CAPILLARY COLUMN CHROMATOGRAPHIC CONDITIONS:

Instrument: Perkin-Elmer Model 800

Type Detector: Flame Ionization

Column: 0.02" X 100' SE-52 Silicone Gum Rubber Support Coated Open Tubular (S.C.O.T.)

Column Temperature: 200° to 260°C @ 2°/Min.

Detector Block Temperature: 260°C

Injection Port Temperature: 340°C

Sample Volume Injected: 0.010 ml. Split 1 to 30

Solvent: CS_2

Sample Concentration: 20%

Flow Rates

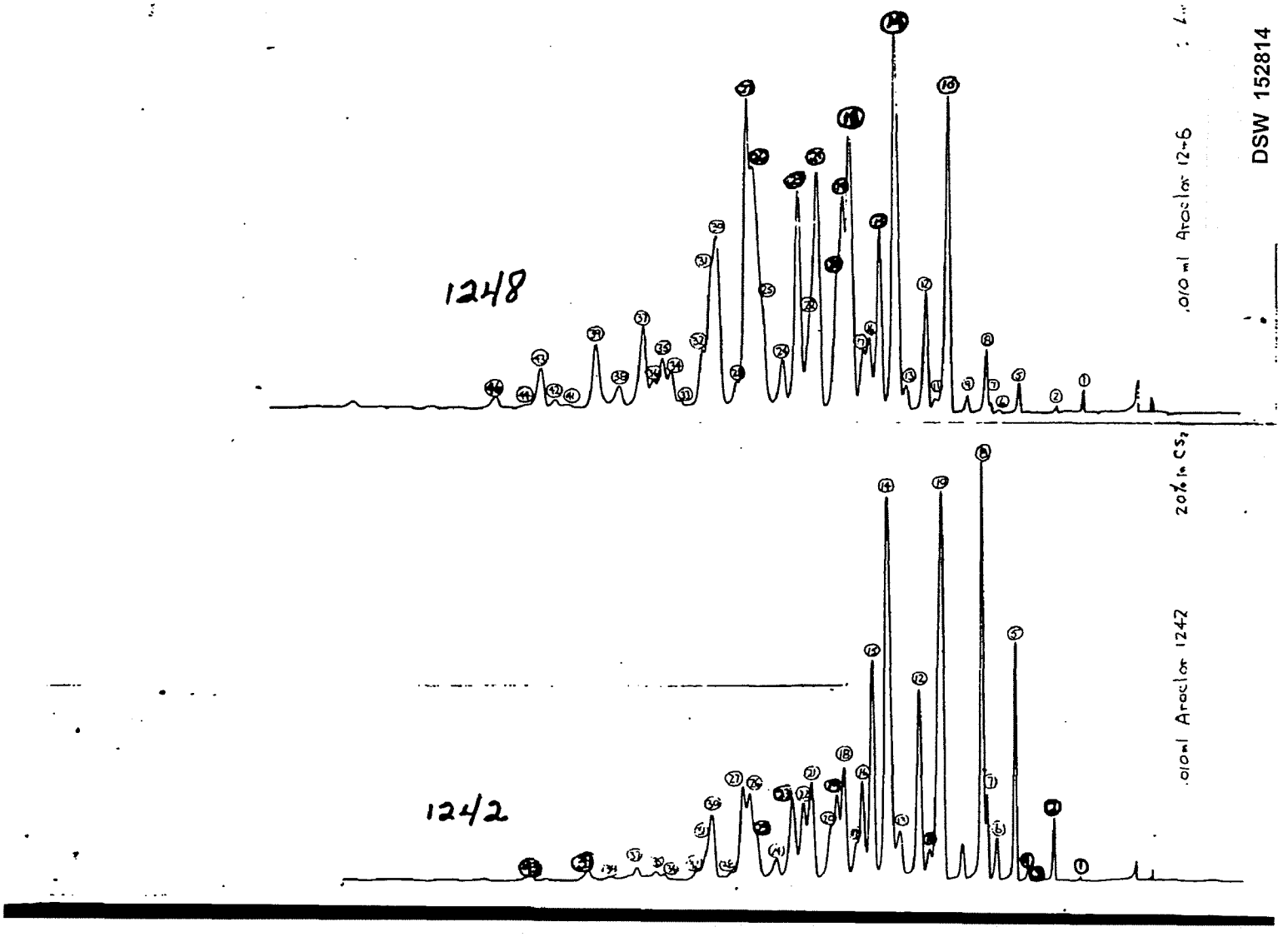
Nitrogen: 10 ml./min.

