# SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

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 method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use 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 included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute 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 RCRA analytes have been determined by this method:

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| Acenaphthene | 83-32-9 | X | X | X | X | X |
| Acenaphthylene | 208-96-8 | X | X | X | X | X |
| Acetophenone | 98-86-2 | X | ND | ND | ND | X |
| 2-Acetylaminofluorene | 53-96-3 | X | ND | ND | ND | X |
| 1-Acetyl-2-thiourea | 591-08-2 | LR | ND | ND | ND | LR |
| Aldrin | 309-00-2 | X | X | X | X | X |
| 2-Aminoanthraquinone | 117-79-3 | X | ND | ND | ND | X |
| Aminoazobenzene | 60-09-3 | X | ND | ND | ND | X |
| 4-Aminobiphenyl | 92-67-1 | X | ND | ND | ND | X |
| 3-Amino-9-ethylcarbazole | 132-32-1 | X | X | ND | ND | ND |
| Anilazine | 101-05-3 | X | ND | ND | ND | X |
| Aniline | 62-53-3 | X | X | ND | X | X |
| *o*-Anisidine | 90-04-0 | X | ND | ND | ND | X |
| Anthracene | 120-12-7 | X | X | X | X | X |
| Aramite | 140-57-8 | HS | ND | ND | ND | X |
| Aroclor 1016 | 12674-11-2 | X | X | X | X | X |
| Aroclor 1221 | 11104-28-2 | X | X | X | X | X |
| Aroclor 1232 | 11141-16-5 | X | X | X | X | X |
| Aroclor 1242 | 53469-21-9 | X | X | X | X | X |
| Aroclor 1248 | 12672-29-6 | X | X | X | X | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| Aroclor 1254 | 11097-69-1 | X | X | X | X | X |
| Aroclor 1260 | 11096-82-5 | X | X | X | X | X |
| Azinphos-methyl | 86-50-0 | HS | ND | ND | ND | X |
| Barban | 101-27-9 | LR | ND | ND | ND | LR |
| Benzidine | 92-87-5 | CP | CP | CP | CP | CP |
| Benzoic acid | 65-85-0 | X | X | ND | X | X |
| Benz(a)anthracene | 56-55-3 | X | X | X | X | X |
| Benzo(b)fluoranthene | 205-99-2 | X | X | X | X | X |
| Benzo(k)fluoranthene | 207-08-9 | X | X | X | X | X |
| Benzo(g,h,i)perylene | 191-24-2 | X | X | X | X | X |
| Benzo(a)pyrene | 50-32-8 | X | X | X | X | X |
| *p*-Benzoquinone | 106-51-4 | OE | ND | ND | ND | X |
| Benzyl alcohol | 100-51-6 | X | X | ND | X | X |
| α-BHC | 319-84-6 | X | X | X | X | X |
| β-BHC | 319-85-7 | X | X | X | X | X |
| δ-BHC | 319-86-8 | X | X | X | X | X |
| γ-BHC (Lindane) | 58-89-9 | X | X | X | X | X |
| Bis(2-chloroethoxy)methane | 111-91-1 | X | X | X | X | X |
| Bis(2-chloroethyl) ether | 111-44-4 | X | X | X | X | X |
| Bis(2-chloroisopropyl) ether | 39638-32-9 | X | X | X | X | X |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | X | X | X | X | X |
| 4-Bromophenyl phenyl ether | 101-55-3 | X | X | X | X | X |
| Bromoxynil | 1689-84-5 | X | ND | ND | ND | X |
| Butyl benzyl phthalate | 85-68-7 | X | X | X | X | X |
| Captafol | 2425-06-1 | HS | ND | ND | ND | X |
| Captan | 133-06-2 | HS | ND | ND | ND | X |
| Carbaryl | 63-25-2 | X | ND | ND | ND | X |
| Carbofuran | 1563-66-2 | X | ND | ND | ND | X |
| Carbophenothion | 786-19-6 | X | ND | ND | ND | X |
| Chlordane (NOS) | 57-74-9 | X | X | X | X | X |
| Chlorfenvinphos | 470-90-6 | X | ND | ND | ND | X |
| 4-Chloroaniline | 106-47-8 | X | ND | ND | ND | X |
| Chlorobenzilate | 510-15-6 | X | ND | ND | ND | X |
| 5-Chloro-2-methylaniline | 95-79-4 | X | ND | ND | ND | X |
| 4-Chloro-3-methylphenol | 59-50-7 | X | X | X | X | X |
| 3-(Chloromethyl)pyridine hydrochloride | 6959-48-4 | X | ND | ND | ND | X |
| 1-Chloronaphthalene | 90-13-1 | X | X | X | X | X |
| 2-Chloronaphthalene | 91-58-7 | X | X | X | X | X |
| 2-Chlorophenol | 95-57-8 | X | X | X | X | X |
| 4-Chloro-1,2-phenylenediamine | 95-83-0 | X | X | ND | ND | ND |
| 4-Chloro-1,3-phenylenediamine | 5131-60-2 | X | X | ND | ND | ND |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | X | X | X | X | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| Chrysene | 218-01-9 | X | X | X | X | X |
| Coumaphos | 56-72-4 | X | ND | ND | ND | X |
| p-Cresidine | 120-71-8 | X | ND | ND | ND | X |
| Crotoxyphos | 7700-17-6 | X | ND | ND | ND | X |
| 2-Cyclohexyl-4,6-dinitro-phenol | 131-89-5 | X | ND | ND | ND | LR |
| 4,4'-DDD | 72-54-8 | X | X | X | X | X |
| 4,4'-DDE | 72-55-9 | X | X | X | X | X |
| 4,4'-DDT | 50-29-3 | X | X | X | X | X |
| Demeton-O | 298-03-3 | HS | ND | ND | ND | X |
| Demeton-S | 126-75-0 | X | ND | ND | ND | X |
| Diallate (*cis* or *trans*) | 2303-16-4 | X | ND | ND | ND | X |
| 2,4-Diaminotoluene | 95-80-7 | DC, OE | ND | ND | ND | X |
| Dibenz(a,j)acridine | 224-42-0 | X | ND | ND | ND | X |
| Dibenz(a,h)anthracene | 53-70-3 | X | X | X | X | X |
| Dibenzofuran | 132-64-9 | X | X | ND | X | X |
| Dibenzo(a,e)pyrene | 192-65-4 | ND | ND | ND | ND | X |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | X | X | ND | ND | ND |
| Di-n-butyl phthalate | 84-74-2 | X | X | X | X | X |
| Dichlone | 117-80-6 | OE | ND | ND | ND | X |
| 1,2-Dichlorobenzene | 95-50-1 | X | X | X | X | X |
| 1,3-Dichlorobenzene | 541-73-1 | X | X | X | X | X |
| 1,4-Dichlorobenzene | 106-46-7 | X | X | X | X | X |
| 3,3'-Dichlorobenzidine | 91-94-1 | X | X | X | X | X |
| 2,4-Dichlorophenol | 120-83-2 | X | X | X | X | X |
| 2,6-Dichlorophenol | 87-65-0 | X | ND | ND | ND | X |
| Dichlorovos | 62-73-7 | X | ND | ND | ND | X |
| Dicrotophos | 141-66-2 | X | ND | ND | ND | X |
| Dieldrin | 60-57-1 | X | X | X | X | X |
| Diethyl phthalate | 84-66-2 | X | X | X | X | X |
| Diethylstilbestrol | 56-53-1 | AW, OS | ND | ND | ND | X |
| Diethyl sulfate | 64-67-5 | LR | ND | ND | ND | LR |
| Dimethoate | 60-51-5 | HE, HS | ND | ND | ND | X |
| 3,3'-Dimethoxybenzidine | 119-90-4 | X | ND | ND | ND | LR |
| Dimethylaminoazobenzene | 60-11-7 | X | ND | ND | ND | X |
| 7,12-Dimethylbenz(a)-anthracene | 57-97-6 | CP | ND | ND | ND | CP |
| 3,3'-Dimethylbenzidine | 119-93-7 | X | ND | ND | ND | X |
| α,α-Dimethylphenethylamine | 122-09-8 | ND | ND | ND | ND | X |
| 2,4-Dimethylphenol | 105-67-9 | X | X | X | X | X |
| Dimethyl phthalate | 131-11-3 | X | X | X | X | X |
| 1,2-Dinitrobenzene | 528-29-0 | X | ND | ND | ND | X |
| 1,3-Dinitrobenzene | 99-65-0 | X | ND | ND | ND | X |
| 1,4-Dinitrobenzene | 100-25-4 | HE | ND | ND | ND | X |
| 4,6-Dinitro-2-methylphenol | 534-52-1 | X | X | X | X | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 2,4-Dinitrophenol | 51-28-5 | X | X | X | X | X |
| 2,4-Dinitrotoluene | 121-14-2 | X | X | X | X | X |
| 2,6-Dinitrotoluene | 606-20-2 | X | X | X | X | X |
| Dinocap | 39300-45-3 | CP, HS | ND | ND | ND | CP |
| Dinoseb | 88-85-7 | X | ND | ND | ND | X |
| Diphenylamine | 122-39-4 | X | X | X | X | X |
| 5,5-Diphenylhydantoin | 57-41-0 | X | ND | ND | ND | X |
| 1,2-Diphenylhydrazine | 122-66-7 | X | X | X | X | X |
| Di-n-octyl phthalate | 117-84-0 | X | X | X | X | X |
| Disulfoton | 298-04-4 | X | ND | ND | ND | X |
| Endosulfan I | 959-98-8 | X | X | X | X | X |
| Endosulfan II | 33213-65-9 | X | X | X | X | X |
| Endosulfan sulfate | 1031-07-8 | X | X | X | X | X |
| Endrin | 72-20-8 | X | X | X | X | X |
| Endrin aldehyde | 7421-93-4 | X | X | X | X | X |
| Endrin ketone | 53494-70-5 | X | X | ND | X | X |
| EPN | 2104-64-5 | X | ND | ND | ND | X |
| Ethion | 563-12-2 | X | ND | ND | ND | X |
| Ethyl carbamate | 51-79-6 | DC | ND | ND | ND | X |
| Ethyl methanesulfonate | 62-50-0 | X | ND | ND | ND | X |
| Famphur | 52-85-7 | X | ND | ND | ND | X |
| Fensulfothion | 115-90-2 | X | ND | ND | ND | X |
| Fenthion | 55-38-9 | X | ND | ND | ND | X |
| Fluchloralin | 33245-39-5 | X | ND | ND | ND | X |
| Fluoranthene | 206-44-0 | X | X | X | X | X |
| Fluorene | 86-73-7 | X | X | X | X | X |
| 2-Fluorobiphenyl (surr) | 321-60-8 | X | X | X | X | X |
| 2-Fluorophenol (surr) | 367-12-4 | X | X | X | X | X |
| Heptachlor | 76-44-8 | X | X | X | X | X |
| Heptachlor epoxide | 1024-57-3 | X | X | X | X | X |
| Hexachlorobenzene | 118-74-1 | X | X | X | X | X |
| Hexachlorobutadiene | 87-68-3 | X | X | X | X | X |
| Hexachlorocyclopentadiene | 77-47-4 | X | X | X | X | X |
| Hexachloroethane | 67-72-1 | X | X | X | X | X |
| Hexachlorophene | 70-30-4 | AW, CP | ND | ND | ND | CP |
| Hexachloropropene | 1888-71-7 | X | ND | ND | ND | X |
| Hexamethylphosphoramide | 680-31-9 | X | ND | ND | ND | X |
| Hydroquinone | 123-31-9 | ND | ND | ND | ND | X |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | X | X | X | X | X |
| Isodrin | 465-73-6 | X | ND | ND | ND | X |
| Isophorone | 78-59-1 | X | X | X | X | X |
| Isosafrole | 120-58-1 | DC | ND | ND | ND | X |
| Kepone | 143-50-0 | X | ND | ND | ND | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| Leptophos | 21609-90-5 | X | ND | ND | ND | X |
| Malathion | 121-75-5 | HS | ND | ND | ND | X |
| Maleic anhydride | 108-31-6 | HE | ND | ND | ND | X |
| Mestranol | 72-33-3 | X | ND | ND | ND | X |
| Methapyrilene | 91-80-5 | X | ND | ND | ND | X |
| Methoxychlor | 72-43-5 | X | ND | ND | ND | X |
| 3-Methylcholanthrene | 56-49-5 | X | ND | ND | ND | X |
| 4,4'-Methylenebis (2-chloroaniline) | 101-14-4 | OE, OS | ND | ND | ND | LR |
| 4,4'-Methylenebis(*N*,*N*-dimethyl-aniline) | 101-61-1 | X | X | ND | ND | ND |
| Methyl methanesulfonate | 66-27-3 | X | ND | ND | ND | X |
| 2-Methylnaphthalene | 91-57-6 | X | X | ND | X | X |
| Methyl parathion | 298-00-0 | X | ND | ND | ND | X |
| 2-Methylphenol | 95-48-7 | X | ND | ND | ND | X |
| 3-Methylphenol | 108-39-4 | X | ND | ND | ND | X |
| 4-Methylphenol | 106-44-5 | X | ND | ND | ND | X |
| Mevinphos | 7786-34-7 | X | ND | ND | ND | X |
| Mexacarbate | 315-18-4 | HE, HS | ND | ND | ND | X |
| Mirex | 2385-85-5 | X | ND | ND | ND | X |
| Monocrotophos | 6923-22-4 | HE | ND | ND | ND | X |
| Naled | 300-76-5 | X | ND | ND | ND | X |
| Naphthalene | 91-20-3 | X | X | X | X | X |
| 1,4-Naphthoquinone | 130-15-4 | X | ND | ND | ND | X |
| 1-Naphthylamine | 134-32-7 | OS | ND | ND | ND | X |
| 2-Naphthylamine | 91-59-8 | X | ND | ND | ND | X |
| Nicotine | 54-11-5 | DC | ND | ND | ND | X |
| 5-Nitroacenaphthene | 602-87-9 | X | ND | ND | ND | X |
| 2-Nitroaniline | 88-74-4 | X | X | ND | X | X |
| 3-Nitroaniline | 99-09-2 | X | X | ND | X | X |
| 4-Nitroaniline | 100-01-6 | X | X | ND | X | X |
| 5-Nitro-*o*-anisidine | 99-59-2 | X | ND | ND | ND | X |
| Nitrobenzene | 98-95-3 | X | X | X | X | X |
| 4-Nitrobiphenyl | 92-93-3 | X | ND | ND | ND | X |
| Nitrofen | 1836-75-5 | X | ND | ND | ND | X |
| 2-Nitrophenol | 88-75-5 | X | X | X | X | X |
| 4-Nitrophenol | 100-02-7 | X | X | X | X | X |
| 5-Nitro-o-toluidine | 99-55-8 | X | X | ND | ND | X |
| Nitroquinoline-1-oxide | 56-57-5 | X | ND | ND | ND | X |
| *N*-Nitrosodi-*n*-butylamine | 924-16-3 | X | ND | ND | ND | X |
| *N*-Nitrosodiethylamine | 55-18-5 | X | ND | ND | ND | X |
| *N*-Nitrosodimethylamine | 62-75-9 | X | X | X | X | X |
| *N*-Nitrosodiphenylamine | 86-30-6 | X | X | X | X | X |
| *N*-Nitrosodi-*n*-propylamine | 621-64-7 | X | X | X | X | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| *N*-Nitrosomethylethylamine | 10595-95-6 | X | ND | ND | ND | X |
| *N*-Nitrosomorpholine | 59-89-2 | ND | ND | ND | ND | X |
| *N*-Nitrosopiperidine | 100-75-4 | X | ND | ND | ND | X |
| *N*-Nitrosopyrrolidine | 930-55-2 | X | ND | ND | ND | X |
| Octamethyl pyrophosphoramide | 152-16-9 | LR | ND | ND | ND | LR |
| 4,4'-Oxydianiline | 101-80-4 | X | ND | ND | ND | X |
| Parathion | 56-38-2 | X | X | ND | ND | X |
| Pentachlorobenzene | 608-93-5 | X | ND | ND | ND | X |
| Pentachloronitrobenzene | 82-68-8 | X | ND | ND | ND | X |
| Pentachlorophenol | 87-86-5 | X | X | X | X | X |
| Phenacetin | 62-44-2 | X | ND | ND | ND | X |
| Phenanthrene | 85-01-8 | X | X | X | X | X |
| Phenobarbital | 50-06-6 | X | ND | ND | ND | X |
| Phenol | 108-95-2 | DC | X | X | X | X |
| 1,4-Phenylenediamine | 106-50-3 | X | ND | ND | ND | X |
| Phorate | 298-02-2 | X | ND | ND | ND | X |
| Phosalone | 2310-17-0 | HS | ND | ND | ND | X |
| Phosmet | 732-11-6 | HS | ND | ND | ND | X |
| Phosphamidon | 13171-21-6 | HE | ND | ND | ND | X |
| Phthalic anhydride | 85-44-9 | CP, HE | ND | ND | ND | CP |
| 2-Picoline (2-Methylpyridine) | 109-06-8 | X | X | ND | ND | ND |
| Piperonyl sulfoxide | 120-62-7 | X | ND | ND | ND | X |
| Pronamide | 23950-58-5 | X | ND | ND | ND | X |
| Propylthiouracil | 51-52-5 | LR | ND | ND | ND | LR |
| Pyrene | 129-00-0 | X | X | X | X | X |
| Resorcinol | 108-46-3 | DC, OE | ND | ND | ND | X |
| Safrole | 94-59-7 | X | ND | ND | ND | X |
| Strychnine | 57-24-9 | AW, OS | ND | ND | ND | X |
| Sulfallate | 95-06-7 | X | ND | ND | ND | X |
| Terbufos | 13071-79-9 | X | ND | ND | ND | X |
| 1,2,4,5-Tetrachlorobenzene | 95-94-3 | X | ND | ND | ND | X |
| 2,3,4,6-Tetrachlorophenol | 58-90-2 | X | ND | ND | ND | X |
| Tetrachlorvinphos | 961-11-5 | X | ND | ND | ND | X |
| Tetraethyl dithiopyrophosphate | 3689-24-5 | X | X | ND | ND | ND |
| Tetraethyl pyrophosphate | 107-49-3 | X | ND | ND | ND | X |
| Thionazine | 297-97-2 | X | ND | ND | ND | X |
| Thiophenol (Benzenethiol) | 108-98-5 | X | ND | ND | ND | X |
| Toluene diisocyanate | 584-84-9 | HE | ND | ND | ND | X |
| *o*-Toluidine | 95-53-4 | X | ND | ND | ND | X |
| Toxaphene | 8001-35-2 | X | X | X | X | X |
| 1,2,4-Trichlorobenzene | 120-82-1 | X | X | X | X | X |
| 2,4,5-Trichlorophenol | 95-95-4 | X | X | ND | X | X |
| 2,4,6-Trichlorophenol | 88-06-2 | X | X | X | X | X |

