

METHOD 8270D

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

1.0 SCOPE AND APPLICATION

1.1 Method 8270 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 compounds can be determined by this method:

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|--------------------------|---------------------|---|------|---------------|------|------|
| | | 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(43) | 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 |
| 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(62) | 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 |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|--|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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 | 108-60-1 | 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(55) | ND | ND | ND | X |
| Captan | 133-06-2 | HS(40) | 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 |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|---------------------------------|---------------------|---|------|---------------|------|------|
| | | 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(68) | 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,0E(42) | 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,0S(67) | ND | ND | ND | X |
| Diethyl sulfate | 64-67-5 | LR | ND | ND | ND | LR |
| Dimethoate | 60-51-5 | HE,HS(31) | 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(45) | ND | ND | ND | CP |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|----------------------------|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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(14) | ND | ND | ND | X |
| 4,6-Dinitro-2-methylphenol | 534-52-1 | X | X | X | X | X |
| 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(28) | 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(28) | 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 |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|---|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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(62) | 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(46) | ND | ND | ND | X |
| Kepone | 143-50-0 | X | ND | ND | ND | X |
| Leptophos | 21609-90-5 | X | ND | ND | ND | X |
| Malathion | 121-75-5 | HS(5) | 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(0) | 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(68) | ND | ND | ND | X |
| Mirex | 2385-85-5 | X | ND | ND | ND | X |
| Monocrotophos | 6923-22-4 | HE | ND | ND | ND | X |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|--------------------------------|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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(44) | ND | ND | ND | X |
| 2-Naphthylamine | 91-59-8 | X | ND | ND | ND | X |
| Nicotine | 54-11-5 | DE(67) | 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-Nitrosomethylethylamine | 10595-95-6 | X | ND | ND | ND | X |
| N-Nitrosodiphenylamine | 86-30-6 | X | X | X | X | X |
| N-Nitrosodi-n-propylamine | 621-64-7 | X | X | X | X | 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 pyrophosphoramidate | 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(28) | X | X | X | X |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|-----------------------------------|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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(65) | ND | ND | ND | X |
| Phosmet | 732-11-6 | HS(15) | ND | ND | ND | X |
| Phosphamidon | 13171-21-6 | HE(63) | ND | ND | ND | X |
| Phthalic anhydride | 85-44-9 | CP,HE(1) | 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(10) | ND | ND | ND | X |
| Safrole | 94-59-7 | X | ND | ND | ND | X |
| Strychnine | 57-24-9 | AW,OS(55) | 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(6) | 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 |
| 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(60) | 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 |

| Compounds | CAS No ^a | Appropriate Preparation Techniques ^b | | | | |
|---------------------------------|---------------------|---|------|---------------|------|------|
| | | 3510 | 3520 | 3540/ 3541 | 3550 | 3580 |
| 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 (number in parenthesis is percent recovery).
 HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
 HS = Hydrolysis during storage (number in parenthesis is percent stability).
 LR = Low response.
 ND = Not determined.
 OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
 OS = Oxidation during storage (number in parenthesis is percent stability).
 X = Greater than 70 percent recovery by this technique.

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 (Tables 16 and 17 contain performance data). Method 3542 describes sample preparation for semivolatile organic compounds in air sampled by Method 0010 (Table 11 contains surrogate performance data), Method 3545 describes an automated solvent extraction device for semivolatiles in solids (Table 12 contains performance data), and Method 3561 describes a supercritical fluid device for the extraction of PAHs from solids (see Tables 13, 14, and 15 for performance data).

1.3 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and 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, Method 8270 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 Sec. 7.0 of 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.

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.

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

1.4.7 Pyridine may perform poorly at the GC injection port temperatures listed in the 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) or supercritical fluid extraction, which involve smaller extract volumes, thereby reducing or eliminating the need for evaporative concentration techniques for many applications.

1.4.8 Toluene diisocyanate rapidly hydrolyses 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.

1.4.9 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 estimated quantitation limit (EQL) of Method 8270 for determining an individual compound is approximately 660 $\mu\text{g}/\text{kg}$ (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). EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced 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) 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 a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 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.

3.2 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.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete 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.

4.1.2 Column - 30-m x 0.25-mm ID (or 0.32-mm ID) 1- μ m film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

4.1.3 Mass spectrometer

4.1.3.1 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 in Table 3 when 1 μ L of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

4.1.3.2 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 in Table 3 when 5 or 50 ng are introduced.

4.1.4 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection 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.

4.1.5 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.

4.1.6 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 joiners (Hewlett-Packard Catalog No. 5062-3556, or equivalent).

4.2 Syringe - 10- μ L.

4.3 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers.

4.4 Balance - Analytical, capable of weighing 0.0001 g.

4.5 Bottles - glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

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

5.3.1 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.

5.3.2 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less 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.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.3.4 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.

5.3.5 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.

5.4 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 specifications in Sec. 7.3.2 are met.

5.4.1 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 at -10°C or less when not in use. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.4.2 If a more sensitive mass spectrometer is employed to achieve lower detection 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.

5.5 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. Store at -10°C or less when not in use. If a more sensitive mass spectrometer is employed to achieve lower detection 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.

5.6 Calibration standards - A minimum of five calibration standards should be prepared at five 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 should contain each analyte for detection by this method.

5.6.1 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).

5.6.2 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 at -10°C or less, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The calibration verification standard should be prepared weekly and stored at 4°C. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.7 Surrogate standards - The recommended surrogates are phenol-d₆, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d₅, 2-fluorobiphenyl, and p-terphenyl-d₁₄. See Method 3500 for instructions on preparing the surrogate solutions.

5.7.1 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.

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

5.8 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).

5.8.1 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.

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

5.8.3 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.

5.9 Solvents - Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents. All solvents should be pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

7.0 PROCEDURE

7.1 Sample preparation

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

| <u>Matrix</u> | <u>Methods</u> |
|--------------------------------------|--|
| Air (particulates and sorbent resin) | 3542 |
| Water (including TCLP leachates) | 3510, 3520, 3535 |
| Soil/sediment | 3540, 3541, 3545, 3550, 3560, 3561 |
| Waste | 3540, 3541, 3545, 3550, 3560, 3561, 3580 |

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

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis.

| <u>Analytes of interest</u> | <u>Methods</u> |
|--|-------------------------------|
| Aniline & aniline derivatives | 3620 |
| Phenols | 3630, 3640, 8041 ^a |
| Phthalate esters | 3610, 3620, 3640 |
| Nitrosamines | 3610, 3620, 3640 |
| Organochlorine pesticides & PCBs | 3610, 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.

7.3 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°C, hold for 4 minutes |
| Temperature program: | 40-270°C at 10°C/min |
| Final temperature: | 270°C, hold until benzo[g,h,i]perylene elutes |
| Injector temperature: | 250-300°C |
| Transfer line temperature: | 250-300°C |
| 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.

7.3.1 The GC/MS system must be hardware-tuned using a 50-ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach has been shown to be useful: 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 no more than 20 scans prior to 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.

7.3.1.2 Use the DFTPP mass intensity criteria in Table 3 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions), provided that method performance is not adversely affected.

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

7.3.1.3 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 Sec. 8.0 of Method 8081 for the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

7.3.1.4 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 6-12 in. of the capillary column. The use of a guard column (Sec. 4.1.6) between the injection port and the analytical column may help prolong analytical column performance.

