RESIDUES AND TRACE ELEMENTS

Extraction Methods for Recovery of Volatile Organic Compounds from Fortified Dry Soils

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Recovery of 8 volatile organic compounds (VOCs) from dry soils, each fortified at 800 ng/g soil, was studied in relation to the extraction method and time of extraction. Extraction procedures studied on 2 desiccator-dried soils were modifications of EPA low- and high-level purge-and-trap extractions (SW-846 Method 5030A): treatment 1, unmodified low-level procedure; treatment 2,18 h water pre-soak followed by low-level procedure; treatment 3, 24 h methanol extract at room temperature followed by high-level procedure; and treatment 4, 24 h methanol extract at 65/C followed by high-level procedure. VOC recoveries from replicate soil samples increased in the treatment order 1 through 4. With Charleston soil (8% clay and 3.6% organic carbon), highly significant differences (p#10.001) in recoveries among treatments were observed for trichloroethene (TCE), tetrachloroethene (PCE), toluene, ethylbenzene, and 0-xylene, with 2- to 3-fold increased recoveries between treatments 1 and 3. With Hayesville soil (32% clay and 0.2% organic carbon), significant improvements (p l 0.05) in recoveries of toluene, ethylbenzene, o-xylene, 1,1,1- trichloroethane, TCE, and PCE were observed for heated methanol (treatment 4) rather than water extraction (treatment 1), but the increases were less than 2-fold.

Dry soils can be fortified with volatile organic compounds (VOCs) to provide relatively stable and reliable quality assurance samples (1-3). Dry \oil adsorbs 2 4 orders of magnitude more compound than moist soil. depending on the soil and compound characteristics (&6). and VOC degradation rates are markedly reduced in dry soil (3). The U.S. Environmental Protection Agency (EPA) Characterization Research Division. Las Vegas (CRD-LV) is currently seeking ways to develop quality assurance sample or performance evaluation materials (PEMs) to verify analytical accuracy during routine sample analyses. From a research perspective. generation of relatively stable replicate samples will increase the accuracy and precision of numerous studies involving VOCs in soil.

Dry soil can he fortified with VOCs by spiking soil with neat compounds (1) or hy allowing soil to adsorb vapors (2. 3). With the former method. the initial fortification level is explicitly controlled. whereas with the latter method, the fortification level is dependent on soil and compound properties, time of exposure and relative concentrations in the fortification solution. With either method, the resulting concentration must be verified by analyzing subsamples.

Once fortified. soil and compound properties can affect the measurement of VOCs. Earlier studies on the generation of VOC-fortified dry \oil\ demonstrated that room temperature methanol extraction (modified from the EPA high-level extraction) was superior to extraction by the standard EPA low-level purge (soil + water purged at 40/C for 11 min) for 6 of 8 compounds studied in 3 soil types (1). In one soil, extraction by the low-level purge recovered 25-49% of the fortified analytes. whereas extraction by methanol recovered 92-106%. Ancillary studies indicated that recovery of VOCs by the low-level procedure increased. however, in proportion to the time that the soil soaked in water while queued on the autosampler.

The low-level purge originated as a method of analyzing VOCs in water samples. In soil. VOCs reside in vapor, dissolved. nonaqueous liquid (NAPL). and sorbed phases. Vapor and dissolved phase VOCs present a sampling problem (because these forms are readily lost during sampling operations), whereas the NAPL and sorbed phases present an extraction problem. The east of extraction of soil VOCs depends on the amount of time that the compounds have diffused and the complexity of the sorption sites. The relatively small size and large vapor pressure of VOC molecules allow these molecules to diffuse into extremely small spaces. pores 100 small for microorganisms to fit (7). or into amorphous humic material (8). Pignatello and Xing (9) recently reviewed evidence for and mechanisms of slow sorption of organic chemicals in soil. After many days, months, or years of diffusion within a soil, extraction of VOCs will reflect slow desorption by reverse diffusive pathways (7,9,10). Pulverization of soil has been shown to

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release entrapped VOCs (11–13). However, the most effective extraction procedure, superior to solvent extraction at room temperature, purge-and-trap, or Soxhlet techniques, is a heated extraction with water-miscible solvents (9, 14).

