Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Unexpected formation of [5]-helicenes from hexaarylbenzenes containing pyrrole moieties

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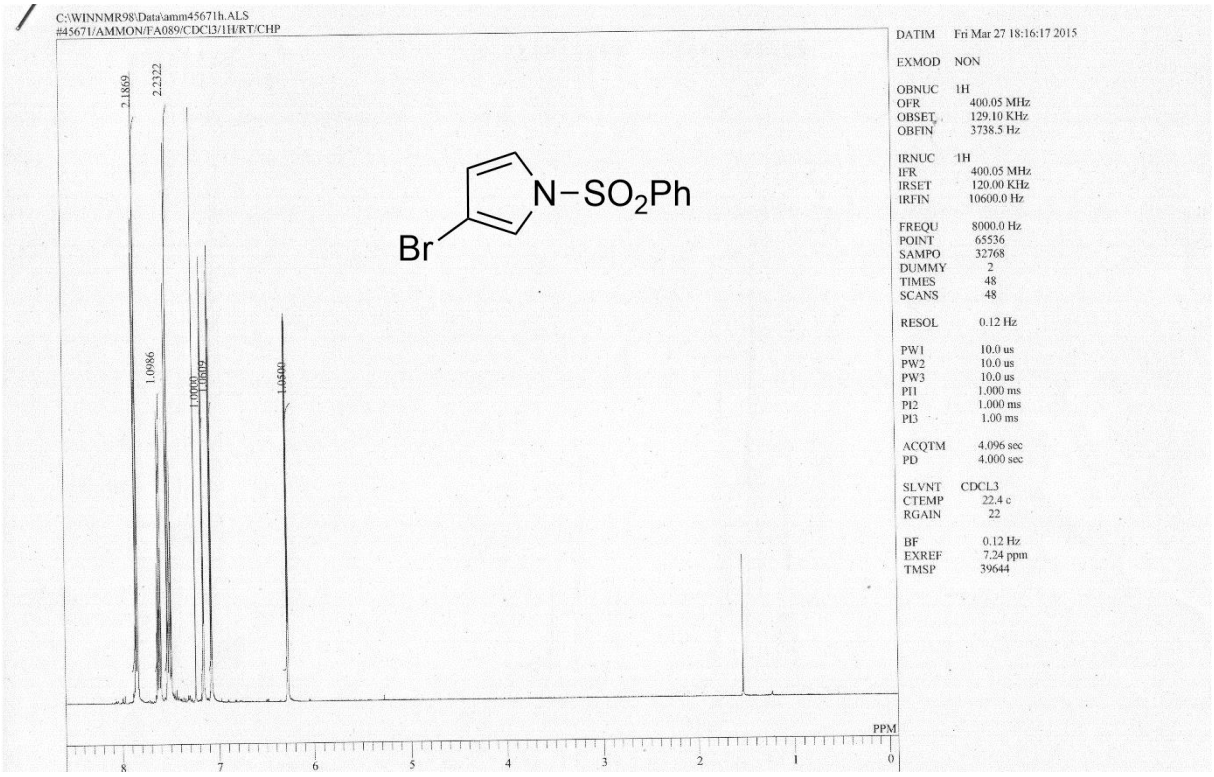
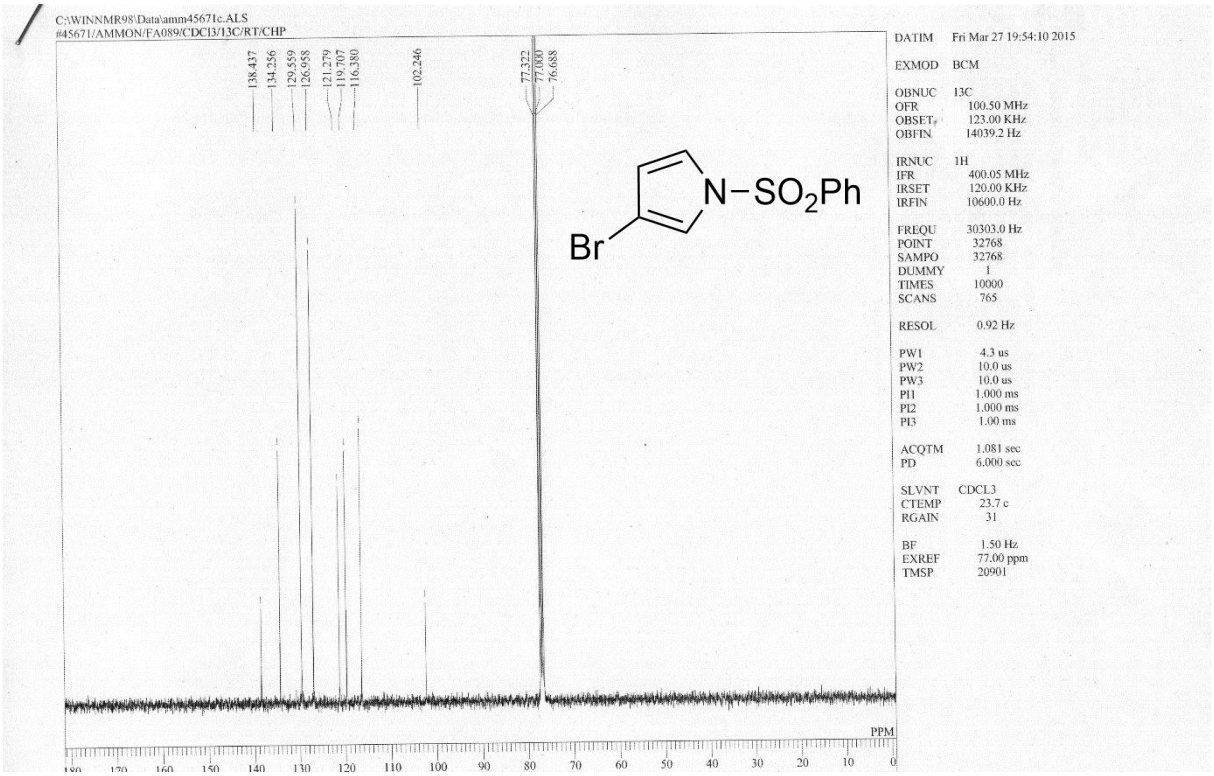
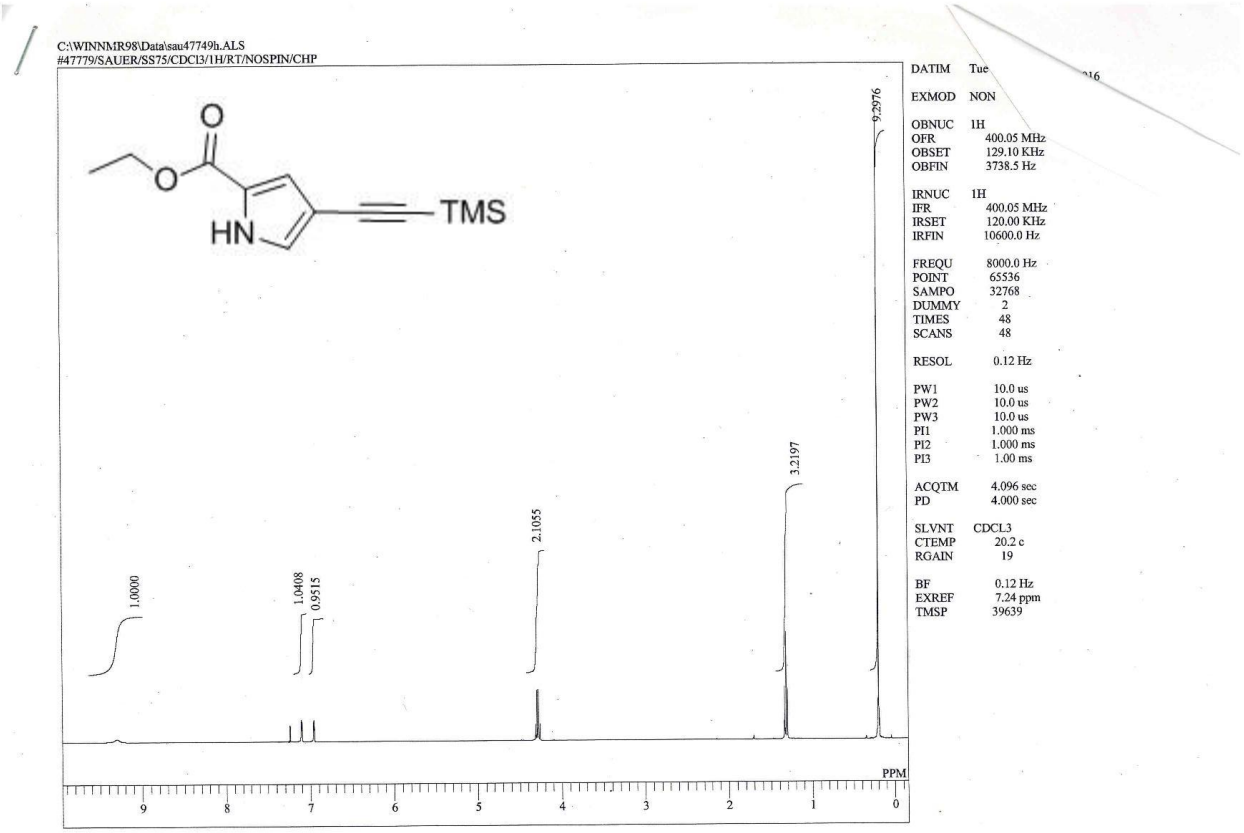
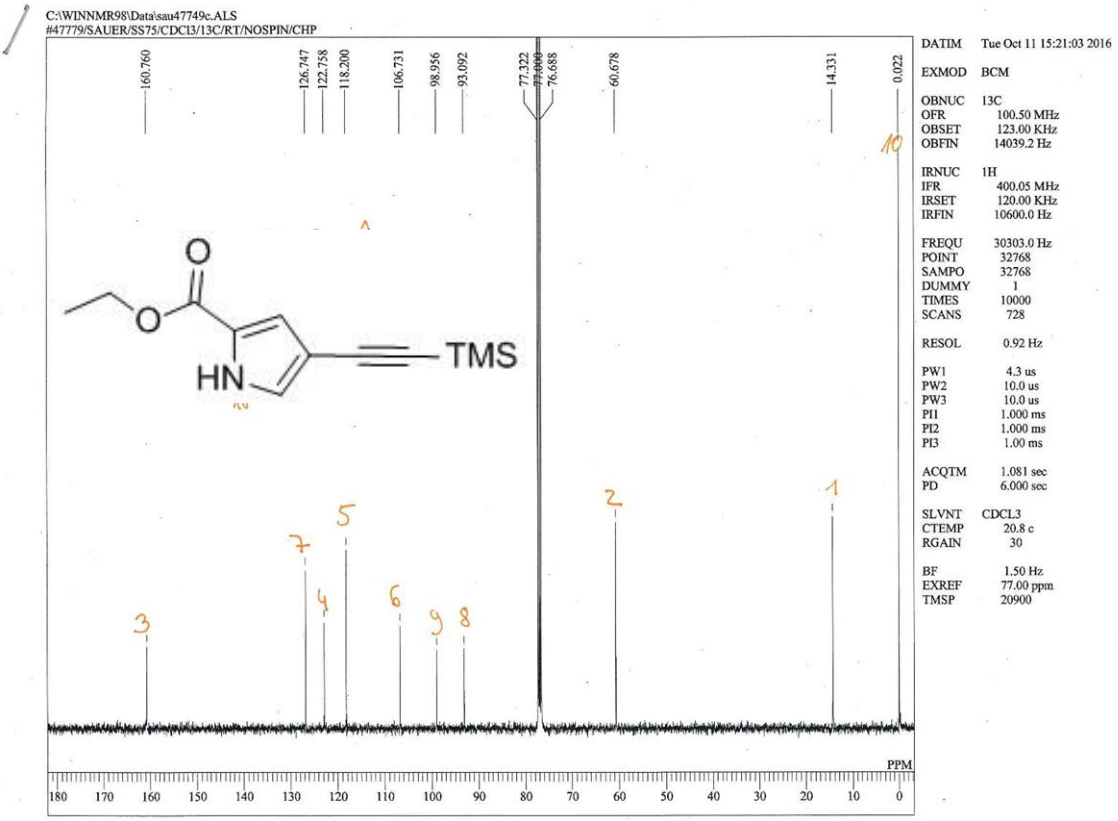
^{1}H and ^{13}C-NMR data for compound **3**





^{1}H and ^{13}C-NMR data for compound **9**





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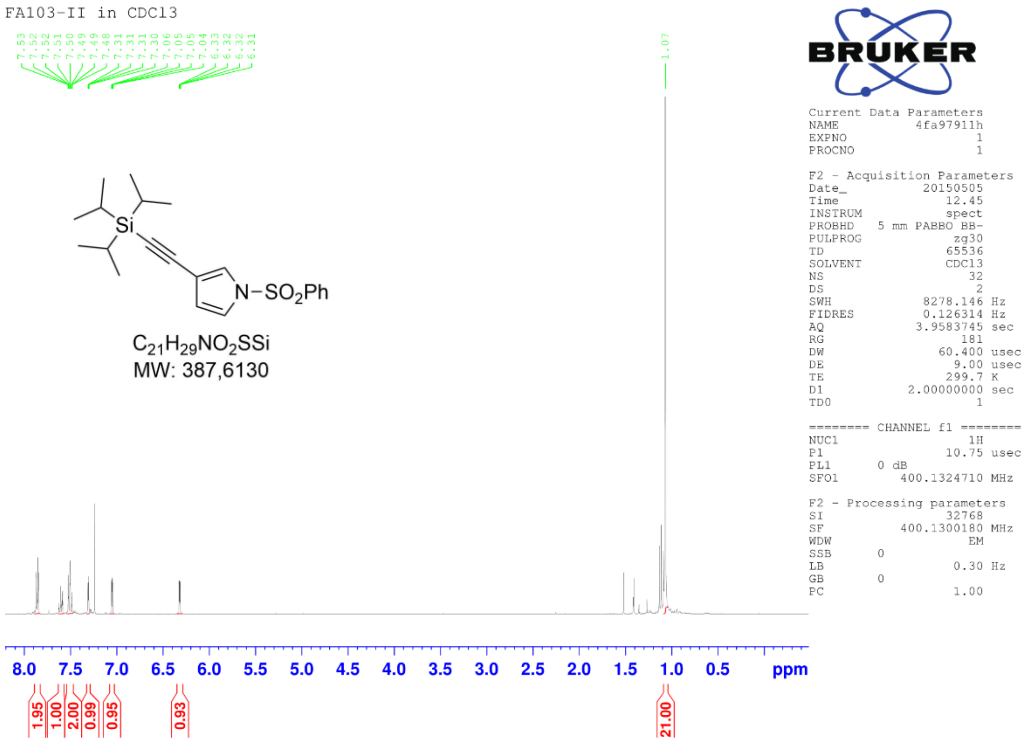
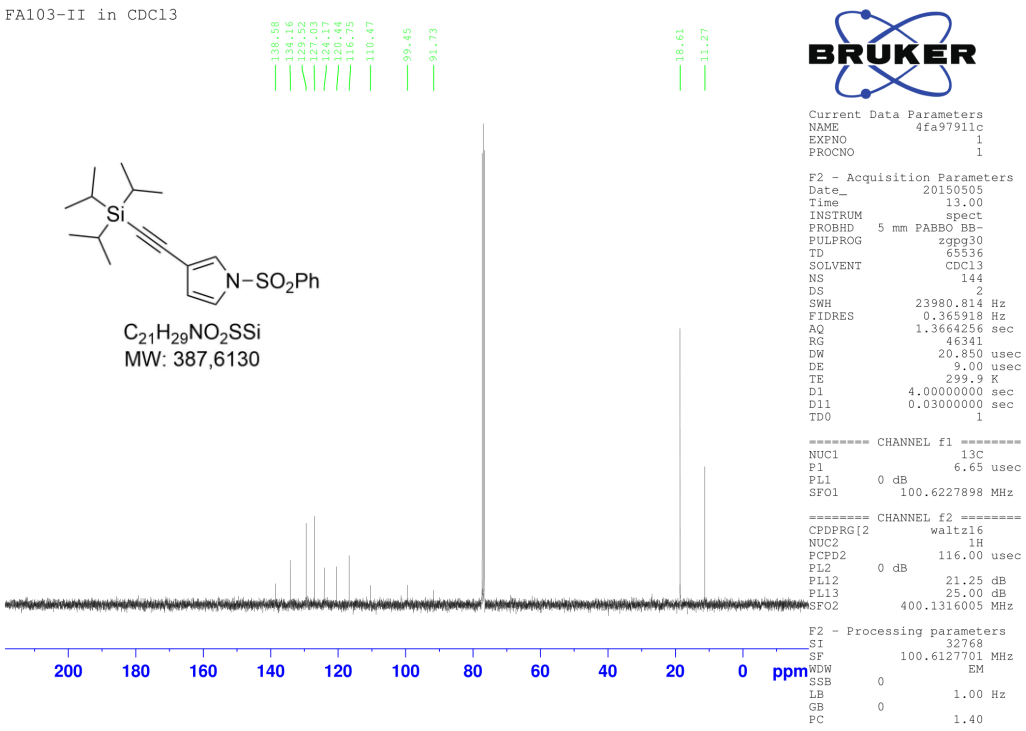
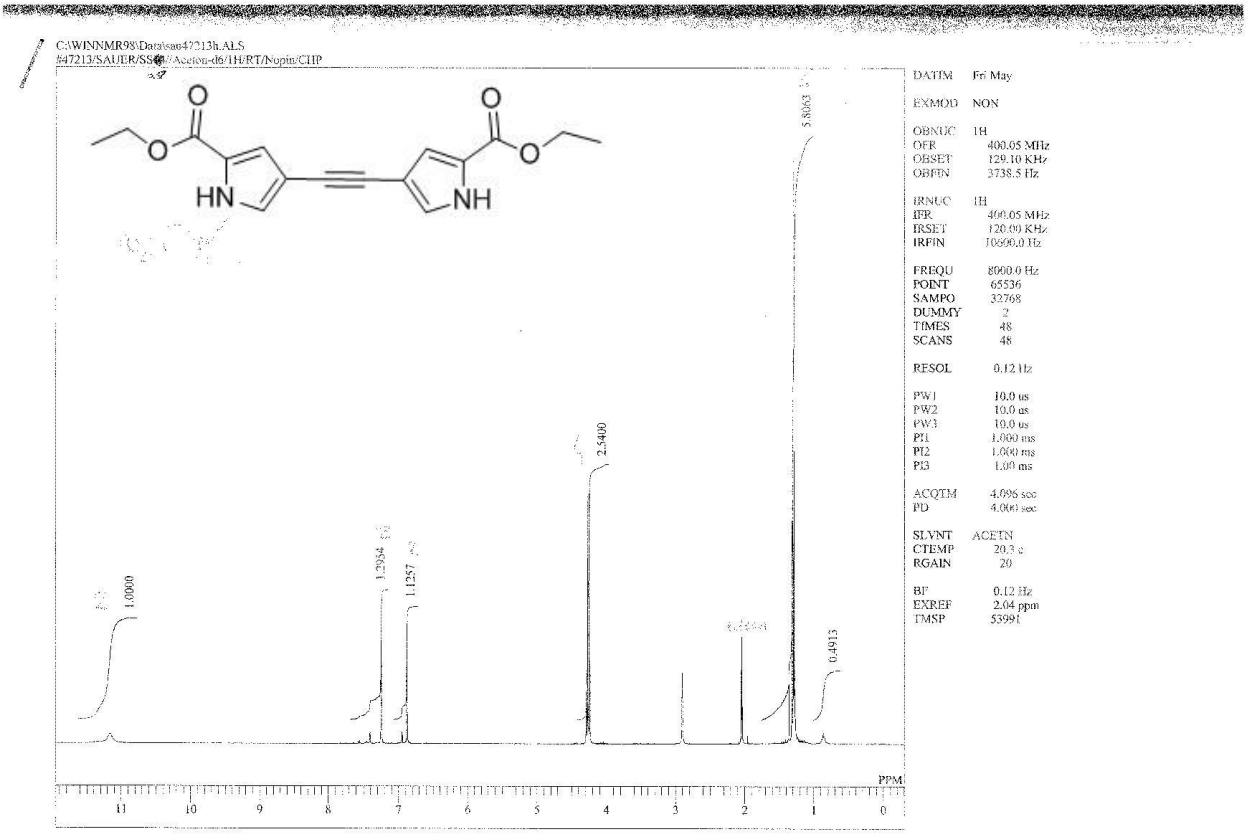
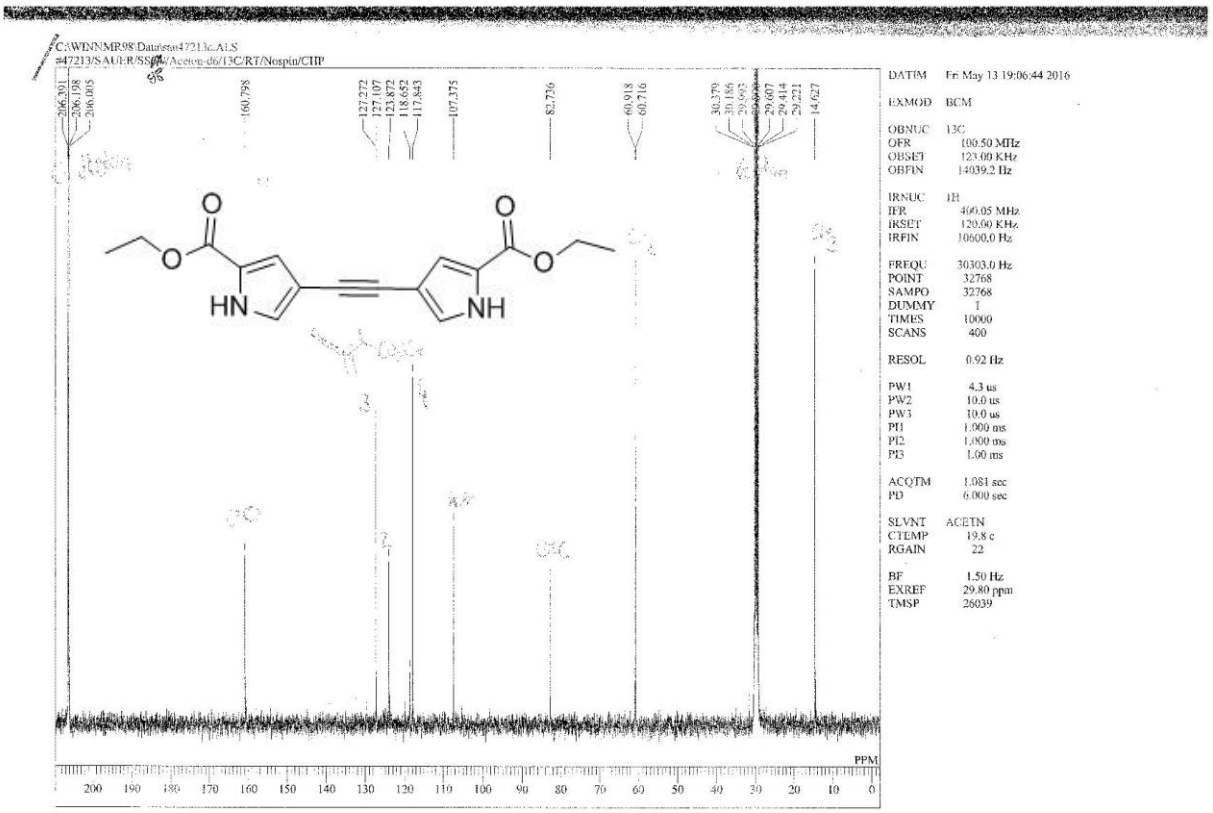


