METHOD 8270E

SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required methods used for the analysis of method-defined parameters (MDPs), are intended to be guidance methods that contain general information on how to perform an analytical procedure or technique. A laboratory can use this guidance as a basic starting point for generating its own detailed standard operating procedure (SOP), either for its own general use or for a specific project application. The performance data referenced in this method are for guidance purposes only and are not intended to be and must not be used as absolute quality control (QC) acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. The following analytes have been determined by this method (shown below):

		Appropriate Preparation Techniques[b]					
Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
Acenaphthene	83-32-9	✓	✓	✓	✓	✓	✓
Acenaphthylene	208-96-8	✓	✓	✓	✓	✓	✓
Acetophenone	98-86-2	✓	✓	✓	✓	✓	✓
2-Acetylaminofluorene	53-96-3	✓	✓	-	✓	✓	-
1-Acetyl-2-thiourea	591-08-2	*	-	-	-	-	*
Aldrin	309-00-2	✓	✓	✓	-	✓	✓
2-Aminoanthraquinone	117-79-3	✓	-	-	-	-	✓
Aminoazobenzene	60-09-3	✓	-	-	-	-	✓
4-Aminobiphenyl	92-67-1	✓*	✓*	-	-	*	✓
3-Amino-9-ethylcarbazole	132-32-1	✓	✓	-	-	-	-
Anilazine	101-05-3	✓	-	-	-	-	✓
Aniline	62-53-3	✓*	✓*	✓*	*	✓*	✓
o-Anisidine	90-04-0	✓	-	-	-	-	✓
Anthracene	120-12-7	✓	✓	✓	✓	✓	✓
Aramite	140-57-8	✓*	✓*	-	-	✓	-
Aroclor 1016 (PCB-1016)	12674-11-2	✓	✓	✓	-	✓	✓
Aroclor 1221 (PCB-1221)	11104-28-2	✓	✓	✓	-	✓	✓
Aroclor 1232 (PCB-1232)	11141-16-5	✓	✓	✓	-	✓	✓
Aroclor 1242 (PCB-1242)	53469-21-9	✓	✓	✓	-	✓	✓
Aroclor 1248 (PCB-1248)	12672-29-6	✓	✓	✓	-	✓	✓
Aroclor 1254 (PCB-1254)	11097-69-1	✓	✓	✓	-	✓	✓
Aroclor 1260 (PCB-1260)	11096-82-5	✓	✓	✓	-	✓	✓
Atrazine	1912-24-9	✓*	✓*	✓*	✓*	✓*	-
Azinphos-methyl (Guthion)	86-50-0	*	*	-	-	-	✓
Azobenzene	103-33-3	✓	✓	-	-	✓	-
Barban	101-27-9	*	-	-	-	-	*
Benzaldehyde	100-52-7	✓*	✓*	✓*	✓*	✓*	-
Benzidine	92-87-5	*	*	*	✓*	*	*
Benzoic acid	65-85-0	*	✓*	✓*	*	✓*	✓*
Benzo(a)anthracene	56-55-3	✓	✓	✓	✓	✓	✓
Benzo(b)fluoranthene	205-99-2	✓	✓	✓	✓	✓	✓
Benzo(k)fluoranthene	207-08-9	✓	✓	✓	✓	✓	✓
Benzo(g,h,i)perylene	191-24-2	✓	✓	✓	✓	✓	✓
Benzo(a)pyrene	50-32-8	✓	✓	✓	✓	✓	✓
Benzo(e)pyrene	192-97-2	✓	-	-	-	✓	-
p-Benzoquinone	106-51-4	*	*	-	-	-	✓
Benzyl alcohol	100-51-6	✓*	✓*	✓*	✓*	✓*	✓*
α-BHC	319-84-6	✓*	✓*	✓	-	✓	✓
β-BHC	319-85-7	✓	✓	✓	-	✓	✓
δ-BHC	319-86-8	✓	✓	✓	-	✓	✓
γ-BHC (Lindane)	58-89-9	✓*	✓*	✓	-	✓	✓
1,1'-Biphenyl	92-52-4	✓	✓	✓	✓	✓	-

Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
Bis(2-chloroethoxy)methane	111-91-1	✓	✓	✓	✓	✓	✓
Bis(2-chloroethyl)ether	111-44-4	✓	✓	✓	✓	✓	✓
Bis(2-chloro-1-methylethyl)ether[c]	108-60-1	✓	✓	✓	-	✓	✓
Bis(2-ethylhexyl)phthalate	117-81-7	✓	✓	✓	✓	✓	✓
4-Bromophenyl phenyl ether	101-55-3	✓	✓	✓	✓	✓	✓
Bromoxynil (Brominal)	1689-84-5	✓	-	-	-	-	✓
Butyl benzyl phthalate	85-68-7	✓	✓	✓	✓	✓	✓
Caprolactam	105-60-2	*	*	✓	✓	✓	-
Captafol	2425-06-1	*	*	-	-	-	✓
Captan	133-06-2	*	*	-	-	-	✓
Carbaryl (Sevin)	63-25-2	✓	-	-	-	-	✓
Carbazole	86-74-8	✓	✓	✓	✓	✓	-
Carbofuran (Furaden)	1563-66-2	✓	-	-	-	-	✓
Carbophenothion	786-19-6	✓	-	-	-	-	✓
Chlordane (NOS)	57-74-9	✓	✓	✓	-	✓	✓
Chlorfenvinphos	470-90-6	✓	-	-	-	-	✓
4-Chloroaniline	106-47-8	✓	✓	✓	✓	✓	✓
Chlorobenzilate	510-15-6	✓	✓	-	✓	✓	✓
5-Chloro-2-methylaniline	95-79-4	✓	-	-	-	-	✓
4-Chloro-3-methylphenol	59-50-7	✓	✓	✓	✓	✓	✓
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	✓	-	-	-	-	✓
1-Chloronaphthalene	90-13-1	✓	✓	✓	-	✓	✓
2-Chloronaphthalene	91-58-7	✓	✓	✓	✓	✓	✓
2-Chlorophenol	95-57-8	✓	✓	✓	✓	✓	✓
4-Chlorophenyl phenyl ether	7005-72-3	✓	✓	✓	✓	✓	✓
4-Chloro-1,2-phenylenediamine	95-83-0	✓	✓	-	-	-	-
4-Chloro-1,3-phenylenediamine	5131-60-2	✓	✓	-	-	-	-
Chrysene	218-01-9	✓	✓	✓	✓	✓	✓
Coumaphos	56-72-4	✓	-	-	-	-	✓
p-Cresidine	120-71-8	✓	-	-	-	-	✓
Crotoxyphos	7700-17-6	✓	-	-	-	-	✓
2-Cyclohexyl-4,6-dinitrophenol	131-89-5	✓	-	-	-	-	*
4,4'-DDD	72-54-8	✓	✓	✓	-	✓	✓
4,4'-DDE	72-55-9	✓	✓	✓	-	✓	✓
4,4'-DDT	50-29-3	✓	✓	✓	-	✓	✓
Demeton-O	298-03-3	*	*	-	-	-	✓
Demeton-S	126-75-0	*	*	-	-	-	✓
Diallate (cis or trans)	2303-16-4	✓	✓	-	✓	✓	✓
2,4-Diaminotoluene	95-80-7	*	*	-	-	-	✓
Dibenz(a,j)acridine	224-42-0	✓	-	-	-	✓†	✓
Dibenz(a,h)anthracene	53-70-3	✓	✓	✓	✓	✓	✓
Dibenzofuran	132-64-9	✓	✓	✓	✓	✓	✓
Dibenzo(a,e)pyrene	192-65-4	✓	-	-	-	✓	✓
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	✓	✓	-	-	-	-
Di-*n*-butyl phthalate	84-74-2	✓	✓	✓	✓	✓	✓
Dichlone	117-80-6	*	*	-	-	-	✓
1,2-Dichlorobenzene	95-50-1	✓*	✓*	✓*	✓*	✓*	✓
1,3-Dichlorobenzene	541-73-1	✓*	✓*	✓*	✓*	✓*	✓*

Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
1,4-Dichlorobenzene	106-46-7	✓*	✓*	✓*	✓*	✓*	✓
3,3'-Dichlorobenzidine	91-94-1	✓*	✓*	✓*	✓*	✓*	✓*
2,4-Dichlorophenol	120-83-2	✓	✓	✓	✓	✓	✓
2,6-Dichlorophenol	87-65-0	✓	✓	✓	✓	✓	✓
Dichlorovos (DDVP, Dichlorvos)	62-73-7	✓	-	-	-	-	✓
Dicrotophos	141-66-2	✓	-	-	-	-	✓
Dieldrin	60-57-1	✓	✓	✓	-	✓	✓
Diethyl phthalate	84-66-2	✓	✓	✓	✓	✓	✓
Diethyl sulfate	64-67-5	*	-	-	-	-	*
Diethylstilbestrol	56-53-1	*	*	-	-	-	✓
Dimethoate	60-51-5	✓*	✓*	-	-	✓*	✓
3,3'-Dimethoxybenzidine	119-90-4	✓	-	-	-	-	*
Dimethyl phthalate	131-11-3	✓	✓	✓	✓	✓	✓
Dimethylaminoazobenzene	60-11-7	✓	✓	-	✓	✓	✓
7,12-Dimethylbenz(a)anthracene	57-97-6	✓*	✓*	-	✓*	✓*	*
3,3'-Dimethylbenzidine	119-93-7	*	*	-	*	*	✓
α,α-Dimethylphenethylamine	122-09-8	-	-	-	-	-	✓
2,4-Dimethylphenol	105-67-9	✓	✓	✓	✓	✓	✓
1,2-Dinitrobenzene	528-29-0	✓	✓	-	-	✓	✓
1,3-Dinitrobenzene (1,3-DNB)	99-65-0	✓	✓	-	✓	✓	✓
1,4-Dinitrobenzene	100-25-4	✓*	-	-	-	✓	✓
4,6-Dinitro-2-methylphenol	534-52-1	✓*	✓*	✓*	✓*	✓*	✓*
2,4-Dinitrophenol	51-28-5	✓*	✓*	✓*	✓*	✓*	✓*
2,4-Dinitrotoluene (2,4-DNT)	121-14-2	✓	✓	✓	✓	✓	✓
2,6-Dinitrotoluene (2,6-DNT)	606-20-2	✓	✓	✓	✓	✓	✓
Dinocap	39300-45-3	*	*	-	-	-	*
Di-*n*-octyl phthalate	117-84-0	✓	✓	✓	✓	✓	✓
Dinoseb (DNBP)	88-85-7	✓	✓	✓	-	✓	✓
1,4-Dioxane	123-91-1	*	✓*	*	*	*	-
Diphenylamine	122-39-4	✓	✓	✓	-	✓	✓
5,5-Diphenylhydantoin	57-41-0	✓	-	-	-	-	✓
1,2-Diphenylhydrazine	122-66-7	✓*	✓*	✓*	✓*	✓*	✓*
Disulfoton	298-04-4	✓	✓	-	-	✓	✓
Endosulfan I	959-98-8	✓*	✓*	✓	-	✓	✓
Endosulfan II	33213-65-9	✓*,	✓*	✓	-	✓	✓
Endosulfan sulfate	1031-07-8	✓	✓	✓	-	✓	✓
Endrin	72-20-8	✓*	✓*	✓	-	✓	✓
Endrin aldehyde	7421-93-4	✓	✓	✓	-	✓	✓
Endrin ketone	53494-70-5	✓	✓	-	-	✓	✓
EPN	2104-64-5	✓	-	-	-	-	✓
Ethion	563-12-2	✓	-	-	-	-	✓
Ethyl carbamate	51-79-6	*	-	-	-	-	✓
Ethyl methanesulfonate	62-50-0	✓	✓	-	✓	✓	✓
Famphur	52-85-7	✓*	-	-	-	*	✓
Fensulfothion	115-90-2	✓	-	-	-	-	✓
Fenthion	55-38-9	✓	-	-	-	-	✓
Fluchloralin	33245-39-5	✓	-	-	-	-	✓
Fluoranthene	206-44-0	✓	✓	✓	✓	✓	✓
Fluorene	86-73-7	✓	✓	✓	✓	✓	✓
Heptachlor	76-44-8	✓	✓	✓	-	✓	✓

Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
Heptachlor epoxide	1024-57-3	✓	✓	✓	-	✓	✓
Hexachlorobenzene	118-74-1	✓	✓	✓	✓	✓	✓
Hexachlorobutadiene	87-68-3	✓	✓	✓	✓	✓	✓
Hexachlorocyclopentadiene	77-47-4	✓*	*	✓*	✓*	✓*	✓*
Hexachloroethane	67-72-1	✓	✓	✓	✓	✓	✓
Hexachlorophene	70-30-4	✓*	-	-	-	-	*
Hexachloropropene	1888-71-7	✓	*	✓	✓	✓	✓
Hexamethyl phosphoramide (HMPA)	680-31-9	✓	-	-	-	-	✓
Hydroquinone	123-31-9	-	-	-	-	-	✓
Indeno(1,2,3-cd)pyrene	193-39-5	✓	✓	✓	✓	✓	✓
Isodrin	465-73-6	✓	✓	-	✓	✓	✓
Isophorone	78-59-1	✓	✓	✓	✓	✓	✓
Isosafrole	120-58-1	✓	✓	-	✓	✓	✓
Kepone	143-50-0	✓*	-	-	-	✓	✓
Leptophos	21609-90-5	✓	-	-	-	-	✓
Malathion	121-75-5	*	*	-	-	-	✓
Maleic anhydride	108-31-6	*	*	-	-	-	✓
Mestranol	72-33-3	✓	-	-	-	-	✓
Methapyrilene	91-80-5	*	-	-	-	-	✓
Methoxychlor	72-43-5	✓	-	-	-	✓	✓
Methyl methanesulfonate	66-27-3	✓*	✓*	-	-	✓*	✓
Methyl parathion	298-00-0	✓	✓	-	-	✓	✓
3-Methylcholanthrene	56-49-5	✓	✓	-	✓	✓	✓
4,4'-Methylenebis(2-chloroaniline)	101-14-4	*	*	-	-	-	*
4,4'-Methylenebis(*N,N*-dimethyl-aniline)	101-61-1	✓	✓	-	-	-	-
1-Methylnaphthalene	90-12-0	✓	✓	✓	✓	✓	-
2-Methylnaphthalene	91-57-6	✓	✓	✓	✓	✓	✓
2-Methylphenol (*o*-Cresol)	95-48-7	✓	✓	✓	✓	✓	✓
3-Methylphenol (*m*-Cresol)	108-39-4	✓	✓	✓	✓	✓	✓
4-Methylphenol (*p*-Cresol)	106-44-5	✓	✓	✓	✓	✓	✓
Mevinphos	7786-34-7	✓	-	-	-	-	✓
Mexacarbate	315-18-4	*	*	-	-	-	✓
Mirex	2385-85-5	✓	-	-	-	-	✓
Monocrotophos	6923-22-4	*	*	-	-	-	✓
Naled	300-76-5	✓	-	-	-	-	✓
Naphthalene	91-20-3	✓	✓	✓	✓	✓	✓
1,4-Naphthoquinone	130-15-4	✓*	✓*	-	✓*	✓*	✓
1-Naphthylamine	134-32-7	✓*	✓*	-	-	*	✓
2-Naphthylamine	91-59-8	✓*	✓*	-	-	*	✓
Nicotine	54-11-5	*	-	-	-	-	✓
5-Nitroacenaphthene	602-87-9	✓	-	-	-	-	✓
2-Nitroaniline	88-74-4	✓*	✓*	✓*	✓*	✓*	✓*
3-Nitroaniline	99-09-2	✓*	✓*	✓*	✓*	✓*	✓*
4-Nitroaniline	100-01-6	✓*	✓	-	-	✓*	✓
5-Nitro-*o*-anisidine	99-59-2	✓	-	-	-	-	✓
Nitrobenzene (NB)	98-95-3	✓	✓	✓	✓	✓	✓
4-Nitrobiphenyl	92-93-3	✓	-	-	-	-	✓
Nitrofen	1836-75-5	✓	-	-	-	-	✓

Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
2-Nitrophenol	88-75-5	✓	✓	✓	✓	✓	✓
4-Nitrophenol	100-02-7	*	✓*	✓*	✓*	✓*	✓*
4-Nitroquinoline-1-oxide	56-57-5	✓	✓	-	✓	✓	✓
N-Nitroso-di-*n*-butylamine	924-16-3	✓	✓	✓	-	✓	✓
N-Nitrosodiethylamine	55-18-5	✓	✓	✓	✓	✓	✓
N-Nitrosodimethylamine	62-75-9	*	✓*	✓*	✓*	✓*	✓*
N-Nitrosodiphenylamine	86-30-6	✓*	✓*	✓*	✓*	✓*	✓*
N-Nitroso-di-*n*-propylamine	621-64-7	✓	✓	✓	✓	✓	✓
N-Nitrosomethylethylamine	10595-95-6	✓	✓	✓	✓	✓	✓
N-Nitrosomorpholine	59-89-2	✓	✓	-	✓	✓	✓
N-Nitrosopiperidine	100-75-4	✓	✓	✓	-	✓	✓
N-Nitrosopyrrolidine	930-55-2	✓	✓	✓	✓	✓	✓
5-Nitro-*o*-toluidine	99-55-8	✓	✓	-	✓	✓	✓
Octamethyl pyrophosphoramide	152-16-9	*	-	-	-	-	*
4,4'-Oxydianiline	101-80-4	✓	-	-	-	-	✓
Parathion	56-38-2	✓	✓	-	-	✓	✓
Pentachlorobenzene	608-93-5	✓	✓	✓	✓	✓	✓
Pentachloronitrobenzene (PCNB)	82-68-8	✓	✓	-	✓	✓	✓
Pentachlorophenol	87-86-5	✓*	✓*	✓*	✓*	✓*	✓*
Perylene	198-55-0	✓	-	-	-	-	✓
Phenacetin	62-44-2	✓	✓	-	✓	✓	✓
Phenanthrene	85-01-8	✓	✓	✓	✓	✓	✓
Phenobarbital	50-06-6	✓	-	-	-	-	✓
Phenol	108-95-2	*	✓*	✓*	✓*	✓*	✓*
1,4-Phenylenediamine	106-50-3	✓	-	-	*	-	✓
Phorate	298-02-2	✓	✓	-	-	✓	✓
Phosalone	2310-17-0	*	*	-	-	-	✓
Phosmet (Imidan)	732-11-6	*	*	-	-	-	✓
Phosphamidon	13171-21-6	*	*	-	-	-	✓
Phthalic anhydride	85-44-9	*	*	-	-	-	*
2-Picoline (2-Methylpyridine)	109-06-8	✓	✓	-	✓	✓	-
Piperonyl sulfoxide	120-62-7	✓	-	-	-	-	✓
Polychlorinated biphenyls (NOS)	1336-36-3	-	-	-	-	-	-
Pronamide (Kerb)	23950-58-5	✓	✓	-	✓	✓	✓
Propylthiouracil	51-52-5	*	-	-	-	-	*
Pyrene	129-00-0	✓	✓	✓	✓	✓	✓
Pyridine	110-86-1	*	✓*	✓*	*	✓*	-
Resorcinol	108-46-3	*	*	-	-	-	✓
Safrole	94-59-7	✓	✓	-	✓	✓	✓
Strychnine	57-24-9	*	*	-	-	-	✓
Sulfallate	95-06-7	✓	-	-	-	-	✓
Terbufos	13071-79-9	✓	-	-	-	-	✓
1,2,4,5-Tetrachlorobenzene	95-94-3	✓	✓	✓	✓	✓	✓
2,3,4,6-Tetrachlorophenol	58-90-2	✓	✓	✓	✓	✓	✓
Tetrachlorvinphos (Stirophos, Gardona)	961-11-5	✓	-	-	-	-	✓
Tetraethyl dithiopyrophosphate	3689-24-5	✓	✓	-	-	✓	-
Tetraethyl pyrophosphate (TEPP)	107-49-3	✓	-	-	-	-	✓
Thionazine	297-97-2	✓	✓	-	-	✓	✓
Thiophenol (Benzenethiol)	108-98-5	✓	-	-	-	-	✓

Compounds	CAS No[a]	3510	3520	3540/3541	3545	3550	3580
2,4-Toluene diisocyanate	584-84-9	*	*	-	-	-	✓
o-Toluidine	95-53-4	✓	✓	-	✓	*	✓
Toxaphene	8001-35-2	✓	✓	✓	-	✓	✓
1,2,4-Trichlorobenzene	120-82-1	✓	✓	✓	✓	✓	✓
2,4,5-Trichlorophenol	95-95-4	✓	✓	✓	✓	✓	✓
2,4,6-Trichlorophenol	88-06-2	✓	✓	✓	✓	✓	✓
O,O,O-Triethyl phosphorothioate	126-68-1	✓	✓	-	-	✓	-
Trifluralin (Treflan)	1582-09-8	✓	-	-	-	-	✓
Trimethyl phosphate	512-56-1	*	*	-	-	-	✓
2,4,5-Trimethylaniline	137-17-7	✓	-	-	-	-	✓
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4	✓	✓	-	✓	✓	✓
Tris(2,3-dibromopropyl)phosphate	126-72-7	✓	-	-	-	-	*
Tri-*p*-tolyl phosphate	78-32-0	✓	-	-	-	-	✓

[a] Chemical Abstract Service (CAS) Registry Number
[b] See Sec. 1.2 for other acceptable preparation methods.
[c] Chemical name changed by Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This analyte is also known as 2,2'-oxybis(1-chloropropane) (CAS index name). See the link at http://www.epa.gov/iris/subst/0407.htm, Sec. VII for the "Revision History" and Sec. VIII for synonyms of this chemical.

KEY TO ANALYTE LIST

✓ Historically, adequate recovery and precision can be obtained for this analyte by this technique. However, actual recoveries may vary depending on the sample matrix, the number of constituents being analyzed concurrently, analytical instrumentation, and the preparation method used. Performance data from a large multi-site laboratory control sample (LCS) study were used to update this table (data can be found in Reference 13 in Sec. 16 and in Table 2). If the average % recovery (%R) fell between 50 - 150% in this study, the preparation technique was considered adequate.

NOTE: Not every analyte has sufficient data points in the study for consideration. The ✓ is also used for analytes if the previous version of this method listed the preparation technique as adequate. See Table 2 for study data. Refer to Sec. 9 for guidance on establishing LCS acceptance criteria.

- This analyte was not determined by this preparation method.

* This analyte exhibits known difficulties with reproducibility, response, recovery, stability, and/or chromatography that may reduce the overall quality or confidence in the result when using this preparation method combined with analysis by Method 8270. This analyte may require special treatment to improve extraction efficiency and analytical performance to a level that would meet the needs of the project and, where necessary, may also require the use of appropriate data qualification. See Sec. 1.4 for specific information regarding this analyte.

✓* This analyte met the criteria for adequate performance using this preparation technique (see definition for ✓). However, the analyte is known to exhibit the problems listed in Sec. 1.4 (see definition for *).

1.2 In addition to the sample preparation methods listed in the above analyte list, the following methods may be used for extraction of semivolatile organic compounds provided the method can be demonstrated to meet the needs of the project:

Air (particulates and sorbent resin)
Method 3542 Extraction of Semivolatile Analytes Collected Using Method 0010

Water (including Toxicity Characteristic Leaching procedure (TCLP) leachates)
Method 3511 Microextraction
Method 3535 Solid-Phase Extraction (SPE)

Soil, Sediment, and Waste
Method 3546 Microwave Extraction
Method 3561 Supercritical Fluid Extraction of Polynuclear Aromatic Hydrocarbons (PAHs)

1.3 This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride (or other suitable solvents provided that the desired performance data can be generated) and are capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include PAHs, chlorinated hydrocarbons, chlorinated pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols (including nitrophenols). See Table 1 for a list of compounds and their characteristic ion(s) that have been evaluated.

In most cases, this method is not appropriate for the quantitation of multicomponent analytes (e.g., polychlorinated biphenyls (PCBs) as Aroclors, technical toxaphene, chlordane, etc.) because of limited sensitivity for these analytes or potential for measurement bias using gas chromatograph/mass spectrometer (GC/MS) technology. Tandem mass spectrometry (GC/MS/MS) may provide adequate sensitivity and selectivity for performing multi-component analyses. Individual components (e.g., a subset of PCB congeners) may be determined with any technology provided sensitivity is sufficient for the data application and interference from other components is minimal. When these analytes have been identified by another technique, Method 8270 may be appropriate for confirmation of the identification of these analytes when concentration in the extract permits. See Sec. 11.7.5 for more information.

1.4 The following compounds may require special treatment when being determined by this method:

NOTE: Some compounds may appear in more than one paragraph.

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration.

1.4.2 Under the alkaline conditions of the extraction step from aqueous matrices, α-BHC, γ-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction should be performed if these compounds are to be reported.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the GC, chemical reaction in acetone solution, and photochemical decomposition. Protecting this analyte from light during heated extraction steps in the procedure (such as concentration) is recommended.

1.4.4 N-Nitrosodimethylamine may be difficult to separate from the solvent peak under the chromatographic conditions described.

1.4.5 N-Nitrosodiphenylamine decomposes in the GC inlet and cannot be separated from diphenylamine. For this reason, it is acceptable to report the combined result for n-nitrosodiphenylamine and diphenylamine for either of these compounds as a combined concentration.

1.4.6 1,2-Diphenylhydrazine is unstable (even at room temperature) and readily converts to azobenzene. Given this analyte's stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using azobenzene. Under these circumstances (poor compound separation) the results for either of these compounds should be reported as a combined concentration.

1.4.7 Benzidine, benzyl alcohol, benzoic acid, 7,12-dimethylbenz(a)anthracene, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, dinocap, hexachlorophene, kepone, mathapyrilene, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, pentachlorophenol, 1,4-phenylenediamine, phthalic anhydride, and o-toluidine are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.8 Analytes that readily ionize in solution may not recover from water matrices unless the pH is adjusted to acidic conditions (e.g., phenols with low acid dissociation constant (pKa)) or to basic conditions (e.g., aniline and pyridine) prior to extraction.

1.4.9 Some analytes may perform poorly at the GC injection port temperatures listed in this method. Lowering the injection port temperature may reduce the amount of degradation. However, the analyst must use caution in modifying the injection port temperature, as the performance of other analytes may be adversely affected. A programmable temperature inlet may also be used.

1.4.10 More volatile analytes such as dichlorobenzenes, 1,4-dioxane, and pyridine may be lost during the evaporative concentration step during sample preparation. As a result, many of the extraction methods listed above may yield low recoveries unless great care is exercised during the concentration steps. To better assess the performance of these analytes, it may be appropriate to use additional surrogates which have similar physicochemical properties such as 1,2-dichlorobenzene-d_4, 1,4-dioxane-d_8, and pyridine-d_5, respectively.

1.4.11 2,4-Toluene diisocyanate rapidly hydrolyzes in water (it has a half-life of less than 30 minutes). Therefore, recoveries of this compound from aqueous matrices should not be expected. In addition, in solid matrices, 2,4-toluene diisocyanate often reacts with alcohols and amines to produce urethane and ureas and consequently cannot usually coexist in a solution containing these materials.

1.4.12 The following analytes may be subject to oxidation or hydrolysis during extraction from water matrices which may be accelerated by acidic or basic conditions (Methods 3510 and 3520): aramite, p-benzoquinone, captafol, demeton-O, demeton-S, 2,4-diaminotoluene, dichlone, dimethoate, 1,4-dinitrobenzene, dinocap, maleic anhydride, malathion, 4,4'-methylenebis(2-chloroaniline), mexacarbate, monocrotophos,

phosalone, phosmet, phthalic anhydride, phthalates, phosphamidon, resorcinol, and trimethylphosphate.

1.4.13 The following analytes may be subject to hydrolysis in water matrices during storage (Methods 3510 and 3520): aramite, azinphos-methyl, captafol, captan, demeton-O, dimethoate, dinocap, malathion, mexacarbate, phosalone, and phosmet.

1.4.14 The following analytes may be subject to degradation during storage: 4-aminobiphenyl, atrazine, benzidine, benzaldehyde, 3,3'-dichlorobenzidine, 3,3'-dimethylbenzidine, diethylstilbestrol, 7,12-dimethylbenz(a)anthracene, famfur, hexachlorophene, kepone, 4,4'-methylenebis(2-chloroaniline), methyl methanesulfonate, 1,4-naphthoquinone, 1-naphthylamine, 2-naphthylamine, and strychnine. This degradation may be accelerated when combined with incompatible analytes or solvents such as in calibration standards (e.g., amines and aldehydes are incompatible).

1.4.15 The following analytes may have low response and/or low recovery: 1-acetyl-2-thiourea, 2-cyclohexyl-4,6-dinitro-phenol, barban, diethyl sulfate, 3,3'-dimethoxybenzidine, 4,4'-methylenebis(2-chloroaniline), octamethylpyrophosphoramide, propylthiouracil, and tris(2,3-dibromopropyl)phosphate.

1.4.16 The following analytes are known to adhere to surfaces during extraction and storage in water matrices (Methods 3510 and 3520): diethylstilbestrol, hexachlorophene, and strychnine.

1.4.17 The following analytes have an unfavorable distribution coefficient when extracting from water matrices which may be of more concern when preparing samples using Method 3510: benzoic acid, caprolactam, 2,4-diaminotoluene, ethyl carbamate, nicotine, 4-nitrophenol, N-nitrosodimethylamine, phenol, and resorcinol.

1.4.18 In addition, analytes in the list provided above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.5 This method includes the optional use of an alternate carrier gas (hydrogen) and GC/MS/MS. See Appendix B and Sec. 6.1.3.3.

1.6 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on QC procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the SW-846 manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by Environmental Protection Agency (EPA or the Agency) as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives (DQOs) for the intended application.

1.7 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use of the GC/MS and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The samples are prepared for analysis by GC/MS using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a GC equipped with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with an MS connected to the GC.

2.3 Analytes eluted from the capillary column are introduced into the MS via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra and retention times (RT) with the mass spectra and RTs of known standards for the target compounds. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard (IS) using an appropriate calibration curve for the intended application.

2.4 This method includes specific calibration and QC steps that supersede the general recommendations provided in Method 8000.

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 Solvents, reagents, gases, other samples, and the environment in which the analysis is performed may yield artifacts and/or interferences for target analytes. The sample preparation and analysis process must be demonstrated to be free from observable interferences by the analysis of method blanks (MBs). Refer to each method to be used for specific guidance on QC procedures and to Chapter Four for general guidance on the cleaning of glassware. Refer to Method 8000 for a discussion of interferences.

4.2 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem. Subtracting blank values from sample results is not permitted. If measured analyte concentrations are suspected of being biased or false positive results for a sample, the laboratory should qualify the affected data or otherwise inform the data user(s) of any suspected data quality issues.

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Some contamination may be eliminated

by baking out the column between analyses. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination. Low-level samples that immediately follow high-level samples need to be inspected for possible carryover. See Method 8000, Sec. 4.2 for further guidance.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals listed in this method. A reference file of safety data sheets (SDSs) must be available to all personnel involved in these analyses. See Appendix B, Sec. B1.4 for safety guidance on using hydrogen carrier gas.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 GC/MS system

6.1.1 GC – An analytical system equipped with a temperature-programmable GC suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases (see Appendix B for guidance on using hydrogen carrier gas). The injection port can be split/splitless, temperature-programmable split/splitless (programmable temperature vaporization or PTV), or on-column. The capillary column should be directly coupled to the source. The GC should be equipped with flow controllers such that the column flow rate will remain constant throughout temperature program operation.

6.1.2 Column – 30 m x 0.25 mm ID (or 0.32 mm ID) 0.25, 0.5, or 1 µm film thickness silicone-coated fused-silica capillary column (5% phenyl-methylpolysiloxane, 5% phenyl-arylene dimethylpolysiloxane, or equivalent). The columns listed in this section were the columns used in developing the method. The listing of these columns in this method is not intended to exclude the use of other columns that may be developed. Laboratories may use these columns or other capillary columns provided that the laboratories document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity) that are appropriate for the intended application.

6.1.3 MS

6.1.3.1 Capable of acquiring mass spectra from mass/charge (*m/z)* 35 to 500 at a rate fast enough to acquire at least 5 (but preferably 10 or more)

mass spectra across each chromatographic peak of interest, using 70 volts (nominal) electron energy in the electron impact ionization mode. The MS must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria as outlined in Sec. 11.3.1.

6.1.3.2 An ion trap MS may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact like spectra that match those in the EPA/National Institute of Standards and Technology (NIST) library (or equivalent). The MS must be capable of producing a mass spectrum for DFTPP which meets the criteria as outlined in Sec. 11.3.1.

6.1.3.3 An MS/MS detector may be used if the detector has the necessary pumps, collision cell, collision gases, and high-vacuum system capable of performing transitions in product ion scan mode or the selected reaction monitoring mode (SRM) for the target analytes of interest. Recommendations for specific precursor and product ions in SRM are available for some target analytes from the manufacturers of the equipment. When analysis is performed using product ions for quantitation, it is not an appropriate verification of the system to perform DFTPP analysis and meet the criteria outlined in Sec. 11.3.1. The system, however, must be capable of documenting the performance of both MSs against manufacturer specifications for mass resolution, mass assignment, and sensitivity using the internal calibrant (e.g., Perfluorotributylamine). The performance of the system should be checked at least weekly, or at a frequency appropriate to meet the needs of the project. At a minimum, the performance of the system must be checked just prior to the initial calibration (ICAL).

6.1.3.4 Selected ion monitoring (SIM) or chemical ionization (CI) mass spectrometry are acceptable techniques for applications requiring quantitation limits below the normal range of electron impact mass spectrometry or to reduce interferences from the sample matrix. DFTPP analysis is not appropriate when CI analysis is used for quantitative purposes. See Sec. 11.3.1.

6.1.4 GC/MS interface – Any GC-to-MS interface may be used that gives acceptable calibration points for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary direct into the MS source.

