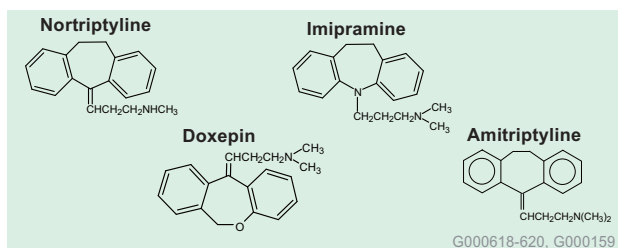


Sample Preparation Applications Drugs



Efficiency of Recovery

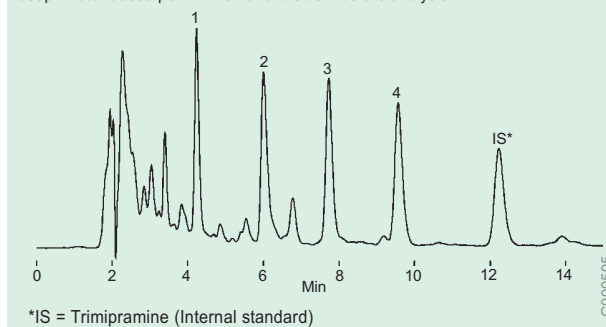
Compound	Concentration (µg/mL)	%Recovery	%RSD (n=6)
1. Nortriptyline	0.10	103.6	±4.5
	0.50	97.5	±4.5
2. Doxepin	0.10	102.2	±3.0
	0.50	100.8	±1.8
3. Imipramine	0.10	92.0	±1.5
	0.50	97.5	±1.7
4. Amitriptyline	0.10	93.6	±1.2
	0.50	95.7	±1.4

SPE Procedure, Using Zymark RapidTrace SPE Workstation

Step	Solvent/Solution	Volume (mL)	Flow Rate (mL/min)	Comments
1. Condition	MeOH	2.0	5.0	conditions sorbent
2. Condition	H ₂ O	2.0	5.0	conditions sorbent
3. Load	spiked porcine serum	2.0 ^A	0.75	applies serum sample
4. Rinse	20% MeOH in H ₂ O	2.0	5.0	washes sorbent
5. Purge-Cannula	H ₂ O	4.0	30.0	cleans sample cannula
6. Rinse	vent	0.1	2.0	positions SPE tube over waste port
7. Dry	N ₂	Time = 10 min		dries sorbent
8. Purge-Cannula	MeOH	4.0	30.0	cleans sample cannula
9. Collect	MeOH	1.0	1.0	elutes analytes into collection vessel
10. Collect	vent	6.0	3.0	pushes residual eluent into vessel ^B
11. Purge-Cannula	H ₂ O	4.0	30.0	cleans sample cannula

^A 1mL porcine serum spiked with 0.1 µg/mL or 0.5 µg/mL each analyte basified with 3 µL 10N KOH, then diluted with 1mL water

^B 350 µL water added per mL methanolic eluent before analysis.



Antidepressants (Tricyclic) From Serum Using Zymark RapidTrace SPE Workstation (SPE/GC)

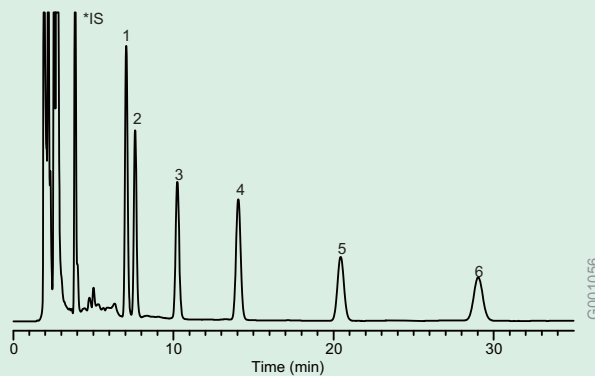
SPE Tube: Discovery DSC-18, 100mg/1mL
Cat. No.: 52602-U
HPLC Column: Discovery C18, 15cm x 4.6mm, 5µm particles, preceded by a 2cm C18 guard column and 0.5µm frit filter
Cat. No.: 504955
Mobile Phase: MeCN:MeOH:25mM KH₂PO₄ (pH 7 with triethylamine) (45:25:30)
Flow Rate: 1mL/min
Temp.: ambient
Det.: UV, 254nm
Inj.: 50 µL diluted porcine serum extract

Efficiency of Recovery

Compound	Concentration (µg/mL)	%Recovery	%RSD (n=6)
1. Phenobarbital	0.5	96.2	±1.6
	1.0	94.9	±1.7
2. Aprobartital	0.5	98.5	±2.1
	1.0	100.8	±0.8
3. Butabartital	0.5	97.2	±1.9
	1.0	98.7	±1.8
4. Mephobarbital	0.5	99.7	±2.4
	1.0	101.0	±2.0
5. Pentobarbital	0.5	96.4	±1.7
	1.0	96.4	±1.9
6. Secobarbital	0.5	98.2	±1.7
	1.0	97.7	±1.8

SPE Method For RapidTrace SPE Workstation Application

- Condition & equilibrate each tube/well with 2mL MeOH & 2mL DI Water
- Load sample
- Wash each tube/well with 2mL 5% MeOH
- Vacuum or air dry with for 5-10 min
This removes any excess water from the sorbent. The presence of water in the final eluent may prolong eluent evaporation.
- Elute with 1-2mL MeOH
- Dry eluate with nitrogen purge (40°C; 15-20 min)
- Reconstitute with 200 µL mobile phase
- Quantify against internal or external standards via HPLC analyses



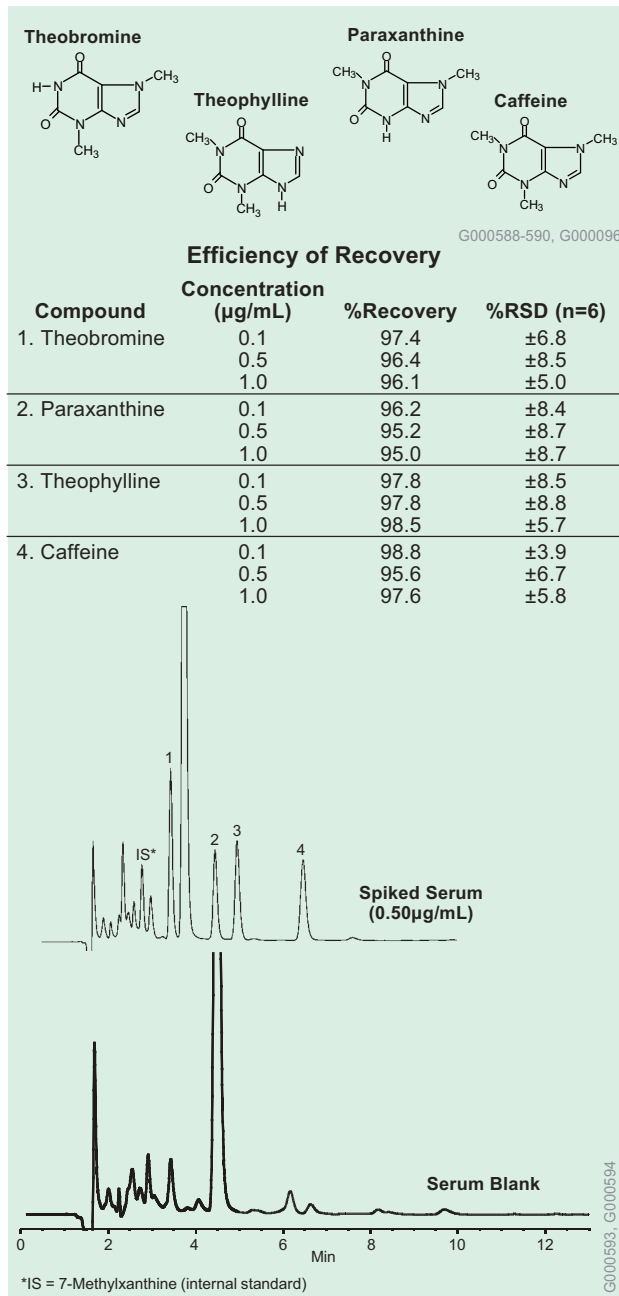
Barbiturates from serum using 500mg/3mL Discovery DSC-18Lt SPE tubes and Zymark's RapidTrace SPE Workstation. *IS = Barbitol (internal standard).