The objective of this research was to refine our knowledge of extraction procedures for recovery of VOCs from fortified dry soils by EPA SW-846 methods (15). Specifically, we studied extraction differences that should not be dismissed, should these dry soils be used as PEMs. The factors studied were (*1*) the effect of the length of time that dry soil soaks before a water purge, (*2*) the extent of the difference between room-temperature and heated methanol extraction, and (*3*) differences among compounds.

Estimates of Compound Partitioning and Sorbent Coverage

Successful fortification of dry soil requires a large soil–vapor partition coefficient, K_d', to retain the compounds on the sorbent against vapor loss. The concept of the vapor K_d' is analogous to an aqueous partition coefficient, K_d. The equilibrium soil uptake versus equilibrium vapor concentration is measured in headspace vials, varying the amount of soil or sorbent in the vial and correcting for concentrations observed in blank vials (5). The slope of the data provide K_d' values as

$$K_d' = \frac{\Delta \text{ soil uptake(ng/g)}}{\Delta \text{ vapor concentration(ng/mL)}}$$

Ong and Lion (5) reported K_d' values for trichloroethene (TCE) on various oven-dried minerals. Vapor sorption was highly correlated with the surface area of the sorbents. Thus, kaolinite, iron oxide, and humic-coated alumina had N_2 sorption as a modifier of surface area measurements of 8.5, 11, and 189 m^2/g, respectively, and corresponding K_d' values of 240, 470, and 13 800 mL/g. Using these values as rough guides, we can calculate estimates of TCE headspace concentrations over soil fortified at 800 ng/g (the rate used in this study). Therefore, soils with surface areas analogous to kaolinite, iron oxide, or humic-coated alumina spiked with TCE at 800 ng/g might sustain equilibrium headspace concentrations of 3.3, 1.7, and 0.06 ng TCE/mL, respectively.

For this study, soils were fortified with 8 compounds and the vapor partitioning coefficients were not known. As a rough check on the sorption capacity of the soils, we estimated the mass of the largest compound if it existed as a monolayer covering the soil with the smallest surface area. By staying well below monolayer coverage on this soil, ample sorption capacity was assumed for the VOCs added to the soil. *o*-Xylene, the largest molecule in this study, occupies approximately 1 nm^2. If it was added to the soil at 8000 ng/g (10 times the actual rate), the molecules would occupy approximately 5.2×10^{-2} m^2/g. This is approximately 2 orders of magnitude smaller than the surface area of the Charleston soil (4.3 m^2/g; Table 1).

Experimental

Materials

Soils used in these studies were the Bt2 horizon of the Hayesville series (clayey, oxidic, mesic Typic Hapludult) collected in Fannin County, Georgia, and the A horizon of the Charleston soil (sandy-skeletal, carbonatic, mesic Fluventic Haplustoll) sampled in Clark County, Nevada. [*Note*: No soil series have been assigned in this part of Nevada; the soil designated as Charleston was collected from the same streamreach as that denoted "surface 4, fir-pine" in Amundson et al. (16).] Selected properties of these soils are given in Table 1.

Eight target compounds were used in the study, 4 gasoline components, benzene, toluene, ethyl benzene, and *o*-xylene and 4 chlorinated solvents, 1,1,1-trichloroethane (TCA), TCE, tetrachloroethene (PCE), and 1,1,2,2-tetrachloroethane (TTCA) (Table 2). Two-system monitoring compounds (SMCs), *cis*-1,2-dichloroethane and 1-chloro-2-fluorobenzene, were added at 150 ng/vial to each sample, blank, and standard to monitor the analytical system. The target compounds were purchased neat and the SMCs as 2000 µg/mL standards.

Soil Fortification and Sampling

Sieved (2 mm), air-dried soil was placed over anhydrous $CaSO_4$ in a desiccator 2–4 days prior to fortification. Subsequently, desiccator-dried soil (1 kg) was placed in a wide-mouth 2 L glass jar with a Teflon-lined lid and fortified with 800 ng each VOC/g soil. The 8 target compounds (neat) were added in order of increasing vapor pressure (*o*-xylene through TCA) by microsyringe; compounds were injected 4 to 5 cm below the soil surface. The jars were then sealed and tumbled end-over-end for 12 h on a rotary mixer.