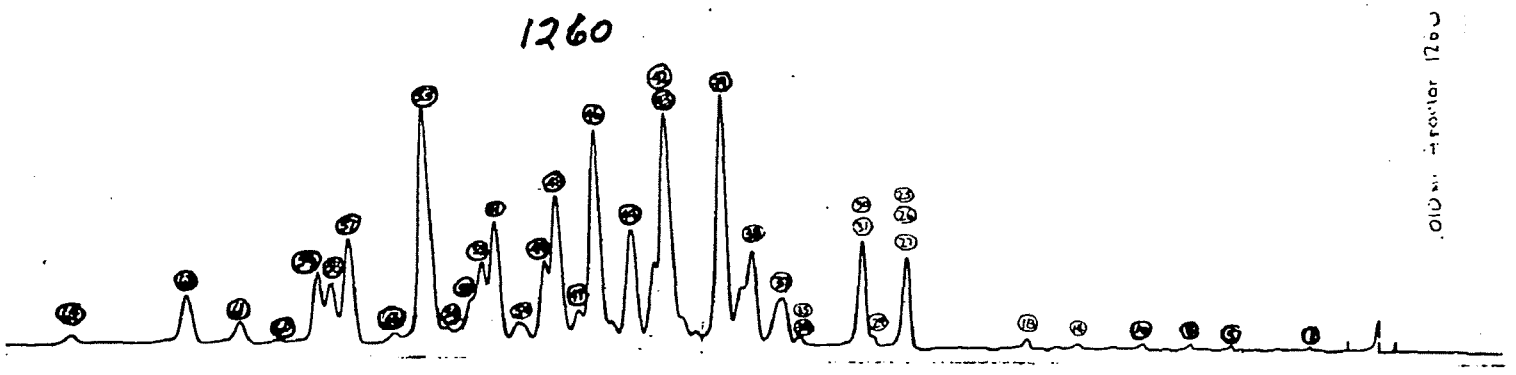
Auxiliary Gas: 10 ml./min.

Hydrogen: 30 ml./min.

Air: 400 ml./min.

