

ABSTRACT

The En Core™ sampler is a volumetric sampling device designed to store soil samples containing Volatile Organic Compounds (VOCs). This study was performed to demonstrate that the sampler is capable of retaining low level VOCs, including gases, in a sandy soil and a clay fortified garden soil for up to seven days. Two concentration levels were used. Samplers were filled, spiked with a VOC standard which contained compounds of varying volatility and stored at 4°C for 2 and 7 days.

EXPERIMENTAL DESIGN

For the first experiment, soil consisting of coarse sand, farm topsoil, and clay was mixed at En Chem, Inc. Sieve analysis was performed and the soil was >80% sand. Actual soil grain percentages are in the Results section. Thirty milliliters of deionized water were added to 570 grams of the soil mixture resulting in a theoretical value of 5% moisture for the high level study. Fifty-four milliliters of deionized water were added to 546 grams of the soil mixture resulting in a theoretical value of 9% moisture for the low level.

For the second experiment, a soil was prepared consisting of 900 grams of garden soil and 225 of grams of the clay. Sieve analysis was performed, actual soil grain percentages are in the Results section. Soil was also submitted for bioenumeration analysis. No deionized water was added as there was moisture present from the garden soil.

The most common method of preparing performance samples for VOCs is to spike a soil sample with a methanol based standard. This method may not be representative since soil is most often contaminated by either vapor or aqueous phase exposure. The spiking technique which was used is an aqueous method which was developed by Alan Hewitt of the Army Corps of Engineers. An aqueous spike was made by exposing 1 milliliter of commercial gasoline to 80 milliliters of deionized water in a 100 milliliter volumetric flask. This mixture was stirred for approximately 24 hours. The gasoline was decanted and the gasoline saturated water was placed in a 20 milliliter vial. Four hundred milliliters of a 1000 mg/L VOC stock standard (En Chem #s 570-46A & 570-46B) containing the VOC analytes of interest were added through the septa of the vial.

Five replicates of time zero samples and five replicates of spikes held for 2 days and 7 days for each concentration level and soil type were used. Five gram En Core™ samplers were filled with soil, the mouth of the sampler was scraped flush, and the sealing surface was wiped clean with a paper towel. The samplers were then placed in a freezer and frozen. The samplers were removed and spiked when the soil thawed just enough to allow a spike to be introduced.* For the low level study, the 5 gram samplers were spiked with 50 uLs of the spiking solution and were then capped. Sample collection was alternated between the initial samples which were extruded into methanol (T=0) and the corresponding replicates which were held for 2 and 7 days. Appropriate amounts of the spike were also injected into 5 milliliters of methanol before and after the spiking procedure in order to obtain a theoretical spike value. Time zero samples were spiked and immediately extruded into 5 milliliters of methanol. Two and seven day spikes were held at 4°C. Upon extrusion, samples were shaken for 2 minutes and sonicated for 20 minutes before analysis.

The high level studies followed the same experimental design. The 5 gram samplers were spiked with 250 uLs of the spiking solution and were then capped. Sample collection again was alternated between the initial samples which were extruded into methanol (T=0) and the

corresponding replicates which were held for 2 and 7 days. Appropriate amounts of the spike were also injected into 5 milliliters of methanol before and after the spiking procedure in order to obtain a theoretical spike value. Time zero samples were spiked and immediately extruded into 5 mLs of methanol. Two and seven day spikes were held at 4°C. Upon extrusion, samples were shaken for 2 minutes and sonicated for 20 minutes before analysis.

RESULTS

Soil grain size analysis for the sandy soil was performed by United Engineering Consultants, Inc., Milwaukee, WI. The sandy soil consisted of 82.6% sand and 17.4% silt and clay. Soil moisture was 4.1% for the high level and 6.6% for the low level before the addition of the aqueous spike. The final moisture is approximately 10%. Soil grain size analysis for the garden soil was performed by CQM, Inc., Green Bay, WI. The garden soil consisted of 62.6% sand, 24.4% silt and 13% clay. Soil was classified as a silty sand with organic fines, fine grained, very dark brown. Soil moisture was 11.9% before the addition of the spike. Bioenumeration results are attached. Petroleum degrader population was determined at 2.6×10^7 colonies per gram dry weight. This microbial concentration is an order of magnitude greater than the level which is considered sufficient to support measurable biodegradation.

