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Method 1625C Semivolatile Organic Compounds by Isotope Dilution GCMS

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June 1989

U.S. Environmental Protection Agency
Office of Science and Technology
Engineering and Analysis Division
401 M Street S.W.
Washington, D.C. 20460

Method 1625C
Semivolatile Organic Compounds
by Isotope Dilution GCMS

1.0 Scope and Application

1.1 This method is designed to meet the survey requirements of the USEPA ITD. The method is used to determine the semivolatile toxic organic pollutants associated with the Clean Water Act (as amended 1987); the Resource Conservation and Recovery Act (as amended 1986); the Comprehensive Environmental Response, Compensation and Liability Act (as amended 1986); and other compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GCMS).

1.2 The chemical compounds listed in Tables 1-4 may be determined in waters, soils, and municipal sludges by the method.

1.3 The detection limits of the method are usually dependent on the level of interferences rather than instrumental limitations. The limits in Tables 5 and 6 typify the minimum quantities that can be detected with no interferences present.

1.4 The GCMS portions of the method are for use only by analysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analysis of environmental samples by GCMS should run the performance tests in Reference 1 before beginning.

TABLE 1. BASE/NEUTRAL EXTRACTABLE COMPOUNDS DETERMINED BY GCMS USING ISOTOPE DILUTION AND INTERNAL STANDARD TECHNIQUES

	Pollutant				Labeled Compound		
Compound	Storet	CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
acenaphthene	34205	83-32-9	001 B	001 B	d_{10}	15067-20-2	201 B
acenaphthylene	34200	208-96-8	077 B	002 B	d_{8}	93951-97-4	277 B
anthracene	34220	120-12-7	078 B	003 B	d_{10}	1719-06-8	278 B
benzidine	39120	92-87-5	005 B	004 B	d_{8}	92890-63-6	205 B
benzo(a)anthracene	34526	56-55-3	072 B	005 B	d_{12}	1718-53-2	272 B
benzo(b)fluoranthene	34230	205-99-2	074 B	007 B	d_{12}	93951-98-5	274 B
benzo(k)fluoranthene	34242	207-08-9	075 B	009 B	d_{12}	93952-01-3	275 B
benzo(a)pyrene	34247	50-32-8	073 B	006 B	d_{12}	63466-71-7	273 B
benzo(ghi)perylene	34521	191-24-2	079 B	008 B	d_{12}	93951-66-7	279 B
biphenyl (Appendix C)	81513	92-52-4	512 B		d_{10}	1486-01-7	612 B
bis(2-chloroethyl) ether	34273	111-44-4	018 B	011 B	d_{8}	93952-02-4	218 B
bis(2-chloroethoxy) methane	34278	111-91-1	043 B	010 B	d_{8}	93966-78-0	243 B
bis(2-chloroisopropyl) ether	34283	108-60-1	042 B	012 B	d_{12}	93951-67-8	242 B
bis(2-ethylhexyl) phthalate	39100	117-81-7	066 B	013 B	d_{4}	93951-87-2	266 B

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	Pollutant				Labeled Compound		
Compound	Storet	CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
4-bromophenyl phenyl ether	34636	101-55-3	041 B	014 B	d_5	93951-83-8	241 B
butyl benzyl phthalate	34292	85-68-7	067 B	015 B	d_4	93951-88-3	267 B
n-C10 (Appendix C)	77427	124-18-5	517 B		d_{22}	16416-29-8	617 B
n-C12 (Appendix C)	77588	112-40-3	506 B		d_{26}	16416-30-1	606 B
n-C14 (Appendix C)	77691	629-59-4	518 B	618 B			
n-C16 (Appendix C)	77757	544-76-3	519 B		d_{34}	15716-08-2	619 B
n-C18 (Appendix C)	77804	593-45-3	520 B	620 B			
n-C20 (Appendix C)	77830	112-95-8	521 B		d_{42}	62369-67-9	621 B
n-C22 (Appendix C)	77859	629-97-0	522 B	622 B			
n-C24 (Appendix C)	77886	646-31-1	523 B		d_{50}	16416-32-3	623 B
n-C26 (Appendix C)	77901	630-01-3	524 B	624 B			
n-C28 (Appendix C)	78116	630-02-4	525 B	625 B			
n-C30 (Appendix C)	78117	638-68-6	526 B		d_{62}	93952-07-9	626 B
carbazole (4c)	77571	86-74-8	528 B		d_8	38537-24-5	628 B
2-chloronaphthalene	34581	91-58-7	020 B	016 B	d_7	93951-84-9	220 B
4-chlorophenyl phenyl ether	34641	7005-72-3	040 B	017 B	d_5	93951-85-0	240 B
chrysene	34320	218-01-9	076 B	018 B	d_{12}	1719-03-5	276 B
p-cymene (Appendix C)	77356	99-87-6	513 B		d_{14}	93952-03-5	613 B
dibenzo(a,h)anthracene	34556	53-70-3	082 B	019 B	d_{14}	13250-98-1	282 B
dibenzofuran (Appendix C & 4c)	81302	132-64-9	505 B		d_8	93952-04-6	605 B
dibenzothiophene (Synfuel)	77639	132-65-0	504 B		d_8	33262-29-2	604 B
di-n-butyl phthalate	39110	84-74-2	068 B	026 B	d_4	93952-11-5	268 B
1,2-dichlorobenzene	34536	95-50-1	025 B	020 B	d_4	2199-69-1	225 B
1,3-dichlorobenzene	34566	541-73-1	026 B	021 B	d_4	2199-70-4	226 B
1,4-dichlorobenzene	34571	106-46-7	027 B	022 B	d_4	3855-82-1	227 B
3,3'-dichlorobenzidine	34631	91-94-1	028 B	023 B	d_6	93951-91-8	228 B
diethyl phthalate	34336	84-66-2	070 B	024 B	d_4	93952-12-6	270 B
2,4-dimethylphenol	34606	105-67-9	034 A	003 A	d_3	93951-75-8	234 A
dimethyl phthalate	34341	131-11-3	071 B	025 B	d_4	93951-89-4	271 B
2,4-dinitrotoluene	34611	121-14-2	035 B	027 B	d_3	93951-68-9	235 B
2,6-dinitrotoluene	34626	606-20-2	036 B	028 B	d_3	93951-90-7	236 B
di-n-octyl phthalate	34596	117-84-0	069 B	029 B	d_4	93952-13-7	269 B
diphenylamine (Appendix C)	77579	122-39-4	507 B		d_{10}	37055-51-9	607 B
diphenyl ether (Appendix C)	77587	101-84-8	508 B		d_{10}	93952-05-7	608 B
1,2-diphenylhydrazine	34346	122-66-7	037 B	030 B	d_{10}	93951-92-9	237 B
fluoranthene	34376	206-44-0	039 B	031 B	d_{10}	93951-69-0	231 B
fluorene	34381	86-73-7	080 B	032 B	d_{10}	81103-79-9	280 B
hexachlorobenzene	39700	118-74-1	009 B	033 B	$^{13}C_6$	93952-14-8	209 B
hexachlorobutadiene	34391	87-68-3	052 B	034 B	$^{13}C_4$	93951-70-3	252 B
hexachloroethane	34396	67-72-1	012 B	036 B	^{13}C	93952-15-9	212 B
hexachlorocyclopentadiene	34386	77-47-4	053 B	035 B	$^{13}C_4$	93951-71-4	253 B

TABLE 1. BASE/NEUTRAL EXTRACTABLE COMPOUNDS DETERMINED BY GCMS USING ISOTOPE DILUTION AND INTERNAL STANDARD TECHNIQUES

	Pollutant				Labeled Compound		
Compound	Storet	CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
indeno(1,2,3-cd)pyrene	34403	193-39-5	083 B	037 B			
isophorone	34408	78-59-1	054 B	038 B	d_8	93952-16-0	254 B
naphthalene	34696	91-20-3	055 B	039 B	d_8	1146-65-2	255 B
beta-naphthylamine (Appendix C)	82553	91-59-8	502 B		d_7	93951-94-1	602 B
nitrobenzene	34447	98-95-3	056 B	040 B	d_5	4165-60-0	256 B
N-nitrosodimethylamine	34438	62-75-9	061 B	041 B	d_6	17829-05-9	261 B
N-nitrosodi-n-proplyamine	34428	621-64-7	063 B	042 B	d_{14}	93951-96-3	263 B
N-nitrosodiphenylamine	34433	86-30-6	062 B	043 B	d_6	93951-95-2	262 B
phenanthrene	34461	85-01-8	081 B	044 B	d_{10}	1517-22-2	281 B
phenol	34694	108-95-2	065 A	010 A	d_5	4165-62-2	265 A
alpha-picoline (Synfuel)	77088	109-06-8	503 B		d_7	93951-93-0	603 B
pyrene	34469	129-00-0	084 B	045 B	d_{10}	1718-52-1	284 B
styrene (Appendix C)	77128	100-42-5	510 B		d_5	5161-29-5	610 B
alpha-terpineol (Appendix C)	77493	98-55-5	509 B		d_3	93952-06-8	609 B
1,2,3-trichlorobenzene (4c)	77613	87-61-6	529 B		d_3	3907-98-0	629 B
1,2,4-trichlorobenzene	34551	120-82-1	008 B	046 B	d_3	2199-72-6	208 B

TABLE 2. ACID EXTRACTABLE COMPOUNDS DETERMINED BY GCMS USING ISOTOPE DILUTION AND INTERNAL STANDARD TECHNIQUES

	Pollutant				Labeled Compound		
Compound	Storet	CAS Registry	EPA-EGD	NPDES	Analog	CAS Registry	EPA-EGD
4-chloro-3-methylphenol	34452	59-50-7	022 A	008 A	d_2	93951-72-5	222 A
2-chlorophenol	34586	95-57-8	024 A	001 A	d_4	93951-73-6	224 A
2,4-dichlorophenol	34601	120-83-2	031 A	002 A	d_3	93951-74-7	231 A
2,4-dinitrophenol	34616	51-28-5	059 A	005 A	d_3	93951-77-0	259 A
2-methyl-4,6-dinitrophenol	34657	534-52-1	060 A	004 A	d_2	93951-76-9	260 A
2-nitrophenol	34591	88-75-5	057 A	006 A	d_4	93951-75-1	257 A
4-nitrophenol	34646	100-02-7	058 A	007 A	d_4	93951-79-2	258 A
pentachlorophenol	39032	87-86-5	064 A	009 A	$^{13}C_6$	85380-74-1	264 A
2,3,6-trichlorophenol (4c)	77688	933-75-5	530 A		d_2	93951-81-6	630 A
2,4,5-trichlorophenol (4c)		95-95-4	531 A		d_2	93951-82-7	631 A
2,4,6-trichlorophenol	34621	88-06-2	021 A	011 A	d_2	93951-80-5	221 A

TABLE 3. BASE/NEUTRAL EXTRACTABLE COMPOUNDS TO BE DETERMINED BY REVERSE SEARCH AND QUANTITATION USING KNOWN RETENTION TIMES, RESPONSE FACTORS, REFERENCE COMPOUND, AND MASS SPECTRA

EGD No.	Compound	CAS Registry
555	acetophenone	98-86-2
556	4-aminobiphenyl	92-67-1
557	aniline	62-53-3
558	o-anisidine	90-04-0
559	aramite	140-57-8
560	benzanthrone	82-05-3
561	1,3-benzenediol(resorcinol)	108-46-3
562	benzenethiol	108-98-5
563	2,3-benzofluorene	243-17-4
564	benzyl alcohol	100-51-6
565	2-bromochlorobenzene	694-80-4
566	3-bromochlorobenzene	108-37-2
567	4-chloro-2-nitroaniline	89-63-4
568	5-chloro-o-toluidine	95-79-4
569	4-chloroaniline	106-47-8
570	3-chloronitrobenzene	121-73-3
571	o-cresol	95-48-7
572	crotoxyphos	7700-17-6
573	2,6-di-tert-butyl-p-benzoquinone	719-22-2
574	2,4-diaminotoluene	95-80-7
575	1,2-dibromo-3-chloropropane	96-12-8
576	2,6-dichloro-4-nitroaniline	99-30-9
577	1,3-dichloro-2-propanol	96-23-1
578	2,3-dichloroaniline	608-27-5
579	2,3-dichloronitro-benzene	3209-22-1
580	1,2:3,4-diepoxybutane	1464-53-5
581	3,3'-dimethoxybenzidine	119-90-4
582	dimethyl sulfone	67-71-0
583	p-dimethylamino-azobenzene	60-11-7
584	7,12-dimethylbenz-(a)anthracene	57-97-6
585	N,N-dimethylformamide	68-12-2
586	3,6-dimethylphenanthrene	1576-67-6
587	1,4-dinitrobenzene	100-25-4
588	diphenyldisulfide	882-33-7
589	ethyl methanesulfonate	62-50-0
590	ethylenethiourea	96-45-7
591	ethynylestradiol3-methyl ether	72-33-3
592	hexachloropropene	1888-71-7
593	2-isopropylnaphthalene	2027-17-0
594	isosafrole	120-58-1
595	longifolene	475-20-7
596	malachite green	569-64-2
597	methapyrilene	91-80-5
598	methyl methanesulfonate	66-27-3
599	2-methylbenzothioazole	120-75-2
900	3-methylcholanthrene	56-49-5

TABLE 3. BASE/NEUTRAL EXTRACTABLE COMPOUNDS TO BE DETERMINED BY REVERSE SEARCH AND QUANTITATION USING KNOWN RETENTION TIMES, RESPONSE FACTORS, REFERENCE COMPOUND, AND MASS SPECTRA

EGD No.	Compound	CAS Registry
901	4,4'-methylene-bis(2-chloroaniline)	101-14-4
902	4,5-methylene-phenanthrene	203-64-5
903	1-methylfluorene	1730-37-6
904	2-methylnaphthalene	91-57-6
905	1-methylphenanthrene	832-69-9
906	2-(methylthio)-benzothiazole	615-22-5
907	1,5-naphthalenediamine	2243-62-1
908	1,4-naphthoquinone	130-15-4
909	alpha-naphthylamine	134-32-7
910	5-nitro-o-toluidine	99-55-8
911	2-nitroaniline	88-74-4
912	3-nitroaniline	99-09-2
913	4-nitroaniline	100-01-6
914	4-nitrobiphenyl	92-93-3
915	N-nitrosodi-n-butylamine	924-16-3
916	N-nitrosodiethylamine	55-18-5
917	N-nitrosomethyl-ethylamine	10595-95-6
918	N-nitrosomethyl-phenylamine	614-00-6
919	N-nitrosomorpholine	59-89-2
920	N-nitrosopiperidine	100-75-4
921	pentachlorobenzene	608-93-5
922	pentachloroethane	76-01-7
923	pentamethylbenzene	700-12-9
924	perylene	198-55-0
925	phenacetin	62-44-2
926	phenothiazine	92-84-2
927	1-phenylnaphthalene	605-02-7
928	2-phenylnaphthalene	612-94-2
929	pronamide	23950-58-5
930	pyridine	110-86-1
931	safrole	94-59-7
932	squalene	7683-64-9
933	1,2,4,5-tetra-chlorobenzene	95-94-3
934	thianaphthene(2,3-benzothiophene)	95-15-8
935	thioacetamide	62-55-5
936	thioxanthone	492-22-8
937	o-toluidine	95-53-4
938	1,2,3-trimethoxybenzene	634-36-6
939	2,4,5-trimethylaniline	137-17-7
940	triphenylene	217-59-4
941	tripropyleneglycolmethyl ether	20324-33-8
942	1,3,5-trithiane	291-21-4

2.0 Summary of Method

2.1 The percent solids content of a sample is determined. Stable isotopically labeled analogs of the compounds of interest are added to the sample. If the solids content is less than 1%, a 1 L sample is extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. If the solids content is 30% or less, the sample is diluted to 1% solids with reagent water, homogenized ultrasonically, and extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques. If the solids content is greater than 30%, the sample is extracted using ultrasonic techniques. Each extract is dried over sodium sulfate, concentrated to a volume of 5 mL, cleaned up using gel permeation chromatography (GPC), if necessary, and concentrated. Extracts are concentrated to 1 mL if GPC is not performed, and to 0.5 mL if GPC is performed. An internal standard is added to the extract, and a 1 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

TABLE 4. ACID EXTRACTABLE COMPOUNDS TO BE DETERMINED BY REVERSE SEARCH AND QUANTITATION USING KNOWN RETENTION TIMES, RESPONSE FACTORS, REFERENCE COMPOUND, AND MASS SPECTRA

EGD No.	Compound	CAS Registry
943	benzoic acid	65-85-0
944	p-cresol	106-44-5
945	3,5-dibromo-4-hydroxybenzonitrile	1689-84-5
946	2,6-dichlorophenol	87-65-0
947	hexanoic acid	142-62-1
948	2,3,4,6-tetrachlorophenol	58-90-2

2.2 Identification of a pollutant (qualitative analysis) is performed in one of three ways: (1) For compounds listed in Tables 1 and 2, and for other compounds for which authentic standards are available, the GCMS system is calibrated and the mass spectrum and retention time for each standard are stored in a user created library. A compound is identified when its retention time and mass spectrum agree with the library retention time and spectrum. (2) For compounds listed in Tables 3 and 4, and for other compounds for which standards are not available, a compound is identified when the retention time and mass spectrum agree with those specified in this method. (3) For chromatographic peaks which are not identified by (1) and (2) above, the background corrected spectrum at the peak maximum is compared with spectra in the EPA/NIH Mass Spectral File (Reference 2). Tentative identification is established when the spectrum agrees (see Section 13).