DSW 152813

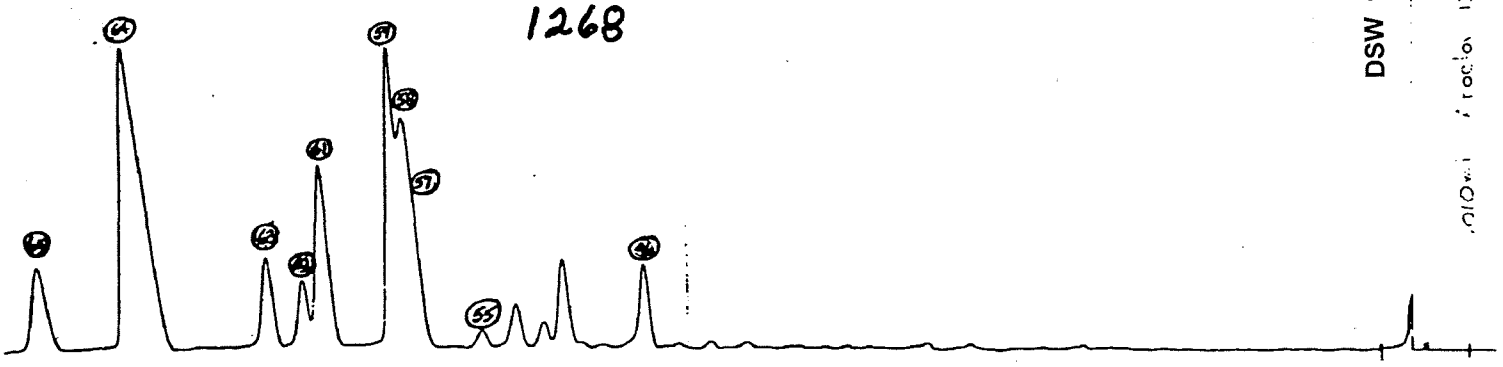

1248
.010 ml Aroclor 1248
DSW 152814
1242
.010ml Aroclor 1242
20% in CS2


1260

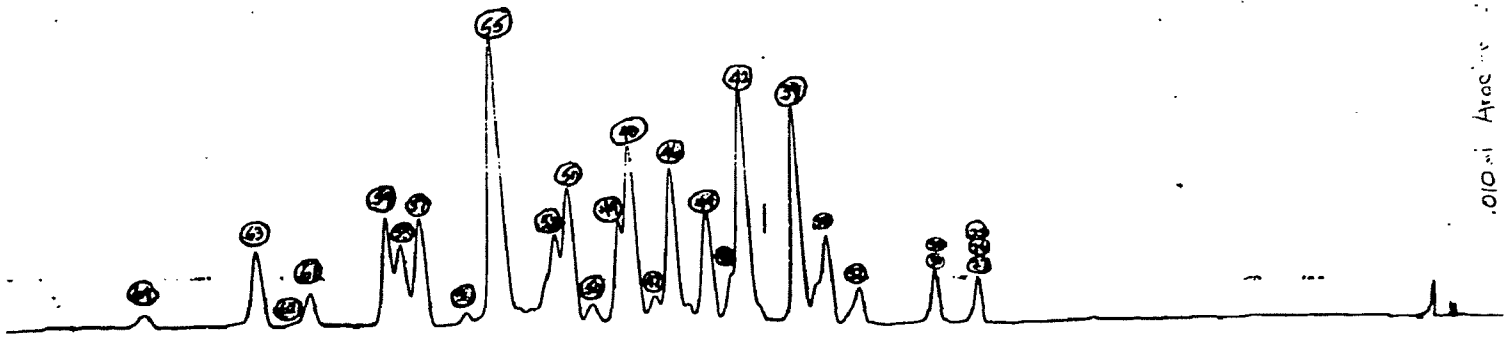

1254

DSW 152816

1268



1262



GENERAL ANALYTICAL OUTLINE

CONCENTRATION

Kundrna-Danish Evap. Conc.
Rotatory Evap. Conc.

Sample(s)	Extraction	Clean Up	Separation	Identification	Quantification
- Air - Water - Soil - Sediment - Fish, Fowl, & Animals Tissues & Excrement - Misc.	- Soxhelt - Wahring Blender - Virtis Homogenizer - Direct Solv. Extraction	-Polar/Non Polar Solvent Partition -Column Chromatography Alumina Silica -Chemical Oxodation → H_2SO_4/HNO_3 CR_2O_7/HOA_c Saponification → Alc - KOH Glycol - KOH	TLC GC	- Two Column GC-EC -DMC -FID - GC/Mass - Reaction/GC → - IR - S.C.O.T. Col. fingerprint (product ident.)	EC DMC FID IR

DSW 152817

STLCOPCB4040163

SUPPLEMENT TO GENERAL ANALYTICAL OUTLINE

WATER

(Measure)
Extract and Hexane
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

SEDIMENT

Dry Grind Measure
Extract with acetonitrile
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

TISSUES

Measure
Extract and/or Homogenize with Hexane
Polar/Non-polar Solvent Part.
Concentrate
Alumina Column
Concentrate
Check with EC/GC
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

4-18-69

DSW 152818

STLCOPCB4040164

DSW 152819

GENERAL ANALYTICAL OUTLINE

CONCENTRATION

Kundrna-Danish Evap. Conc.
Rotatory Evap. Conc.

Sample(s)

- Air
- Water
- Soil
- Sediment
- Fish, Fowl, & Animals Tissues & Excrement
- Misc.

Extraction

- Soxhelt
- Wahring Blender
- Virtis Homogenizer
- Direct Solv. Extraction

Clean Up

-Polar/Non Polar Solvent Partition

-Column Chromatography
- Alumina
- Silica

-Chemical Oxodation →
- H_2SO_4/HNO_3
- CR_2O_7/HOA_c

Saponification →
- Alc - KOH
- Glycol - KOH

Separation

- TLC
- GC

Identification

- Two Column GC-EC -DMC -FID
- GC/Mass
- Reaction/GC →
- IR
- S.C.O.T. Col. fingerprint (product ident.)