Theoretical or true values were established by analysis of samples in which the spike solution was injected directly into methanol. Two samples were prepared directly in methanol for each study. They were the first and last sample prepared in each study. By using the theoretical samples to bracket the time course samples, losses in the spiking solution during sample preparation can be monitored. Theoretical results can be found on page three of Appendix 1 for the sandy soil study and page 3 of Appendix 2 for the garden soil study. Time=zero (T=0) samples consisted of spiking filled En Core samplers and extruding directly into methanol within a minute of spiking. Average recoveries for T=0 samples were divided by theoretical values to evaluate efficiency of the experimental design and spiking technique.

Sandy Soil Study

Low level samples are defined by EPA method 5035 as having concentrations between 1-200 ug/Kg. High level is defined as greater than 200 ug/Kg. For the low level study, all compounds were within range with the exception of Methylene Chloride (203.4 ug/Kg), Benzene (302.5 ug/Kg), and Toluene (371.2 ug/Kg). The source of the Benzene and Toluene which elevates their concentrations relative to the other compounds is the gasoline saturated water which was the base of the spiking solution.

Average recovery for all VOC compounds in the low level study was 89% at two days and 88% at seven days. The recovery values ranged from 82% for Vinyl chloride to 94% for Bromoform at two days in the low level study. At seven days, the values ranged from 66% for Vinyl Chloride and Bromoform to 155% for Naphthalene. The high recovery for Naphthalene may be explained by differential response between analytical instruments. Samples were prepared in methanol to eliminate matrix effects associated with the low level sample preparation method. The difference between the T=0 average of 94.8 ug/Kg and the seven day average of 146.8 ug/Kg represents a one part per billion difference on column. This difference however is magnified by the 50 fold dilution inherent to the analytical method.

* The reverse situation also performs well, i.e., supercooling the samplers to just the point of freezing, then introducing the spike.

Average recovery for all VOC compounds in the high level study was 89% at two days and 80% at seven days. The recovery values ranged from 73% for Vinyl chloride to 95% for MTBE at two days in the high level study. At seven days, the values ranged from 57% for Vinyl Chloride to 103% for Methylene Chloride.

Average percent recoveries for samples held for two and seven days are in Table 1. Complete data can be found in Appendix 1.

Garden Soil Study

All compounds were within range with the exception of Methylene Chloride (222.4 ug/Kg), Benzene (289.7 ug/Kg), and Toluene (352.1ug/Kg) for the low level study. The source of the Benzene and Toluene which elevates their concentrations relative to the other compounds is the gasoline saturated water which was the base of the spiking solution.

Average recovery for all VOC compounds in the low level study was 92% at two days and 90% at seven days. The recovery values ranged from 76% for Benzene to 101% for Chloroform at two days in the low level study. At seven days, the values ranged from 46% for Vinyl Chloride to 116% for Methylene Chloride. At the seven day point, aromatic compounds showed lower recoveries which could be attributed to biodegradation. For example, Benzene and Toluene had 59% and 65% recovery respectively.

Average recovery for all VOC compounds in the high level study was 104% at two days and 111% at seven days. The recovery values ranged from 97% for Vinyl Chloride to 110% for 1,2,4-Trimethylbenzene at two days in the high level study. At seven days, the values ranged from 86% for Vinyl Chloride to 125% for Bromodichloromethane.

Average percent recoveries for samples held for two and seven days are in Table 2. Complete data can be found in Appendix 2.

Table 1. Sandy Soil Percent Recoveries for 2 and 7 Day Storage

<u>Compound</u>	High Concentration Spike		Low Concentration Spike	
	<u>2 Day Storage</u>	<u>7 Day Storage</u>	<u>2 Day Storage</u>	<u>7 Day Storage</u>
	<u>Average % Recovery</u>	<u>Average % Recovery</u>	<u>Average % Recovery</u>	<u>Average % Recovery</u>
Vinyl chloride	73	57	82	66
Methylene chloride	90	103	89	137
Methyl tert-butyl ether	95	99	92	91
Chloroform	89	82	91	76
Carbon Tetrachloride	87	73	90	76
Bromodichloromethane	92	77	88	76
Benzene	89	77	87	73
1,1,2-Trichloroethane	93	92	90	93
Ethylene Dibromide	93	90	90	97
Toluene	88	73	90	74
Ethylbenzene	90	79	87	71
Styrene	86	66	83	67
Bromoform	92	65	94	66
m/p-Xylene	89	73	90	77
o-Xylene	89	72	88	76
1,3,5-Trimethylbenzene	88	78	90	102
1,2,4-Trimethylbenzene	89	75	89	112
Naphthalene	92	101	86	155