Sample Info: 0.5mL porcine serum spike with 0.5 µg/mL or 1.0 µg/mL each analyte then diluted with 0.5mL water.

Barbiturates From Serum Using Zymark RapidTrace SPE Workstation (SPE/HPLC)

SPE Tube: Discovery DSC-18Lt, 500mg/3mL
Cat. No.: 52613-U
HPLC Column: Discovery C18, 15cm x 4.6mm, 5µm particles, preceded by a 2cm C18 guard column and 0.5µm frit filter
Cat. No.: 504955
Mobile Phase: MeOH/H₂O (40:60)
Flow Rate: 1mL/min
Temp.: 30°C
Det.: UV, 214nm
Inj.: 30 µL diluted porcine serum extract

Sample Preparation Applications Drugs



Bronchodilator: Theophylline and Other Caffeine Metabolites from Serum (SPE/HPLC)

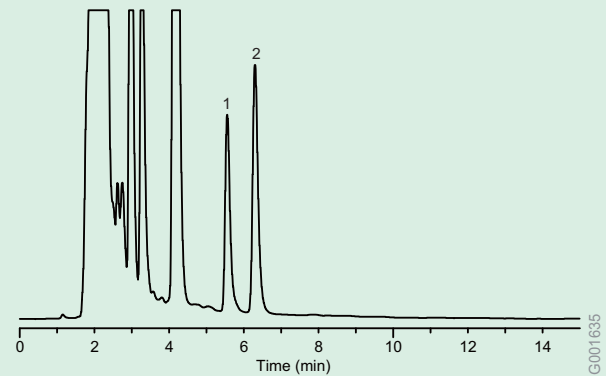
SPE Tube: Discovery DSC-18, 500mg/3mL
Cat. No.: 52603-U
Condition: 2mL methanol, then 2mL water
Apply Sample: 1mL porcine serum spiked with 0.1µg/mL, 0.50µg/mL, or 1.0µg/mL each analyte
Wash and Dry: 2mL 5% methanol in water; dry tube 10 min with nitrogen stream
Elute: 1mL methanol; evaporate to dryness with nitrogen stream at room temperature; reconstitute in 200µL mobile phase containing 0.2µg/mL or 7-methylxanthine (IS)
Column: Discovery RP-AmideC16, 15cm x 4.6mm, 5µm particles, preceded by a 2cm RP-AmideC16 guard column and 0.5µm frit filter
Cat. No.: 505013
Mobile Phase: methanol:1% acetic acid (17:38)
Flow Rate: 1mL/min
Temp.: 30°C
Det.: UV, 272nm
Inj.: 20µL reconstituted porcine serum extract

Efficiency of Recovery

Compound (µg/mL)	Discovery DSC-SCX (n=3)		Leading Competitor SCX (n=2)	
	% Recovery	%RSD	%Recovery	%RSD
1. 3-methylpyrazole (1.0)	89.4	±10.2%	67.1	±20%
2. 4-methylpyrazole (1.0)	79.4	±6.8%	50.5	±30%

SPE Procedure

1. Condition & equilibrate with 2mL MeOH & 2mL DI water.
2. Load 1mL urine sample spiked with 1µg/mL of each analyte.
3. Wash with 2mL DI water.
4. Elute with 2mL 5% MeOH in 250mM phosphate buffer, pH 7.4.
5. Quantify against external standards via HPLC analyses.

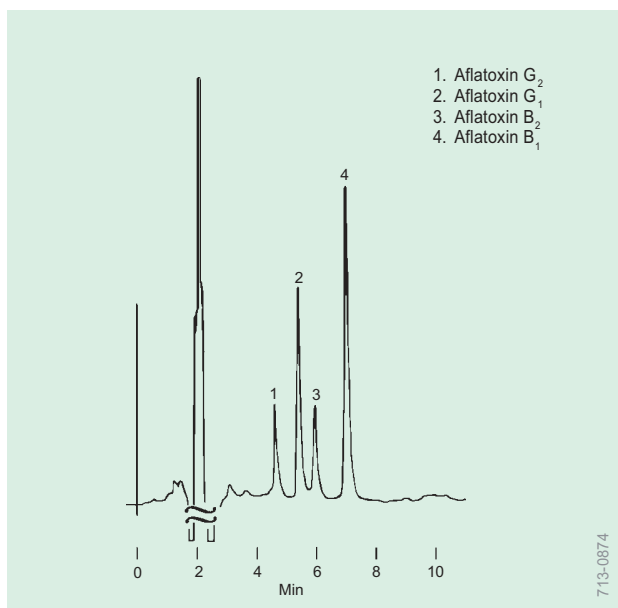


Competitor Comparison of 3-methylpyrazole and 4-methylpyrazole From Urine Using Discovery DSC-SCX (SPE/HPLC)

SPE Tube: Discovery DSC-SCX, 500mg/3mL
Cat. No.: 52686-U
HPLC Column: Discovery C18, 15cm x 4.6mm, 5µm particle, preceded by a 2cm guard column and 0.5µm frit filter
Cat. No.: 504955
Mobile Phase: MeOH:5mM phosphate buffer, pH 6 (20:80)
Flow Rate: 1mL/min
Temp.: 30°C
Det.: UV, 220nm
Inj.: 25µL diluted urine extract

Sample Preparation Applications

Mycotoxins, Herbicides



Aflatoxins (SPE/HPLC)

Sample: cornmeal spiked with aflatoxins (30ppb G₂ and B₂, 100ppb G₁ and B₁) Blend 50g sample for 1 min in 100mL methanol:water (8:2) and filter.

Extraction Tube: Supelclean LC-CN, 500mg/3mL
Cat. No.: 57013

Conditioning: 2mL 0.5% aqueous acetic acid

Sample Addition: 1mL filtered extract + 4mL 0.5% aqueous acetic acid

Washing: 500µL 20% THF in 0.5% aqueous acetic acid, then 2mL hexane. Dry packing under nitrogen. 3mL 25% THF in hexane. Dry packing 1 min under nitrogen.

Elution: 2 x 2mL 1% THF in methylene chloride. Evaporate eluate to dryness under nitrogen. Reconstitute with 100µL of methanol, then dilute with 100µL of 0.5% aqueous acetic acid.

Column: SUPELCOSIL LC-18, 25cm x 4.6mm ID, 5µm particles (with guard column)

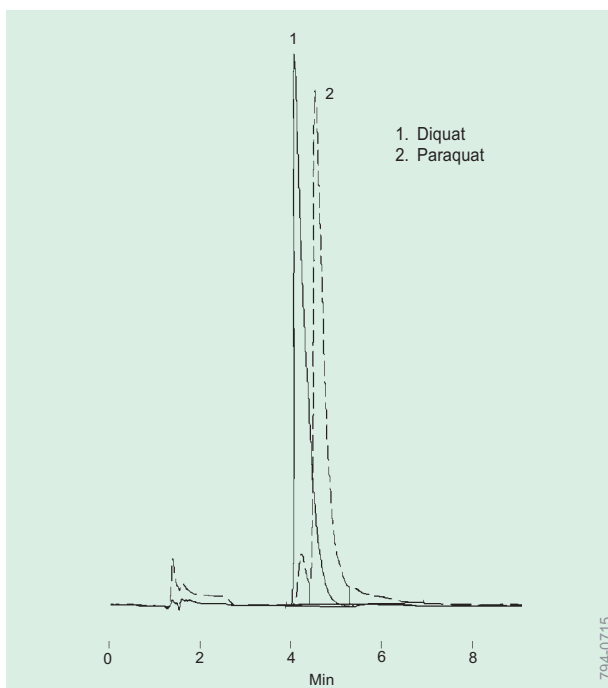
Cat. No.: 58298

Mobile Phase: methanol:acetonitrile:water (22.5:22.5:55)

Flow Rate: 1.5mL/min

Det.: VIS, 365nm

Inj.: 100µL



Paraquat and Diquat (SPE/HPLC)

Refer to US EPA Method 549.1 for full details.