2.3 Quantitative analysis is performed in one of four ways by GCMS using extracted ion current profile (EICP) areas: (1) For compounds listed in Tables 1 and 2, and for other compounds for which standards and labeled analogs are available, the GCMS system is calibrated and the compound concentration is determined using an isotope dilution technique. (2) For compounds listed in Tables 1 and 2, and for other compounds for which authentic standards but no labeled compounds are available, the GCMS system is calibrated and the compound concentration is determined using an internal standard technique. (3) For compounds listed in Tables 3 and 4, and for other compounds for which standards are not available, compound concentrations are determined using

known response factors. (4) For compounds for which neither standards nor known response factors are available, compound concentration is determined using the sum of the EICP areas relative to the sum of the EICP areas of the internal standard.

2.4 The quality of the analysis is assured through reproducible calibration and testing of the extraction and GCMS systems.

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

		Retention Time				Method Detection Limit [4]	
EGD No.[1]	Compound	Mean (sec)	EGD Ref	Relative [2]	Minimum Level [3] (µg/L)	Low solids (µg/kg)	High Solids (µg/kg)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000–1.000	10		
930	pyridine	378	164	0.325			
261	N-nitrosodimethylamine-d_6[5]	378	164	0.286- 0.364	50		
361	N-nitrosodimethylamine[5]	385	261	1.006–1.028	50	16	27
585	N,N-dimethylformamide	407	164	0.350			
580	1,2:3,4-diepoxybutane	409	164	0.352			
603	alpha picoline-d_7	417	164	0.326–0.393	50		
703	alpha picoline	426	603	1.006–1.028	50	25	87
917	N-nitrosomethylethylamine	451	164	0.338			
598	methyl methanesulfonate	511	164	0.439			
610	styrene-d_5	546	164	0.450–0.488	10		
710	styrene	549	610	1.002–1.009	10	149*	17
916	N-nitrosodiethylamine	570	164	0.490			
577	1,3-dichloro-2-propanol	589	164	0.506			
589	ethyl methanesulfonate	637	164	0.548			
582	dimethyl sulfone	649	164	0.558			
562	benzenethiol	667	164	0.574			
922	pentachloroethane	680	164	0.585			
557	aniline	694	164	0.597			
613	p-cymene-d_{14}	742	164	0.624–0.652	10		
713	p-cymene	755	613	1.008–1.023	10	426*	912*
265	phenol-d_5	696	164	0.584–0.613	10		
365	phenol	700	265	0.995–1.010	10	2501*	757*
218	bis(2-chloroethyl) ether-d_8	696	164	0.584–0.607	10		
318	bis(2-chloroethyl) ether	704	218	1.007–1.016	10	32	22
617	n-C10-d_{22}	698	164	0.585–0.615	10		
717	n-C10	720	617	1.022–1.038	10	299*	1188*
226	1,3-dichlorobenzene-d_4	722	164	0.605–0.636	10		
326	1,3-dichlorobenzene	724	226	0.998–1.008	10	46	26
227	1,4-dichlorobenzene-d_4	737	164	0.601–0.666	10		
327	1,4-dichlorobenzene	740	227	0.997–1.009	10	35	20
225	1,2-dichlorobenzene-d_4	758	164	0.632–0.667	10		
325	1,2-dichlorobenzene	760	225	0.995–1.008	10	63	16
935	thioacetamide	768	164	0.660			
564	benzyl alcohol	785	164	0.675			

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low solids (µg/kg)	High Solids (µg/kg)
242	bis(2-chloroisopropyl) ether-d_{12}	788	164	0.664–0.691	10		
342	bis(2-chloroisopropyl) ether	799	242	1.010–1.016	10	24	39
571	o-cresol	814	164	0.700			
263	N-nitrosodi-n-propylamine-d_{14}[5]	817	164	0.689–0.716	20		
363	N-nitrosodi-n-propylamine[5]	830	263	1.008–1.023	20	46	47
555	acetophenone	818	164	0.703			
212	hexachloroethane-^{13}C	819	164	0.690–0.717	10		
312	hexachloroethane	823	212	0.999–1.001	10	58	55
937	o-toluidine	830	164	0.714			
919	N-nitrosomorpholine	834	164	0.717			
575	1,2-dibromo-3-chloropropane	839	164	0.721			
256	nitrobenzene-d_5	845	164	0.706–0.727	10		
356	nitrobenzene	849	256	1.002–1.007	10	39	28
566	3-bromochlorobenzene	854	164	0.734			
565	2-bromochlorobenzene	880	164	0.757			
941	tripropylene glycol methyl ether	881	164	0.758			
254	isophorone-d_8	881	164	0.747–0.767	10		
354	isophorone	889	254	0.999–1.017	10	8	5
942	1,3,5-trithiane	889	164	0.764			
920	N-nitrosopiperidine	895	164	0.770			
234	2,4-dimethylphenol-d_3	921	164	0.781–0.803	10		
334	2,4-dimethylphenol	924	234	0.999–1.003	10	26	13
243	bis(2-chloroethoxy) methane-d_6[5]	933	164	0.792–0.807	10		
343	bis(2-chloroethoxy) methane[5]	939	243	1.000–1.013	10	26	23
208	1,2,4-trichlorobenzene-d_3	955	164	0.813–0.830	10		
308	1,2,4-trichlorobenzene	958	208	1.000–1.005	10	49	24
558	o-anisidine	962	164	0.827			
255	naphthalene-d_8	963	164	0.819–0.836	10		
355	naphthalene	967	255	1.001–1.006	10	62	42
934	thianapthene	971	164	0.835			
609	alpha-terpineol-d_3	973	164	0.829–0.844	10		
709	alpha-terpineol	975	609	0.998–1.008	10	nd	nd
606	n-C12-d_{26}	953	164	0.730–0.908	10		
706	n-C12	981	606	0.986–1.051	10	860*	3885*
629	1,2,3-trichlorobenzene-d_3[5]	1000	164	0.852–0.868	10		
729	1,2,3-trichlorobenzene[5]	1003	629	1.000–1.005	10	260*	164*
252	hexachlorobutadiene-$^{13}C_4$	1005	164	0.856–0.871	10		
352	hexachlorobutadiene	1006	252	0.999–1.002	10	46	22

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low solids (µg/kg)	High Solids (µg/kg)
918	N-nitrosomethylphenylamine	1006	164	0.865			
592	hexachloropropene	1013	164	0.871			
569	4-chloroaniline	1016	164	0.874			
570	3-chloronitrobenzene	1018	164	0.875			
915	N-nitrosodi-n-butylamine	1063	164	0.914			
923	pentamethylbenzene	1083	164	0.931			
561	1,3-benzenediol	1088	164	0.936			
931	safrole	1090	164	0.937			
939	2,4,5-trimethylaniline	1091	164	0.938			
904	2-methylnaphthalene	1098	164	0.944			
599	2-methylbenzothiazole	1099	164	0.945			
568	5-chloro-o-toluidine	1101	164	0.947			
938	1,2,3-trimethoxybenzene	1128	164	0.970			
933	1,2,4,5-tetrachlorobenzene	1141	164	0.981			
253	hexachlorocyclo-pentadiene-$^{13}C_4$	1147	164	0.976–0.986	10		
353	hexachlorocyclopentadiene	1142	253	0.999–1.001	10	nd	nd
594	isosafrole (cis or trans)	1147	164	0.986			
594	isosafrole (cis or trans)	1190	164	1.023			
578	2,3-dichloroaniline	1160	164	0.997			
574	2,4-diaminotoluene	1187	164	1.021			
220	2-chloronaphthalene-d_7	1185	164	1.014–1.024	10		
320	2-chloronaphthalene	1200	220	0.997–1.007	10	80	59
518	n-C14	1203	164	1.034	10	256	3533
612	biphenyl-d_{10}	1195	164	1.016–1.027	10		
712	biphenyl	1205	612	1.001–1.006	10	67	55
608	diphenyl ether-d_{10}	1211	164	1.036–1.047	10		
708	diphenyl ether	1216	608	0.997–1.009	10	44	12
579	2,3-dichloronitrobenzene	1214	164	1.044			
911	2-nitroaniline	1218	164	1.047			
908	1,4-naphthoquinone	1224	164	1.052			
595	longifolene	1225	164	1.053			
277	acenaphthylene-d_8	1265	164	1.080–1.095	10		
377	acenaphthylene	1247	277	1.000–1.004	10	57	18
593	2-isopropylnaphthalene	1254	164	1.078			
587	1,4-dinitrobenzene	1255	164	1.079			
576	2,6-dichloro-4-nitroaniline	1259	164	1.083			
271	dimethyl phthalate-d_4	1269	164	1.083–1.102	10		
371	dimethyl phthalate	1273	271	0.998–1.005	10	62	21
573	2,6-di-t-butyl-p-benzoquinone	1273	164	1.095			
236	2,6-dinitrotoluene-d_3	1283	164	1.090–1.112	10		
336	2,6-dinitrotoluene	1300	236	1.001–1.005	10	55	47
912	3-nitroaniline	1297	164	1.115			

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low solids (µg/kg)	High Solids (µg/kg)
201	acenaphthene-d_{10}	1298	164	1.107–1.125	10		
301	acenaphthene	1304	201	0.999–1.009	10	64	55
605	dibenzofuran-d_8	1331	164	1.134–1.155	10		
705	dibenzofuran	1335	605	0.998–1.007	10	77	210*
921	pentachlorobenzene	1340	164	1.152			
909	alpha-naphthylamine	1358	164	1.168			
235	2,4-dinitrotoluene-d_3	1359	164	1.152–1.181	10		
335	2,4-dinitrotoluene	1364	235	1.000–1.002	10	65	209*
602	beta-naphthylamine-d_7	1368	164	1.163–1.189	50		
702	beta-naphthylamine	1371	602	0.996–1.007	50	49	37
590	ethylenethiourea	1381	164	1.187			
280	fluorene-d_{10}	1395	164	1.185–1.214	10		
380	fluorene	1401	281	0.999–1.008	10	69	61
240	4-chlorophenyl phenyl ether-d_5	1406	164	1.194–1.223	10		
340	4-chlorophenyl phenyl ether	1409	240	0.990–1.015	10	73	59
270	diethyl phthalate-d_4	1409	164	1.197–1.229	10		
370	diethyl phthalate	1414	270	0.996–1.006	10	52	16
906	2-(methylthio)benzothiazole	1415	164	1.217			
567	4-chloro-2-nitroaniline	1421	164	1.222			
910	5-nitro-o-toluidine	1422	164	1.223			
913	4-nitroaniline	1430	164	1.230			
619	n-C16-d_{34}	1447	164	1.010–1.478	10		
719	n-C16	1469	619	1.013–1.020	10	116*	644*
237	1,2-diphenylhydrazine-$_d$8	1433	164	1.216–1.248	20		
337	1,2-diphenylhydrazine[6]	1439	237	0.999–1.009	20	48	27
607	diphenylamine-d_{10}	1437	164	1.213–1.249	20		
707	diphenylamine	1439	607	1.000–1.007	20	58	54
262	N-nitrosodiphenylamine-d_6	1447	164	1.225–1.252	20		
362	N-nitrosodiphenylamine[7]	1464	262	1.000–1.002	20	55	36
241	4-bromophenyl phenyl ether-d_5[5]	1495	164	1.271–1.307	10		
341	4-bromophenyl phenyl ether[5]	1498	241	0.990–1.015	10	55	17
925	phenacetin	1512	164	1.300			
903	1-methylfluorene	1514	164	1.302			
209	hexachlorobenzene-$^{13}C_6$	1521	164	1.288–1.327	10		
309	hexachlorobenzene	1522	209	0.999–1.001	10	51	48
556	4-aminobiphenyl	1551	164	1.334			
929	pronamide	1578	164	1.357			
281	phenanthrene-d_{10}	1578	164	1.334–1.380	10		
520	n-C18	1580	164	1.359	10	134*	844*
381	phenanthrene	1583	281	1.000–1.005	10	42	22
278	anthracene-d_{10}	1588	164	1.342–1.388	10		

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low solids (µg/kg)	High Solids (µg/kg)
378	anthracene	1592	278	0.998–1.006	10	52	21
604	dibenzothiophene-d_8	1559	164	1.314–1.361	10		
704	dibenzothiophene	1564	604	1.000–1.006	10	72	71
588	diphenyldisulfide	1623	164	1.396			
914	4-nitrobiphenyl	1639	164	1.409			
927	1-phenylnaphthalene	1643	164	1.413			
628	carbazole-d_8[5]	1645	164	1.388–1.439	20		
728	carbazole[5]	1650	628	1.000–1.006	20	47	24
621	n-C20-d_{42}	1655	164	1.184–1.662	10		
721	n-C20	1677	621	1.010–1.021	10	83	229*
907	1,5-naphthalenediamine	1676	164	1.441			
902	4,5-methylenephenanthrene	1690	164	1.453			
905	1-methylphenanthrene	1697	164	1.459			
268	di-n-butyl phthalate-d_4	1719	164	1.446–1.510	10		
368	di-n-butyl phthalate	1723	268	1.000–1.003	10	64	80
928	2-phenylnaphthalene	1733	164	1.490			
586	3,6-dimethylphenanthrene	1763	164	1.516			
597	methapyrilene	1781	164	1.531			
926	phenothiazine	1796	164	1.544			
239	fluoranthene-d_{10}	1813	164	1.522–1.596	10		
339	fluoranthene	1817	239	1.000–1.004	10	54	22
572	crotoxyphos	1822	164	1.567			
936	thioxanthone	1836	164	1.579			
284	pyrene-d_{10}	1844	164	1.523–1.644	10		
384	pyrene	1852	284	1.001–1.003	10	40	48
205	benzidine-d_8	1854	164	1.549–1.632	50		
305	benzidine	1853	205	1.000–1.002	50	nd	nd
522	n-C22	1889	164	1.624	10	432*	447*
559	aramite	1901	164	1.635			
559	aramite	1916	164	1.647			
583	p-dimethylaminoazobenzene	1922	164	1.653			
563	2,3-benzofluorene	1932	164	1.661			
623	n-C24-d_{50}	1997	164	1.671–1.764	10		
723	n-C24	2025	612	1.012–1.015	10	--	--
932	squalene	2039	164	1.753			
267	butylbenzyl phthalate-d_4[5]	2058	164	1.715–1.824	10		
367	butylbenzyl phthalate[5]	2060	267	1.000–1.002	10	60	65
276	chrysene-d_{12}	2081	164	1.743–1.837	10		
376	chrysene	2083	276	1.000–1.004	10	51	48
901	4,4'methylenebis (2-chloroaniline)	2083	164	1.791			
272	benzo(a)anthracene-d_{12}	2082	164	1.735–1.846	10		
372	benzo(a)anthracene	2090	272	0.999–1.007	10	61	47