Quantification

- EC
- DMC
- FID
- IR

STLCOPCB4040165

SUPPLEMENT TO GENERAL ANALYTICAL OUTLINE

WATER

(Measure)
Extract and Hexane
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

SEDIMENT

Dry Grind Measure
Extract with acetonitrile
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

TISSUES

Measure
Extract and/or Homogenize with Hexane
Polar/Non-polar Solvent Part.
Concentrate
Alumina Column
Concentrate
Check with EC/GC
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

4-18-69

DSW 152820

STLCOPCB4040166

RELATIVE RETENTION TIMES OF AROCLORS AND SOME PESTICIDES ON QF-1 FLUOROSILICONE COLUMN (p,p' DDE = 8.1 Min. = 100)

Instrument: F&M Model 402 High Efficiency Gas Chromatograph

Type of Detector: Electron Capture - high temperature
Ni^{63} cell (pulsed voltage type)
glass U shaped

Column: 6 MM (OD) X 6 M 6% QF-1 on 80/100 Mesh Chromosorb W

Column Temperature: 190°C

Detector Temperature: 250°C

Injection Port Temperature: 225°C

Sample Volume: 1-5 µl

Flow Rates

Helium (carrier gas) 60 ml/min.
Argon Methane (purge gas) 185 ml/min.

. . . . . .

Aroclor 1242: 9, 13, 18, 20, 24, 28, 33, 38, 42, 45, 54, 65, 71, 82, 95, 103, 123, 153.

Aroclor 1248: 24, 29, 33, 39, 43, 46, 55, 66, 72, 80, 83, 96, 104, 125, 154, 164, 206, 252, 314.

Aroclor 1254: 23, 28, 45, 54, 71, 77, 94, 103, 125, 132, 145, 154, 164, 206, 250, 314.

Aroclor 1260: 24, 29, 38, 46, 66, 77, 90, 109, 133, 145, 165, 202, 252, 311, 381, 476.

Aroclor 1262: 66, 78, 90, 100, 110, 133, 145, 166, 202, 253, 310, 381, 478, 531.

Aroclor 1268: 73, 131, 165, 180, 228, 251, 300, 317, 342, 474, 541.

. . . . . .

1) Lindane - 41
2) Heptachler - 45
3) Aldrin - 51
4) pp' DDE - 100
5) pp' DDD - 120
6) op' DDT - 127
7) Dieldrin - 142
8,9) Endrin, pp' DDD - 170
10) Methoxychlor - 292

