OM100R[a]

Analysis of Semivolatile Organic Compounds Using Capillary Gas Chromatography with Ion Trap Mass Spectrometric Detection

Note: **This is a draft method and is made available for use at your discretion. The method has been used in at least one laboratory, but has not been extensively reviewed. Performance parameters, such as precision and bias, may not be available. If you use this method, please provide the editors of *DOE Methods* with comments. Please also provide any performance data you may generate. Your input will assist us in verifying this method.**

The decision to use this method or a modified version of this method should be made during project planning (see Chapter 1). Project personnel, together with stakeholders, should determine the activities that are needed to ensure that this method is appropriate for use in the project (see Appendix B).

1.0 Scope and Application

1.1 This method is very similar to SW-846 Method 8270B (EPA 1994) in that it describes the determination of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and groundwater. It is included as an appendix to Chapter 8 because in addition, it provides method performance data for mixed waste samples found at U. S. Department of Energy (DOE) sites. It differs from Method 8270B because it incorporates the use of a gas chromatograph/ion trap mass spectrometer (GC-ITS) in place of the GC-quadrupole mass selective detector (GC-MSD) systems typically used. Use of the GC-ITS is specifically allowed in the proposed update III to SW-846 Method 8270C.

1.2 The reader is referred to Method 8270 for most details. Information is provided here as necessary to incorporate the distinctive features of the new method.

1.3 As noted in Method 8270, phenols, anilines, benzoic acid, 3,3'-dichlorobenzidine, and the late eluting phthalates and polycyclic aromatic hydrocarbons (PAHs) are subject to poor and/or erratic chromatographic performance. Improved performance for these compounds was obtained using cold on-column injection with a temperature-programmable injection port. Incorporation of a pressure-controlled flow system may further enhance the chromatographic behavior of these compounds. Phthalates, phenol, and benzoic acid also may be subject to background contamination and/or carryover. Multiple blanks can be performed to help minimize this problem.

(a) This method was submitted by S-A. Barschick, S. Worthy, and W. H. Griest (Oak Ridge National Laboratory, Oak Ridge, Tennessee).

1.4 The analysis of radioactive materials requires that health physics personnel monitor the instrument and laboratory. Additionally, extracts prepared from radioactive samples should be surveyed by a health physicist before transfer to the GC-ITS laboratory.

2.0 Summary of Method

This method provides the conditions for the determination of semivolatile organic compounds using capillary GC with an ion trap detector when the conditions are different from those used in SW-846 Method 8270B.

3.0 Interferences

See SW-846 Method 8270B.

4.0 Safety

Gloves and protective clothing should be worn to protect against unnecessary exposure to organic solvents and contaminants. When handling radioactive samples, all applicable radiochemical handling procedures and health physics monitoring practices should be followed.

5.0 Apparatus and Materials

5.1 Gas Chromatograph/Ion Trap Mass Spectrometer System

5.1.1 **Gas Chromatograph** - A GC equipped with a temperature-programmable oven and a suitable mass spectrometric (MS) interface. An injection port capable of operation in the splitless injection mode is suitable providing the GC is equipped with a pressure-controlled flow system. For flow-controlled systems, a temperature-programmable injection port capable of cold on-column injections provides better overall chromatographic performance. A Varian model 3300 equipped with both a 1077 split/splitless injection port and a septum-equipped programmable injector (SPI) was used in the method development and performance evaluation.

5.1.2 **Capillary Columns** - 30 m long x 0.25-mm ID, 0.25-µm film thickness DB-5MS fused silica capillary column (J&W Scientific or equivalent ultra-low bleed column). When on-column injection is used, the analytical column is connected to a ≤ 0.5 m long x 0.53-mm ID deactivated fused-silica guard column for on-column injection. The columns are connected using a glasSeal connector (Supelco).

5.1.3 **Ion Trap Mass Spectrometer -** An ITS capable of scanning from 35 to 500 amu every 1 sec or less and operating in the electron-impact ionization mode using 70 volts electron energy. A Finnigan MAT Magnum ion trap spectrometer and a GC-MS interface were used in the method development and performance evaluation.

5.1.4 **Data System -** See Method 8270.

5.2 Other Equipment - see Method 8270.

6.0 Reagents

6.1 Solvents - Pesticide-grade methylene chloride, methanol, and other appropriate solvents used in the extraction cleanup procedures.

6.2 Semivolatile Organic Compound Standards - U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) standards, including semivolatile (SV) calibration mix numbers 1, 2, 3, 4, 5, and 7, and 3,3'-dichlorobenzidine (Restek or equivalent). Ampules contain 2,000 µg/mL of each compound in methylene chloride or methanol. Fifty microliters of each mixture should be combined in a total volume of 1 mL diluted with methylene chloride to prepare a 100-µg/mL stock calibration solution from which to prepare calibration standards. Appropriate dilutions of this stock solution are used to prepare a minimum of five calibration standards.

6.3 Internal Standards - The internal standards used included 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. These were purchased as EPA CLP standards in ampules containing 4,000 µg/mL of each component in methylene chloride. Twenty-five microliters of this standard are diluted in 1 mL of methylene chloride to prepare a 100-µg/mL internal standard stock solution to be used for spiking calibration standards and extracts. Each 1-mL calibration standard or sample extract undergoing analysis should be spiked with 10 µL of the 100-µg/mL internal standard solution, resulting in a concentration of 1 ng/µL of each internal standard. Note that this is a different concentration from that in Method 8270B.

6.4 Surrogate Standards - The surrogate standards used included phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. These were purchased as EPA CLP ampules containing 2,000 µg/mL acid surrogate standard mix in methanol and 1,000 µg/mL of base/neutral surrogate standard mix in methylene chloride. For preparation of calibration standards, 50 µL of acid surrogate mix and 100 µL of base/neutral surrogate mix are added to the 1-mL stock calibration solution to obtain 100 µg/mL of each surrogate. For surrogate spiking of samples, a 10-µg/mL surrogate stock spiking solution is prepared by diluting 50 µL of acid surrogate mix and 100 µL of base/neutral surrogate mix to a total volume of 10 mL with methylene chloride. Each sample undergoing extraction should be spiked with the surrogate stock-spiking solution to obtain 2 µg/mL of base/neutral and acid surrogates in the resulting 1 mL of extract. Note that this is a different concentration from that in Method 8270B.

6.5 GC-MS Tuning Standard - A methylene chloride solution containing 10 ng/µL of decafluorotriphenylphosphine (DFTPP) can be used as a GC-MS tuning standard to evaluate MS performance. This standard also could contain 10 ng/µL each of pentachlorophenol and benzo(g,h,i)perylene to verify injection port inertness and GC column performance. Note that these are different concentrations than those in Method 8270B.

7.0 Sample Collection, Preservation, and Handling

Sample collection, preservation, and handling should be addressed in the planning process. Radioactive samples and resulting extracts should be handled according to facility health physics procedures.