Table 2. Garden Soil Percent Recoveries for 2 and 7 Day Storage

<u>Compound</u>	High Concentration Spike		Low Concentration Spike	
	<u>2 Day Storage</u>	<u>7 Day Storage</u>	<u>2 Day Storage</u>	<u>7 Day Storage</u>
	<u>Average % Recovery</u>	<u>Average % Recovery</u>	<u>Average % Recovery</u>	<u>Average % Recovery</u>
Vinyl Chloride	97	86	82	46
Methylene Chloride	101	97	92	116
Methyl tert-butyl ether	106	117	98	90
Chloroform	105	121	101	97
Carbon Tetrachloride	105	108	96	111
Bromodichloromethane	103	125	98	106
Benzene	101	107	76	59
1,1,2-Trichloroethane	107	124	95	107
Ethylene Dibromide	102	114	92	80
Toluene	102	110	86	65
Ethylbenzene	104	107	95	93
Styrene	103	97	86	65
Bromoform	106	120	83	91
m/p-Xylene	101	104	96	89
o-Xylene	103	109	91	86
1,3,5-Trimethylbenzene	107	113	98	106
1,2,4-Trimethylbenzene	110	116	99	102
Naphthalene	104	118	96	108

DISCUSSION

Two soils were used in this study. One soil was predominantly sand (82.6%) the other was a biologically active loamy topsoil. Losses which are due to spiking technique and transfer are inherent to the experimental design and are independent of storage within the En Core. To normalize the data set, recoveries for En Cores held for two and seven days are calculated relative to T=0 values.

The samplers were spiked with an aqueous standard to minimize volatilization during the spiking procedure. The deionized water was first exposed to gasoline then a small injection of a methanol based standard was added to introduce other compounds of interest. This technique aids in the retention of compounds with low water solubility. Not only does this spiking technique allow a higher level of precision but it also closely mimics the way contaminants are distributed in “real world” sites since contaminants are often transferred in an aqueous phase and not in a methanol solution. This technique was developed by Alan Hewitt and is also used by ERA, a performance sample manufacturer.

En Core samplers and the spiking solution were chilled prior to spiking to further limit volatilization losses during spiking. This technique was effective in limiting volatilization losses during the spiking procedure. Vinyl Chloride in the sandy soil study for example had a 81% recovery in the high level T=0 samples relative to the theoretical value when the soil and spike was chilled.

In the sandy soil study, Vinyl Chloride in all cases had the lowest recovery as would be expected due to its low boiling point. In all cases however, average VOC recovery was $\geq 80\%$ up to seven days. These recoveries meet the criteria for precision which would be applied to an analytical instrument per EPA method 8260.

The garden soil study exhibited excellent recoveries for total VOCs $\geq 100\%$ through seven days for the high level portion. As with the sandy soil study, Vinyl Chloride showed the lowest recovery of all compounds used. The majority of the recoveries were greater than 100% probably due to the low recoveries in Replicate 2 of the Time Zero samples. In this particular replicate, the syringe used to spike the sample developed a plug and it is questionable whether the entire spike was added to the sample. This lowered the average of the Time Zero samples and thus increased two and seven day recoveries to greater than 100%.

The low level study also exhibited excellent recoveries for total VOCs $\geq 90\%$ through seven days. Vinyl Chloride did show losses at the seven day point (46% recovery). This loss could partially be attributed to the storage of the extract prior to analysis. Biodegradation may have occurred at the seven day point in particular with Benzene and Toluene. The reduction of Benzene from 76% at two days to 59% at seven days is most likely biodegradation. Other chlorinated compounds with similar volatility did not exhibit this loss. This study could be deemed a worst case scenario since not only was the soil biologically active but was also aerated during the homogenization of the soil.

