Synthesis of mono- and di-arylated acenaphthylenes and programmed access to dibenzo[*j*,*l*]fluoranthenes *via* palladium-catalysed C-H bond functionalisation

Xinzhe Shi,[a] *Thierry Roisnel,*[a] *Jean-François Soulé,**[a] *Henri Doucet**[a]

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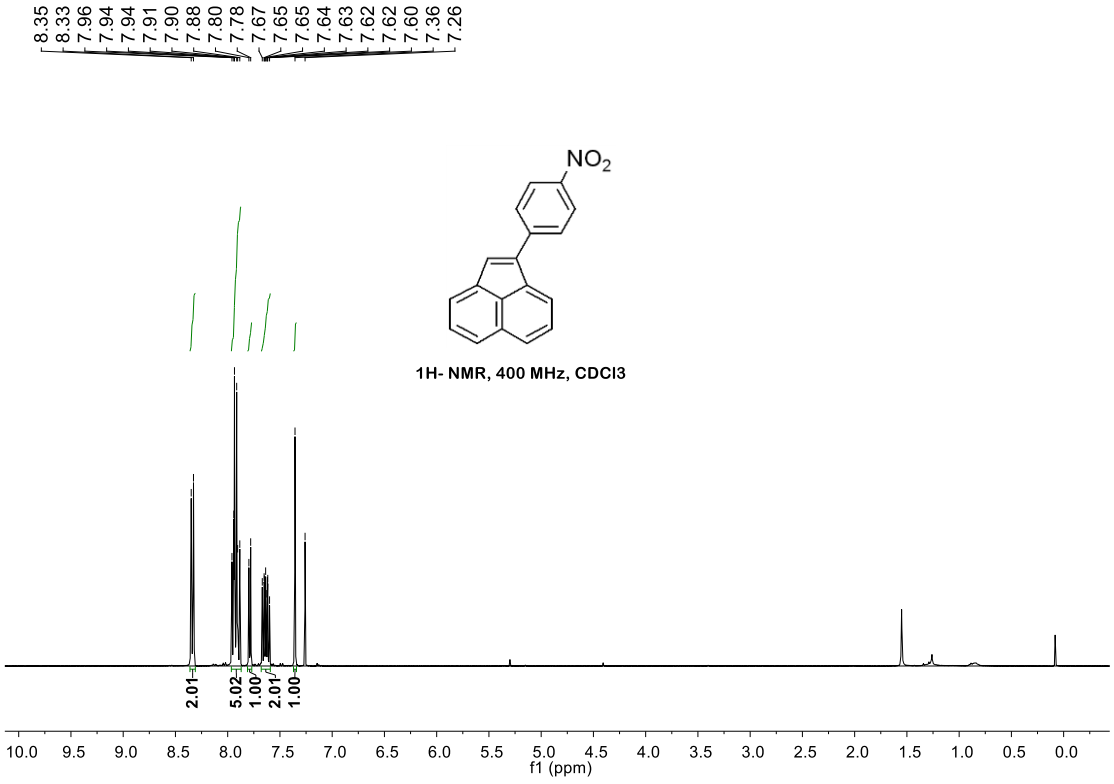
Contents

1a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7.62
7.60
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7.26
NO2
1H- NMR, 400 MHz, CDCl3
2.01
5.02
1.00
2.01
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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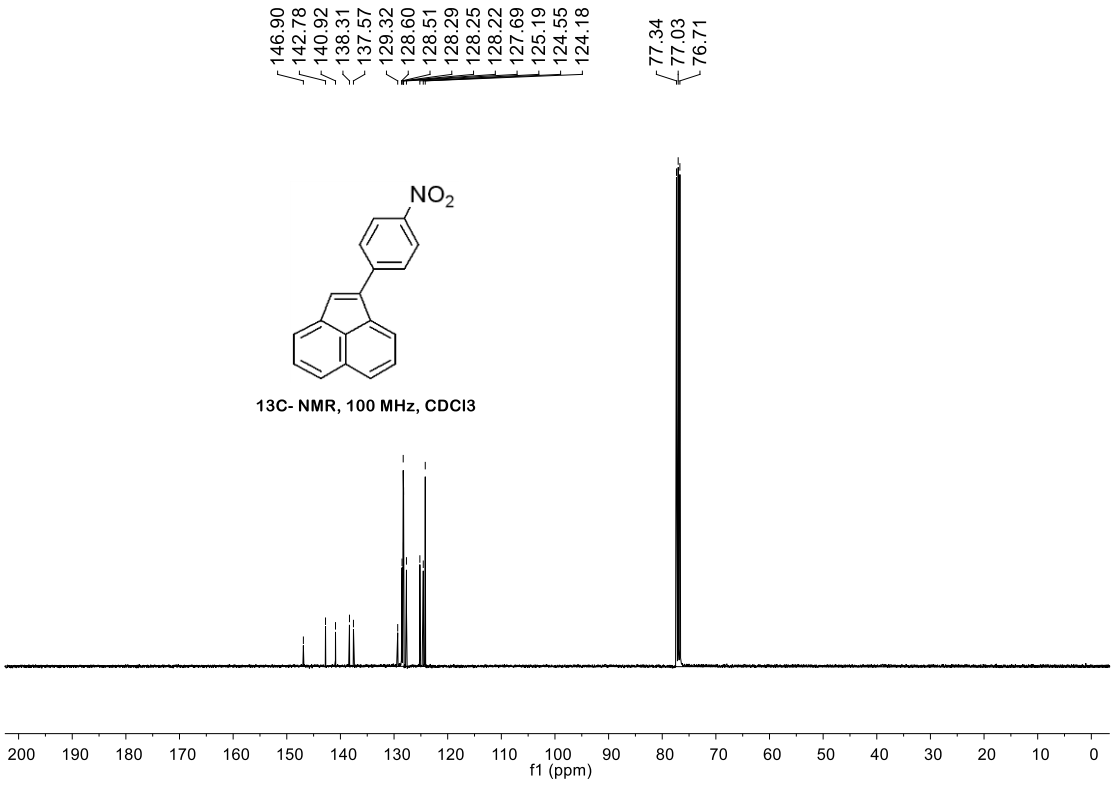
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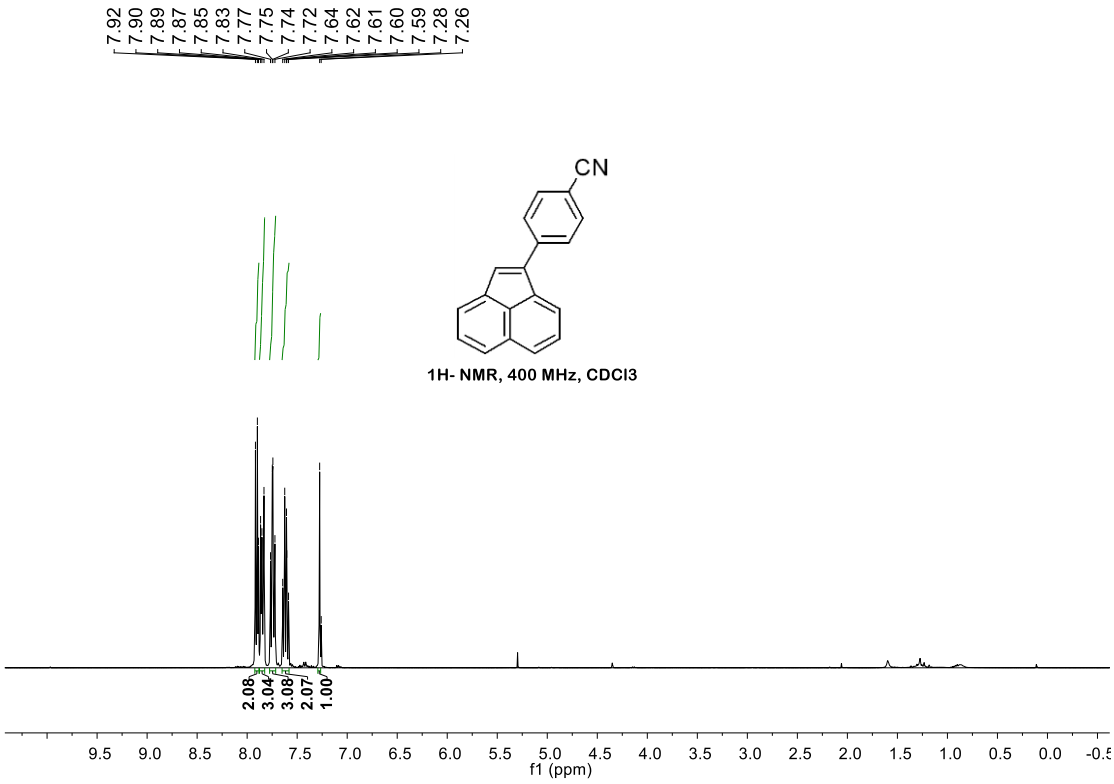
2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CN
1H- NMR, 400 MHz, CDCl3
2.08
3.04
3.08
2.07
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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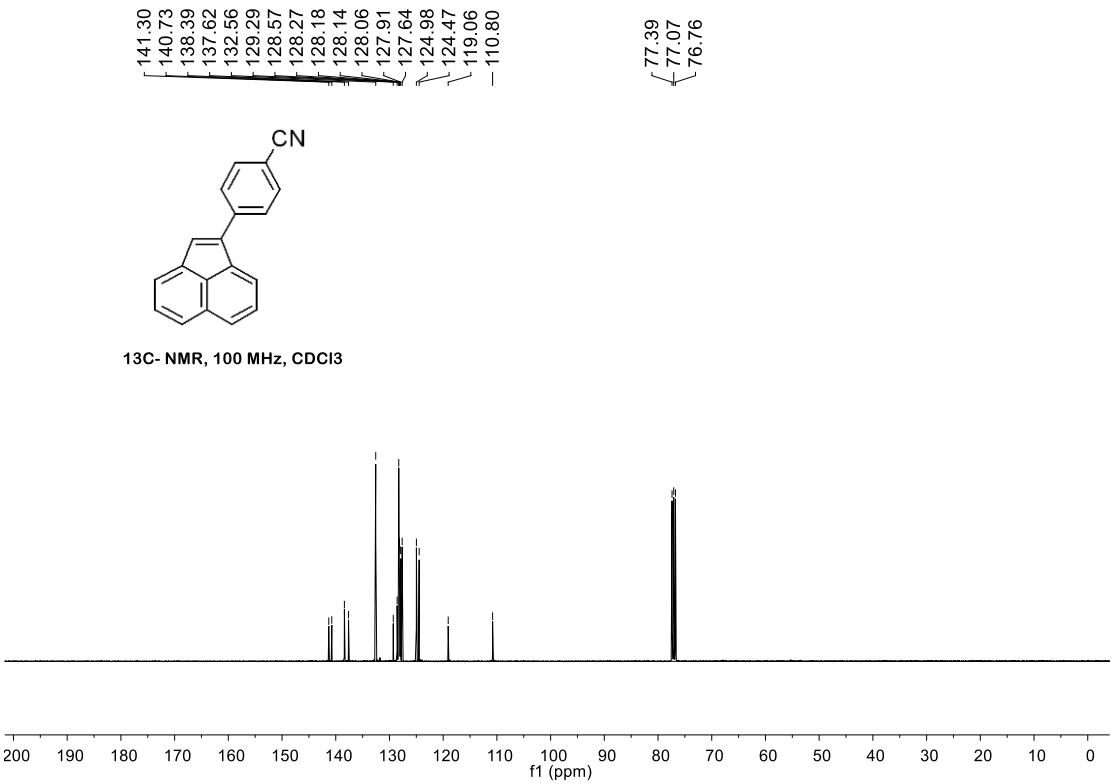
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f1 (ppm)

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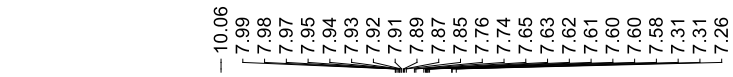

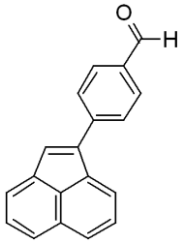

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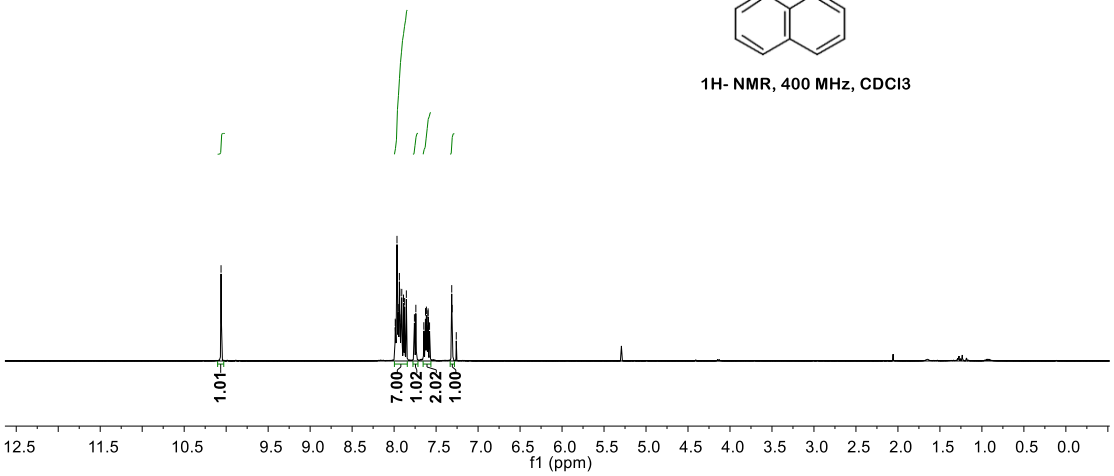

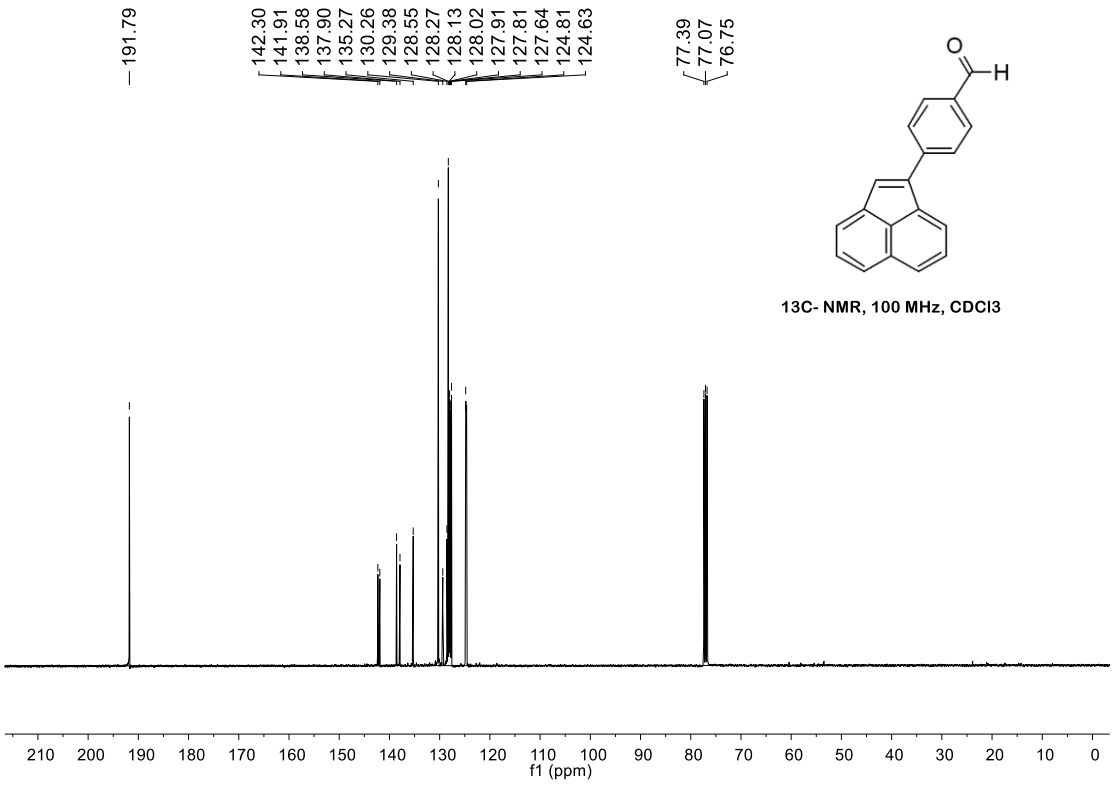

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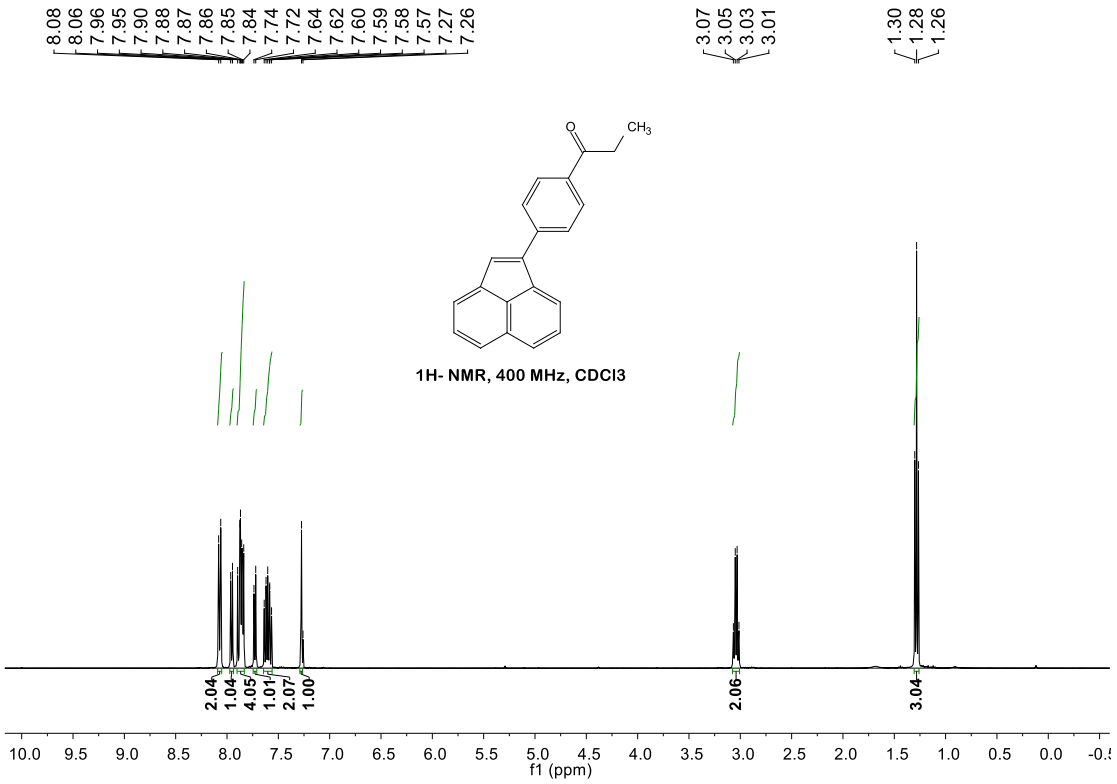
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7.57
7.27
7.26
3.07
3.05
3.03
3.01
1.30
1.28
1.26
O
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1H- NMR, 400 MHz, CDCl3
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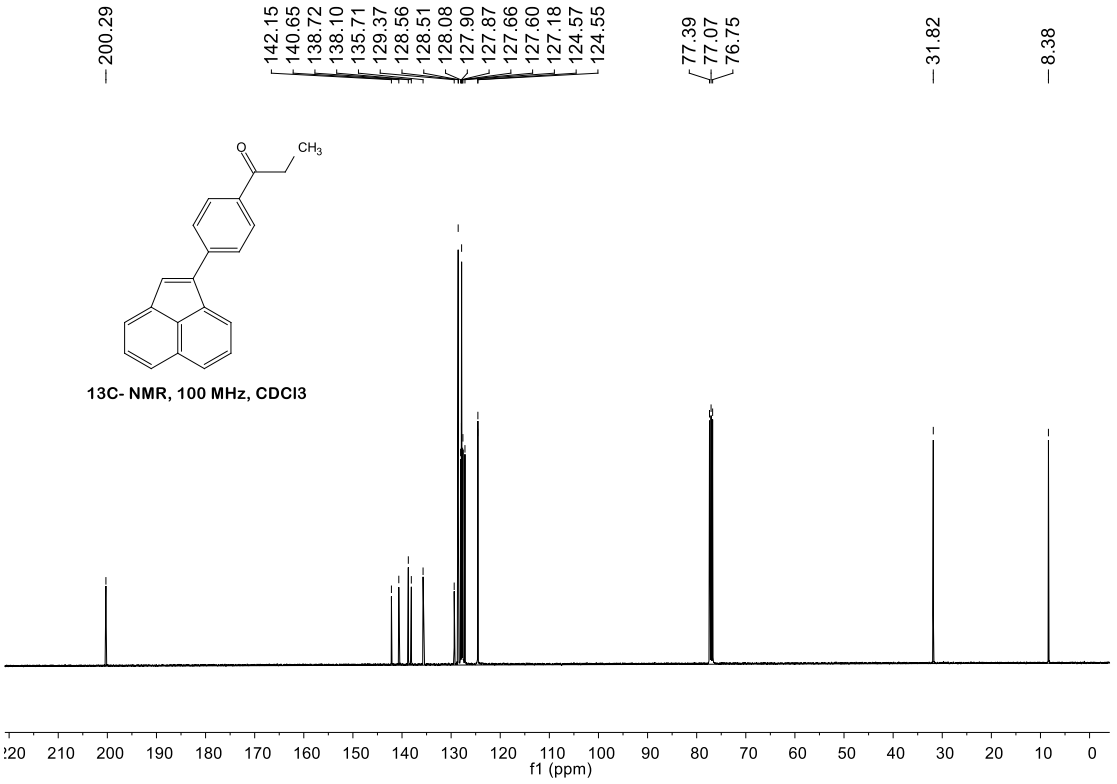
4.05
1.01
2.07
1.00
2.06
3.04
f1 (ppm)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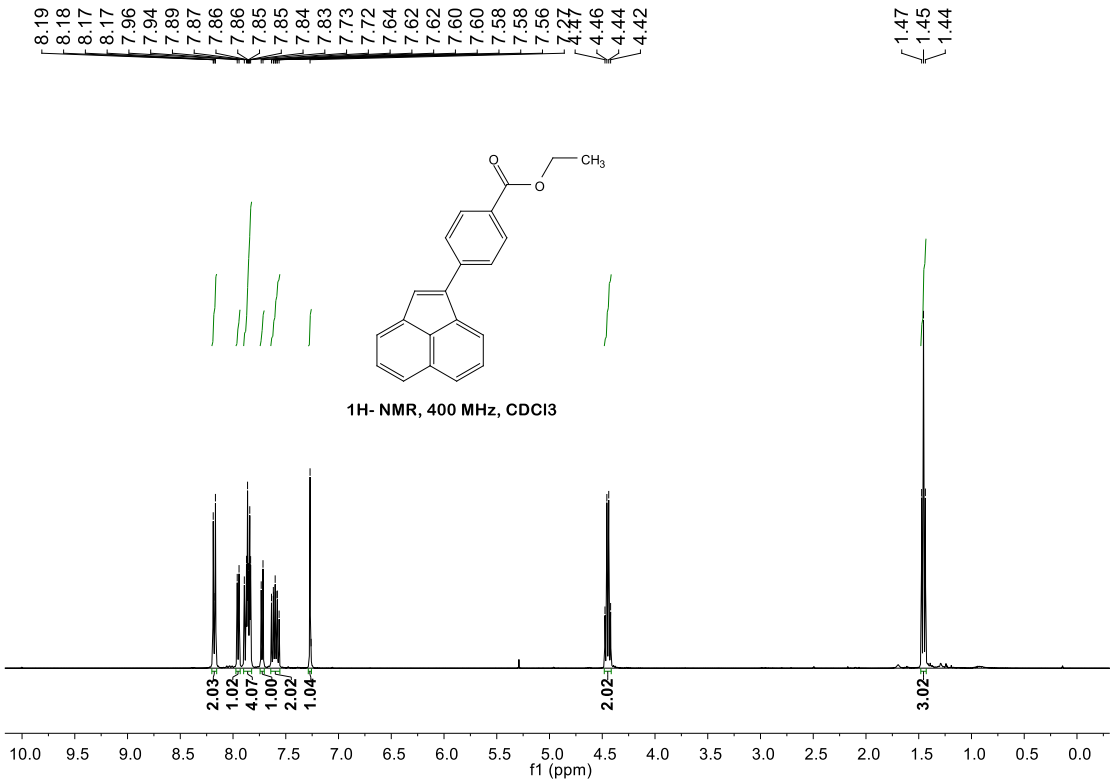
O
CH3
13C- NMR, 100 MHz, CDCl3
f1 (ppm)