8.0 Procedure

8.1 GC-MS Operating Parameters

8.1.1 Recommended Operating Conditions

Mass Range: 45 - 500 amu
Scan Rate: 1 sec/scan
Automatic Gain Control: on
Filament Delay: 300 sec
Source Temperature: 220°C
Emission Current: 50 µA

Transfer Line Temperature: 290°C
Initial Column Temperature: 40°C hold 4 min
Temperature Program: 40-280°C @ 8°C/min

Injection parameters
Injection mode: splitless, 2 min
Injection port temperature: 270°C
Injection volume: 1 µL
Column head pressure: 15 psi
Carrier gas: helium @ 45 cm/s (@ 40°C)

Injection parameters	
Injection mode:	on-column
Injection port temperature:	35°C hold 0.1 min
Temperature program:	35-280°C @ 180°C/min
Column head pressure:	16 psi
Carrier gas:	helium @ 50 cm/s (@ 40°C)

8.1.2 **MS Tuning Procedure -** To ensure proper operation of the MS, a weekly tuning procedure should be followed. The automatic tuning procedure monitors air and water abundances to check for leaks, electronic noise zero, and calibration gas pressure. The tuning procedure also allows automatic optimization of multiplier voltage, filament emission current, and automatic gain control (AGC) target and performs mass calibration under the optimized parameters. Preferably, multiplier voltage, filament emission current, and AGC target can be tuned manually. The appropriate MS operation manual should be referred to if parameters do not fall within set limits or for guidelines on manual tuning procedures. If manually tuning, it is best to perform the mass calibration at the mid-range temperature of the GC program used for analysis (200°C).

8.1.3 **GC-ITS Tuning Procedure -** To test system performance before samples are analyzed, the GC-MS tuning standard should be analyzed. A 1-µL injection of DFTPP should result in a mass spectrum that meets the criteria listed in Table 1. The tailing factor (at 10% peak height) for pentachlorophenol should be ≤ 5. The peak width (at 10% peak height) for benzo(g,h,i)perylene should be ≤ 0.4 min. Failure to meet these criteria should result in corrective actions (see below).

Table 1. DFTPP Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	24 to 28% of mass 442
69	17 to 20% of mass 442
127	10 to 12% of mass 442
198	39 to 48% of mass 442
199	<10% of mass 198
275	~10% of mass 442
424	4% of mass 442
442	Base peak, 100% relative abundance
443	18 to 20% of mass 442
444	≤2% of mass 442

8.2 **Initial Calibration -** A minimum of five calibration standards should be prepared covering the working range of the GC-MS system. Ten calibration standards, covering a working range of 50 to 2,000 ng/mL, were found to work best. Each of the calibration standards (spiked with 10 µL of internal standard solution) should be analyzed and the extracted ion current profile (EICP) integrated using the primary characteristic ion of each compound. Table 2 lists the internal standards with the corresponding analytes assigned for quantitation, the characteristic ions, and retention times. (Note that elution order and retention times may vary slightly from those listed in the table, depending on the column and chromatographic operating conditions used.)

Table 2. Quantitation Parameters for SVOCs

Compound Name	Retention Time	Quant.Ion	Characteristic Ions
1,4-dichlorobenzene-d_4 (IS)	10:27	150	152, 115
2-fluorophenol	6:58	112	92, 64
phenol-d_6	9:38	99	71
phenol	9:41	94	66
2-chlorophenol	9:52	128	130, 64, 63
bis(2-chloroethyl) ether	9:52	93	95, 63, 49
1,3-dichlorobenzene	10:16	146	148, 111
1,4-dichlorobenzene	10:30	146	148, 111
1,2-dichlorobenzene	10:54	146	148, 111
benzyl alcohol	10:59	79	108, 107, 77
2-methylphenol	11:22	107	108, 79, 77
bis(2-chloroisopropyl) ether	11:26	45	121, 77
N-nitrosodi-n-propylamine	11:47	70	130, 113
4-methylphenol	11:51	107	108, 79, 77
hexachloroethane	11:52	117	203, 201, 166, 119
naphthalene-d_8 (IS)	14:12	136	108, 66
nitrobenzene-d_5	12:05	82	128, 54
nitrobenzene	12:08	77	123, 51
isophorone	12:55	82	138, 54
2-nitrophenol	13:05	139	109, 81, 65
2,4-dimethylphenol	13:24	107	122, 121, 77
benzoic acid	13:34	105	122, 77, 49
bis(2-chloroethoxy)methane	13:42	93	95, 63
2,4-dichlorophenol	13:51	162	164, 98, 63
1,2,4-trichlorobenzene	14:03	182	184, 180, 145, 109
naphthalene	14:15	128	129, 127, 102, 63
4-chloroaniline	14:32	127	129, 92, 65
hexachlorobutadiene	14:43	225	260, 227, 223, 190
4-chloro-3-methylphenol	16:09	107	144, 142, 77
2-methylnaphthalene	16:25	141	142, 115
acenaphthene-d_{10} (IS)	19:35	162	164, 160, 80

Table 2. Quantitation Parameters for SVOCs (Cont.)

Compound Name (Cont.)	Retention Time	Quant.Ion	Characteristic Ions
hexachlorocyclopentadiene	16:55	237	272, 239, 235
2,4,6-trichlorophenol	17:20	198	196, 132, 97
2,4,5-trichlorophenol	17:27	198	196, 132, 97
2-fluorobiphenyl	17:39	172	173, 171, 50
2-chloronaphthalene	17:52	162	164, 127
2-nitroaniline	18:17	65	138, 108, 92, 80
dimethylphthalate	19:00	163	133, 77
2,6-dinitrotoluene	19:07	165	89, 77, 63, 51
acenaphthylene	19:08	152	153, 151, 76, 63
3-nitroaniline	19:35	65	138, 92, 80
acenaphthene	19:41	153	154, 152, 76, 63
2,4-dinitrophenol	19:54	154	184, 107, 63
dibenzofuran	20:14	168	139, 63
4-nitrophenol	20:17	109	139, 81, 65
2,4-dinitrotoluene	20:20	165	119, 89, 63
diethylphthalate	21:14	149	177, 176, 76, 65
fluorene	21:18	165	166, 139, 83, 63
4-chlorophenyl phenyl ether	21:26	204	206, 141, 51
4-nitroaniline	21:26	108	138, 92, 80, 65
phenanthrene-d_{10} (IS)	24:11	188	160, 94, 80
4,6-dinitro-2-methylphenol	21:33	198	121, 105, 51
N-nitrosodiphenylamine	21:50	169	168, 167, 77, 51
2,4,6-tribromophenol	22:04	332	334, 330, 143, 141
4-bromophenyl phenyl ether	22:56	250	248, 141, 77, 51
hexachlorobenzene	22:59	284	286, 249, 214, 142
pentachlorophenol	23:39	266	268, 264, 230, 167
phenanthrene	24:16	178	179, 177, 152, 76
anthracene	24:26	178	179, 152, 76
carbazole	25:01	167	168, 166, 139
di-n-butyl phthalate	26:25	149	150, 104
fluoranthene	27:58	202	203, 201, 101, 88
chrysene-d_{12} (IS)	32:28	240	241, 237, 118
pyrene	28:39	202	203, 201,101, 88
terphenyl-d_{14}	29:22	244	245, 213,122
butyl benzyl phthalate	31:04	149	206, 104, 91
benz(a)anthracene	32:26	228	229, 227, 113, 101
3,3'-dichlorobenzidine	32:30	252	254, 253, 127, 77
chrysene	32:33	228	229, 227, 113, 101
bis(ethylhexyl) phthalate	33:00	149	279, 167, 71
perylene-d_{12} (IS)	37:12	264	265, 261, 132
di-n-octyl phthalate	35:01	149	279
benzo(b)fluoranthene	35:48	252	253, 251, 126, 125, 113
benzo(k)fluoranthene	35:54	252	253, 251, 126, 125, 113

Table 2. Quantitation Parameters for SVOCs (Cont.)

