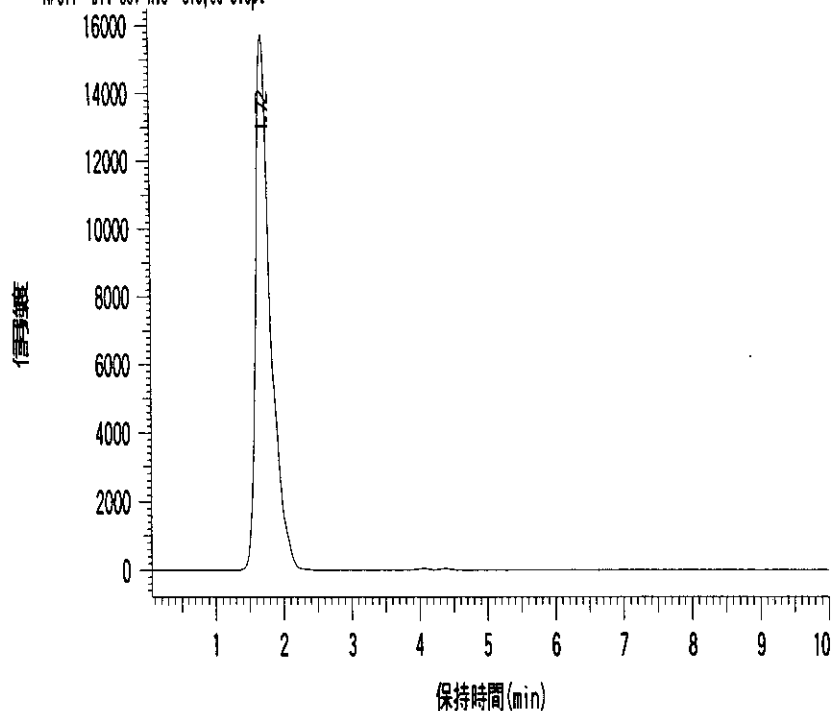


## 2. 4. 8 Thiramの検討結果

Ziram-APCI - 1390  $\mu$ g/mL 1 注入 1  $\mu$ L 100mg/L - スタンダードスキャン 241 - 03/01/21 02:54 午後

APCI+ DV: 30V N:5 G:5,60 S:3pt

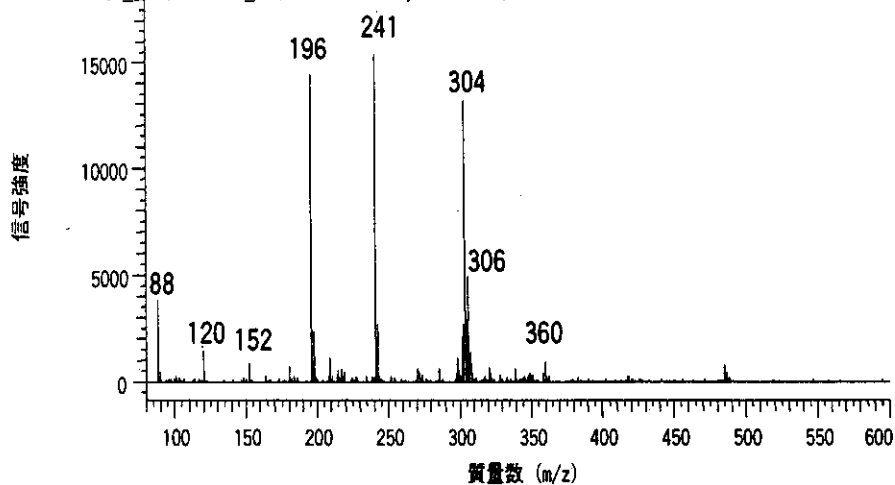


### 測定条件

サンプル	Thiram 100mg/L 20 $\mu$ L	イオン源	APCI正イオン検出
カラム	C6H12N2S4 MW240	ドリフト電圧	30V
溶離液	ODP-50 4.6mmIDx50mm	霧化器温度	180°C
流量	MeOH/H <sub>2</sub> O=80/20	脱溶媒室温度	350°C
UV検出	1mL/min	第一細孔温度	120°C
	230nm	質量数カットオフ	60

図2. 4. 16 Thiramのクロマトグラム

Ziram-APCI - 1390  $\mu$ g/mL 1 注入 1  $\mu$ L 100mg/L - スタンダードスキャン - 03/01/21 02:54 午後  
RT: 1.64\_1.80 - 1.09\_1.32 min BI: m/z 241 Int 15422 AT: 200 ms APCI+ DV: 30V



### 測定条件

サンプル	Thiram 100mg/L 20 $\mu$ L	イオン源	APCI正イオン検出
カラム	C6H12N2S4 MW 240	ドリフト電圧	30V
溶離液	ODP-50 4.6mmIDx50mm	霧化器温度	180°C
流量	MeOH/H <sub>2</sub> O=80/20	脱溶媒室温度	350°C
UV検出	1mL/min	第一細孔温度	120°C
	230nm	質量数カットオフ	60

図2. 4. 17 Thiramのマスペクトル

## 2.5 カーバメート系農薬の検討結果

### 2.5.1 Propargiteの検討結果

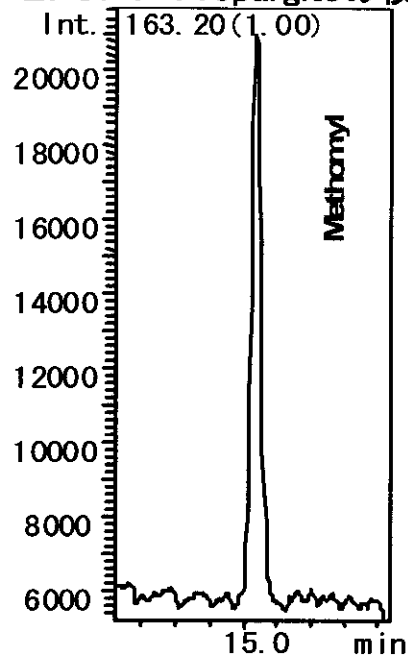


図2.5.1 Propargiteのクロマトグラム

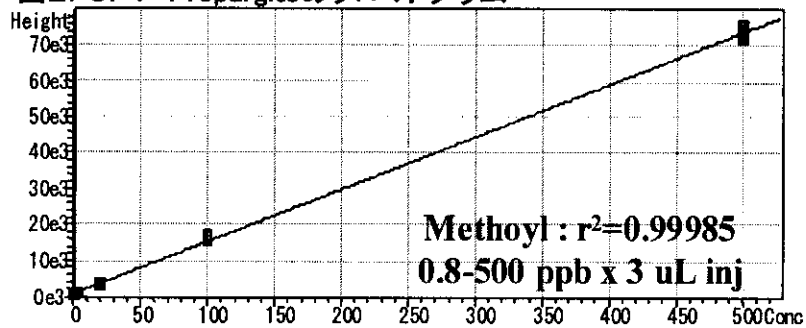


図2.5.2 Propargiteの検量線

表2.5.1 Propargiteの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 μm)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) → 95%B (40-50 min) → 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 °C
Sample store temp.	: 4 °C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 °C
Heat Block temp.	: 200 °C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 163(M+H)+

