Experimental data belonging to the paper

Porphyrin Co(III)-nitrene radical mediated pathway for synthesis of *o*-aminoazobenzenes

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X-ray single crystal determination Compound C

$C_{20}H_{28}N_4$ Fw = 324.46, orange plate, 0.80 x 0.29 x 0.08 mm, monoclinic, P 21/c (no. 14), a = 11.7911(5), b = 6.7919(3), c = 11.5759(5)Å, α = 90 °, β = 94.530(2), γ = 90 °, V = 924.15(7) Å^3, Z = 2, D_x = 1.166 g cm^{-3}, μ = 0.071 mm^{-1}. In total, 7644 reflections were measured on a Bruker D8 Quest Eco diffractometer, equipped with a TRIUMPH monochromator and a CMOS PHOTON 50 detector (λ = 0.71073 Å) up to a resolution of $(\sin\theta/\lambda)_{max}$ = 0.84 Å^{-1} at a temperature of 150(2) K. The intensity data were integrated with the Bruker APEX2 software.[1] Absorption correction and scaling was performed with SADABS.[2] (0.82–0.99 correction range). In total, 1615 reflections were unique (R_{int}= 0.110), of which 1280 were observed [I>2σ(I)]. The structure was solved with Intrinsic Phasing Methods using SHELXT[3] and refined with SHELXL-2013[4] against F^2 of all reflections. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were introduced in calculated positions and refined with a riding model. R1/wR2 [I > 2σ(I)]: 0.0428/0.1482. S = 1.142. Residual electron density between -0.192 and 0.168 e Å^{-3}. Geometry calculations and checking for higher symmetry was performed with the PLATON program.[5]

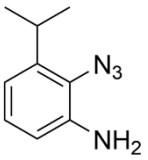
[1] Bruker, APEX2 software, Madison, WI, USA, **2014**.

[2] SAINT, version 6.02, and SADABS, version 2.03; Bruker AXS, Inc., Madison, WI, **2002**.

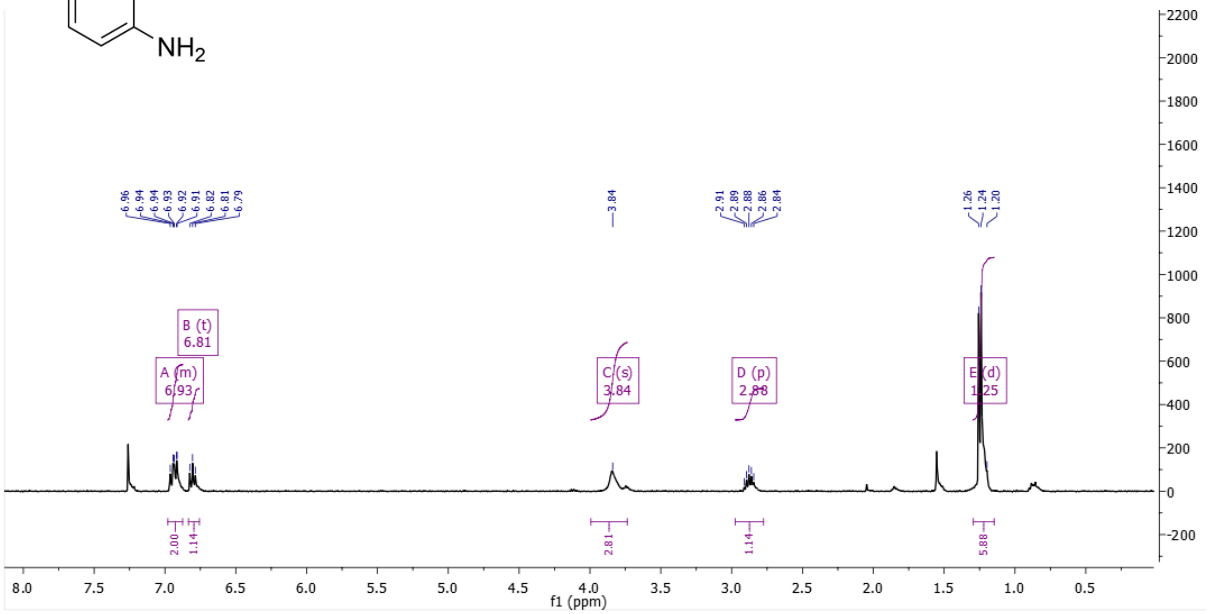
[3] G.M. Sheldrick, SHELXT - Universität Göttingen, Germany, **2012**.

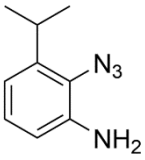
[4] G.M. Sheldrick, *Acta Cryst*. **2015**, A71, 3–8.

[5] A.L. Spek, *Acta Cryst*. **2009**, D65, 148–155.