6.1.5 Data system – A computer system that allows the continuous acquisition and storage of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the MS. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan number limits. A recent version of the EPA/NIST mass spectral library (or equivalent) should also be available.

6.1.6 Guard column (optional) – Between the injection port and the analytical column, joined with column connectors or may be purchased integrated into the analytical column.

6.2 Syringes – various

6.3 Volumetric flasks, Class A – Appropriate sizes equipped with ground-glass stoppers

6.4 Balance – Analytical, capable of weighing 0.0001 g

6.5 Bottles – Glass equipped with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops

6.6 Vials – for GC autosampler

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available at: http://pubs.acs.org/reagents/comminfo/techquestions.html. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Organic-free reagent water – All references to water in this method refer to organic-free reagent water.

7.3 Standard solutions

The following sections describe the preparation of stock, intermediate, and working standards for the compounds of interest. This discussion is provided as an example. Other approaches and concentrations of the target compounds may be used if appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards. Commercially prepared stock standards may be used at any concentration if they are certified by an accredited supplier or third party.

7.4 Stock standard solutions (1000 mg/L) – Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

7.4.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in a suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.4.2 Transfer the stock standard solutions into bottles equipped with PTFE-lined screw caps. Store the vials (protected from light) at ≤6 °C or as recommended by the standard manufacturer. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

7.4.3 Certified solutions purchased from a vendor must be replaced per the manufacturer's recommended expiration date. Stock standard solutions prepared in-

house must be replaced after one year or sooner if comparison with QC check samples indicates a problem. When solutions are mixed together, regardless of the source, they must be replaced after the manufacturer's expiration date or one year (whichever occurs first) or sooner if problems are indicated.

7.4.4 It is recommended that nitrosamine compounds be placed together in a separate calibration mix and not combined with other calibration mixes. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

7.4.5 Mixes with hydrochloride salts may contain hydrochloric acid, which can cause analytical difficulties. When using a premixed certified standard, consult the manufacturer's instructions for additional guidance.

7.5 IS solutions – The recommended ISs are: 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (see Table 5). See Sec. 11.4.3 of Method 8000 for additional information. Other compounds may be used as ISs as long as they have RTs similar to their target compounds, they can be unambiguously identified and meet any applicable acceptance criteria described in Sec. 11. See Sec. 11.4.3 of Method 8000 for additional information.

7.5.1 Dissolve 0.200 g of each compound with a small volume of carbon disulfide. Transfer to a 50-mL volumetric flask and dilute to volume with methylene chloride so that the final solvent is approximately 20% carbon disulfide. Most of the compounds are also soluble in small volumes of methanol, acetone, or toluene, except for perylene-d_{12}. The resulting solution will contain each standard at a concentration of 4,000 ng/µL. Each 1-mL sample extract undergoing analysis should be spiked with 10 µL of the IS solution, resulting in a concentration of 40 ng/µL of each IS. Store away from any light source at ≤6 °C when not in use (−10 °C is recommended). When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.5.2 A more dilute internal standard solution may be employed to achieve lower detection levels.

7.6 GC/MS tune check solution – It is recommended that DFTPP solutions are prepared at 50 ng/µl or less in methylene chloride. Preparation in alternate solvents may result in degradation of DFTPP. The standard should also contain 50 ng/µL each of 4,4'- dichlorodiphenyltrichloroethane (DDT), pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Alternate concentrations may be used to compensate for different injection volumes if the total amount injected is 50 ng or less. Store away from any light source at ≤6 °C when not in use (−10 °C is recommended). If a more dilute IS is employed to achieve lower quantitation levels, a more dilute tune check solution may be necessary. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.7 Calibration standards – There are two types of calibration standards used for this method: standards made from the primary source for ICAL and continuing calibration verification (CCV), and standards made from a second source for initial calibration verification (ICV). When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.7.1 ICAL standards must be prepared at a minimum of five different concentrations from the secondary dilution of stock standards or from a premixed certified solution. Include a minimum of five different concentrations in the calibration for average response factor (RF) or linear (first-order) calibration models and six different concentrations for a quadratic (second-order) model with the low standard at or below the lower limit of quantitation (LLOQ) (see Sec. 9.9 and Method 8000). At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the DQOs of the project. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the GC/MS system. Each standard and/or series of calibration standards prepared at a given concentration should contain all the desired project-specific target analytes for which qualitative and quantitative results are to be reported by this method.

7.7.2 CCV standards should be prepared at a concentration near the mid-point of the ICAL from the same source as the ICAL or be a midlevel standard used in the ICAL.

7.7.3 Second source standards for ICV should be prepared at a concentration near the mid-point of the calibration range with standards from a second manufacturer or from a manufacturer's batch prepared independently from the batch used for calibration. A second lot number from the same manufacturer may be adequate to meet this requirement. The standard should contain all target analytes that will be reported for the project, if readily available. See Secs. 9.3.2 and 11.3.7 for guidance and acceptance limits.

7.7.4 It is the intent of EPA that all target analytes for a particular analysis be included in the calibration standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

7.7.5 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the IS solution prior to analysis. All standards should be stored away from any light source at ≤6 °C when not in use (−10 °C is recommended), and should be freshly prepared once a year, or sooner if check standards indicate a problem. The ICV and CCV standards should be prepared, as necessary, and stored at ≤6 °C.

7.8 Surrogate standards – The recommended surrogates are: phenol-d_6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14}. Other compounds with physicochemical properties more similar to the analyte classes of interest may be used as surrogates (e.g., deuterated monitoring compounds in the EPA Contract Laboratory Program's (CLP) current statement of work (SOW), which can be found in Reference 17 in Sec. 16), provided they are not found in field samples, can be unambiguously identified, and meet any applicable acceptance criteria described in Sec. 11 for ICAL and CCV. See Method 3500 for instructions on preparing the surrogate solutions. See Sec. 1.4.10 for surrogate suggestions for more volatile analytes.

NOTE: In the presence of samples containing residual chlorine, phenol-d_6 has been known to react to form chlorinated phenolic compounds. Sample preservation precautions outlined in Chapter Four should be used when residual chlorine is known to be present

in order to minimize degradation of deuterated phenols or any other susceptible target analyte.

NOTE: It is a good practice to analyze surrogates spiking solutions prior to their use to verify they are prepared at the correct concentrations. This is also recommended as a troubleshooting step when surrogate recoveries in blanks and LCSs are problematic.

7.9 Matrix spike and LCSs – See Method 3500 for instructions on preparing the matrix spike standard. Matrix spikes and LCSs should be prepared with target analytes from the same source as used for the ICAL standards to restrict the influence of standard accuracy on the determination of recovery through preparation and analysis. Matrix spike and LCS standards should be prepared with targets representative of the compounds being investigated. It is recommended that all reported target analytes be included in all LCS and matrix spike samples. For some applications, a limited set of representative analytes is acceptable.

NOTE: It is a good practice to also analyze/verify target compound spiking solutions prior to use or as a troubleshooting step when trying to determine the root cause of poor target compound spike recoveries in the LCS.

7.10 Solvents – Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents may be used. All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use, if necessary.

7.11 Carrier gas – Helium or hydrogen may be used. If hydrogen is used, analytical conditions may need to be adjusted for optimum performance, and calibration and all QC tests in Sec. 9.0 must be performed with hydrogen carrier gas. See Appendix B for guidance.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample collection, preservation and storage requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. Where such requirements are specified in a regulation, follow those requirements. In the absence of specific regulatory requirements, use the following information as guidance in determining the sample collection, preservation and storage requirements.

8.1 See the introductory material to Chapter Four, "Organic Analytes" for storage conditions and holding times.

8.2 Store the sample extracts at ≤6 °C (protected from light) in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling

and analysis plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results. Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and QC data should be maintained for reference or inspection.

9.2 Refer to Method 8000 for specific determinative method QC procedures. Refer to Method 3500 or 5000 for QC procedures to ensure the proper operation of the various sample preparation techniques. If an extract cleanup procedure is performed, refer to Method 3600 for the appropriate QC procedures. Any more specific QC procedures provided in this method will supersede those noted in Methods 3500, 3600, 5000 or 8000.

9.3 QC procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of RT windows, calibration verification and chromatographic analysis of samples. In addition, discussions regarding the instrument QC criteria listed below can be found in the referenced sections of this method, and a summary is provided in Table 6. Quantitative sample analyses should not proceed for those analytes that do not meet the QC acceptance criteria. However, analyses may continue for those analytes that exceed the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values.

9.3.1 The GC/MS must meet DFTPP criteria prior to the ICAL. See Secs. 11.3.1 and 11.4.1 for further details. Acceptance criteria are primarily intended to verify sensitivity, mass assignments and mass resolution under the same conditions used for analysis.

9.3.2 There must be an ICAL of the GC/MS system as described in Sec. 11.3. Prior to analyzing samples, verify the ICAL standards using a second source ICV standard, if readily available (See Secs. 7.7.1 and 11.3.7).

9.3.3 The GC/MS system must meet the CCV acceptance criteria in Sec. 11.4.

9.4 Initial demonstration of proficiency (IDP)

Prior to implementation of a method, each laboratory must perform an IDP consisting of at least four replicate reference samples spiked into a clean matrix taken through the entire sample preparation and analysis. Whenever a significant change to instrumentation or procedure occurs, the laboratory must demonstrate that acceptable precision and bias can still be obtained by the changed conditions. Also, whenever new staff members are trained, each analyst must perform an IDP for the method or portion of the method for which the analyst is responsible. This demonstration should document that the new analyst is capable of successfully following the SOP established by the laboratory and meeting any applicable acceptance criteria specified therein. Refer to Sec. 9.3 of Method 8000 for more information on how to perform an IDP.

9.5 Blanks

9.5.1 Before processing any samples, the analyst must demonstrate through the analysis of a MB or instrument blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. As a continuing check, each time a batch of samples is extracted, cleaned up, and analyzed, and when there is a change in reagents, a MB must be prepared and analyzed for the compounds of interest

as a safeguard against chronic laboratory contamination. MBs and field blanks must be carried through all stages of sample preparation and analysis. At least one MB or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

9.5.2 Blanks are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations/responses are ≥10X the blank). Other criteria may be used depending on the needs of the project.

9.5.3 If an analyte of interest is found in a sample in the batch near a concentration confirmed in the blank (refer to Sec. 9.5.2), the presence and/or concentration of that analyte should be considered suspect and may require qualification. Contaminants in the blank should meet most or all of the qualitative identifiers in Sec. 11.6 to be considered. Samples may require re-extraction and/or re-analysis if the blanks do not meet laboratory-established or project-specific criteria. Re-extraction and/or re-analysis is *not* necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project.

9.5.4 When new reagents or chemicals are received, the laboratory should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks should be prepared for each set of reagents.

9.5.5 The laboratory should not subtract the results of the MB from those of any associated samples. Such "blank subtraction" may lead to negative sample results. If the MB results do not meet the project-specific acceptance criteria and reanalysis is not practical, then the data user should be provided with the sample results, the MB results, and a discussion of the corrective actions undertaken by the laboratory.

9.6 Sample QC for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (i.e., precision, bias, and method sensitivity). At a minimum, this must include the analysis of a MB and LCS, and where practical, a matrix spike, and a duplicate in each preparation batch, as well as monitoring the recovery of surrogates. These QC samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on field samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples and project goals and should be addressed in the project planning documents. If samples are expected to contain target analytes, laboratories may use a matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, then laboratories should use a matrix spike and matrix spike duplicate pair. Consult Method 8000 for information on developing acceptance criteria for the matrix spike and matrix spike duplicate.

9.6.2 An LCS must be included with each preparation batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 A MB or instrument blank must be included with each analytical batch. MBs consist of an aliquot of clean (control) matrix similar to the sample and of a similar weight or volume. Other types of blanks (e.g., equipment rinsates, storage blanks, etc.) should be included when appropriate but they are distinct from MBs.

9.6.4 Also see Method 8000 for the details on carrying out QC procedures for preparation and analysis. In-house method performance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.7 Surrogates must be added to every blank, field sample, laboratory QC, and field QC. The laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in the laboratory's SOP or in an approved project plan.

9.8 Monitor IS responses to ensure sensitivity is maintained and to limit the potential for measurement bias of associated target analyte concentrations. IS responses in field samples are compared to responses of the same IS in the ICAL standards or CCV standards, with suggested acceptance criteria provided in Sec. 11.5.4.1. When IS responses fall outside the acceptance limit, further investigation is warranted and results may require qualification for detects and non-detects.

9.9 Lower limit of quantitation (LLOQ)

General guidance for LLOQ is provided in this section and in Method 8000. The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence. The LLOQ must be greater than or equal to the lowest point in the calibration curve. The laboratory shall establish the LLOQ at concentrations where both quantitative and qualitative requirements can consistently be met (see Sec. 11.6). The laboratory shall verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 - 2 times the established LLOQ. Additional LLOQ verifications may be useful on a project-specific basis if a matrix is expected to contain significant interferences at the LLOQ. The verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired decision level or regulatory action level based on the stated DQOs.

9.9.1 LLOQ Verification

9.9.1.1 The verification of LLOQs using spiked clean control material represents a best-case scenario because it does not evaluate the potential matrix effects of real-world samples. For the application of LLOQs on a project-specific basis, with established DQOs, a representative matrix-specific LLOQ verification may provide a more reliable estimate of the lower quantitation limit capabilities.

9.9.1.2 The LLOQ verification is prepared by spiking a clean control material with the analyte(s) of interest at 0.5 - 2 times the LLOQ concentration level(s). Alternatively, a representative sample matrix free of targets may be spiked with the analytes of interest at 0.5 - 2 times the LLOQ concentration levels. The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples. It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab should rotate the verification among similar analytical instruments such that all are included within three years.

9.9.1.3 Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria. This practice acknowledges the potential for greater uncertainty at the low end of the calibration curve. Practical, historically based LLOQ acceptance criteria should be determined once sufficient data points have been acquired.

9.9.2 Reporting concentrations below LLOQ – Concentrations that are below the established LLOQ may still be reported; however, these analytes must be qualified as estimated. The procedure for reporting analytes below the LLOQ should be documented in the laboratory's SOP or in a project-specific plan. Analytes below the LLOQ that are reported should meet most or all of the qualitative identification criteria in Sec. 11.6.

9.10 It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 CALIBRATION AND STANDARDIZATION

See Secs. 11.3 and 11.4 for information on calibration and standardization.

11.0 PROCEDURE

11.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis:

Matrix	Methods
Air (particulates and sorbent resin)	3542
Water (including TCLP leachates)	3510, 3511, 3520, 3535
Soil/sediment	3540, 3541, 3545, 3546, 3550, 3561
Waste	3540, 3541, 3545, 3546, 3550, 3561, 3580

11.2 Extract cleanup – Cleanup procedures may not be necessary for relatively clean sample matrices. Extracts from environmental and waste samples may require additional cleanup steps prior to analysis. The specific cleanup procedure used will depend upon the analytes of interest, the nature of the interferences, and the DQOs for the project. General guidance for sample extract cleanup is provided in this section and in Method 3600.

Extracts may be cleaned up by any of the following methods prior to GC/MS analysis:

Analytes of Interest	Methods
All base, neutral, and acid Priority Pollutants	3640
Aniline and aniline derivatives	3620
Chlorinated hydrocarbons	3620, 3640
Haloethers	3620, 3640
Nitroaromatics and cyclic ketones	3620, 3640
Nitrosamines	3610, 3620, 3640
Organochlorine pesticides	3610, 3620, 3630, 3640, 3660
Organophosphorus pesticides	3620, 3640
PAHs	3611, 3630, 3640
PCBs	3620, 3630, 3660, 3665
Petroleum waste	3611, 3650
Phenols	3630, 3640
Phthalate esters	3610, 3620, 3640

11.3 Initial calibration

Establish the GC/MS operating conditions, using the following settings as guidance:

Analytes of Interest	Methods
Mass range:	*m/z* 35 - 500
Acquisition rate:	Sufficient to acquire at least 5 (but preferably 10 or more) mass spectra across a peak
Initial temperature:	40 °C, hold for 4 minutes
Temperature program:	40 - 320 °C at 10 °C/minutes
Final temperature:	320 °C, hold until 2 min after benzo(g,h,i)perylene elutes
Injector temperature:	225 - 300 °C
Transfer line temperature:	250 - 300 °C
Source temperature:	According to manufacturer's specifications
Injector:	Grob-type, split/splitless
Injection volume:	0.5 - 2 µL
Carrier gas:	Hydrogen at 50 cm/second or helium at 30 cm/second
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

Split injection is allowed if the sensitivity of the MS is sufficient.

11.3.1 The GC/MS system must be hardware-tuned such that injecting 50 ng or less of DFTPP meets the manufacturer's specified acceptance criteria or as listed in Table 3. The tuning criteria as outlined in Table 3 were developed using quadrupole MS instrumentation with helium carrier gas and it is recognized that other tuning criteria may be more effective depending on the type of instrumentation (e.g., time-of-flight, ion trap, etc.). In these cases, it would be appropriate to follow the manufacturer's tuning instructions or some other consistent tuning criteria. However, no matter which tuning criteria are selected, sample analyses must be performed under the same conditions as the calibration standards.

