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LC/oaTOFMSによる臭素系難燃剤の分析

日本ウォーターズ(株)

分析条件

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- 試料
  - 試料10mgを5mLのトルエンで溶解後、5mlのアセトニトリルを加え、全量で10 mLとした(1mg/mL溶液)。
- HPLC
  - 装置:Alliance 2695
  - ｶﾗﾑ:Symmetry C18 150x2.1 I.D. 3.5u
  - 移動相:90%アセトニトリル
  - 流速:0.3 mL/min
  - 注入量:5μL
- MS
  - 装置:LCT Premier
  - イオン化モード:ESIネガティブ
  - キャピラリー電圧:3300 V
  - コーン電圧:100 V
  - ソース温度:120 ℃
  - 脱溶媒ガス温度:400 ℃
  - 脱溶媒ガス流量:600 L/Hr
  - コーンガス流量:50 L/Hr
  - TOF モード:Wモード(分解能約10,000)
  - スキャン時間:1 sec
  - インタースキャンディレイ:0.1 sec
  - ロックスプレー:使用
  - ロックマス:Leucine Enkepharine (m/z 556.2771)1ug/ml 5uL/min
  - DRE:OFF

Octa-BDEのクロマトグラム

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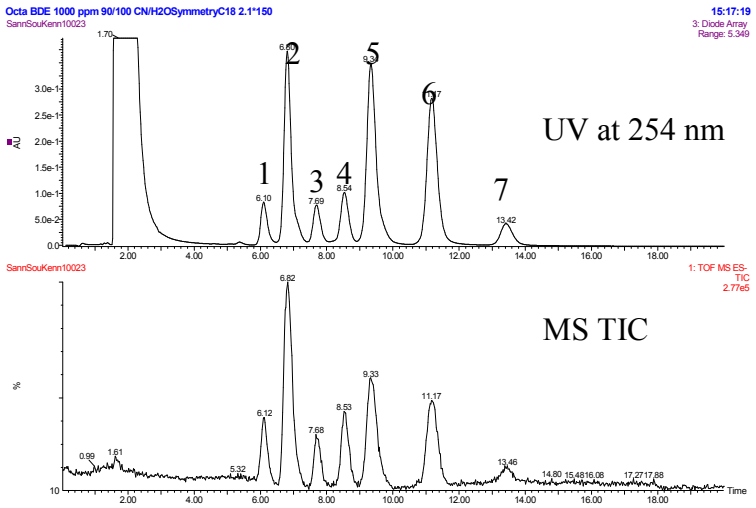

 3

Deca-BDEのクロマトグラム

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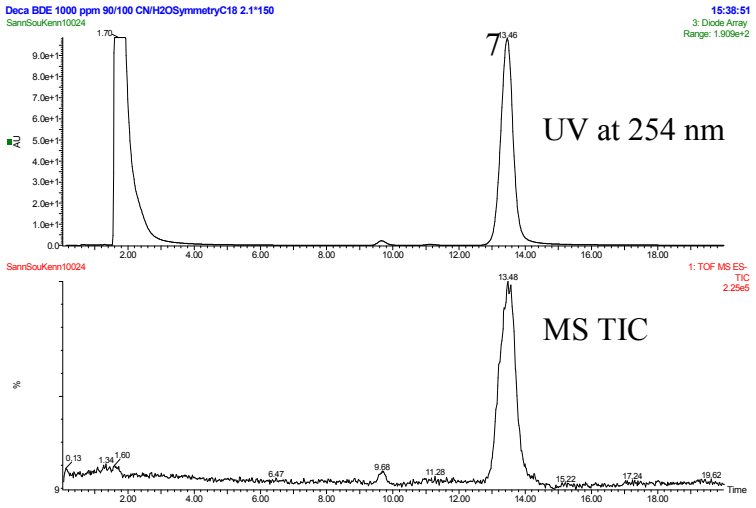

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ピーク 7のスペクトル (Deca-BDE)