TABLE 5. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low solids (µg/kg)	High Solids (µg/kg)
581	3,3'-dimethoxybenzidine	2090	164	1.797			
228	3,3'-dichlorobenzidine-d_6	2088	164	1.744–1.848	50		
328	3,3'-dichlorobenzidine	2086	228	1.000–1.001	50	62	111
940	triphenylene	2088	164	1.795			
560	benzanthrone	2106	164	1.811			
266	bis(2-ethylhexyl) phthalate-d_4	2123	164	1.771–1.880	10		
366	bis(2-ethylhexyl) phthalate	2124	266	1.000–1.002	10	553*	1310*
524	n-C26	2147	164	1.846	10	609*	886*
591	ethynylestradiol 3-methyl ether	2209	164	1.899			
269	di-n-octyl phthalate-d_4	2239	164	1.867–1.982	10		
369	di-n-octyl phthalate	2240	269	1.000–1.002	10	72	62
525	n-C28	2272	164	1.954	10	492*	1810*
584	7,12-dimethylbenz(a)-anthracene	2284	164	1.964			
274	benzo(b)fluoranthene-d_{12}	2281	164	1.902–2.025	10		
374	benzo(b)fluoranthene	2293	274	1.000–1.005	10	54	30
275	benzo(k)fluoranthene-d_{12}	2287	164	1.906–2.033	10		
375	benzo(k)fluoranthene	2293	275	1.000–1.005	10	95	20
924	perylene	2349	164	2.020			
273	benzo(a)pyrene-d_{12}	2351	164	1.954–2.088	10		
373	benzo(a)pyrene	2350	273	1.000–1.004	10	52	15
626	n-C30-d_{62}	2384	164	1.972–2.127	10		
726	n-C30	2429	626	1.011–1.028	10	252*	658*
596	malachite green	2382	164	2.048			
900	3-methylcholanthrene	2439	164	2.097			
083	indeno(1,2,3-cd)pyrene	2650	164	2.279	20	67	263*
282	dibenzo(a,h)anthracene-d_{14}[5]	2649	164	2.107–2.445	20		
382	dibenzo(a,h)anthracene[5]	2660	282	1.000–1.007	20	49	125
279	benzo(ghi)perylene-d_{12}	2741	164	2.187–2.524	20		
379	benzo(ghi)perylene	2750	279	1.001–1.006	20	44	nd

[1]Reference numbers beginning with 0, 1, 5, or 9 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2]Single values in this column are based on single laboratory data.
[3]This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. The concentration in the aqueous or solid phase is determined using the equations in Section 14.0.

[4]Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).
[5]Specification derived from related compound.
[6]Detected as azobenzene.
[7]Detected as diphenylamine.

nd = not detected when spiked into the sludge tested
*Background levels of these compounds were present in the sludge tested, resulting in higher than expected MDL's. The MDL for these compounds is expected to be approximately 50 µg/kg with no interferences present.

Column: 30 ±2 m x 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.

Temperature program: Five minutes at 30°C; 30-280°C at 8°C per min; isothermal at 280°C until benzo(ghi)perylene elutes.

Gas velocity: 30 ±5 cm/sec at 30°C.

TABLE 6. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR ACID EXTRACTABLE COMPOUNDS

		Retention Time				Method Detection Limit [4]	
EGD No.[1]	Compound	Mean (sec)	EGD Ref	Relative [2]	Minimum Level [3] (µg/L)	Low Solids (µg/kg)	High Solids (µg/kg)
164	2,2'-difluorobiphenyl (int std)	1163	164	1.000–1.000	10		
224	2-chlorophenol-d_4	701	164	0.587–0.618	10		
324	2-chlorophenol	705	224	0.997–1.010	10	18	10
947	hexanoic acid	746	164	0.641			
944	p-cresol	834	164	0.717			
257	2-nitrophenol-d_4	898	164	0.761–0.783	20		
357	2-nitrophenol	900	257	0.994–1.009	20	39	44
231	2,4-dichlorophenol-d_3	944	164	0.802–0.822	10		
331	2,4-dichlorophenol	947	231	0.997–1.006	10	24	116
943	benzoic acid	971	164	0.835			
946	2,6-dichlorophenol	981	164	0.844			
222	4-chloro-3-methylphenol-d_2	1086	164	0.930–0.943	10		
322	4-chloro-3-methylphenol	1091	222	0.998–1.003	10	41	62
221	2,4,6-trichlorophenol-d_2	1162	164	0.994–1.005	10	46	111
321	2,4,6-trichlorophenol	1165	221	0.998–1.004	10		
631	2,4,5-trichlorophenol-d_2[5]	1167	164	0.998–1.009	10		
731	2,4,5-trichlorophenol	1170	631	0.998–1.004	10	32	55
530	2,3,6-trichlorophenol	1195	164	1.028	10	58	37
259	2,4-dinitrophenol-d_3	1323	164	1.127–1.149	50		
359	2,4-dinitrophenol	1325	259	1.000–1.005	50	565	642
258	4-nitrophenol-d_4	1349	164	1.147–1.175	50		

TABLE 6. GAS CHROMATOGRAPHIC RETENTION TIMES AND DETECTION LIMITS FOR ACID EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention Time			Minimum Level[3] (µg/L)	Method Detection Limit[4]	
		Mean (sec)	EGD Ref	Relative[2]		Low Solids (µg/kg)	High Solids (µg/kg)
358	4-nitrophenol	1354	258	0.997–1.006	50	287	11
948	2,3,4,6-tetrachlorophenol	1371	164	1.179			
260	2-methyl-4,6-dinitrophenol-d_2	1433	164	1.216–1.249	20		
360	2-methyl-4,6-dinitrophenol	1435	260	1.000–1.002	20	385	83
945	3,5-dibromo-4-hydroxybenzonitrile	1481	164	1.273			
264	pentachlorophenol-$^{13}C_6$	1559	164	1.320–1.363	50		
364	pentachlorophenol	1561	264	0.998–1.002	50	51	207

[1]Reference numbers beginning with 0, 1, 5, or 9 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2]Single values in this column are based on single laboratory data.
[3]This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. The concentration in the aqueous or solid phase is determined using the equations in section 14.
[4]Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).
[5]Specification derived from related compound.

Column: 30 ±2 m x 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.

Temperature program: Five minutes at 30°C; 30-250°C or until pentachlorophenol elutes.

Gas velocity: 30 ±5 cm/sec at 30°C.

3.0 Contamination And Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given eight hour shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450°C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

4.0 Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 3-5.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene, 3,3'-dichlorobenzidine, dibenzo(a,h)anthracene, benzo(a)pyrene, N-nitrosodimethylamine, and beta-naphthylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5.0 Apparatus And Materials

5.1 Sampling Equipment—For discrete or composite sampling.

5.1.1 Sample bottles and caps

5.1.1.1 Liquid samples (waters, sludges and similar materials that contain less than 5% solids)—Sample bottle, amber glass, 1.1 L minimum, with screw cap.

5.1.1.2 Solid samples (soils, sediments, sludges, filter cake, compost, and similar materials that contain more than 5% solids)—Sample bottle, wide mouth, amber glass, 500 mL minimum.

5.1.1.3 If amber bottles are not available, samples shall be protected from light.

5.1.1.4 Bottle caps—Threaded to fit sample bottles. Caps shall be lined with Teflon.

5.1.1.5 Cleaning

5.1.1.5.1 Bottles are detergent water washed, then solvent rinsed or baked at 450°C for one hour minimum before use.

5.1.1.5.2 Cap liners are washed with detergent and water, rinsed with reagent water (see Section 6.5.1) and then solvent, and then baked for at least one hour at approximately 200°C.

5.1.2 Compositing equipment—Automatic or manual compositing system incorporating glass containers cleaned per bottle cleaning procedure above. Sample containers

are kept at 0-4°C during sampling. Only glass or Teflon tubing shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used only in the pump. Before use, the tubing shall be thoroughly rinsed with methanol, followed by repeated rinses with reagent water (Section 6.5.1) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Equipment—For determining percent moisture.

5.2.1 Oven—Capable of maintaining a temperature of 110 ±5°C.

5.2.2 Desiccator.

5.3 Sonic Disruptor—375 watt with pulsing capability and 3/4 in. disruptor horn (Ultrasonics, Inc, Model 375C, or equivalent).

5.4 Extraction Apparatus

5.4.1 Continuous liquid-liquid extractor—Teflon or glass connecting joints and stopcocks without lubrication, 1.5-2 L capacity (Hershberg-Wolf Extractor, Ace Glass 6841-10, or equivalent).

5.4.2 Beakers

5.4.2.1 1.5-2 L borosilicate glass beakers calibrated to 1 L.

5.4.2.2 400-500 mL borosilicate glass beakers.

5.4.2.3 Spatulas—Stainless steel.

5.4.3 Filtration apparatus

5.4.3.1 Glass funnel—125-250 mL.

5.4.3.2 Filter paper for above (Whatman 41, or equivalent)

5.5 Drying Column—15-20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.6 Concentration Apparatus

5.6.1 Concentrator tube—Kuderna-Danish (K-D) 10 mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.6.2 Evaporation flask—Kuderna-Danish (K-D) 500 mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012).

5.6.3 Snyder column—Kuderna-Danish (K-D) three-ball macro (Kontes K-503000-0232, or equivalent).

5.6.4 Snyder column—Kuderna-Danish (K-D) two-ball micro (Kontes K-469002-0219, or equivalent).

5.6.5 Boiling chips—Approximately 10/40 mesh, extracted with methylene chloride and baked at 450°C for one hour minimum.

5.6.6 Nitrogen evaporation device—Equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent) is suitable.

5.7 Water Bath—Heated, with concentric ring cover, capable of temperature control (±2°C), installed in a fume hood.

5.8 Sample Vials—Amber glass, 2-5 mL with Teflon-lined screw cap.

5.9 Balances

5.9.1 Analytical—Capable of weighing 0.1 mg.

5.9.2 Top loading—Capable of weighing 10 mg.

5.10 Automated Gel Permeation Chromatograph—Analytical Biochemical Labs, Inc., Columbia, MO, Model GPC Autoprep 1002, or equivalent.

5.10.1 Column—600-700 mm x 25 mm i.d., packed with 70 g of SX-3 Bio-beads (Bio-Rad Laboratories, Richmond, CA).

5.10.2 UV detectors—254 mu, preparative or semi-prep flow cell:

5.10.2.1 Schmadzu—5 mm path length.

5.10.2.2 Beckman—Altex 152W, 8 µL micro-prep flow cell, 2 mm path.

5.10.2.3 Pharmacia UV-1— 3 mm flow cell.

5.10.2.4 LDC Milton-Roy UV-3—Monitor #1203.

5.11 Gas Chromatograph—Shall have splitless or on-column injection port for capillary column, temperature program with 30°C hold, and shall meet all of the performance specifications in Section 12.

5.11.1 Column—30 ±5 m x 0.25 ±0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J&W DB-5, or equivalent).

5.12 Mass Spectrometer—70 eV electron impact ionization, shall repetitively scan from 35-450 amu in 0.95-1.00 second, and shall produce a unit resolution (valleys between m/z 441-442 less than 10 percent of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in Table 7 (Reference 6). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or

ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

TABLE 7. DFTPP MASS-INTENSITY SPECIFICATIONS*

Mass	Intensity Required
51	8-82% of m/z 198
68	less than 2% of m/z 69
69	11-91% of m/z 198
70	less than 2% of m/z 69
127	32-59% of m/z 198
197	less than 1% of m/z 198
198	base peak, 100% abundance
199	4-9% of m/z 198
275	11-30% of m/z 198
441	44-110% of m/z 443
442	30-86% of m/z 198
443	14-24% of m/z 442

*Reference 6.

5.13 Data System—Shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data, generate reports, and shall compute and record response factors.

5.13.1 Data acquisition—Mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.13.2 Mass spectral libraries—User created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (Section 7.2).

5.13.3 Data processing—The data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.13.4 Response factors and multipoint calibrations—The data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multi-point calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are used for testing calibration linearity. Statistics on initial (Section 8.2) and on-going (Section 12.7) performance shall be computed and maintained.

6.0 Reagents and Standards

6.1 Reagents for Adjusting Sample pH

6.1.1 Sodium hydroxide—Reagent grade, 6 N in reagent water.

6.1.2 Sulfuric acid—Reagent grade, 6 N in reagent water.

6.2 Sodium Sulfate—Reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g), baked at 450°C for one hour minimum, cooled in a desiccator, and stored in a pre-cleaned glass bottle with screw cap which prevents moisture from entering.

6.3 Methylene Chloride—Distilled in glass (Burdick and Jackson, or equivalent).

6.4 GPC Calibration Solution—Containing 300 mg/mL corn oil, 15 mg/mL bis(2-ethylhexyl) phthalate, 1.4 mg/mL pentachlorophenol, 0.1 mg/mL perylene, and 0.5 mg/mL sulfur.

6.5 Reference Matrices

6.5.1 Reagent water—Water in which the compounds of interest and interfering compounds are not detected by this method.

6.5.2 High solids reference matrix—Playground sand or similar material in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard Solutions—Purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96% or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at -20 to -10°C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of Stock Solutions—Prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in Section 4. The large number of labeled and unlabeled acid and base/neutral compounds used for combined calibration (Section 7) and calibration verification (Section 12.5) require high concentrations (approximately 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10-200 µg/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check standards indicates a change in concentration.

6.8 Labeled Compound Spiking Solution—From stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 µg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary Standard—Using stock solutions (Section 6.7), prepare a secondary standard containing all of the compounds in Tables 1 and 2 at a concentration of 400 µg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal Standard Solution—Prepare 2,2'-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP Solution—Prepare at 50 µg/mL in acetone.

6.12 Solutions for Obtaining Authentic Mass Spectra (Section 7.2)—Prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.

6.13 Calibration Solutions—Combine 5 aliquots of 0.5 mL each of the solution in Section 6.8 with 25, 50, 125, 250, and 500 µL of the solution in Section 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100 and 200 µg/mL of the pollutants and a constant nominal 100 µg/mL of the labeled compounds. Spike each solution with 10 µL of the internal standard solution (Section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (Section 7.4).

6.14 Precision and Recovery Standard—Used for determination of initial (Section 8.2) and on-going (Section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 µg/mL.

6.15 Stability of Solutions—All standard solutions (Sections 6.8 through 6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ±15% of the area obtained in the initial analysis of the standard.

7.0 Calibration

7.1 Assemble the GCMS and establish the operating conditions in Table 5. Analyze standards per the procedure in Section 11 to demonstrate that the analytical system meets the minimum levels in Tables 5 and 6, and the mass-intensity criteria in Table 7 for 50 ng DFTPP.

