Rapid In Situ Collection and Analysis of Semivolatile Organics by Thermal Extraction Cone Penetrometry Gas Chromatography/Mass Spectrometry

Alexander Gorshteyn and Albert Robbat, Jr.
Department of Chemistry, Center for Field Analytical Studies & Technology, Tufts University, 62 Talbot Avenue, Medford, Massachusetts 02155

Received 7 February 2000; revised 5 March 2000; accepted 7 March 2000

Abstract: A thermal extraction cone penetrometer (TECP) has been developed to detect subsurface contaminants in situ without bringing soil to the surface or into a collection chamber. Coupled with thermal desorption gas chromatography/mass spectrometry (TD-GC/MS) sample collection and analysis can be accomplished in ~20 min for the full range of U.S. Environmental Protection Agency Target Compounds (EPA). TECP extraction efficiencies of 50 to 100% can be obtained for most EPA Method 8270 compounds. Results of 99 volatile and semivolatile organics analyzed from the same TECP extracted soil in 16 and 40 min are presented. Measurement precision and accuracy were well within the Method 8270 benchmarks required for solvent-extracted soils analyzed by GC/MS. The total ion and reconstructed ion current chromatograms are shown for chlorinated solvents and gasoline constituents extracted from a hazardous waste site located at Hanscom Air Force Base (Bedford, MA). Data compared favorably against traditional purge and trap GC/MS. © 2000 John Wiley & Sons, Inc. Field Analyt Chem Technol 4: 85–92, 2000

Keywords: semivolatile organics; cone penetrometer; subsurface sample collection; field analysis; thermal desorption; gas chromatography/mass spectrometry

Introduction

Over the past decade we have developed in situ rapid sample collection tools,[1] field-practical analytical instrumentation, and methods,[2,3] and a dynamic site investigation process[4,5] that has been used to investigate and support hazardous waste site cleanups at 35 Superfund, RCRA, and Brownfield sites. We showed that data produced in the field can be used by on-site project managers to determine where samples should be collected and what types of analyses should be performed.[6] In some projects, data were produced under rapid screening conditions. For example, 18 chlorinated solvents and gasoline constituents were chromatographically separated and detected in less than 1 min by desorbing the volatile organic compounds (VOCs) from soil into a heated sampling probe, which was connected to a gas chromatography/mass spectrometry (GC/MS) instrument. With the use of the same thermal desorption technique, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) were separated and detected in approximately 3 min. In both cases, soil samples were brought to the surface for analysis. In other projects, risk-analysis quality VOC data were provided by purging soil from water, with the organics trapped by Tenax and analyzed by GC/MS in 12 min. SVOCs were analyzed by TD-GC/MS after solvent/soil extraction. In contrast to the direct soil TD analyses, the TD module we used for SVOCs was a large-volume GC inlet capable of desorbing soil or extracts (up to 500 μl) without the need to change sample introduction systems. The TD method increased instrument sensitivity so that SVOCs met the Soil Screening Levels (U.S. EPA 1994), including chlorinated compounds normally analyzed by electron capture detection. Risk quality data were produced in 12 min for the simultaneous detection of chlorinated pesticides, PCBs, and PAHs, with PCBs or trinitrotoluene and its derivatives analyzed in 3 min/sample when detected separately. Our field data supported the development of guidelines aimed at streamlining the site investigation process.[7,8]

Every site investigation must determine whether risk to human health or the environment exists. If the data obtained support the notion that no risk or an acceptable level of risk exists for the intended land usage, then no further action is required. If sufficient risk is determined, then the site investigation must delineate the nature, extent, direction, concentration, and rate of movement of the contamination. The abil-

Correspondence to: Albert Robbat, Jr.

ity to rapidly assess the disposition of environmental contaminants at purported or existing hazardous waste sites is an essential component of the nation's environmental restoration program. Field analytics can support these frameworks only when data are produced quickly enough to make next-step decisions on site.[9–11] Cost savings are achieved when sufficient data are produced to fully characterize the site in one or two site visits and when sample collectors and analysts do not sit idle waiting for one another.

Geoprobe™ and Cone Penetrometry (CP) have become the most widely used tools to rapidly collect soil samples and bring them to the surface. Much effort has been made to develop sensors for the purpose of characterizing the subsurface geology.[9] Cone penetrometry has also been used to detect organics in situ by laser induced fluorescence,[13–16] Raman and infrared spectroscopy,[10,11] and thermal extraction mass spectrometry.[12] In addition, laser induced breakdown,[13,14] γ-,[15] and x-ray fluorescence[23–25] spectroscopies have been described for the detection of metals by CP.