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
Cat. No.: 57172

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: SUPELCOSIL LC-18, 15cm x 4.6mm ID, 5µm particles
Cat. No.: 58230-U

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, quantify
Diquat -308nm, Paraquat -257nm

Inj.: 100µL

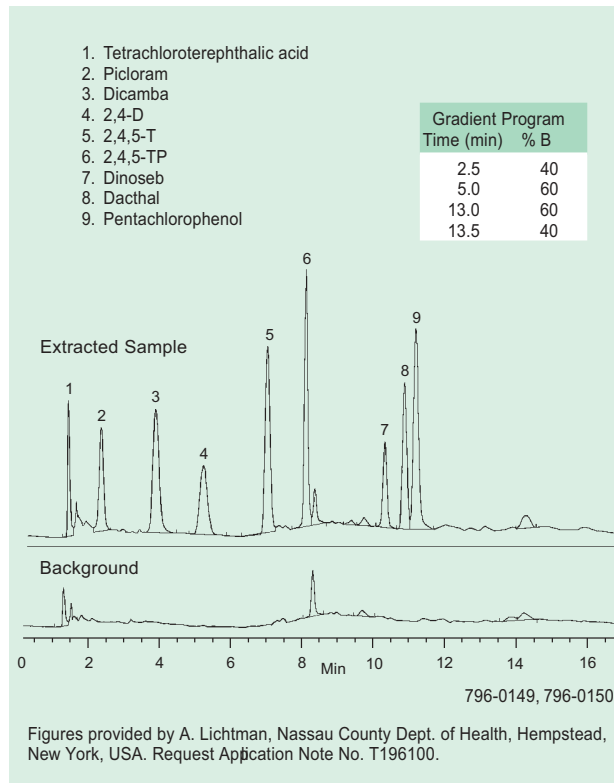
Sample
Preparation

Order: 1.800.325.3010 Technical Service: 1.800.359.3041 Web: www.sigma-aldrich.com/supelco

SUPELCO

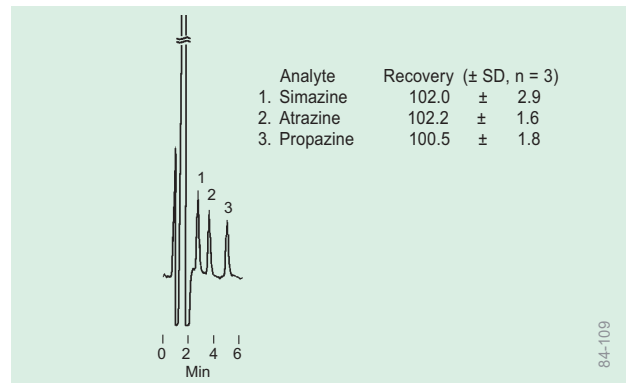
Sample Preparation Applications

Herbicides



Acidic Herbicides in Water (SPE/HPLC)

Extraction Tube: ENVI-Carb, 250mg/6mL
 Cat. No.: 57092
 Column: polymeric-coated silica-based PAH specialty column, 20cm x 3mm ID, 5µm particles
 Supelco Equivalent: SUPELCOSIL LC-PAH (available on request)
 Mobile Phase: gradient, A = water/0.05% H₃PO₄, B = acetonitrile
 Temp.: 50°C
 Flow Rate: 0.5mL/min
 Det.: photodiode array: peak width — 0.053 min sampling interval — 0.320 sec, monitor 210nm & 225nm
 Inj.: 10µL of extract (4-5ppb each analyte in water)



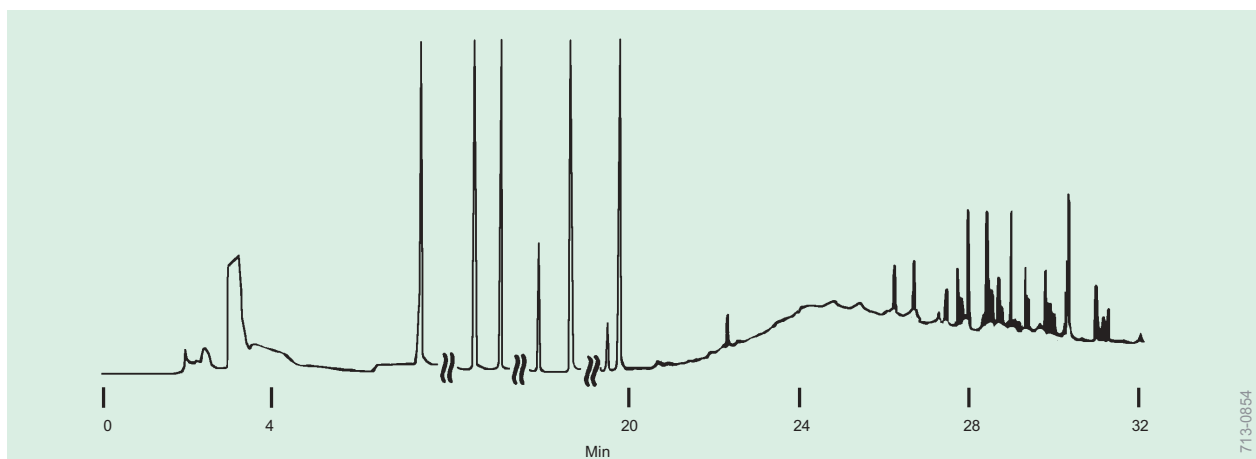
Triazine Herbicides from Grass (SPE/HPLC)

Sample: 5g fresh grass clippings spiked with 2ppm each herbicide. Add 4g anhydrous sodium sulfate and 20mL methylene chloride:acetone (80:20) Shake 20 min and allow mixture to stand 1 min
 Extraction Tube: Supelclean LC-SCX, 500mg/3mL
 Cat. No.: 57018
 Conditioning: 1mL methylene chloride
 Sample Addition: 2mL grass extract
 Washing: 2 x 2mL acetonitrile
 Dry packing for 5 min under nitrogen 2 x 2mL deionized water
 Elution: 1.5mL methanol
 Dilute to 2mL with deionized water.
 Column: SUPELCOSIL LC-8-DB, 15cm x 4.6mm ID, 5µm particles *
 Cat. No.: 58347
 Mobile Phase: acetonitrile:water (45:55)
 Flow Rate: 1.5mL/min
 Det.: UV, 254nm
 Inj.: 100µL