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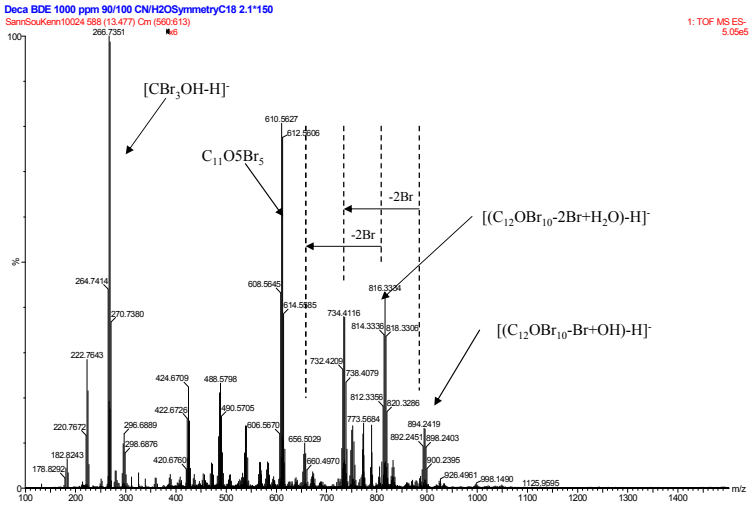

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ピーク 7の組成分析結果1 (Deca-BDE)

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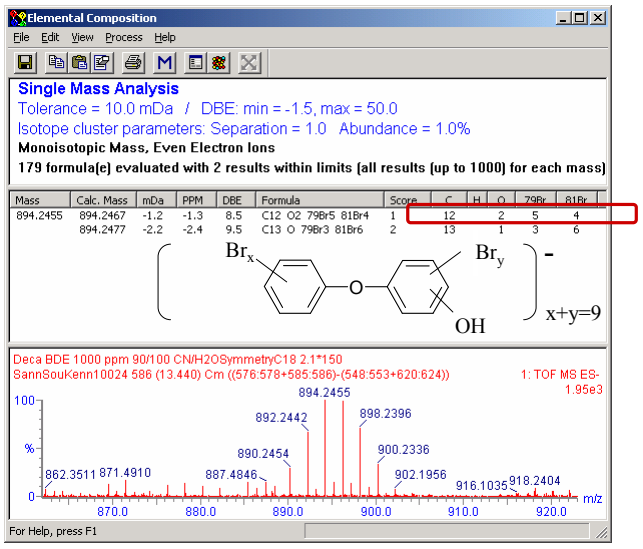

 6

$[(C_{12}OBr_{10}\text{-}Br+OH)\text{-}H]^-$

Waters
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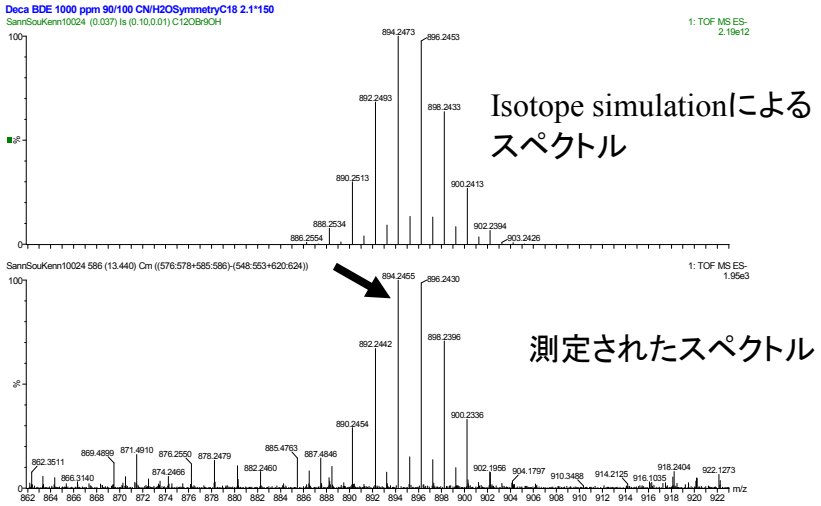

Isotope simulationによる
スペクトル

測定されたスペクトル

 7

ピーク 7の組成分析結果2 (Deca-BDE)