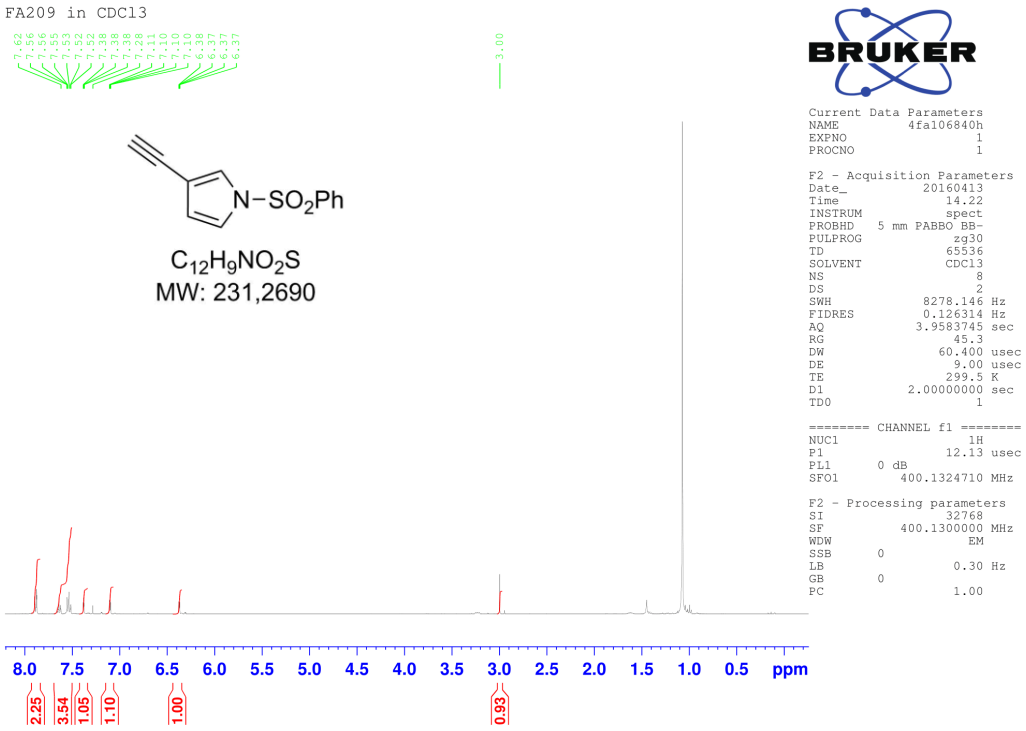


^{1}H and ^{13}C-NMR data for compound **11**





^{1}H NMR data for compound **5**

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PROCNO                   1

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PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9583745 sec
RG                   45.3
DW                 60.400 usec
DE                  9.00 usec
TE                 299.5 K
D1              2.00000000 sec
TD0                      1

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PL1        0 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
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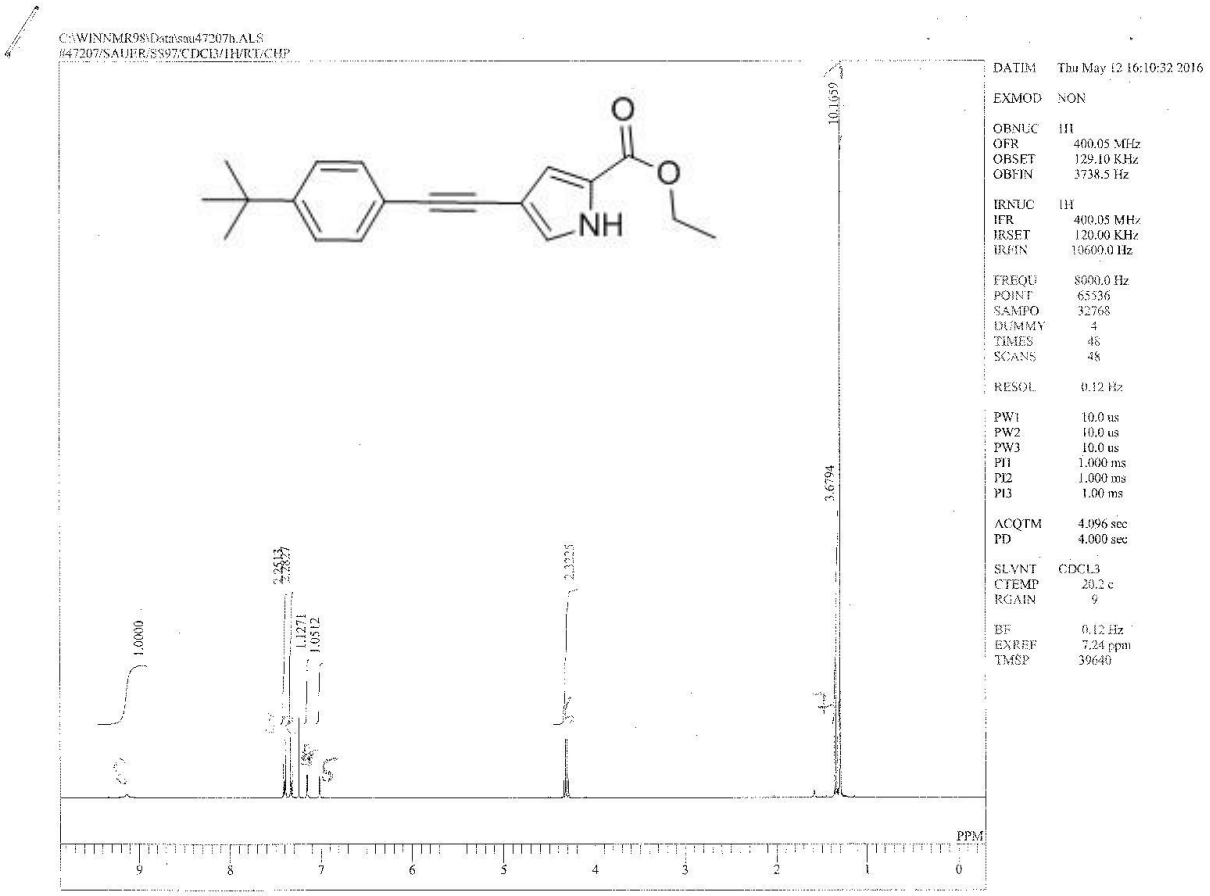

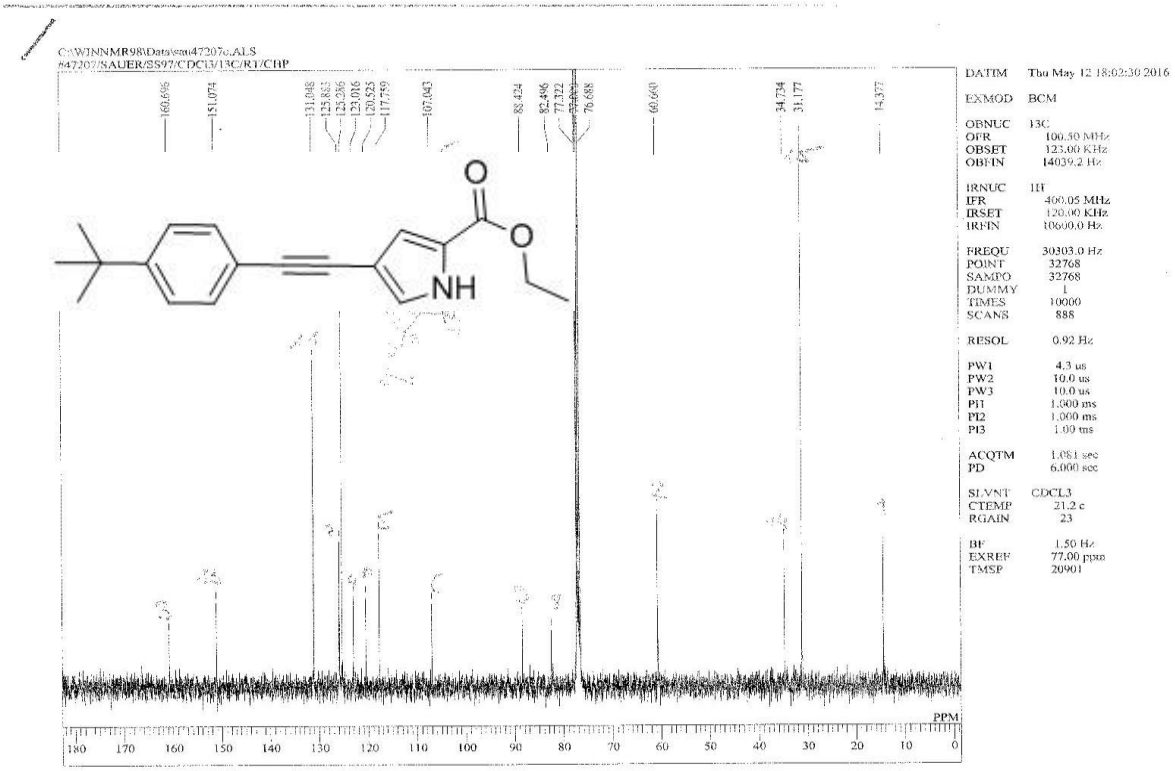

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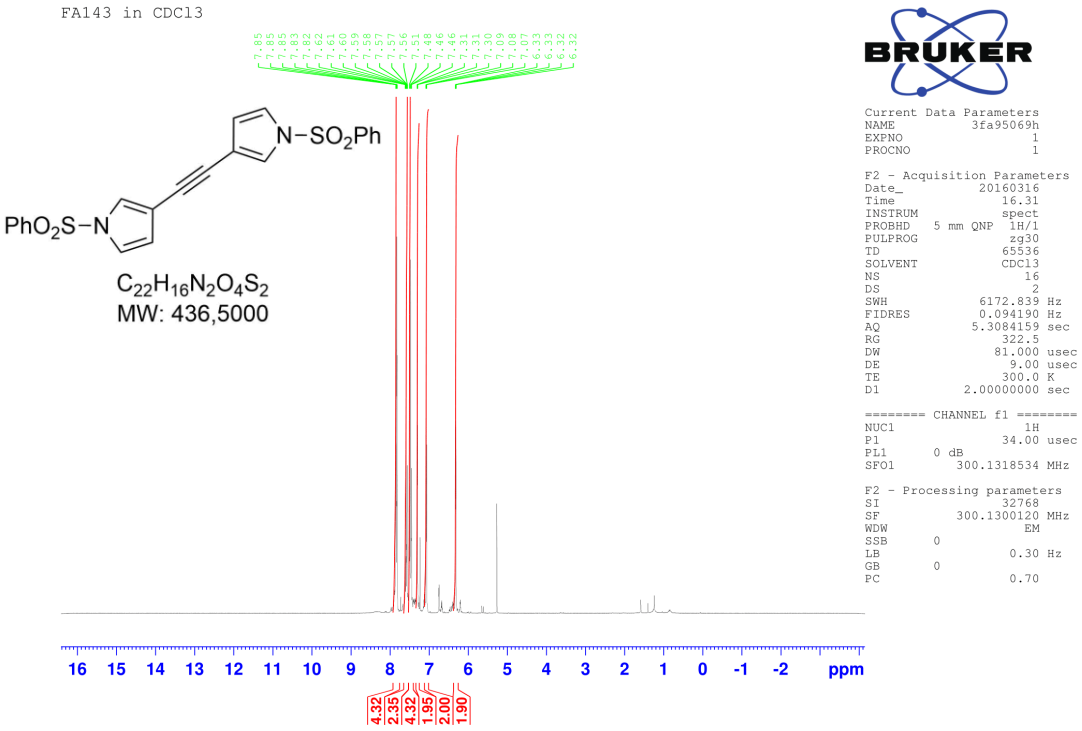