Acceptable system performance may also be demonstrated by meeting manufacturer specifications for mass resolution, mass accuracy, and sensitivity using the internal calibrant (e.g., perfluorotributylamine, also known as PFTBA). Tuning the instrument should only be performed prior to initial calibration. Other reference compounds may also be appropriate for demonstrating acceptable MS performance depending on the system or conditions used for analysis (e.g., octafluoronaphthalene for negative ion CI). Regardless of how MS performance is evaluated, system calibration must not begin until performance criteria are met**,** and calibration standards and samples must be analyzed under the same conditions. If CI, SIM or tandem MS is used, the manufacturer's MS tuning criteria or one of the alternative procedures listed above may be substituted for the DFTPP tune verification requirement.

11.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach should be used: Use a single spectrum at the apex of the DFTPP peak, an average spectrum of the three highest points of the peak, or an average spectrum across the entire peak to evaluate the performance of the system. Background subtraction is allowed and is accomplished using a single mass spectrum acquired within 20 seconds of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument

background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

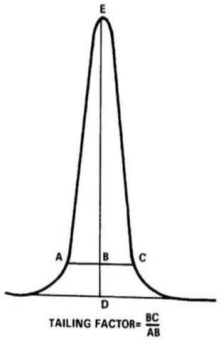
11.3.1.2 Use the DFTPP mass intensity criteria in the manufacturer's instructions as primary tuning acceptance criteria or those in Table 3 as default tuning acceptance criteria if the primary tuning criteria are not available. Alternatively, other documented tuning criteria may be used (e.g., CLP or Method 625), provided that method performance is not adversely affected. The analyst is always free to choose criteria that are more stringent than those included in this method or to use other documented criteria provided they are used consistently throughout the ICAL, calibration verification, and sample analyses.

NOTE: All subsequent standards, field samples, and QC samples associated with a DFTPP analysis must use identical MS instrument conditions with the exception of SIM analysis. DFTPP may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM. As an alternative to DFTPP for SIM analysis, the laboratory may use an alternate detector verification, such as PFTBA, or the manufacturer's recommended detector check.

NOTE: DFTPP tune checks are not appropriate for CI analysis or tandem MS analysis using SRM. However, the laboratory must demonstrate, prior to the ICAL, that the MS system achieves mass accuracy and mass resolution criteria specified by the instrument manufacturer for the perfluorotributylamine (PFTBA) internal calibrant or another appropriate chemical.

11.3.1.3 The GC/MS tune check solution should also be used to assess the GC column performance and injection port inertness. Degradation of DDT to dichlorodiphenyldichloroethylene (DDE) and dichlorodiphenyldichloroethane (DDD) should not exceed 20% (See Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{Tailing Factor} = \frac{BC}{AB}$$



where the peak is defined as follows (see Figure 1 for a full-page version of this image with additional information): AB is the line segment from the center to point A; AC is the width at 10% height; BC is the line segment from the center to point C; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

NOTE: Degradation and tailing factor checks are performed to verify injection port inertness and are important when the target list includes a broad range of analyte chemistries, especially reactive phenols and pesticides. These checks are optional when the analytes of interest are not subject to the same chromatography or reactivity problems (e.g., PAHs, PCBs) or

when using alternate MS techniques where routine full scan analysis is not performed (e.g., CI, MS/MS).

11.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require maintenance. It may also be necessary to cut off the first six to 12 inches of the capillary column to remove high boiling contaminants in the column. The use of a guard column (Sec. 6.1.6) between the injection port and the analytical column may help prolong analytical column performance life.

11.3.2 The base peak *m/z* of each target analyte and IS is appropriate for use as the primary *m/z* for quantitation (see Table 1), but another prominent *m/z* in the mass spectrum may also be used for quantitation provided it is used consistently. If interferences are noted, use the next most intense ion as the quantitation ion (e.g., for 1,4-dichlorobenzene-d_4, use *m/z* 152 for quantitation).

11.3.3 Analyze a consistent volume of each calibration standard (i.e., containing the compounds for quantitation and the appropriate surrogates and ISs) and tabulate the response of the primary ion against the concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards must be analyzed with the low standard at or below the LLOQ (see Secs. 7.7, 9.9 and Method 8000). Alternate injection volumes may be used if the applicable QC requirements for using this method are met. The injection volume must be the same for the analysis of all standards and sample extracts. Figure 2 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

NOTE: LLOQs should be established at concentrations where both quantitative and qualitative requirements can be consistently and reliably met (see Secs. 9.9 and 11.6). Target analyte peaks in the calibration standard at the LLOQ should be visually inspected to ensure that peak signal is adequately distinguishable from background and meets the qualitative requirements outlined in 11.6.

11.3.4 Initial calibration calculations

Tabulate the response of the quantitation ions (see Table 1 for suggested ions) against the concentration for each target analyte and each IS. Calculate RFs for each target analyte relative to one of the ISs as follows:

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

where:

A_s = Peak response of the analyte or surrogate
A_{is} = Peak response of the IS
C_s = Concentration of the analyte or surrogate (e.g., in µg/ml)
C_{is} = Concentration of the IS (e.g., in µg/ml)

11.3.4.1 Calculate the mean RF and the relative standard deviation (RSD) of the RFs for each target analyte using the following equations:

$$\text{mean RF} = \overline{\text{RF}} = \frac{\sum_{i=1}^{n} \text{RF}_i}{n} \qquad \text{RSD} = \frac{\text{SD}}{\overline{\text{RF}}} \times 100 \qquad \text{SD} = \sqrt{\frac{\sum_{i=1}^{n} (\text{RF}_i - \overline{\text{RF}})^2}{n - 1}}$$

where:

RF_i = RF for each of the calibration standards
$\overline{\text{RF}}$ = Mean RF for each compound from the ICAL
n = Number of calibration standards (e.g., 5)
SD = Standard deviation

11.3.4.2 The RSD should be ≤20% for each target analyte (see Sec. 11.3.5). Table 4 contains minimum RFs that may be used as guidance in determining if the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 4 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information is provided as guidance only. The laboratory should establish procedures in its SOP (e.g., laboratory established minimum RFs, signal-to-noise (S/N) checks, etc.) to ensure that the instrument is working properly and that calibration standards were correctly prepared.

NOTE: For a target analyte, whose RF <0.01 (response of peak is <1/100 the response of the IS), it is recommended that its concentration in relation to other analytes be increased to make the response more comparable to other analytes.

11.3.5 Linearity of target analytes – If the RSD of any target analyte is 20% or less, then the RF is assumed to be constant over the calibration range, and the average RF may be used for quantitation (Sec. 11.7.2). The average RF should not be used for compounds that have an RSD greater than 20% unless the concentration is reported as estimated.

11.3.5.1 If the RSD of any target analyte is greater than 20%, refer to Sec. 11.5 in Method 8000 for guidance in selecting an alternate calibration model. One of the options must be applied to GC/MS calibration in this situation, or a new ICAL must be performed.

11.3.5.2 When the RSD exceeds 20%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

11.3.5.3 If more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard error (RSE) ≤20%) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Correct the source of the problem; then repeat the calibration procedure beginning with Sec. 11.3. If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated. In order to report non-detects, it must be demonstrated that there is sufficient accuracy to detect the failed compounds at

the applicable LLOQ (see Secs. 11.3.6 for refitting standards and 11.4.4.2 for CCV). Refer to Method 8000 for further discussion of RSE. Example RSE calculations can be found in Reference 19.

11.3.5.4 Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project. The analyst should strive to place more emphasis on meeting the calibration criteria for those compounds that are critical project compounds, rather than meeting the criteria for those less important compounds. It is not necessary to meet criteria for compounds that will not be reported.

NOTE: It is considered inappropriate once the calibration models have been finalized to select an alternate fit solely to pass the recommended QC criteria for samples and associated QC on a case-by-case basis.

11.3.6 Calibration, especially when using linear regression models, has the potential for a significant bias at the lower portion of the calibration curve. The lowest calibration point should be recalculated (not reanalyzed) using the final calibration curve in which this standard is used (i.e., re-fitting the response from the low concentration calibration standard back into the curve). See Method 8000 for additional details. The recalculated concentration of the low calibration point (especially where linear regression fits are used) should be within ±50% of the standard's true concentration, and the recalculated concentrations of any calibration standards above the LLOQ should be within ±30%. Alternate criteria may be applied depending on the needs of the project; however, those criteria should be clearly defined in a laboratory SOP or a project-specific QAPP. Analytes which do not meet the re-fitting criteria should be evaluated for corrective action. If a failure occurs in the low point and it is equivalent to the LLOQ, the analyte should be reported as estimated near that concentration or the LLOQ should be reestablished at a higher concentration.

11.3.7 ICV - Prior to analyzing samples, verify the ICAL using a standard obtained from a second source to the calibration standard, if possible, such as a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration, if readily available. Suggested acceptance criteria for the analyte concentrations in this standard are 70 - 130% of the expected analyte concentration(s). Alternative criteria may be appropriate based on project-specific DQOs. Quantitative sample analyses should not proceed for those analytes that do not meet the ICAL verification criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values.

11.3.8 Additional considerations for SIM and SRM analysis

SIM and SRM may be useful for applications requiring quantitation limits below the normal range of electron impact quadrupole mass spectrometry, and both are allowable options for this method. Using the primary *m/z* (or product ion for SRM detectors) for quantitation and at least one secondary *m/z (*or product ion*)* for confirmation, set up the collection groups based on their chromatographic RTs. The selected *m/z* (or product ion) values should include any mass defect noted in the target analyte mass spectra acquired on the instrument, usually less than 0.2 amu. The dwell time for each ion may be automatically calculated by the instrument software or may be calculated based on the peak widths of the analytes of interest, the number of spectra

needed to be acquired across each peak, and the number of concurrent ions that need to be acquired in each segment. When fewer masses are monitored in each segment, the acquisition time for each mass can be increased, thereby increasing the sensitivity of the system. The total cycle time for the MS should be short enough that at least five but preferably ten or more spectra are acquired per chromatographic peak.

When samples are analyzed in SIM or SRM mode (generally to achieve lower reporting limits or reduce interferences) the following best practices are recommended:

- At least two ions should be monitored for each target analyte and use the mid-point of the calibration curve to establish proper ion ratios for each compound. The ratios of primary and secondary ions are the only qualitative tool available in SIM and SRM runs (other than RT), which increases their importance in proper identification. When interferences are expected or observed in a given matrix, acquiring multiple secondary ions may aid in qualitative identification.

- All monitored ions must be correctly integrated in order to achieve proper ion ratios. The primary/secondary ion ratios and the reference mass spectrum should be updated from the mid-point ICAL standard.

  Additional guidance for performing SIM analyses, in particular for PAHs and phenol target analyte compounds, can be found in the most recent CLP semivolatile organic methods SOW. See the SIM sections from the following CLP SOW for further details: EPA CLP Multi-concentration Organics Analysis, SOM01.2 SOW (Reference 12), or the current version of Method 625.

11.4 CCV – A CCV standard must be analyzed at the beginning of each 12-hour analytical period prior to any sample analysis.

11.4.1 Daily analysis of the GC/MS tune check solution is no longer required as part of the CCV. The analyst should, however, closely monitor chromatography as well as target and IS responses in the CCV for deterioration in the system. See the note in Sec. 11.4.4.2 for additional detail.

11.4.2 The ICAL (Sec. 11.3) for each compound of interest must be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for analysis of ICAL standards and samples. This is accomplished by analyzing a calibration standard (containing all the compounds that will be reported) prepared from the same stock solutions or source materials used for the ICAL standards at a concentration near the midpoint of the calibration range of the GC/MS. The results must be compared against the most recent ICAL curve and should meet the verification acceptance criteria provided in Secs. 11.4.4 through 11.4.6.

NOTE: A CCV may be omitted if samples are analyzed within 12 hours of ICAL, and the injection of the last ICAL standard may be used as the starting time reference for evaluation.

11.4.3 A MB or instrument blank must be analyzed after the CCV and prior to samples in order to ensure that the total system (i.e., introduction device, transfer lines and GC/MS system) is free of contaminants. If the MB indicates contamination, then it may be appropriate to analyze an instrument blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Method 8000 for information regarding MB performance criteria.

11.4.4 CCV standard criteria

11.4.4.1 The calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20% of the expected value.

NOTE: For the RF calibration model, % difference (%D) between the calculated RF of an analyte in the calibration verification standard and the RF_{avg} of that analyte from the ICAL is the same value as % drift for calculated versus expected concentration. Refer to Method 8000 for guidance on calculating %D and % drift.

11.4.4.2 If the %D or percent drift for a compound is ≤20%, then the ICAL for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. The analyst should strive to place more emphasis on meeting the CCV criteria for those compounds that are critical to the project. If the criterion is not met (i.e., greater than ±20%D or drift) for more than 20% of the compounds included in the ICAL (or more than 20% of those that will be reported), then corrective action must be taken prior to the analysis of samples. Target analytes that do not meet the CCV criteria and are reported in the associated samples must be qualified to indicate the reported concentrations are potentially estimated or biased values. In cases where compounds fail low, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the LLOQ or project specific level of interest (e.g., by calibrating below the established LLOQ to confirm the non-detect, or by analyzing a standard near that level to confirm the analyte could be qualitatively identified if it were present [See Sec. 11.7 of Method 8000]). Alternatively, the non-detect could be qualified or the LLOQ raised to a higher level. In cases where compounds fail high in the CCV and are not found in the associated field samples, they may be reported without qualification.

NOTE: Daily tailing and degradation checks are good indicators of reactivity in the system and the need for maintenance. Because these are no longer required daily, the analyst must closely monitor responses and chromatography in the CCV for signs that the system is too reactive for analysis to continue (e.g., losses of reactive analytes, unusual tailing, loss of resolution). If significant losses of target analytes/ISs occur (<50% recovery) or if significant degradation of the chromatography occurs (tailing factor >2), system maintenance must be performed or the analyst must demonstrate there is adequate sensitivity at the LLOQ.

11.4.4.3 Problems similar to those listed under ICAL could affect the ability to pass the CCV standard analysis. If the problem cannot be corrected by other measures, a new ICAL must be generated. The CCV criteria must be met before sample analysis begins.

11.4.5 IS RT – If the absolute RT for any IS changes by more than 30 seconds from that in the mid-point standard level of the most recent ICAL sequence (or the most recent CCV), then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.4.6 IS responses – In order to demonstrate continued stability of the measurement system after ICAL, IS responses in the CCVs must be evaluated by comparing them to the responses of the same ISs in the ICAL standard(s). If the response of an IS changes by more than a factor of 2 (50 - 200%) relative to the response of that IS in the mid-point ICAL standard or the average of responses in the suite of ICAL standards (as defined in the laboratory's SOP), then corrective actions should be taken as needed. These corrective actions may include but are not limited to replacing and/or reanalyzing the CCV standard or retuning the MS and re-calibrating. When IS responses do not meet these criteria, system sensitivity may have been compromised, and sample reanalysis is recommended, especially if any action limits for the project are near the LLOQ.

11.5 GC/MS analysis of samples

11.5.1 It is recommended that sample extracts be screened on a GC/flame ionization detector (FID) or GC/photo ionization detector (PID) using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. GC/MS calibration verification criteria must be met before analyzing samples.

11.5.2 Allow the sample extract to warm to room temperature. Prior to analysis, add 10 µL of the IS solution to the 1 mL of concentrated sample extract obtained from sample preparation.

11.5.3 Inject an aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 11.3). The injection volume must be the same volume that was used for the calibration standards.

11.5.4 If the concentration for any analyte exceeds the ICAL range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional IS solution must be added to the diluted extract to maintain the same concentration as in the calibration standards. Secondary ion quantitation should be used only when there are sample interferences with the primary ion.

11.5.4.1 IS responses (area counts) and RTs must be monitored in all samples, spikes and blanks to effectively check method performance and to anticipate the need for system maintenance. If the response of the primary *m/z* for any of the ISs in samples, spikes, and blanks changes by a factor of two (from 50% to 200%) from the responses determined in the mid-point standard level of the most recent ICAL sequence or CCV standard (whichever was analyzed more recently), corrective action should be taken. The samples, spikes, or blanks should be reanalyzed or the associated data should be qualified.

11.5.4.2 When ions from a compound in the sample saturate the detector, this analysis should be followed by the analysis of an instrument blank consisting of clean solvent. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences. Contamination from one sample to the next on the instrument usually takes place in the syringe. If adequate syringe washes are employed, then carryover from high concentration samples can usually be avoided.

11.5.4.3 It is recommended to target the response in the mid to upper half of calibration range for target analytes that exceeded the calibration range and required dilution.

11.6 Analyte identification

11.6.1 Target Identification - The qualitative identification of compounds determined by this method is based on RT and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. Compounds are identified when the following criteria are met.

11.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other.

11.6.1.2 The RT should be within ±10 seconds of the RT for this analyte in the CCV run at the beginning of the 12-hour period (delta RT 0.17 minute) or within ±10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds). Chromatograms should be carefully inspected to minimize the occurrence of both false positive and false negative results. If the RT for the IS has shifted, the sample should be inspected for similar shifts for the associated target analytes. If RT drift is significant, relative retention time (RRT) may be used as an alternative to delta RTs. See Sec. 11.4. of Method 8000 for additional information.

NOTE: Some analytes such as phenols may have RT shifting that is much greater than the associated IS (greater than ± 10 seconds relative to the IS shift) and is still the target analyte. In those cases, it may be more useful to compare the delta RT with compounds that have similar chemistries (e.g., phenolic surrogates) to help identify the target. Also, dilutions or spiked samples are recommended to help minimize the effects of matrix on the elution of the target and assist in target identification.

11.6.1.3 The relative intensities of the qualifier ion(s) (i.e., secondary characteristic ions, or additional monitored MS/MS transitions) should agree within 30% of the relative intensities of these ions in the reference spectrum. For example, for a qualifier ion with response of 50% of the quantitation ion in the reference spectrum, the corresponding qualifier ion ratio in a sample mass spectrum can range between 20% and 80%. The reference mass spectrum used for this comparison should be generated by the laboratory using the conditions of this method (typically a mid-level calibration standard). Use professional judgment in interpretation where interferences are observed. Qualitative identification of sample mass spectra not acquired in limited ion acquisition modes (i.e. SIM or SRM) may also be supported by comparison to a reference library as described in Sec. 11.6.2.

11.6.1.4 Unresolved structural isomers with similar mass spectra are identified as isomeric pairs. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is ≤ 50% of the average of the two peak heights, or 1−[valley height]/[average peak height] is ≥50%). The resolution should be verified on the mid-point concentration of the ICAL as well as the laboratory-designated CCV level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and

benzo(k)fluoranthene). It is important to check the separation of structural isomers in the ICV and the daily CCV check standards to verify if the instrument performance is adequate regarding separation of compounds of interest which are structural isomers.

11.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

11.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

11.6.2 Tentative Identification - For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the Resource Conservation and Recovery Act (RCRA) permit or waste delisting requirements may require the reporting of non-target analytes. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

(1) Major ions in the library reference spectrum (ions greater than 10% of the most abundant ion) are present in the sample spectrum at similar relative intensities.

(2) The molecular ion in the library reference spectrum is present in the sample spectrum. If the molecular ion is not present, carefully review library matches in order to avoid misidentification.

(3) Major ions present in the sample spectrum but not in the reference spectrum are reviewed to determine whether they may be contributed by co-eluting compounds.

(4) Ions present in the reference spectrum but not in the sample mass spectra are reviewed for unintended subtraction. Data system library reduction programs can sometimes create these discrepancies.

(5) Mass spectral library search algorithms typically assign a match factor to the peak identity based on comparison of an unknown mass spectrum to library spectra. For spectra meeting the above conditions, match factors greater than 0.8 (80%) may be considered confirming evidence. Where a known limitation in data collection is identified, e.g., the presence of an incompletely resolved spectral interference, a lower match factor may be considered confirmatory. For multiple library spectra with similar match factors (e.g., for hydrocarbons with low abundance molecular ions, or structural isomers), the tentative identification assigned to the unknown may be better represented as a more generic structure

(e.g., unknown hydrocarbon, C4 benzene structural isomer). See Reference 18 in Sec. 16 for more information.

11.7 Quantitation

11.7.1 Once a target compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP. The IS used should be the one nearest the RT of that of a given analyte.

11.7.1.1 It is highly recommended to use the integration produced by the software if the integration is correct because the software should produce more consistent integrations than an analyst will manually. However, manual integrations may be necessary when the software does not produce proper integrations because baseline selection is improper; the correct peak is missed; a co-elution is integrated; the peak is partially integrated; etc. The analyst is responsible for ensuring that the integration is correct whether performed by the software or done manually.

11.7.1.2 Manual integrations should not be substituted for proper maintenance of the instrument or setup of the method (e.g., RT updates, integration parameter files, etc.). The analyst should seek to minimize manual integration by properly maintaining the instrument, updating RTs, and configuring peak integration parameters.

11.7.2 If the RSD is 20% or less, then the RF calibration model is acceptable for ICAL (Sec. 11.3.4). See Method 8000 for the equations describing IS calibration and either linear or non-linear calibrations.

11.7.3 Where applicable, the concentrations of any non-target analytes identified in the sample (Sec. 11.6.2) may be estimated. The same formula (as in Sec. 11.3.4) should be used with the following modifications: The responses A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

11.7.4 The resulting concentration should be reported indicating that the value is an estimate. Use the nearest IS free of interferences.

11.7.5 Quantitation of multicomponent compounds to estimate total concentrations (e.g., toxaphene, Aroclors, chlordane) is beyond the scope of Method 8270. When 8270 is used to confirm PCBs, it is primarily a mass spectral (qualitative) confirmation of the isomer or level of chlorination where individual congeners cannot be resolved. Normally, quantitation is performed using a GC/electron capture detector (ECD), for example by Methods 8081 or 8082. Individual components (e.g., a subset of PCB congeners) may be determined with this method provided sensitivity is sufficient for the data application and interference from other components is minimal. For PCBs, Cochran and Frame (Reference 14 in Sec. 16) provide a literature review of analytical chemistry of PCB congeners, including identification of congeners that coelute under different chromatographic conditions. Table 1 of Parris, et al. (1996) and Table 8 of Kucklick, et al. (2013) (References 15 and 16 in Sec. 16) provide PCB congener lists that might be determined by 8270 under the described conditions, along with identified coeluting congeners. Refer to Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as Aroclors, toxaphene, and chlordane. Refer to Method 680 for information related to calibration and quantitation of PCBs as

homolog groups. Refer to Method 1668 for more information related to quantitation of PCBs as individual congeners. Refer to Method 8276 for guidance on measurement of toxaphene by negative ion chemical ionization GC/MS.

11.7.6 Quantitation of multicomponent parameters such as diesel range organics (DROs) and total petroleum hydrocarbons (TPH) using the IS technique described in this method is not recommended. Typically, quantitation for these parameters is performed using Method 8015 with GC/FID analysis; however, it is acceptable to use the total ion chromatogram that is generated from this method with external standard calibration to quantitate such parameters. External standard calibration is recommended in order to reduce the need to subtract area contributed by multiple non-target peaks (such as the ISs) in the TPH chromatogram. See Sec. 11.4.2 in Method 8000 and Method 8015 for additional guidance.

12.0 DATA ANALYSIS AND CALCULATIONS

See Sec. 11.7 and Method 8000 for information on data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation. The performance data provided in Reference 13 in Sec. 16 are for guidance purposes only.

13.2 Single laboratory initial demonstration of capability data were generated from five replicate measurements using a modified continuous liquid-liquid extractor (Method 3520) with hydrophobic membrane. In this case, only a single acid pH extraction was performed using the CLP calibration criteria and the applicable CLP target analytes. These data are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry. Laboratories should generate their own acceptance criteria depending on the extraction and instrument conditions. See Method 8000 for more detailed guidance.

13.3 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of gas chromatographic performance. These recoveries (using Method 3510 extraction) are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry. These data are provided for guidance purposes only.

13.4 Method performance data using Method 3541 (i.e., automated Soxhlet extraction) are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately one hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three extractions were performed and each extract was analyzed by GC/MS following Method 8270. The low recovery of the

more volatile compounds is probably due to volatilization losses during equilibration. These data as listed were taken from Reference 5 and are provided for guidance purposes only.

13.5 Surrogate precision and accuracy data are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by this method (i.e., 8270). These data are provided for guidance purposes only.

13.6 Single-laboratory precision and bias data using Method 3545 (i.e., pressurized fluid extraction) for semivolatile organic compounds are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (i.e., 250, 2500, and 12,500 µg/kg) on three types of soil (i.e., clay, loam, and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction (ASE) system and by the Perstorp Environmental Soxtec™ (i.e., automated Soxhlet). The data are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry represent seven replicate extractions and analyses for each individual sample and were taken from Reference 7. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam 98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low – 101.2%, mid – 97.2% and high – 99.2%. These data are provided for guidance purposes only.

13.7 Single-laboratory precision and bias data using Method 3561 (i.e., supercritical fluid extraction (SFE) extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (i.e., EC-1, a lake sediment from Environment Canada and HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions ranged from 85 to 148%, with an overall average of 100%, based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions ranged from 73 to 133%, with an overall average of 92%, based on the certified value for the marine sediment. The data are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry and were taken from Reference 8. These data are provided for guidance purposes only.

13.8 Single laboratory precision and accuracy using Method 3561 (i.e., SFE extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for 12 of the method analytes by the extraction of a certified reference material (i.e., a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions ranged from 60 to 122%, with an overall average of 89%, based on the certified value. The instrument conditions that were utilized to extract a 3.4 g sample were as follows: Pressure - 300 atm; time - 60 min; extraction fluid - CO_2; modifier - 10% 1:1 (v/v) methanol/methylene chloride; oven temperature - 80 °C; restrictor temperature - 120 °C; and, trapping fluid - chloroform (methylene chloride has also been used). The data are located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry and were taken from Reference 9. These data are provided for guidance purposes only.

13.9 Tables located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry contain single-laboratory precision and accuracy data for SPE of TCLP buffer solutions spiked at two levels and extracted using Method 3535. These data are provided for guidance purposes only.

13.10 The table located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry contains multiple-laboratory data for SPE of spiked TCLP soil leachates extracted using Method 3535. These data are provided for guidance purposes only.

13.11 Tables located at https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry contain single-laboratory PAH recovery data for microwave extraction of contaminated soils and standard reference materials using Method 3546. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult http://www.acs.org/content/dam/acsorg/about/governance/committees/chemicalsafety/publications/less-is-better.pdf.

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available at: http://www.labsafety.org/FreeDocs/WasteMgmt.pdf.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS[a]

Compound[b]	Primary Ion	Secondary Ions
Acenanaphthene-d_{10} (IS)	164	162, 160
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
2-Acetylaminofluorene	181	180, 223, 152
1-Acetyl-2-thiourea	118	43, 42, 76
Aldrin	66	263, 220
2-Aminoanthraquinone	223	167, 195
Aminoazobenzene	197	92, 120, 65, 77
4-Aminobiphenyl	169	168, 170, 115
Anilazine	239	241, 143, 178, 89
Aniline	93	66, 65
o-Anisidine	108	80, 123, 52
Anthracene	178	176, 179
Aramite	185	191, 319, 334, 197, 321
Atrazine	200	173, 215
Azinphos-methyl (Guthion)	160	132, 93, 104, 105
Azobenzene	182	105, 77
Barban	222	51, 87, 224, 257, 153
Benzaldehyde	77	105, 106
Benz(a)anthracene	228	229, 226
Benzidine	184	92, 185
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	122	105, 77
Benzo(g,h,i)perylene	276	138, 277
Benzo(a)pyrene	252	253, 125
Benzo(e)pyrene	252	253, 125
p-Benzoquinone	108	54, 82, 80
Benzyl alcohol	108	79, 77
α-BHC	183	181, 109
β-BHC	181	183, 109
γ-BHC (Lindane)	183	181, 109
δ-BHC	183	181, 109
1,1'-Biphenyl	154	153, 76
Bis(2-chloro-1-methylethyl)ether	45	77, 121
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-ethylhexyl)phthalate	149	167, 279
Bromoxynil (Brominal)	277	279, 88, 275, 168
4-Bromophenyl phenyl ether	248	250, 141
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55, 56
Captafol	79	77, 80, 107
Captan	79	149, 77, 119, 117
Carbaryl (Sevin)	144	115, 116, 201
Carbazole	167	166, 139

Compound[b]	Primary Ion	Secondary Ions
Carbofuran (Furaden)	164	149, 131, 122
Carbophenothion	157	97, 121, 342, 159, 199
Chlorfenvinphos	267	269, 323, 325, 295
4-Chloroaniline	127	129, 65, 92
Chlorobenzilate	251	139, 253, 111, 141
5-Chloro-2-methylaniline	106	141, 140, 77, 89
4-Chloro-3-methylphenol	107	144, 142
1-Chloronaphthalene	162	127, 164
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
4-Chlorophenyl phenyl ether	204	206, 141
4-Chloro-1,2-phenylenediamine	142	144, 80
4-Chloro-1,3-phenylenediamine	142	144, 80
Chrysene	228	226, 229
Chrysene-d_{12} (IS)	240	120, 236
Coumaphos	362	226, 210, 364, 97, 109
p-Cresidine	122	94, 137, 77, 93
Crotoxyphos	127	105, 193, 166
2-Cyclohexyl-4,6-dinitrophenol	231	185, 41, 193, 266
4,4'-DDD	235	237, 165
4,4'-DDE	246	248, 176
4,4'-DDT	235	237, 165
Demeton-O	88	89, 60, 61, 115, 171
Demeton-S	88	60, 81, 89, 114, 115
Diallate (cis or trans)	86	234, 43, 70
2,4-Diaminotoluene	121	122, 94, 77, 104
Dibenz(a,j)acridine	279	280, 277, 250
Dibenz(*a,h*)anthracene	278	139, 279
Dibenzo(a,e)pyrene	302	151, 150, 300
Dibenzofuran	168	139
1,2-Dibromo-3-chloropropane (DBCP)	75	155, 157
Di-*n*-butyl phthalate	149	150, 104
Dichlone	191	163, 226, 228, 135, 193
1,2-Dichlorobenzene	146	148, 111
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene-d_4 (IS)	152	150, 115
3,3'-Dichlorobenzidine	252	254, 126
2,4-Dichlorophenol	162	164, 98
2,6-Dichlorophenol	162	164, 98
Dichlorovos (DDVP, Dichlorvos)	109	185, 79, 145
Dicrotophos	127	67, 72, 109, 193, 237
Dieldrin	79	263, 279
Diethyl phthalate	149	177, 150
Diethyl sulfate	139	45, 59, 99, 111, 125
Diethylstilbestrol	268	145, 107, 239, 121, 159
Dimethoate	87	93, 125, 143, 229
3,3'-Dimethoxybenzidine	244	201, 229
Dimethyl aminoazobenzene	225	120, 77, 105, 148, 42
Dimethyl phthalate	163	194, 164
7,12-Dimethylbenz(a)anthracene	256	241, 239, 120

Compound[b]	Primary Ion	Secondary Ions
3,3'-Dimethylbenzidine	212	106, 196, 180
2,4-Dimethylphenol	122	107, 121
α,α-Dimethylphenylamine	58	91, 65, 134, 42
1,2-Dinitrobenzene	168	50, 63, 74
1,3-Dinitrobenzene (1,3-DNB)	168	76, 50, 75, 92, 122
1,4-Dinitrobenzene	168	75, 50, 76, 92, 122
4,6-Dinitro-2-methylphenol	198	51, 105
2,4-Dinitrophenol	184	63, 154
2,6-Dinitrophenol	162	164, 126, 98, 63
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
Dinocap	69	41, 39
Dinoseb (DNBP)	211	163, 147, 117, 240
Di-n-octyl phthalate	149	167, 43
1,4-Dioxane	88	43, 58
5,5-Diphenylhydantoin	180	104, 252, 223, 209
1,2-Diphenylhydrazine	77	105, 182
Diphenylamine	169	168, 167
Disulfoton	88	97, 89, 142, 186
Endosulfan I	195	339, 341
Endosulfan II	337	339, 341
Endosulfan sulfate	272	387, 422
Endrin	263	82, 81
Endrin aldehyde	67	345, 250
Endrin ketone	317	67, 319
EPN	157	169, 185, 141, 323
Ethion	231	97, 153, 125, 121
Ethyl carbamate	62	44, 45, 74
Ethyl methanesulfonate	79	109, 9745, 65
Famphur	218	125, 93, 109, 217
Fensulfothion	293	97, 308, 125, 292
Fenthion	278	125, 109, 169, 153
Fluchloralin	306	63, 326, 328, 264, 65
Fluoranthene	202	101, 203
Fluorene	166	165, 167
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Heptachlor	100	272, 274
Heptachlor epoxide	353	355, 351
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Hexachlorophene	196	198, 209, 211, 406, 408
Hexachloropropene	213	211, 215, 117, 106, 141
Hexamethylphosphoramide (HPMA)	135	44, 179, 92, 42
Hydroquinone	110	81, 53, 55
Indeno(1,2,3-cd)pyrene	276	138, 277
Isodrin	193	66, 195, 263, 265, 147
Isophorone	82	95, 138
Isosafrole	162	131, 104, 77, 51