## 2. 5. 2 Carbarylの検討結果

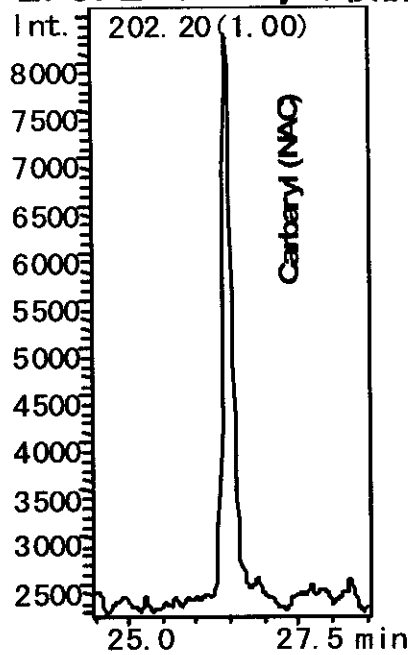


図2. 5. 3 Carbarylのクロマトグラム

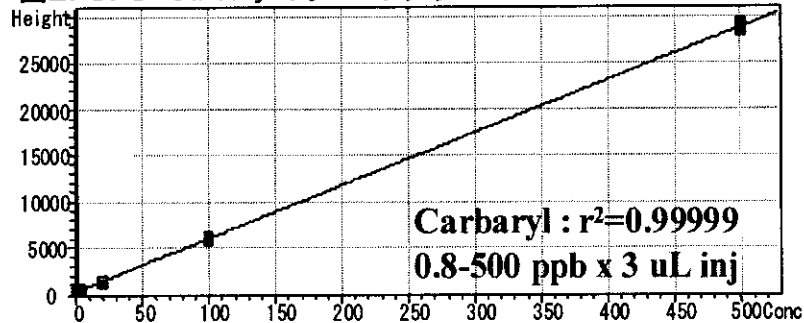


図2. 5. 4 Propargiteの検量線

表2. 5. 2 Carbarylの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 202(M+H) $^{+}$

### 2. 5. 3 Benfuracarbの検討結果

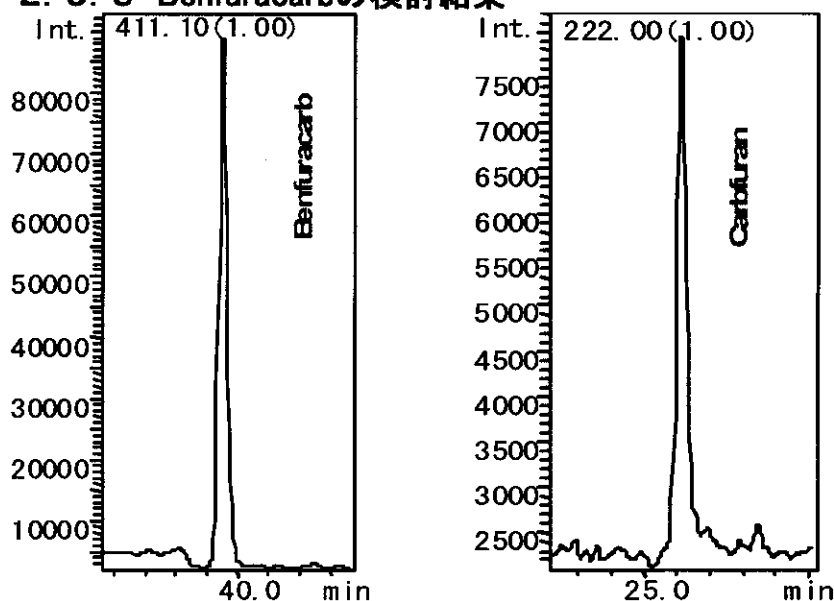


図2. 5. 5 BenfuracarbとCarbofuranのクロマトグラム

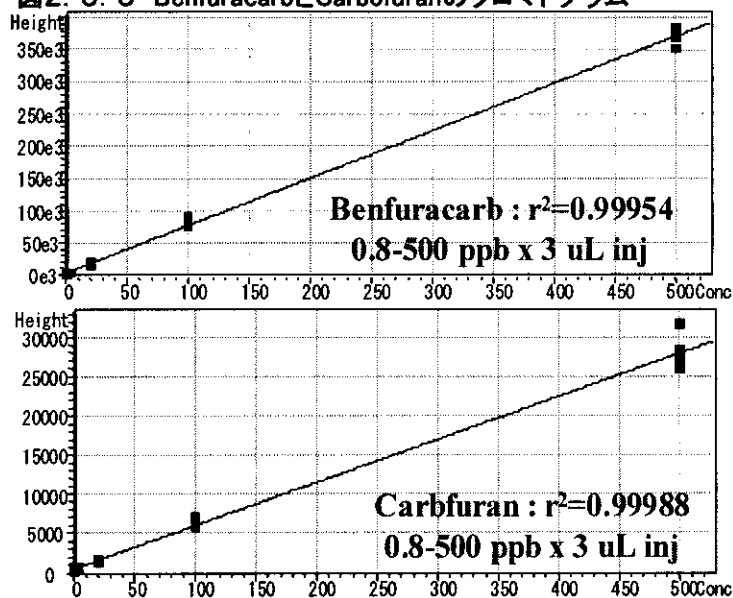


図2. 5. 6 BenfuracarbとCarbofuranの検量線

表2. 5. 3 BenfuracarbとCarbofuranの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 μm)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) → 95%B (40-50 min) → 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 °C
Sample store temp.	: 4 °C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 °C
Heat Block temp.	: 200 °C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 222(M+H) <sup>+</sup> , 411(M+H) <sup>+</sup>

## 2. 5. 4 Oxamylの検討結果

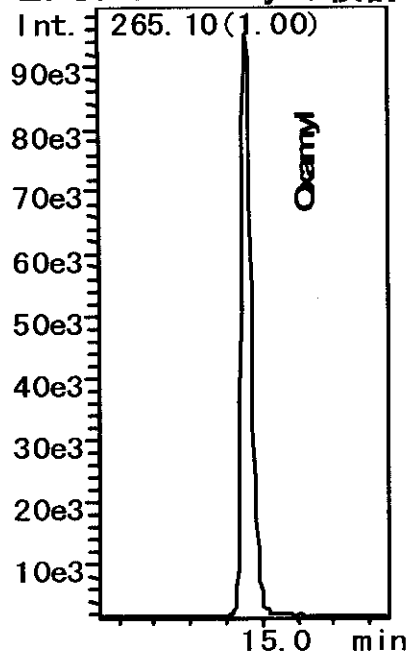


図2. 5. 7 Oxamylのクロマトグラム

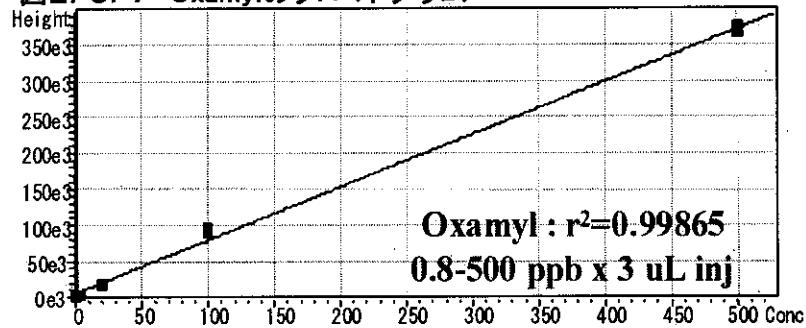


図2. 5. 8 Oxamylの検量線

表2. 5. 4 Oxamylの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 285(M+46) $^{+}$