5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7.27
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1.44
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O
CH3
1H- NMR, 400 MHz, CDCl3
2.03
1.02
4.07
1.00
2.02
1.04
2.02
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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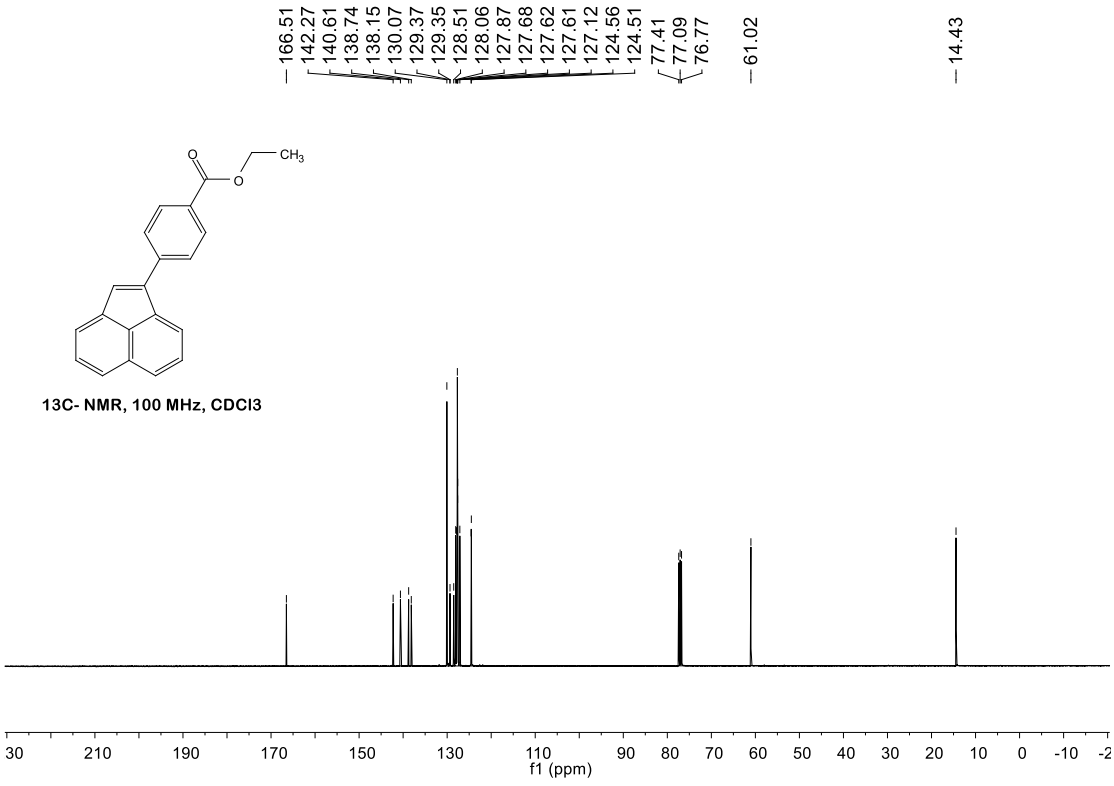
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127.61
127.12
124.56
124.51
77.41
77.09
76.77
61.02
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CH3
13C- NMR, 100 MHz, CDCl3
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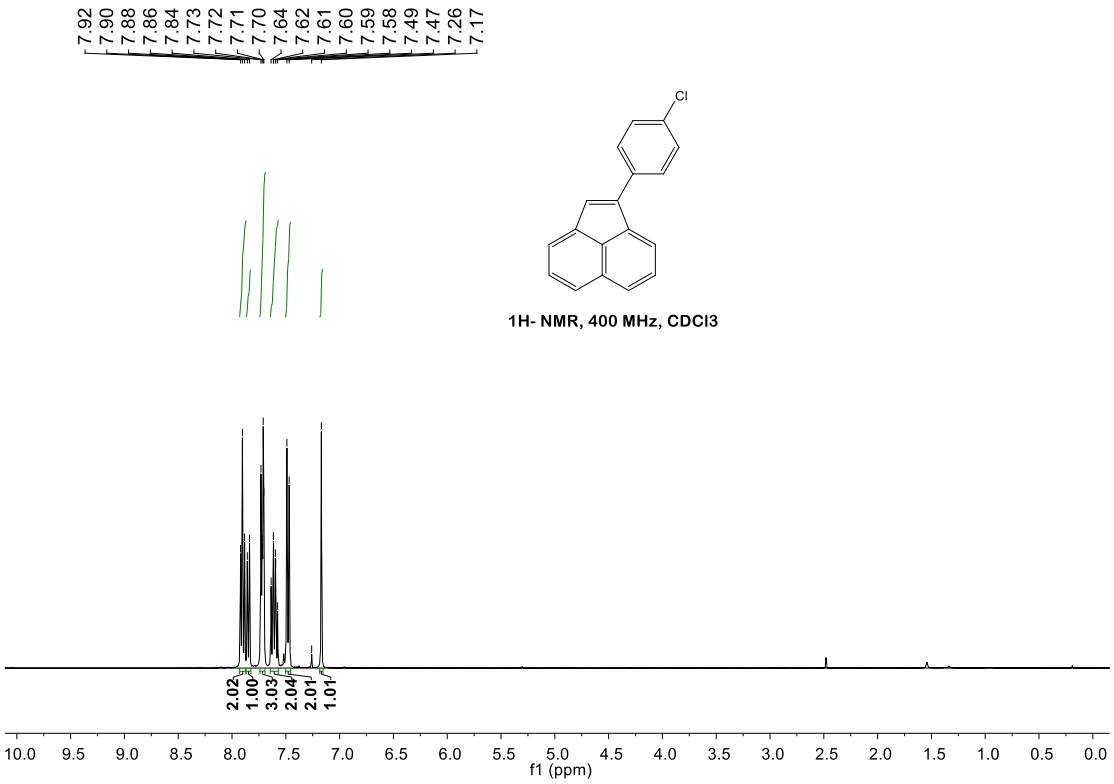
f1 (ppm)

6



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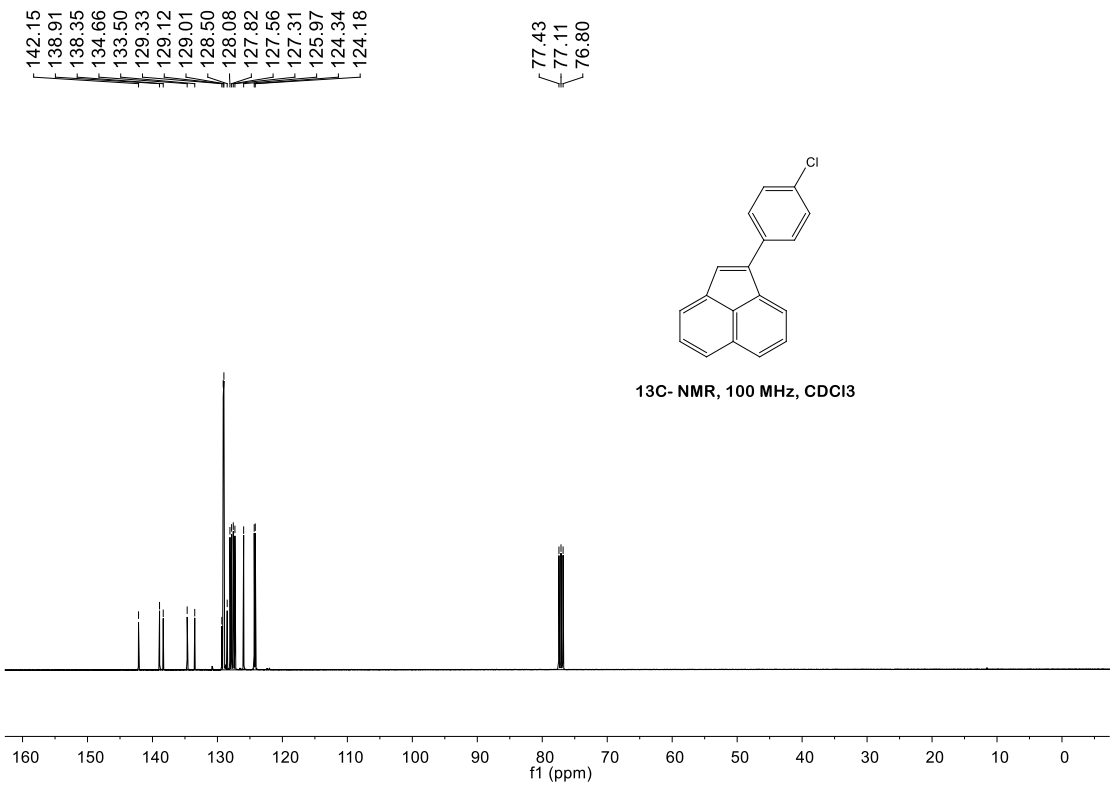

13C- NMR, 100 MHz, CDCl3

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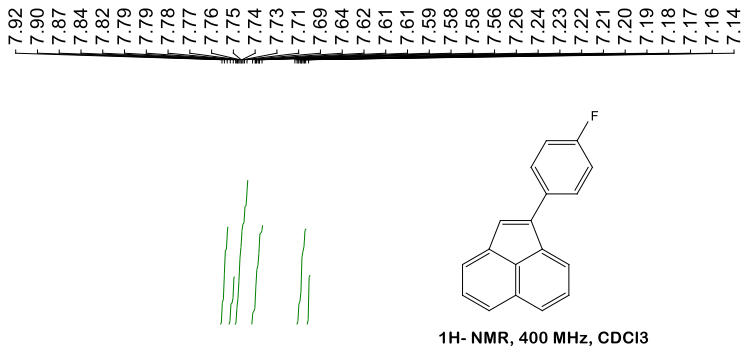

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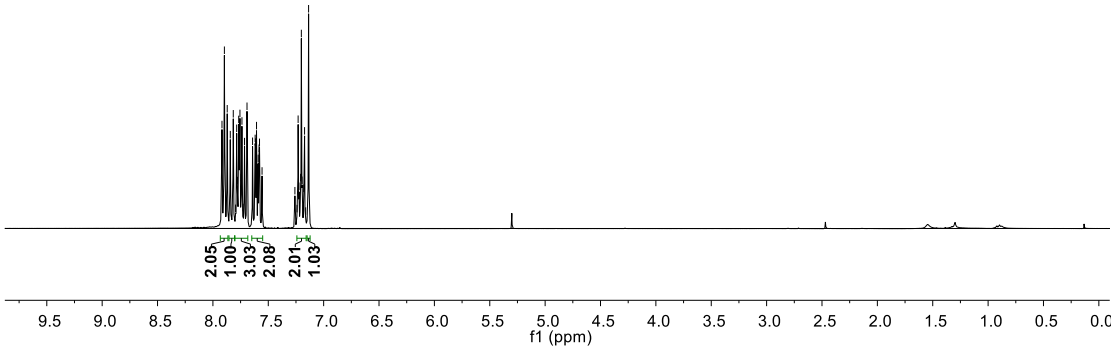

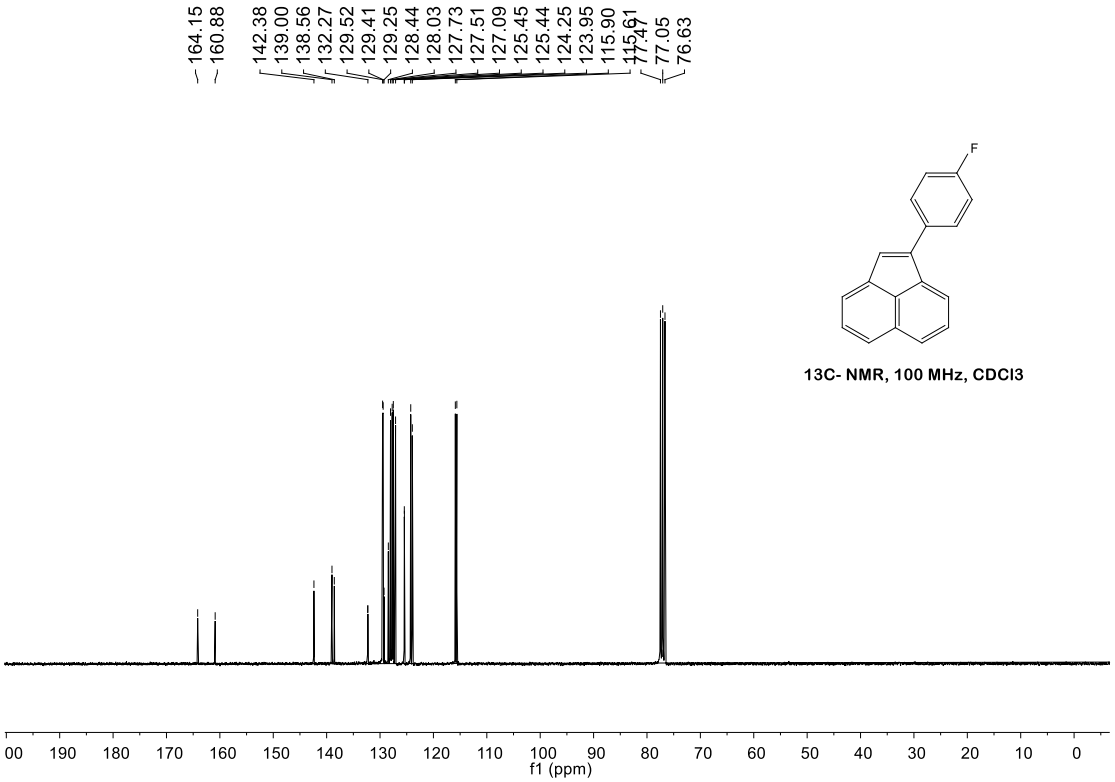

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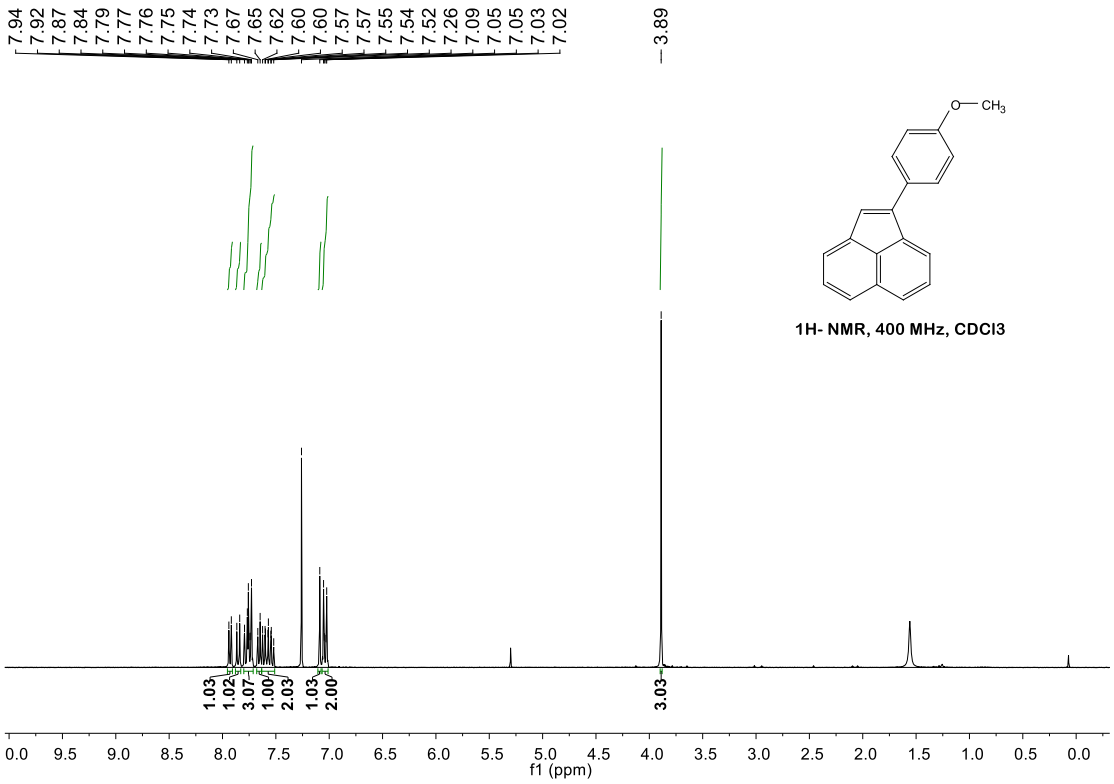

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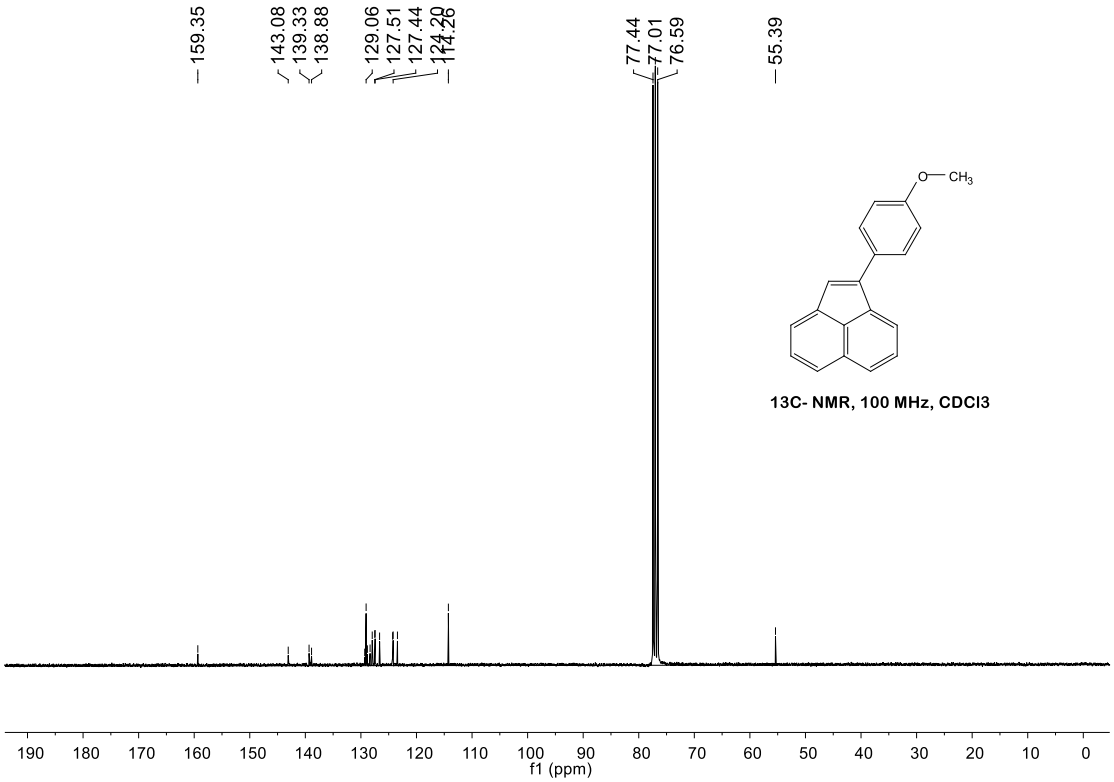