Compound Name (Cont.)	Retention Time	Quant.Ion	Characteristic Ions
benzo(a)pyrene	36:59	252	253, 251, 126, 125, 113
indeno(1,2,3-cd)pyrene	42:25	276	274, 138
dibenz(a,h)anthracene	42:43	278	279, 277, 139, 138
benzo(g,h,i)perylene	43:59	276	275, 138

8.3 **GC-ITS Performance Checks -** A calibration standard at mid-concentration range (containing all analytes and surrogates) should be analyzed routinely to verify the initial calibration. A 1,000 ng/mL calibration standard was used in this study. Response factors (RFs) for each compound should be compared to the average RFs from the initial calibration and should meet criteria similar to those described in Method 8270B under "initial calibration" and "daily GC-MS calibration." Caution: radioactive material from low-level mixed waste sample extracts may accumulate in deposits in the injection port liner or guard column. Items should be handled carefully when attempting to correct chromatographic problems.

8.4 GC-ITS Analysis

8.4.1 Sample extracts are obtained from appropriate cleanup procedures. Each 1-mL extract should be spiked with 10 µL of the 100-µg/mL internal standard solution to obtain 1 µg/mL each of the internal standards before analysis. Each 1-mL extract is analyzed using the GC-ITS method described above. The injected volume for sample extracts should ideally contain 2 ng of the base/neutral and acid surrogates.

8.4.2 Qualitative and quantitative analysis should follow the guidelines found in Method 8270B. The MS operation manual should be consulted for guidelines on quantitation software.

9.0 Quality Control

9.1 The use of this method either as is or with modifications should be supported by appropriate quality control (QC) procedures (e.g., as outlined in Chapter 3).

10.0 Method Performance

The evaluation of method performance was based on the results of the initial calibration procedure, the ability to meet the performance checks, limits of detection, and results from the analysis of performance evaluation samples and comparison of GC-ITS and GC-MSD results for solid and/or mixed waste extracts.

10.1 **Initial Calibration -** Initial calibrations were performed on four separate occasions under slightly different conditions to attempt to meet the calibration and quantitation criteria (discussed below). Wide variations in analyte sensitivities presented a problem in meeting the calibration criteria. Contributing to this problem was the decrease in analyte response as a function of elution temperature when operated in the splitless injection mode. This was overcome to some extent using the cold on-column injection technique and the temperature-programmable injector. Another contribution to this problem is the limited dynamic range of the ion trap. In this case, difficulty was encountered in determining a suitable concentration range for calibration such that certain compounds would not go undetected, while other compounds would not overload the capillary column or ion trap. This was overcome to some extent by using more than five calibration standards and choosing a much narrower concentration range.

10.1.1 System performance checks are performed to ensure that minimum RFs are met and the validity of the initial calibration is checked before the calibration curve is used. Three attempts at an initial calibration were made using the splitless injection parameters. The first attempt used six calibration standards ranging from 10 ng/mL to 5 µg/mL. The second used nine calibration standards from 10 ng/mL to 75 µg/mL. A much narrower concentration range, 200 ng/mL to 5 µg/mL with six standards, was used for the third attempt. In all three initial calibration trials, the SW-846 Method 8270A system performance check compound (SPCC) and calibration check compound (CCC) quantitation criteria for the GC-MSD could not be met. The SPCCs that did not meet the minimum response factor of 0.05 included 2,4-dinitrophenol and 4-nitrophenol. The CCCs that had response factors with percent differences greater than 30% included pentachlorophenol, di-n-octyl phthalate, and benzo(a)pyrene. A final attempt was made to prepare an initial calibration meeting all quantitation criteria. This attempt used the SPI injection parameters and a concentration range from 50 ng/mL to 2 µg/mL using 9 calibration standards. Using the narrower concentration range and the on-column injection technique, all quantitation criteria were met for this initial calibration. Note that it was necessary to not include some of the lower concentration standards in the initial calibration, but a minimum of five standards were included for the determination of the RF for every compound.

10.1.2 Certified reporting limits (CRLs) and method detection limits (MDLs) were determined for each compound. Calibration standards were prepared from 5 ng/mL to 20 µg/mL and analyzed in triplicate using the splitless injection mode.

This unusually wide concentration range was chosen to determine and encompass the range of analyte sensitivities detected using an ion trap spectrometer. Quantitation limits for each of the compounds are reported in Table 3. Values are listed for the entire concentration range analyzed (concentration range ≥ 4 orders of magnitude) and for the most linear region of this concentration range covering a three-order linear dynamic range. With this high sensitivity and dynamic range, the regulatory limits for each analyte easily can be met and exceeded using the GC-ITS system. The reason for the somewhat higher detection limits for some of the analytes can be attributed to a greater difficulty in chromatographing those compounds that tended to tail or be subject to peak broadening.

Table 3. Quantitation Limits for SVOCs

Compound Name	Quant. range[(a)]	CRLs[(b)]	MDLs[(c)]	Quant. range[(a)]	CRLs[(b)]	MDLs[(c)]
2-fluorophenol	5 to 10000	520	380	5 to 500	19	14
d_6-phenol	5 to 10000	1200	840	5 to 500	36	26
phenol	5 to 10000	800	580	5 to 500	43	31
2-chlorophenol	5 to 10000	730	530	5 to 500	36	26
bis(2-chloroethyl) ether	5 to 10000	1200	890	5 to 500	36	26
1,3-dichlorobenzene	5 to 10000	780	560	5 to 500	22	16
1,4-dichlorobenzene	5 to 10000	910	660	5 to 500	37	27
1,2-dichlorobenzene	5 to 10000	780	560	5 to 500	36	26
benzyl alcohol	5 to 10000	920	670	5 to 500	54	40
2-methylphenol	5 to 10000	950	690	5 to 500	38	27
bis(2-chloroisopropyl) ether	5 to 10000	920	660	5 to 500	58	42
N-nitrosodi-n-propylamine	5 to 10000	850	620	5 to 500	50	36
4-methylphenol	5 to 10000	980	700	5 to 500	58	42
hexachloroethane	5 to 10000	760	550	5 to 500	27	20
d_5-nitrobenzene	5 to 10000	1200	860	5 to 500	70	51
nitrobenzene	5 to 10000	690	500	5 to 500	100	76
isophorone	5 to 10000	470	340	5 to 500	63	46
2-nitrophenol	5 to 10000	1400	1000	5 to 500	74	54
2,4-dimethylphenol	5 to 10000	890	640	5 to 500	52	38
benzoic acid	5 to 10000	1100	820	5 to 1000	280	210
bis(2-chloroethoxy) methane	5 to 10000	710	510	5 to 500	77	56
2,4-dichlorophenol	5 to 10000	370	270	5 to 500	50	36
1,2,4-trichlorobenzene	5 to 10000	320	230	5 to 500	46	34
naphthalene	5 to 10000	500	360	5 to 500	29	21
4-chloroaniline	5 to 10000	990	720	5 to 500	96	70

