CHEMISTRY
A European Journal

Supporting Information

C-Functionalized Cationic Diazaoxatriangulenes: Late-Stage Synthesis and Tuning of Physicochemical Properties

Irene Hernández Delgado,[a] Simon Pascal,[a] Céline Besnard,[b] Silvia Voci,[c] Laurent Bouffier,[c] Neso Sojic,[c] and Jérôme Lacour*[a]

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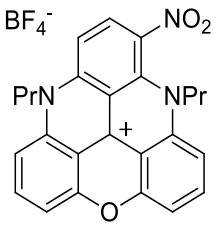
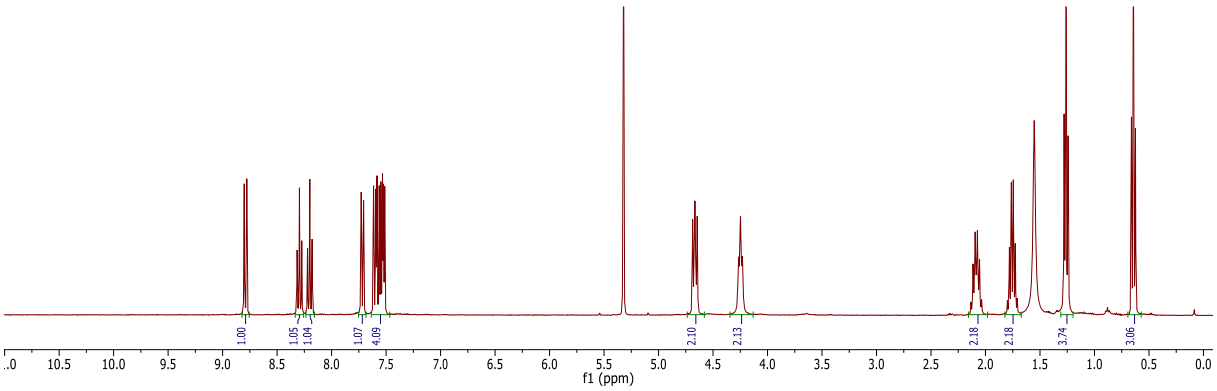
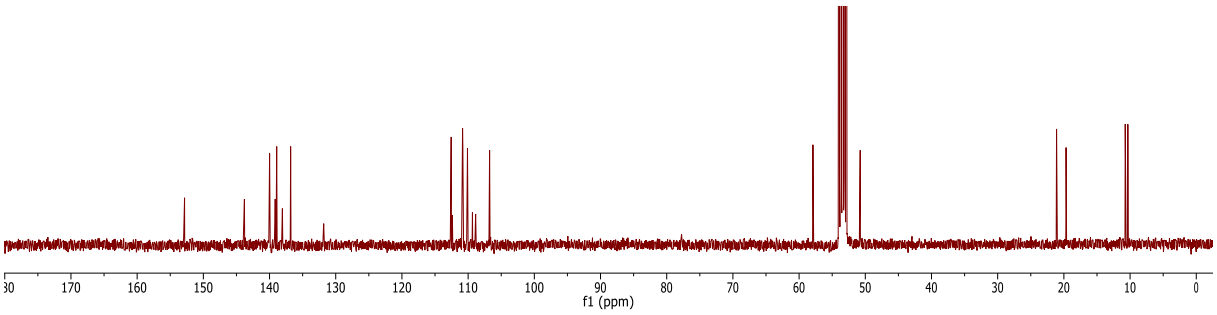
Compound 2



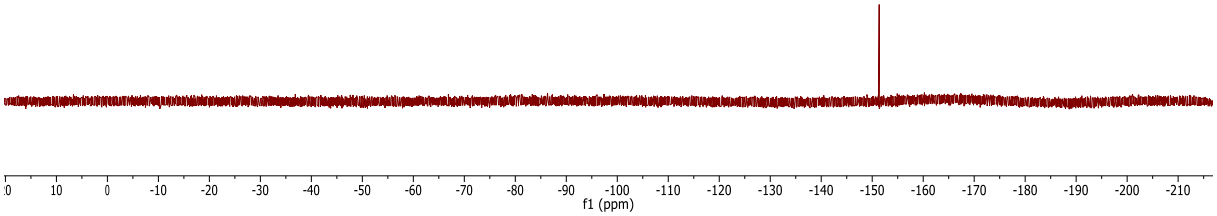
^{1}H NMR (400 MHz, CD_2Cl_2)



^{13}C NMR (101 MHz, CD_2Cl_2)



^{19}F NMR (282 MHz, CD_3CN)



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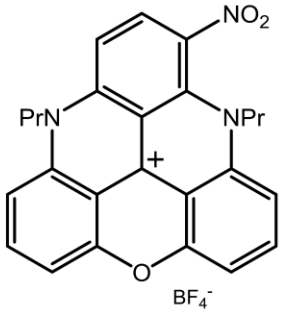
Faculty of Sciences

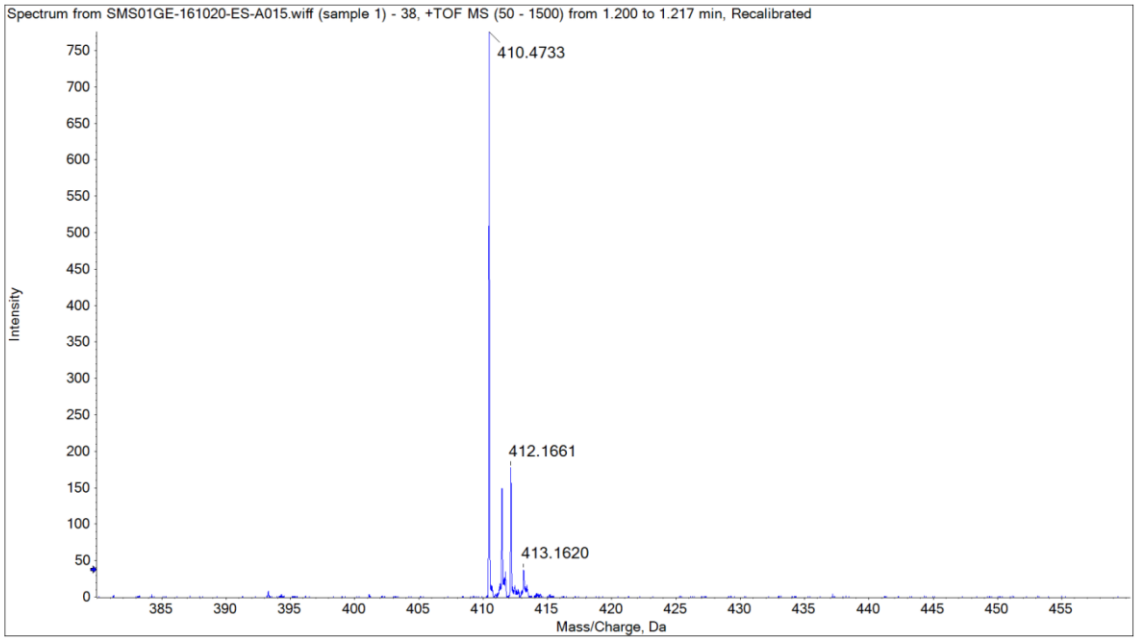
Sciences Mass Spectrometry

Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-NO2	Date of certificate:	26/10/2016
Sample number:	8511	Data filename:	SMS10GE-161020-ES-A015
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

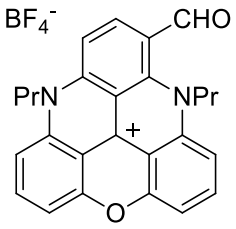
Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{25}H_{22}N_3O_2$	412.1661	412.1656	1.2



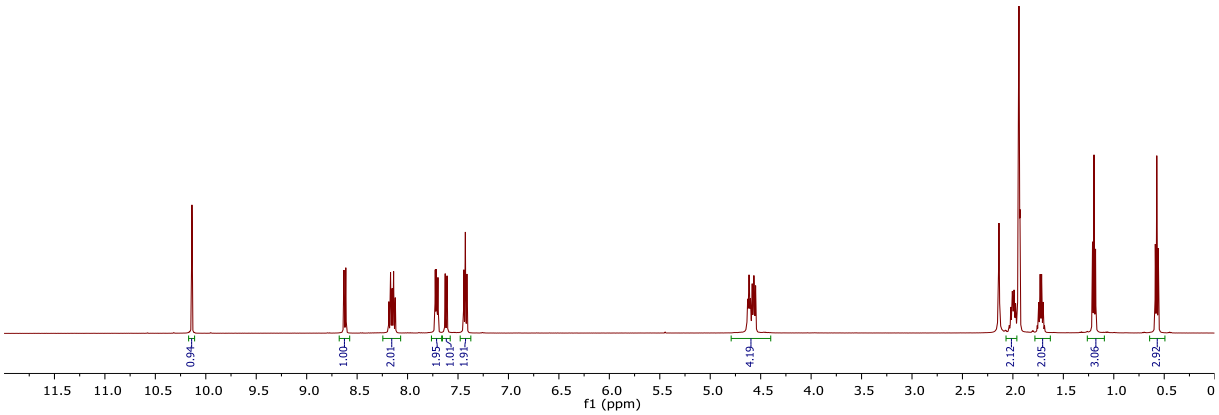
Chemical Formula:
$C_{25}H_{22}BF_4N_3O_3$ IH-DAOTA-NO_2
Exact Mass: 499.17



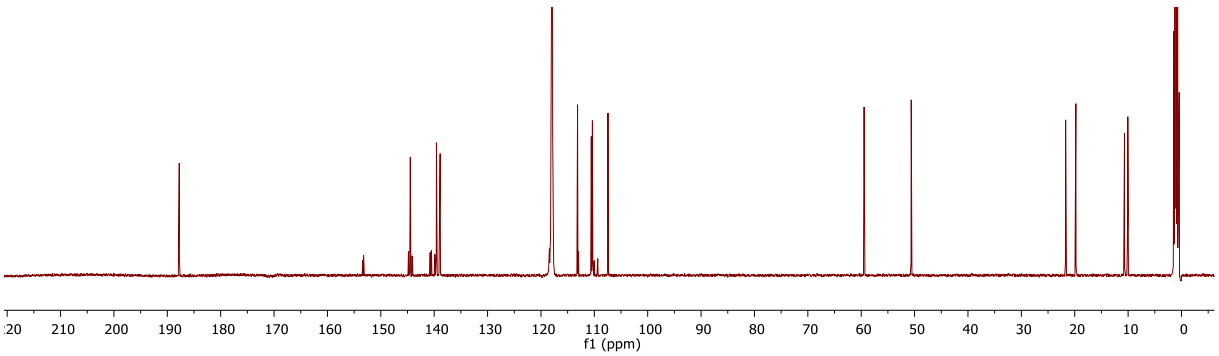
Compound 3



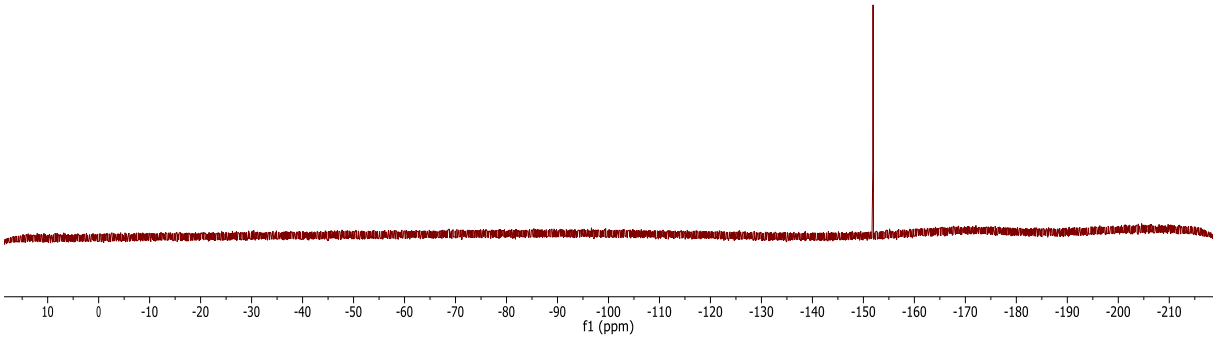
^{1}H NMR (400 MHz, CD_3CN)