APPENDIX 1(Sandy Soil)

High Concentration Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Replicate 1	605.5	700.5	740.0	748.5	635.5	701.5	1340.5	732.5	716.5	1706.0	766.5	694.0	661.5	1802.5	891.0	785.5	848.5	692.0
Replicate 2	688.0	753.0	790.0	791.5	679.5	737.0	1277.0	770.0	766.0	1780.5	804.0	727.0	726.0	1850.0	919.0	825.5	906.5	777.0
Replicate 3	652.0	749.0	781.0	791.5	684.0	735.5	1404.0	772.0	776.0	1798.5	800.5	704.5	729.0	1833.5	913.0	814.5	919.5	721.5
Replicate 4	663.0	739.0	818.0	790.5	678.5	767.0	1433.5	815.0	802.0	1802.0	794.0	722.5	776.5	1875.0	945.5	816.5	903.5	800.0
Replicate 5	566.5	730.0	804.0	778.0	620.5	746.0	1382.5	790.5	795.5	1803.0	785.0	711.0	746.5	1852.5	920.5	793.0	891.0	708.5
ave	635.0	734.3	786.6	780.0	659.6	737.4	1367.5	776.0	771.2	1778.0	790.0	711.8	727.9	1842.7	917.8	807.0	893.8	739.8
RSD	8	3	4	2	4	3	4	4	4	2	2	2	6	1	2	2	3	6

TIME = 2 Days -- >80% sand

Replicate 1	493.5	667.5	715.5	712.0	591.0	672.0	1233.0	725.0	722.0	1570.5	723.5	618.5	651.0	1640.0	814.0	709.5	801.5	662.0
Replicate 2	478.5	667.5	763.5	735.0	576.5	675.5	1244.0	712.5	716.5	1613.5	727.5	615.0	671.5	1665.5	830.5	736.0	815.0	672.0
Replicate 3	421.0	639.5	751.0	690.5	569.0	692.0	1211.5	750.0	737.0	1582.5	723.5	623.5	691.0	1695.5	844.0	741.0	826.0	743.5
Replicate 4	459.5	658.0	720.0	672.5	562.5	661.5	1177.0	694.5	697.5	1489.0	672.0	597.0	653.0	1575.5	779.5	683.0	769.0	668.0
Replicate 5	468.0	673.0	767.0	668.0	555.5	696.0	1210.5	737.0	699.0	1542.5	708.0	616.0	674.0	1621.5	825.5	690.5	786.0	651.0
ave	464.1	661.1	743.4	695.6	570.9	679.4	1215.2	723.8	714.4	1559.6	710.9	614.0	668.1	1639.6	818.7	712.0	799.5	679.3
RSD	6	2	3	4	2	2	2	3	2	3	3	2	2	3	3	4	3	5
% rel. rec.	73	90	95	89	87	92	89	93	93	88	90	86	92	89	89	88	89	92

Total Average % VOC Recovery = **89**

TIME = 7 Days -- >80% sand

Replicate 1	388.0	731.5	819.0	654.0	529.5	561.0	1073.0	758.0	702.5	1371.0	697.0	505.5	500.0	1447.0	714.5	665.5	694.0	747.5
Replicate 2	264.5	722.0	740.0	631.0	423.5	581.0	1054.5	737.0	768.5	1326.5	608.0	486.0	440.0	1382.5	677.5	651.5	711.0	768.0
Replicate 3	414.0	790.0	814.0	663.5	491.5	599.5	1095.5	710.0	711.0	1299.5	587.0	459.5	452.5	1316.0	626.5	607.5	645.5	733.5
Replicate 4	368.5	739.5	780.0	625.5	497.5	564.0	1040.0	647.5	640.5	1271.5	654.0	470.0	472.0	1325.0	658.5	624.5	663.0	751.5
Replicate 5	384.5	804.0	759.5	624.0	468.5	538.0	1030.0	698.0	658.0	1250.5	564.0	428.5	491.5	1285.0	639.5	595.0	635.5	717.0
ave	363.9	757.4	782.5	639.6	482.1	568.7	1058.6	710.1	696.1	1303.8	622.0	469.9	471.2	1351.1	663.3	628.8	669.8	743.5
RSD	16	5	4	3	8	4	2	6	7	4	9	6	5	5	5	5	5	3
% rel. rec.	57	103	99	82	73	77	77	92	90	73	79	66	65	73	72	78	75	101