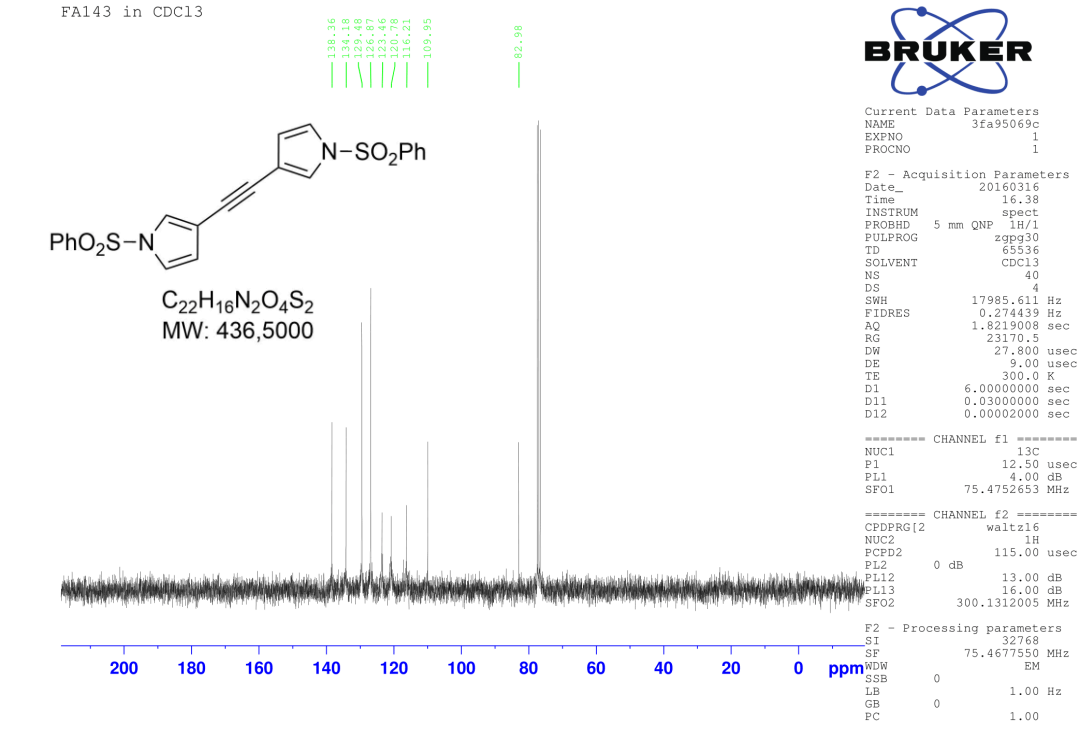

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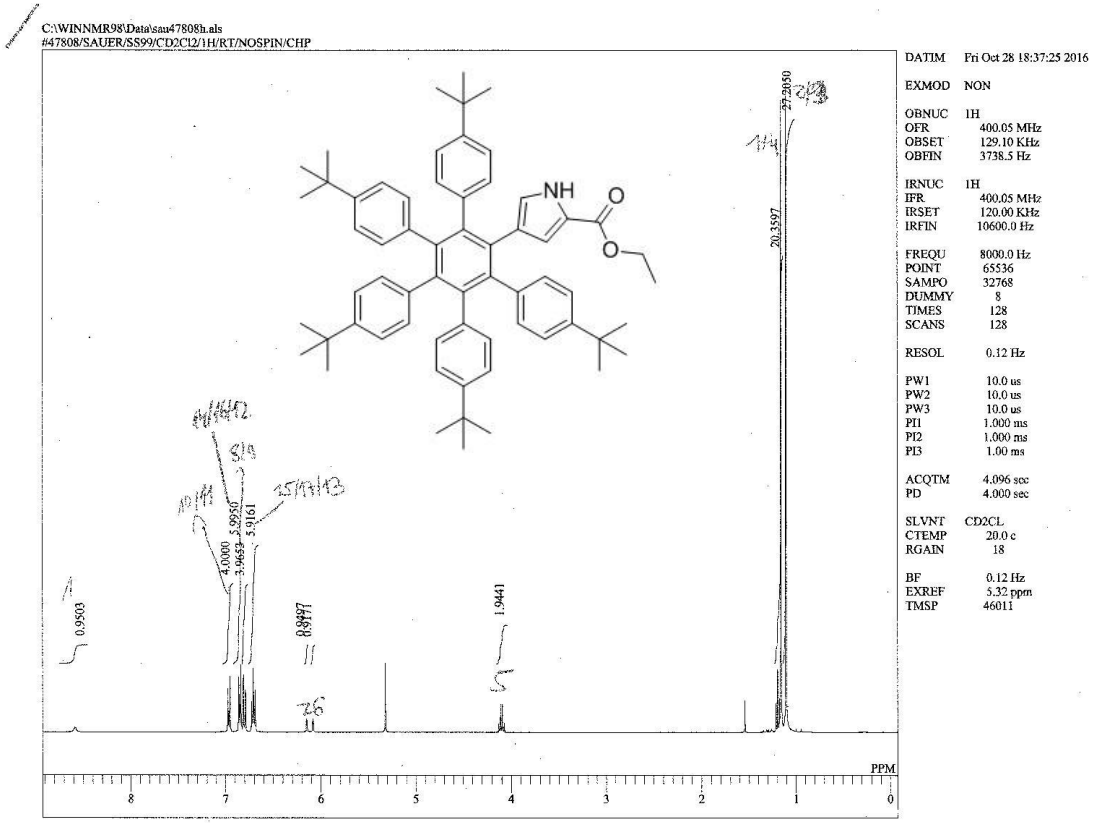
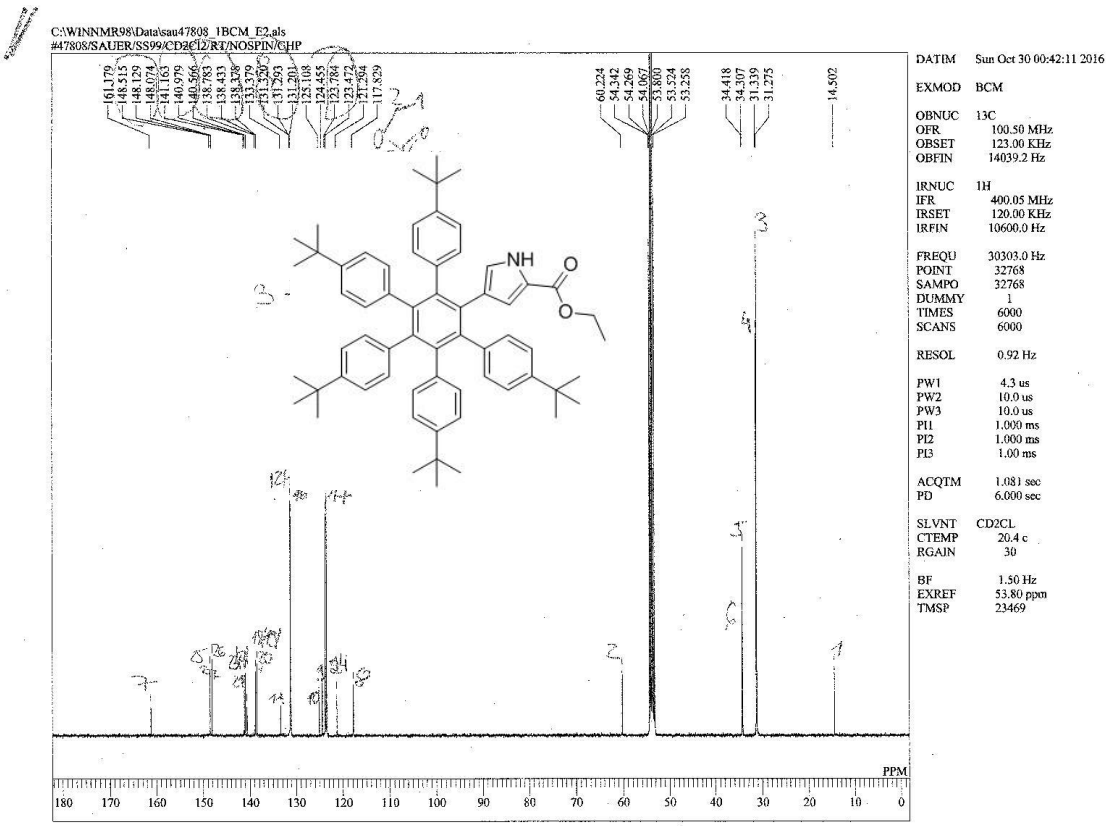
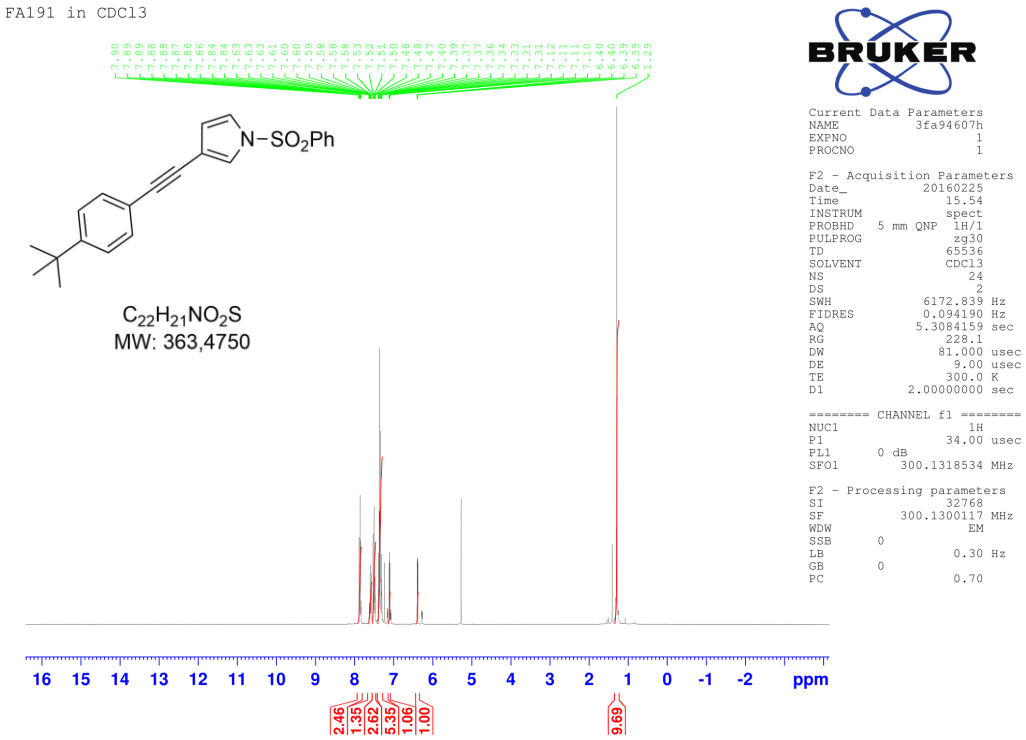
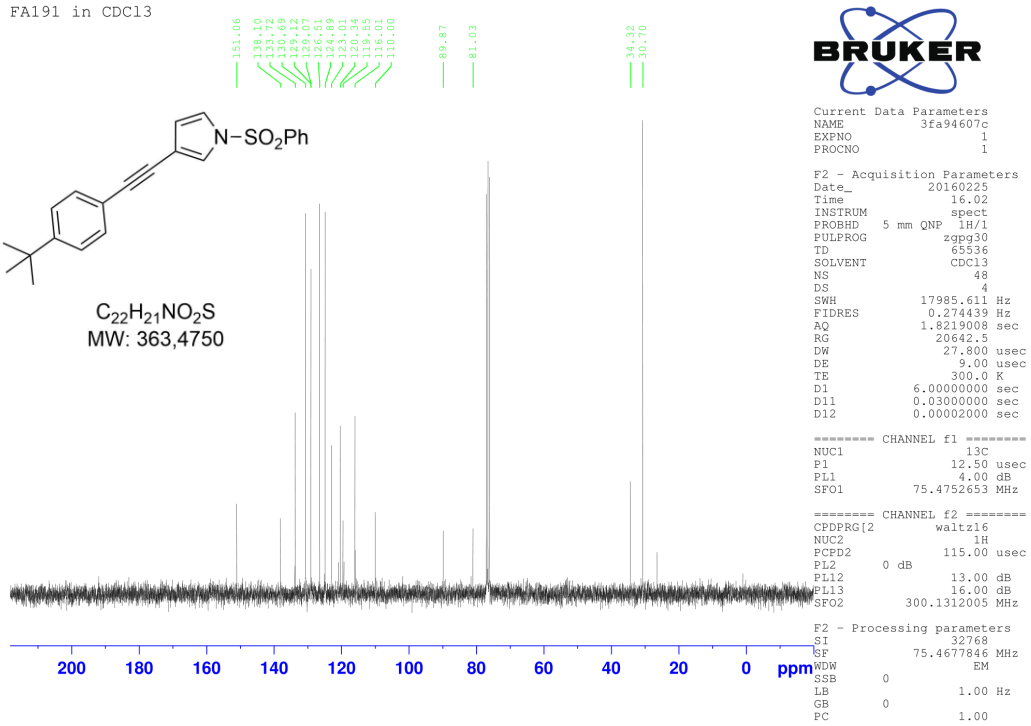




^{1}H and ^{13}C-NMR data for compound **7**





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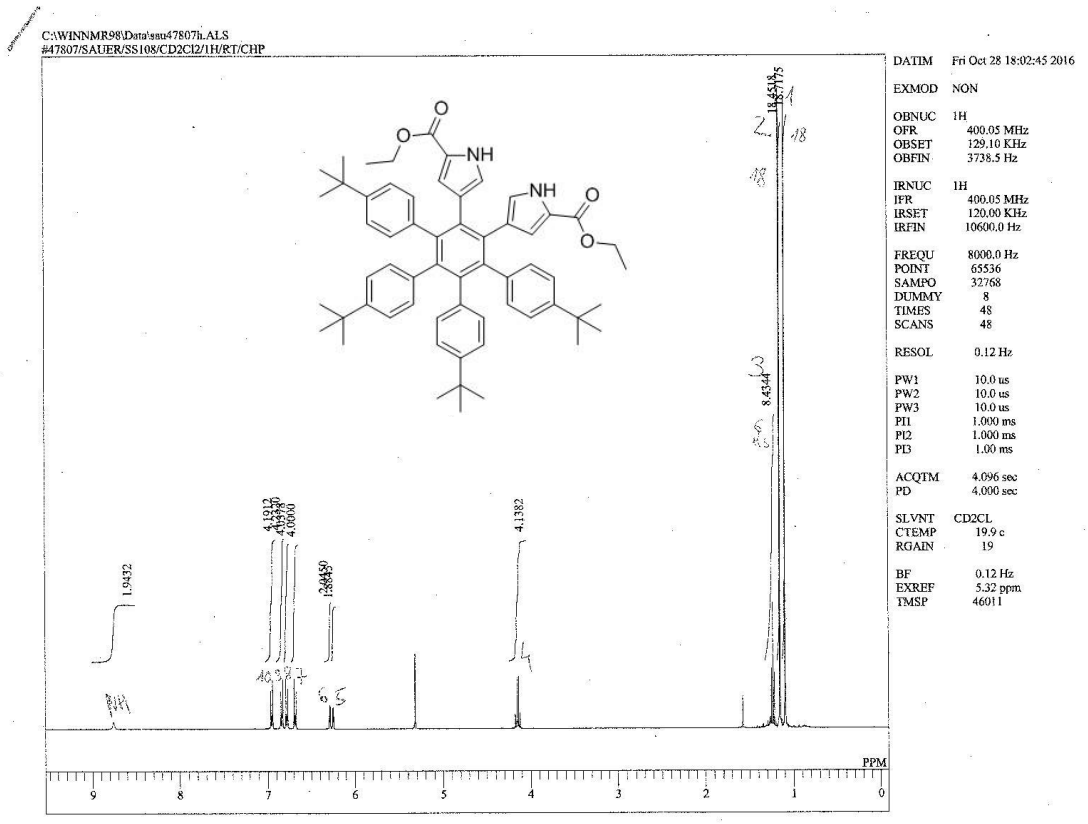

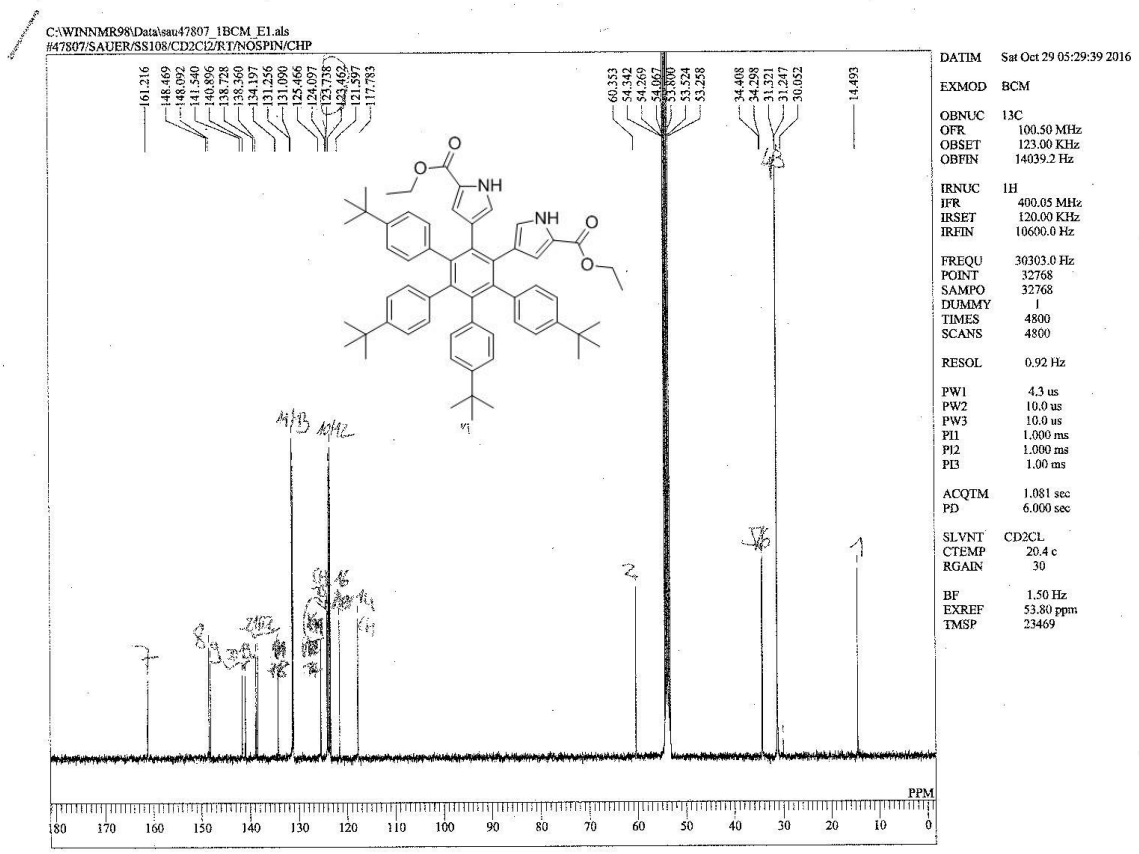

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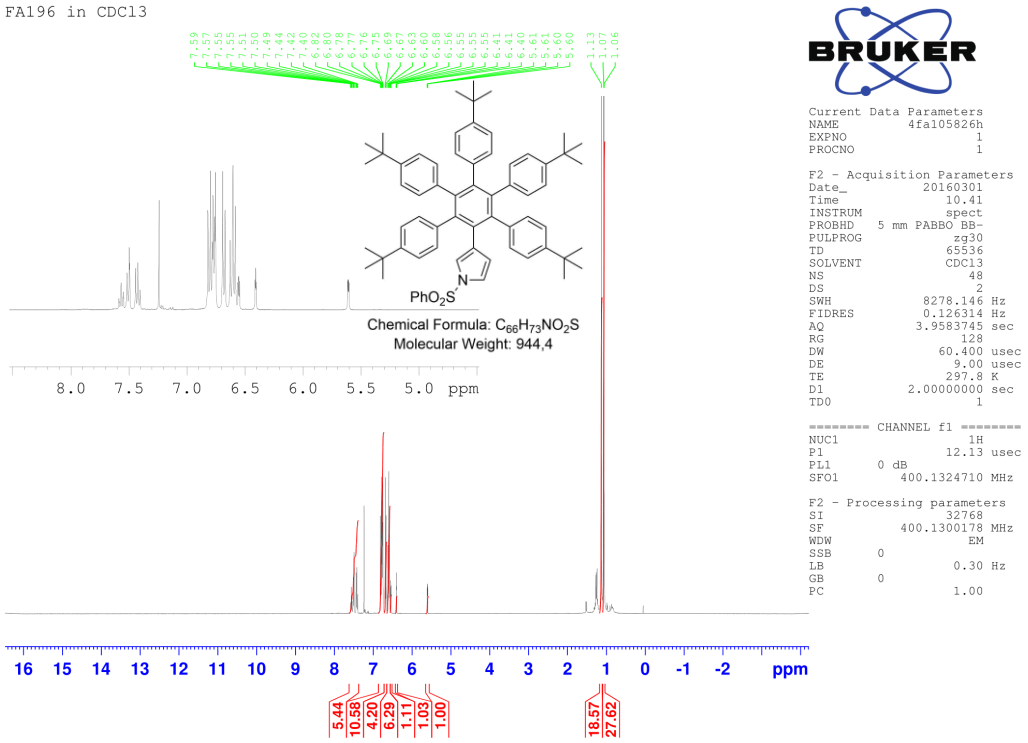
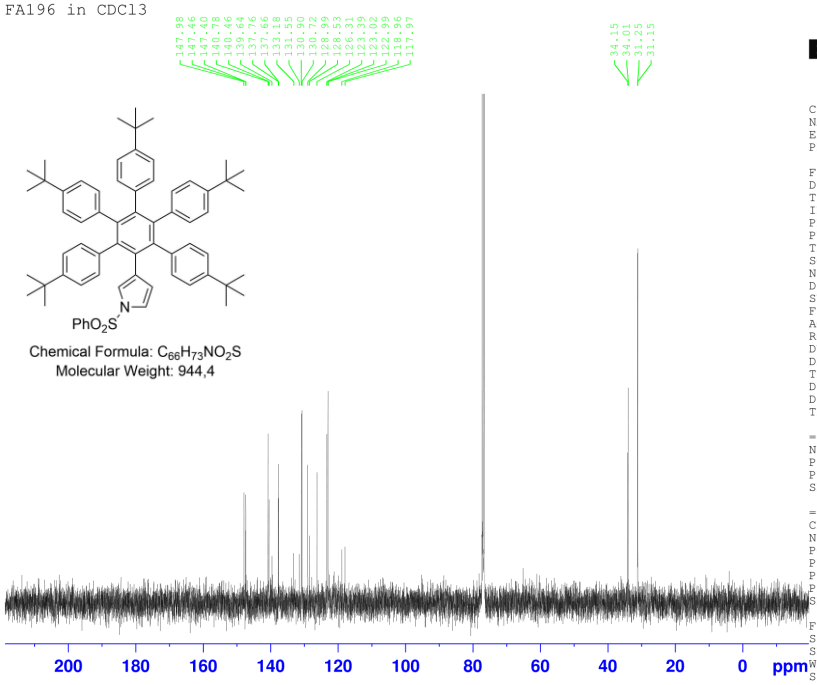


FA196 in CDCl3



BRUKER

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PROCNO                 1

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PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 360
DS                   4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664256 sec
RG                4634
DW              20.850 usec
DE               9.00 usec
TE               298.0 K
D1          4.00000000 sec
D11         0.03000000 sec
TD0                  1

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NUC1                13C
P1                6.05 usec
PL1       0 dB
SFO1        100.6227898 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2           116.00 usec
PL2       0 dB
PL12             21.51 dB
PL13            120.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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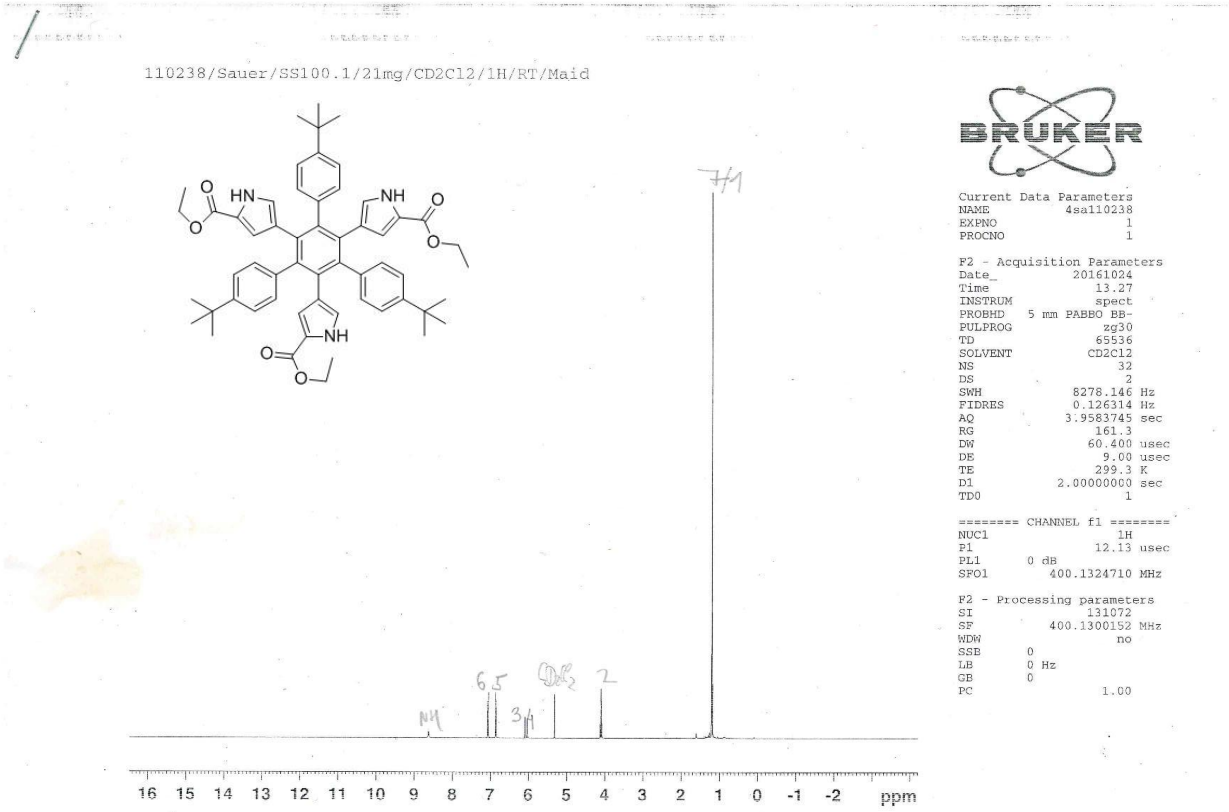