| | | Appropriate Preparation Techniques[b] | | | | |
|---|---|---|---|---|---|---|
| Compounds | CAS No[a] | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| Trifluralin | 1582-09-8 | X | ND | ND | ND | X |
| 2,4,5-Trimethylaniline | 137-17-7 | X | ND | ND | ND | X |
| Trimethyl phosphate | 512-56-1 | HE | ND | ND | ND | X |
| 1,3,5-Trinitrobenzene | 99-35-4 | X | ND | ND | ND | X |
| Tris(2,3-dibromopropyl) phosphate | 126-72-7 | X | ND | ND | ND | LR |
| Tri-*p*-tolyl phosphate | 78-32-0 | X | ND | ND | ND | X |
| *O*,*O*,*O*-Triethyl phosphorothioate | 126-68-1 | X | ND | ND | ND | X |

[a] Chemical Abstract Service Registry Number
[b] See Sec. 1.2 for other acceptable preparation methods.

KEY TO ANALYTE LIST

AW = Adsorption to walls of glassware during extraction and storage.
CP = Nonreproducible chromatographic performance.
DC = Unfavorable distribution coefficient.
HE = Hydrolysis during extraction accelerated by acidic or basic conditions.
HS = Hydrolysis during storage potential.
LR = Low response.
ND = Not determined.
OE = Oxidation during extraction accelerated by basic conditions.
OS = Oxidation during storage potential.
X = Historically, adequate recovery can be obtained by this technique. However, actual recoveries may vary depending on the extraction efficiency, the number of constituents being analyzed concurrently, and the analytical instrumentation.

1.2 In addition to the sample preparation methods listed in the above analyte list, Method 3535 describes a solid-phase extraction procedure that may be applied to the extraction of semivolatiles from TCLP leachates (see Tables 16 and 17 of this method for performance data). Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (see Table 11 of this method for surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (see Table 12 of this method for performance data), Method 3561 describes a supercritical fluid device for the extraction of PAHs from solids (see Tables 13, 14, and 15 of this method for performance data), and Method 3546 provides an extraction procedure employing commercially available microwave equipment to extract semivolatiles while using less solvent and taking less time than procedures such as a Soxhlet extraction (see Tables 19 through 23 of this method for the applicable performance data). (The tabulated data are provided for guidance purposes only.)

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 polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and 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 ions that have been evaluated.

In most cases, this method is not appropriate for the quantitation of multicomponent analytes, e.g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analytes. 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. Refer to Methods 8081 and 8082 for guidance on calibration and quantitation of multicomponent analytes such as the Aroclors, Toxaphene, and Chlordane.

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

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

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 expected to be present.

1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

1.4.5 N-nitrosodiphenylamine decomposes in the gas chromatographic 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 the stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using azobenzene. Under these poor compound separation circumstances the results for either of these compounds should be reported as a combined concentration.

1.4.7 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4.8 Pyridine 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. Therefore, if pyridine is to be determined in addition to other target analytes, it may be necessary to perform separate analyses. In addition, pyridine may be lost during the evaporative concentration of the sample extract. As a result, many of the extraction methods listed above may yield low recoveries unless great care is exercised during the concentration steps. For this reason, analysts may wish to consider the use of extraction techniques such as pressurized fluid extraction (Method 3545), microwave extraction (Method 3546), or supercritical fluid extraction, which involve smaller extract volumes, thereby reducing or eliminating the need for evaporative concentration techniques for many applications.

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

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

1.5 The lower limits of quantitation for this method when determining an individual compound are approximately 660 µg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples (see Table 2). Lower limits of quantitation will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector. The lower limits of quantitation listed in Table 2 are provided for guidance and may not always be achievable.

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 quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the 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 EPA 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 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 gas chromatograph/mass spectrometers 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 gas chromatography/mass spectrometry (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 gas chromatograph (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 a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using an appropriate calibration curve for the intended application.

2.4 This method includes specific calibration and quality control 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, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Four for general guidance on the cleaning of glassware. Also 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.

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. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

## 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 file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

## 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 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph -- An analytical system equipped with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

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 (J&W Scientific DB-5 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 Mass spectrometer

6.1.3.1 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer 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 mass spectrometer 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/NIST Library. The mass spectrometer must be capable of producing a mass spectrum for DFTPP which meets the criteria as outlined in 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 mass spectrometer source.

6.1.5 Data system -- A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should 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. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

6.1.6 Guard column (optional) -- (J&W deactivated fused-silica, 0.25-mm ID x 6-m, or equivalent) between the injection port and the analytical column joined with column connectors (Agilent Catalog No. 5062-3556, or equivalent).

6.2 Syringe -- 10-µL.

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.

## 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, where such specifications are available. 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, and other approaches and concentrations of the target compounds may be used, as appropriate for the intended application. See Method 8000 for additional information on the preparation of calibration standards.

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 pesticide quality acetone or other 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, protected from light, at #6 EC 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 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

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 Internal standard solutions -- The internal standards recommended 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). Other compounds may be used as internal standards as long as the criteria in Sec. 11.3.2 are met.

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 internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store away from any light source at #6 EC when not in use (-10 EC is recommended). When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

7.5.2 If a more sensitive mass spectrometer is employed to achieve lower quantitation levels, a more dilute internal standard solution may be required. Area counts of the internal standard peaks should be between 50-200% of the area of the target analytes in the mid-point calibration analysis.

7.6 GC/MS tuning standard -- A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL each of 4,4'-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 EC when not in use (-10 EC is recommended). If a more sensitive mass spectrometer is employed to achieve lower quantitation levels, a more dilute tuning 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 -- A minimum of five calibration standards should be prepared at different concentrations. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives 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 quantitation and quantitative results are to be reported by this method.

7.7.1 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).

7.7.2 Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the internal standard solution prior to analysis. All standards should be stored away from any light source at #6 EC when not in use (-10 EC is recommended), and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared, as necessary, and stored at #6 EC. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

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-$d_{14}$. See Method 3500 for instructions on preparing the surrogate solutions.

NOTE: In the presence of samples containing residual chlorine, phenol-$d_6$ has been known to react to form chlorinated phenolic compounds that are not detected as the original spiked surrogate. 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.

7.8.1 Surrogate standard check -- Determine what the appropriate concentration should be for the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards. It is recommended that this check be done whenever a new surrogate spiking solution is prepared.

NOTE: Method 3561 (SFE Extraction of PAHs) recommends the use of bromobenzene and p-quaterphenyl to better cover the range of PAHs listed in the method.

7.8.2 If a more sensitive mass spectrometer is employed to achieve lower quantitation levels, a more dilute surrogate solution may be necessary.

7.9 Matrix spike and laboratory control standards -- See Method 3500 for instructions on preparing the matrix spike standard. The same standard may be used as the laboratory control standard (LCS) and the spiking solution should be the same source as used for the initial calibration standards to restrict the influence of standard accuracy on the determination of recovery through preparation and analysis.

7.9.1 Matrix spike check -- Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery. It is recommended that this check be done whenever a new matrix spiking solution is prepared.

7.9.2 If a more sensitive mass spectrometer is employed to achieve lower quantitation levels, a more dilute matrix and LCS spiking solution may be necessary.

7.9.3 Some projects may require the spiking of the specific compounds of interest, since the spiking compounds listed in Method 3500 would not be representative of the compounds of interest required for the project. When this occurs, the matrix and LCS spiking standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

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

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Four, "Organic Analytes."

8.2 Store the sample extracts at #6 EC, 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 quality control (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 that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control 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 8000, 5000, 3500, or 3600.

9.3 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, discussions regarding the instrument QC requirements listed below can be found in the referenced sections of this method:

- The GC/MS must be tuned to meet the recommended DFTPP criteria prior to the initial calibration and for each 12-hr period during which analyses are performed. See Secs. 11.3.1 and 11.4.1 for further details.

- There must be an initial calibration of the GC/MS system as described in Sec. 11.3. In addition, the initial calibration curve should be verified immediately after performing the standard analyses using a second source standard (prepared using standards different from the calibration standards). The suggested acceptance limits for this initial calibration verification analysis are 70 - 130%. Alternative acceptance limits may be appropriate based on the desired project-specific data quality objectives. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results could be used for screening purposes and would be considered estimated values.

- The GC/MS system must meet the calibration verification acceptance criteria in Sec. 11.4, each 12 hrs.

- The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 11.6.1.

9.4 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.5 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, a method blank must be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the lab should monitor the preparation and/or analysis 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 need to be prepared for each set of reagents.

9.6 Sample quality control for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual 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 a knowledge of the samples in the sample batch. 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 MS/MSD.

9.6.2 A laboratory control sample (LCS) should be included with each analytical 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 Also see Method 8000 for the details on carrying out sample quality control 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 Surrogate recoveries

If surrogates are used, 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 an approved project plan.

9.8 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. When any changes are made to the system (e.g., the column is changed, a septum is changed), see the guidance in Method 8000 regarding whether recalibration of the system must take place.

9.9 It is recommended that the laboratory adopt additional quality assurance 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 Sec 11.3 for information on calibration and standardization.

## 11.0 PROCEDURE

11.1 Sample preparation

11.1.1 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, 3520, 3535 |
| Soil/sediment | 3540, 3541, 3545, 3546, 3550, 3560, 3561 |
| Waste | 3540, 3541, 3545, 3546, 3550, 3560, 3561, 3580 |

11.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10-µL syringe may be appropriate. The quantitation limit is very high (approximately 10,000 µg/L). Therefore, it is only appropriate where concentrations in excess of 10,000 µg/L are expected.

11.2 Extract cleanup -- Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. 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 |
|---|---|
| Aniline and aniline derivatives | 3620 |
| Phenols | 3630, 3640, 8041[a] |
| Phthalate esters | 3610, 3620, 3640 |
| Nitrosamines | 3610, 3620, 3640 |
| Organochlorine pesticides | 3610, 3620, 3630, 3640, 3660 |
| PCBs | 3620, 3630, 3660, 3665 |
| Nitroaromatics and cyclic ketones | 3620, 3640 |
| Polynuclear aromatic hydrocarbons | 3611, 3630, 3640 |
| Haloethers | 3620, 3640 |
| Chlorinated hydrocarbons | 3620, 3640 |
| Organophosphorus pesticides | 3620 |
| Petroleum waste | 3611, 3650 |
| All base, neutral, and acid priority pollutants | 3640 |

[a] Method 8041 includes a derivatization technique and a GC/ECD analysis, if interferences are encountered on GC/FID.