Total Average % VOC Recovery = **80**

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene

Low Concentration Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Replicate 1	129.0	216.5	172.0	177.5	155.0	154.0	319.0	157.0	145.0	346.0	197.0	152.5	133.0	424.0	209.5	148.5	132.0	98.5
Replicate 2	145.5	190.5	160.0	169.5	149.0	155.5	310.0	164.0	142.0	395.0	181.0	145.5	114.0	386.5	192.5	144.0	138.5	101.0
Replicate 3	160.0	213.0	169.5	178.5	150.0	154.0	319.5	160.0	152.5	389.0	187.5	147.0	142.5	408.5	198.0	139.0	138.0	101.5
Replicate 4	127.5	213.0	156.5	165.5	140.0	151.0	284.0	141.5	129.5	372.5	179.5	137.5	107.0	350.5	176.0	124.0	126.0	91.5
Replicate 5	106.0	184.0	144.0	158.0	131.0	140.5	280.0	132.5	127.5	353.5	154.5	124.5	113.0	340.5	170.5	129.0	117.5	81.5
ave	133.6	203.4	160.4	169.8	145.0	151.0	302.5	151.0	139.3	371.2	179.9	141.4	121.9	382.0	189.3	136.9	130.4	94.8
RSD	15	7	7	5	7	4	6	9	8	6	9	8	12	9	8	7	7	9

TIME = 2 Days -- >80% sand

Replicate 1	100.0	180.0	145.5	153.0	127.5	127.0	274.0	142.5	129.0	343.0	151.0	122.5	107.5	346.0	165.5	125.5	117.0	89.5
Replicate 2	119.0	190.0	152.0	161.5	135.5	131.0	264.0	130.0	124.0	336.5	165.0	123.0	128.0	344.0	170.0	132.0	121.0	85.5
Replicate 3	106.0	177.5	152.0	154.0	138.0	127.0	262.5	150.5	132.5	339.5	162.0	119.0	113.0	346.5	168.0	119.0	115.5	82.5
Replicate 4	106.0	175.0	133.5	150.5	126.0	139.5	251.5	122.0	119.5	323.5	151.5	111.5	106.5	346.0	165.0	119.5	111.0	74.5
Replicate 5	113.5	180.5	151.0	156.0	123.0	138.0	265.0	136.5	119.5	334.0	151.0	113.5	118.5	328.5	165.0	118.0	114.5	74.0
ave	108.9	180.6	146.8	155.0	130.0	132.5	263.4	136.3	124.9	335.3	156.1	117.9	114.7	342.2	166.7	122.8	115.8	81.2
RSD	7	3	5	3	5	4	3	8	5	2	4	4	8	2	1	5	3	8
% rel. rec.	82	89	92	91	90	88	87	90	90	90	87	83	94	90	88	90	89	86

Total Average % VOC Recovery = **89**

TIME = 7 Days -- >80% sand

Replicate 1	85.0	292.0	141.5	129.0	100.0	109.5	215.0	122.0	138.5	266.5	133.0	97.5	79.0	296.5	153.0	140.5	150.0	166.5
Replicate 2	86.0	257.0	144.5	143.0	103.0	123.5	225.0	146.0	143.5	290.5	125.5	97.0	88.5	289.5	144.0	141.5	150.5	142.5
Replicate 3	88.0	298.0	151.0	121.0	113.5	90.0	214.0	135.5	141.5	278.0	134.0	93.0	53.5	297.0	153.0	139.0	145.0	129.0
Replicate 4	91.0	279.0	150.0	132.0	125.0	128.5	224.5	134.0	114.5	281.0	128.5	99.5	96.5	290.5	138.5	147.5	147.0	140.5
Replicate 5	93.0	263.5	143.5	120.5	110.0	119.0	220.5	164.0	139.5	254.0	122.0	84.5	83.0	288.5	133.5	128.0	138.0	155.5
ave	88.6	277.9	146.1	129.1	110.3	114.1	219.8	140.3	135.5	274.0	128.6	94.3	80.1	292.4	144.4	139.3	146.1	146.8
RSD	4	6	3	7	9	13	2	11	9	5	4	6	20	1	6	5	3	10
% rel. rec.	66	137	91	76	76	76	73	93	97	74	71	67	66	77	76	102	112	155