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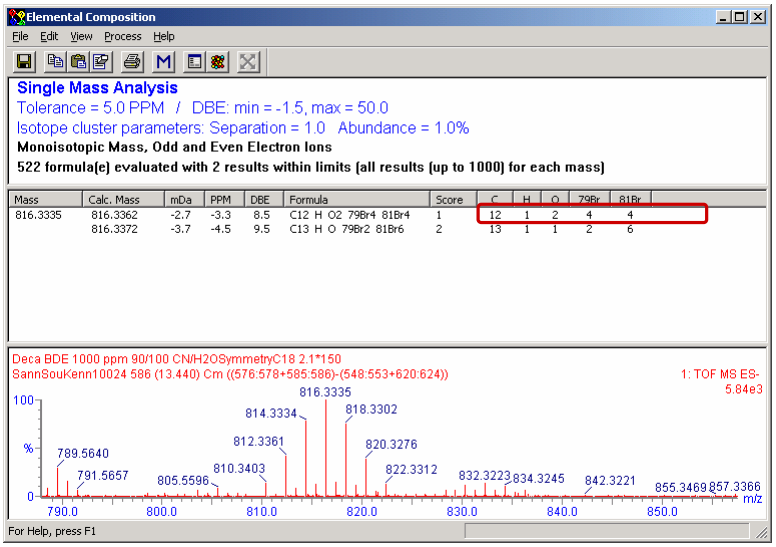

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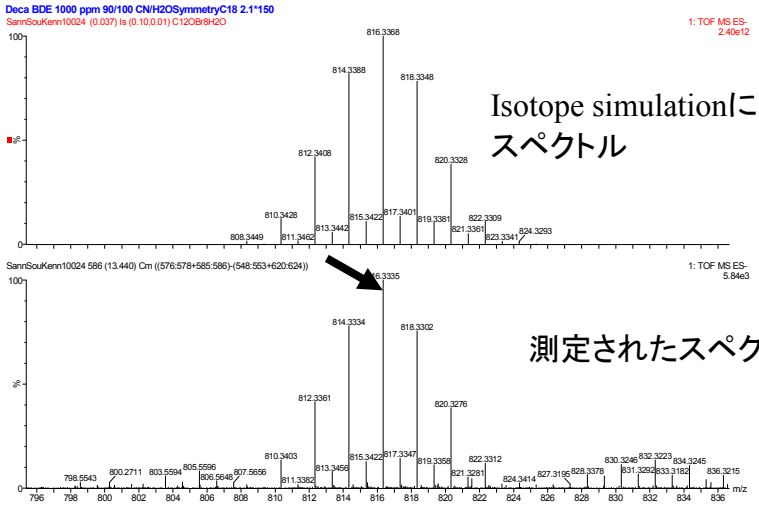

Isotope simulationによる
スペクトル

測定されたスペクトル

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ピーク 7の組成分析結果3 (Deca-BDE)

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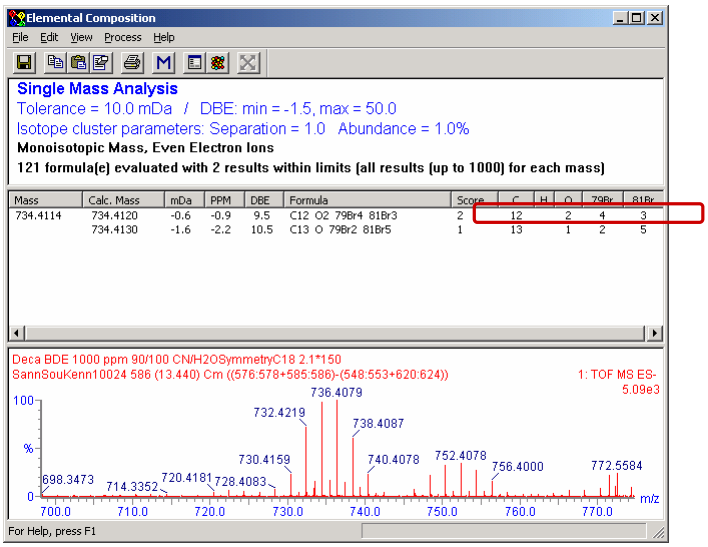

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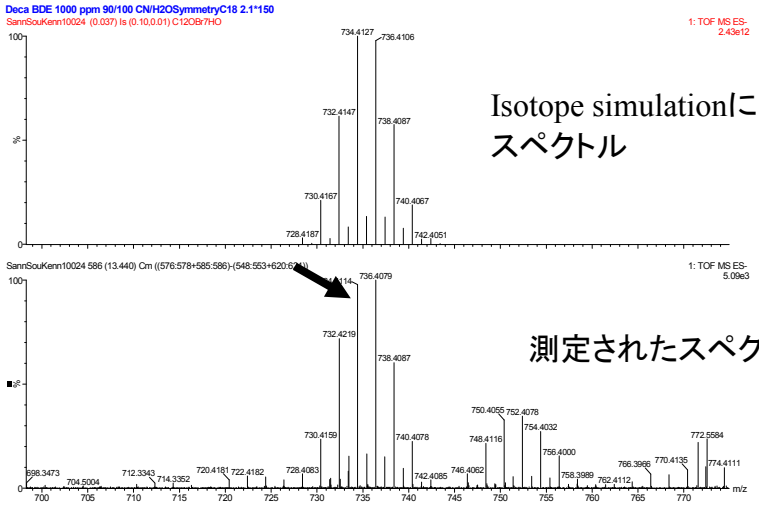