11.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

| | |
|---|---|
| Mass range: | 35-500 amu |
| Scan time: | #1 sec/scan |
| Initial temperature: | 40 EC, hold for 4 min |
| Temperature program: | 40-320 EC at 10 EC/min |
| Final temperature: | 320 EC, hold until 2 min after benzo[g,h,i]perylene elutes |
| Injector temperature: | 250-300 EC |
| Transfer line temperature: | 250-300 EC |
| Source temperature: | According to manufacturer's specifications |
| Injector: | Grob-type, splitless |
| Injection volume: | 1-2 µL |
| Carrier gas: | Hydrogen at 50 cm/sec or helium at 30 cm/sec |
| Ion trap only: | Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations |

Split injection is allowed if the sensitivity of the mass spectrometer 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 mass spectrometer instrumentation 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 is selected, the system calibration must not begin until the tuning acceptance criteria are met with the sample analyses performed under the same conditions as the calibration standards.

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: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans 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 tighter than those included in this method or to use other documented criteria provided they are used consistently throughout the initial calibration, calibration verification, and sample analyses.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

11.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and 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:

$$\text{TailingFactor} = \frac{BC}{AB}$$

Where the peak is defined as follows: AC is the width at 10% height; 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.)

11.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6 to12 in. of the capillary 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 internal standards selected in Sec. 7.5 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). 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 150 for quantitation).

11.3.3 Analyze 1-2 µL of each calibration standard (containing the compounds for quantitation and the appropriate surrogates and internal standards) and tabulate the area of the primary ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 7.7 and Method 8000). Alternate injection volumes may be used if the applicable quality control requirements for using this method are met. The injection volume must be the same for all standards and sample extracts. Figure 2 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

11.3.4 Initial calibration calculations

Calculate response factors (RFs) for each target analyte relative to one of the internal standards (see Table 5) as follows:

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

where:

$A_s$ = Peak area (or height) of the analyte or surrogate.
$A_{is}$ = Peak area (or height) of the internal standard.
$C_s$ = Concentration of the analyte or surrogate, in µg/L.
$C_{is}$ = Concentration of the internal standard, in µg/L.

11.3.4.1 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes, as noted in Table 4, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also 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.

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

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

$RF_i$ = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

11.3.4.2 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec. 11.3.

11.3.5 Evaluation of retention times -- The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

$$RRT = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

11.3.6 Linearity of target analytes -- If the RSD of any target analyte is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Sec. 11.7.2).

11.3.6.1 If the RSD of any target analyte is greater than 20%, refer to Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed. The average RF should not be used for compounds that have an RSD greater than 20% unless the concentration is reported as estimated.

11.3.6.2 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.

11.3.6.3 Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet either the 20% RSD, minimum correlation coefficient criteria (0.99), or the acceptance criteria for alternative calibration procedures in Method 8000. Any calibration method described in Method 8000 may be used, but it should be used consistently. It is considered inappropriate once the calibration analyses are completed to select an alternative calibration procedure in order to pass the recommended criteria on a case-by-case basis. 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 adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit.

11.4 GC/MS calibration verification -- Calibration verification consists of three steps that are performed at the beginning of each 12-hr analytical shift.

11.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng or less of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria as outlined in Sec. 11.3.1 before sample analysis begins. These criteria must be demonstrated each 12-hr shift during which samples are analyzed.

11.4.2 The initial calibration function for each target analyte should be checked immediately after the first occurrence in the region of the middle of the calibration range with a standard from a source different from that used for the initial calibration. The value determined from the second source check should be within 30% of the expected concentration. An alternative recovery limit may be appropriate based on the desired project-specific data quality objectives. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results could be used for screening purposes and would be considered estimated values.

11.4.3 The initial calibration (Sec. 11.3) for each compound of interest should be verified once every 12 hrs prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard (containing all the compounds for quantitation) at a concentration either near the midpoint concentration for the calibrating range of the GC/MS or near the action level for the project. The results must be compared against the most recent initial calibration curve and should meet the verification acceptance criteria provided in Secs. 11.4.5 through 11.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

11.4.4 A method blank should be analyzed prior to sample analyses in order to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Method 8000 for information regarding method blank performance criteria.

11.4.5 Calibration verification standard criteria

11.4.5.1 Each of the most common target analytes in the calibration verification standard should meet the minimum response factors as noted in Table 4. This criteria is particularly important when the common target analytes are also critical project-required compounds. This is the same check that is applied during the initial calibration.

11.4.5.2 If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

11.4.5.3 All target compounds of interest must be evaluated using a 20% criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Method 8000 for guidance on calculating percent difference and drift.

11.4.5.4 If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration 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. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

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

11.4.5.6 The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve (see Method 8000 for additional details). It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Other recovery criteria may be applicable depending on the project's data quality objectives and for those situations the minimum quantitation check criteria should be outlined in a laboratory standard operating procedure, or a project-specific Quality Assurance Project Plan. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

11.4.6 Internal standard retention time -- The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 sec from that in the mid-point standard level of the most recent initial calibration sequence, 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.

11.4.7 Internal standard response -- If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.5 GC/MS analysis of samples

11.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/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.

11.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard 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 volume to be injected should include an appropriate concentration that is within the calibration range of base/neutral and acid surrogates using the surrogate solution as noted in Sec. 7.8. The injection volume must be the same volume that was used for the calibration standards.

11.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard solution must be added to the diluted extract to maintain the same concentration as in the calibration standards (usually 40 ng/µL, or other concentrations as appropriate, if a more sensitive GC/MS system is being used). Secondary ion quantitation should be used only when there are sample interferences with the primary ion.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times in all samples, spikes, blanks, and standards to effectively check drifting, method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance. Internal standard responses (area counts) must be monitored in all samples, spikes, blanks for similar reasons. If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified.

11.5.4.1 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.

11.5.4.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

11.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below:

$$\text{Dwell Time for the Group} = \frac{\text{Scan Time (msec)}}{\text{Total Ions in the Group}}$$

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 statement of work (SOW). See the SIM sections from the following CLP SOW for further details: EPA CLP Organics SOW. (Reference 14)

11.6 Analyte identification

11.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference 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. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

11.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

11.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) Use professional judgement in interpretation where interferences are observed.

11.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

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 coelute (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 coeluting compound.

11.6.2 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. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

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.

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. 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 coelution 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. retention time updates, integration parameter files, etc). The analyst should seek to minimize manual integration by properly maintaining the instrument, updating retention times, and configuring peak integration parameters.

11.7.2 If the RSD of a compound's response factor is 20% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (Sec. 11.3.4). See Method 8000 for the equations describing internal standard calibration and either linear or non-linear calibrations.

11.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 11.6.2) should be estimated. The same formula as in Sec. 11.3.4 should be used with the following modifications: The areas $A_x$ 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 internal standard free of interferences.

11.7.5 Quantitation of multicomponent compounds (e.g., Toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, for example by using Methods 8081 or 8082. However, this method (8270) may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/µL in the concentrated sample extract.

11.7.6 Quantitation of multicomponent parameters such as diesel range organics (DROs) and total petroleum hydrocarbons (TPH) using the Method 8270 recommended internal standard quantitation technique is beyond the scope of this method. Typically, analyses for these parameters are performed using GC/FID or GC with a MS detector capability that is available with Method 8015.

11.7.7 Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

## 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.

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 presented in Table 6. Laboratories should generate their own acceptance criteria depending on the extraction and instrument conditions. (See Method 8000.)

13.3 Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of GC performance. These recoveries (using Method 3510 extraction) are presented in Table 7. These data are provided for guidance purposes only.

13.4 Method performance data using Method 3541 (automated Soxhlet extraction) are presented in Tables 8 and 9. 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 1 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 gas chromatography/mass spectrometry 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 7 and are provided for guidance purposes only.

13.5 Surrogate precision and accuracy data are presented in Table 10 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 (8270). These data are provided for guidance purposes only.

13.6 Single laboratory precision and bias data using Method 3545 (pressurized fluid extraction) for semivolatile organic compounds are presented in Table 11. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (250, 2500, and 12,500 µg/kg) on three types of soil (clay, loam and sand). Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by the Perstorp Environmental Soxtec™ (automated Soxhlet). The data in Table 11 represent seven replicate extractions and analyses for each individual sample and were taken from Reference 9. 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 (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 (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 range from 85 to 148% (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 range from 73 to 133% (overall average of 92%) based on the certified value for the marine sediment. The data are found in Tables 12 and 13 and were taken from Reference 10. These data are provided for guidance purposes only.

13.8 Single laboratory precision and accuracy data using Method 3561 (SFE extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (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 range from 60 to 122% (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 EC; Restrictor temperature -- 120 EC; and, trapping fluid -- chloroform (methylene chloride has also been used). The data are found in Table 14 and were taken from Reference 11. These data are provided for guidance purposes only.

13.9 Tables 15 and 16 contain single-laboratory precision and accuracy data for solid-phase extraction of TCLP buffer solutions spiked at two levels and extracted using Method 3535. These data are provided for guidance purposes only.

13.10 Table 17 contains multiple-laboratory data for solid-phase extraction of spiked TCLP soil leachates extracted using Method 3535. These data are provided for guidance purposes only.

13.11 Tables 18 through 22 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 *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency 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 from the American Chemical Society at the address listed in Sec. 14.2.

## 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 IN APPROXIMATE RETENTION TIME ORDER [a]

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| 2-Picoline | 93 | 66,92 |
| Aniline | 93 | 66,65 |
| Phenol | 94 | 65,66 |
| Bis(2-chloroethyl) ether | 93 | 63,95 |
| 2-Chlorophenol | 128 | 64,130 |
| 1,3-Dichlorobenzene | 146 | 148,111 |
| 1,4-Dichlorobenzene-$d_4$ (IS) | 152 | 150,115 |
| 1,4-Dichlorobenzene | 146 | 148,111 |
| Benzyl alcohol | 108 | 79,77 |
| 1,2-Dichlorobenzene | 146 | 148,111 |
| N-Nitrosomethylethylamine | 88 | 42,43,56 |
| Bis(2-chloroisopropyl) ether | 45 | 77,121 |
| Ethyl carbamate | 62 | 44,45,74 |
| Thiophenol (Benzenethiol) | 110 | 66,109,84 |
| Methyl methanesulfonate | 80 | 79,65,95 |
| N-Nitrosodi-n-propylamine | 70 | 42,101,130 |
| Hexachloroethane | 117 | 201,199 |
| Maleic anhydride | 54 | 98,53,44 |
| Nitrobenzene | 77 | 123,65 |
| Isophorone | 82 | 95,138 |
| N-Nitrosodiethylamine | 102 | 42,57,44,56 |
| 2-Nitrophenol | 139 | 109,65 |
| 2,4-Dimethylphenol | 122 | 107,121 |
| p-Benzoquinone | 108 | 54,82,80 |
| Bis(2-chloroethoxy)methane | 93 | 95,123 |
| Benzoic acid | 122 | 105,77 |
| 2,4-Dichlorophenol | 162 | 164,98 |
| Trimethyl phosphate | 110 | 79,95,109,140 |
| Ethyl methanesulfonate | 79 | 109,97,45,65 |
| 1,2,4-Trichlorobenzene | 180 | 182,145 |
| Naphthalene-$d_8$ (IS) | 136 | 68 |
| Naphthalene | 128 | 129,127 |
| Hexachlorobutadiene | 225 | 223,227 |
| Tetraethyl pyrophosphate | 99 | 155,127,81,109 |
| Diethyl sulfate | 139 | 45,59,99,111,125 |
| 4-Chloro-3-methylphenol | 107 | 144,142 |
| 2-Methylnaphthalene | 142 | 141 |
| 2-Methylphenol | 107 | 108,77,79,90 |
| Hexachloropropene | 213 | 211,215,117,106,141 |
| Hexachlorocyclopentadiene | 237 | 235,272 |
| N-Nitrosopyrrolidine | 100 | 41,42,68,69 |
| Acetophenone | 105 | 71,51,120 |
| 3/4-Methylphenol[b] | 107 | 108,77,79,90 |

TABLE 1
(continued)

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| 2,4,6-Trichlorophenol | 196 | 198,200 |
| o-Toluidine | 106 | 107,77,51,79 |
| 2-Chloronaphthalene | 162 | 127,164 |
| N-Nitrosopiperidine | 114 | 42,55,56,41 |
| 1,4-Phenylenediamine | 108 | 80,53,54,52 |
| 1-Chloronaphthalene | 162 | 127,164 |
| 2-Nitroaniline | 65 | 92,138 |
| 5-Chloro-2-methylaniline | 106 | 141,140,77,89 |
| Dimethyl phthalate | 163 | 194,164 |
| Acenaphthylene | 152 | 151,153 |
| 2,6-Dinitrotoluene | 165 | 63,89 |
| Phthalic anhydride | 104 | 76,50,148 |
| o-Anisidine | 108 | 80,123,52 |
| 3-Nitroaniline | 138 | 108,92 |
| Acenaphthene-$d_{10}$ (IS) | 164 | 162,160 |
| Acenaphthene | 154 | 153,152 |
| 2,4-Dinitrophenol | 184 | 63,154 |
| 2,6-Dinitrophenol | 162 | 164,126,98,63 |
| 4-Chloroaniline | 127 | 129,65,92 |
| Isosafrole | 162 | 131,104,77,51 |
| Dibenzofuran | 168 | 139 |
| 2,4-Diaminotoluene | 121 | 122,94,77,104 |
| 2,4-Dinitrotoluene | 165 | 63,89 |
| 4-Nitrophenol | 139 | 109,65 |
| 2-Naphthylamine | 143 | 115,116 |
| 1,4-Naphthoquinone | 158 | 104,102,76,50,130 |
| p-Cresidine | 122 | 94,137,77,93 |
| Dichlorovos | 109 | 185,79,145 |
| Diethyl phthalate | 149 | 177,150 |
| Fluorene | 166 | 165,167 |
| 2,4,5-Trimethylaniline | 120 | 135,134,91,77 |
| N-Nitrosodi-n-butylamine | 84 | 57,41,116,158 |
| 4-Chlorophenyl phenyl ether | 204 | 206,141 |
| Hydroquinone | 110 | 81,53,55 |
| 4,6-Dinitro-2-methylphenol | 198 | 51,105 |
| Resorcinol | 110 | 81,82,53,69 |
| N-Nitrosodiphenylamine | 169 | 168,167 |
| Safrole | 162 | 104,77,103,135 |
| Hexamethyl phosphoramide | 135 | 44,179,92,42 |
| 3-(Chloromethyl)pyridine hydrochloride | 92 | 127,129,65,39 |
| Diphenylamine | 169 | 168,167 |
| 1,2,4,5-Tetrachlorobenzene | 216 | 214,179,108,143,218 |
| 1-Naphthylamine | 143 | 115,89,63 |
| 1-Acetyl-2-thiourea | 118 | 43,42,76 |
| 4-Bromophenyl phenyl ether | 248 | 250,141 |

TABLE 1
(continued)