* With Supelguard LC-8-DB column, 2.0cm x 4.6mm ID, 5µm particles

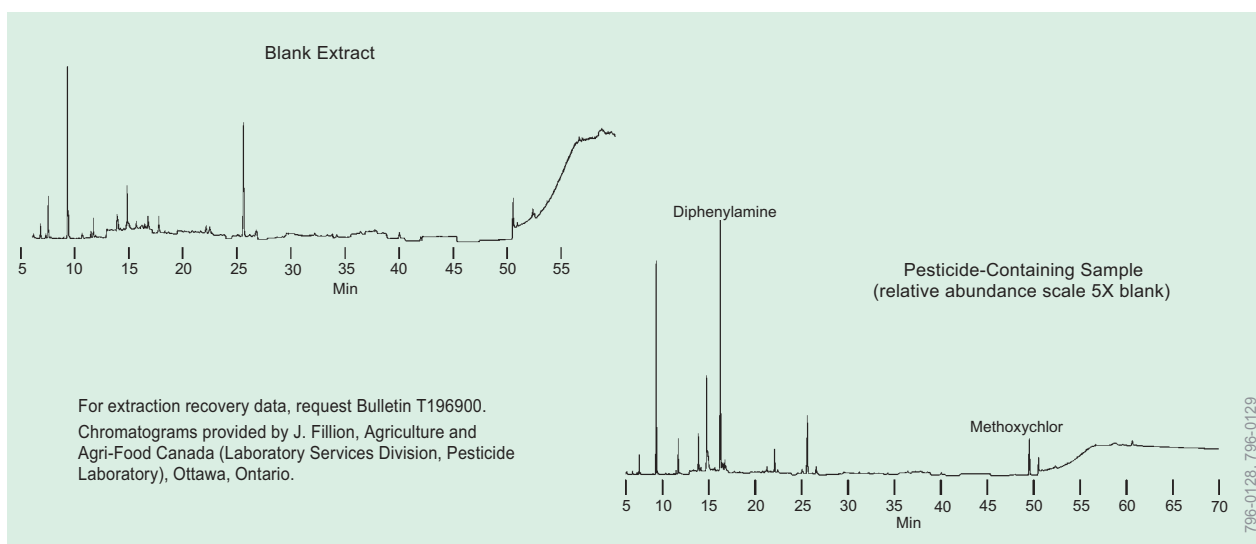
Sample Preparation Applications

PCBs, Pesticides



PCB (SPE/GC)

Sample: used transformer oil containing 50ppb Aroclor 1254
 Extraction Tube: Supelclean LC-Florisil, 1.0g/6mL
 Cat. No.: 57057
 Extraction Tube: Supelclean LC-Si, 1.0g/6mL
 Cat. No.: 57051
 Connect tubes in series, LC-Florisil on top
 Conditioning: 500µL isooctane
 Sample Addition: 0.2g, weigh onto top of frit of tube
 Drying: none
 Elution: 5 x 2mL isooctane, up to 10 mL in volumetric flask
 Column: SPB-5, 30m x 0.32mm ID, 0.25µm film
 Cat. No.: 24048
 Oven: 40°C (4 min) to 300°C at 10°C/min, hold 5 min
 Carrier: nitrogen
 Det.: ECD
 Inj.: 1µL splitless (30 sec delay), then split (50:1)



For extraction recovery data, request Bulletin T196900.
 Chromatograms provided by J. Fillion, Agriculture and
 Agri-Food Canada (Laboratory Services Division, Pesticide
 Laboratory), Ottawa, Ontario.

Pesticides in Fruits and Vegetables (SPE/GC)

Sample: homogenize 50g sample in 100mL acetonitrile then add 10g NaCl and homogenize 5min
 Extraction Tube: ENVI-Carb, 500mg/6mL
 Cat. No.: 57094
 Extraction: Centrifuge 13mL of acetonitrile layer at high speed for 5 min. Evaporate 10mL aliquot to 0.5mL under nitrogen at 35° C. Transfer sample to SPE tube. Elute pesticides with 20mL acetonitrile:toluene (3:1). Concentrate to 2mL by rotary evaporation. Add 2 x 10mL acetone, concentrating material to 2mL after each addition. Add 50µL cis-chlordane in acetone (500ng/µL) then dilute to 2.5mL with acetone.
 Supelco Equivalent: SPB-1701 (available on request)
 Column: 14% cyanopropylphenyl/86% dimethylsiloxane, 30m x 0.25mm ID, 0.15µm film
 Oven: 70°C (hold 2 min) to 130°C at 25°C; 130°C to 220°C at 2°C/min; 220°C to 280°C at 10°C/min and hold 4.6 min
 Carrier: helium
 Det.: MSD, 285°C
 Inj.: 2µL, splitless

Order: 1.800.325.3010 Technical Service: 1.800.359.3041 Web: www.sigma-aldrich.com/supelco

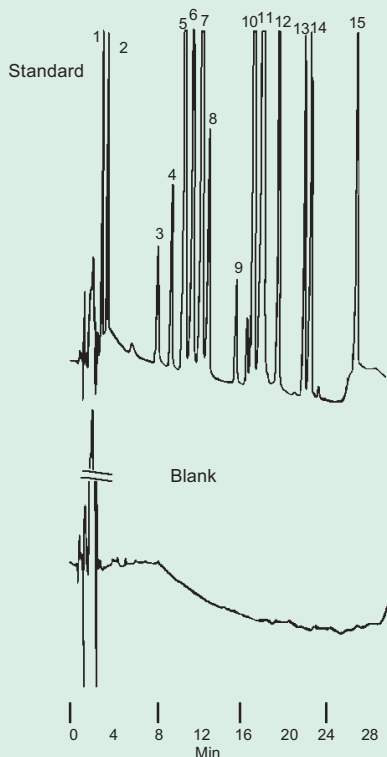
Sample
Preparation

SUPELCO

Sample Preparation Applications

Pesticides

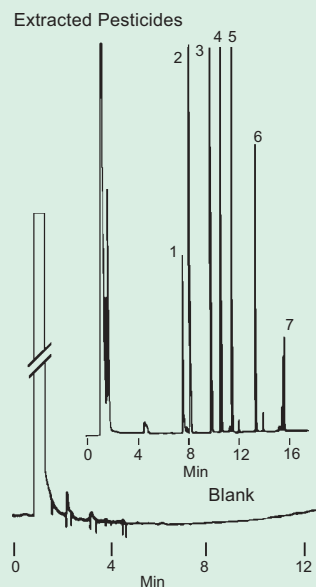
Analyte	% Recovery (\pm RSD, n = 5)
1. Oxamyl	111 \pm 9.6
2. Methomyl	105 \pm 5.0
3. N-1-Naphthylthiourea	—
4. Aldicarb	92 \pm 0.8
5. Simazine	91 \pm 6.5
6. Monuron	99 \pm 3.2
7. Cyanazine	90 \pm 5.4
8. Metribuzin	97 \pm 3.9
9. Carbofuran	106 \pm 6.2
10. Atrazine	89 \pm 5.7
11. Carbaryl	97 \pm 3.5
12. Diuron	88 \pm 5.7
13. Propham	95 \pm 3.2
14. Propachlor	96 \pm 3.8
15. Linuron	88 \pm 5.4



Nonvolatile Pesticides (SPE/HPLC)

Sample: water spiked with pesticides (10-50 μ g/L each component)
 Extraction Tube: Supelclean ENVI-Carb, 250mg/3mL
 Cat. No.: 57088
 Conditioning: 5mL methylene chloride:methanol (80:20)
 1mL methanol
 10mL 2% acetic acid in water
 Keep bed moist until sample is added.
 Sample Addition: 100mL, 5mL/min
 Drying: 1 min with vacuum suction
 Elution: 0.8mL methanol
 2 x 3.5mL methylene chloride:methanol (80:20)
 Dry eluate to 500 μ L under gentle stream of nitrogen
 Reconstitute to 1mL with methanol.
 Column: SUPELCOSIL LC-18-DB, 25cm x 4.6mm ID, 5 μ m particles
 Cat. No.: 58355-U
 Mobile Phase: A = water:acetonitrile (90:10), B = acetonitrile
 80% A for 5 min then to 30% A over 30 min
 Flow Rate: 1.5mL/min
 Det.: UV, 220nm
 Inj.: 20 μ L

Analyte	% Recovery (\pm CV)
1. Hexachlorobenzene	87 \pm 11
2. γ -BHC (Lindane)	99 \pm 13
3. Heptachlor	96 \pm 12
4. Aldrin	94 \pm 13
5. Heptachlor epoxide	98 \pm 13
6. Endrin	93 \pm 11
7. Methoxychlor	110 \pm 13