(a) Concentration range, in ng/mL, includes zero

(b) Certified Reporting Limits (CRLs), in pg injected, are based on 95% confidence limits (5% positive and negative intervals).

(c) Method Detection Limits (MDLs), in pg injected, are based on a 99% confidence limit (1% positive and 0% negative intervals).

Table 3. Quantitation Limits for SVOCs (Cont.)

Compound Name (Cont.)	Quant. range[a]	CRLs[b]	MDLs[c]	Quant. range[a]	CRLs[b]	MDLs[c]
hexachlorobutadiene	5 to 10000	320	230	5 to 500	20	15
4-chloro-3-methylphenol	5 to 10000	490	360	5 to 1000	57	41
2-methylnaphthalene	5 to 10000	320	230	5 to 500	28	21
hexachlorocyclopentadiene	5 to 10000	1200	860	5 to 500	39	29
2,4,6-trichlorophenol	5 to 10000	740	530	5 to 500	31	23
2,4,5-trichlorophenol	5 to 10000	1700	1200	5 to 500	51	37
2-fluorobiphenyl	5 to 10000	660	480	5 to 500	64	46
2-chloronaphthalene	5 to 10000	700	500	5 to 500	59	43
2-nitroaniline	5 to 10000	560	400	5 to 500	280	200
dimethylphthalate	5 to 10000	430	310	5 to 500	46	34
2,6-dinitrotoluene	5 to 10000	1100	760	5 to 500	55	40
acenaphthylene	5 to 10000	560	400	5 to 500	39	29
3-nitroaniline	5 to 10000	1300	950	5 to 500	97	71
acenaphthene	5 to 10000	580	420	5 to 500	42	31
2,4-dinitrophenol	5 to 10000	1800	1300	5 to 500	130	94
dibenzofuran	5 to 10000	830	600	5 to 500	62	45
4-nitrophenol	5 to 10000	2200	1600	5 to 500	70	51
2,4-dinitrotoluene	5 to 10000	950	680	5 to 500	100	74
diethylphthalate	5 to 10000	680	490	5 to 500	43	31
fluorene	5 to 10000	670	490	5 to 500	36	26
4-chlorophenyl phenyl ether	5 to 10000	730	520	5 to 500	58	42
4-nitroaniline	5 to 10000	850	610	5 to 500	74	54
4,6-dinitro-2-methylphenol	5 to 10000	2100	1500	5 to 500	81	59
N-nitrosodiphenylamine	5 to 10000	470	340	5 to 500	53	38
2,4,6-tribromophenol	5 to 10000	700	500	5 to 500	36	26
4-bromophenyl phenyl ether	5 to 10000	860	620	5 to 500	34	25
hexachlorobenzene	5 to 10000	730	530	5 to 500	37	27
pentachlorophenol	5 to 10000	1600	1200	5 to 1000	290	210
phenanthrene	5 to 10000	580	420	5 to 500	36	26
anthracene	5 to 10000	600	430	5 to 500	33	24
carbazole	5 to 10000	750	540	5 to 500	39	29
di-n-butyl phthalate	5 to 10000	520	380	5 to 500	60	44
fluoranthene	5 to 10000	600	440	5 to 500	74	54
pyrene	5 to 10000	860	620	5 to 500	73	53
d14-terphenyl	5 to 10000	1300	910	5 to 500	53	39
butyl benzyl phthalate	5 to 10000	1200	900	5 to 500	100	76
benz(a) anthracene	5 to 10000	980	700	5 to 500	62	46
3,3'-dichlorobenzidine	5 to 10000	2900	2100	5 to 500	120	86
chrysene	5 to 10000	820	590	5 to 500	69	50
bis(ethylhexyl) phthalate	5 to 10000	1300	940	5 to 500	360	260
di-n-octyl phthalate	5 to 10000	2300	1700	5 to 500	210	160
benzo(b) fluoranthene	5 to 10000	1600	1100	5 to 500	150	110
benzo(k) fluoranthene	5 to 10000	1500	1100	5 to 500	44	32

(a) Concentration range, in ng/mL, includes zero

(b) Certified Reporting Limits (CRLs), in pg injected, are based on 95% confidence limits (5% positive and negative intervals).

(c) Method Detection Limits (MDLs), in pg injected, are based on a 99% confidence limit (1% positive and 0% negative intervals).

Table 3. Quantitation Limits for SVOCs (Cont.)

Compound Name (Cont.)	Quant. range[(a)]	CRLs[(b)]	MDLs[(c)]	Quant. range[(a)]	CRLs[(b)]	MDLs[(c)]
benzo(a) pyrene	5 to 10000	1100	800	5 to 500	85	62
indeno(1,2,3-cd) pyrene	5 to 10000	1500	1100	5 to 500	73	53
dibenz(a,h) anthracene	5 to 10000	1200	860	5 to 500	170	120
benzo(g,h,i) perylene	5 to 10000	750	540	5 to 500	170	120

(a) Concentration range, in ng/mL, includes zero

(b) Certified Reporting Limits (CRLs), in pg injected, are based on 95% confidence limits (5% positive and negative intervals).

(c) Method Detection Limits (MDLs), in pg injected, are based on a 99% confidence limit (1% positive and 0% negative intervals).

10.2 Analysis of Performance Evaluation Samples - Performance evaluation standards were obtained from Analytical Products Group, Inc. (APG). The samples were prepared according to the Proficiency Environmental Testing Program. Extracts were divided into two equal portions for analysis by the GC-MSD and GC-ITS. The extract was analyzed as obtained by the GC-MSD system. For GC-ITS analysis, the extract was diluted 1/10, 1/100, and 1/1000 to encompass and validate the working range of this system. Appropriate aliquots of internal standards were added before analysis. Results for the analysis of two sets of APG standards are shown in Tables 4a-4d. Note that two levels (high and low) were analyzed for each set. The data presented in Table 4 were analyzed using the splitless injection parameters. Results from APG using the SPI injection parameters will be added in future upgrades to this method. For each APG standard, acceptable results were obtained using the GC-ITS system for at least one of the dilutions. Acceptable results were obtained for a number of the analytes in the 1/1000 dilution. The majority of the analytes were detected within acceptable limits for the 1/100 and 1/10 dilutions. Compounds that fell outside the acceptance limits were either below their detection limit for the given dilution or had response factors that did not meet the quantitation criteria in the concentration range of the given dilution. These results, however, do demonstrate an increase in sensitivity of at least a factor of 100 for the GC-ITS system over the GC-MSD for almost all of the target analytes.