Myers et al. have reported the detection of volatile organic compounds (VOCs) by the so-called site characterization and analysis penetrometer system (SCAPS).[19] In their device, the sampling chamber opens by pneumatically releasing the locking lugs once the CP reaches depth. A second, ~2 in., push results in the sampling chamber filling with soil (3.5–5 g). When the chamber, heated to ~185 °C, is purged by carrier gas, VOCs are swept to the surface for analysis. In contrast, we designed a cone penetrometer sampling probe capable of thermally extracting (TE) SVOCs bound to soil.[1,26] The TECP collects organics *in situ* without bringing soil into the collection probe. Initial results showed that 60% to 90% extraction efficiency for PAH and chlorinated pesticides was possible, with recoveries compound specific. TECP recovery of Aroclor 1248 (total PCB) was 95%, while tri- and dinitrotoluene were extracted at 90% efficiency. These results were from dry soil. TECP efficiency was the same for dry and wet soils up to 20% moisture content. When soil moisture was between 20 and 35%, the TECP extraction efficiency decreased. Under these conditions soil temperature maximized at 100 °C until soil-water evaporated. Then, dry and wet soil recoveries were in agreement. In this article, we describe the TECP recovery of EPA Method 8270C organics from 10% soil-water and the simultaneous detection of these compounds in 16 min. Results indicate that the TECP in combination with fast GC/MS should provide an excellent tool for rapidly determining the extent, concentration, and movement of hazardous substances.

Experiment

Thermal Extraction Cone Penetrometry System

Construction details for the heated transfer line and sampling probe are described elsewhere. The TECP system is shown schematically in Figure 1. Silcosteel® (30 m × 1 mm, ID 0.76 mm) from Restec Corp. was used to transfer organics from soil to the sample collection chamber (Figure 1, Part 8, see also Figure 2). Electrical leads were placed between the sample collection chamber and the end of the sample collection probe in order to heat the transfer line by resistive heating. The amount of power needed to heat the steel tube can be calculated by

$$P = BV\,[\gamma S^2\,\Delta T/\rho\tau]^{0.5}, \qquad (1)$$

where V is the voltage; B is estimated heat loss; γ is the density of steel; S is the cross section of the tube; ΔT is the temperature difference from ambient to 300 °C; ρ is equal to R^*S/L, the specific electrical resistance of steel, where R

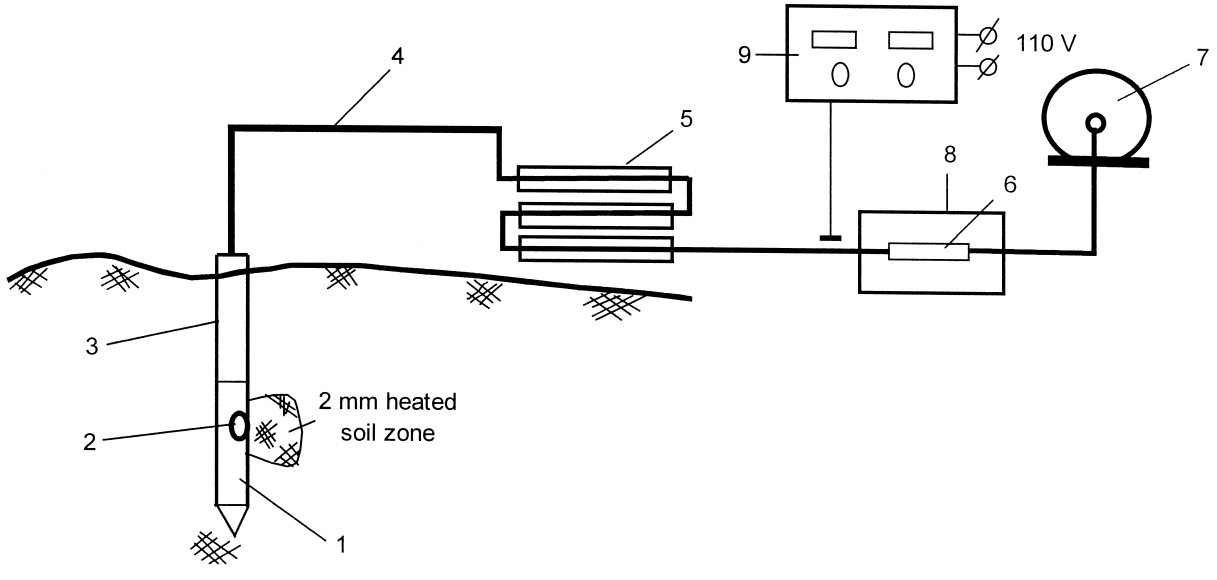

FIG. 1. Schematic of the thermal extraction cone pentrometer system. 1, 1-ft sample collection probe; 2, sample collection window; 3,5, pipe; 4, transfer line; 6, Tenax tube and/or empty glass sleeve; 7, vacuum pump; 8, sample collection chamber; 9, TECP control system.