7.3.2 The internal standards selected in Sec. 5.4 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 (i.e. for 1,4-dichlorobenzene-d₄, use 152 m/z for quantitation).

7.3.3 Analyze 1-2 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 5.6 and Method 8000). The injection volume must be the same for all standards and sample extracts. Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

Calculate response factors (RFs) for each target analyte relative to one of the internal standards 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 $\mu\text{g/L}$.
 C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

7.3.4 System performance check compounds (SPCCs)

7.3.4.1 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System performance check compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol.

7.3.4.2 The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

7.3.4.3 If the minimum response factors are not met, the system must be evaluated, and corrective action must 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. This check must be met before sample analysis begins.

7.3.5 Calibration check compounds (CCCs)

7.3.5.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.5.2 Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC (see Table 4) must be less than or equal to 30%.

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

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

7.3.5.3 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec. 7.3.

7.3.5.4 If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, then refer to Sec. 7.0 of Method 8000.

7.3.6 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.

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

7.3.7 Linearity of target analytes - If the RSD of any target analytes is 15% 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. 7.6.2).

7.3.7.1 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 in 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.

NOTE: Method 8000 designates a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.7.2 When the RSD exceeds 15%, 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.

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

7.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The

results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

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.

7.4.3 A method blank should be analyzed either after the calibration standard, or at any other time during the analytical shift, 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 Sec. 8.0 of Method 8000B for method blank performance criteria.

7.4.4 System performance check compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must 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. This check must be met before sample analysis begins.

7.4.5 Calibration check compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Table 4 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 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 seconds 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.

7.4.7 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.

7.5 GC/MS analysis of samples

7.5.1 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.

7.5.2 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 concentrated sample extract obtained from sample preparation.

7.5.3 Inject a 1-2 μL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 7.3). The volume to be injected should contain 100 ng of base/neutral and 200 ng of acid surrogates (assuming 100% recovery), unless a more sensitive GC/MS system is being used and the surrogate solution is less concentrated than that listed in Sec. 5.7. The injection volume must be the same volume used for the calibration standards.

7.5.4 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 (40 ng/ μL , unless 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 and responses (area counts) 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.

7.5.4.1 When ions from a compound in the sample saturate the detector, this analysis must 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.

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

7.5.5 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 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.

7.6.1.1 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.

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

7.6.1.3 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%.)

7.6.1.4 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 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

7.6.1.5 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.

7.6.1.6 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.

7.6.2 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 $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- (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.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

7.7.2 If the RSD of a compound's response factor is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data (Sec. 7.3.5). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae 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.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

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

7.7.6 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 25% of the sum of the two peak heights. Otherwise, structural isomers are quantitated as isomeric pairs. Diastereomeric pairs (e.g., Aramite and Isosafrol) that may be separable by the GC should be summed and reported as the sum of both compounds.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Sec. 7.0 of Method 8000 and include calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8270:

8.2.1 The GC/MS system must be tuned to meet the DFTPP criteria discussed in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 7.4, each 12 hours.

8.2.4 The RRT of the sample component must fall within the RRT window of the standard component provided in Sec. 7.6.1.

8.3 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 are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 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, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, 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.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 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, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 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 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.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 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. If any changes are made to the system (e.g., the column changed, a septum is changed), see the guidance in Sec 8.2 of Method 8000 regarding whether recalibration of the system must take place.

8.7 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.

9.0 METHOD PERFORMANCE

9.1 Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial

wastewaters spiked at six concentrations ranging from 5 to 1,300 µg/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7. These values are presented as guidance only and are not intended as absolute acceptance criteria. Laboratories should generate their own acceptance criteria for capillary column method performance. (See Method 8000.)

9.2 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 8.

9.3 Method performance data using Method 3541 (automated Soxhlet extraction) are presented in Table 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 are listed in Table 10 and were taken from Reference 7.

9.4 Surrogate precision and accuracy data are presented in Table 11 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 Method 8270.

9.5 Single laboratory precision and bias data using Method 3545 (pressurized fluid extraction) for semivolatile organic compounds are presented in Table 12. 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 12 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%.

9.6 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 13 and 14 and were taken from Reference 10.

9.7 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. Following are the instrument conditions that were utilized to extract a 3.4 g sample: Pressure - 300 atm; Time - 60 min.; Extraction fluid - CO₂; Modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80°C; Restrictor temperature - 120°C; and, Trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 15 and were taken from Reference 11.

9.8 Tables 16 and 17 contain single-laboratory precision and accuracy data for solid-phase extraction of TCLP buffer solutions spiked at two levels and extracted using Method 3535.

9.9 Table 18 contains multiple-laboratory data for solid-phase extraction of spiked TCLP soil leachates extracted using Method 3535.

10.0 REFERENCES

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TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|---|----------------------|-------------|---------------------|
| 2-Picoline | 3.75 ^a | 93 | 66,92 |
| Aniline | 5.68 | 93 | 66,65 |
| Phenol | 5.77 | 94 | 65,66 |
| Bis(2-chloroethyl) ether | 5.82 | 93 | 63,95 |
| 2-Chlorophenol | 5.97 | 128 | 64,130 |
| 1,3-Dichlorobenzene | 6.27 | 146 | 148,111 |
| 1,4-Dichlorobenzene-d ₄ (IS) | 6.35 | 152 | 150,115 |
| 1,4-Dichlorobenzene | 6.40 | 146 | 148,111 |
| Benzyl alcohol | 6.78 | 108 | 79,77 |
| 1,2-Dichlorobenzene | 6.85 | 146 | 148,111 |
| N-Nitrosomethylethylamine | 6.97 | 88 | 42,43,56 |
| Bis(2-chloroisopropyl) ether | 7.22 | 45 | 77,121 |
| Ethyl carbamate | 7.27 | 62 | 44,45,74 |
| Thiophenol (Benzenethiol) | 7.42 | 110 | 66,109,84 |
| Methyl methanesulfonate | 7.48 | 80 | 79,65,95 |
| N-Nitrosodi-n-propylamine | 7.55 | 70 | 42,101,130 |
| Hexachloroethane | 7.65 | 117 | 201,199 |
| Maleic anhydride | 7.65 | 54 | 98,53,44 |
| Nitrobenzene | 7.87 | 77 | 123,65 |
| Isophorone | 8.53 | 82 | 95,138 |
| N-Nitrosodiethylamine | 8.70 | 102 | 42,57,44,56 |
| 2-Nitrophenol | 8.75 | 139 | 109,65 |
| 2,4-Dimethylphenol | 9.03 | 122 | 107,121 |
| p-Benzoquinone | 9.13 | 108 | 54,82,80 |
| Bis(2-chloroethoxy)methane | 9.23 | 93 | 95,123 |
| Benzoic acid | 9.38 | 122 | 105,77 |
| 2,4-Dichlorophenol | 9.48 | 162 | 164,98 |
| Trimethyl phosphate | 9.53 | 110 | 79,95,109,140 |
| Ethyl methanesulfonate | 9.62 | 79 | 109,97,45,65 |
| 1,2,4-Trichlorobenzene | 9.67 | 180 | 182,145 |
| Naphthalene-d ₈ (IS) | 9.75 | 136 | 68 |
| Naphthalene | 9.82 | 128 | 129,127 |
| Hexachlorobutadiene | 10.43 | 225 | 223,227 |
| Tetraethyl pyrophosphate | 11.07 | 99 | 155,127,81,109 |
| Diethyl sulfate | 11.37 | 139 | 45,59,99,111,125 |
| 4-Chloro-3-methylphenol | 11.68 | 107 | 144,142 |
| 2-Methylnaphthalene | 11.87 | 142 | 141 |
| 2-Methylphenol | 12.40 | 107 | 108,77,79,90 |
| Hexachloropropene | 12.45 | 213 | 211,215,117,106,141 |
| Hexachlorocyclopentadiene | 12.60 | 237 | 235,272 |
| N-Nitrosopyrrolidine | 12.65 | 100 | 41,42,68,69 |

