Supporting Information

Carbonylative Coupling of *N*-Chloroamines with Alcohols: Synthesis of Esterification Reagents

Zhiping Yin,[a] Zechao Wang,[a] and Xiao-Feng Wu*[a]

Leibniz-Institut für Katalyse e.V. an der Universität Rostock, Albert Einstein Straße 29a, 18059 Rostock, Germany

E-mail: xiao-feng.wu@catalysis.de

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General comments

Chemicals were purchased from Sigma-Aldrich, Strem, Acros, TCI or Alfa Aesar and used as such unless stated otherwise. NMR spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. All solvents and reagents were purchased from Sigma-Aldrich and used as received. Chemical shifts (ppm) are given relative to solvent: references for $CDCl_3$ were 7.26 ppm (^{1}H NMR) and 77.00 ppm (^{13}C NMR). Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), m (multiplet) and br.s (broad singlet). GC-yields were calculated using hexadecane as internal standard. 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-7890A 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).

N-Chloroamines were prepared according literature from the corresponding amines (W. Li, X. F. Wu, *Chem. Eur. J.* 2015, **21**, 7374; T. V. Hughes, S. D. Hammond, M. P. Cava, *J. Org. Chem.*, 1998, **63**, 401; A. Fraix, T. Le Gall, M. Berchel, C. Denis, P. Lehn, T. Montier, P. A. Jaffrès, *Org. Biomol. Chem.*, 2013, **11**, 1650).

General procedure

Synthesis of benzotriazole-carboxylates: To a screw-cap vial (4 mL) equipped with a septum, a small cannula, and a stirring bar was added 1-chloro-1*H*-benzo[*d*][1,2,3]triazole **1a** (1.0 mmol, 153 mg), CH_3CN (2 mL), [(iPr)CuCl] (5 mol%, 12 mg) and MeOH (0.5 mmol, 16 mg, 20 ul). The vial then was purged with argon three times before placed on an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr instruments under air. After flushing the autoclave three times with CO, a pressure of 50 bar CO was set and the reaction was performed for 16 hours at 70 °C. Afterwards, the autoclave was cooled to room temperature and the pressure was released carefully. The solvent was removed under reduced pressure and the crude products were purified by column chromatography on silica gel (eluent: pentane/ethyl acetate = 15:1) to give the pure product **2a** as white solid in 80% yield (70.8 mg).

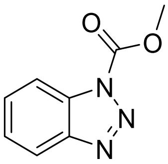
Synthesis of carbamates: To a screw-cap vial (4 mL) equipped with a septum, a small cannula, and a stirring bar was added 4-chloromorpholine **1d** (1.0 mmol, 121 mg), CH_3CN (2 mL), TBAB (5 mmol%, 16.3 mg)**,** Pd/C (2 mmol%, 21.2 mg) and MeOH (10 mmol, 320 mg). The vial then was purged with argon three times before placed on an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr instruments under air. After flushing the autoclave three times with CO, a pressure of 50 bar CO was set and the reaction was performed for 16 hours at 70 °C. Afterwards, the autoclave was cooled to room temperature and the pressure was released carefully. The solvent was removed under reduced pressure and the crude products were purified by column chromatography on silica gel (eluent: pentane/ethyl acetate = 10:1) to give the pure product **2n** as colorless oil in 60% yield (87 mg).

Synthesis of methyl-2-cyano-2-phenylacetate: To a solution of LDA (2.0 mmol, 1M in Et_2O, 2ml) in THF (2 mL) at 0 °C under argon was added the phenylacetonitrile (1 mmol, 117 mg) in THF (1 mL), and the mixture was stirred for 30 min at room temperature. This solution was syringed into a solution of methyl 1*H*-

benzo[*d*][1,2,3]triazole-1-carboxylate (354 mg, 2.0 mmol) in THF (2 mL) at 0 °C under argon, and the reaction mixture was stirred 20 hours at room temperature. A solution of NH_4Cl (3 mL) was added, and extracted with EtOAc (3 × 4 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The pure products were obtained after purification by column chromatography (ethyl acetate/pentane = 1:20) to give the pure product as colorless oil in 74% yield (129 mg).

Spectroscopic Data of Products

Methyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate[1] (2a)



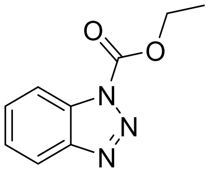
Yield: 71 mg (80%); white solid, M.p. 69-71 °C.

^{1}H NMR (300 MHz, CDCl$_3$) δ 8.12 – 7.98 (m, 2H), 7.59 (ddd, J = 8.4, 7.1, 0.8 Hz, 1H), 7.50 – 7.37 (m, 1H), 4.17 (s, 3H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 149.38, 145.80, 131.70, 130.23, 125.79, 120.37, 113.32, 55.24.

GC-MS (EI, 70ev): m/z (%) = 177 (M$^+$, 100), 134 (64), 120 (17), 106 (36), 90 (95), 78 (30), 59 (62).

Ethyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate[1] (2b)



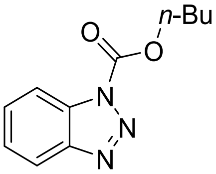
Yield: 74.5 mg (79%); white solid, M.p. 70-72 °C.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.15 – 8.06 (m, 2H), 7.68 – 7.59 (m, 1H), 7.51 – 7.43 (m, 1H), 4.68 (q, J = 7.1 Hz, 2H), 1.56 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 148.87, 145.83, 131.73, 130.10, 125.68, 120.34, 113.39, 65.17, 14.23.

GC-MS (EI, 70ev): m/z (%) = 191 (M$^+$, 47), 135 (42), 119 (17), 91 (100), 79 (21), 64 (36).

Butyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate[1] (2c)



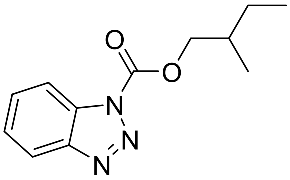
Yield: 78.8 mg, colorless oil, 72%,

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.15 – 8.06 (m, 2H), 7.64 (ddd, J = 8.2, 7.1, 1.1 Hz, 1H), 7.48 (ddd, J = 8.2, 7.1, 1.1 Hz, 1H), 4.62 (t, J = 6.7 Hz, 2H), 1.96 – 1.85 (m, 2H), 1.61 – 1.47 (m, 2H), 1.01 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 149.01, 145.87, 131.75, 130.10, 125.67, 120.37, 113.38, 68.90, 30.51, 18.97, 13.61.

GC-MS (EI, 70ev): m/z (%) = 219 (M$^+$, 5), 135 (8), 119 (6), 91 (14).

2-Methylbutyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2d)



Yield: 85.0 mg, colorless oil, 73%,

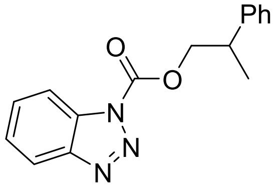
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.17 – 8.07 (m, 2H), 7.65 (ddd, J = 8.3, 7.1, 1.1 Hz, 1H), 7.48 (ddd, J = 8.2, 7.1, 1.1 Hz, 1H), 4.50 (dd, J = 10.5, 6.1 Hz, 1H), 4.42 (dd, J = 10.5, 6.8 Hz, 1H), 2.03 (m, 1H), 1.67 – 1.53 (m, 1H), 1.42 – 1.28 (m, 1H), 1.09 (d, J = 6.8 Hz, 3H), 0.99 (t, J = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 149.09, 145.89, 131.74, 130.09, 125.65, 120.39, 113.35, 73.48, 34.20, 25.85, 16.28, 11.15.

GC-MS (EI, 70ev): m/z (%) = 233 (M$^+$, 10), 135 (27), 136 (18), 119 (34), 91 (55), 55 (14).

HRMS (EI) Calc. for $C_{12}H_{15}O_2N_3$ (M+): 233.11588; found: 233.11613.

2-Phenylpropyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2e)



Yield: 94 mg, colorless oil, 67%

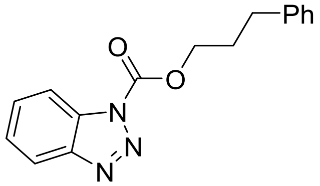
^{1}H NMR (300 MHz, CDCl$_3$) δ 8.07 – 8.00 (m, 1H), 7.78 (dt, *J* = 8.3, 1.0 Hz, 1H), 7.49 (ddd, *J* = 8.3, 7.1, 1.1 Hz, 1H), 7.38 (ddd, *J* = 8.2, 7.1, 1.1 Hz, 1H), 7.29 – 7.24 (m, 4H), 7.22 – 7.17 (m, 1H), 4.67 – 4.58 (m, 2H), 3.34 (m, 1H), 1.40 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 148.83, 145.85, 141.91, 131.59, 130.04, 128.74, 127.33, 127.13, 125.65, 120.34, 113.35, 73.38, 38.99, 17.90.

GC-MS (EI, 70ev): m/z (%) = 281 (M$^+$, 3), 163 (7), 134 (12), 118 (65), 105 (18), 91 (100), 77 (12).

HRMS (EI) Calc. for $C_{16}H_{15}O_2N_3$ (M+): 281.11588; found: 281.11671.

3-Phenylpropyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2f)



Yield: 116.6 mg, colorless oil, 83%

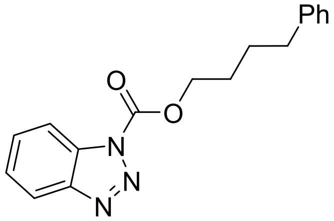
^{1}H NMR (300 MHz, CDCl$_3$) δ 8.08 – 7.98 (m, 2H), 7.57 (ddd, *J* = 8.3, 7.1, 1.1 Hz, 1H), 7.41 (ddd, *J* = 8.2, 7.1, 1.1 Hz, 1H), 7.25 – 7.07 (m, 5H), 4.55 (t, *J* = 6.6 Hz, 2H), 2.78 (dd, *J* = 8.4, 6.8 Hz, 2H), 2.27 – 2.12 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 148.92, 145.85, 140.35, 131.73, 130.12, 128.51, 128.35, 126.19, 125.70, 120.38, 113.36, 68.21, 31.85, 29.94.

GC-MS (EI, 70ev): m/z (%) = 281 (M$^+$, 4), 252 (5), 207 (20), 180 (7), 118 (52), 104 (6), 91 (100), 44 (28).

HRMS (EI) Calc. for $C_{16}H_{15}O_2N_3$ (M+): 281.11588; found: 281.11633.

4-Phenylbutyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2g)



Yield: 97 mg, colorless oil, 66%,

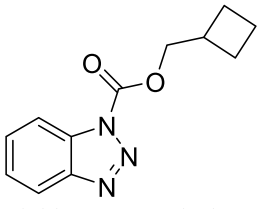
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.14 (dt, *J* = 8.3, 0.9 Hz, 1H), 8.10 (dt, *J* = 8.3, 1.0 Hz, 1H), 7.66 (ddd, *J* = 8.3, 7.1, 1.0 Hz, 1H), 7.50 (ddd, *J* = 8.2, 7.1, 1.0 Hz, 1H), 7.31 – 7.26 (m, 2H), 7.24 – 7,18 (m, 3H), 4.64 (t, *J* = 6.6 Hz, 2H), 2.73 (t, *J* = 7.4 Hz, 2H), 2.03 – 1.92 (m, 2H), 1.91 – 1.80 (m, 2H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 149.02, 145.91, 141.54, 131.79, 130.16, 128.40, 125.97, 125.73, 120.44, 113.42, 68.92, 35.30, 28.07, 27.44.

GC-MS (EI, 70ev): m/z (%) = 295 (M$^+$, 3), 266 (5), 239 (7), 180 (7), 130 (10), 104 (21), 91 (100).

HRMS (EI) Calc. for $C_{17}H_{17}O_2N_3$ (M$^+$): 295.13153; found: 295.13126.

Cyclobutylmethyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2h)

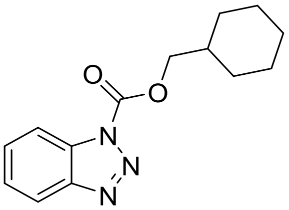


Yield: 64 mg, colorless oil, 55%,

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.13 (dt, *J* = 8.3, 1.0 Hz, 1H), 8.08 (dt, *J* = 8.3, 0.9 Hz, 1H), 7.64 (ddd, *J* = 8.3, 7.1, 1.0 Hz, 1H), 7.48 (ddd, *J* = 8.2, 7.1, 1.0 Hz, 1H), 4.60 (d, *J* = 6.9 Hz, 2H), 3.10-2.85 (m, 1H), 2.25 – 2.15 (m, 2H), 2.04 – 1.90 (m, 4H).

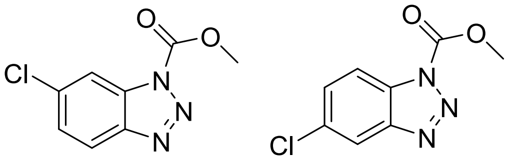
^{13}C NMR (100 MHz, CDCl$_3$) δ 149.11, 145.88, 131.74, 130.09, 125.66, 120.39, 113.40, 72.58, 33.84, 24.72, 18.38.
GC-MS (EI, 70ev): m/z (%) = 231 (M$^+$, 5), 135 (6), 119 (19), 90 (32), 64 (22), 41 (100).
HRMS (EI) Calc. for $C_{12}H_{13}O_2N_3$ (M+): 231.10023; found: 231.10026.

Cyclohexylmethyl 1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2i)



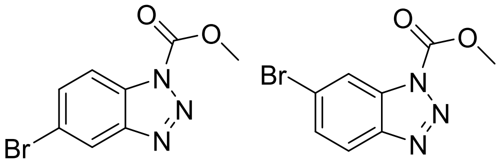
Yield: 88.1 mg, colorless oil, 68%,
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.13 (ddd, *J* = 9.2, 8.3, 1.0 Hz, 2H), 7.66 (ddd, *J* = 8.2, 7.1, 1.1 Hz, 1H), 7.50 (ddd, *J* = 8.2, 7.1, 1.1 Hz, 1H), 4.44 (d, *J* = 6.6 Hz, 2H), 2.05 – 1.85 (m, 3H), 1.85 – 1.66 (m, 4H), 1.35 – 1.27 (m, 2H), 1.19 – 1.07 (m, 2H).
^{13}C NMR (100 MHz, CDCl$_3$) δ 149.12, 145.92, 131.79, 130.13, 125.68, 120.42, 113.42, 73.93, 37.08, 29.48, 26.18, 25.50.
GC-MS (EI, 70ev): m/z (%) = 259 (M$^+$, 6), 164 (6), 136 (18), 119 (28), 95 (42), 55 (100).
HRMS (EI) Calc. for $C_{14}H_{17}O_2N_3$ (M$^+$): 259.13153; found: 259.13129.