## 2. 5. 5 Thiodicarbの検討結果

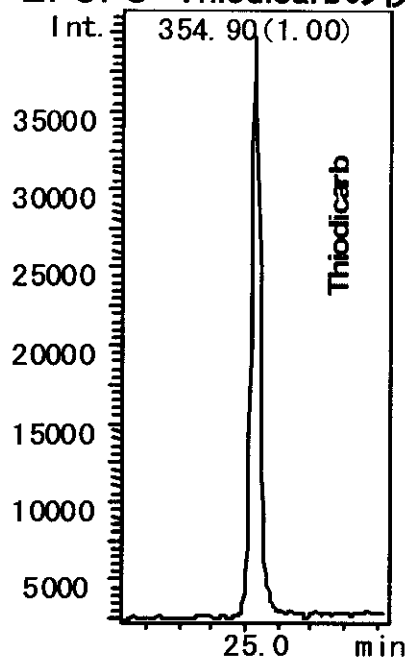


図2. 5. 9 Thiodicarbのクロマトグラム

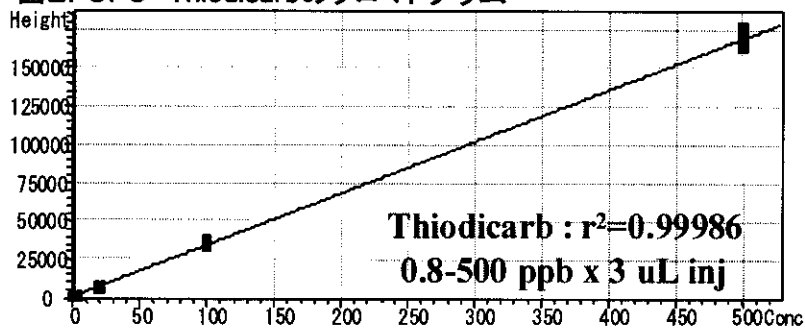


図2. 5. 10 Thiodicarbの検量線

表2. 5. 5 Thiodicarbの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 354.90

## 2. 5. 6 Isoprocarbの検討結果

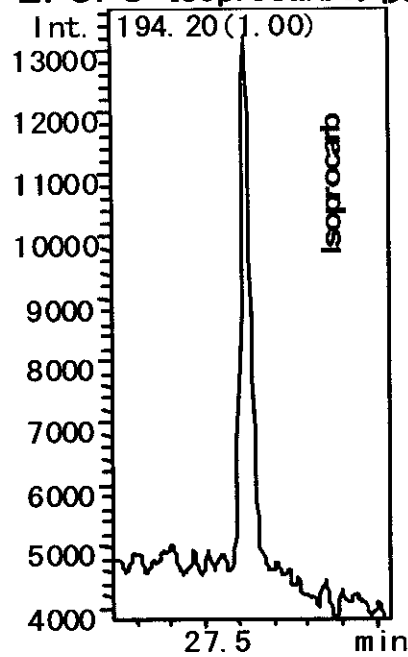


図2. 5. 11 Isoprocarbのクロマトグラム

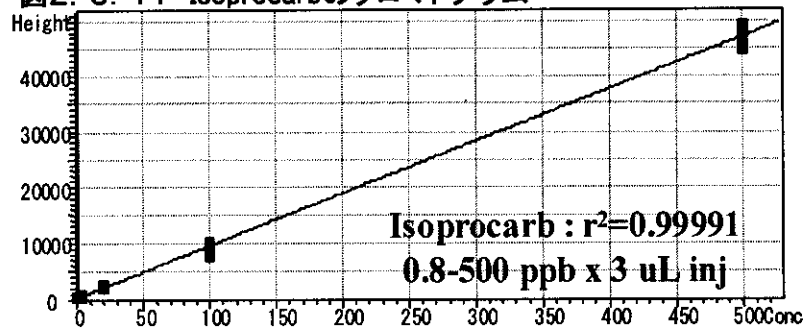


図2. 5. 12 Isoprocarbの検量線

### 表2. 5. 6 Isoprocarbの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 μm)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) → 95%B (40-50 min) → 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 °C
Sample store temp.	: 4 °C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 °C
Heat Block temp.	: 200 °C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 194(M+H) <sup>+</sup>

## 2. 5. 7 XMCの検討結果

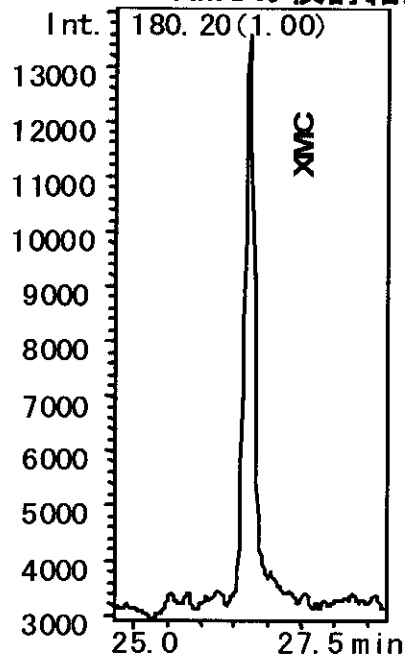


図2. 5. 13 XMCのクロマトグラム

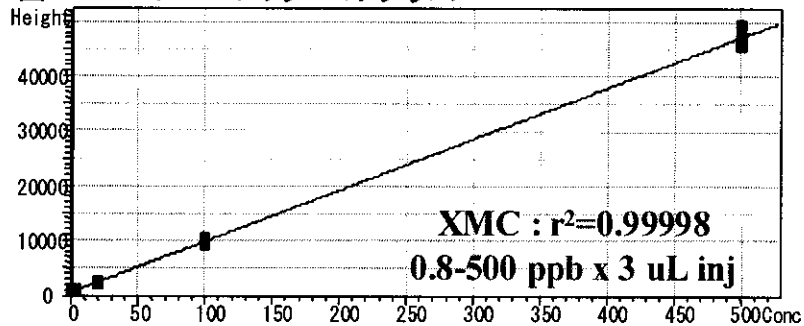


図2. 5. 14 XMCの検量線

### 表2. 5. 7 XMCの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 180(M+H) $^{+}$



## 2.5.8 Benomylの検討結果

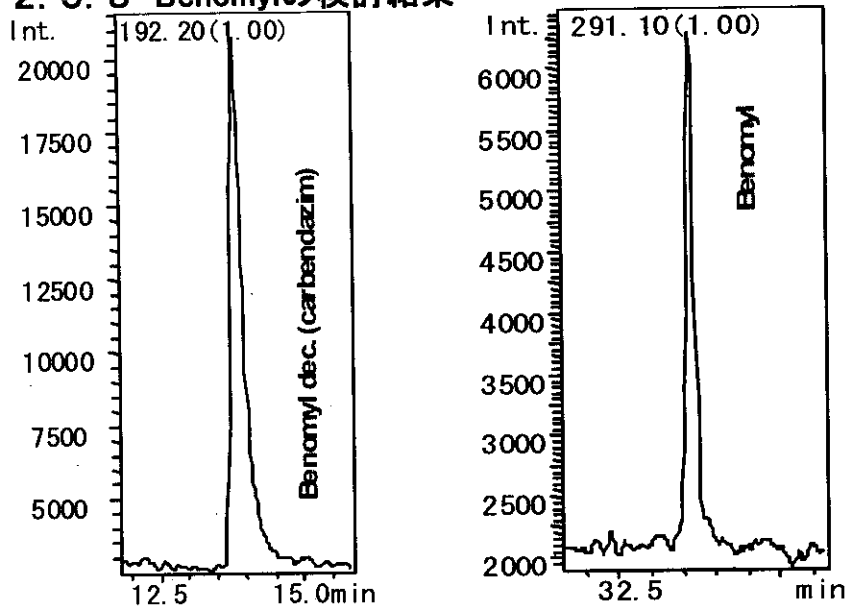


図2.5.15 BenomylとCarbedazimのクロマトグラム

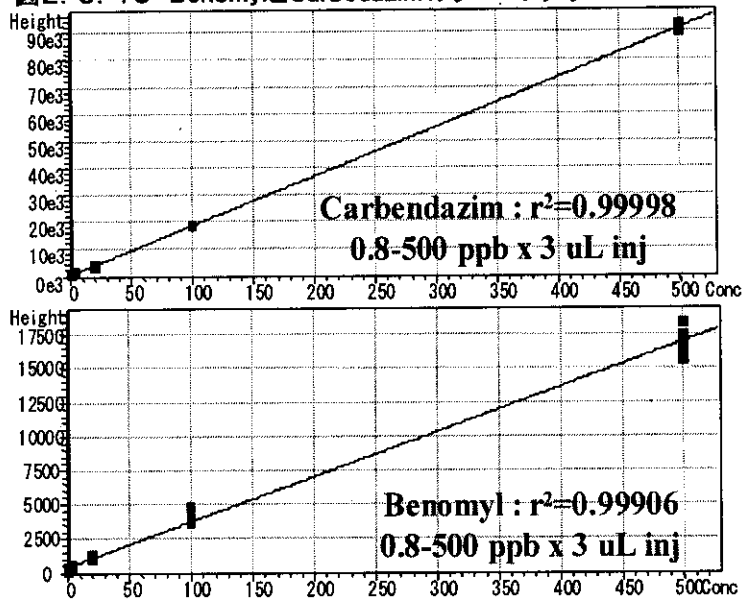


図2.5.16 BenomylとCarbedazimの検量線

表2.5.8 BenomylとCarbedazimの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 192(M+H) $^{+}$ , 291(M+H) $^{+}$