TABLE 1
(continued)

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|-----------------------------------|----------------------|-------------|-------------------|
| Acetophenone | 12.67 | 105 | 71,51,120 |
| 4-Methylphenol | 12.82 | 107 | 108,77,79,90 |
| 2,4,6-Trichlorophenol | 12.85 | 196 | 198,200 |
| o-Toluidine | 12.87 | 106 | 107,77,51,79 |
| 3-Methylphenol | 12.93 | 107 | 108,77,79,90 |
| 2-Chloronaphthalene | 13.30 | 162 | 127,164 |
| N-Nitrosopiperidine | 13.55 | 114 | 42,55,56,41 |
| 1,4-Phenylenediamine | 13.62 | 108 | 80,53,54,52 |
| 1-Chloronaphthalene | 13.65 ^a | 162 | 127,164 |
| 2-Nitroaniline | 13.75 | 65 | 92,138 |
| 5-Chloro-2-methylaniline | 14.28 | 106 | 141,140,77,89 |
| Dimethyl phthalate | 14.48 | 163 | 194,164 |
| Acenaphthylene | 14.57 | 152 | 151,153 |
| 2,6-Dinitrotoluene | 14.62 | 165 | 63,89 |
| Phthalic anhydride | 14.62 | 104 | 76,50,148 |
| o-Anisidine | 15.00 | 108 | 80,123,52 |
| 3-Nitroaniline | 15.02 | 138 | 108,92 |
| Acenaphthene-d ₁₀ (IS) | 15.05 | 164 | 162,160 |
| Acenaphthene | 15.13 | 154 | 153,152 |
| 2,4-Dinitrophenol | 15.35 | 184 | 63,154 |
| 2,6-Dinitrophenol | 15.47 | 162 | 164,126,98,63 |
| 4-Chloroaniline | 15.50 | 127 | 129,65,92 |
| Isosafrole | 15.60 | 162 | 131,104,77,51 |
| Dibenzofuran | 15.63 | 168 | 139 |
| 2,4-Diaminotoluene | 15.78 | 121 | 122,94,77,104 |
| 2,4-Dinitrotoluene | 15.80 | 165 | 63,89 |
| 4-Nitrophenol | 15.80 | 139 | 109,65 |
| 2-Naphthylamine | 16.00 ^a | 143 | 115,116 |
| 1,4-Naphthoquinone | 16.23 | 158 | 104,102,76,50,130 |
| p-Cresidine | 16.45 | 122 | 94,137,77,93 |
| Dichlorovos | 16.48 | 109 | 185,79,145 |
| Diethyl phthalate | 16.70 | 149 | 177,150 |
| Fluorene | 16.70 | 166 | 165,167 |
| 2,4,5-Trimethylaniline | 16.70 | 120 | 135,134,91,77 |
| N-Nitrosodi-n-butylamine | 16.73 | 84 | 57,41,116,158 |
| 4-Chlorophenyl phenyl ether | 16.78 | 204 | 206,141 |
| Hydroquinone | 16.93 | 110 | 81,53,55 |
| 4,6-Dinitro-2-methylphenol | 17.05 | 198 | 51,105 |
| Resorcinol | 17.13 | 110 | 81,82,53,69 |
| N-Nitrosodiphenylamine | 17.17 | 169 | 168,167 |
| Safrole | 17.23 | 162 | 104,77,103,135 |
| Hexamethyl phosphoramidate | 17.33 | 135 | 44,179,92,42 |

TABLE 1
(continued)

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|--|----------------------|-------------|---------------------|
| 3-(Chloromethyl)pyridine hydrochloride | 17.50 | 92 | 127,129,65,39 |
| Diphenylamine | 17.54 ^a | 169 | 168,167 |
| 1,2,4,5-Tetrachlorobenzene | 17.97 | 216 | 214,179,108,143,218 |
| 1-Naphthylamine | 18.20 | 143 | 115,89,63 |
| 1-Acetyl-2-thiourea | 18.22 | 118 | 43,42,76 |
| 4-Bromophenyl phenyl ether | 18.27 | 248 | 250,141 |
| Toluene diisocyanate | 18.42 | 174 | 145,173,146,132,91 |
| 2,4,5-Trichlorophenol | 18.47 | 196 | 198,97,132,99 |
| Hexachlorobenzene | 18.65 | 284 | 142,249 |
| Nicotine | 18.70 | 84 | 133,161,162 |
| Pentachlorophenol | 19.25 | 266 | 264,268 |
| 5-Nitro-o-toluidine | 19.27 | 152 | 77,79,106,94 |
| Thionazine | 19.35 | 107 | 96,97,143,79,68 |
| 4-Nitroaniline | 19.37 | 138 | 65,108,92,80,39 |
| Phenanthrene-d ₁₀ (IS) | 19.55 | 188 | 94,80 |
| Phenanthrene | 19.62 | 178 | 179,176 |
| Anthracene | 19.77 | 178 | 176,179 |
| 1,4-Dinitrobenzene | 19.83 | 168 | 75,50,76,92,122 |
| Mevinphos | 19.90 | 127 | 192,109,67,164 |
| Naled | 20.03 | 109 | 145,147,301,79,189 |
| 1,3-Dinitrobenzene | 20.18 | 168 | 76,50,75,92,122 |
| Diallate (cis or trans) | 20.57 | 86 | 234,43,70 |
| 1,2-Dinitrobenzene | 20.58 | 168 | 50,63,74 |
| Diallate (trans or cis) | 20.78 | 86 | 234,43,70 |
| Pentachlorobenzene | 21.35 | 250 | 252,108,248,215,254 |
| 5-Nitro-o-anisidine | 21.50 | 168 | 79,52,138,153,77 |
| Pentachloronitrobenzene | 21.72 | 237 | 142,214,249,295,265 |
| 4-Nitroquinoline-1-oxide | 21.73 | 174 | 101,128,75,116 |
| Di-n-butyl phthalate | 21.78 | 149 | 150,104 |
| 2,3,4,6-Tetrachlorophenol | 21.88 | 232 | 131,230,166,234,168 |
| Dihydrosaffrole | 22.42 | 135 | 64,77 |
| Demeton-O | 22.72 | 88 | 89,60,61,115,171 |
| Fluoranthene | 23.33 | 202 | 101,203 |
| 1,3,5-Trinitrobenzene | 23.68 | 75 | 74,213,120,91,63 |
| Dicrotophos | 23.82 | 127 | 67,72,109,193,237 |
| Benzidine | 23.87 | 184 | 92,185 |
| Trifluralin | 23.88 | 306 | 43,264,41,290 |
| Bromoxynil | 23.90 | 277 | 279,88,275,168 |
| Pyrene | 24.02 | 202 | 200,203 |
| Monocrotophos | 24.08 | 127 | 192,67,97,109 |
| Phorate | 24.10 | 75 | 121,97,93,260 |
| Sulfallate | 24.23 | 188 | 88,72,60,44 |

TABLE 1
(continued)

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|--------------------------------------|----------------------|-------------|---------------------|
| Demeton-S | 24.30 | 88 | 60,81,89,114,115 |
| Phenacetin | 24.33 | 108 | 180,179,109,137,80 |
| Dimethoate | 24.70 | 87 | 93,125,143,229 |
| Phenobarbital | 24.70 | 204 | 117,232,146,161 |
| Carbofuran | 24.90 | 164 | 149,131,122 |
| Octamethyl pyrophosphoramidate | 24.95 | 135 | 44,199,286,153,243 |
| 4-Aminobiphenyl | 25.08 | 169 | 168,170,115 |
| Dioxathion | 25.25 | 97 | 125,270,153 |
| Terbufos | 25.35 | 231 | 57,97,153,103 |
| α,α -Dimethylphenylamine | 25.43 | 58 | 91,65,134,42 |
| Pronamide | 25.48 | 173 | 175,145,109,147 |
| Aminoazobenzene | 25.72 | 197 | 92,120,65,77 |
| Dichlone | 25.77 | 191 | 163,226,228,135,193 |
| Dinoseb | 25.83 | 211 | 163,147,117,240 |
| Disulfoton | 25.83 | 88 | 97,89,142,186 |
| Fluchloralin | 25.88 | 306 | 63,326,328,264,65 |
| Mexacarbate | 26.02 | 165 | 150,134,164,222 |
| 4,4'-Oxydianiline | 26.08 | 200 | 108,171,80,65 |
| Butyl benzyl phthalate | 26.43 | 149 | 91,206 |
| 4-Nitrobiphenyl | 26.55 | 199 | 152,141,169,151 |
| Phosphamidon | 26.85 | 127 | 264,72,109,138 |
| 2-Cyclohexyl-4,6-Dinitrophenol | 26.87 | 231 | 185,41,193,266 |
| Methyl parathion | 27.03 | 109 | 125,263,79,93 |
| Carbaryl | 27.17 | 144 | 115,116,201 |
| Dimethylaminoazobenzene | 27.50 | 225 | 120,77,105,148,42 |
| Propylthiouracil | 27.68 | 170 | 142,114,83 |
| Benz(a)anthracene | 27.83 | 228 | 229,226 |
| Chrysene-d ₁₂ (IS) | 27.88 | 240 | 120,236 |
| 3,3'-Dichlorobenzidine | 27.88 | 252 | 254,126 |
| Chrysene | 27.97 | 228 | 226,229 |
| Malathion | 28.08 | 173 | 125,127,93,158 |
| Kepone | 28.18 | 272 | 274,237,178,143,270 |
| Fenthion | 28.37 | 278 | 125,109,169,153 |
| Parathion | 28.40 | 109 | 97,291,139,155 |
| Anilazine | 28.47 | 239 | 241,143,178,89 |
| Bis(2-ethylhexyl) phthalate | 28.47 | 149 | 167,279 |
| 3,3'-Dimethylbenzidine | 28.55 | 212 | 106,196,180 |
| Carbophenothion | 28.58 | 157 | 97,121,342,159,199 |
| 5-Nitroacenaphthene | 28.73 | 199 | 152,169,141,115 |
| Methapyrilene | 28.77 | 97 | 50,191,71 |
| Isodrin | 28.95 | 193 | 66,195,263,265,147 |
| Captan | 29.47 | 79 | 149,77,119,117 |

TABLE 1
(continued)

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|------------------------------------|----------------------|-------------|---------------------|
| Chlorfenvinphos | 29.53 | 267 | 269,323,325,295 |
| Crotoxyphos | 29.73 | 127 | 105,193,166 |
| Phosmet | 30.03 | 160 | 77,93,317,76 |
| EPN | 30.11 | 157 | 169,185,141,323 |
| Tetrachlorvinphos | 30.27 | 329 | 109,331,79,333 |
| Di-n-octyl phthalate | 30.48 | 149 | 167,43 |
| 2-Aminoanthraquinone | 30.63 | 223 | 167,195 |
| Barban | 30.83 | 222 | 51,87,224,257,153 |
| Aramite | 30.92 | 185 | 191,319,334,197,321 |
| Benzo(b)fluoranthene | 31.45 | 252 | 253,125 |
| Nitrofen | 31.48 | 283 | 285,202,139,253 |
| Benzo(k)fluoranthene | 31.55 | 252 | 253,125 |
| Chlorobenzilate | 31.77 | 251 | 139,253,111,141 |
| Fensulfothion | 31.87 | 293 | 97,308,125,292 |
| Ethion | 32.08 | 231 | 97,153,125,121 |
| Diethylstilbestrol | 32.15 | 268 | 145,107,239,121,159 |
| Famphur | 32.67 | 218 | 125,93,109,217 |
| Tri-p-tolyl phosphate ^b | 32.75 | 368 | 367,107,165,198 |
| Benzo(a)pyrene | 32.80 | 252 | 253,125 |
| Perylene-d ₁₂ (IS) | 33.05 | 264 | 260,265 |
| 7,12-Dimethylbenz(a)anthracene | 33.25 | 256 | 241,239,120 |
| 5,5-Diphenylhydantoin | 33.40 | 180 | 104,252,223,209 |
| Captafol | 33.47 | 79 | 77,80,107 |
| Dinocap | 33.47 | 69 | 41,39 |
| Methoxychlor | 33.55 | 227 | 228,152,114,274,212 |
| 2-Acetylaminofluorene | 33.58 | 181 | 180,223,152 |
| 4,4'-Methylenebis(2-chloroaniline) | 34.38 | 231 | 266,268,140,195 |
| 3,3'-Dimethoxybenzidine | 34.47 | 244 | 201,229 |
| 3-Methylcholanthrene | 35.07 | 268 | 252,253,126,134,113 |
| Phosalone | 35.23 | 182 | 184,367,121,379 |
| Azinphos-methyl | 35.25 | 160 | 132,93,104,105 |
| Leptophos | 35.28 | 171 | 377,375,77,155,379 |
| Mirex | 35.43 | 272 | 237,274,270,239,235 |
| Tris(2,3-dibromopropyl) phosphate | 35.68 | 201 | 137,119,217,219,199 |
| Dibenz(a,j)acridine | 36.40 | 279 | 280,277,250 |
| Mestranol | 36.48 | 277 | 310,174,147,242 |
| Coumaphos | 37.08 | 362 | 226,210,364,97,109 |
| Indeno(1,2,3-cd)pyrene | 39.52 | 276 | 138,227 |
| Dibenz(a,h)anthracene | 39.82 | 278 | 139,279 |
| Benzo(g,h,i)perylene | 41.43 | 276 | 138,277 |
| 1,2:4,5-Dibenzopyrene | 41.60 | 302 | 151,150,300 |
| Strychnine | 45.15 | 334 | 334,335,333 |

TABLE 1
(continued)

| Compound | Retention Time (min) | Primary Ion | Secondary Ion(s) |
|------------------------------------|----------------------|-------------|---------------------|
| Piperonyl sulfoxide | 46.43 | 162 | 135,105,77 |
| Hexachlorophene | 47.98 | 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 |
| 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

^aEstimated retention times

^bSubstitute for the non-specific mixture, tricresyl phosphate

TABLE 2

ESTIMATED QUANTITATION LIMITS (EQLs) FOR SEMIVOLATILE ORGANICS

| Compound | Estimated Quantitation Limits ^a | |
|------------------------------|--|---|
| | 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 |

TABLE 2
(Continued)

| Compound | Estimated Quantitation Limits ^a | |
|--|--|---|
| | Ground water (µg/L) | Low Soil/Sediment ^b (µg/kg) |
| 5-Chloro-2-methylaniline | 10 | ND |
| 4-Chloro-3-methylphenol | 20 | 1300 |
| 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 |

TABLE 2
(Continued)

| Compound | Estimated Quantitation Limits ^a | |
|-----------------------------|--|---|
| | Ground water (µg/L) | Low Soil/Sediment ^b (µg/kg) |
| 3,3'-Dimethylbenzidine | 10 | ND |
| 2,4-Dimethylphenol | 10 | 660 |
| Dimethyl phthalate | 10 | 660 |
| 1,2-Dinitrobenzene | 40 | ND |
| 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 |

TABLE 2
(Continued)

| Compound | Estimated Quantitation Limits ^a | |
|--------------------------|--|---|
| | Ground water (µg/L) | Low Soil/Sediment ^b (µg/kg) |
| Kepone | 20 | ND |
| Leptophos | 10 | ND |
| Malathion | 50 | ND |
| Mestranol | 20 | ND |
| Methapyrilene | 100 | ND |
| Methoxychlor | 10 | ND |
| 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 |

TABLE 2
(Continued)

| Compound | Estimated Quantitation Limits ^a | |
|--------------------------------|--|---|
| | Ground water (µg/L) | Low Soil/Sediment ^b (µg/kg) |
| N-Nitrosodiphenylamine | 10 | 660 |
| N-Nitroso-di-n-propylamine | 10 | 660 |
| N-Nitrosopiperidine | 20 | ND |
| N-Nitrosopyrrolidine | 40 | ND |
| Octamethyl pyrophosphoramidate | 200 | ND |
| 4,4'-Oxydianiline | 20 | ND |
| Parathion | 10 | ND |
| Pentachlorobenzene | 10 | ND |
| 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 |

TABLE 2
(Continued)

| Compound | Estimated Quantitation Limits ^a | |
|-----------------------------------|--|---|
| | Ground water (µg/L) | Low Soil/Sediment ^b (µg/kg) |
| 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 |

^a Sample EQLs are highly matrix-dependent. The EQLs listed here are provided for guidance and may not always be achievable.

^b EQLs listed for soil/sediment are based on wet weight. Normally, data are reported on a dry weight basis, therefore, EQLs will be higher based on the % dry weight of each sample. These EQLs 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
Non-water miscible waste

7.5
75

^cEQL = (EQL 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 | 30-60% of mass 198 |
| 68 | < 2% of mass 69 |
| 70 | < 2% of mass 69 |
| 127 | 40-60% of mass 198 |
| 197 | < 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5-9% of mass 198 |
| 275 | 10-30% of mass 198 |
| 365 | > 1% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | > 40% of mass 198 |
| 443 | 17-23% of mass 442 |

^a Data taken from Reference 3.

^b Alternate tuning criteria may be employed, (e.g., CLP, Method 525, or manufacturers' instructions), provided that method performance is not adversely affected.

TABLE 4

CALIBRATION CHECK COMPOUNDS (CCC)

| Base/Neutral Fraction | Acid Fraction |
|-----------------------|-------------------------|
| Acenaphthene | 4-Chloro-3-methylphenol |
| 1,4-Dichlorobenzene | 2,4-Dichlorophenol |
| Hexachlorobutadiene | 2-Nitrophenol |
| Diphenylamine | Phenol |
| Di-n-octyl phthalate | Pentachlorophenol |
| Fluoranthene | 2,4,6-Trichlorophenol |
| Benzo(a)pyrene | |

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- | Dimethyl phthalate |
| Ethyl methanesulfonate | phenethylamine | 2,4-Dinitrophenol |
| 2-Fluorophenol (surr) | 2,4-Dimethylphenol | 2,4-Dinitrotoluene |
| Hexachloroethane | Hexachlorobutadiene | 2,6-Dinitrotoluene |
| Methyl methanesulfonate | Isophorone | Fluorene |
| 2-Methylphenol | 2-Methylnaphthalene | 2-Fluorobiphenyl (surr) |
| 4-Methylphenol | Naphthalene | Hexachlorocyclopentadiene |
| N-Nitrosodimethylamine | Nitrobenzene | 1-Naphthylamine |
| N-Nitroso-di-n-propylamine | Nitrobenzene-d ₈ (surr) | 2-Naphthylamine |
| Phenol | 2-Nitrophenol | 2-Nitroaniline |
| Phenol-d ₆ (surr) | N-Nitrosodi-n-butylamine | 3-Nitroaniline |
| 2-Picoline | N-Nitrosopiperidine | 4-Nitroaniline |
| | 1,2,4-Trichlorobenzene | 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 | |
| Hexachlorobenzene | Pyrene | |
| N-Nitrosodiphenylamine | Terphenyl-d ₁₄ (surr) | |
| Pentachlorophenol | 7,12-Dimethylbenz(a)anthracene | |
| Pentachloronitrobenzene | Di-n-octyl phthalate | |
| Phenacetin | Indeno(1,2,3-cd) pyrene | |
| Phenanthrene | 3-Methylcholanthrene | |
| Pronamide | | |

(surr) = surrogate

TABLE 6
MULTILABORATORY PERFORMANCE DATA^a

| Compound | Test conc. (µg/L) | Limit for s (µg/L) | Range for \bar{x} (µg/L) | Range p, p _s (%) |
|------------------------------|----------------------|-----------------------|-------------------------------|--------------------------------|
| Acenaphthene | 100 | 27.6 | 60.1-132.3 | 47-145 |
| Acenaphthylene | 100 | 40.2 | 53.5-126.0 | 33-145 |
| Aldrin | 100 | 39.0 | 7.2-152.2 | D-166 |
| Anthracene | 100 | 32.0 | 43.4-118.0 | 27-133 |
| Benz(a)anthracene | 100 | 27.6 | 41.8-133.0 | 33-143 |
| Benzo(b)fluoranthene | 100 | 38.8 | 42.0-140.4 | 24-159 |
| Benzo(k)fluoranthene | 100 | 32.3 | 25.2-145.7 | 11-162 |
| Benzo(a)pyrene | 100 | 39.0 | 31.7-148.0 | 17-163 |
| Benzo(g,h,i)perylene | 100 | 58.9 | D-195.0 | D-219 |
| Benzyl butyl phthalate | 100 | 23.4 | D-139.9 | D-152 |
| β-BHC | 100 | 31.5 | 41.5-130.6 | 24-149 |
| δ-BHC | 100 | 21.6 | D-100.0 | D-110 |
| Bis(2-chloroethyl) ether | 100 | 55.0 | 42.9-126.0 | 12-158 |
| Bis(2-chloroethoxy)methane | 100 | 34.5 | 49.2-164.7 | 33-184 |
| Bis(2-chloroisopropyl) ether | 100 | 46.3 | 62.8-138.6 | 36-166 |
| Bis(2-ethylhexyl) phthalate | 100 | 41.1 | 28.9-136.8 | 8-158 |
| 4-Bromophenyl phenyl ether | 100 | 23.0 | 64.9-114.4 | 53-127 |
| 2-Chloronaphthalene | 100 | 13.0 | 64.5-113.5 | 60-118 |
| 4-Chlorophenyl phenyl ether | 100 | 33.4 | 38.4-144.7 | 25-158 |
| Chrysene | 100 | 48.3 | 44.1-139.9 | 17-168 |
| 4,4'-DDD | 100 | 31.0 | D-134.5 | D-145 |
| 4,4'-DDE | 100 | 32.0 | 19.2-119.7 | 4-136 |
| 4,4'-DDT | 100 | 61.6 | D-170.6 | D-203 |
| Dibenzo(a,h)anthracene | 100 | 70.0 | D-199.7 | D-227 |
| Di-n-butyl phthalate | 100 | 16.7 | 8.4-111.0 | 1-118 |
| 1,2-Dichlorobenzene | 100 | 30.9 | 48.6-112.0 | 32-129 |
| 1,3-Dichlorobenzene | 100 | 41.7 | 16.7-153.9 | D-172 |
| 1,4-Dichlorobenzene | 100 | 32.1 | 37.3-105.7 | 20-124 |
| 3,3'-Dichlorobenzidine | 100 | 71.4 | 8.2-212.5 | D-262 |
| Dieldrin | 100 | 30.7 | 44.3-119.3 | 29-136 |
| Diethyl phthalate | 100 | 26.5 | D-100.0 | D-114 |
| Dimethyl phthalate | 100 | 23.2 | D-100.0 | D-112 |
| 2,4-Dinitrotoluene | 100 | 21.8 | 47.5-126.9 | 39-139 |
| 2,6-Dinitrotoluene | 100 | 29.6 | 68.1-136.7 | 50-158 |
| Di-n-octyl phthalate | 100 | 31.4 | 18.6-131.8 | 4-146 |
| Endosulfan sulfate | 100 | 16.7 | D-103.5 | D-107 |
| Endrin aldehyde | 100 | 32.5 | D-188.8 | D-209 |
| Fluoranthene | 100 | 32.8 | 42.9-121.3 | 26-137 |

TABLE 6
(continued)

| Compound | Test conc. ($\mu\text{g/L}$) | Limit for s ($\mu\text{g/L}$) | Range for \bar{x} ($\mu\text{g/L}$) | Range p, p_s (%) |
|----------------------------|-----------------------------------|------------------------------------|--|-----------------------|
| Fluorene | 100 | 20.7 | 71.6-108.4 | 59-121 |
| Heptachlor | 100 | 37.2 | D-172.2 | D-192 |
| Heptachlor epoxide | 100 | 54.7 | 70.9-109.4 | 26-155 |
| Hexachlorobenzene | 100 | 24.9 | 7.8-141.5 | D-152 |
| Hexachlorobutadiene | 100 | 26.3 | 37.8-102.2 | 24-116 |
| Hexachloroethane | 100 | 24.5 | 55.2-100.0 | 40-113 |
| Indeno(1,2,3-cd)pyrene | 100 | 44.6 | D-150.9 | D-171 |
| Isophorone | 100 | 63.3 | 46.6-180.2 | 21-196 |
| Naphthalene | 100 | 30.1 | 35.6-119.6 | 21-133 |
| Nitrobenzene | 100 | 39.3 | 54.3-157.6 | 35-180 |
| N-Nitrosodi-n-propylamine | 100 | 55.4 | 13.6-197.9 | D-230 |
| Aroclor 1260 | 100 | 54.2 | 19.3-121.0 | D-164 |
| Phenanthrene | 100 | 20.6 | 65.2-108.7 | 54-120 |
| Pyrene | 100 | 25.2 | 69.6-100.0 | 52-115 |
| 1,2,4-Trichlorobenzene | 100 | 28.1 | 57.3-129.2 | 44-142 |
| 4-Chloro-3-methylphenol | 100 | 37.2 | 40.8-127.9 | 22-147 |
| 2-Chlorophenol | 100 | 28.7 | 36.2-120.4 | 23-134 |
| 2,4-Chlorophenol | 100 | 26.4 | 52.5-121.7 | 39-135 |
| 2,4-Dimethylphenol | 100 | 26.1 | 41.8-109.0 | 32-119 |
| 2,4-Dinitrophenol | 100 | 49.8 | D-172.9 | D-191 |
| 2-Methyl-4,6-dinitrophenol | 100 | 93.2 | 53.0-100.0 | D-181 |
| 2-Nitrophenol | 100 | 35.2 | 45.0-166.7 | 29-182 |
| 4-Nitrophenol | 100 | 47.2 | 13.0-106.5 | D-132 |
| Pentachlorophenol | 100 | 48.9 | 38.1-151.8 | 14-176 |
| Phenol | 100 | 22.6 | 16.6-100.0 | 5-112 |
| 2,4,6-Trichlorophenol | 100 | 31.7 | 52.4-129.2 | 37-144 |

s = Standard deviation of four recovery measurements, in $\mu\text{g/L}$

\bar{x} = Average recovery for four recovery measurements, in $\mu\text{g/L}$

p, p_s = Measured percent recovery

D = Detected; result must be greater than zero

^a Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION^a

| Compound | Accuracy, as recovery, x' ($\mu\text{g/L}$) | Single analyst precision, s_r' ($\mu\text{g/L}$) | Overall precision, S' ($\mu\text{g/L}$) |
|------------------------------|---|--|---|
| Acenaphthene | 0.96C+0.19 | 0.15 \bar{x} -0.12 | 0.21 \bar{x} -0.67 |
| Acenaphthylene | 0.89C+0.74 | 0.24 \bar{x} -1.06 | 0.26 \bar{x} -0.54 |
| Aldrin | 0.78C+1.66 | 0.27 \bar{x} -1.28 | 0.43 \bar{x} +1.13 |
| Anthracene | 0.80C+0.68 | 0.21 \bar{x} -0.32 | 0.27 \bar{x} -0.64 |
| Benz(a)anthracene | 0.88C-0.60 | 0.15 \bar{x} +0.93 | 0.26 \bar{x} -0.21 |
| Benzo(b)fluoranthene | 0.93C-1.80 | 0.22 \bar{x} +0.43 | 0.29 \bar{x} +0.96 |
| Benzo(k)fluoranthene | 0.87C-1.56 | 0.19 \bar{x} +1.03 | 0.35 \bar{x} +0.40 |
| Benzo(a)pyrene | 0.90C-0.13 | 0.22 \bar{x} +0.48 | 0.32 \bar{x} +1.35 |
| Benzo(g,h,i)perylene | 0.98C-0.86 | 0.29 \bar{x} +2.40 | 0.51 \bar{x} -0.44 |
| Benzyl butyl phthalate | 0.66C-1.68 | 0.18 \bar{x} +0.94 | 0.53 \bar{x} +0.92 |
| β -BHC | 0.87C-0.94 | 0.20 \bar{x} -0.58 | 0.30 \bar{x} +1.94 |
| δ -BHC | 0.29C-1.09 | 0.34 \bar{x} +0.86 | 0.93 \bar{x} -0.17 |
| Bis(2-chloroethyl) ether | 0.86C-1.54 | 0.35 \bar{x} -0.99 | 0.35 \bar{x} +0.10 |
| Bis(2-chloroethoxy)methane | 1.12C-5.04 | 0.16 \bar{x} +1.34 | 0.26 \bar{x} +2.01 |
| Bis(2-chloroisopropyl) ether | 1.03C-2.31 | 0.24 \bar{x} +0.28 | 0.25 \bar{x} +1.04 |
| Bis(2-ethylhexyl) phthalate | 0.84C-1.18 | 0.26 \bar{x} +0.73 | 0.36 \bar{x} +0.67 |
| 4-Bromophenyl phenyl ether | 0.91C-1.34 | 0.13 \bar{x} +0.66 | 0.16 \bar{x} +0.66 |
| 2-Chloronaphthalene | 0.89C+0.01 | 0.07 \bar{x} +0.52 | 0.13 \bar{x} +0.34 |
| 4-Chlorophenyl phenyl ether | 0.91C+0.53 | 0.20 \bar{x} -0.94 | 0.30 \bar{x} -0.46 |
| Chrysene | 0.93C-1.00 | 0.28 \bar{x} +0.13 | 0.33 \bar{x} -0.09 |
| 4,4'-DDD | 0.56C-0.40 | 0.29 \bar{x} -0.32 | 0.66 \bar{x} -0.96 |
| 4,4'-DDE | 0.70C-0.54 | 0.26 \bar{x} -1.17 | 0.39 \bar{x} -1.04 |
| 4,4'-DDT | 0.79C-3.28 | 0.42 \bar{x} +0.19 | 0.65 \bar{x} -0.58 |
| Dibenzo(a,h)anthracene | 0.88C+4.72 | 0.30 \bar{x} +8.51 | 0.59 \bar{x} +0.25 |
| Di-n-butyl phthalate | 0.59C+0.71 | 0.13 \bar{x} +1.16 | 0.39 \bar{x} +0.60 |
| 1,2-Dichlorobenzene | 0.80C+0.28 | 0.20 \bar{x} +0.47 | 0.24 \bar{x} +0.39 |
| 1,3-Dichlorobenzene | 0.86C-0.70 | 0.25 \bar{x} +0.68 | 0.41 \bar{x} +0.11 |
| 1,4-Dichlorobenzene | 0.73C-1.47 | 0.24 \bar{x} +0.23 | 0.29 \bar{x} +0.36 |
| 3,3'-Dichlorobenzidine | 1.23C-12.65 | 0.28 \bar{x} +7.33 | 0.47 \bar{x} +3.45 |
| Dieldrin | 0.82C-0.16 | 0.20 \bar{x} -0.16 | 0.26 \bar{x} -0.07 |
| Diethyl phthalate | 0.43C+1.00 | 0.28 \bar{x} +1.44 | 0.52 \bar{x} +0.22 |
| Dimethyl phthalate | 0.20C+1.03 | 0.54 \bar{x} +0.19 | 1.05 \bar{x} -0.92 |
| 2,4-Dinitrotoluene | 0.92C-4.81 | 0.12 \bar{x} +1.06 | 0.21 \bar{x} +1.50 |
| 2,6-Dinitrotoluene | 1.06C-3.60 | 0.14 \bar{x} +1.26 | 0.19 \bar{x} +0.35 |
| Di-n-octyl phthalate | 0.76C-0.79 | 0.21 \bar{x} +1.19 | 0.37 \bar{x} +1.19 |
| Endosulfan sulfate | 0.39C+0.41 | 0.12 \bar{x} +2.47 | 0.63 \bar{x} -1.03 |
| Endrin aldehyde | 0.76C-3.86 | 0.18 \bar{x} +3.91 | 0.73 \bar{x} -0.62 |

TABLE 7
(Continued)

| Compound | Accuracy, as recovery, x' ($\mu\text{g/L}$) | Single analyst precision, s'_i ($\mu\text{g/L}$) | Overall precision, S' ($\mu\text{g/L}$) |
|----------------------------|---|--|---|
| Fluoranthene | 0.81C+1.10 | 0.22 \bar{x} -0.73 | 0.28 \bar{x} -0.60 |
| Fluorene | 0.90C-0.00 | 0.12 \bar{x} +0.26 | 0.13 \bar{x} +0.61 |
| Heptachlor | 0.87C-2.97 | 0.24 \bar{x} -0.56 | 0.50 \bar{x} -0.23 |
| Heptachlor epoxide | 0.92C-1.87 | 0.33 \bar{x} -0.46 | 0.28 \bar{x} +0.64 |
| Hexachlorobenzene | 0.74C+0.66 | 0.18 \bar{x} -0.10 | 0.43 \bar{x} -0.52 |
| Hexachlorobutadiene | 0.71C-1.01 | 0.19 \bar{x} +0.92 | 0.26 \bar{x} +0.49 |
| Hexachloroethane | 0.73C-0.83 | 0.17 \bar{x} +0.67 | 0.17 \bar{x} +0.80 |
| Indeno(1,2,3-cd)pyrene | 0.78C-3.10 | 0.29 \bar{x} +1.46 | 0.50 \bar{x} -0.44 |
| Isophorone | 1.12C+1.41 | 0.27 \bar{x} +0.77 | 0.33 \bar{x} +0.26 |
| Naphthalene | 0.76C+1.58 | 0.21 \bar{x} -0.41 | 0.30 \bar{x} -0.68 |
| Nitrobenzene | 1.09C-3.05 | 0.19 \bar{x} +0.92 | 0.27 \bar{x} +0.21 |
| N-Nitrosodi-n-propylamine | 1.12C-6.22 | 0.27 \bar{x} +0.68 | 0.44 \bar{x} +0.47 |
| Aroclor 1260 | 0.81C-10.86 | 0.35 \bar{x} +3.61 | 0.43 \bar{x} +1.82 |
| Phenanthrene | 0.87C+0.06 | 0.12 \bar{x} +0.57 | 0.15 \bar{x} +0.25 |
| Pyrene | 0.84C-0.16 | 0.16 \bar{x} +0.06 | 0.15 \bar{x} +0.31 |
| 1,2,4-Trichlorobenzene | 0.94C-0.79 | 0.15 \bar{x} +0.85 | 0.21 \bar{x} +0.39 |
| 4-Chloro-3-methylphenol | 0.84C+0.35 | 0.23 \bar{x} +0.75 | 0.29 \bar{x} +1.31 |
| 2-Chlorophenol | 0.78C+0.29 | 0.18 \bar{x} +1.46 | 0.28 \bar{x} +0.97 |
| 2,4-Dichlorophenol | 0.87C-0.13 | 0.15 \bar{x} +1.25 | 0.21 \bar{x} +1.28 |
| 2,4-Dimethylphenol | 0.71C+4.41 | 0.16 \bar{x} +1.21 | 0.22 \bar{x} +1.31 |
| 2,4-Dinitrophenol | 0.81C-18.04 | 0.38 \bar{x} +2.36 | 0.42 \bar{x} +26.29 |
| 2-Methyl-4,6-dinitrophenol | 1.04C-28.04 | 0.10 \bar{x} +42.29 | 0.26 \bar{x} +23.10 |
| 2-Nitrophenol | 0.07C-1.15 | 0.16 \bar{x} +1.94 | 0.27 \bar{x} +2.60 |
| 4-Nitrophenol | 0.61C-1.22 | 0.38 \bar{x} +2.57 | 0.44 \bar{x} +3.24 |
| Pentachlorophenol | 0.93C+1.99 | 0.24 \bar{x} +3.03 | 0.30 \bar{x} +4.33 |
| Phenol | 0.43C+1.26 | 0.26 \bar{x} +0.73 | 0.35 \bar{x} +0.58 |
| 2,4,6-Trichlorophenol | 0.91C-0.18 | 0.16 \bar{x} +2.22 | 0.22 \bar{x} +1.81 |

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in $\mu\text{g/L}$.

s'_i = Expected single analyst standard deviation of measurements at an average concentration of \bar{x} , in $\mu\text{g/L}$.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of \bar{x} , in $\mu\text{g/L}$.

C = True value for the concentration, in $\mu\text{g/L}$.

\bar{x} = Average recovery found for measurements of samples containing a concentration of C, in $\mu\text{g/L}$.

^a Criteria from 40 CFR Part 136 for Method 625, using a packed GC column. These criteria are based directly on the method performance data in Table 7. These values are for guidance only. Appropriate derivation of acceptance criteria for capillary columns should result in much narrower ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 8
EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

| Compound | Percent Recovery, Day 0 | | Percent Recovery, Day 7 | |
|--|-------------------------|-----|-------------------------|-----|
| | 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 9

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

| Compound | Clay Soil | | Topsoil | |
|--|---------------|-----|---------------|-----|
| | 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 10

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 |
| Diethyl phthalate | 74.9 | 5.4 |
| 4-Chlorophenyl-phenyl ether | 67.2 | 3.2 |

TABLE 10
(continued)

| Compound | Mean Recovery | RSD |
|-----------------------------|---------------|-----|
| 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 |
| Dibenzo(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 11
PRECISION AND BIAS VALUES FOR METHOD 3542¹

| 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 11 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 12

ACCELERATED SOLVENT EXTRACTION (METHOD 3545) RECOVERY VALUES
AS PERCENT OF SOXTEC™

| Compound | Clay | | | Loam | | | Sand | | | Mean Rec. |
|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-----------|
| | Low | Mid | High | Low | Mid | High | Low | Mid | High | |
| 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 12
(continued)

| Compound | Clay | | | Loam | | | Sand | | | Mean Rec. |
|-----------------------------|-------|--------|-------|-------|--------|--------|--------|--------|-------|-----------|
| | Low | Mid | High | Low | Mid | High | Low | Mid | High | |
| 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 13

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 14

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 15

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 16

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

| Analyte | Spike Level ($\mu\text{g/L}$) | 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 17

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

| Analyte | Spike Level ($\mu\text{g/L}$) | 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 18

RECOVERY DATA FROM THREE LABORATORIES FOR SOLID-PHASE EXTRACTION
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 18
(continued)

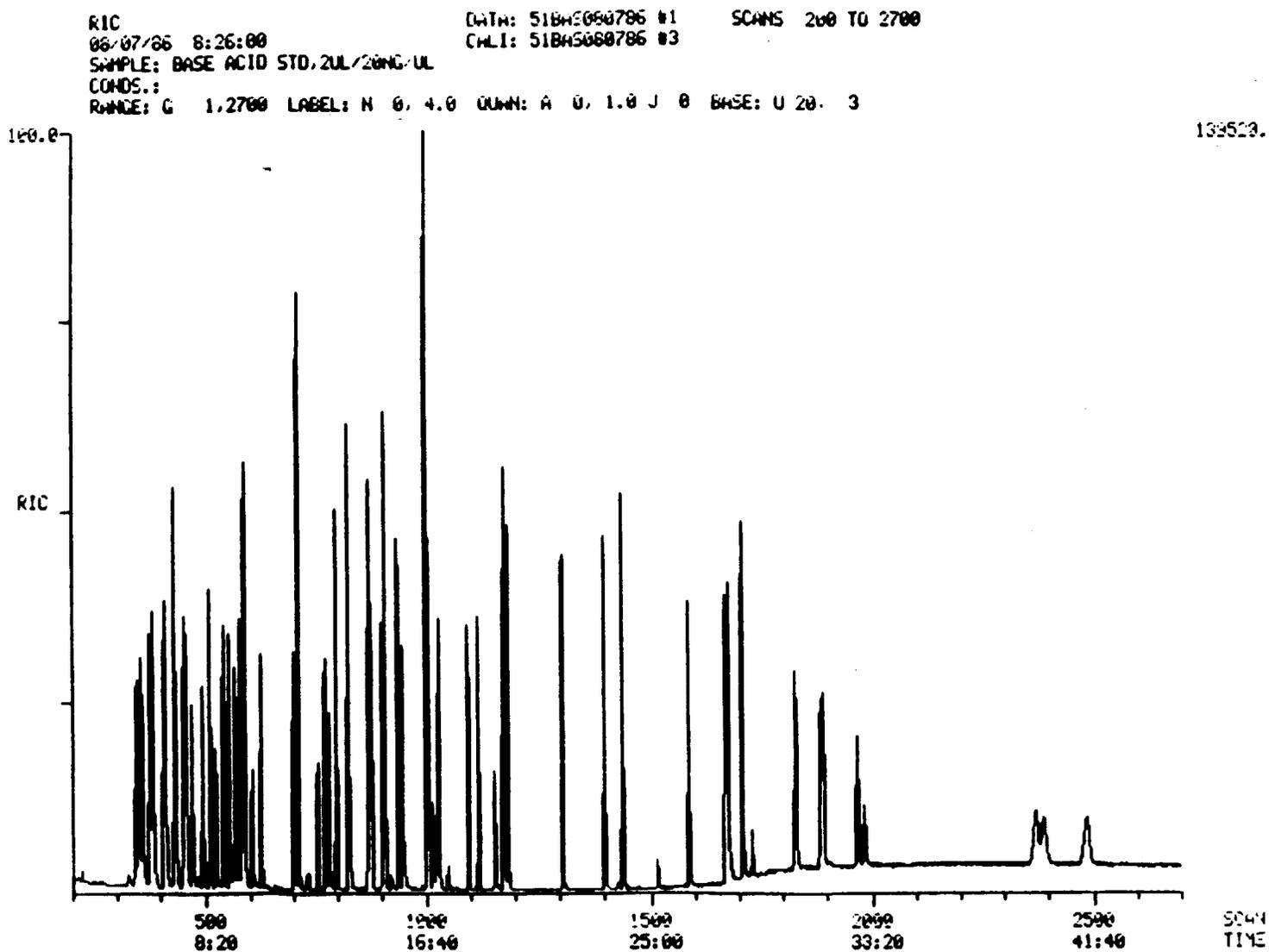
| Buffer 2 pH = 4.937 | | Lab 1 | | | Lab 2 | | | Lab 3 | | |
|-----------------------|---------------------|-------|-----|----|-------|------|----|-------|-----|----|
| Analyte | Spike Level (µg/L)* | %R | RSD | n | %R | RSD | n | %R | RSD | n |
| o-Cresol | 200,00 | 97 | 13 | 7 | 37.8 | 4.5 | 3 | 6.1 | 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 m- and p-Cresol. Lab 2 reported the sum of all three isomers of Cresol.

Data from Reference 12.

FIGURE 1
GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



METHOD 8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS
SPECTROMETRY (GC/MS)