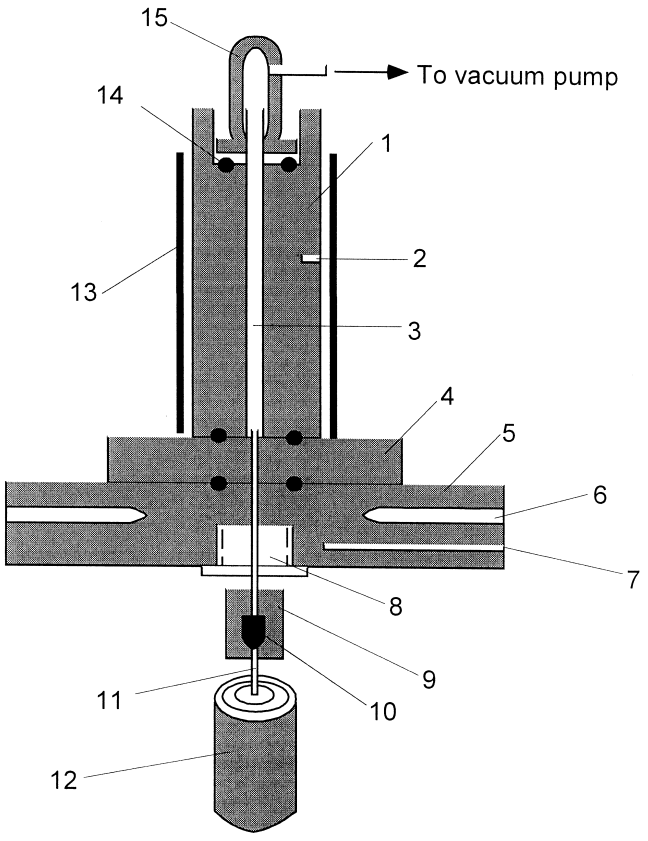

FIG. 2. Illustration of the sample collection unit. 1, Adsorber housing; 2,7, thermocouple; 3, glass sleeve; 4, ceramic isolation plate; 5, adsorber body; 6, heater cartridges; 8–10, column coupler; 11, Silcosteel® ; 12, transfer line; 13, Peltier chamber; 14, O-ring; 15, adsorber cap.

is the electrical resistance over the length (L) of the tube; and τ is the time it takes to heat the steel tube. The heated transfer line, collection probe, and sample chamber unit are controlled by the system's electronics (Figure 1, Part 9). Electrical current was supplied by a step-up isolation transformer (Grainger), with the power (model DCIP-50245-F00, Watlow) and temperature (model 988A-10FD-AARG, Watlow) processor controlled. Thermocouples were placed inside the transfer line, aluminum block, and at the collection window to monitor temperature. A flow meter was used to control and monitor inlet and backflush carrier-gas flow rates.

The sample collection probe was made from a 1-ft section of CP pipe, which housed an aluminum heated block. Both Silcosteel® and the carrier gas lines (Viton tubing) were threaded through the heated block to the sample collection window (Figure 1, Part 2). For field application the transfer line was threaded through several pipe sections (Parts 3 and 5), providing length to reach subsurface depths. Carrier gas, exiting the probe ~10 mm below the TECP inlet, and organic vapor were drawn into the transfer line by vacuum (Figure 1, Part 7), with the organics trapped at the surface (Part 6).

Figure 2 shows how VOCs and SVOCs were collected separately or in series. SVOCs were collected in an empty glass sleeve (Figure 2, Part 3), which was housed (Part 1) in the sample collection chamber (Part 13). SVOCs were freeze-trapped by a Peltier-cooled unit at 2 °C. VOCs were adsorbed onto Tenax, with the tube placed between the adsorber cap (Figure 2, Part 15) and the vacuum pump. The figure also illustrates the construction between the adsorber unit and the Silcosteel® tube (Part 11). Both Silcosteel® and Viton tubing were encapsulated by a shrinkable polyolefin sleeve (Comco). The transfer line (Part 12) consisted of several layers that provided electrical (heat-shrinkable Teflon tubing, Patriot Plastics, and a Nextel 312 sleeve, Omega), thermal (insulated fiberglass wrap, Fisher Scientific), and moisture (polyimid insulation tape, Newark) isolation. Heater cartridges (6, model CIJ5, 110V/60W, Omega) were used to maintain adsorber body temperature (Figure 2, Part 5). A ceramic plate (Part 4) was used to provide electrical and temperature isolation between the adsorber body and the Peltier chamber.

Large-Volume Thermal Desorption GC/MS

Target compounds were analyzed by TD-GC/MS; see Figure 3. The TD module, built at Tufts, was used with a Hewlett Packard GC/MS modified to perform fast GC separations. Samples were introduced by either inserting the TECP glass sleeve or by injecting solutions up to 500 μl into the TD inlet. TECP or syringe-injected sample introduction was made easy by employing a pressure-fitted GC inlet cap rather than the typical screw-type nut inlet, which facilitated the alternation of sample types when analyzing QC check samples. Reference 1 describes the details of the large volume TD GC inlet system. The TD was ballistically heated from ambient temperature to 320 °C in <10 s. Organics were swept from the TD onto the capillary column by helium at 2 ml/min. After desorption the TD was backflushed by switching the heated six-port valve. A constant flow of carrier gas was maintained through the column at all times. The fused silica column, 15 m × 0.32-mm ID with 0.25-μm film thickness of 5% diphenyl, 94% dimethyl, and 1% vinylpolysiloxane (Supelco SPB-5), was held at 10 °C for 1 min then heated to 290 °C at 33 °C/min, and then held isothermal for 1.5 min. The Ion Fingerprint Detection™ (IFD) mass spectrometry software (Ion Signature Technology) was used to analyze GC/MS data. IFD searches each MS scan for a particular compound's mass spectral profile, then extracts the selected ion signal computing its match against predetermined relative abundance error ratios. Quantitation was made by normalizing the least matrix affected target ion against the main ion for each compound's integrated ion signal. This approach decreased nonuniform matrix background signals and minimized variances in the scan-to-scan chromatographic envelope. Details of IFD operation have been discussed previously in this journal.[9]

Reagents, Standards, and Soil Samples

Standard solutions were made of Method 8270 compounds, surrogates, and internal standards, which were pur-

FIG. 3. Picture of specially constructed thermal desorption inlet with HP GC/MS system.

chased from Ultra Scientific and used to prepare fortified soil samples. Soils were a poorly to moderately sorted fine-to-medium sand and an organically mixed loam collected from university property. Soils samples were analyzed before fortification and found to be free of contamination. Soil from Hanscom Air Force Base (HAFB, Bedford, MA) consisted of sandy loam to loam; when it was dry it was dark grayish brown (Munsell designation 10YR4/2). Methylene chloride, HPLC grade, was used to extract all organics from soil.