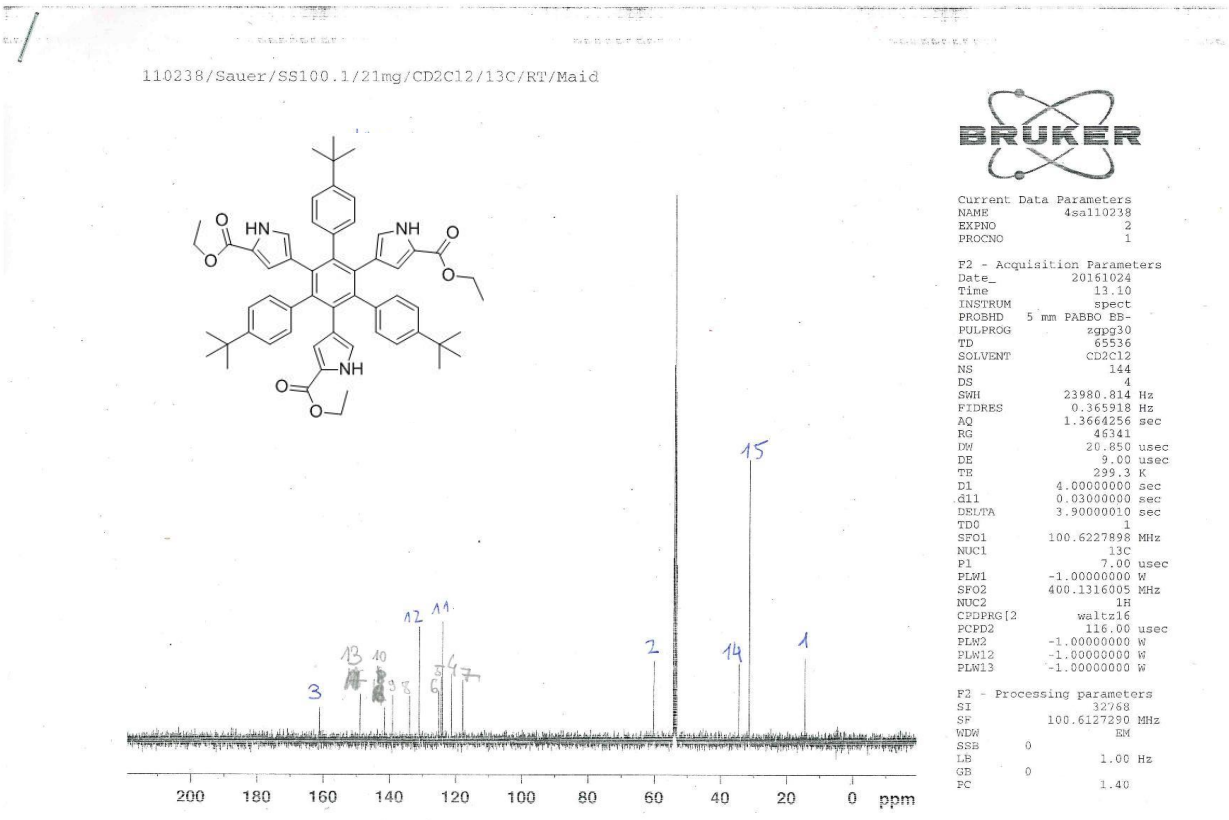

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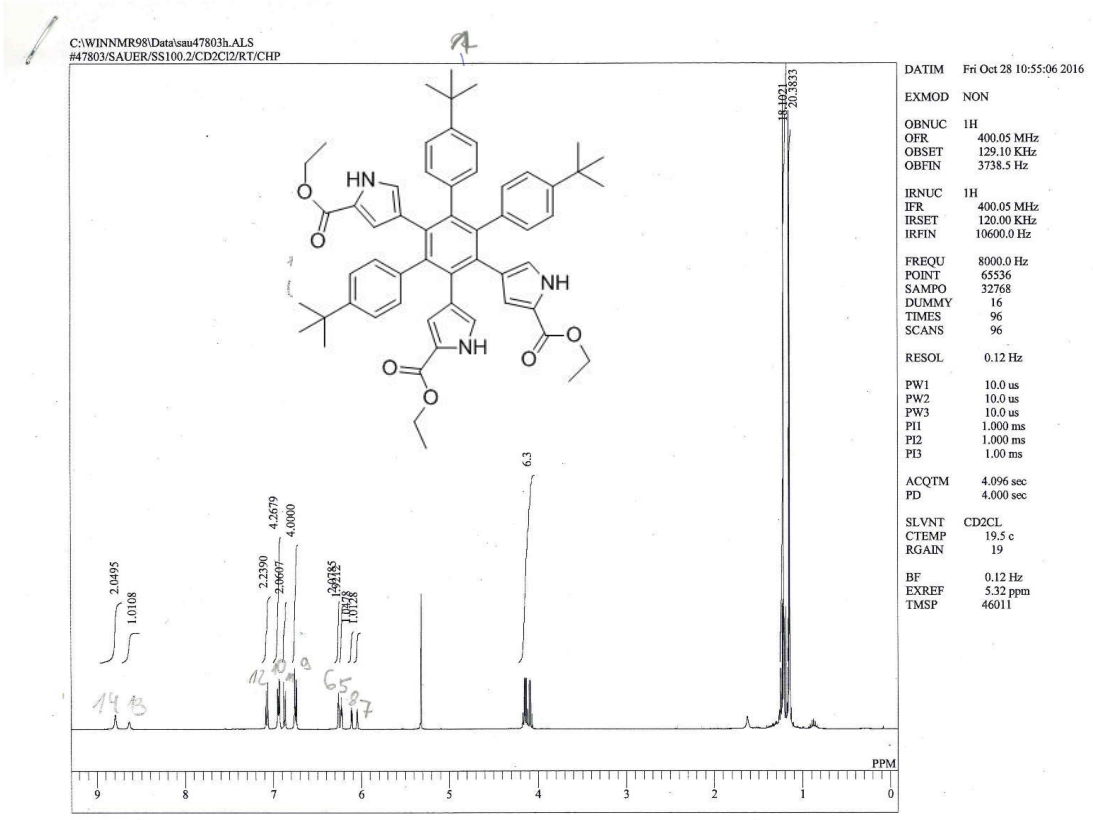

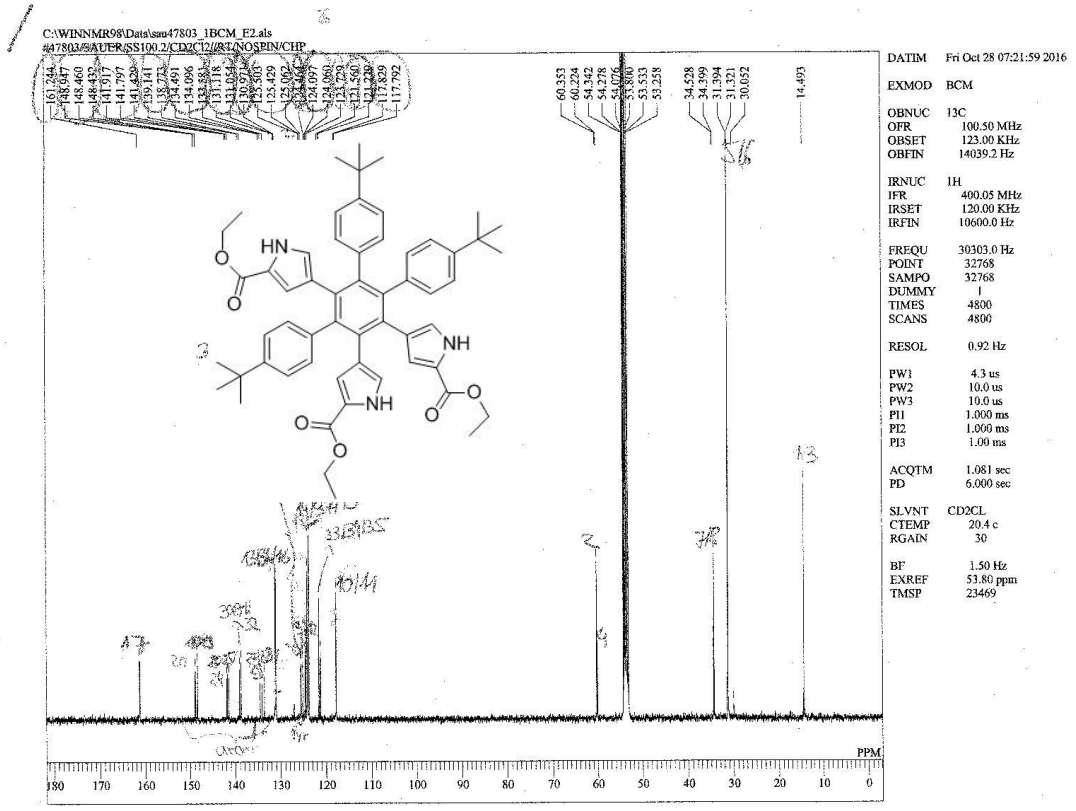

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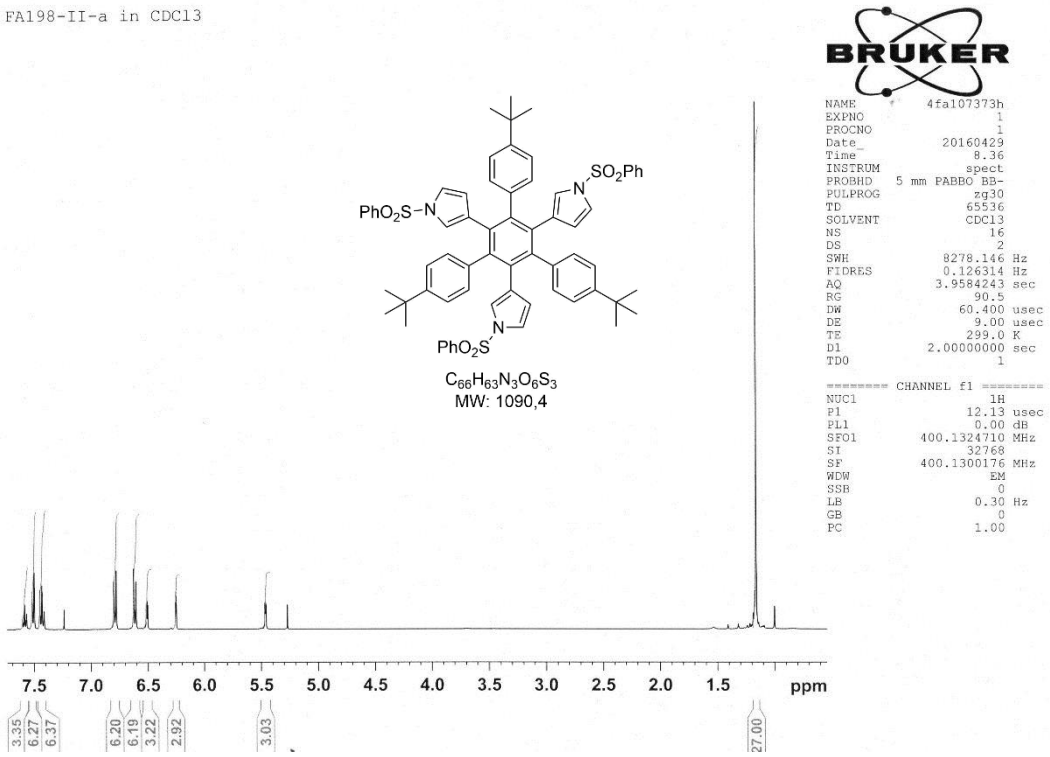
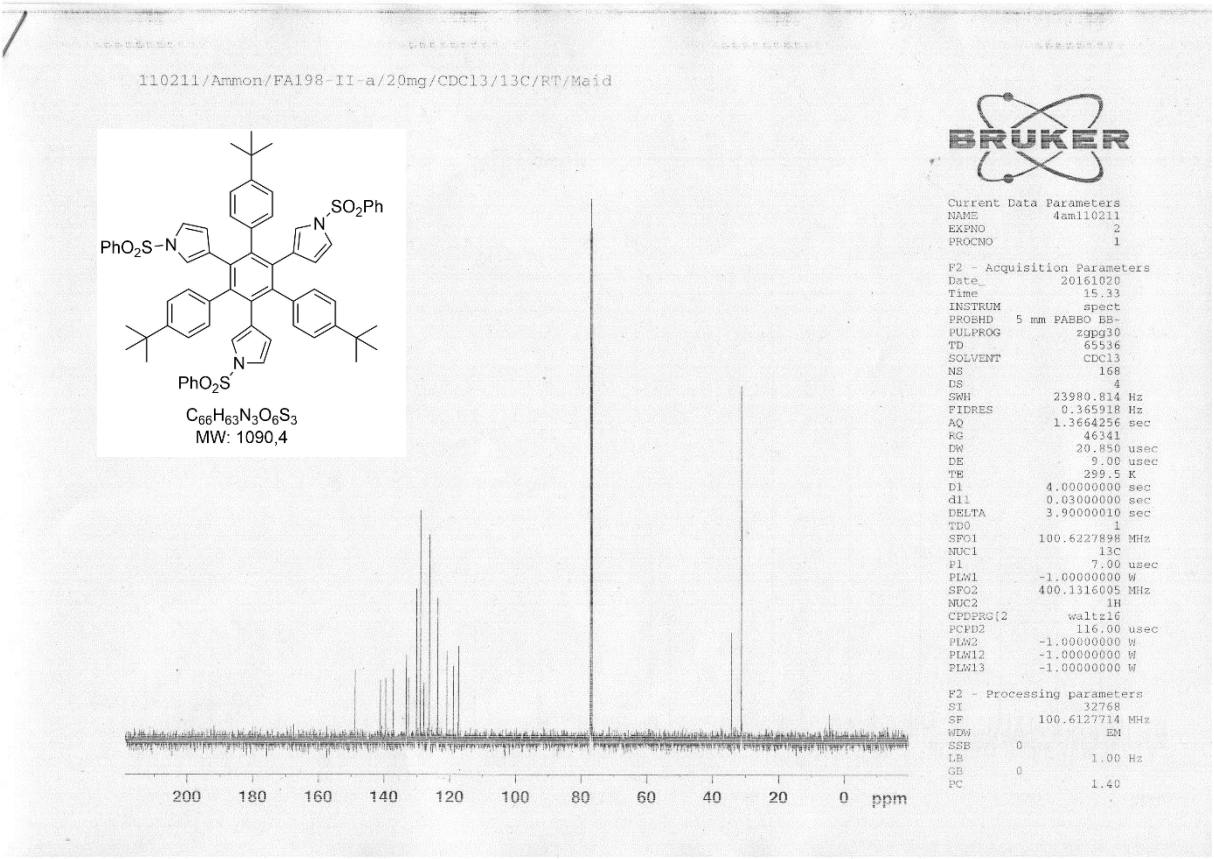
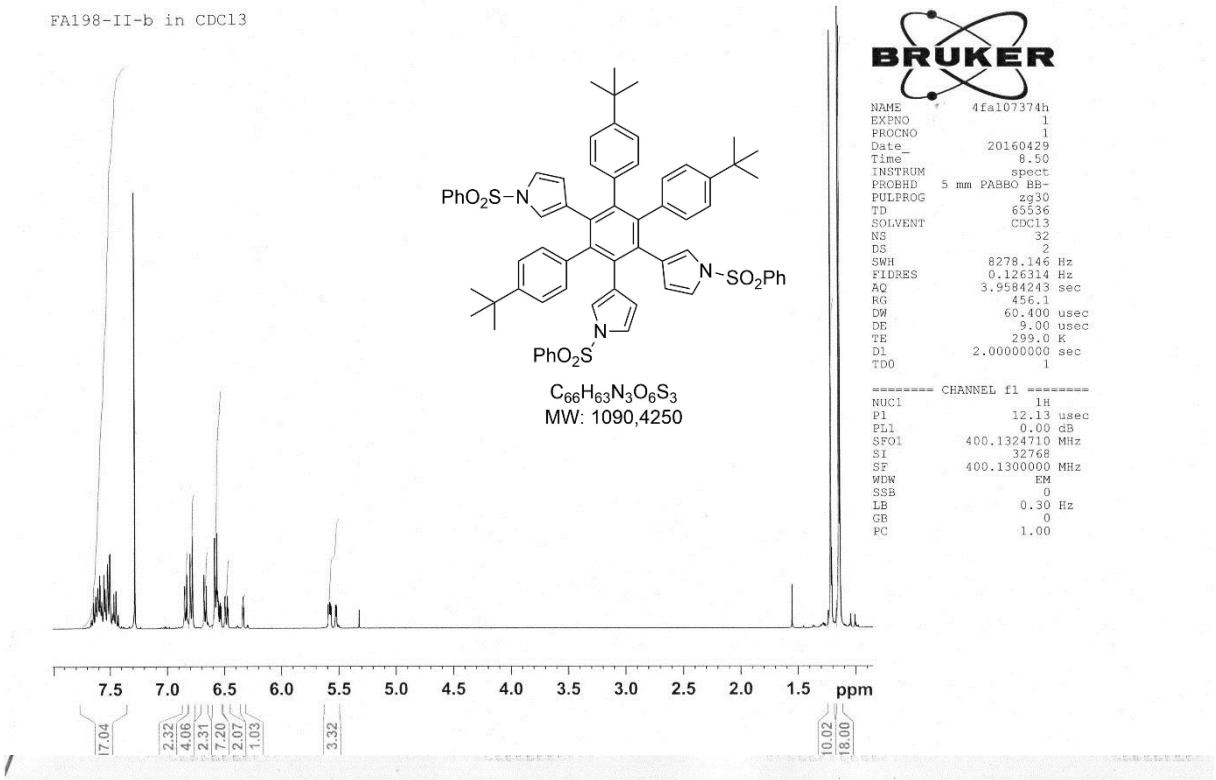




^{1}H and ^{13}C-NMR data for compound **19**

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TD                      65536
SOLVENT                 CDCl3
NS                         32
DS                          2
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FIDRES                0.126314 Hz
AQ                 3.9584243 sec
RG                       456.1
DW                      60.400 usec
DE                       9.00 usec
TE                      299.0 K
D1                 2.00000000 sec
TD0                          1

======== CHANNEL f1 ========
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PL1                      0.00 dB
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SI                       32768
SF             400.1300000 MHz
WDW                        EM
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PC                       1.00
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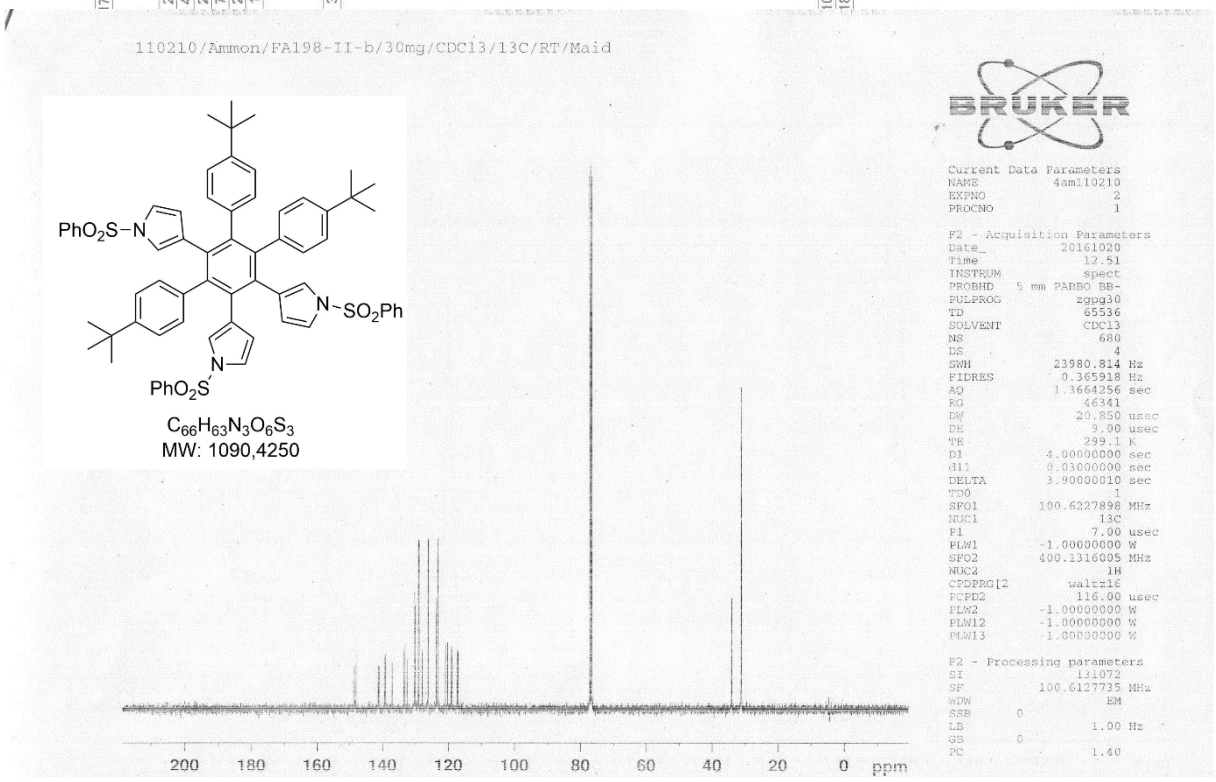


