vapor fortification

Table 2 shows the average analyte concentrations and standard deviations for triplicate subsamples of TB, RMA, CRREL, and PBA soils and a set of empty ampoules that were vapor-fortified together as a single batch treatment. These subsamples were all

analyzed by aqueous extraction HS/GC analysis. The results show that the fortification treatment level is soil-specific and that the concentrations sorbed by the soils were much greater than those determined for the empty ampoules. The relationship between the sorption capacity of a soil and this treatment process is beyond the scope of this study, but the observation of this characteristic is unique to vapor fortification and does not occur when treatment is by direct injection of either aqueous or methanolic solutions.

### Precision of vapor fortification

Table 3 shows results for the analysis of triplicate subsamples of the TB standard soil from five different vapor fortification batches prepared over the course of four months. In each of these fortification batches only TB soil subsamples were treated. Four of the batches were analyzed by aqueous extraction HS/GC analysis and the fifth by MeOH extraction PT/GC/MS analysis. A statistical comparison of mean concentrations for any of the four VOCs of interest, performed using an analysis of variance (ANOVA) at the 95% confidence level, failed to establish any significant differences between the five treatment batches. The means, standard deviations, relative standard deviations, and analyte concentration ranges for a 95% confidence range based on the entire data set ( $n=15$ ) are also provided in Table 3.

Under the assumption that this set of analyses provides a true estimate of the four VOC concentrations in this soil, 95% confidence bands around the mean could be used in tests of the performance of other laboratories. Furthermore, the percent relative standard deviation for all of the analyses was  $\leq 6.2\%$ . These findings demonstrate that there was good precision both within and among the treatment batches.

Table 2. Soil-specific vapor fortification concentrations.

Compound	Empty <sup>†</sup>	Soils ( $\mu$ g/g)			
		TB	RMA	CRREL	PBA
TDCE	5.1 $\pm$ 0.5*	6.7 $\pm$ 0.8	16 $\pm$ 0.6	10 $\pm$ 1.3	46 $\pm$ 1.3
Benzene	1.8 $\pm$ 0.2	7.3 $\pm$ 0.3	16 $\pm$ 0.3	8.8 $\pm$ 0.5	41 $\pm$ 0.7
TCE	2.1 $\pm$ 0.2	9.2 $\pm$ 0.2	18 $\pm$ 0.4	13 $\pm$ 0.9	51 $\pm$ 1.8
Toluene	1.5 $\pm$ 0.08	11 $\pm$ 0.1	22 $\pm$ 0.5	14 $\pm$ 0.7	54 $\pm$ 2.3

<sup>†</sup> ( $\mu$ g/vial)

\* Mean and standard deviation

**Table 3. Analyte concentrations ( $\mu\text{g/g}$ ) established for vapor-fortified TB soil subsamples from five different treatment batches.**

Batch and analysis method	Compound			
	TDCE	Benzene	TCE	Toluene
A HS/GC	8.1	9.0	10	12
	7.7	8.5	10	12
	8.1	8.9	11	13
	$8.0 \pm 0.2^*$	$8.8 \pm 0.3$	$10 \pm 0.6$	$12 \pm 0.6$
B HS/GC	7.4	8.4	9.9	12
	7.9	8.6	10	12
	8.2	9.0	11	13
	$7.8 \pm 0.4$	$8.7 \pm 0.3$	$10 \pm 0.6$	$12 \pm 0.6$
C HS/GC	9.4	8.7	11	12
	7.4	8.2	10	12
	7.8	8.3	11	12
	$8.2 \pm 1.1$	$8.4 \pm 0.3$	$11 \pm 0.6$	$12 \pm 0.0$
D HS/GC	7.9	7.9	11	10
	7.6	8.4	11	11
	8.2	8.7	12	12
	$7.9 \pm 0.3$	$8.3 \pm 0.4$	$11 \pm 0.6$	$11 \pm 1.0$
E PT/GC/MS	8.0	8.3	9.7	12
	8.0	8.0	11	12
	7.6	7.6	11	11
	$7.9 \pm 0.2$	$8.0 \pm 0.4$	$11 \pm 0.8$	$12 \pm 0.6$
Total ( $n=15$ )	$8.0 \pm 0.48^*$	$8.4 \pm 0.40$	$11 \pm 0.65$	$12 \pm 0.74$
% relative SD	6.0%	4.8%	5.9%	6.2%
	(7.0-9.0)**	(7.5-9.3)	(9.6-12.4)	(10.4-13.6)