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| Toluene diisocyanate | 174 | 145,173,146,132,91 |
| 2,4,5-Trichlorophenol | 196 | 198,97,132,99 |
| Hexachlorobenzene | 284 | 142,249 |
| Nicotine | 84 | 133,161,162 |
| Pentachlorophenol | 266 | 264,268 |
| 5-Nitro-o-toluidine | 152 | 77,79,106,94 |
| Thionazine | 107 | 96,97,143,79,68 |
| 4-Nitroaniline | 138 | 65,108,92,80,39 |
| Phenanthrene-$d_{10}$ (IS) | 188 | 94,80 |
| Phenanthrene | 178 | 179,176 |
| Anthracene | 178 | 176,179 |
| 1,4-Dinitrobenzene | 168 | 75,50,76,92,122 |
| Mevinphos | 127 | 192,109,67,164 |
| Naled | 109 | 145,147,301,79,189 |
| 1,3-Dinitrobenzene | 168 | 76,50,75,92,122 |
| Diallate (cis or trans) | 86 | 234,43,70 |
| 1,2-Dinitrobenzene | 168 | 50,63,74 |
| Diallate (trans or cis) | 86 | 234,43,70 |
| Pentachlorobenzene | 250 | 252,108,248,215,254 |
| 5-Nitro-o-anisidine | 168 | 79,52,138,153,77 |
| Pentachloronitrobenzene | 237 | 142,214,249,295,265 |
| 4-Nitroquinoline-1-oxide | 174 | 101,128,75,116 |
| Di-n-butyl phthalate | 149 | 150,104 |
| 2,3,4,6-Tetrachlorophenol | 232 | 131,230,166,234,168 |
| Dihydrosaffrole | 135 | 64,77 |
| Demeton-O | 88 | 89,60,61,115,171 |
| Fluoranthene | 202 | 101,203 |
| 1,3,5-Trinitrobenzene | 75 | 74,213,120,91,63 |
| Dicrotophos | 127 | 67,72,109,193,237 |
| Benzidine | 184 | 92,185 |
| Trifluralin | 306 | 43,264,41,290 |
| Bromoxynil | 277 | 279,88,275,168 |
| Pyrene | 202 | 200,203 |
| Monocrotophos | 127 | 192,67,97,109 |
| Phorate | 75 | 121,97,93,260 |
| Sulfallate | 188 | 88,72,60,44 |
| Demeton-S | 88 | 60,81,89,114,115 |
| Phenacetin | 108 | 180,179,109,137,80 |
| Dimethoate | 87 | 93,125,143,229 |
| Phenobarbital | 204 | 117,232,146,161 |
| Carbofuran | 164 | 149,131,122 |
| Octamethyl pyrophosphoramide | 135 | 44,199,286,153,243 |
| 4-Aminobiphenyl | 169 | 168,170,115 |
| Dioxathion | 97 | 125,270,153 |
| Terbufos | 231 | 57,97,153,103 |

TABLE 1
(continued)

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| α,α-Dimethylphenylamine | 58 | 91,65,134,42 |
| Pronamide | 173 | 175,145,109,147 |
| Aminoazobenzene | 197 | 92,120,65,77 |
| Dichlone | 191 | 163,226,228,135,193 |
| Dinoseb | 211 | 163,147,117,240 |
| Disulfoton | 88 | 97,89,142,186 |
| Fluchloralin | 306 | 63,326,328,264,65 |
| Mexacarbate | 165 | 150,134,164,222 |
| 4,4'-Oxydianiline | 200 | 108,171,80,65 |
| Butyl benzyl phthalate | 149 | 91,206 |
| 4-Nitrobiphenyl | 199 | 152,141,169,151 |
| Phosphamidon | 127 | 264,72,109,138 |
| 2-Cyclohexyl-4,6-Dinitrophenol | 231 | 185,41,193,266 |
| Methyl parathion | 109 | 125,263,79,93 |
| Carbaryl | 144 | 115,116,201 |
| Dimethylaminoazobenzene | 225 | 120,77,105,148,42 |
| Propylthiouracil | 170 | 142,114,83 |
| Benz(a)anthracene | 228 | 229,226 |
| Chrysene-$d_{12}$ (IS) | 240 | 120,236 |
| 3,3'-Dichlorobenzidine | 252 | 254,126 |
| Chrysene | 228 | 226,229 |
| Malathion | 173 | 125,127,93,158 |
| Kepone | 272 | 274,237,178,143,270 |
| Fenthion | 278 | 125,109,169,153 |
| Parathion | 109 | 97,291,139,155 |
| Anilazine | 239 | 241,143,178,89 |
| Bis(2-ethylhexyl) phthalate | 149 | 167,279 |
| 3,3'-Dimethylbenzidine | 212 | 106,196,180 |
| Carbophenothion | 157 | 97,121,342,159,199 |
| 5-Nitroacenaphthene | 199 | 152,169,141,115 |
| Methapyrilene | 97 | 50,191,71 |
| Isodrin | 193 | 66,195,263,265,147 |
| Captan | 79 | 149,77,119,117 |
| Chlorfenvinphos | 267 | 269,323,325,295 |
| Crotoxyphos | 127 | 105,193,166 |
| Phosmet | 160 | 77,93,317,76 |
| EPN | 157 | 169,185,141,323 |
| Tetrachlorvinphos | 329 | 109,331,79,333 |
| Di-n-octyl phthalate | 149 | 167,43 |
| 2-Aminoanthraquinone | 223 | 167,195 |
| Barban | 222 | 51,87,224,257,153 |
| Aramite | 185 | 191,319,334,197,321 |
| Benzo(b)fluoranthene | 252 | 253,125 |
| Nitrofen | 283 | 285,202,139,253 |
| Benzo(k)fluoranthene | 252 | 253,125 |

TABLE 1
(continued)

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| Chlorobenzilate | 251 | 139,253,111,141 |
| Fensulfothion | 293 | 97,308,125,292 |
| Ethion | 231 | 97,153,125,121 |
| Diethylstilbestrol | 268 | 145,107,239,121,159 |
| Famphur | 218 | 125,93,109,217 |
| Tri-p-tolyl phosphate[c] | 368 | 367,107,165,198 |
| Benzo(a)pyrene | 252 | 253,125 |
| Perylene-$d_{12}$ (IS) | 264 | 260,265 |
| 7,12-Dimethylbenz(a)anthracene | 256 | 241,239,120 |
| 5,5-Diphenylhydantoin | 180 | 104,252,223,209 |
| Captafol | 79 | 77,80,107 |
| Dinocap | 69 | 41,39 |
| Methoxychlor | 227 | 228,152,114,274,212 |
| 2-Acetylaminofluorene | 181 | 180,223,152 |
| 4,4'-Methylenebis(2-chloroaniline) | 231 | 266,268,140,195 |
| 3,3'-Dimethoxybenzidine | 244 | 201,229 |
| 3-Methylcholanthrene | 268 | 252,253,126,134,113 |
| Phosalone | 182 | 184,367,121,379 |
| Azinphos-methyl | 160 | 132,93,104,105 |
| Leptophos | 171 | 377,375,77,155,379 |
| Mirex | 272 | 237,274,270,239,235 |
| Tris(2,3-dibromopropyl) phosphate | 201 | 137,119,217,219,199 |
| Dibenz(a,j)acridine | 279 | 280,277,250 |
| Mestranol | 277 | 310,174,147,242 |
| Coumaphos | 362 | 226,210,364,97,109 |
| Indeno(1,2,3-cd)pyrene | 276 | 138,277 |
| Dibenz(a,h)anthracene | 278 | 139,279 |
| Benzo(g,h,i)perylene | 276 | 138,277 |
| 1,2:4,5-Dibenzopyrene | 302 | 151,150,300 |
| Strychnine | 334 | 334,335,333 |
| Piperonyl sulfoxide | 162 | 135,105,77 |
| Hexachlorophene | 196 | 198,209,211,406,408 |
| Aldrin | 66 | 263,220 |
| Aroclor 1016 | 222 | 260,292 |
| Aroclor 1221 | 190 | 224,260 |
| Aroclor 1232 | 190 | 224,260 |
| Aroclor 1242 | 222 | 256,292 |
| Aroclor 1248 | 292 | 362,326 |
| Aroclor 1254 | 292 | 362,326 |
| Aroclor 1260 | 360 | 362,394 |
| α-BHC | 183 | 181,109 |
| β-BHC | 181 | 183,109 |
| δ-BHC | 183 | 181,109 |
| γ-BHC (Lindane) | 183 | 181,109 |
| 4,4'-DDD | 235 | 237,165 |

TABLE 1
(continued)

| Compound | Primary Ion | Secondary Ion(s) |
|---|---|---|
| 4,4'-DDE | 246 | 248,176 |
| 4,4'-DDT | 235 | 237,165 |
| Dieldrin | 79 | 263,279 |
| 1,2-Diphenylhydrazine | 77 | 105,182 |
| 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 |
| 2-Fluorobiphenyl (surr) | 172 | 171 |
| 2-Fluorophenol (surr) | 112 | 64 |
| Heptachlor | 100 | 272,274 |
| Heptachlor epoxide | 353 | 355,351 |
| Nitrobenzene-$d_5$ (surr) | 82 | 128,54 |
| N-Nitrosodimethylamine | 42 | 74,44 |
| Phenol-$d_6$ (surr) | 99 | 42,71 |
| Terphenyl-$d_{14}$ (surr) | 244 | 122,212 |
| 2,4,6-Tribromophenol (surr) | 330 | 332,141 |
| Toxaphene | 159 | 231,233 |

IS = internal standard
surr = surrogate
[a] The data presented are representative of DB-5 type analytical columns
[b] Compounds cannot be separated for quantitation
[c] Substitute for the non-specific mixture, tricresyl phosphate

TABLE 2

EXAMPLE LOWER LIMITS OF QUANTITATION FOR SEMIVOLATILE ORGANICS

| | Lower Limits of Quantitation[a] | |
|---|---|---|
| Compound | Ground water (µg/L) | Low Soil/Sediment[b] (µg/kg) |
| Acenaphthene | 10 | 660 |
| Acenaphthylene | 10 | 660 |
| Acetophenone | 10 | ND |
| 2-Acetylaminofluorene | 20 | ND |
| 1-Acetyl-2-thiourea | 1000 | ND |
| 2-Aminoanthraquinone | 20 | ND |
| Aminoazobenzene | 10 | ND |
| 4-Aminobiphenyl | 20 | ND |
| Anilazine | 100 | ND |
| o-Anisidine | 10 | ND |
| Anthracene | 10 | 660 |
| Aramite | 20 | ND |
| Azinphos-methyl | 100 | ND |
| Barban | 200 | ND |
| Benz(a)anthracene | 10 | 660 |
| Benzo(b)fluoranthene | 10 | 660 |
| Benzo(k)fluoranthene | 10 | 660 |
| Benzoic acid | 50 | 3300 |
| Benzo(g,h,i)perylene | 10 | 660 |
| Benzo(a)pyrene | 10 | 660 |
| p-Benzoquinone | 10 | ND |
| Benzyl alcohol | 20 | 1300 |
| Bis(2-chloroethoxy)methane | 10 | 660 |
| Bis(2-chloroethyl) ether | 10 | 660 |
| Bis(2-chloroisopropyl) ether | 10 | 660 |
| 4-Bromophenyl phenyl ether | 10 | 660 |
| Bromoxynil | 10 | ND |
| Butyl benzyl phthalate | 10 | 660 |
| Captafol | 20 | ND |
| Captan | 50 | ND |
| Carbaryl | 10 | ND |
| Carbofuran | 10 | ND |
| Carbophenothion | 10 | ND |
| Chlorfenvinphos | 20 | ND |
| 4-Chloroaniline | 20 | 1300 |
| Chlorobenzilate | 10 | ND |
| 5-Chloro-2-methylaniline | 10 | ND |
| 4-Chloro-3-methylphenol | 20 | 1300 |

TABLE 2
(continued)

| Compound | Lower Limits of Quantitation[a] Ground water (µg/L) | Low Soil/Sediment[b] (µg/kg) |
|---|---|---|
| 3-(Chloromethyl)pyridine hydrochloride | 100 | ND |
| 2-Chloronaphthalene | 10 | 660 |
| 2-Chlorophenol | 10 | 660 |
| 4-Chlorophenyl phenyl ether | 10 | 660 |
| Chrysene | 10 | 660 |
| Coumaphos | 40 | ND |
| p-Cresidine | 10 | ND |
| Crotoxyphos | 20 | ND |
| 2-Cyclohexyl-4,6-dinitrophenol | 100 | ND |
| Demeton-O | 10 | ND |
| Demeton-S | 10 | ND |
| Diallate (cis or trans) | 10 | ND |
| Diallate (trans or cis) | 10 | ND |
| 2,4-Diaminotoluene | 20 | ND |
| Dibenz(a,j)acridine | 10 | ND |
| Dibenz(a,h)anthracene | 10 | 660 |
| Dibenzofuran | 10 | 660 |
| Dibenzo(a,e)pyrene | 10 | ND |
| Di-n-butyl phthalate | 10 | ND |
| Dichlone | NA | ND |
| 1,2-Dichlorobenzene | 10 | 660 |
| 1,3-Dichlorobenzene | 10 | 660 |
| 1,4-Dichlorobenzene | 10 | 660 |
| 3,3'-Dichlorobenzidine | 20 | 1300 |
| 2,4-Dichlorophenol | 10 | 660 |
| 2,6-Dichlorophenol | 10 | ND |
| Dichlorovos | 10 | ND |
| Dicrotophos | 10 | ND |
| Diethyl phthalate | 10 | 660 |
| Diethylstilbestrol | 20 | ND |
| Diethyl sulfate | 100 | ND |
| Dimethoate | 20 | ND |
| 3,3'-Dimethoxybenzidine | 100 | ND |
| Dimethylaminoazobenzene | 10 | ND |
| 7,12-Dimethylbenz(a)anthracene | 10 | ND |
| 3,3'-Dimethylbenzidine | 10 | ND |
| 2,4-Dimethylphenol | 10 | 660 |
| Dimethyl phthalate | 10 | 660 |
| 1,2-Dinitrobenzene | 40 | ND |

TABLE 2
(continued)

| Compound | Lower Limits of Quantitation[a] Ground water (µg/L) | Low Soil/Sediment[b] (µg/kg) |
|---|---|---|
| 1,3-Dinitrobenzene | 20 | ND |
| 1,4-Dinitrobenzene | 40 | ND |
| 4,6-Dinitro-2-methylphenol | 50 | 3300 |
| 2,4-Dinitrophenol | 50 | 3300 |
| 2,4-Dinitrotoluene | 10 | 660 |
| 2,6-Dinitrotoluene | 10 | 660 |
| Dinocap | 100 | ND |
| Dinoseb | 20 | ND |
| 5,5-Diphenylhydantoin | 20 | ND |
| Di-n-octyl phthalate | 10 | 660 |
| Disulfoton | 10 | ND |
| EPN | 10 | ND |
| Ethion | 10 | ND |
| Ethyl carbamate | 50 | ND |
| Bis(2-ethylhexyl) phthalate | 10 | 660 |
| Ethyl methanesulfonate | 20 | ND |
| Famphur | 20 | ND |
| Fensulfothion | 40 | ND |
| Fenthion | 10 | ND |
| Fluchloralin | 20 | ND |
| Fluoranthene | 10 | 660 |
| Fluorene | 10 | 660 |
| Hexachlorobenzene | 10 | 660 |
| Hexachlorobutadiene | 10 | 660 |
| Hexachlorocyclopentadiene | 10 | 660 |
| Hexachloroethane | 10 | 660 |
| Hexachlorophene | 50 | ND |
| Hexachloropropene | 10 | ND |
| Hexamethylphosphoramide | 20 | ND |
| Indeno(1,2,3-cd)pyrene | 10 | 660 |
| Isodrin | 20 | ND |
| Isophorone | 10 | 660 |
| Isosafrole | 10 | ND |
| Kepone | 20 | ND |
| Leptophos | 10 | ND |
| Malathion | 50 | ND |
| Mestranol | 20 | ND |
| Methapyrilene | 100 | ND |
| Methoxychlor | 10 | ND |