Compound[b]	Primary Ion	Secondary Ions
Kepone	272	274, 237, 178, 143, 270
Leptophos	171	377, 375, 77, 155, 379
Malathion	173	125, 127, 93, 158
Maleic anhydride	54	98, 53, 44
Mestranol	277	310, 174, 147, 242
Methapyrilene	97	50, 191, 71
Methoxychlor	227	228, 152, 114, 274, 212
Methyl methanesulfonate	80	79, 65, 95
Methyl parathion	109	125, 263, 79, 93
3-Methylcholanthrene	268	252, 253, 126, 134, 113
4,4'-Methylenebis(2-chloroaniline)	231	266, 268, 140, 195
4,4'-Methylenebis(N,N-dimethyl-aniline)	254	253, 134
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
2-Methylphenol	107	108, 77, 79, 90
3/4-Methylphenol[c]	107	108, 77, 79, 90
Mevinphos	127	192, 109, 67, 164
Mexacarbate	165	150, 134, 164, 222
Mirex	272	237, 274, 270, 239, 235
Monocrotophos	127	192, 67, 97, 109
Naled	109	145, 147, 301, 79, 189
Naphthalene	128	129, 127
Naphthalene-d_8 (IS)	136	68
1,4-Naphthoquinone	158	104, 102, 76, 50, 130
1-Naphthylamine	143	115, 89, 63
2-Naphthylamine	143	115, 116
Nicotine	84	133, 161, 162
5-Nitroacenaphthene	199	152, 169, 141, 115
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	108, 92
4-Nitroaniline	138	65, 108, 92, 80, 39
5-Nitro-*o*-anisidine	168	79, 52, 138, 153, 77
Nitrobenzene	77	123, 65
Nitrobenzene-d_5 (surr)	82	128, 54
4-Nitrobiphenyl	199	152, 141, 169, 151
Nitrofen	283	285, 202, 139, 253
2-Nitrophenol	139	109, 65
4-Nitrophenol	139	109, 65
4-Nitroquinoline-1-oxide	174	101, 128, 75, 116
N-Nitrosodi-*n*-butylamine	84	57, 41, 116, 158
N-Nitrosodiethylamine	102	42, 57, 44, 56
N-Nitrosodimethylamine	42	74, 44
N-Nitrosodiphenylamine	169	168, 167
N-Nitrosodi-*n*-propylamine	70	42, 101, 130
N-Nitrosomethylethylamine	88	42, 43, 56
N-Nitrosomorpholine	56	116, 86
N-Nitrosopiperidine	114	42, 55, 56, 41
N-Nitrosopyrrolidine	100	41, 42, 68, 69
5-Nitro-o-toluidine	152	106, 79
Octamethylpyrophosphoramide	135	44, 199, 286, 153, 243
4,4'-Oxydianiline	200	108, 171, 80, 65

Compound[b]	Primary Ion	Secondary Ions
Parathion	109	97, 291, 139, 155
Pentachlorobenzene	250	252, 108, 248, 215, 254
Pentachloronitrobenzene	237	142, 214, 249, 295, 265
Pentachlorophenol	266	264, 268
Perylene	252	253, 125
Perylene-d_{12} (IS)	264	260, 265
Phenacetin	108	180, 179, 109, 137, 80
Phenanthrene	178	179, 176
Phenanthrene-d_{10} (IS)	188	94, 80
Phenobarbital	204	117, 232, 146, 161
Phenol	94	65, 66
Phenol-d_6 (surr)	99	42, 71
1,4-Phenylenediamine	108	80, 53, 54, 52
Phorate	75	121, 97, 93, 260
Phosalone	182	184, 367, 121, 379
Phosmet (Imidan)	160	77, 93, 317, 76
Phosphamidon	127	264, 72, 109, 138
Phthalic anhydride	104	76, 50, 148
2-Picoline	93	66, 92
Piperonyl sulfoxide	162	135, 105, 77
Pronamide (Kerb)	173	175, 145, 109, 147
Propylthiouracil	170	142, 114, 83
Pyrene	202	200, 203
Pyridine	79	52, 50
3-(Chloromethyl)pyridine hydrochloride	92	127, 129, 65, 39
Resorcinol	110	81, 82, 53, 69
Safrole	162	104, 77, 103, 135
Strychnine	334	334, 335, 333
Sulfallate	188	88, 72, 60, 44
Terbufos	231	57, 97, 153, 103
Terphenyl-d_{14} (surr)	244	122, 212
1,2,4,5-Tetrachlorobenzene	216	214, 179, 108, 143, 218
2,3,4,6-Tetrachlorophenol	232	131, 230, 166, 234, 168
Tetrachlorvinphos (Stirophos, Gardona)	329	109, 331, 79, 333
Tetraethyl dithiopyrophosphate	97	202, 238, 266
Tetraethyl pyrophosphate (TEPP)	99	155, 127, 81, 109
Thionazine	107	96, 97, 143, 79, 68
Thiophenol (Benzenethiol)	110	66, 109, 84
2,4-Toluene diisocyanate	174	145, 173, 146, 132, 91
o-Toluidine	106	107, 77, 51, 79
Toxaphene	159	231, 233
2,4,6-Tribromophenol (surr)	330	332, 141
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	198, 97, 132, 99
2,4,6-Trichlorophenol	196	198, 200
O,O,O-Triethyl phosphorothioate	198	121, 93
Trifluralin (Treflan)	306	43, 264, 41, 290
Trimethyl phosphate	110	79, 95, 109, 140
2,4,5-Trimethylaniline	120	135, 134, 91, 77
1,3,5-Trinitrobenzene	75	74, 213, 120, 91, 63
Tris(2,3-dibromopropyl)phosphate	201	137, 119, 217, 219, 199

Compound[b]	Primary Ion	Secondary Ions
Tri-*p*-tolyl phosphate[d]	368	367, 107, 165, 198

IS = internal standard

surr = surrogate

[a] The data presented are representative of DB-5 type analytical columns.

[b] Aroclors, Chlordane (NOS), and PCBs (NOS) are not included in this table because the quantitation ions vary with the individual components.

[c] Compounds cannot be separated for quantitation

[d] Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

2012 DEPARTMENT OF DEFENSE LABORATORY CONTROL SAMPLE CONTROL LIMIT STUDY AVERAGE % RECOVERY PER PREPARATION TECHNIQUE

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
Acenaphthene	83-32-9	80	83	77/89	80	76	-
Acenaphthylene	208-96-8	79	85	77/92	79	79	86
Acetophenone	98-86-2	81	77	65/61	70	73	-
2-Acetylaminofluorene	53-96-3	87	100	-/-	91	80	-
1-Acetyl-2-thiourea	591-08-2	-	-	-/-	-	-	-
Aldrin	309-00-2	95	-	-/-	-	97	-
2-Aminoanthraquinone	117-79-3	-	-	-/-	-	-	-
Aminoazobenzene	60-09-3	-	-	-/-	-	-	-
4-Aminobiphenyl	92-67-1	82	67	-/-	-	32	-
3-Amino-9-ethylcarbazole	132-32-1	-	-	-/-	-	-	-
Anilazine	101-05-3	-	-	-/-	-	-	-
Aniline	62-53-3	60	61	53/51	49	63	-
o-Anisidine	90-04-0	-	-	-/-	-	-	-
Anthracene	120-12-7	86	86	80/89	83	82	-
Aramite	140-57-8	83	90	-/-	-	71	-
Atrazine	1912-24-9	91	94	90/-	86	86	-
Azinphos-methyl (Guthion)	86-50-0	-	-	-/-	-	-	-
Azobenzene	103-33-3	83	92	-/-	-	82	-
Barban	101-27-9	-	-	-/-	-	-	-
Benzaldehyde	100-52-7	71	86	66/-	68	68	-
Benzidine	92-87-5	46	26	-/12	61	37	-
Benzo(a)anthracene	56-55-3	89	89	81/91	86	84	101
Benzo(b)fluoranthene	205-99-2	89	89	75/91	87	86	98
Benzo(k)fluoranthene	207-08-9	91	90	83/95	89	87	100
Benzoic acid	65-85-0	29	58	46/64	48	71	93
Benzo(g,h,i)perylene	191-24-2	89	90	78/98	84	86	101
Benzo(a)pyrene	50-32-8	88	87	78/95	88	84	91
Benzo(e)pyrene	192-97-2	84	-	-/-	-	77	-
p-Benzoquinone	106-51-4	-	-	-/-	-	-	-
Benzyl alcohol	100-51-6	66	80	75/82	63	74	-
α-BHC	319-84-6	95	-	-/-	-	95	-
β-BHC	319-85-7	91	-	-/-	-	95	-
δ-BHC	319-86-8	96	-	-/-	-	97	-
γ-BHC (Lindane)	58-89-9	94	-	-/-	-	98	-
1,1'-Biphenyl	92-52-4	80	77	73/82	69	73	-
Bis(2-chloroethoxy)methane	111-91-1	81	83	76/89	70	75	94
Bis(2-chloroethyl)ether	111-44-4	77	79	75/88	68	71	-
Bis(2-chloro-1-methylethyl)ether	108-60-1	76	83	-/-	-	76	-
Bis(2-ethylhexyl)phthalate	117-81-7	93	94	87/94	83	90	103
4-Bromophenyl phenyl ether	101-55-3	86	89	78/91	78	82	-
Bromoxynil (Brominal)	1689-84-5	-	-	-/-	-	-	-
Butyl benzyl phthalate	85-68-7	91	94	84/95	82	88	99
Caprolactam	105-60-2	21	42	76/-	86	80	-
Captafol	2425-06-1	-	-	-/-	-	-	-
Captan	133-06-2	-	-	-/-	-	-	-
Carbaryl (Sevin)	63-25-2	-	-	-/-	-	-	-

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
Cabriole	86-74-8	89	90	75/88	84	87	-
Carbofuran (Furaden)	1563-66-2	-	-	-/-	-	-	-
Carbophenothion	786-19-6	-	-	-/-	-	-	-
Chlordane (NOS)*	57-74-9	95	-	-/-	-	94	-
Chlorfenvinphos	470-90-6	-	-	-/-	-	-	-
4-Chloroaniline	106-47-8	74	77	56/71	54	63	-
Chlorobenzilate	510-15-6	93	103	-/-	93	76	-
5-Chloro-2-methylaniline	95-79-4	-	-	-/-	-	-	-
4-Chloro-3-methylphenol	59-50-7	82	86	79/91	76	81	-
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	-	-	-/-	-	-	-
1-Chloronaphthalene	90-13-1	-	83	-/-	-	80	-
2-Chloronaphthalene	91-58-7	75	78	75/85	72	75	-
2-Chlorophenol	95-57-8	73	77	77/87	68	74	93
4-Chloro-1,2-phenylenediamine	95-83-0	-	-	-/-	-	-	-
4-Chloro-1,3-phenylenediamine	5131-60-2	-	-	-/-	-	-	-
4-Chlorophenyl phenyl ether	7005-72-3	84	87	79/91	76	80	-
Chrysene	218-01-9	89	89	82/91	87	85	100
Coumaphos	56-72-4	-	-	-/-	-	-	-
p-Cresidine	120-71-8	-	-	-/-	-	-	-
Crotoxyphos	7700-17-6	-	-	-/-	-	-	-
2-Cyclohexyl-4,6-dinitrophenol	131-89-5	-	-	-/-	-	-	-
4,4'-DDD	72-54-8	96	-	-/-	-	101	-
4,4'-DDE	72-55-9	94	-	-/-	-	100	-
4,4'-DDT	50-29-3	94	-	-/-	-	96	-
Demeton-O	298-03-3	-	-	-/-	-	-	-
Demeton-S	126-75-0	-	-	-/-	-	-	-
Diallate (cis or trans)	2303-16-4	89	93	-/-	87	79	-
2,4-Diaminotoluene	95-80-7	-	-	-/-	-	-	-
Dibenz(a,j)acridine	224-42-0	-	-	-/-	-	89	-
Dibenz(a,h)anthracene	53-70-3	89	90	80/97	86	87	101
Dibenzofuran	132-64-9	82	86	75/92	73	80	-
Dibenzo(a,e)pyrene	192-65-4	94	-	-/-	-	93	-
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	-	-	-/-	-	-	-
Di-n-butyl phthalate	84-74-2	90	93	83/93	82	87	-
Dichlone	117-80-6	-	-	-/-	-	-	-
1,2-Dichlorobenzene	95-50-1	68	69	70/86	65	72	-
1,3-Dichlorobenzene	541-73-1	65	67	68/84	63	69	93
1,4-Dichlorobenzene	106-46-7	66	69	72/85	64	70	-
3,3'-Dichlorobenzidine	91-94-1	79	75	57/83	78	73	78
2,4-Dichlorophenol	120-83-2	81	82	80/91	72	77	-
2,6-Dichlorophenol	87-65-0	83	81	96/84	71	78	-
Dichlorovos (DDVP, Dichlorvos)	62-73-7	-	-	-/-	-	-	-
Dicrotophos	141-66-2	-	-	-/-	-	-	-
Dieldrin	60-57-1	96	-	-/-	-	98	-
Diethyl phthalate	84-66-2	88	91	80/93	79	84	-
Diethyl sulfate	64-67-5	-	-	-/-	-	-	-
Diethylstilbestrol	56-53-1	-	-	-/-	-	-	-
Dimethoate	60-51-5	88	108	-/-	-	80	-

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
3,3'-Dimethoxybenzidine	119-90-4	-	-	-/-	-	-	-
Dimethyl aminoazobenzene	60-11-7	87	99	-/-	92	88	-
Dimethyl phthalate	131-11-3	85	90	80/93	77	83	-
7,12-Dimethylbenz(a)-anthracene	57-97-6	98	89	-/-	79	91	-
3,3'-Dimethylbenzidine	119-93-7	47	48	-/-	48	23	-
α,α-Dimethylphenethylamine	122-09-8	-	-	-/-	-	-	-
2,4-Dimethylphenol	105-67-9	75	68	54/87	71	74	95
1,2-Dinitrobenzene	528-29-0	86	77	-/-	-	80	-
1,3-Dinitrobenzene (1,3-DNB)	99-65-0	83	96	-/-	82	81	-
1,4-Dinitrobenzene	100-25-4	85	-	-/-	-	74	-
4,6-Dinitro-2-methylphenol	534-52-1	88	90	72/75	69	82	85
2,4-Dinitrophenol	51-28-5	80	82	59/63	57	74	70
2,4-Dinitrotoluene	121-14-2	90	91	84/95	81	85	98
2,6-Dinitrotoluene	606-20-2	88	90	83/94	78	82	95
Dinocap	39300-45-3	-	-	-/-	-	-	-
Dinoseb (DNBP)	88-85-7	92	95	-/67	-	52	-
Di-*n*-octyl phthalate	117-84-0	93	94	84/96	87	89	108
1,4-Dioxane	123-91-1	41	57	49/40	48	48	-
Diphenylamine	122-39-4	82	89	-/90	-	77	-
5,5-Diphenylhydantoin	57-41-0	-	-	-/-	-	-	-
1,2-Diphenylhydrazine	122-66-7	83	85	84/88	73	82	97
Disulfoton	298-04-4	87	95	-/-	-	72	-
Endosulfan I	959-98-8	102	-	-/-	-	102	-
Endosulfan II	33213-65-9	-	-	-/-	-	-	-
Endosulfan sulfate	1031-07-8	-	-	-/-	-	-	-
Endrin	72-20-8	99	-	-/-	-	99	-
Endrin aldehyde	7421-93-4	99	-	-/-	-	100	-
Endrin ketone	53494-70-5	-	-	-/-	-	-	-
EPN	2104-64-5	-	-	-/-	-	-	-
Ethion	563-12-2	-	-	-/-	-	-	-
Ethyl carbamate	51-79-6	-	-	-/-	-	-	-
Ethyl methanesulfonate	62-50-0	87	76	-/-	64	75	-
Famphur	52-85-7	136	-	-/-	-	174	-
Fensulfothion	115-90-2	-	-	-/-	-	-	-
Fenthion	55-38-9	-	-	-/-	-	-	-
Fluchloralin	33245-39-5	-	-	-/-	-	-	-
Fluoranthene	206-44-0	89	90	82/92	87	86	100
Fluorene	86-73-7	84	86	79/90	82	80	-
Heptachlor	76-44-8	93	-	-/-	-	96	-
Heptachlor epoxide	1024-57-3	95	-	-/-	-	99	-
Hexachlorobenzene	118-74-1	86	86	79/89	78	81	-
Hexachlorobutadiene	87-68-3	68	72	73/89	70	74	-
Hexachlorocyclopentadiene	77-47-4	59	39	57/46	53	69	70
Hexachloroethane	67-72-1	64	66	72/84	64	69	-
Hexachlorophene	70-30-4	74	-	-/-	-	-	-
Hexachloropropene	1888-71-7	59	45	-/78	60	66	-
Hexamethylphosphoramide (HMPA)	680-31-9	-	-	-/-	-	-	-
Hydroquinone	123-31-9	-	-	-/-	-	-	-
Indeno(1,2,3-cd)pyrene	193-39-5	89	89	81/98	85	87	102