Table 4a. Method Validation Results: APG 1, Level 1

Compound Name	Reported GC-MSD	Reported GC-ITS 1/1000	1/100	Mean	Acceptance Range
bis(2-chloroethyl)ether	55.7	64.3	62.8	60.0	38.3 - 81.7
1,4-dichlorobenzene	47.0	42.7	46.7	46.3	13.0 - 79.6
hexachloroethane	14.0	13.3	14.0	15.0	4.6 - 25.4
isophorone	27.9	21.3	25.8	26.9	14.7 - 39.1
2-nitrophenol	11.9	**7.8**(a)	9.0	11.9	8.9 - 14.9
2,4-dimethylphenol	35.1	21.7	26.2	29.9	18.9 - 40.9
2,4-dichlorophenol	12.1	10.7	8.5	12.3	6.6 - 18.0
naphthalene	21.3	17.2	16.7	22.0	9.4 - 34.6
4-chloro-3-methylphenol	27.2	20.4	27.5	24.5	17.0 - 32.0
hexachlorocyclopentadiene	16.7	17.8	19.5	17.8	0.0 - 36.5
2,4,6-trichlorophenol	16.1	13.7	18.3	18.3	10.0 - 26.6
2,4-dinitrophenol(b)	43.5	29.3	34.7	37.4	9.4 - 65.4
4-nitrophenol	83.4	16.5	38.4	53.1	15.9 - 90.3
fluorene	37.3	30.5	33.6	33.3	22.9 - 43.7
hexachlorobenzene	45.1	31.8	32.8	34.6	19.6 - 49.6
anthracene	28.5	20.8	24.5	24.0	16.3 - 31.7
di-n-butyl phthalate	56.6	31.4	41.1	37.6	20.3 - 54.9
fluoranthene	35.0	29.0	32.5	28.0	19.5 - 36.5
bis(2-ethylhexyl) phthalate	72.0	**23.3**	41.4	52.9	30.4 - 75.4

Performance evaluation standards prepared according to the Proficiency Environmental Testing Program by APG

(a) Numbers listed in bold indicate concentrations outside of the acceptance range.

(b) Compounds listed in bold indicate that initial calibration requirements for these compounds were not met.

Table 4b. Method Validation Results: APG 1, Level 2

	Reported	Reported GC-ITS			
Compound Name	GC-MSD	1/1000	1/100	Mean	Acceptance Range
bis(2-chloroethyl)ether	124.2	140.1	142.5	141.9	48.8 - 235.0
1,4-dichlorobenzene	96.6	99.8	101.7	118.8	33.9 - 203.7
hexachloroethane	35.8	40.0	36.3	45.3	10.0 - 80.6
isophorone	104.2	100.6	110.5	86.1	44.6 - 127.6
2-nitrophenol	69.6	**28.7**[(a)]	43.3	71.2	42.0 - 100.4
2,4-dimethylphenol	94.8	57.8	68.5	83.0	40.2 - 125.8
2,4-dichlorophenol	56.3	**32.0**	49.2	63.8	37.2 - 90.4
naphthalene	82.6	86.5	80.4	96.6	31.3 - 161.9
4-chloro-3-methylphenol	193.9	127.4	187.2	162.5	100.6 - 224.4
hexachlorocyclopentadiene	68.9	36.7	43.2	57.3	0.0 - 120.0
2,4,6-trichlorophenol	127.5	109.5	115.1	137.2	79.2 - 195.3
2,4-dinitrophenol(b)	193.8	49.6	99.2	124.9	13.2 - 236.6
4-nitrophenol	181.1	22.4	91.0	111.6	7.6 - 215.6
fluorene	58.5	49.7	52.5	49.1	30.5 - 67.7
hexachlorobenzene	114.2	97.6	116.3	86.9	51.3 - 122.5
anthracene	95.5	92.7	96.5	81.8	64.3 - 99.3
di-n-butyl phthalate	115.0	143.6	201.1	132.0	27.8 - 236.2
fluoranthene	72.9	53.2	61.4	55.4	34.0 - 76.8
bis(2-ethylhexyl) phthalate	142.9	**33.8**	103.6	127.5	37.7 - 217.3

Performance evaluation standards prepared according to the Proficiency Environmental Testing Program by APG

(a) Numbers listed in bold indicate concentrations outside of the acceptance range.

(b) Compounds listed in bold indicate that initial calibration requirements for these compounds were not met.

Table 4c. Method Validation Results: APG 2, Level 1

	Reported	Reported GC-ITS				
Compound Name	GC-MSD	1/1000	1/100	1/10	Mean	Acceptance Range
phenol	10.5	**29.5**(a)	13.3	9.8	11.7	0.0 - 24.1
1,4-dichlorobenzene	38.3	38.2	50.7	43.4	51.0	18.8 - 83.3
hexachloroethane	13.9	12.6	15.6	13.6	18.1	3.0 - 33.2
nitrobenzene	20.3	20.1	29.4	35.3	27.1	15.9 - 38.4
2,4-dimethylphenol	21.0	17.1	21.7	27.1	24.2	5.7 - 42.7
1,2,4-trichlorobenzene	22.7	25.2	27.1	28.7	30.5	14.8 - 46.3
2,4,6-trichlorophenol	20.7	19.2	26.3	**37.2**	26.8	16.6 - 36.9
acenaphthene	24.1	28.5	27.7	30.1	31.8	16.8 - 46.9
4-nitrophenol	45.9	0.0	14.3	51.3	35.8	0.0 - 82.3
2,6-dinitrotoluene	13.3	7.9	10.2	26.9	17.8	6.5 - 29.1
diethylphthalate	28.3	18.0	31.6	39.2	26.1	0.0 - 56.8
4,6-dinitro-2-methylphenol(b)	49.4	**6.6**	**9.0**	55.4	58.3	12.1 - 104.5
pentachlorophenol	43.2	11.5	19.1	56.2	44.0	5.1 - 82.8
anthracene	29.1	32.4	40.9	50.4	38.4	22.6 - 54.4
butyl benzyl phthalate	55.4	19.3	36.9	**183.7**	43.3	0.0 - 94.4
chrysene	19.4	20.0	21.1	25.4	23.3	12.4 - 34.2

Performance evaluation standards prepared according to the Proficiency Environmental Testing Program by APG

(a) Numbers listed in bold indicate concentrations outside of the acceptance range.

(b) Compounds listed in bold indicate that initial calibration requirements for these compounds were not met.