TABLE 2
(continued)

| Compound | Lower Limits of Quantitation[a] Ground water (µg/L) | Low Soil/Sediment[b] (µg/kg) |
|---|---|---|
| 3-Methylcholanthrene | 10 | ND |
| Methyl methanesulfonate | 10 | ND |
| 2-Methylnaphthalene | 10 | 660 |
| Methyl parathion | 10 | ND |
| 2-Methylphenol | 10 | 660 |
| 3-Methylphenol | 10 | ND |
| 4-Methylphenol | 10 | 660 |
| Mevinphos | 10 | ND |
| Mexacarbate | 20 | ND |
| Mirex | 10 | ND |
| Monocrotophos | 40 | ND |
| Naled | 20 | ND |
| Naphthalene | 10 | 660 |
| 1,4-Naphthoquinone | 10 | ND |
| 1-Naphthylamine | 10 | ND |
| 2-Naphthylamine | 10 | ND |
| Nicotine | 20 | ND |
| 5-Nitroacenaphthene | 10 | ND |
| 2-Nitroaniline | 50 | 3300 |
| 3-Nitroaniline | 50 | 3300 |
| 4-Nitroaniline | 20 | ND |
| 5-Nitro-o-anisidine | 10 | ND |
| Nitrobenzene | 10 | 660 |
| 4-Nitrobiphenyl | 10 | ND |
| Nitrofen | 20 | ND |
| 2-Nitrophenol | 10 | 660 |
| 4-Nitrophenol | 50 | 3300 |
| 5-Nitro-o-toluidine | 10 | ND |
| 4-Nitroquinoline-1-oxide | 40 | ND |
| N-Nitrosodi-n-butylamine | 10 | ND |
| N-Nitrosodiethylamine | 20 | ND |
| N-Nitrosodiphenylamine | 10 | 660 |
| N-Nitroso-di-n-propylamine | 10 | 660 |
| N-Nitrosopiperidine | 20 | ND |
| N-Nitrosopyrrolidine | 40 | ND |
| Octamethyl pyrophosphoramide | 200 | ND |
| 4,4'-Oxydianiline | 20 | ND |
| Parathion | 10 | ND |
| Pentachlorobenzene | 10 | ND |

TABLE 2
(continued)

| Compound | Lower Limits of Quantitation[a] Ground water (µg/L) | Low Soil/Sediment[b] (µg/kg) |
|---|---|---|
| Pentachloronitrobenzene | 20 | ND |
| Pentachlorophenol | 50 | 3300 |
| Phenacetin | 20 | ND |
| Phenanthrene | 10 | 660 |
| Phenobarbital | 10 | ND |
| Phenol | 10 | 660 |
| 1,4-Phenylenediamine | 10 | ND |
| Phorate | 10 | ND |
| Phosalone | 100 | ND |
| Phosmet | 40 | ND |
| Phosphamidon | 100 | ND |
| Phthalic anhydride | 100 | ND |
| 2-Picoline | ND | ND |
| Piperonyl sulfoxide | 100 | ND |
| Pronamide | 10 | ND |
| Propylthiouracil | 100 | ND |
| Pyrene | 10 | 660 |
| Resorcinol | 100 | ND |
| Safrole | 10 | ND |
| Strychnine | 40 | ND |
| Sulfallate | 10 | ND |
| Terbufos | 20 | ND |
| 1,2,4,5-Tetrachlorobenzene | 10 | ND |
| 2,3,4,6-Tetrachlorophenol | 10 | ND |
| Tetrachlorvinphos | 20 | ND |
| Tetraethyl pyrophosphate | 40 | ND |
| Thionazine | 20 | ND |
| Thiophenol (Benzenethiol) | 20 | ND |
| o-Toluidine | 10 | ND |
| 1,2,4-Trichlorobenzene | 10 | 660 |
| 2,4,5-Trichlorophenol | 10 | 660 |
| 2,4,6-Trichlorophenol | 10 | 660 |
| Trifluralin | 10 | ND |
| 2,4,5-Trimethylaniline | 10 | ND |
| Trimethyl phosphate | 10 | ND |
| 1,3,5-Trinitrobenzene | 10 | ND |
| Tris(2,3-dibromopropyl) phosphate | 200 | ND |
| Tri-p-tolyl phosphate(h) | 10 | ND |

TABLE 2
(continued)

[a] Sample lower limits of quantitation are highly matrix-dependent and those listed here are provided for guidance and may not always be achievable.

[b] Lower limits of quantitation listed for soil/sediment are based on wet weight. When data are reported on a dry weight basis, the lower limits will be higher based on the % dry weight of each sample. These lower limits are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined

NA = Not Applicable

| Other Matrices | Factor[c] |
|---|---|
| High-concentration soil and sludges by ultrasonic extractor | 7.5 |
| Non-water miscible waste | 75 |

[c]Lower limit of quantitation = (Lower limit of quantitation for low soil/sediment given above in Table 2) x (Factor)

TABLE 3

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

| Mass | Ion Abundance Criteria |
|---|---|
| 51 | 10-80% of Base Peak |
| 68 | < 2% of mass 69 |
| 70 | < 2% of mass 69 |
| 127 | 10-80% of Base Peak |
| 197 | < 2% of mass 198 |
| 198 | Base peak, or > 50% of Mass 442 |
| 199 | 5-9% of mass 198 |
| 275 | 10-60% of Base Peak |
| 365 | > 1% of mass 198 |
| 441 | present but < 24% of mass 442 |
| 442 | Base Peak, or > 50% of mass 198 |
| 443 | 15-24% of mass 442 |

[a] The majority of the data are taken from Reference 13 (Method 525.2).

[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

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| Benzaldehyde | 0.010 |
| Phenol | 0.800 |
| Bis(2-chloroethyl)ether | 0.700 |
| 2-Chlorophenol | 0.800 |
| 2-Methylphenol | 0.700 |
| 2,2'-Oxybis-(1-chloropropane) | 0.010 |
| Acetophenone | 0.010 |
| 4-Methylphenol | 0.600 |
| N-Nitroso-di-n-propylamine | 0.500 |
| Hexachloroethane | 0.300 |
| Nitrobenzene | 0.200 |
| Isophorone | 0.400 |
| 2-Nitrophenol | 0.100 |
| 2,4-Dimethylphenol | 0.200 |
| Bis(2-chloroethoxy)methane | 0.300 |
| 2,4-Dichlorophenol | 0.200 |
| Naphthalene | 0.700 |
| 4-Chloroaniline | 0.010 |
| Hexachlorobutadiene | 0.010 |
| Caprolactam | 0.010 |
| 4-Chloro-3-methylphenol | 0.200 |
| 2-Methylnaphthalene | 0.400 |
| Hexachlorocyclopentadiene | 0.050 |
| 2,4,6-Trichlorophenol | 0.200 |
| 2,4,5-Trichlorophenol | 0.200 |
| 1,1'-Biphenyl | 0.010 |
| 2-Chloronaphthalene | 0.800 |

TABLE 4
(continued)

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| 2-Nitroaniline | 0.010 |
| Dimethyl phthalate | 0.010 |
| 2,6-Dinitrotoluene | 0.200 |
| Acenaphthylene | 0.900 |
| 3-Nitroaniline | 0.010 |
| Acenaphthene | 0.900 |
| 2,4-Dinitrophenol | 0.010 |
| 4-Nitrophenol | 0.010 |
| Dibenzofuran | 0.800 |
| 2,4-Dinitrotoluene | 0.200 |
| Diethyl phthalate | 0.010 |
| 1,2,4,5-Tetrachlorobenzene | 0.010 |
| 4-Chlorophenyl-phenyl ether | 0.400 |
| Fluorene | 0.900 |
| 4-Nitroaniline | 0.010 |
| 4,6-Dinitro-2-methylphenol | 0.010 |
| 4-Bromophenyl-phenyl ether | 0.100 |
| N-Nitrosodiphenylamine | 0.010 |
| Hexachlorobenzene | 0.100 |
| Atrazine | 0.010 |
| Pentachlorophenol | 0.050 |
| Phenanthrene | 0.700 |
| Anthracene | 0.700 |
| Carbazole | 0.010 |
| Di-n-butyl phthalate | 0.010 |
| Fluoranthene | 0.600 |
| Pyrene | 0.600 |
| Butyl benzyl phthalate | 0.010 |
| 3,3'-Dichlorobenzidine | 0.010 |
| Benzo(a)anthracene | 0.800 |

TABLE 4
(continued)

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| Chrysene | 0.700 |
| Bis-(2-ethylhexyl)phthalate | 0.010 |
| Di-n-octyl phthalate | 0.010 |
| Benzo(b)fluoranthene | 0.700 |
| Benzo(k)fluoranthene | 0.700 |
| Benzo(a)pyrene | 0.700 |
| Indeno(1,2,3-cd)pyrene | 0.500 |
| Dibenz(a,h)anthracene | 0.400 |
| Benzo(g,h,i)perylene | 0.500 |
| 2,3,4,6-Tetrachlorophenol | 0.010 |

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 |
| Benzyl alcohol | Benzoic acid | Acenaphthylene |
| Bis(2-chloroethyl) ether | Bis(2-chloroethoxy)methane | 1-Chloronaphthalene |
| Bis(2-chloroisopropyl) ether | 4-Chloroaniline | 2-Chloronaphthalene |
| 2-Chlorophenol | 4-Chloro-3-methylphenol | 4-Chlorophenyl phenyl ether |
| 1,3-Dichlorobenzene | 2,4-Dichlorophenol | Dibenzofuran |
| 1,4-Dichlorobenzene | 2,6-Dichlorophenol | Diethyl phthalate |
| 1,2-Dichlorobenzene | α,α-Dimethyl-phenethylamine | Dimethyl phthalate |
| Ethyl methanesulfonate | 2,4-Dimethylphenol | 2,4-Dinitrophenol |
| 2-Fluorophenol (surr) | Hexachlorobutadiene | 2,4-Dinitrotoluene |
| Hexachloroethane | Isophorone | 2,6-Dinitrotoluene |
| Methyl methanesulfonate | 2-Methylnaphthalene | Fluorene |
| 2-Methylphenol | Naphthalene | 2-Fluorobiphenyl (surr) |
| 4-Methylphenol | Nitrobenzene | Hexachlorocyclopentadiene |
| N-Nitrosodimethylamine | Nitrobenzene-$d_8$ (surr) | 1-Naphthylamine |
| N-Nitroso-di-n-propylamine | 2-Nitrophenol | 2-Naphthylamine |
| Phenol | N-Nitrosodi-n-butylamine | 2-Nitroaniline |
| Phenol-$d_6$ (surr) | N-Nitrosopiperidine | 3-Nitroaniline |
| 2-Picoline | 1,2,4-Trichlorobenzene | 4-Nitroaniline |
| | | 4-Nitrophenol |
| | | Pentachlorobenzene |
| | | 1,2,4,5-Tetrachlorobenzene |
| | | 2,3,4,6-Tetrachlorophenol |
| | | 2,4,6-Tribromophenol (surr) |
| | | 2,4,6-Trichlorophenol |
| | | 2,4,5-Trichlorophenol |

(surr) = surrogate

TABLE 5
(continued)

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

(surr) = surrogate

TABLE 6

EXAMPLE SINGLE LABORATORY PERFORMANCE DATA[a]

| Compound | Test conc. (µg/L) | **&** of 5 replicates (µg/L) | % Recovery of Avg. |
|---|---|---|---|
| Acenaphthene | 50 | 46.7 | 93.4 |
| Acenaphthylene | 50 | 46.1 | 92.2 |
| Aniline | 50 | 8.3 | 16.7 |
| Anthracene | 50 | 48.4 | 96.8 |
| Benzoic acid | 50 | 43.7 | 87.4 |
| Benz(a)anthracene | 50 | 49.6 | 99.2 |
| Benzo(b)fluoranthene | 50 | 49.8 | 99.6 |
| Benzo(k)fluoranthene | 50 | 50.6 | 101 |
| Benzo(a)pyrene | 50 | 47.7 | 95.5 |
| Benzo(g,h,i)perylene | 50 | 52.6 | 105 |
| Benzyl alcohol | 50 | 44.4 | 88.8 |
| Bis(2-chloroethyl) ether | 50 | 44.2 | 88.4 |
| Bis(2-chloroethoxy)methane | 50 | 46.6 | 93.1 |
| Bis(2-chloroisopropyl) ether | 50 | 43.4 | 86.8 |
| Bis(2-ethylhexyl) phthalate | 50 | 50.2 | 100 |
| 4-Bromophenyl phenyl ether | 50 | 48.6 | 97.2 |
| Butyl benzyl phthalate | 50 | 49.6 | 99.3 |
| Carbazole | 50 | 52.1 | 104 |
| 2-Chloroaniline | 50 | 38.9 | 77.7 |
| 4-Chloro-3-methylphenol | 50 | 47.3 | 94.6 |
| 2-Chloronaphthalene | 50 | 45.3 | 90.8 |
| 2-Chlorophenol | 50 | 43.1 | 86.2 |
| 4-Chlorophenyl phenyl ether | 50 | 47.3 | 94.6 |
| Chrysene | 50 | 50.3 | 101 |
| Dibenzofuran | 50 | 47.4 | 94.7 |
| Dibenz(a,h)anthracene | 50 | 51.6 | 103 |
| Di-n-butyl phthalate | 50 | 50.5 | 101 |
| 1,2-Dichlorobenzene | 50 | 35.8 | 71.6 |
| 1,3-Dichlorobenzene | 50 | 33.3 | 66.7 |
| 1,4-Dichlorobenzene | 50 | 34.4 | 68.7 |
| 3,3'-Dichlorobenzidine | 50 | 32.0 | 64.0 |
| 2,4-Dichlorophenol | 50 | 47.4 | 94.8 |
| Diethyl phthalate | 50 | 50.0 | 99.9 |
| Dimethyl phthalate | 50 | 48.5 | 97.0 |
| 2,4-Dimethylphenol | 50 | 31.2 | 62.3 |
| 4,6-Dinitro-2-methylphenol | 50 | 57.6 | 115 |
| 2,4-Dinitrophenol | 50 | 58.7 | 117 |
| 2,4-Dinitrotoluene | 50 | 51.3 | 103 |