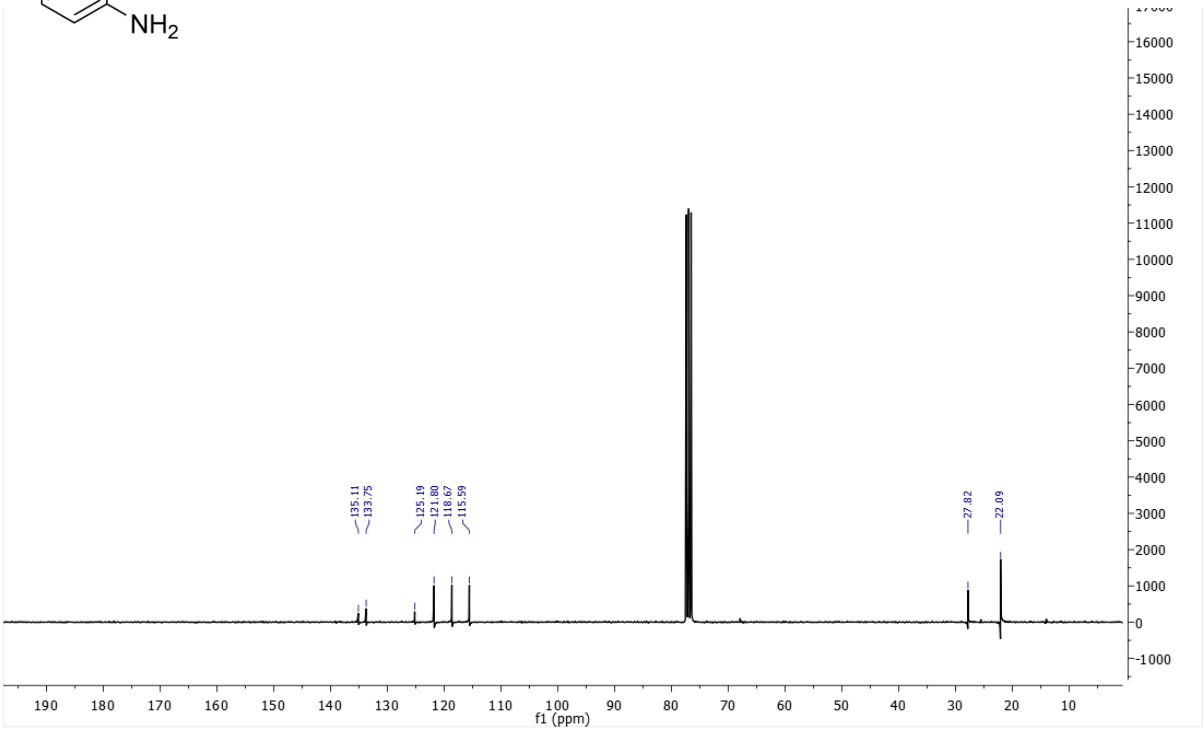


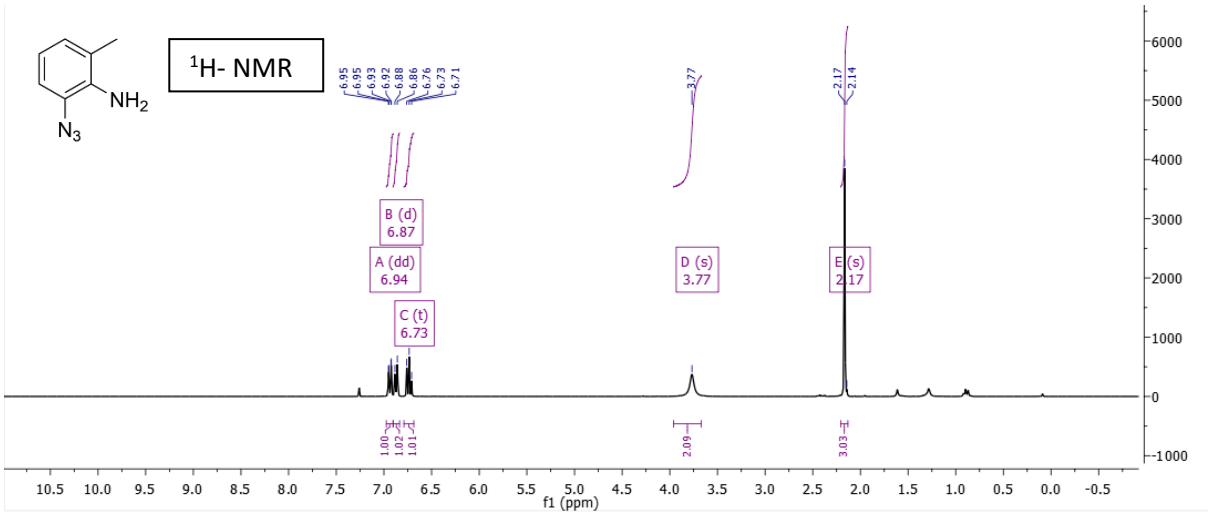
^{1}H- NMR

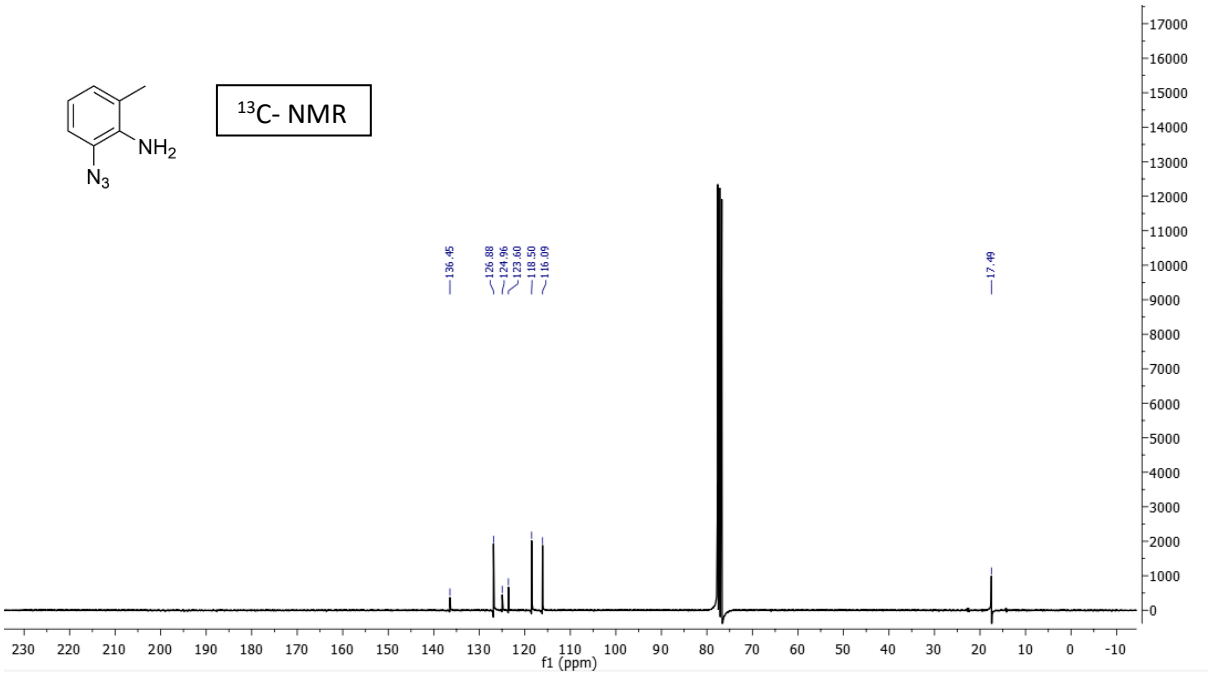