Results and Discussion

Past research showed that the effective soil temperature, 300 °C, extended 3 mm from the TECP collection window, with temperature decreasing as a function of distance. Equa-

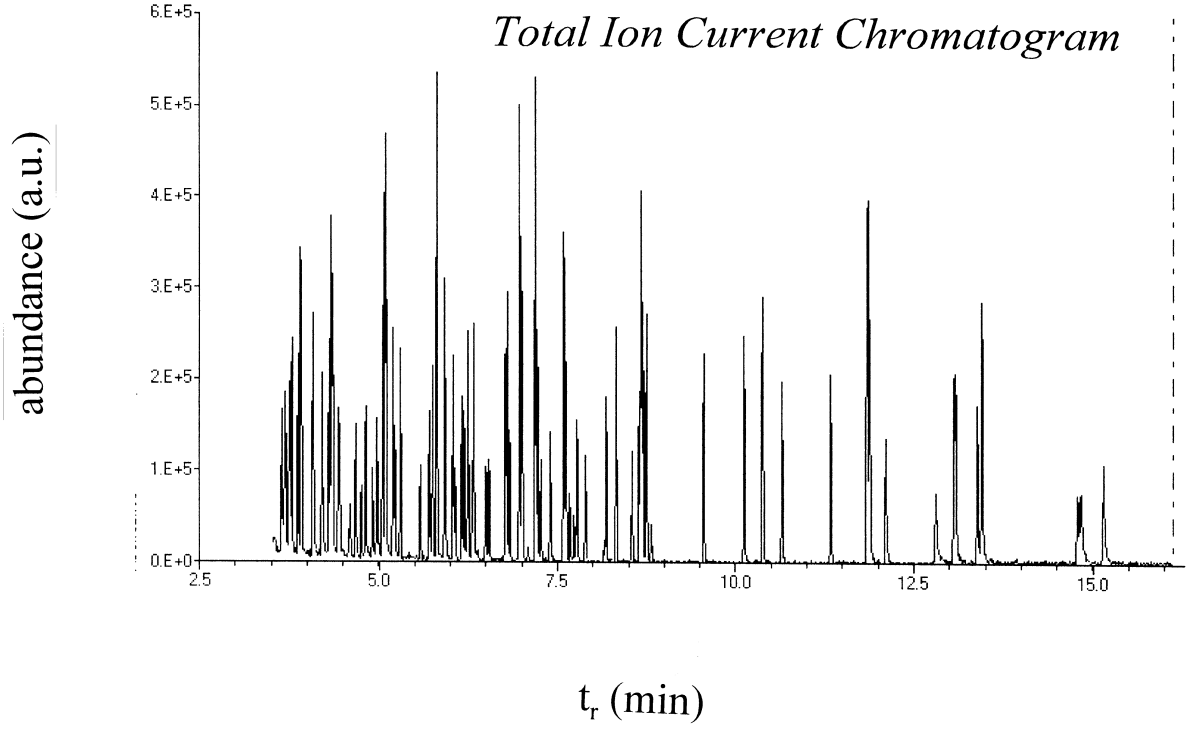

FIG. 4. Total ion current chromatogram of a standard mixture containing 99 volatile and semivolatile organics. The mixture was separated under fast GC conditions, with spectral deconvolution of coeluting compounds made by the IFD data analysis software.

TABLE 1. TECP recovery of a fortified soil sample analyzed by TD-GC/MS in 16 and 40 min.