4-18-69

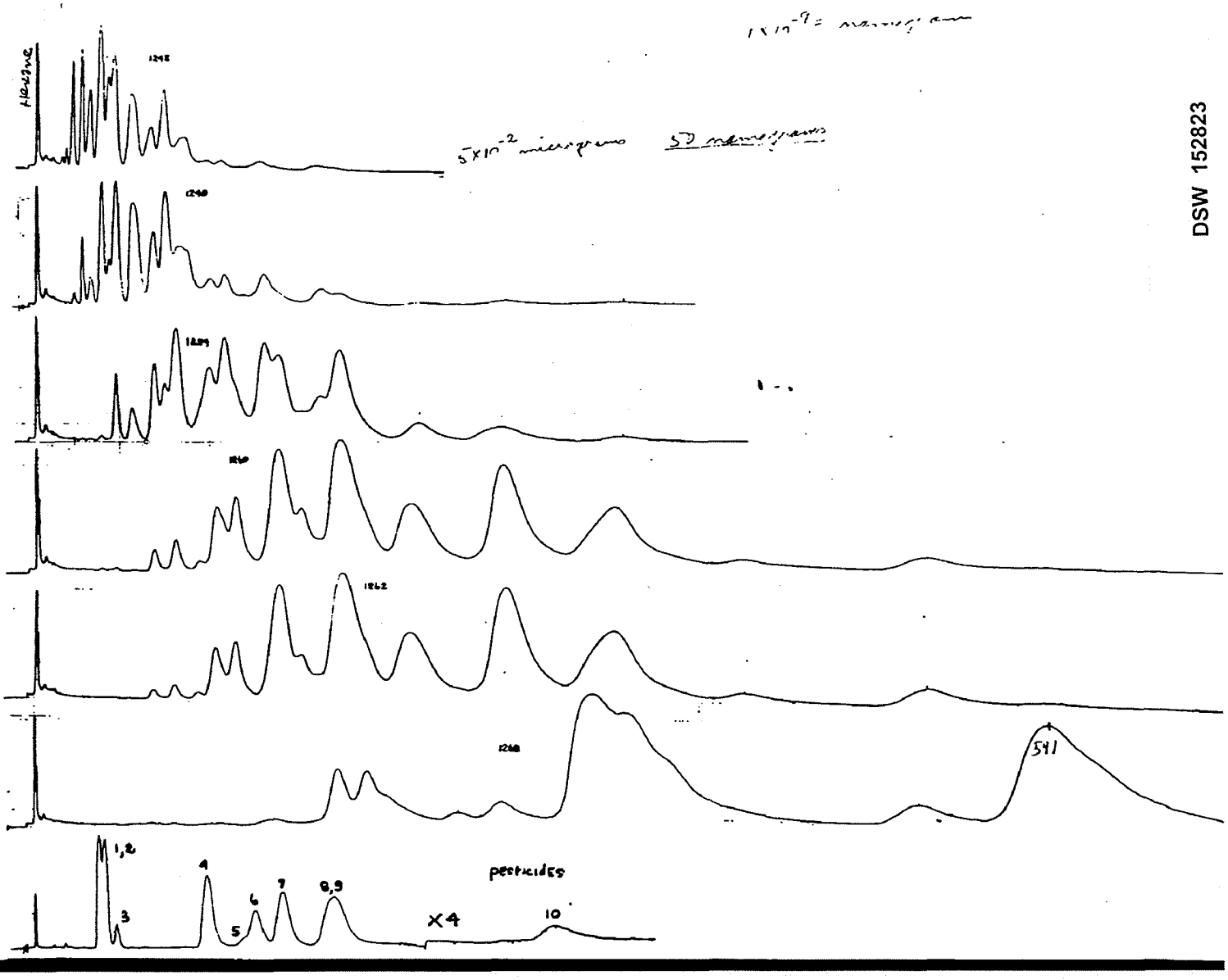
DSW 152821

AROCLORS	g X 10^{-8}
1242	5.18
1248	5.11
1254	5.11
1260	5.35
1262	5.03
1268	5.00

	PESTICIDES	g X 10^{-9}
8	Endrin	5.13
2	Heptachlor	5.22
1	Lindane	5.50
10	Methoxychlor	5.17
6	o,p DDT	5.46
4	pp' DDE	5.18
5 & 9	pp' DDD	5.63
3	Aldrin	5.08
7	Dieldrin	5.04

4-18-69

DSW 152822


Hexane
1242
1248
1254
1260
1262
1268
541
1,2
3
4
5
6
7
8,9
pesticides
X4
10

DSW 152823

GENERAL ANALYTICAL OUTLINE

CONCENTRATION

Kundrna-Danish Evap. Conc.
Rotatory Evap. Conc.

Sample(s)	Extraction	Clean Up	Separation	Identification	Quantification
- Air - Water - Soil - Sediment - Fish, Fowl, & Animals Tissues & Excrement - Misc.	- Soxhelt - Wahring Blender - Virtis Homogenizer - Direct Solv. Extraction	-Polar/Non Polar Solvent Partition -Column Chromatography Alumina Silica -Chemical Oxodation → H_2SO_4/HNO_3 CR_2O_7/HOA_c Saponification → Alc - KOH Glycol - KOH	TLC GC	- Two Column GC-EC -DMC -FID - GC/Mass - Reaction/GC → - IR - S.C.O.T. Col. fingerprint (product ident.)	EC DMC FID IR

DSW 152824

SUPPLEMENT TO GENERAL ANALYTICAL OUTLINE

WATER

(Measure)
Extract and Hexane
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

SEDIMENT

Dry Grind Measure
Extract with acetonitrile
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

TISSUES

Measure
Extract and/or Homogenize with Hexane
Polar/Non-polar Solvent Part.
Concentrate
Alumina Column
Concentrate
Check with EC/GC
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

4-18-69

DSW 152825

RELATIVE RETENTION TIMES OF AROCLORS AND SOME PESTICIDES ON QF-1 FLUOROSILICONE COLUMN (p,p' DDE = 8.1 Min. = 100)

Instrument: F&M Model 402 High Efficiency Gas Chromatograph

Type of Detector: Electron Capture - high temperature
Ni^{63} cell (pulsed voltage type)
glass U shaped

Column: 6 MM (OD) X 6 M 6% QF-1 on 80/100 Mesh Chromosorb W

Column Temperature: 190°C

Detector Temperature: 250°C

Injection Port Temperature: 225°C

Sample Volume: 1-5 µl

Flow Rates

Helium (carrier gas) 60 ml/min.
Argon Methane (purge gas) 185 ml/min.

. . . . . .

Aroclor 1242: 9, 13, 18, 20, 24, 28, 33, 38, 42, 45, 54, 65, 71, 82, 95, 103, 123, 153.

Aroclor 1248: 24, 29, 33, 39, 43, 46, 55, 66, 72, 80, 83, 96, 104, 125, 154, 164, 206, 252, 314.