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PROCNO                       1

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SOLVENT                 CDCl3
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DS                           4
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FIDRES                0.365918 Hz
AQ                 1.3664256 sec
RG                       46341
DW                      20.850 usec
DE                       9.00 usec
TE                      299.1 K
D1                 4.00000000 sec
d11                0.03000000 sec
DELTA              3.90000010 sec
TD0                          1
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NUC1                       13C
P1                       7.00 usec
PLW1              -1.00000000 W
SFO2           400.1316005 MHz
NUC2                       1H
CPDPRG[2              waltz16
PCPD2                  116.00 usec
PLW2              -1.00000000 W
PLW12             -1.00000000 W
PLW13             -1.00000000 W

F2 - Processing parameters
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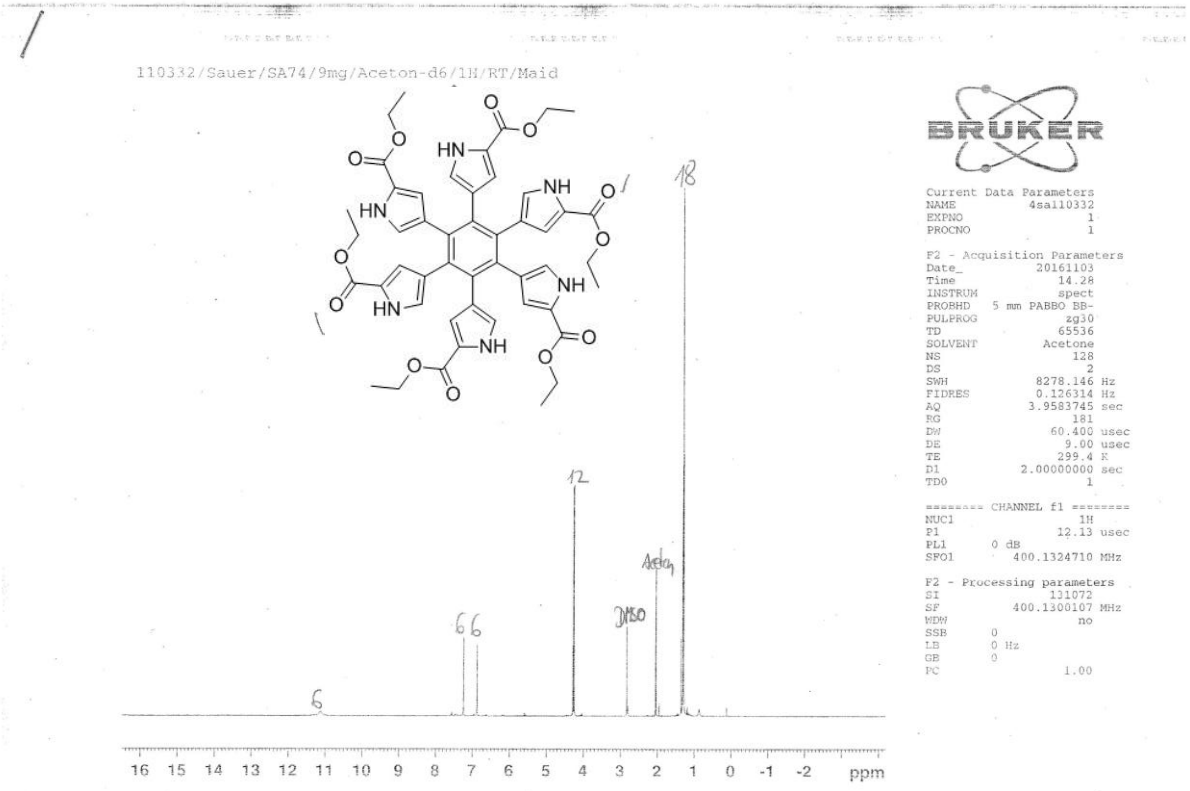
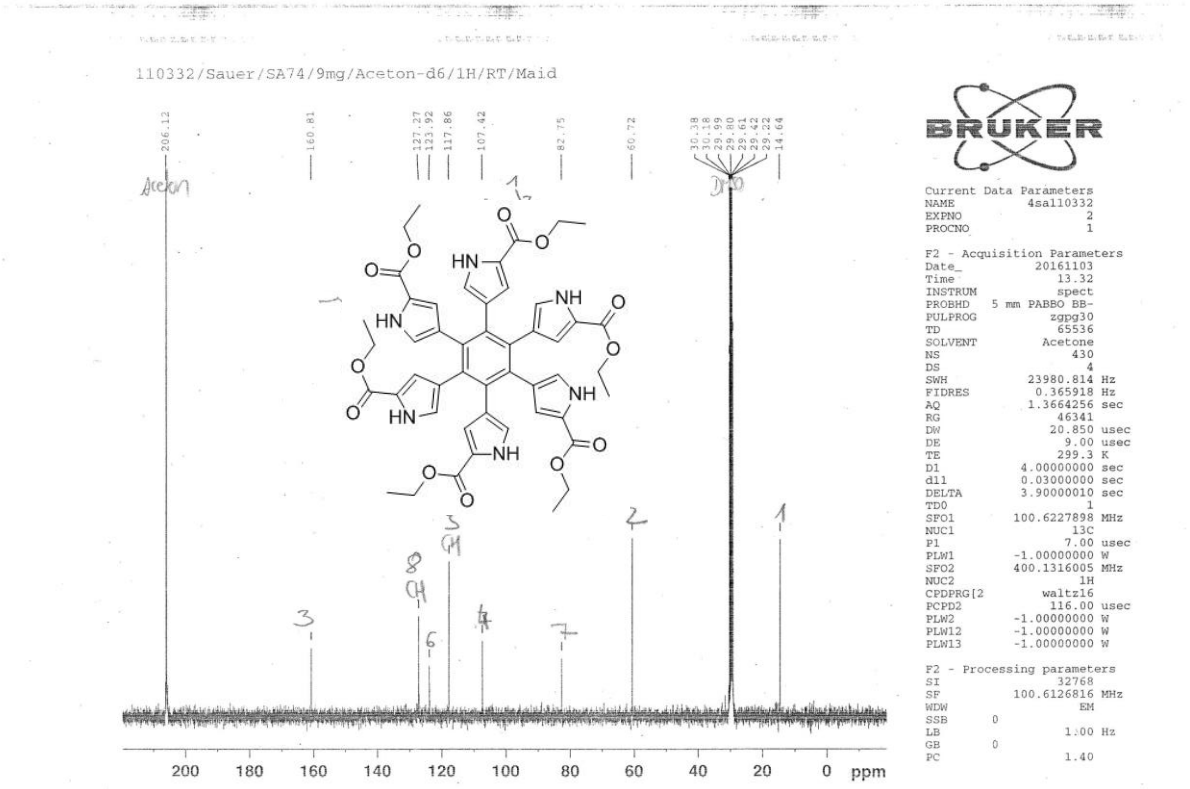
^{1}H and ^{13}C-NMR data for compound **23**





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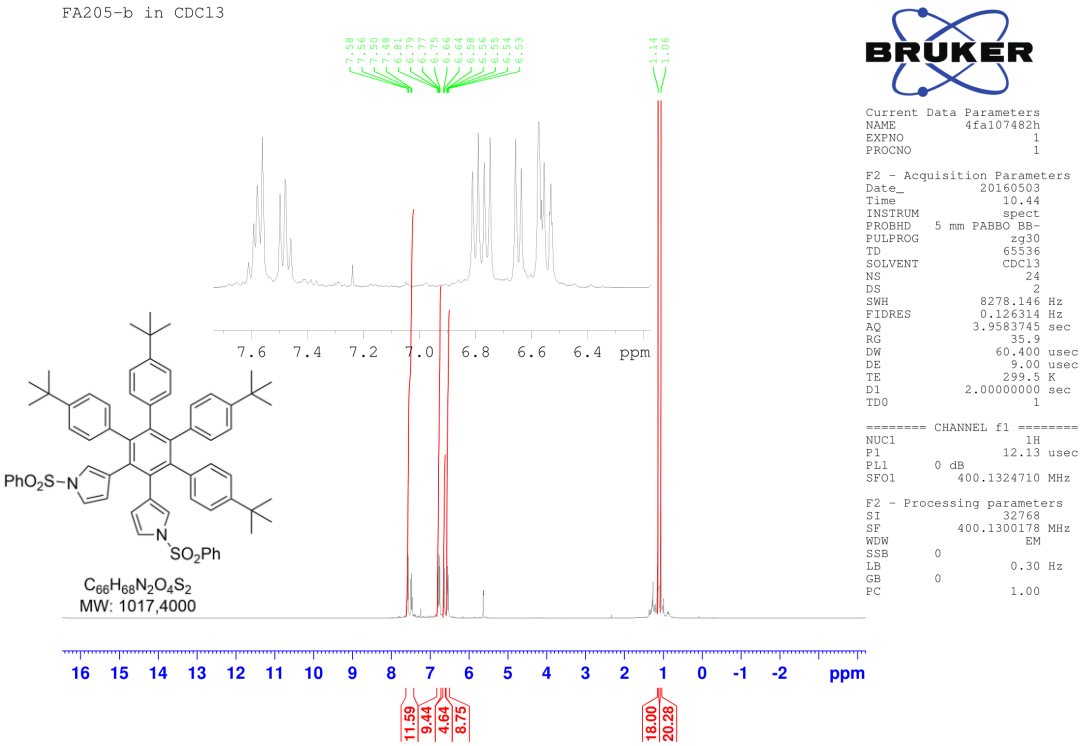

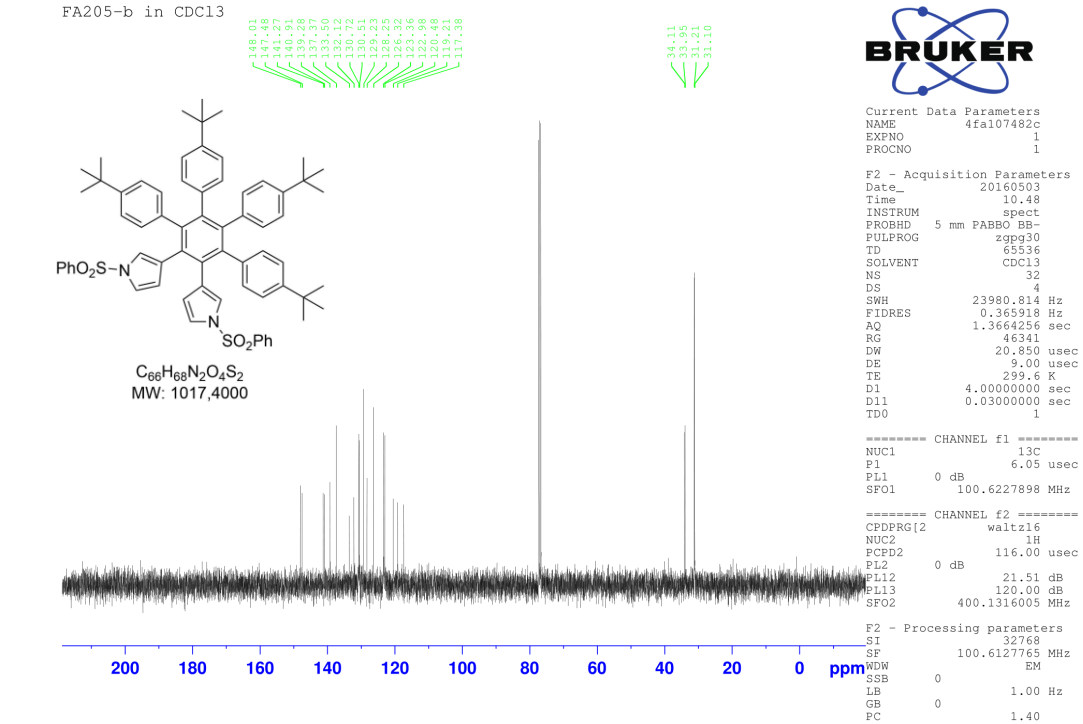

^{1}H and ^{13}C-NMR data for compound **22**

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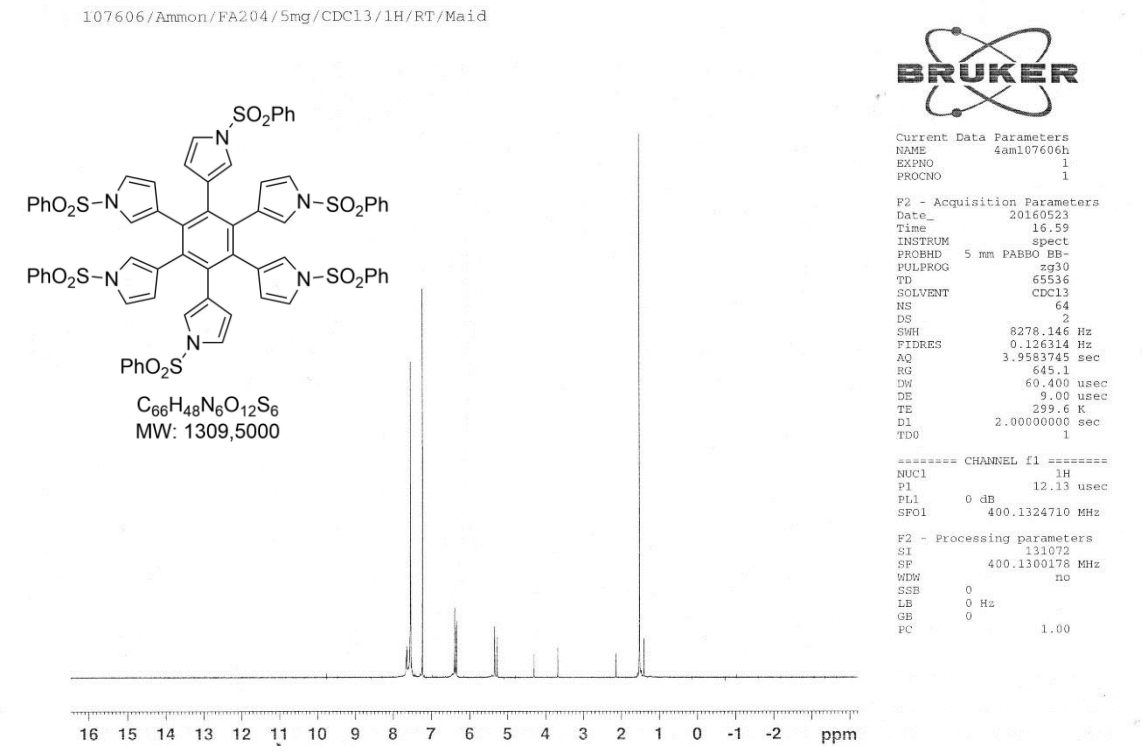
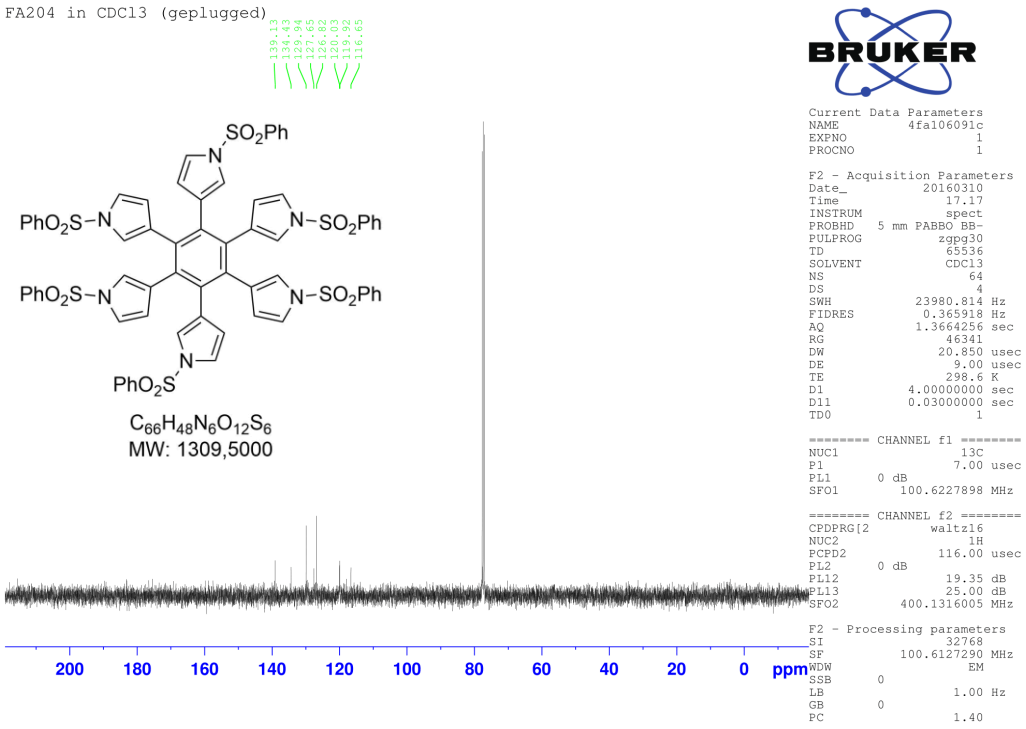
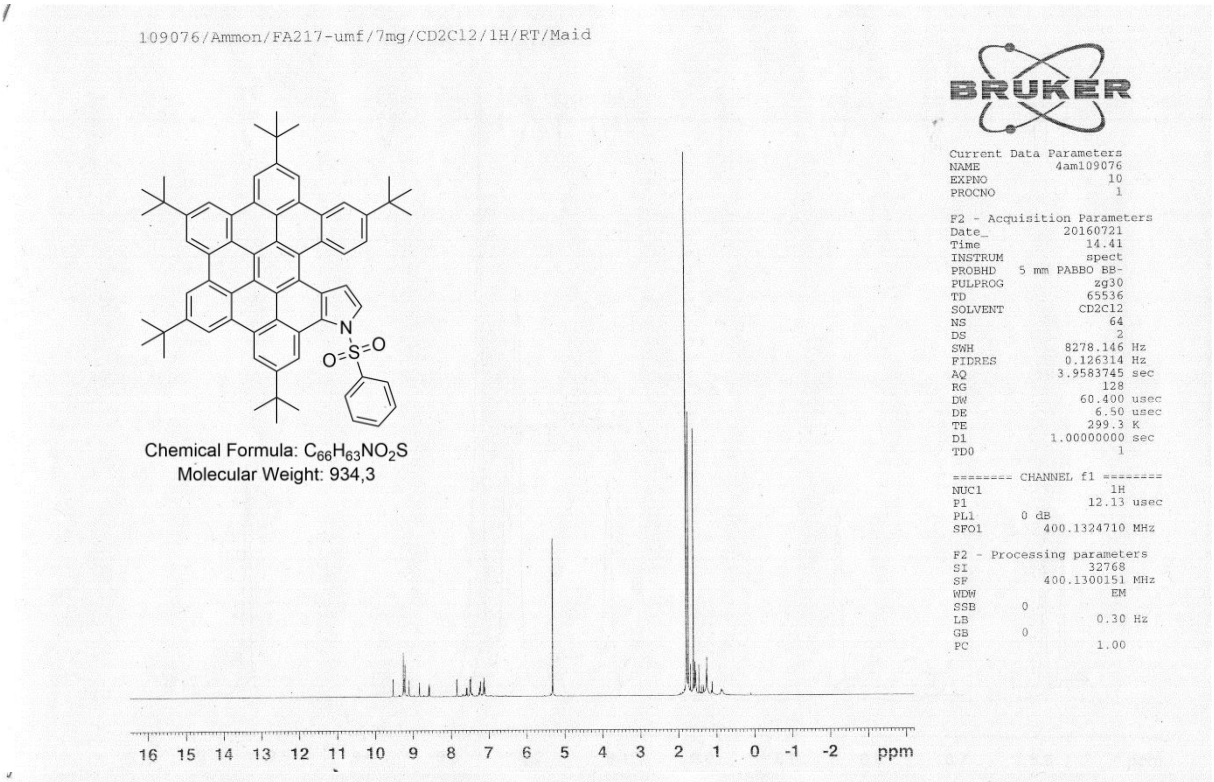
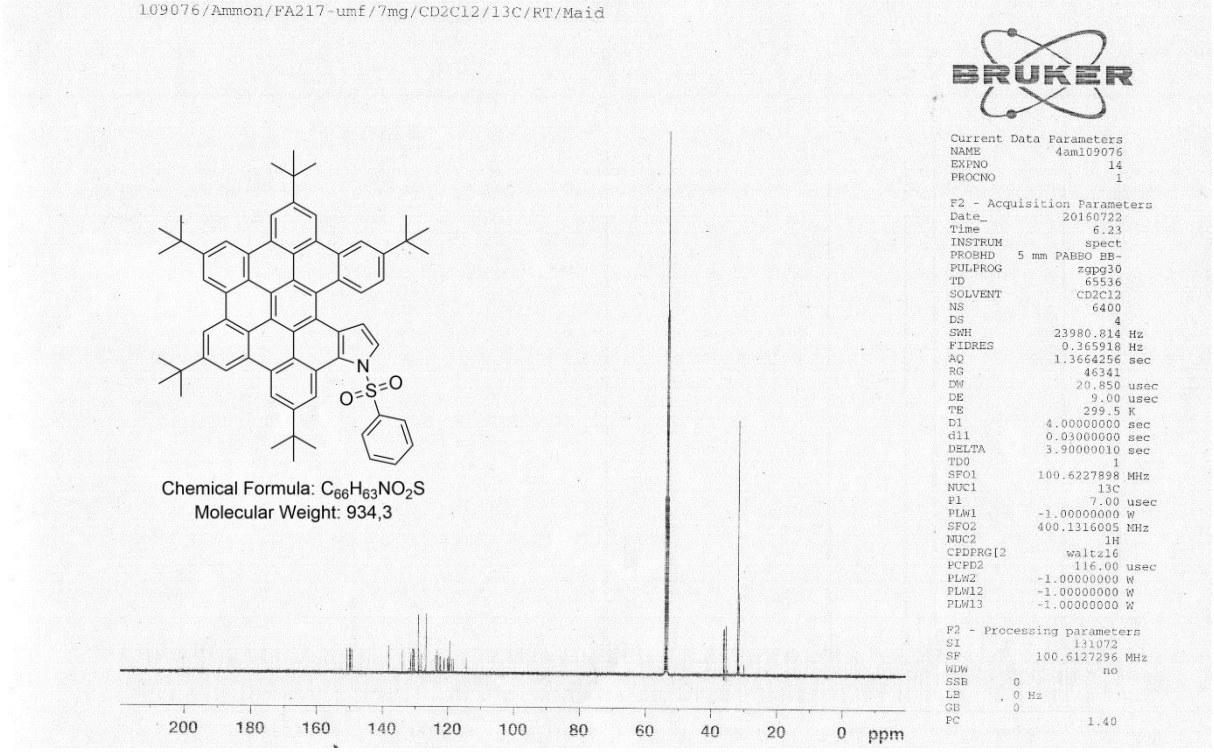