		16 min						40 min	EPA 8270C	Difference (%)
	Compound	Spiked (ng)	TECP extracted (ng) $n = 1$	$n = 2$	$n = 3$	Average	RSD (%)	Recovery (%)	Recovery (%)	recovery range	($n = 3$) 40 and 16 min
1*	Phenol-d6	80	44	56	68	56	21	70	75	77–116	3
2	Aniline	150	110	81	96	96	15	64	60		−3
3	Phenol	150	192	104	166	154	29	103	89	5–112	−7
4	Pentachloroethane	100	74	52	66	64	17	64	70		4
5	2-chlorophenol	150	154	178	136	156	14	104	89	23–134	−8
6	1,3-dichlorobenzene	100	69	106	79	85	23	85	80	D–116	−3
7	1,4-dichlorobenzene	100	67	85	71	74	13	74	80	20–124	4
8	Benzyl alcohol	150	110	100	98	103	6	68	91		14
9	1,2-dichlorobenzene	100	69	87	92	83	15	83	80	32–129	−2
10	Bis(2-chloroisopropyl)ether	200	200	120	165	162	25	81	85	36–166	3
11	N-nitrosopyrrolidine	150	160	88	115	121	30	81	87		4
12	1,2,4-trimethylbenzene	150	92	120	103	105	13	70	55		−12
13	3-methylphenol	150	120	85	112	106	17	70	86		10
14	N-nitrosodi-n-propylamine	150	110	130	121	120	8	80	80	D–230	0
15	N-nitrosomorpholine	150	160	118	103	127	23	85	78		−4
16	o-Toluidine	150	95	64	78	79	20	53	65		10
17	Hexachloroethane	100	72	99	66	79	22	79	75	40–113	−3
18	2-methylphenol	150	162	120	174	152	19	101	80		−12
19	Acetophenone	150	92	110	96	99	10	66	50		−14
20*	Nitrobenzene-d5	80	72	54	60	62	15	78	80	68–120	2
21	Nitrobenzene	150	120	130	121	124	4	82	75	35–180	−5
22	N-nitrosopiperidine	150	120	87	95	101	17	67	88		13
23	Isophorone	150	120	89	102	104	15	69	90	21–196	13
24	4-nitrophenol	150	110	140	109	120	15	80	91	4–132	7
25	2,4-dimethylphenol	150	99	69	79	82	19	55	56	32–119	1
26	Bis(2-chloroethoxy)methane	200	91	159	86	112	36	56	80	33–184	18
27	2,4-dichlorophenol	150	134	130	176	147	17	98	88	39–135	−5
28	1,2,4-trichlorobenzene	100	63	76	59	66	13	66	93	44–142	17
29	Naphthalene	150	150	103	151	135	20	90	84	21–133	−3
30	4-chloroaniline	150	100	71	88	86	17	58	50		−7
31	Hexachloropropene	100	49	56	76	60	23	60	81		15
32	alpha, alpha-Dimethylphenethylamine	150	120	130	172	141	20	94	98		2
33	Hexachlorobutadiene	100	62	73	72	69	9	69	96	24–116	16
34	Dichlorophenol	150	130	184	138	151	19	101	90		−6
35	1,4-phenylenediamine	150	140	130	122	131	7	87	75		−7
36	N-nitroso-n-butylamine	150	180	110	122	137	27	92	86		−3
37	4-chloro-3-methyl phenol	150	110	72	95	92	21	62	88	22–147	18
38	Isosafrole	150	67	82	56	68	19	46	67		19
39	2-methylnaphthalene	150	156	142	154	151	5	100	110		5
40	Bis(2-chloroethyl)ether	200	130	198	129	152	26	76	82	12–158	4
41	Hexachlorocyclopentadiene	100	70	92	76	79	14	79	60		−14
42	1,2,4,5-tetrachlorobenzene	100	61	75	89	75	19	75	86		7
43	2,4,5-trichlorophenol	150	138	144	176	153	13	102	114		6
44*	2-fluorobiphenyl	80	70	64	64	66	5	83	93	81–113	6
45	2,4,6-trichlorophenol	150	69	62	84	72	16	48	39	37–144	−10
46	Safrole	150	70	76	86	77	10	52	45		−7
47	2-chloronaphthalene	100	68	76	84	76	11	76	86	60–118	6
48	2-nitroaniline	150	86	62	77	75	16	50	75		20
49	1,4-naphthoquinone	150	80	71	68	73	9	49	45		−4
50	Dimethyl phthalate	200	93	143	89	108	28	54	91	D–112	25
51	1,3-dinitrobenzene	150	70	88	88	82	13	55	34		−23
52	Acenaphthylene	150	140	129	133	134	4	89	65	33–145	−16
53	2,6-dinitrotoluene	150	72	62	92	75	20	50	65	50–158	13

(Continued)