Aroclor 1254: 23, 28, 45, 54, 71, 77, 94, 103, 125, 132, 145, 154, 164, 206, 250, 314.

Aroclor 1260: 24, 29, 38, 46, 66, 77, 90, 109, 133, 145, 165, 202, 252, 311, 381, 476.

Aroclor 1262: 66, 78, 90, 100, 110, 133, 145, 166, 202, 253, 310, 381, 478, 531.

Aroclor 1268: 73, 131, 165, 180, 228, 251, 300, 317, 342, 474, 541.

. . . . . .

1) Lindane - 41
2) Heptachler - 45
3) Aldrin - 51
4) pp' DDE - 100
5) pp' DDD - 120
6) op' DDT - 127
7) Dieldrin - 142
8,9) Endrin, pp' DDD - 170
10) Methoxychlor - 292

4-18-69

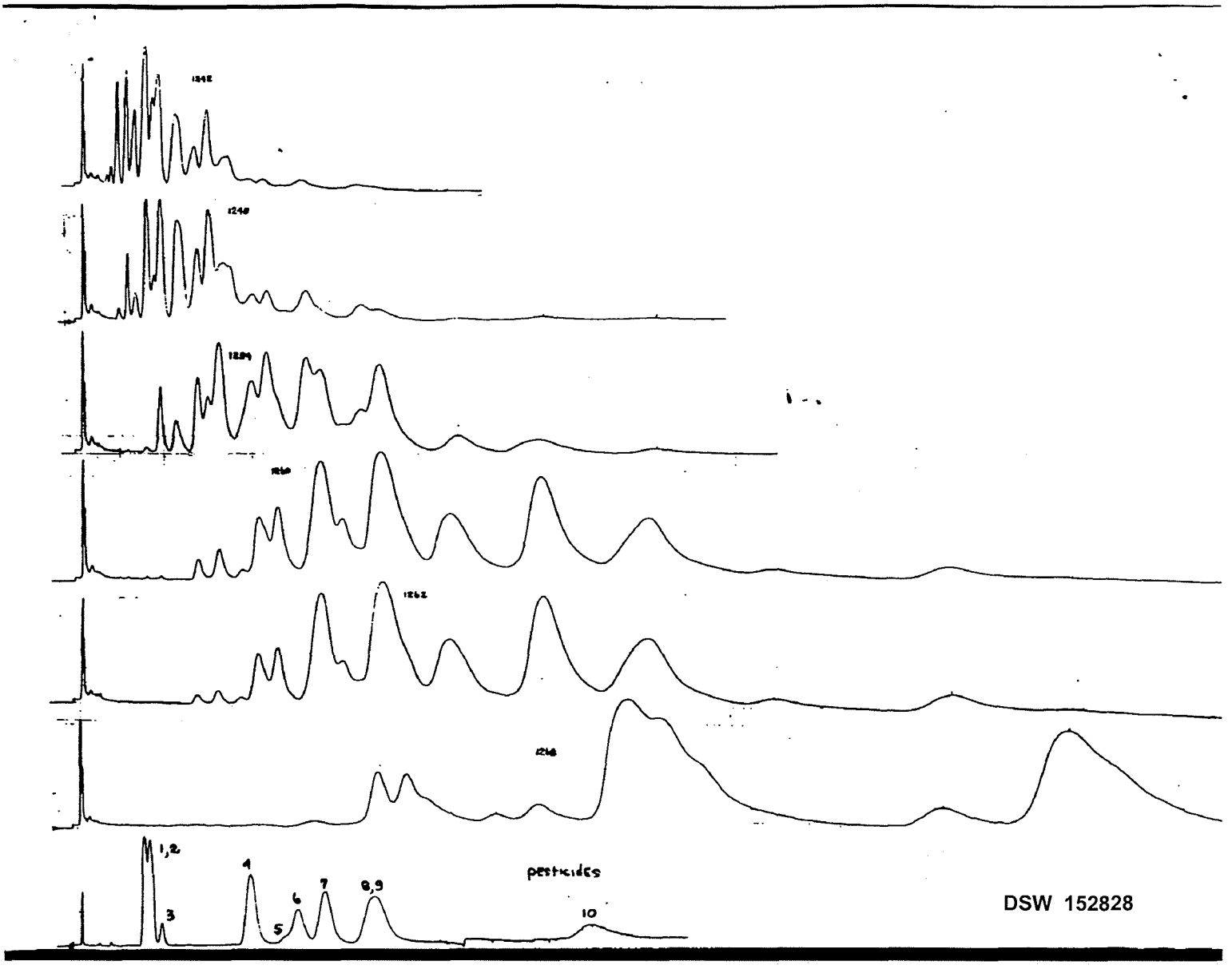
DSW 152826

AROCLORS	g X 10^{-8}
1242	5.18
1248	5.11
1254	5.11
1260	5.35
1262	5.03
1268	5.00

	PESTICIDES	g X 10^{-9}
8	Endrin	5.13
2	Heptachlor	5.22
1	Lindane	5.50
10	Methoxychlor	5.17
6	o,p DDT	5.46
4	pp' DDE	5.18
5 & 9	pp' DDD	5.63
3	Aldrin	5.08
7	Dieldrin	5.04

4-18-69

DSW 152827


1,2
3
4
5
6
7
8,9
pesticides
10
DSW 152828

DSW 152829

GENERAL ANALYTICAL OUTLINE

CONCENTRATION

Kundrna-Danish Evap. Conc.
Rotatory Evap. Conc.

Sample(s)

- Air
- Water
- Soil
- Sediment
- Fish, Fowl, & Animals Tissues & Excrement
- Misc.

Extraction

- Soxhelt
- Wahring Blender
- Virtis Homogenizer
- Direct Solv. Extraction

Clean Up

-Polar/Non Polar Solvent Partition

-Column Chromatography
Alumina
Silica

-Chemical Oxodation →
H_2SO_4/HNO_3
CR_2O_7/HOA_c

Saponification →
Alc - KOH
Glycol - KOH

Separation

TLC
GC

Identification

- Two Column GC-EC
  -DMC
  -FID
- GC/Mass
- Reaction/GC →
- IR
- S.C.O.T. Col. fingerprint (product ident.)

Quantification

EC
DMC
FID
IR

STLCOPCB4040175

SUPPLEMENT TO GENERAL ANALYTICAL OUTLINE