Soil subsamples were obtained by scooping soil from the fortified bulk sample with a glass weighing funnel. Weights of

Table 1. Description of soils used in this study

Soil designation	Sand[a], %	Clay[a], %	pH water	pH 0.01M $CaCl_2$	EC[b], dS/m	Surface area[c], m^2/g	Organic carbon[d], %	Carbonate carbon[d], %
Hayesville	46	32	5.0	4.4	0.018	25.5	0.2	0
Charleston[e]	61	8	7.7	7.3	0.313	4.3	3.8	3.1

[a] Determined by pipet method after pretreatment for removal of organics and carbonates.
[b] Electrical conductivity.
[c] Measured by 3-point N_2 sorption.
[d] Organic carbon calculated as difference between total carbon (dry combustion) and carbonate carbon (manometric method).
[e] Soil was collected from the same streamreach as that designated "surface 4 fir-pine," described in ref. 16.

Table 2. Selected physical-chemical properties of compounds used in this study

Compound	Density, g/mL	Solubility at 25°C, mg/L	Vapor pressure at 25°C, mm Hg	Log K_{ow}[a]
TCA	1.339	1495[b]	123.7[b]	2.49[b]
Benzene	0.879	1791[b]	95.19[b]	2.13[b]
TCE	1.464	1100[b]	69.0[b]	2.42[b]
Toluene	0.867	534.8[b]	28.4[b]	2.73[b]
PCE	1.623	150.3[b]	18.49[b]	3.40[b]
Ethylbenzene	0.867	161[c]	9.53[c]	3.15[c]
o-Xylene	0.880	175[b]	6.6[b]	3.12[b]
TTCA	1.541	1100[b]	12.03[b]	3.03[b]

[a] Log octanol–water partition coefficient.
[b] Ref. 17.
[c] Ref. 18.

ca 1 and 5 g, needed for the water/purge and methanol/purge procedures, respectively, were transferred through a standard glass funnel and into 5 mL ampules, alternating between the 2 sample sizes. Ampules were sealed in the order filled and weighed to the nearest 0.01 g. Sealed ampules were stored for 3 months at room temperature (25°C) before the experiment was conducted.

Ampules were assigned to 1 of 4 treatments, based on the assumption that volatile losses were occurring during the subsampling/ampulation procedure. As the ampules were filled, they were divided into 7 blocks, or "rounds." Ampules were randomly assigned to treatments within each round.

Treatments

Sample preparation for treatments 1 and 2 (low-level water/purge procedures) consisted of opening the ampules, transferring the soil into 40 mL VOA (volatile organics analysis) vials, and sealing the vials immediately with lids designed to fit on the purge-and-trap autosampler (Associated Design & Manufacturing Co., Alexandria, VA). Sample preparation for treatments 3 and 4 (high-level methanol/purge procedures) consisted of opening the ampules, transferring the soil into 40 mL VOA vials containing 5 mL methanol, sealing, and manually shaking the vials for 1 min. Subsequent procedures varied by treatment and are described below.

(a) *Treatment 1, water.*—Vials were secured on the autosampler, water and SMCs were added through a 3-port sampling valve, and samples were analyzed in the order that subsamples were collected from the bulk soil.

(b) *Treatment 2, water, presoak.*—Vials were secured on the autosampler, 3 mL water was added, and samples were presoaked for 18 h at room temperature (25°C). After the presoak, SMCs and 2 mL water was added, and the samples were analyzed sequentially on the autosampler as in treatment 1.

(c) *Treatment 3, methanol, 25°C.*—Samples were soaked in methanol for 18–48 h at room temperature (25°C). Vials were then opened, 100 µL was removed by syringe, and the extract was placed in a 40 mL vial. Vials were attached to the autosampler, 5 mL water plus SMCs were added through the 3-port sampling valve, and samples were analyzed sequentially on the autosampler as in treatment 1.