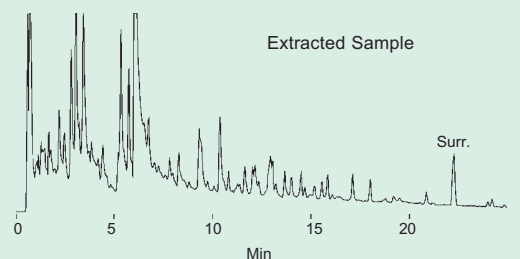
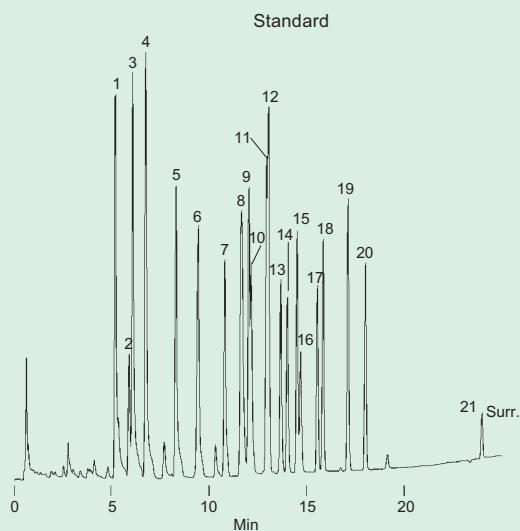
Chlorinated Pesticides (SPE/GC)

Sample: drinking water spiked with pesticides
 Extraction Tube: Supelclean ENVI-18, 500mg/6mL
 Cat. No.: 57064
 Conditioning: 2 x 6mL hexane:ethyl ether (1:1)
 6mL methanol
 6mL deionized water
 Sample Addition: 250mL, 10mL/min
 Drying: 10 min
 Elution: 2 x 1.5mL hexane:ethyl ether (1:1) Concentrate eluate to 2mL under stream of nitrogen.
 Column: PTE-5, 30m x 0.25mm ID, 0.25 μ m film
 Cat. No.: 24135-U
 Oven: 150 $^{\circ}$ C (2 min) to 275 $^{\circ}$ C at 10 $^{\circ}$ C/min
 Carrier: helium
 Det.: ECD, 310 $^{\circ}$ C
 Inj.: 1 μ L

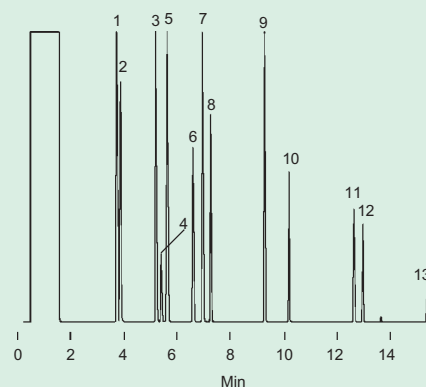
Sample Preparation Applications

Pesticides, Phenols

- | | |
|----------------------------|--------------------------------|
| 1. α -BHC | 12. 4,4'-DDE |
| 2. β -BHC | 13. Endrin |
| 3. γ -BHC (Lindane) | 14. Endosulfan II |
| 4. δ -BHC | 15. 4,4'-DDD |
| 5. Heptachlor | 16. Endrin aldehyde |
| 6. Aldrin | 17. Endosulfan sulfate |
| 7. Heptachlor epoxide | 18. 4,4'-DDT |
| 8. γ -Chlordane | 19. Endrin ketone |
| 9. Endosulfan I | 20. Methoxychlor |
| 10. α -Chlordane | 21. Decachlorobiphenyl (Surr.) |
| 11. Dieldrin | |



Analyte	% Recovery (\pm CV)
1. Phenol	101.8 \pm 3.7
2. 2-Chlorophenol	104.7 \pm 1.7
3. 2-Methylphenol	107.0 \pm 1.9
4. 2-Bromophenol (Int. Std.)	—
5. 3-Methylphenol	104.9 \pm 1.6
6. 2-Nitrophenol	96.3 \pm 2.8
7. 2,4-Dimethylphenol	105.3 \pm 2.2
8. 2,4-Dichlorophenol	106.3 \pm 2.2
9. 4-Chloro-3-methylphenol	104.6 \pm 2.0
10. 2,4,6-Trichlorophenol	103.9 \pm 2.1
11. 4-Nitrophenol	99.9 \pm 4.1
12. 2,3,4,6-Tetrachlorophenol	104.6 \pm 1.5
13. Pentachlorophenol	97.7 \pm 4.8



Phenols (SPE/GC)

Sample: water spiked with phenols
 Extraction Tube: Supelclean ENVI-Chrom P, 250mg/6mL
 Cat. No.: 57225-U
 Conditioning: 6mL methyl t-butyl ether*
 6mL methanol
 6mL deionized water
 Sample Addition: 100mL
 Drying: 10 min, vacuum suction
 Elution: 2mL methyl t-butyl ether,* allow to soak with vacuum off
 2mL methyl t-butyl ether,* elute dropwise
 Additional methyl t-butyl ether until 5mL of eluate is collected.
 Column: PTE-5 QTM, 15m x 0.53mm ID, 0.5 μ m film
 Cat. No.: 25355
 Oven: 65°C to 185°C at 10°C/min, hold 1 min, then to 275°C at
 20°C/min, hold 5 min
 Carrier: helium
 Det.: FID, 300°C
 Inj.: 1 μ L splitless (45 sec hold)

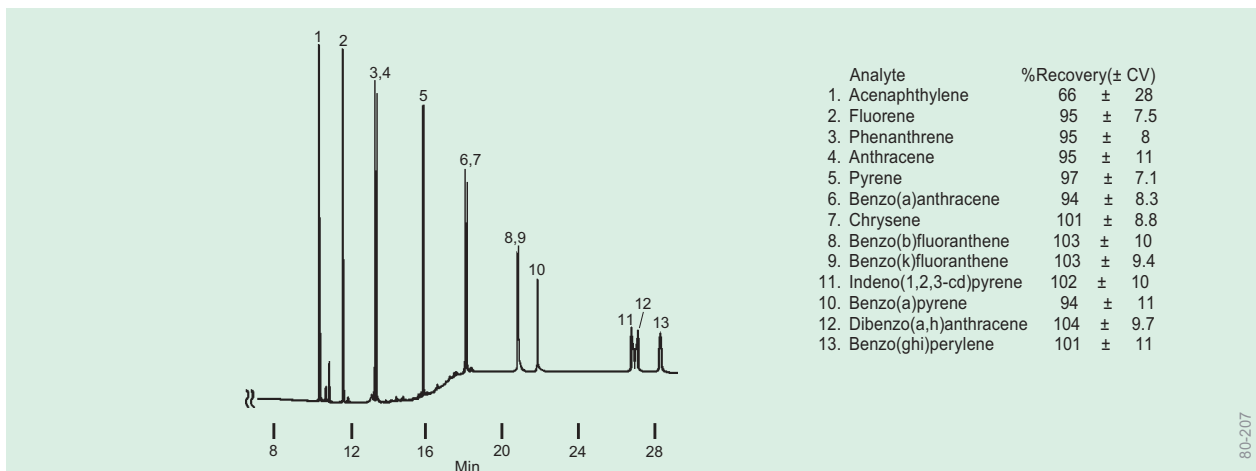
* Can substitute ethyl acetate.