## 2. 5. 9 Thiophanate methylの検討結果

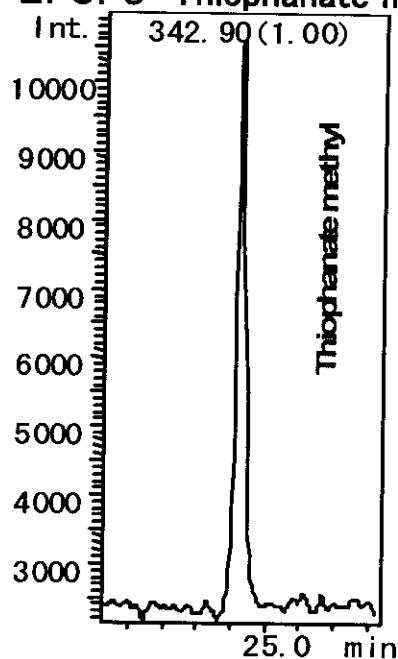


図2. 5. 17 Thiophanate methylのクロマトグラム

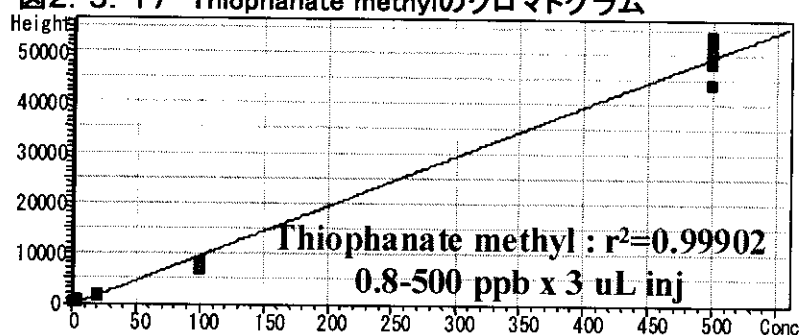


図2. 5. 18 Thiophanate methylの検量線

## 表2. 5. 9 Thiophanate methylの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 343(M+H) $^{+}$

## 2. 5. 10 Asulamの検討結果

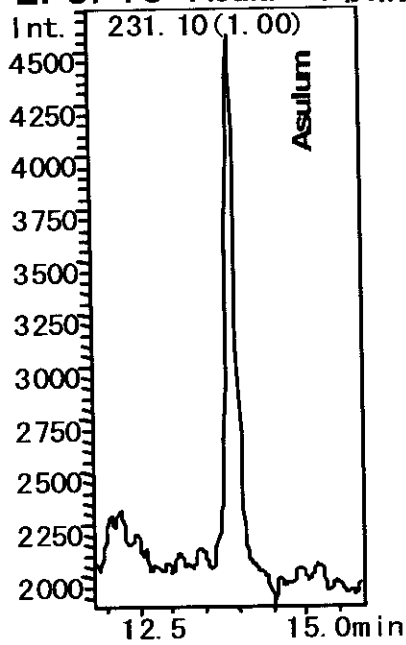


図2. 5. 19 Asulamのクロマトグラム

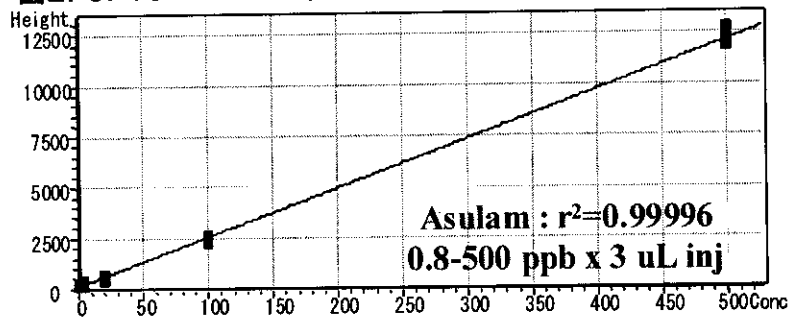


図2. 5. 20 Asulamの検量線

### 表2. 5. 10 Asulamの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 231(M+H) $^{+}$

## 2. 5. 11 Thiramの検討結果

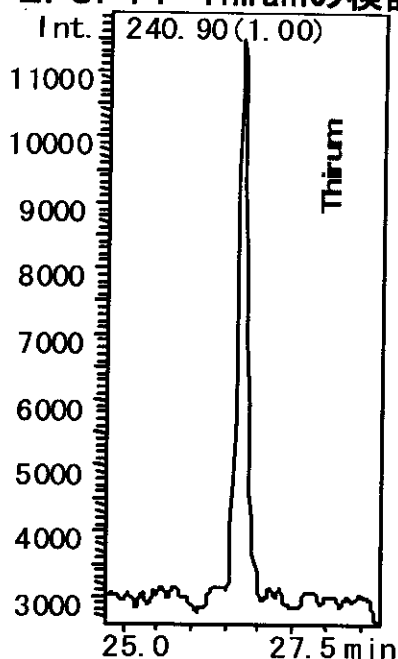


図2. 5. 21 Thiramのクロマトグラム

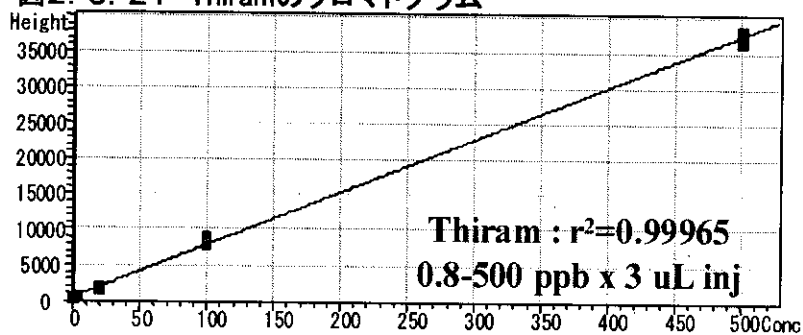


図2. 5. 22 Thiramの検量線

### 表2. 5. 11 Thiramの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 $\mu$ m)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) $\rightarrow$ 95%B (40-50 min) $\rightarrow$ 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 $^{\circ}$ C
Sample store temp.	: 4 $^{\circ}$ C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 $^{\circ}$ C
Heat Block temp.	: 200 $^{\circ}$ C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 241(M+H) $^{+}$