Isotope simulationによる
スペクトル

測定されたスペクトル

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ピーク 7の組成分析結果4 (Deca-BDE)

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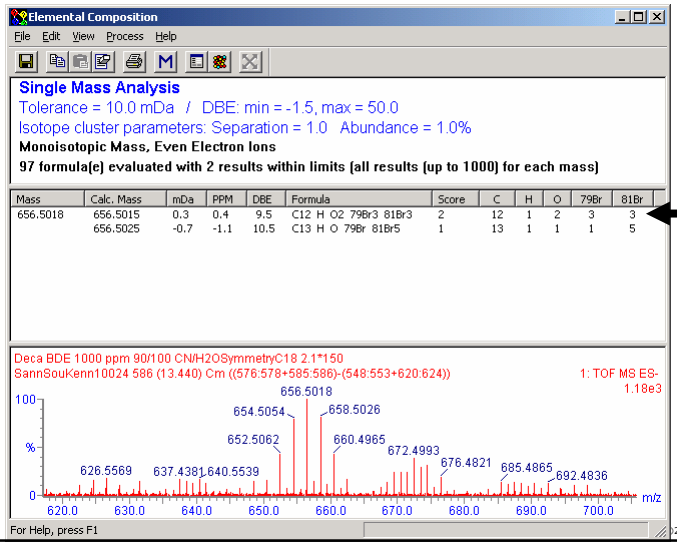

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
656.5018	656.5015	0.3	0.4	9.5	C12 H O2 79Br3 81Br3	2	12	1	2	3	3
	656.5025	-0.7	-1.1	10.5	C13 H O 79Br 81Br5	1	13	1	1	1	5

 12

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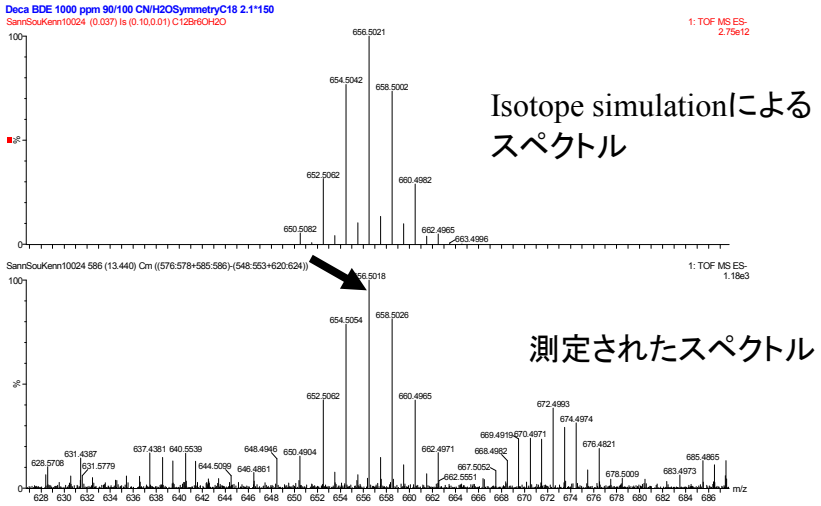

Isotope simulationによる
スペクトル

測定されたスペクトル

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ピーク 7の組成分析結果5 (Deca-BDE)

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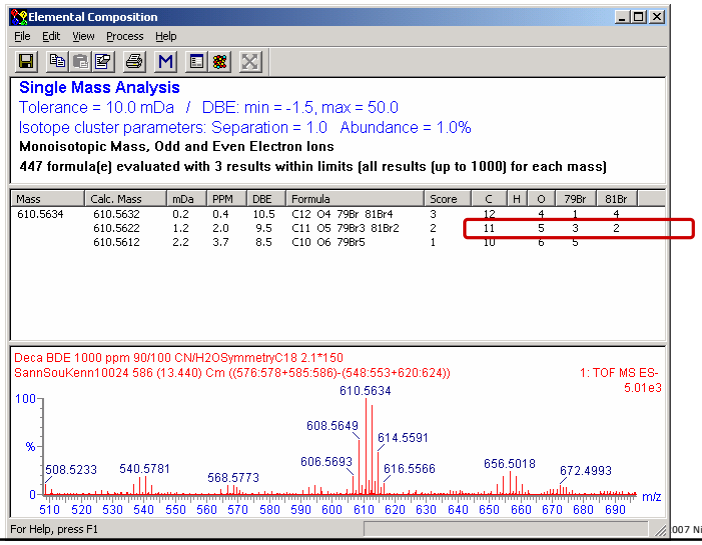