TABLE 1. *(Continued)*

	Compound	16 min Spiked (ng)	TECP extracted (ng) $n = 1$	$n = 2$	$n = 3$	Average	RSD (%)	Recovery (%)	40 min Recovery (%)	EPA 8270C recovery range	Difference (%) ($n = 3$) 40 and 16 min
54	3-nitroaniline	150	110	110	103	108	4	72	64		−6
55	Acenaphthene	150	141	131	135	136	4	90	65	33–145	−16
56	2,4-dinitrophenol	150	160	130	152	147	11	98	83	D–191	−8
57	2-nitrophenol	150	77	82	69	76	9	51	50	29–182	−1
58	Dibenzofuran	150	96	82	73	84	14	56	73		13
59	Pentachlorobenzene	100	81	79	75	78	4	78	96		10
60	2,4-dinitrotoluene	150	67	66	89	74	18	49	39	39–139	−12
61	2,3,4,6-tetrachlorophenol	150	61	88	75	75	18	50	85		26
62	Diethyl phthalate	200	109	159	132	133	19	67	91	D–114	15
63	Fluorene	150	141	121	131	131	8	87	60	59–121	−19
64	4-chlorophenyl phenyl ether	200	162	136	148	149	9	74	94	25–158	12
65	5-nitro-o-toluidine	150	67	77	89	78	14	52	62		9
66	4-nitroaniline	150	74	76	84	78	7	52	96		30
67	4,6-dinitro-2-methylphenol	200	90	162	108	120	31	60	95	D–181	23
68*	4-aminobiphenyl	80	60	44	56	53	16	67	75		6
69	N-nitrosodiphenylamine	150	75	75	64	71	9	48	72		20
70	Tribromophenol	150	143	92	115	117	22	78	85		4
71	1,3,5-trinitrobenzene	150	130	120	145	132	10	88	85		−2
72	4-Bromophenyl phenyl ether	200	74	132	141	116	31	58	82	53–127	17
73	Phenacetin	150	98	91	102	97	6	65	45		−18
74	Hexachlorobenzene	100	76	134	98	103	29	103	79	D–152	−13
75	Diphenylamine	150	57	98	85	80	26	53	45		−8
76	Pentachlorophenol	150	146	162	128	145	12	97	134	14–176	16
77	Pentachloronitrobenzene	150	61	73	81	72	14	48	55		7
78	Phenanthrene	150	133	126	129	129	3	86	83	54–120	−2
79	Anthracene	150	132	126	145	134	7	90	85	27–133	−3
80	Dinoseb	150	87	100	92	93	7	62	100		23
81	Di-n-butyl phthalate	200	106	151	156	138	20	69	61	1–118	−6
82	Fluoranthene	150	122	135	109	122	11	81	74	26–137	−5
83	Pyrene	150	131	114	141	129	11	86	90	52–115	2
84*	4-terphenyl-d14	80	77	55	76	69	18	87	90	49–146	2
85	Dimethylaminoazobenzene	150	64	100	95	86	23	58	63		5
86	3,3′-dimethylbenzidine	150	87	74	77	79	9	53	55		2
87	Butyl-benzyl-phthalate	200	68	130	113	104	31	52	85	D–152	24
88	2-acetylaminofluorene	200	130	215	175	173	25	87	85		−1
89	Benzo[a]anthracene	150	121	140	115	125	10	84	75	33–143	−5
90	3,3′-dichlorobenzidine	200	180	160	162	167	7	84	63	D–152	−14
91	Chrysene	150	142	97	132	124	19	82	85	17–168	2
92	Bis (2EH) phthalate	200	161	107	163	144	22	72	85	8–158	8
93	Di-n-octyl phthalate	200	130	213	175	173	24	86	72	4–146	−9
94	Benzo[b]fluoranthene	150	130	107	132	123	11	82	75	24–159	−4
95	Benzo[k]fluoranthene	150	90	165	174	143	32	95	75	11–162	−12
96	Benzo(a)pyrene	150	133	110	121	121	9	81	65	17–163	−11
97	Indeno(1,2,3-c,d)pyrene	150	141	117	152	137	13	91	74	D–171	−10
98	Dibenz(a,h)anthracene	150	140	130	115	128	10	86	76	D–227	−6
99	Benzo(g,h,i)perylene	150	130	120	109	120	9	80	84	D–219	3

* Surrogates.

tion (2) can be used to calculate the total amount of heat, Q_{total}, transferred from the sample probe to the soil, where

$$Q_{\text{total}} = B[\Sigma\, m_i c_{pi}\, \Delta T_i + m_j\, \Delta_{\text{tr}} H_j + \ldots\ ]\,. \qquad (2)$$

B is the assumed heat loss into the environment; m and c are the mass and specific heat capacity of the soil-water *and* sample probe (i.e., aluminum block and steel), i and j are the respective subscripts for each component; ΔT_i is the temperature difference (final and initial) for each material; $\Delta_{\text{tr}} H_j$ is the enthalpy of phase transition from liquid to gas. From this, optimum TECP extraction efficiency requires that the pipe be heated to 450 °C in order to obtain soil temper-

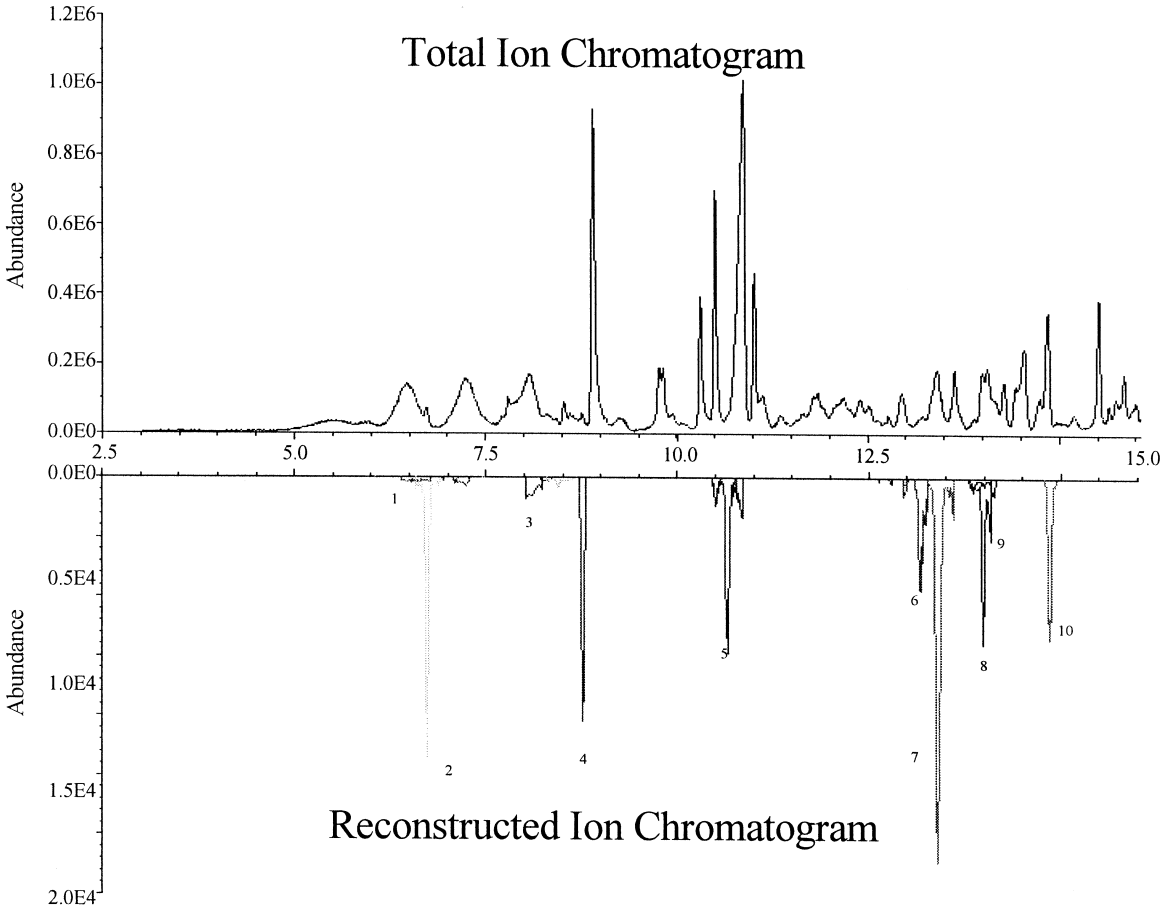

FIG. 5. TECP+TD-GC/MS analysis of a soil sample collected from Hanscom Air Force Base (Bedford, MA). Compounds found are (1) 1,1,1-trichloroethene, (2) methylene chloride, (3) 1,1 dichloroethene, (4) 1,4-difluorobenzene (surrogate), (5) toluene-d_8 (internal standard), (6) ethylbenzene, (7) m/p-xylene, (8) o-xylene, (9) styrene, (10) 4-bromofluorobenzene (surrogate).

ature approximating 300 °C for soils containing upwards of 20% moisture. All experiments were conducted with soil that contained at least 10% moisture.

Figure 4 illustrates a typical 10-μl injection of a standard solution containing 99 compounds. This standard was used to prepare fortified soil samples, which were then placed around the sample probe. TECP recoveries are shown in Table 1. The table lists the amount of each compound spiked into the soil *and* the combined TECP+TD GC/MS measurement accuracy and precision ($n = 3$) for the 16-min analysis. Because of the difficulty of preparing fortified VOC soil samples, the most volatile chlorinated gases were not included in this study. Therefore, Tenax was not used to trap the VOCs. Instead, all organics were freeze-trapped with the use of the Peltier-cooled sample collection chamber. The table also shows the same extract analyzed in 40 min by EPA Method 8270C, GC/MS procedure, as well as the acceptable EPA recovery ranges. TECP recoveries as found by the 16- and 40-min analyses were within 20% for all compounds but seven (50, 51, 61, 66, 67, 80, 87); see RPD in table. No bias was observed in the data with 46 of the 99 measurements less than the average recovery obtained by the different GC separation times. IFD correctly extracted ion signal whether peaks contained single or multiple compounds. Importantly, all compounds fall within the EPA recovery range whether analyzed in 16 or 40 min except phenol-d_6, a surrogate. Failure to fall within the recovery range was due to the TECP extraction efficiency rather than to mixture complexity or analysis rates. TECP precision; that is, the ability to reproducibly extract organics from soil, was excellent. The percent relative standard deviation (%RSD) was less than 30% for all but five compounds, with $4 < 32\%$ and the last one at 36%. In previous field investigations we conducted,[8,9] the EPA and the corresponding state agencies where these studies occurred set measurement accuracy for field surrogate soil/solvent recovery at 30 to 200% as compared to the Method 8270B benchmark of 20 to 140% *and* field analysis precision the same as method 8270B, namely, $<60\%$ RPD. Precision and accuracy of the combined (TECP+TD GC/MS) analyses were well within the required benchmarks for laboratories following Method 8270B or 8270C criteria and certainly were well within the site-specific criteria.

TECP+TD GC/MS data were compared against results obtained for soil (Hanscom Air Force Base, Bedford, MA) collected in a 3-ft tube by Geoprobe™ and analyzed by EPA

Method 8260A, purge-and-trap GC/MS. Except for 1,1,1-trichloroethene and 1,1-dichloroethene, not found in the laboratory analysis, results were within 30%. Figure 5 illustrates typical total ion current (TIC) and reconstructed ion current (RIC) chromatograms for the TECP-extracted soil. Note that most compounds produced extremely low signals of 10^2 to 10^4 when compared to the matrix, 10^6. Loss of VOCs by laboratory analysis may be due to the time between sample collection and analysis (i.e., the transport of sample from the field and the time the sample was actually analyzed), masking of target compounds by the matrix, or the common laboratory practice of diluting highly concentrated samples prior to analysis. In any case, the data show that field analysis with our technology provides cost and time advantages over static investigations where samples are collected and shipped off site for analysis. Although TECP extraction efficiencies range from approximately 50% to nearly quantitative recoveries, VOCs and SVOCs bound to soil can be released and collected in as little as 5 min or as much as 15 min when water content is between 20 and 35%. Analyzing preceding extracts while collecting subsequent soil samples increases field personnel efficiencies and provides information about the extent, direction, concentration, and rate of contaminant movement at the lowest cost.

Acknowledgement

Support for this research was provided by the U.S. Environmental Protection Agency's NCERQA program under Grant No. R826184010.

References

1. Gorshteyn A, Smarason S, Robbat A, Jr. Environ Sci Technol 1999; 33:2474–2480.
2. Gankin YV, Smarason S, Robbat A, Jr. Field determination of VOCs in soil and water by purge-and-trap and thermal desorption GC/MS. In: Lopez-Avila V, editor. Current protocols in field analytical chemistry. New York: John Wiley & Sons; 1998. p 1B.1.1–1B.1.12.
3. Gankin YV, Smarason S, Robbat A, Jr. Field determination of SVOCs in soil by thermal desorption GC/MS. In: Lopez-Avila V, editor. Current protocols in field analytical chemistry. New York: John Wiley & Sons; 1998. p 3.1–3.10.
4. Robbat A, Jr. Field analytics, dynamic workplans. In: Meyers RA, editor. The encyclopedia of environmental analysis and remediation. New York: John Wiley & Sons; 1998.
5. Robbat A, Jr. Dynamic workplans and field analytics. In: Lopez-Avila V, editor. Current protocols in field analytical chemistry. New York: John Wiley & Sons; 1998.
6. Robbat A, Jr., Smarason S, Gankin Y. Dynamic work plans and field analytics, the keys to cost-effective hazardous waste site investigations. Field Anal Chem Technol 1998;2:253–265.
7. Standard practice for expedited site characterization of vadose zone and groundwater contamination at hazardous waste sites. West Conshohocken, PA: ASTM Publication No. D6235-98.
8. Innovations in site characterization. Case study: Hanscom Air Force Base (Sites 1, 2, and 3). Washington, DC: U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response; September 1998. EPA-542-R-98-006 [http://www.clu-in.org/download/char/hafbcs2.pdf and videotape of the Dynamic Site Investigation Process http://www.clu-in.org/video/hanscom.htm].
9. Robbat A, Jr., Smarason S, Gankin Y. Fast gas chromatography/mass spectrometry analysis in support of risk-based decisions. Field Anal Chem Technol 1999;3:55–66.
10. Pepper JW, Wright AO, Kenny JE, Smarason S, Robbat A, Jr. Laser-induced fluorescence and fast gas chromatography/mass spectrometry with subsurface thermal extraction of organics: Field analytical technologies for expediting site characterization and cleanup. Remediation 1998(Winter): 95–111.
11. Johnson R, Quinn J, Durham L, Williams G, Robbat A, Jr. Adaptive sampling and analysis programs for contaminated soils. Remediation 1997;2:81–96.
12. Aldstat JH, Martin AF. Mikrochim Acta 1997;127:1–18.
13. Bloch J, Johnson B, Newbury N, Germaine J, Hemond H, Sinfiled J. Appl Spectrosc 1998;52:1299–1304.
14. Hart SJ, Chen YM, Kenny JE, Lien BK, Best TW. Field Anal Chem Technol 1997;1:343–355.
15. Robbat A, Jr., Kenny JE, Smarason S, Pepper JW, Wright AO, Remediation 1998;9:95–111.
16. Davis WM, Cespedes ER, Lee TL, Powell JF, Goodson RA. Environ Geol 1997;29:228–237.
17. Nau G, McVicker JA, Bucholtz F, Ewing KJ, Vohra ST, Sanghera JS, Aggarwal ID, Adams JW. Proc SPIE Int Soc Opt Eng 1997; 3211:491–495.
18. Kyle KR, Brown SB. Proc Electrochem Soc 1997;97:1066–1079.
19. Myers KF, Karn RA, Eng DY, Hewitt AD, Strong AB, Brannon JM. Field Anal Chem Technol 1998;2:163–171.
20. Theriault GA, Bodensteiner S, Lieberman SH. Field Anal Chem Technol 1998;2:117–125.
21. Miles B, Cortes J. Field Anal Chem Technol 1998;2:75–87.
22. Morgan JC, Adams JW, Ballard JW. Field Anal Chem Technol 1998; 2:111–115.
23. McDonald BJ, Unsell CW, Elam WT, Hudson KR, Adams JW. Nucl Instrum Methods Phys Res Sect A 1999;422:805–808.
24. Elam WT, Gilfrich JV. Adv X-Ray Anal 1997;39:861–867.
25. Elam WT, Gilfrich JV. Adv X-Ray Anal 1995;38:699–704.
26. Gorshteyn A, Kataenko Z, Smarason S, Robbat A, Jr. Instrum Sci Technol 1999;27:111–121.