## 2. 5. 12 Dimepiperateの検討結果

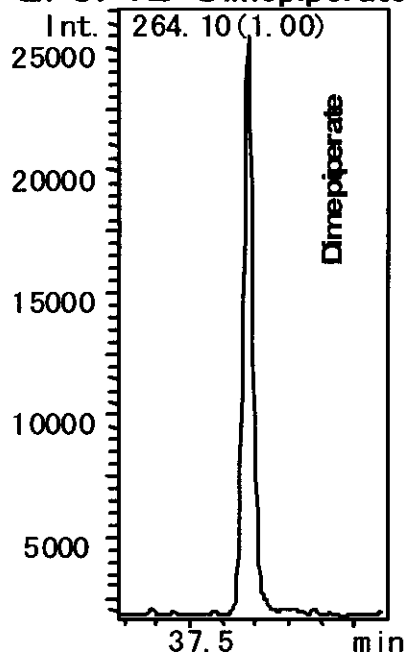


図2. 5. 23 Dimepiperateのクロマトグラム

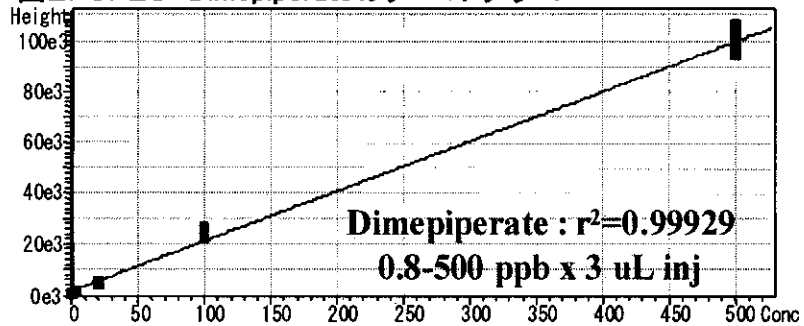


図2. 5. 24 Dimepiperateの検量線

### 表2. 5. 12 Dimepiperateの分析条件

Column	: Shim-pack FC-ODS (2.0 mmI.D. x 150 mmL, 3 μm)
Mobile phase A	: acetonitrile
Mobile phase B	: 0.1% formic acid-water
Gradient program	: 0%B (0 min) → 95%B (40-50 min) → 0%B (50.1-60 min)
Flow rate	: 0.2 mL/min
Column temp.	: 40 °C
Sample store temp.	: 4 °C
Ionization mode	: positive ESI
Applied voltage	: 4.5 kV
CDL temp.	: 200 °C
Heat Block temp.	: 200 °C
CDL voltage	: S-mode
Q-array voltage	: S-mode
Scan range	: m/z 50-600
m/z of selected ions	: 264(M+H) <sup>+</sup>

## 2.6 4級アンモニウム系農薬の検討結果

### 2.6.1 Diquatの検討結果

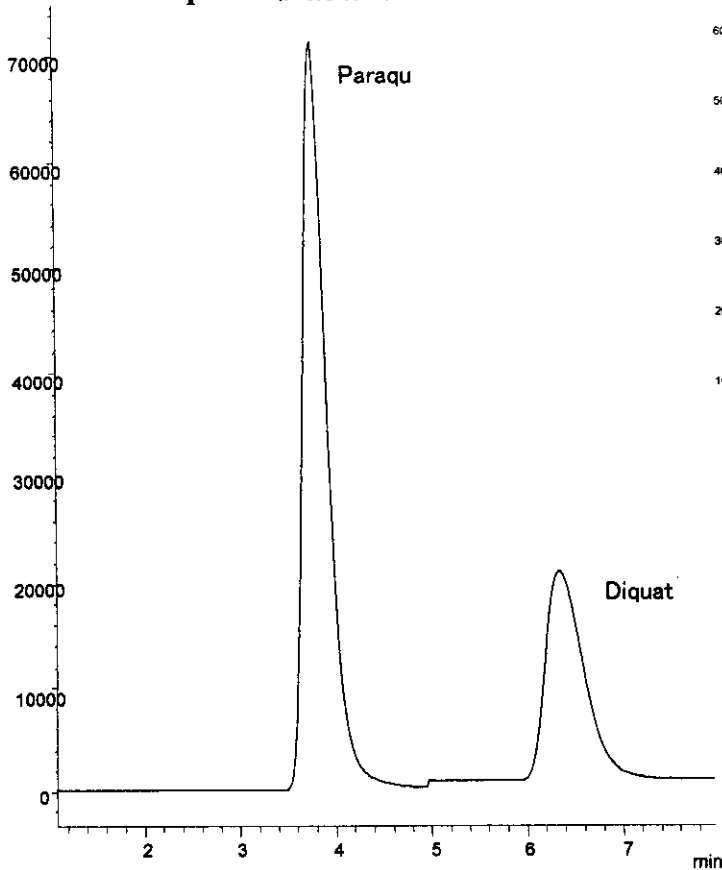


図2.6.1 Diquatのクロマトグラム

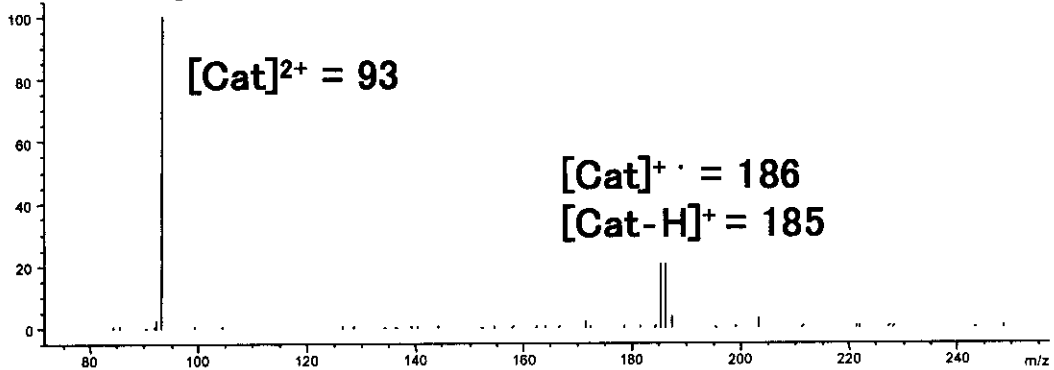


図2.6.2 Diquatのマスペクトル

表2.6.1 Diquatの分析条件

#### HPLC

Instrument Agilent 1100  
 Column TSK GEL SP NPR  
 i.d. 4.6 x 35 mm  
 Mobile phase A=80mM HCO<sub>2</sub>NH<sub>4</sub> /H<sub>2</sub>O  
 B=CH<sub>3</sub>CN  
 B 20% isocratic  
 Flow rate 1.2 mL/min.  
 Injection vol. SIM 100 μL

#### MS

Instrument Agilent 1100 LC/MSD SL  
 Ionization Electrospray  
 Fragmentor 100V  
 Nebulizer N<sub>2</sub> (60 psi)  
 Drying gas N<sub>2</sub> (13 L/min.)  
 Vcap 1500 V  
 Polarity Positive  
 SIM (m/z) 93 ; PQ

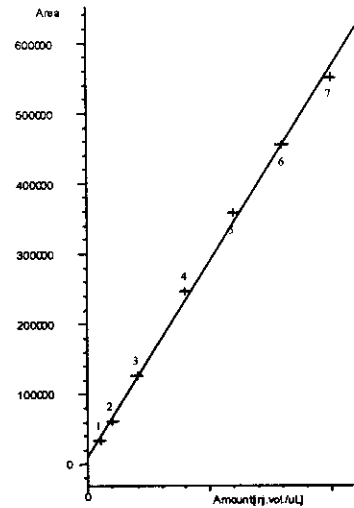


図2.6.3 Diquatの検量線

注入量-面積 (5~100μL)

相関係数: 0.99874

注入量-面積 (5~60μL)

相関係数: 0.99968

## 2. 6. 2 Paraquatの検討結果

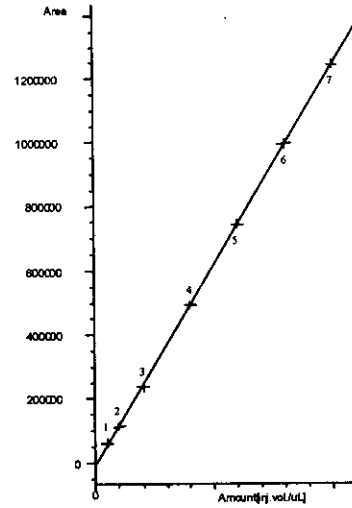
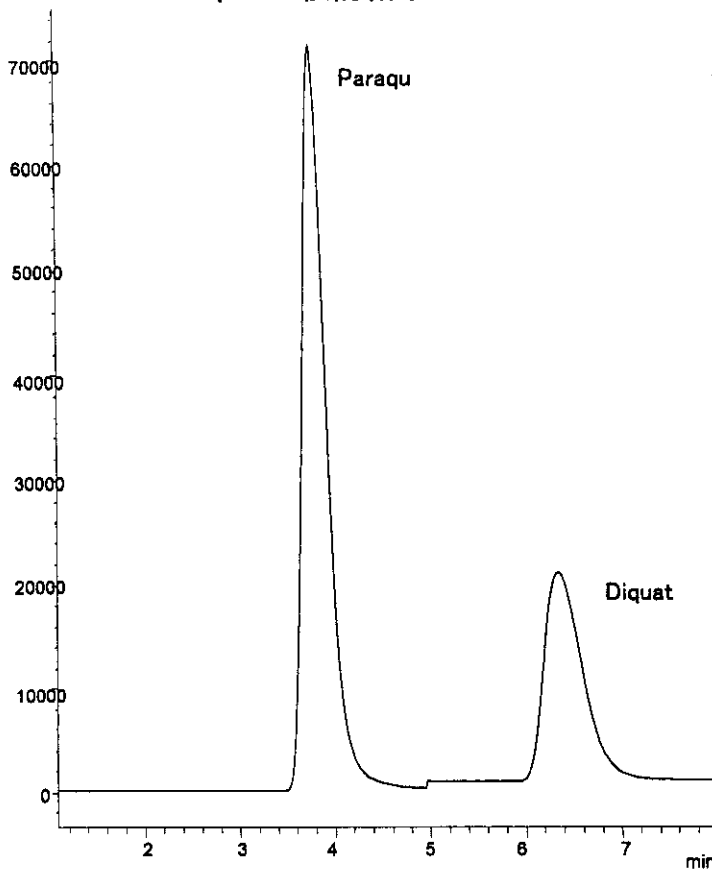