7.2 Mass Spectral Libraries—Detection and identification of compounds of interest are dependent upon spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. Examine the spectrum to determine that only a single compound is present. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (Section 7.1 and Table 7) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the five most intense mass spectral peaks and all other mass spectral peaks greater than 10% of the base peak. The spectrum may be further edited to remove common interfering masses. If five mass spectral peaks cannot be obtained under the scan conditions given in Section 5.12, the mass spectrometer may be scanned to an m/z lower than 35 to gain additional spectral information. The spectrum obtained is stored for reverse search and for compound confirmation.

7.2.5 For the compounds in Tables 3 and 4 and for other compounds for which the mass spectra, quantitation m/z's, and retention times are known but the instrument is not to be calibrated, add the retention time and reference compound (Tables 5 and 6); the response factor and the quantitation m/z (Tables 8 and 9); and spectrum (Appendix A) to the reverse search library. Edit the spectrum per Section 7.2.4, if necessary.

7.3 Analytical Range—Demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (Section 15.3). The 20 µg/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection—Demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 µg/mL level (per all criteria in Section 13). The 50 µg/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

TABLE 8. CHARACTERISTIC M/Z's AND RESPONSE FACTORS OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

Compound	Labeled Analog	Primary m/z[1]	Response Factor[2]
acenaphthene	d_{10}	154 / 164	
acenaphthylene	d_8	152 / 160	
acetophenone		105	0.79
4-aminobiphenyl		169	0.81
aniline		93	1.04
o-anisidine		108	0.43
anthracene	d_{10}	178 / 188	
aramite		185	0.19
benzanthrone		230	0.15
1,3-benzenediol		110	0.78
benzenethiol		110	0.18
benzidine	d_8	184 / 192	
benzo(a)anthracene	d_{12}	228 / 240	
benzo(b)fluoranthene	d_{12}	252 / 264	
benzo(k)fluoranthene	d_{12}	252 / 264	
benzo(a)pyrene	d_{12}	252 / 264	
benzo(ghi)perylene	d_{12}	276 / 288	
2,3-benzofluorene		216	0.35
benzoic acid		105	0.16
benzyl alcohol		79	0.47
biphenyl	d_{10}	154 / 164	
bis(2-chloroethyl) ether	d_8	93 / 101	
bis(2-chloroethoxy)methane	d_6	93 / 99	
bis(2-chloroisopropyl) ether	d_{12}	121 / 131	
bis(2-ethylhexyl) phthalate	d_4	149 / 153	
2-bromochlorobenzene		111	0.33
3-bromochlorobenzene		192	0.40
4-bromophenyl phenyl ether	d_5	248 / 253	
butyl benzyl phthalate	d_4	149 / 153	
n-C10	d_{22}	57 / 82	
n-C12	d_{26}	57 / 66	
n-C14		57	
n-C16	d_{34}	57 / 66	
n-C18		57	
n-C20	d_{42}	57 / 66	
n-C22		57	
n-C24	d_{50}	57 / 66	
n-C26		57	
n-C28		57	
n-C30	d_{62}	57 / 66	
carbazole	d_8	167 / 175	
4-chloro-2-nitroaniline		172	0.20
5-chloro-o-toluidine		106	0.50
4-chloroaniline		127	0.73
2-chloronaphthalene	d_7	162 / 169	
3-chloronitrobenzene		157	0.18
4-chlorophenyl phenyl ether	d_5	204 / 209	

TABLE 8. CHARACTERISTIC M/Z's AND RESPONSE FACTORS OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

Compound	Labeled Analog	Primary m/z[1]	Response Factor[2]
3-chloropropionitrile		54	0.42
chrysene	d_{12}	228 / 240	
o-cresol		108	0.59
crotoxyphos		127	0.017
p-cymene	d_{14}	119 / 130	
2,6-di-tert-butyl-p-benzoquinone		220	0.078
di-n-butyl phthalate	d_4	149 / 153	
2,4-diaminotoluene		122	0.059
dibenzo(a,h)anthracene	d_{14}	278 / 292	
dibenzofuran	d_8	168 / 176	
dibenzothiophene	d_8	184 / 192	
1,2-dibromo-3-chloropropane		157	0.22
2,6-dichloro-4-nitroaniline		124	0.019
1,3-dichloro-2-propanol		79	0.68
2,3-dichloroaniline		161	0.47
1,2-dichlorobenzene	d_4	146 / 152	
1,3-dichlorobenzene	d_4	146 / 152	
1,4-dichlorobenzene	d_4	146 / 152	
3,3'-dichlorobenzidine	d_6	252 / 258	
2,2'-difluorobiphenyl (int std)		190	
2,3-dichloronitrobenzene		191	0.11
1,2:3,4-diepoxybutane		55	0.27
diethyl phthalate	d_4	149 / 153	
3,3'-dimethoxybenzidine		244	0.19
dimethyl phthalate	d_4	163 / 167	
dimethyl sulfone		79	0.40
p-dimethylaminoazobenzene		120	0.23
7,12-dimethylbenz(a) anthracene		256	0.58
N,N-dimethylformamide		73	0.51
3,6-dimethylphenanthrene		206	0.72
2,4-dimethylphenol	d_3	122 / 125	
1,4-dinitrobenzene		168	0.24
2,4-dinitrotoluene	d_3	165 / 168	
2,6-dinitrotoluene	d_3	165 / 167	
di-n-octyl phthalate	d_4	149 / 153	
diphenylamine	d_{10}	169 / 179	
diphenyl ether	d_{10}	170 / 180	
diphenyldisulfide		218	0.25
1,2-diphenylhydrazine[3]	d_{10}	77 / 82	
ethyl methanesulfonate		109	0.28
ethylenethiourea		102	0.22
ethynylestradiol 3-methyl ether		227	0.28
fluoranthene	d_{10}	202 / 212	
fluorene	d_{10}	166 / 176	
hexachlorobenzene	$^{13}C_6$	284 / 292	

TABLE 8. CHARACTERISTIC M/Z's AND RESPONSE FACTORS OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

Compound	Labeled Analog	Primary m/z[1]	Response Factor[2]
hexachlorobutadiene	$^{13}C_4$	225 / 231	
hexachloroethane	$^{13}C_2$	201 / 204	
hexachlorocyclopentadiene	$^{13}C_4$	237 / 241	
hexachloropropene		213	0.23
indeno(1,2,3-cd)pyrene		276	
isophorone	d_8	82 / 88	
2-isopropylnaphthalene		170	0.32
isosafrole		162	0.33
longifolene		161	0.14
malachite green		330	
methapyrilene		97	0.43
methyl methanesulfonate		80	0.20
2-methylbenzothiazole		149	0.59
3-methylcholanthrene		268	0.59
4,4'-methylenebis (2-chloroaniline)		231	0.21
4,5-methylenephenanthrene		190	0.44
1-methylfluorene		180	0.37
2-methylnaphthalene		142	0.99
1-methylphenanthrene		192	0.65
2-(methylthio)benzothiazole		181	0.42
naphthalene	d_8	128 / 136	
1,5-naphthalenediamine		158	0.085
1,4-naphthoquinone		158	0.021
alpha-naphthylamine		143	0.89
beta-naphthylamine	d_7	143 / 150	
5-nitro-o-toluidine		152	0.31
2-nitroaniline		138	0.39
3-nitroaniline		138	0.27
4-nitroaniline		138	0.11
nitrobenzene	d_5	123 / 128	
4-nitrobiphenyl		199	0.35
N-nitrosodi-n-butylamine		84	0.47
N-nitrosodi-n-propylamine	d_{14}	70 / 78	
N-nitrosodiethylamine		102	0.45
N-nitrosodimethylamine	d_6	74 / 80	
N-nitrosodiphenylamine[4]	d_6	169 / 175	
N-nitrosomethylethylamine		88	0.33
N-nitrosomethylphenylamine		106	0.024
N-nitrosomorpholine		56	0.49
N-nitrosopiperidine		114	0.41
pentachlorobenzene		248	0.25
pentachloroethane		117	0.20
pentamethylbenzene		148	0.42
perylene		252	0.30
phenacetin		108	0.38
phenanthrene	d_{10}	178 / 188	

TABLE 8. CHARACTERISTIC M/Z's AND RESPONSE FACTORS OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

Compound	Labeled Analog	Primary m/z[1]	Response Factor[2]
phenol	d_5	94/71	
phenothiazine		199	0.15
1-phenylnaphthalene		204	0.48
2-phenylnaphthalene		204	0.73
alpha-picoline	d_7	93/100	
pronamide		173	0.31
pyrene	d_{10}	202/212	
pyridine		79	0.68
safrole		162	0.45
squalene		69	0.042
styrene	d_5	104/109	
alpha-terpineol	d_3	59/62	
1,2,4,5-tetrachlorobenzene		216	0.43
thianaphthene		134	1.52
thioacetamide		75	0.28
thioxanthone		212	0.23
o-toluidine		106	1.04
1,2,3-trichlorobenzene	d_3	180/183	
1,2,4-trichlorobenzene	d_3	180/183	
1,2,3-trimethoxybenzene		168	0.48
2,4,5-trimethylaniline		120	0.28
triphenylene		228	1.32
tripropylene glycol methyl ether		59	0.092
1,3,5-trithiane		138	0.15

[1]Native/labeled
[2]Referenced to 2,2'-difluorobiphenyl
[3]Detected as azobenzene
[4]Detected as diphenylamine

NOTE: *Because the composition and purity of commercially-supplied isotopically labeled standards may vary, the primary m/z of the labeled analogs given in this table should be used as guidance. The appropriate m/z of the labeled analogs should be determined prior to use for sample analysis. Deviations from the m/z's listed here must be documented by the laboratory and submitted with the data.*

TABLE 9. CHARACTERISTIC M/Z'S AND RESPONSE FACTORS OF ACID EXTRACTABLE COMPOUNDS

Compound	Labeled Analog	Primary m/z[1]	Response Factor[2]
benzoic acid		105	0.16
4-chloro-3-methylphenol	d_2	107 / 109	
2-chlorophenol	d_4	128 / 132	
p-cresol		108	0.61
3,5-dibromo-4-hydroxybenzonitrile		277	0.12
2,4-dichlorophenol	d_3	162 / 167	
2,6-dichlorophenol		162	0.42
2,4-dinitrophenol	d_3	184 / 187	
hexanoic acid		60	0.62
2-methyl-4,6-dinitrophenol	d_2	198 / 200	
2-nitrophenol	d_4	65 / 109	
4-nitrophenol	d_4	65 / 109	
pentachlorophenol	$^{13}C_6$	266 / 272	
2,3,4,6-tetrachlorophenol		232	0.17
2,3,6-trichlorophenol	d_2	196 / 200	
2,4,5-trichlorophenol	d_2	196 / 200	
2,4,6-trichlorophenol	d_2	196 / 200	

[1]Native / labeled.
[2]Referenced to 2,2'-difluorobiphenyl.

NOTE: *Because the composition and purity of commercially-supplied isotopically labeled standards may vary, the primary m/z of the labeled analogs given in this table should be used as guidance. The appropriate m/z of the labeled analogs should be determined prior to use for sample analysis. Deviations from the m/z's listed here must be documented by the laboratory and submitted with the data.*

7.4 Calibration with Isotope Dilution—Isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation m / z (Tables 8 and 9) extracted ion current profile (EICP) area for the compound is in the calibration range. Alternate labeled compounds and quantitation m / z's may be used based on availability. If any of the above conditions preclude isotope dilution, the internal standard method (Section 7.5) is used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d_5 as the isotopic diluent. Also shown are the ±10% error limits (dotted lines). Relative Response (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

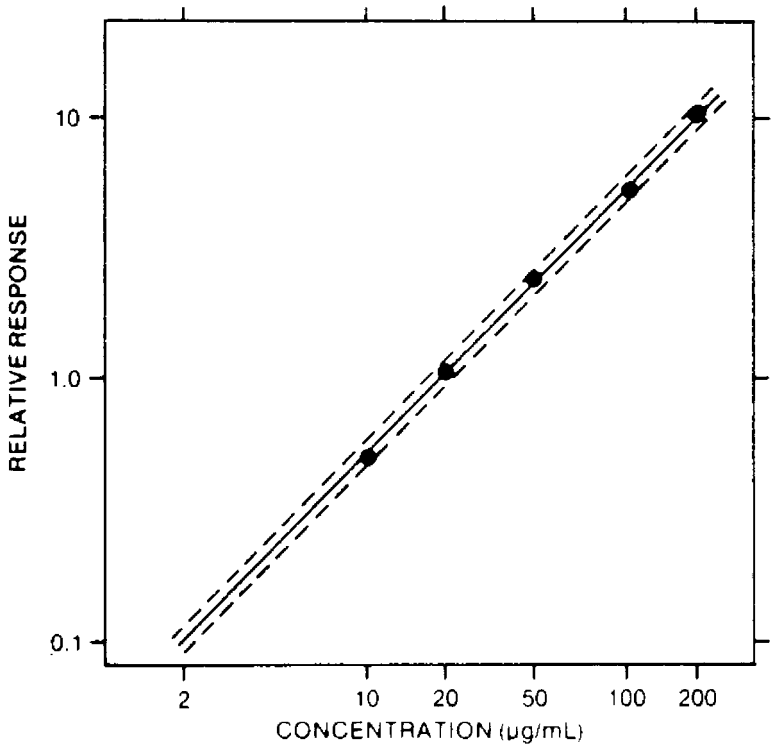

FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ± 10 Percent Error Window.

7.4.2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure pollutant.

R_y = the isotope ratio measured for the labeled compound.

R_m = the isotope ratio of an analytical mixture of pollutant and labeled compounds.

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by the internal standard method.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (Figure 2). For this case,

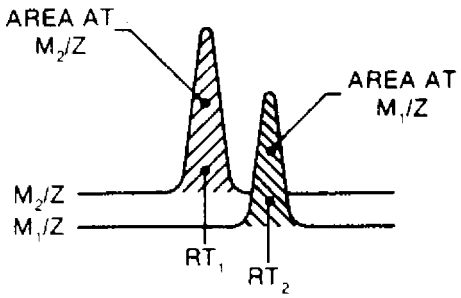

FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_2/z) and Unlabeled (m_1/z) Pairs.

$$R_x = \frac{[\text{area } m_1/z \text{ (at } RT_2)]}{1}$$

$$R_y = \frac{1}{[\text{area } m_2/z \text{ (at } RT_1)]}$$

$$R_m = \frac{[\text{area } m_1/z \text{ (at } RT_2)]}{[\text{area } m_2/z \text{ (at } RT_1)]}$$

as measured in the mixture of the pollutant and labeled compounds (Figure 2), and RR = R_m.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in Section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10% of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation:

$$RR = \frac{(R_y - R_m)(R_x + 1)}{(R_m - R_x)(R_y + 1)}$$

where,

R_x = Measured as shown in Figure 3A.
R_y = Measured as shown in Figure 3B.
R_m = Measured as shown in Figure 3C.

For the example,

$$R_x = \frac{46100}{4780} = 9.644$$

$$R_y = \frac{2650}{43600} = 0.06078$$

$$R_m = \frac{49200}{48300} = 1.019$$

$$RR = 1.115$$

The data from these analyses are reported to three significant figures (see Section 14.6). Therefore, in order to prevent rounding errors from affecting the values to be reported, all calculations performed prior to the final determination of concentrations should be carried out using at least four significant figures.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 µL aliquot of each of the calibration standards (Section 6.13) using the procedure in Section 11.0. Compute the RR at each concentration.