(d) *Treatment 4, methanol, 65°C.*—Vials containing soil and methanol were heated in a convection oven at 65°C for 24 h. Vials were then cooled to room temperature (25°C) and analyzed within 3 days as described in treatment 3.

Analytical Procedures

Extractions were performed by EPA SW-846 preparatory Method 5030A and the extracts were analyzed by SW-846 Method 8021 (15). EPA protocols were followed except that (*1*) the soil–methanol ratio (high-level procedure) was in-

Table 3. Minimum method detection limits (ng/g)

	Low level		High level	
Compound	Hayesville	Charleston	Hayesville	Charleston
Benzene	1.7	2.7	27	43
Toluene	12.1	6.9	86	75
Ethylbenzene	1.7	2.8	61	61
o-Xylene	2.9	4.6	70	84
TCA	2.1	2.4	25	79
TCE	1.9	3.1	27	107
PCE	2.7	3.9	23	88
TTCA	5.0	4.0	53	139

creased to 5 g soil in 5 mL methanol to ensure that VOC concentrations were above the minimum detection limits, and (2) the methanol extraction time was 24 h rather than 2 min.

A purge-and-trap O.I. Analytical (College Station, TX) Model 4460A Sample Concentrator with a multiple purging module (MPM-16) autosampler was used for sample analysis. Desorbed vapors were separated on a Hewlett-Packard Series II Model 5890 gas chromatograph equipped with a J&W DB 624 30 m × 0.53 mm id, fused silica column. Detectors were an O.I. Analytical Model 4420 electrolytic conductivity detector and an O.I. Analytical Model 4430 photoionization detector, arranged in series. Five-point calibration curves (analyzed in triplicate) were used to determine sample concentrations. SMCs were selected to bracket the retention times of the target compounds without peak interference. Acceptance criteria for sample data required recovery of SMCs at 100 ± 30%.

Minimum detection limits.—Seven soil replicates of each soil were prepared and analyzed by procedures analogous to treatments 1 and 3. The low level replicates were spiked with 10 ng of each compound (in methanol) just prior to analysis. The replicates analyzed by the high-level procedure were prepared to yield the same quantity on the detector as the low-level replicates; these were spiked with the compounds at 100 ng/g, diluted 1:1 in methanol, and the extract was analyzed. The minimum detection limits by each procedure for each soil (Table 3) were calculated as the product of the standard deviation of the replicate analyses and the Student's t value for a 2-sided, 99% confidence level, with 6 degrees of freedom (19).

Statistical procedures.—Data were examined for normality and homogeneity of variances by graphical and univariate procedures. A multivariate analysis of variance (MANOVA) was performed to test for significant differences in compound concentrations as a function of the independent variables: soil, treatment, and soil-by-treatment interaction. Significant effects were followed by a least significant difference test (LSD) for comparison of treatment means (Table 4).

Table 4. MANOVA results

Compound	Source[a]	Degrees of freedom	F ratio
Benzene	Soil	1	7.88[b]
	Trt	3	2.69
	Soil x trt	3	0.33
Toluene	Soil	1	2.76
	Trt	3	21.41[b]
	Soil x trt	3	6.00[c]
Ethylbenzene	Soil	1	39.84[b]
	Trt	3	59.16[b]
	Soil x trt	3	25.67[b]
o-Xylene	Soil	1	47.73[b]
	Trt	3	60.44[b]
	Soil x trt	3	25.42[b]
TCA	Soil	1	6.65[d]
	Trt	3	10.02[b]
	Soil x trt	3	2.01
TCE	Soil	1	81.06[b]
	Trt	3	33.21[b]
	Soil x trt	3	9.42[b]
PCE	Soil	1	23.80[b]
	Trt	3	19.84[b]
	Soil x trt	3	7.06[b]
TTCA	Soil	1	43.10[b]
	Trt	3	4.82[c]
	Soil x trt	3	10.97[b]

[a] Sources of variability: soil = soil; trt = treatment; soil x trt = soil-by-treatment interaction.
[b] Significant at $p \leq 0.001$.
[c] Significant at $p \leq 0.01$.
[d] Significant at $p \leq 0.05$.