図2. 6. 6 Paraquatの検量線  
 注入量-面積(5~100µL)  
 相関係数:0.99994

図2. 6. 4 Paraquatのクロマトグラム

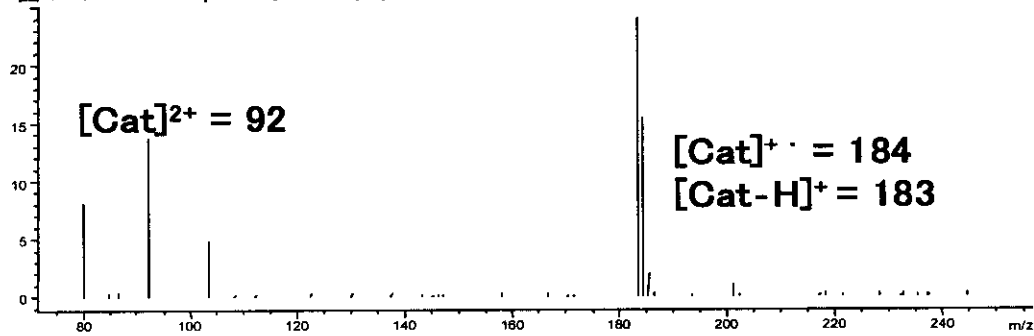


図2. 6. 6 Diquatのマスペクトル

表2. 6. 2 Diquatの分析条件

HPLC		MS	
Instrument	Agilent 1100	Instrument	Agilent 1100 LC/MSD SL
Column	TSK GEL SP NPR i.d. 4.6 x 35 mm	Ionization	Electrospray Fragmentor 100V
Mobile phase	A=80mM HCO <sub>2</sub> NH <sub>4</sub> / B=CH <sub>3</sub> CN B 20% isocratic	Neblizer	N <sub>2</sub> (60 psi)
Flow rate	1.2 mL/min.	Drying gas	N <sub>2</sub> (13 L/min.)
Injection vol.	SIM 100 µL	Vcap	1500 V
		Polarity	Positive
		SIM (m/z)	93 ; PQ

## 2.7 陰イオン系農薬の検討結果

### 2.7.1 Fosetylの検討結果

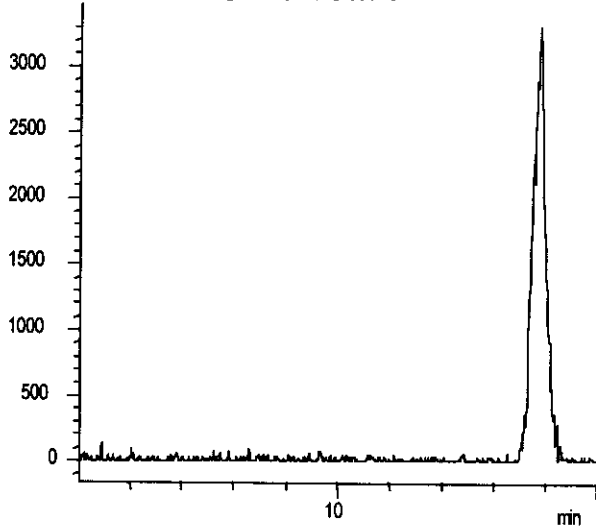


図2.7.1 Fosetylのクロマトグラム

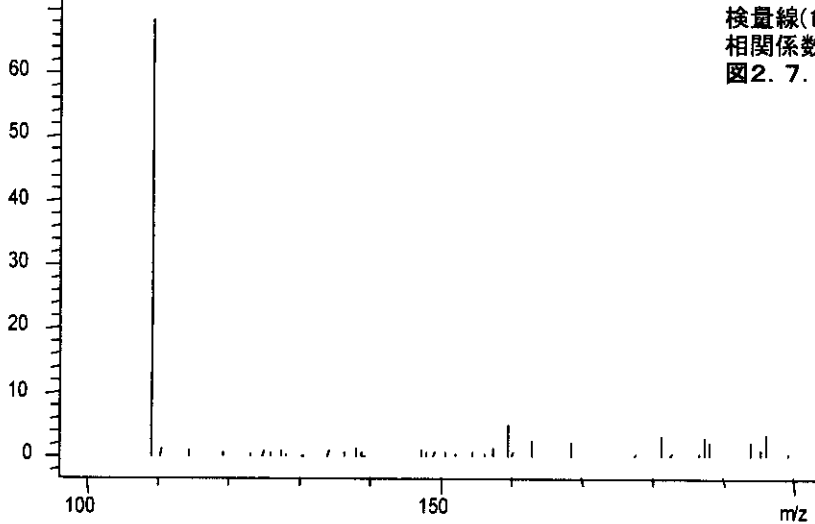
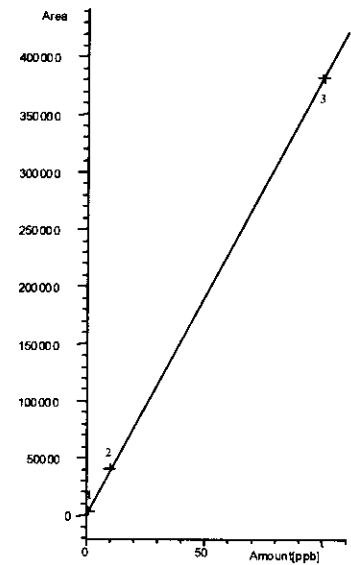


図2.7.2 Fosetylのマスペクトル

表2.7.1 Fosetylの分析条件

HPLC		MS	
Instrument	Agilent 1100	Instrument	Agilent 1100 LC/MSD SL
Column	TSK GEL SuperIC-AP i.d. 4.6 x 75 mm	Mass range	$m/z$ 80 ~ 500
Mobile phase	0.2% Formic acid	Ionization	Electrospray
Flow rate	1.0 mL/min.	Fragmentor	100V
Injection vol.	Scan 1.3mg/mL x 10 $\mu$ L SIM 10ng/mL x 100 $\mu$ L	Neblizer	N2 (60 psi)
		Drying gas	N2 (13 L/min.)
		Vcap	2500 V
		Polarity	Negative
			110/AMPA
			180/Glufocinate
			168/Glyphosate
			109/Fosetyl aluminium



検量線(1~100mg/L), 100 $\mu$ L注入  
相関係数: 0.99996

図2.7.3 Fosetylの検量線



## 2. 7. 2 Glufosinateの検討結果

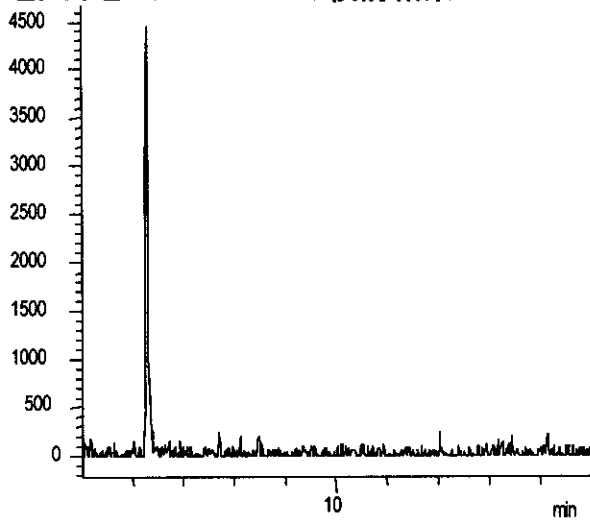


図2. 7. 4 Glufosinateのクロマトグラム

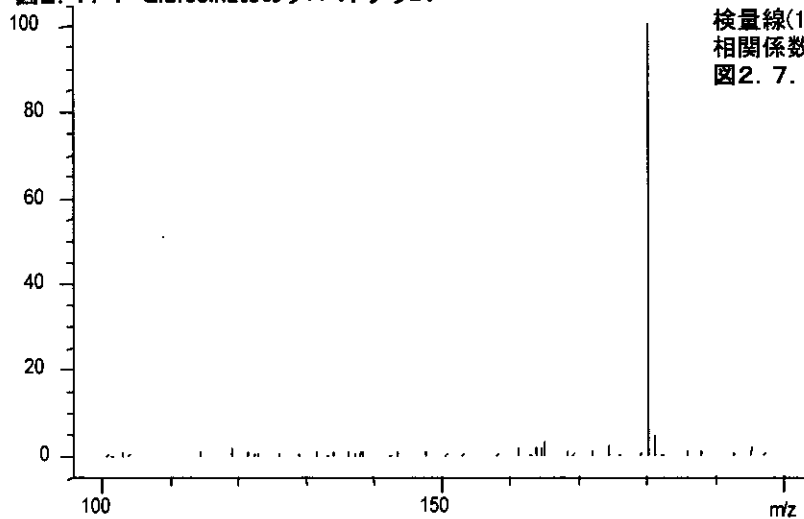
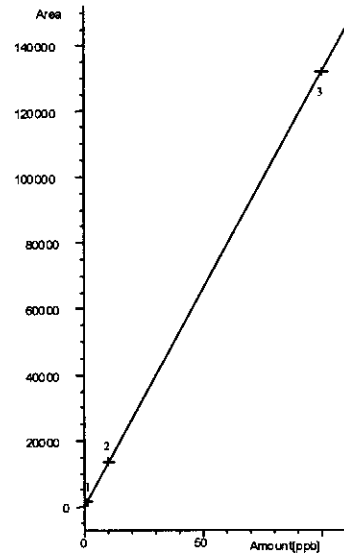


図2. 7. 5 Glufosinateのマスペクトル

表2. 7. 2 Glufosinateの分析条件

HPLC		MS	
Instrument	Agilent 1100	Instrument	Agilent 1100 LC/MSD SL
Column	TSK GEL SuperIC-AP i.d. 4.6 x 75 mm	Mass range	$m/z$ 80 ~ 500
Mobile phase	0.2% Formic acid	Ionization	Electrospray
Flow rate	1.0 mL/min.	Fragmentor	100V
Injection vol.	Scan 1.3mg/mL x 10 $\mu$ L	Neblizer	N2 (60 psi)
	SIM 10ng/mL x 100 $\mu$ L	Drying gas	N2 (13 L/min.)
	Vcap		2500 V
	Polarity		Negative
			110/AMPA
			180/Glufocinate
			168/Glyphosate
			109/Fosetyl aluminium



検量線(1~100mg/L), 100 $\mu$ L注入  
相関係数:0.99996

図2. 7. 6 Glufosinateの検量線

### 2. 7. 3 Glyphosateの検討結果

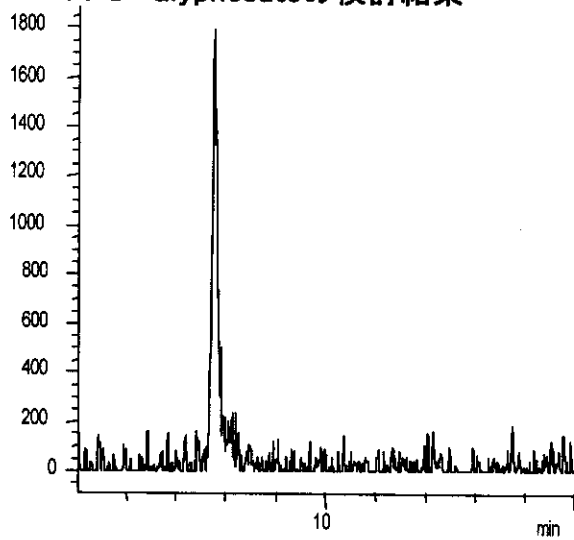


図2. 7. 7 Glyphosateのクロマトグラム

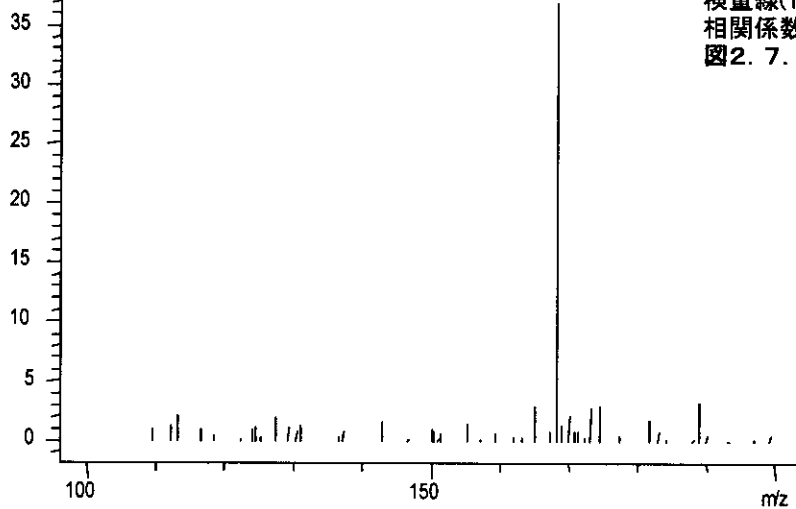
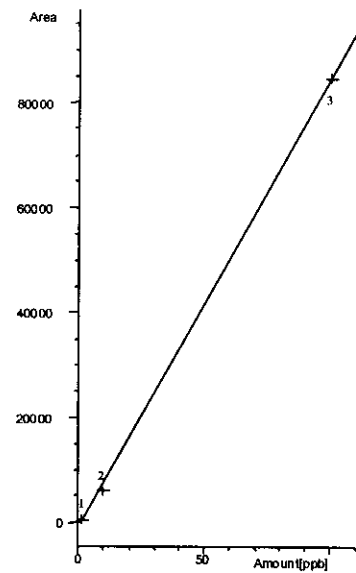


図2. 7. 8 Glyphosateのマススペクトル

表2. 7. 3 Glyphosateの分析条件

HPLC		MS	
Instrument	Agilent 1100	Instrument	Agilent 1100 LC/MSD SL
Column	TSK GEL SuperIC-AP i.d. 4.6 x 75 mm	Mass range	$m/z$ 80 ~ 500
Mobile pha	0.2% Formic acid	Ionization	Electrospray
Flow rate	1.0 mL/min.	Fragmentor	100V
Injection vol	Scan 1.3mg/mL x 10 $\mu$ L	Neblizer	N2 (60 psi)
	SIM 10ng/mL x 100 $\mu$ L	Drying gas	N2 (13 L/min.)
		Vcap	2500 V
		Polarity	Negative
			110/AMPA
			180/Glufocinate
			168/Glyphosate
			109/Fosetyl aluminium