7.4.6 Linearity—If the ratio of relative response to concentration for any compound is constant (less than 20% coefficient of variation) over the five-point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

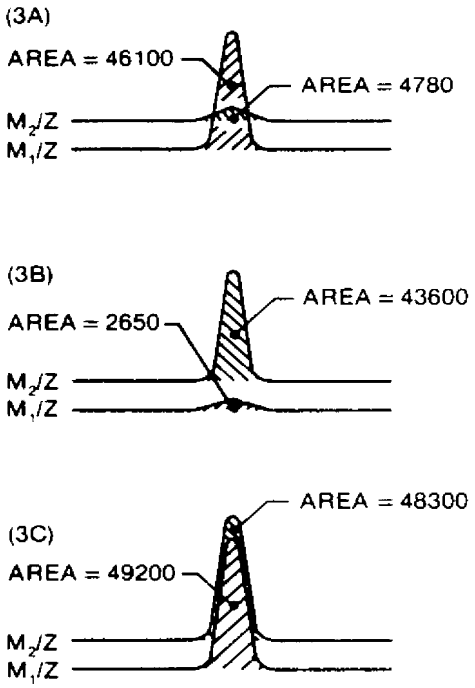

FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

7.5 Calibration by Internal Standard—Used when criteria for isotope dilution (Section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'-difluorobi-phenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (Sections 8.4 and 12.7.4).

7.5.1 Response factors—Calibration requires the determination of response factors (RF) which are defined by the following equation:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

where,

A_s = The area of the characteristic mass for the compound in the daily standard.
A_{is} = The area of the characteristric mass for the internal standard.
C_{is} = The concentration of the internal standard (µg/mL).
C_s = The concentration of the compound in the daily standard (µg/mL).

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (Section 6.13); nominally, 10, 20, 50, 100, and 200 µg/mL. The amount of internal standard added to each extract is the same (100 µg/mL) so that C_{is} remains constant. The RF is plotted vs concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.1.2 Linearity—If the response factor (RF) for any compound is constant (less than 35% coefficient of variation) over the five-point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five-point range.

7.6 Combined Calibration—By using calibration solutions (Section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 12.5) by analyzing the 100 µg/mL calibration standard (Section 6.13). Re-calibration is required only if calibration verification (Section 12.5) criteria cannot be met.

8.0 Quality Assurance/Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 7). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method. If the method is to be applied routinely to samples containing high solids with very little moisture (e.g., soils, filter cake, compost), the high solids reference matrix (Section 6.5.2) is substituted for the reagent water (Section 6.5.1) in all performance tests, and the high solids method (Section 10) is used for these tests.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in Section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (Section 15).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 6.14) that the analysis system is in control. These procedures are described in Sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Section 8.4.

8.2 Initial Precision and Accuracy—To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 For low solids (aqueous samples), extract, concentrate, and analyze two sets of four 1 L aliquots (eight aliquots total) of the precision and recovery standard (Section 6.14) according to the procedure in Section 10. For high solids samples, two sets of four 30 g aliquots of the high solids reference matrix are used.

8.2.2 Using results of the first set of four analyses, compute the average recovery (X) in µg/mL and the standard deviation of the recovery(s) in µg/mL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy in Table 10. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: *The large number of compounds in Table 10 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed.*

To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and X for only those compounds which failed the test of the first set of four analyses (Section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (Section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method beginning in Section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (Section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in Table 10. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample is complex. Water samples are diluted, and smaller amounts of soils, sludges, and sediments are reanalyzed per Section 15.

8.4 As part of the QA program for the laboratory, method accuracy for samples shall be assessed and records shall be maintained. After the analysis of five samples of a given matrix type (water, soil, sludge, sediment) for which the labeled compounds pass the tests in Section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from P - $2s_p$ to P + $2s_p$ for each matrix.

For example: If P = 90% and s_p = 10% for five analyses of compost, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each compound in each matrix on a regular basis (e.g., after each 5-10 new accuracy measurements).

TABLE 10. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Labeled and native compound initial precision and accuracy (Section 8.2.3) (µg/L)		Labeled compound recovery (Sections 8.3 and 14.2)	Calibration verification (Section 12.5)	Labeled and native compound ongoing accuracy (Section 12.7)
		s	X	P (%)	(µg/L)	R (µg/L)
301	acenaphthene	21	79–134		80–125	72–144
201	acenaphthene-d_{10}	38	38–147	20–270	71–141	30–180
377	acenaphthylene	38	69–186		60–166	61–207
277	acenaphthylene-d_8	31	39–146	23–239	66–152	33–168
378	anthracene	41	58–174		60–168	50–199
278	anthracene-d_{10}	49	31–194	14–419	58–171	23–242
305	benzidine	119	16–518		34–296	11–672
205	benzidine-d_8	269	ns[2]–ns	ns–ns	ns–ns	ns–ns
372	benzo(a)anthracene	20	65–168		70–142	62–176
272	benzo(a)anthracene-d_{12}	41	25–298	12–605	28–357	22–329
374	benzo(b)fluoranthene	183	32–545		61–164	20–ns
274	benzo(b)fluoranthene-d_{12}	168	11–577	ns–ns	14–ns	ns–ns
375	benzo(k)fluoranthene	26	59–143		13–ns	53–155
275	benzo(k)fluoranthene-d_{12}	114	15–514	ns–ns	13–ns	ns–685
373	benzo(a)pyrene	26	62–195		78–129	59–206
273	benzo(a)pyrene-d_{12}	24	35–181	21–290	12–ns	32–194
379	benzo(ghi)perylene	21	72–160		69–145	58–168
279	benzo(ghi)perylene-d_{12}	45	29–268	14–529	13–ns	25–303
712	biphenyl (Appendix C)	41	75–148		58–171	62–176
612	biphenyl-d_{10}	43	28–165	ns–ns	52–192	17–267
318	bis(2-chloroethyl)ether	34	55–196		61–164	50–213
218	bis(2-chloroethyl) ether d_8	33	29–196	15–372	52–194	25–222
343	bis(2-chloroethoxy) methane	27	43–153		44–228	39–166

TABLE 10. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Labeled and native compound initial precision and accuracy (Section 8.2.3) (µg/L) s	X	Labeled compound recovery (Sections 8.3 and 14.2) P (%)	Calibration verification (Section 12.5) (µg/L)	Labeled and native compound ongoing accuracy (Section 12.7) R (µg/L)
243	bis(2-chloroethoxy) methane[3]	33	29–196	15–372	52–194	25–222
342	bis(2-chloroisopropyl) ether	17	81–138		67–148	77–145
242	bis(2-chloroisopropyl) ether-d_{12}	27	35–149	20–260	44–229	30–169
366	bis(2-ethylhexyl) phthalate	31	69–220		76–131	64–232
266	bis(2-ethylhexyl) phthalate-d_4	29	32–205	18–364	43–232	28–224
341	4-bromophenyl phenyl ether	44	44–140		52–193	35–172
241	4-bromophenylphenyl ether-d_5[3]	52	40–161	19–325	57–175	29–212
367	butyl benzyl phthalate	31	19–233		22–450	35–170
267	butyl benzyl phthalate-d_4[3]	29	32–205	18–364	43–232	28–224
717	n-C10 (Appendix C)	51	24–195		42–235	19–237
617	n-C10-d_{22}	70	ns–298	ns–ns	44–227	ns–504
706	n-C12 (Appendix C)	74	35–369		60–166	29–424
606	n-C12-d_{26}	53	ns–331	ns–ns	41–242	ns–408
518	n-C14 (Appendix C)[3]	109	ns–ns		37–268	ns–ns
719	n-C16 (Appendix C)	33	80–162		72–138	71–181
619	n-C16-d_{34}	46	37–162	18–308	54–186	28–202
520	n-C18 (Appendix C)[3]	39	42–131		40–249	35–167
721	n-C20 (Appendix C)	59	53–263		54–184	46–301
621	n-C20-d_{42}	34	34–172	19–306	62–162	29–198
522	n-C22 (Appendix C)[3]	31	45–152		40–249	39–195
723	n-C24 (Appendix C)	11	80–139		65–154	78–142
623	n-C24-d_{50}	28	27–211	15–376	50–199	25–229
524	n-C26 (Appendix C)[3]	35	35–193		26–392	31–212
525	n-C28 (Appendix C)[3]	35	35–193		26–392	31–212
726	n-C30 (Appendix C)	32	61–200		66–152	56–215
626	n-C30-d_{62}	41	27–242	13–479	24–423	23–274
728	carbazole (4c)	38	36–165		44–227	31–188
628	carbazole-d_8[3]	31	48–130	29–215	69–145	40–156
320	2-chloronaphthalene	100	46–357		58–171	35–442
220	2-chloronaphthalene-d_7	41	30–168	15–324	72–139	24–204
322	4-chloro-3-methylphenol	37	76–131		85–115	62–159
222	4-chloro-3-methylphenol-d_2	111	30–174	ns–613	68–147	14–314
324	2-chlorophenol	13	79–135		78–129	76–138
224	2-chlorophenol-d_4	24	36–162	23–255	55–180	33–176
340	4-chlorophenyl phenyl ether	42	75–166		71–142	63–194

TABLE 10. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Labeled and native compound initial precision and accuracy (Section 8.2.3) (µg/L) s	X	Labeled compound recovery (Sections 8.3 and 14.2) P (%)	Calibration verification (Section 12.5) (µg/L)	Labeled and native compound ongoing accuracy (Section 12.7) R (µg/L)
240	4-chlorophenyl phenyl ether-d_5	52	40–161	19–325	57–175	29–212
376	chrysene	51	59–186		70–142	48–221
276	chrysene-d_{12}	69	33–219	13–512	24–411	23–290
713	p-cymene (Appendix C)	18	76–140		79–127	72–147
613	p-cymene-d_{14}	67	ns–359	ns–ns	66–152	ns–468
382	dibenzo(a,h)anthracene	55	23–299		13–761	19–340
282	dibenzo(a,h) anthracene-d_{14}[3]	45	29–268	14–529	13–ns	25–303
705	dibenzofuran (Appendix C)	20	85–136		73–136	79–146
605	dibenzofuran-d_8	31	47–136	28–220	66–150	39–160
704	dibenzothiophene (Synfuel)	31	79–150		72–140	70–168
604	dibenzothiophene-d_8	31	48–130	29–215	69–145	40–156
368	di-n-butyl phthalate	15	76–165		71–142	74–169
268	di-n-butyl phthalate-d_4	23	23–195	13–346	52–192	22–209
325	1,2-dichlorobenzene	17	73–146		74–135	70–152
225	1,2-dichlorobenzene-d_4	35	14–212	ns–494	61–164	11–247
326	1,3-dichlorobenzene	43	63–201		65–154	55–225
226	1,3-dichlorobenzene-d_4	48	13–203	ns–550	52–192	ns–260
327	1,4-dichlorobenzene	42	61–194		62–161	53–219
227	1,4-dichlorobenzene-d_4	48	15–193	ns–474	65–153	11–245
328	3,3'-dichlorobenzidine	26	68–174		77–130	64–185
228	3,3'-dichlorobenzidine-d_6	80	ns–562	ns–ns	18–558	ns–ns
331	2,4-dichlorophenol	12	85–131		67–149	83–135
231	2,4-dichlorophenol-d_3	28	38–164	24–260	64–157	34–182
370	diethyl phthalate	44	75–196		74–135	65–222
270	diethyl phthalate-d_4	78	ns–260	ns–ns	47–211	ns–ns
334	2,4-dimethylphenol	13	62–153		67–150	60–156
234	2,4-dimethylphenol-d_3	22	15–228	ns–449	58–172	14–242
371	dimethyl phthalate	36	74–188		73–137	67–207
271	dimethyl phthalate-d_4	108	ns–640	ns–ns	50–201	ns–ns
359	2,4-dinitrophenol	18	72–134		75–133	68–141
259	2,4-dinitrophenol-d_3	66	22–308	ns–ns	39–256	17–378
335	2,4-dinitrotoluene	18	75–158		79–127	72–164
235	2,4-dinitrotoluene-d_3	37	22–245	10–514	53–187	19–275
336	2,6-dinitrotoluene	30	80–141		55–183	70–159
236	2,6-dinitrotoluene-d_3	59	44–184	17–442	36–278	31–250
369	di-n-octyl phthalate	16	77–161		71–140	74–166
269	di-n-octyl phthalate-d_4	46	12–383	ns–ns	21–467	10–433
707	diphenylamine (Appendix C)	45	58–205		57–176	51–231
607	diphenylamine-d_{10}	42	27–206	11–488	59–169	21–249

TABLE 10. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Labeled and native compound initial precision and accuracy (Section 8.2.3) (µg/L) s	X	Labeled compound recovery (Sections 8.3 and 14.2) P (%)	Calibration verification (Section 12.5) (µg/L)	Labeled and native compound ongoing accuracy (Section 12.7) R (µg/L)
708	diphenyl ether (Appendix C)	19	82–136		83–120	77–144
608	diphenyl ether-d_{10}	37	36–155	19–281	77–129	29–186
337	1,2-diphenylhydrazine	73	49–308		75–134	40–360
237	1,2-diphenylhy-drazine-d_{10}	35	31–173	17–316	58–174	26–200
339	fluoranthene	33	71–177		67–149	64–194
239	fluoranthene-d_{10}	35	36–161	20–278	47–215	30–187
380	fluorene	29	81–132		74–135	70–151
280	fluorene-d_{10}	43	51–131	27–238	61–164	38–172
309	hexachlorobenzene	16	90–124		78–128	85–132
209	hexachlorobenzene-$^{13}C_6$	81	36–228	13–595	38–265	23–321
352	hexachlorobutadiene	56	51–251		74–135	43–287
252	hexachlorobutadiene-$^{13}C_4$	63	ns–316	ns–ns	68–148	ns–413
312	hexachloroethane	227	21–ns		71–141	13–ns
212	hexachloroethane-^{13}C	77	ns–400	ns–ns	47–212	ns–563
353	hexachlorocyclo-pentadiene	15	69–144		77–129	67–148
253	hexachlorocyclo-pentadiene-$^{13}C_4$	60	ns–ns	ns–ns	47–211	ns–ns
083	ideno(1,2,3-cd)pyrene[3]	55	23–299		13–761	19–340
354	isophorone	25	76–156		70–142	70–168
254	isophorone-d_8	23	49–133	33–193	52–194	44–147
360	2-methyl-4,6-dinitrophenol	19	77–133		69–145	72–142
260	2-methyl-4,6-dinitrophenol-d_2	64	36–247	16–527	56–177	28–307
355	naphthalene	20	80–139		73–137	75–149
255	naphthalene-d_8	39	28–157	14–305	71–141	22–192
702	beta-naphthylamine (Appendix C)	49	10–ns		39–256	ns–ns
602	beta-naphthylamine-d_7	33	ns–ns	ns–ns	44–230	ns–ns
356	nitrobenzene	25	69–161		85–115	65–169
256	nitrobenzene-d_5	28	18–265	ns–ns	46–219	15–314
357	2-nitrophenol	15	78–140		77–129	75–145
257	2-nitrophenol-d_4	23	41–145	27–217	61–163	37–158
358	4-nitrophenol	42	62–146		55–183	51–175
258	4-nitrophenol-d_4	188	14–398	ns–ns	35–287	ns–ns
361	N-nitrosodimethylamine	49	10–ns		39–256	ns–ns
261	N-nitrosodimethyl-amine-d_6[3]	33	ns–ns	ns–ns	44–230	ns–ns
363	N-nitrosodi-n-propylamine	45	65–142		68–148	53–173
263	N-nitrosodi-n-propylamine[3]	37	54–126	26–256	59–170	40–166
362	N-nitrosodiphenylamine	45	65–142		68–148	53–173

TABLE 10. ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Labeled and native compound initial precision and accuracy (Section 8.2.3) (µg/L) s	X	Labeled compound recovery (Sections 8.3 and 14.2) P (%)	Calibration verification (Section 12.5) (µg/L)	Labeled and native compound ongoing accuracy (Section 12.7) R (µg/L)
262	N-nitrosodiphenyl-amine-d_6	37	54–126	26–256	59–170	40–166
364	pentachlorophenol	21	76–140		77–130	71–150
264	pentachlorophenol-$^{13}C_6$	49	37–212	18–412	42–237	29–254
381	phenanthrene	13	93–119		75–133	87–126
281	phenanthrene-d_{10}	40	45–130	24–241	67–149	34–168
365	phenol	36	77–127		65–155	62–154
265	phenol-d_5	161	21–210	ns–ns	48–208	ns–ns
703	alpha-picoline (Synfuel)	38	59–149		60–165	50–174
603	alpha-picoline-d_7	138	11–380	ns–ns	31–324	ns–608
384	pyrene	19	76–152		76–132	72–159
284	pyrene-d_{10}	29	32–176	18–303	48–210	28–196
710	styrene (Appendix C)	42	53–221		65–153	48–244
610	styrene-d_5	49	ns–281	ns–ns	44–228	ns–348
709	alpha-terpineol (Appendix C)	44	42–234		54–186	38–258
609	alpha-terpineol-d_3	48	22–292	ns–672	20–502	18–339
729	1,2,3-trichloro-benzene (4c)	69	15–229		60–167	11–297
629	1,2,3-trichloro-benzene-d_3[3]	57	15–212	ns–592	61–163	10–282
308	1,2,4-trichlorobenzene	19	82–136		78–128	77–144
208	1,2,4-trichlorobenzene-d_3	57	15–212	ns–592	61–163	10–282
530	2,3,6-trichloro-phenol (4c)[3]	30	58–137		56–180	51–153
731	2,4,5-trichlorophenol (4c)	30	58–137		56–180	51–153
631	2,4,5-trichlorophenol-d_2[3]	47	43–183	21–363	69–144	34–226
321	2,4,6-trichlorophenol	57	59–205		81–123	48–244
221	2,4,6-trichlorophenol-d_2	47	43–183	21–363	69–144	34–226

[1]Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2]ns = no specification: limit is outside the range that can be measured reliably.
[3]This compound is to be determined by internal standard; specification is derived from related compound.