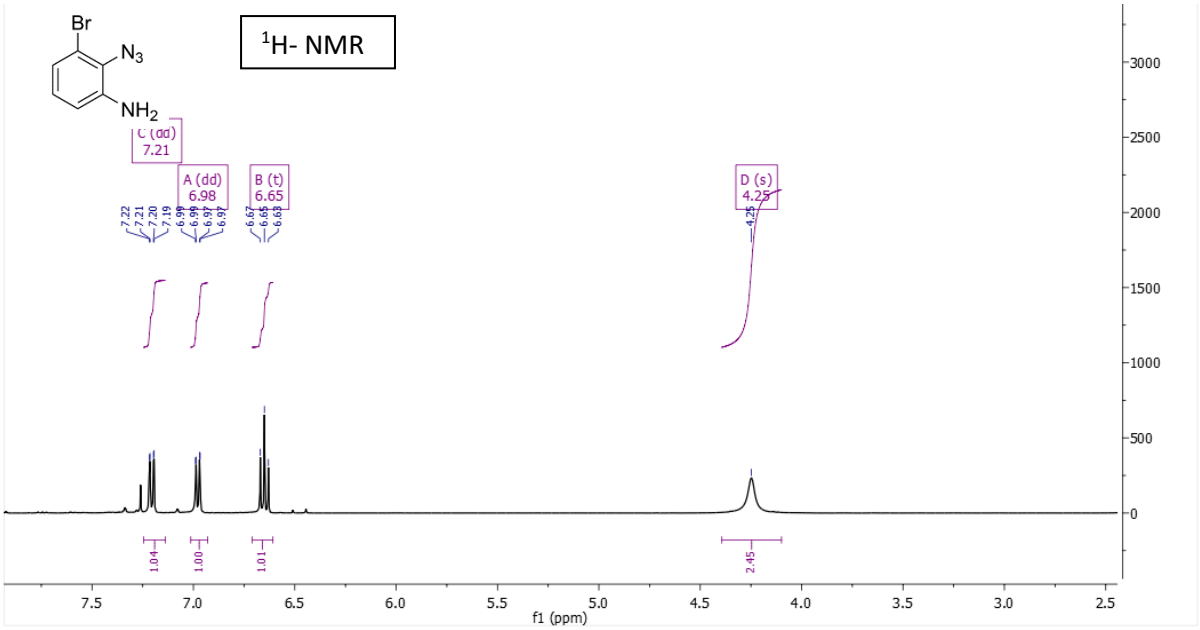


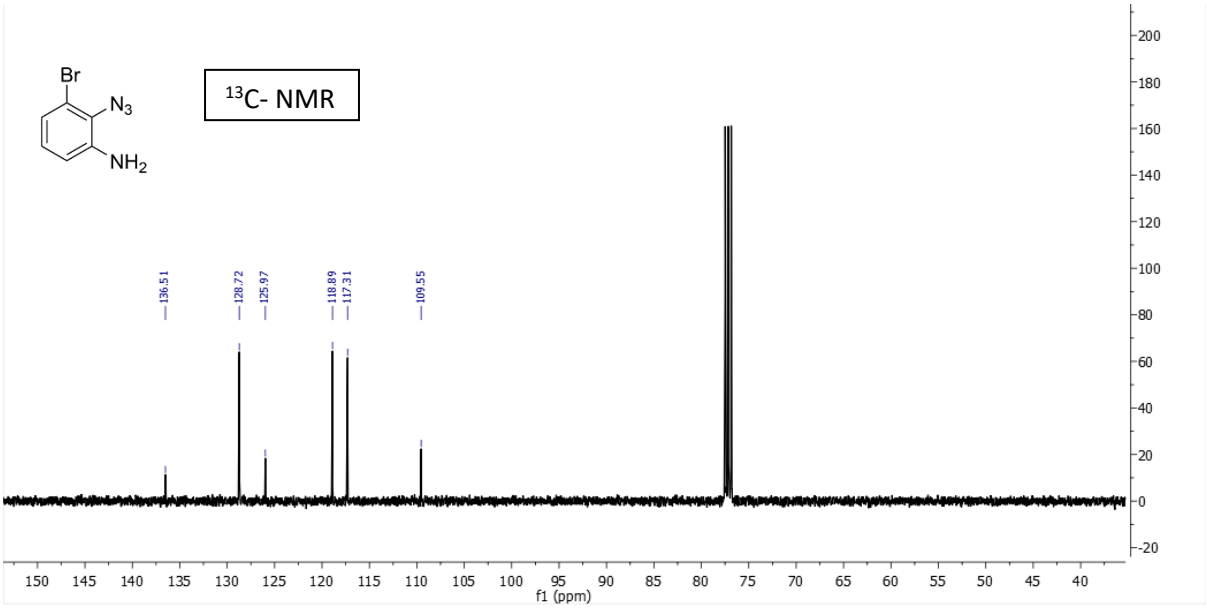
^{13}C- NMR

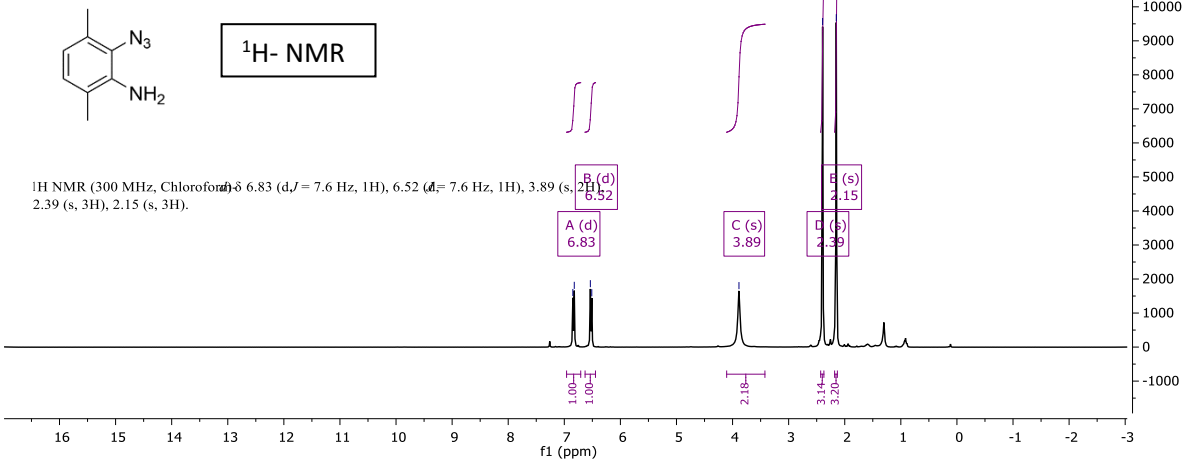



^{1}H- NMR
NH2
N3
6.95
6.95
6.93
6.92
6.88
6.86
6.76
6.73
6.71
3.77
2.17
2.14
B (d)
6.87
A (dd)
6.94
C (t)
6.73
D (s)
3.77
E (s)
2.17
1.00
1.02
1.01
2.09
3.03
f1 (ppm)