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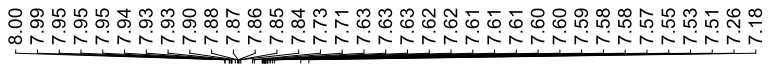

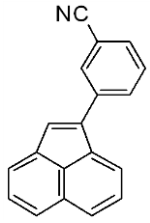

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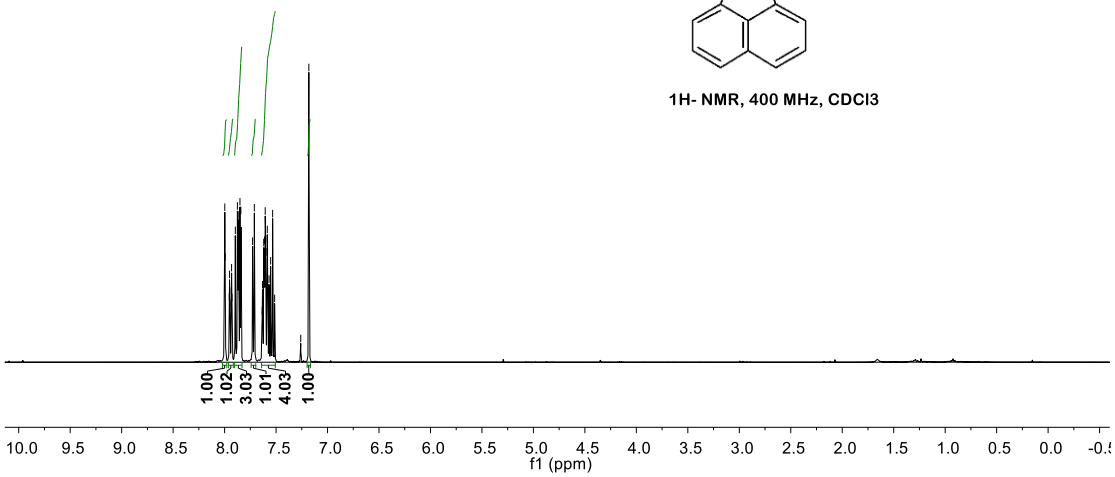

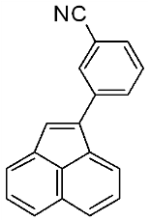

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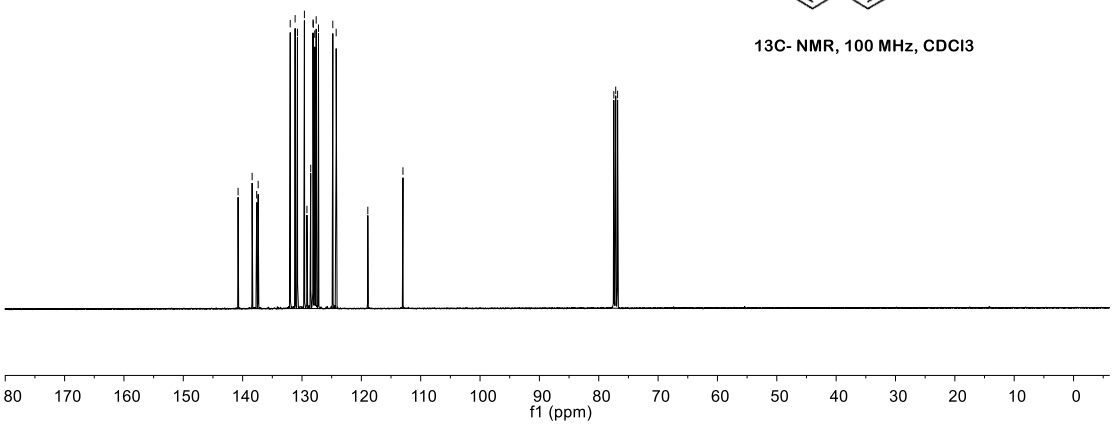

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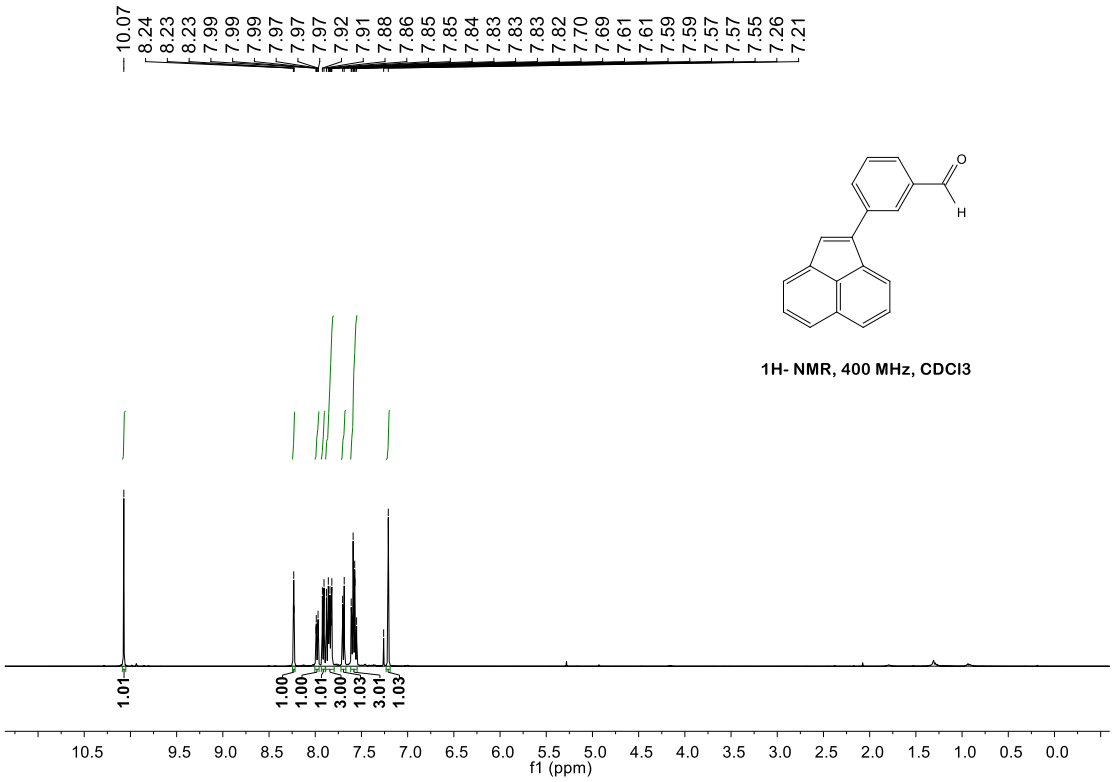
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7.21
O
H
1H- NMR, 400 MHz, CDCl3
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1.00
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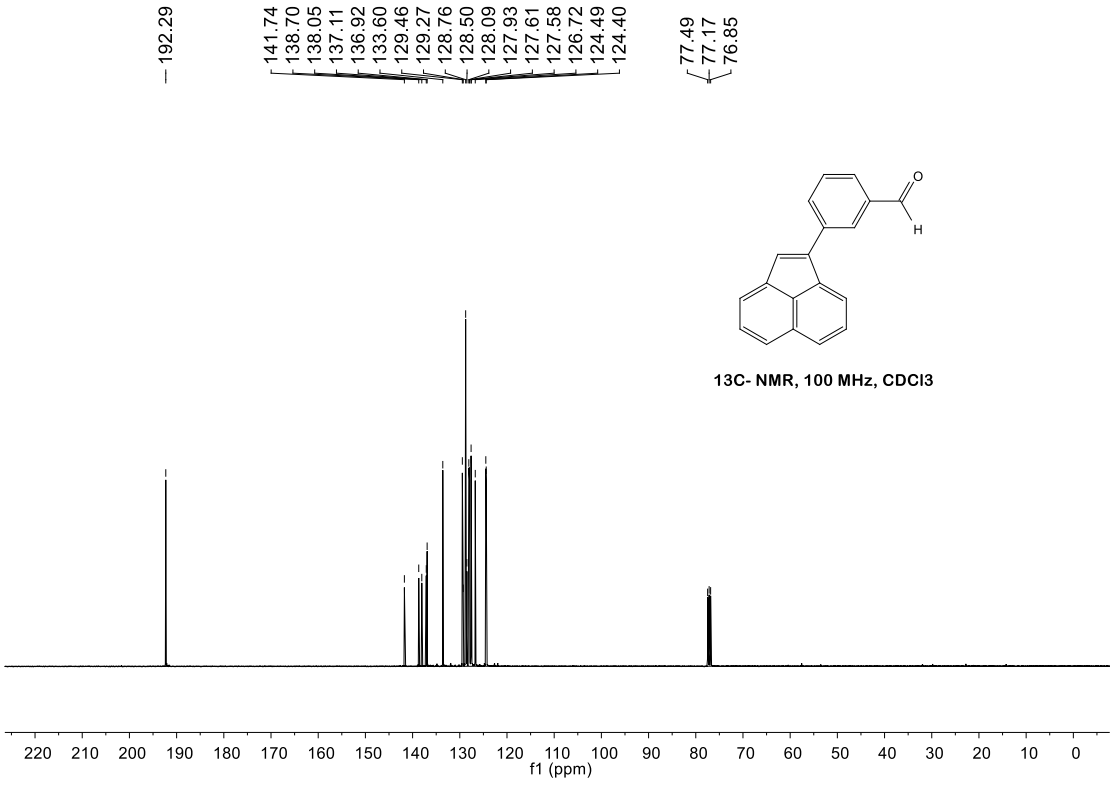
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O
H
13C- NMR, 100 MHz, CDCl3
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200
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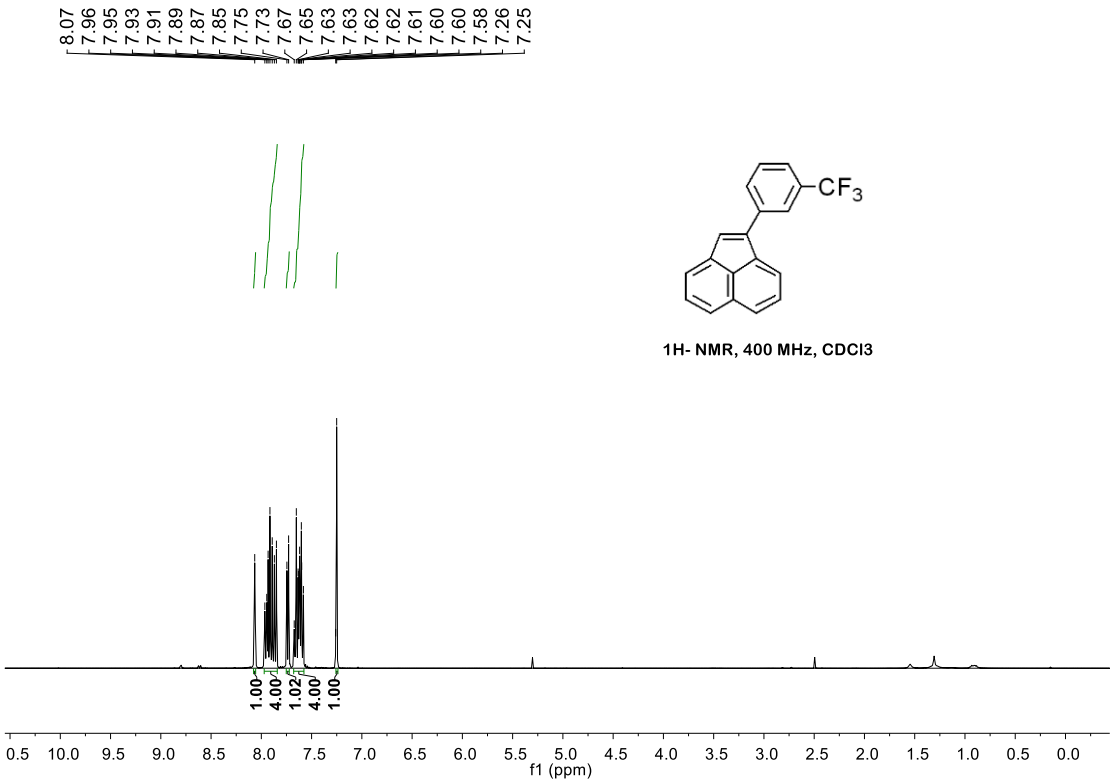
f1 (ppm)

11



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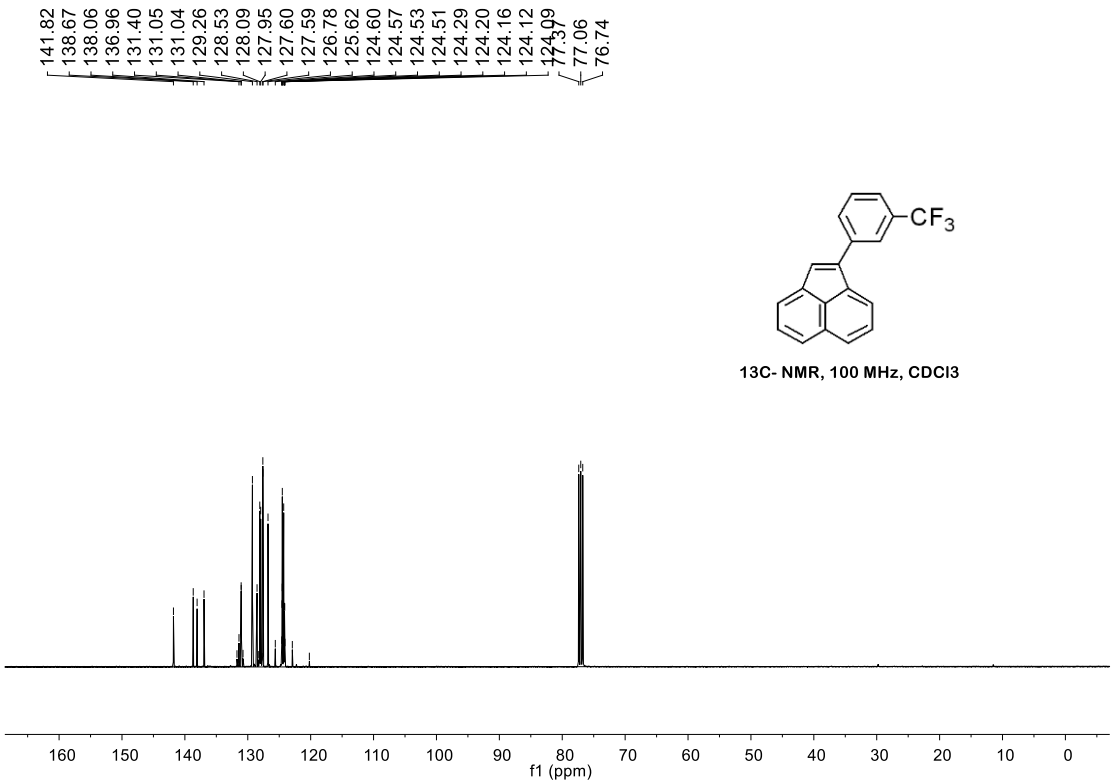

13C- NMR, 100 MHz, CDCl3

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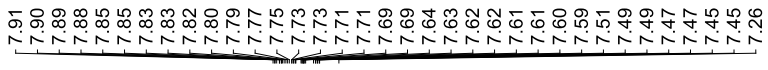

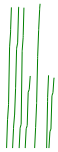

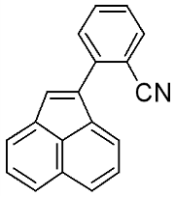

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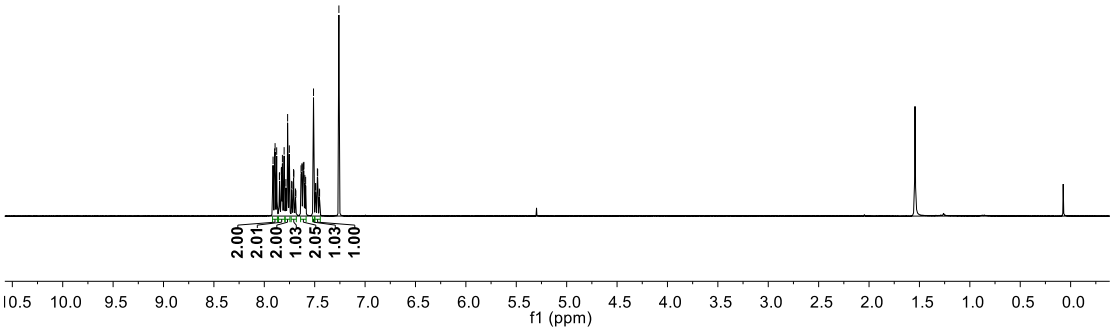

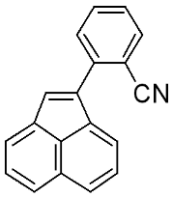

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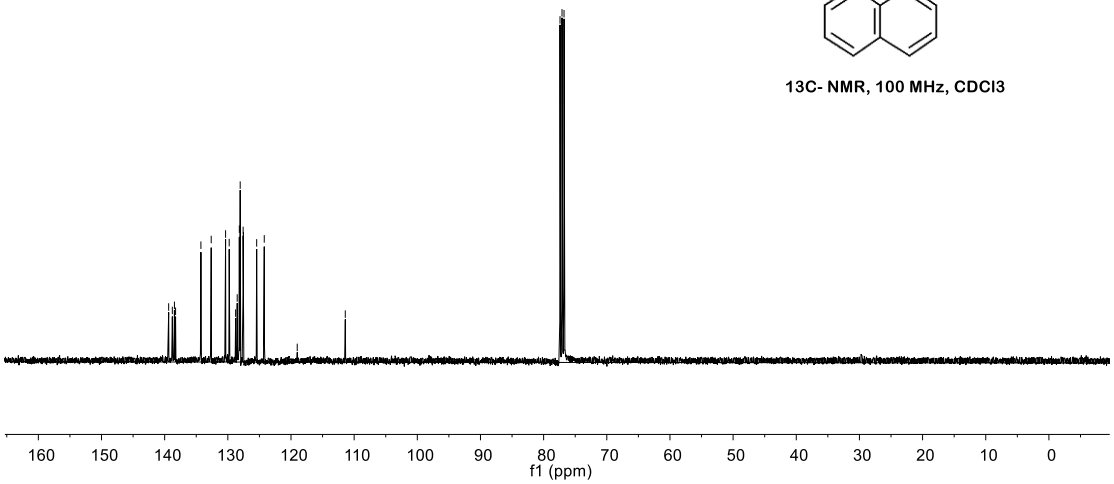

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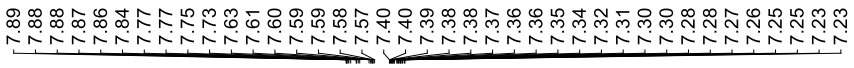

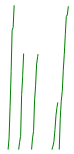

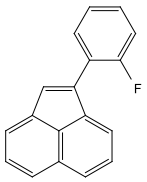

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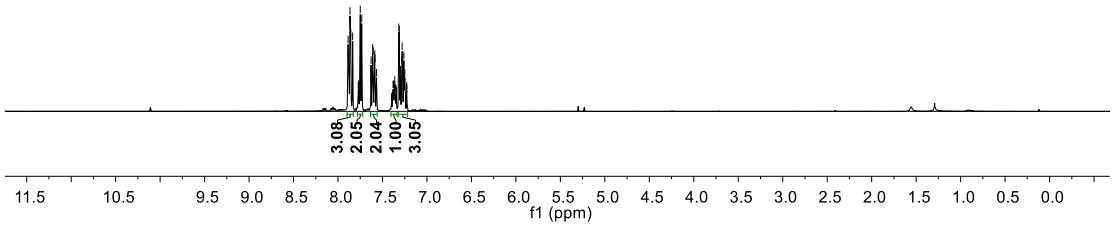

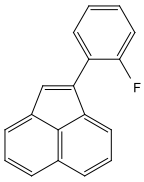

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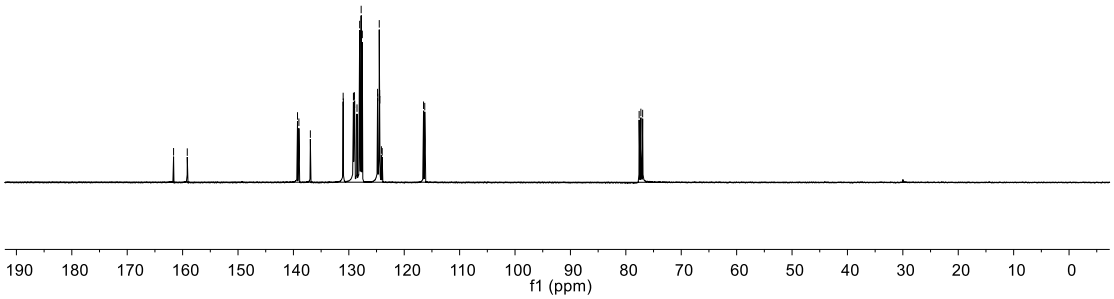

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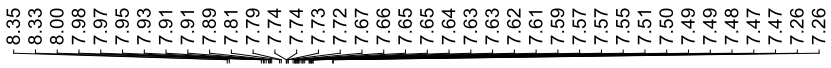