8.5 Blanks—Reagent water and high solids reference matrix blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a 1 L reagent water blank or a high solids reference matrix blank with each sample lot (samples started through the extraction process

on the same eight hour shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (Section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (Tables 1–4) or any potentially interfering compound is found in an aqueous blank at greater than 10 µg/L, or in a high solids reference matrix blank at greater than 100 µg/kg (assuming a response factor of 1 relative to the internal standard for compounds not listed in Tables 1-4), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (Section 7), calibration verification (Section 12.5), and for initial (Section 8.2) and on-going (Section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of semivolatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal standard method is used.

9.0 Sample Collection, Preservation, and Handling

9.1 Collect samples in glass containers following conventional sampling practices (Reference 8). Aqueous samples which flow freely are collected in refrigerated bottles using automatic sampling equipment. Solid samples are collected as grab samples using wide mouth jars.

9.2 Maintain samples at 0-4°C from the time of collection until extraction. If residual chlorine is present in aqueous samples, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine (Reference 9).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10.0 Sample Extraction, Concentration, and Cleanup

Samples containing 1% solids or less are extracted directly using continuous liquid/liquid extraction techniques (Section 10.2.1 and Figure 4). Samples containing 1-30% solids are diluted to the 1% level with reagent water (Section 10.2.2) and extracted using continuous liquid/liquid extraction techniques. Samples containing greater than 30% solids are extracted using ultrasonic techniques (Section 10.2.5)

10.1 Determination of Percent Solids

10.1.1 Weigh 5-10 g of sample into a tared beaker.

10.1.2 Dry overnight (12 hours minimum) at 110 ±5°C, and cool in a desiccator.

10.1.3 Determine percent solids as follows:

$$\% \text{ solids} = \frac{\text{weight of dry sample}}{\text{weight of wet sample}} \times 100$$

10.2 Preparation of Samples for Extraction

10.2.1 Samples containing 1% solids or less—Extract sample directly using continuous liquid/liquid extraction techniques.

10.2.1.1 Measure 1.00 ±0.01 L of sample into a clean 1.5-2.0 L beaker.

10.2.1.2 Dilute aliquot—For samples which are expected to be difficult to extract, concentrate, or clean-up, measure an additional 100.0 ±1.0 mL into a clean 1.5-2.0 L beaker and dilute to a final volume of 1.00 ±0.1 L with reagent water.

10.2.1.3 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into the sample aliquots. Proceed to preparation of the QC aliquots for low solids samples (Section 10.2.3).

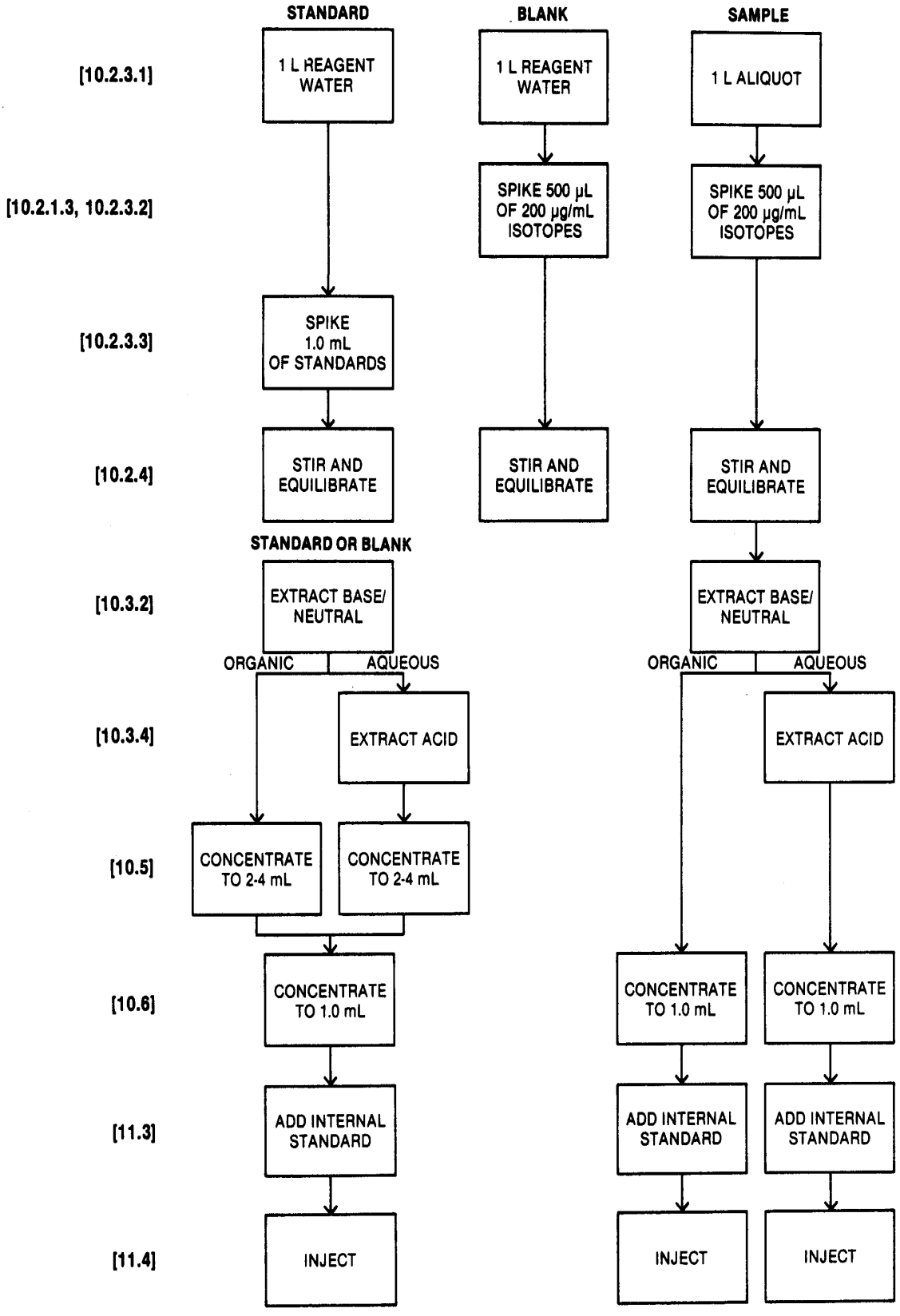

FIGURE 4 Flow Chart for Extraction/Concentration of Low Solids Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

10.2.2 Samples containing 1-30% solids

10.2.2.1 Mix sample thoroughly.

10.2.2.2 Using the percent solids found in Section 10.1.3, determine the weight of sample required to produce 1 L of solution containing 1% solids as follows:

$$\text{sample weight, (grams)} = \frac{1000}{\%\ \text{solids}}$$

10.2.2.3 Discard all sticks, rocks, leaves and other foreign material prior to weighing. Place the weight determined in Section 10.2.2.2 in a clean 1.5-2.0 L beaker.

10.2.2.4 Dilute aliquot—For samples which are expected to be difficult to extract, concentrate, or clean up, weigh an amount of sample equal to one-tenth the amount determined in Section 10.2.2.2 into a second clean 1.5-2.0 L beaker. When diluted to 1.0 L, this dilute aliquot will contain 0.1% solids.

10.2.2.5 Bring the sample aliquot(s) above to 100-200 mL volume with reagent water.

10.2.2.6 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into each sample aliquot.

10.2.2.7 Using a clean metal spatula, break any solid portions of the sample into small pieces.

10.2.2.8 Place the 3/4 inch horn on the ultrasonic probe approx ½ inch below the surface of each sample aliquot and pulse at 50% for three minutes at full power. If necessary, remove the probe from the solution and break any large pieces using the metal spatula or a stirring rod and repeat the sonication. Clean the probe with methylene chloride:acetone (1:1) between samples to preclude cross-contamination.

10.2.2.9 Bring the sample volume to 1.0 ±0.1 L with reagent water.

10.2.3 Preparation of QC aliquots for samples containing low solids (<30%).

10.2.3.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.0 ±0.01 L aliquots of reagent water in clean 1.5-2.0 L beakers.

10.2.3.2 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into one reagent water aliquot. This aliquot will serve as the blank.

10.2.3.3 Spike 1.0 mL of the precision and recovery standard (Section 6.14) into the two remaining reagent water aliquots.

10.2.4 Stir and equilibrate all sample and QC solutions for one to two hours. Extract the samples and QC aliquots per Section 10.3.

10.2.5 Samples containing 30% solids or greater

10.2.5.1 Mix the sample thoroughly.

10.2.5.2 Discard all sticks, rocks, leaves and other foreign material prior to weighing. Weigh 30 ±0.3 g into a clean 400-500 mL beaker.

10.2.5.3 Dilute aliquot—For samples which are expected to be difficult to extract, concentrate, or clean-up, weigh 3 ±0.03 g into a clean 400-500 mL beaker.

10.2.5.4 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into each sample aliquot.

10.2.5.5 QC aliquots—For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 30 ±0.3 g aliquots of the high solids reference matrix in clean 400-500 mL beakers.

10.2.5.6 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into one high solids reference matrix aliquot. This aliquot will serve as the blank.

10.2.5.7 Spike 1.0 mL of the precision and recovery standard (Section 6.14) into the two remaining high solids reference matrix aliquots. Extract, concentrate, and clean up the high solids samples and QC aliquots per Sections 10.4 through 10.8.

10.3 Continuous Extraction of Low Solids (Aqueous) Samples—Place 100-150 mL methylene chloride in each continuous extractor and 200-300 mL in each distilling flask.

10.3.1 Pour the sample(s), blank, and QC aliquots into the extractors. Rinse the glass containers with 50-100 mL methylene chloride and add to the respective extractors. Include all solids in the extraction process.

10.3.2 Base/neutral extraction—Adjust the pH of the waters in the extractors to 12-13 with 6 N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, one to two drops of methylene chloride per second will fall from the condensor tip into the water. Test and adjust the pH of the waters during the first to second hour and during the fifth to tenth hour of extraction. Extract for 24-48 hours.

10.3.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7-10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30-50 mL of methylene chloride and pour through the drying column. Collect the solution in

a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per Sections 10.5 through 10.6.

10.3.4 Acid extraction—Adjust the pH of the waters in the extractors to 2 or less using 6 N sulfuric acid. Charge clean distilling flasks with 300-400 mL of methylene chloride. Test and adjust the pH of the waters during the first one to two hours and during the fifth to tenth hour of extraction. Extract for 24-48 hours. Repeat Section 10.3.3, except label as the acid fraction.

10.4 Ultrasonic Extraction of High Solids Samples

10.4.1 Add 60 g of anhydrous sodium sulfate the sample and QC aliquot(s) (Section 10.2.5) and mix thoroughly.

10.4.2 Add 100 ±10 mL of acetone:methylene chloride (1:1) to the sample and mix thoroughly.

10.4.3 Place the 3/4 in. horn on the ultrasonic probe approx ½ in. below the surface of the solvent but above the solids layer and pulse at 50% for three minutes at full power. If necessary, remove the probe from the solution and break any large pieces using the metal spatula or a stirring rod and repeat the sonication.

10.4.4 Decant the extracts through Whatman 41 filter paper using glass funnels and collect in 500-1000 mL graduated cylinders.

10.4.5 Repeat the extraction steps (Sections 10.4.2 through 10.4.4) twice more for each sample and QC aliquot. On the final extraction, swirl the sample or QC aliquot, pour into its respective glass funnel, and rinse with acetone:methylene chloride. Record the total extract volume.

10.4.6 Pour each extract through a drying column containing 7-10 cm of anhydrous sodium sulfate. Rinse the graduated cylinder with 30-50 mL of methylene chloride and pour through the drying column. Collect each extract in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube. Seal and label as the high solids semivolatile fraction. Concentrate and clean up the samples and QC aliquots per Sections 10.5 through 10.8.

10.5 Macro Concentration—Concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes.

10.5.1 Add one to two clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approx 1 mL of methylene chloride through the top. Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the

concentrator tube with 1-2 mL of methylene chloride. A 5 mL syringe is recommended for this operation.

10.5.2 For performance standards (Sections 8.2 and 12.7) and for blanks (Section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for aqueous samples.

10.6 Micro Concentration

10.6.1 Kuderna-Danish (K-D)—Add a clean boiling chip and attach a two-ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0.2 mL of methylene chloride. Adjust the final volume to 5.0 mL if the extract is to be cleaned up by GPC, to 1.0 mL if it does not require clean-up, or to 0.5 mL if it has been cleaned up.

10.6.2 Nitrogen blowdown—Place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: *New plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.*

The internal wall of the tube must be rinsed down several times with methylene chloride during the operation. During evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry. Adjust the final volume to 5.0 mL if the extract is to be cleaned up by GPC, to 1.0 mL if it does not require clean-up, or to 0.5 mL if it has been cleaned up.

10.7 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at -20 to -10°C until ready for analysis.

10.8 GPC Setup and Calibration

10.8.1 Column packing

10.8.1.1 Place 75 ±5 g of SX-3 Bio-beads in a 400-500 mL beaker.

10.8.1.2 Cover the beads and allow to swell overnight (12 hours minimum).

10.8.1.3 Transfer the swelled beads to the column and pump solvent through the column, from bottom to top, at 4.5-5.5 mL/min prior to connecting the column to the detector.

10.8.1.4 After purging the column with solvent for one to two hours, adjust the column head pressure to 7-10 psig, and purge for four to five hours to remove air from the column. Maintain a head pressure of 7-10 psig. Connect the column to the detector.

10.8.2 Column calibration

10.8.2.1 Load 5 mL of the calibration solution (Section 6.4) into the sample loop.

10.8.2.2 Inject the calibration solution and record the signal from the detector. The elution pattern will be corn oil, bis(2-ethylhexyl) phthalate, pentachlorophenol, perylene, and sulfur.