Table 4d. Method Validation Results: APG 2, Level 2

Compound Name	Reported GC-MSD	Reported GC-ITS 1/1000	Reported GC-ITS 1/100	Reported GC-ITS 1/10	Mean	Acceptance Range
phenol	45.5	**181.9**(b)	42.3	53.2	46.8	0.0 - 98.1
1,4-dichlorobenzene	122.8	135.5	135.5	164.9	146.8	54.1 - 239.5
hexachloroethane	44.1	40.3	40.2	48.7	52.4	11.0 - 93.9
nitrobenzene	40.4	39.2	43.7	56.0	48.1	22.6 - 73.6
2,4-dimethylphenol	127.4	91.9	101.3	129.9	125.4	40.6 - 210.1
1,2,4-trichlorobenzene	47.1	47.5	44.8	55.8	68.7	6.4 - 131.0
2,4,6-trichlorophenol	63.7	64.7	72.7	105.2	78.7	29.2 - 128.1
acenaphthene	58.2	63.2	53.6	64.1	77.8	45.3 - 91.5
4-nitrophenol	169.4	0.0	47.2	212.9	121.5	0.0 - 271.4
2,6-dinitrotoluene	58.4	**23.8**	**32.0**	81.4	59.6	35.8 - 83.5
diethylphthalate	106.3	113.1	120.7	118.2	93.9	0.0 - 220.6
4,6-dinitro-2-methylphenol(a)	114.3	**12.5**	**19.7**	149.8	130.1	36.8 - 223.5
pentachlorophenol	96.3	**29.0**	38.2	124.3	95.2	32.0 - 158.3
anthracene	93.3	108.3	121.6	164.0	115.1	52.0 - 178.1
butyl benzyl phthalate	143.8	51.2	189.9	**534.5**	125.8	0.0 - 276.1
chrysene	61.3	55.9	60.9	78.4	69.7	40.5 - 98.8

Performance evaluation standards prepared according to the Proficiency Environmental Testing Program by APG

(a) Numbers listed in bold indicate concentrations outside of the acceptance range.

(b) Compounds listed in bold indicate that initial calibration requirements for these compounds were not met.

10.3 Analysis of Solid Waste Extracts - The ability to detect analytes in solid matrices also was tested. Sand was used as a surrogate solid matrix for this study. A 10-g aliquot of sand was spiked with all analytes and surrogates at a level of 10 µg/g and extracted according to appropriate procedures. The resulting extract was divided for analysis by the GC-MSD and GC-ITS systems. Appropriate levels of internal standards were added before analysis. The extract was diluted 1/100 and 1/1000 for analysis by GC-ITS and analyzed as obtained by GC-MSD. The results for the analysis of two separate samples are given in Tables 5 and 6. The majority of analytes can be detected with relative percent differences ≤ 30% for the 1/100 dilution.

Tables 7 and 8 compare the performance of the GC-ITS with that of the GC-MSD on extracts of transuranic waste sludges and on PAH-containing wastes. The transuranic waste sludges contained percent concentrations of hydrocarbon oils, and their extracts had to be diluted 60-fold for the GC-MSD analysis to prevent overloading and chemically contaminating the system. Three 100-fold dilutions of the extracts were run on the GC-ITS. In general, the results of the GC-ITS analysis agreed well with those of the GC-MSD (Table 7). The GC-ITS also determined more sample analytes and matrix-spike compounds than did the GC-MSD. However, the results for the GC-ITS measurement of phenol appeared to have a high bias relative to those for the GC-MSD. The reason for this bias is not clear. A high bias for substituted phenols was not observed in the comparison of results for method performance sample extracts (Table 4). The GC-ITS and GC-MSD also agreed reasonably well in their results for PAH wastes (Table 8), where the sample for the GC-ITS was diluted 1:100 before analysis. The GC-ITS detected three more compounds than did the GC-MSD, but was low relative to the GC-MSD for benzo(k)fluoranthene. Overall, the results for the GC-ITS appear at least equivalent to, but more sensitive than, those of the GC-MSD.

11.0 Reference

U.S. Environmental Protection Agency (EPA). 1994. *Test Methods for Evaluating Solid Waste.* “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique.” EPA/SW-846, Method 8270B. Revision 1, Office of Solid Waste and Emergency Response, Washington, DC.

12.0 Further Reading

136 CFR 537. 1990. “Definition and Procedure for the Determination of the Method Detection Limit.” Appendix B. U. S. Code of Federal Regulations.

Finnigan MAT. 1994. “Magnum GC/MS Systems Software Reference Manuals I & II.” Instrument Control & Post Acquisition Applications, Revision B.

Lee, M. L., F. J. Yang, and K. D. Bartle. 1984. *Open Tubular Column Gas Chromatography: Theory and Practice*. Wiley-Intercession Publication.

U.S. Army Toxic and Hazardous Materials Agency (USATHAMA). 1990. “USATHAMA: Quality Assurance Program.” Aberdeen Proving Ground, Maryland.

Table 5. Analysis of Solid Waste Extracts

Compound Name	Reported GC-MSD(a)	GC-ITS:(b) 1/1000	relative % diff.	1/100	relative % diff.
2-fluorophenol	4.8	1.9	**86**(c)	5.0	4
d_6-phenol	5.3	4.4	18	6.4	19
phenol	5.3	12.0	**77**	5.6	5
2-chlorophenol	5.5	5.3	4	5.6	2
bis(2-chloroethyl) ether	5.4	3.2	**51**	5.2	4
1,3-dichlorobenzene	5.6	5.4	4	5.3	6
1,4-dichlorobenzene	5.2	5.3	2	5.3	2
1,2-dichlorobenzene	5.5	5.4	2	4.9	12
benzyl alcohol	5.9	ND	-	5.9	0
2-methylphenol	5.5	4.4	22	5.9	7
bis(2-chloroisopropyl) ether	5.6	4.5	22	5.8	4
N-nitrosodi-n-propylamine	6.0	4.2	**35**	6.6	10
4-methylphenol	6.0	2.5	**82**	5.4	10
hexachloroethane	4.8	5.7	17	5.1	6
d_5-nitrobenzene	5.6	3.7	**41**	6.5	15
nitrobenzene	5.4	4.2	25	5.1	6
isophorone	6.2	5.4	14	6.4	3
2-nitrophenol	6.2	2.8	**76**	4.7	28
2,4-dimethylphenol	6.2	3.4	**58**	3.5	**56**
benzoic acid	NR	ND	-	2.4	-
bis(2-chloroethoxy) methane	5.8	2.6	**76**	5.8	0
2,4-dichlorophenol	6.2	2.7	**79**	5.7	8
1,2,4-trichlorobenzene	5.8	5.6	4	5.6	4
naphthalene	5.7	6.0	5	6.1	7
4-chloroaniline	1.8	ND	-	0.5	**113**
hexachlorobutadiene	5.9	5.7	3	5.8	2
4-chloro-3-methylphenol	7.7	4.0	**63**	7.2	7
2-methylnaphthalene	6.2	6.0	3	6.4	3
hexachlorocyclopentadiene	6.0	5.3	12	4.9	20
2,4,6-trichlorophenol	7.7	4.7	**48**	6.3	20
2,4,5-trichlorophenol	8.2	9.6	16	7.6	8
2-fluorobiphenyl	6.9	7.2	4	7.3	6
2-chloronaphthalene	6.8	7.7	12	6.4	6
2-nitroaniline	7.5	2.4	**103**	6.3	17
dimethylphthalate	8.3	8.6	4	8.2	1
2,6-dinitrotoluene	8.9	5.2	**52**	6.9	25

(a) Reported concentration, in µg/g, for the extraction of a 10-g sample spiked to a level of 10 µg/g

(b) Reported concentration, in µg/g, for 1/1000 and 1/100 dilutions of the extract resulting from the extract of the original 10-g sample

(c) ND = not detected, NR = not reported, bold = relative percent differences ≥ 30

Table 5. Analysis of Solid Waste Extracts (cont.)