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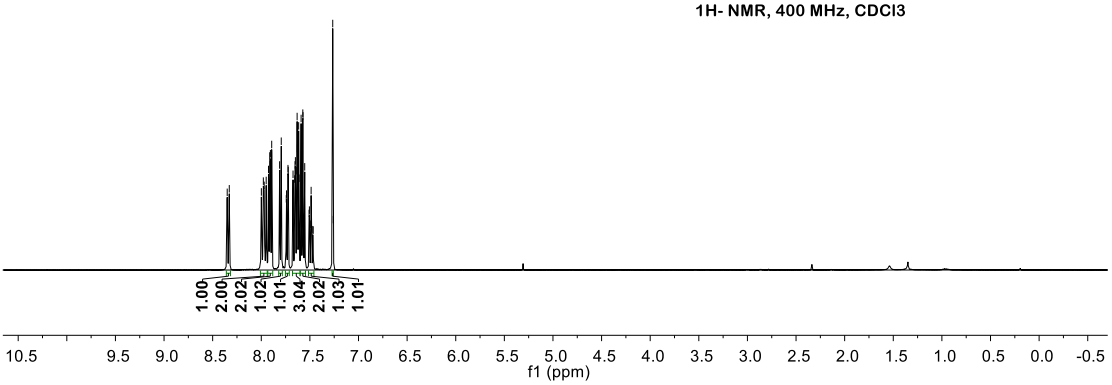
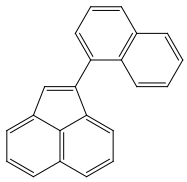
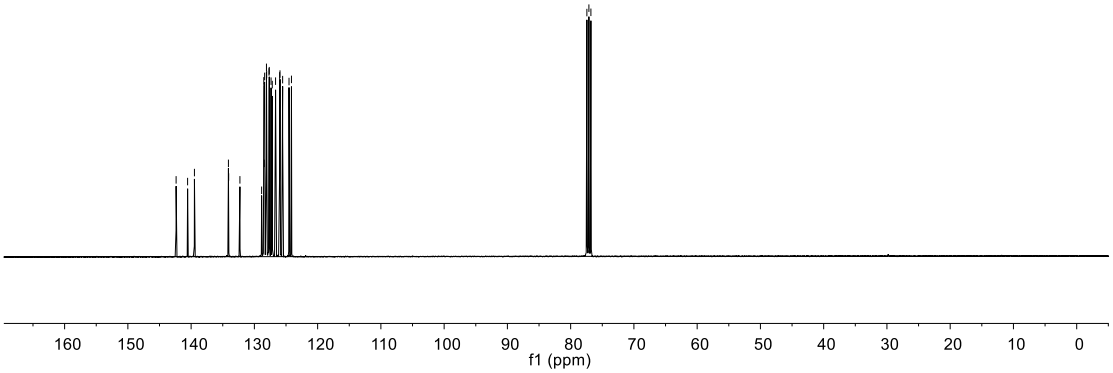




13C- NMR, 100 MHz, CDCl3



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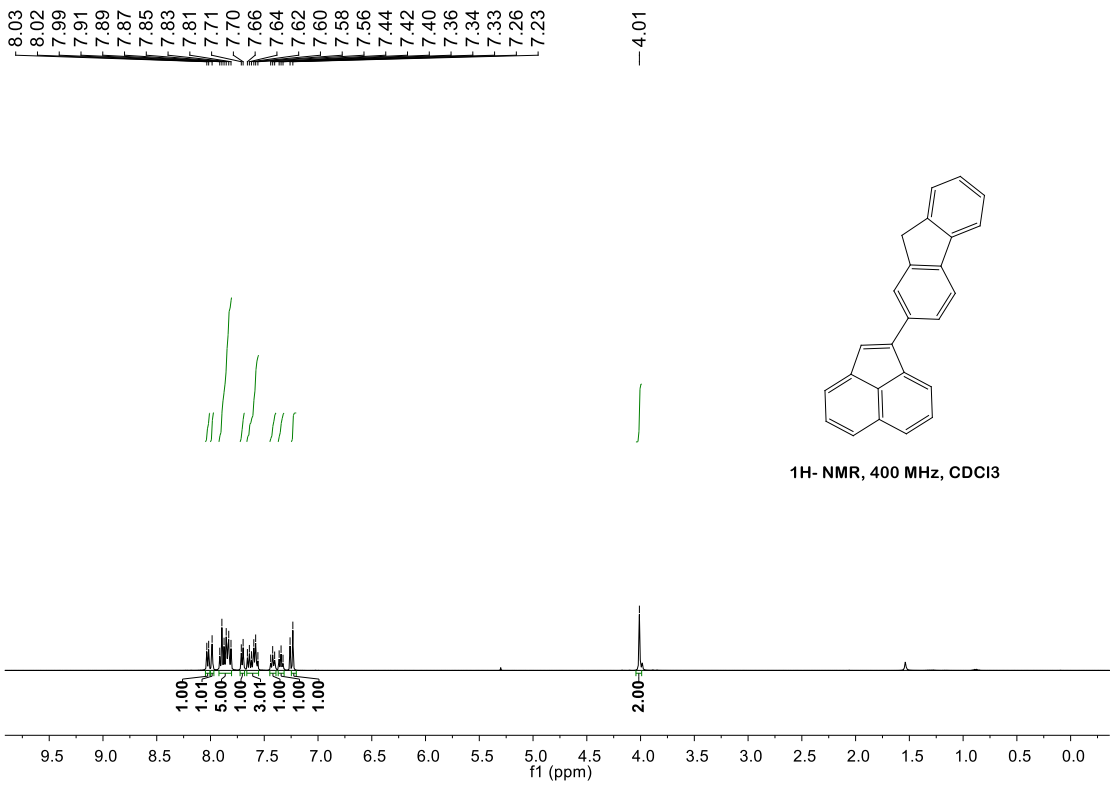
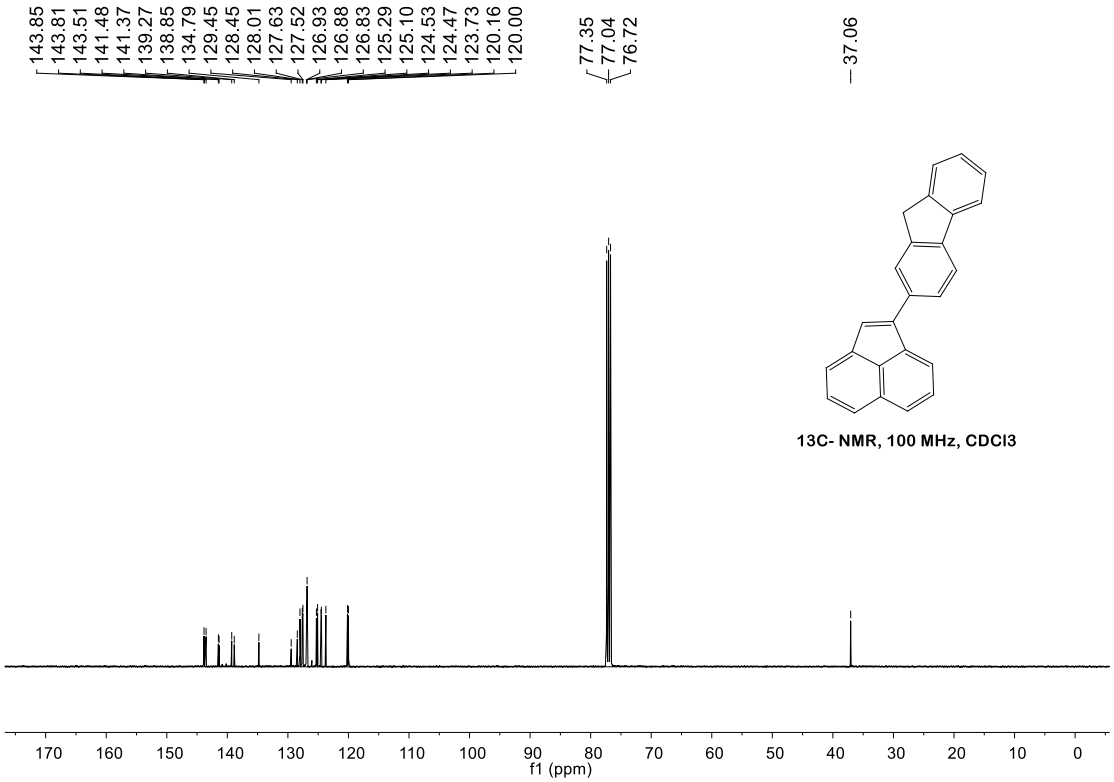
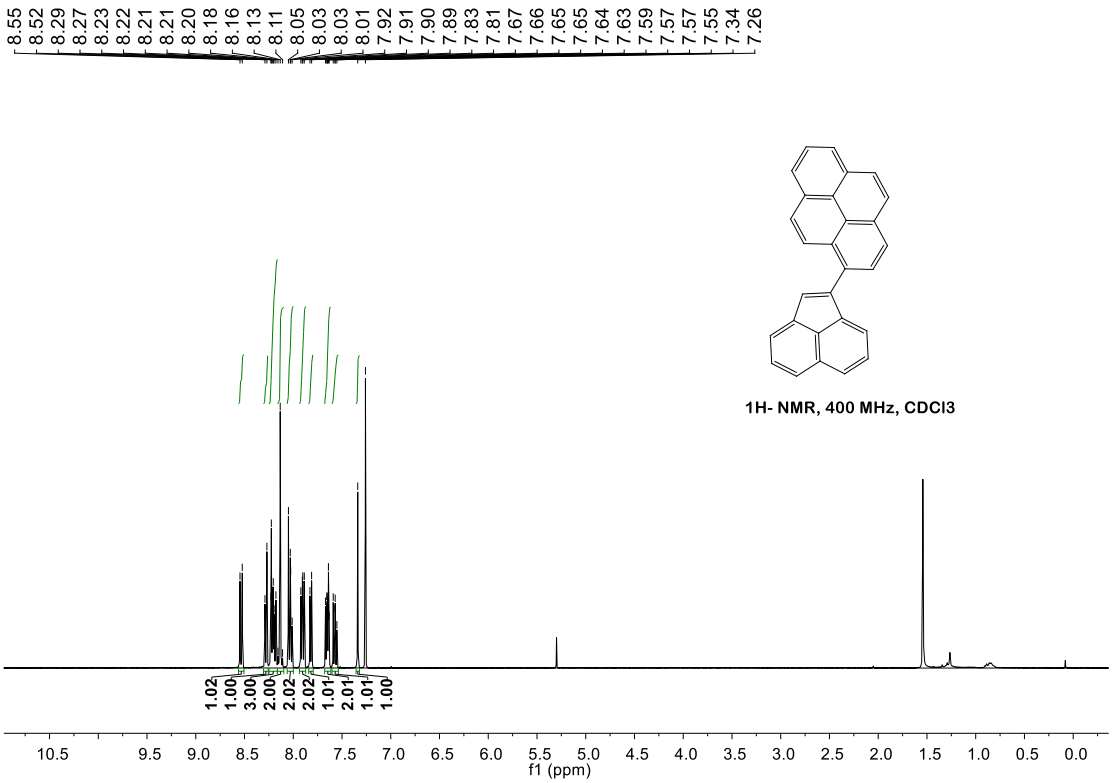


1H- NMR, 400 MHz, CDCl3

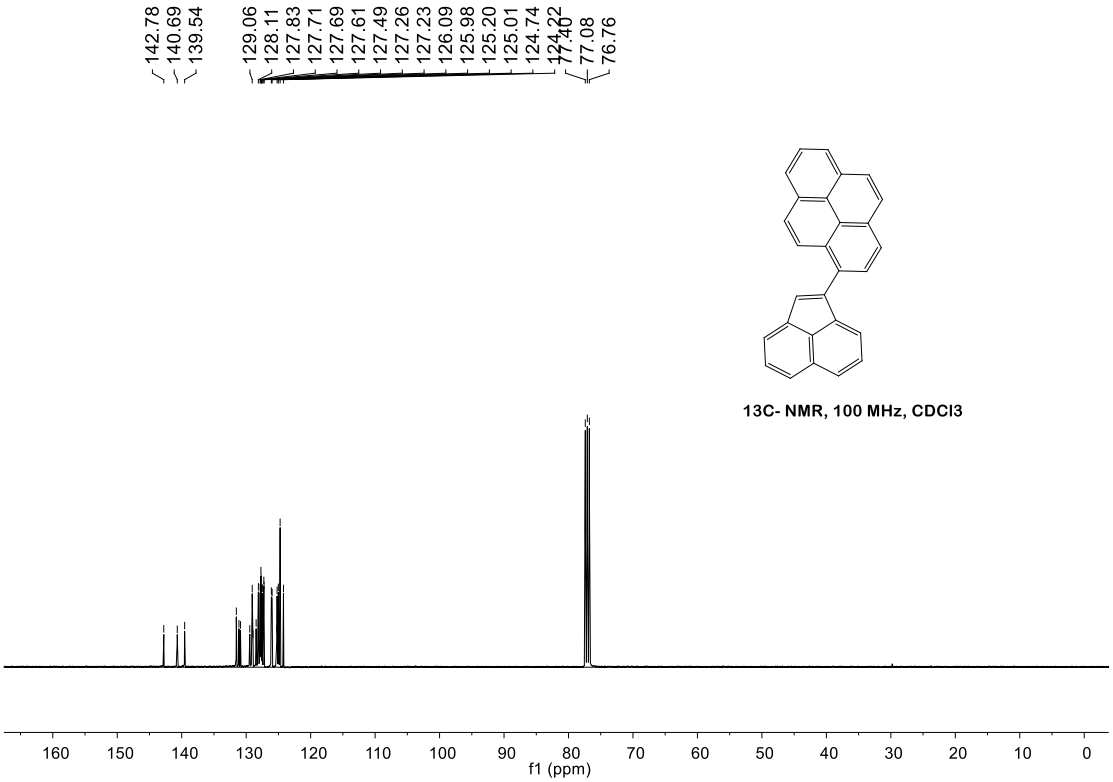


13C- NMR, 100 MHz, CDCl3

16



1H- NMR, 400 MHz, CDCl3



13C- NMR, 100 MHz, CDCl3

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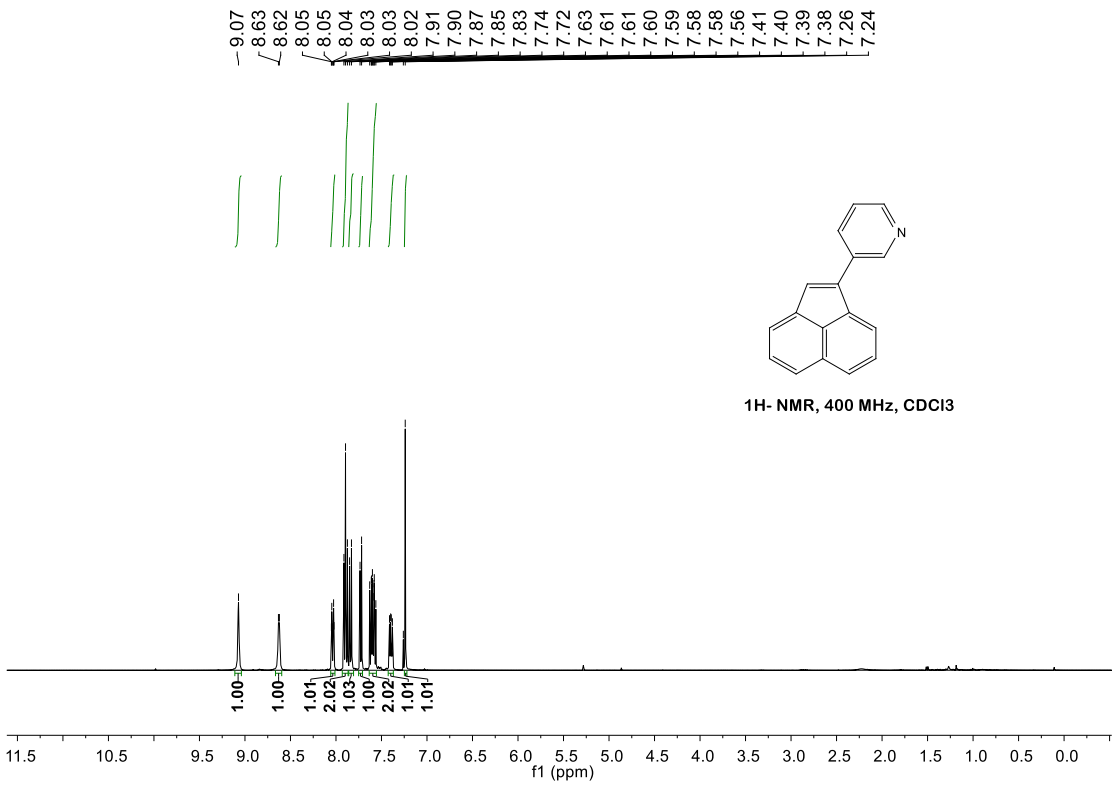
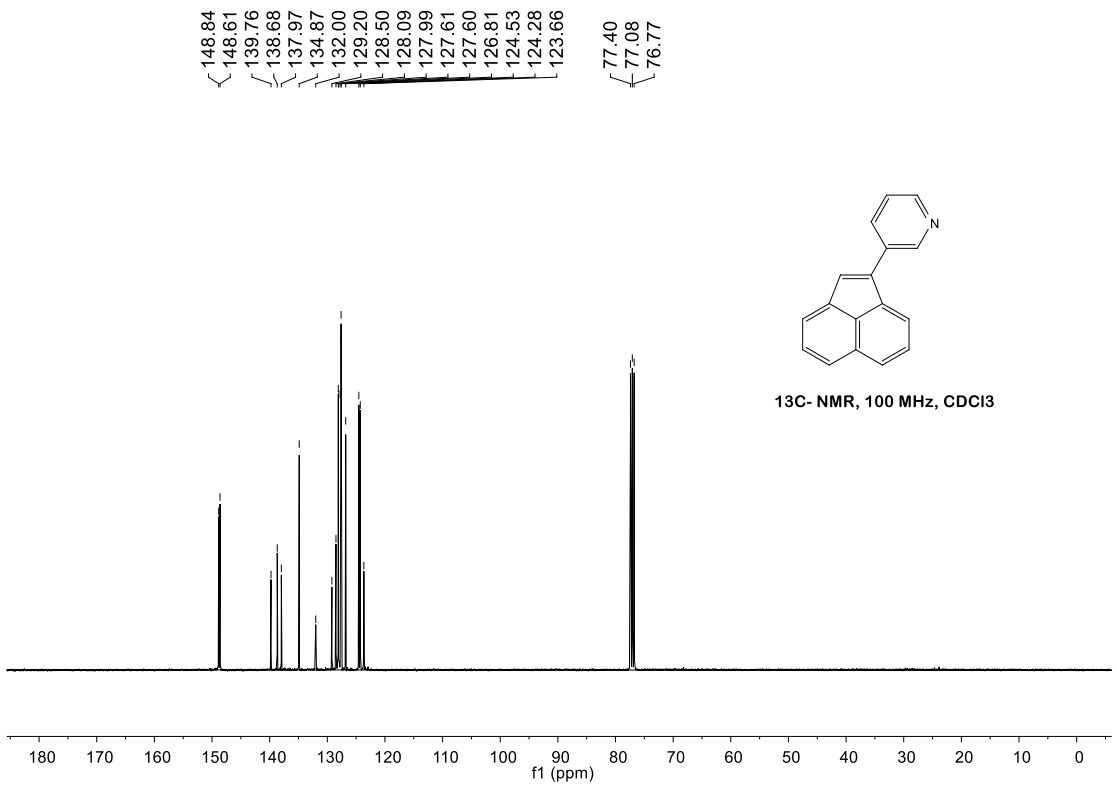


1H- NMR, 400 MHz, CDCl3



13C- NMR, 100 MHz, CDCl3

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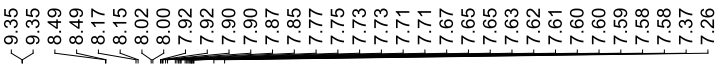
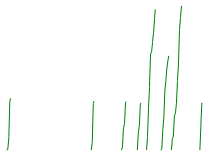
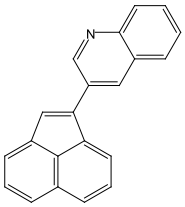
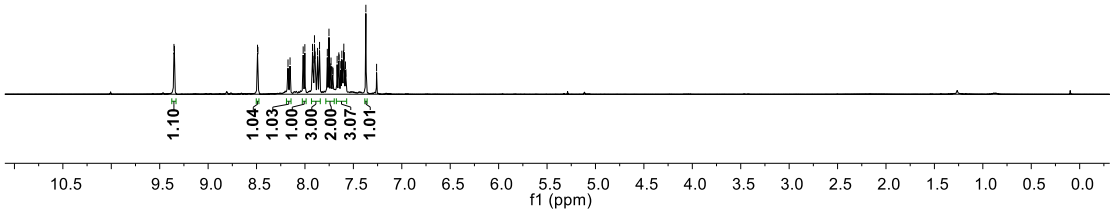
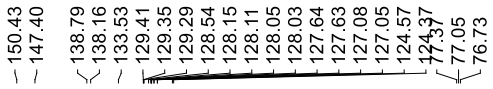