Chlorinated Pesticides in Hazardous Waste (SPE/GC)

Sample: 100mL aqueous hazardous waste, adjust pH to 5-7 if necessary,
 add 5mL methanol
 Extraction Tube: ENVI-8, glass, 500mg/6mL
 Cat. No.: 57107 (0.5g) or 57108 (1g)
 Conditioning: 3mL methanol, (do not allow bed to dry out)
 2mL 5% methanol in water
 Sample Addition: adjust vacuum to flow rate of 5mL/min
 Dry: purge tube with nitrogen for 2-3 min (use Visidry attachment)
 Extraction: 2 x 4mL hexane:acetone (90:10), allow to soak,
 elute dropwise
 Column: PTE-5 QTM, 15m x 0.53mm ID, 0.5 μ m film
 Cat. No.: 25355
 Oven: 150°C (0.5min) to 275°C (5min)
 Carrier: helium
 Det.: ECD, 300°C
 Inj.: 1 μ L, 200°C, split/splitless (45 sec delay)

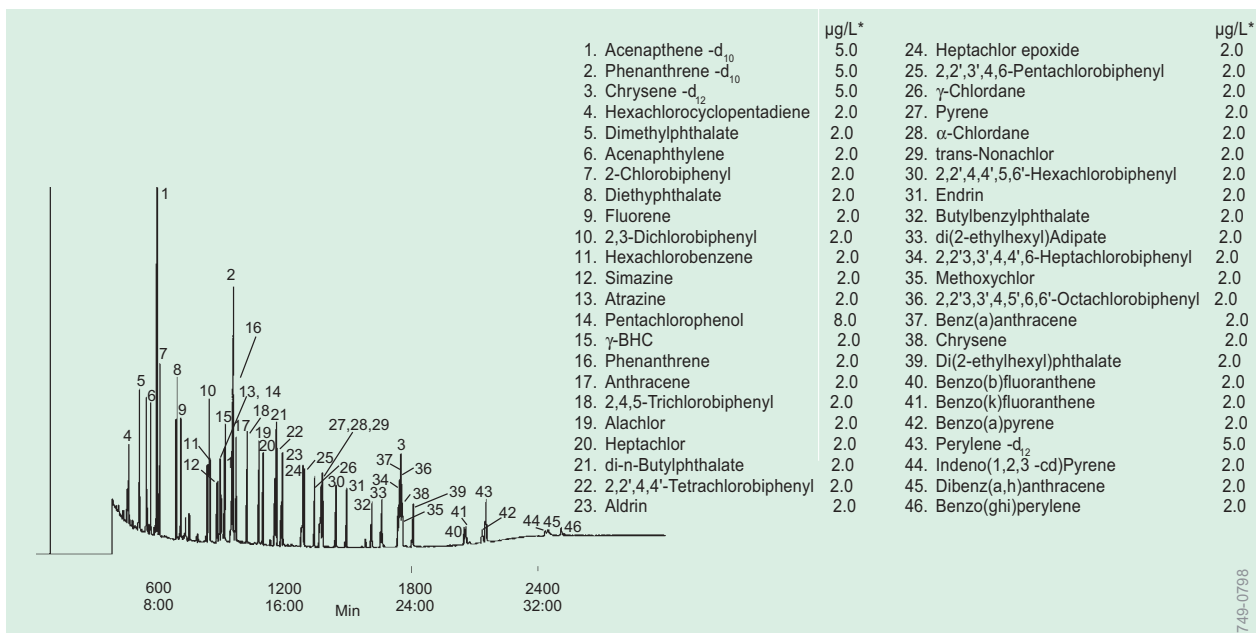
Sample Preparation Applications

Polynuclear Aromatic Hydrocarbons, Semivolatiles



PAHs in Water (SPE/GC)

Sample: water spiked with PAHs
 Extraction Tube: Supelclean ENVI-18, 500mg/6mL
 Cat. No.: 57064
 Conditioning: 2 x 6mL toluene:methanol (10:1), 6mL methanol, 6mL deionized water
 Sample Addition: 250mL, 10mL/min
 Drying: 10 min
 Elution: 2 x 1mL toluene:methanol (10:1)
 Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
 Cat. No.: 24135-U
 Oven: 70°C (2 min) to 280°C at 8°C/min
 Carrier: helium
 Det.: FID, 310°C
 Inj.: 1µL



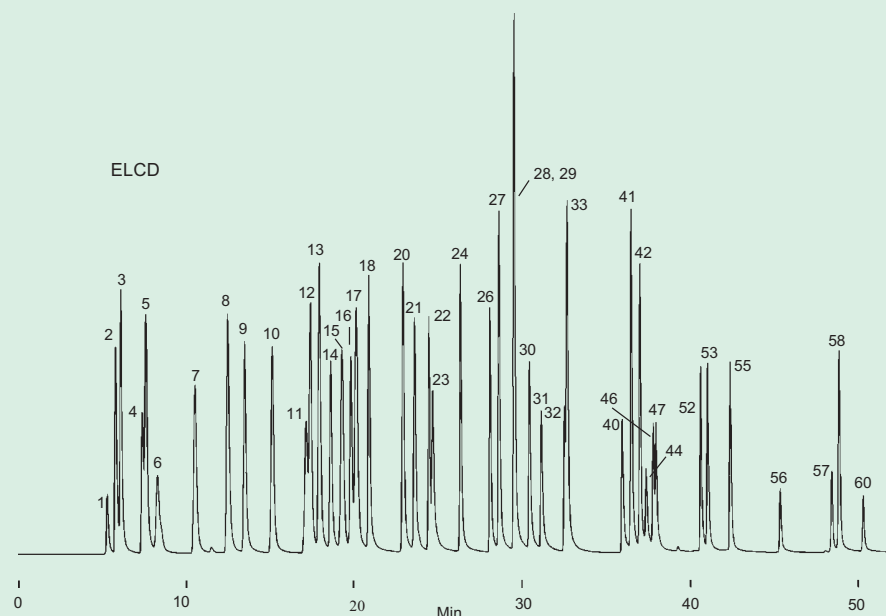
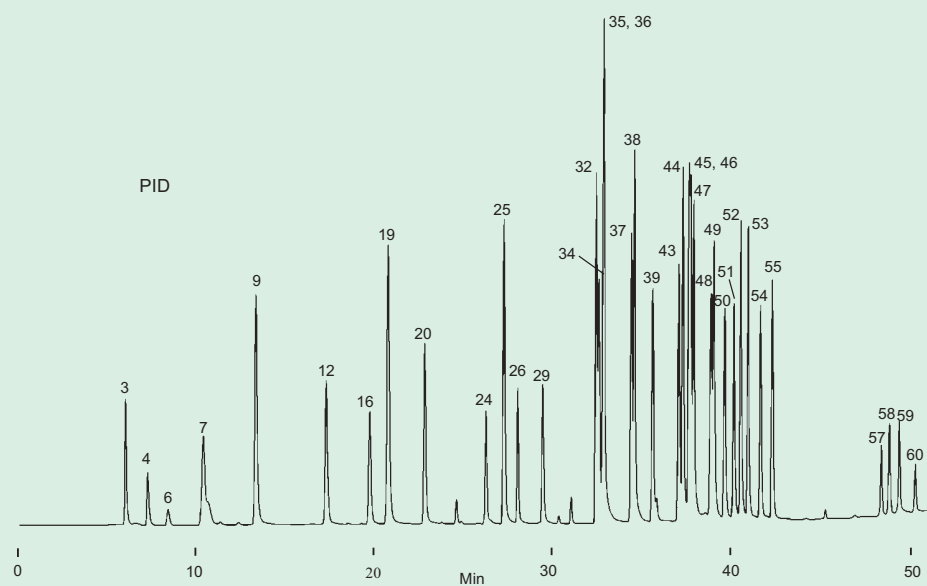
Semivolatiles (SPE/GC)

Sample: 1L drinking water, pH to <2 with 6N HCl, add 5mL methanol and mix thoroughly
 Extraction Disk: ENVI-18 DSK, 47mm
 Cat. No.: 57171
 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 2 x 5mL acetonitrile, extract disk with solution
 Column: 5% diphenyl/95% dimethyl silicone capillary, 30m x 0.25mm ID, 0.25µm film
 Oven: 40°C to 160°C (3 min), then to 300°C (3 min) at 6°C/min
 Supelco Equivalent: Equity-5 (Cat. No. 28089-U)
 Carrier: helium, 33cm/sec
 Det.: MS, scan range m/z=45-450
 Inj.: 1µL, split/splitless, 1min delay

Sample Preparation Applications

Volatiles

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



Chromatograms provided courtesy of O I Analytical, College Station, TX.