Compound Name	Reported GC-MSD(a)	GC-ITS:(b) 1/1000	Relative% diff.	1/100	Relative % diff.
acenaphthylene	7.0	7.0	0	6.7	4
3-nitroaniline	7.8	1.5	**135**	5.9	28
acenaphthene	7.3	8.3	13	7.2	1
2,4-dinitrophenol	9.6	ND	-	3.7	**89**
dibenzofuran	7.6	8.4	10	7.2	5
4-nitrophenol	7.7	ND	-	4.9	**44**
2,4-dinitrotoluene	9.2	2.4	**117**	6.5	**34**
diethylphthalate	8.1	8.0	1	8.2	1
fluorene	7.4	8.3	11	7.3	1
4-chlorophenyl phenyl ether	8.3	8.6	4	7.8	6
4-nitroaniline	9.0	ND	-	5.9	**42**
4,6-dinitro-2-methylphenol	8.4	1.2	**150**	4.8	**54**
N-nitrosodiphenylamine	4.8	3.5	**31**	4.7	2
2,4,6-tribromophenol	7.7	4.9	**44**	8.4	9
4-bromophenyl phenyl ether	8.0	7.5	6	8.6	7
hexachlorobenzene	7.8	8.4	7	8.1	4
pentachlorophenol	9.0	2.2	**121**	7.4	20
phenanthrene	7.7	8.5	10	8.6	11
anthracene	7.6	7.4	3	7.5	1
carbazole	NR	6.8	-	7.7	-
di-n-butyl phthalate	11.1	7.7	**36**	13.5	20
fluoranthene	7.6	7.5	1	8.8	15
pyrene	10.1	7.4	**31**	8.1	22
d_{14}-terphenyl	10.2	8.5	18	9.4	8
butyl benzyl phthalate	8.9	1.9	**130**	8.2	8
benz(a) anthracene	9.1	4.5	**68**	8.1	12
3,3'-dichlorobenzidine	6.0	ND	-	1.3	**129**
chrysene	9.1	7.1	25	8.1	12
bis(ethylhexyl) phthalate	8.9	2.1	**124**	6.9	25
di-n-octyl phthalate	8.1	1.6	**134**	6.4	23
benzo(b) fluoranthene	6.6	3.2	**69**	6.3	5
benzo(k) fluoranthene	8.4	6.8	21	8.5	1
benzo(a) pyrene	8.4	2.6	**105**	8.2	2
indeno(1,2,3-cd) pyrene	9.6	1.6	**143**	6.4	**40**
dibenz(a,h) anthracene	11.6	2.4	**131**	6.4	**58**
benzo(g,h,i) perylene	11.9	3.1	**117**	8.1	**38**

(a) Reported concentration, in µg/g, for the extraction of a 10-g sample spiked to a level of 10 µg/g

(b) Reported concentration, in µg/g, for 1/1000 and 1/100 dilutions of the extract resulting from the extract of the original 10-g sample

(c) ND = not detected, NR = not reported, bold = relative percent differences ≥ 30

Table 6. Analysis of Solid Waste Extracts

Compound Name	Reported GC-MSD(a)	GC-ITS:(b) 1/1000	Relative % diff.	1/100	Relative % diff.
2-fluorophenol	5.7	3.6	**45**(c)	6.9	19
d_6-phenol	6.4	5.8	10	8.9	**33**
phenol	6.4	6.6	3	8.3	26
2-chlorophenol	6.6	6.6	0	7.1	7
bis(2-chloroethyl) ether	6.5	4.3	**41**	6.1	6
1,3-dichlorobenzene	6.6	7.3	10	7.0	6
1,4-dichlorobenzene	6.3	7.2	13	7.0	10
1,2-dichlorobenzene	6.5	7.1	9	6.9	6
benzyl alcohol	7.2	ND	-	7.5	4
2-methylphenol	7.1	5.7	22	7.1	0
bis(2-chloroisopropyl) ether	6.6	6.2	6	8.4	24
N-nitrosodi-n-propylamine	7.1	5.6	24	8.0	12
4-methylphenol	7.3	4.0	**58**	7.7	5
hexachloroethane	5.7	7.6	28	7.2	23
d_5-nitrobenzene	6.7	5.6	18	9.0	29
nitrobenzene	6.5	4.7	**32**	7.3	12
isophorone	7.5	6.2	19	8.7	15
2-nitrophenol	7.6	3.0	**87**	7.2	5
2,4-dimethylphenol	7.7	3.6	**72**	6.1	23
benzoic acid	NR	ND	-	2.6	-
bis(2-chloroethoxy) methane	7.1	4.2	**51**	8.2	14
2,4-dichlorophenol	7.8	3.7	**71**	7.7	1
1,2,4-trichlorobenzene	7.2	6.8	6	7.5	4
naphthalene	6.8	7.3	7	7.6	11
4-chloroaniline	2.5	ND	-	1.0	**86**
hexachlorobutadiene	7.0	7.1	1	7.6	8
4-chloro-3-methylphenol	8.6	5.4	**46**	9.1	6
2-methylnaphthalene	7.4	7.2	3	8.8	17
hexachlorocyclopentadiene	7.2	5.9	20	7.0	3
2,4,6-trichlorophenol	8.7	5.3	**48**	8.7	0
2,4,5-trichlorophenol	9.2	11.9	26	8.4	9
2-fluorobiphenyl	8.0	8.3	4	9.5	17
2-chloronaphthalene	7.9	9.2	15	9.6	20
2-nitroaniline	8.1	3.1	**89**	9.2	13
dimethylphthalate	9.2	9.2	0	9.4	2
2,6-dinitrotoluene	9.6	4.5	**72**	9.4	2

(a) Reported concentration, in µg/g, for the extraction of a 10-g sample spiked to a level of 10 µg/g

(b) Reported concentration, in µg/g, for 1/1000 and 1/100 dilutions of the extract resulting from the extract of the original 10-g sample

(c) ND = not detected, NR = not reported, bold = relative percent differences ≥ 30

Table 6. (contd.)

