Efficient Palladium-Catalyzed Double Carbonylation of *o*-dibromobenzenes: Synthesis of thalidomide

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General Methods

NMR spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. Multiplets were assigned as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), m (multiplet) and br. s (broad singlet). All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HRMS) were recorded on Agilent 6210. The data are given as mass units per charge (*m/z*). Gas chromatography analysis was performed on an Agilent HP-5890 instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck).

X-ray crystal structure analysis of 5-methyl-2-(pyridin-2-yl)isoindoline-1,3-dione:

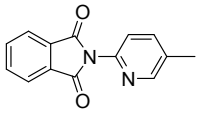
Data were collected on a Bruker Kappa APEX II Duo diffractometer. The structure was solved by direct methods and refined by full-matrix least-squares procedures on F2 with the SHELXTL software package (G. M. Sheldrick, Acta Crystallogr. 2008, A64, 112–122.).

Crystal data for 5-methyl-2-(pyridin-2-yl)isoindoline-1,3-dione: $C_{14}H_{10}N_2O_2$, M = 238.24, monoclinic, space group P21/c, a = 12.9282(5), b = 13.3172(6), c = 6.6540(3) Å, β = 102.290(1)°, V = 1119.35(8) Å3, T = 150(2) K, Z = 4, 15678 reflections measured, 2926 independent reflections (Rint = 0.0349), final R values (I > 2σ(I)): R1 = 0.0432, wR2 = 0.1058, final R values (all data): R1 = 0.0641, wR2 = 0.1185, 164 parameters.

General Procedure:

The reaction was carried out in a Parr Instruments 4560 series 300 mL autoclave containing an alloy plate with wells for six 4 mL Wheaton vials. $Pd(OAc)_2$, (4.49 mg, 2.0 mol%), *CataC*Xium A (21,48 mg, 6.0 mol%), amino pyridine (1.1 mmol), and a magnetic stir bar were placed in each vials, which were then capped with a septum equipped with an inlet needle and flushed with argon. Then TEA (3 mmol, 3.0 equiv.), *o*-dibromobenzene (1.0 mmol) and DMAc (5 mL) were added to the vial *via*

syringe. The vials were placed in an autoclave, which was then purged several times with argon. Subsequently it was filled with 30 bars of CO at room temperature and heated at 100 °C for 20 h. After the reaction the autoclave was cooled to room temperature and vented to discharge N_2. The product was extracted with ethyl acetate (5×3 mL). The organic layers were washed with brine, dried over Na_2SO_4, and evaporated to yield the crude reaction mixture. The purification occurred by flash chromatography on silica gel (eluent: heptane/EtOAc 50:50).

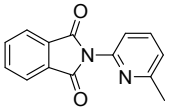


2-(5-Methylpyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.46 (s, 1*H*), 8.06 – 7.94 (m, 4*H*), 7.91 – 7.78 (m, 1*H*), 7.44 (d, *J* = 8.0 Hz, 1*H*), 2.38 (s, 3*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.67, 149.53, 143.49, 138.96, 135.02, 133.85, 131.41, 123.68, 122.55, 17.60.

MS (EI, 70 eV): *m/z* (%) = 238 ([M]$^+$, 70), 210 (100), 193 (9), 182 (10), 104(14), 76(20).

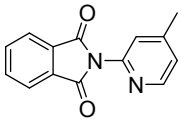


2-(6-Methylpyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6)) δ 8.49 (dd, *J* = 5.1, 0.8 Hz, 1*H*), 8.02 – 7.86 (m, 4*H*), 7.46 – 7.28 (m, 2*H*), 2.41 (s, 3*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.58, 149.81, 149.06, 145.92, 135.06, 131.37, 125.03, 123.71, 123.63, 20.46.

MS (EI, 70 eV): *m/z* (%) = 238 ([M]$^+$, 65), 210 (100), 193 (12), 104 (14), 76 (20).

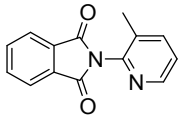


2-(4-Methylpyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.47 (d, *J* = 2.4 Hz, 1*H*), 8.02 – 7.90 (m, 4*H*), 7.85 (ddd, *J* = 7.9, 2.6, 0.9 Hz, 1*H*), 7.44 (d, *J* = 8.0 Hz, 1*H*), 2.38 (s, 3*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.58, 149.81, 149.06, 145.92, 135.06, 131.37, 125.03, 123.71, 123.63, 20.46.

MS (EI, 70 eV): *m/z* (%) = 238 ([M]$^+$, 41), 210 (100), 193 (4), 181 (7), 104 (14), 76 (23).

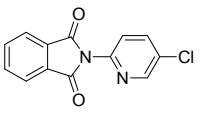


2-(3-Methylpyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.48 (dd, *J* = 5.0, 1.8 Hz, 1*H*), 8.10 – 7.86 (m, 5*H*), 7.50 (dd, *J* = 7.7, 4.7 Hz, 1*H*), 2.20 (s, 3*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.46, 147.28, 144.81, 140.14, 135.17, 132.37, 131.31, 124.97, 123.88, 16.59.

MS (EI, 70 eV): *m/z* (%) = 238 ([M]$^+$, 100), 209 (21), 194 (29), 181 (19).

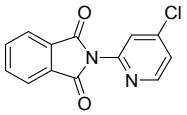


2-(5-Chloropyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.72 (s, 1*H*), 8.20 (ddd, *J* = 8.5, 2.7, 1.0 Hz, 1*H*), 8.03 – 7.86 (m, 4*H*), 7.72 – 7.57 (m, 1*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.19, 147.95, 144.45, 138.49, 135.08, 131.31, 131.07, 124.13, 123.77.

MS (EI, 70 eV): *m/z* (%) = 258 ([M]$^+$, 65), 230 (100), 179 (14), 104 (23), 76 (36).

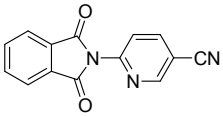


2-(4-Chloropyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.65 (d, *J* = 5.4 Hz, 1*H*), 8.06 – 7.98 (m, 2*H*), 7.96 – 7.91 (m, 2*H*), 7.81 – 7.74 (m, 1*H*), 7.70 (dd, *J* = 5.4, 2.0 Hz, 1*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.18, 150.69, 147.12, 144.27, 135.24, 131.30, 124.27, 123.90, 123.03.

MS (EI, 70 eV): *m/z* (%) = 258 ([M]$^+$, 57), 230 (100), 202 (9), 179 (8), 104 (16), 76 (21).

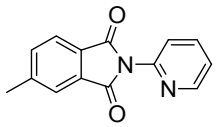


6-(1,3-Dioxoisoindolin-2-yl)nicotinonitrile

^{1}H NMR (300 MHz, DMSO-d_6) δ 9.12 (dd, *J* = 2.3, 0.8 Hz, 1*H*), 8.56 (dd, *J* = 8.4, 2.3 Hz, 1*H*), 8.06 – 8.00 (m, 2*H*), 7.96 – 7.91 (m, 2*H*), 7.78 (dd, *J* = 8.4, 0.8 Hz, 1*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 165.78, 152.60, 148.84, 142.49, 135.31, 131.26, 123.98, 122.42, 116.65, 108.65.

MS (EI, 70 eV): *m/z* (%) = 249 ([M]$^+$, 43), 221 (100), 204 (8), 193 (9), 104 (17), 76 (27).



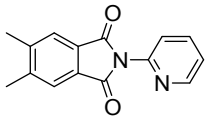
5-Methyl-2-(pyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.68 – 8.59 (m, 1*H*), 8.04 (dddd, *J* = 7.9, 7.4, 1.9, 0.4 Hz, 1*H*), 7.98 – 7.81 (m, 2*H*), 7.74 (ddt, *J* = 7.6, 1.3, 0.6 Hz, 1*H*), 7.60 – 7.43 (m, 2*H*), 2.59 – 2.34 (m, 3*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.55, 166.44, 149.37, 146.05, 145.94, 138.65, 135.40, 131.71, 128.74, 124.06, 124.03, 123.61, 123.06, 21.42.

MS (EI, 70 eV): *m/z* (%) = 238 ([M]$^+$, 46), 210 (100), 193 (11), 181 (12), 118 (20), 89 (32).

HRMS (EI) (m/z): [M+H]$^+$ calcd. for: $C_{14}H_{10}O_2N_2$ 238.07368, found 238.07313.



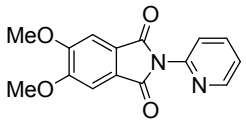
5, 6-Dimethyl-2-(pyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.63 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1*H*), 8.03 (td, *J* = 7.7, 1.9 Hz, 1*H*), 7.80 (s, 2*H*), 7.59 – 7.43 (m, 2*H*), 2.43 (s, 6*H*)

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.64, 149.34, 146.01, 144.68, 138.61, 129.27, 124.36, 123.94, 123.04, 20.04.

MS (EI, 70 eV): *m/z* (%) = 252 ([M]$^+$, 48), 224 (100), 207 (10), 132 (12),103 (18), 78 (17).

HRMS (EI) (m/z): [M+H]$^+$ calcd. for: $C_{15}H_{12}O_2N_2$ 252.08933, found 252.08906.



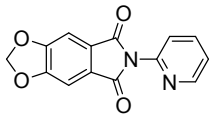
5,6-Dimethoxy-2-(pyridin-2-yl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 7.80 (ddd, *J* = 4.9, 1.9, 0.9 Hz, 1*H*), 7.20 (ddd, *J* = 8.0, 7.5, 1.9 Hz, 1*H*), 6.78 – 6.59 (m, 4*H*), 3.14 (s, 6*H*).

^{13}C NMR (75 MHz, DMSO-d_6) 166.44, 154.12, 149.33, 146.09, 138.60, 124.51, 123.81, 122.97, 105.99, 56.52.

MS (EI, 70 eV): *m/z* (%) = 284 ([M]$^+$, 100), 256 (89), 239 (15), 136 (24).

HRMS (EI) (m/z): $[M+H]^+$ calcd. for: $C_{15}H_{12}O_4N_2$ 284.07916, found 284. 07862.



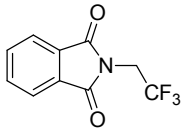
6-(Pyridin-2-yl)-5*H*-[1,3]dioxolo[4,5-*f*]isoindole-5,7(6*H*)-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 8.62 (ddd, *J* = 4.8, 1.9, 1.0 Hz, 1*H*), 8.03 (ddd, *J* = 8.0, 7.5, 2.0 Hz, 2*H*), 7.51 (d, *J* = 1.7 Hz, 3*H*), 6.33 (s, 2*H*).

^{13}C NMR (75 MHz, DMSO-d_6) 165.85, 153.04, 149.34, 145.90, 138.63, 126.80, 123.91, 122.93, 103.84, 103.61.

MS (EI, 70 eV): *m/z* (%) = 268 ($[M]^+$, 80), 240 (100), 223 (13), 120 (24).

HRMS (EI) (m/z): $[M+H]^+$ calcd. for: $C_{14}H_8O_4N_2$ 268,04786, found 268. 04761.

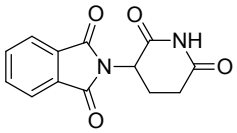


2-(2,2,2-Trifluoroethyl)isoindoline-1,3-dione

^{1}H NMR (300 MHz, DMSO-d_6) δ 7.93 – 7.91 (m, 1*H*), 7.90 (d, *J* = 0.5 Hz, 1*H*), 7.86 (d, *J* = 0.5 Hz, 1*H*), 7.85 – 7.83 (m, 1*H*), 4.37 (d, *J* = 9.4 Hz, 2*H*).

^{13}C NMR (75 MHz, DMSO-d_6) δ 166.65 , 134.98 , 131.08 , 125.51 , 124.48 , 121.80 .

MS (EI, 70 eV): *m/z* (%) = 229 ($[M]^+$, 20), 210 (4), 160 (100), 133 (7), 104 (10), 76 (19).

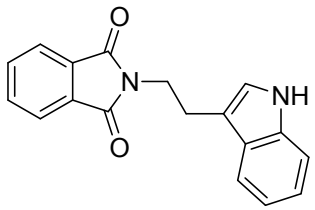


Thalidomide

^{1}H NMR (300 MHz, DMSO-d_6) δ 11.15 (s, 1*H*), 7.92 (dq, *J* = 5.8, 2.1 Hz, 3*H*), 5.17 (dd, *J* = 12.9, 5.4 Hz, 1*H*), 2.65 – 2.41 (m, 4*H*), 2.16 – 1.90 (m, 1*H*).

^{13}C NMR (75 MHz, DMSO-d_6) 172.73, 169.82, 167.12, 134.85, 131.19, 131.05, 123.38, 48.94, 40.31, 38.58, 30.90, 21.94.

MS (EI, 70 eV): *m/z* (%) = 258 ($[M]^+$, 25), 230 (24), 213 (12), 202 (22), 187 (17), 173 (100), 160 (7), 148 (45), 113 (23), 117 (7), 104 (45), 76 (42).

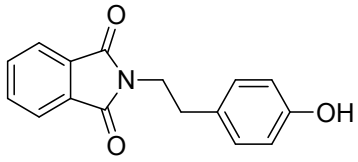


2-(2-(1*H*-Indol-3-yl)ethyl)isoindoline-1,3-dione

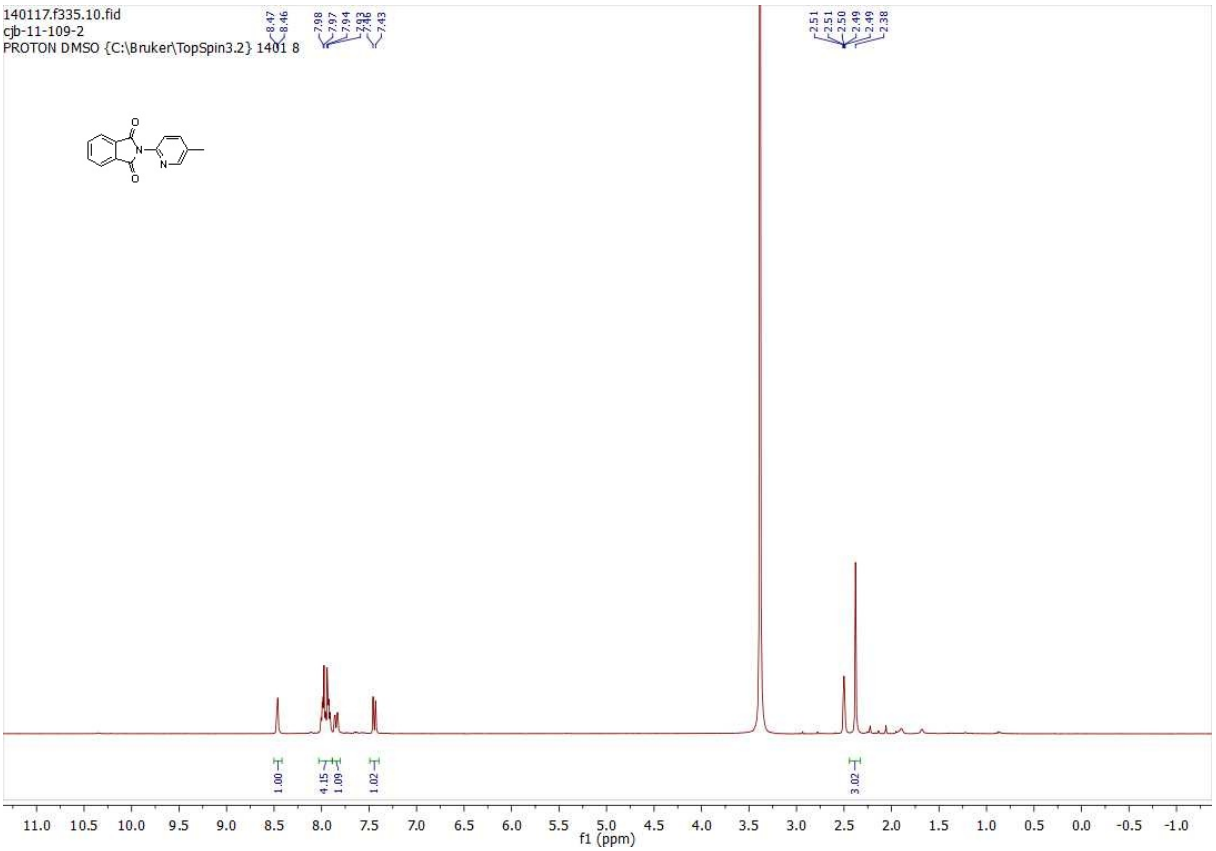
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 3.1 Hz, 1*H*), 7.87 – 7.84 (m, 1H), 7.78 (ddt, *J* = 7.7, 1.6, 0.8 Hz, 1*H*), 7.74 – 7.70 (m, 2*H*), 7.41 – 7.34 (m, 1*H*), 7.23 (dd, *J* = 7.0, 1.3 Hz, 2*H*), 7.18 – 7.11 (m, 2*H*), 4.14 – 3.91 (m, 2*H*), 3.25 – 3.11 (m, 2*H*).

^{13}C NMR (75 MHz, $CDCl_3$) 170.77, 168.40, 136.30, 133.90, 132.20, 127.39, 123.19, 122.17, 122.00, 119.39, 118.82, 112.21, 111.21, 77.53, 77.10, 76.68, 38.57, 24.53.

MS (EI, 70 eV): m/z (%) = 290 ([M]+, 35), 230 (24), 160 (8), 143 (20), 130 (100), 173 (100), 103 (7), 77 (11).



2-(4-Hydroxyphenethyl)isoindoline-1,3-dione
1H NMR (300 MHz, DMSO-*d*$_6$) δ 7.81 – 7.71 (m, 4*H*), 6.90 (d, J = 8.4 Hz, 2*H*), 6.56 (d, J = 8.4 Hz, 2*H*), 5.31 (s, 1*H*), 3.79 – 3.56 (m, 2*H*), 2.73 (t, J = 7.3 Hz, 2*H*).
^{13}C NMR (75 MHz, DMSO-*d*$_6$) 167.63, 155.74, 134.35, 131.43, 129.48, 128.15, 122.95, 115.15, 40. 28, 32.80.
MS (EI, 70 eV): m/z (%) = 267 ([M]+, 15), 160 (27), 120 (100), 107 (32), 77 (18), 51 (5).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3.02
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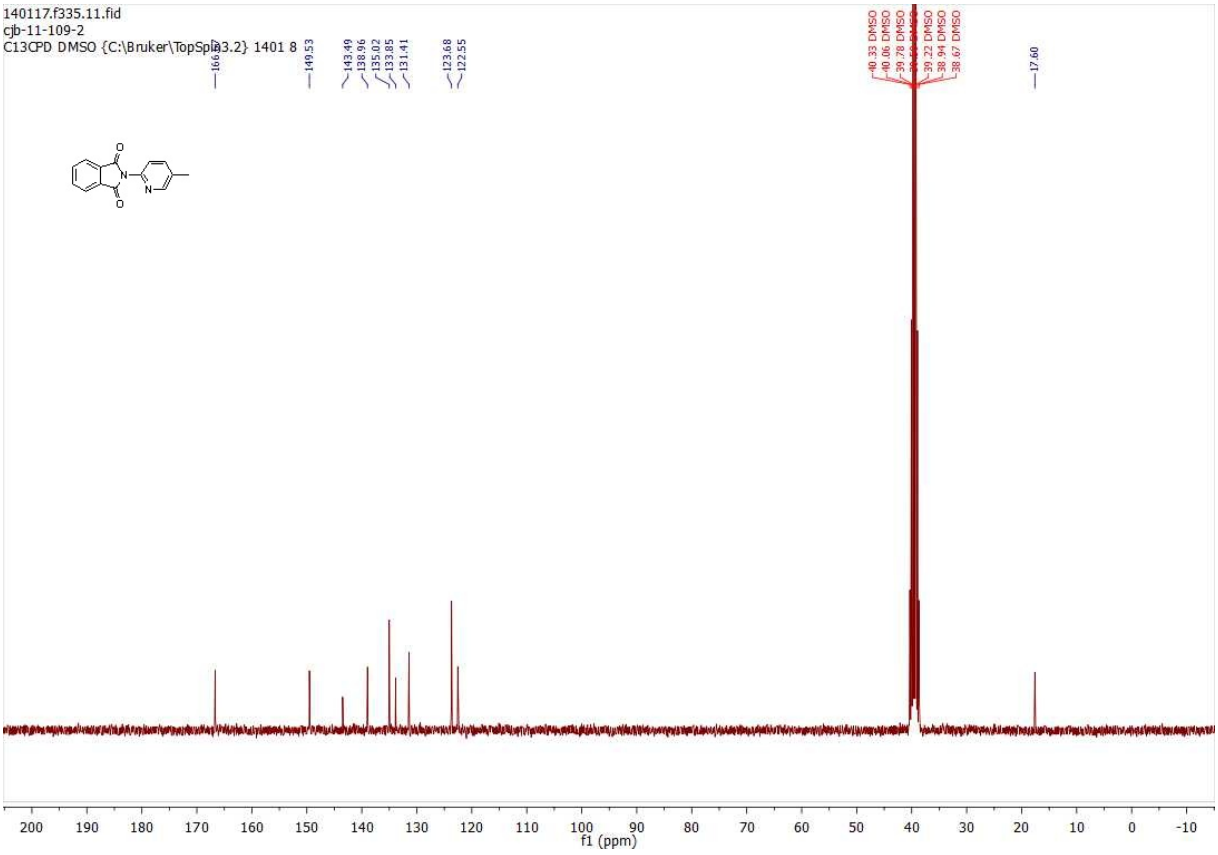
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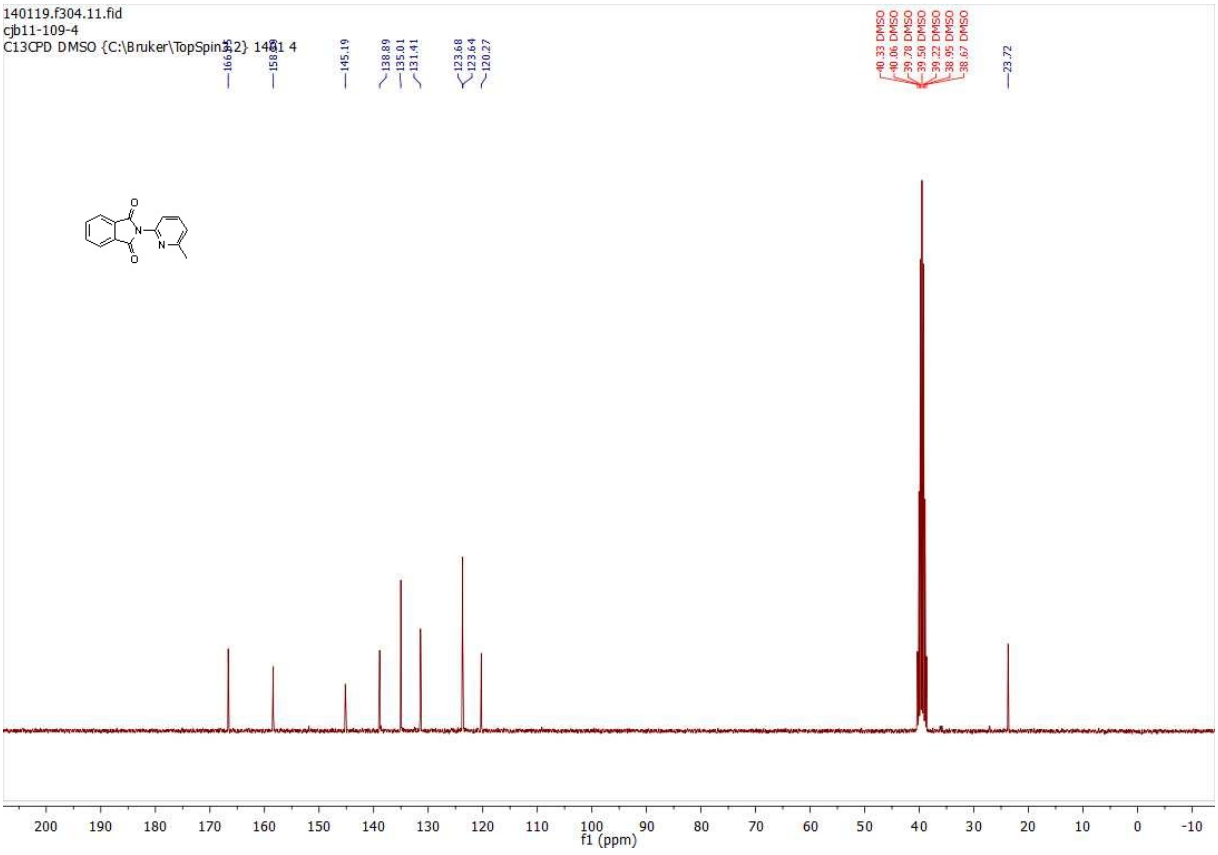
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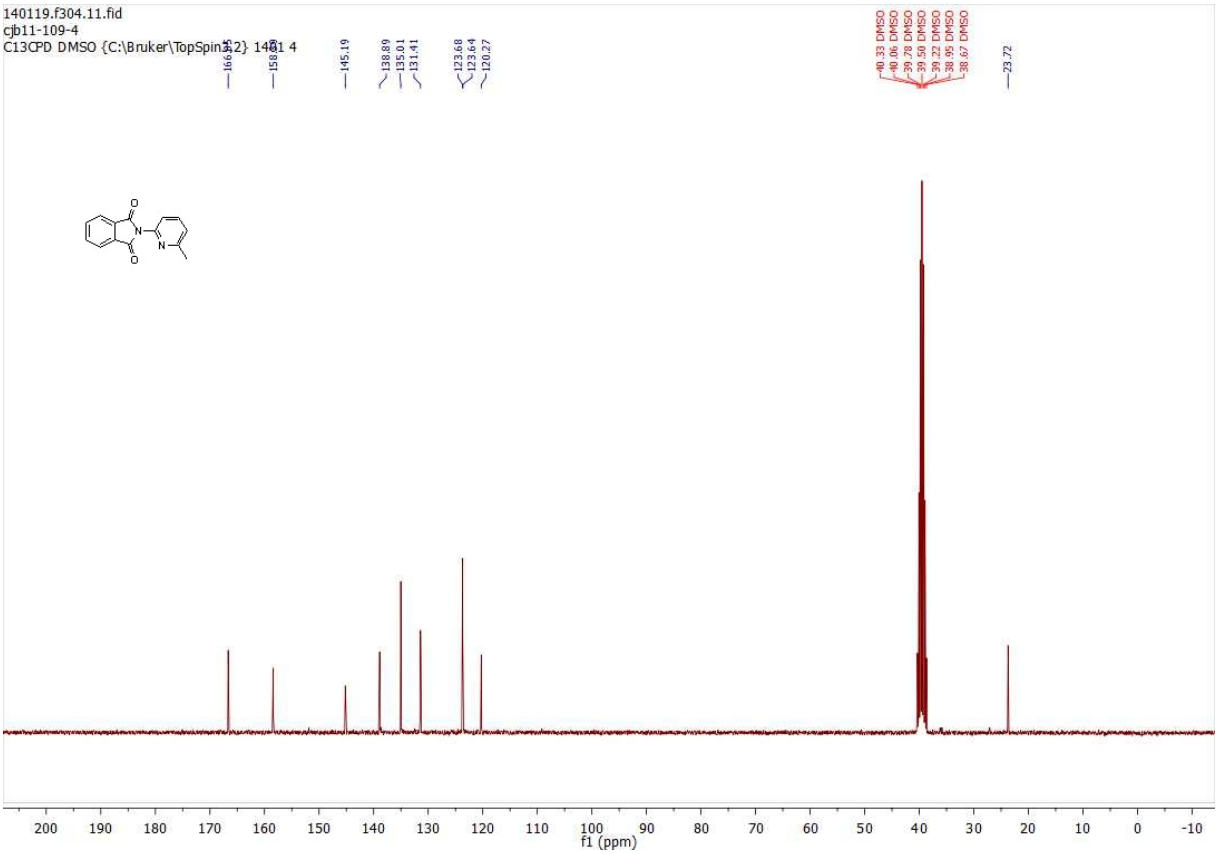
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40.06 DMSO
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39.50 DMSO
39.22 DMSO
38.95 DMSO
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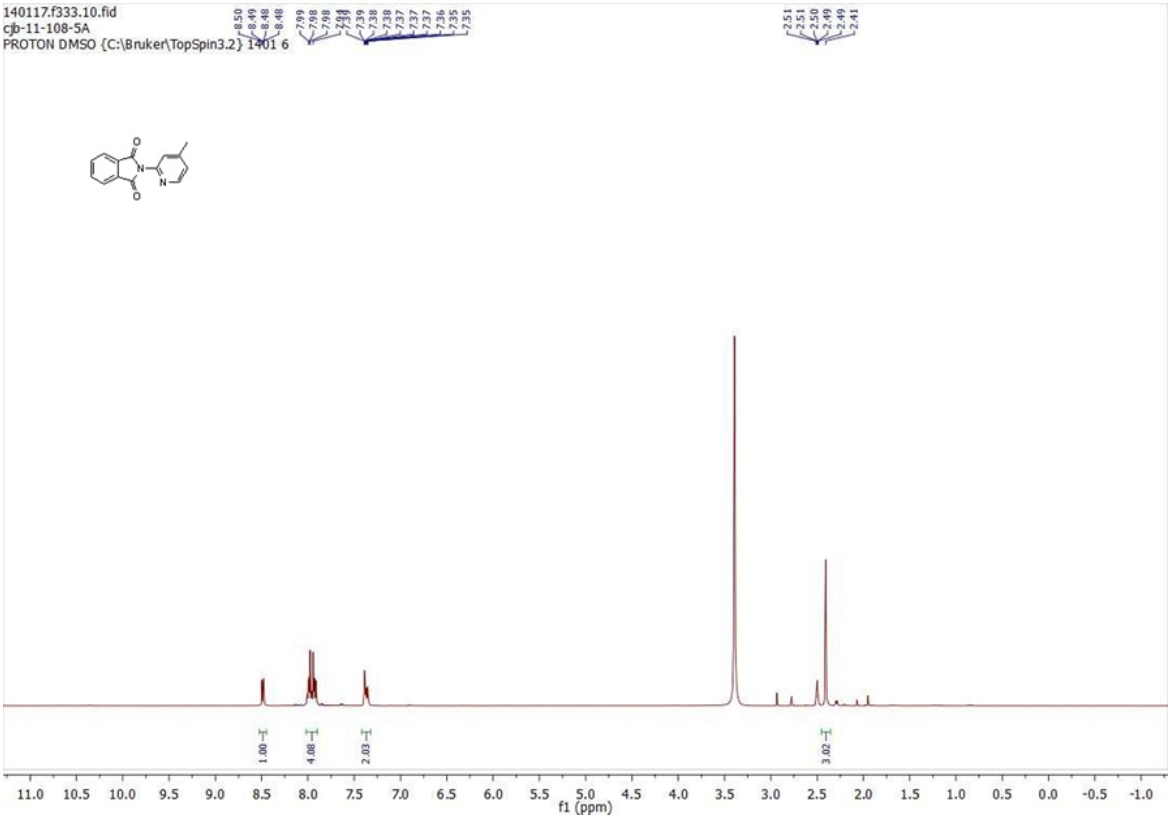
23.72
f1 (ppm)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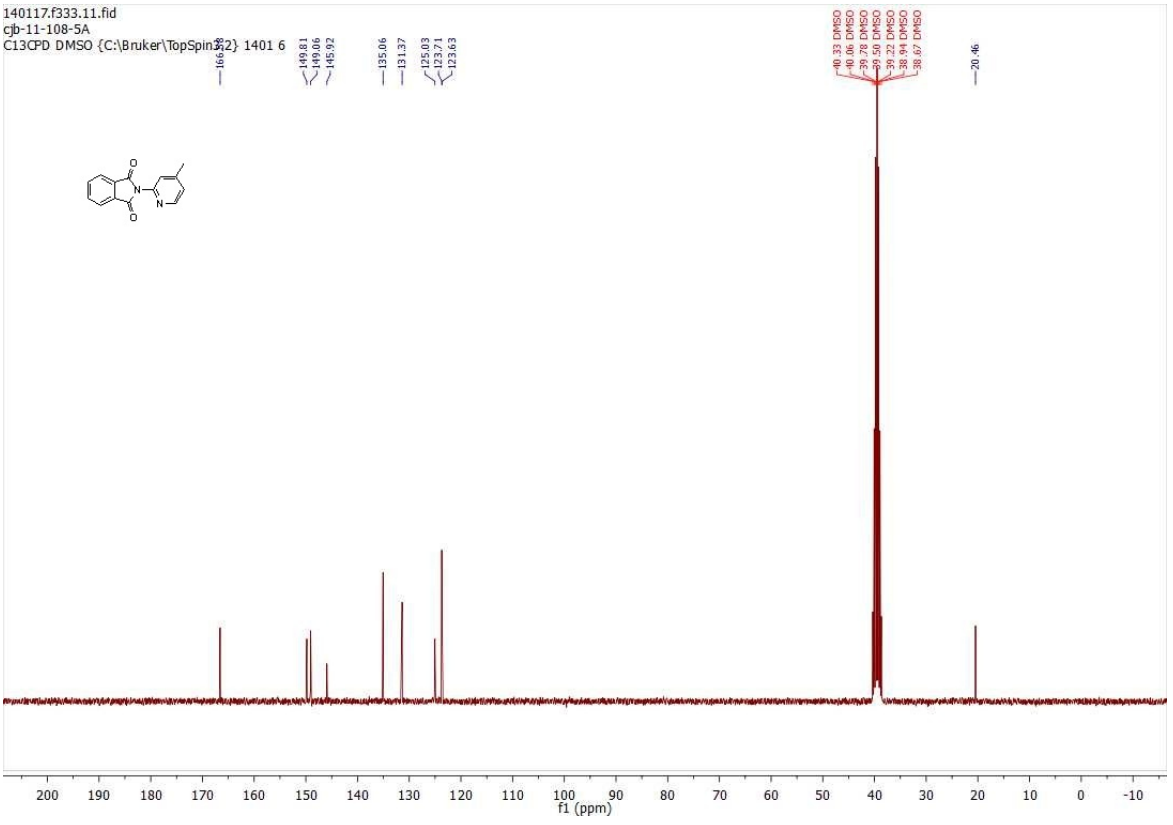
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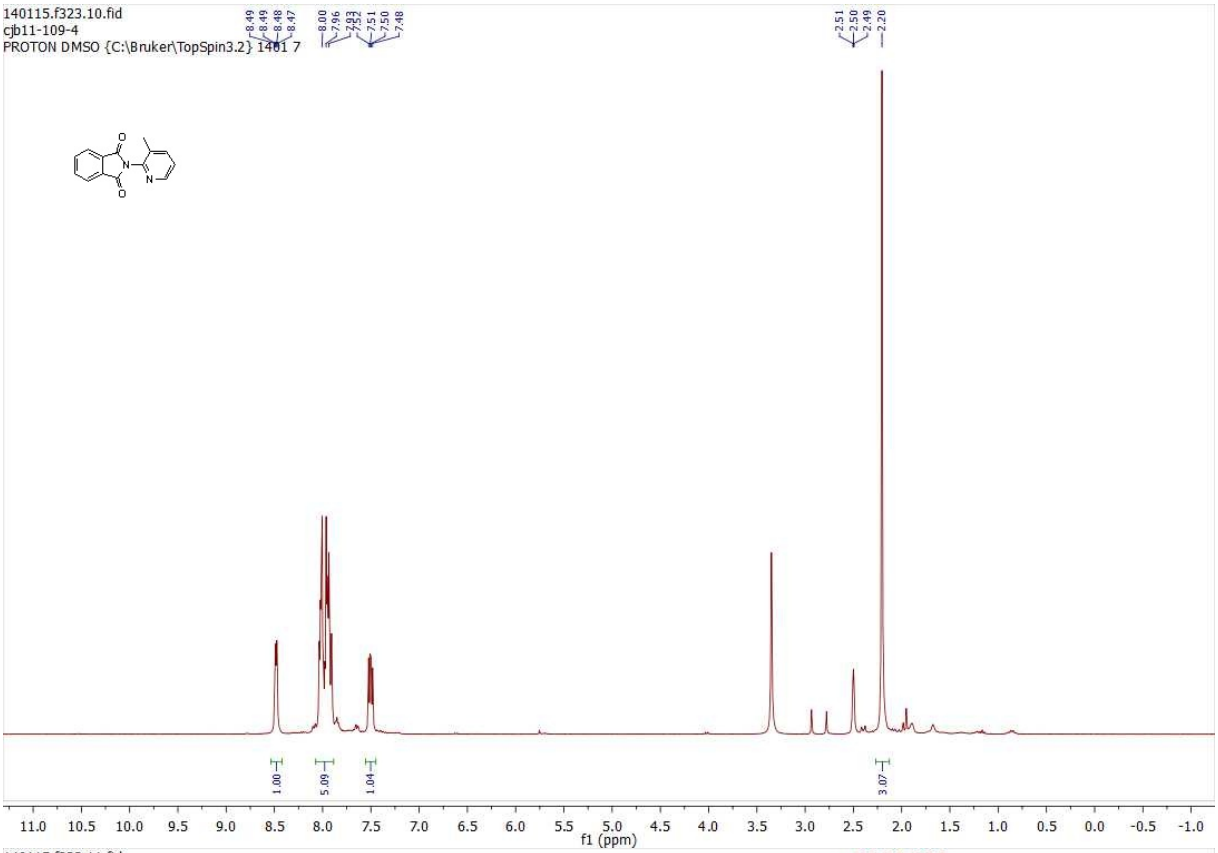
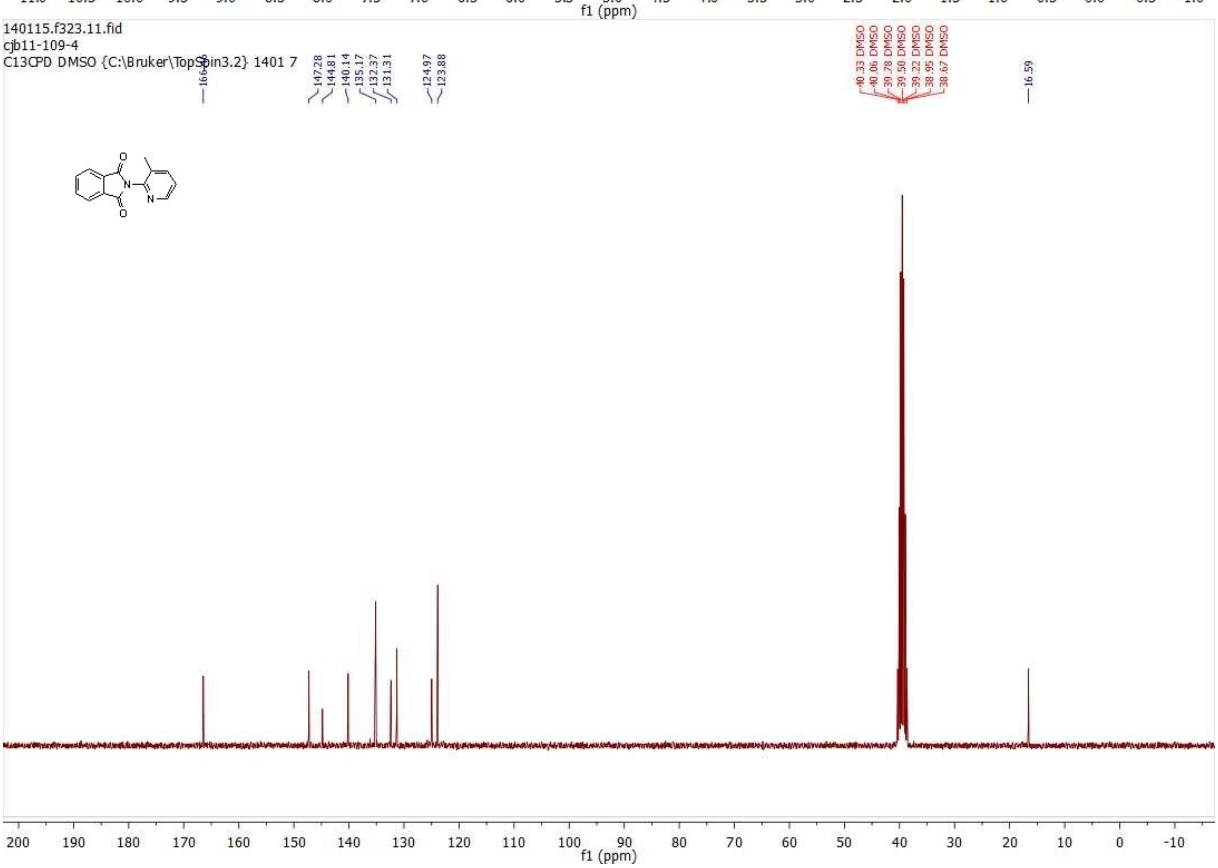
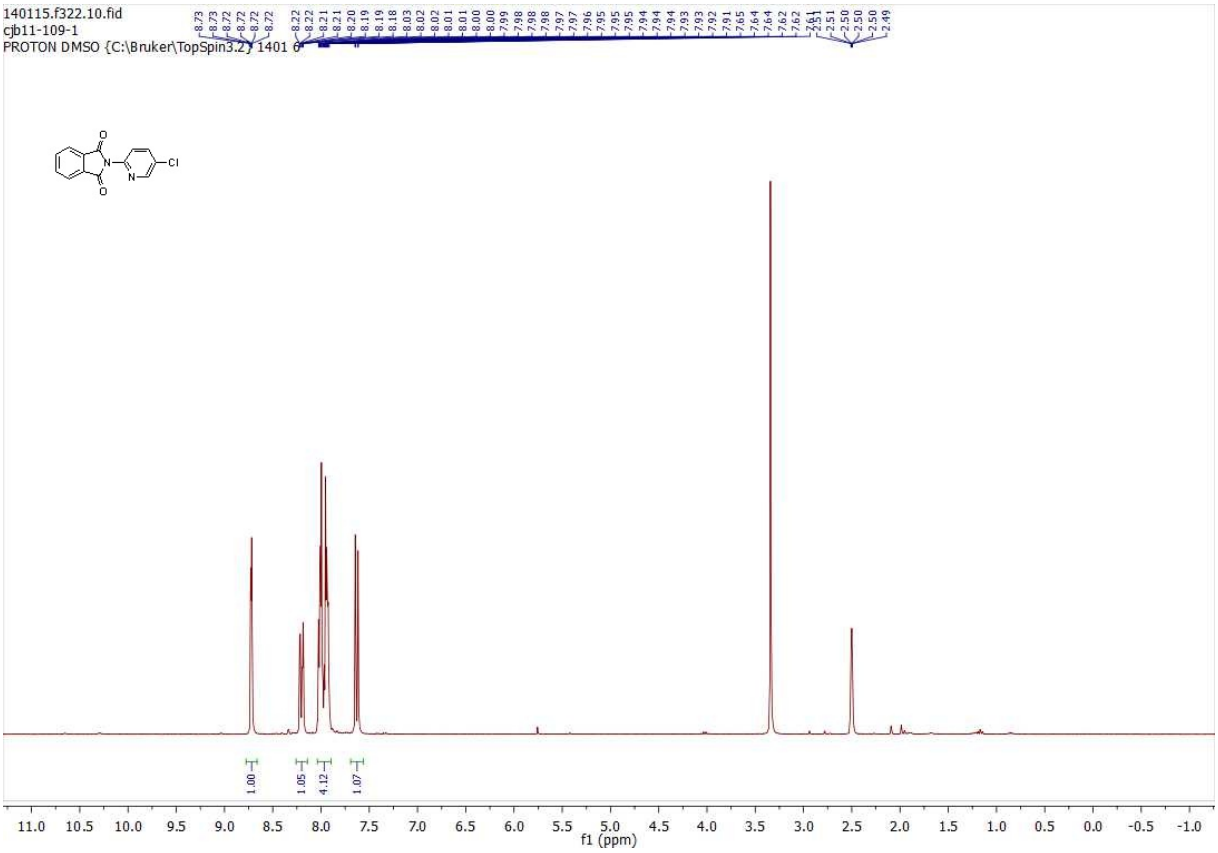
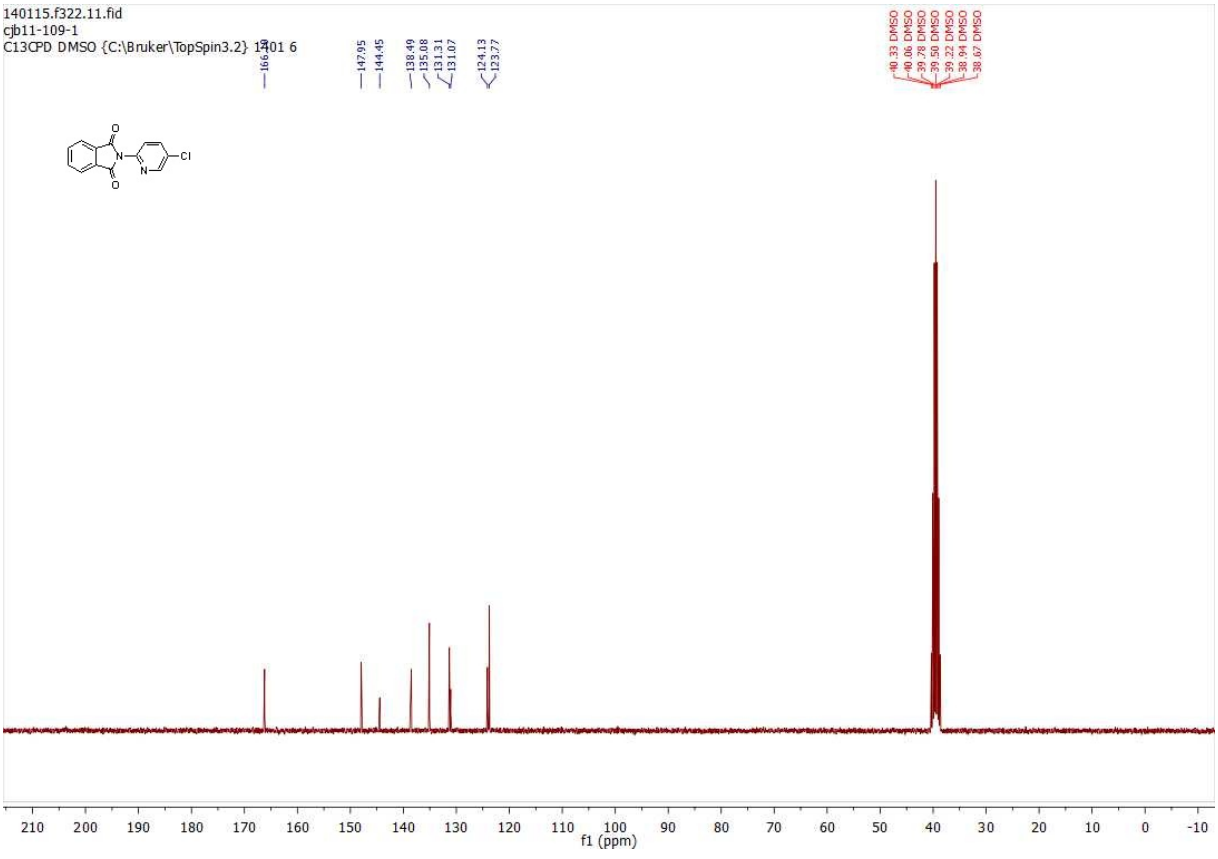
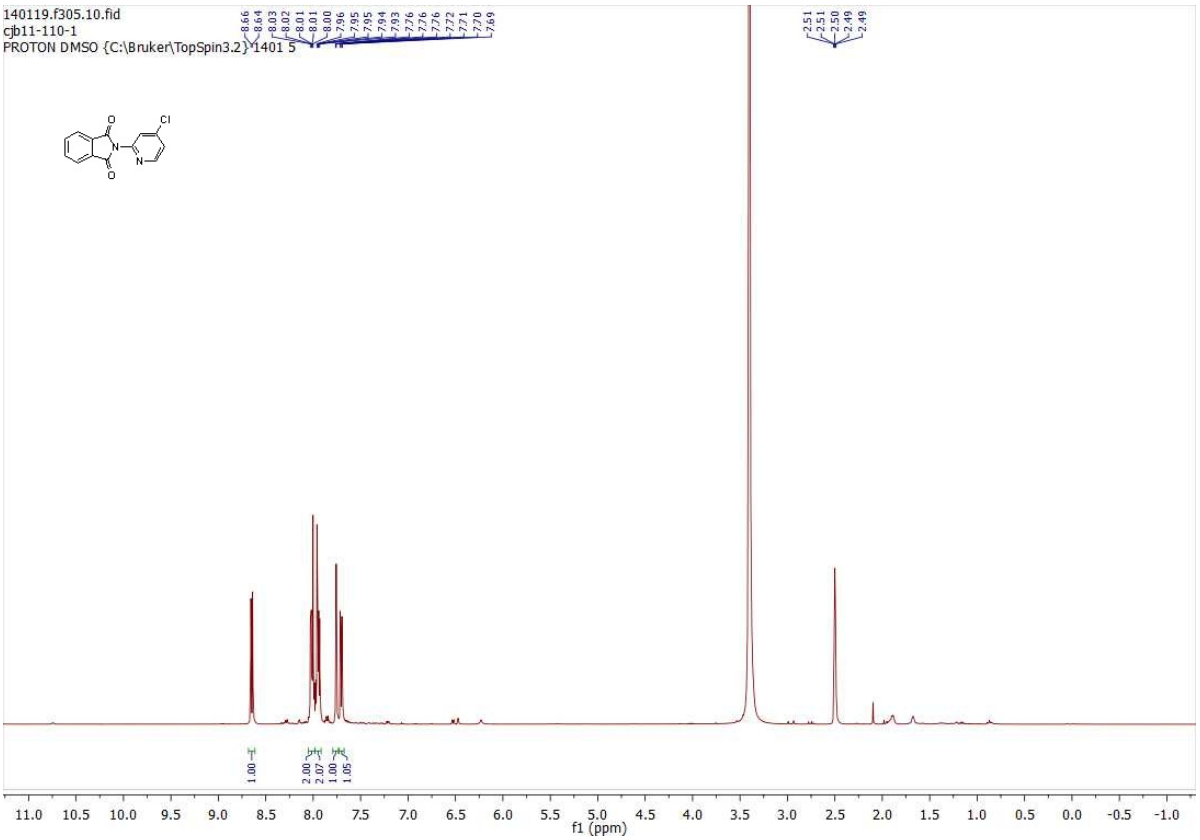
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 6
f1 (ppm)

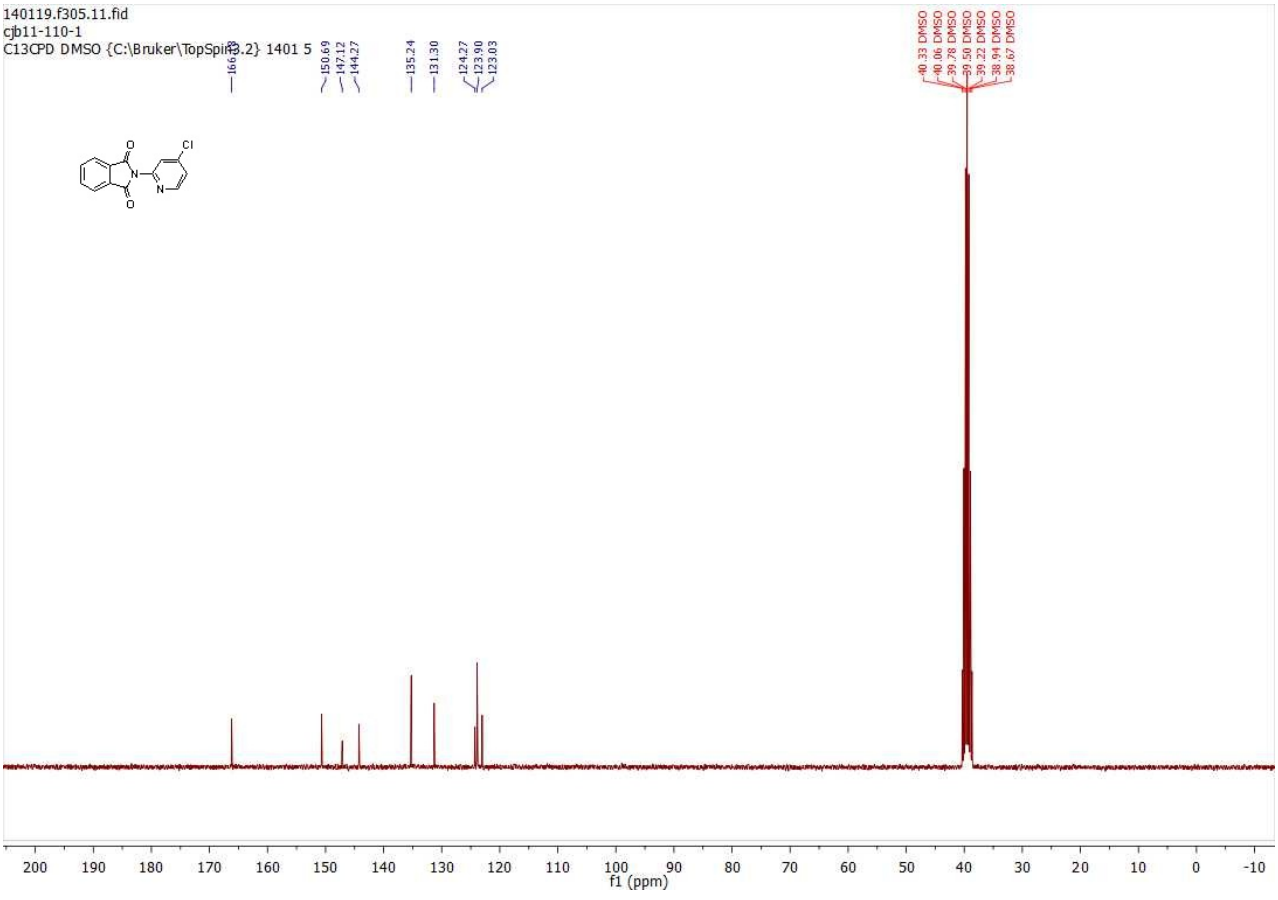

140115.f323.10.fid
cjb11-109-4
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 7
8.49
8.49
8.48
8.48
8.47
8.00
7.96
7.83
7.82
7.51
7.50
7.48
2.51
2.50
2.49
2.20
1.00
5.09
1.04
3.07
f1 (ppm)

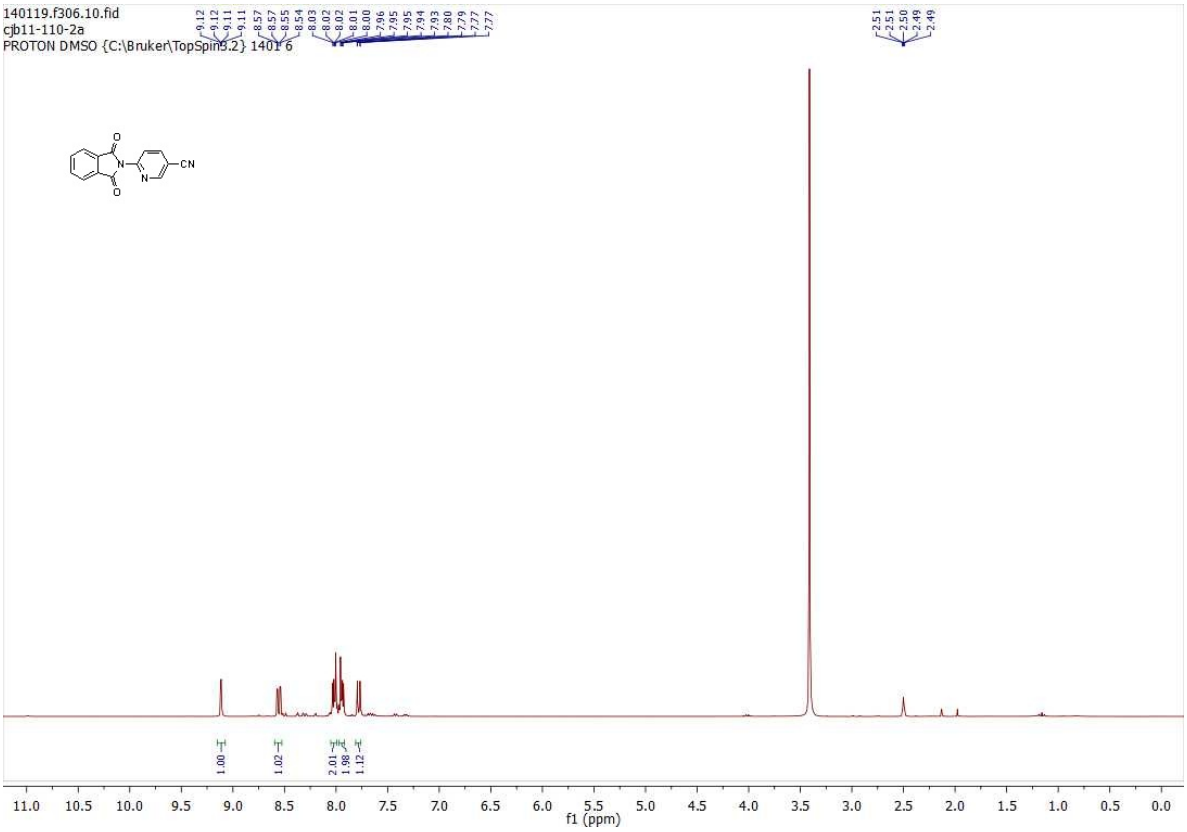

140115.f323.11.fid
cjb11-109-4
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 7
166.69
147.28
144.81
140.14
135.17
132.37
131.31
124.97
123.88
40.33 DMSO
40.06 DMSO
39.78 DMSO
39.50 DMSO
39.22 DMSO
38.95 DMSO
38.67 DMSO
16.59
f1 (ppm)

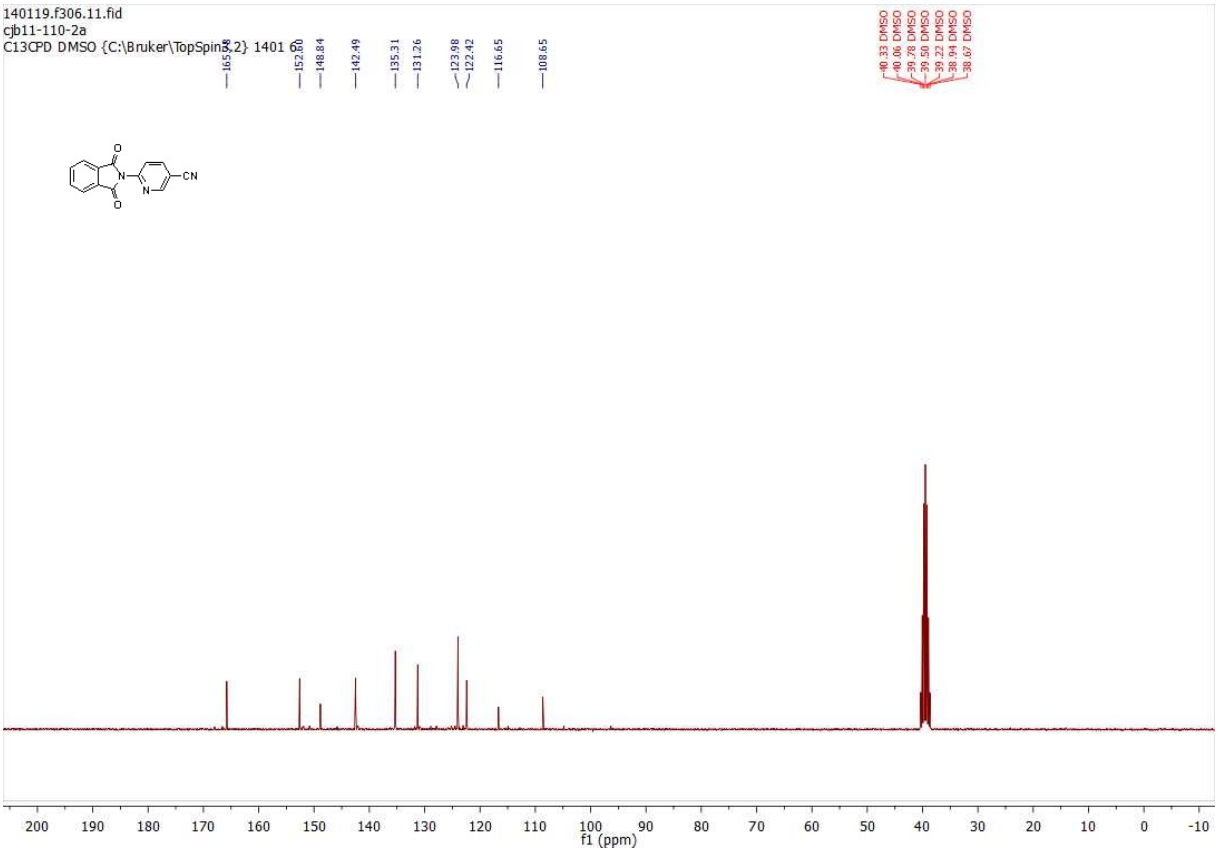

140115.f322.10.fid
cjb11-109-1
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 6
1.00
1.05
4.12
1.07
f1 (ppm)

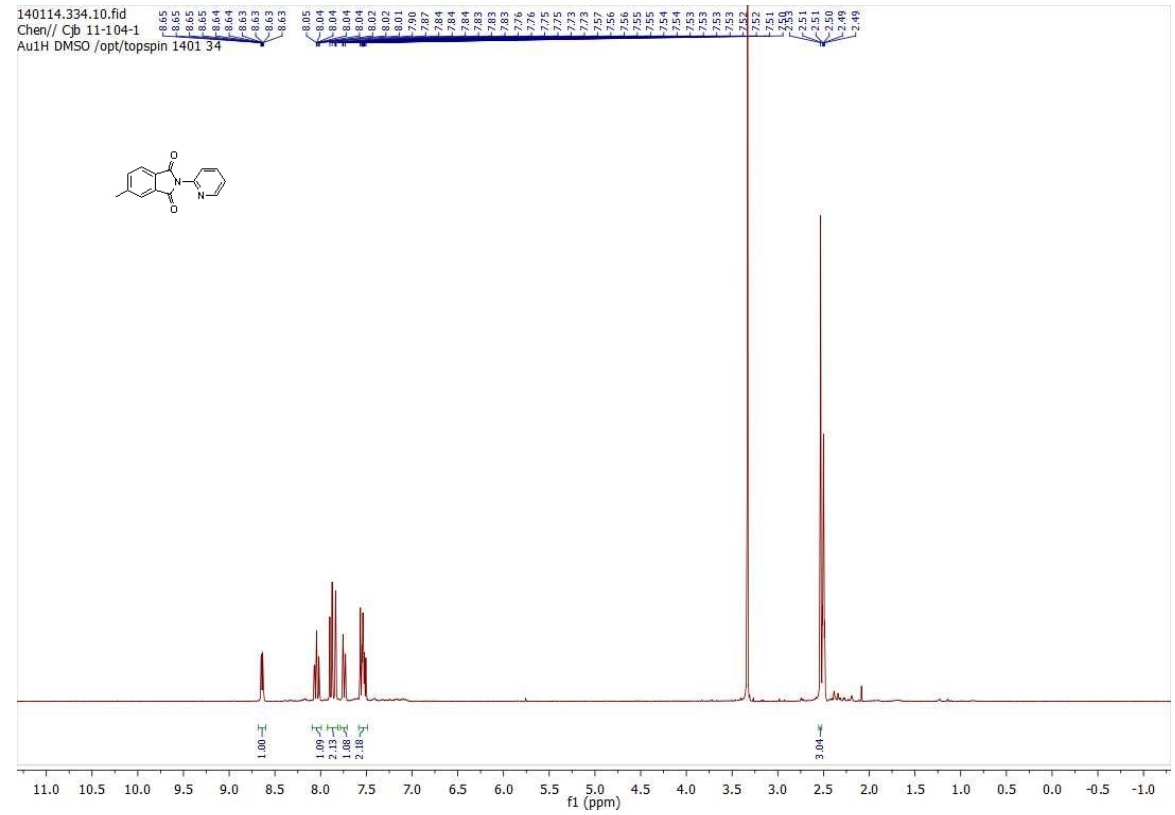

140115.f322.11.fid
cjb11-109-1
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 6
166.24
147.95
144.45
138.49
135.08
131.31
131.07
124.13
123.77
40.33 DMSO
40.06 DMSO
39.78 DMSO
39.50 DMSO
39.22 DMSO
38.94 DMSO
38.67 DMSO
f1 (ppm)

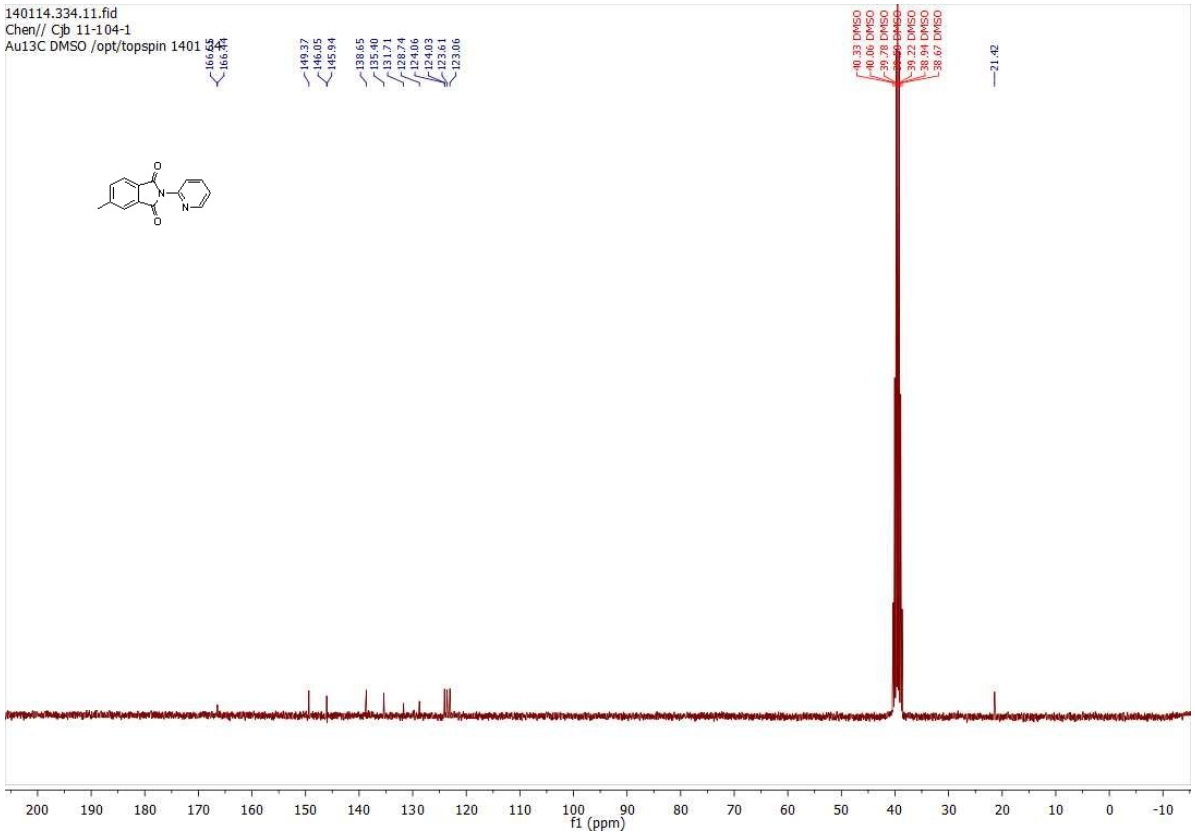

140119.f305.10.fid
cjb11-110-1
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 5
8.66
8.64
8.03
8.02
8.01
8.01
8.00
7.96
7.95
7.95
7.94
7.93
7.76
7.76
7.76
7.72
7.71
7.70
7.69
2.51
2.51
2.50
2.49
2.49
Cl
O
N
O
1.00
2.00
2.07
1.08
1.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

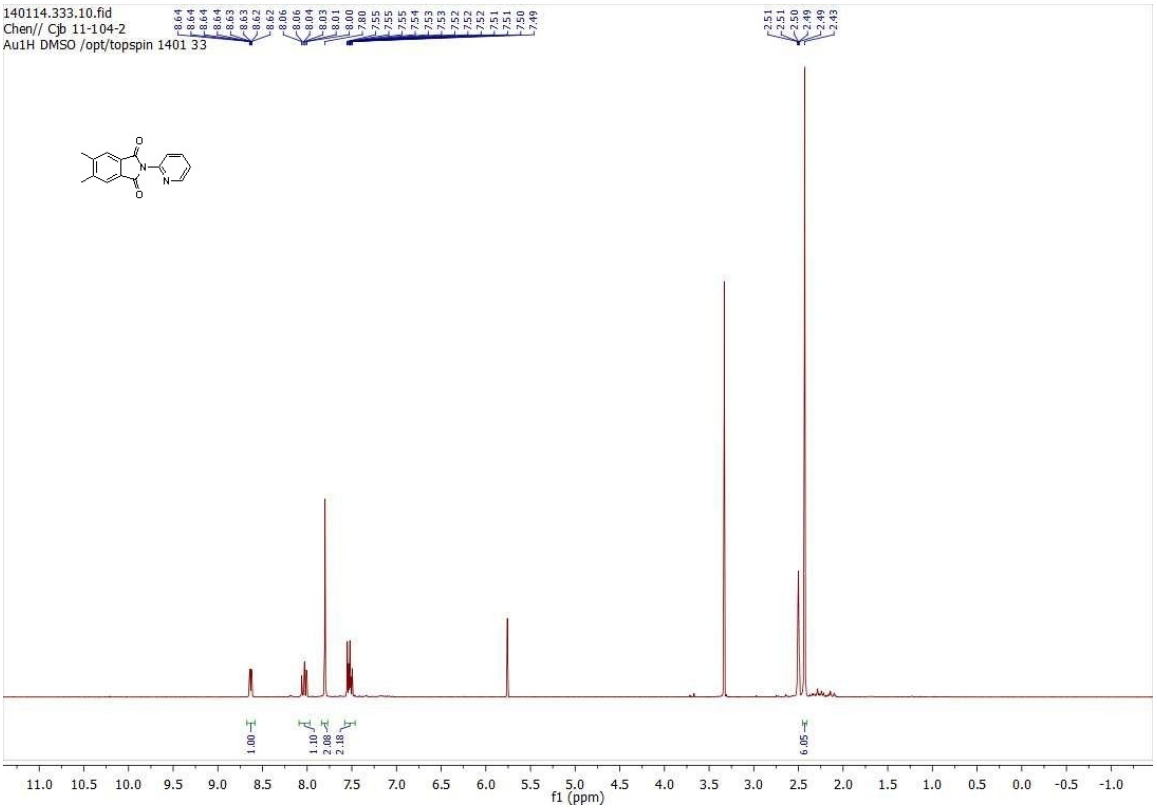

140119.f305.11.fid
cjb11-110-1
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 5
166.83
150.69
147.12
144.27
135.24
131.30
124.27
123.90
123.03
40.33 DMSO
40.06 DMSO
39.78 DMSO
39.50 DMSO
39.22 DMSO
38.94 DMSO
38.67 DMSO
Cl
O
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

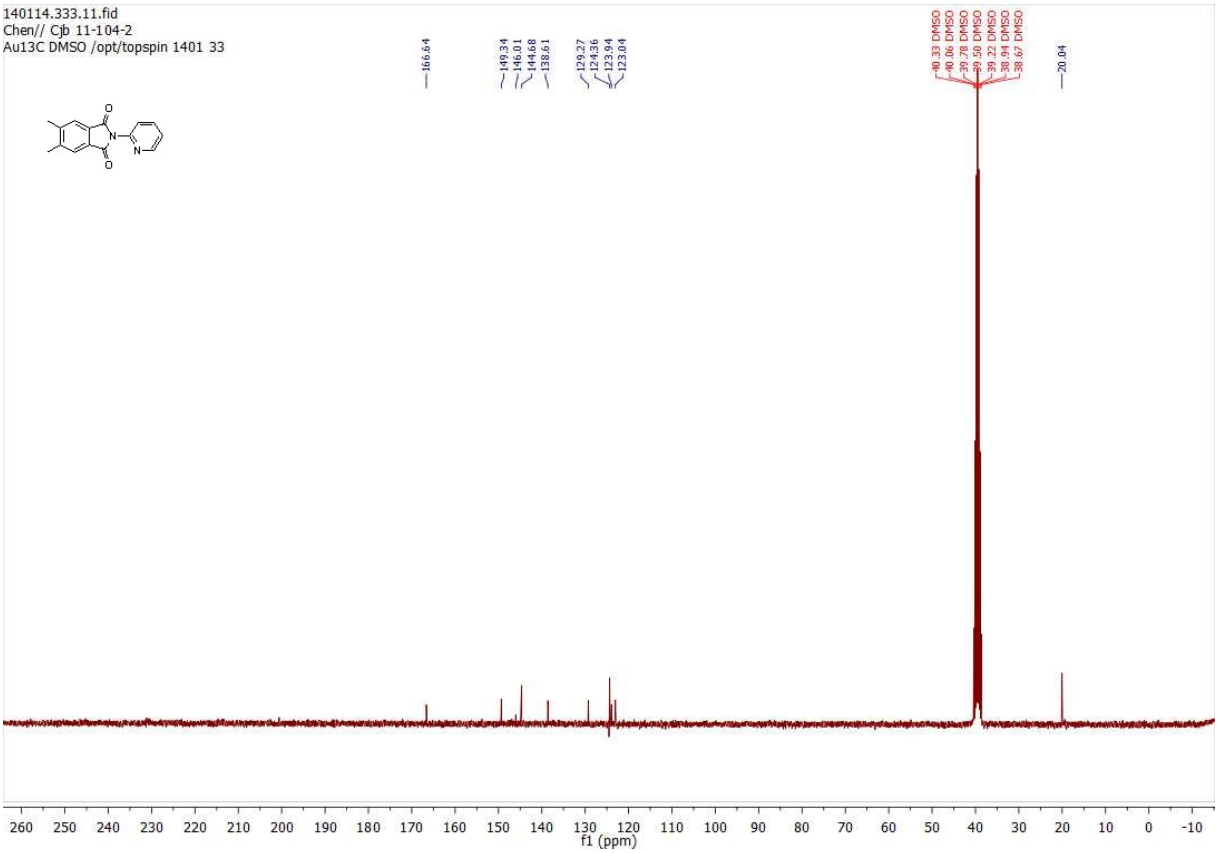

140119.f306.10.fid
cjb11-110-2a
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 6
9.12
9.12
9.11
9.11
8.57
8.57
8.55
8.54
8.03
8.02
8.02
8.01
8.00
7.96
7.95
7.94
7.93
7.80
7.79
7.77
7.77
2.51
2.51
2.50
2.49
2.49
O
N
CN
N
O
1.00
1.02
2.01
1.98
1.12
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

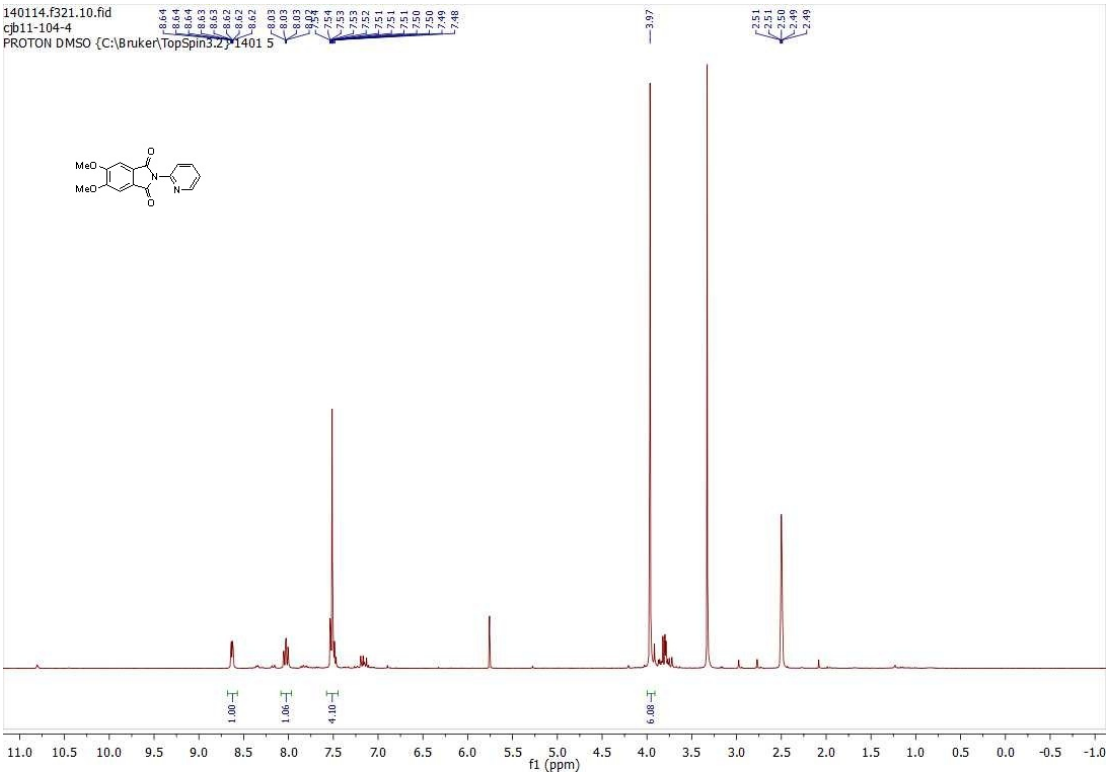

140119.f306.11.fid
cjb11-110-2a
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 60
165.48
152.60
148.84
142.49
135.31
131.26
123.98
122.42
116.65
108.65
40.33 DMSO
40.06 DMSO
39.78 DMSO
39.50 DMSO
39.22 DMSO
38.94 DMSO
38.67 DMSO
O
N
CN
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

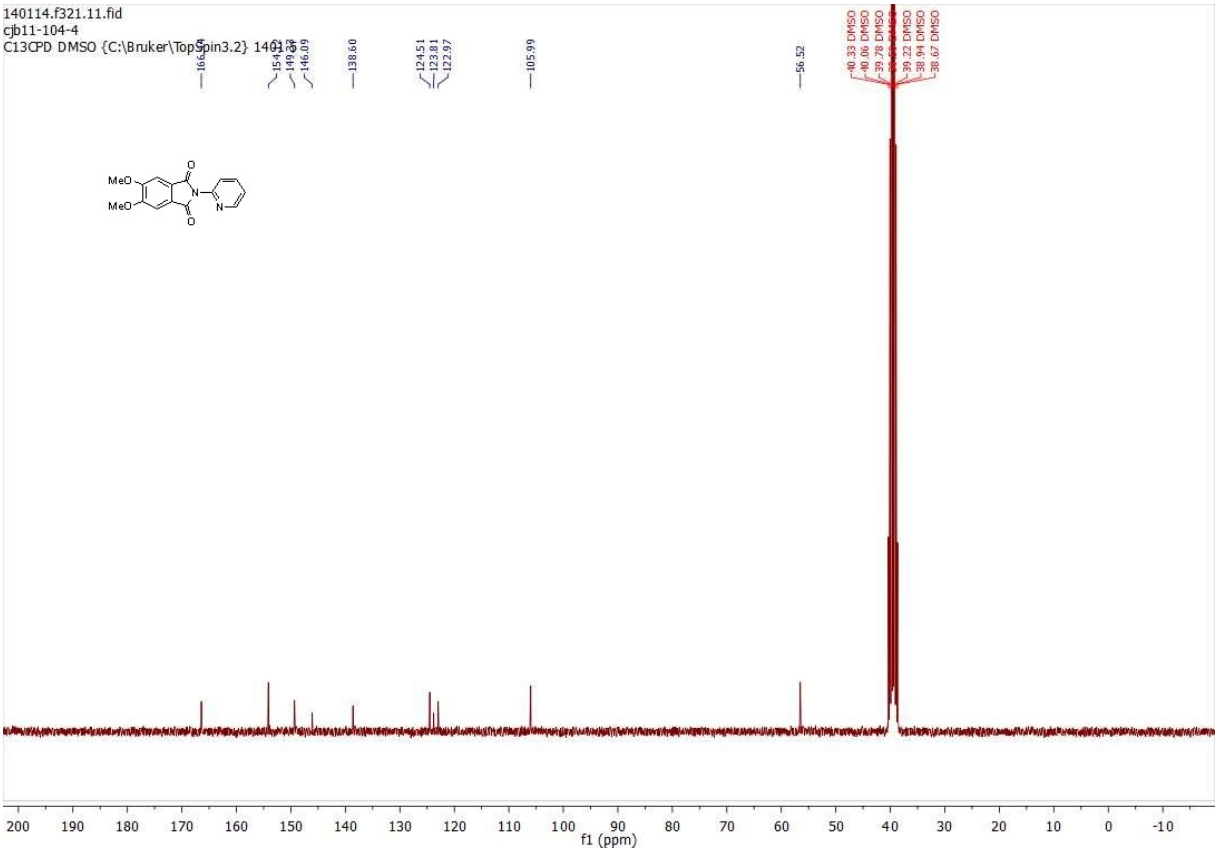

140114.334.10.fid
Chen// Cjb 11-104-1
Au1H DMSO /opt/topspin 1401 34
f1 (ppm)

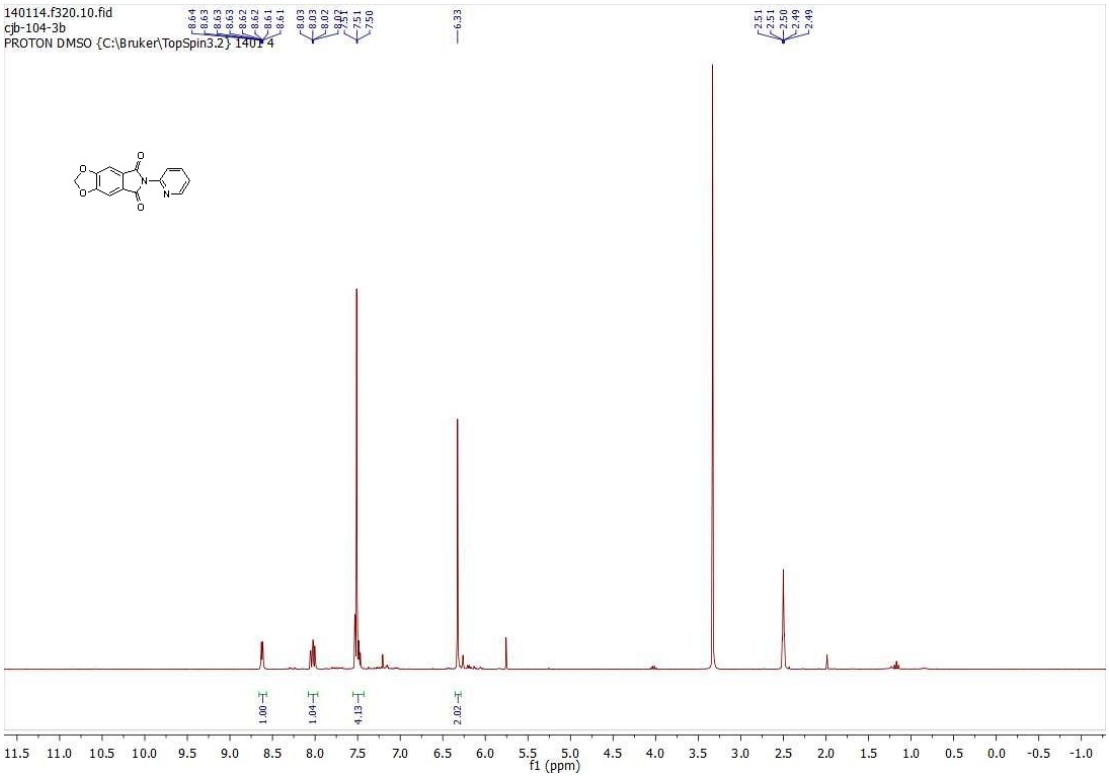

140114.334.11.fid
Chen// Cjb 11-104-1
Au13C DMSO /opt/topspin 1401 34
f1 (ppm)

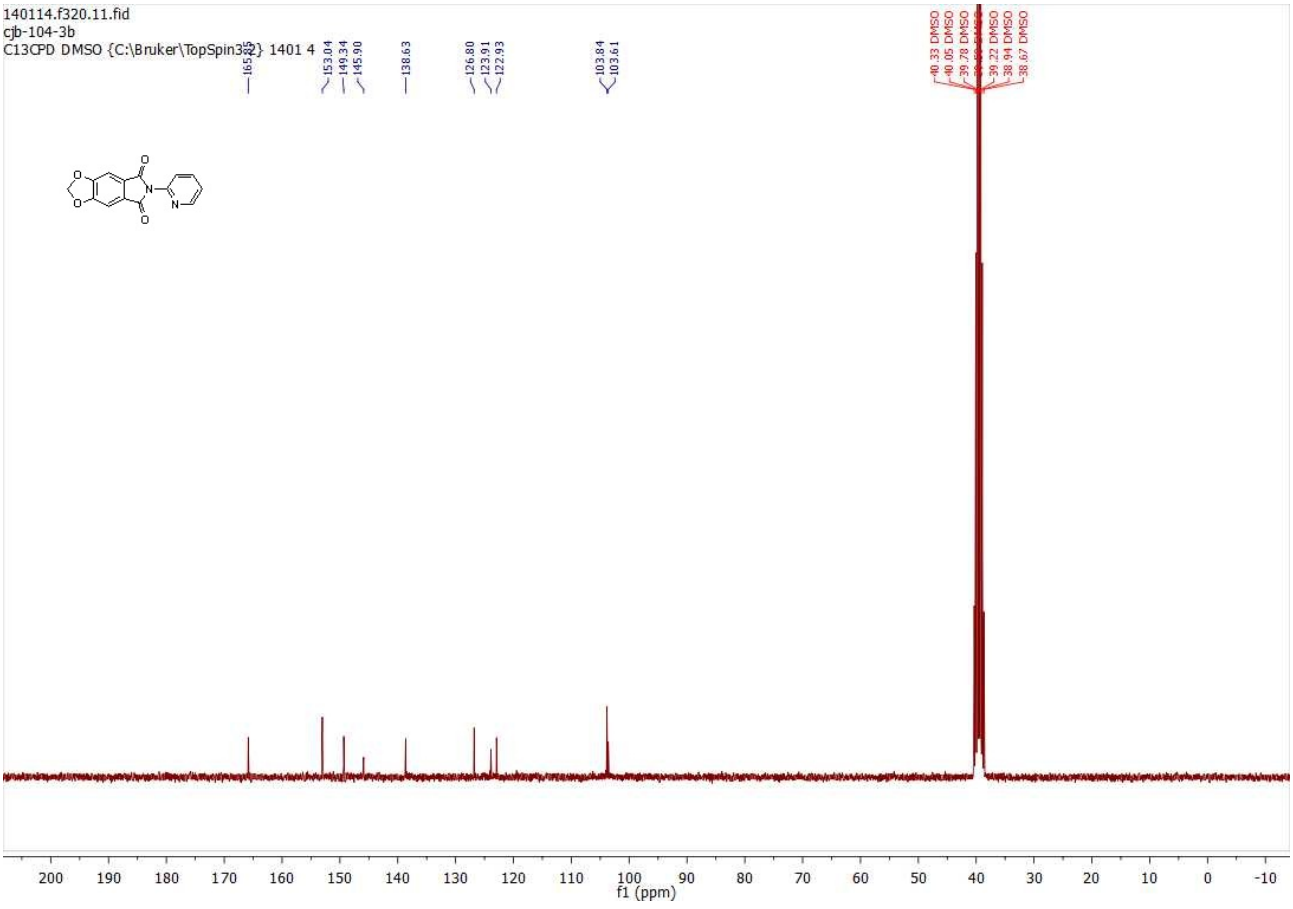

140114.333.10.fid
Chen// Cjb 11-104-2
Au1H DMSO /opt/topspin 1401 33
f1 (ppm)

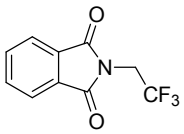

140114.333.11.fid
Chen// Cjb 11-104-2
Au13C DMSO /opt/topspin 1401 33
f1 (ppm)


140114.f321.10.fid
cjb11-104-4
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 5
MeO
MeO
O
N
O
f1 (ppm)

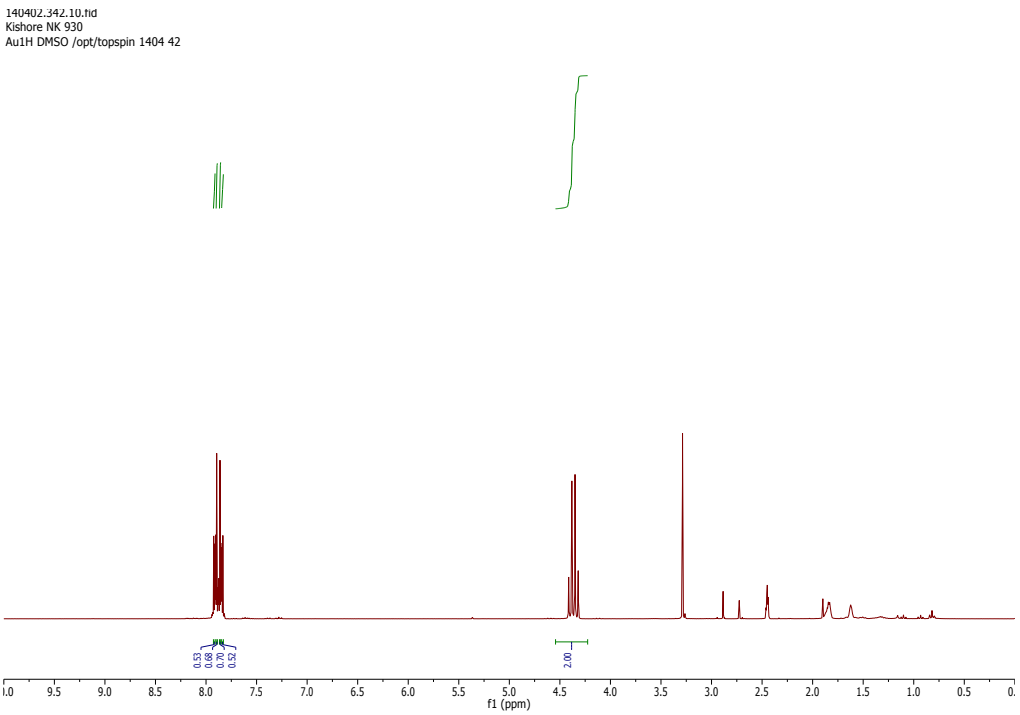

140114.f321.11.fid
cjb11-104-4
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 5
MeO
MeO
O
N
O
f1 (ppm)

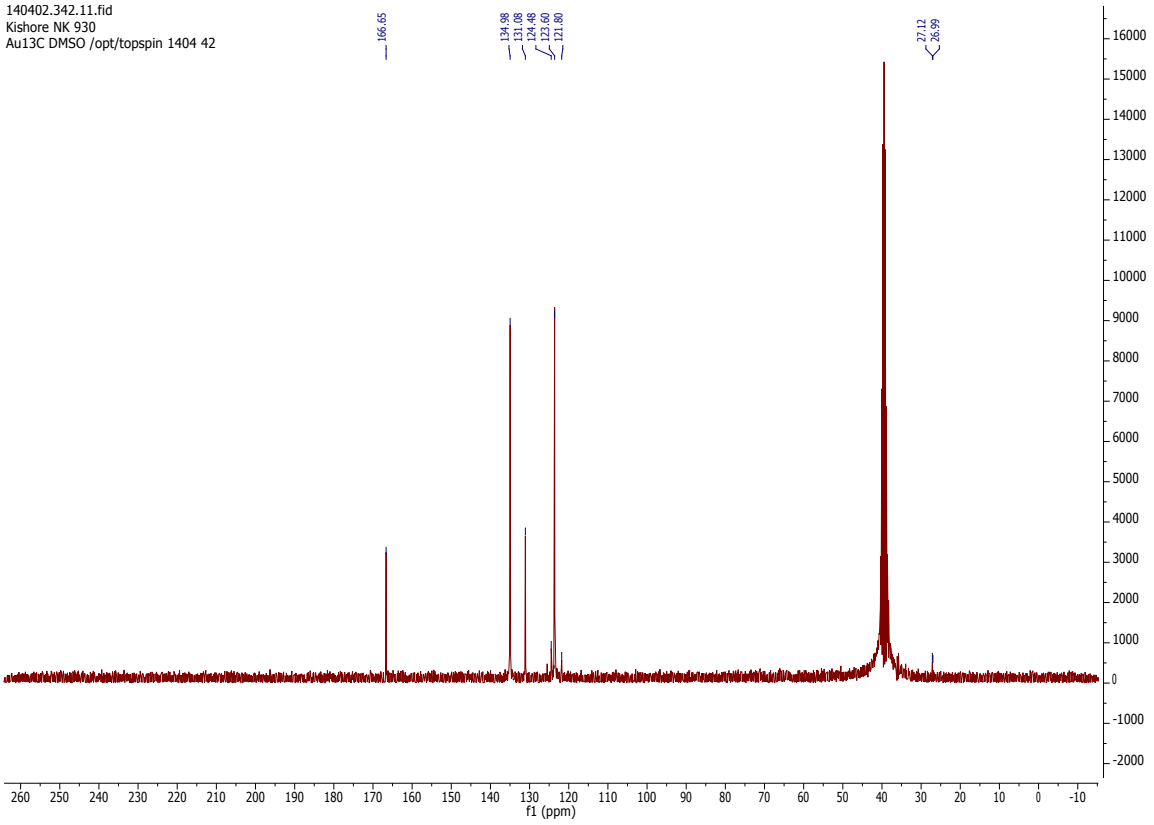

140114.f320.10.fid
cjb-104-3b
PROTON DMSO {C:\Bruker\TopSpin3.2} 1401 4
f1 (ppm)

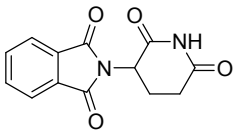

140114.f320.11.fid
cjb-104-3b
C13CPD DMSO {C:\Bruker\TopSpin3.2} 1401 4
f1 (ppm)


O
N
CF3
O

^{1}HNMR

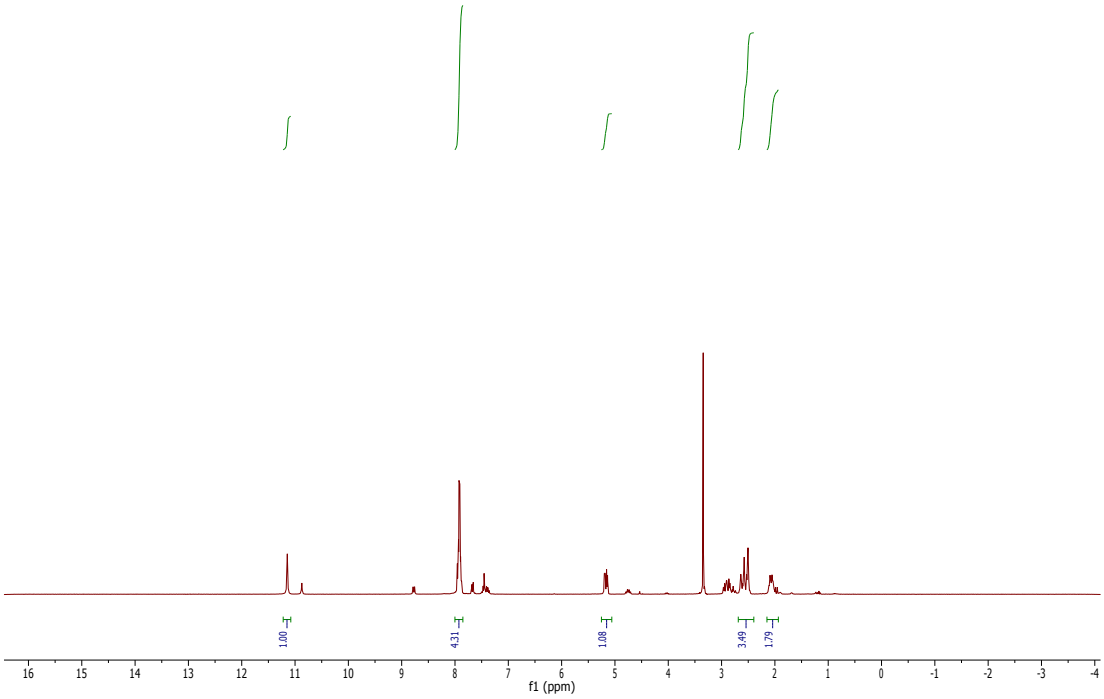

140402.342.10.fid
Kishore NK 930
Au1H DMSO /opt/topspin 1404 42
0.53
0.68
0.70
0.52
2.00
f1 (ppm)


140402.342.11.fid
Kishore NK 930
Au13C DMSO /opt/topspin 1404 42
166.65
134.98
131.08
124.48
123.60
121.80
27.12
26.99
f1 (ppm)



^{1}HNMR

140321.360.10.fid
Kishore/ NK 874
Au1H DMSO /opt/topspin 1403 60



^{13}CNMR

140321.360.11.fid
Kishore/ NK 874
Au13C DMSO /opt/topspin 1403 60