WATER

(Measure)
Extract and Hexane
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

SEDIMENT

Dry Grind Measure
Extract with acetonitrile
Concentrate
Check with EC/GC
Concentrate
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

TISSUES

Measure
Extract and/or Homogenize with Hexane
Polar/Non-polar Solvent Part.
Concentrate
Alumina Column
Concentrate
Check with EC/GC
TLC or Chemical Separation
Check with EC/GC
S.C.O.T. Col/FID fingerprint
GC/Mass

4-18-69

DSW 152830

RELATIVE RETENTION TIMES OF AROCLORS AND SOME PESTICIDES ON QF-1 FLUOROSILICONE COLUMN (p,p' DDE = 8.1 Min. = 100)

Instrument: F&M Model 402 High Efficiency Gas Chromatograph

Type of Detector: Electron Capture - high temperature
Ni^{63} cell (pulsed voltage type)
glass U shaped

Column: 6 MM (OD) X 6 M 6% QF-1 on 80/100 Mesh Chromosorb W

Column Temperature: 190°C

Detector Temperature: 250°C

Injection Port Temperature: 225°C

Sample Volume: 1-5 µl

Flow Rates

Helium (carrier gas) 60 ml/min.
Argon Methane (purge gas) 185 ml/min.

. . . . . .

Aroclor 1242: 9, 13, 18, 20, 24, 28, 33, 38, 42, 45, 54, 65, 71, 82, 95, 103, 123, 153.

Aroclor 1248: 24, 29, 33, 39, 43, 46, 55, 66, 72, 80, 83, 96, 104, 125, 154, 164, 206, 252, 314.

Aroclor 1254: 23, 28, 45, 54, 71, 77, 94, 103, 125, 132, 145, 154, 164, 206, 250, 314.

Aroclor 1260: 24, 29, 38, 46, 66, 77, 90, 109, 133, 145, 165, 202, 252, 311, 381, 476.

Aroclor 1262: 66, 78, 90, 100, 110, 133, 145, 166, 202, 253, 310, 381, 478, 531.

Aroclor 1268: 73, 131, 165, 180, 228, 251, 300, 317, 342, 474, 541.

. . . . . .