^{13}C NMR (101 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



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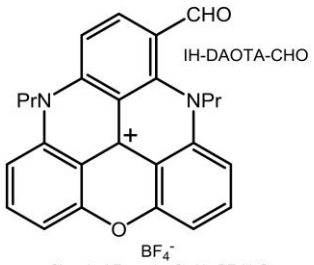
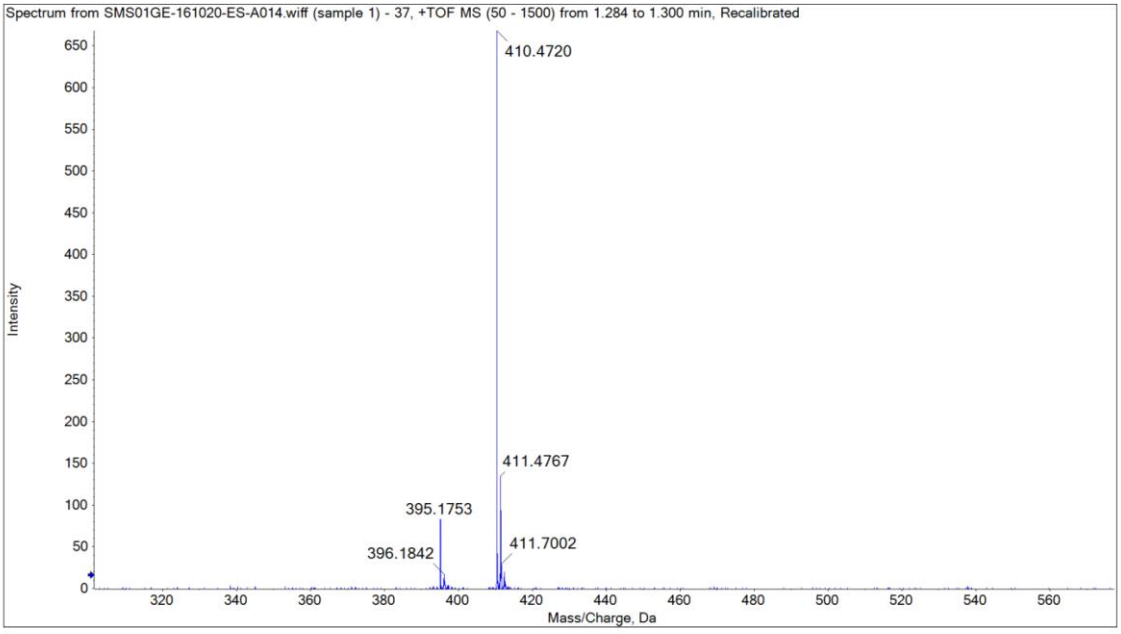
Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-CHO	Date of certificate:	26/10/2016
Sample number:	8510	Data filename:	SMS10GE-161020-ES-A014
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

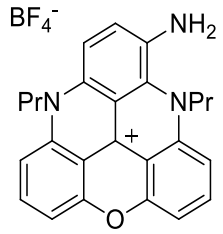
Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{26}H_{23}N_2O_2$	395.1753	395.1754	-0.3



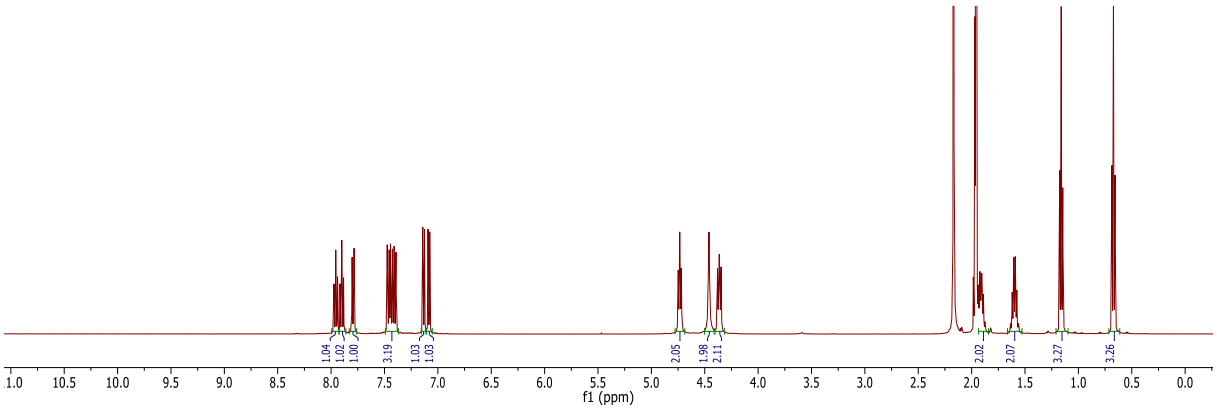
Chemical Formula: $C_{26}H_{23}BF_4N_2O_2$
Exact Mass: 482.18



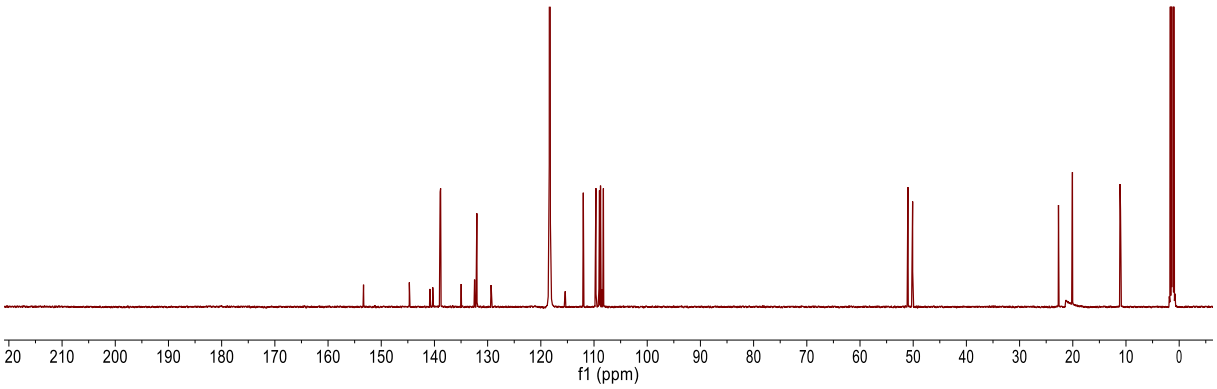
Compound 4



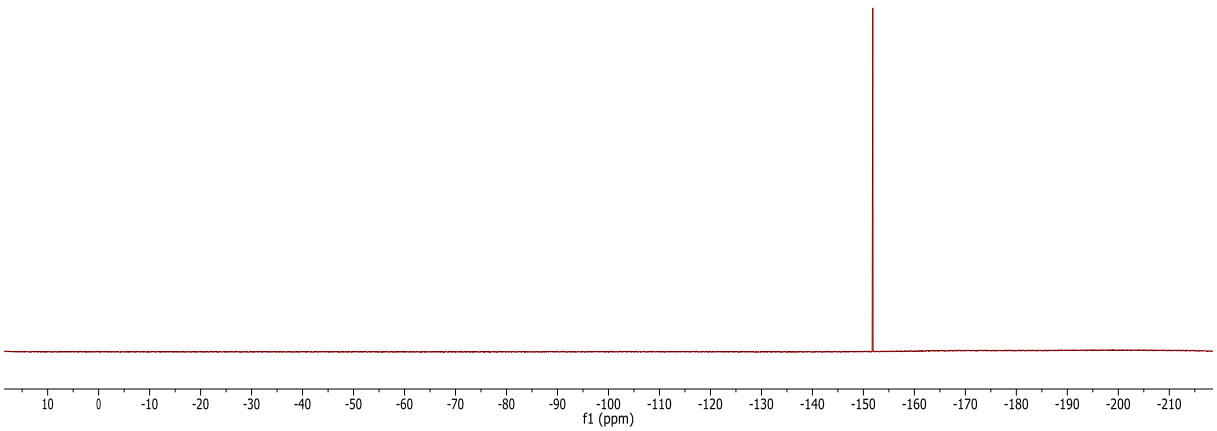
^{1}H NMR (400 MHz, CD_3CN)



^{13}C NMR (101 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



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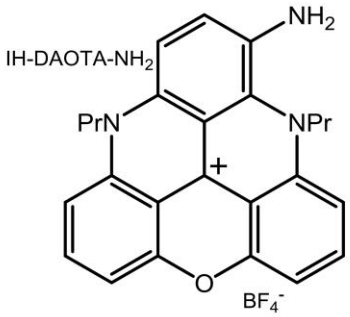
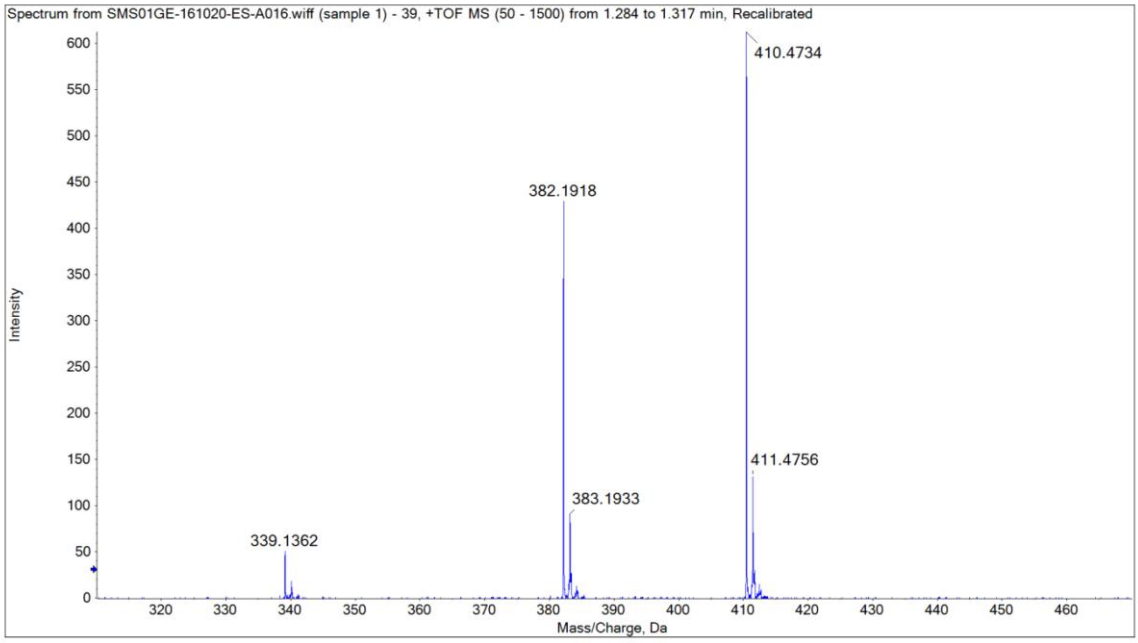
Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-NH2	Date of certificate:	26/10/2016
Sample number:	8512	Data filename:	SMS10GE-161020-ES-A016
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{25}H_{24}N_3O$	382.1918	382.1914	1.2



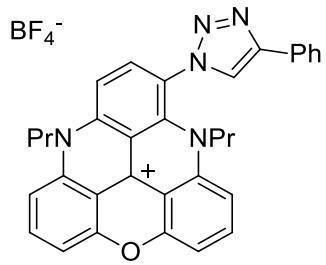
Chemical Formula: $C_{25}H_{24}BF_4N_3O$
Exact Mass: 469.19



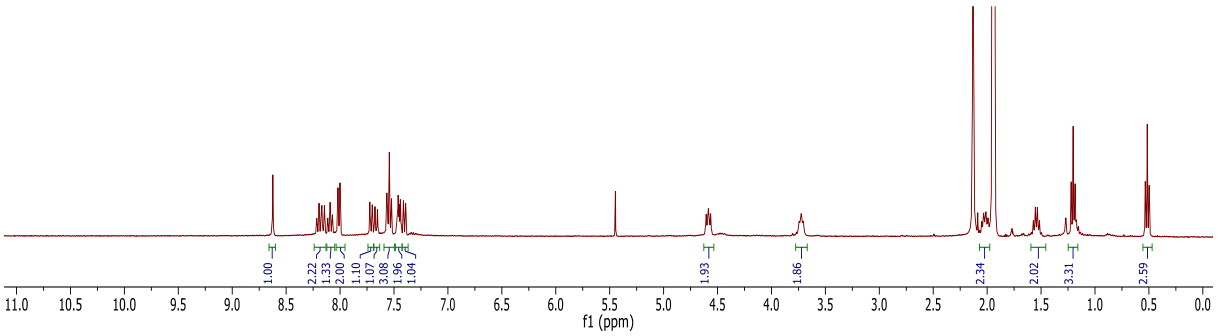
Compound 2

Compound 2

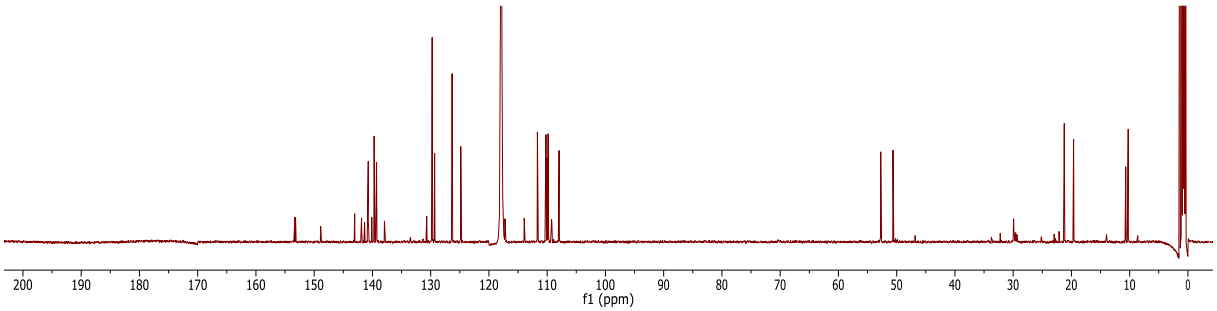
Compound 5



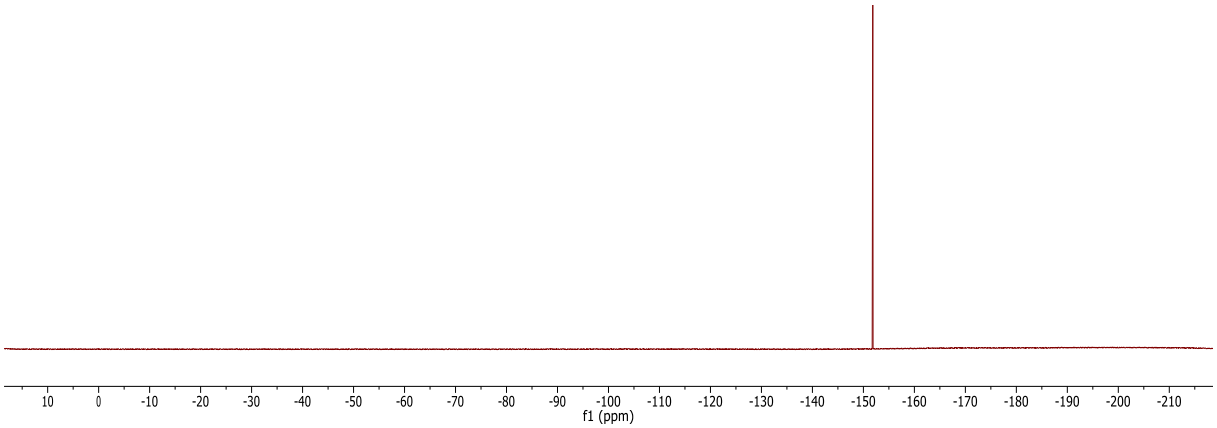
^{1}H NMR (500 MHz, CD_3CN)



^{13}C NMR (126 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



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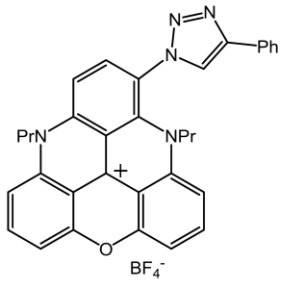
Sciences Mass Spectrometry

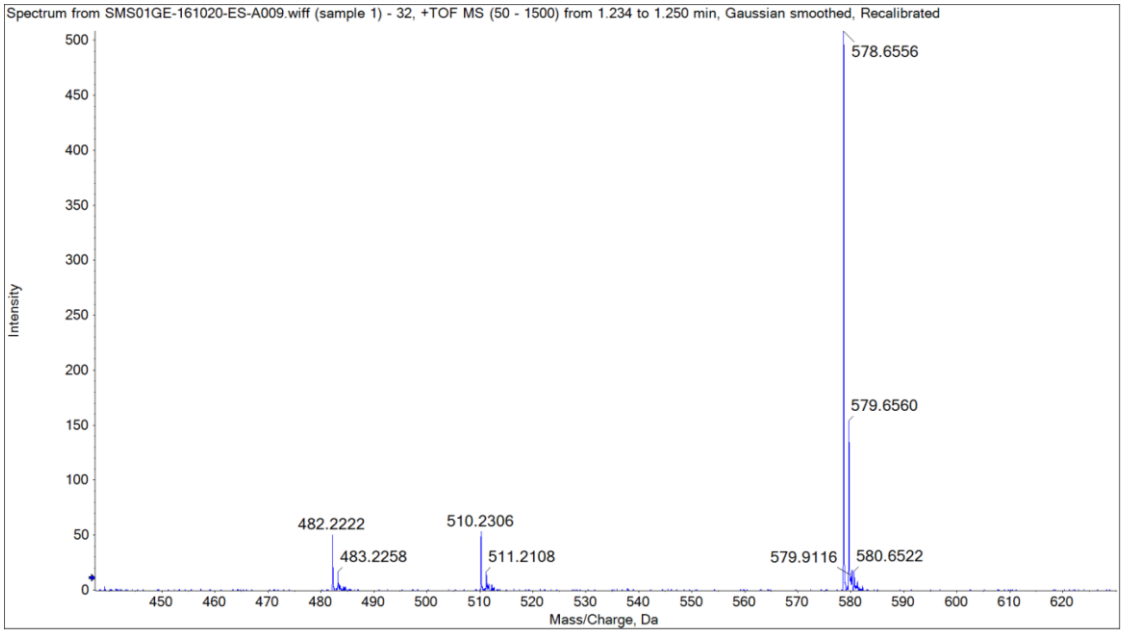
Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-TRIAZOL	Date of certificate:	24/10/2016
Sample number:	8507	Data filename:	SMS10GE-161020-ES-A009
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{33}H_{28}N_5O$	510.2306	510.2288	3.4

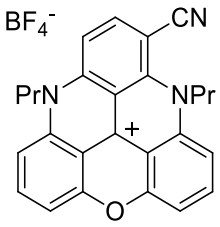


Chemical Formula: $C_{33}H_{28}BF_4N_5O$
Exact Mass: 597.23

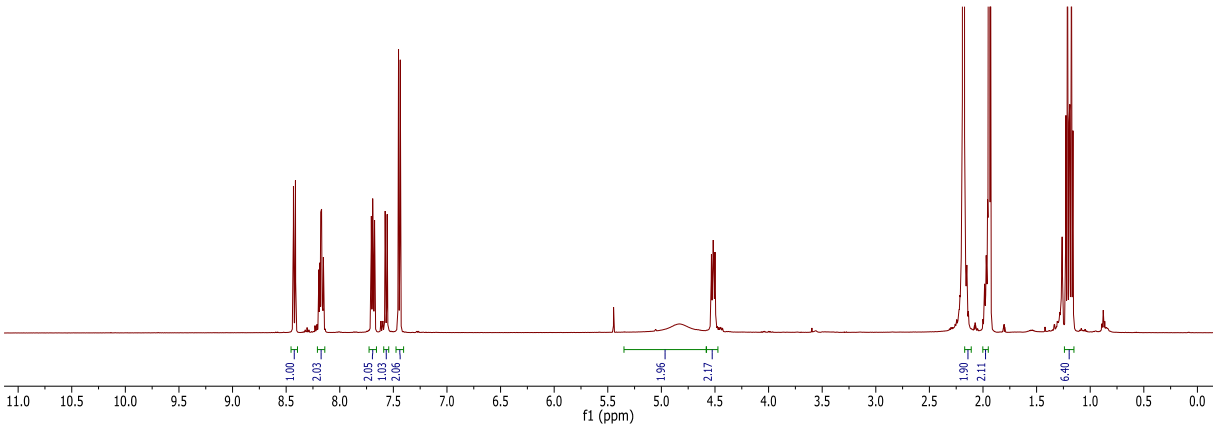
IH-DAOTA-Triazolo



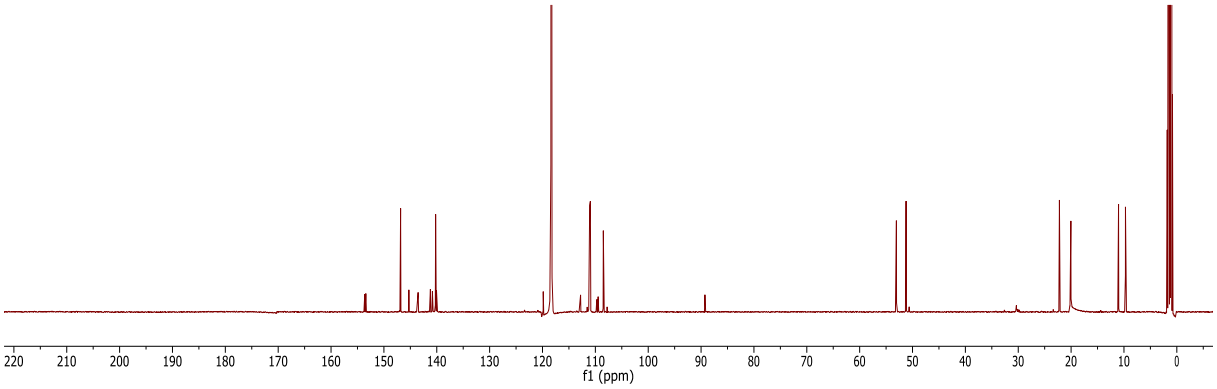
Compound 6



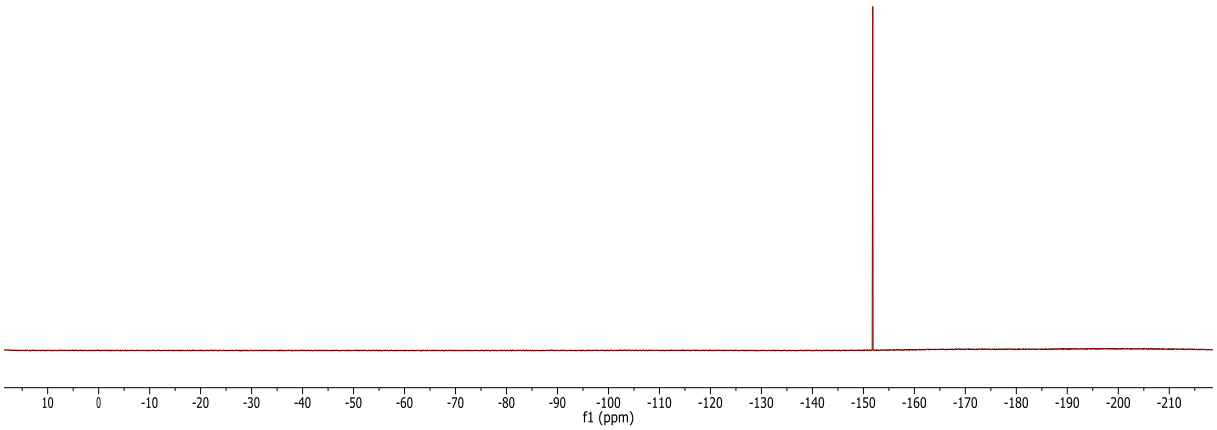
^{1}H NMR (500 MHz, CD_3CN)



^{13}C NMR (126 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



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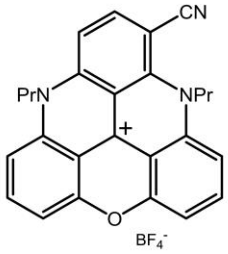
Sciences Mass Spectrometry

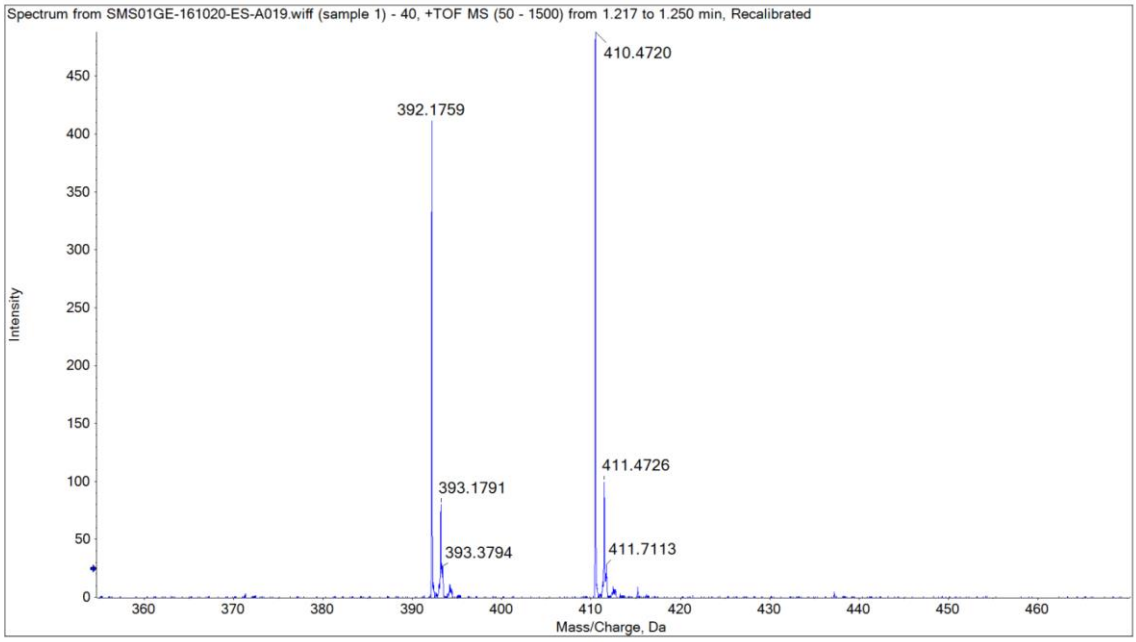
Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-CN	Date of certificate:	26/10/2016
Sample number:	8513	Data filename:	SMS10GE-161020-ES-A019
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{26}H_{22}N_3O$	392.1759	392.1757	0.5

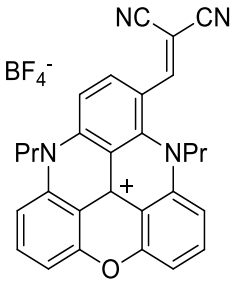


Chemical Formula:
$C_{26}H_{22}BF_4N_3O$
Exact Mass: 479.18

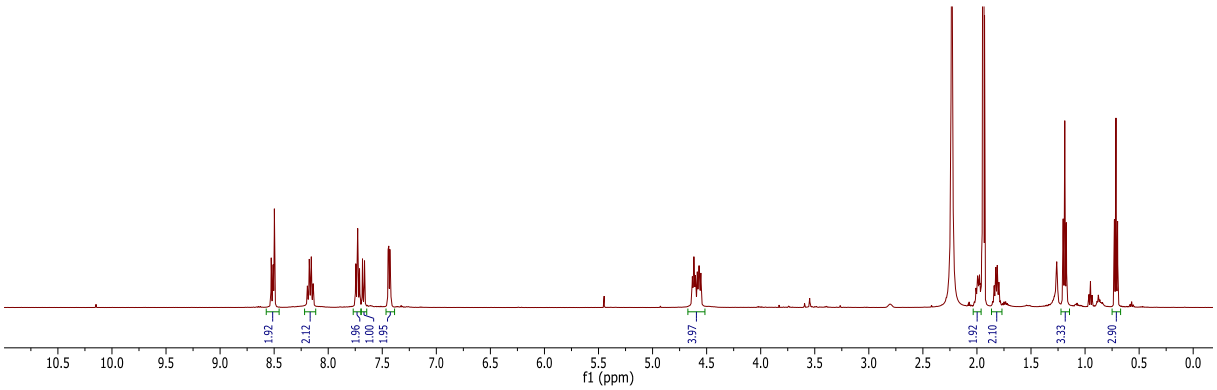
IH-DAOTA-CN



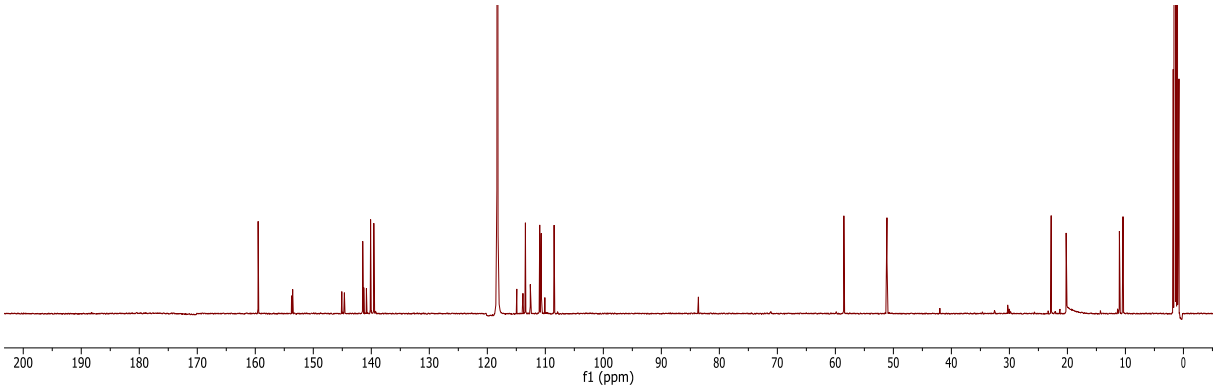
Compound 7



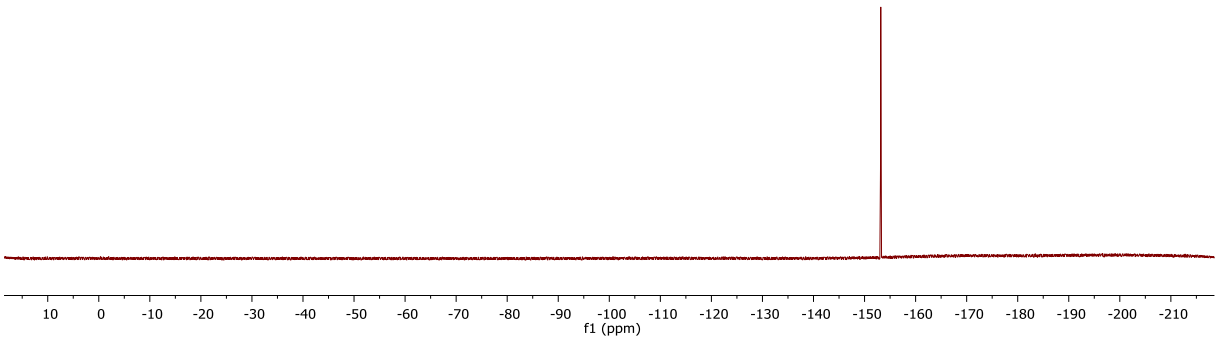
^{1}H NMR (500 MHz, CD_3CN)



^{13}C NMR (126 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



Mass Spectrometry Core Facility

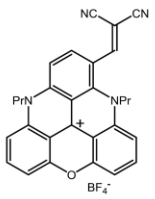
Faculty of Sciences – University of Geneva

UNIVERSITÉ DE GENÈVE
FACULTÉ DES SCIENCES

ESI-HRMS – Certificate of Analysis

Applicant:	Irene Hernandez	Date of reception:	September 25th, 2017
Group/Company:	Prof. LACOUR	Date of certificate:	September 27th, 2017
Sample name:	IH-DAOTA-KNOEV	Data filename:	SMS-XL-170926-ES-A003
Sample number:	8941	Instrument:	QSTAR XL (AB/MDS Sciex)
Analyst:	**Eliane SANDMEIER**	Ionisation mode:	ESI (positive polarity)
Contact:	**esi-hrms@unige.ch**		

Expected Formula	Ion type	Theoretical *m/z*	Observed *m/z*	Accuracy *(ppm)* [a]
$C_{29}H_{23}N_4O$	$[M]^+$	443.1866	443.1860	-1.4

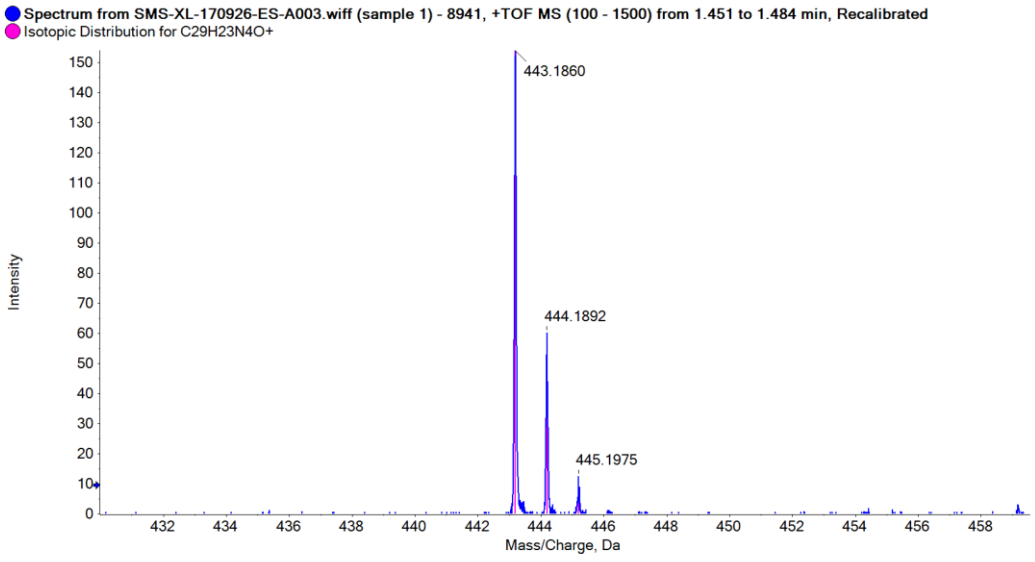


IH-DAOTA-Knoev

Chemical Formula: $C_{29}H_{23}BF_4N_4O^+$
Molecular Weight: 443.53
m/z: 443.19 (100.0%), 444.19 (31.4%), 445.19 (4.7%), 444.18 (1.5%)

[a] *Mass accuracy is determined after spectrum re-calibration (internal calibration with standards added to the FIA mobile phase)*

Recalibrated mass spectrum



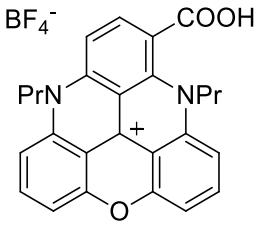
Compound 4

Compound 4

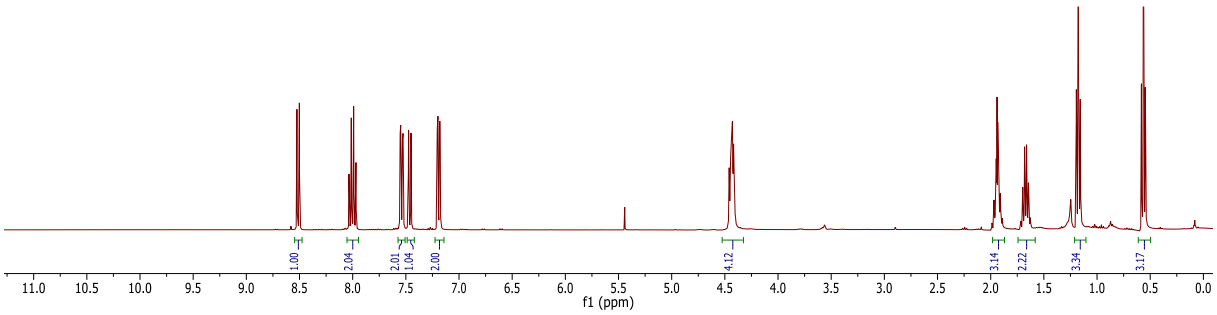
Compound 7

Compound 7

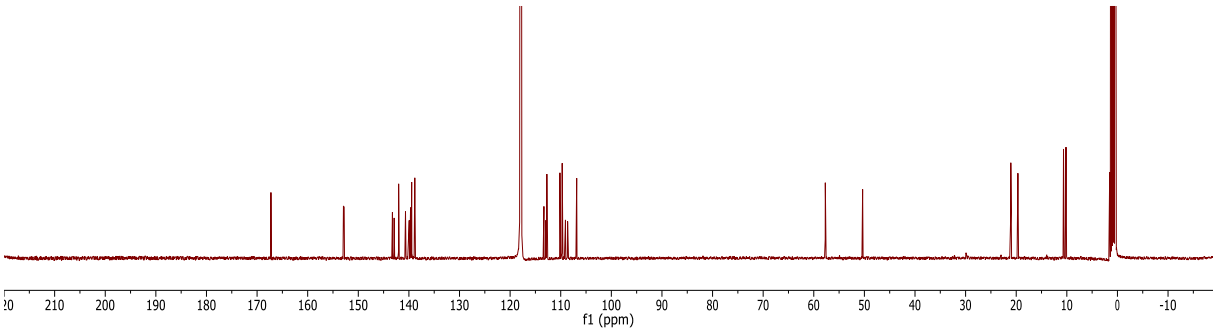
Compound 8-H$^+$



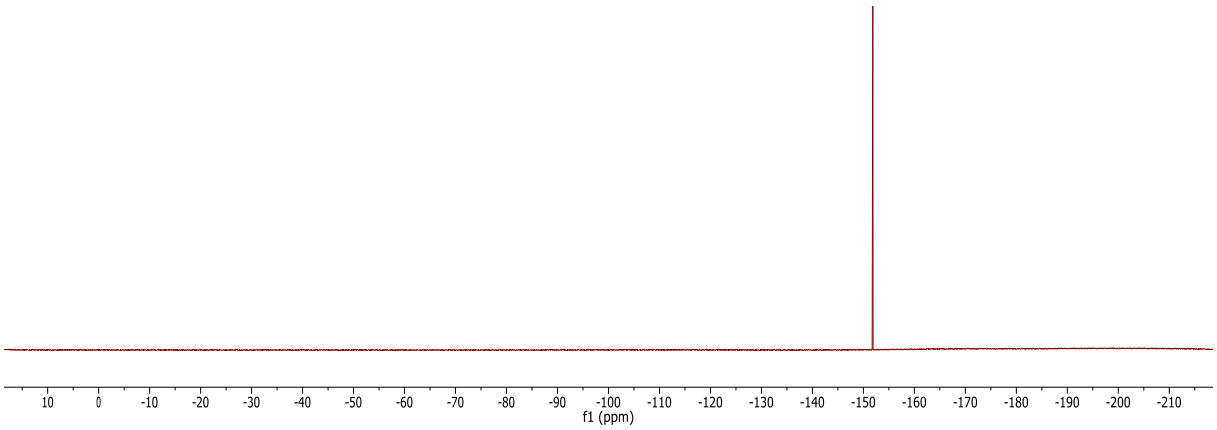
^{1}H NMR (400 MHz, CD_3CN)



^{13}C NMR (101 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



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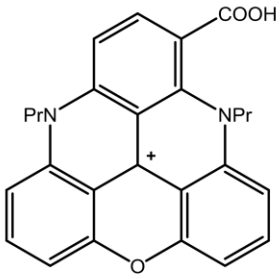
Submitter: HERNANDEZ	Date of reception: 18/10/2016
Sample name: DAOTA-COOH	Date of certificate: 07/11/2016
Sample number: 8545	Data filename: SMS10GE-161102-ES-A028
Operator: Eliane Sandmeier	Instrument: QSTAR Pulsar (AB/MDS Sciex)
Principal investigator: Dr. Sophie Michalet	Ionisation mode: ESI (positive mode)

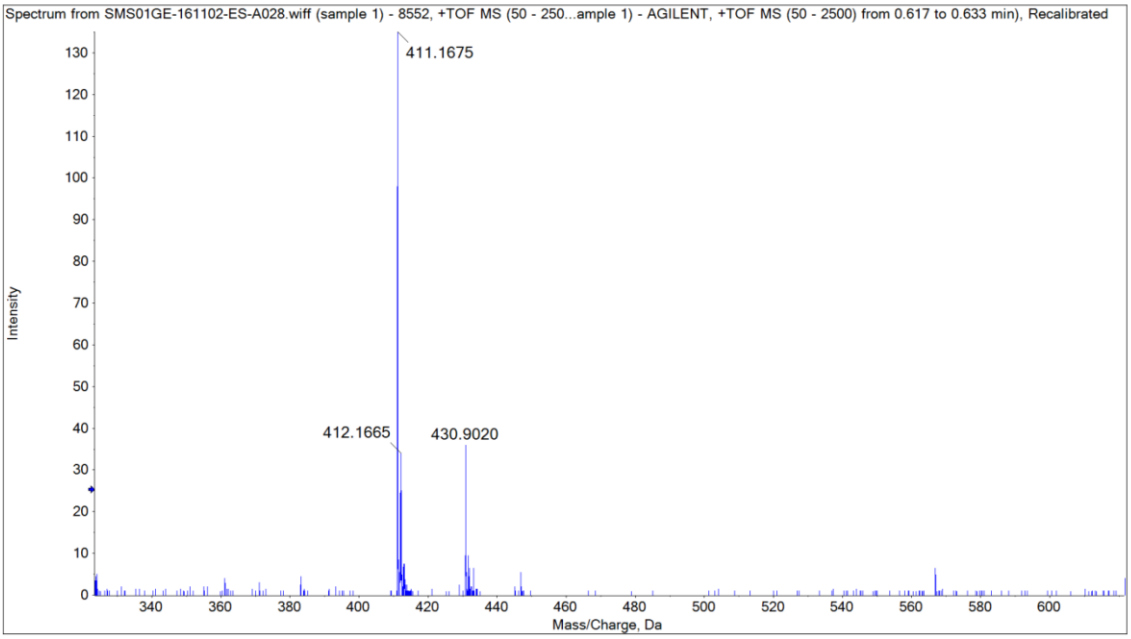
Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{26}H_{23}N_2O$	411.1675	411.1703	-2.9



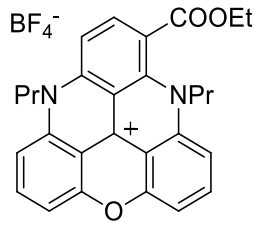
BF_4^-

Chemical Formula: $C_{26}H_{23}BF_4N_2O_3$

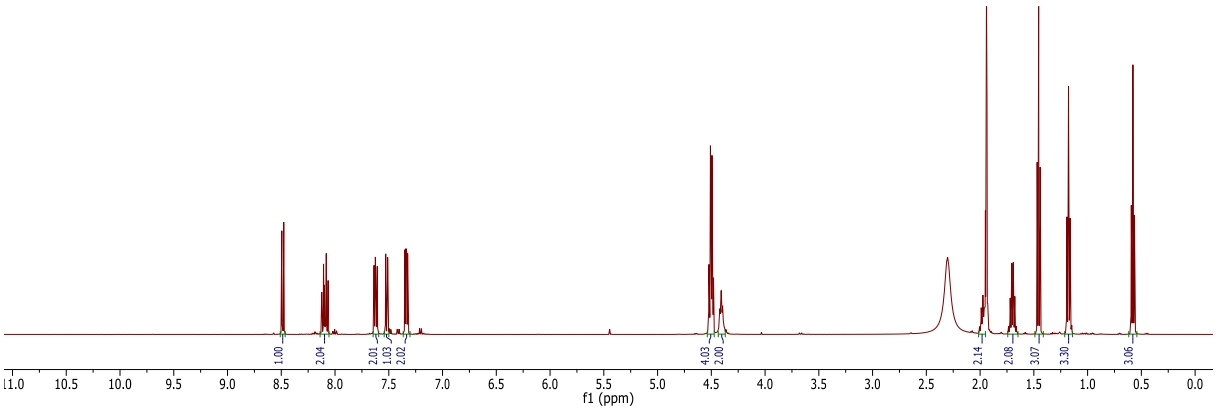
Exact Mass: 498.17



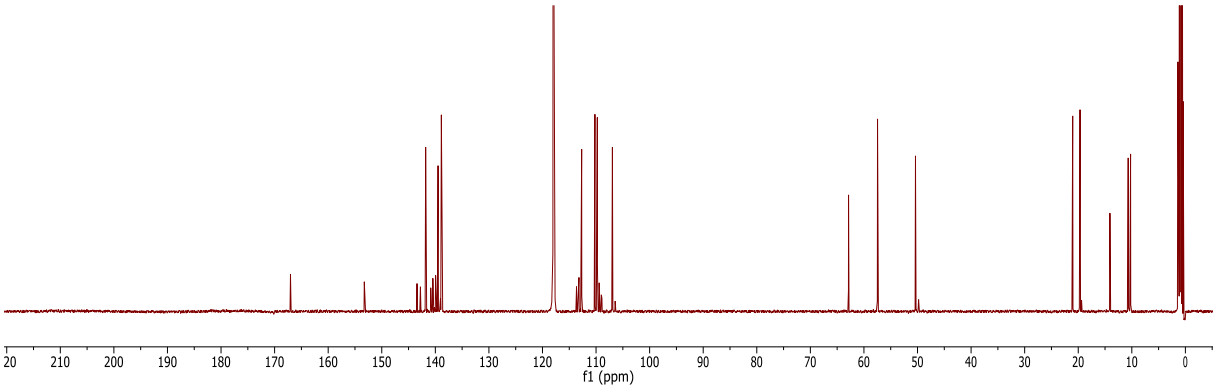
Compound 9



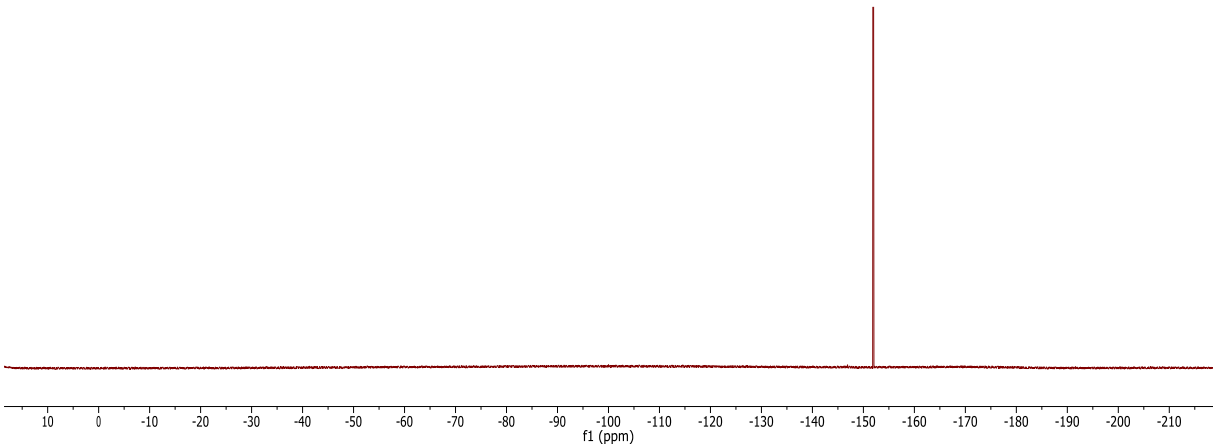
^{1}H NMR (500 MHz, CD_3CN



^{13}C NMR (126 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



Mass Spectrometry Core Facility

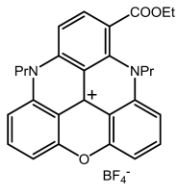
Faculty of Sciences – University of Geneva

UNIVERSITÉ DE GENÈVE
FACULTÉ DES SCIENCES

ESI-HRMS – Certificate of Analysis

Applicant:	Irene Hernandez	Date of reception:	September 25th, 2017
Group/Company:	Prof. LACOUR	Date of certificate:	September 27th, 2017
Sample name:	IH-DAOTA-CO2Et	Data filename:	SMS-XL-170926-ES-A012
Sample number:	8950	Instrument:	QSTAR XL (AB/MDS Sciex)
Analyst:	**Eliane SANDMEIER**	Ionisation mode:	ESI (positive polarity)
Contact:	**esi-hrms@unige.ch**		

Expected Formula	Ion type	Theoretical *m/z*	Observed *m/z*	Accuracy *(ppm)* [a]
$C_{24}H_{275}N_2O_3$	$[M]^+$	439.2016	439.2012	1.0

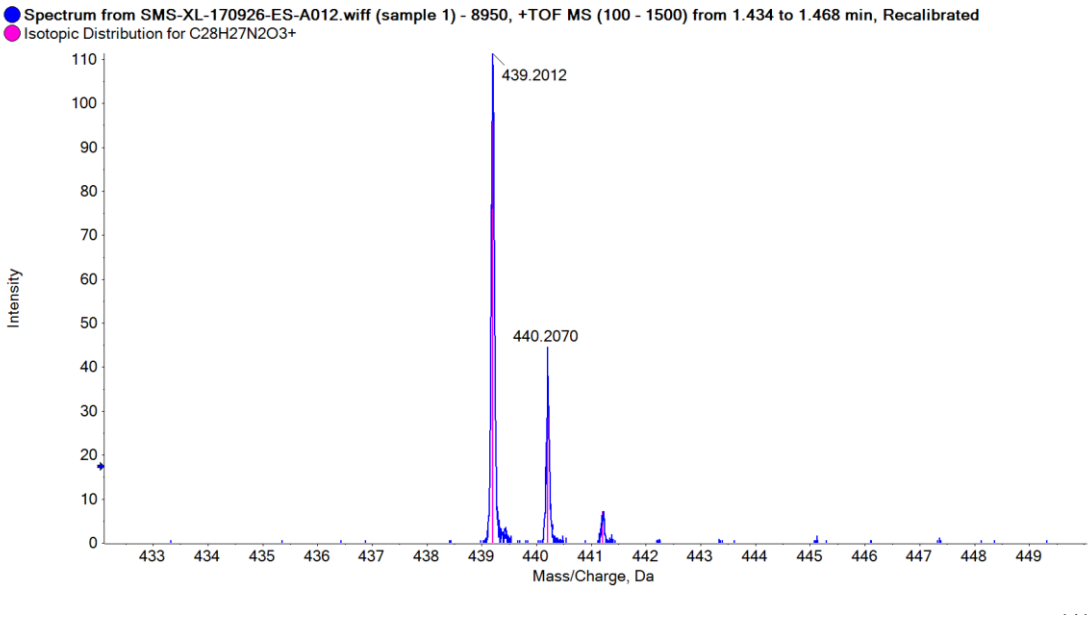


IH-DAOTA-COOEt

Chemical Formula: $C_{28}H_{27}BF_4N_2O_3^+$
Molecular Weight: 526.34
m/z: 439.20 (100.0%), 440.20 (30.3%), 441.21 (2.7%), 441.21 (1.7%)

[a] *Mass accuracy is determined after spectrum re-calibration (internal calibration with standards added to the FIA mobile phase)*

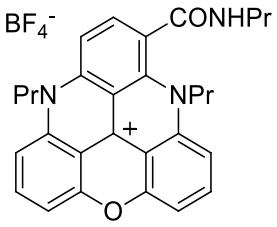
Recalibrated mass spectrum



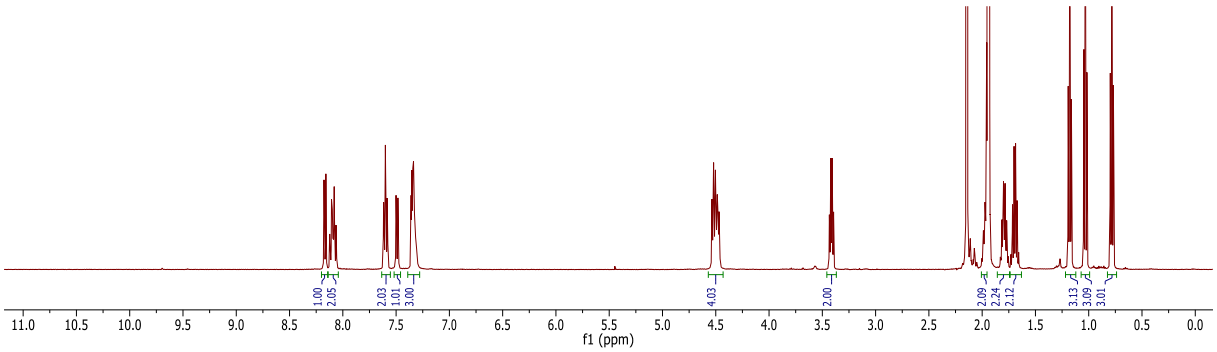
Compound 7

Compound 7

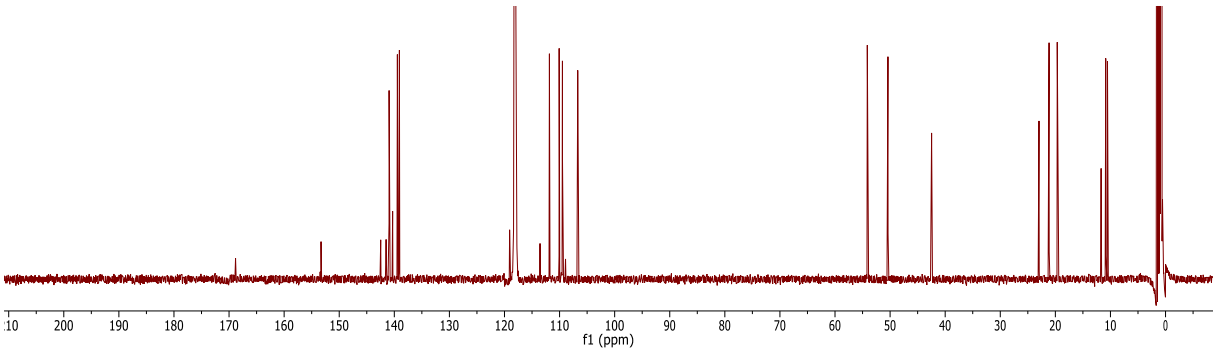
Compound 10



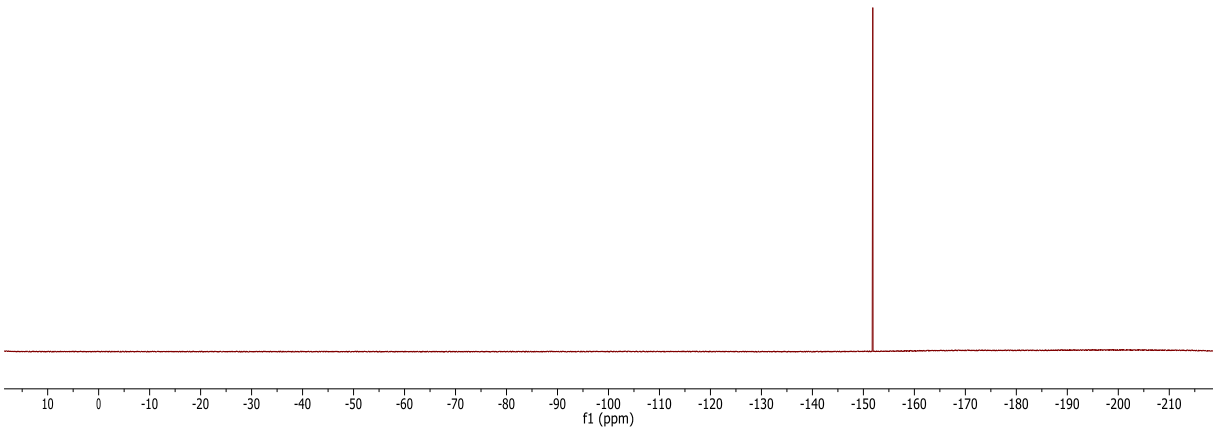
^{1}H NMR (500 MHz, CD_3CN)



^{13}C NMR (126 MHz, CD_3CN)



^{19}F NMR (282 MHz, CD_3CN)



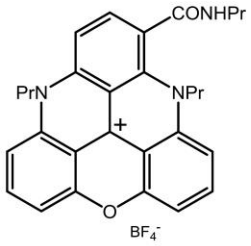
UNIVERSITY OF GENEVA

Faculty of Sciences

Sciences Mass Spectrometry

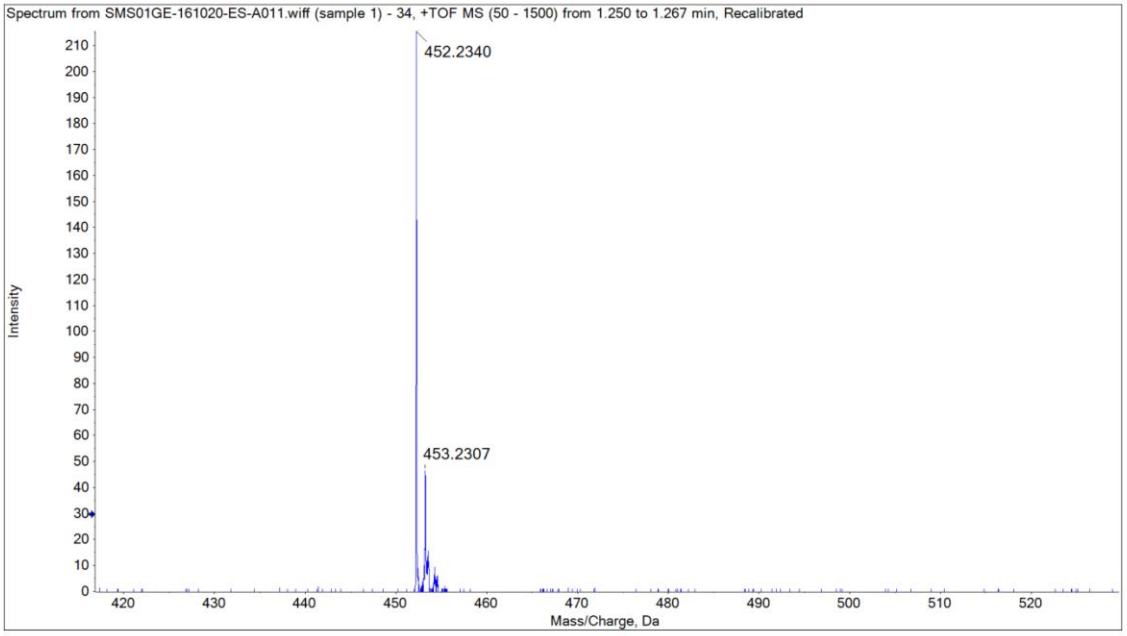
Submitter:	HERNANDEZ	Date of reception:	18/10/2016
Sample name:	DAOTA-AMIDE	Date of certificate:	26/10/2016
Sample number:	8509	Data filename:	SMS10GE-161020-ES-A011
Operator:	Eliane Sandmeier	Instrument:	QSTAR Pulsar (AB/MDS Sciex)
Principal investigator:	Dr. Sophie Michalet	Ionisation mode:	ESI (positive mode)

Expected Formula	Observed *m/z* $[M]^+$	Expected *m/z* (amu)	Accuracy (ppm)
$C_{29}H_{30}N_3O_2$	452.2340	452.2333	1.7



Chemical Formula:
$C_{29}H_{30}BF_4N_3O_2$
Exact Mass: 539.24

IH-DAOTA-CONHPr



II. X-ray diffraction

Details for the refinement for each structure can be found below. Tables of hydrogen bonds are also presented for each structure.

Compound 1

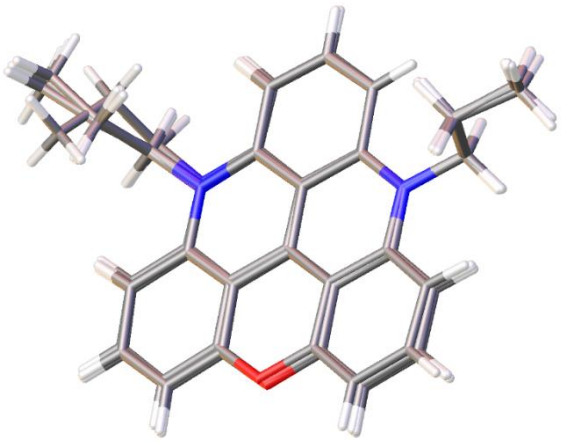


Figure S1. X-ray structure of DAOTA **1** (superimposition of the four symmetrically independent catinic triangule)

Table S2. Crystal data and structure refinement for **1**

Identification code	**ih_daota**
Empirical formula	$C_{51}H_{48}B_2Cl_2F_8N_4O_2$
Formula weight	993.45
Temperature/K	180.00(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	15.5373(3)
b/Å	25.9899(4)
c/Å	22.5918(4)
α/°	90
β/°	102.1432(17)
γ/°	90
Volume/Å^3	8918.7(3)
Z	8
ρ_{calc}g/cm^3	1.480
μ/mm^{-1}	2.015
F(000)	4112.0
Crystal size/mm^3	0.181 × 0.05 × 0.037
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.33 to 147.682

Index ranges	-17 ≤ h ≤ 19, -21 ≤ k ≤ 31, -27 ≤ l ≤ 26
Reflections collected	35452
Independent reflections	17572 [R_{int} = 0.0218, R_{sigma} = 0.0331]
Data/restraints/parameters	17572/301/1362
Goodness-of-fit on F^2	1.025
Final R indexes [I>=2σ (I)]	R_1 = 0.0785, wR_2 = 0.2273
Final R indexes [all data]	R_1 = 0.1033, wR_2 = 0.2612
Largest diff. peak/hole / e $Å^{-3}$	0.64/-1.23

Table S3. Hydrogen bonds in **1**.

donor	H	acceptor	Symmetry code for the acceptor	D…A (Å)	D-H…A angle (°)
C1S	H1SA	O1C	2555	3.508(6)	153
C1S	H1SB	F3E	4564	3.316(7)	145
C3C	H3C	F3E	3566	3.237(5)	146
C4C	H4C	F1E	3566	3.312(9)	145
C4D	H4D	F1D	2545	3.245(16)	145
C5	H5	F1C	4464	3.327(7)	140
C5C	H5C	F2F	1555	3.27(4)	145
C5D	H5D	F2C	4564	3.346(11)	154
C8	H8B	F4D	2445	3.47(2)	163
C8B	H8BB	F2E	3566	3.402(6)	153
C8B	H8BB	F3F	3566	3.44(3)	168'
C10B	H10G	F7AA	3566	3.354(5)	139
C12C	H12C	F4C	1555	3.314(13)	153
C12D	H12D	F4F	4564	3.25(3)	144
C13	H13	F4AA	3566	3.265(3)	135
C8D	H8DA	F2C	4564	3.492(12)	167
C13B	H13B	F2B	3565	3.400(8)	151
C13C	H13C	F3C	1555	3.288(10)	141
C13D	H13D	F3F	4564	3.33(2)	148
C14	H14	F6AA	3566	3.357(4)	165
C18C	H18A	F4AA	4464	3.416(3)	169
C18	H18D	F6AA	1455	3.246(4)	155
C18D	H18G	F3A	4565	3.440(6)	156
C19B	H19F	F1C	4464	3.177(7)	124
C20	H20E	F1F	1555	2.85(4)	103
C22B	H22B	F3B	1555	3.406(14)	156
C23B	H23B	F3AA	4464	3.357(4)	149

4464 = -1/2+x,3/2-y,-1/2+z
2445 = -1/2-x,-1/2+y,1/2-z
3566 = -x,1-y,1-z
1455 = -1+x,y,z
3565 = -x,1-y,-z
2545 = 1/2-x,-1/2+y,1/2-z
4565 = 1/2+x,3/2-y,1/2+z
2555 = 1/2-x,1/2+y,1/2-z

Compound 2

Table S3. Crystal data and structure refinement for **2**.

Identification code	**ih363b**
Empirical formula	$C_{25}H_{22}BF_4N_3O_3$
Formula weight	499.26
Temperature/K	179.95(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.65357(14)
b/Å	12.04731(17)
c/Å	18.5986(3)
α/°	90
β/°	94.0845(14)
γ/°	90
Volume/Å^3	2157.52(5)
Z	4
ρ_{calc}g/cm^3	1.537
μ/mm^{-1}	1.062
F(000)	1032.0
Crystal size/mm^3	0.238 × 0.1457 × 0.1311
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.752 to 146.75
Index ranges	-7 ≤ h ≤ 11, -14 ≤ k ≤ 14, -21 ≤ l ≤ 23
Reflections collected	14409
Independent reflections	4274 [R_{int} = 0.0177, R_{sigma} = 0.0191]
Data/restraints/parameters	4274/193/439
Goodness-of-fit on F^2	1.051
Final R indexes [I>=2σ (I)]	R_1 = 0.0451, wR_2 = 0.1184
Final R indexes [all data]	R_1 = 0.0491, wR_2 = 0.1228
Largest diff. peak/hole / e Å^{-3}	0.46/-0.41

Table S4. Hydrogen bonds in **2**.

donor	H	acceptor	Symmetry code for the acceptor	D…A (Å)	D-H…A angle (°)
C(5A)	H(5A)	F(5B)	1655	2.936(7)	128
C(16)	H(16A)	O(27)		2.800(2)	122
C(17)	H(17A)	N(25)		3.110(2)	127
C(17)	H(17B)	F(3)	1555	3.264(2)	140
C(17)	H(17C)	F(3)	1555	3.264(2)	140
C(17)	H(17D)	N(25)		3.110(2)	125
C(18A)	H(18C)	F(5A)	2646	3.039(10)	117
C(18A)	H(18F)	F(5A)	2646	3.039(10)	156
C(22A)	H(22A)	F(1)	2746	3.300(8)	154
C(22A)	H(22B)	F(1)	2746	3.300(8)	150
C(29B)	H(30A)	O(26)	2756	3.603(3)	138
C(29A)	H(30C)	F(5A)	1655	3.465(6)	164
C(29A)	H(30C)	F(5B)	1655	3.364(5)	165

2756 = 2-x,1/2+y,3/2-z
1655 = 1+x,y,z
2646 = 1-x,-1/2+y,3/2-z
2746 = 2-x,-1/2+y,3/2-z

Compound 3

Table S5. Crystal data and structure refinement for **3**.

Identification code	**daota_CHO**
Empirical formula	$C_{26}H_{23}BF_4N_2O_2$
Formula weight	482.27
Temperature/K	180.01(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	7.8647(3)
b/Å	21.0247(5)
c/Å	13.4118(4)
α/°	90
β/°	105.170(3)
γ/°	90
Volume/Å^3	2140.40(12)
Z	4
ρ_{calc}g/cm^3	1.497
μ/mm^{-1}	0.998
F(000)	1000.0
Crystal size/mm^3	0.177 × 0.073 × 0.025
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.02 to 147.508
Index ranges	-9 ≤ h ≤ 9, -26 ≤ k ≤ 26, -16 ≤ l ≤ 16
Reflections collected	20330
Independent reflections	4293 [R_{int} = 0.0305, R_{sigma} = 0.0219]
Data/restraints/parameters	4293/102/355
Goodness-of-fit on F^2	1.058
Final R indexes [I>=2σ (I)]	R_1 = 0.0711, wR_2 = 0.2063
Final R indexes [all data]	R_1 = 0.0873, wR_2 = 0.2207
Largest diff. peak/hole / e Å^{-3}	0.35/-0.48

Table S6. Hydrogen bonds in **3**.

donor	H	acceptor	Symmetry code for the acceptor	D…A (Å)	D-H…A angle (°)
C(4)	H(4)	F(1A)	1555	3.030(9)	130
C(4)	H(4)	F(1B)	1555	3.051(11)	133
C(5)	H(5)	O(18)	2655	3.109(4)	138
C(9)	H(9B)	F(4B)	4564	3.267(10)	133
C(15)	H(15)	O(18)		2.774(5)	102
C(21)	H(21A)	O(18)	4555	3.354(5)	141
C(24)	H(24)	F(2B)	2746	3.303(9)	144
C(25)	H(25)	F(4B)	2746	3.293(10)	152

2655 = 1-x,1/2+y,1/2-z 4555 = x,1/2-y,1/2+z

4564 = x,3/2-y,-1/2+z 2746 = 2-x,-1/2+y,3/2-z

Compound 4

Table S7. Crystal data and structure refinement for **4**.

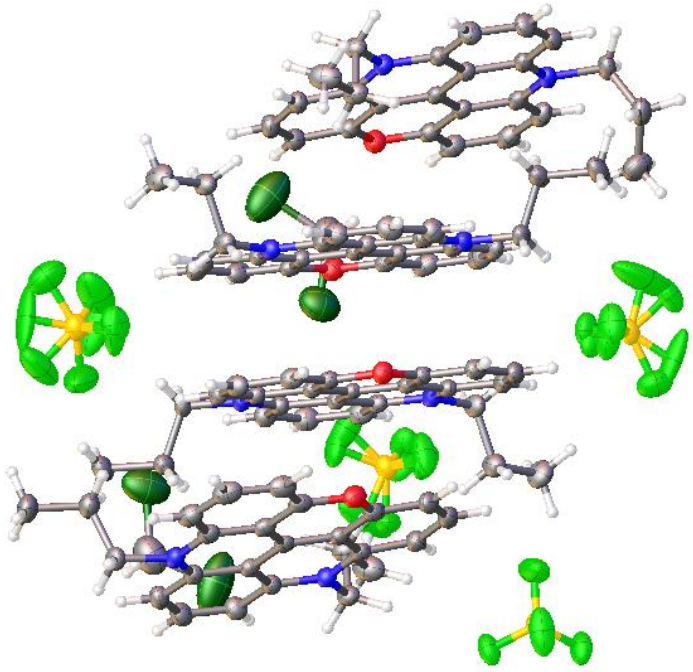
Identification code	**irene**
Empirical formula	$C_{25}H_{24}BF_4N_3O$
Formula weight	469.28
Temperature/K	180.15
Crystal system	triclinic
Space group	P-1
a/Å	9.5617(6)
b/Å	10.7543(6)
c/Å	10.8834(6)
α/°	79.037(5)
β/°	74.517(5)
γ/°	85.404(5)
Volume/Å^3	1058.33(11)
Z	2
ρ_{calc}g/cm^3	1.473
μ/mm^{-1}	0.968
F(000)	488.0
Crystal size/mm^3	0.176 × 0.071 × 0.059
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.378 to 147.412
Index ranges	-11 ≤ h ≤ 11, -13 ≤ k ≤ 13, -13 ≤ l ≤ 13
Reflections collected	15797
Independent reflections	4202 [R_{int} = 0.0246, R_{sigma} = 0.0208]
Data/restraints/parameters	4202/0/317
Goodness-of-fit on F^2	1.036
Final R indexes [I>=2σ (I)]	R_1 = 0.0492, wR_2 = 0.1282
Final R indexes [all data]	R_1 = 0.0607, wR_2 = 0.1385
Largest diff. peak/hole / e Å^{-3}	0.55/-0.42

Table S8. Hydrogen bonds in **4**.

donor	H	acceptor	Symmetry code for the acceptor	D…A (Å)	D-H…A angle (°)
N(21)	H(21A)	F(4)	2765	3.052(2)	161
N(21)	H(21B)	F(4)	1554	3.067(2)	159
C(11)	H(11)	F(1)	2666	3.351(2)	160
C(16)	H(16A)	F(3)	1554	3.441(3)	154
C(16)	H(16B)	N(21)		2.919(3)	130
C(22)	H(22)	F(2)	2765	3.455(3)	168

1554 = x,y,-1+z
2765 = 2-x,1-y,-z
2666 = 1-x,1-y,1-z

Compound 1

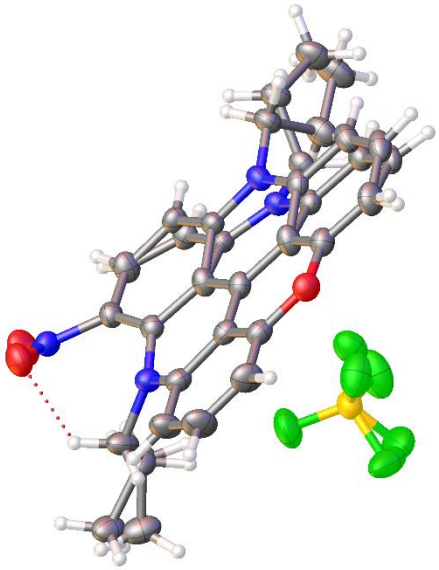


View of the asymmetric unit

The crystal was very small so that the diffraction data are weak. Three of the tetrafluoroborate ions are disordered and were refined using eight fluorine atoms each. Restraints were applied on 1-2 and 1-3 distances (SADI) as well as on anisotropic displacement parameters.

Compound	**1 (H)**
CCDC	**1824707**
Formula	$C_{51}H_{48}B_2Cl_2F_8N_4O_2$
$D_{calc.}$/ g cm^{-3}	1.480
μ/mm^{-1}	2.015
Formula Weight	993.45
Colour	red
Shape	prism
Size/mm^3	0.18×0.05×0.04
T/K	180.00(10)
Crystal System	monoclinic
Space Group	$P2_1/n$
a/Å	15.5373(3)
b/Å	25.9899(4)
c/Å	22.5918(4)
α/°	90
β/°	102.1432(17)
γ/°	90
V/Å^3	8918.7(3)
Z	8
Z'	2
Wavelength/Å	1.54184
Radiation type	CuK_α
Θ_{min}/°	3.165
Θ_{max}/°	73.841
Measured Refl.	35452
Independent Refl.	17572
Reflections Used	12240
R_{int}	0.0218
Parameters	1362
Restraints	301
Largest Peak	0.639
Deepest Hole	-1.230
GooF	1.025
wR_2 (all data)	0.2612
wR_2	0.2273
R_1 (all data)	0.1033
R_1	0.0785

Compound 2

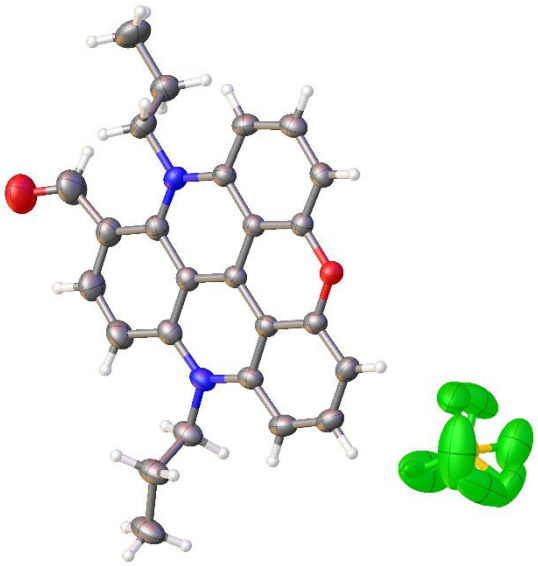


View of the asymmetric unit

The cation is disordered and part of it was refined using 2 components. Geometrical restraints (SADI on 1-2 distances) and restraints on anisotropic displacement parameters were used. A small disorder on the other alkyl arm was also modelled with restrains on 1-2 and 1-3 distances. The BF4- anion is also disordered and was refined as 2 parts.

Compound	**2 (NO_2)**
CCDC	**1443634**
Formula	$C_{25}H_{22}BF_4N_3O_3$
$D_{calc.}$/ g cm^{-3}	1.537
μ/mm^{-1}	1.062
Formula Weight	499.26
Colour	red
Shape	prism
Size/mm^3	0.24×0.15×0.13
T/K	179.95(10)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	9.65357(14)
b/Å	12.04731(17)
c/Å	18.5986(3)
α/°	90
β/°	94.0845(14)
γ/°	90
V/Å^3	2157.52(5)
Z	4
Z'	1
Wavelength/Å	1.54184
Radiation type	CuK_α
Θ_{min}/°	4.376
Θ_{max}/°	73.375
Measured Refl.	14409
Independent Refl.	4274
Reflections Used	3892
R_{int}	0.0177
Parameters	439
Restraints	193
Largest Peak	0.460
Deepest Hole	-0.415
GooF	1.035
wR_2 (all data)	0.1236
wR_2	0.1191
R_1 (all data)	0.0491
R_1	0.0451

Compound 3

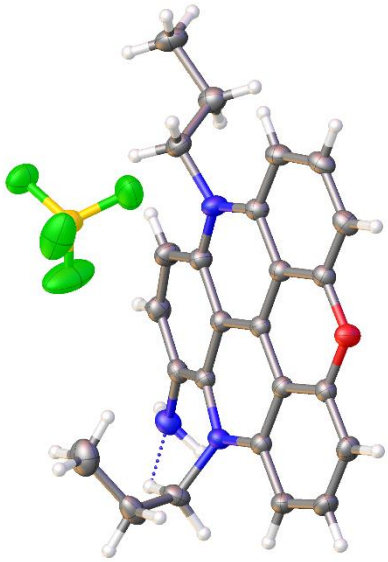


View of the asymmetric unit

The tetrafluoroborate anion is disordered and was modelled using eight fluorine atoms. Restraints (SADI) were used on B-F and F-F distances. The anisotropic displacement parameters were also restrained (RIGU).

Compound	**3 (CHO)**
CCDC	**1824709**
Formula	$C_{26}H_{23}BF_4N_2O_2$
$D_{calc.}$/ g cm^{-3}	1.497
μ/mm^{-1}	0.998
Formula Weight	482.27
Colour	red
Shape	prism
Size/mm^3	0.18×0.07×0.03
T/K	180.01(10)
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	7.8647(3)
b/Å	21.0247(5)
c/Å	13.4118(4)
α/°	90
β/°	105.170(3)
γ/°	90
V/Å^3	2140.40(12)
Z	4
Z'	1
Wavelength/Å	1.54184
Radiation type	CuK_α
Θ_{min}/°	4.010
Θ_{max}/°	73.754
Measured Refl.	20330
Independent Refl.	4293
Reflections Used	3319
R_{int}	0.0305
Parameters	355
Restraints	102
Largest Peak	0.353
Deepest Hole	-0.481
GooF	1.058
wR_2 (all data)	0.2209
wR_2	0.2065
R_1 (all data)	0.0873
R_1	0.0711

Compound 4



View of the asymmetric unit

Compound	**4 (NH_2)**
CCDC	**1824706**
Formula	$C_{25}H_{24}BF_4N_3O$
$D_{calc.}$/ g cm^{-3}	1.473
μ/mm^{-1}	0.968
Formula Weight	469.28
Colour	red
Shape	prism
Size/mm^3	0.18×0.07×0.06
T/K	180.15
Crystal System	triclinic
Space Group	P-1
a/Å	9.5617(6)
b/Å	10.7543(6)
c/Å	10.8834(6)
α/°	79.037(5)
β/°	74.517(5)
γ/°	85.404(5)
V/Å^3	1058.33(11)
Z	2
Z'	1
Wavelength/Å	1.54184
Radiation type	CuK_α
Θ_{min}/°	4.189
Θ_{max}/°	73.706
Measured Refl.	15797
Independent Refl.	4202
Reflections Used	3417
R_{int}	0.0246
Parameters	317
Restraints	0
Largest Peak	0.545
Deepest Hole	-0.425
GooF	1.035
wR_2 (all data)	0.1385
wR_2	0.1282
R_1 (all data)	0.0607
R_1	0.0492