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
Isodrin	465-73-6	92	97	-/-	82	79	-
Isophorone	78-59-1	79	84	75/89	70	74	89
Isosafrole	120-58-1	85	78	-/-	68	78	-
Kepone	143-50-0	64	-	-/-	-	52	-
Leptophos	21609-90-5	-	-	-/-	-	-	-
Malathion	121-75-5	-	-	-/-	-	-	-
Maleic anhydride	108-31-6	-	-	-/-	-	-	-
Mestranol	72-33-3	-	-	-/-	-	-	-
Methapyrilene	91-80-5	41	-	-/-	-	-	-
Methoxychlor	72-43-5	95	-	-/-	-	94	-
Methyl methanesulfonate	66-27-3	72	69	-/-	-	73	-
Methyl parathion	298-00-0	100	109	-/-	-	73	-
3-Methylcholanthrene	56-49-5	84	91	-/-	86	82	-
4,4'-Methylenebis(2-chloroaniline)	101-14-4	-	-	-/-	-	-	-
4,4'-Methylenebis(*N,N*-dimethyl-aniline)	101-61-1	-	-	-/-	-	-	-
1-Methylnaphthalene	90-12-0	76	78	78/-	78	77	-
2-Methylnaphthalene	91-57-6	75	80	75/89	77	77	-
2-Methylphenol	95-48-7	67	79	73/87	69	74	93
3-Methylphenol	108-39-4	65	81	75/91	71	77	-
4-Methylphenol	106-44-5	65	81	75/91	71	77	-
Mevinphos	7786-34-7	-	-	-/-	-	-	-
Mexacarbate	315-18-4	-	-	-/-	-	-	-
Mirex	2385-85-5	-	-	-/-	-	-	-
Monocrotophos	6923-22-4	-	-	-/-	-	-	-
Naled	300-76-5	-	-	-/-	-	-	-
Naphthalene	91-20-3	75	77	74/89	77	74	98
1,4-Naphthoquinone	130-15-4	68	66	-/-	80	72	-
1-Naphthylamine	134-32-7	68	64	-/-	-	30	-
2-Naphthylamine	91-59-8	69	53	-/-	-	31	-
Nicotine	54-11-5	-	-	-/-	-	-	-
5-Nitroacenaphthene	602-87-9	-	-	-/-	-	-	-
2-Nitroaniline	88-74-4	88	90	79/88	78	84	91
3-Nitroaniline	99-09-2	80	87	66/78	70	76	71
4-Nitroaniline	100-01-6	91	-	-/-	-	86	-
5-Nitro-*o*-anisidine	99-59-2	-	-	-/-	-	-	-
Nitrobenzene	98-95-3	80	80	73/89	68	75	95
4-Nitrobiphenyl	92-93-3	-	-	-/-	-	-	-
Nitrofen	1836-75-5	-	-	-/-	-	-	-
2-Nitrophenol	88-75-5	81	82	77/89	70	76	-
4-Nitrophenol	100-02-7	38	83	70/86	74	80	94
4-Nitroquinoline-1-oxide	56-57-5	100	76	-/-	63	96	-
N-Nitroso-di-*n*-butylamine	924-16-3	87	86	-/90	-	86	-
N-Nitrosodiethylamine	55-18-5	76	78	-/80	64	78	-
N-Nitrosodimethylamine	62-75-9	48	75	67/75	61	69	86
N-Nitrosodiphenylamine	86-30-6	85	81	77/91	78	80	-
N-Nitroso-di-*n*-propylamine	621-64-7	82	84	75/86	71	75	-
N-Nitrosomethylethylamine	10595-95-6	70	77	-/74	64	75	-
N-Nitrosomorpholine	59-89-2	80	83	-/-	73	91	-
N-Nitrosopiperidine	100-75-4	83	82	-/85	-	80	-

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
N-Nitrosopyrrolidine	930-55-2	78	81	-/84	76	79	-
5-Nitro-*o*-toluidine	99-55-8	84	90	-/-	90	63	-
Octamethyl pyrophosphoramide	152-16-9	-	-	-/-	-	-	-
4,4'-Oxydianiline	101-80-4	-	-	-/-	-	-	-
Parathion	56-38-2	96	112	-/-	-	72	-
Pentachlorobenzene	608-93-5	87	88	-/87	74	87	-
Pentachloronitrobenzene	82-68-8	91	98	-/-	85	80	-
Pentachlorophenol	87-86-5	83	84	66/79	79	78	97
Perylene	198-55-0	87	-	-/-	-	75	-
Phenacetin	62-44-2	91	96	-/-	88	80	-
Phenanthrene	85-01-8	87	88	80/90	82	83	-
Phenobarbital	50-06-6	-	-	-/-	-	-	-
Phenol	108-95-2	37	76	76/88	69	74	86
1,4-Phenylenediamine	106-50-3	-	-	-/-	12	-	-
Phorate	298-02-2	79	100	-/-	-	69	-
Phosalone	2310-17-0	-	-	-/-	-	-	-
Phosmet (Imidan)	732-11-6	-	-	-/-	-	-	-
Phosphamidon	13171-21-6	-	-	-/-	-	-	-
Phthalic anhydride	85-44-9	-	-	-/-	-	-	-
2-Picoline (2-Methylpyridine)	109-06-8	59	69	-/-	60	64	-
Piperonyl sulfoxide	120-62-7	-	-	-/-	-	-	-
Polychlorinated biphenyls (NOS)	1336-36-3	-	-	-/-	-	-	-
Pronamide (Kerb)	23950-58-5	87	99	-/-	86	75	-
Propylthiouracil	51-52-5	-	-	-/-	-	-	-
Pyrene	129-00-0	89	90	81/93	86	85	98
Pyridine	110-86-1	42	53	67/-	44	52	-
Resorcinol	108-46-3	-	-	-/-	-	-	-
Safrole	94-59-7	84	82	-/-	68	77	-
Strychnine	57-24-9	-	-	-/-	-	-	-
Sulfallate	95-06-7	-	-	-/-	-	-	-
Terbufos	13071-79-9	-	-	-/-	-	-	-
1,2,4,5-Tetrachlorobenzene	95-94-3	75	84	87/85	70	76	-
2,3,4,6-Tetrachlorophenol	58-90-2	87	89	92/83	83	80	-
Tetrachlorvinphos (Stirophos, Gardona)	961-11-5	-	-	-/-	-	-	-
Tetraethyl dithiopyrophosphate	3689-24-5	86	101	-/-	-	69	-
Tetraethyl pyrophosphate (TEPP)	107-49-3	-	-	-/-	-	-	-
Thionazine	297-97-2	103	105	-/-	-	81	-
Thiophenol (Benzenethiol)	108-98-5	-	-	-/-	-	-	-
2,4-Toluene diisocyanate	584-84-9	-	-	-/-	-	-	-
o-Toluidine	95-53-4	75	62	-/-	56	39	-
Toxaphene	8001-35-2	100	-	-/-	-	99	-
1,2,4-Trichlorobenzene	120-82-1	68	72	74/89	68	72	-
2,4,5-Trichlorophenol	95-95-4	85	87	80/93	76	79	98
2,4,6-Trichlorophenol	88-06-2	84	86	80/90	74	79	98
O,O,O-Triethyl phosphorothioate	126-68-1	87	90	-/-	-	75	-
Trifluralin (Treflan)	1582-09-8	-	-	-/-	-	-	-
Trimethyl phosphate	512-56-1	-	-	-/-	-	-	-
2,4,5-Trimethylaniline	137-17-7	-	-	-/-	-	-	-
1,3,5-Trinitrobenzene	99-35-4	85	98	-/-	95	85	-

Compounds	CAS No	3510	3520	3540/ 3541	3545	3550	3580
Tris(2,3-dibromopropyl)phosphate	126-72-7	-	-	-/-	-	-	-
Tri-*p*-tolyl phosphate	78-32-0						

* Average % recovery for Chlordane (NOS) taken from gamma-chlordane results in Department of Defense (DOD) Study.

TABLE 3

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA[a,b]

Mass	Ion Abundance Criteria
68	<2% of *m/z* 69
69	Present
70	<2% of *m/z* 69
197	<2% of *m/z* 198
198	Base peak or present
199	5-9% of *m/z* 198
365	>1% of Base Peak
441	<150% of *m/z* 443
442	Base peak or present
443	15-24% of *m/z* 442

[a] The criteria are taken from Reference 11 (Method 525.3).

[b] *The criteria in this table are intended to be used as default criteria for quadrupole instrumentation if optimized manufacturer's operating conditions are not available. Alternate tuning criteria may be employed (e.g., CLP or Method 625), provided that method performance is not adversely affected. See Sec. 11.3.1.*

TABLE 4

GUIDANCE RESPONSE FACTOR CRITERIA FOR INITIAL CALIBRATION USING THE SUGGESTED IONS FROM TABLE 1 (See Sec. 11.3.4.2)

Semivolatile Compounds	Guidance Min Response Factor (RF)
Acenaphthene	0.9
Acenaphthylene	0.9
Acetophenone	0.01
Anthracene	0.7
Atrazine	0.01
Benzaldehyde	0.01
Benzo(a)anthracene	0.8
Benzo(a)pyrene	0.7
Benzo(b)fluoranthene	0.7
Benzo(g,h,i)perylene	0.5
Benzo(k)fluoranthene	0.7
1,1'-Biphenyl	0.01
Bis(2-chloroethoxy)methane	0.3
Bis(2-chloroethyl)ether	0.7
Bis-(2-ethylhexyl)phthalate	0.01
4-Bromophenyl-phenyl ether	0.1
Butyl benzyl phthalate	0.01
Caprolactam	0.01
Carbazole	0.01
4-Chloroaniline	0.01
4-Chloro-3-methylphenol	0.2
2-Chloronaphthalene	0.8
2-Chlorophenol	0.8
4-Chlorophenyl-phenyl ether	0.4
Chrysene	0.7
Dibenz(*a,h*)anthracene	0.4
Dibenzofuran	0.8
Di-*n*-butyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
2,4-Dichlorophenol	0.2
Diethyl phthalate	0.01
Dimethyl phthalate	0.01
2,4-Dimethylphenol	0.2
4,6-Dinitro-2-methylphenol	0.01
2,4-Dinitrophenol	0.01
2,4-Dinitrotoluene	0.2
2,6-Dinitrotoluene	0.2
Di-n-octyl phthalate	0.01
Fluoranthene	0.6
Fluorene	0.9
Hexachlorobenzene	0.1
Hexachlorobutadiene	0.01
Hexachlorocyclopentadiene	0.05
Hexachloroethane	0.3
Indeno(1,2,3-cd)pyrene	0.5

Semivolatile Compounds	Guidance Min Response Factor (RF)
Isophorone	0.4
2-Methylnaphthalene	0.4
2-Methylphenol	0.7
4-Methylphenol	0.6
Naphthalene	0.7
2-Nitroaniline	0.01
3-Nitroaniline	0.01
4-Nitroaniline	0.01
Nitrobenzene	0.2
2-Nitrophenol	0.1
4-Nitrophenol	0.01
N-Nitroso-di-*n*-propylamine	0.5
N-Nitrosodiphenylamine	0.01
2,2'-Oxybis-(1-chloropropane)	0.01
Pentachlorophenol	0.05
Phenanthrene	0.7
Phenol	0.8
Pyrene	0.6
1,2,4,5-Tetrachlorobenzene	0.01
2,3,4,6-Tetrachlorophenol	0.01
2,4,5-Trichlorophenol	0.2
2,4,6-Trichlorophenol	0.2

These RFs are provided as guidance only and are not intended to be a requirement.

See Sec. 11.3.4.2 and Appendix B for additional information.

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzaldehyde	Benzoic acid	Acenaphthylene
Benzyl alcohol	Bis(2-chloroethoxy)methane	1,1'-Biphenyl
Bis(2-chloroethyl)ether	Caprolactam	1-Chloronaphthalene
Bis(2-chloro-1-methylethyl)ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,4-Dichlorobenzene	α,α-Dimethylphenethylamine	Dimethyl phthalate
1,4-Dioxane	2,4-Dimethylphenol	2,4-Dinitrophenol
Ethyl methanesulfonate	Hexachlorobutadiene	2,4-Dinitrotoluene
2-Fluorophenol (surr)	Isophorone	2,6-Dinitrotoluene
Hexachloroethane	2-Methylnaphthalene	Fluorene
Methyl methanesulfonate	1-Methylnaphthalene	2-Fluorobiphenyl (surr)
2-Methylphenol	Naphthalene	Hexachlorocyclopentadiene
4-Methylphenol	Nitrobenzene	1-Naphthylamine
N-Nitrosodimethylamine	Nitrobenzene-d_8 (surr)	2-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Nitroaniline
Phenol	N-Nitrosodi-n-butylamine	3-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosopiperidine	4-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitrophenol
Pyridine		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
Atrazine	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)perylene
4-Bromophenyl phenyl ether	Butyl benzyl phthalate	Benzo(a)pyrene
Carbazole	Chrysene	Benzo(e)pyrene
Di-n-butyl phthalate	3,3'-Dichlorobenzidine	Dibenz(a,j)acridine
4,6-Dinitro-2-methylphenol	Dimethyl aminoazobenzene	Dibenz(a,h)anthracene
Diphenylamine	Di-*n*-octyl phthalate	7,12-Dimethylbenz(a)anthracene
Fluoranthene	Pyrene	Indeno(1,2,3-cd)pyrene
Hexachlorobenzene	Terphenyl-d_{14} (surr)	3-Methylcholanthrene
4-Nitroquinoline-1-oxide		Perylene
N-Nitrosodiphenylamine		
Pentachlorophenol		
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr) = surrogate

TABLE 6

SUMMARY OF QC CRITERIA FOR USE WITH 8270E[a]

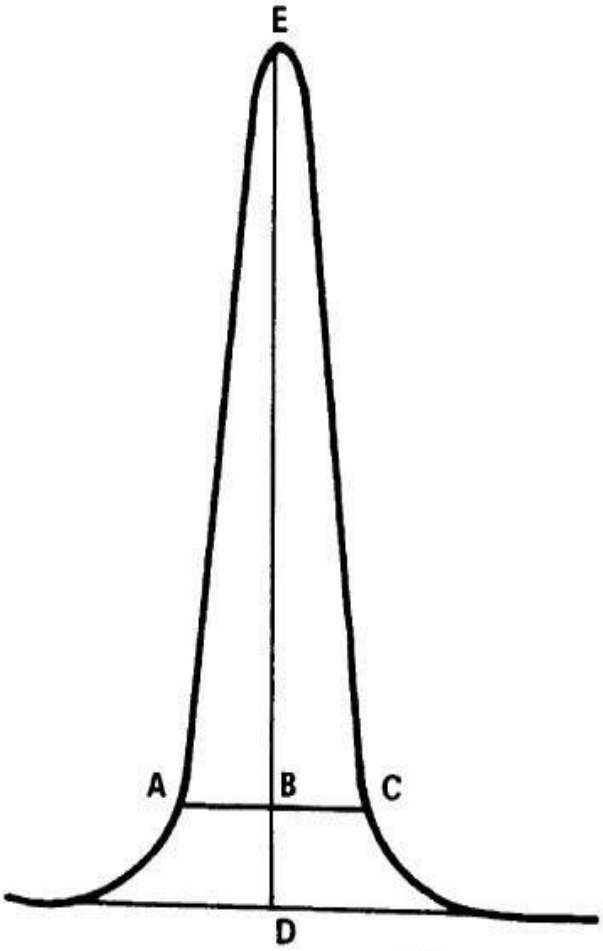
Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
Instrument performance check (Secs. 9.3.1, 11.3.1)	Prior to initial calibration	≤50 ng Decafluorotriphenylphosphine (DFTPP) injected	Meet ion ratio criteria for reference compound: DFTPP (Table 3), or alternative documented criteria; Tailing factor ≤2 and degradation ≤20%
Initial calibration (ICAL) (Secs. 9.3.2, 11.3)	Prior to analyzing samples, and as needed if continuing performance criteria cannot be met	5 points minimum for response factor (RF) and linear regressions (LR), 6 points minimum for quadratic regression (QR) >90% of reported target analytes meet ICAL criteria	For average RF calibration model: ≤20% relative standard deviation (RSD) of RFs For LR or QR model: R≥0.995, R^2≥0.99. Independent of calibration model: Low standard recalculation (refit) should be ±50% of true value; other standards >lower limit of quantitation (LLOQ) are recommended to be ±30% of true value. Or, relative standard error (RSE) ≤20% (Refer to Method 8000 and reference 19 for calculation). See Method 8000 for additional criteria.
Initial Calibration Verification (ICV) (Secs. 9.3.2, 11.3.7)	After each ICAL and prior to analyzing samples	Prepared from different source of target analytes than ICAL standards	Calculated concentrations of target analytes are ±30% of true value
Continuing Calibration Verification (CCV) (Secs. 9.3.3, 11.4)	Once at least every 12 hours	>80% of target analytes meet CCV criteria	Targets are ≤20% difference or drift; IS responses are within 50% to 200% of mid-point of ICAL or average of ICAL ISs; and retention times for ISs have not shifted >30 seconds relative to ICAL
Blanks (Sec. 9.5)	One method blank (MB) per preparation	NA	Target analyte concentrations in blanks are <1/2 LLOQ, or ≤10% of

Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
	batch of 20 or fewer samples; Instrument blanks as needed		concentration in field samples
Laboratory Control Sample (LCS) (Sec. 9.6.2)	One per preparation batch of 20 or fewer samples	NA	Meets recovery criteria
Duplicates and Matrix Spikes (Sec. 9.6.1)	A duplicate and matrix spike, or matrix spike/matrix spike duplicate per preparation of 20 or fewer samples (not required per batch)	NA	Performance-based or project-defined recovery criteria for matrix spikes; Relative percent difference (RPD) criteria between measured concentrations in sample and laboratory duplicate or in matrix spike and matrix spike duplicate
Surrogates (Sec. 9.7)	Added to each sample	NA	Performance-based recovery criteria established by the laboratory or criteria chosen for the project
Internal Standards (IS) (Secs. 9.8, 11.5.4)	Added to each sample	NA	IS response is within 50-200% of the response of the same IS in the midpoint ICAL standard (or average of ICAL) or most recent CCV
Qualitative Analyte Identification (Sec. 11.6.1)	Each target analyte	NA	RT in sample is within ±10 seconds of RT in midpoint ICAL or CCV standard) or within ±10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds) Characteristic ion(s) within ±30% of expected ion ratio in reference spectrum; or, match to reference library spectra ≥0.8 (only for full mass range acquisition modes)

[a] Default acceptance criteria; alternative criteria may be specified for a given application. Refer to Sec. 9 for more information.