713-1165, 1166

Volatile Compounds by US EPA Method 502.2 (PT/GC)

Sample: 5mL water (5ppb each analyte)
 Instrument: OI Analytical Model 4560
 Trap: VOCARB 3000
 Cat. No.: 21131-U
 Purge: 11min, at 25°C, 37mL/min
 Desorption: 0.5min at 220°C

Column: VOCOL, 105m x 0.53mm ID, 3.0µm film
 Cat. No.: 25358
 Oven: 35°C (10 min) to 200°C at 4°C/min, hold
 Carrier: helium, 8.5mL/min
 Det.: ELCD (OI Model 5220)/PID(OI Model 5230) in series

Order: 1.800.325.3010 Technical Service: 1.800.359.3041 Web: www.sigma-aldrich.com/supelco

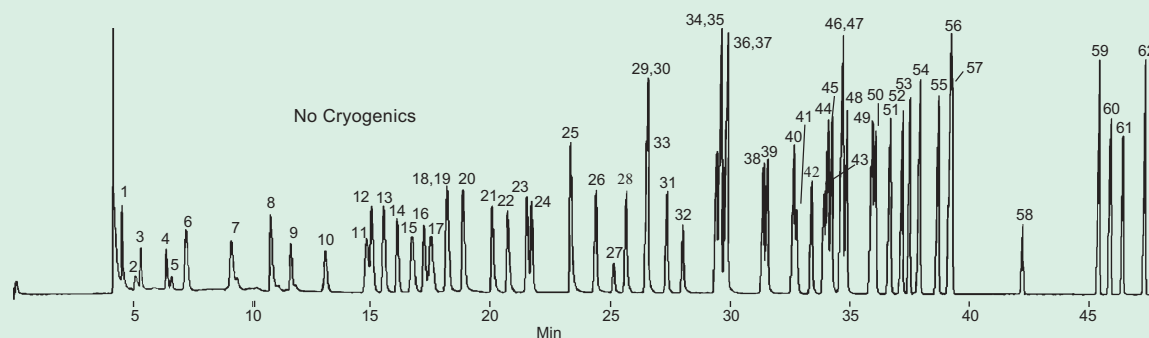
Sample
Preparation

SUPELCO

Sample Preparation Applications

Volatiles

- | | | | |
|-------------------------------|-------------------------------|-------------------------------|--|
| 1. Dichlorodifluoromethane | 17. Carbon tetrachloride | 33. Chlorobenzene | 49. tert-Butylbenzene |
| 2. Chloromethane | 18. 1,2-Dichloroethane | 34. 1,1,1,2-Tetrachloroethane | 50. 1,2,4-Trimethylbenzene |
| 3. Vinyl chloride | 19. Benzene | 35. Ethylbenzene | 51. sec-Butylbenzene |
| 4. Bromomethane | 20. Fluorobenzene (int std) | 36. m-Xylene | 52. p-Isopropyltoluene |
| 5. Chloroethane | 21. Trichloroethylene | 37. p-Xylene | 53. 1,3-Dichlorobenzene |
| 6. Trichlorofluoromethane | 22. 1,2-Dichloropropane | 38. o-Xylene | 54. 1,4-Dichlorobenzene |
| 7. 1,1-Dichloroethylene | 23. Bromodichloromethane | 39. Styrene | 55. n-Butylbenzene |
| 8. Methylene chloride | 24. Dibromomethane | 40. Isopropylbenzene | 56. 1,2-Dichlorobenzene-d ₄ (int std) |
| 9. trans-1,2-Dichloroethylene | 25. cis-1,3-Dichloropropene | 41. Bromoform | 57. 1,2-Dichlorobenzene |
| 10. 1,1-Dichloroethane | 26. Toluene | 42. 1,1,2,2-Tetrachloroethane | 58. 1,2-Dibromo-3-chloropropane |
| 11. 2,2-Dichloropropane | 27. trans-1,3-Dichloropropene | 43. 1,2,3-Trichloropropane | 59. 1,2,4-Trichlorobenzene |
| 12. cis-1,2-Dichloroethylene | 28. 1,1,2-Trichloroethane | 44. n-Propylbenzene | 60. Hexachlorobutadiene |
| 13. Chloroform | 29. 1,3-Dichloropropane | 45. Bromobenzene | 61. Naphthalene |
| 14. Bromochloromethane | 30. Tetrachloroethylene | 46. 1,3,5-Trimethylbenzene | 62. 1,2,3-Trichlorobenzene |
| 15. 1,1,1-Trichloroethane | 31. Chlorodibromomethane | 47. 2-Chlorotoluene | |
| 16. 1,1-Dichloropropene | 32. 1,2-Dibromoethane | 48. 4-Chlorotoluene | |



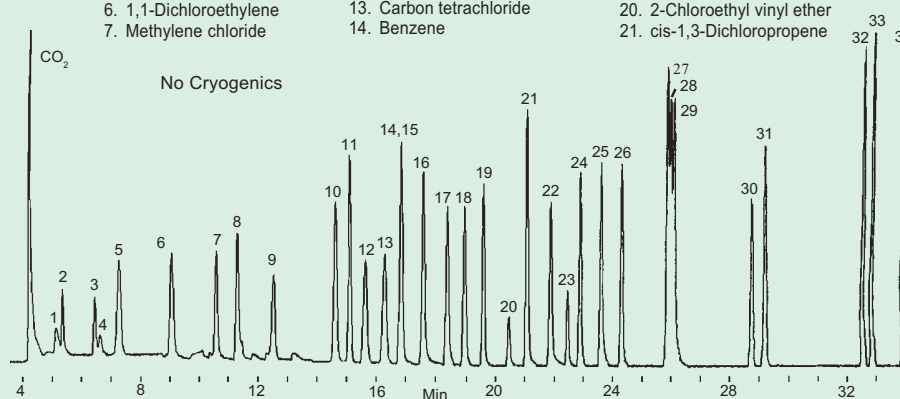
92-0134

Volatile Compounds by US EPA Method 524.2 (PT/GC)

Sample: 10ppb each component in 5mL water
 Trap: VOCARB 3000
 Cat. No.: 21066-U
 Purge: 11min, 40mL/min
 Dry: 3 min
 Desorb. Temp: 250°C for 4 min
 Bake: 280°C for 10 min

Column: VOCOL, 105m x 0.53mm ID, 3.0µm film
 Cat. No.: 25358
 Oven: 35°C (10 min) to 200°C at 4°C/min, hold 10 min
 Carrier: helium, 10mL/min
 Det.: MS, Scan Range m/z = 35-260 at 0.6 sec/scan

- | | | | |
|---------------------------|-------------------------------|-------------------------------|---------------------------------------|
| 1. Chloromethane | 8. trans-1,2-Dichloroethylene | 15. 1,2-Dichloroethane | 22. Toluene |
| 2. Vinyl chloride | 9. 1,1,-Dichloroethane | 16. Difluorobenzene (IS) | 23. trans-1,3-Dichloropropene |
| 3. Bromomethane | 10. Chloroform | 17. Trichloroethylene | 24. 1,1,2-Trichloroethane |
| 4. Chloroethane | 11. Bromochloromethane (IS) | 18. 1,2-Dichloropropane | 25. Tetrachloroethylene |
| 5. Trichlorofluoromethane | 12. 1,1,1-Trichloroethane | 19. Bromodichloromethane | 26. Chlorodibromomethane |
| 6. 1,1-Dichloroethylene | 13. Carbon tetrachloride | 20. 2-Chloroethyl vinyl ether | 27. Chlorobenzene-d ₆ (IS) |
| 7. Methylene chloride | 14. Benzene | 21. cis-1,3-Dichloropropene | 28. Chlorobenzene |