TABLE 6
(continued)

| Compound | Test conc. (µg/L) | & of 5 replicates (µg/L) | % Recovery of Avg. |
|---|---|---|---|
| 2,6-Dinitrotoluene | 50 | 50.2 | 100 |
| Di-n-octyl phthalate | 50 | 51.1 | 102 |
| Fluoranthene | 50 | 51.0 | 102 |
| Fluorene | 50 | 48.5 | 97.0 |
| Hexachlorobenzene | 50 | 49.0 | 97.9 |
| Hexachlorobutadiene | 50 | 34.7 | 69.5 |
| Hexachlorocyclopentadiene | 50 | 1.9 | 3.8 |
| Hexachloroethane | 50 | 29.9 | 58.8 |
| Indeno(1,2,3-cd)pyrene | 50 | 51.7 | 103 |
| Isophorone | 50 | 47.1 | 94.3 |
| 2-Methylnaphthalene | 50 | 44.7 | 89.4 |
| 2-Methylphenol | 50 | 41.7 | 83.4 |
| 4-Methylphenol | 50 | 42.6 | 85.2 |
| Naphthalene | 50 | 43.4 | 86.8 |
| 2-Nitroaniline | 50 | 48.4 | 96.7 |
| 3-Nitroaniline | 50 | 46.8 | 93.6 |
| 4-Nitroaniline | 50 | 56.1 | 112 |
| Nitrobenzene | 50 | 47.1 | 94.1 |
| 2-Nitrophenol | 50 | 47.3 | 94.6 |
| 4-Nitrophenol | 50 | 55.4 | 111 |
| N-Nitrosodiphenylamine | 50 | 46.7 | 93.4 |
| N-Nitroso-di-propylamine | 50 | 44.6 | 89.3 |
| Pentachlorophenol | 50 | 56.9 | 114 |
| Phenanthrene | 50 | 49.7 | 99.4 |
| Phenol | 50 | 40.9 | 81.8 |
| Pyrene | 50 | 49.2 | 98.4 |
| 1,2,4-Trichlorobenzene | 50 | 39.1 | 78.2 |
| 2,4,5-Trichlorophenol | 50 | 47.7 | 95.4 |
| 2,4,6-Trichlorophenol | 50 | 49.2 | 98.4 |

x = Average recovery for five initial demonstration of capability measurements, in µg/L

[a] Extraction using acidic pH only with a modified continuous liquid-liquid extractor with hydrophobic membrane according to Method 3520. These values are for guidance only. Appropriate derivation of acceptance criteria for similar extraction conditions may result in much different recovery ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

| | Percent Recovery, Day 0 | | Percent Recovery, Day 7 | |
|---|---|---|---|---|
| Compound | Mean | RSD | Mean | RSD |
| 3-Amino-9-ethylcarbazole | 80 | 8 | 73 | 3 |
| 4-Chloro-1,2-phenylenediamine | 91 | 1 | 108 | 4 |
| 4-Chloro-1,3-phenylenediamine | 84 | 3 | 70 | 3 |
| 1,2-Dibromo-3-chloropropane | 97 | 2 | 98 | 5 |
| Dinoseb | 99 | 3 | 97 | 6 |
| Parathion | 100 | 2 | 103 | 4 |
| 4,4'-Methylenebis(N,N-dimethylaniline) | 108 | 4 | 90 | 4 |
| 5-Nitro-o-toluidine | 99 | 10 | 93 | 4 |
| 2-Picoline | 80 | 4 | 83 | 4 |
| Tetraethyl dithiopyrophosphate | 92 | 7 | 70 | 1 |

Data taken from Reference 6.

TABLE 8

MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE ORGANIC FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)[a]

| | Clay Soil | | Topsoil | |
|---|---|---|---|---|
| Compound | Mean Recovery | RSD | Mean Recovery | RSD |
| 1,3-Dichlorobenzene | 0 | -- | 0 | -- |
| 1,2-Dichlorobenzene | 0 | -- | 0 | -- |
| Nitrobenzene | 0 | -- | 0 | -- |
| Benzal chloride | 0 | -- | 0 | -- |
| Benzotrichloride | 0 | -- | 0 | -- |
| 4-Chloro-2-nitrotoluene | 0 | -- | 0 | -- |
| Hexachlorocyclopentadiene | 4.1 | 15 | 7.8 | 23 |
| 2,4-Dichloronitrobenzene | 35.2 | 7.6 | 21.2 | 15 |
| 3,4-Dichloronitrobenzene | 34.9 | 15 | 20.4 | 11 |
| Pentachlorobenzene | 13.7 | 7.3 | 14.8 | 13 |
| 2,3,4,5-Tetrachloronitrobenzene | 55.9 | 6.7 | 50.4 | 6.0 |
| Benefin | 62.6 | 4.8 | 62.7 | 2.9 |
| alpha-BHC | 58.2 | 7.3 | 54.8 | 4.8 |
| Hexachlorobenzene | 26.9 | 13 | 25.1 | 5.7 |
| delta-BHC | 95.8 | 4.6 | 99.2 | 1.3 |
| Heptachlor | 46.9 | 9.2 | 49.1 | 6.3 |
| Aldrin | 97.7 | 12 | 102 | 7.4 |
| Isopropalin | 102 | 4.3 | 105 | 2.3 |
| Heptachlor epoxide | 90.4 | 4.4 | 93.6 | 2.4 |
| trans-Chlordane | 90.1 | 4.5 | 95.0 | 2.3 |
| Endosulfan I | 96.3 | 4.4 | 101 | 2.2 |
| Dieldrin | 129 | 4.7 | 104 | 1.9 |
| 2,5-Dichlorophenyl-4-nitrophenyl ether | 110 | 4.1 | 112 | 2.1 |
| Endrin | 102 | 4.5 | 106 | 3.7 |
| Endosulfan II | 104 | 4.1 | 105 | 0.4 |
| p,p'-DDT | 134 | 2.1 | 111 | 2.0 |
| 2,3,6-Trichlorophenyl-4'-nitrophenyl ether | 110 | 4.8 | 110 | 2.8 |
| 2,3,4-Trichlorophenyl-4'-nitrophenyl ether | 112 | 4.4 | 112 | 3.3 |
| Mirex | 104 | 5.3 | 108 | 2.2 |

[a] The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4-nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 9

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY AUTOMATED SOXHLET (METHOD 3541)[a]

| Compound | Mean Recovery | RSD |
|---|---|---|
| Phenol | 47.8 | 5.6 |
| Bis(2-chloroethyl)ether | 25.4 | 13 |
| 2-Chlorophenol | 42.7 | 4.3 |
| Benzyl alcohol | 55.9 | 7.2 |
| 2-Methylphenol | 17.6 | 6.6 |
| Bis(2-chloroisopropyl)ether | 15.0 | 15 |
| 4-Methylphenol | 23.4 | 6.7 |
| N-Nitroso-di-n-propylamine | 41.4 | 6.2 |
| Nitrobenzene | 28.2 | 7.7 |
| Isophorone | 56.1 | 4.2 |
| 2-Nitrophenol | 36.0 | 6.5 |
| 2,4-Dimethylphenol | 50.1 | 5.7 |
| Benzoic acid | 40.6 | 7.7 |
| Bis(2-chloroethoxy)methane | 44.1 | 3.0 |
| 2,4-Dichlorophenol | 55.6 | 4.6 |
| 1,2,4-Trichlorobenzene | 18.1 | 31 |
| Naphthalene | 26.2 | 15 |
| 4-Chloroaniline | 55.7 | 12 |
| 4-Chloro-3-methylphenol | 65.1 | 5.1 |
| 2-Methylnaphthalene | 47.0 | 8.6 |
| Hexachlorocyclopentadiene | 19.3 | 19 |
| 2,4,6-Trichlorophenol | 70.2 | 6.3 |
| 2,4,5-Trichlorophenol | 26.8 | 2.9 |
| 2-Chloronaphthalene | 61.2 | 6.0 |
| 2-Nitroaniline | 73.8 | 6.0 |
| Dimethyl phthalate | 74.6 | 5.2 |
| Acenaphthylene | 71.6 | 5.7 |
| 3-Nitroaniline | 77.6 | 5.3 |
| Acenaphthene | 79.2 | 4.0 |
| 2,4-Dinitrophenol | 91.9 | 8.9 |
| 4-Nitrophenol | 62.9 | 16 |
| Dibenzofuran | 82.1 | 5.9 |
| 2,4-Dinitrotoluene | 84.2 | 5.4 |
| 2,6-Dinitrotoluene | 68.3 | 5.8 |

| Compound | Mean Recovery | RSD |
|---|---|---|
| Diethyl phthalate | 74.9 | 5.4 |
| 4-Chlorophenyl-phenyl ether | 67.2 | 3.2 |
| Fluorene | 82.1 | 3.4 |
| 4-Nitroaniline | 79.0 | 7.9 |
| 4,6-Dinitro-2-methylphenol | 63.4 | 6.8 |
| N-Nitrosodiphenylamine | 77.0 | 3.4 |
| 4-Bromophenyl-phenyl ether | 62.4 | 3.0 |
| Hexachlorobenzene | 72.6 | 3.7 |
| Pentachlorophenol | 62.7 | 6.1 |
| Phenanthrene | 83.9 | 5.4 |
| Anthracene | 96.3 | 3.9 |
| Di-n-butyl phthalate | 78.3 | 40 |
| Fluoranthene | 87.7 | 6.9 |
| Pyrene | 102 | 0.8 |
| Butyl benzyl phthalate | 66.3 | 5.2 |
| 3,3'-Dichlorobenzidine | 25.2 | 11 |
| Benzo(a)anthracene | 73.4 | 3.8 |
| Bis(2-ethylhexyl) phthalate | 77.2 | 4.8 |
| Chrysene | 76.2 | 4.4 |
| Di-n-octyl phthalate | 83.1 | 4.8 |
| Benzo(b)fluoranthene | 82.7 | 5.0 |
| Benzo(k)fluoranthene | 71.7 | 4.1 |
| Benzo(a)pyrene | 71.7 | 4.1 |
| Indeno(1,2,3-cd)pyrene | 72.2 | 4.3 |
| Dibenz(a,h)anthracene | 66.7 | 6.3 |
| Benzo(g,h,i)perylene | 63.9 | 8.0 |
| 1,2-Dichlorobenzene | 0 | -- |
| 1,3-Dichlorobenzene | 0 | -- |
| 1,4-Dichlorobenzene | 0 | -- |
| Hexachloroethane | 0 | -- |
| Hexachlorobutadiene | 0 | -- |

[a] Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

TABLE 10

PRECISION AND BIAS VALUES FOR METHOD 3542[1]

| Compound | Mean Recovery | Standard Deviation | % RSD |
|---|---|---|---|
| 2-Fluorophenol | 74.6 | 28.6 | 38.3 |
| Phenol-$d_5$ | 77.8 | 27.7 | 35.6 |
| Nitrobenzene-$d_5$ | 65.6 | 32.5 | 49.6 |
| 2-Fluorobiphenyl | 75.9 | 30.3 | 39.9 |
| 2,4,6-Tribromophenol | 67.0 | 34.0 | 50.7 |
| Terphenyl-$d_{14}$ | 78.6 | 32.4 | 41.3 |

[1] The surrogate values shown in Table 10 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 11

PRESSURIZED FLUID EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

| | Clay | | | Loam | | | Sand | | | Mean |
|---|---|---|---|---|---|---|---|---|---|---|
| Compound | Low | Mid | High | Low | Mid | High | Low | Mid | High | Rec. |
| Phenol | 93.3 | 78.7 | 135.9 | 73.9 | 82.8 | 124.6 | 108.8 | 130.6 | 89.7 | 102.0 |
| Bis(2-chloroethyl) ether | 102.1 | 85.1 | 109.1 | 96.0 | 88.0 | 103.6 | 122.3 | 119.9 | 90.8 | 101.9 |
| 2-Chlorophenol | 100.8 | 82.6 | 115.0 | 93.8 | 88.9 | 111.1 | 115.0 | 115.3 | 91.9 | 101.6 |
| 1,3-Dichlorobenzene | 127.7 | 129.7 | 110.0 | *364.2 | 129.9 | 119.0 | *241.3 | *163.7 | 107.1 | 120.6 |
| 1,4-Dichlorobenzene | 127.9 | 127.0 | 110.5 | *365.9 | 127.8 | 116.4 | *309.6 | *164.1 | 105.8 | 119.2 |
| 1,2-Dichlorobenzene | 116.8 | 115.8 | 101.3 | *159.2 | 113.4 | 105.5 | *189.3 | 134.0 | 100.4 | 112.5 |
| 2-Methylphenol | 98.9 | 82.1 | 119.7 | 87.6 | 89.4 | 111.0 | 133.2 | 128.0 | 92.1 | 104.7 |
| Bis(2-chloroisopropyl)ether | 109.4 | 71.5 | 108.0 | 81.8 | 81.0 | 88.6 | 118.1 | 148.3 | 94.8 | 100.2 |
| o-Toluidine | 100.0 | 89.7 | 117.2 | 100.0 | *152.5 | 120.3 | 100.0 | *199.5 | 102.7 | 110.3 |
| N-Nitroso-di-n-propylamine | 103.0 | 79.1 | 107.7 | 83.9 | 88.1 | 96.2 | 109.9 | 123.3 | 91.4 | 98.1 |
| Hexachloroethane | 97.1 | 125.1 | 111.0 | *245.4 | 117.1 | 128.1 | *566.7 | 147.9 | 103.7 | 118.6 |
| Nitrobenzene | 104.8 | 82.4 | 106.6 | 86.8 | 84.6 | 101.7 | 119.7 | 122.1 | 93.3 | 100.2 |
| Isophorone | 100.0 | 86.4 | 98.2 | 87.1 | 87.5 | 109.7 | 135.5 | 118.4 | 92.7 | 101.7 |
| 2,4-Dimethylphenol | 100.0 | 104.5 | 140.0 | 100.0 | 114.4 | 123.1 | 100.0 | *180.6 | 96.3 | 109.8 |
| 2-Nitrophenol | 80.7 | 80.5 | 107.9 | 91.4 | 86.7 | 103.2 | 122.1 | 107.1 | 87.0 | 96.3 |
| Bis(chloroethoxy)methane | 94.4 | 80.6 | 94.7 | 86.5 | 84.4 | 99.6 | 130.6 | 110.7 | 93.2 | 97.2 |
| 2,4-Dichlorophenol | 88.9 | 87.8 | 111.4 | 85.9 | 87.6 | 103.5 | 123.3 | 107.0 | 92.1 | 98.6 |
| 1,2,4-Trichlorobenzene | 98.0 | 97.8 | 98.8 | 123.0 | 93.7 | 94.5 | 137.0 | 99.4 | 95.3 | 104.2 |
| Naphthalene | 101.7 | 97.2 | 123.6 | 113.2 | 102.9 | 129.5 | *174.5 | 114.0 | 89.8 | 106.1 |
| 4-Chloroaniline | 100.0 | *150.2 | *162.4 | 100.0 | 125.5 | *263.6 | 100.0 | *250.8 | 114.9 | 108.1 |
| Hexachlorobutadiene | 101.1 | 98.7 | 102.2 | 124.1 | 90.3 | 98.0 | 134.9 | 96.1 | 96.8 | 104.7 |
| 4-Chloro-3-methylphenol | 90.4 | 80.2 | 114.7 | 79.0 | 85.2 | 109.8 | 131.6 | 116.2 | 90.1 | 99.7 |
| 2-Methylnaphthalene | 93.2 | 89.9 | 94.6 | 104.1 | 92.2 | 105.9 | 146.2 | 99.1 | 93.3 | 102.1 |
| Hexachlorocyclopentadiene | 100.0 | 100.0 | 0.0 | 100.0 | 100.0 | 6.8 | 100.0 | 100.0 | *238.3 | 75.8 |
| 2,4,6-Trichlorophenol | 94.6 | 90.0 | 112.0 | 84.2 | 91.2 | 103.6 | 101.6 | 95.9 | 89.8 | 95.9 |
| 2,4,5-Trichlorophenol | 84.4 | 91.9 | 109.6 | 96.1 | 80.7 | 103.6 | 108.9 | 83.9 | 87.9 | 94.1 |
| 2-Chloronaphthalene | 100.0 | 91.3 | 93.6 | 97.6 | 93.4 | 98.3 | 106.8 | 93.0 | 92.0 | 96.2 |
| 2-Nitroaniline | 90.0 | 83.4 | 97.4 | 71.3 | 88.4 | 89.9 | 112.1 | 113.3 | 87.7 | 92.6 |
| 2,6-Dinitrotoluene | 83.1 | 90.6 | 91.6 | 86.4 | 90.6 | 90.3 | 104.3 | 84.7 | 90.9 | 90.3 |
| Acenaphthylene | 104.9 | 95.9 | 100.5 | 99.0 | 97.9 | 108.8 | 118.5 | 97.8 | 92.0 | 101.7 |
| 3-Nitroaniline | *224.0 | 115.6 | 97.6 | 100.0 | 111.8 | 107.8 | 0.0 | 111.7 | 99.0 | 92.9 |
| Acenaphthene | 102.1 | 92.6 | 97.6 | 97.2 | 96.9 | 104.4 | 114.2 | 92.0 | 89.0 | 98.4 |
| 4-Nitrophenol | 0.0 | 93.2 | 121.5 | 18.1 | 87.1 | 116.6 | 69.1 | 90.5 | 84.5 | 75.6 |
| 2,4-Dinitrotoluene | 73.9 | 91.9 | 100.2 | 84.7 | 93.8 | 98.9 | 100.9 | 84.3 | 87.3 | 90.7 |