Results

The mean concentrations of VOCs generally increased in the treatment order 1 through 4 (Figure 1), with the exception of TTCA on the Hayesville soil. Differences caused by treatments were significant at $p \leq 0.01$ or 0.001 for all compounds except benzene (Tables 4 and 5). Mean recoveries by treatment (excluding TTCA recoveries on the Hayesville soil) were as follows: treatment 1, 46.8% (range, 29–86%); treatment 2, 59.6% (range, 30–89%); treatment 3, 77.9% (range, 35–126%); and treatment 4, 85.0% (range, 53–121%). For most compounds, differences caused by soil and the soil-by-treatment interaction term were highly significant (Table 4), indicating that the 2 soils behaved differently, i.e., the effect of treatments was greater on the Charleston soil than on the Hayesville soil (Figure 1).

In the Charleston soil, soaking samples before the low-level purge (treatment 2 versus treatment 1) resulted in a 1.5- to 2-fold increase in all compound recoveries except benzene (Figure 1 and Table 5). Room temperature methanol extraction (treatment 3 versus treatment 1) produced a 2- to 3-fold increase in recoveries for all compounds except benzene and TCA. Heated methanol (treatment 4 versus treatment 1) produced greater than 3-fold increases in recoveries of TCE, ethylbenzene, and o-xylene. TCE was the only compound for which significantly increased concentrations ($p \leq 0.05$) occurred in the heated methanol treatment when compared with room temperature methanol (treatment 4 versus treatment 3).

In contrast to the Charleston soil, differences in recoveries caused by treatments were much smaller in the Hayesville soil. Soaking the Hayesville soil in water (treatment 2) did not improve extraction of any of the compounds as compared with treatment 1 (Figure 1 and Table 5). Only toluene exhibited a significant increase in recovery as a result of room temperature methanol extraction (treatment 3 versus treatment 1). Heated methanol (treatment 4 versus treatment 1) significantly ($p \leq 0.05$) improved recoveries of TCA, TCE, PCE, toluene, ethylbenzene, and o-xylene; the increases, however, were less than 2-fold.

Discussion

The disparity in the initial soil volumes between the 2 treatment types, 1 and 5 g, created a disparity in the ampule headspace volumes, approximately 9 and 5 mL, respectively. This