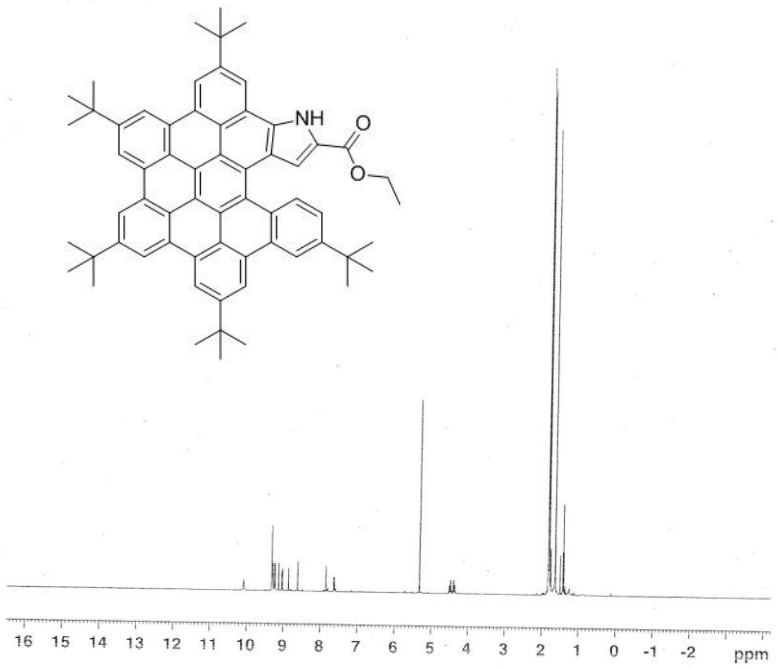
^{1}H and ^{13}C-NMR data for compound **24**





^{1}H and ^{13}C-NMR data for compound **25**

109302/Sauer/SS102/21mg/1H/CD2Cl2/RT/Maid



BRUKER

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PROCNO                        1

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INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                 CD2Cl2
NS                           64
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FIDRES                 0.126314 Hz
AQ                     3.9583745 sec
RG                          114
DW                       60.400 usec
DE                        9.00 usec
TE                       299.3 K
D1                    2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       12.13 usec
PL1          0 dB
SFO1              400.1324710 MHz

F2 - Processing parameters
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SF                    400.1300151 MHz
WDW                          no
SSB          0
LB           0 Hz
GB           0
PC                         1.00
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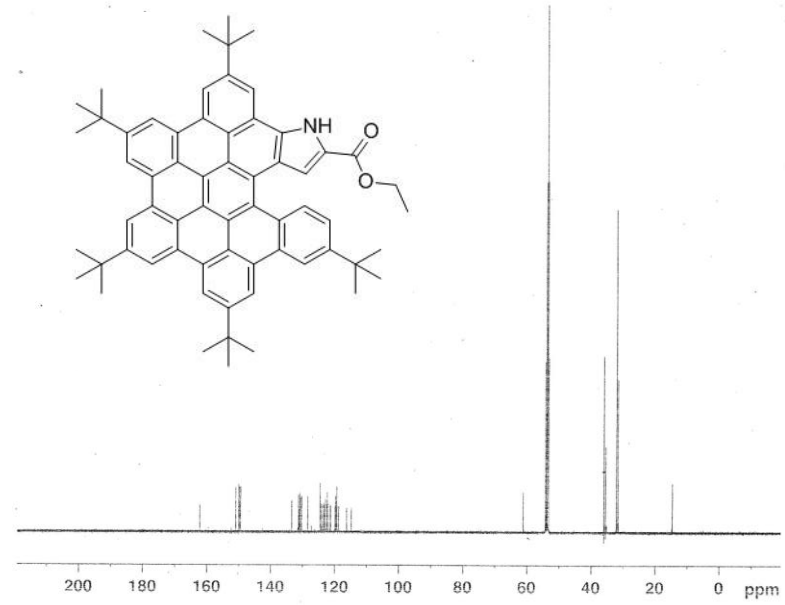
109302/Sauer/SS102/21mg/13C/CD2Cl2/RT/Maid



BRUKER

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PROCNO                        1

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TD                       65536
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FIDRES                 0.365918 Hz
AQ                     1.3664256 sec
RG                         46341
DW                       20.850 usec
DE                        9.00 usec
TE                       299.4 K
D1                    4.00000000 sec
d11                   0.03000000 sec
DELTA                 3.90000010 sec
TD0                           1
SFO1             100.6227898 MHz
NUC1                         13C
P1                        7.00 usec
PLW1             -1.00000000 W
SFO2             400.1316005 MHz
NUC2                         1H
CPDPRG[2              waltz16
PCPD2                   116.00 usec
PLW2             -1.00000000 W
PLW12            -1.00000000 W
PLW13            -1.00000000 W

F2 - Processing parameters
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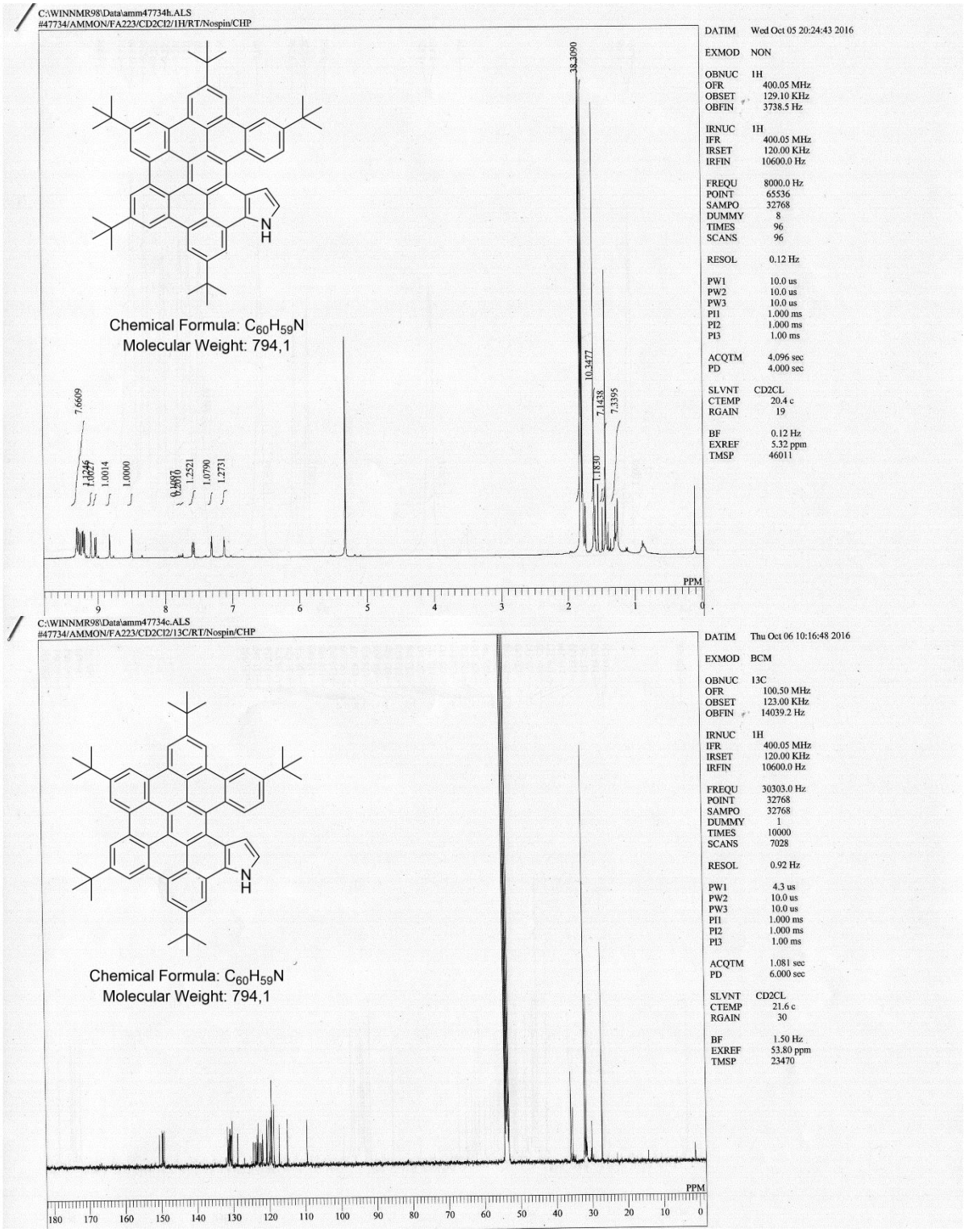
^{1}H and ^{13}C-NMR data for compound **26**



X-ray crystallographic data for compound **6**

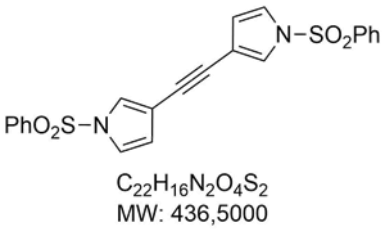


Table 1 Crystal data and structure refinement for 16Jux-FA01.

Identification code	16Jux-FA01
Empirical formula	$C_{22}H_{16}N_2O_4S_2$
Formula weight	218.24
Temperature/K	173.00(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	5.69674(11)
b/Å	8.92923(16)
c/Å	19.6982(5)
α/°	90
β/°	94.927(2)
γ/°	90
Volume/Å^3	998.30(4)
Z	2
ρ_{calc}g/cm^3	1.452
μ/mm^{-1}	2.703
F(000)	452.0
Crystal size/mm^3	0.404 × 0.117 × 0.085
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.012 to 122.832
Index ranges	$-6 \le h \le 6$, $-10 \le k \le 10$, $-22 \le l \le 20$
Reflections collected	4209
Independent reflections	1524 [R_{int} = 0.0297, R_{sigma} = 0.0250]
Data/restraints/parameters	1524/0/136
Goodness-of-fit on F^2	1.054
Final R indexes [I>=2σ (I)]	R_1 = 0.0430, wR_2 = 0.1146
Final R indexes [all data]	R_1 = 0.0465, wR_2 = 0.1186
Largest diff. peak/hole / e Å^{-3}	0.30/-0.25

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16Jux-FA01. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
S7	962.3(10)	5709.0(6)	6323.7(3)	35.0(3)
N10	1618(3)	4182(2)	5890.5(10)	32.5(5)
O9	-1540(3)	5702(2)	6316.4(10)	47.7(5)

C14	378(4)	581(3)	5110.4(12)	32.6(6)
O8	2186(3)	6910.6(19)	6036.0(9)	44.2(5)
C1	2177(4)	5418(3)	7160.2(12)	32.7(6)
C11	3690(4)	3956(3)	5588.6(12)	34.2(6)
C5	5211(5)	5896(4)	8041.3(14)	55.3(8)
C2	943(5)	4590(3)	7607.0(14)	46.9(7)
C15	156(4)	2951(3)	5762.5(12)	32.3(6)
C13	1293(4)	1977(3)	5371.6(11)	30.2(5)
C3	1888(7)	4422(3)	8275.2(15)	57.7(9)
C12	3531(4)	2620(3)	5266.1(12)	36.0(6)
C6	4306(4)	6086(3)	7370.5(13)	43.8(7)
C4	4017(6)	5068(4)	8484.5(14)	56.3(8)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16Jux-FA01. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
S7	32.9(4)	29.5(4)	40.9(4)	-5.3(2)	-5.9(3)	0.9(2)
N10	30.6(10)	32.0(11)	33.9(11)	-5.9(8)	-3.5(8)	-5.2(8)
O9	30(1)	47.8(12)	63.3(13)	-16.2(9)	-7.7(9)	5.5(8)
C14	34.9(13)	33.1(13)	28.6(13)	-0.9(9)	-3.6(10)	-1.7(10)
O8	55.9(11)	29.5(9)	45.3(10)	2.2(8)	-5.9(8)	-5.9(8)
C1	32.5(12)	29.0(12)	36.2(13)	-6.3(10)	1.6(10)	1.2(10)
C11	32.5(13)	37.3(13)	32.5(13)	-2(1)	1.5(10)	-6.6(11)
C5	45.3(16)	78(2)	41.0(16)	-11.5(14)	-6.7(13)	-7.9(14)
C2	53.5(16)	38.0(14)	49.7(17)	-3.8(12)	6.5(13)	-13.6(13)
C15	27.0(11)	32.2(13)	36.6(13)	-2.9(10)	-3.2(10)	-5.3(10)
C13	32.7(12)	29.6(12)	26.7(12)	-0.7(9)	-5.7(9)	-2(1)
C3	94(2)	37.5(16)	42.6(17)	1.4(12)	13.7(16)	-14.1(15)
C12	37.5(13)	38.4(14)	32.3(13)	-4.6(10)	3(1)	-3.0(11)
C6	36.5(14)	58.5(17)	36.5(15)	-5.1(12)	3.3(11)	-9.4(13)
C4	80(2)	51.3(18)	35.7(15)	-4.0(14)	-4.7(14)	2.9(17)

Table 4 Bond Lengths for 16Jux-FA01.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
S7	N10	1.668(2)	C1	C6	1.383(3)
S7	O9	1.4244(18)	C11	C12	1.351(3)
S7	O8	1.4236(18)	C5	C6	1.387(4)
S7	C1	1.751(2)	C5	C4	1.369(4)
N10	C11	1.382(3)	C2	C3	1.387(4)

N10	C15	1.389(3)	C15	C13	1.362(3)
C14	C14[1]	1.191(5)	C13	C12	1.430(3)
C14	C13	1.429(3)	C3	C4	1.374(4)
C1	C2	1.387(4)			

[1]-X,-Y,1-Z

Table 5 Bond Angles for 16Jux-FA01.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
N10	S7	C1	105.83(11)	C6	C1	C2	121.3(2)
O9	S7	N10	105.00(10)	C12	C11	N10	107.8(2)
O9	S7	C1	108.85(12)	C4	C5	C6	120.6(3)
O8	S7	N10	105.88(11)	C1	C2	C3	118.9(3)
O8	S7	O9	121.57(11)	C13	C15	N10	107.5(2)
O8	S7	C1	108.60(11)	C14	C13	C12	126.9(2)
C11	N10	S7	125.21(16)	C15	C13	C14	125.7(2)
C11	N10	C15	109.13(19)	C15	C13	C12	107.4(2)
C15	N10	S7	125.65(17)	C4	C3	C2	120.0(3)
C14[1]	C14	C13	179.6(4)	C11	C12	C13	108.1(2)
C2	C1	S7	119.70(19)	C1	C6	C5	118.5(3)
C6	C1	S7	118.94(19)	C5	C4	C3	120.6(3)

[1]-X,-Y,1-Z

Table 6 Torsion Angles for 16Jux-FA01.

A	**B**	**C**	**D**	**Angle/°**	**A**	**B**	**C**	**D**	**Angle/°**
S7	N10	C11	C12	-177.75(17)	O8	S7	N10	C15	-149.19(19)
S7	N10	C15	C13	177.55(16)	O8	S7	C1	C2	163.5(2)
S7	C1	C2	C3	-177.4(2)	O8	S7	C1	C6	-13.4(2)
S7	C1	C6	C5	177.6(2)	C1	S7	N10	C11	-85.8(2)
N10	S7	C1	C2	-83.2(2)	C1	S7	N10	C15	95.6(2)
N10	S7	C1	C6	99.9(2)	C1	C2	C3	C4	-0.2(4)
N10	C11	C12	C13	-0.4(3)	C11	N10	C15	C13	-1.2(3)
N10	C15	C13	C14	-178.3(2)	C2	C1	C6	C5	0.7(4)
N10	C15	C13	C12	0.9(3)	C2	C3	C4	C5	0.8(5)
O9	S7	N10	C11	159.1(2)	C15	N10	C11	C12	1.0(3)
O9	S7	N10	C15	-19.4(2)	C15	C13	C12	C11	-0.3(3)
O9	S7	C1	C2	29.2(2)	C6	C1	C2	C3	-0.6(4)
O9	S7	C1	C6	-147.7(2)	C6	C5	C4	C3	-0.7(5)

C14 C13 C12 C11 178.8(2) C4 C5 C6 C1 -0.1(4)
O8 S7 N10 C11 29.3(2)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16Jux-FA01.

Atom	*x*	*y*	*z*	U(eq)
H11	4967	4608	5605	41
H5	6643	6336	8191	66
H2	-496	4154	7461	56
H15	-1329	2816	5916	39
H3	1081	3872	8582	69
H12	4678	2193	5019	43
H6	5114	6649	7069	53
H4	4653	4941	8932	68

Experimental

Single crystals of $C_{22}H_{16}N_2O_4S_2$ [16Jux-FA01] were [crystallized from toluene/hexanes]. A suitable crystal was selected and [mounted on a loop] on a SuperNova, Dual, Cu at zero, Atlas diffractometer. The crystal was kept at 173.00(10) K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [16Jux-FA01]

Crystal Data for $C_{22}H_{16}N_2O_4S_2$ (*M* =218.24 g/mol): monoclinic, space group $P2_1/c$ (no. 14), *a* = 5.69674(11) Å, *b* = 8.92923(16) Å, *c* = 19.6982(5) Å, *β* = 94.927(2)°, *V* = 998.30(4) Å^3, *Z* = 2, *T* = 173.00(10) K, μ(CuKα) = 2.703 mm^{-1}, *Dcalc* = 1.452 g/cm^3, 4209 reflections measured (9.012° ≤ 2Θ ≤ 122.832°), 1524 unique (R_{int} = 0.0297, R_{sigma} = 0.0250) which were used in all calculations. The final R_1 was 0.0430 (I > 2σ(I)) and wR_2 was 0.1186 (all data).