Methyl 6-chloro-1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate and Methyl 5-chloro-1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2l + 2l')



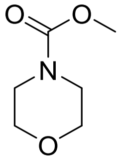
Yield: 74 mg, white solid,70%,
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.13 (ddd, *J* = 13.6, 1.9, 0.7 Hz, 1H), 8.06 (td, *J* = 8.7, 0.7 Hz, 1H), 7.63 (dd, *J* = 8.8, 1.9 Hz, 0.4H), 7.48 (dd, *J* = 8.8, 1.9 Hz, 0.6H), 4.25 (s, 1.5H), 4.25 (s, 1H).
^{13}C NMR (100 MHz, CDCl$_3$) δ 149.08, 146.49, 144.39, 137.04, 132.39, 131.74, 131.04, 127.00, 121.24, 119.93, 114.26, 113.47, 55.52.
GC-MS (EI, 70ev): m/z (%) = 213 (M+2$^+$, 10), 211 (M$^+$, 27), 168 (27), 124 (46), 59 (33).
HRMS (EI) Calc. for $C_8H_6O_2N_3{}^{37}Cl$ (M$^+$): 213.01136; found: 213.01125; $C_8H_6O_2N_3Cl$ (M$^+$): 211.01431; found: 211.01408.

Methyl 6-bromo-1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate and Methyl 5-bromo-1*H*-benzo[*d*][1,2,3]triazole-1-carboxylate (2m + 2m')



Yield: 98 mg, white solid, 77%,
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.33 (dd, *J* = 1.8, 0.6 Hz, 1H), 8.28 (dd, *J* = 1.7, 0.6 Hz, 0.3H), 8.02 (dd, *J* = 8.7, 0.7 Hz, 0.3H), 7.99 (dd, *J* = 8.7, 0.7 Hz, 1H), 7.75 (dd, *J* = 8.8, 1.7 Hz, 0.4H), 7.61 (dd, *J* = 8.7, 1.7 Hz, 1H), 4.25 (s, 3H), 4.24 (s, 1H).
^{13}C NMR (100 MHz, CDCl$_3$) δ 149.06, 144.71, 133.57, 132.69, 129.62, 125.13, 123.13, 121.43, 116.49, 114.57, 55.53.
GC-MS (EI, 70ev): m/z (%) = 257 (M+2$^+$, 21), 255 (M$^+$, 20), 214 (17), 212 (20), 170 (29), 168 (32), 59 (40).
HRMS (EI) Calc. for $C_8H_6O_2N_3{}^{81}Br$ (M$^+$): 256.96174; found: 256.96198; $C_8H_6O_2N_3Br$ (M$^+$): 254.96379; found: 256.96374.

Methyl morpholine-4-carboxylate[2] (2n)



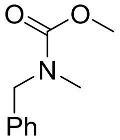
Yield: 87 mg, colorless oil, 60%,

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.70 (s, 3H), 3.67 – 3.60 (m, 4H), 3.46-3.43 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 155.90, 66.55, 52.65, 44.04.

GC-MS (EI, 70ev): m/z (%) = 145 (M$^+$, 29), 130 (62), 114 (13), 100 (22), 86 (12), 56 (24), 42 (41).

Methyl benzyl(methyl)carbamate[3] (2o)



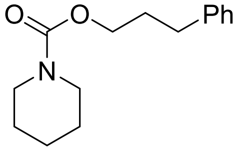
Yield: 95 mg, colorless oil, 53%,

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.33 – 7.04 (m, 5H), 4.45-4.35 (m, 2H), 3.67 (s, 3H), 2.78 – 2.67 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 137.42, 128.52, 127.75, 127.29, 127.18, 52.69, 52.48, 52.22.

GC-MS (EI, 70ev): m/z (%) = 179 (M$^+$, 65), 164 (34), 168 (27), 147 (12), 120 (47), 91 (100), 65 (19), 42 (26).

3-Phenylpropyl piperidine-1-carboxylate (2p)



Yield: 163 mg, colorless oil, 66%,

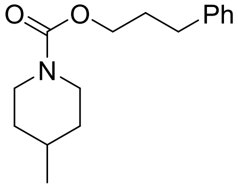
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 2H), 7.25 – 7.18 (m, 3H), 4.13 (t, *J* = 6.5 Hz, 2H), 3.50 – 3.35 (m, 4H), 2.81 – 2.63 (m, 2H), 2.08 – 1.91 (m, 2H), 1.59 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.42, 141.38, 128.29, 125.79, 64.41, 44.64, 32.22, 30.59, 25.60, 24.32.

GC-MS (EI, 70ev): m/z (%) = 247 (M+, 3), 118 (84), 91 (37), 65 (5), 41 (5).

HRMS (EI) Calc. for $C_{15}H_{21}O_2N$ (M$^+$): 247.15668; found: 247.15669.

3-Phenylpropyl 4-methylpiperidine-1-carboxylate (2q)



Yield: 128 mg, colorless oil, 49%,

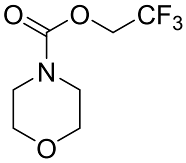
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.15 (m, 2H), 7.13 – 7.06 (m, 3H), 4.05 – 3.95 (m, 4H), 2.73 – 2.55 (m, 4H), 1.96 – 1.81 (m, 2H), 1.60 – 1.32 (m, 3H), 1.10 – 0.91 (m, 2H), 0.86 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 155.38, 141.35, 128.27, 128.26, 125.77, 64.41, 44.00, 33.85, 32.19, 30.79, 30.57, 21.77.

GC-MS (EI, 70ev): m/z (%) = 261 (M+, 3), 144 (5), 91 (37), 55 (11).

HRMS (EI) Calc. for $C_{16}H_{23}O_2N$ (M$^+$): 261.17233; found: 261.17234.

2,2,2-Trifluoroethyl morpholine-4-carboxylate (2r)



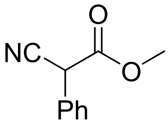
Yield: 119 mg, colorless oil, 56%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.49 (q, *J* = 8.5 Hz, 2H), 3.62 – 3.71 (m, 4H), 3.54 – 3.47 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.28, 122.50 (q, J = 1098.4 Hz), 66.40 (d, J = 68 Hz), 61.30 (q, J = 144.8 Hz), 44.30 (d, J = 83.6 Hz),

GC-MS (EI, 70ev): m/z (%) = 213 (M^+, 54), 198 (100), 170 (20), 127 (38), 100 (60), 83 (88), 56 (48).
HRMS (EI) Calc. for $C_7H_{10}O_3N_1F_3$ (M+): 213.06073; found: 213.06037.

Methyl 2-cyano-2-phenylacetate[4]

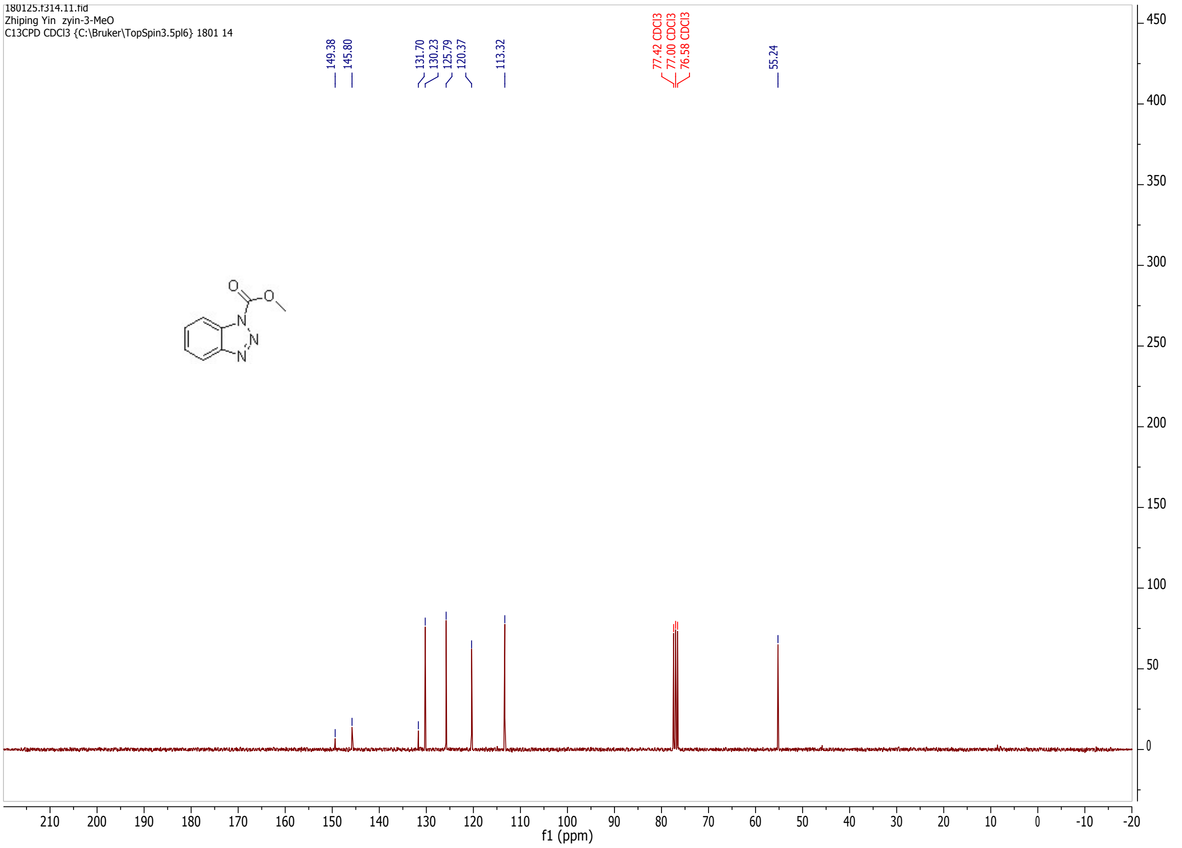


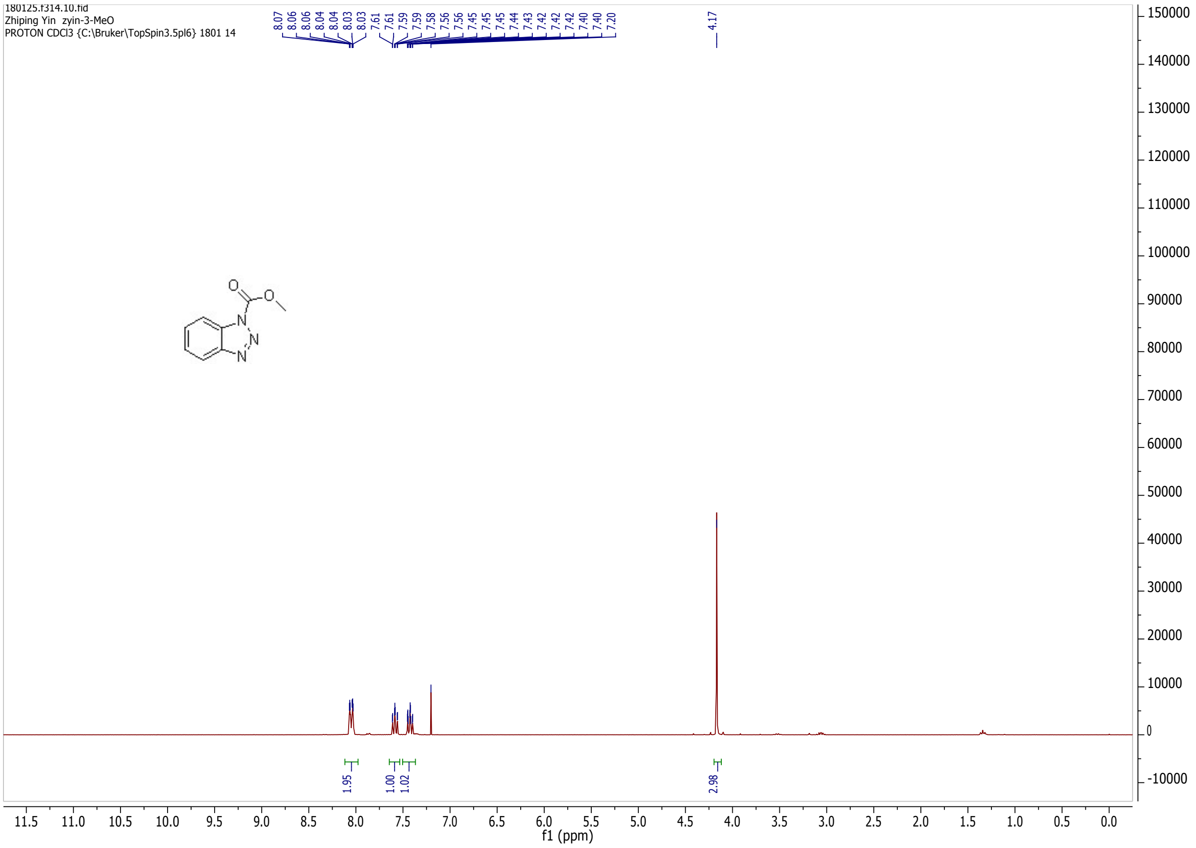
Yield: 129 mg, colorless oil, 74%Yield,
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.38 (m, 5H), 4.75 (s, 1H), 3.80 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 165.41, 129.74, 129.31, 129.23, 127.86, 115.50, 53.88, 43.45.
GC-MS (EI, 70ev): m/z (%) = 175 (M^+, 10), 130 (20), 116 (96), 89 (21), 59 (11).

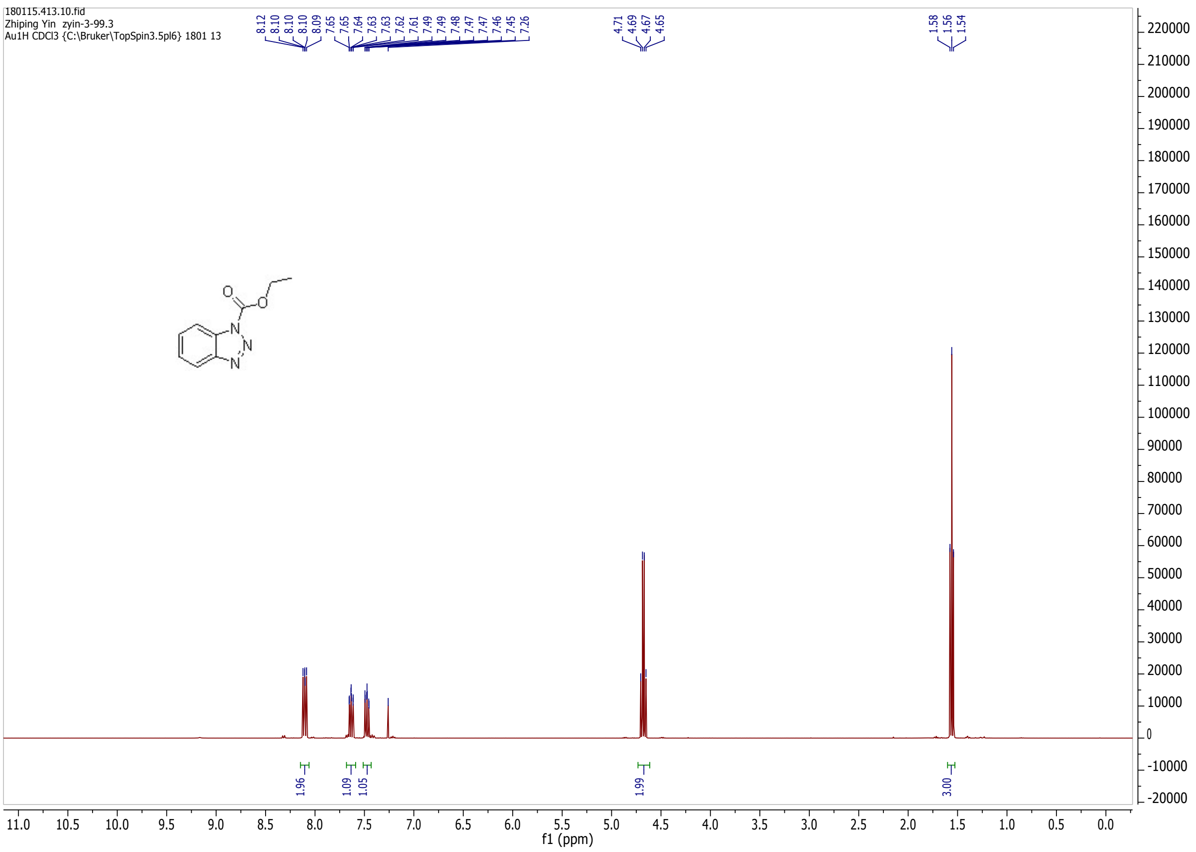
References

(1) Katritzky, A. R.; Zhang, G. F.; Fan, W. Q.; Wu, J.; Pernak, J. *J. Phys. Org. Chem.* **1993**, *6*, 567.
(2) Zeng, R.; Bao, L.; Sheng, H.; Sun, L.; Chen, M.; Feng, Y.; Zhu, M. *RSC Advances* **2016**, *6*, 78576.
(3) Peterson, S. L.; Stucka, S. M.; Dinsmore, C. J. *Org. Lett.* **2010**, *12*, 1340.
(4) Park, E. J.; Lee, S.; Chang, S. *J. Org. Chem.* **2010**, *75*, 2760.

Copy of ^{1}H and ^{13}C NMR Spectra of Products



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8.09
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7.63
7.63
7.62
7.61
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.26
4.71
4.69
4.67
4.65
1.58
1.56
1.54
O
O
N
N
N
1.96
1.09
1.05
1.99
3.00
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190000
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160000
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50000
40000
30000
20000
10000
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10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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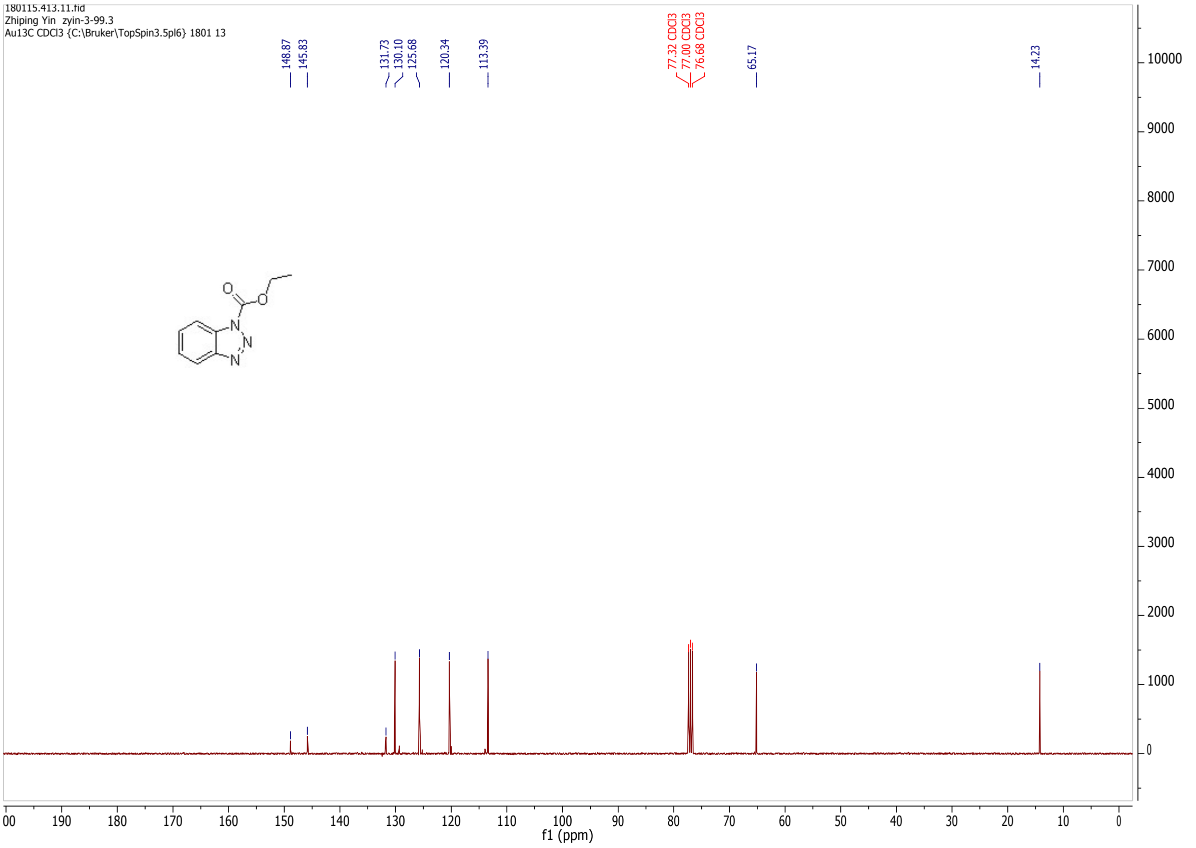
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125.68
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113.39
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65.17
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O
O
N
N
N
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00
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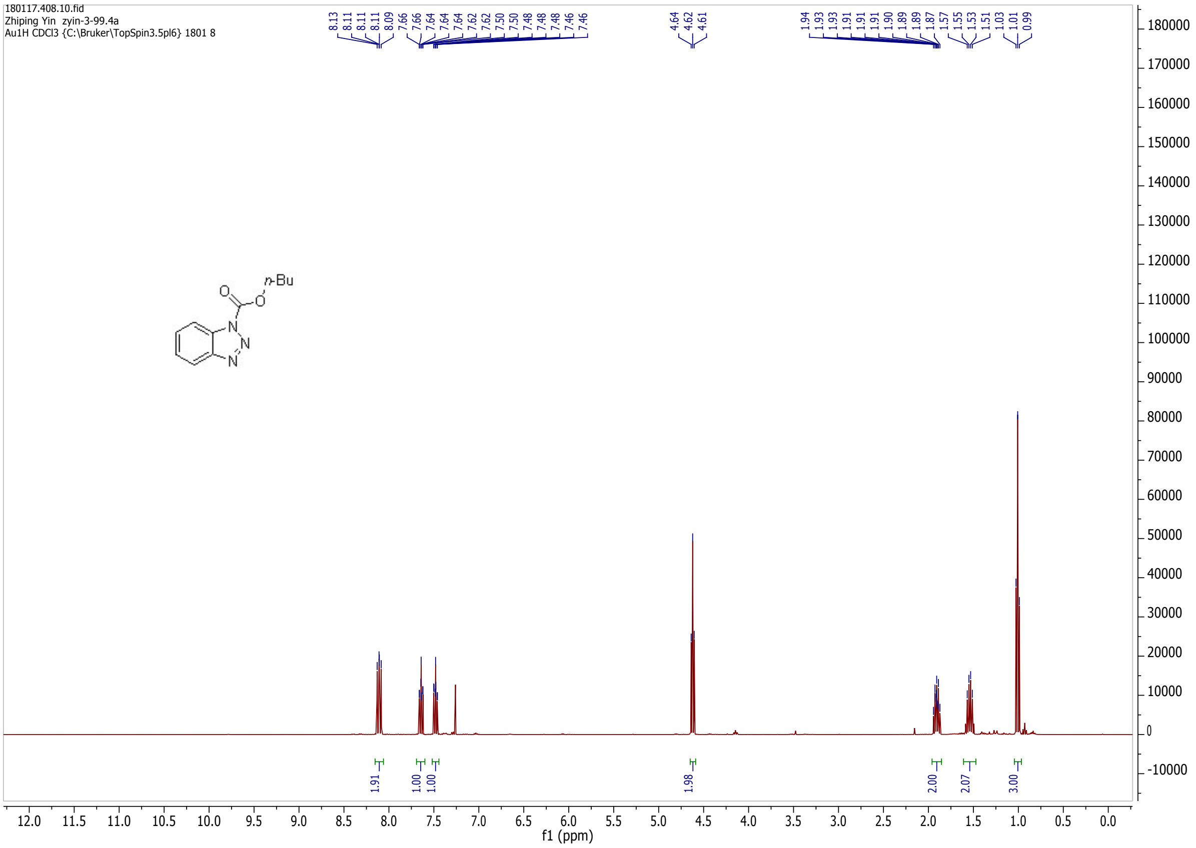
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4.64 4.62 4.61
1.94 1.93 1.93 1.91 1.91 1.91 1.90 1.89 1.89 1.87 1.57 1.55 1.53 1.51 1.03 1.01 0.99
n-Bu
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O
N
N
N
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1.00
1.00
1.98
2.00
2.07
3.00
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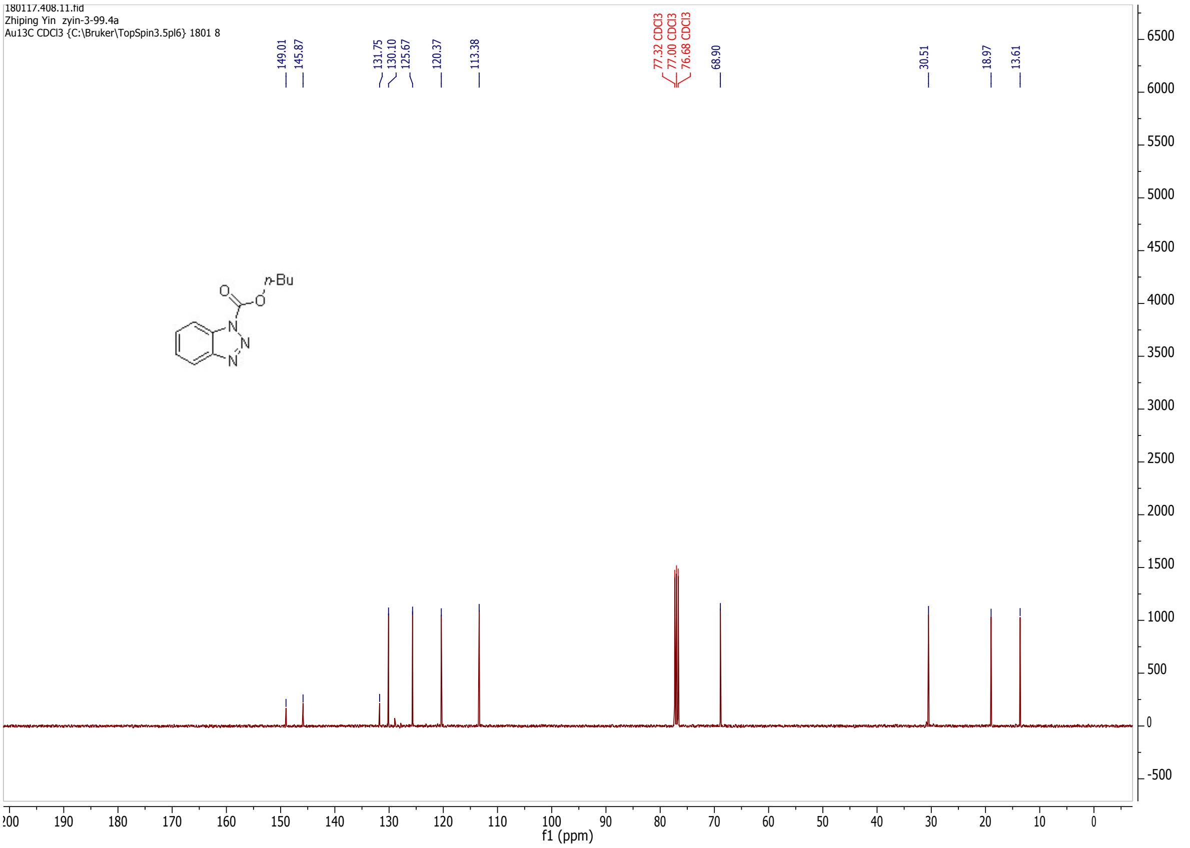
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68.90
30.51
18.97
13.61
n-Bu
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O
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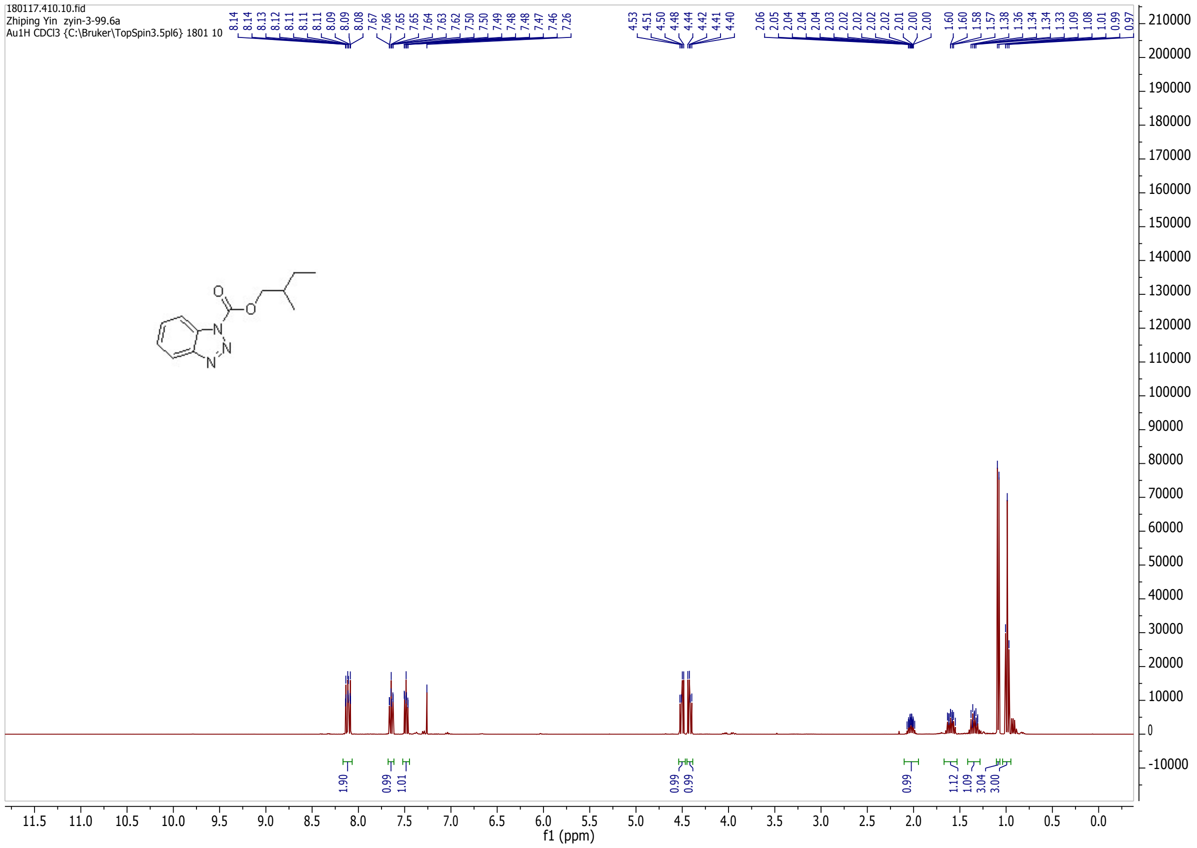
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4.53 4.51 4.50 4.48 4.44 4.42 4.41 4.40
2.06 2.05 2.04 2.04 2.04 2.03 2.02 2.02 2.02 2.02 2.01 2.00 2.00
1.60 1.60 1.58 1.57 1.38 1.36 1.34 1.34 1.33 1.09 1.08 1.01 0.99 0.97
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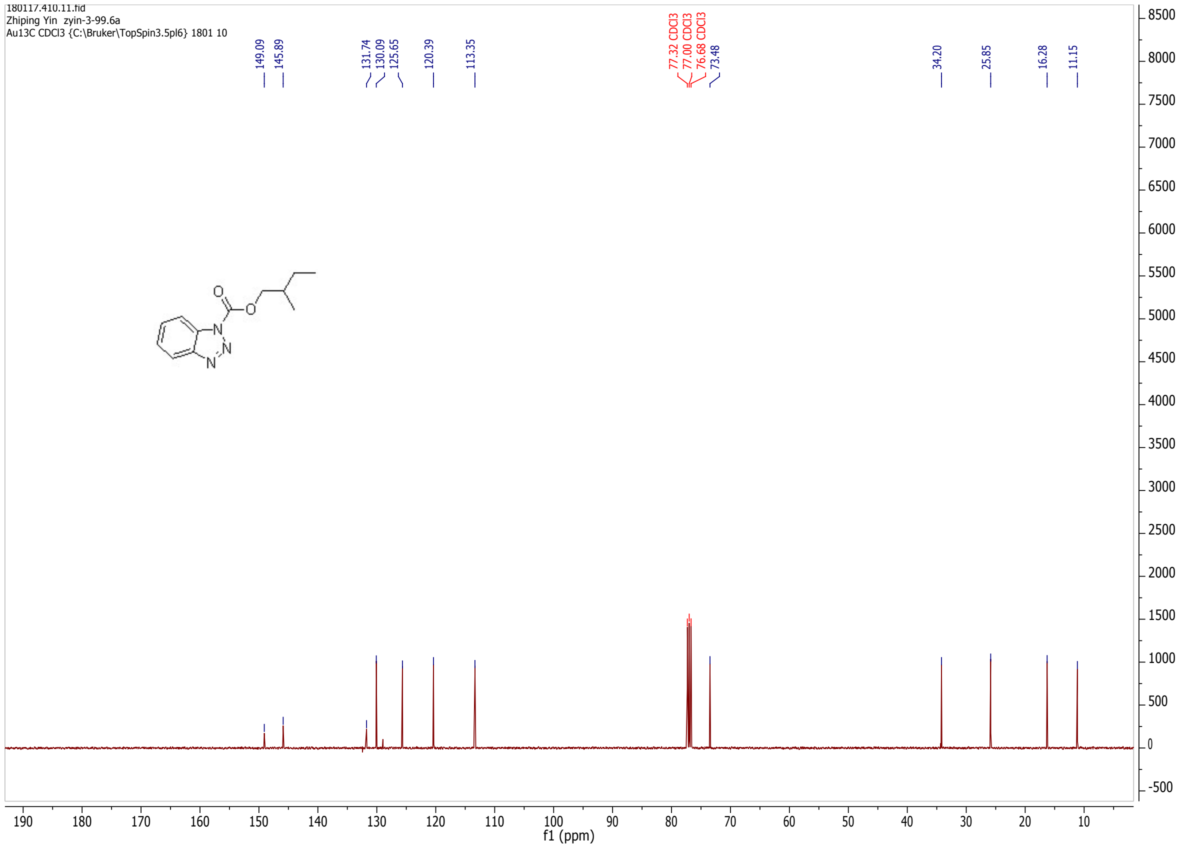
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113.35
77.32 CDCl3
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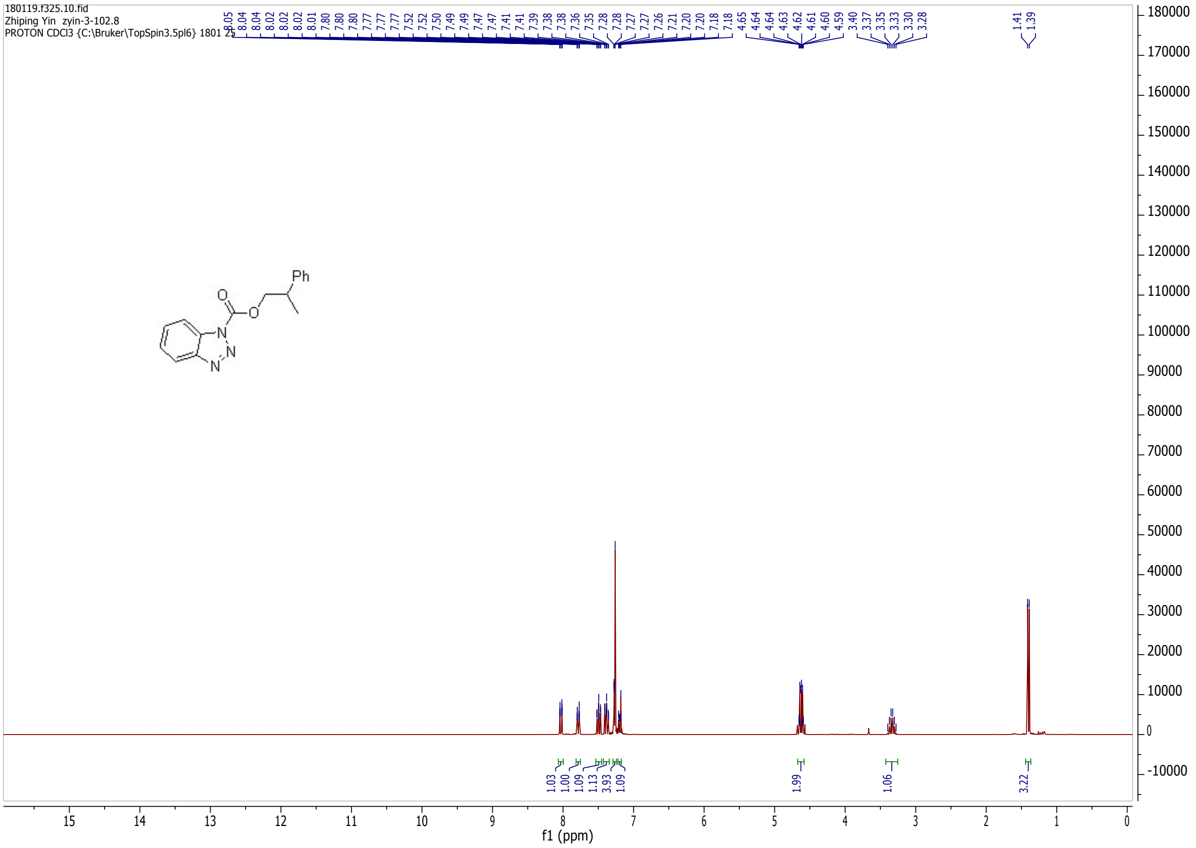
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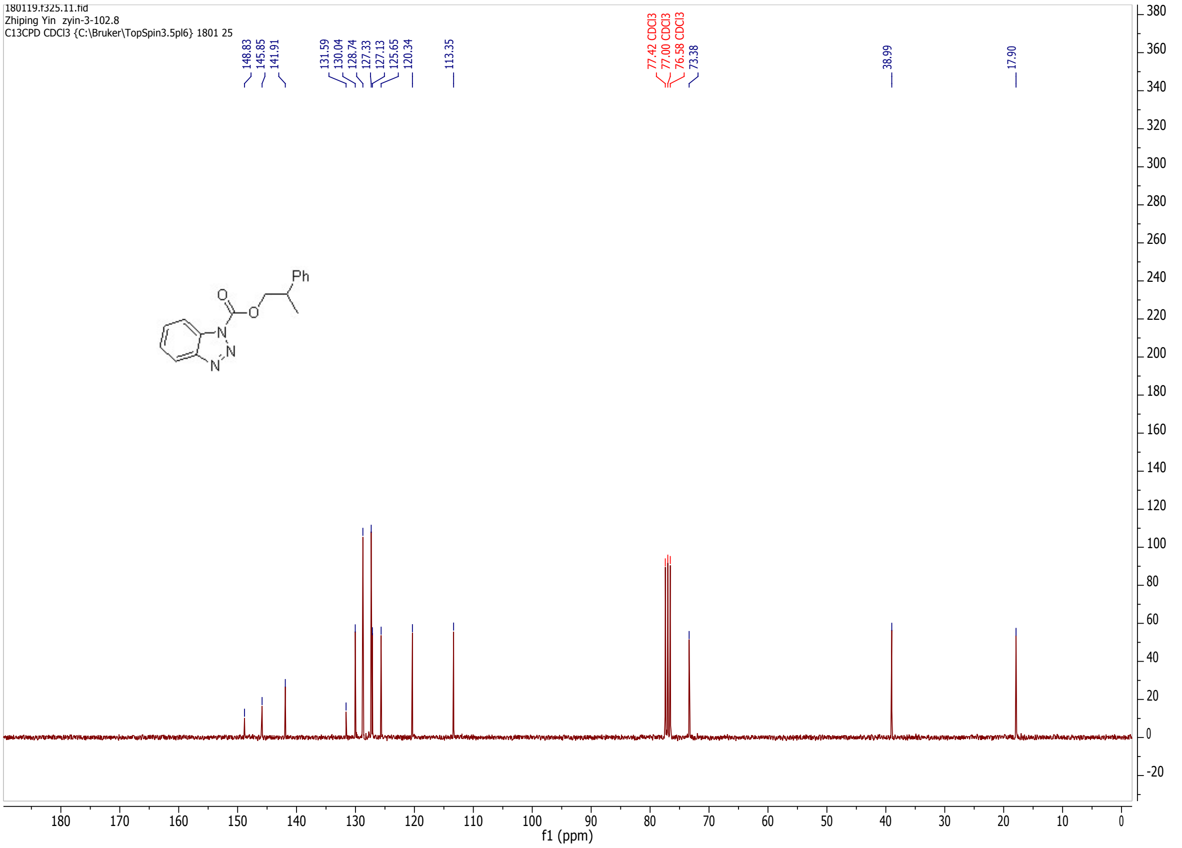
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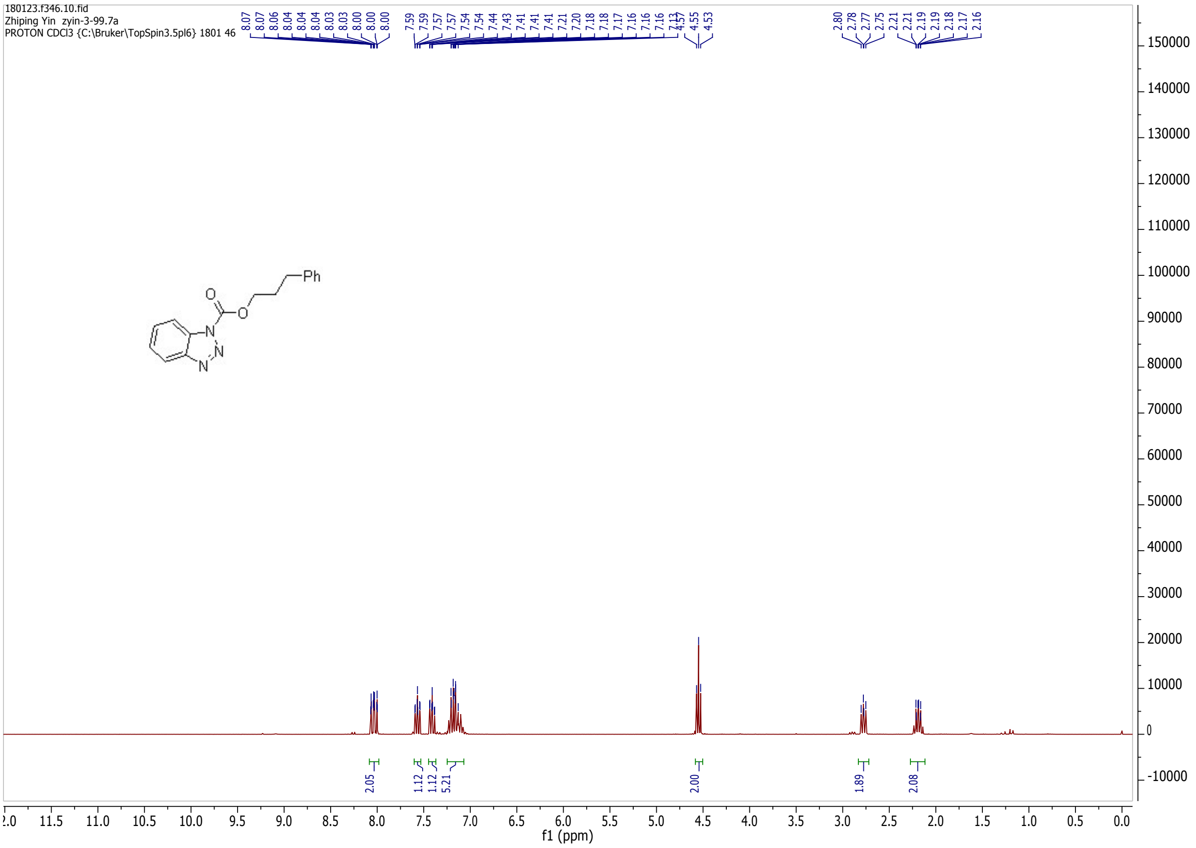
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2.80 2.78 2.77 2.75 2.21 2.21 2.19 2.19 2.18 2.17 2.16
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140000
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120000
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60000
50000
40000
30000
20000
10000
0
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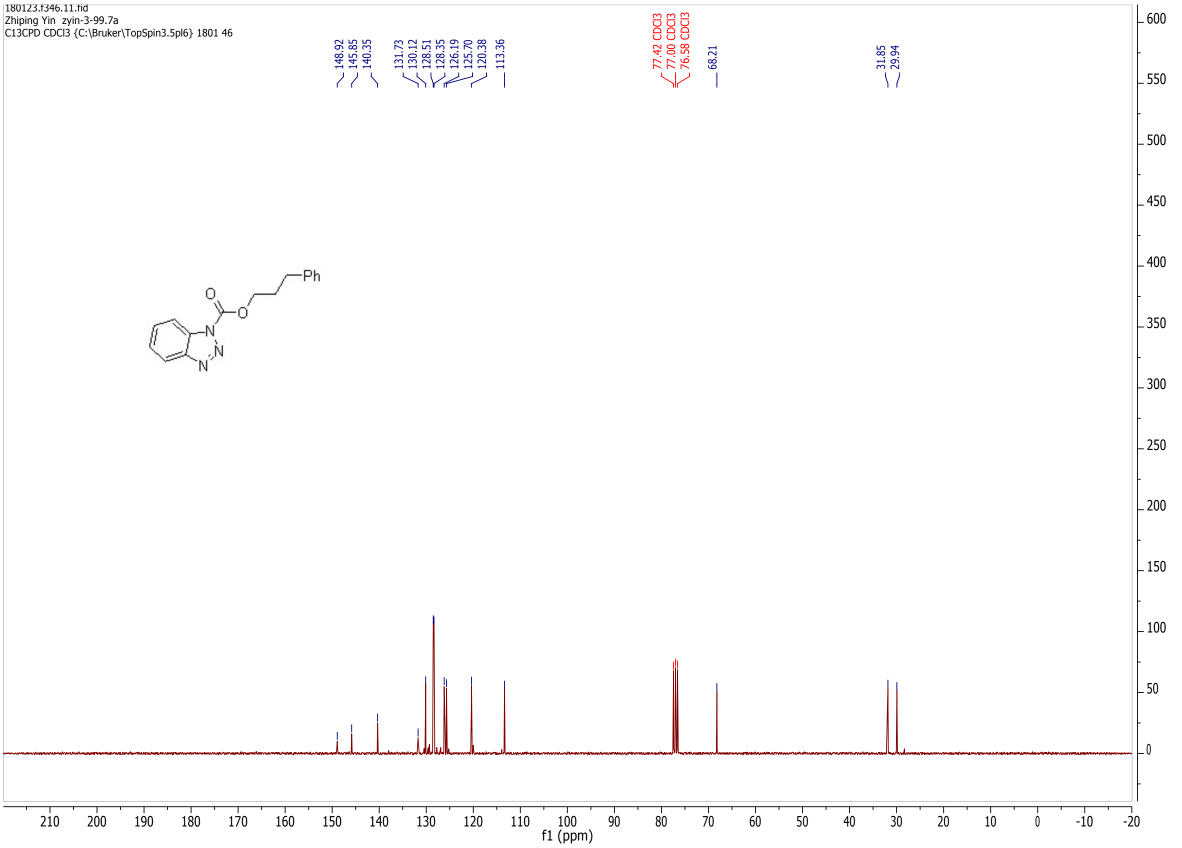
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128.35
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125.70
120.38
113.36
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
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31.85
29.94
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N
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500
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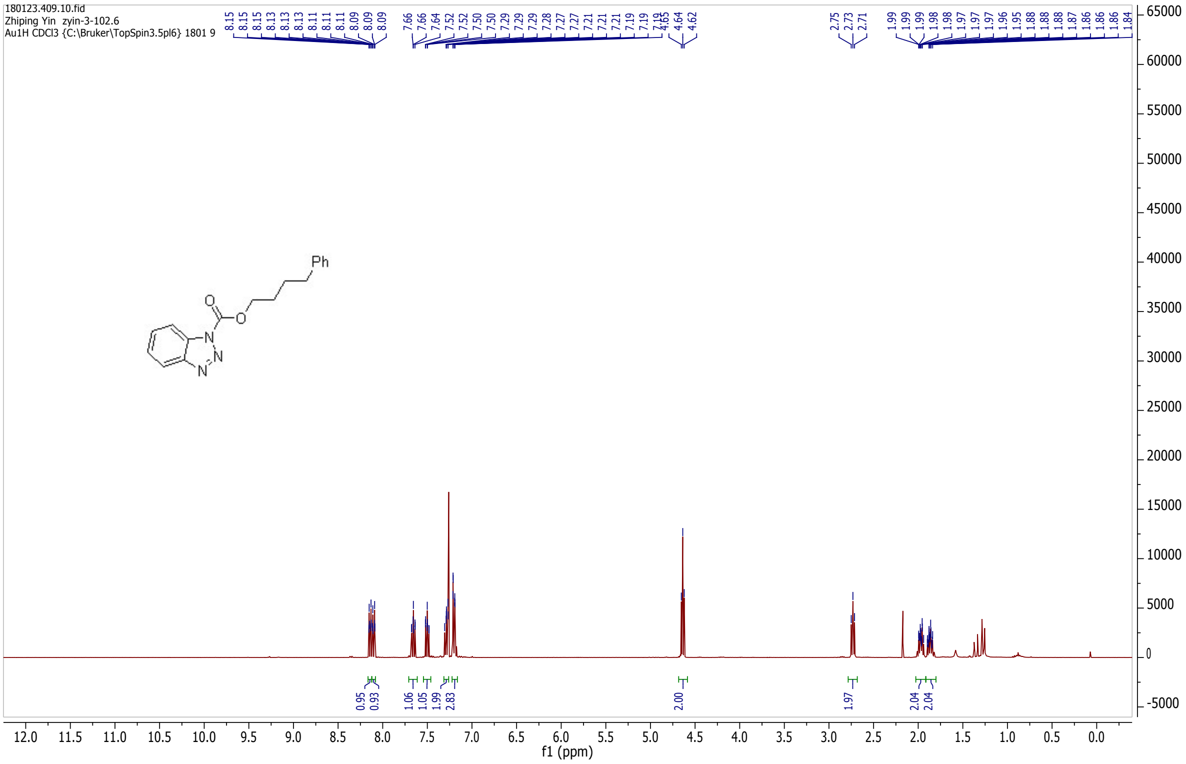
f1 (ppm)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8.11
8.11
8.09
8.09
8.09
7.66
7.66
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7.52
7.50
7.50
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7.29
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7.21
7.21
7.19
7.19
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4.64
4.62
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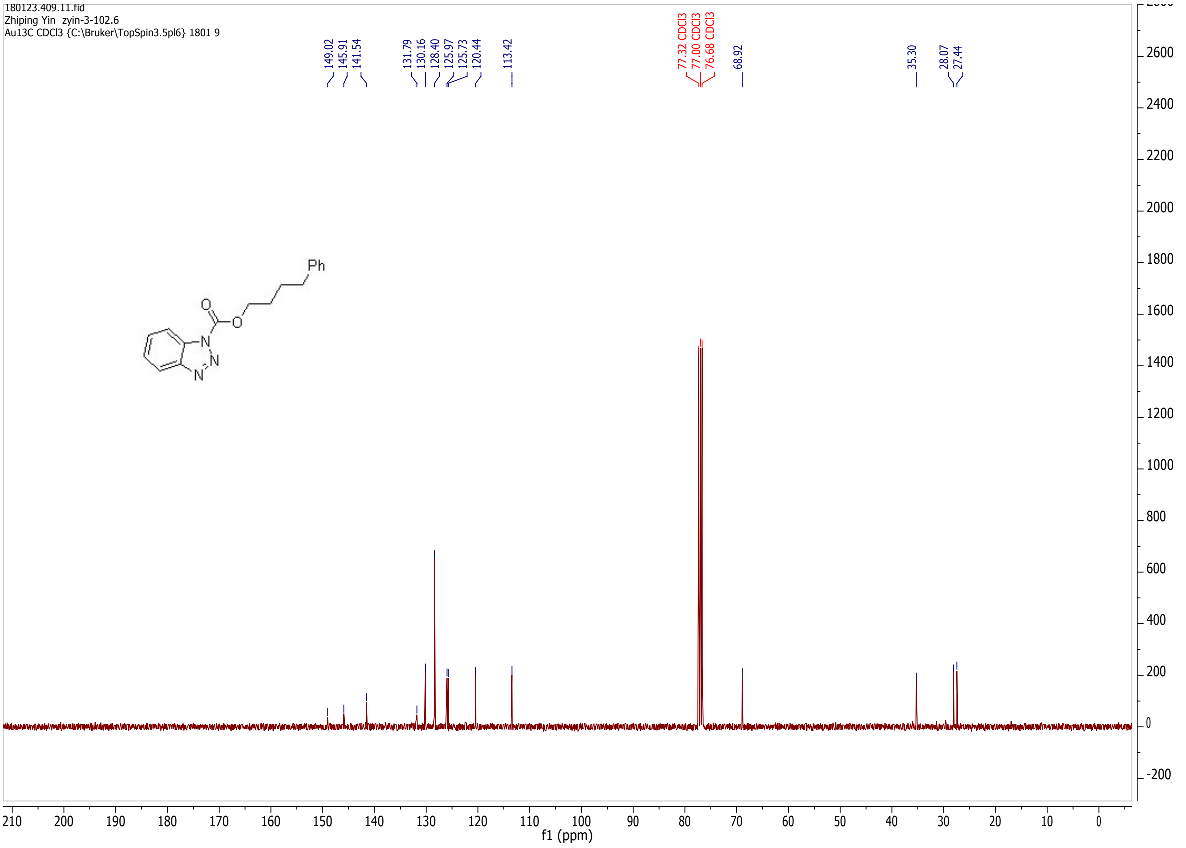
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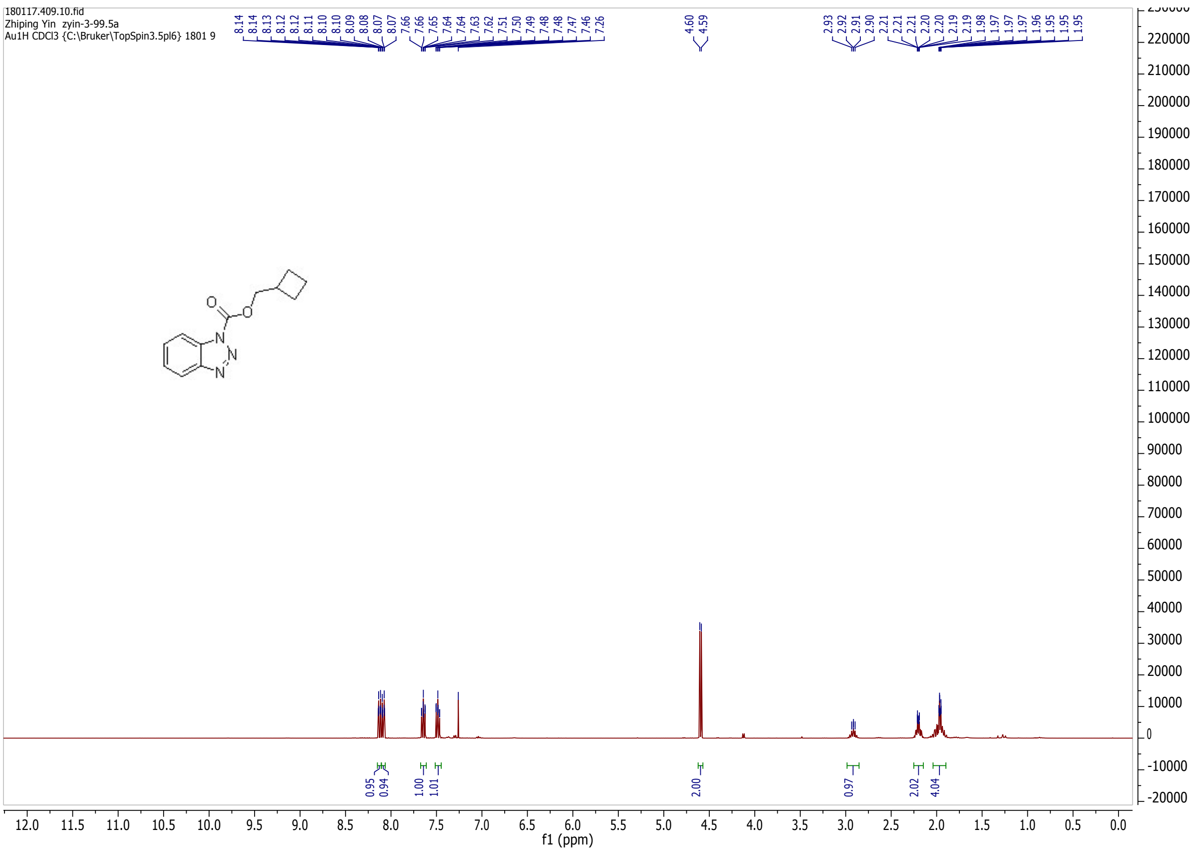
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4.60 4.59
2.93 2.92 2.91 2.90 2.21 2.21 2.21 2.20 2.20 2.19 2.19 1.98 1.97 1.97 1.97 1.96 1.95 1.95 1.95
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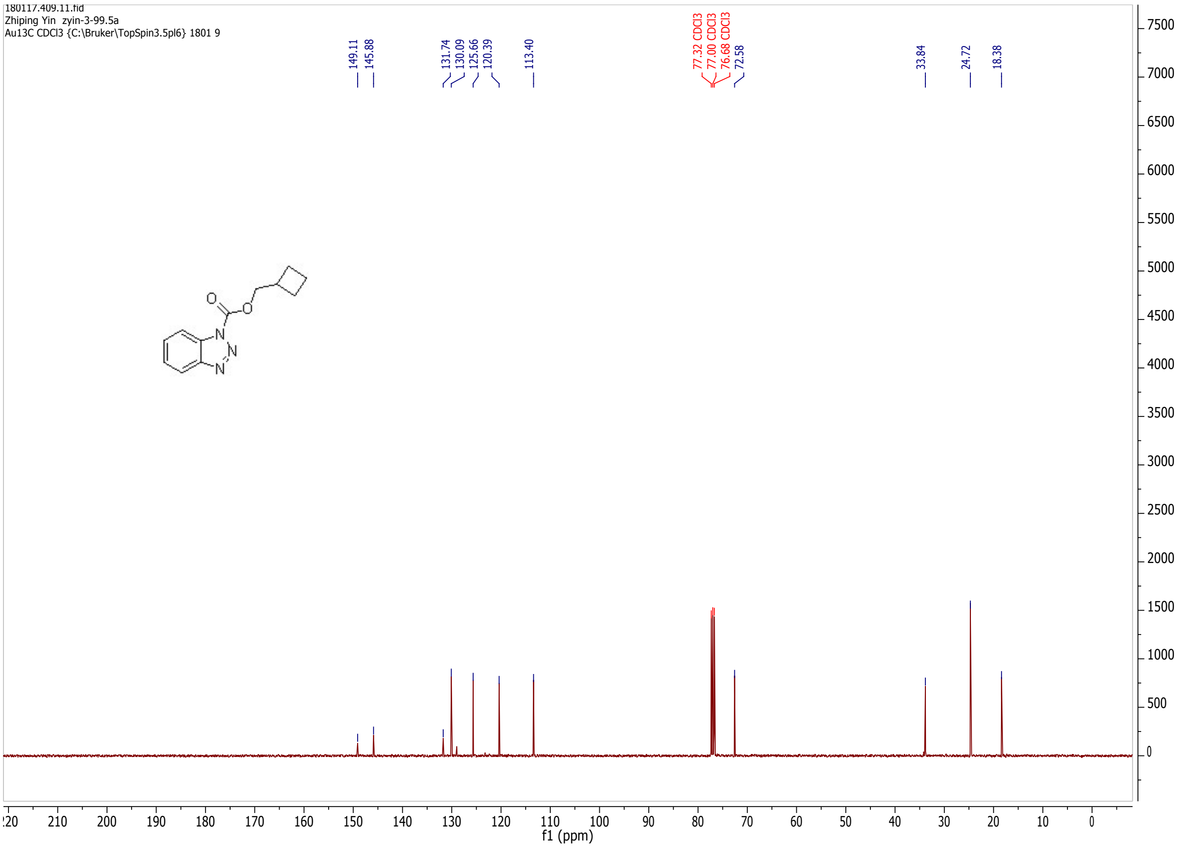
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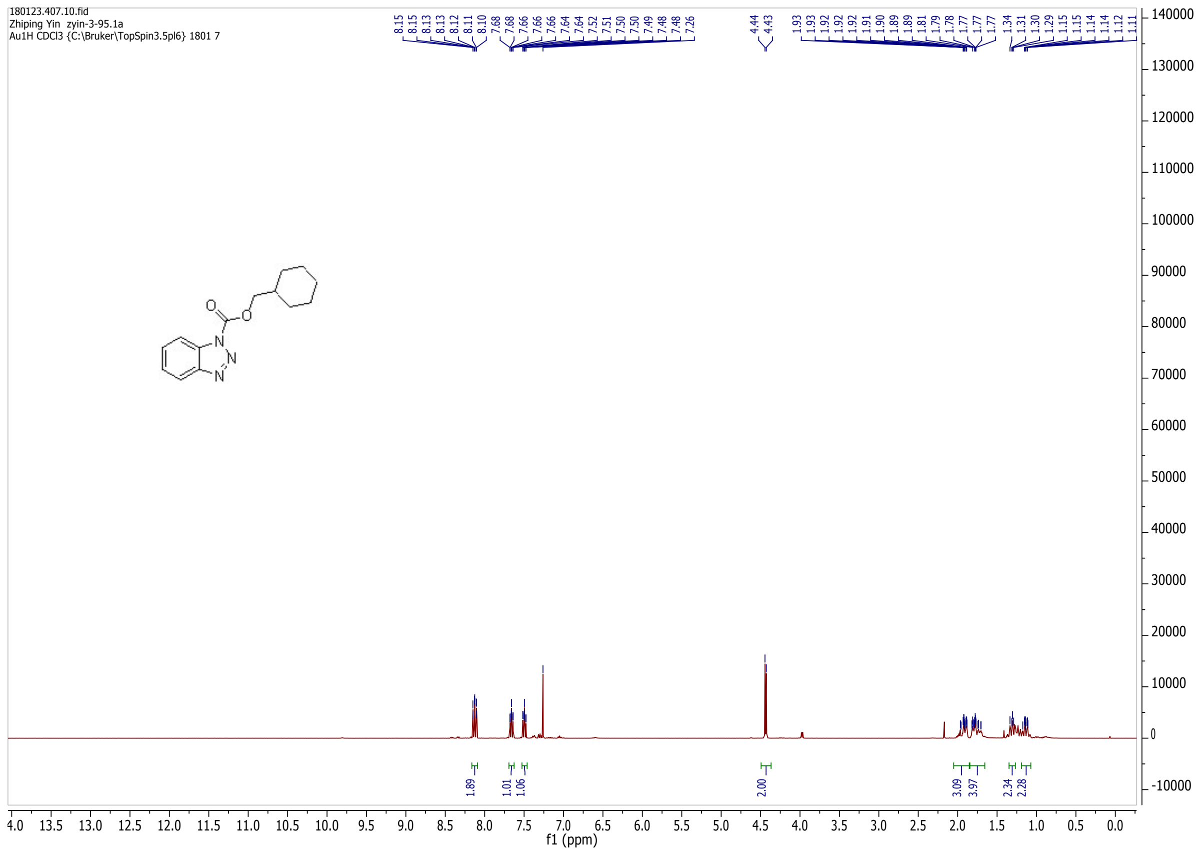
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4.44 4.43
1.93 1.93 1.92 1.92 1.92 1.91 1.90 1.89 1.89 1.81 1.79 1.78 1.77 1.77 1.77
1.34 1.31 1.30 1.29 1.15 1.15 1.14 1.14 1.12 1.11
1.89 1.01 1.06 2.00 3.09 3.97 2.34 2.28
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f1 (ppm)
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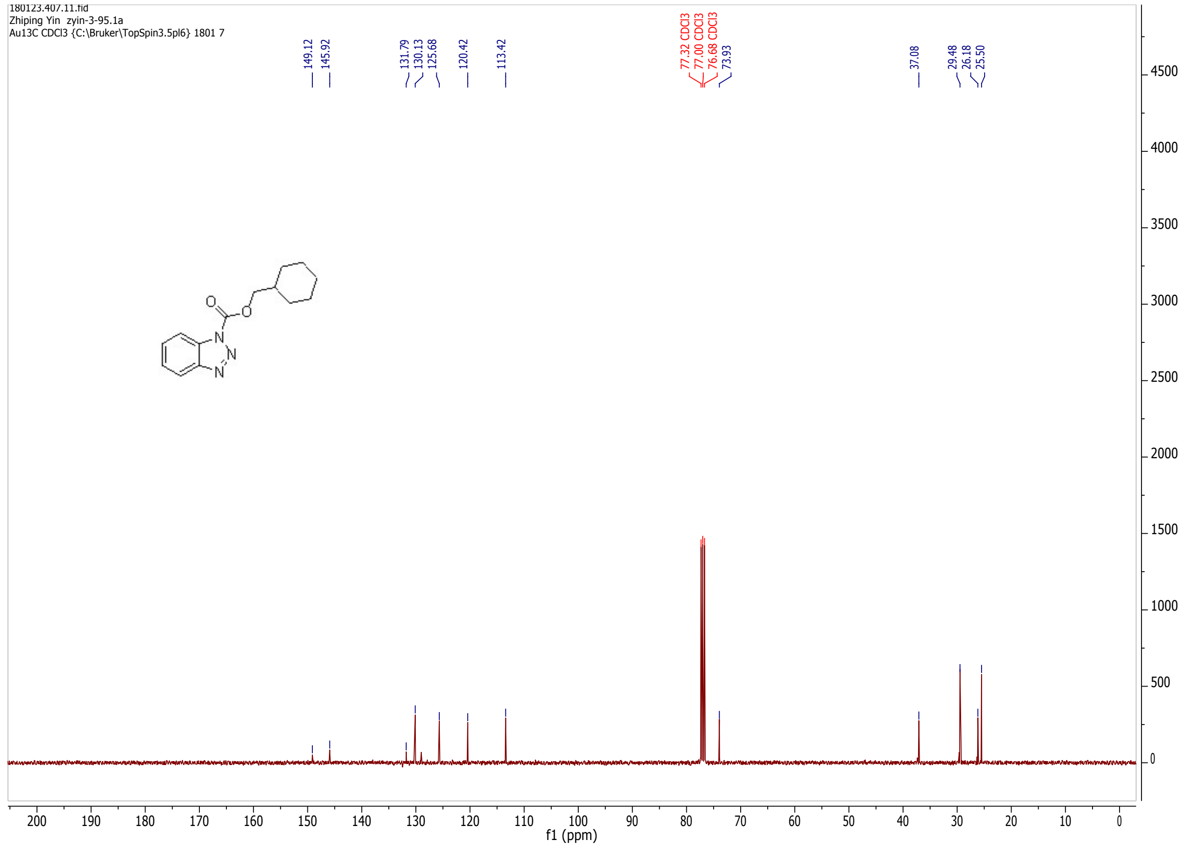
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131.79 130.13 125.68
120.42
113.42
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37.08
29.48 26.18 25.50
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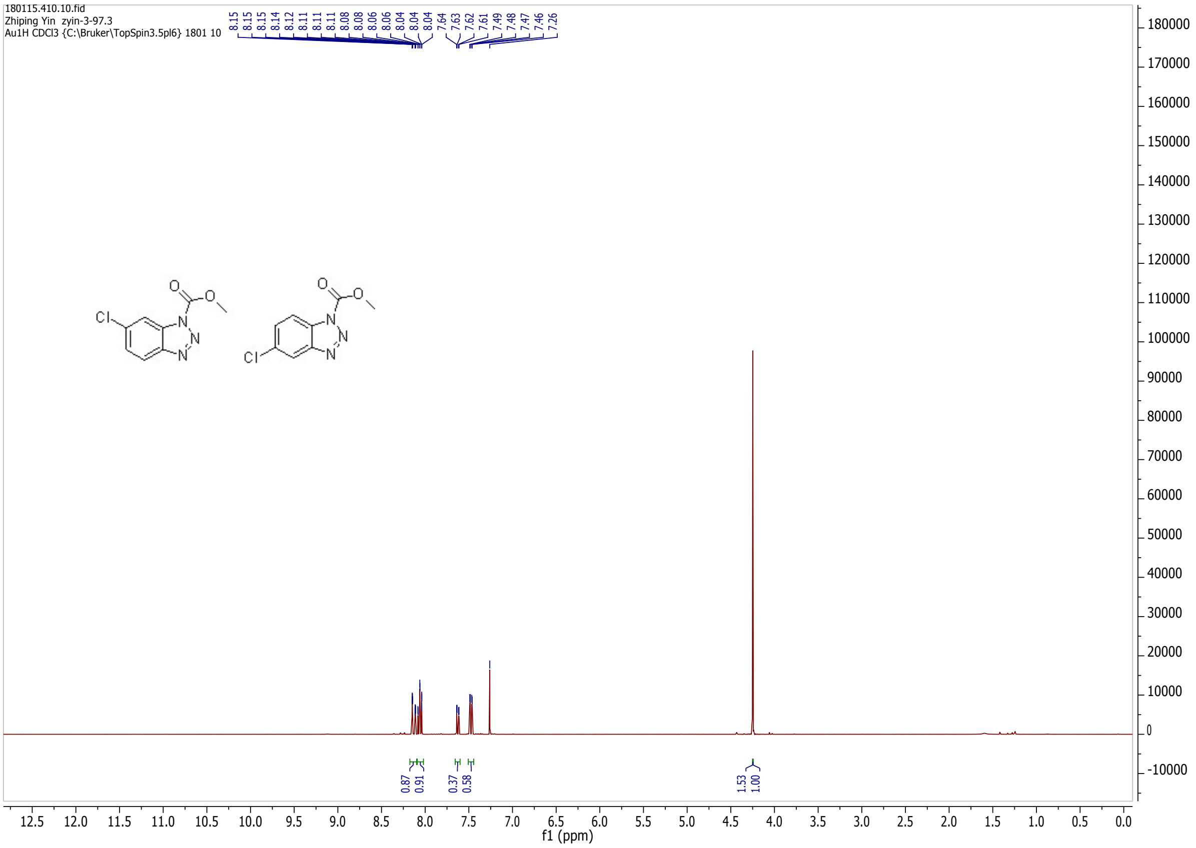

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8.11
8.08
8.08
8.06
8.06
8.04
8.04
8.04
7.64
7.63
7.62
7.61
7.49
7.48
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7.26
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170000
160000
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140000
130000
120000
110000
100000
90000
80000
70000
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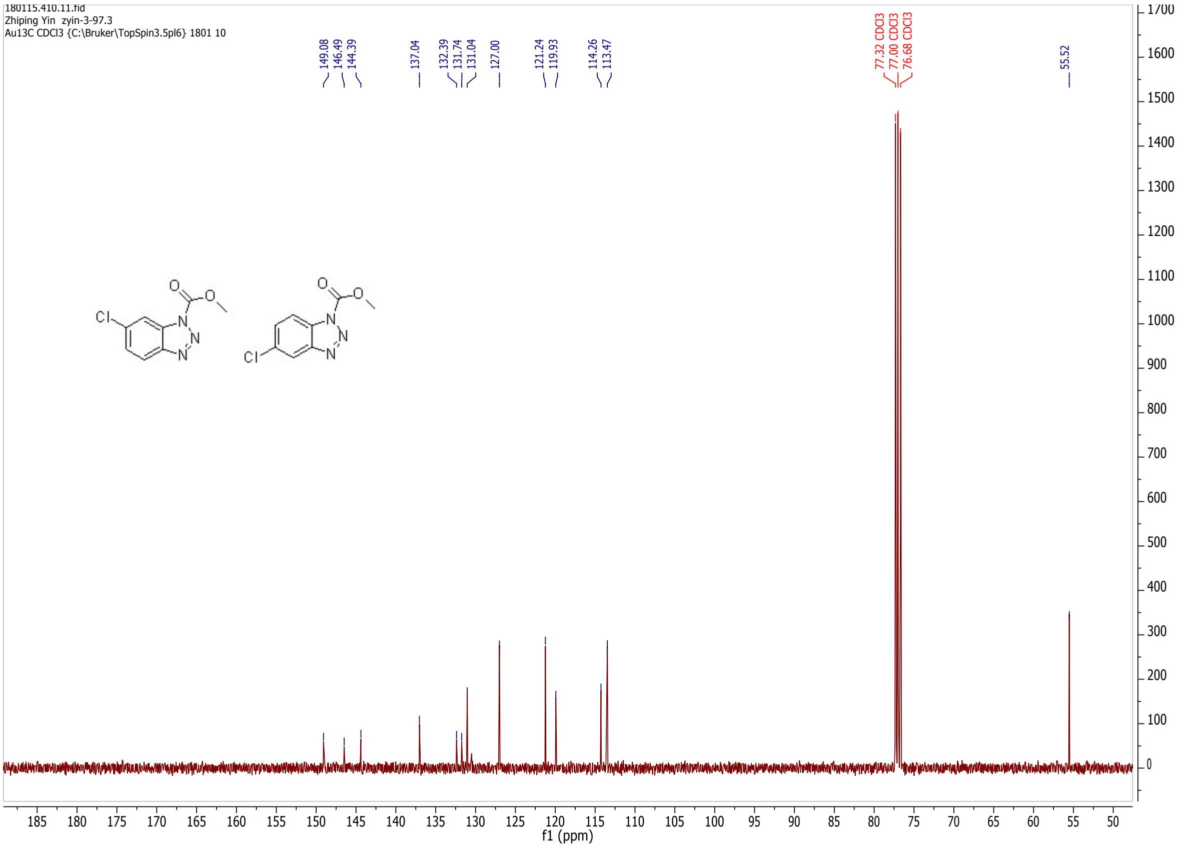
f1 (ppm)



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131.04
127.00
121.24
119.93
114.26
113.47
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.52
Cl
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1600
1500
1400
1300
1200
1100
1000
900
800
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0
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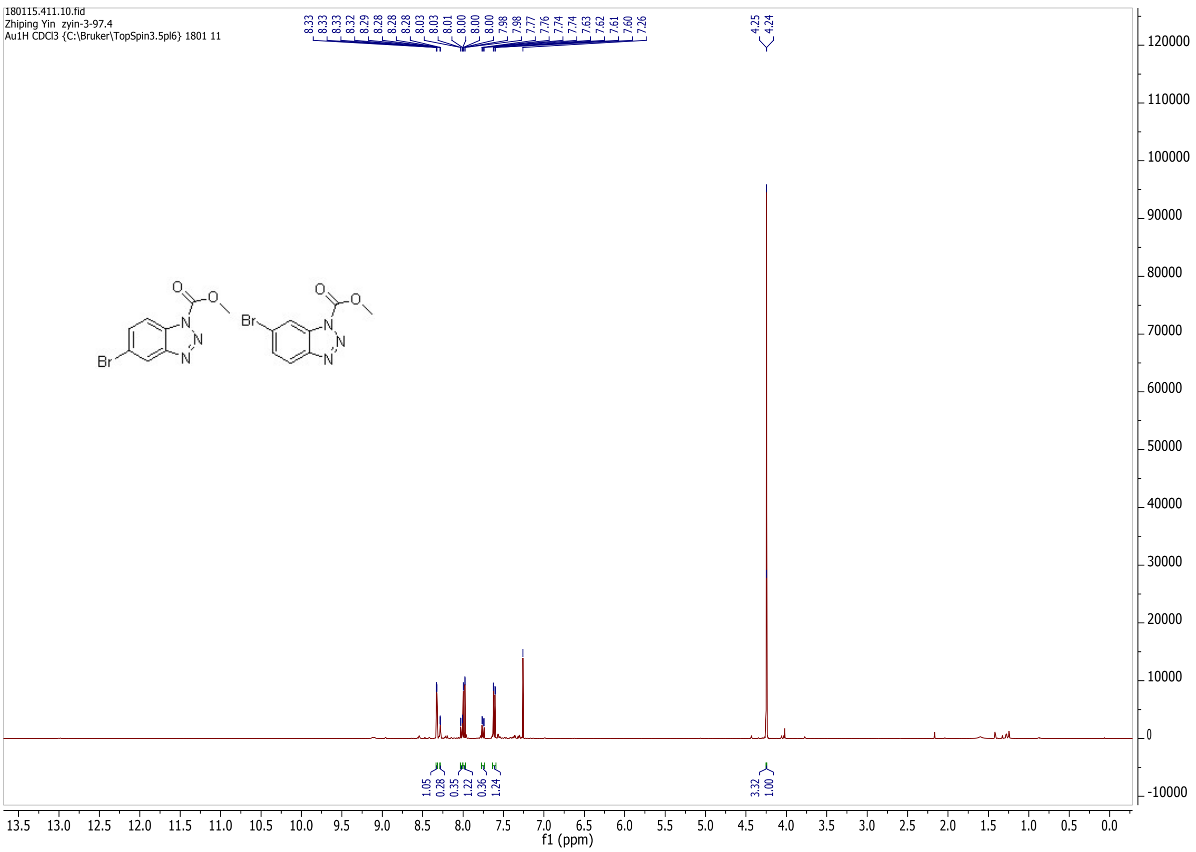
f1 (ppm)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8.01
8.00
8.00
8.00
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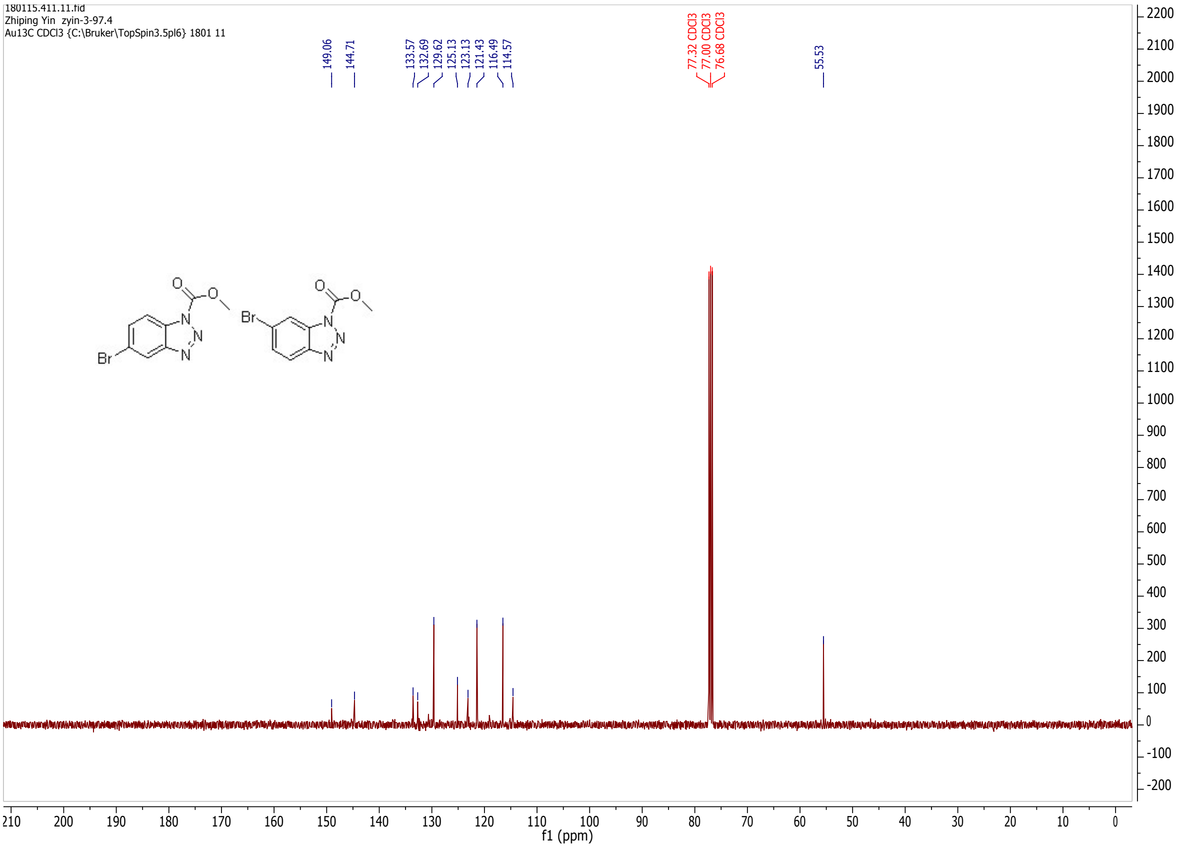
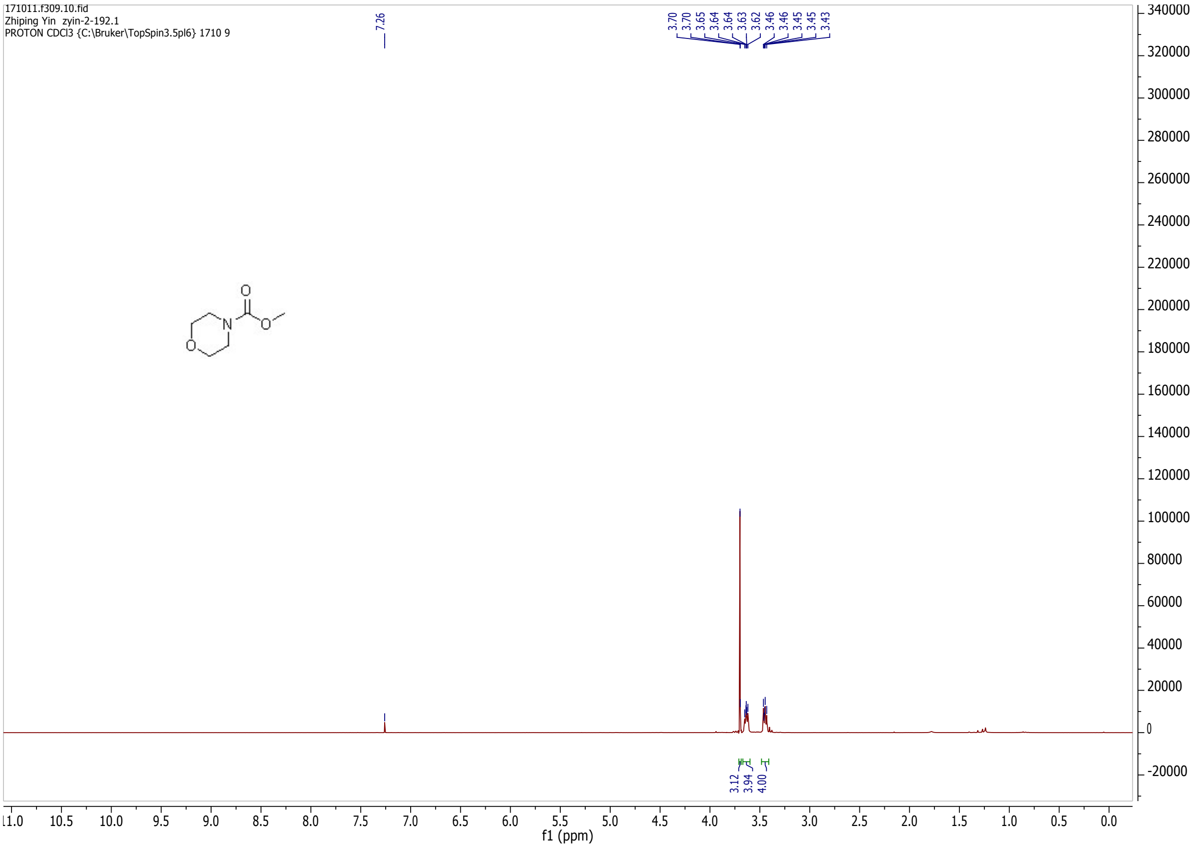
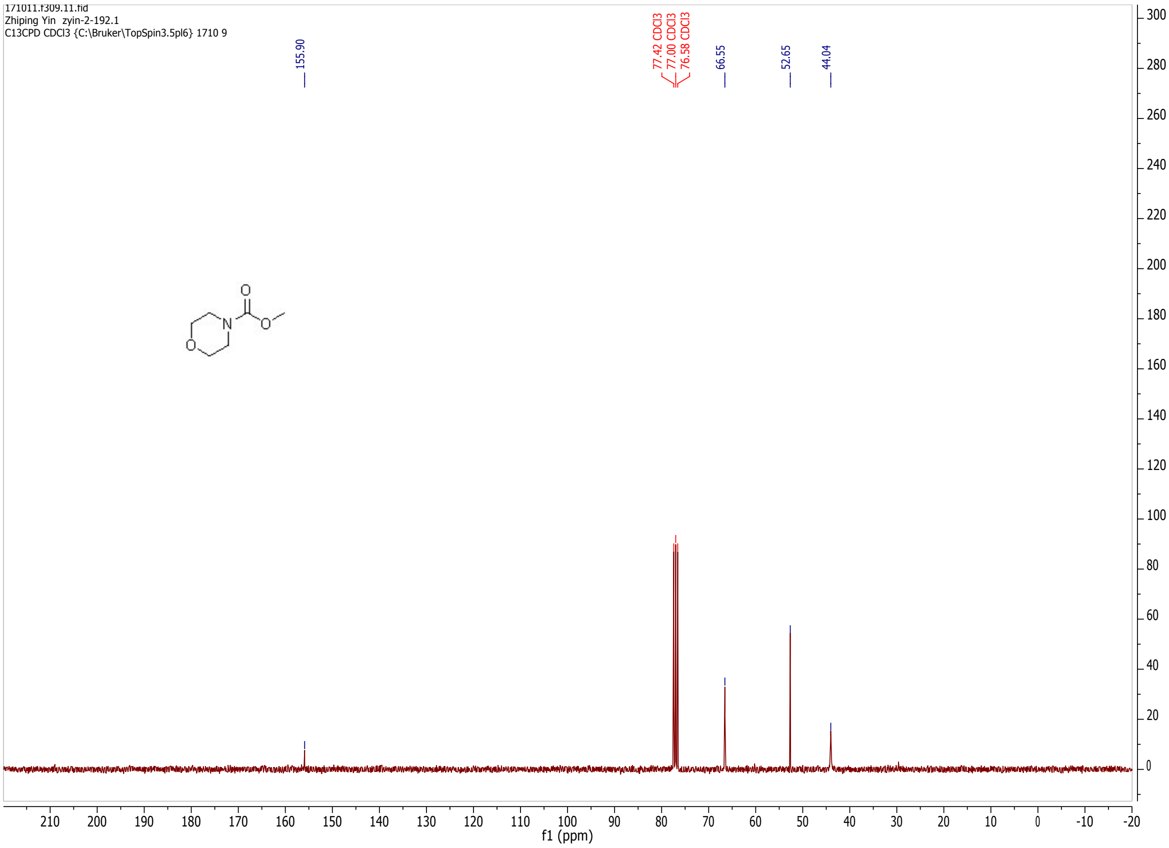
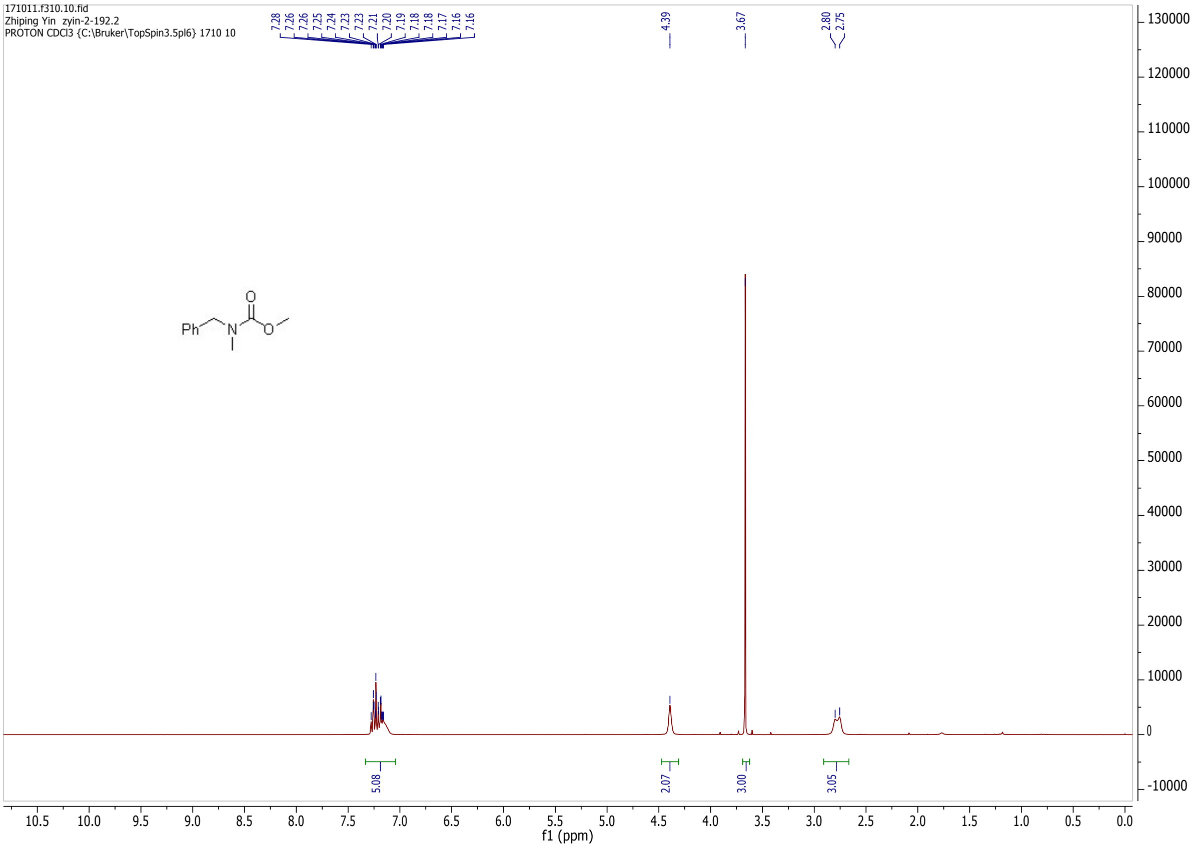
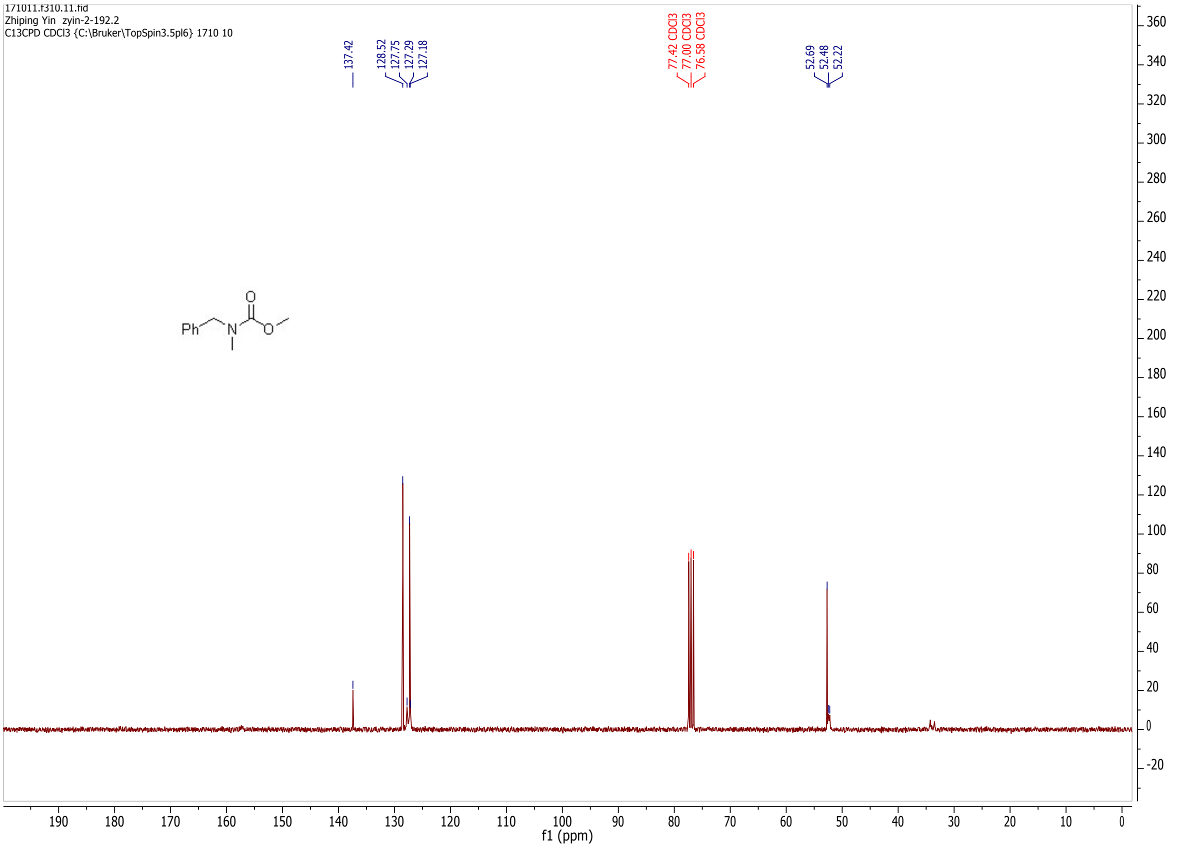
7.98
7.77
7.76
7.74
7.74
7.63
7.62
7.61
7.60
7.26
4.25
4.24
O
O
N
N
N
Br
O
O
Br
N
N
N
1.05
0.28
0.35
1.22
0.36
1.24
3.32
1.00
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
13.5
13.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