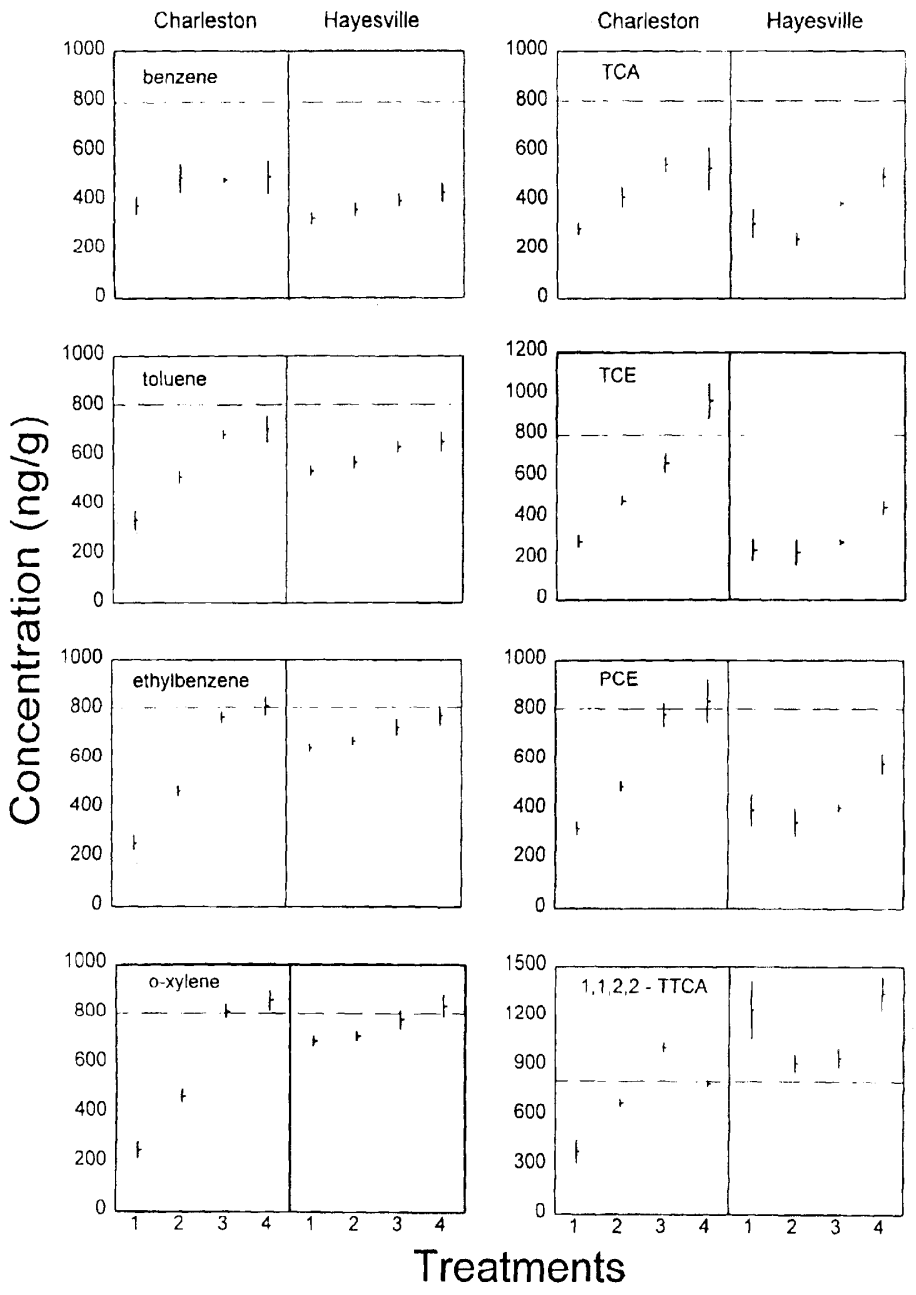

Figure 1. Mean VOC concentrations ($n = 7$) and standard errors of means (error bars) measured by treatment on Charleston and Hayesville soils. Treatments were: (1) water, (2) water, 18 h pre-soak, (3) 25°C methanol extract, and (4) 65°C methanol extract, followed by purge-and-trap analyses. Dotted line marks the nominal fortification rate, 800 ng/g.

Table 5. Mean VOC recoveries for each compound/treatment/soil combination ($n = 7$)

Compound	Treatment	Recovery, % Charleston	Hayesville
TCA	4	66 a[a]	62 a
	3	68 a	48 ab
	2	52 ab	30 c
	1	35 b	38 bc
Benzene	4	61 a	53 a
	3	61 a	50 a
	2	60 a	45 a
	1	47 a	40 a
TCE	4	121 a	56 a
	3	83 b	35 b
	2	61 c	31 b
	1	36 d	29 b
Toluene	4	88 a	82 a
	3	85 a	79 a
	2	64 b	72 ab
	1	42 c	67 b
PCE	4	104 a	73 a
	3	98 a	50 b
	2	61 b	43 b
	1	40 c	50 b
Ethylbenzene	4	102 a	97 a
	3	96 a	91 ab
	2	58 b	84 b
	1	32 c	81 b
TTCA	4	99 b	167 a
	3	126 a	118 b
	2	85 b	114 b
	1	48 c	155 ab
o-Xylene	4	107 a	104 a
	3	102 a	97 ab
	2	59 b	89 b
	1	31 c	86 b

[a] Different lowercase letters indicate treatment differences ($p < 0.05$) in the extraction of a given compound from the soil indicated, as measured by Fisher's least significant difference test.