10.8.2.3 Set the "dump time" to allow >85% removal of the corn oil and >85% collection of the phthalate.

10.8.2.4 Set the "collect time" to the peak minimum between perylene and sulfur.

10.8.2.5 Verify the calibration with the calibration solution after every 20 extracts. Calibration is verified if the recovery of the pentachlorophenol is greater than 85%. If calibration is not verified, the system shall be recalibrated using the calibration solution, and the previous 20 samples shall be re-extracted and cleaned up using the calibrated GPC system.

10.9 Extract Cleanup

10.9.1 Filter the extract or load through the filter holder to remove particulates. Load the 5.0 mL extract onto the column. The maximum capacity of the column is 0.5-1.0 g. If necessary, split the extract into multiple aliquots to prevent column overload.

10.9.2 Elute the extract using the calibration data determined in Section 10.8.2. Collect the eluate in a clean 400-500 mL beaker.

10.9.3 Concentrate the cleaned up extract to 5.0 mL per Section 10.5.

10.9.4 Rinse the sample loading tube thoroughly with methylene chloride between extracts to prepare for the next sample.

10.9.5 If a particularly dirty extract is encountered, a 5.0 mL methylene chloride blank shall be run through the system to check for carry-over.

10.9.6 Concentrate the extract to 0.5 mL and transfer to a screw-cap vial per Sections 10.6 and 10.7. Concentrating extracts cleaned up by GPC to 0.5 mL will place the analytes in the same part of the GCMS calibration range as in samples not subjected to GPC.

11.0 GCMS Analysis

11.1 Establish the operating conditions given in Tables 5 or 6 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (Sections 10.5.2 and 10.9.6), use the operating conditions in Table 5.

11.2 Bring the concentrated extract (Section 10.7) or standard (Sections 6.13 through 6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (Sections 6.6 and 10.7) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (Section 6.10) to the extract (use 1.0 µL of solution per 0.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.0 µL. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo(ghi)perylene or pentachlorophenol peak elutes for the base/neutral (or semi-volatile) or acid fraction, respectively. Return the column to the initial temperature for analysis of the next sample.

12.0 System and Laboratory Performance

12.1 At the beginning of each 8 hr shift during which analyses are performed, GCMS system performance and calibration are verified for all pollutants and labeled compounds. For these tests, analysis of the 100 µg/mL calibration standard (Section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP Spectrum Validity—Inject 1 µL of the DFTPP solution (Section 6.11) either separately or within a few seconds of injection of the standard (Section 12.1) analyzed at the beginning of each shift. The criteria in Table 7 shall be met.

12.3 Retention Times—The absolute retention time of 2,2'-difluorobiphenyl shall be within the range of 1078-1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in Tables 5 and 6.

12.4 GC Resolution—The valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Calibration Verification—Compute the concentration of each pollutant (Tables 1 and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in Section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in Table 10. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound

fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (Section 12.1), or recalibrate (Section 7).

12.6 Multiple Peaks—Each compound injected shall give a single, distinct GC peak.

12.7 On-going Precision and Accuracy

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (Section 10) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (Tables 1 and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in Table 10. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound.

NOTE: *The large number of compounds in Table 10 present a substantial probability that one or more will fail when all compounds are analyzed.*

To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standards (Section 10).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (Section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (Section 10) and repeat the on-going precision and recovery test (Section 12.7).

12.7.4 Add results which pass the specifications in Section 12.7.3 to initial and previous on-going data for each compound in each matrix. Update QC charts to form a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound in each matrix type by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from $R-2s_r$ to $R+2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85-105%.

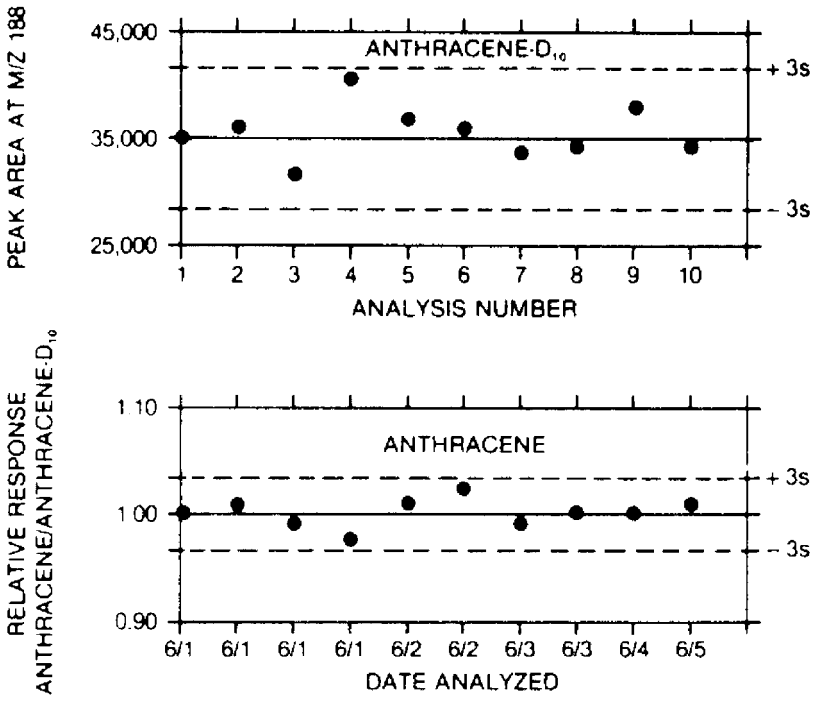

FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-d_{10} (lower graph) Plotted as a Function of Time or Analysis Number.

13.0 Qualitative Determination

Identification is accomplished by comparison of data from analysis of a sample or blank with data stored in the mass spectral libraries. For compounds for which the relative retention times and mass spectra are known, identification is confirmed per Sections 13.1 and 13.2. For unidentified GC peaks, the spectrum is compared to spectra in the EPA/NIH mass spectral file per Section 13.3.

13.1 Labeled Compounds and Pollutants Having No Labeled Analog (Tables 1-4)

13.1.1 The signals for all characteristic m/z's stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.1.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (one-half to two times) for all masses stored in the library.

13.1.3 For the compounds for which the system has been calibrated (Tables 1 and 2), the retention time shall be within the windows specified in Tables 5 and 6, or within

±15 scans or ±15 seconds (whichever is greater) for compounds for which no window is specified.

13.1.4 The system has not been calibrated for the compounds listed in Tables 3 and 4, however, the relative retention times and mass spectra of these compounds are known. Therefore, for a compound in Table 3 or 4 to be identified, its retention time relative to the internal standard 2,2'-difluorobiphenyl must fall within a retention time window of ±30 seconds, or ±30 scans (whichever is greater) of the nominal retention time of the compound specified in Table 5 or 6.

13.2 Pollutants Having a Labeled Analog (Tables 1 and 2)

13.2.1 The signals for all characteristic m/z's stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.2.3 The relative retention time between the pollutant and its labeled analog shall be within the windows specified in Tables 5 and 6.

13.3 Unidentified GC Peaks

13.3.1 The signals for masses specific to a GC peak shall all maximize within ±1 scan.

13.3.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two with the masses stored in the EPA/NIH Mass Spectral File.

13.4 The m/z's present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, or if identification is ambiguous, an experienced spectrometrist (Section 1.4) is to determine the presence or absence of the compound.

14.0 Quantitative Determination

14.1 Isotope Dilution—Because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography, correction for recovery of the pollutant can be made by adding a known amount of a labeled compound to every sample prior to extraction. Relative response (RR) values for sample mixtures are used in conjunction with the calibration curves described in Section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenol example given in Figure 1 (Section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in Figure 1 indicates a concentration of 27 µg/mL in the sample extract (C_{ex}).

14.2 Internal Standard—Compute the concentration in the extract using the response factor determined from calibration data (Section 7.5) and the following equation:

$$C_{ex}\ (\mu g/mL) = \frac{(A_s \times C_{is})}{(A_{is} \times RF)}$$

where,

C_{ex} = The concentration of the compound in the extract, and the other terms are as defined in Section 7.5.1.

14.3 The concentration of the pollutant in the solid phase of the sample is computed using the concentration of the pollutant in the extract and the weight of the solids (Section 10), as follows:

$$\text{Concentration in solid } (\mu g/kg) = \frac{(C_{ex} \times V_{ex})}{W_s}$$

where,

V_{ex} = The extract volume in mL, and W_s is the sample weight in kg.

14.4 Dilution of Samples—If the EICP area at the quantitation m/z for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (Section 10) is analyzed by isotope dilution. For water samples, where the base/neutral and acid extracts are not combined, re-analysis is only required for the extract (B/N or A) in which the compound exceeds the calibration range. If further dilution is required and the sample holding time has not been exceeded, a smaller sample aliquot is extracted per Sections 14.4.1 through 14.4.3. If the sample holding time has been exceeded, the sample extract is diluted by successive factors of 10, internal standard is added to give a concentration of 100 µg/mL in the diluted extract, and the diluted extract is analyzed by the internal standard method.

14.4.1 For samples containing one percent solids or less for which the holding time has not been exceeded, dilute 10 mL, 1.0 mL, 0.1 mL etc. of sample to one liter with reagent water and extract per Section 10.2.1.

14.4.2 For samples containing 1-30% solids for which the holding time has not been exceeded, extract an amount of sample equal to 1/100 the amount determined in Section 10.2.2.2. Extract per Section 10.2.2.

14.4.3 For samples containing 30% solids or greater for which the holding time has not been exceeded, extract 0.30 ±0.003 g of sample per Section 10.2.5.

14.5 Dilution of samples containing high concentrations of compounds to be identified per Section 13.3—When the EICP area of the quantitation m/z of a compound to be identified per Section 13.3 exceeds the linear range of the GCMS system, or when any peak is saturated, dilute the sample per Sections 14.4.1 through 14.4.3.

14.6 Results are reported to three significant figures for all pollutants, labeled compounds, and tentatively identified compounds found in all standards, blanks, and samples. For aqueous samples, the units are µg/L, and for samples containing one percent solids or greater (soils, sediments, filter cake, compost), the units are µg/kg, based on the dry weight of the solids.

14.6.1 Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 14.4), or at which no m/z in the spectrum is saturated (Section 14.5). For compounds having a labeled analog, results are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 14.4) and the labeled compound recovery is within the normal range for the method (Section 15.4).

15.0 Analysis of Complex Samples

15.1 Some samples may contain high levels (>1000 µg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to 1 mL (Section 10.6); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (Section 10) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (Section 9.3) has not been exceeded, dilute an aliquot of an aqueous sample with reagent water, or weigh a dilute aliquot of a high solids sample and re-extract (Section 10); otherwise, dilute the extract (Section 14.4) and analyze by the internal standard method (Section 14.2).

15.3 Recovery of Internal Standard—The EICP area of the internal standard should be within a factor of two of the area in the shift standard (Section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then loss of the internal standard in the extract has occurred. In this case, use one of the labeled compounds (preferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of Labeled Compounds—In most samples, labeled compound recoveries will be similar to those from reagent water or from the high solids reference matrix (Section 12.7). If the labeled compound recovery is outside the limits given in Table 10, the extract from the dilute aliquot (Section 10) is analyzed as in Section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 µg/mL calibration standard (Section 12.1) shall be analyzed and calibration verified (Section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in Section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not apply to the sample being analyzed, and the result may not be reported for regulatory compliance purposes.

16.0 Method Performance

16.1 Interlaboratory performance for this method is detailed in Reference 10. Reference mass spectra, retention times, and response factors are from References 11 and 12. Results of initial tests of this method on municipal sludge can be found in Reference 13.

16.2 A chromatogram of the 100 µg/mL acid/base/neutral calibration standard (Section 6.13) is shown in Figure 6.

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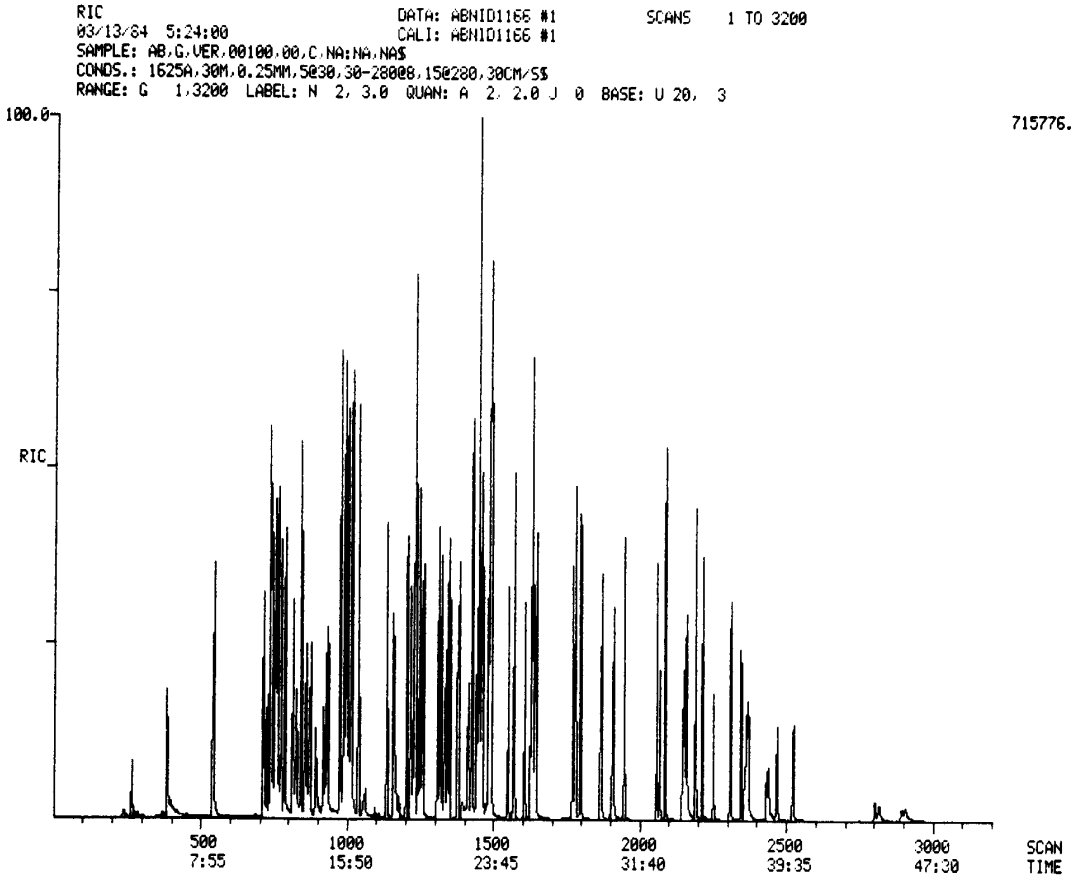

FIGURE 6 Chromatogram of Combined Acid/Base/Neutral Standard.