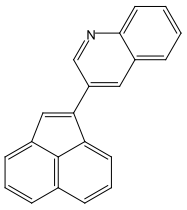




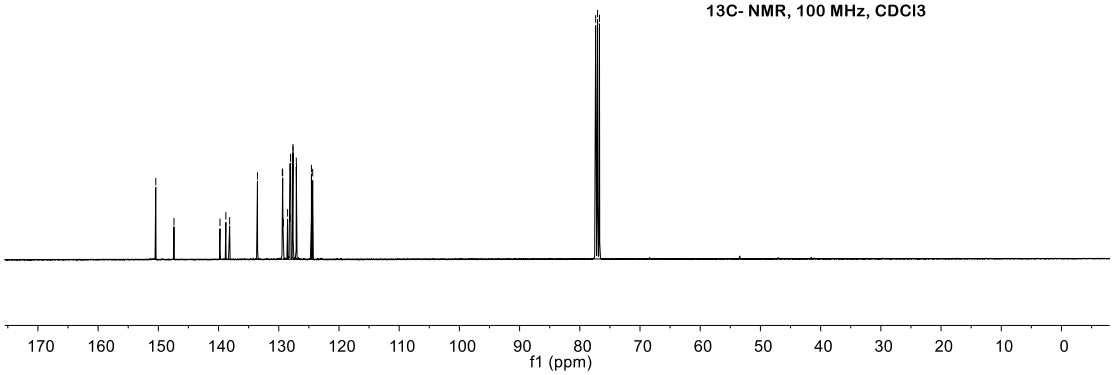
1H- NMR, 400 MHz, CDCl3







13C- NMR, 100 MHz, CDCl3



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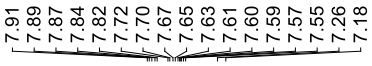
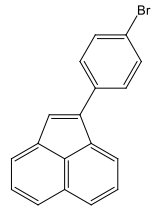
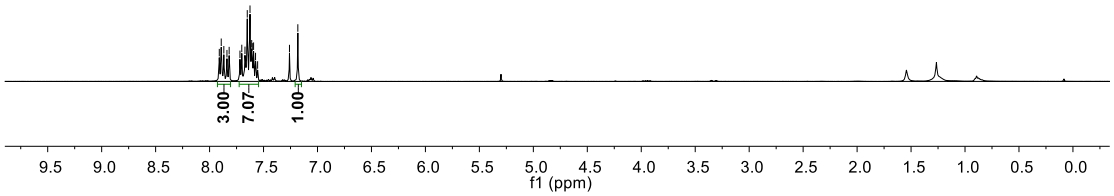
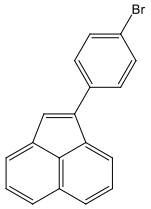


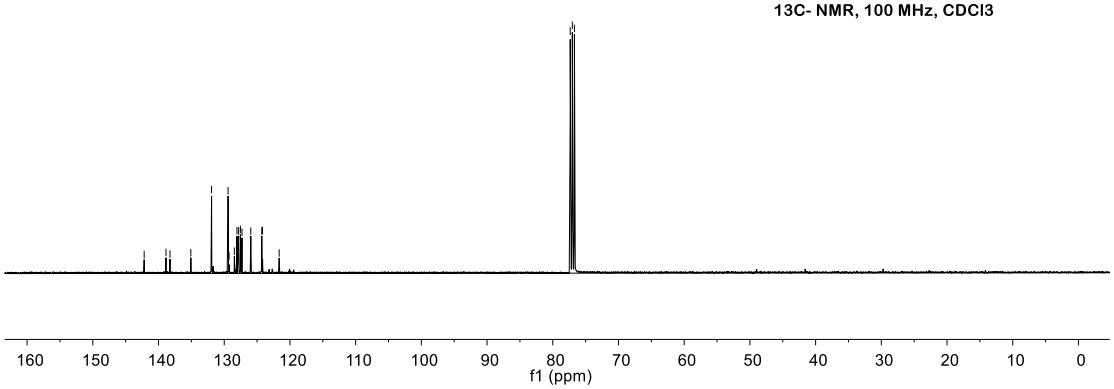


1H- NMR, 400 MHz, CDCl3





13C- NMR, 100 MHz, CDCl3



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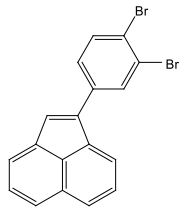
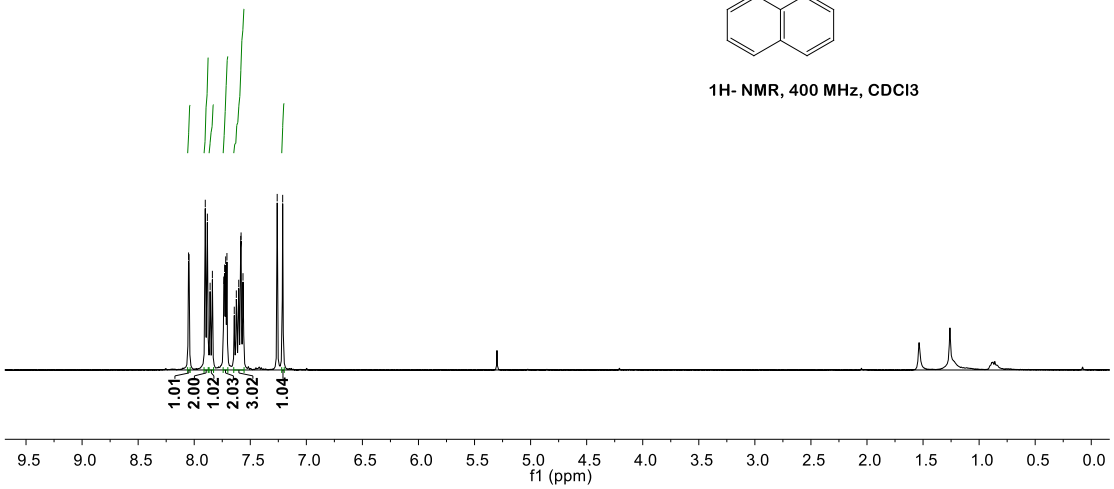
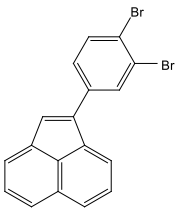


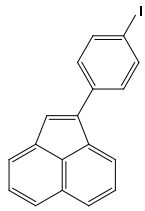
1H- NMR, 400 MHz, CDCl3



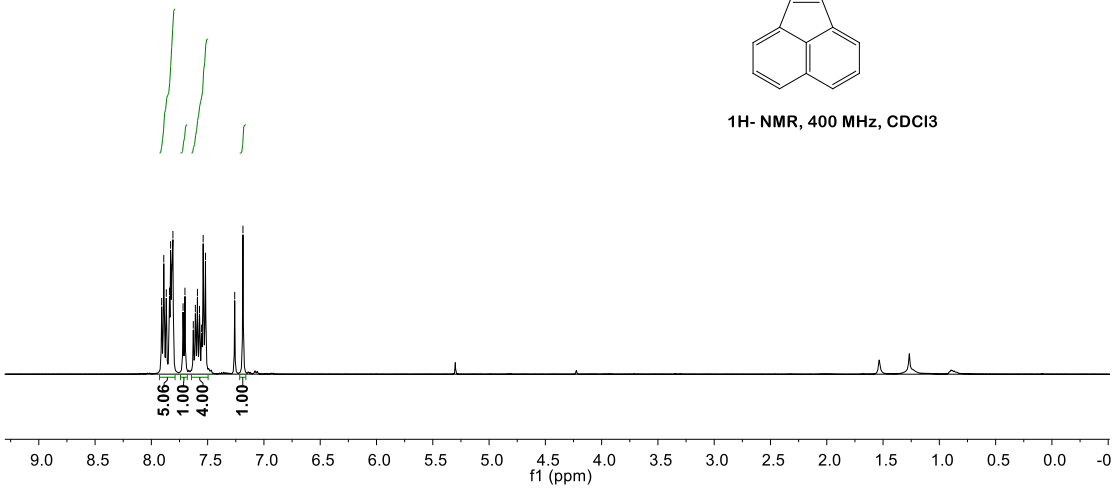


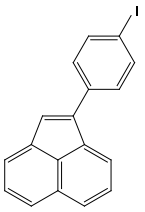
13C- NMR, 100 MHz, CDCl3

21



1H- NMR, 400 MHz, CDCl3





13C- NMR, 100 MHz, CDCl3

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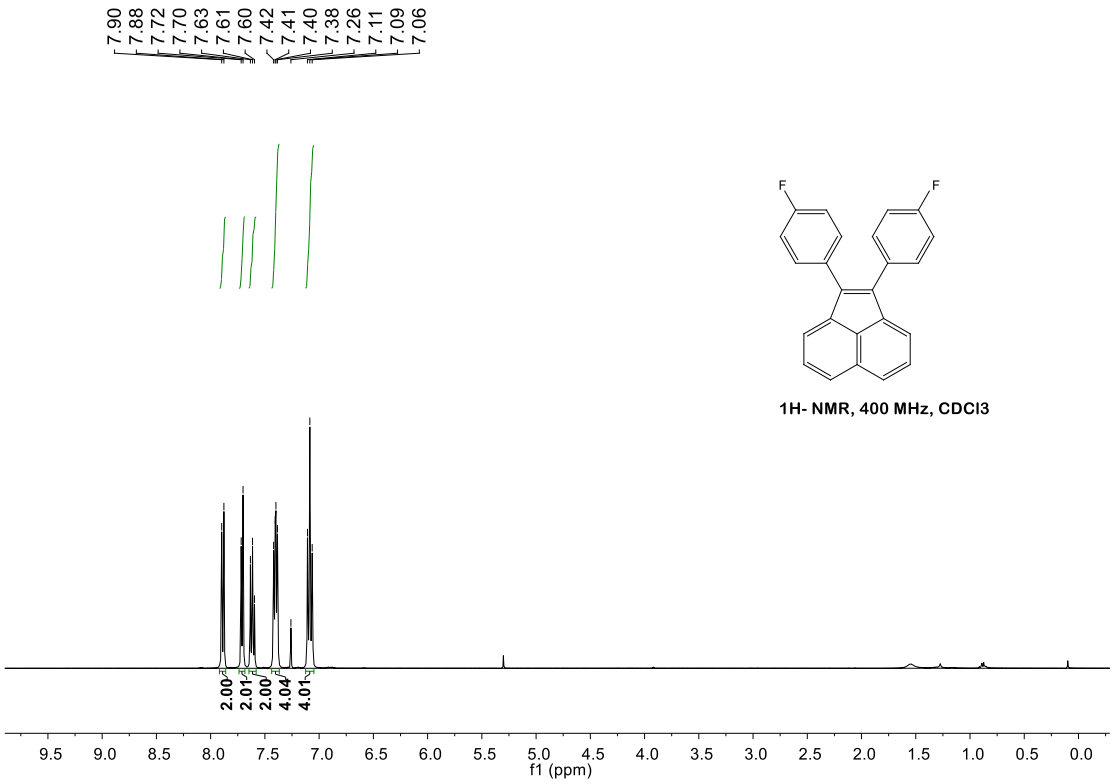
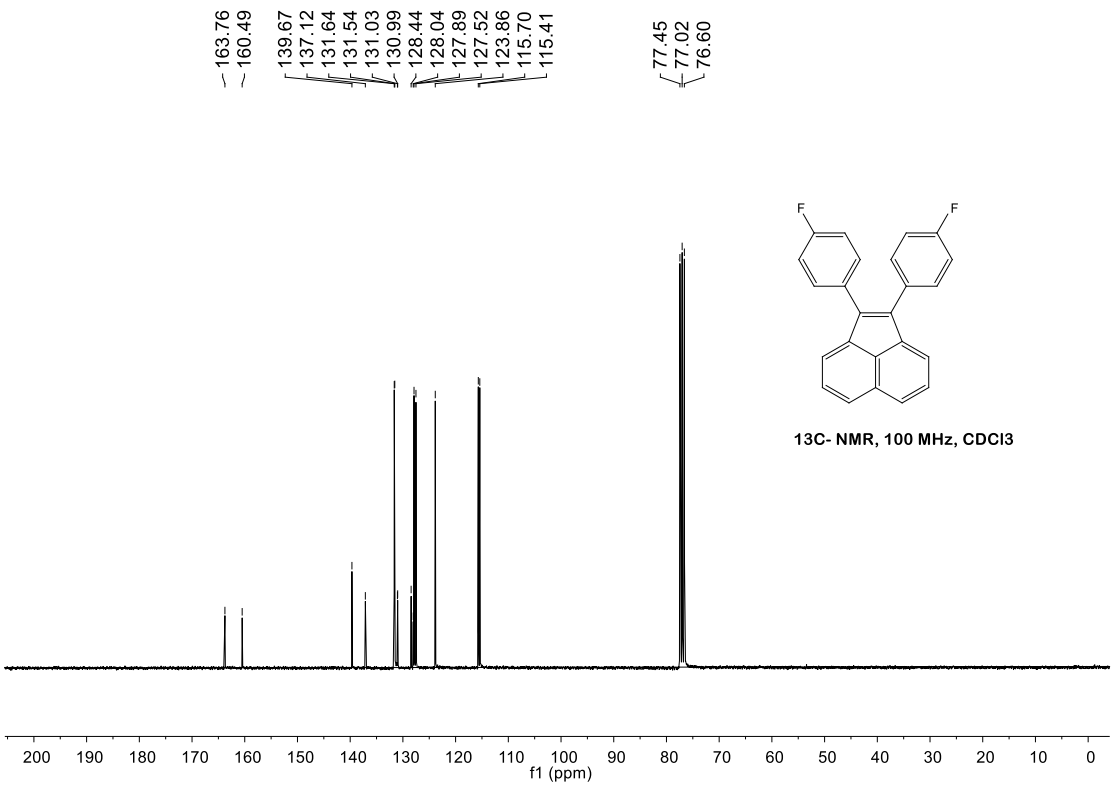


1H- NMR, 400 MHz, CDCl3



13C- NMR, 100 MHz, CDCl3

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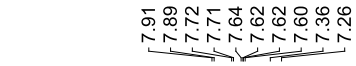
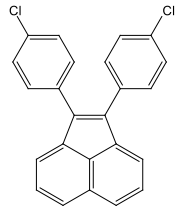
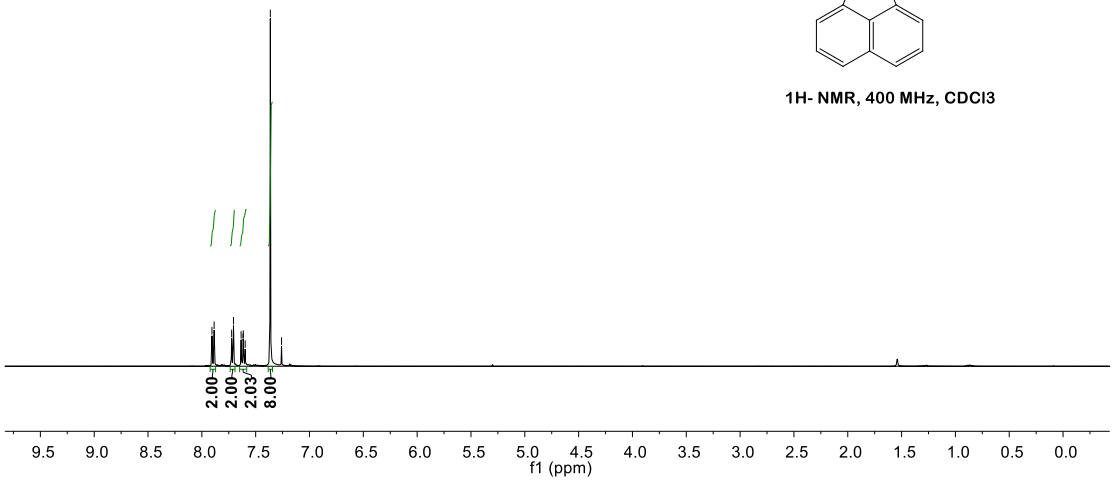
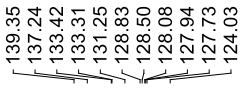
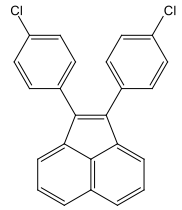




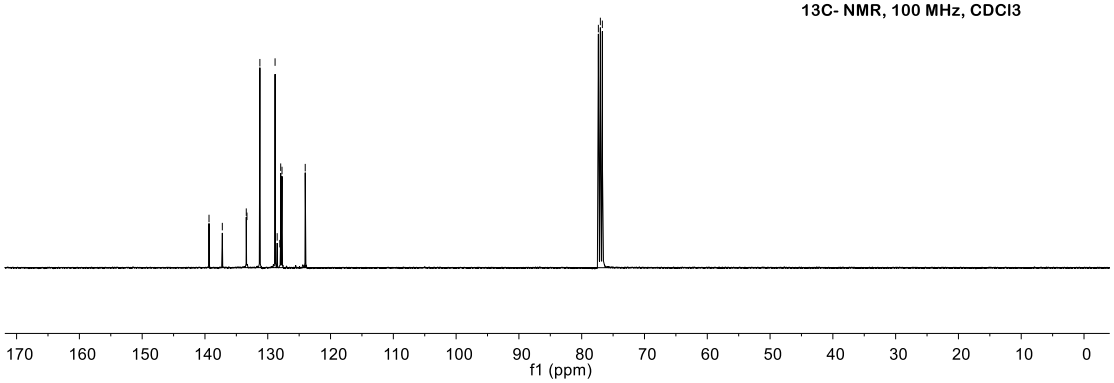
1H- NMR, 400 MHz, CDCl3







13C- NMR, 100 MHz, CDCl3



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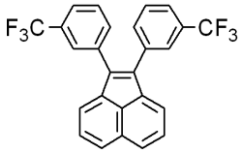
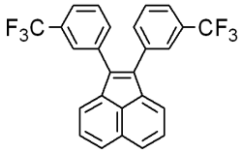
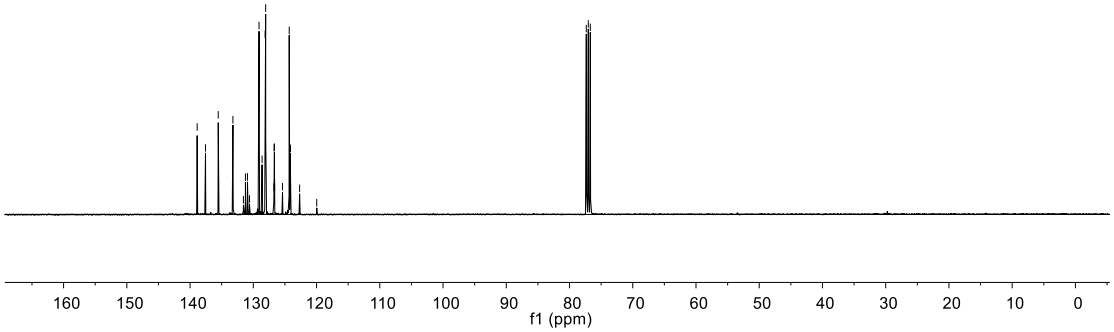


1H- NMR, 400 MHz, CDCl3



13C- NMR, 100 MHz, CDCl3



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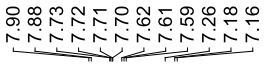
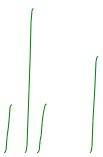
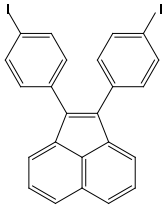
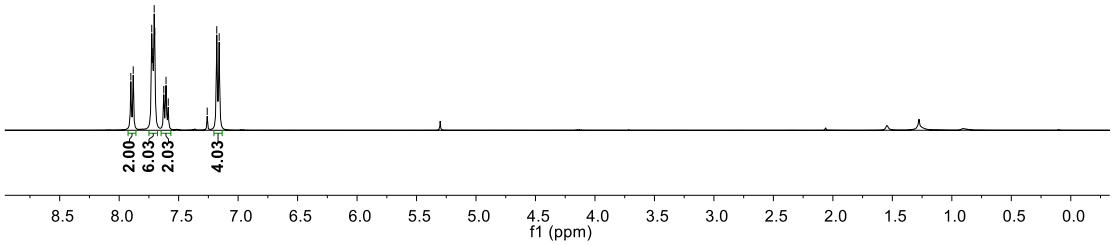
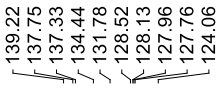


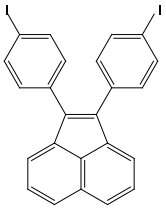




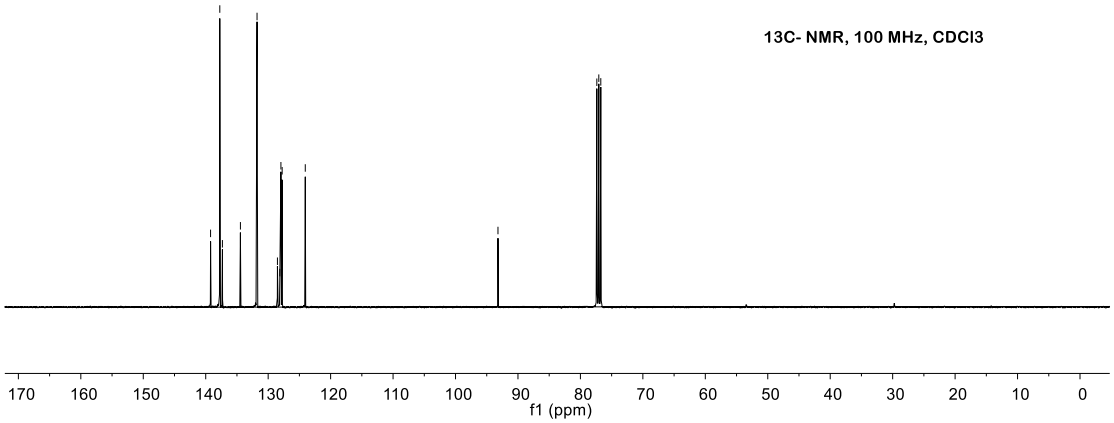
1H- NMR, 400 MHz, CDCl3







13C- NMR, 100 MHz, CDCl3



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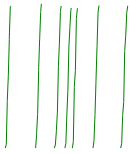
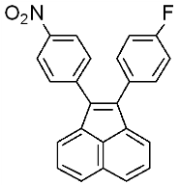
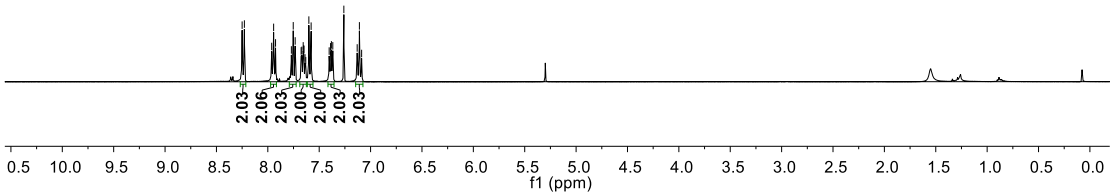
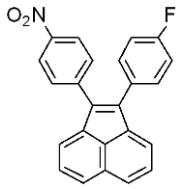