TABLE 11
(continued)

| Compound | Clay | | | Loam | | | Sand | | | Mean |
|---|---|---|---|---|---|---|---|---|---|---|
| | Low | Mid | High | Low | Mid | High | Low | Mid | High | Rec. |
| Dibenzofuran | 89.5 | 91.7 | 109.3 | 98.5 | 92.2 | 111.4 | 113.8 | 92.7 | 90.4 | 98.8 |
| 4-Chlorophenyl phenyl ether | 83.0 | 94.5 | 98.7 | 95.7 | 94.3 | 94.2 | 111.4 | 87.7 | 90.3 | 94.4 |
| Fluorene | 85.2 | 94.9 | 89.2 | 102.0 | 95.5 | 93.8 | 121.3 | 85.7 | 90.9 | 95.4 |
| 4-Nitroaniline | 77.8 | 114.8 | 94.5 | 129.6 | 103.6 | 95.4 | *154.1 | 89.3 | 87.5 | 99.1 |
| N-Nitrosodiphenylamine | 82.6 | 96.7 | 93.8 | 92.9 | 93.4 | 116.4 | 97.5 | 110.9 | 86.7 | 96.8 |
| 4-Bromophenyl phenyl ether | 85.6 | 92.9 | 92.8 | 91.1 | 107.6 | 89.4 | 118.0 | 97.5 | 87.1 | 95.8 |
| Hexachlorobenzene | 95.4 | 91.7 | 92.3 | 95.4 | 93.6 | 83.7 | 106.8 | 94.3 | 90.0 | 93.7 |
| Pentachlorophenol | 68.2 | 85.9 | 107.7 | 53.2 | 89.8 | 88.1 | 96.6 | 59.8 | 81.3 | 81.2 |
| Phenanthrene | 92.1 | 93.7 | 93.3 | 100.0 | 97.8 | 113.3 | 124.4 | 101.0 | 89.9 | 100.6 |
| Anthracene | 101.6 | 95.0 | 93.5 | 92.5 | 101.8 | 118.4 | 123.0 | 94.5 | 90.6 | 101.2 |
| Carbazole | 94.4 | 99.3 | 96.6 | 105.5 | 96.7 | 111.4 | 115.7 | 83.2 | 88.9 | 99.1 |
| Fluoranthene | 109.9 | 101.4 | 94.3 | 111.6 | 96.6 | 109.6 | 123.2 | 85.4 | 92.7 | 102.7 |
| Pyrene | 106.5 | 105.8 | 107.6 | 116.7 | 90.7 | 127.5 | 103.4 | 95.5 | 93.2 | 105.2 |
| 3,3'-Dichlorobenzidine | 100.0 | *492.3 | 131.4 | 100.0 | *217.6 | *167.6 | 100.0 | *748.8 | 100.0 | 116.5 |
| Benzo(a)anthracene | 98.1 | 107.0 | 98.4 | 119.3 | 98.6 | 104.0 | 105.0 | 93.4 | 89.3 | 101.5 |
| Chrysene | 100.0 | 108.5 | 100.2 | 116.8 | 93.0 | 117.0 | 106.7 | 93.6 | 90.2 | 102.9 |
| Benzo(b)fluoranthene | 106.6 | 109.9 | 75.6 | 121.7 | 100.7 | 93.9 | 106.9 | 81.9 | 93.6 | 99.0 |
| Benzo(k)fluoranthene | 102.4 | 105.2 | 88.4 | 125.5 | 99.4 | 95.1 | 144.7 | 89.2 | 78.1 | 103.1 |
| Benzo(a)pyrene | 107.9 | 105.5 | 80.8 | 122.3 | 97.7 | 104.6 | 101.7 | 86.2 | 92.0 | 99.9 |
| Indeno(1,2,3-cd)pyrene | 95.1 | 105.7 | 93.8 | 126.0 | 105.2 | 90.4 | 133.6 | 82.6 | 91.9 | 102.7 |
| Dibenz(a,h)anthracene | 85.0 | 102.6 | 82.0 | 118.8 | 100.7 | 91.9 | 142.3 | 71.0 | 93.1 | 98.6 |
| Benzo(g,h,i)perylene | 98.0 | 0.0 | 81.2 | 0.0 | 33.6 | 78.6 | 128.7 | 83.0 | 94.2 | 66.4 |
| Mean | 95.1 | 94.3 | 101.0 | 95.5 | 96.5 | 104.1 | 113.0 | 100.9 | 92.5 | |

* Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 12

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561 (SFE - SOLID TRAP)

| Compound | Certified Value (mg/kg) | SFE Value[a] (mg/kg) | Percent of Certified Value | SFE RSD |
|---|---|---|---|---|
| Naphthalene | (27.9)[b] | 41.3 ± 3.6 | (148) | 8.7 |
| Acenaphthylene | (0.8) | 0.9 ± 0.1 | (112) | 11.1 |
| Acenaphthene | (0.2) | 0.2 ± 0.01 | (100) | 0.05 |
| Fluorene | (15.3) | 15.6 ± 1.8 | (102) | 11.5 |
| Phenanthrene | 15.8 ± 1.2 | 16.1 ± 1.8 | 102 | 11.2 |
| Anthracene | (1.3) | 1.1 ± 0.2 | (88) | 18.2 |
| Fluoranthene | 23.2 ± 2.0 | 24.1 ± 2.1 | 104 | 8.7 |
| Pyrene | 16.7 ± 2.0 | 17.2 ± 1.9 | 103 | 11.0 |
| Benz(a)anthracene | 8.7 ± 0.8 | 8.8 ± 1.0 | 101 | 11.4 |
| Chrysene | (9.2) | 7.9 ± 0.9 | (86) | 11.4 |
| Benzo(b)fluoranthene | 7.9 ± 0.9 | 8.5 ± 1.1 | 108 | 12.9 |
| Benzo(k)fluoranthene | 4.4 ± 0.5 | 4.1 ± 0.5 | 91 | 12.2 |
| Benzo(a)pyrene | 5.3 ± 0.7 | 5.1 ± 0.6 | 96 | 11.8 |
| Indeno(1,2,3-cd)pyrene | 5.7 ± 0.6 | 5.2 ± 0.6 | 91 | 11.5 |
| Benzo(g,h,i)perylene | 4.9 ± 0.7 | 4.3 ± 0.5 | 88 | 11.6 |
| Dibenz(a,h)anthracene | (1.3) | 1.1 ± 0.2 | (85) | 18.2 |

[a] Relative standard deviations for the SFE values are based on six replicate extractions.

[b] Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 13

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

| Compound | Certified Value (mg/kg) | SFE Value[a] (mg/kg) | Percent of Certified Value | SFE RSD |
|---|---|---|---|---|
| Naphthalene | 9.0 ± 0.7 | 7.4 ± 0.6 | 82 | 8.1 |
| Acenaphthylene | 0.3 ± 0.1 | 0.4 ± 0.1 | 133 | 25.0 |
| Acenaphthene | 4.5 ± 1.5 | 3.3 ± 0.3 | 73 | 9.0 |
| Fluorene | 13.6 ± 3.1 | 10.4 ± 1.3 | 77 | 12.5 |
| Phenanthrene | 85.0 ± 20.0 | 86.2 ± 9.5 | 101 | 11.0 |
| Anthracene | 13.4 ± 0.5 | 12.1 ± 1.5 | 90 | 12.4 |
| Fluoranthene | 60.0 ± 9.0 | 54.0 ± 6.1 | 90 | 11.3 |
| Pyrene | 39.0 ± 9.0 | 32.7 ± 3.7 | 84 | 11.3 |
| Benz(a)anthracene | 14.6 ± 2.0 | 12.1 ± 1.3 | 83 | 10.7 |
| Chrysene | 14.1 ± 2.0 | 12.0 ± 1.3 | 85 | 10.8 |
| Benzo(b)fluoranthene | 7.7 ± 1.2 | 8.4 ± 0.9 | 109 | 10.7 |
| Benzo(k)fluoranthene | 2.8 ± 2.0 | 3.2 ± 0.5 | 114 | 15.6 |
| Benzo(a)pyrene | 7.4 ± 3.6 | 6.6 ± 0.8 | 89 | 12.1 |
| Indeno(1,2,3-cd)pyrene | 5.0 ± 2.0 | 4.5 ± 0.6 | 90 | 13.3 |
| Benzo(g,h,i)perylene | 5.4 ± 1.3 | 4.4 ± 0.6 | 82 | 13.6 |
| Dibenz(a,h)anthracene | 1.3 ± 0.5 | 1.1 ± 0.3 | 85 | 27.3 |

[a] Relative standard deviations for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 14

SINGLE LABORATORY ACCURACY AND PRECISION FOR THE EXTRACTION OF PAHs FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561 (SFE - LIQUID TRAP)

| Compound | Certified Value (mg/kg) | SFE Value[a] (mg/kg) | Percent of Certified Value | SFE RSD |
|---|---|---|---|---|
| Naphthalene | 32.4 ± 8.2 | 29.55 | 91 | 10.5 |
| 2-Methylnaphthalene | 62.1 ± 11.5 | 76.13 | 122 | 2.0 |
| Acenaphthene | 632 ± 105 | 577.28 | 91 | 2.9 |
| Dibenzofuran | 307 ± 49 | 302.25 | 98 | 4.1 |
| Fluorene | 492 ± 78 | 427.15 | 87 | 3.0 |
| Phenanthrene | 1618 ± 340 | 1278.03 | 79 | 3.4 |
| Anthracene | 422 ± 49 | 400.80 | 95 | 2.6 |
| Fluoranthene | 1280 ± 220 | 1019.13 | 80 | 4.5 |
| Pyrene | 1033 ± 285 | 911.82 | 88 | 3.1 |
| Benz(a)anthracene | 252 ± 8 | 225.50 | 89 | 4.8 |
| Chrysene | 297 ± 26 | 283.00 | 95 | 3.8 |
| Benzo(a)pyrene | 97.2 ± 17.1 | 58.28 | 60 | 6.5 |
| Benzo(b)fluoranthene + Benzo(k)fluoranthene | 153 ± 22 | 130.88 | 86 | 10.7 |

[a] Relative standard deviations for the SFE values are based on four replicate extractions.

Data are taken from Reference 11.

TABLE 15

SINGLE LABORATORY RECOVERY DATA FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF BASE/NEUTRAL/ACID EXTRACTABLES FROM SPIKED TCLP BUFFERS
LOW SPIKE LEVEL

| Analyte | Spike Level (µg/L) | Buffer 1 (pH = 2.886) | | Buffer 2 (pH = 4.937) | |
|---|---|---|---|---|---|
| | | Recovery (%) | RSD | Recovery (%) | RSD |
| 1,4-Dichlorobenzene | 3,750 | 63 | 10 | 63 | 9 |
| Hexachloroethane | 1,500 | 55 | 6 | 77 | 4 |
| Nitrobenzene | 1,000 | 82 | 10 | 100 | 5 |
| Hexachlorobutadiene | 250 | 65 | 3 | 56 | 4 |
| 2,4-Dinitrotoluene | 65 | 89 | 4 | 101 | 5 |
| Hexachlorobenzene | 65 | 98 | 5 | 95 | 6 |
| o-Cresol | 100,000 | 83 | 10 | 85 | 5 |
| m-Cresol* | 100,000 | 86 | 8 | 85 | 3 |
| p-Cresol* | 100,000 | * | * | * | * |
| 2,4,6-Trichlorophenol | 1,000 | 84 | 12 | 95 | 12 |
| 2,4,5-Trichlorophenol | 200,000 | 83 | 11 | 88 | 3 |
| Pentachlorophenol | 50,000 | 82 | 9 | 78 | 9 |

Results from seven replicate spiked buffer samples.

* In this study, m-cresol and p-cresol co-eluted and were quantitated as a mixture of both isomers.

Data from Reference 12.