92-0010

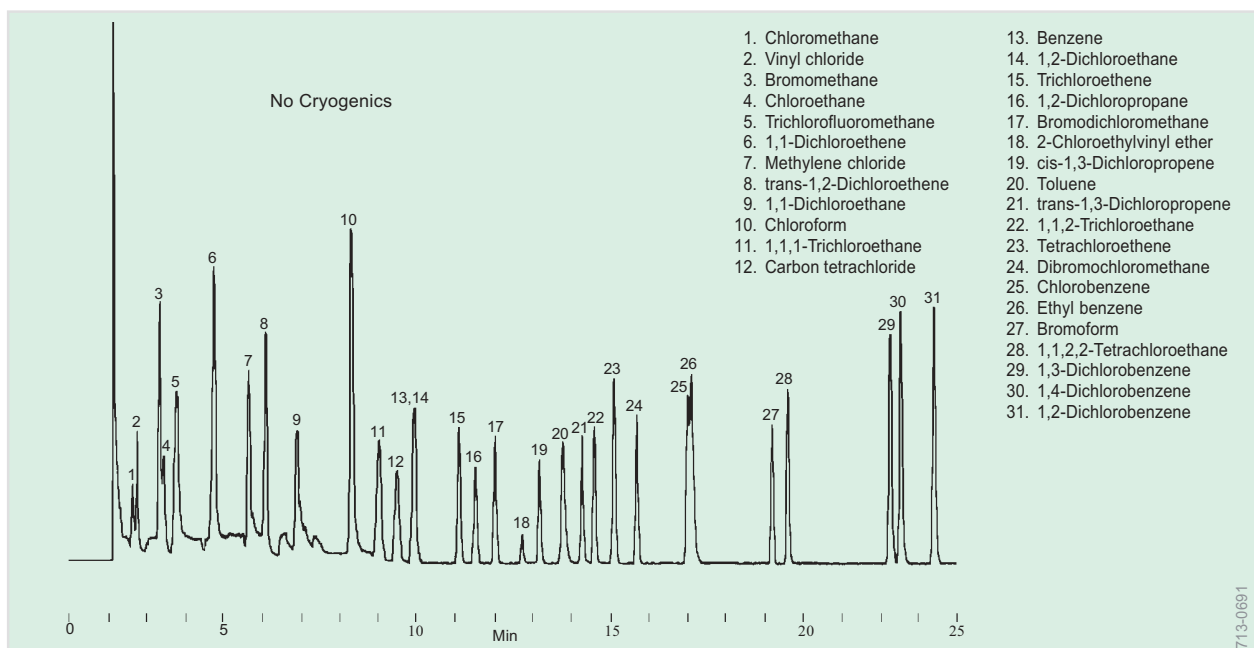
Volatile Compounds by US EPA Method 624 (PT/GC)

Sample: 20ppb each component in 5mL water
 Trap: VOCARB 3000
 Cat. No.: 21066-U
 Purge: 11 min, 35mL/min
 Dry Purge: 3 min
 Desorb. Temp: 250°C for 4 min

Bake: 60°C for 10 min
 Column: VOCOL, 105m x 0.53mm ID, 3.0µm film
 Cat. No.: 25358
 Oven: 35°C (4 min) to 200°C at 6°C/min
 Carrier: helium, 7.5mL/min
 Det.: MS, Scan Range m/z = 35-260 at 0.6 sec/scan

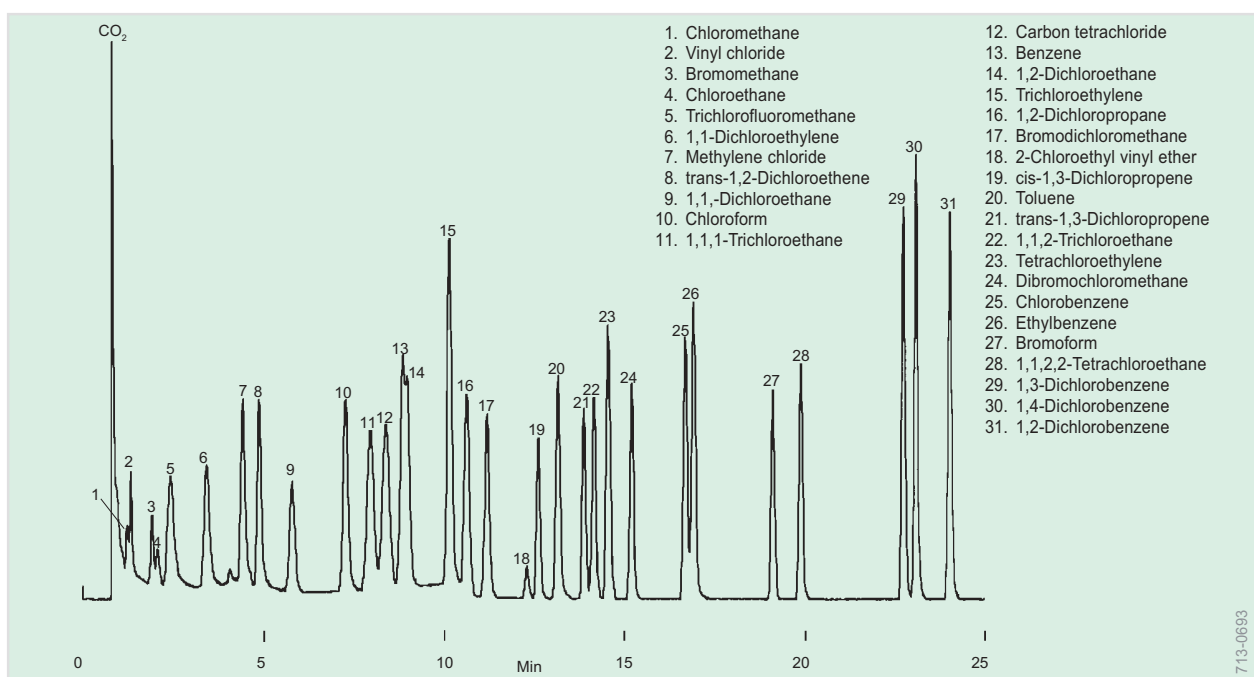
Sample Preparation Applications

Volatiles



Volatile Compounds by US EPA Method 624 (PT/GC)

Sample: 20ppb each component in 5mL water	Column: VOCOL, 60m x 0.53mm ID, 3.0µm film
Trap: VOCARB 3000	Cat. No.: 25381
Cat. No.: 21066-U	Oven: 35°C (4 min) to 200°C at 6°C/min
Purge: 11min, 35mL/min	Carrier: helium, 7.5mL/min
Dry Purge: 3 min	Det.: MS, Scan Range m/z = 35-260 at 0.6 sec/scan
Desorb.: 250°C for 4 min	
Bake: 260°C for 10 min	



Volatile Compounds by US EPA Method 624 (PT/GC)

Sample: 20ppb each component in 5mL water	Column: VOCOL, 30m x 0.53mm ID, 3.0µm film
Trap: VOCARB 3000	Cat. No.: 25320-U
Cat. No.: 21066-U	Oven: 5°C (2 min) to 200°C at 5°C/min
Purge: 11min, 40mL/min	Carrier: helium, 7.5mL/min
Dry Purge: 3 min	Det.: MS, Scan Range m/z = 35-260 at 0.6 sec/scan
Desorb.: 250°C for 4 min	
Bake: 280°C for 10 min	