検量線(1~100mg/L), 100 $\mu$ L注入  
相関係数:0.99996

図2. 7. 9 Glyphosateの検量線

## 2.7.4 AMPAの検討結果

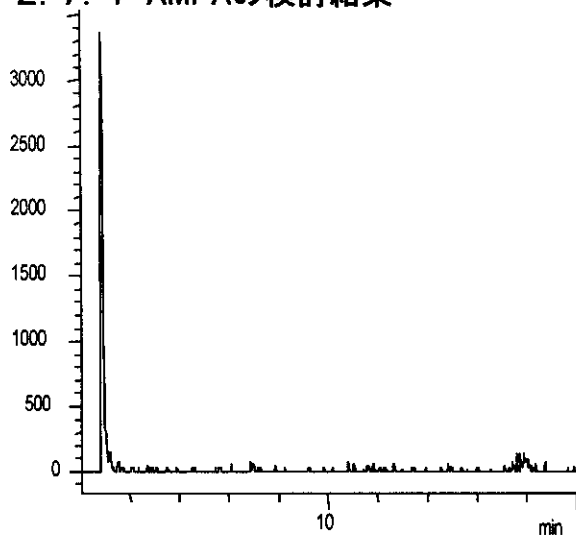


図2.7.10 AMPAのクロマトグラム

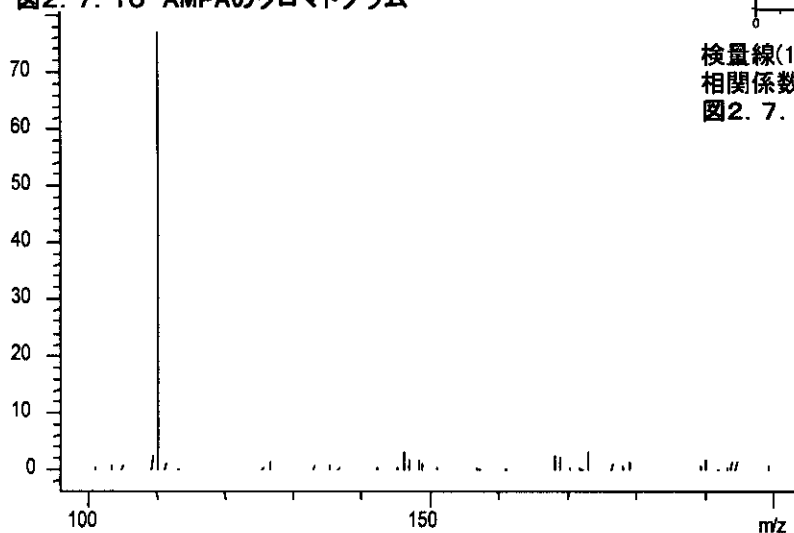
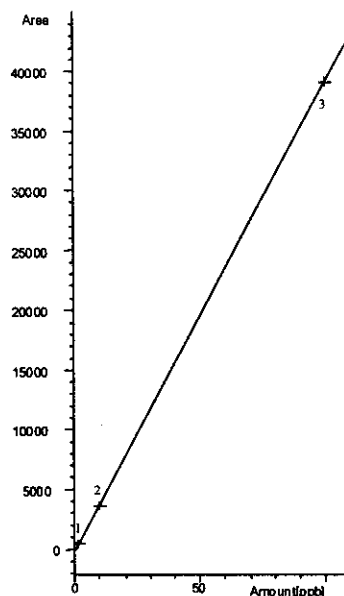


図2.7.11 AMPAのマスペクトル

表2.7.4 AMPAの分析条件

HPLC		MS	
Instrument	Agilent 1100	Instrument	Agilent 1100 LC/MSD SL
Column	TSK GEL SuperIC-AP i.d. 4.6 x 75 mm	Mass range	$m/z$ 80 ~ 500
Mobile pha:	0.2% Formic acid	Ionization	Electrospray
Flow rate	1.0 mL/min.	Fragmentor	100V
Injection v	Scan 1.3mg/mL x 10 $\mu$ L	Neblizer	N2 (60 psi)
	SIM 10ng/mL x 100 $\mu$ L	Drying gas	N2 (13 L/min.)
		Vcap	2500 V
		Polarity	Negative



検量線(1~100mg/L), 100 $\mu$ L注入  
相関係数:0.99996

図2.7.12 AMPAの検量線

## 2.8 ウレア系農薬の検討結果

### 2.8.1 Dymronの検討結果

精製水への添加回収実験

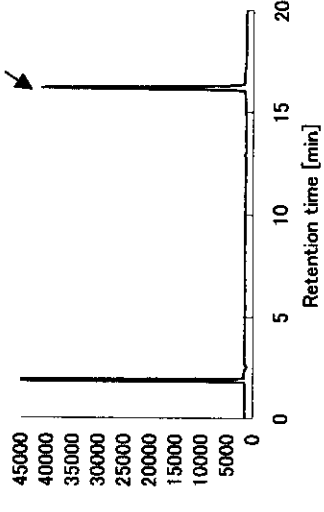


図2.8.1 PS2を用いた結果

水道水への添加回収実験

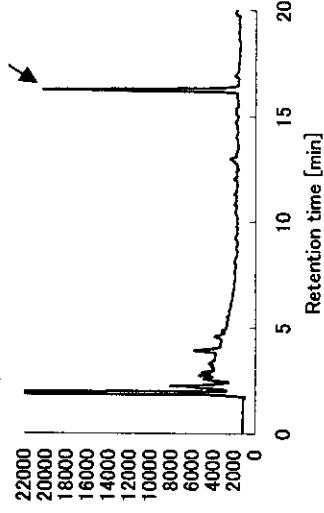


図2.8.2 PS2を用いた結果

河川水への添加回収実験

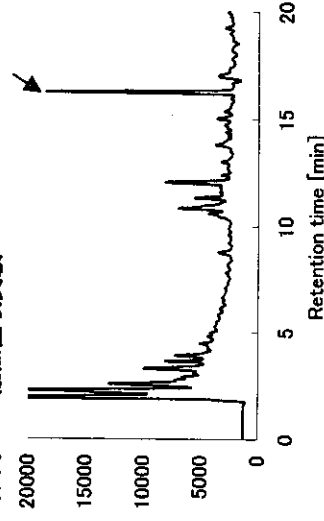


図2.8.3 PS2を用いた結果

図2.8.4 C18を用いた場合

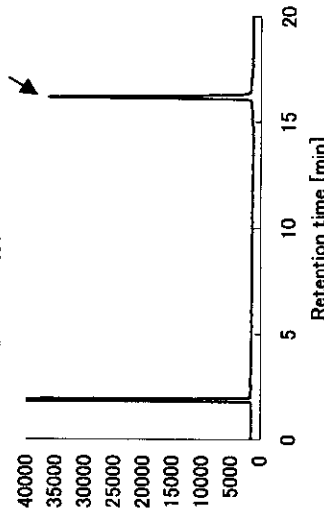


図2.8.5 C18を用いた場合

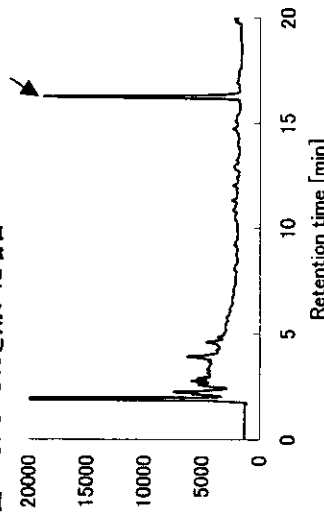


図2.8.6 C18を用いた場合

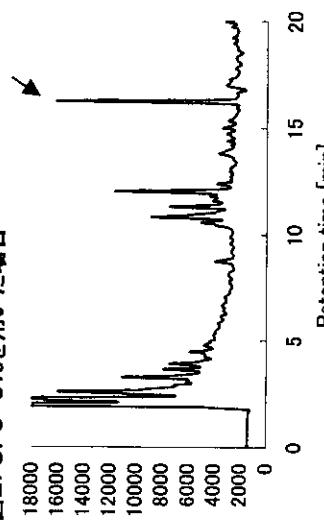


図2.8.7 Oasisを用いた場合

図2.8.8 Oasisを用いた場合

図2.8.9 Oasisを用いた場合