\* Mean and standard deviation (SD)

\*\* 95% confidence range for individual values

### Holding time

Table 4 shows the results obtained for the aqueous extraction of the TB soil subsamples held for 28, 60, 120, and 240 days, and for the RMA and PBA soils extracted with MeOH after holding for 0 and 30 days. All subsamples were stored at room temperature ( $22^\circ\text{C}$ ) in sealed ampoules, and analyses were performed by HS/GC. An ANOVA of the TB subsamples and a Students *t*-test analysis of the RMA and PBA subsamples failed to establish any significant differences at the 95% confidence level between the various holding periods. Thus, once the treated soils were sealed in glass ampoules they could serve as evaluation materials over a period of at least 30 days. The results in Table 4 also show that vapor fortification is a precise treatment method for the RMA reference soil and the PBA site-specific soil.

### Extraction solvent dependency

Table 5 shows the results obtained from both an aqueous and MeOH extraction HS/GC analysis of the PBA treated soil. The aqueous HS/GC concentrations are different from those shown in Table

2, because the PBA soil was sieved removing some small organic debris (roots, twigs, etc.) prior to subsampling and treatment. The mean concentrations established by the analysis of these two different extraction solvents was significantly different for benzene, trichloroethylene, and toluene at the 95% confidence level as determined by the Students *t*-test after finding that the variances were homogeneous with an *F*-ratio test. Thus, this method of soil fortification also results in analyte soil sorption that is extraction-solvent-dependent. This finding agrees with a previous study that attributed the stronger binding forces, associated with organic carbon as compared to mineral grain surfaces, as causing the significant differences in the analyte concentrations established between aqueous and MeOH extractions (Hewitt et al. 1992). The fortified TB soil, which showed no significant analyte concentration differences when extracted with these two solvents (Table 3), has much less organic carbon in comparison (Table 1). Because of this characteristic, vapor-fortified soils could be used to evaluate both the extraction and analysis steps in a laboratory procedure. However, this phenomenon would not occur for



**Table 4. Analyte concentrations ( $\mu\text{g/g}$ ) established for vapor-fortified TB, RMA, and PBA soil subsamples triplicates vs. holding time.**

Holding time	Compound			
	TDCE	Benzene	TCE	Toluene
<b>TB soil</b>				
28 days	7.8, 8.3, 8.0	8.9, 9.4, 9.1	10, 11, 11	11, 12, 11
	$8.0 \pm 0.3^*$	$9.1 \pm 0.3$	$10 \pm 0.6$	$11 \pm 0.6$
60 days	8.2, 9.3, 7.3	9.3, 9.7, 9.0	11, 12, 11	12, 13, 12
	$8.2 \pm 0.9$	$9.1 \pm 0.3$	$11 \pm 0.6$	$12 \pm 0.6$
120 days	8.9, 7.9, 5.8	9.5, 9.2, 8.2	12, 12, 10	13, 13, 12
	$7.5 \pm 1.6$	$9.0 \pm 0.7$	$12 \pm 0.6$	$13 \pm 0.6$
240 days	7.0, 7.0, 6.1	8.6, 8.4, 8.0	11, 11, 10	13, 12, 12
	$6.7 \pm 0.5$	$8.3 \pm 0.3$	$11 \pm 0.6$	$12 \pm 0.6$
<b>RMA soil</b>				
0 days	14, 12, 13	15, 15, 15	16, 16, 17	23, 22, 22
	$13 \pm 1.0$	$15 \pm 0.0$	$16 \pm 0.6$	$22 \pm 0.6$
30 days	15, 15, 14	15, 16, 17	19, 18, 17	22, 21, 21
	$15 \pm 0.6$	$16 \pm 1.0$	$18 \pm 1.0$	$21 \pm 0.6$
<b>PBA soil</b>				
0 days	37, 37, 38	38, 39, 38	56, 56, 55	66, 66, 63
	$37 \pm 0.6$	$38 \pm 0.6$	$56 \pm 0.6$	$65 \pm 1.7$
30 days	36, 38, 35	38, 39, 37	55, 57, 54	65, 69, 65
	$36 \pm 1.5$	$38 \pm 1.0$	$55 \pm 1.5$	$66 \pm 2.3$