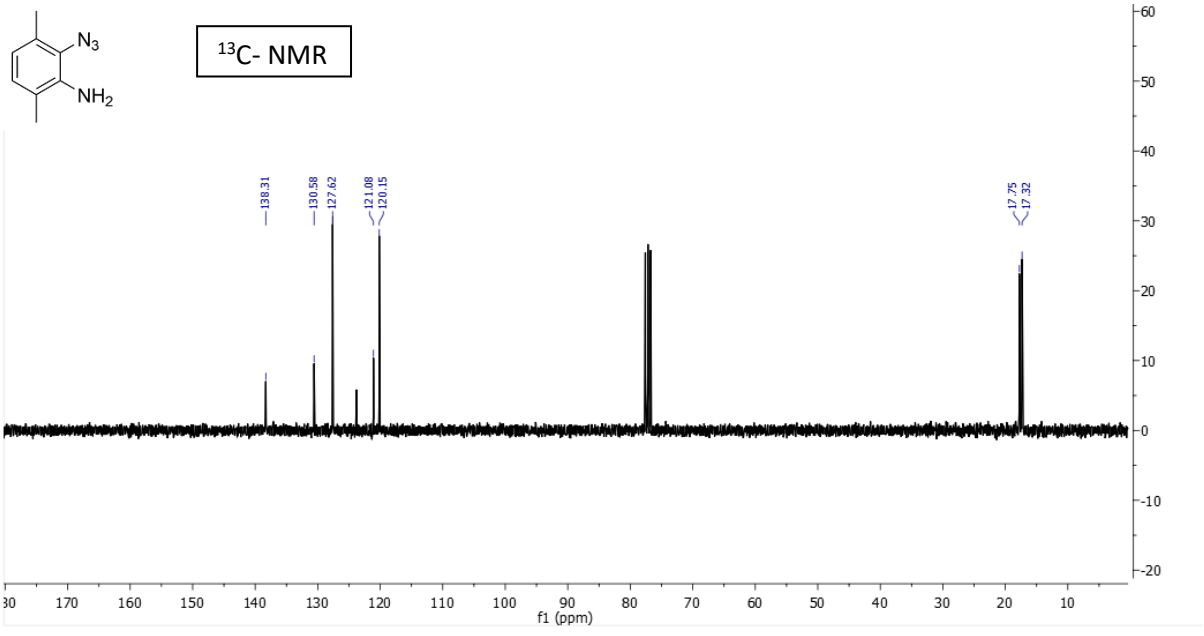

^{13}C- NMR
NH2
N3
136.45
126.88
124.96
123.60
118.50
116.09
17.49
f1 (ppm)


Br
N3
NH2
1H- NMR
C (dd)
7.21
A (dd)
6.98
B (t)
6.65
D (s)
4.25
7.22
7.21
7.20
7.19
6.99
6.98
6.97
6.97
6.67
6.65
6.65
6.63
4.25
1.04
1.00
1.01
2.45
3000
2500
2000
1500
1000
500
0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
f1 (ppm)

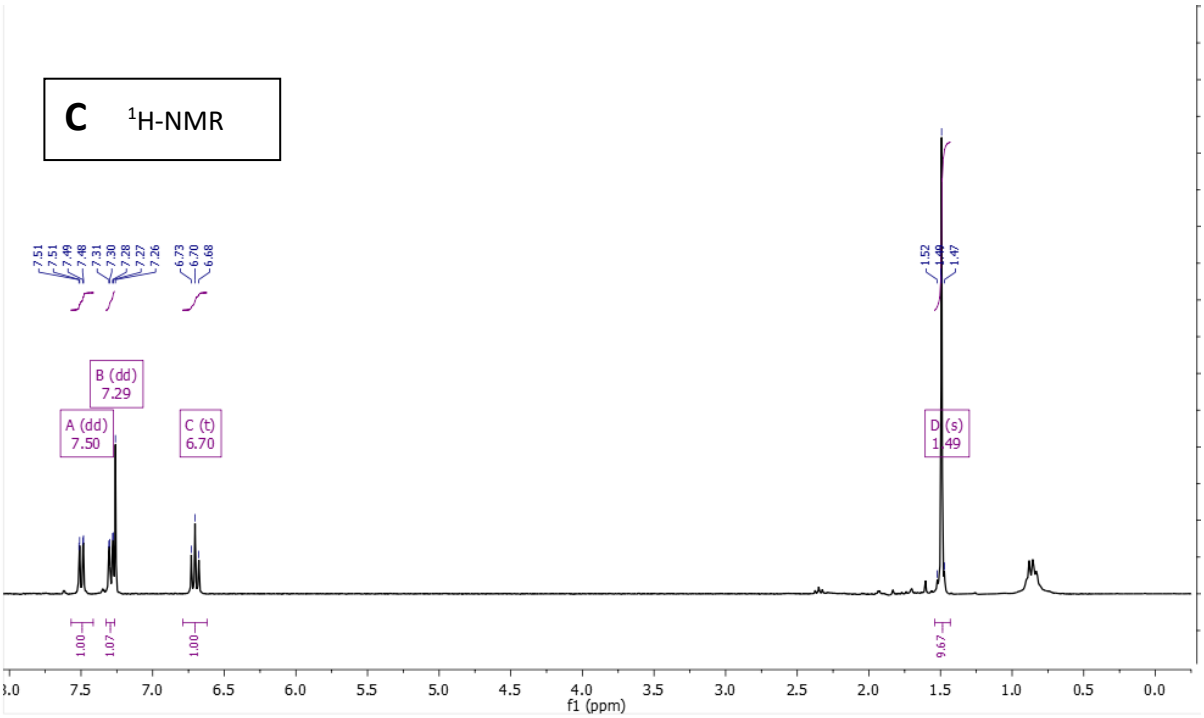

Br
N3
NH2
13C- NMR
136.51
128.72
125.97
118.89
117.31
109.55
200
180
160
140
120
100
80
60
40
20
0
-20
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
f1 (ppm)