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
610.5634	610.5632	0.2	0.4	10.5	C12 O4 79Br 81Br4	3	12		4	1	4
	610.5622	1.2	2.0	9.5	C11 O5 79Br3 81Br2	2	11		5	3	2
	610.5612	2.2	3.7	8.5	C10 O6 79Br5	1	10		6	5	

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$C_{11}O_5Br_5$

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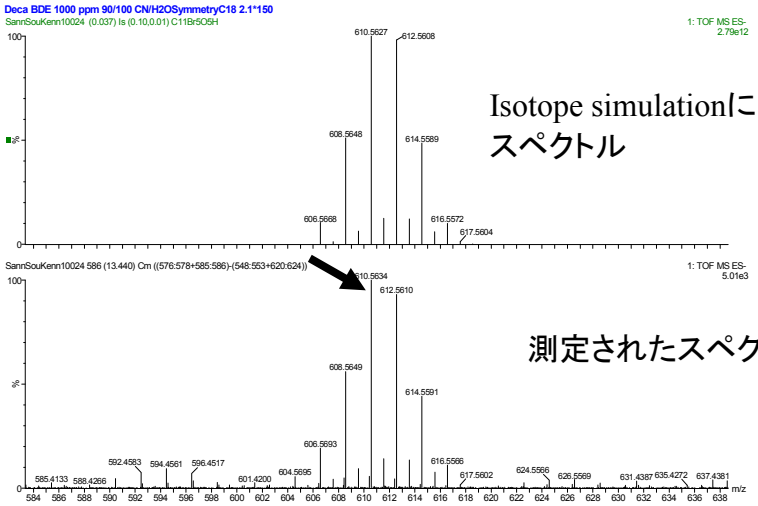

Isotope simulationによる
スペクトル

測定されたスペクトル

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ピーク 7の組成分析結果6 (Deca-BDE)

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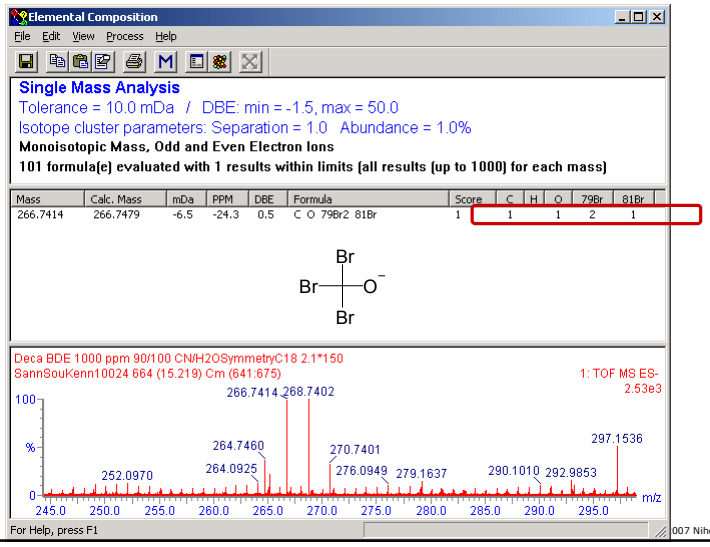