1) Lindane - 41
2) Heptachler - 45
3) Aldrin - 51
4) pp' DDE - 100
5) pp' DDD - 120
6) op' DDT - 127
7) Dieldrin - 142
8,9) Endrin, pp' DDD - 170
10) Methoxychlor - 292

4-18-69

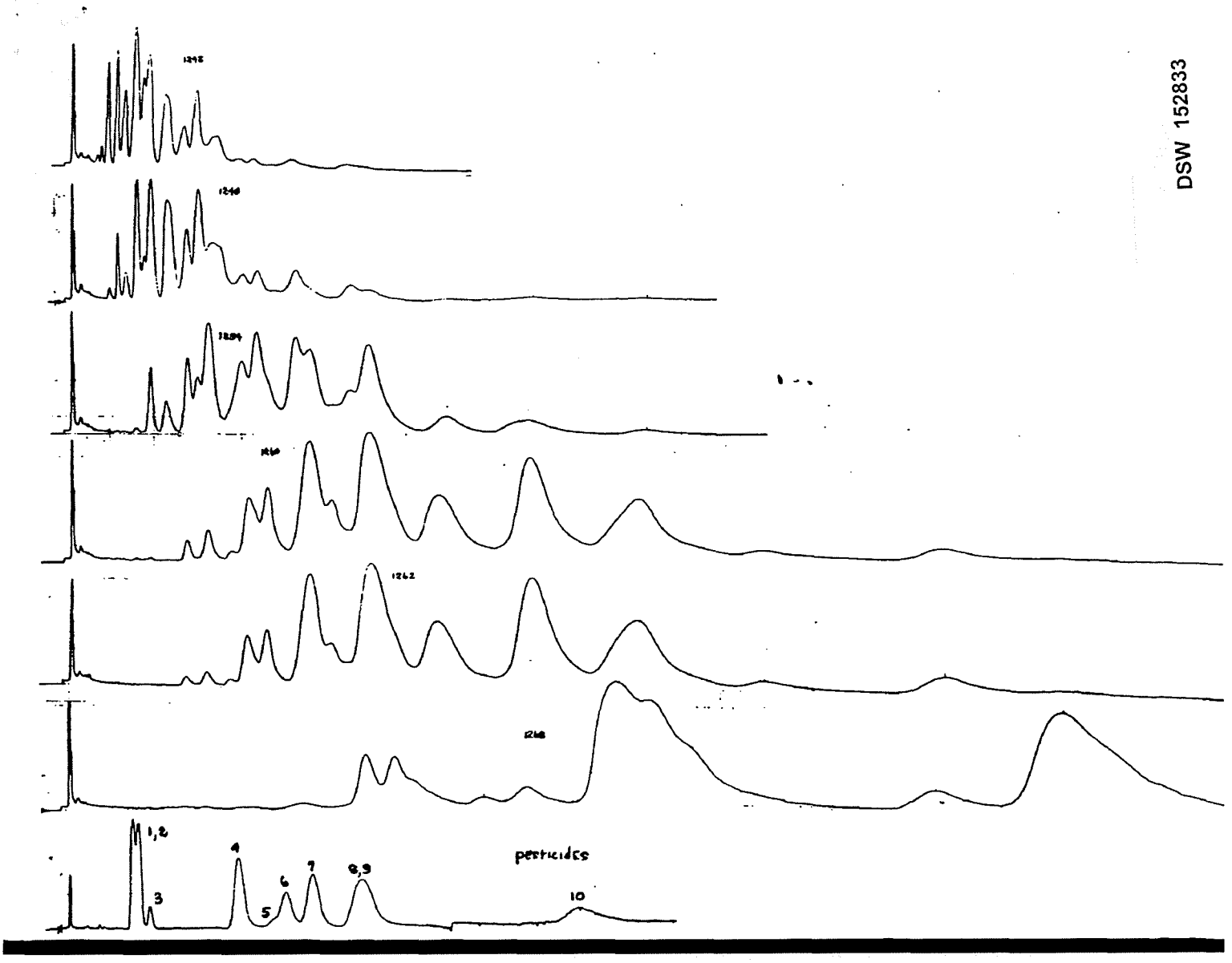
DSW 152831

AROCLORS	g X 10^{-8}
1242	5.18
1248	5.11
1254	5.11
1260	5.35
1262	5.03
1268	5.00

	PESTICIDES	g X 10^{-9}
8	Endrin	5.13
2	Heptachlor	5.22
1	Lindane	5.50
10	Methoxychlor	5.17
6	o,p DDT	5.46
4	pp' DDE	5.18
5 & 9	pp' DDD	5.63
3	Aldrin	5.08
7	Dieldrin	5.04

4-18-69

DSW 152832



DSW 152833