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

555 acetophenone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	21	43	245	49	19	50	221	51	524	52	75
61	13	62	26	63	422	65	31	73	13	74	64
75	36	76	62	77	941	78	11	89	12	91	22
105	1000	106	87	120	479	121	38				

556 4-aminobiphenyl

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	55	63	65	72	82	83	73	85	163	115	142
139	65	141	132	167	163	168	280	169	1000	170	216

557 aniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	65	41	66	42	16	46	11	47	75	50	40
51	47	52	54	53	12	54	40	61	17	62	28
63	59	64	33	65	226	66	461	74	11	78	14
91	10	92	136	93	1000	94	73				

558 o-anisidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	22	41	43	42	10	50	60	51	106	52	202
53	286	54	39	61	12	62	25	63	43	64	24
65	142	66	20	76	13	77	36	68	32	79	25
80	915	81	41	92	47	93	14	94	18	105	18
108	1000	109	55	122	123	844	124	56			

559 aramite

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	606	57	758	59	328	63	782	65	285	74	113
77	155	91	339	105	153	107	239	121	107	123	120
163	143	175	182	185	1000	187	328	191	346	197	191
319	270	334	137								

560 benzanthrone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	69	75	71	87	97	88	160	99	69	100	215
101	278	150	58	174	67	199	63	200	350	201	236
202	762	203	126	230	1000	231	177				

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

561 1,3-benzenediol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	64	41	19	52	42	43	36	49	11	50	43
51	54	52	29	53	184	54	89	55	97	61	15
62	27	63	74	64	61	65	13	68	56	69	119
71	16	81	201	82	251	95	13	109	11	110	1000
111	51										

562 benzenethiol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	128	50	149	51	205	65	175	66	505	69	114
77	161	84	259	109	316	110	1000	111	102		

563 2,3-benzofluorene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	52	81	69	94	143	95	253	106	60	107	205
108	491	187	75	189	90	213	233	214	60	215	987
216	1000	217	166								

943 benzoic acid

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	29	50	221	51	413	52	45	66	11	74	53
75	25	76	81	77	778	78	76	105	1000	122	868

564 benzyl alcohol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	17	59	16	50	155	51	319	52	78	53	84
61	11	62	31	63	70	64	12	65	75	74	35
75	13	76	18	77	565	78	116	79	1000	80	73
89	65	90	64	91	125	105	38	106	18	107	523
108	737	109	43								

565 2-bromochlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	237	50	890	51	183	73	158	74	506	75	1000
76	202	111	961	113	287	190	638	192	809	194	193

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

566 3-bromochlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	201	50	834	51	174	73	169	74	509	75	914
76	197	111	1000	113	301	190	625	192	802	194	191

567 4-chloro-2-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	119	50	174	51	260	52	531	61	205	62	394
63	1000	64	315	65	192	73	290	74	105	75	156
76	127	78	152	90	724	91	253	101	232	114	312
126	766	128	234	142	211	172	915	174	289		

568 5-chloro-o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	115	51	261	52	257	53	137	77	420	78	134
79	140	89	152	106	1000	140	599	141	964	142	265
143	313										

569 4-chloroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	60	62	55	63	147	64	135	65	329	73	51
91	63	92	186	99	67	100	115	127	1000	128	81
129	292										

570 3-chloronitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	619	51	189	73	144	74	330	75	1000	76	169
85	101	99	258	111	851	113	266	157	424	159	137

571 o-cresol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	102	51	181	53	144	77	358	79	380	80	159
89	114	90	231	107	783	108	1000				

944 p-cresol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	136	51	224	52	106	53	196	77	420	79	308
80	145	90	122	107	822	108	1000				

572 crotoxyphos

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	633	44	448	67	42	77	70	79	41	104	100
105	484	109	21	127	1000	166	180	193	401	194	20

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

573 2,6-di-t-butyl-p-benzoquinone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	392	53	586	55	325	57	668	65	416	67	927
77	376	79	308	91	456	95	322	107	248	121	255
135	538	136	240	149	429	163	292	177	1000	205	203
220	410										

574 2,4-diaminotoluene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	70	42	55	51	76	52	70	53	51	61	91
67	50	77	147	78	69	93	63	94	224	104	128
105	134	106	67	121	958	122	1000	123	79		

575 1,2-dibromo-3-chloropropane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	38	59	341	51	104	61	38	75	1000	76	75
77	331	81	43	93	117	95	106	97	12	105	67
106	17	119	74	121	66	155	635	157	784	158	20
159	204	187	10								

945 3,5-dibromo-4-hydroxybenzonitrile

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	148	61	193	62	222	88	632	117	137	168	152
170	141	275	489	277	1000	279	451				

576 2,6-dichloro-4-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	206	52	1000	61	523	62	828	63	588	73	470
65	137	89	218	90	443	97	458	124	954	126	401
133	218	160	401	176	431	178	134	206	378		

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

577 1,3-dichloro-2-propanol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	14	42	55	43	503	44	22	47	12	58	15
49	113	50	15	51	37	57	10	61	12	75	14
78	11	79	1000	80	25	81	310				

578 2,3-dichloroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
52	138	61	151	62	265	63	455	64	142	65	105
73	130	90	460	99	202	125	108	126	149	161	1000
163	626	165	101								

579 2,3-dichloronitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	220	50	257	61	150	62	120	63	173	73	336
74	976	75	743	84	351	85	166	86	125	109	1000
110	204	111	303	133	701	135	435	145	580	147	368
161	190	163	121	191	411	193	263				

946 2,6-dichlorophenol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	111	62	160	63	714	73	132	98	293	99	117
126	260	162	1000	164	613	166	101				

580 1,2:3,4-diepoxybutane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	37	41	29	42	83	43	60	55	1000	56	67
57	155	58	16	85	13						

581 3,3'-dimethoxybenzidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
65	44	79	222	85	69	93	84	107	46	115	110
122	115	158	154	186	144	201	552	229	162	244	1000
245	152										

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

582 dimethyl sulfone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	10	45	94	46	29	47	18	48	69	62	14
63	69	64	22	65	19	79	1000	81	36	94	528
96	23										

583 p-dimethylaminoazobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	483	51	181	77	447	78	120	79	147	91	109
104	142	105	190	120	1000	148	160	225	676		

584 7,12-dimethylbenzo(a)anthracene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
101	24	112	34	113	112	114	38	119	212	120	296
125	46	126	81	127	60	128	76	215	24	226	47
237	23	239	313	240	230	241	433	242	61	250	32
252	68	253	33	255	84	256	1000	257	180		

585 N,N-dimethylformamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	58	41	79	42	497	43	115	44	1000	45	19
57	17	58	83	72	89	73	994	74	35		

586 3,6-dimethylphenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
76	113	89	129	94	179	101	142	102	151	189	388
190	193	191	430	205	246	206	1000	207	159		

587 1,4-dinitrobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	1000	51	131	63	228	64	218	74	311	75	623
76	664	92	240	122	166	168	399				

588 diphenyldisulfide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	153	51	293	65	671	59	282	77	141	109	1000
110	132	154	191	185	117	218	418				

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

589 ethyl methanesulfonate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	16	43	72	45	208	48	40	59	19	63	23
64	22	65	93	79	1000	80	127	81	42	96	16
97	206	109	579	111	18	123	15	124	33		

590 ethylenethiourea

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	46	42	126	45	97	46	42	59	14	72	89
73	151	102	1000								

591 ethynylestradiol 3-methyl ether

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	155	53	101	91	157	115	143	147	226	159	132
160	115	173	199	174	313	227	1000	228	149	242	153
310	516										

592 hexachloropropene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	131	71	333	106	334	108	200	117	329	119	320
141	206	143	196	211	631	213	1000	215	623	217	186

947 hexanoic acid

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	627	42	535	43	214	45	186	46	19	55	128
56	90	57	102	60	1000	61	66	69	21	70	20
73	412	74	56	87	98						

593 2-isopropylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	100	63	111	76	157	77	129	115	147	127	131
128	216	152	133	153	184	154	114	155	1000	156	139
170	368										

594 isosafrole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	110	51	222	63	127	77	277	78	208	103	355
104	441	131	371	132	107	135	129	161	250	162	1000

595 longifolene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	438	55	719	65	346	67	453	77	566	69	713
91	1000	93	611	94	546	95	404	105	614	107	475
119	394	133	338	161	568	204	172				

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

596 malachite green

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
118	113	126	313	165	369	208	135	209	233	210	181
237	158	253	1000	254	160	329	189	330	775	331	170

597 methapyriline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	72	45	47	53	40	58	1000	71	188	72	225
78	54	79	48	97	516	190	40	191	67		

598 methyl methanesulfonate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	178	56	15	48	108	50	26	63	35	64	48
65	285	78	27	79	821	80	1000	81	44	82	33
95	137	109	59	110	60						

599 2-methylbenzothiozole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	152	50	133	58	153	62	106	63	309	69	513
82	204	108	392	109	102	148	279	149	1000	150	110

900 3-methylcholanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
113	58	119	55	125	83	126	305	132	99	133	122
134	160	250	56	252	322	253	271	263	59	265	106
266	50	267	192	268	1000	269	185				

901 4,4'-methylenebis(2-chloroaniline)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
77	190	84	107	98	299	104	133	115	226	140	316
195	352	229	228	231	1000	233	227	265	171	266	631
267	144	268	358								

902 4,5-methylenephenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	50	62	55	63	95	74	69	81	145	86	53
87	60	94	255	95	659	163	80	187	213	188	137
189	900	190	1000								

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

903 1-methylfluorene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	66	51	87	62	57	63	137	74	64	75	85
76	196	83	135	87	53	88	78	89	203	90	58
139	54	151	73	152	124	163	57	164	58	165	1000
166	136	176	96	177	52	178	202	179	182	180	686
181	99										

904 2-methylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	29	51	39	57	28	58	47	62	26	63	65
65	19	69	56	70	25	71	126	74	25	75	23
76	14	77	15	86	13	87	18	89	42	113	19
114	13	115	303	116	25	126	13	139	98	140	24
141	748	142	1000	143	105						

905 1-methylphenanthrene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	54	63	86	70	62	74	51	81	52	83	164
96	132	163	55	165	217	189	165	191	532	192	1000
193	152										

906 2-(methylthio)benzothiazole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	790	50	212	63	383	69	578	82	233	108	627
136	239	148	938	180	250	181	1000				

907 1,5-naphthalenediamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	48	65	83	77	75	79	111	103	86	118	52
130	262	131	40	141	43	157	89	158	1000	159	117

908 1,4-naphthoquinone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	445	51	62	52	52	66	69	74	189	75	205
76	590	101	51	102	613	103	52	104	550	130	433
158	1000	159	100								

909 alpha-naphthylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	25	51	31	57	36	59	46	62	28	63	59
65	27	71	58	72	104	89	62	113	22	114	34
115	401	116	212	142	53	143	1000	144	101		

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

910 5-nitro-o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	194	52	159	53	121	77	766	78	176	79	619
94	168	104	120	106	691	152	1000				

911 2-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	64	50	51	51	89	52	207	53	74	62	58
63	181	64	155	65	960	66	96	80	212	91	86
92	566	108	170	138	1000	139	63				

912 3-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	101	52	120	53	59	62	58	63	143	64	121
65	1000	66	114	80	169	91	62	92	764	93	62
108	87	138	717	139	51						

913 4-nitroaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
52	228	53	160	62	110	63	216	64	164	65	1000
66	124	80	266	92	300	108	636	138	520		

914 4-nitrobiphenyl

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	131	63	104	76	179	115	134	141	277	151	259
152	902	153	284	169	374	199	1000	200	125		

915 N-nitroso-di-n-butylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	1000	42	536	43	570	44	313	55	129	56	167
57	994	84	985	86	103	99	197	115	158	116	237
158	161										

916 N-nitrosodiethylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	170	42	079	43	69	44	1000	45	20	54	18
56	525	57	492	70	24	71	28	85	25	87	31
102	807	103	35								

917 N-nitrosomethylethylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	117	42	1000	43	667	44	26	54	17	56	189
57	99	59	13	71	60	73	57	88	772	89	20

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

918 N-nitrosomethylphenylamine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	181	51	434	52	104	63	110	77	1000	78	194
79	331	104	147	106	673	107	220	212	137		

919 N-nitrosomorpholine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	181	42	192	43	52	44	17	54	85	55	95
56	1000	57	49	85	13	86	333	87	14	116	337

920 N-nitrosopiperidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	320	42	1000	43	43	51	14	52	12	53	32
54	58	55	444	56	224	57	17	67	21	82	26
83	28	84	47	114	491	115	26				

921 pentachlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
73	160	108	239	125	102	178	102	213	179	215	218
217	106	248	648	250	1000	252	642	254	199		

922 pentachloroethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	203	60	398	62	119	83	378	85	218	94	114
95	165	117	1000	119	979	121	306	130	293	132	272
165	716	167	901	169	422						

923 pentamethylbenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	126	53	84	63	61	65	99	77	145	79	64
91	218	105	128	115	120	117	91	133	1000	134	105
147	60	148	420								

924 perylene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	33	111	43	112	70	113	111	124	132	125	251
126	243	224	49	248	75	249	52	250	284	251	86
252	1000	253	219								

925 phenacetin

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
43	443	51	33	52	112	53	164	63	39	64	30
65	47	79	31	80	179	31	154	108	1000	109	196
110	50	137	461	138	40	179	672	180	64		

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

926 phenothiazine¶

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	145	51	120	63	134	69	190	100	128	154	149
166	240	167	607	198	186	199	1000	200	143		

927 1-phenylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	132	51	156	63	148	74	124	75	142	76	136
87	101	88	183	89	162	100	155	101	527	102	111
200	144	201	136	202	643	203	1000	204	999	205	159

928 2-phenylnaphthalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	108	63	101	76	136	88	133	89	158	101	333
102	188	202	398	203	270	204	1000	205	157		

929 pronamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	270	66	109	74	112	75	137	84	194	109	186
145	334	147	198	173	1000	175	615	254	133	255	211
256	102	257	122								

930 pyridine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	45	48	11	49	62	50	324	51	414	52	879
53	112	54	12	55	16	75	21	76	19	77	22
78	151	79	1000	80	101	81	58				

931 safrole

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	132	51	369	63	108	77	391	78	228	103	348
104	477	105	130	131	437	132	166	161	298	162	1000
163	109										

932 squalene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
53	62	55	94	67	105	68	119	69	1000	70	57
79	43	81	465	82	52	93	70	95	104	107	43
109	47	121	46	137	41						

933 1,2,4,5-tetrachlorobenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	125	49	176	61	127	72	183	73	332	74	448
84	197	108	284	109	231	143	194	145	117	179	237
181	224	214	791	216	1000	218	482	220	101		

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

948 2,3,4,6-tetrachlorophenol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
61	234	65	167	66	105	83	134	84	178	96	202
97	107	131	463	133	270	166	298	168	273	194	168
196	164	230	793	232	1000	234	471				

934 thianaphthene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	80	50	91	51	65	62	82	63	162	67	78
69	139	74	55	89	191	90	136	108	82	134	1000
135	104	136	52								

935 thioacetamide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	225	42	485	43	44	46	18	57	36	58	93
59	165	60	437	75	1000	76	25	77	43		

936 thioxanthone

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	262	63	180	69	320	74	116	69	176	82	121
92	188	108	129	139	385	152	227	183	112	184	951
185	137	212	1000	213	145						

937 o-toluidine

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	51	41	38	42	35	49	10	50	88	51	169
52	164	53	192	53	86	62	26	63	68	64	30
65	59	66	24	74	19	65	14	76	21	77	313
78	113	79	243	80	80	89	107	90	76	91	52
104	45	106	1000	107	90						

938 1,2,3-trimethoxybenzene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
50	257	51	459	52	139	53	276	63	112	65	341
67	114	77	246	79	132	82	117	93	483	95	801
107	190	108	144	110	898	125	578	153	759	168	1000

939 2,4,5-trimethylaniline

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
41	80	52	58	51	63	53	66	65	150	67	74
79	62	91	167	93	51	117	54	118	65	119	93
120	1000	121	87	134	670	135	978	136	99		

Appendix A
Mass Spectra in the Form of Mass/Intensity Lists

940 triphenylene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
74	52	87	55	100	107	101	108	112	131	113	244
114	181	200	67	202	56	224	84	225	56	226	313
227	132	228	1000	229	184						

941 tripropylene glycol methyl ether

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
45	492	46	15	47	19	55	17	57	68	58	43
59	1000	60	34	71	16	72	44	73	363	74	232
103	57	117	92	161	21						

942 1,3,5-trithiane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
46	1000	47	150	48	98	59	93	60	76	64	136
73	102	91	92	92	111	110	58	138	259		