 16

CBr_3OH

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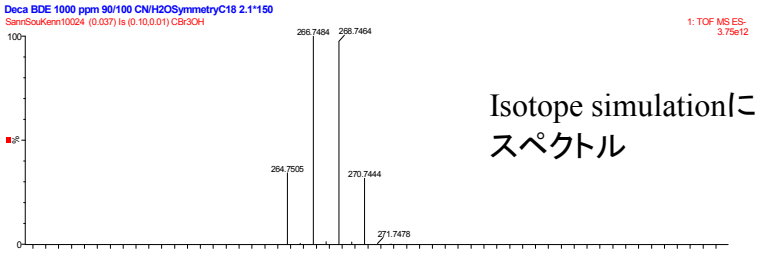
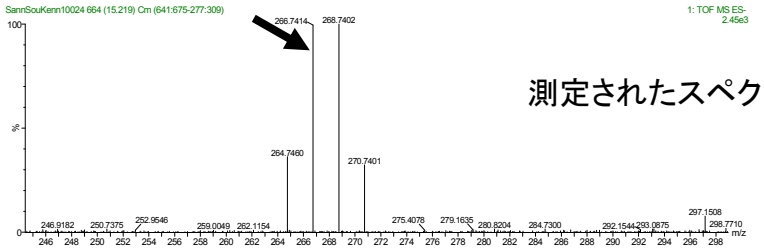


Isotope simulationによる
スペクトル



測定されたスペクトル

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ピーク5のスペクトル（Octa-BDE）

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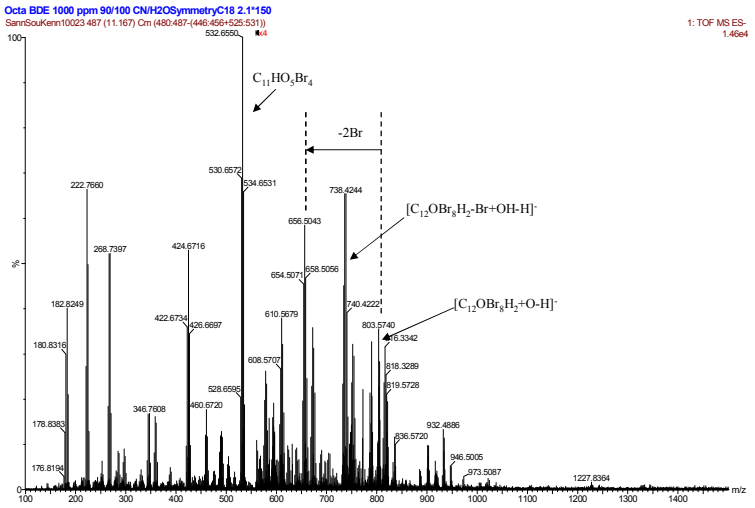

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ピーク 5の組成分析結果1 (Octa-BDE)

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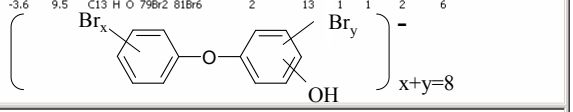
Elemental Composition

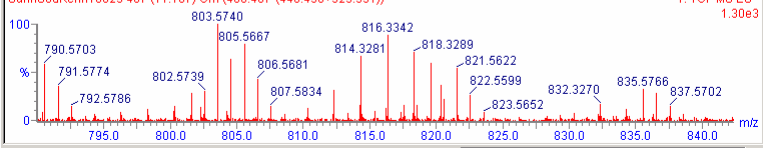
File Edit View Process Help

Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%
Monoisotopic Mass, Odd and Even Electron Ions
483 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
816.3342	816.3352	-1.0	-1.2	7.5	C11 H O3 79Br6 81Br2	1	11	1	3	6	2
	816.3362	-2.0	-2.4	8.5	C12 H O2 79Br4 81Br4	3	12	1	2	4	4
	816.3372	-3.0	-3.6	9.5	C13 H O 79Br2 81Br6	2	13	1	1	2	6



Octa BDE 1000 ppm 90/100 CN/H2OSymmetryC18 2.1*150
SannSouKenn10023 487 (11.167) Cm (480:487-(446:456+525:531))
1: TOF MS ES-
1.30e3



For Help, press F1

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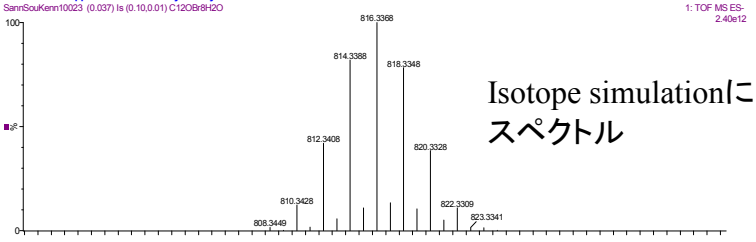
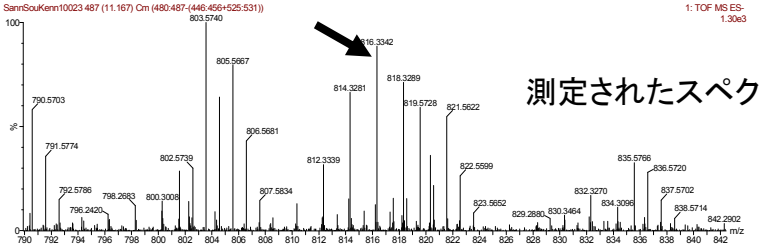
Octa BDE 1000 ppm 90/100 CN/H2OSymmetryC18 2.1*150
SannSouKenn10023 (0.037) Is (0.10,0.01) C12OBr8H2O
1: TOF MS ES-
2.40e12