Total Average % VOC Recovery = **88**

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene

High Concentration Theoretical Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Prespike	786.5	804.0	788.5	830.0	738.0	777.5	1499.0	781.0	766.5	1852.0	847.0	759.5	729.0	1943.0	948.5	843.0	924.5	701.5
Postspike	782.0	784.0	833.5	831.5	721.0	740.0	1480.0	820.0	779.0	1839.0	796.0	725.0	754.5	1812.5	932.0	763.5	858.5	693.0
ave	784.3	794.0	811.0	830.8	729.5	758.8	1489.5	800.5	772.8	1845.5	821.5	742.3	741.8	1877.8	940.3	803.3	891.5	697.3
T=0/direct	81	92	97	94	90	97	92	97	100	96	96	96	98	98	98	100	100	106

Low Concentration Theoretical Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Prespike	172.5	227.5	337.0	181.5	184.5	157.0	325.5	150.5	143.0	395.0	172.0	153.0	143.0	403.0	206.5	142.0	136.0	96.0
Postspike	184.0	241.0	176.0	189.5	168.5	154.5	325.0	153.5	144.0	409.0	172.5	146.5	137.5	397.5	199.0	143.5	135.5	95.0
ave	178.3	234.3	256.5	185.5	176.5	155.8	325.3	152.0	143.5	402.0	172.3	149.8	140.3	400.3	202.8	142.8	135.8	95.5
T=0/direct	75	87	63	92	82	97	93	99	97	92	104	94	87	95	93	96	96	99

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene

APPENDIX 2(Biologically Active Soil) High Concentration Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Replicate 1	476.5	796.0	592.5	577.0	526.5	508.0	1037.5	554.5	547.0	1164.5	608.0	476.5	337.0	1297.5	594.0	607.5	609.0	564.0
Replicate 2*	362.5	673.0	558.0	531.0	403.0	400.0	872.0	473.0	482.5	996.5	498.5	372.0	319.0	1087.0	523.0	492.0	504.5	495.5
Replicate 3	516.5	792.5	694.0	615.5	579.5	512.0	1092.5	618.5	608.5	1336.5	664.0	530.5	428.5	1429.0	712.5	603.5	627.5	613.5
Replicate 4	532.5	816.5	635.5	656.0	519.0	524.5	1086.5	578.5	586.0	1305.0	619.0	482.5	376.5	1407.5	688.0	634.5	662.5	597.5
Replicate 5	498.0	792.5	647.0	602.5	507.5	500.0	1014.0	610.5	606.0	1225.0	560.0	465.5	352.5	1306.5	602.5	571.5	605.5	540.5
ave	477.2	774.1	625.4	596.4	507.1	488.9	1020.5	567.0	566.0	1205.5	589.9	465.4	362.7	1305.5	624.0	581.8	601.8	562.2
RSD	14	7	8	8	13	10	9	10	9	11	11	12	12	10	12	9	10	8

* syringe plugged during spiking

TIME = 2 Days -- Garden Soil

Replicate 1	455.5	785.0	664.0	611.0	543.0	498.0	1039.5	594.0	569.0	1216.5	624.0	495.0	371.0	1338.5	628.5	649.5	665.5	611.5
Replicate 2	441.5	792.0	649.0	635.5	525.0	514.0	1027.5	590.5	618.0	1227.5	639.0	485.0	357.0	1347.5	648.0	627.0	643.5	574.0
Replicate 3	532.0	864.0	719.0	655.0	568.0	519.0	1073.5	661.0	619.5	1265.5	658.0	487.5	424.0	1309.0	636.5	652.0	703.5	619.5
Replicate 4	407.0	677.5	609.0	603.0	488.2	496.5	994.0	559.5	525.0	1188.5	577.5	459.5	413.0	1276.5	655.0	600.5	667.0	541.0
Replicate 5	469.0	803.0	683.0	614.5	535.0	494.5	1026.0	624.5	565.5	1221.0	561.5	459.5	365.0	1293.0	639.5	595.5	632.5	589.5
ave	461.0	784.3	664.8	623.8	531.8	504.4	1032.1	605.9	579.4	1223.8	612.0	477.3	386.0	1312.9	641.5	624.9	662.4	587.1
RSD	10	9	6	3	5	2	3	6	7	2	7	3	8	2	2	4	4	5
% rel. rec.	97	101	106	105	105	103	101	107	102	102	104	103	106	101	103	107	110	104