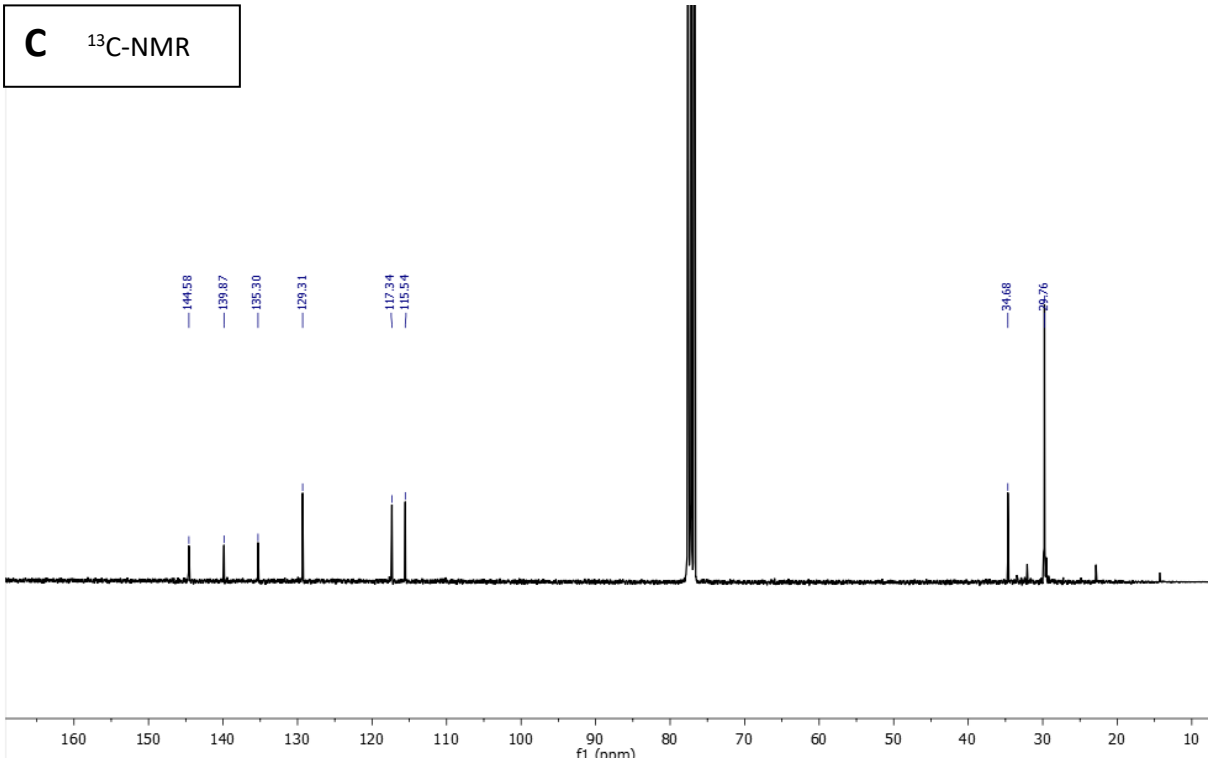
1H NMR (300 MHz, Chloroform-*d*) δ 6.83 (d, *J* = 7.6 Hz, 1H), 6.52 (d, *J* = 7.6 Hz, 1H), 3.89 (s, 2H), 2.39 (s, 3H), 2.15 (s, 3H).