Isotope simulationによる
スペクトル

SannSouKenn10023 487 (11.167) Cm (480:487-(446:456+525:531))
1: TOF MS ES-
1.30e3



測定されたスペクトル

ピーク 5の組成分析結果3 (Octa-BDE)

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Elemental Composition

File Edit View Process Help

Single Mass Analysis

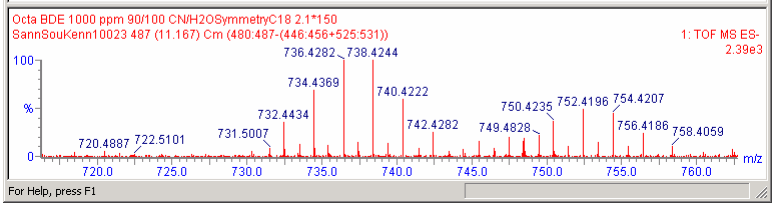
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

389 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
736.4282	736.4287	-0.5	-0.7	9.5	C13 H2 O 79Br2 81Br5	2	13	2	1	2	5
	736.4277	0.5	0.7	8.5	C12 H2 O2 79Br4 81Br3	3	12	2	2	4	3
	736.4267	1.5	2.0	7.5	C11 H2 O3 79Br6 81Br	4	11	2	3	6	1
	736.4233	4.9	6.7	11.5	C14 O 79Br7	1	14		1	7	



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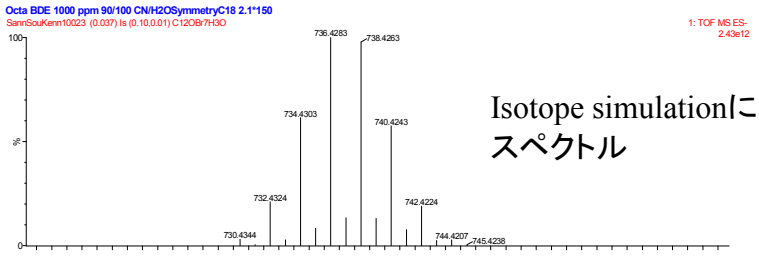
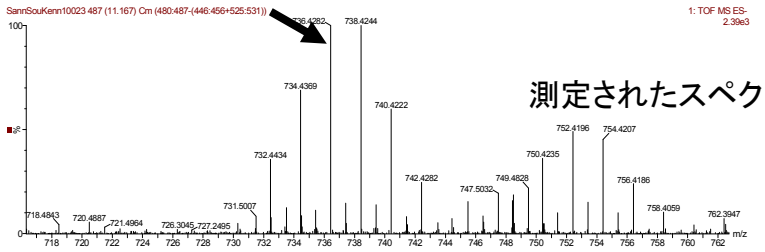


Isotope simulationによるスペクトル



測定されたスペクトル

ピーク 5の組成分析結果4 (Octa-BDE)

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Elemental Composition

File Edit View Process Help

Single Mass Analysis

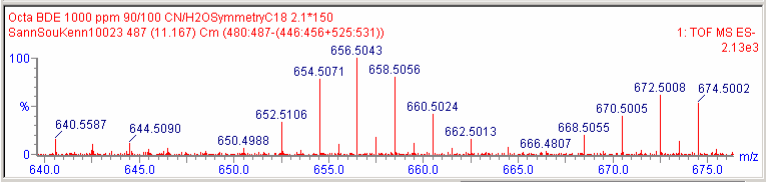
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

307 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
656.5043	656.5025	1.8	2.7	10.5	C13 H O 79Br 81Br5	1	13	1	1	1	5
	656.5015	2.8	4.2	9.5	C12 H O2 79Br3 81Br3	2	12	1	2	3	3
	656.5005	3.8	5.7	8.5	C11 H O3 79Br5 81Br	3	11	1	3	5	1