Total Average % VOC Recovery = 104

TIME = 7 Days -- Garden Soil

Replicate 1	440.5	783.0	766.0	747.5	579.0	628.0	1134.0	734.0	700.0	1389.0	669.0	475.5	427.0	1425.0	713.5	673.5	735.5	657.5
Replicate 2	439.5	790.5	748.5	760.5	556.5	623.0	1125.5	700.5	640.5	1362.0	646.0	453.5	452.0	1345.5	690.5	668.5	698.5	643.5
Replicate 3	410.5	728.5	699.5	713.0	515.5	589.5	1053.0	689.0	621.5	1281.0	619.0	450.0	426.0	1359.5	662.5	660.5	704.0	692.5
Replicate 4	415.0	752.0	746.0	744.0	546.5	629.0	1106.5	714.0	639.5	1341.0	624.0	449.5	450.0	1352.0	685.5	659.0	692.5	687.5
Replicate 5	340.5	712.5	684.0	658.0	534.5	593.5	1019.5	684.5	614.5	1277.0	610.5	431.5	417.5	1286.5	641.0	615.0	672.0	630.0
ave	409.2	753.3	728.8	724.6	546.4	612.6	1087.7	704.4	643.2	1330.0	633.7	452.0	434.5	1353.7	678.6	655.3	700.5	662.2
RSD	10	4	5	6	4	3	5	3	5	4	4	3	4	4	4	4	3	4
% rel. rec.	86	97	117	121	108	125	107	124	114	110	107	97	120	104	109	113	116	118

Total Average % VOC Recovery = 111

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene

Low Concentration Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Replicate 1	191.5	200.0	182.0	181.0	123.0	124.5	309.0	150.0	144.0	361.0	146.0	129.0	104.5	345.5	176.5	139.0	158.5	152.0
Replicate 2	198.0	225.0	173.0	171.5	122.0	129.0	287.5	135.0	135.5	362.5	160.5	127.5	71.0	356.5	188.5	152.0	145.5	150.5
Replicate 3	205.5	234.5	174.0	157.0	111.0	118.5	283.5	144.0	125.5	361.0	147.0	124.0	80.5	336.0	174.5	142.5	150.5	145.0
Replicate 4	180.5	231.5	174.0	128.5	120.0	124.5	300.0	137.5	133.5	355.5	146.5	120.0	78.0	328.5	176.0	141.0	151.0	139.0
Replicate 5	181.5	221.0	165.5	157.5	104.5	103.0	268.5	124.0	119.5	320.5	126.0	111.0	92.0	301.0	150.5	129.5	138.0	130.0
ave	191.4	222.4	173.7	159.1	116.1	119.9	289.7	138.1	131.6	352.1	145.2	122.3	85.2	333.5	173.2	140.8	148.7	143.3
RSD	6	6	3	12	7	8	5	7	7	5	8	6	15	6	8	6	5	6

TIME = 2 Days -- Garden Soil

Replicate 1	172.0	231.0	180.0	167.5	113.5	115.0	237.0	134.0	125.5	293.5	138.5	108.0	93.0	344.0	166.5	146.5	157.5	151.5
Replicate 2	134.0	173.5	158.5	145.0	102.0	116.0	202.0	131.0	110.5	323.5	137.0	97.5	52.5	304.5	149.0	136.0	137.0	128.5
Replicate 3	162.0	232.5	165.0	166.5	111.0	114.0	215.5	125.5	128.0	267.5	129.5	101.5	68.5	301.5	153.0	129.5	144.0	118.0
Replicate 4	152.5	194.5	173.5	153.0	105.5	119.0	220.5	132.5	120.0	278.5	138.0	105.5	73.5	329.5	160.5	138.5	148.5	140.5
Replicate 5	165.5	197.0	176.0	175.0	127.0	122.5	220.5	132.5	123.0	357.0	149.0	116.0	64.5	327.5	158.5	138.5	147.0	147.0
ave	157.2	205.7	170.6	161.4	111.8	117.3	219.1	131.1	121.4	304.0	138.4	105.7	70.4	321.4	157.5	137.8	146.8	137.1
RSD	9	12	5	8	9	3	6	3	6	12	5	7	21	6	4	4	5	10
% rel. rec.	82	92	98	101	96	98	76	95	92	86	95	86	83	96	91	98	99	96