Compound Name	Reported GC-MSD(a)	GC-ITS:(b) 1/1000	Relative % diff.	1/100	Relative % diff.
acenaphthylene	7.9	7.7	2	9.1	14
3-nitroaniline	9.0	0.9	**164**	9.2	2
acenaphthene	8.0	8.8	10	9.0	12
2,4-dinitrophenol	10.2	ND	-	4.6	**76**
dibenzofuran	8.3	8.7	5	9.2	10
4-nitrophenol	8.3	ND	-	7.7	8
2,4-dinitrotoluene	10.4	2.5	**122**	8.7	18
diethylphthalate	8.2	7.9	4	10.3	23
fluorene	8.1	8.4	4	9.4	15
4-chlorophenyl phenyl ether	9.4	9.3	1	10.1	7
4-nitroaniline	10.8	ND	-	8.0	**30**
4,6-dinitro-2-methylphenol	9.1	1.2	**153**	6.9	28
N-nitrosodiphenylamine	4.6	4.2	9	6.5	**34**
2,4,6-tribromophenol	8.3	5.4	**42**	11.4	**31**
4-bromophenyl phenyl ether	8.8	8.7	1	10.8	20
hexachlorobenzene	8.3	9.2	10	9.5	13
pentachlorophenol	9.6	3.0	**105**	10.1	5
phenanthrene	7.9	9.9	22	10.3	26
anthracene	8.0	8.6	7	9.7	19
carbazole	--	7.7	-	11.4	-
di-n-butyl phthalate	8.9	6.0	**39**	14.3	**46**
fluoranthene	8.6	8.5	1	10.7	22
pyrene	10.0	8.0	22	10.5	5
d_{14}-terphenyl	10.3	9.3	10	12.0	15
butyl benzyl phthalate	9.6	2.0	**131**	8.3	14
benz(a) anthracene	10.0	4.9	**68**	9.7	3
3,3'-dichlorobenzidine	7.6	0.3	**185**	4.3	**55**
chrysene	9.3	9.7	4	10.3	10
bis(ethylhexyl) phthalate	9.5	1.8	**136**	10.8	13
di-n-octyl phthalate	8.2	1.8	**128**	9.8	18
benzo(b) fluoranthene	9.0	3.0	**100**	9.0	0
benzo(k) fluoranthene	9.1	6.9	28	10.4	13
benzo(a) pyrene	9.1	3.5	**89**	10.8	17
indeno(1,2,3-cd) pyrene	10.4	2.2	**130**	9.3	11
dibenz(a,h) anthracene	12.4	2.9	**124**	10.4	18
benzo(g,h,i) perylene	12.3	4.0	**102**	10.6	15

(a) Reported concentration, in μg/g, for the extraction of a 10-g sample spiked to a level of 10 μg/g

(b) Reported concentration, in μg/g, for 1/1000 and 1/100 dilutions of the extract resulting from the extract of the original 10-g sample

(c) ND = not detected, NR = not reported, bold = relative percent differences ≥ 30

Table 7. Comparison of Results (µg/g) for the Analysis of Transuranic Waste Sludge Extracts (Sample extract diluted 1:300 for GC-ITS and 1:60 for GC-MSD)

Compound	Type	Spike Conc (µg/g)	Sample 102S2 GC-ITS	Sample 102S2 GC-MSD	Sample 10CM6 GC-ITS	Sample 10CM6 GC-MSD	Sample 10CM6-MS(a) GC-ITS	Sample 10CM6-MS(a) GC-MSD
2-fluorophenol	acid surr.	10	4.9	6.0	4.5	6.4	2.7	5.0
d_5-phenol	acid surr.	10	11.4	10.9	10.6	11.1	8.6	10.3
phenol	acid matrix	10(a)	46.9	11.9	35.6	12.7	39.5	21.1
2-chlorophenol	acid matrix	10(a)	—	—	—	—	6.5	7.7
1,4-dichlorobenzene	B/N matrix	5(a)	—	—	—	—	2.8	—
N-nitrosodi-n-propylamine	B/N matrix	5(a)	—	—	—	—	5.0	6.2
d_5-nitrobenzene	B/N surr.	5	3.9	4.4	3.4	4.3	3.2	3.8
isophorone	analyte	—	—	—	—	—	—	1.9
1,2,4-trichlorobenzene	B/N matrix	5(a)	—	—	—	—	4.6	—
naphthalene	analyte	—	0.6	—	0.6	—	0.5	—
4-chloro-3-methylphenol	acid matrix	10(a)	—	—	—	—	9.1	12.7
2-methylnaphthalene	analyte	—	2.8	—	2.3	—	2.1	—
2-fluorobiphenyl	B/N surr.	5	6.8	6.3	5.0	6.0	5.5	5.8
dimethylphthalate	analyte	—	—	—	—	—	—	26.7
acenaphthene	B/N matrix	5(a)	—	—	—	—	4.8	7.1
diethylphthalate	analyte	—	0.7	—	0.5	—	—	—
N-nitrosodi-n-phenylamine	analyte	—	—	—	2.0	—	1.6	—
2,4,6-tribromophenol	acid surr.	10	2.8	2.7	3.6	2.4	2.4	1.6
phenanthrene	analyte	—	2.0	—	2.1	—	3.7	—
di-n-butylphthalate	analyte	—	1.9	—	2.0	—	2.4	—
fluoranthene	analyte	—	29.5	31.4	23.9	29.2	27.0	30.2
pyrene	B/N matrix	5(a)	—		—	—	5.7	—
d_{14}-terphenyl	B/N surr.	5	6.2	7.1	6.3	7.4	4.7	7.3
bis(2-ethylhexyl)phthalate	analyte	—	129.8	100.6	130.4	98.4	105.0	90.1

(a) Spiked only into matrix spike (MS) samples

Table 8. Comparison of Results (µg/mL) for Analysis of Polycyclic Aromatic Hydrocarbon Wastes (Sample for GC-ITS diluted 1:100)

	Sample 025		Sample 029	
Compound	GC-ITS	GC-MSD	GC-ITS	GC-MSD
phenol	73.8	—	189	—
naphthalene	122	123	2.3	—
2-methylnaphthalene	534	545	7.3	—
acenaphthene	38.8	—	166	203
acenaphthylene	—	—	3.5	21.2
dibenzofuran	16.5	—	65.3	80.6
fluorene	43.0	—	182	278
phenanthrene	101	160	1260	1600
anthracene	—	—	1150	1220
di-n-butylphthalate	41.7	44.7	—	—
carbazole	—	—	107	128
fluoranthene	—	—	3980	3490
pyrene	17.1	26.6	3350	2800
benzo(a)anthracene	—	—	1170	1240
chrysene	—	—	978	1280
bis(2-ethylhexyl)phthalate	69.3	73.5	—	—
benzo(b)fluoranthene	—	—	1430	1150
benzo(k)fluoranthene	—	—	307	1380
benzo(a)pyrene	—	—	713	964
indeno(123-cd)pyrene	—	—	520	360
dibenz(a,h)anthracene	—	—	81.7	153
benzo(ghi)perylene	—	—	342	415