disparity could bias the results by allowing differential losses when the ampules were broken open for analysis. Although the volume discrepancy certainly introduced some bias, evidence from previous studies indicates that the headspace loss was small relative to other factors. Minnich et al. (1) reported on initial studies of the differences between the low- and high-level procedures using the same compounds and soils, but without the intermediate step placing and sealing the soil in the ampules. Large differences between the low- and high-level procedures were observed on the Charleston soil, and lesser differences were seen on the Hayesville soil in the absence of a headspace discrepancy. Furthermore, in replicate 1 g samples of Charleston soil that sat open for 40 min before sealing, there were no apparent differences in concentration between the open set and samples that were sealed immediately. On the Hayesville soil, volatile losses over the first 20 min were not observed, but decreases in benzene, TCA, and TCE were seen after 20 min. Therefore, the bias introduced by the headspace volume discrepancy in the present study would certainly be greatest for the most volatile compounds, benzene and TCA, and least for the less volatile compounds, o-xylene and ethylbenzene. Because the effect does not follow the previously documented vapor losses, the extraction factor is held largely responsible for the observed treatment differences.

Benzene and TCA had conspicuously low recoveries (<70%), regardless of soil or treatment (Figure 1 and Table 5). These compounds have the highest vapor pressures of any compounds studied and, therefore, were likely to have the greatest losses during the soil subsampling procedure. An initial loss of these compounds from the bulk sample jar headspace occurred upon opening along with a second loss when ampules were broken open during the transfer step. Any further studies will incorporate chilling soil before subsampling or transfer steps to minimize the losses of compounds with high vapor pressures.

TTCA recovery from Hayesville soil was greater than 100% for each treatment (range, 114–167%) and did not follow the treatment order exhibited by the other soil/compound combinations. This TTCA recovery was checked in earlier studies by repeated fortification and analyses of the Hayesville soil (1). The fortification solution used was the same stock as that used for the calibration standards. TTCA recovery from the Hayesville soil was high every time it was fortified and analyzed. The Hayesville soil is a weathered subsoil from southeastern United States, containing a large proportion of iron and aluminum oxides.

Finally, the recalcitrance of desorption from the Charleston soil is attributed to the organic carbon content of this soil. Vapor sorption, generally postulated as surface adsorption (4–6), should be greater for the Hayesville soil than for the Charleston soil, because the surface area of the Hayesville soil (25.5 m^2/g) is greater than that of the Charleston soil (4.3 m^2/g). Drying the soils in this study in a desiccator rather than an oven resulted in 0.7% moisture remaining in the Charleston soil and only 0.3% in the Hayesville soil. This moisture appeared to be sufficient to maintain partially hydrated soil organic matter, allowing for vapor diffusion within the organic material during the contaminant exposure period and subsequent slow VOC desorption.

Conclusions

Soil and compound properties influence the ease of VOC extractions from dry, VOC-fortified soils. Differences in recoveries among extraction procedures were much greater in the Charleston soil (8% clay and 3.8% organic carbon) than in the Hayesville soil (32% clay and 0.2% organic carbon). Soaking in water prior to purging increased recovery of all compounds except benzene from the Charleston soil. In contrast, soaking in water had essentially no effect on VOC recoveries from the Hayesville soil. Methanol extraction was superior to low-level purge-and-trap for recovery of toluene, ethylbenzene, o-xylene, TCA, TCE, and PCE from either soil. Heated methanol (65°C) resulted in superior VOC recoveries of 3 out of 4 chlo-

rinated solvents in the Hayesville soil and of TCE only in the Charleston soil.

The data confirm a previous observation (1), that the extraction of VOCs from certain dry soils by EPA low-level purge-and-trap procedure can be improved by soaking in water on an autosampler. If commercial laboratories were to analyze fortified dry soils by the low-level purge-and-trap procedure (as performance evaluation samples), low recoveries would be expected from some soils, and extraction efficiencies would be expected to increase as soaking time increased. Analysis by the high-level purge-and-trap procedure, after a 24 h methanol soak, should provide relatively consistent results, regardless of the soil type or soaking time on the autosampler. Further studies are needed to determine the stability of chlorinated VOCs, particularly TTCA, on soils high in iron and aluminum oxides.

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