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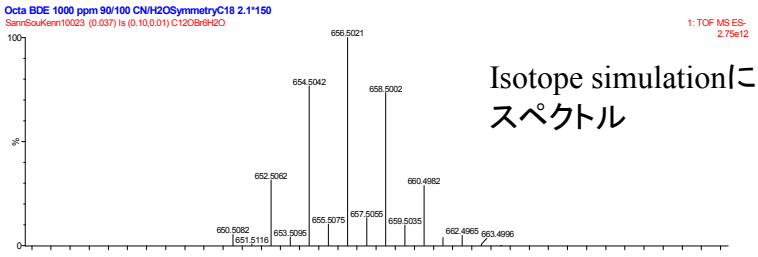

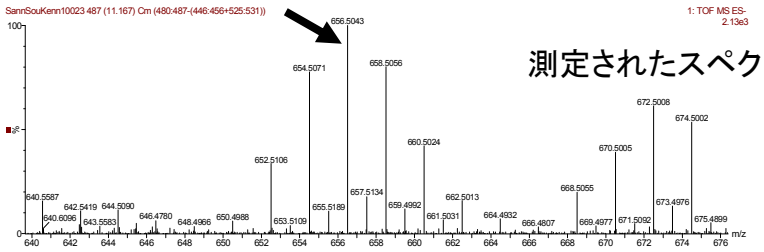

ピーク 5の組成分析結果4 (Octa-BDE)

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Elemental Composition

File Edit View Process Help

Single Mass Analysis

Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

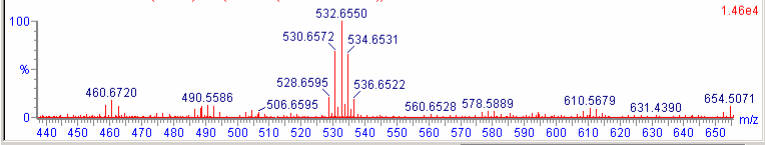
Monoisotopic Mass, Odd and Even Electron Ions

234 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula	Score	C	H	O	79Br	81Br
532.6550	532.6526	2.4	4.4	10.5	C12 H O4 81Br4	1	12	1	4		4
	532.6517	3.3	6.3	9.5	C11 H O5 79Br2 81Br2	3	11	1	5	2	2
	532.6507	4.3	8.2	8.5	C10 H O6 79Br4	2	10	1	6	4	

Octa BDE 1000 ppm 90/100 CN/H2OSymmetryC18 2.1*150
SannSouKenn10023 487 (11.167) Cm (480:487-(446:456+525:531))

1: TOF MS ES-
1.46e4



For Help, press F1

$C_{11}HO_5Br_4$

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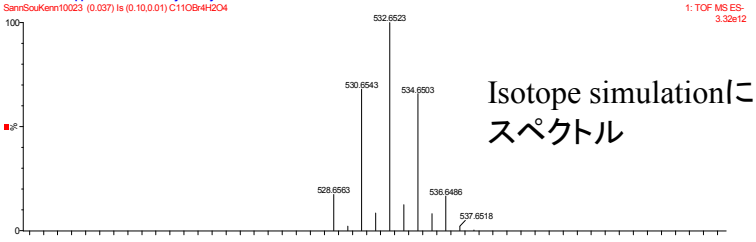
Octa BDE 1000 ppm 90/100 CN/H2OSymmetryC18 2.1*150
SannSouKenn10023 (0.037) Is (0.10,0.01) C11OBr4H2O4

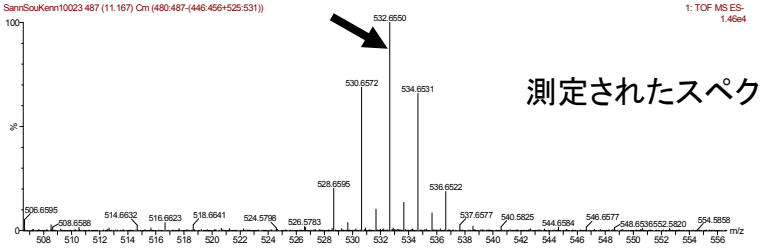
1: TOF MS ES-
3.32e12



Isotope simulationによる
スペクトル

SannSouKenn10023 487 (11.167) Cm (480:487-(446:456+525:531))

1: TOF MS ES-
1.46e4



測定されたスペクトル