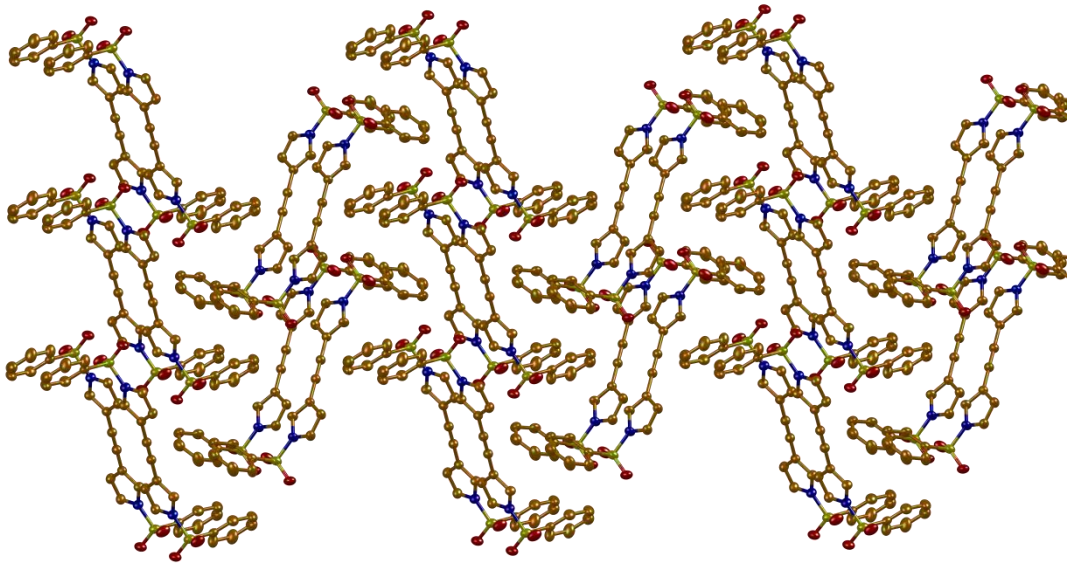
Refinement model description

Number of restraints - 0, number of constraints - unknown.