TABLE 16

SINGLE LABORATORY RECOVERY DATA FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF BASE/NEUTRAL/ACID EXTRACTABLES FROM SPIKED TCLP BUFFERS
HIGH SPIKE LEVEL

| Analyte | Spike Level (µg/L) | Buffer 1 (pH = 2.886) | | Buffer 2 (pH = 4.937) | |
|---|---|---|---|---|---|
| | | Recovery (%) | RSD | Recovery (%) | RSD |
| 1,4-Dichlorobenzene | 15,000 | 63 | 10 | 63 | 9 |
| Hexachloroethane | 6,000 | 54 | 7 | 46 | 7 |
| Nitrobenzene | 4,000 | 81 | 4 | 81 | 13 |
| Hexachlorobutadiene | 1,000 | 81 | 5 | 70 | 11 |
| 2,4-Dinitrotoluene | 260 | 99 | 8 | 98 | 3 |
| Hexachlorobenzene | 260 | 89 | 8 | 91 | 9 |
| o-Cresol* | 400,000 | 92 | 15 | 90 | 4 |
| m-Cresol* | 400,000 | 95 | 8 | 82 | 6 |
| p-Cresol* | 400,000 | 82 | 14 | 84 | 7 |
| 2,4,6-Trichlorophenol | 4,000 | 93 | 12 | 104 | 12 |
| 2,4,5-Trichlorophenol | 800,000 | 93 | 14 | 97 | 23 |
| Pentachlorophenol | 200,000 | 84 | 9 | 73 | 8 |

Results from seven replicate spiked buffer samples.

* In this study, recoveries of these compounds were determined from triplicate spikes of the individual compounds into separate buffer solutions.

Data from Reference 12.

TABLE 17

RECOVERY DATA FROM THREE LABORATORIES FOR SOLID-PHASE EXTRACTION (METHOD 3535) OF BASE/NEUTRAL/ACID EXTRACTABLES FROM SPIKED TCLP LEACHATES FROM SOIL SAMPLES

| Buffer 1 pH = 2.886 | | Lab 1 | | | Lab 2 | | | Lab 3 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Spike Level (µg/L)* | %R | RSD | n | %R | RSD | n | %R | RSD | n |
| o-Cresol | 200,000 | 86 | 8 | 7 | 35.3 | 0.7 | 3 | 7.6 | 6 | 3 |
| m-Cresol** | -- | 77 | 8 | 7 | -- | -- | -- | -- | -- | -- |
| p-Cresol** | -- | -- | -- | -- | -- | -- | -- | 7.7 | 11 | 3 |
| 2,4,6-Trichlorophenol | 2,000 | 106 | 6 | 7 | 96.3 | 3.9 | 3 | 44.8 | 5 | 3 |
| 2,4,5-Trichlorophenol | 400,000 | 93 | 3 | 7 | 80.5 | 4.5 | 3 | 63.3 | 11 | 3 |
| Pentachlorophenol | 100,000 | 79 | 2 | 7 | 33.8 | 12.2 | 3 | 29.2 | 13 | 3 |
| 1,4-Dichlorobenzene | 7,500 | 51 | 5 | 7 | 81.3 | 5.3 | 3 | 19.2 | 7 | 3 |
| Hexachloroethane | 3,000 | 50 | 5 | 7 | 66.2 | 2.1 | 3 | 12.6 | 11 | 3 |
| Nitrobenzene | 2,000 | 80 | 8 | 7 | 76.3 | 5.3 | 3 | 63.9 | 12 | 3 |
| Hexachlorobutadiene | 500 | 53 | 8 | 7 | 63.3 | 4.8 | 3 | 9.6 | 9 | 3 |
| 2,4-Dinitrotoluene | 130 | 89 | 8 | 7 | 35.7 | 2.6 | 3 | 58.2 | 17 | 3 |
| Hexachlorobenzene | 130 | 84 | 21 | 7 | 92.3 | 1.6 | 3 | 71.7 | 9 | 3 |

(continued)

TABLE 17
(continued)

| Buffer 2 pH = 4.937 | | Lab 1 | | | Lab 2 | | | Lab 3 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Spike Level (µg/L)* | %R | RSD | n | %R | RSD | n | %R | RSD | n |
| o-Cresol | 200,00 | 97 | 13 | 7 | 37.8 | 4.5 | 3 | 6.1 | 24 | 3 |
| m-Cresol** | -- | 83 | 4 | 7 | -- | -- | -- | 6.0 | 25 | 3 |
| p-Cresol** | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 2,4,6-Trichlorophenol | 2,000 | 104 | 4 | 7 | 91.7 | 8.0 | 3 | 37.7 | 25 | 3 |
| 2,4,5-Trichlorophenol | 400,000 | 94 | 4 | 7 | 85.2 | 0.4 | 3 | 64.4 | 10 | 3 |
| Pentachlorophenol | 100,000 | 109 | 11 | 7 | 41.9 | 28.2 | 3 | 36.6 | 32 | 3 |
| 1,4-Dichlorobenzene | 7,500 | 50 | 5 | 7 | 79.7 | 1.0 | 3 | 26.5 | 68 | 3 |
| Hexachloroethane | 3,000 | 51 | 3 | 7 | 64.9 | 2.0 | 3 | 20.3 | 90 | 3 |
| Nitrobenzene | 2,000 | 80 | 4 | 7 | 79.0 | 2.3 | 3 | 59.4 | 6 | 3 |
| Hexachlorobutadiene | 500 | 57 | 5 | 7 | 60 | 3.3 | 3 | 16.6 | 107 | 3 |
| 2,4-Dinitrotoluene | 130 | 86 | 6 | 7 | 38.5 | 5.2 | 3 | 62.2 | 6 | 3 |
| Hexachlorobenzene | 130 | 86 | 7 | 7 | 91.3 | 0.9 | 3 | 75.5 | 5 | 3 |

* 250-mL aliquots of leachate were spiked. Lab 1 spiked at one-half these levels.

** m-Cresol and p-Cresol coelute. Lab 1 and Lab 3 reported o-Cresol and the sum of — and p-Cresol. Lab 2 reported the sum of all three isomers of Cresol.

Data from Reference 12.

TABLE 18

SINGLE-LABORATORY PAH ANALYSIS DATA FROM A REAL SOIL CONTAMINATED WITH CREOSOTE, USING METHOD 3546
(MICROWAVE EXTRACTION)

| Compound | Concentration (µg/kg) | RSD (%) | REAC values (µg/kg) |
|---|---|---|---|
| Naphthalene | 2,170 | 12.4 | 710,000 |
| 2-Methylnaphthalene | 28,710 | 3.1 | N/R |
| 1-Methylnaphthalene | 33,180 | 2.4 | N/R |
| Biphenyl | 13,440 | 6.0 | N/R |
| 2,6-Dimethylnaphthalene | 52,990 | 3.8 | N/R |
| Acenaphthylene | 16,320 | 3.1 | 21,000 |
| Acenaphthene | 801,210 | 6.0 | 1,700,000 |
| Fluorene | 789,980 | 3.4 | 990,000 |
| Phenanthrene | 1,627,480 | 0.7 | 3,300,000 |
| Anthracene | 346,010 | 4.0 | 360,000 |
| Benzo(a)anthracene | 300,380 | 2.7 | 310,000 |
| Fluoranthene | 1,331,690 | 1.6 | 1,600,000 |
| Pyrene | 1,037,710 | 3.0 | 1,100,000 |
| Chrysene | 293,200 | 3.4 | 320,000 |
| Benzo(b)fluoranthene | 152,000 | 3.8 | 140,000 |
| Benzo(k)fluoranthene | 127,740 | 3.6 | 130,000 |
| Benzo(e)pyrene | 87,610 | 3.9 | N/R |
| Benzo(a)pyrene | 128,330 | 3.9 | 110,000 |
| Perylene | 35,260 | 4.3 | N/R |
| Indeno(123-cd)pyrene | 63,900 | 5.0 | 25,000 |
| Dibenz(a,h)anthracene | 17,290 | 6.9 | N/R |
| Benzo(ghi)perylene | 42,720 | 6.9 | 20,000 |

*n = 4

Soil samples obtained from US EPA Emergency Response Center archive bank through their contract laboratory REAC (Edison, NJ). The standard Soxhlet extraction procedures were performed by REAC three years earlier; this long storage period is believed to account for the low naphthalene recovery data in the present study

REAC data labeled N/R = not reported

TABLE 19

SINGLE-LABORATORY PAH RECOVERY DATA FROM HS-5 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

| Compound | Certified Value (µg/kg) | Confidence Interval (µg/kg) | Recovery (%) |
|---|---|---|---|
| Naphthalene | 250 | 180 - 320 | 76 |
| Acenaphthylene | 150 | * | 107 |
| Acenaphthene | 230 | 130 - 330 | 61 |
| Fluorene | 400 | 300 - 500 | 63 |
| Phenanthrene | 5,200 | 4,200 - 6,200 | 72 |
| Anthracene | 380 | 230 - 530 | 84 |
| Fluoranthene | 8,400 | 5,800 - 10,000 | 81 |
| Pyrene | 5,800 | 4,000 - 7,600 | 69 |
| Benzo(a)anthracene | 2,900 | 1,700 - 4,100 | 53 |
| Chrysene | 2,800 | 1,900 - 3,700 | 76 |
| Benzo(b)fluoranthene | 2,000 | 1,000 - 3,000 | 84 |
| Benzo(k)fluoranthene | 1,000 | 600 - 1,400 | 137 |
| Benzo(a)pyrene | 1,700 | 900 - 2,500 | 52 |
| Indeno(123-cd) pyrene | 1,300 | 600 - 2,000 | 63 |
| Dibenz(a,h)anthracene | 200 | 100 - 300 | 125 |
| Benzo(ghi)perylene | 1,300 | 1000 - 1600 | 64 |

n = 3

* values not certified

The uncertainties represent 90% confidence intervals

TABLE 20

SINGLE-LABORATORY PAH RECOVERY DATA FROM HS-4 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

| Compound | Certified Value (µg/kg) | Confidence Interval (µg/kg) | Recovery (%) |
|---|---|---|---|
| Naphthalene | 150 | * | 54 |
| Acenaphthylene | 150 | * | 82 |
| Acenaphthene | 150 | * | 63 |
| Fluorene | 150 | * | 81 |
| Phenanthrene | 680 | 600 - 760 | 81 |
| Anthracene | 140 | 70 - 210 | 108 |
| Fluoranthene | 1250 | 1,150 - 1,350 | 84 |
| Pyrene | 940 | 820 - 1,060 | 85 |
| Benzo(a)anthracene | 530 | 470 - 580 | 78 |
| Chrysene | 650 | 570 - 730 | 84 |
| Benzo(b)fluoranthene | 700 | 550 - 850 | 84 |
| Benzo(k)fluoranthene | 360 | 310 - 410 | 156 |
| Benzo(a)pyrene | 650 | 570 - 730 | 73 |
| Indeno(123-cd) pyrene | 510 | 360 - 660 | 88 |
| Dibenz(a,h)anthracene | 120 | 70 - 170 | 117 |
| Benzo(ghi)perylene | 580 | 360 - 800 | 91 |

n = 3

* values not certified

The uncertainties represent 90% confidence intervals

TABLE 21

SINGLE-LABORATORY PAH RECOVERY DATA FROM HS-3 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

| Compound | Certified Value (µg/kg) | Confidence Interval (µg/kg) | Recovery (%) |
|---|---|---|---|
| Naphthalene | 9,000 | 8300 - 9,700 | 61 |
| Acenaphthylene | 300 | 200 - 400 | 199 |
| Acenaphthene | 4,500 | 3,000 - 6,000 | 80 |
| Fluorene | 13,300 | 10,200 -16,400 | 58 |
| Phenanthrene | 85,000 | 65000 -105,000 | 87 |
| Anthracene | 13,400 | 12,900 -13,900 | 48 |
| Fluoranthene | 60,000 | 51,000-69,000 | 91 |
| Pyrene | 39,000 | 30,000-48,000 | 86 |
| Benzo(a)anthracene | 14,600 | 12,600-16,600 | 78 |
| Chrysene | 14,100 | 12,100-16,100 | 91 |
| Benzo(b)fluoranthene | 7,700 | 6,500-8,900 | 101 |
| Benzo(k)fluoranthene | 2,800 | 800-4,800 | 275 |
| Benzo(a)pyrene | 7,400 | 3,000-7,000 | 74 |
| Indeno(123-cd)pyrene | 5,400 | 4,100-6,700 | 100 |
| Dibenz(a,h)anthracene | 1,300 | 800-1,800 | 118 |
| Benzo(ghi)perylene | 5,000 | 3,000-7,000 | 99 |

n = 3

* values not certified

The uncertainties represent 90% confidence intervals

TABLE 22

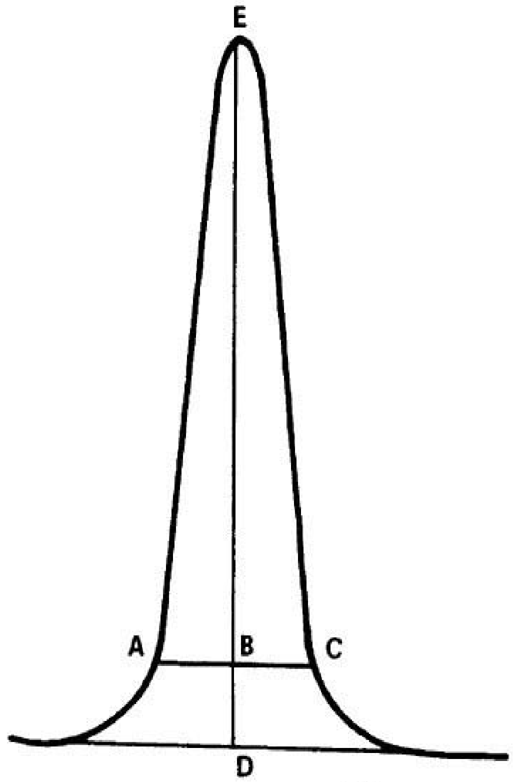
SINGLE-LABORATORY PAH RECOVERY DATA FROM SRM 1941 MARINE SEDIMENT, USING METHOD 3546 (MICROWAVE EXTRACTION)

| Compound | Certified Value (µg/kg) | Recovery (%) |
|---|---|---|
| Naphthalene | 1010 | 97.4 |
| Fluorene | 100 | 100.0 |
| Phenanthrene | 490 | 102.0 |
| Fluoranthene | 980 | 116.7 |
| Pyrene | 810 | 97.3 |
| Benz(a)anthracene | 430 | 89.8 |
| Chrysene | 380 | 130.3 |
| Benzo(b)fluoranthene | 740 | 95.8 |
| Benzo(k)fluoranthene | 360 | 130.2 |
| Benz(e)pyrene | 550 | 81.0 |
| Benzo(a)pyrene | 630 | 76.0 |
| Perylene | 450 | 72.4 |
| Indeno(123-cd)pyrene | 500 | 126.0 |
| Dibenz(a,h)anthracene | 110 | 78.7 |
| Benz(ghi)perylene | 530 | 85.2 |

n = 3

All RSDs < 10%

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

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

FIGURE 2
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD