

GC/HPLC Analyses of Organic Compounds in Drinking Water: US EPA Procedures

This bulletin summarizes the analyses for organic compounds in drinking water according to US Environmental Protection Agency Series 500 methodology. Sample preparation, chromatography columns, and detection are described for each class of compounds. Most of the analyses are illustrated with chromatograms. Supelco products, most of which are tested for environmental applications, are listed for each method.

Key Words:

- drinking water
- US EPA Series 500 methods
- water quality

In the United States, drinking water quality is monitored according to the US Environmental Protection Agency's Series 500 methods (1).^{*} Most of these methods call for analysis by capillary gas chromatography. Some methods suggest using a confirmational column to verify identities of organic compounds found in a sample. Most methods call for detection by conventional means (electron capture, nitrogen-phosphorus, photoionization, etc.) but some call for mass spectrometry. Volatile organic compounds can be analyzed with photoionization and electrolytic/conductivity detection, in series (Method 502.2), or by GC/mass spectrometry (Method 524.2). Two methods (Method 531.1 for carbamate herbicides and Method 547 for glyphosphates) call for analysis by HPLC, with a postcolumn reaction system as part of the detection process. Methods 549.1 and 550 also specify HPLC.

Sample Preparation

Organic pollutants in drinking water samples normally are present in parts-per-billion or lower concentrations, and must be concentrated prior to chromatographic analysis. Volatile organics are purged from the sample and trapped on solid adsorbents (methods 502.2 and 524.2), then rapidly heat desorbed from the trap onto the chromatographic column. Recommended traps are described for each method. Nonvolatile organics are extracted in solvent or, in one method, by solid phase extraction (Method 525). Extraction techniques and cleanup procedures are described in detail in the EPA methodology.

Previous EPA restrictions limited the use of adsorbents for collecting volatile organic compounds (VOCs) to those specified in the method. Now the EPA officially allows the use of alternative adsorbents. This allowance is published in the Federal Register (59 FR 62456-71), which lists changes to several EPA methods contained in the document, *Technical Note for Drinking Water Meth-*

ods (Technical Notes) (2). This Technical Note amends two methods with the following sentence:

The use of alternative sorbents is acceptable provided the data acquired meets all quality control criteria described in section 10, and provided the purge and desorption procedures specified in section 11 of the method are not changed.

This sentence is added to Method 502.2, Rev. 2.0, at the end of Section 6.2.2, and replaces the last sentence in Method 524.2, Rev. 4.0, in Section 6.2.2.

Anticipating these allowances in future methods, scientists now can look at ways to greatly enhance the performance of their system by the selection of modern adsorbents. The VOCARB™ 4000 purge trap contains a combination of carbon adsorbents that efficiently trap and release the broad range of analytes in methods 502.2 and 524.2. Excellent thermal stability of the adsorbents in the trap allows higher desorption temperatures (250°C), with less background contamination, than traps specified in the methods, providing more rapid transfer of analytes and improved chromatography.

Chromatography

Supelco columns and packings were used to obtain most of the chromatograms in this bulletin. Supelco products are listed by name in many of these EPA methods. For most of the other methods, Supelco materials are generically equivalent alternatives to listed materials. Specific testing for environmental applications ensures consistent performance from these products.

VOCOL™ capillary columns are tested specifically for analyses of volatile priority pollutants in wastewater, according to EPA methods 601, 602, and 624. Thus, they are ideal for monitoring these same compounds in drinking water at all stages of treatment. A 0.75mm ID VOCOL column is listed in EPA Method 524.2. Because 0.75mm ID VOCOL columns have high optimum carrier gas flow rates, analytes can be desorbed directly from the adsorbent trap to the column. In contrast, sample cryofocusing is necessary with narrower bore columns. In addition, 0.75mm ID VOCOL columns can be used with either capillary or packed column injection systems and detectors.

PTE™-5 capillary columns are tested specifically for analyses of acidic and basic organic compounds in water. These inert, 0.25mm ID columns are excellent for analyses of nitrogen- and phosphorus-containing pesticides (Method 507) and chlorinated pesticides, herbicides, and PCBs (methods 508 and 515). Very low column bleed ensures minimal interference with analyte detection in GC/MS systems.

Table 1. Regulated Drinking Water Compounds

Contaminants	Methods
Benzene	502.2, 524.2
Carbon tetrachloride	502.2, 524.2, 551
Chlorobenzene	502.2, 524.2
1,2-Dichlorobenzene	502.2, 524.2
1,4-Dichlorobenzene	502.2, 524.2
1,2-Dichloroethane	502.2, 524.2
cis-Dichloroethylene	502.2, 524.2
trans-Dichloroethylene	502.2, 524.2
Dichloromethane	502.2, 524.2
1,2-Dichloropropane	502.2, 524.2
Ethylbenzene	502.2, 524.2
Styrene	502.2, 524.2
Tetrachloroethylene	502.2, 524.2, 551
1,1,1-Trichloroethane	502.2, 524.2, 551
Trichloroethylene	502.2, 524.2, 551
Toluene	502.2, 524.2
1,2,4-Trichlorobenzene	502.2, 524.2
1,1-Dichloroethylene	502.2, 524.2
1,1,2-Trichloroethane	502.2, 524.2
Vinyl chloride	502.2, 524.2
Xylenes (total)	502.2, 524.2
2,3,7,8-TCDD (Dioxin)	1613
2,4-D	515.2, 555, 515.1
2,4,5-TP (Silvex®)	515.2, 555, 515.1
Alachlor	505 [■] , 507, 525.2, 508.1
Atrazine	505 [■] , 507, 525.2, 508.1
Benzo(a)pyrene	525.2, 550, 550.1
Carbofuran	531.1, 6610
Chlordane	505, 508, 525.2, 508.1
Dalapon	552.1, 515.1
Di(2-ethylhexyl)adipate	506, 525.2
Di(2-ethylhexyl)phthalate	506, 525.2
Dibromochloropropane	504.1, 551
Dinoseb	515.2, 555, 515.1
Diquat	549.1
Endothall	548.1
Endrin	505, 508, 525.2, 508.1
Ethylene dibromide	504.1, 551
Glyphosate	547, 6651
Heptachlor	505, 508, 525.2, 508.1
Heptachlor epoxide	505, 508, 525.2, 508.1
Hexachlorobenzene	505, 508, 525.2, 508.1
Hexachlorocyclopentadiene	505, 525.2, 508, 508.1
Lindane	505, 508, 525.2, 508.1
Methoxychlor	505, 508, 525.2, 508.1
Oxamyl	531.1, 6610
PCBs	
as Decachlorobiphenyl [▲]	508A
as Aroclors	505, 508
Pentachlorophenol	515.2, 525.2, 555, 515.1
Picloram	515.2, 555, 515.1
Simazine	505 [■] , 507, 525.2, 508.1
Toxaphene	505, 508, 525.2
Trihalomethanes (total)	502.2, 524.2, 551

[■] If lower detection limits of alachlor, atrazine, and simazine are required, a nitrogen-phosphorous detector should be substituted for the electron capture detector in Method 505; or, another approved method should be used.

[▲]PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl using Method 508A.

*Regulations specified by the US EPA require monitoring for certain contaminants to which maximum contaminant levels do not apply. These chemicals are called "unregulated" contaminants, and presently include sulfate, 34 volatile organic chemicals (VOCs) and 13 synthetic organic chemicals (SOCs).

Table 2. Unregulated Drinking Water Compounds*

VOC Contaminants	Methods
Chloroform	502.2, 524.2, 555
Bromodichloromethane	502.2, 524.2, 551
Bromoform	502.2, 524.2, 551
Chlorodibromomethane	502.2, 524.2, 551
Bromobenzene	502.2, 524.2
Bromochloromethane	502.2, 524.2
Bromomethane	502.2, 524.2
n-Butylbenzene	502.2, 524.2
sec-Butylbenzene	502.2, 524.2
tert-Butylbenzene	502.2, 524.2
Chloroethane	502.2, 524.2
Chloromethane	502.2, 524.2
o-Chlorotoluene	502.2, 524.2
p-Chlorotoluene	502.2, 524.2
Dibromomethane	502.2, 524.2
m-Dichlorobenzene	502.2, 524.2
Dichlorodifluoromethane	502.2, 524.2
1,1-Dichloroethane	502.2, 524.2
1,3-Dichloropropane	502.2, 524.2
2,2-Dichloropropane	502.2, 524.2
1,1-Dichloropropene	502.2, 524.2
1,3-Dichloropropene	502.2, 524.2
Fluorotrichloromethane	502.2, 524.2
Hexachlorobutadiene	502.2, 524.2
Isopropylbenzene	502.2, 524.2
p-Isopropyltoluene	502.2, 524.2
Naphthalene	502.2, 524.2
n-Propylbenzene	502.2, 524.2
1,1,2,2-Tetrachloroethane	502.2, 524.2
1,1,1,2-Tetrachloroethane	502.2, 524.2
1,2,3-Trichlorobenzene	502.2, 524.2
1,2,3-Trichloropropane	502.2, 524.2, 504.1
1,2,4-Trimethylbenzene	502.2, 524.2
1,3,5-Trimethylbenzene	502.2, 524.2

SOC Contaminants	Methods
Aldicarb	531.1, 6610
Aldicarb sulfone	531.1, 6610
Aldicarb sulfoxide	531.1, 6610
Aldrin	505, 508, 525.2, 508.1
Butachlor	507, 525.2
Carbaryl	531.1, 6610
Dicamba	515.1, 515.2, 555
Dieldrin	505, 508, 525.2, 508.1
3-Hydroxycarbofuran	531.1, 6610
Methomyl	531.1, 6610
Metolachlor	507, 525.2, 508.1
Metribuzin	507, 525.2, 508.1
Propachlor	508, 525.2, 508.1

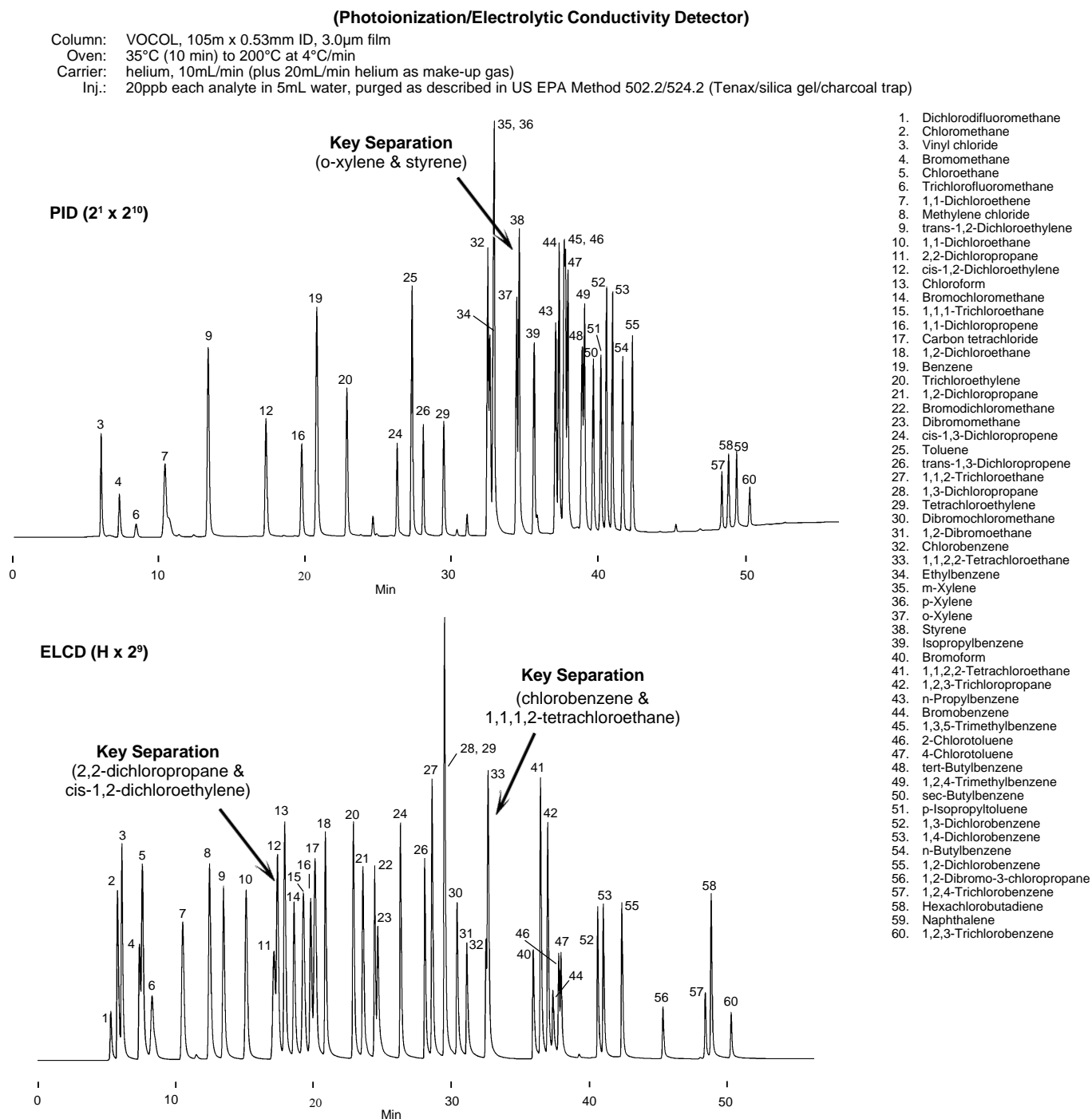
References

1. Methods 502.2, 505, 507, 508, 508A, 515.1, and 531.1 are in *Methods for the Determination of Organic Compounds in Drinking Water*. Order number PB 91-231480.
Methods 506, 547, 550, 508, 550.1, and 551 are in *Methods for the Determination of Organic Compounds in Drinking Water — Supplement II*. Order number PB 91-146027.
Methods 515.2, 524.2, 548.1, 549.1, 552.1, and 555 are in *Methods for the Determination of Organic Compounds in Drinking Water — Supplement II*. Order number PB 92-207703.
Methods 502.1 and 503.1 are replaced by Method 502.2, effective 7/1/96.
Method 524.1 is replaced by Method 524.2 effective 7/1/96.
Current methods are available from NTIS, 5285 Port Royal Road, Springfield, VA 22161.
2. National Technical Information Service, US Department of Commerce, *Technical Note for Drinking Water*, NTIS Document #PB95-104766, NTIS, 5285 Port Royal Road, Springfield, VA 22161 USA. Phone 800-553-6847.

*Methods listed in this bulletin are included in reference 1. Obtain methods for other compounds separately from the same source.

Method 502.2 – Volatile Organics (Replaces Methods 502.1 and 503.1)

Figure A. Volatile Compounds by Method 502.2



Purge and Trap Concentrator: O.I. Analytical Corporation Model 4460A
 Tandem PID/ELCD detector: O.I. Analytical Corporation Model 4440
 Chromatograms provided courtesy of O.I. Analytical, College Station, TX.

713-1165, 1166

Sample Preparation: Purge and trap using a VOCARB 4000 trap (8.5cm Carbopack™ C/10cm Carbopack B/6cm Carboxen 1000/1cm Carboxen 1001).

GC Column: VOCOL wide bore capillary column, 105m x 0.53mm ID fused silica, 3.0µm phase film.

Confirmational Column: None listed.

Detector: Photoionization and electrolytic/conductivity, in series.

Chemical Standards: See page 13.

Qualifications: Current EPA regulations require water utilities to monitor for 49 volatile compounds (27 are unregulated) which could be monitored with a VOCOL column.

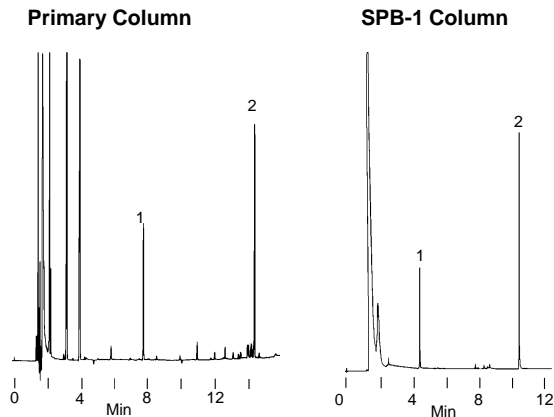
Also see listings for Method 524.2.

Method 504 – 1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane

Figure B. Ethylene Dibromide and Dibromochloropropane by Method 504

Column: *Primary* – SPB-1/Carbowax 20M (25:75), 30m x 0.32mm ID, 0.25µm film
SPB-1 Column – 30m x 0.32mm ID, 0.25µm film
Oven: 40°C (4 min) to 190°C at 8°C/min
Carrier: helium, 25cm/sec
Det.: ECD, 4×10^{-11} (primary column) or 128×10^{-11} AFS
Inj.: 1µL hexane containing 2ng each analyte, split 100:1

1. Ethylene dibromide
2. Dibromochloropropane



796-0317, 0318

Sample Preparation: Extract with hexane.

GC Column: SPB™-1 methyl polysiloxane/Carbowax® 20M polyethylene glycol capillary column (25:75), 30m x 0.32mm ID fused silica, 0.25µm film.

Confirmational Column: SPB-1 capillary column, 30m x 0.32mm ID fused silica, 1.0µm phase film. Figure B shows a 30m x 0.32mm ID, 0.25µm film SPB-1 column also provides excellent results.

Detector: Electron capture.

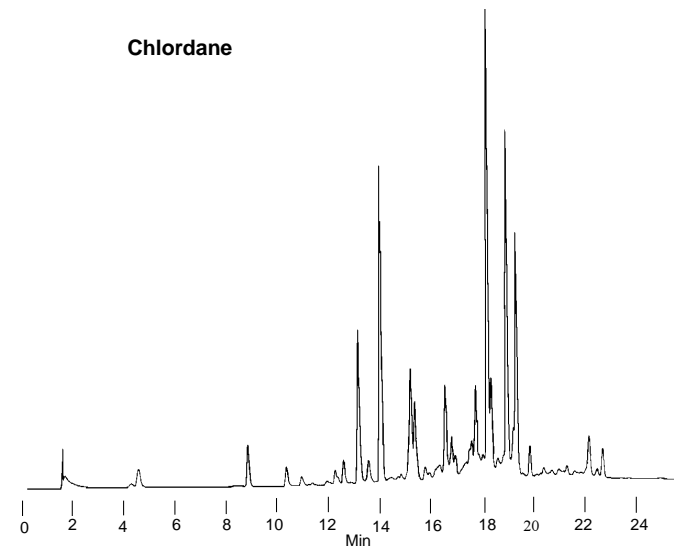
Chemical Standard: EPA EDB/DBCP Mix.

Qualifications: The capillary GC system must be equipped with a splitless injector.

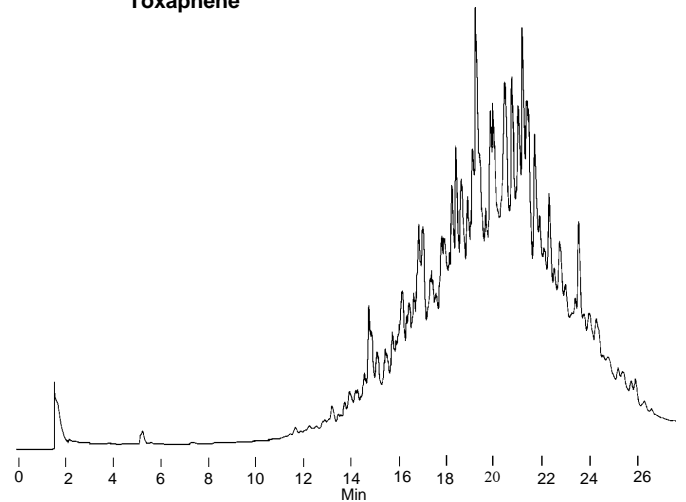
Method 505 – Organohalide Pesticides & PCBs/Microextraction GC/ECD

Figure C. Chlordane or Toxaphene by Method 505

Column: SPB-1, 30m x 0.32mm ID, 1.0µm film
Oven: 180°C to 260°C at 4°C/min
Carrier: helium, 25cm/sec
Det.: ECD, 32 (chlordane) or 16×10^{-11} AFS
Inj.: 1µL hexane containing 2ng each analyte, split 100:1



Toxaphene

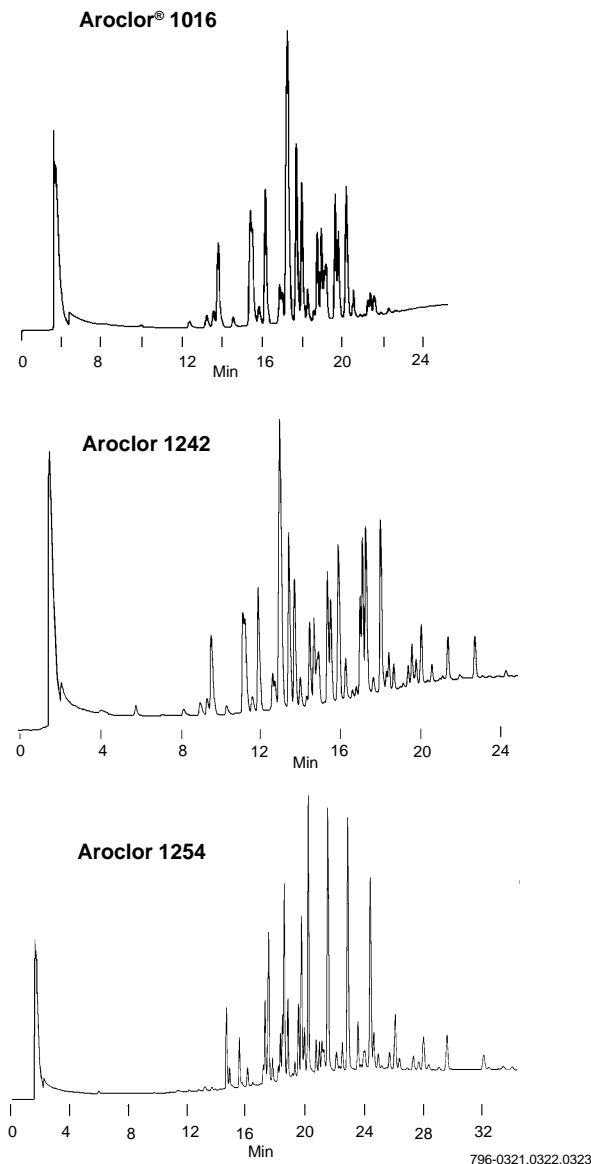


796-0319, 0320

Method 505 contd. on next page.

Figure D. Polychlorinated Biphenyls Mixtures by Method 505

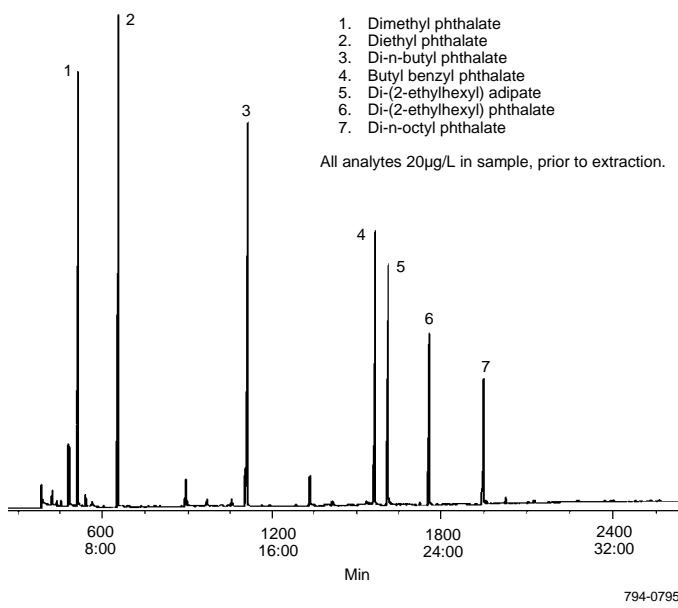
Column: SPB-1, 30m x 0.32mm ID, 1.0µm film
 Oven: 180°C to 260°C at 4°C/min
 Carrier: helium, 25cm/sec
 Det.: ECD, 4 x 10⁻¹¹ AFS
 Inj.: 1µL hexane containing 2ng Aroclor PCBs mixture, split 100:1



Method 506 – Phthalate and Adipate Esters

Figure E. Phthalate and Adipate Esters

Sample: 1L drinking water, add 5mL methanol and mix thoroughly
 Extraction Disk: ENVI-18 DSK, 47mm
 Conditioning: 5mL dichloromethane (pull completely through disk)
 5mL methanol (do not allow disk to dry out)
 5mL reagent water
 Sample Addition: adjust vacuum to flow rate of 100mL/min
 Extraction: rinse sample container with 5mL acetonitrile,
 extract disk with solution, rinse container with
 2 x 5mL dichloromethane, extract disk with solution
 Column: 5% diphenyl/95% dimethyl silicone capillary (PTE-5
 equivalent), 30m x 0.25mm ID, 0.25µm film
 Oven: 40°C (1 min) to 160°C (3 min),
 then to 300°C (3 min) at 6°C/min
 Carrier: helium, 30cm/sec
 Det.: MS, scan range m/z=45-450
 Inj.: 1µL, split/splitless, 45 sec delay, 240°C



Sample Preparation: ENVI™-18 DSK extraction disk, 47mm.

GC Column: 5% diphenyl/95% dimethyl silicone capillary, 30m x 0.25mm ID, 0.25µm film (PTE-5 equivalent).

Confirmational Column: SPB-1 capillary column, 30m x 0.32mm ID fused silica, 0.25µm phase film.

Detector: PID, 10 volt.

Chemical Standards: See page 13.

Sample Preparation: Extract with hexane.

GC Column: SPB-1 methyl polysiloxane capillary column, 30m x 0.32mm ID fused silica, 1.0µm phase film.

Confirmational Column: SPB-1/Carbowax 20M PEG (50:50) capillary column, 30m x 0.32mm ID fused silica, 0.25µm phase film, or Methyl/phenyl silicone (50:50) capillary column (e.g. SPB-50), 25m x 0.25mm ID fused silica, 1.5µm phase film.

Detector: Electron capture.

Chemical Standards: See page 13.

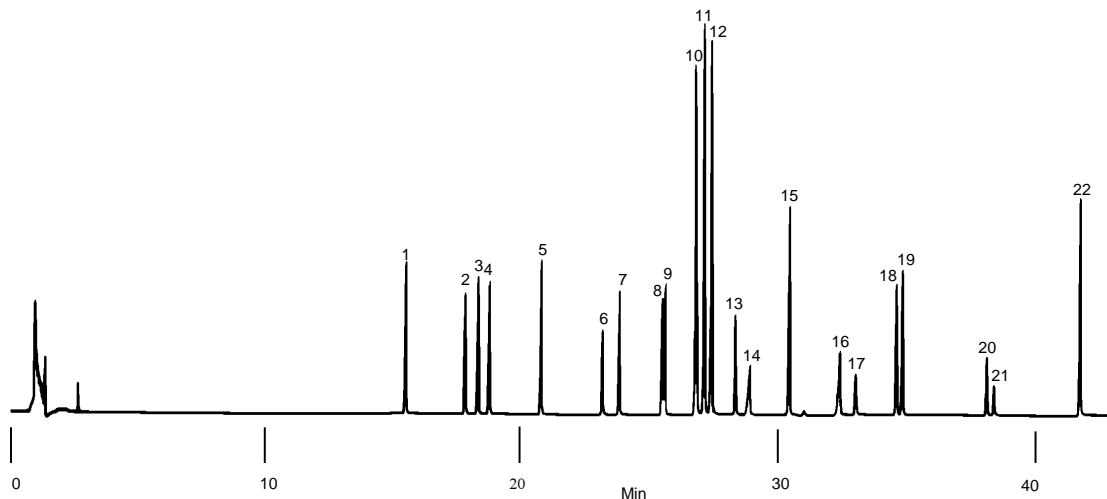
Qualifications: Either on-column or splitless injection may be used.

Method 507 – Nitrogen- & Phosphorus-Containing Pesticides

Figure F. Nitrogen-Containing Herbicides by Method 507

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 60°C to 300°C at 4°C/min
Carrier: helium, 30cm/sec
Det.: TSD, 64 x 10⁻¹² AFS
Inj.: 2µL ethyl acetate containing 5ng each herbicide, splitless

1. Eptam®
2. Sutan®
3. Vernam®
4. Tillam®
5. Ordram®
6. Propachlor
7. Ro-Neet®
8. Treflan®
9. Balan®
10. Simazine
11. Atrazine
12. Propazine
13. Tolban®
14. Terbacil
15. Sencor®
16. Bromacil
17. Dual®
18. Paarlan®
19. Prowl®
20. Oxadiazon
21. Goal®
22. Hexazinone

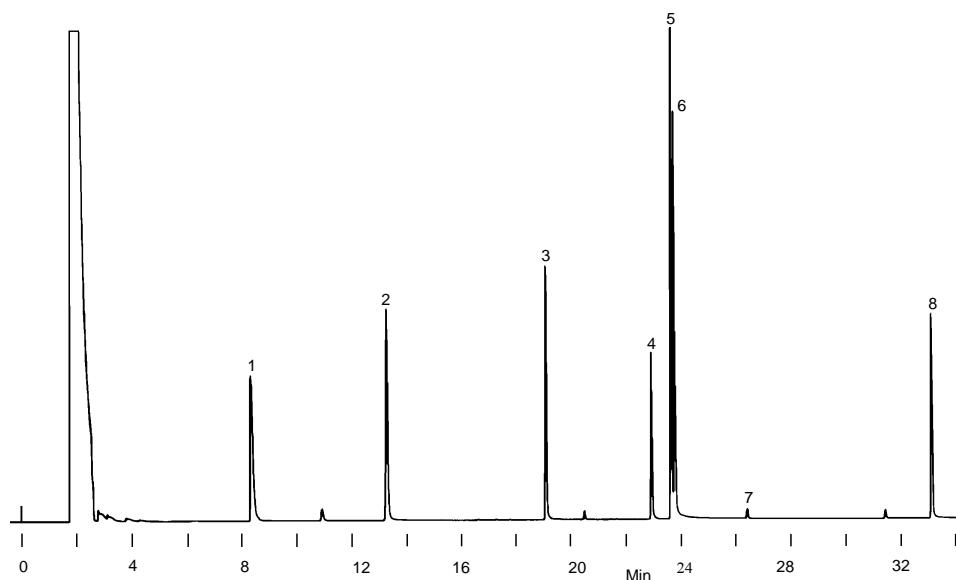


713-1062

Figure G. Organophosphorus Compounds and Alachlor by Method 507

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 100°C to 300°C at 4°C/min
Carrier: helium, 20cm/sec
Det.: TSD, 256 x 10⁻¹¹ AFS
Inj.: 1µL methyl tertiary butyl ether containing 10ng each analyte, splitless

1. Dichlorvos
2. Mevinphos
3. Ethoprop
4. Terbufos
5. Disulfoton
6. Diazinon
7. Alachlor
8. Merphos



713-1063

Sample Preparation: Extract with methylene chloride, concentrate, transfer to methyl tert-butyl ether.

GC Column: PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column: SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Detector: Nitrogen-phosphorus (NPD) or TSD.

Chemical Standards: See page 13.

Qualifications: Splitless injection is recommended.

Method 508 – Chlorinated Pesticides

Method 508.1 – Chlorinated Pesticides

Method 508A (screen test) — Perchlorinated PCBs

Figure H. Chlorinated Pesticides by Method 508

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 60°C to 300°C at 4°C/min
Carrier: helium, 30cm/sec
Det.: ECD, 32 x 10⁻¹¹ AFS
Inj.: 2µL methyl tertiary butyl ether containing 20-2000pg each analyte, splitless

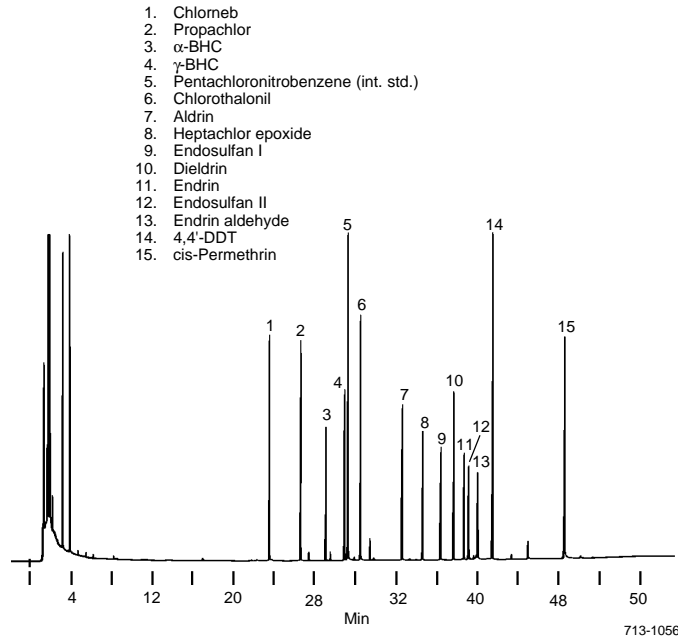
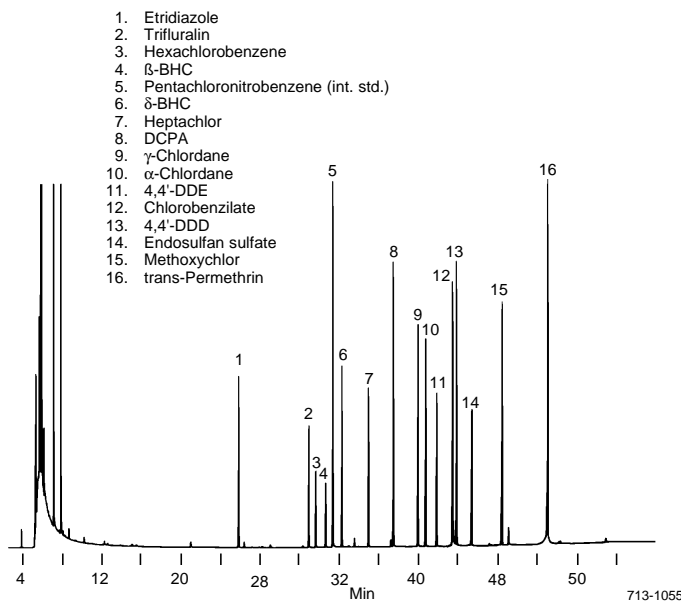


Figure I. Chlorinated Pesticides by Method 508

Conditions: same as Figure H



Method 508

Sample Preparation: Extract 1 liter water sample with methylene chloride, concentrate, transfer to methyl tert-butyl ether.

GC Column: PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column: SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Detector: Electron capture.

Chemical Standards: See page 14.

Qualifications: Refer to the EPA method for relative retention times for pesticides.

Method 508A

Sample Preparation: Extract 1L water sample with methylene chloride, dry, concentrate, transfer to chloroform. Perchlorinate PCBs to decachlorobiphenyl for GC confirmation.

GC Column: PTE-5 capillary column, 30m x 0.32mm ID fused silica, 0.25µm phase film.

Detector: Electron capture.

Chemical Standards: See page 14.

Method 515.1 (revision 4) – Chlorinated Herbicides

Method 515.2– Chlorinated Acids

Sample Preparation:

515.1: 1 liter sample volume adjusted to pH 12; extract in ethyl ether before conversion to methyl esters with diazomethane.

515.2: 250mL sample volume; extract acids with 47mm resin disk.

GC Column:

PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column:

SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

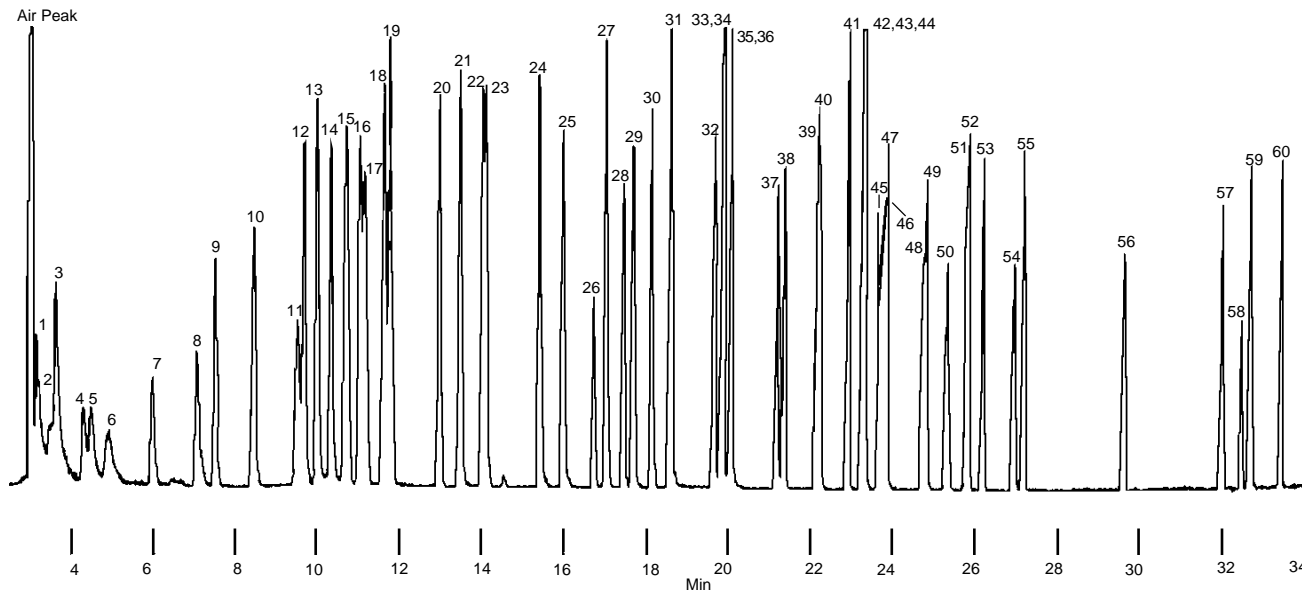
Method 524.2 – Purgeable Organics (GC/MS) (Replaces Method 524.1)

Figure J. Purgeable Organics by Method 524.2

(Using Tekmar® LCS 2000 Purge & Trap and GC/MS)

Column: VOCOL, 60m x 0.75mm ID, 1.5µm film
Oven: 10°C (4 min) to 170°C at 4°C/min
Carrier: 10mL/min
Det.: MS, scan range m/z=33-275, 1 scan/0.700 sec
Inj.: composite of VOC mixes, 100ppb each compound in 5mL water

- | | | | |
|-----------------------------|-------------------------------|-------------------------------|------------------------------|
| 1. Dichlorodifluoromethane | 16. 1,1-Dichloropropene | 31. 1,2-Dibromoethane | 46. 1,2,4-Trimethylbenzene |
| 2. Chloromethane | 17. Tetrachloromethane | 32. Chlorobenzene | 47. 4-Chlorotoluene |
| 3. Vinyl chloride | 18. Benzene | 33. 1,1,1,2-Tetrachloroethane | 48. tert-Butylbenzene |
| 4. Bromomethane | 19. 1,2-Dichloroethane | 34. Ethyl benzene | 49. 1,2,4-Trimethylbenzene |
| 5. Chloroethane | 20. Trichloroethene | 35. p-Xylene | 50. sec-Butylbenzene |
| 6. Trichlorofluoromethane | 21. 1,2-Dichloropropane | 36. m-Xylene | 51. Isopropyltoluene |
| 7. 1,1-Dichloroethene | 22. Bromodichloromethane | 37. o-Xylene | 52. 1,3-Dichlorobenzene |
| 8. Dichloromethane | 23. Dibromomethane | 38. Styrene | 53. 1,4-Dichlorobenzene |
| 9. trans-1,2-Dichloroethene | 24. cis-1,3-Dichloropropene | 39. Bromoform | 54. Butylbenzene |
| 10. 1,1-Dichloroethane | 25. Toluene | 40. Isopropylbenzene | 55. 1,2-Dichlorobenzene |
| 11. 2,2-Dichloropropane | 26. trans-1,3-Dichloropropene | 41. 1,1,2,2-Tetrachloroethane | 56. 1,2-Dibromochloropropane |
| 12. cis-1,2-Dichloroethene | 27. 1,1,2-Trichloroethane | 42. Bromobenzene | 57. 1,2,4-Trichlorobenzene |
| 13. Trichloromethane | 28. Tetrachloroethene | 43. 1,2,3-Trichloropropane | 58. Hexachlorobutadiene |
| 14. Bromochloromethane | 29. 1,3-Dichloropropane | 44. Propylbenzene | 59. Naphthalene |
| 15. 1,1,1-Trichloroethane | 30. Dibromochloromethane | 45. 2-Chlorotoluene | 60. 1,2,3-Trichlorobenzene |



796-0324

Sample Preparation: Purge and trap using a VOCARB 4000 trap (8.5cm Carboxen 1000 / 10cm Carboxen 1001 / 6cm Carboxen 1000 / 1cm Carboxen 1001).

GC Column: VOCOL wide bore capillary column, 60m x 0.75mm ID borosilicate glass, 1.5µm phase film.

Confirmational Column: VOCOL wide bore capillary column, 30m x 0.53mm ID fused silica, 3µm phase film.

Detector: Mass spectrometer (70eV, 35-260amu scanning capability).

Chemical Standards: See page 14.

Qualifications: When used with this method, 30m x 0.53mm ID capillary columns must be cooled to temperatures below 10°C.

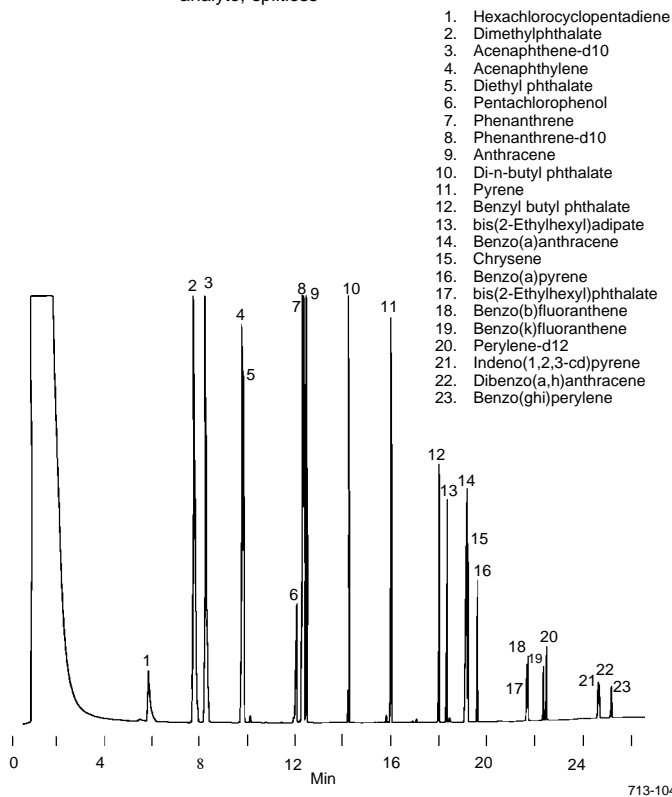
Method 525 – General Purpose Organics

Method 525.1 – General Purpose Organics

Method 525.2 – General Purpose Organics

Figure K. General Purpose Organic Compounds by Method 525

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 120°C (4 min) to 320°C at 10°C/min
(Trap Desorption Temp.: 240°C)
Carrier: helium, 40cm/sec
Det.: FID (32 x 10⁻¹¹ AFS)
Inj.: 1µL methanol/methylene chloride containing 100ng each analyte, splitless



Sample Preparation:

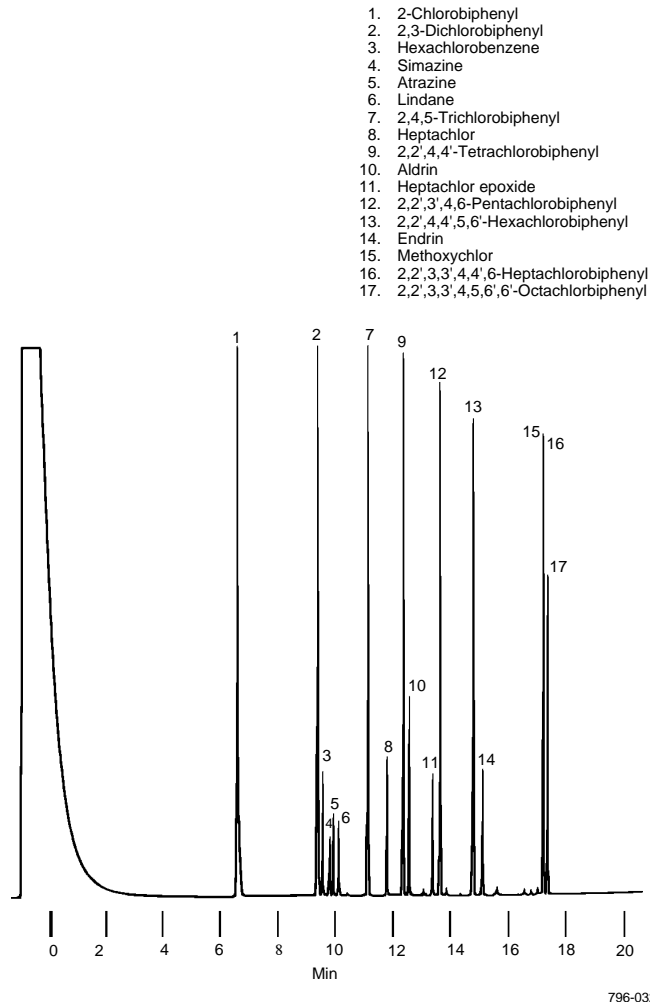
525: Extract 1 liter water sample on ENVI-18 solid phase extraction tube, recover in methylene chloride.

525.1: Extract on ENVI-18 solid phase extraction tube or extraction disk, recover in methylene chloride.

525.2: Extract on ENVI-18 solid phase extraction tube or extraction disk, recover in ethyl acetate followed by methylene chloride.

Figure L. General Purpose Organic Compounds by Method 525

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 120°C (4 min) to 320°C at 10°C/min
(trap desorbed at 240°C)
Carrier: helium, 40cm/sec
Det.: FID (32 x 10⁻¹¹ AFS)
Inj.: 1µL methanol/methylene chloride containing 100ng each pesticide, 250ng each PCB, splitless



GC Column: PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column: None listed.

Detector: Mass spectrometer (70eV, 45-450amu scanning capability).

Chemical Standards: See page 14.

Method 531.1 – N-Methyl Carbomoyloximes & N-Methyl Carbamates

Sample Preparation: Direct injection.

HPLC Column: SUPELCOSM LC-18, 15cm x 4.6mm ID, 5µm packing.

Confirmational Column: None listed.

Detector: Fluorescence (postcolumn derivatization of analytes to methyl amines with o-phthalaldehyde and 2-mercaptoethanol).

Chemical Standards: Custom prepared mix.

Qualifications: A postcolumn reaction system is required.

Method 548.1 – Endothall

Sample Preparation: 100mL sample extracted using an SPE tube (8mL) using ion exchange or extraction disk.

GC Column: PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column: VOCOL, 30m x 0.53mm ID fused silica, 3µm film.

Detector: FID or mass spectrometry.

Chemical Standards: Custom prepared mix.

Method 549.1 – Diquat and Paraquat

Figure M. Diquat and Paraquat by Method 549.1

Sample: 250mL drinking water, adjust sample pH to 10.5 ± 0.2 with sodium hydroxide solution (10% w/v) or hydrochloric acid solution (10% v/v)

Extraction Disk: ENVI-8 DSK, 47mm

Conditioning: 10mL methanol
2 x 10 mL reagent water
10mL conditioning solvent A (0.5g cetyl trimethyl ammonium bromide and 5mL conc. ammonium hydroxide in 500mL water, dilute to 1L)
2 x 10mL reagent water
10mL conditioning solvent B (10.0g hexanesulfonic acid, sodium salt and 10mL ammonium hydroxide in 250mL deionized water, dilute to 500mL)

Sample Addition: adjust vacuum to flow rate of 100mL/min

Extraction: 0.5 to 1.0mL methanol (to cover disk)
2 x 4mL eluting solution (13.5mL orthophosphoric acid and 10.3mL diethylamine in 500mL water, dilute to 1L)

Column: C18, 15cm x 4.6mm ID, 5µm particles

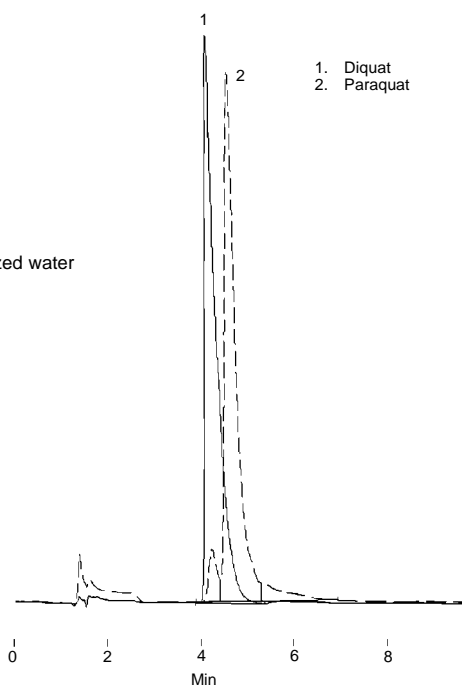
Mobile Phase: 3.5mL triethylamine and 1.0g 1-hexane-sulfonic acid, sodium salt to 800mL deionized water add orthophosphoric acid to pH = 2.5, dilute to 1L

Flow Rate: 1.0mL/min

Temp.: 35°C

Det.: Photodiode array, quantitate Diquat -308nm, Paraquat -257nm

Inj.: 100µL



794-0715

Sample Preparation: C8 SPE tube (500mg) or extraction disk.

HPLC Column: LC-18, 15m x 4.6mm ID, 5µm particles.

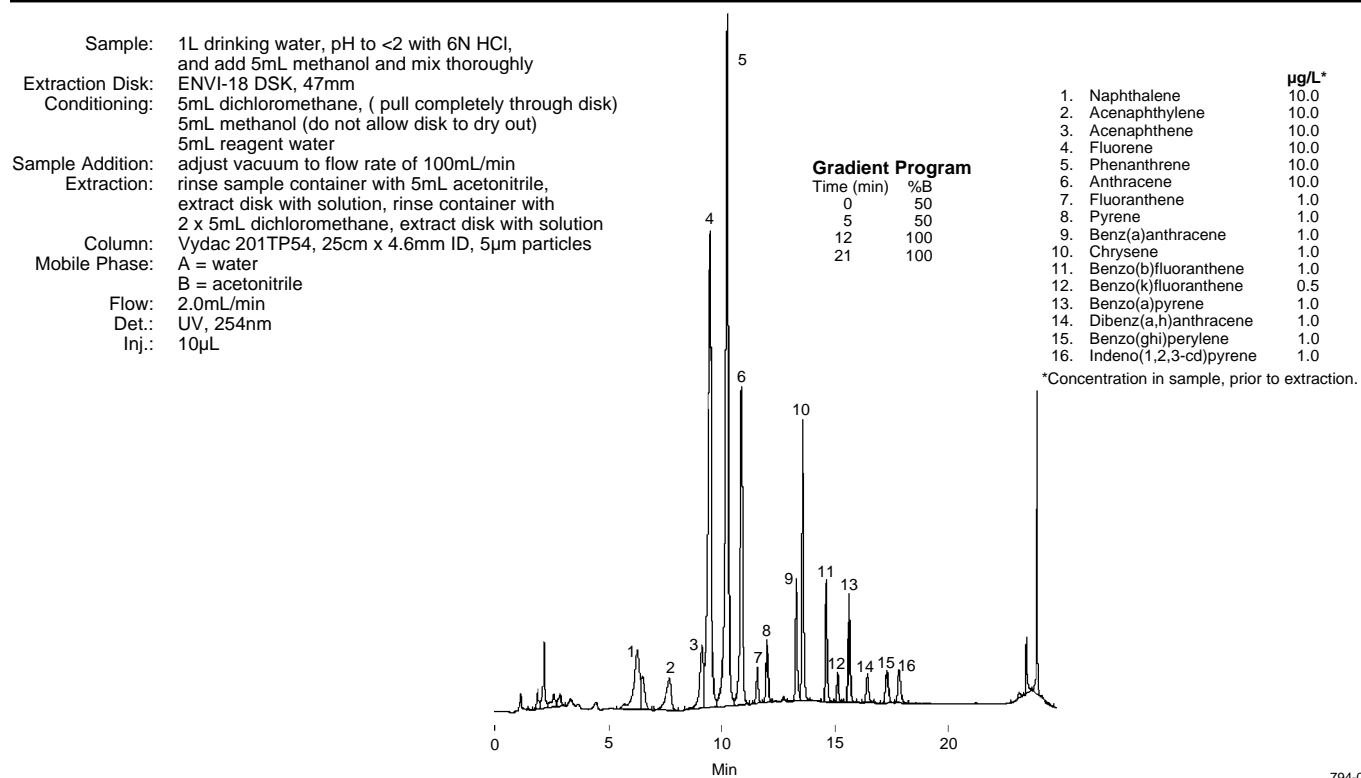
Confirmational Column: None listed.

Detector: Photodiode array, 308nm for diquat, 257nm for paraquat.

Chemical Standards: Custom prepared mix.

Method 550.1 – Polycyclic Aromatic Hydrocarbons

Figure N. Polycyclic Aromatic Hydrocarbons by Method 550.1



Sample Preparation: ENVI-18 DSK extraction disk, 47mm.

HPLC Column: 25cm x 4.6mm ID, 5µm particles.

Confirmational Column: None listed.

Detector: UV, 254nm.

Chemical Standards: Custom prepared mix.

Method 551 – Chlorinated Disinfection Byproducts and Chlorinated Solvents

Sample Preparation: 35mL sample extracted with 2mL methyl-tert-butyl ether.

GC Column: SPB-1 capillary column, 30m x 0.32mm ID fused silica, 1.0µm phase film.

Confirmational Column: SPB-2401 capillary column, 30m x 0.32mm ID fused silica, 0.5µm phase film.

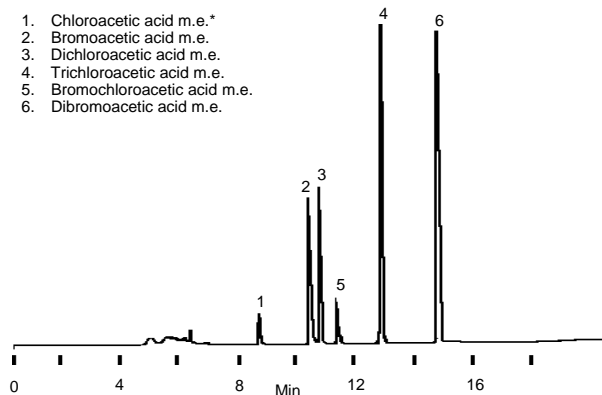
Chemical Standards: See page 15.

Method 552.1 – Haloacetic Acids and Dalapon

Method 552.2 – Haloacetic Acids and Dalapon

Figure O. Haloacetic Acid Methyl Esters Using PTE-5 Column

Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Oven: 50°C (10 min) to 200°C at 10°C/min
Carrier: helium, 25cm/sec
Det.: ECD, 300°C
Inj.: 1µL MTBE (~0.02ng bromochloroacetic acid, other analytes 0.2ng), splitless, 200°C

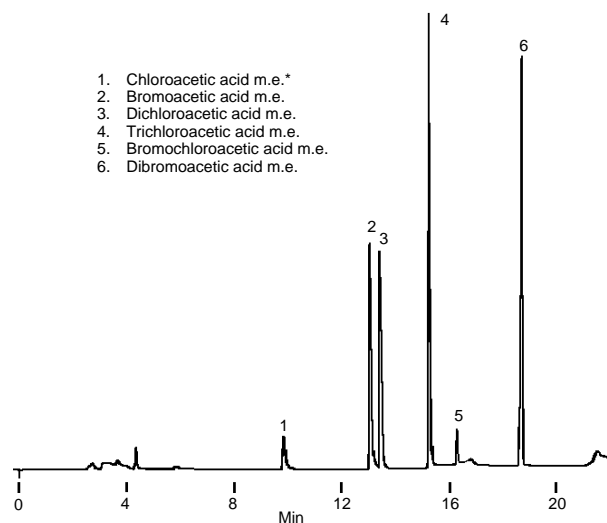


*Compounds analyzed as diazomethane-derivatized methyl esters.

712-0129

Figure P. Haloacetic Acid Methyl Esters Using SPB-1701 Column

Column: SPB-1701, 30m x 0.32mm ID, 0.25µm film
Oven: 50°C (10 min) to 120°C at 10°C/min, hold 3 min
Carrier: helium, 25cm/sec
Det.: ECD, 300°C
Inj.: 1µL MTBE (20ng/mL bromochloroacetic acid, all others 200ng/mL each), splitless 200°C



*Compounds analyzed as diazomethane-derivatized methyl esters.

712-0128

Sample Preparation: 40mL sample is adjusted to pH <0.5, extracted with 4mL methyl tert-butyl ether.

GC Column: PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Confirmational Column: SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm phase film.

Detector: Electron capture.

Chemical Standards: See page 15.

Method 555 – Chlorinated Acids

Sample Preparation: 100mL sample adjusted to pH 12, 20mL aliquot extracted with C18 SPE tube or disk.

HPLC Column: SUPELCOSIL C18, 25cm x 4.6mm ID, 5µm particles.

Confirmational Column: None listed.

Detector: Photodiode array UV.

Chemical Standards: Custom prepared mix.

Ordering Information:

Product	Description	Cat. No.
502.2 Volatile Organics		
Columns		
	VOCOL capillary column, 60m x 0.75mm ID glass, 1.5µm film	23731
	VOCOL capillary column, 105m x 0.53mm ID fused silica, 3µm film	25358
	VOCOL capillary column, 30m x 0.53mm ID fused silica, 3µm film	25320-U
Sample Preparation		
	VOCARB 4000 adsorbent trap, 30.5cm x 0.125" x 0.105" ID, containing Carboxen C, Carboxen B, Carboxen S-III adsorbents	20308*
Calibration Standards[▲]		
	VOC Calibration Standards Kit	48804
	Volatile Organic Compounds Mix 1, 1mL	48775*
	Volatile Organic Compounds Mix 2, 1mL	48777*
	Volatile Organic Compounds Mix 3, 1mL	48779*
	Volatile Organic Compounds Mix 4, 1mL	48786*
	Volatile Organic Compounds Mix 5, 1mL	48797*
	Volatile Organic Compounds Mix 6, 1mL	48799-U*
	Volatile Organic Compounds Mix 7, 1mL	48802-U*
	Volatile Organic Compounds Mix 8, 1mL	48803
	Volatile Organic Compounds Mix 9, 1mL	47399
Internal Standards[▲]		
	1-Chloro-2-fluorobenzene, 2000µg/mL in methanol, 1mL	48369
	2-Bromo-1-chloropropane, 2000µg/mL in methanol, 1mL	48713
	1,4-Dichlorobutane, 2000µg/mL in methanol, 1mL	48066
	EPA 502 Internal Standard Mix, 1mL	48950-U

504.1 1,2-Dibromoethane & 1,2-Dibromo-3-chloropropane

Product	Description	Cat. No.
Columns		
	SPB-1/Carbowax 20M capillary column (25:75), 30m x 0.32mm ID fused silica, 0.25µm film	custom
	SPB-1 capillary column, 30m x 0.32mm ID fused silica, 1.0µm film	24045-U
	VOCOL capillary column, 30m x 0.53mm ID fused silica, 3µm film	25320-U
Sample Preparation		
	Pre-assembled vials, 40mL, pk. of 100	27181
Calibration Standards[▲]		
	EPA EDB/DBCP Mix, 1mL	48225-U

505 Organohalide Pesticides & Polychlorinated Biphenyls (PCBs)

Product	Description	Cat. No.
Columns		
	SPB-1 capillary column, 30m x 0.32mm ID fused silica, 1.0µm film	24045-U
	SPB-1/Carbowax 20M capillary column (25:75), 30m x 0.32mm ID fused silica, 0.25µm film	custom
	SPB-50 capillary column, 30m x 0.32mm ID fused silica, 1.5µm film	custom
Sample Preparation		
	Pre-assembled vials, 40mL, pk. of 100	27181
Calibration Standards[▲]		
	PCB Kit 3	48825
	EPA 505/525 Update Pesticides Mix A, 1mL	47727-U
	EPA 505/525 Update Pesticides Mix B, 1mL	47728-U
	Chlordane, 5000µg/mL in methanol, 1mL	40089
	α-Chlordane, 100µg/mL in hexane, 1mL	48192
	γ-Chlordane, 100µg/mL in hexane, 1mL	48193
	Toxaphene, 5000µg/mL in methanol, 1mL	40111

Product	Description	Cat. No.
506 Phthalate & Adipate Esters		
Columns		
	PTE-5 capillary column, 30m x 0.32mm ID fused silica, 0.25µm film	24143
	SPB-1 capillary column, 30m x 0.32mm ID fused silica, 0.25µm film	24044
Sample Preparation		
	ENVI-18 solid phase extraction tubes, 6mL (1g packing), box of 30	57065
	ENVI-18 DSK extraction disks, 47mm, pk. of 24	57171
	Sample bottle, amber glass, 32oz., with Teflon-lined screw cap	24559
	Separatory funnel, 2 liter, Teflon stopcock	64806
	Kuderna-Danish macro concentrator, w/ground joints, 500mL	64685-U
Calibration Standards[▲]		
	EPA 506 Phthalate Esters Mix 1, 1mL	48223
	EPA Phthalate Esters Mix, 1mL	48805-U
	Di-n-octyl phthalate, 5000µg/mL in methanol, 1mL	40067
	Di(2-ethylhexyl)phthalate, 2000µg/mL in methanol, 1mL	47994
	Di(2-ethylhexyl)adipate, 2000µg/mL in methanol, 1mL	47995-U
507 Nitrogen & Phosphorus Pesticides		
Columns		
	PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24135-U
	SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24113
Sample Preparation		
	Sample bottle, amber glass, 32oz., with Teflon-lined screw cap	24559
	Separatory funnel, 2 liter, Teflon stopcock	64806
	Kuderna-Danish macro concentrator, with ground joints, 500mL	64685-U
Calibration Standards		
	custom prepared	custom
Internal Standard		
	Triphenyl phosphate, 500µg/mL in methyl tert-butyl ether, 1mL	48064
Surrogate Standard		
	1,3-Dimethyl-2-nitrobenzene, 250µg/mL in methyl tert-butyl ether, 1mL	48063
Performance Standard[▲]		
	507 Laboratory Performance Check Solution, 1mL	48946

*For Tekmar LSC purge/trap units. For traps for other models, refer to the Supelco catalog.

▲For a listing of the components of mixes and kits, refer to the Supelco catalog.

*Separate source standard is available for this product.

Product	Description	Cat. No.	Product	Description	Cat. No.
508, 508.1, 508A Chlorinated Pesticides & PCBs			524.2 Purgeable Organics		
Columns[▲]			Columns		
	PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24135-U		VOCOL capillary column, 60m x 0.75mm ID fused silica, 1.5µm film	23731
	SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24113		VOCOL capillary column, 30m x 0.53mm ID fused silica, 3µm film	25320-U
Sample Preparation				PTE-5 capillary column, 30m x 0.32mm ID fused silica, 1µm film	24159
	ENVI-18 solid phase extraction tubes, 6mL (0.5g packing), box of 30	57064	Sample Handling		
	ENVI-18 solid phase extraction tubes, 6mL (1g packing), box of 30	57065		VOCARB 4000 adsorbent trap, 30.5cm x 0.125" x 0.105", containing Carbopack C, Carbopack B, & Carboxen 1000	20308*
	Sample bottle, amber glass, 32 oz., with Teflon-lined screw cap	24559	Calibration Standards[▲]		
	Separatory funnel, 2 liter, Teflon stopcock	64806		VOC Calibration Standards Kit	48804
	Kuderna-Danish macro concentrator, with ground joints, 500mL	64685-U		EPA 524 Volatile Organic Compounds Kit	47936
Calibration Standards[▲]				EPA 524.2 Volatile Organic Compounds Mix, 1mL	47932
	PCB Kit 3	48825-U		EPA 524 Volatile Organic Compounds Mix A, 1mL	47933
	TCL Pesticides Mix, 1mL	48913 ♦		EPA 524 Volatile Organic Compounds Mix B, 1mL	47934
	EPA Phase V 508 Pesticide/SOC MCL Mix, 1mL	47361		Volatile Organic Compounds Mix 1, 1mL	48775*
	Standard Mix A-1, 1mL	47977		Volatile Organic Compounds Mix 2, 1mL	48777*
	Chlordane, 5000µg/mL in methanol, 1mL	40089		Volatile Organic Compounds Mix 3, 1mL	48779*
	α-Chlordane, 100µg/mL in hexane, 1mL	48192		Volatile Organic Compounds Mix 4, 1mL	48786*
	γ-Chlordane, 100µg/mL in hexane, 1mL	48193		Volatile Organic Compounds Mix 5, 1mL	48797*
	Toxaphene, 5000µg/mL in methanol, 1mL	40111		Volatile Organic Compounds Mix 6, 1mL	48799-U*
Internal Standard			Internal Standards[▲]		
	Pentachloronitrobenzene, 5000µg/mL in methanol, 1mL	40156		Fluorobenzene, 2000µg/mL in methanol, 1mL	48943
Surrogate Standard				EPA 524 Internal Standard Mix, 1mL	48948
	4,4'-Dichlorobiphenyl, 500µg/mL in isooctane, 1mL	48260		EPA 524.2 Fortification Solution, 1mL	47358
Performance Standards[▲]			Surrogate Standards[▲]		
	DDT-Endrin Mix, 1mL	48282		EPA 524 Surrogate Standard Mix, 1mL	48466
	EPA Pesticide-Herbicide QC Mix, 5mL	49145		4-Bromofluorobenzene, 2000µg/mL in methanol, 1mL	48083
515.1, 515.2 Chlorinated Herbicides				1,2-Dichlorobenzene-d4, 1mL	48952
Columns			QC Standards[▲]		
	PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24135-U		Discretionary Aromatic Volatiles Mix NC, 1mL [■]	47273
	SPB-1701 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24113		Volatile Organic Contaminants Mix 1 NC, 1mL [■]	47274
Sample Handling				Volatile Organic Contaminants Mix 2 NC, 1mL [■]	47275
	ENVI-18 solid phase extraction tubes, 6mL (0.5g packing), box of 30	57064			
	ENVI-18 solid phase extraction tubes, 6mL (1g packing), box of 30	57065			
	Sample bottle, amber glass, 32oz., with Teflon-lined screw cap	24559			
	Separatory funnel, 2 liter, Teflon stopcock	64806			
	Kuderna-Danish macro concentrator, with ground joints, 500mL	64685-U			

*For Tekmar LSC purge/trap units. For traps for other models, refer to the Supelco catalog.

▲For a listing of the components of mixes and kits, refer to the Supelco catalog.

♦Separate source standard is available for this product.

■This mix is the Supelco equivalent of the former EPA-certified QC sample.

Product	Description	Cat. No.	Product	Description	Cat. No.
525.2 General Purpose Organics			550, 550.1 Polycyclic Aromatic Hydrocarbons		
Column			Column		
	PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24135-U		SUPELCO SIL LC-PAH HPLC column, 25cm x 4.6mm ID, 5µm packing	58229
Sample Handling			Sample Handling		
	ENVI-18 solid phase extraction tubes, 6mL (1g packing), box of 30	57065		ENVI-18 solid phase extraction tubes, 6mL (1g packing), box of 30	57065
	ENVI-18 DSK extraction disks, 47mm, pk. of 24	57171		ENVI-18 DSK extraction disks, 47mm, pk. of 24	57171
Calibration Standards[▲]				Sample bottle, amber glass, 32 oz., with Teflon-lined screw cap	24559
	EPA 505/525 Update Pesticide Mix A, 1mL	47727-U		Separatory funnel, 2 liter, Teflon stopcock	64806
	EPA 505/525 Update Pesticide Mix B, 1mL	47728-U		Kuderna-Danish macro concentrator, with ground joints, 500mL	64685-U
	EPA 525 Update Phthalate Esters Mix, 1mL	47973	Calibration Standard[▲]		
	Volatile Organic Compounds Mix 9, 1mL	47399		TCL Polynuclear Aromatic Hydrocarbons Mix, 1mL	49156
	EPA Phase V 525.1 MCL Pesticides Mix, 1mL	47400	551 Chlorinated Disinfection Byproducts & Chlorinated Solvents		
	525 Polynuclear Aromatic Hydrocarbons Mix A, 1mL	48953	Columns		
	EPA 525 Polynuclear Aromatic Hydrocarbons Mix A, 1mL	48249		SPB-1 capillary column, 30m x 0.32mm ID fused silica, 1.0µm film	24045-U
	EPA 525/525.1 PCB Mix, 1mL	48246		SP-2401 capillary column, 30m x 0.32mm ID fused silica, 0.5µm film	custom
	Toxaphene, 500µg/mL in methanol, 1mL	48243	Sample Handling		
Internal Standards[▲]				Pre-assembled vials, 40mL, pk. of 100	27181
	EPA 525/525.1 Internal Standard Mix, 1mL	48242	Calibration Standards[▲]		
Surrogate Standard				EPA 551 Disinfection By-Products Kit	48112
	Perylene-d12, 2000µg/mL in methylene chloride, 1mL	48081		EPA 551A Halogenated Volatiles Mix, 1mL	48045
Fortification Standards[▲]				EPA 551B Halogenated Volatiles Mix, 1mL	48046
	EPA 525 Fortification Solution A, 1mL	48230		Chloral hydrate, 1000µg/mL in acetonitrile, 1mL	47335
	EPA 525 Fortification Solution B, 1mL	48099	552.1, 552.2 Haloacetic Acids & Dalapon		
QC Standard[▲]			Columns		
	EPA Pesticide-Herbicide QC Mix, 5mL	49145		SPB-1701 capillary column, 30m x 0.32mm ID fused silica, 0.25µm film	24184
531.1 N-Methylcarbamoyloximes & N-Methylcarbamates				SPB-210 capillary column, 30m x 0.32mm ID fused silica, 0.50µm film	24329
Column			Sample Handling		
	SUPELCO SIL LC-18 HPLC column, 15cm x 4.6mm ID, 5µm packing	58230		ENVI-18 solid phase extraction tubes, 1mL, box of 108	57023
	For components of postcolumn reaction system, see our general catalog.		Calibration Standards[▲]		
Calibration Standards				EPA 552.1 Acids Calibration Mix ICR, 1mL	47629
	custom prepared	custom		EPA 552.1 Esters Calibration Mix ICR, 1mL	47630
547 Glyphosate				EPA 552.1 Acids Calibration Mix with Surrogate, 1mL	47652
				EPA 552.1 Esters Calibration Mix with Surrogate, 1mL	47653
	For components of postcolumn reaction system, see our general catalog.		Internal Standard		
Calibration Standards				1,2,3-Trichloropropane, 1000µg/mL in methyl tert-butyl ether, 1mL	47669
	custom prepared	custom	Surrogate Standards		
548.1 Endothal				2,3-Dibromopropionic acid, 1000µg/mL in methyl tert-butyl ether, 1mL	47789
Columns				Methyl-2-bromopropionate, 1000µg/mL in methyl tert-butyl ether, 1mL	47668
	PTE-5 capillary column, 30m x 0.25mm ID fused silica, 0.25µm film	24135-U	555 Chlorinated Acids		
	VOCOL capillary column, 30m x 0.53mm ID fused silica, 3µm film	25320-U	Column		
Calibration Standards				SUPELCO SIL LC-18 HPLC column, 25cm x 4.6mm ID, 5µm packing	58298
	custom prepared	custom	Calibration Standards		
549.1 Diquat & Paraquat				custom prepared	custom
Sample Handling			*For Tekmar LSC purge/trap units. For traps for other models, refer to the Supelco catalog.		
	ENVI-8 solid phase extraction tubes, 6mL (0.5g packing), box of 30	57232	▲For a listing of the components of mixes and kits, refer to the Supelco catalog.		
	ENVI-8 DSK extraction disks, 47mm, pk. of 24	57172	*Separate source standard is available for this product.		
Calibration Standards					
	custom prepared	custom			

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