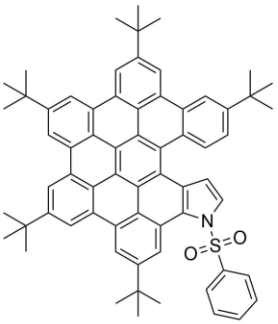
Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
2.a Aromatic/amide H refined with riding coordinates:
 C11(H11), C5(H5), C2(H2), C15(H15), C3(H3), C12(H12), C6(H6), C4(H4)
```

Packing motif of Compound 6



X-ray crystallographic data for compound **24**



Chemical Formula: $C_{66}H_{63}NO_2S$
Molecular Weight: 934,3

Table 1 Crystal data and structure refinement for 16Jux_FA02_2.

Identification code	16Jux_FA02_2
Empirical formula	$C_{67.5}H_{66}Cl_3NO_2S$
Formula weight	1061.62
Temperature/K	152.7(10)
Crystal system	triclinic
Space group	P-1
a/Å	14.0225(7)
b/Å	15.4902(10)
c/Å	15.6494(5)
α/°	109.303(4)
β/°	105.791(4)
γ/°	106.459(5)
Volume/Å^3	2814.2(3)
Z	2
ρ_{calc}g/cm^3	1.253
μ/mm^{-1}	2.173
F(000)	1122.0
Crystal size/mm^3	0.181 × 0.128 × 0.071
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.186 to 113.446
Index ranges	-14 ≤ h ≤ 14, -16 ≤ k ≤ 16, -16 ≤ l ≤ 14
Reflections collected	11987
Independent reflections	7226 [R_{int} = 0.0257, R_{sigma} = 0.0357]
Data/restraints/parameters	7226/1/700
Goodness-of-fit on F^2	1.021
Final R indexes [I>=2σ (I)]	R_1 = 0.0729, wR_2 = 0.1995
Final R indexes [all data]	R_1 = 0.0797, wR_2 = 0.2094
Largest diff. peak/hole / e Å^{-3}	1.59/-0.77

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 16Jux_FA02_2. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
S1	2997.7(7)	-708.9(6)	-542.7(6)	27.5(3)
O2	3201(2)	-920.1(17)	-1416.4(17)	34.9(6)
O1	3019(2)	-1365.0(17)	-83.9(17)	32.5(6)

N1	3925(2)	463(2)	258(2)	25.0(6)
C23	4625(3)	2099(2)	1343(2)	22.2(7)
C24	4793(3)	2978(2)	2186(2)	21.4(7)
C49	3977(3)	3660(2)	3298(2)	21.9(7)
C48	4585(3)	4635(2)	3451(2)	22.1(7)
C25	5567(3)	3969(2)	2503(2)	22.1(7)
C22	4932(3)	2022(2)	526(2)	25.6(8)
C51	3435(3)	1825(2)	2486(2)	20.8(7)
C60	2811(3)	1688(2)	3038(2)	22.0(7)
C52	3459(3)	957(2)	1800(2)	22.3(7)
C44	5989(3)	5784(2)	3186(2)	23.7(7)
C1AA	4394(3)	5471(2)	4000(2)	22.7(7)
C21	4498(3)	1042(3)	-116(3)	27.5(8)
C62	3184(3)	3503(2)	3718(2)	23.2(7)
C50	4066(3)	2828(2)	2647(2)	21.0(7)
C74	3667(3)	7963(2)	5510(2)	24.0(7)
C43	5772(3)	6598(2)	3680(2)	25.1(8)
C0BA	3987(3)	1148(2)	1161(2)	21.9(7)
C0AA	3597(3)	5321(2)	4393(2)	23.8(7)
C27	7996(3)	3724(2)	2022(2)	27.2(8)
C61	2584(3)	2521(2)	3576(2)	23.1(7)
C53	2994(3)	33(2)	1792(2)	25.0(8)
C59	2381(3)	742(2)	3019(2)	25.2(8)
C45	5391(3)	4786(2)	3060(2)	22.4(7)
C54	2472(3)	-88(2)	2409(2)	24.7(8)
C26	6554(3)	4180(2)	2317(2)	23.3(7)
C28	8540(3)	4654(2)	2052(2)	25.2(8)
C65	1434(3)	3135(2)	4309(2)	25.6(8)
C72	3978(3)	7107(2)	5031(2)	23.0(7)
C63	2961(3)	4304(2)	4230(2)	23.5(7)
C37	6989(3)	7673(2)	3232(3)	27.2(8)
C34	7123(3)	5144(2)	2410(2)	24.7(8)
C2AA	4978(3)	6449(2)	4130(2)	24.4(8)
C42	6285(3)	7523(3)	3699(3)	29.3(8)
C36	7210(3)	6869(2)	2768(3)	28.3(8)
C67	383(3)	2884(2)	4478(3)	27.4(8)
C55	2023(3)	-1126(2)	2390(3)	28.7(8)
C20	7037(3)	3491(2)	2146(2)	24.8(8)
C35	6757(3)	5935(2)	2763(2)	25.3(8)
C11	1760(3)	-584(3)	-755(2)	31.6(9)
C66	1721(3)	2360(2)	3864(2)	26.4(8)
C73	4768(3)	7238(2)	4653(2)	26.2(8)

C75	4493(3)	8987(3)	5745(3)	34.1(9)
C33	8092(3)	5340(2)	2256(3)	27.1(8)
C29	9608(3)	4881(3)	1909(3)	30.0(8)
C38	7499(3)	8702(3)	3246(3)	32.7(9)
C76	2559(3)	7766(3)	4780(3)	32.3(8)
C71	3410(3)	6144(2)	4898(2)	25.5(8)
C77	3582(3)	7999(3)	6475(3)	33.0(8)
C64	2085(3)	4097(2)	4514(2)	26.7(8)
C56	1326(3)	-1922(3)	1321(3)	39.9(10)
C69	391(3)	2320(3)	5120(3)	37.3(9)
C70	-587(3)	2205(3)	3472(3)	37.3(9)
C16	874(3)	-1353(3)	-850(3)	41.6(10)
C68	213(3)	3827(3)	4978(3)	41.5(10)
C12	1666(3)	231(3)	-882(3)	44(1)
C58	1332(4)	-1165(3)	2989(3)	43.4(10)
C30	10412(3)	4683(3)	2639(3)	43.7(10)
C31	9376(4)	4197(3)	850(3)	51.3(11)
C32	10155(3)	5977(3)	2119(4)	46.7(11)
C57	2991(3)	-1360(3)	2824(3)	44.9(10)
C40	7940(4)	9538(3)	4298(3)	51.6(11)
C13	658(4)	279(4)	-1102(3)	59.0(13)
C15	-128(3)	-1297(4)	-1082(3)	56.5(12)
C14	-217(4)	-478(4)	-1185(3)	60.0(13)
C39	8442(4)	8793(3)	2931(4)	61.7(14)
C41	6626(4)	8857(4)	2561(4)	60.5(13)
Cl11	2917.3(16)	5841.0(15)	1771.4(15)	78.7(5)
Cl12	3599.4(16)	7047(2)	779.5(16)	92.1(7)
Cl22	1743(2)	3132.7(19)	-183(2)	143.0(13)
Cl21	3964(3)	3896(3)	57(2)	140.1(11)
C200	3056(6)	3449(6)	579(6)	95(3)
C100	3881(7)	6436(9)	1478(11)	137(5)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 16Jux_FA02_2. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
S1	31.5(5)	21.4(5)	25.3(5)	5.3(4)	13.9(4)	8.6(4)
O2	43.1(15)	29.3(13)	27.0(13)	5.1(11)	20.3(12)	10.6(12)
O1	41.8(15)	22.4(12)	34.4(14)	11.4(11)	18.2(12)	13.4(11)
N1	28.8(15)	22.1(15)	23.9(15)	7.8(12)	14.9(13)	9.0(13)
C23	24.1(17)	22.1(17)	24.8(18)	12.0(15)	12.9(14)	10.8(14)

C24	22.0(17)	21.5(17)	24.6(17)	12.3(14)	11.4(14)	9.8(14)
C49	22.8(17)	22.3(18)	20.4(17)	10.4(14)	10.6(14)	6.2(14)
C48	23.0(17)	19.7(17)	22.3(17)	8.9(14)	10.4(14)	6.4(14)
C25	25.2(18)	20.8(17)	21.7(17)	10.3(14)	11.9(14)	7.7(14)
C22	29.0(19)	24.7(19)	24.8(18)	11.5(15)	14.2(15)	9.4(15)
C51	20.5(16)	18.1(17)	21.7(17)	9.2(14)	8.0(14)	5.2(14)
C60	21.2(17)	18.0(17)	21.4(17)	6.4(14)	8.7(14)	3.3(14)
C52	22.7(17)	21.1(17)	21.5(17)	7.8(14)	10.4(14)	7.3(14)
C44	24.8(18)	20.4(17)	26.0(18)	10.1(14)	13.1(15)	6.9(14)
C1AA	22.7(17)	20.2(17)	23.2(17)	8.4(14)	10.8(14)	5.9(14)
C21	30.5(19)	31(2)	27.7(19)	13.5(16)	19.8(16)	13.4(16)
C62	23.5(17)	20.7(17)	22.5(17)	7.9(14)	10.7(14)	5.9(14)
C50	22.0(17)	19.7(17)	20.2(17)	8.5(14)	9.3(14)	6.8(14)
C74	25.5(18)	22.5(17)	24.7(18)	9.6(14)	11.7(14)	10.4(14)
C43	29.2(18)	21.0(17)	28.1(18)	11.4(15)	16.1(15)	9.4(15)
C0BA	24.2(17)	19.3(17)	21.2(17)	6.5(14)	9.9(14)	9.9(14)
C0AA	25.7(18)	20.8(17)	23.5(17)	8.7(14)	12.2(15)	6.7(14)
C27	28.7(19)	23.8(18)	31.2(19)	11.1(15)	15.7(16)	11.3(15)
C61	23.4(17)	21.4(17)	22.9(17)	9.2(14)	11.0(14)	6.4(14)
C53	28.1(18)	20.6(17)	25.4(18)	8.2(15)	13.1(15)	8.7(15)
C59	25.6(18)	22.2(18)	27.0(18)	10.3(15)	14.1(15)	6.3(15)
C45	23.4(17)	19.6(17)	23.4(17)	9.7(14)	11.0(14)	6.2(14)
C54	27.5(18)	20.2(17)	25.1(18)	10.7(15)	11.4(15)	6.8(14)
C26	25.8(18)	20.0(17)	23.5(17)	8.8(14)	13.8(15)	6.0(14)
C28	27.5(18)	19.8(17)	28.0(18)	8.1(15)	16.3(15)	7.6(15)
C65	28.1(19)	24.1(18)	24.8(18)	9.7(15)	15.3(15)	7.8(15)
C72	23.9(17)	21.1(17)	22.4(17)	7.5(14)	11.6(14)	7.6(14)
C63	23.5(17)	21.3(17)	26.1(18)	9.7(15)	13.4(15)	7.4(14)
C37	30.2(19)	21.1(18)	36(2)	15.1(16)	19.8(16)	9.0(15)
C34	29.6(19)	20.5(17)	25.4(18)	9.6(14)	15.4(15)	8.7(15)
C2AA	24.8(18)	23.0(18)	26.0(18)	10.3(15)	13.3(15)	8.3(15)
C42	34(2)	22.0(18)	37(2)	12.5(16)	21.0(17)	11.7(16)
C36	32.0(19)	24.3(19)	33.7(19)	14.0(16)	20.8(16)	9.8(16)
C67	27.9(18)	25.7(18)	31.2(19)	11.6(15)	19.8(16)	8.2(15)
C55	37(2)	17.5(17)	31.9(19)	11.9(15)	18.1(16)	6.3(15)
C20	26.2(18)	17.9(17)	29.7(18)	9.1(15)	15.6(15)	5.7(14)
C35	27.6(18)	19.7(17)	30.3(19)	11.2(15)	16.4(15)	7.1(15)
C11	31(2)	35(2)	21.7(18)	6.4(16)	9.5(15)	13.5(17)
C66	29.3(19)	21.2(17)	27.6(18)	10.6(15)	14.5(15)	6.1(15)
C73	27.3(18)	18.6(17)	30.9(19)	9.8(15)	13.5(16)	6.8(15)
C75	34(2)	22.4(18)	44(2)	8.3(17)	21.5(18)	11.3(16)
C33	31.3(19)	18.6(17)	32.8(19)	10.8(15)	20.0(16)	6.2(15)

C29	30.5(19)	24.0(18)	39(2)	12.2(16)	22.9(17)	9.6(15)
C38	42(2)	21.8(18)	45(2)	17.9(17)	29.6(19)	12.4(16)
C76	31(2)	32(2)	37(2)	17.1(17)	15.0(17)	14.3(16)
C71	27.2(18)	25.5(18)	28.2(18)	11.5(15)	17.8(15)	10.7(15)
C77	42(2)	33(2)	27.9(19)	10.6(16)	18.6(17)	20.1(17)
C64	29.9(19)	23.0(18)	28.3(18)	9.8(15)	16.7(16)	9.5(15)
C56	50(2)	21.1(19)	40(2)	12.0(17)	19.8(19)	3.5(17)
C69	36(2)	41(2)	41(2)	21.1(19)	24.3(18)	13.3(18)
C70	31(2)	39(2)	37(2)	12.3(18)	16.4(17)	10.8(17)
C16	38(2)	42(2)	30(2)	8.1(18)	12.3(17)	7.4(19)
C68	41(2)	32(2)	57(3)	15.7(19)	36(2)	12.9(18)
C12	47(2)	46(2)	42(2)	20(2)	17(2)	24(2)
C58	62(3)	23.6(19)	52(2)	18.6(18)	39(2)	10.1(19)
C30	35(2)	44(2)	60(3)	28(2)	24(2)	16.8(19)
C31	52(3)	49(3)	48(3)	12(2)	34(2)	14(2)
C32	41(2)	37(2)	74(3)	28(2)	38(2)	15.2(19)
C57	51(2)	36(2)	55(3)	30(2)	21(2)	17.1(19)
C40	67(3)	24(2)	59(3)	15(2)	36(2)	7(2)
C13	61(3)	75(3)	49(3)	28(2)	19(2)	43(3)
C15	30(2)	76(3)	45(3)	16(2)	9.5(19)	14(2)
C14	40(3)	94(4)	37(2)	17(3)	10(2)	34(3)
C39	79(3)	34(2)	106(4)	40(3)	73(3)	25(2)
C41	71(3)	48(3)	74(3)	42(3)	26(3)	25(2)
Cl11	84.4(13)	83.6(13)	81.0(12)	46.7(11)	38.7(10)	34.3(10)
Cl12	66.7(12)	157(2)	89.6(14)	81.9(15)	45.6(11)	49.8(13)
Cl22	90.4(17)	79.3(15)	165(3)	31.1(16)	-39.0(17)	23.1(13)
Cl21	187(3)	170(3)	112(2)	72(2)	76(2)	114(3)
C200	105(6)	86(6)	70(5)	31(4)	-11(5)	58(5)
C100	61(5)	145(9)	255(15)	155(11)	54(7)	38(6)

Table 4 Bond Lengths for 16Jux_FA02_2.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
S1	O2	1.424(2)	C61	C66	1.388(5)
S1	O1	1.427(2)	C53	C54	1.389(5)
S1	N1	1.681(3)	C59	C54	1.389(5)
S1	C11	1.755(4)	C54	C55	1.539(5)
N1	C21	1.410(4)	C26	C34	1.416(5)
N1	C0BA	1.422(4)	C26	C20	1.411(5)
C23	C24	1.453(5)	C28	C33	1.375(5)
C23	C22	1.436(5)	C28	C29	1.534(5)

C23	C0BA	1.374(5)	C65	C67	1.533(5)
C24	C25	1.428(5)	C65	C66	1.400(5)
C24	C50	1.411(4)	C65	C64	1.383(5)
C49	C48	1.410(5)	C72	C73	1.389(5)
C49	C62	1.441(5)	C72	C71	1.390(5)
C49	C50	1.417(5)	C63	C64	1.407(5)
C48	C1AA	1.439(5)	C37	C42	1.392(5)
C48	C45	1.424(5)	C37	C36	1.385(5)
C25	C45	1.409(5)	C37	C38	1.541(5)
C25	C26	1.460(5)	C34	C35	1.461(5)
C22	C21	1.348(5)	C34	C33	1.410(5)
C51	C60	1.409(4)	C2AA	C73	1.387(5)
C51	C52	1.433(4)	C36	C35	1.402(5)
C51	C50	1.449(4)	C67	C69	1.534(5)
C60	C61	1.461(5)	C67	C70	1.537(5)
C60	C59	1.405(5)	C67	C68	1.531(5)
C52	C0BA	1.453(4)	C55	C56	1.529(5)
C52	C53	1.389(5)	C55	C58	1.523(5)
C44	C43	1.408(5)	C55	C57	1.540(5)
C44	C45	1.451(5)	C11	C16	1.389(5)
C44	C35	1.415(5)	C11	C12	1.376(5)
C1AA	C0AA	1.415(5)	C29	C30	1.549(5)
C1AA	C2AA	1.417(5)	C29	C31	1.525(5)
C62	C61	1.422(5)	C29	C32	1.527(5)
C62	C63	1.407(5)	C38	C40	1.533(6)
C74	C72	1.536(4)	C38	C39	1.519(5)
C74	C75	1.527(5)	C38	C41	1.526(6)
C74	C76	1.532(5)	C16	C15	1.389(6)
C74	C77	1.531(5)	C12	C13	1.391(6)
C43	C2AA	1.475(5)	C13	C14	1.376(7)
C43	C42	1.397(5)	C15	C14	1.366(7)
C0AA	C63	1.469(5)	Cl11	C100	1.673(9)
C0AA	C71	1.398(5)	Cl12	C100	1.706(9)
C27	C28	1.404(5)	Cl22	C200	1.721(7)
C27	C20	1.374(5)	Cl21	C200	1.791(9)

Table 5 Bond Angles for 16Jux_FA02_2.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
O2	S1	O1	118.90(15)	C59	C54	C53	118.0(3)
O2	S1	N1	104.99(14)	C59	C54	C55	122.9(3)

O2	S1	C11	109.84(16)	C34	C26	C25	119.8(3)
O1	S1	N1	110.00(14)	C20	C26	C25	122.4(3)
O1	S1	C11	108.76(16)	C20	C26	C34	117.5(3)
N1	S1	C11	103.19(15)	C27	C28	C29	120.1(3)
C21	N1	S1	117.9(2)	C33	C28	C27	116.7(3)
C21	N1	C0BA	106.7(3)	C33	C28	C29	123.1(3)
C0BA	N1	S1	130.6(2)	C66	C65	C67	119.7(3)
C22	C23	C24	130.3(3)	C64	C65	C67	122.6(3)
C0BA	C23	C24	121.4(3)	C64	C65	C66	117.6(3)
C0BA	C23	C22	107.9(3)	C73	C72	C74	122.2(3)
C25	C24	C23	124.2(3)	C73	C72	C71	117.4(3)
C50	C24	C23	116.0(3)	C71	C72	C74	120.2(3)
C50	C24	C25	119.6(3)	C62	C63	C0AA	119.3(3)
C48	C49	C62	119.9(3)	C64	C63	C62	118.9(3)
C48	C49	C50	119.6(3)	C64	C63	C0AA	121.7(3)
C50	C49	C62	120.1(3)	C42	C37	C38	120.4(3)
C49	C48	C1AA	120.3(3)	C36	C37	C42	117.4(3)
C49	C48	C45	119.4(3)	C36	C37	C38	122.2(3)
C45	C48	C1AA	120.4(3)	C26	C34	C35	119.2(3)
C24	C25	C26	123.5(3)	C33	C34	C26	118.8(3)
C45	C25	C24	118.2(3)	C33	C34	C35	121.6(3)
C45	C25	C26	118.3(3)	C1AA	C2AA	C43	119.1(3)
C21	C22	C23	107.8(3)	C73	C2AA	C1AA	119.3(3)
C60	C51	C52	118.3(3)	C73	C2AA	C43	121.6(3)
C60	C51	C50	120.0(3)	C37	C42	C43	122.8(3)
C52	C51	C50	121.6(3)	C37	C36	C35	122.3(3)
C51	C60	C61	119.0(3)	C65	C67	C69	110.7(3)
C59	C60	C51	119.2(3)	C65	C67	C70	108.9(3)
C59	C60	C61	121.7(3)	C69	C67	C70	108.3(3)
C51	C52	C0BA	114.5(3)	C68	C67	C65	112.0(3)
C53	C52	C51	119.5(3)	C68	C67	C69	108.7(3)
C53	C52	C0BA	126.0(3)	C68	C67	C70	108.1(3)
C43	C44	C45	120.7(3)	C54	C55	C57	108.3(3)
C43	C44	C35	119.3(3)	C56	C55	C54	109.5(3)
C35	C44	C45	119.9(3)	C56	C55	C57	109.1(3)
C0AA	C1AA	C48	120.3(3)	C58	C55	C54	112.0(3)
C0AA	C1AA	C2AA	119.1(3)	C58	C55	C56	108.7(3)
C2AA	C1AA	C48	120.6(3)	C58	C55	C57	109.3(3)
C22	C21	N1	109.6(3)	C27	C20	C26	121.5(3)
C61	C62	C49	119.8(3)	C44	C35	C34	118.9(3)
C63	C62	C49	120.7(3)	C36	C35	C44	119.1(3)
C63	C62	C61	119.4(3)	C36	C35	C34	121.8(3)

C24	C50	C49	120.2(3)	C16	C11	S1	118.2(3)
C24	C50	C51	120.2(3)	C12	C11	S1	120.1(3)
C49	C50	C51	119.6(3)	C12	C11	C16	121.6(4)
C75	C74	C72	112.2(3)	C61	C66	C65	122.4(3)
C75	C74	C76	108.8(3)	C2AA	C73	C72	122.7(3)
C75	C74	C77	108.0(3)	C28	C33	C34	123.4(3)
C76	C74	C72	107.8(3)	C28	C29	C30	109.4(3)
C77	C74	C72	110.9(3)	C31	C29	C28	109.0(3)
C77	C74	C76	109.1(3)	C31	C29	C30	109.4(3)
C44	C43	C2AA	119.7(3)	C31	C29	C32	109.7(3)
C42	C43	C44	118.9(3)	C32	C29	C28	112.0(3)
C42	C43	C2AA	121.4(3)	C32	C29	C30	107.3(3)
N1	C0BA	C52	129.5(3)	C40	C38	C37	109.8(3)
C23	C0BA	N1	107.9(3)	C39	C38	C37	112.1(3)
C23	C0BA	C52	122.6(3)	C39	C38	C40	107.4(4)
C1AA	C0AA	C63	119.4(3)	C39	C38	C41	109.9(4)
C71	C0AA	C1AA	118.9(3)	C41	C38	C37	109.0(3)
C71	C0AA	C63	121.7(3)	C41	C38	C40	108.6(3)
C20	C27	C28	121.9(3)	C72	C71	C0AA	122.6(3)
C62	C61	C60	119.4(3)	C65	C64	C63	122.4(3)
C66	C61	C60	121.4(3)	C15	C16	C11	118.9(4)
C66	C61	C62	118.9(3)	C11	C12	C13	118.5(4)
C52	C53	C54	122.1(3)	C14	C13	C12	119.9(5)
C54	C59	C60	122.2(3)	C14	C15	C16	119.6(4)
C48	C45	C44	119.4(3)	C15	C14	C13	121.4(4)
C25	C45	C48	120.8(3)	Cl22	C200	Cl21	109.5(4)
C25	C45	C44	119.7(3)	Cl11	C100	Cl12	119.6(5)
C53	C54	C55	119.1(3)				

Table 6 Torsion Angles for 16Jux_FA02_2.

A	**B**	**C**	**D**	**Angle/°**	**A**	**B**	**C**	**D**	**Angle/°**
S1	N1	C21	C22	160.7(2)	C43	C2AA	C73	C72	175.9(3)
S1	N1	C0BA	C23	-157.5(3)	C0BA	N1	C21	C22	2.5(4)
S1	N1	C0BA	C52	19.5(5)	C0BA	C23	C24	C25	170.2(3)
S1	C11	C16	C15	-175.9(3)	C0BA	C23	C24	C50	-15.4(4)
S1	C11	C12	C13	176.6(3)	C0BA	C23	C22	C21	-1.1(4)
O2	S1	N1	C21	13.4(3)	C0BA	C52	C53	C54	-177.8(3)
O2	S1	N1	C0BA	165.5(3)	C0AA	C1AA	C2AA	C43	-177.1(3)
O2	S1	C11	C16	106.6(3)	C0AA	C1AA	C2AA	C73	1.0(5)
O2	S1	C11	C12	-69.5(3)	C0AA	C63	C64	C65	175.7(3)

O1	S1	N1	C21	142.4(2)	C27	C28	C33	C34	-1.5(5)
O1	S1	N1	C0BA	-65.5(3)	C27	C28	C29	C30	-52.7(4)
O1	S1	C11	C16	-25.1(3)	C27	C28	C29	C31	66.9(4)
O1	S1	C11	C12	158.8(3)	C27	C28	C29	C32	-171.5(3)
N1	S1	C11	C16	-141.9(3)	C61	C60	C59	C54	171.4(3)
N1	S1	C11	C12	42.0(3)	C61	C62	C63	C0AA	178.8(3)
C23	C24	C25	C45	156.7(3)	C61	C62	C63	C64	-5.4(5)
C23	C24	C25	C26	-26.0(5)	C53	C52	C0BA	N1	17.4(5)
C23	C24	C50	C49	-164.0(3)	C53	C52	C0BA	C23	-165.8(3)
C23	C24	C50	C51	18.2(4)	C53	C54	C55	C56	-50.7(4)
C23	C22	C21	N1	-0.9(4)	C53	C54	C55	C58	-171.3(3)
C24	C23	C22	C21	-173.9(3)	C53	C54	C55	C57	68.1(4)
C24	C23	C0BA	N1	176.1(3)	C59	C60	C61	C62	169.1(3)
C24	C23	C0BA	C52	-1.2(5)	C59	C60	C61	C66	-17.6(5)
C24	C25	C45	C48	11.4(5)	C59	C54	C55	C56	129.3(4)
C24	C25	C45	C44	-164.7(3)	C59	C54	C55	C58	8.7(5)
C24	C25	C26	C34	163.0(3)	C59	C54	C55	C57	-111.9(4)
C24	C25	C26	C20	-23.9(5)	C45	C48	C1AA	C0AA	-179.6(3)
C49	C48	C1AA	C0AA	0.1(5)	C45	C48	C1AA	C2AA	2.2(5)
C49	C48	C1AA	C2AA	-178.0(3)	C45	C25	C26	C34	-19.7(5)
C49	C48	C45	C25	1.7(5)	C45	C25	C26	C20	153.5(3)
C49	C48	C45	C44	177.7(3)	C45	C44	C43	C2AA	3.9(5)
C49	C62	C61	C60	2.5(5)	C45	C44	C43	C42	-173.6(3)
C49	C62	C61	C66	-171.1(3)	C45	C44	C35	C34	-14.2(5)
C49	C62	C63	C0AA	-4.1(5)	C45	C44	C35	C36	170.8(3)
C49	C62	C63	C64	171.7(3)	C26	C25	C45	C48	-166.1(3)
C48	C49	C62	C61	-179.0(3)	C26	C25	C45	C44	17.8(4)
C48	C49	C62	C63	3.9(5)	C26	C34	C35	C44	12.3(5)
C48	C49	C50	C24	2.6(5)	C26	C34	C35	C36	-172.9(3)
C48	C49	C50	C51	-179.6(3)	C26	C34	C33	C28	-2.3(5)
C48	C1AA	C0AA	C63	-0.4(5)	C28	C27	C20	C26	-0.5(5)
C48	C1AA	C0AA	C71	-178.2(3)	C63	C62	C61	C60	179.6(3)
C48	C1AA	C2AA	C43	1.1(5)	C63	C62	C61	C66	6.1(5)
C48	C1AA	C2AA	C73	179.2(3)	C63	C0AA	C71	C72	-177.8(3)
C25	C24	C50	C49	10.7(5)	C37	C36	C35	C44	4.5(5)
C25	C24	C50	C51	-167.1(3)	C37	C36	C35	C34	-170.3(3)
C25	C26	C34	C35	4.5(5)	C34	C26	C20	C27	-3.3(5)
C25	C26	C34	C33	178.1(3)	C2AA	C1AA	C0AA	C63	177.8(3)
C25	C26	C20	C27	-176.6(3)	C2AA	C1AA	C0AA	C71	0.0(5)
C22	C23	C24	C25	-17.9(5)	C2AA	C43	C42	C37	-176.2(3)
C22	C23	C24	C50	156.5(3)	C42	C43	C2AA	C1AA	173.3(3)
C22	C23	C0BA	N1	2.6(4)	C42	C43	C2AA	C73	-4.8(5)

C22	C23	C0BA	C52	-174.7(3)	C42	C37	C36	C35	-0.4(5)
C51	C60	C61	C62	-14.0(5)	C42	C37	C38	C40	46.4(5)
C51	C60	C61	C66	159.3(3)	C42	C37	C38	C39	165.7(4)
C51	C60	C59	C54	-5.5(5)	C42	C37	C38	C41	-72.4(4)
C51	C52	C0BA	N1	-162.4(3)	C36	C37	C42	C43	-2.6(5)
C51	C52	C0BA	C23	14.3(4)	C36	C37	C38	C40	-133.2(4)
C51	C52	C53	C54	2.1(5)	C36	C37	C38	C39	-13.9(5)
C60	C51	C52	C0BA	171.4(3)	C36	C37	C38	C41	108.0(4)
C60	C51	C52	C53	-8.5(5)	C67	C65	C66	C61	171.6(3)
C60	C51	C50	C24	172.3(3)	C67	C65	C64	C63	-170.7(3)
C60	C51	C50	C49	-5.5(5)	C20	C27	C28	C33	2.9(5)
C60	C61	C66	C65	-174.8(3)	C20	C27	C28	C29	-179.9(3)
C60	C59	C54	C53	-1.0(5)	C20	C26	C34	C35	-168.9(3)
C60	C59	C54	C55	179.0(3)	C20	C26	C34	C33	4.6(5)
C52	C51	C60	C61	-166.9(3)	C35	C44	C43	C2AA	-179.5(3)
C52	C51	C60	C59	10.0(5)	C35	C44	C43	C42	3.0(5)
C52	C51	C50	C24	-5.1(5)	C35	C44	C45	C48	-177.1(3)
C52	C51	C50	C49	177.0(3)	C35	C44	C45	C25	-1.0(5)
C52	C53	C54	C59	2.7(5)	C35	C34	C33	C28	171.1(3)
C52	C53	C54	C55	-177.3(3)	C11	S1	N1	C21	-101.7(3)
C44	C43	C2AA	C1AA	-4.1(5)	C11	S1	N1	C0BA	50.4(3)
C44	C43	C2AA	C73	177.8(3)	C11	C16	C15	C14	-1.8(6)
C44	C43	C42	C37	1.3(5)	C11	C12	C13	C14	0.1(6)
C1AA	C48	C45	C25	-178.6(3)	C66	C65	C67	C69	58.1(4)
C1AA	C48	C45	C44	-2.5(5)	C66	C65	C67	C70	-60.8(4)
C1AA	C0AA	C63	C62	2.4(5)	C66	C65	C67	C68	179.6(3)
C1AA	C0AA	C63	C64	-173.3(3)	C66	C65	C64	C63	4.6(5)
C1AA	C0AA	C71	C72	0.0(5)	C73	C72	C71	C0AA	-1.0(5)
C1AA	C2AA	C73	C72	-2.2(5)	C75	C74	C72	C73	-14.0(4)
C21	N1	C0BA	C23	-3.1(3)	C75	C74	C72	C71	170.7(3)
C21	N1	C0BA	C52	174.0(3)	C33	C28	C29	C30	124.4(4)
C62	C49	C48	C1AA	-1.8(5)	C33	C28	C29	C31	-116.1(4)
C62	C49	C48	C45	177.9(3)	C33	C28	C29	C32	5.6(5)
C62	C49	C50	C24	175.9(3)	C33	C34	C35	C44	-161.0(3)
C62	C49	C50	C51	-6.2(5)	C33	C34	C35	C36	13.8(5)
C62	C61	C66	C65	-1.4(5)	C29	C28	C33	C34	-178.6(3)
C62	C63	C64	C65	0.0(5)	C38	C37	C42	C43	177.8(3)
C50	C24	C25	C45	-17.5(5)	C38	C37	C36	C35	179.3(3)
C50	C24	C25	C26	159.8(3)	C76	C74	C72	C73	105.7(4)
C50	C49	C48	C1AA	171.5(3)	C76	C74	C72	C71	-69.6(4)
C50	C49	C48	C45	-8.8(5)	C71	C0AA	C63	C62	-179.9(3)
C50	C49	C62	C61	7.7(5)	C71	C0AA	C63	C64	4.4(5)

C50	C49	C62	C63	-169.4(3)	C71	C72	C73	C2AA	2.1(5)
C50	C51	C60	C61	15.6(4)	C77	C74	C72	C73	-134.9(3)
C50	C51	C60	C59	-167.5(3)	C77	C74	C72	C71	49.8(4)
C50	C51	C52	C0BA	-11.1(4)	C64	C65	C67	C69	-126.7(4)
C50	C51	C52	C53	169.0(3)	C64	C65	C67	C70	114.4(4)
C74	C72	C73	C2AA	-173.3(3)	C64	C65	C67	C68	-5.2(5)
C74	C72	C71	C0AA	174.5(3)	C64	C65	C66	C61	-3.9(5)
C43	C44	C45	C48	-0.6(5)	C16	C11	C12	C13	0.6(6)
C43	C44	C45	C25	175.6(3)	C16	C15	C14	C13	2.5(7)
C43	C44	C35	C34	169.2(3)	C12	C11	C16	C15	0.2(6)
C43	C44	C35	C36	-5.8(5)	C12	C13	C14	C15	-1.7(7)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 16Jux_FA02_2.

Atom	***x***	***y***	***z***	**U(eq)**
H22	5363	2565	451	31
H21	4568	783	-726	33
H27	8300	3242	1914	33
H53	3035	-535	1350	30
H59	2015	667	3436	30
H42	6148	8074	4045	35
H36	7685	6953	2441	34
H20	6692	2851	2115	30
H66	1311	1701	3755	32
H73	5181	7894	4756	31
H75A	5218	9094	6163	51
H75B	4294	9514	6096	51
H75C	4496	9010	5128	51
H33	8454	5981	2295	33
H76A	2610	7746	4162	48
H76B	2342	8304	5068	48
H76C	2017	7122	4643	48
H71	2875	6042	5161	31
H77A	4281	8096	6935	49
H77B	3016	7367	6335	49
H77C	3396	8557	6774	49
H64	1935	4639	4859	32
H56A	738	-1751	1025	60
H56B	1015	-2578	1311	60
H56C	1778	-1945	941	60

H69A	466	1698	4792	56
H69B	-291	2158	5211	56
H69C	1004	2742	5766	56
H70A	-620	2562	3059	56
H70B	-1262	2028	3575	56
H70C	-498	1593	3142	56
H16	953	-1909	-758	50
H68A	828	4275	5619	62
H68B	-461	3636	5080	62
H68C	163	4173	4556	62
H12	2277	749	-822	53
H58A	1784	-695	3686	65
H58B	1027	-1847	2926	65
H58C	739	-979	2738	65
H30A	11110	4868	2577	66
H30B	10114	3970	2485	66
H30C	10521	5086	3318	66
H31A	10061	4304	767	77
H31B	8905	4352	388	77
H31C	9012	3495	714	77
H32A	9679	6121	1650	70
H32B	10847	6098	2046	70
H32C	10290	6414	2796	70
H57A	2720	-2030	2794	67
H57B	3432	-861	3514	67
H57C	3437	-1336	2439	67
H40A	7335	9552	4493	77
H40B	8317	10185	4315	77
H40C	8449	9410	4757	77
H13	574	833	-1194	71
H15	-748	-1824	-1168	68
H14	-898	-429	-1317	72
H39A	8980	8653	3348	93
H39B	8781	9475	3003	93
H39C	8174	8310	2237	93
H41A	6342	8332	1884	91
H41B	6942	9516	2575	91
H41C	6033	8824	2784	91
H20A	3120	2855	654	114
H20B	3251	3977	1243	114
H10A	4119	5936	1125	165
H10B	4512	6932	2105	165

Table 8 Atomic Occupancy for 16Jux_FA02_2.

Atom	***Occupancy***	**Atom**	***Occupancy***	**Atom**	***Occupancy***
Cl11	0.75	Cl12	0.75	Cl22	0.75
Cl21	0.75	C200	0.75	H20A	0.75
H20B	0.75	C100	0.75	H10A	0.75
H10B	0.75				

Experimental

Single crystals of $C_{67.5}H_{66}Cl_3NO_2S$ [16Jux_FA02_2] were [crystallized from DCM/EtOH]. A suitable crystal was selected and [mounted on a loop] on a SuperNova, Dual, Cu at zero, Atlas diffractometer. The crystal was kept at 152.7(10) K during data collection. Using Olex2 [1], the structure was solved with the ShelXS [2] structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [16Jux_FA02_2]

Crystal Data for $C_{67.5}H_{66}Cl_3NO_2S$ (M =1061.62 g/mol): triclinic, space group P-1 (no. 2), a = 14.0225(7) Å, b = 15.4902(10) Å, c = 15.6494(5) Å, α = 109.303(4)°, β = 105.791(4)°, γ = 106.459(5)°, V = 2814.2(3) Å^3, Z = 2, T = 152.7(10) K, μ(CuKα) = 2.173 mm^{-1}, *Dcalc* = 1.253 g/cm^3, 11987 reflections measured (7.186° ≤ 2Θ ≤ 113.446°), 7226 unique (R_{int} = 0.0257, R_{sigma} = 0.0357) which were used in all calculations. The final R_1 was 0.0729 (I > 2σ(I)) and wR_2 was 0.2094 (all data).

Refinement model description

Number of restraints - 1, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2. Restrained distances
 Cl21-C200 ≈ Cl22-C200
 with sigma of 0.02
 Cl22-C200 ≈ Cl21-C200
 with sigma of 0.02
3. Uiso/Uaniso restraints and constraints
Cl21 ≈ Cl22: within 1.7A with sigma of 0.04 and sigma for terminal atoms of
0.08
Cl11 ≈ Cl12: within 1.7A with sigma of 0.04 and sigma for terminal atoms of
0.08
```

```
4. Others
 Fixed Sof: Cl11(0.75) Cl12(0.75) Cl22(0.75) Cl21(0.75) C200(0.75)
H20A(0.75)
 H20B(0.75) C100(0.75) H10A(0.75) H10B(0.75)
5.a Secondary CH2 refined with riding coordinates:
 C200(H20A,H20B), C100(H10A,H10B)
5.b Aromatic/amide H refined with riding coordinates:
 C22(H22), C21(H21), C27(H27), C53(H53), C59(H59), C42(H42), C36(H36),
 C20(H20), C66(H66), C73(H73), C33(H33), C71(H71), C64(H64), C16(H16),
C12(H12),
  C13(H13), C15(H15), C14(H14)
5.c Idealised Me refined as rotating group:
 C75(H75A,H75B,H75C), C76(H76A,H76B,H76C), C77(H77A,H77B,H77C),
C56(H56A,H56B,
 H56C), C69(H69A,H69B,H69C), C70(H70A,H70B,H70C), C68(H68A,H68B,H68C),
C58(H58A,
 H58B,H58C), C30(H30A,H30B,H30C), C31(H31A,H31B,H31C), C32(H32A,H32B,H32C),
 C57(H57A,H57B,H57C), C40(H40A,H40B,H40C), C39(H39A,H39B,H39C),
C41(H41A,H41B,
```