Total Average % VOC Recovery = **92**

TIME = 7 Days -- Garden Soil

Replicate 1	92.5	277.0	179.5	184.5	141.5	148.5	197.5	167.5	120.5	273.0	149.0	92.0	71.0	335.0	167.5	173.5	168.0	169.0
Replicate 2*	67.5	220.0	143.5	122.5	104.5	103.0	124.5	123.5	88.0	167.5	104.0	64.0	76.5	242.5	125.5	133.5	130.0	152.5
Replicate 3	97.0	277.5	156.5	151.5	130.5	129.0	175.5	152.0	99.5	243.5	134.0	84.5	81.0	291.5	144.0	142.5	154.0	154.0
Replicate 4	93.0	265.0	163.0	157.5	137.0	131.0	186.0	146.0	120.0	236.5	153.5	81.0	78.5	314.5	158.5	157.5	158.0	159.5
Replicate 5	90.0	251.5	143.0	156.0	130.5	121.0	170.5	147.5	99.0	217.5	137.5	74.5	82.5	296.5	147.5	140.5	151.5	135.5
* spike pooled on surface of soil plug during spiking procedure																		
ave	88.0	258.2	157.1	154.4	128.8	126.5	170.8	147.3	105.4	227.6	135.6	79.2	77.9	296.0	148.6	149.5	152.3	154.1
RSD	13	9	10	14	11	13	16	11	14	17	14	13	6	12	11	11	9	8
% rel. rec.	46	116	90	97	111	106	59	107	80	65	93	65	91	89	86	106	102	108

Total Average % VOC Recovery = **90**

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene

High Concentration Theoretical Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Prespike	672.5	959.0	742.0	788.5	658.5	669.5	1354.5	736.5	720.5	1657.5	760.5	688.0	498.5	1776.0	857.0	761.0	846.5	752.0
Postspike	656.0	954.5	788.0	739.5	639.5	613.5	1328.0	749.0	756.0	1559.5	690.0	621.5	544.0	1608.0	744.5	702.5	755.0	737.5
ave	664.3	956.8	765.0	764.0	649.0	641.5	1341.3	742.8	738.3	1608.5	725.3	654.8	521.3	1692.0	800.8	731.8	800.8	744.8
T=0/direct	72	77	103	102	102	115	102	104	104	111	109	109	140	109	115	110	112	99

Low Concentration Theoretical Samples

	VC	MC	MTBE	CF	CT	BDCM	BZ	TCA	EDB	TOL	EB	STY	BF	MPX	OX	135TMB	124TMB	NAP
TIME = 0																		
Prespike*	200.0	231.0	189.0	196.0	119.5	129.0	311.5	146.5	134.0	455.0	168.5	145.0	77.5	371.0	178.5	155.5	164.5	142.0
Postspike	262.0	270.0	187.5	180.0	124.0	122.0	316.5	154.0	136.5	476.0	164.0	136.0	82.0	366.5	184.0	148.5	150.5	132.0
ave	231.0	250.5	188.3	188.0	121.8	125.5	314.0	150.3	135.3	465.5	166.3	140.5	79.8	368.8	181.3	152.0	157.5	137.0
T=0/direct	83	89	92	85	95	96	92	92	97	76	87	87	107	90	96	93	94	105

* voa vial lid was loosened in sonicator

All results are expressed in ug/kg.

ANALYTE KEY

VC	Vinyl chloride	BZ	Benzene	BF	Bromoform
MC	Methylene chloride	TCA	1,1,2-Trichloroethane	MPX	m/p-Xylene
MTBE	Methyl tert-butyl ether	EDB	Ethylene dibromide	OX	o-Xylene
CF	Chloroform	TOL	Toluene	135TMB	1,3,5-Trimethylbenzene
CT	Carbon tetrachloride	EB	Ethylbenzene	124TMB	1,2,4-Trimethylbenzene
BDCM	Bromodichloromethane	STY	Styrene	NAP	Naphthalene