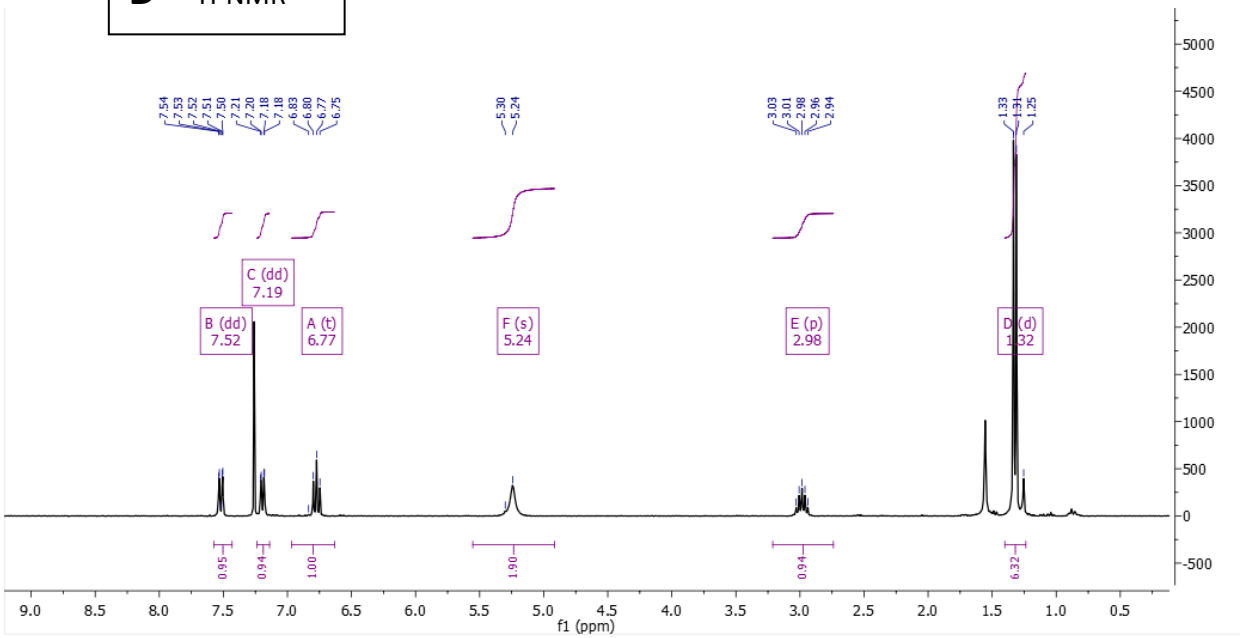
C ^{1}H-NMR



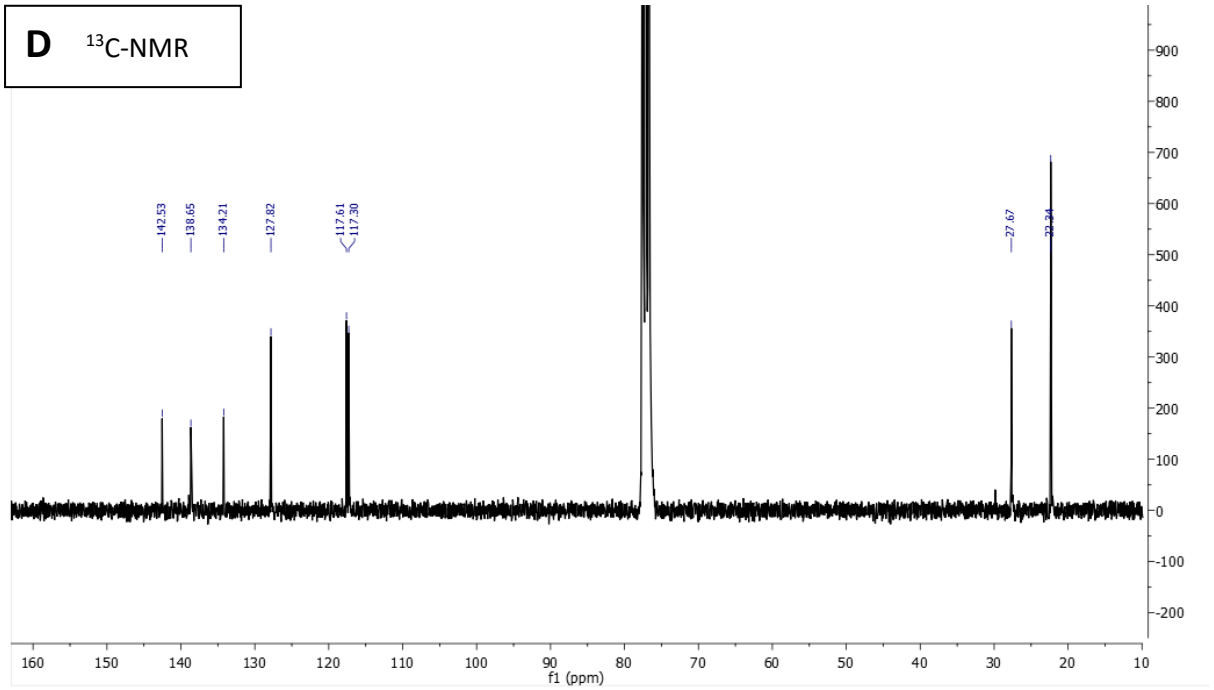
C ^{13}C-NMR



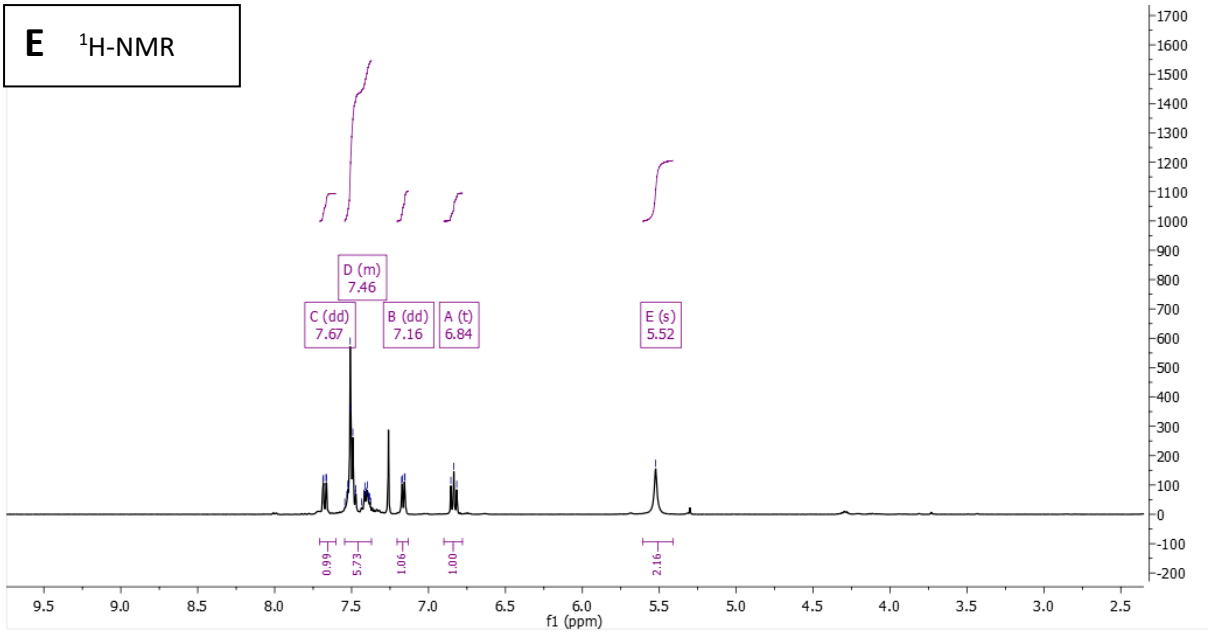
D ^{1}H-NMR



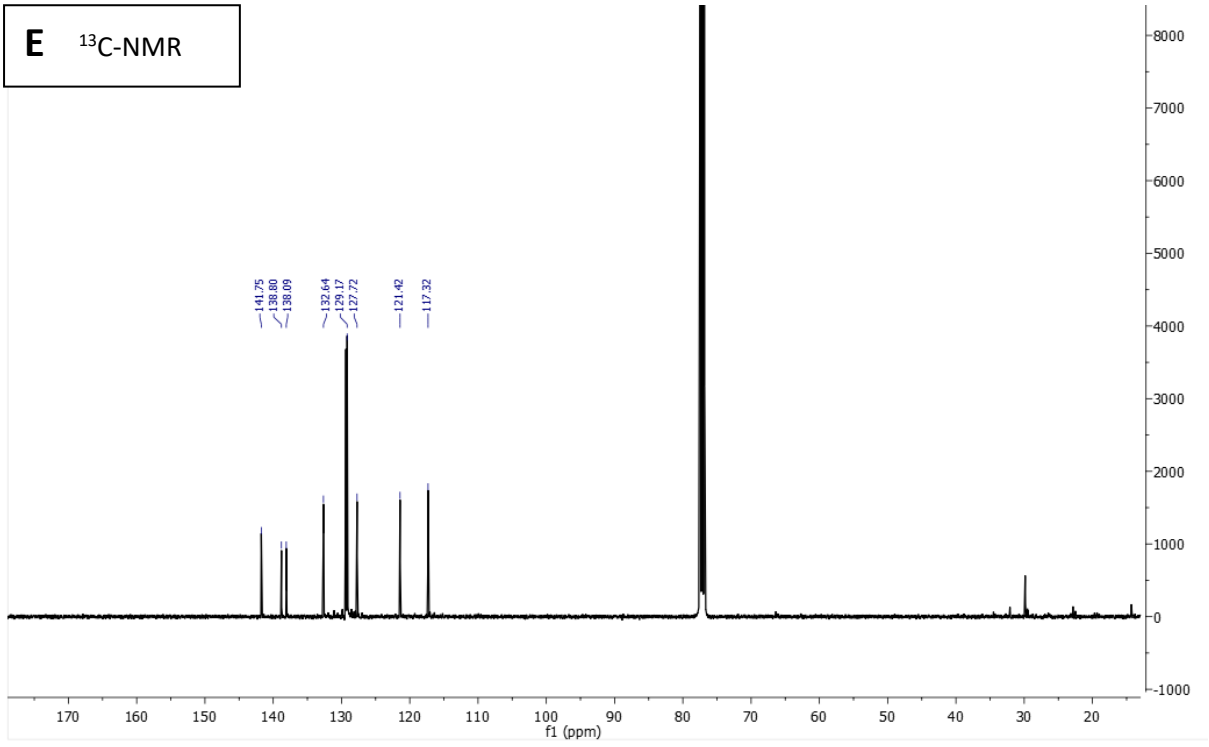
D ^{13}C-NMR



E ^{1}H-NMR



E ^{13}C-NMR



F ^{1}H-NMR