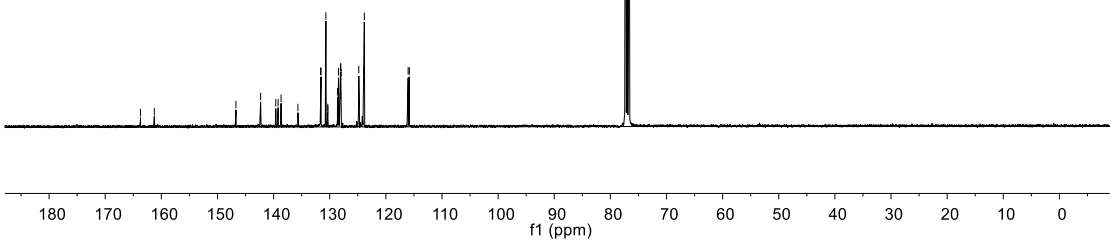


1H- NMR, 400 MHz, CDCl3





13C- NMR, 100 MHz, CDCl3



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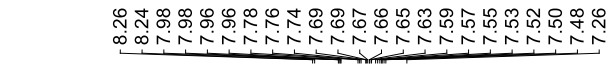
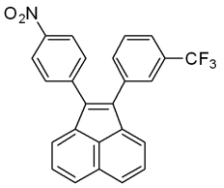
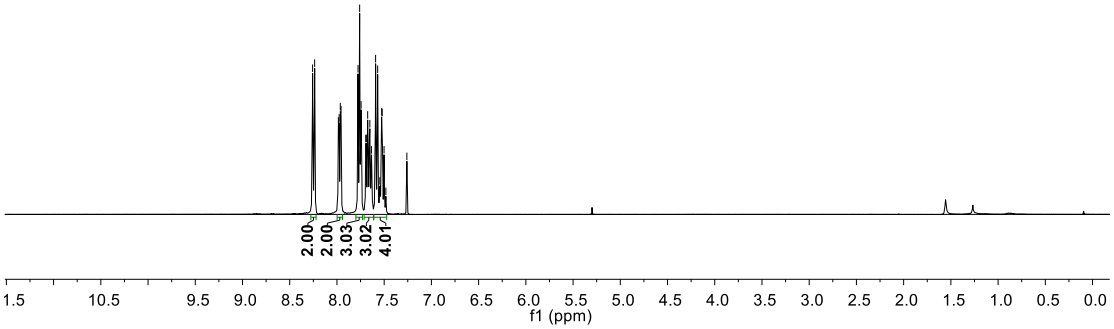
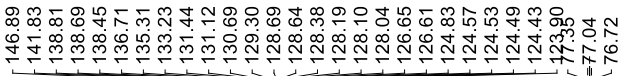
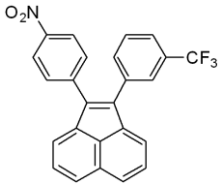




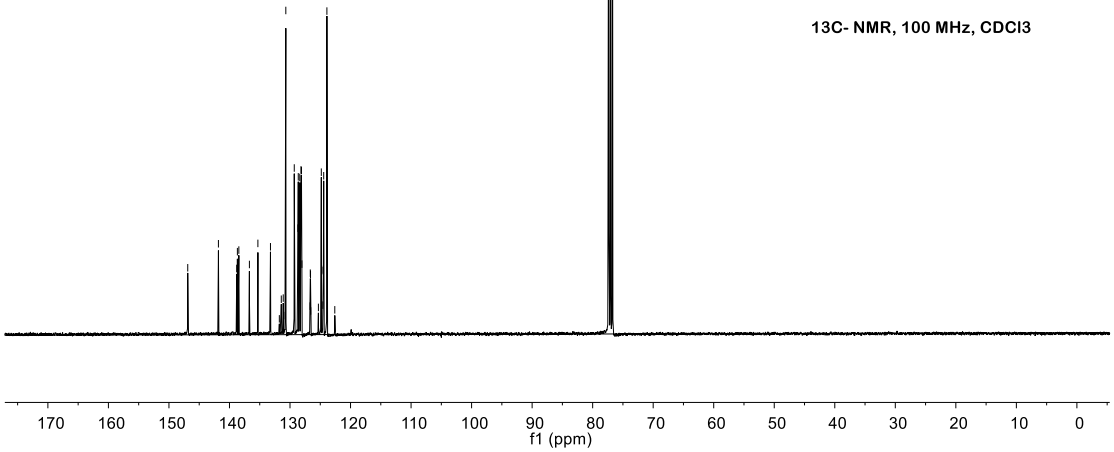
1H- NMR, 400 MHz, CDCl3







13C- NMR, 100 MHz, CDCl3



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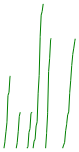
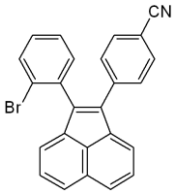
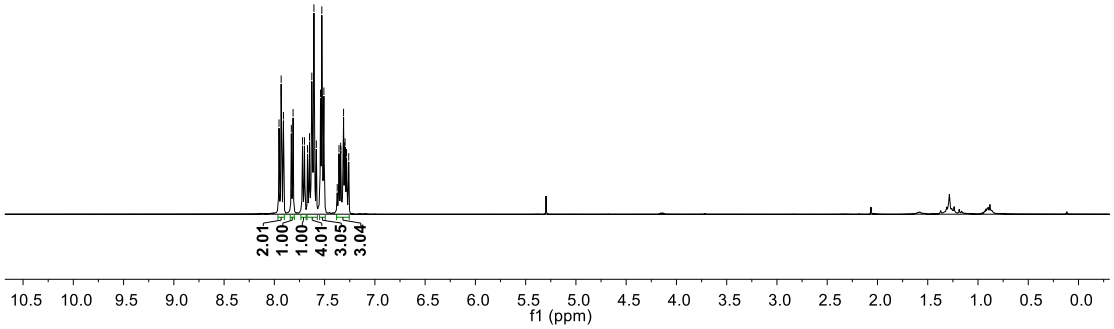
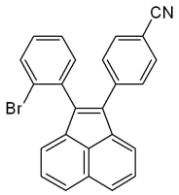


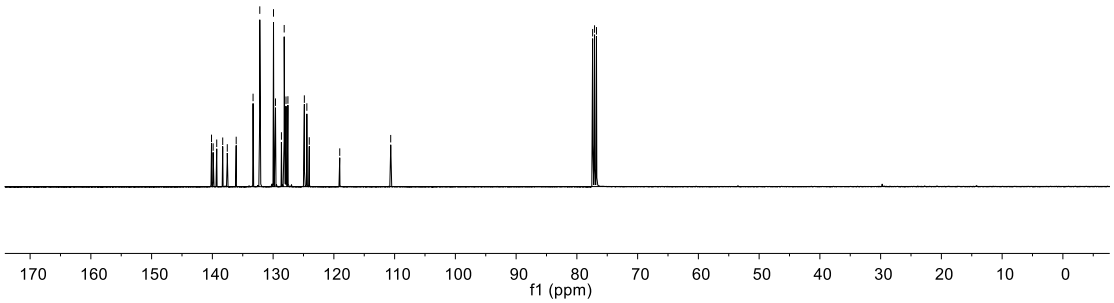


1H- NMR, 400 MHz, CDCl3

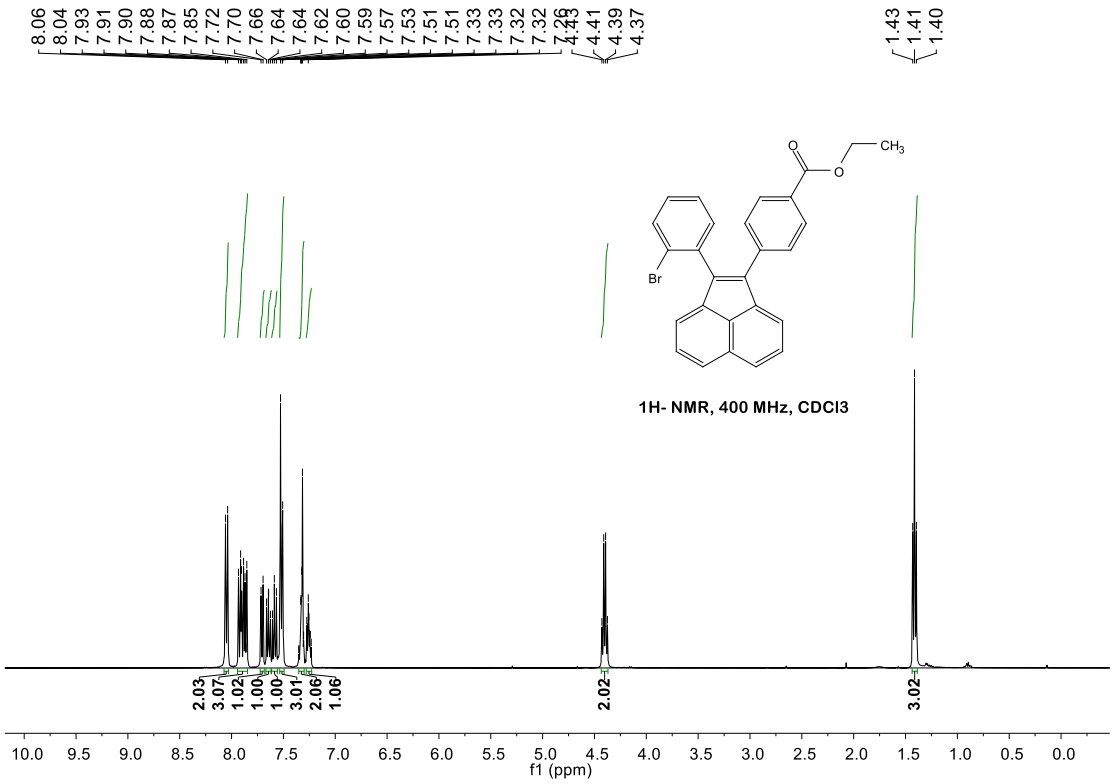




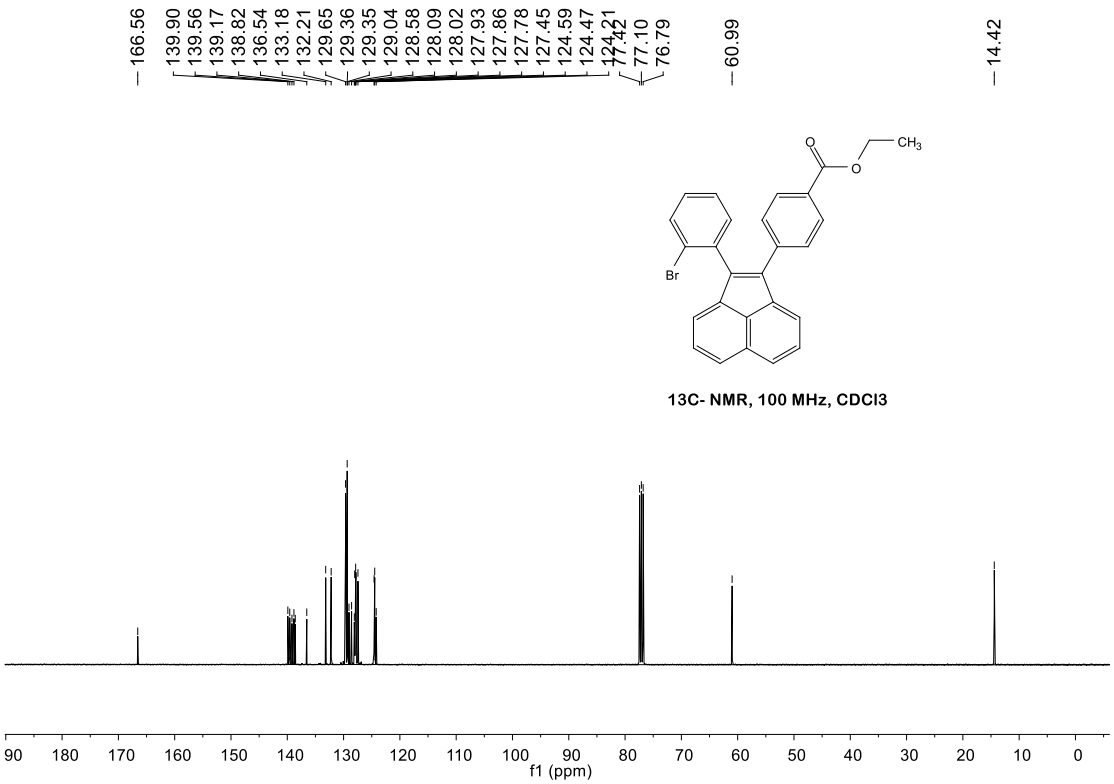
13C- NMR, 100 MHz, CDCl3



29

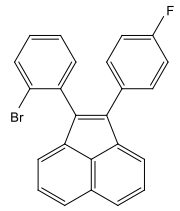


1H- NMR, 400 MHz, CDCl3

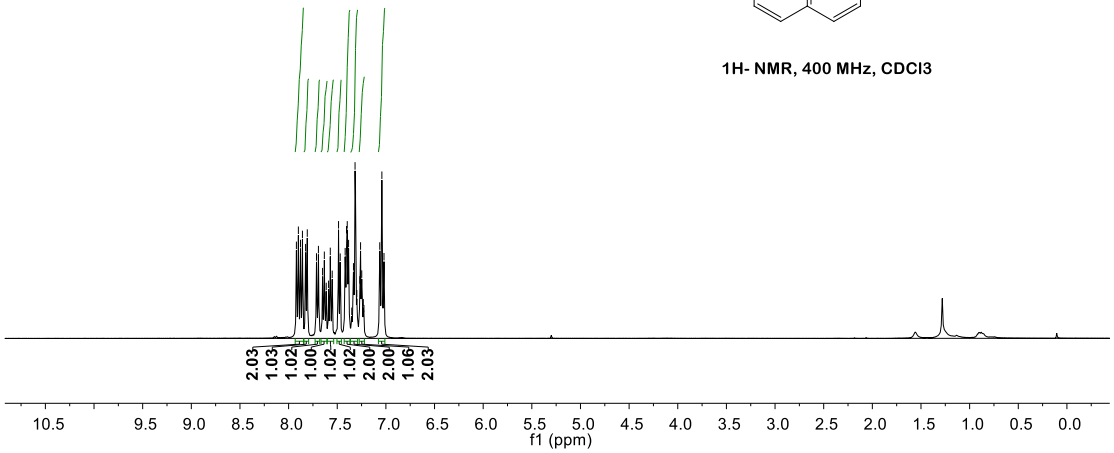


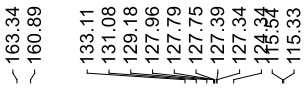
13C- NMR, 100 MHz, CDCl3

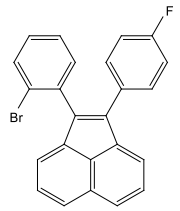
30



1H- NMR, 400 MHz, CDCl3

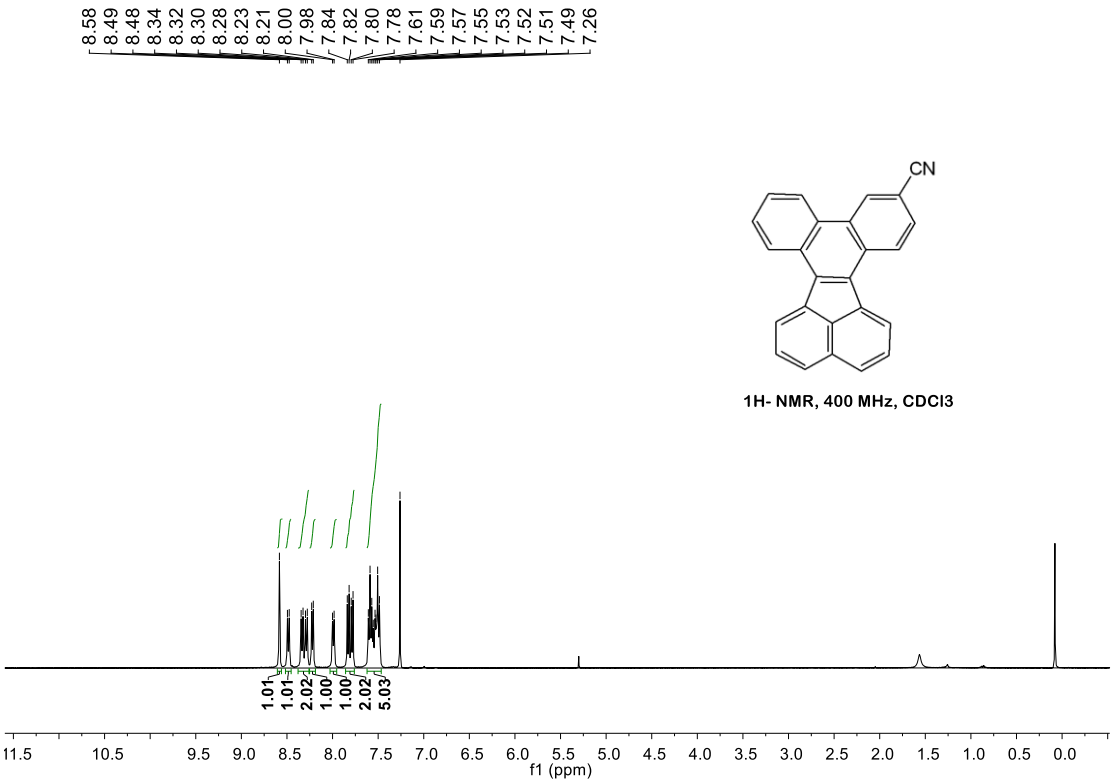




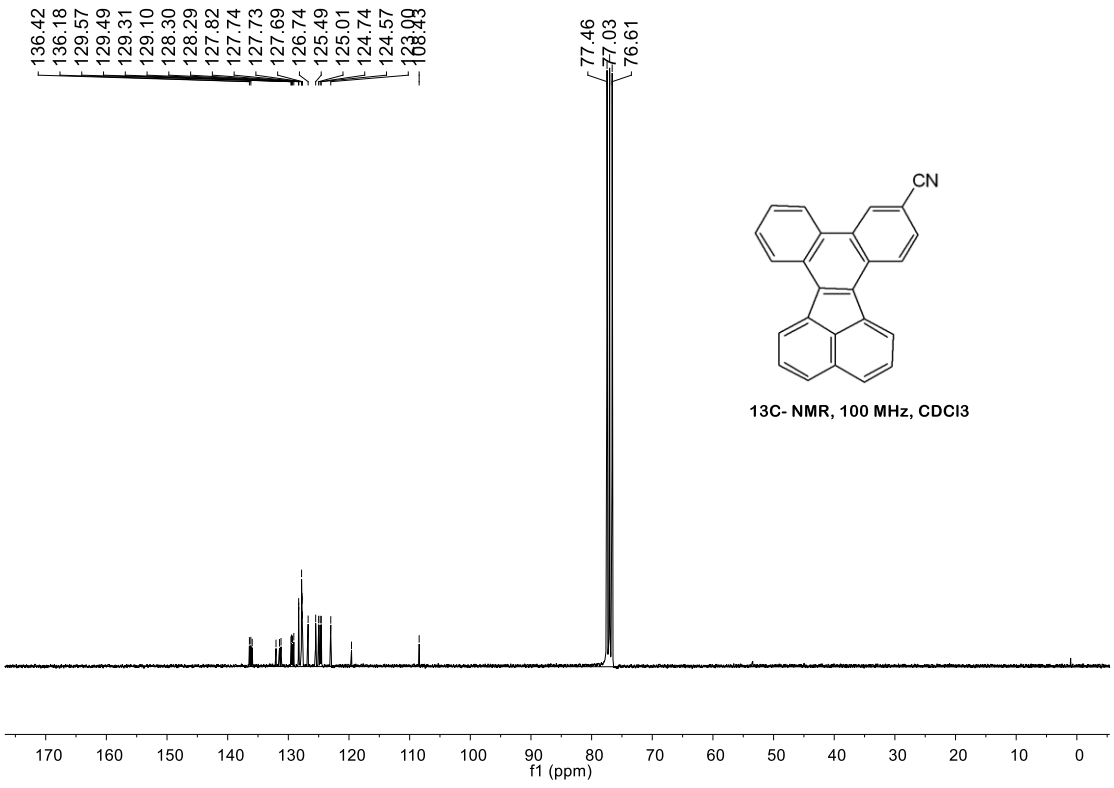


13C- NMR, 100 MHz, CDCl3

31

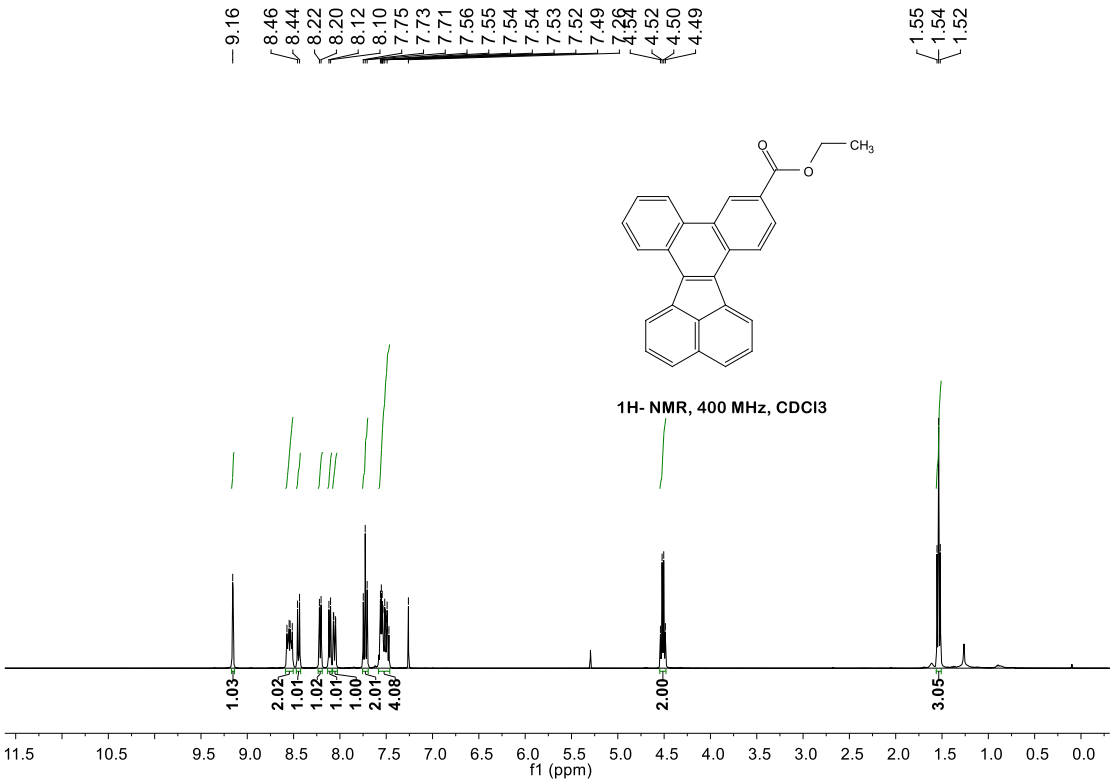


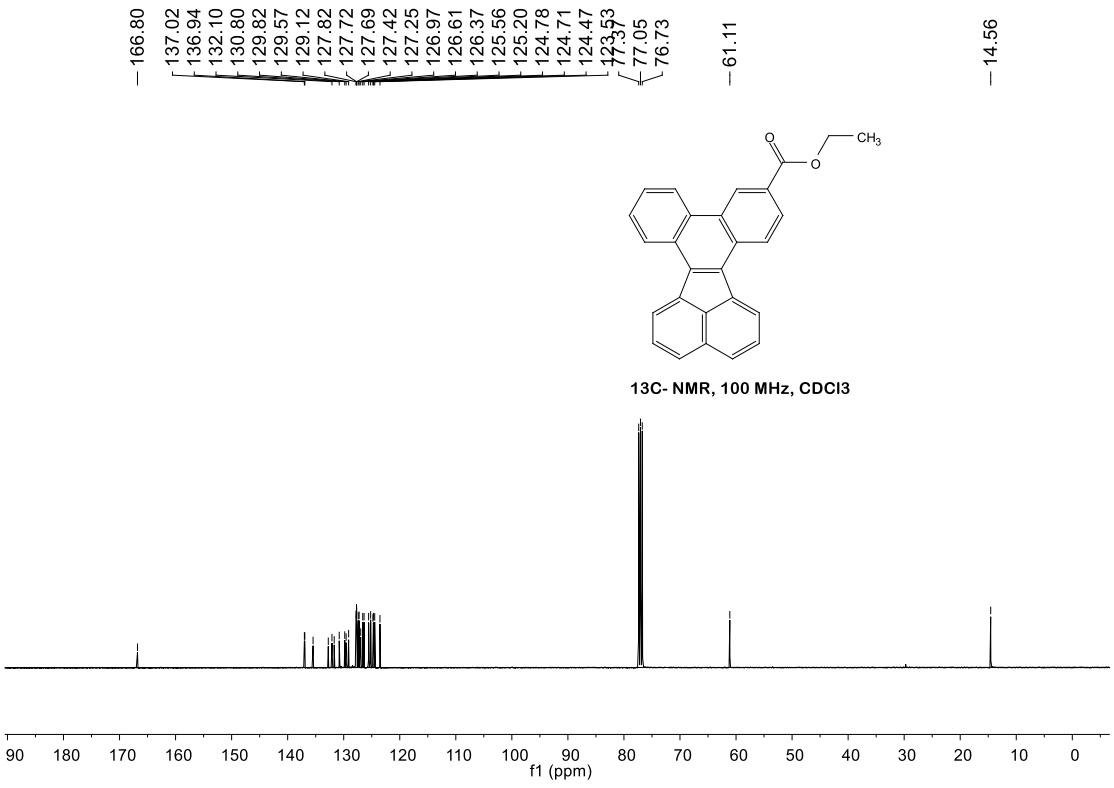
1H- NMR, 400 MHz, CDCl3



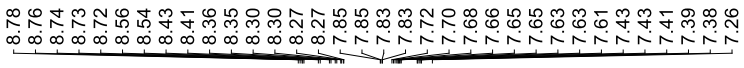
13C- NMR, 100 MHz, CDCl3

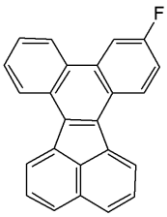
32


9.16
8.46
8.44
8.22
8.20
8.12
8.10
7.75
7.73
7.71
7.56
7.55
7.54
7.54
7.53
7.52
7.49
7.26
4.54
4.52
4.50
4.49
1.55
1.54
1.52
O
O
CH3
1H- NMR, 400 MHz, CDCl3
1.03
2.02
1.01
1.02
1.01
1.00
2.01
4.08
2.00
3.05
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

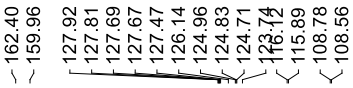

166.80
137.02
136.94
132.10
130.80
129.82
129.57
129.12
127.82
127.72
127.69
127.42
127.25
126.97
126.61
126.37
125.56
125.20
124.78
124.71
124.47
123.53
77.37
77.05
76.73
61.11
14.56
O
O
CH3
13C- NMR, 100 MHz, CDCl3
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

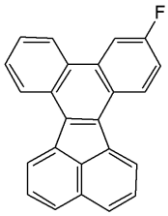
33



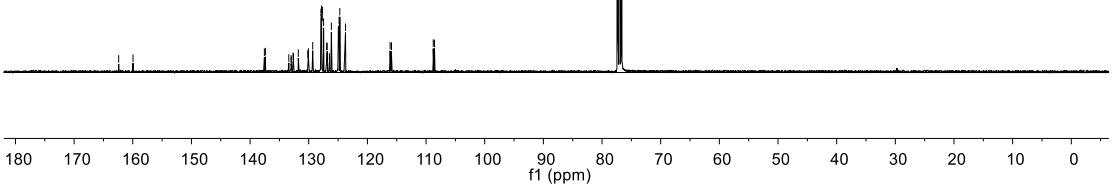


1H- NMR, 400 MHz, CDCl3





13C- NMR, 100 MHz, CDCl3



X-ray analysis of **23** (CDC 1553883) and **33** (CDC 1553881)

Crystal structure report of compound 23:

CDC 1553883

X-ray crystallographic study

(C_{24} H_{14} Cl_2); *M* = 373.25. APEXII, Bruker-AXS diffractometer, Mo-Kα radiation (λ = 0.71073 Å), *T* = 150 K; monoclinic *C* 2/*c* (I.T.#15), a = 16.8237(12), b = 11.3330(7), c = 10.2994(8) Å, β = 114.382(3) °, *V* = 1788.6(2) $Å^3$. *Z* = 4, *d* = 1.386 $g.cm^{-3}$, μ = 0.367 mm^{-1}. The structure was solved by dual-space algorithm using the *SHELXT* program [1], and then refined with full-matrix least-square methods based on F^2 (*SHELXL*) [2]. All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions. A final refinement on F^2 with 2048 unique intensities and 119 parameters converged at ω*R*(F^2) = 0.0828 (*R*(*F*) = 0.0331) for 1756 observed reflections with *I* > 2σ(*I*).

[1] G. M. Sheldrick, Acta Cryst. A71 (2015) 3-8
[2] Sheldrick G.M., Acta Cryst. C71 (2015) 3-8

Structural data

Empirical formula	C_{24} H_{14} Cl_2
Formula weight	373.25
Temperature	150 *K*
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, *C* 2/*c*
Unit cell dimensions	a = 16.8237(12) Å, α = 90 ° b = 11.3330(7) Å, β = 114.382(3) ° c = 10.2994(8) Å, γ = 90 °
Volume	1788.6(2) $Å^3$
Z, Calculated density	4, 1.386 ($g.cm^{-3}$)
Absorption coefficient	0.367 mm^{-1}
F(000)	768
Crystal size	0.500 x 0.370 x 0.220 mm
Crystal color	orange
Theta range for data collection	3.479 to 27.476 °
h_min, h_max	-21, 21
k_min, k_max	-14, 14
l_min, l_max	-9, 13
Reflections collected / unique	4736 / 2048 [R(int)[a] = 0.0210]
Reflections [I>2σ]	1756
Completeness to theta_max	0.995
Absorption correction type	multi-scan
Max. and min. transmission	0.916 , 0.838
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2048 / 0 / 119
[b]S (Goodness-of-fit)	1.039
Final *R* indices [I>2σ]	*R*1[c] = 0.0331, *wR*2[d] = 0.0828
R indices (all data)	*R*1[c] = 0.0404, *wR*2[d] = 0.0889

Largest diff. peak and hole 0.253 and -0.244 $e^{-}.Å^{-3}$

[a] $R_{int} = \sum |F_o^2 - < F_o^2 >| / \sum [F_o^2]$
[b] $S = \{\sum [w(F_o^2 - F_c^2)^2] / (n - p)\}^{1/2}$
[c] $R1 = \sum | |F_o| - |F_c| | / \sum |F_o|$
[d] $wR2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]\}^{1/2}$
$w = 1 / [\sigma(F_o^2) + aP^2 + bP]$ where $P = [2F_c^2 + MAX(F_o^2, 0)] /3$

Atomic coordinates, site occupancy (%) and equivalent isotropic displacement parameters (Å²). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	occ.	U(eq)
C1	0.32828(8)	0.16558(11)	0.44358(15)	1	0.0203(3)
C2	0.34445(9)	0.26123(12)	0.37459(15)	1	0.0215(3)
H2	0.326399	0.260500	0.274177	1	0.026
C3	0.38762(9)	0.35836(12)	0.45508(14)	1	0.0201(3)
H3	0.398624	0.424790	0.408671	1	0.024
C4	0.41517(8)	0.36029(11)	0.60289(14)	1	0.0181(3)
C5	0.39573(8)	0.26281(12)	0.66821(14)	1	0.0201(3)
H5	0.412517	0.263489	0.768315	1	0.024
C6	0.35247(9)	0.16547(12)	0.58942(15)	1	0.0211(3)
H6	0.339618	0.099715	0.634671	1	0.025
C7	0.46354(9)	0.46293(11)	0.68616(14)	1	0.0182(3)
C8	0.43886(9)	0.58702(12)	0.64277(14)	1	0.0194(3)
C9	0.37326(9)	0.64179(13)	0.53047(15)	1	0.0240(3)
H9	0.329807	0.596977	0.457607	1	0.029
C10	0.500000	0.65790(17)	0.750000	1	0.0195(4)
C11	0.500000	0.78079(17)	0.750000	1	0.0229(4)
C12	0.56703(10)	0.83550(13)	0.86939(17)	1	0.0274(3)
H12	0.569725	0.919084	0.876953	1	0.033
C13	0.62801(10)	0.76743(13)	0.97401(16)	1	0.0281(3)
H13	0.672676	0.805752	1.052227	1	0.034
Cl1	0.27588(2)	0.04204(3)	0.34336(4)	1	0.02943(13)

Anisotropic displacement parameters (Å²)

The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \ldots + 2 h k a^* b^* U_{12}]$.

Atom	U11	U22	U33	U23	U13	U12
C1	0.0182(6)	0.0167(6)	0.0244(7)	-0.0038(5)	0.0070(5)	0.0003(5)
C2	0.0218(7)	0.0232(7)	0.0189(6)	-0.0003(5)	0.0078(5)	0.0014(5)
C3	0.0217(7)	0.0200(6)	0.0198(6)	0.0021(5)	0.0097(5)	-0.0002(5)
C4	0.0175(6)	0.0170(6)	0.0203(6)	-0.0003(5)	0.0083(5)	0.0013(5)
C5	0.0212(7)	0.0209(6)	0.0190(6)	0.0010(5)	0.0090(5)	0.0010(5)
C6	0.0210(7)	0.0172(6)	0.0263(7)	0.0030(5)	0.0108(6)	0.0013(5)
C7	0.0212(7)	0.0176(6)	0.0183(6)	0.0009(5)	0.0105(5)	0.0003(5)
C8	0.0220(7)	0.0190(6)	0.0196(6)	-0.0001(5)	0.0111(5)	-0.0003(5)
C9	0.0261(7)	0.0231(7)	0.0217(7)	0.0014(6)	0.0086(6)	0.0011(6)
C10	0.0206(9)	0.0196(9)	0.0204(9)	0.000	0.0105(7)	0.000
C11	0.0279(10)	0.0186(9)	0.0266(10)	0.000	0.0155(8)	0.000
C12	0.0349(8)	0.0177(6)	0.0333(8)	-0.0040(6)	0.0177(7)	-0.0038(6)
C13	0.0322(8)	0.0248(7)	0.0260(7)	-0.0077(6)	0.0109(6)	-0.0080(6)
Cl1	0.0323(2)	0.01927(19)	0.0297(2)	-0.00531(14)	0.00581(16)	-0.00290(13)

Bond lengths [Å]

C1 - C2 = 1.383(2)
C1 - C6 = 1.385(2)
C1 - Cl1 = 1.7468(14)
C2 - C3 = 1.3890(19)
C2 - H2 = 0.9500
C3 - C4 = 1.3966(19)
C3 - H3 = 0.9500
C4 - C5 = 1.4005(19)
C4 - C7 = 1.4747(18)
C5 - C6 = 1.3848(19)
C5 - H5 = 0.9500
C6 - H6 = 0.9500
C7 - C7_#1 = 1.379(3)
C7 - C8 = 1.4822(18)
C8 - C9 = 1.3737(19)
C8 - C10 = 1.4089(17)
C9 - C13_#1 = 1.425(2)
C9 - H9 = 0.9500

C10 - C11 = 1.393(3)
C11 - C12 = 1.4224(17)
C11 - C12_#1 = 1.4224(17)
C12 - C13 = 1.377(2)
C12 - H12 = 0.9500
C13 - H13 = 0.9500

Symmetry transformations used to generate equivalent atoms:
#1 -x, y, -z+1/2 T = [1, 0, 1]

Angles [°]

C2 - C1 - C6 = 121.73(12)
C2 - C1 - Cl1 = 118.87(11)
C6 - C1 - Cl1 = 119.40(10)
C1 - C2 - C3 = 118.66(13)
C1 - C2 - H2 = 120.70
C3 - C2 - H2 = 120.70
C2 - C3 - C4 = 121.29(13)
C2 - C3 - H3 = 119.40
C4 - C3 - H3 = 119.40
C3 - C4 - C5 = 118.23(12)
C3 - C4 - C7 = 120.09(12)
C5 - C4 - C7 = 121.67(12)
C6 - C5 - C4 = 121.16(13)
C6 - C5 - H5 = 119.40
C4 - C5 - H5 = 119.40
C1 - C6 - C5 = 118.88(12)
C1 - C6 - H6 = 120.60
C5 - C6 - H6 = 120.60
C7_#1 - C7 - C4 = 127.92(7)
C7_#1 - C7 - C8 = 108.40(7)
C4 - C7 - C8 = 123.67(11)
C9 - C8 - C10 = 118.37(13)
C9 - C8 - C7 = 135.25(13)
C10 - C8 - C7 = 106.36(12)
C8 - C9 - C13_#1 = 118.36(14)
C8 - C9 - H9 = 120.80
C13_#1 - C9 - H9 = 120.80
C11 - C10 - C8_#1 = 124.76(8)
C11 - C10 - C8 = 124.76(8)
C8_#1 - C10 - C8 = 110.48(17)
C10 - C11 - C12 = 115.84(9)
C10 - C11 - C12_#1 = 115.84(9)
C12 - C11 - C12_#1 = 128.32(18)
C13 - C12 - C11 = 120.06(14)
C13 - C12 - H12 = 120.00
C11 - C12 - H12 = 120.00
C12 - C13 - C9_#1 = 122.57(14)
C12 - C13 - H13 = 118.70
C9_#1 - C13 - H13 = 118.70

Symmetry transformations used to generate equivalent atoms:
#1 -x, y, -z+1/2 T = [1, 0, 1]

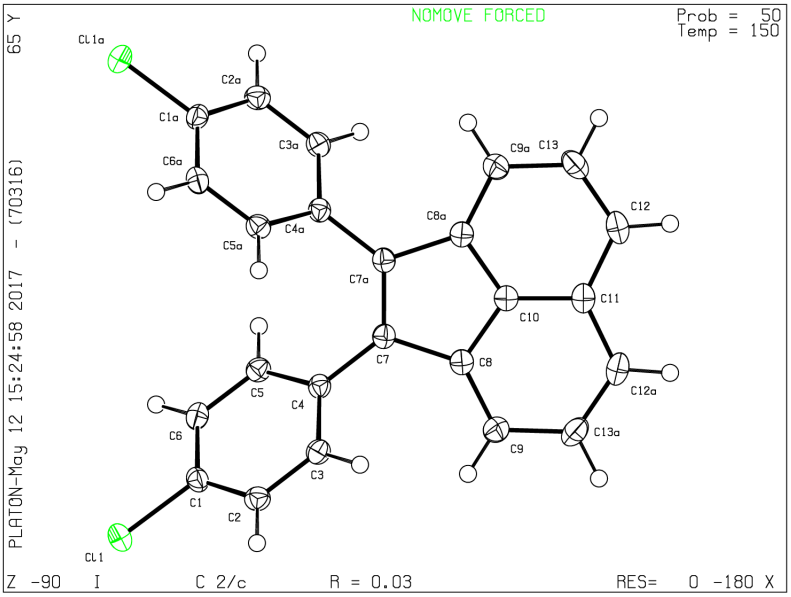
Torsion angles [°]

C6 - C1 - C2 - C3 = 1.20(2)
Cl1 - C1 - C2 - C3 = -178.78(10)
C1 - C2 - C3 - C4 = 0.50(2)
C2 - C3 - C4 - C5 = -2.00(2)
C2 - C3 - C4 - C7 = 177.88(12)
C3 - C4 - C5 - C6 = 1.70(2)
C7 - C4 - C5 - C6 = -178.11(12)
C2 - C1 - C6 - C5 = -1.50(2)
Cl1 - C1 - C6 - C5 = 178.55(10)
C4 - C5 - C6 - C1 = -0.10(2)
C3 - C4 - C7 - C7_#1 = -136.55(18)
C5 - C4 - C7 - C7_#1 = 43.30(2)
C3 - C4 - C7 - C8 = 44.05(19)
C5 - C4 - C7 - C8 = -136.10(14)
C7_#1 - C7 - C8 - C9 = -178.68(16)
C4 - C7 - C8 - C9 = 0.80(2)
C7_#1 - C7 - C8 - C10 = -0.10(17)
C4 - C7 - C8 - C10 = 179.40(11)
C10 - C8 - C9 - C13_#1 = 1.91(19)
C7 - C8 - C9 - C13_#1 = -179.65(15)

C9 - C8 - C10 - C11 = -1.10(14)
C7 - C8 - C10 - C11 = -179.96(6)
C9 - C8 - C10 - C8_#1 = 178.90(14)
C7 - C8 - C10 - C8_#1 = 0.04(6)
C8_#1 - C10 - C11 - C12 = -0.68(10)
C8 - C10 - C11 - C12 = 179.32(10)
C8_#1 - C10 - C11 - C12_#1 = 179.32(10)
C8 - C10 - C11 - C12_#1 = -0.68(10)
C10 - C11 - C12 - C13 = 1.59(16)
C12_#1 - C11 - C12 - C13 = -178.41(16)
C11 - C12 - C13 - C9_#1 = -0.80(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x, y, -z+1/2 T = [1, 0, 1]

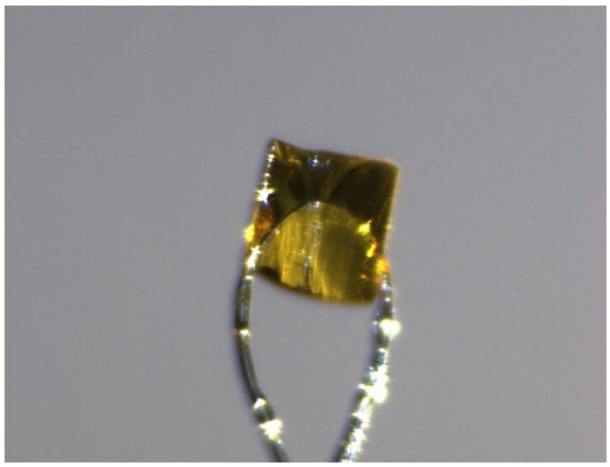
Structure visualisation



Crystal structure report of compound 33:

CDC 1553881

X-ray crystallographic study



(C_{24} H_{13} F); *M* = 320.34. D8 VENTURE Bruker AXS diffractometer [*], Mo-Kα radiation (λ = 0.71073 Å), *T* = 150 K; monoclinic *P* $2_1/n$ (I.T.#14), a = 10.4684(7), b = 7.4280(4), c = 18.7773(11) Å, β = 90.086(3) °, *V* = 1460.11(15) $Å^3$. *Z* = 4, *d* = 1.457 $g.cm^{-3}$, μ = 0.092 mm^{-1}. The structure was solved by dual-space algorithm using the *SHELXT* program [1], and then refined with full-matrix least-square methods based on F^2 (*SHELXL*) [2]. All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions. A final refinement on F^2 with 3338 unique intensities and 230 parameters converged at ω*R*(F^2) = 0.1592 (*R*(*F*) = 0.0647) for 2348 observed reflections with $I > 2\sigma(I)$.

[1] G. M. Sheldrick, Acta Cryst. A71 (2015) 3-8
[2] Sheldrick G.M., Acta Cryst. C71 (2015) 3-8

[*] Thanks to FEDER founds

Structural data

Empirical formula	C_{24} H_{13} F
Formula weight	320.34
Temperature	150 *K*
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, *P* $2_1/n$
Unit cell dimensions	a = 10.4684(7) Å, α = 90 ° b = 7.4280(4) Å, β = 90.086(3) ° c = 18.7773(11) Å, γ = 90 °
Volume	1460.11(15) $Å^3$
Z, Calculated density	4, 1.457 ($g.cm^{-3}$)
Absorption coefficient	0.092 mm^{-1}
F(000)	664
Crystal size	0.390 x 0.340 x 0.045 mm
Crystal color	yellow
Theta range for data collection	3.363 to 27.484 °
h_min, h_max	-13, 13
k_min, k_max	-9, 8
l_min, l_max	-24, 23
Reflections collected / unique	12886 / 3338 [R(int)[a] = 0.0673]
Reflections [I>2σ]	2348
Completeness to theta_max	0.993
Absorption correction type	multi-scan
Max. and min. transmission	0.996 , 0.723
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3338 / 0 / 230
[b]S (Goodness-of-fit)	1.047
Final *R* indices [I>2σ]	*R*1[c] = 0.0647, *wR*2[d] = 0.1592
R indices (all data)	*R*1[c] = 0.0962, *wR*2[d] = 0.1789
Largest diff. peak and hole	0.623 and -0.563 $e^-.Å^{-3}$

[a] $R_{int} = \sum |F_o^2 - \langle F_o^2 \rangle| / \sum [F_o^2]$
[b] $S = \{\sum [w(F_o^2 - F_c^2)^2] / (n - p)\}^{1/2}$
[c] $R1 = \sum ||F_o| - |F_c|| / \sum |F_o|$
[d] $wR2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]\}^{1/2}$
$w = 1 / [\sigma(F_o^2) + aP^2 + bP]$ where $P = [2F_c^2 + MAX(F_o^2, 0)] /3$

Atomic coordinates, site occupancy (%) and equivalent isotropic displacement parameters (Å²). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	occ.	U(eq)
C1	0.3973(2)	0.1902(3)	0.46823(12)	1	0.0247(5)
C2	0.2835(2)	0.1289(3)	0.43615(13)	1	0.0332(6)
H2	0.284957	0.087315	0.388312	1	0.040
C3A	0.1710(3)	0.1286(4)	0.47323(14)	0.595(3)	0.0407(7)
H3A	0.095246	0.087019	0.450655	0.595(3)	0.049
C3B	0.1710(3)	0.1286(4)	0.47323(14)	0.405(3)	0.0407(7)
F4	0.0652(4)	0.0799(7)	0.4415(2)	0.405(3)	0.0558(7)
C5	0.1656(3)	0.1882(4)	0.54322(14)	1	0.0412(7)
H5	0.087139	0.185925	0.568525	1	0.049
C6	0.2748(2)	0.2505(3)	0.57564(13)	1	0.0315(6)
H6	0.270664	0.292199	0.623411	1	0.038
C7	0.3928(2)	0.2539(3)	0.53953(11)	1	0.0235(5)
C8	0.5084(2)	0.3177(3)	0.57184(11)	1	0.0217(5)
C9	0.5305(2)	0.3956(3)	0.64362(11)	1	0.0230(5)
C10	0.4587(2)	0.4345(3)	0.70307(12)	1	0.0297(5)

H10	0.370590	0.404410	0.704526	1	0.036
C11	0.5178(3)	0.5199(4)	0.76206(12)	1	0.0338(6)
H11	0.467190	0.546592	0.802667	1	0.041
C12	0.6443(3)	0.5656(3)	0.76312(12)	1	0.0313(6)
H12	0.679862	0.624784	0.803392	1	0.038
C13	0.7219(2)	0.5239(3)	0.70356(12)	1	0.0270(5)
C14	0.8546(3)	0.5588(3)	0.69658(13)	1	0.0346(6)
H14	0.899922	0.617417	0.733839	1	0.041
C15	0.9168(3)	0.5076(4)	0.63598(14)	1	0.0408(7)
H15	1.006252	0.527921	0.632738	1	0.049
C16	0.8531(2)	0.4252(4)	0.57756(14)	1	0.0359(6)
H16	0.899438	0.391923	0.536135	1	0.043
C17	0.7238(2)	0.3941(3)	0.58151(12)	1	0.0248(5)
C18	0.6621(2)	0.4406(3)	0.64588(11)	1	0.0238(5)
C19	0.6227(2)	0.3190(3)	0.53485(11)	1	0.0227(5)
C20	0.6295(2)	0.2593(3)	0.46229(12)	1	0.0244(5)
C21	0.7431(2)	0.2681(4)	0.42199(13)	1	0.0338(6)
H21	0.818165	0.315117	0.443526	1	0.041
C22	0.7480(3)	0.2110(4)	0.35283(14)	1	0.0415(7)
H22	0.824785	0.220393	0.326197	1	0.050
C23A	0.6387(3)	0.1390(4)	0.32237(13)	0.595(3)	0.0385(6)
F24	0.6447(3)	0.0784(5)	0.25537(14)	0.595(3)	0.0558(7)
C23B	0.6387(3)	0.1390(4)	0.32237(13)	0.405(3)	0.0385(6)
H23B	0.642451	0.094836	0.274945	0.405(3)	0.046
C25	0.5254(2)	0.1296(3)	0.35852(12)	1	0.0314(6)
H25	0.452022	0.080782	0.335804	1	0.038
C26	0.5171(2)	0.1920(3)	0.42931(11)	1	0.0239(5)

Anisotropic displacement parameters (Å²)

The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \ldots + 2 h k a^* b^* U_{12}]$.

Atom	U11	U22	U33	U23	U13	U12
C1	0.0323(12)	0.0204(10)	0.0214(11)	0.0061(8)	-0.0065(9)	-0.0041(9)
C2	0.0414(15)	0.0342(13)	0.0239(12)	0.0050(10)	-0.0122(11)	-0.0119(11)
C3A	0.0353(15)	0.0532(17)	0.0336(14)	0.0123(12)	-0.0124(11)	-0.0201(12)
C3B	0.0353(15)	0.0532(17)	0.0336(14)	0.0123(12)	-0.0124(11)	-0.0201(12)
F4	0.0446(14)	0.0897(19)	0.0330(12)	-0.0144(12)	-0.0059(10)	-0.0068(13)
C5	0.0325(14)	0.0579(18)	0.0331(14)	0.0133(13)	-0.0041(11)	-0.0166(13)
C6	0.0295(13)	0.0402(14)	0.0248(12)	0.0062(10)	-0.0018(10)	-0.0084(10)
C7	0.0278(12)	0.0226(11)	0.0201(11)	0.0060(8)	-0.0061(9)	-0.0037(9)
C8	0.0260(11)	0.0204(10)	0.0186(10)	0.0042(8)	-0.0039(8)	-0.0004(8)
C9	0.0289(12)	0.0232(11)	0.0168(10)	0.0055(8)	-0.0052(9)	-0.0032(9)
C10	0.0325(13)	0.0363(13)	0.0202(11)	0.0043(9)	-0.0014(9)	-0.0033(10)
C11	0.0418(15)	0.0410(14)	0.0185(11)	-0.0002(10)	0.0000(10)	0.0004(11)
C12	0.0469(15)	0.0279(12)	0.0189(11)	0.0019(9)	-0.0102(10)	-0.0034(10)
C13	0.0357(13)	0.0252(11)	0.0202(11)	0.0057(9)	-0.0096(9)	-0.0035(9)
C14	0.0382(14)	0.0385(14)	0.0270(12)	0.0065(10)	-0.0160(11)	-0.0106(11)
C15	0.0286(13)	0.0618(18)	0.0320(14)	0.0084(12)	-0.0102(11)	-0.0132(12)
C16	0.0269(13)	0.0550(17)	0.0259(12)	0.0021(11)	-0.0053(10)	-0.0027(11)
C17	0.0260(12)	0.0285(12)	0.0199(11)	0.0034(9)	-0.0066(9)	-0.0001(9)
C18	0.0286(12)	0.0236(11)	0.0193(10)	0.0050(8)	-0.0059(9)	-0.0013(9)
C19	0.0243(11)	0.0233(11)	0.0206(11)	0.0045(8)	-0.0056(8)	0.0007(9)
C20	0.0256(11)	0.0264(11)	0.0212(11)	0.0016(9)	-0.0045(9)	0.0060(9)
C21	0.0248(12)	0.0485(15)	0.0280(13)	-0.0035(11)	-0.0015(10)	0.0061(11)
C22	0.0353(14)	0.0613(18)	0.0279(13)	-0.0069(12)	0.0017(11)	0.0150(13)
C23A	0.0463(16)	0.0480(16)	0.0213(12)	-0.0088(11)	-0.0040(11)	0.0165(12)
F24	0.0446(14)	0.0897(19)	0.0330(12)	-0.0144(12)	-0.0059(10)	-0.0068(13)
C23B	0.0463(16)	0.0480(16)	0.0213(12)	-0.0088(11)	-0.0040(11)	0.0165(12)
C25	0.0382(14)	0.0325(13)	0.0236(12)	-0.0030(10)	-0.0111(10)	0.0062(10)
C26	0.0320(12)	0.0205(10)	0.0193(10)	0.0020(8)	-0.0073(9)	0.0034(9)

Bond lengths [Å]

C1 - C2 = 1.409(3)
C1 - C7 = 1.421(3)
C1 - C26 = 1.452(3)
C2 - C3B = 1.370(4)
C2 - C3A = 1.370(4)
C2 - H2 = 0.9500
C3A - C5 = 1.388(4)
C3A - H3A = 0.9500
C3B - F4 = 1.308(5)
C3B - C5 = 1.388(4)
C5 - C6 = 1.374(3)

C5 - H5 = 0.9500
C6 - C7 = 1.411(3)
C6 - H6 = 0.9500
C7 - C8 = 1.433(3)
C8 - C19 = 1.384(3)
C8 - C9 = 1.485(3)
C9 - C10 = 1.377(3)
C9 - C18 = 1.418(3)
C10 - C11 = 1.417(3)
C10 - H10 = 0.9500
C11 - C12 = 1.367(4)
C11 - H11 = 0.9500
C12 - C13 = 1.418(4)
C12 - H12 = 0.9500
C13 - C18 = 1.395(3)
C13 - C14 = 1.420(4)
C14 - C15 = 1.366(4)
C14 - H14 = 0.9500
C15 - C16 = 1.422(3)
C15 - H15 = 0.9500
C16 - C17 = 1.375(3)
C16 - H16 = 0.9500
C17 - C18 = 1.414(3)
C17 - C19 = 1.482(3)
C19 - C20 = 1.435(3)
C20 - C21 = 1.411(3)
C20 - C26 = 1.420(3)
C21 - C22 = 1.367(3)
C21 - H21 = 0.9500
C22 - C23B = 1.386(4)
C22 - C23A = 1.386(4)
C22 - H22 = 0.9500
C23A - F24 = 1.338(4)
C23A - C25 = 1.369(4)
C23B - C25 = 1.369(4)
C23B - H23B = 0.9500
C25 - C26 = 1.410(3)
C25 - H25 = 0.9500

Angles [°]

C2 - C1 - C7 = 118.80(2)
C2 - C1 - C26 = 121.20(2)
C7 - C1 - C26 = 120.10(2)
C3B - C2 - C1 = 120.70(2)
C3A - C2 - C1 = 120.70(2)
C3A - C2 - H2 = 119.70
C1 - C2 - H2 = 119.70
C2 - C3A - C5 = 121.10(2)
C2 - C3A - H3A = 119.40
C5 - C3A - H3A = 119.40
F4 - C3B - C2 = 119.80(3)
F4 - C3B - C5 = 119.00(3)
C2 - C3B - C5 = 121.10(2)
C6 - C5 - C3A = 119.50(3)
C6 - C5 - C3B = 119.50(3)
C6 - C5 - H5 = 120.30
C3A - C5 - H5 = 120.30
C5 - C6 - C7 = 121.40(2)
C5 - C6 - H6 = 119.30
C7 - C6 - H6 = 119.30
C6 - C7 - C1 = 118.50(2)
C6 - C7 - C8 = 122.80(2)
C1 - C7 - C8 = 118.70(2)
C19 - C8 - C7 = 121.30(2)
C19 - C8 - C9 = 108.60(19)
C7 - C8 - C9 = 130.10(2)
C10 - C9 - C18 = 117.20(2)
C10 - C9 - C8 = 137.10(2)
C18 - C9 - C8 = 105.61(19)
C9 - C10 - C11 = 119.30(2)
C9 - C10 - H10 = 120.30
C11 - C10 - H10 = 120.30
C12 - C11 - C10 = 122.90(2)

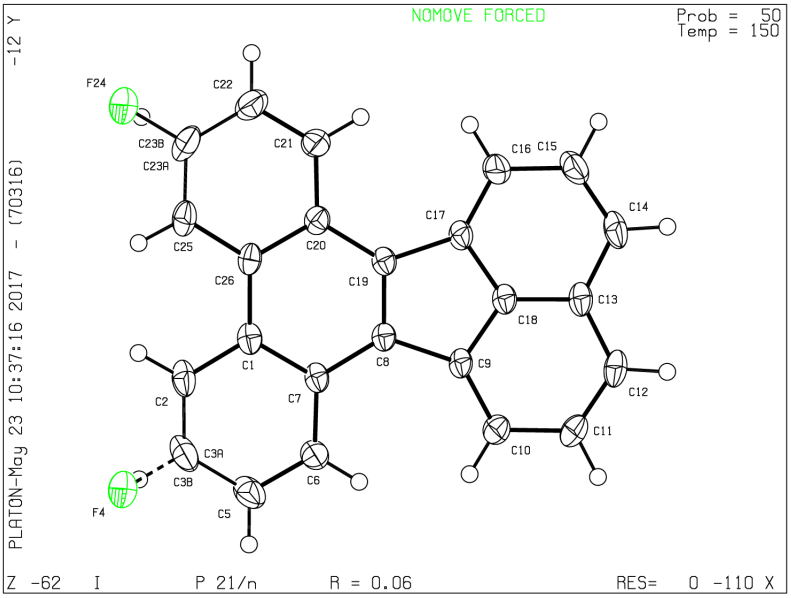
C12 - C11 - H11 = 118.50
C10 - C11 - H11 = 118.50
C11 - C12 - C13 = 119.40(2)
C11 - C12 - H12 = 120.30
C13 - C12 - H12 = 120.30
C18 - C13 - C12 = 116.90(2)
C18 - C13 - C14 = 116.60(2)
C12 - C13 - C14 = 126.50(2)
C15 - C14 - C13 = 119.60(2)
C15 - C14 - H14 = 120.20
C13 - C14 - H14 = 120.20
C14 - C15 - C16 = 122.60(2)
C14 - C15 - H15 = 118.70
C16 - C15 - H15 = 118.70
C17 - C16 - C15 = 119.40(2)
C17 - C16 - H16 = 120.30
C15 - C16 - H16 = 120.30
C16 - C17 - C18 = 117.10(2)
C16 - C17 - C19 = 137.20(2)
C18 - C17 - C19 = 105.71(19)
C13 - C18 - C17 = 124.60(2)
C13 - C18 - C9 = 124.20(2)
C17 - C18 - C9 = 111.21(19)
C8 - C19 - C20 = 121.20(2)
C8 - C19 - C17 = 108.84(19)
C20 - C19 - C17 = 129.90(2)
C21 - C20 - C26 = 118.70(2)
C21 - C20 - C19 = 122.50(2)
C26 - C20 - C19 = 118.70(2)
C22 - C21 - C20 = 121.90(2)
C22 - C21 - H21 = 119.10
C20 - C21 - H21 = 119.10
C21 - C22 - C23B = 118.60(3)
C21 - C22 - C23A = 118.60(3)
C21 - C22 - H22 = 120.70
C23A - C22 - H22 = 120.70
F24 - C23A - C25 = 119.40(3)
F24 - C23A - C22 = 118.50(3)
C25 - C23A - C22 = 122.00(2)
C25 - C23B - C22 = 122.00(2)
C25 - C23B - H23B = 119.00
C22 - C23B - H23B = 119.00
C23B - C25 - C26 = 120.40(2)
C23A - C25 - C26 = 120.40(2)
C23A - C25 - H25 = 119.80
C26 - C25 - H25 = 119.80
C25 - C26 - C20 = 118.30(2)
C25 - C26 - C1 = 121.70(2)
C20 - C26 - C1 = 120.00(2)

Torsion angles [°]

C7 - C1 - C2 - C3B = -0.80(4)
C26 - C1 - C2 - C3B = -179.70(2)
C7 - C1 - C2 - C3A = -0.80(4)
C26 - C1 - C2 - C3A = -179.70(2)
C1 - C2 - C3A - C5 = -0.10(4)
C1 - C2 - C3B - F4 = 176.60(3)
C1 - C2 - C3B - C5 = -0.10(4)
C2 - C3A - C5 - C6 = 0.80(4)
F4 - C3B - C5 - C6 = -175.90(4)
C2 - C3B - C5 - C6 = 0.80(4)
C3A - C5 - C6 - C7 = -0.60(4)
C3B - C5 - C6 - C7 = -0.60(4)
C5 - C6 - C7 - C1 = -0.30(4)
C5 - C6 - C7 - C8 = -179.90(2)
C2 - C1 - C7 - C6 = 1.00(3)
C26 - C1 - C7 - C6 = 179.90(2)
C2 - C1 - C7 - C8 = -179.40(2)
C26 - C1 - C7 - C8 = -0.60(3)
C6 - C7 - C8 - C19 = 179.90(2)
C1 - C7 - C8 - C19 = 0.40(3)
C6 - C7 - C8 - C9 = -2.40(4)
C1 - C7 - C8 - C9 = 178.00(2)

C19 - C8 - C9 - C10 = 178.30(3)
C7 - C8 - C9 - C10 = 0.40(4)
C19 - C8 - C9 - C18 = -0.20(2)
C7 - C8 - C9 - C18 = -178.00(2)
C18 - C9 - C10 - C11 = 1.40(3)
C8 - C9 - C10 - C11 = -176.90(2)
C9 - C10 - C11 - C12 = -0.30(4)
C10 - C11 - C12 - C13 = -1.20(4)
C11 - C12 - C13 - C18 = 1.40(3)
C11 - C12 - C13 - C14 = -178.80(2)
C18 - C13 - C14 - C15 = -1.20(3)
C12 - C13 - C14 - C15 = 178.90(2)
C13 - C14 - C15 - C16 = 2.20(4)
C14 - C15 - C16 - C17 = -0.30(4)
C15 - C16 - C17 - C18 = -2.50(4)
C15 - C16 - C17 - C19 = 178.30(3)
C12 - C13 - C18 - C17 = 178.10(2)
C14 - C13 - C18 - C17 = -1.70(3)
C12 - C13 - C18 - C9 = -0.20(3)
C14 - C13 - C18 - C9 = 180.00(2)
C16 - C17 - C18 - C13 = 3.60(3)
C19 - C17 - C18 - C13 = -176.90(2)
C16 - C17 - C18 - C9 = -177.90(2)
C19 - C17 - C18 - C9 = 1.60(2)
C10 - C9 - C18 - C13 = -1.20(3)
C8 - C9 - C18 - C13 = 177.60(2)
C10 - C9 - C18 - C17 = -179.70(2)
C8 - C9 - C18 - C17 = -0.90(2)
C7 - C8 - C19 - C20 = 1.00(3)
C9 - C8 - C19 - C20 = -177.03(19)
C7 - C8 - C19 - C17 = 179.21(19)
C9 - C8 - C19 - C17 = 1.10(2)
C16 - C17 - C19 - C8 = 177.60(3)
C18 - C17 - C19 - C8 = -1.70(2)
C16 - C17 - C19 - C20 = -4.40(4)
C18 - C17 - C19 - C20 = 176.30(2)
C8 - C19 - C20 - C21 = 176.50(2)
C17 - C19 - C20 - C21 = -1.20(4)
C8 - C19 - C20 - C26 = -2.30(3)
C17 - C19 - C20 - C26 = 180.00(2)
C26 - C20 - C21 - C22 = -1.10(4)
C19 - C20 - C21 - C22 = -179.90(2)
C20 - C21 - C22 - C23B = -1.40(4)
C20 - C21 - C22 - C23A = -1.40(4)
C21 - C22 - C23A - F24 = -178.10(3)
C21 - C22 - C23A - C25 = 2.40(4)
C21 - C22 - C23B - C25 = 2.40(4)
C22 - C23B - C25 - C26 = -0.80(4)
F24 - C23A - C25 - C26 = 179.70(3)
C22 - C23A - C25 - C26 = -0.80(4)
C23B - C25 - C26 - C20 = -1.80(3)
C23A - C25 - C26 - C20 = -1.80(3)
C23B - C25 - C26 - C1 = 177.60(2)
C23A - C25 - C26 - C1 = 177.60(2)
C21 - C20 - C26 - C25 = 2.70(3)
C19 - C20 - C26 - C25 = -178.50(2)
C21 - C20 - C26 - C1 = -176.70(2)
C19 - C20 - C26 - C1 = 2.10(3)
C2 - C1 - C26 - C25 = -1.20(3)
C7 - C1 - C26 - C25 = 179.90(2)
C2 - C1 - C26 - C20 = 178.20(2)
C7 - C1 - C26 - C20 = -0.70(3)

Structure visualisation


NOMOVE FORCED
Prob = 50
Temp = 150
PLATON-May 23 10:37:16 2017 - (70316)
-12 Y
F24
C22
C23B
C23A
C21
C16
C15
C17
C14
C25
C20
C26
C19
C18
C13
C1
C8
C9
C2
C7
C12
C11
C3A
C10
C3B
C6
F4
C5
Z -62 I P 21/n R = 0.06 RES= 0 -110 X