\* Mean and standard deviation

**Table 5. Headspace gas chromatography analysis of aqueous and MeOH extraction concentrations (mg/g) of PBA fortified-soil subsample triplicates.**

Extraction solvent	Compound			
	TDCE	Benzene	TCE	Toluene
Water	37	35	46	48
	37	35	45	47
	39	36	46	50
	$38 \pm 1.1^*$	$35 \pm 0.6$	$46 \pm 0.6$	$48 \pm 1.5$
MeOH	37	38	56	66
	37	39	56	66
	38	38	55	63
	$37 \pm 0.6$	$38 \pm 0.6^\dagger$	$56 \pm 0.6^\dagger$	$65 \pm 1.7^\dagger$

\* Mean and standard deviation

† MeOH extraction concentration significantly greater than water as determined by the Students *t*-test.

methods that create performance evaluation samples just prior to analysis or that use large quantities of an organic solvent to transfer the analytes of interest due to sorption kinetics and competition for sorption sites.

### Summary

Under ideal circumstances, performance evaluation samples used to qualify subsequent environmental analyses should mimic the matrix to which

they are being compared. The most appropriate performance evaluation materials would have the same analytes and matrix composition as the environmental samples of concern. In addition, fortified analyte concentrations must be known within acceptable limits and must remain stable for periods of at least a month and preferably for years. The fortification method briefly described in this report can be applied to both reference and site-specific soils, creating precise performance evaluation materials that show some of the sorption and desorption characteristics often associated with field samples. Moreover, the vapor fortification treatment method and exposure period are more representative of the mechanism

by which unsaturated soil becomes contaminated than are the currently used liquid spiking methods.

### CONCLUSION

Vapor fortification treatment and glass ampoule confinement is a precise means to create and hold soil subsamples contaminated with VOCs for both QA and QC purposes. The subsample concentra-

tions achieved by this method are soil specific and, once sealed in a glass ampoule, maintained analyte concentrations for at least one month. Performance evaluation materials prepared by this procedure also show sorption/desorption characteristics that are consistent with environmental soil samples. These features make the vapor-fortification treatment method more relevant than liquid spiking procedures for assessing the precision and accuracy of a laboratory's sample preparation (extraction) and analysis (determination) procedure.

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# REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE February 1994	3. REPORT TYPE AND DATES COVERED	
4. TITLE AND SUBTITLE Vapor Fortified QA/QC Samples for the Analysis of Volatile Organic Compounds			5. FUNDING NUMBERS	
6. AUTHORS Alan D. Hewitt				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Cold Regions Research and Engineering Laboratory 72 Lyme Road Hanover, New Hampshire 03755-1290			8. PERFORMING ORGANIZATION REPORT NUMBER Special Report 94-2	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Environmental Center Aberdeen Proving Ground, Maryland 21010-5401			10. SPONSORING/MONITORING AGENCY REPORT NUMBER SFIM-AEC-TS-CR-94030	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited. Available from NTIS, Springfield, Virginia 22161			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>Vapor fortification is a precise treatment method for preparing soil subsamples for the analysis of volatile organic compounds (VOCs). This method of spiking is unique in that the fortification level is soil-type specific and the treatment mechanism is analogous to how soil in the vadose zone becomes contaminated by vapors from liquid VOC point sources. Soil subsamples treated by vapor fortification and sealed in glass ampoules until analysis have shown both good precision within batch and among batches, and analyte concentration stability for a month or longer. Vapor-fortified quality assurance/quality control (QA/QC) soils are more environmentally representative than soils fortified by liquid spiking procedures for the analysis of VOCs in vadose zone samples.</p>				
14. SUBJECT TERMS Quality assurance Quality control			15. NUMBER OF PAGES 12	
Spiked Soils			16. PRICE CODE	
Volatile organic compounds				
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	