FIGURE 1

TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm

10% Peak Height = BD = 10 mm

Peak Width at 10% Peak Height = AC = 23 mm

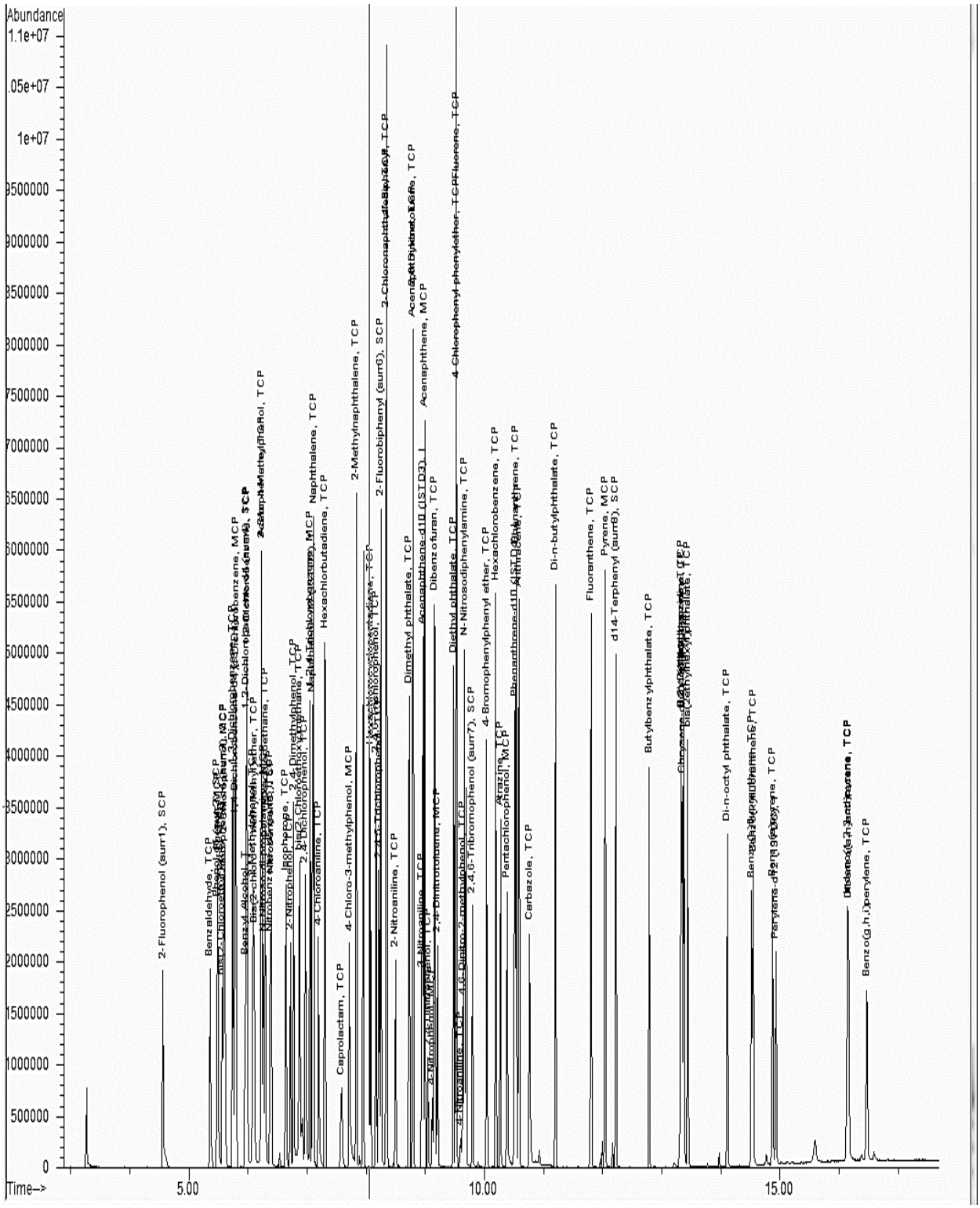
AB = 11 mm

BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

FIGURE 2

GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD[a]



[a] Courtesy of EPA Region 6

Appendix A: Changes to 8270E, Rev. 6 compared to 8270D, Rev. 5

1. Throughout: The term mass was replaced with the ratio of mass/charge (*m/z)* as that is what is actually measured. Area or height was replaced with response.
2. Table, Sec. 1.1: Designation for appropriate preparation technique in the analyte table in Sec. 1.1 was changed from X to ✓ to be more intuitive. All abbreviations for problematic compounds were removed and replaced with * (definitions and specific analytes were added to Sec. 1.4). The analyte table in Sec. 1.1 was updated using data from an EPA statistical analysis by preparation method of data from a DOD LCS study conducted in 2012 (which was published in 2013). If average %R fell between 50-150%, the preparation technique was designated as adequate. If there was inadequate data in the study, a – (dash) or ✓* was used (✓* is for analytes listed as having adequate recovery in previous versions). Method 3545 was added as a preparation technique. Footnote c was updated with additional information. The key to the analyte list has new definitions for symbols used in the table.
3. The following analytes were added to the table: atrazine, azobenzene, benzaldehyde, benzo(e)pyrene, 1,1'-biphenyl, caprolactam, carbazole, 1,4-dioxane, 1-methylnaphthalene, perylene, and pyridine. The following names were updated to match the NIST database: benzo(g,h,i)perylene, 4-nitroquinoline-1-oxide, and 2,4-toluene diisocyanate.
4. Sec. 1.2: Method 3545 was removed from Sec. 1.2 and added to the analyte table in Sec. 1.1 as it is commonly used. Method 3511 was added as an allowable preparation method.
5. Sec. 1.3: Added GC/MS/MS as technology for multi-components.
6. Sec. 1.4: Abbreviations from the analyte table in Sec. 1.1 were moved into subsections of Sec. 1.4 (new Secs. 1.4.10 through 1.4.17). Additional analytes were added if they were known to be problematic (Sec. 1.4.7 and 1.4.14). Sec. 1.4.8 was added regarding adjusting pH prior to water extractions for some analytes. Surrogate compound suggestions were added for more volatile analytes in Sec. 1.4.10.
7. Sec. 1.5: The section about LLOQ and reference to old Table 2 were removed. Language was added discussing the use of hydrogen carrier gas and tandem mass spectrometry instrumentation.
8. Sec. 2.3: Jet separator interface was removed.
9. Sec. 4.2: Added information about blank contamination may not be subtracted from samples and may require sample qualification.
10. Sec. 5.0: Added reference to new Appendix B for using hydrogen carrier gas.
11. Sec. 6.1.1: Added specific GC inlet types. References to column vendors were removed.
12. Sec. 6.1.3: Changed from scan rate to minimum number of spectra per peak. Added subsections for MS/MS (Sec. 6.1.3.3) and SIM/CI (Sec. 6.1.3.4).
13. Sec. 7.4.3: Added language regarding expiration dates of standards and when standards must be discarded.
14. Sec. 7.5: Removed reference to Sec. 11.3.2 for IS criteria and added reference to Sec. 11.4.3 of Method 8000 (IS calibration).
15. Sec. 7.6: Added warning about preparing DFTPP in alternate solvents.
16. Sec. 7.7.1: Added language about minimum number of calibration standards for regression curve types and low point at or below LLOQ.
17. Sec. 7.7.2: Added paragraph for CCV.

18. Sec. 7.7.3: Added paragraph about performing ICV with second source.
19. Sec. 7.8: Added reference for additional surrogates. Reworded note about verifying surrogate solutions.
20. Sec. 7.8.1: Removed note about surrogate requirement from Method 3561.
21. Sec. 7.9: Added recommendation to use full analyte list for LCS spike solution. Added note about verifying solutions prior to use or for troubleshooting.
22. Sec. 7.11: Added carrier gases to Reagents and Standards section.
23. Sec. 9.3.1: Changed tune verification frequency to just prior to ICAL. Added language regarding purpose for verification.
24. Sec. 9.3.2: Clarified when the ICV is required.
25. Sec. 9.3.4: Removed paragraph for relative retention time.
26. Sec. 9.5: Significant revisions/additions were made to blank section. Added clarifying information about concentrations allowed in blanks (1/2 LLOQ), how blank concentration relates to sample concentration (<1/10) and some guidance for qualifying data.
27. Sec. 9.6.3: Added paragraph requiring use of MB in each batch.
28. Sec. 9.7: Reworded to require use of surrogates.
29. Sec. 9.8: Added language for monitoring of ISs in samples.
30. Sec. 9.9: LLOQ language from Method 8000 was added. Additional information/revisions made to LLOQ language (e.g., concentration range of 0.5 - 2X added for LLOQ verification; frequency). Section was added for reporting concentrations below LLOQ.
31. Sec. 11.1: Method 3560 was removed. Method 3511 was added.
32. Sec. 11.2: The reference to derivatization in Method 8041 was removed. Method 3640 was added.
33. Secs. 11.3 & 11.4: Reorganized sections to place all criteria and checks for ICAL (e.g., curve calculations and criteria, refitting low point, and ICVs) in Sec. 11.3 and to place all criteria and checks for CCVs in Sec. 11.4. Redundant information was removed and the requirements were clarified.
34. Sec. 11.3: Updated GC/MS operating conditions.
35. Sec. 11.3.1.2: Updated note about performing tune verification for SIM/scan. Added note about instrument performance check for CI or tandem MS analysis.
36. Sec. 11.3.1.3: Added tailing graphic. Added note that degradation and tailing checks are not needed for limited analyte lists.
37. Sec. 11.3.2: Removed relative time reference for targets compared to IS (0.8-1.2)
38. Sec. 11.3.3: Added note about having adequate sensitivity at LLOQ.
39. Sec. 11.3.4.2: Added language to make minimum RFs in Table 4 guidance only. Added suggestions for laboratories to establish procedure for checking preparation of standards. Added a note about analytes with low responses (RF <0.01).
40. Sec. 11.3.5.1: Added note about curve fit when blank contamination is present.
41. Sec. 11.3.5.3: Added criteria for regression curves and RSE. Removed RRT language.
42. Sec. 11.3.6: Clarified language for refitting low ICAL point. Changed criteria for refitting to ±50%. [Moved from 11.4.5.6]

43. Sec. 11.3.7: Added section about ICV requirements.
44. Sec. 11.3.8: Updated SIM and SRM guidance. [Moved from 11.5.5]
45. Sec. 11.4.1: Decreased DFTPP tune check, tailing, and breakdown frequency from every 12 hours to once prior to ICAL.
46. Sec. 11.4.2: Clarified note to allow the injection time for last ICAL standard as start of 12-hour clock.
47. Sec. 11.4.3: Clarified that a blank is required after calibration.
48. Sec. 11.4.4: Removed requirement to check min RFs. Added allowance for single CCV evaluation report using concentration for mixed calibration models.
49. Sec. 11.4.4.2: Added note about monitoring system performance in absence of degradation and tailing checks.
50. Sec. 11.4.6: Clarified that monitoring of ISs in CCVs is required.
51. Sec. 11.5.4: Expanded dilution target range to include middle of curve.
52. Sec. 11.6.1: Added language requiring the evaluation of the absolute shifts of target analytes. Added clarifying language for analytes with greater shifts. Revised RRT to be an alternate method for evaluating RT shifts.
53. Sec. 11.6.1.4: Updated calculations for verifying peak resolution.
54. Sec. 11.6.2: Revised tentative identification interpretation language.
55. Sec. 11.7.5: Added references for performing PCB analysis as individual congeners using low and high-resolution mass spectrometry and as homolog series.
56. Sec. 11.7.6: Clarified language for TPH (GRO and DRO). Added allowance to use GC/MS analysis.
57. Sec. 13.0: Removed all references to tables and replaced with references to: http://www.epa.gov/hw-sw846/validated-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry.
58. Sec. 14 & 15: Website links to previous ACS documents were updated as the old documents listed are no longer available. Reference was updated to http://www.acs.org/content/dam/acsorg/about/governance/committees/chemicalsafety/publications/less-is-better.pdf and http://www.labsafety.org/FreeDocs/WasteMgmt.pdf.
59. Sec. 16.0: Added reference for DOD data. Added references for PCB analysis.
60. Table 1: Added new analytes with suggested ions. Removed Aroclors.
61. Table 2: LLOQ limits removed. Replaced with 2012 DOD study data.
62. Table 3: DFTPP criteria updated with criteria from EPA Method 525.3.
63. Table 4: Min RF table renamed as guidance. Caution added below table. Compounds were alphabetized by name.
64. Table 5: New analytes added with suggested IS. Compounds were alphabetized by name.
65. Tables 6 - 22: Removed tables 6 - 22 from 8270D containing performance data. See web link in Sec. 13.

B1.0 Guidance for Using Hydrogen Carrier Gas

B1.1 Hydrogen is an acceptable carrier gas to use for this analysis. However, the following modifications may be needed to make the analysis comparable to helium carrier gas:

B1.1.1 It is recommended that the highest purity hydrogen gas (i.e., 99.999% or better) be used, such as from a generator or from high purity cylinders that will have minimal interferences present (e.g., hydrocarbons and water). Use of stainless steel tubing instead of copper tubing may increase the longevity of gas lines as older copper lines may become brittle over time with the use of hydrogen. MS ion source materials should be designed and approved for use with hydrogen. Contact the manufacturer of the MS to confirm the ion source is compatible.

Additionally, the pressure in the source should be reduced when hydrogen is used to prevent chemical ionization or other detrimental reactions from occurring. This may be done by the use of narrower bore columns (0.18 mm ID or smaller), reduction in the flow to the MS, and/or by the use of internal MS vacuum pumps (turbo pumps) with greater volumetric or pumping efficiency. Hydrogen may not be a suitable carrier gas for systems that have internal diffusion pumps.

B1.1.2. Use of hydrogen will clean (scrub) the metal surfaces of the analytical system of compounds that have adhered to the surface (generally hydrocarbons) and increase the background presence of these interferences. A bake-out of the system using high flows of hydrogen may decrease these interferences to a level that would not interfere with analysis. It is also recommended new filters be installed on gas lines prior to switching to hydrogen to prevent the scrubbing of impurities from the filters.

B1.1.3 Methylene chloride used in calibration standards and extracts may form hydrochloric acid (HCl) in the inlet when hydrogen is used as a carrier gas. HCl may build up over time, creating active sites in the chromatographic system, and potentially causing permanent damage to sensitive parts due to corrosion. Using an alternate solvent such as ethyl acetate to prepare calibration standards should reduce the formation of HCl. However, this would also require a solvent switch for extracts from any associated preparation methods, as extracts are required to be in the same final solvent as calibration standards (>80% the same). Use of the lowest practical inlet temperature setting (e.g., 225 °C), use of split injections, and/or frequent replacement of inlet liners may also prevent the formation of HCl if methylene chloride is used. Regular replacement of inlet liners to mitigate performance issues related to active site and HCl formation is recommended. Alternate injection techniques may be used as long as the user can demonstrate adequate perform for their project needs using that introduction method.

B1.2 Use of hydrogen as the carrier gas may also reduce the responses of target analytes (i.e., approximately 2 - 5 times) as compared to helium. RF criteria listed in Table 4 were developed using helium carrier gas and are not appropriate for hydrogen carrier gas due to the reduced response of some analytes. If minimum RFs are used in evaluating the calibration, the laboratory should develop their own criteria or use published RF from the instrument manufacturer. Reactivity of target analytes will vary with instrument conditions. As part of the IDP process, evaluate target analytes for stability under the expected analytical conditions.

B1.3 As with any method modification, all QC procedures listed in Sec. 9.0 of this method should be repeated and passed using hydrogen as the carrier gas prior to the analysis of samples. This would include the use of alternate solvents for extracts and calibration standards, if utilized.

B1.4 Because hydrogen gas is flammable, additional safety controls may be necessary to prevent explosive levels of gas from forming. This may be accomplished by connecting vent lines from the GC inlet and MS rough pump to exhaust systems in the laboratory and leak testing all gas line connections. The flow of hydrogen should also be turned off at the source prior to opening gas lines on the GC and prior to venting the MS (such as when maintenance is performed). The user should consult additional guidelines for the safe use of hydrogen from the instrument manufacturer prior to implementing its use.