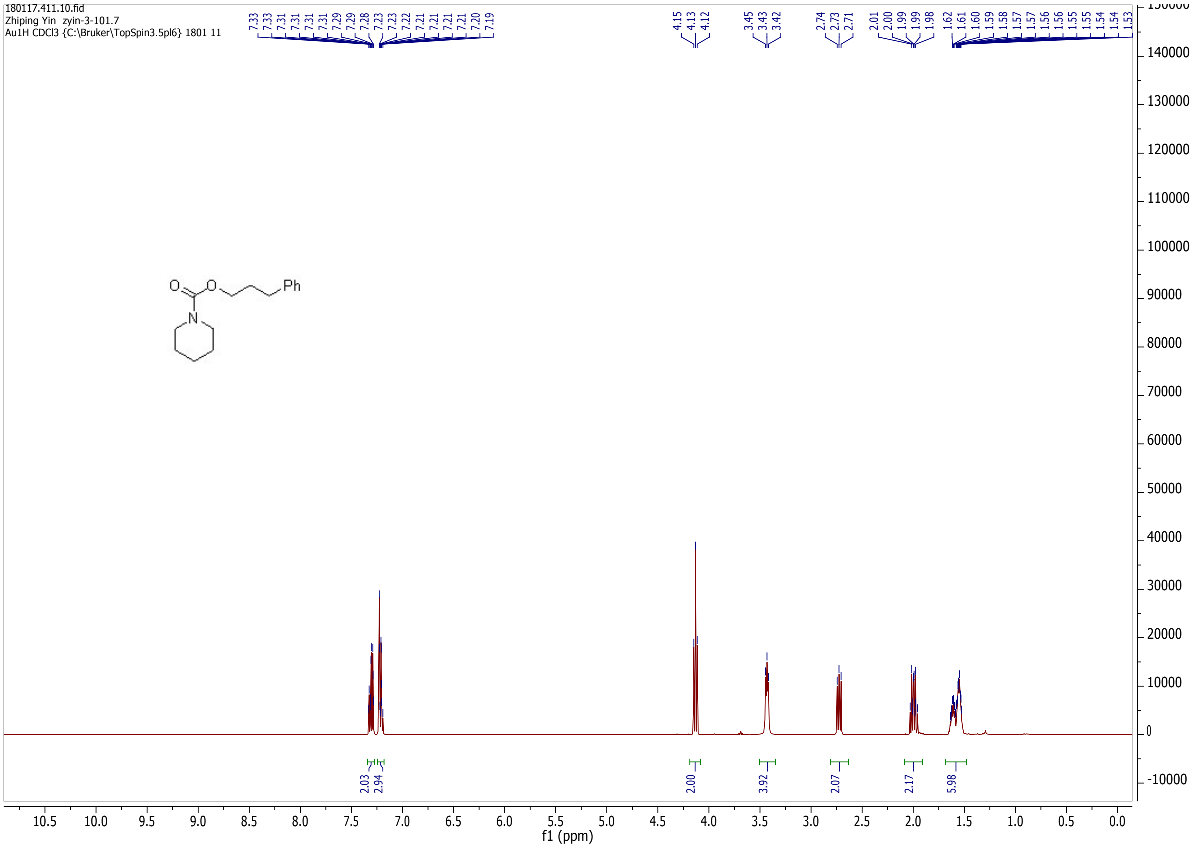

180115.411.11.fid
Zhiping Yin zyin-3-97.4
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 11
149.06
144.71
133.57
132.69
129.62
125.13
123.13
121.43
116.49
114.57
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.53
O
O
N
N
N
Br
O
O
Br
N
N
N
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

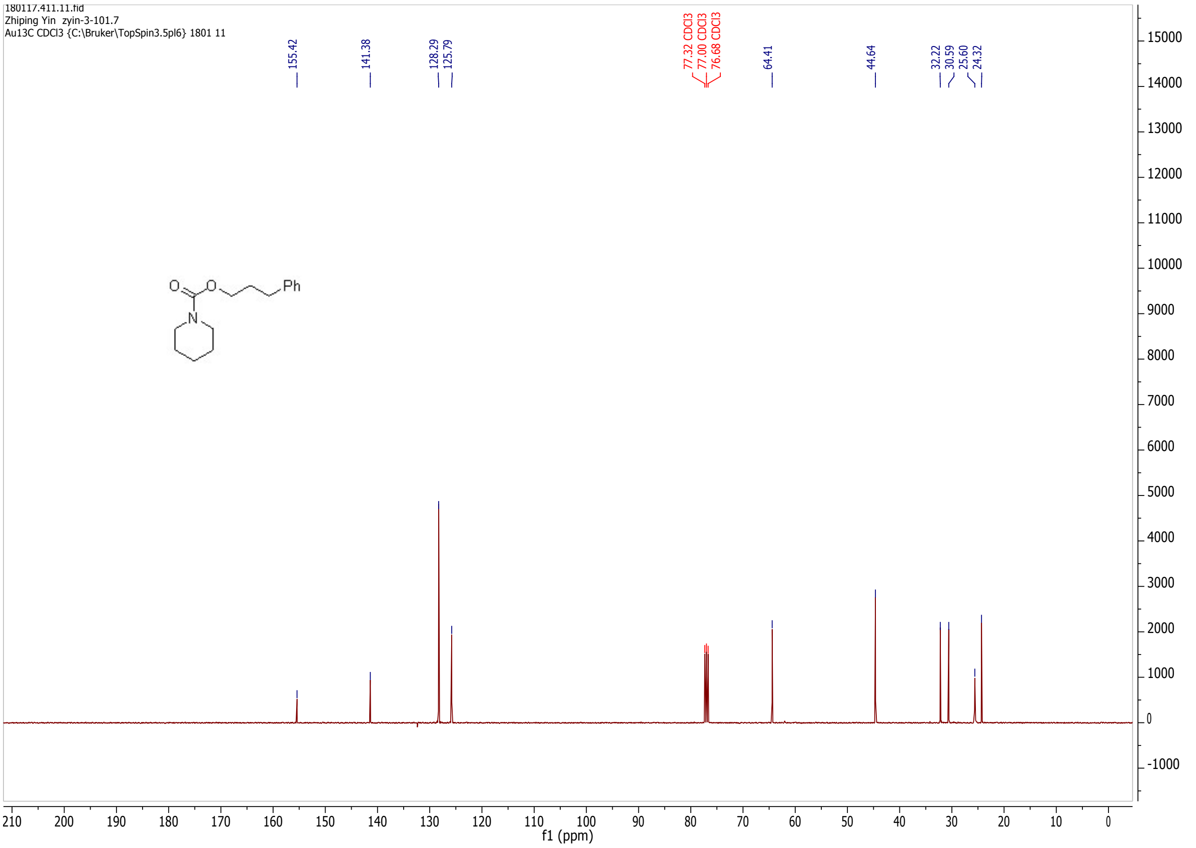

171011.f309.10.fid
Zhiping Yin zyin-2-192.1
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1710 9
7.26
3.70
3.70
3.65
3.64
3.64
3.63
3.62
3.46
3.46
3.45
3.45
3.43
O
N
O
O
3.12
3.94
4.00
340000
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
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)

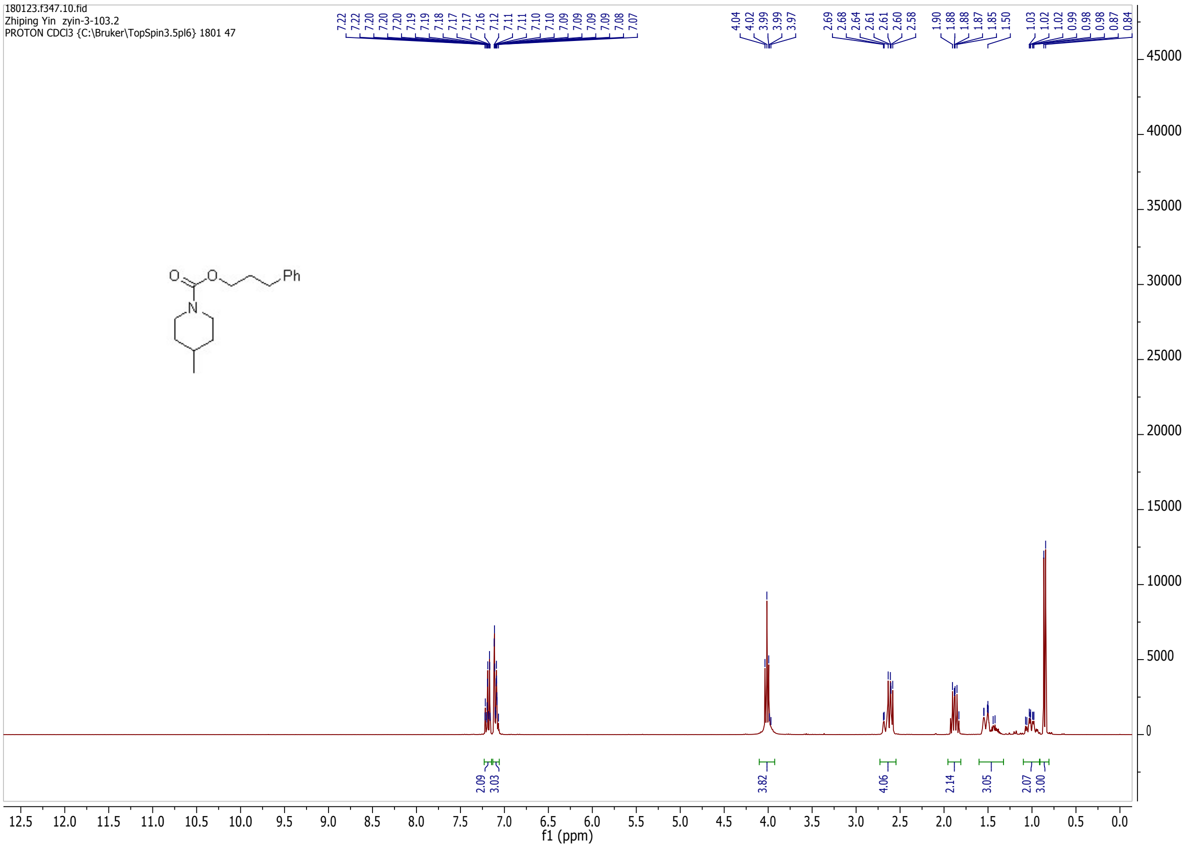

171011.f309.11.fid
Zhiping Yin zyin-2-192.1
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1710 9
155.90
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
66.55
52.65
44.04
O
N
O
O
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

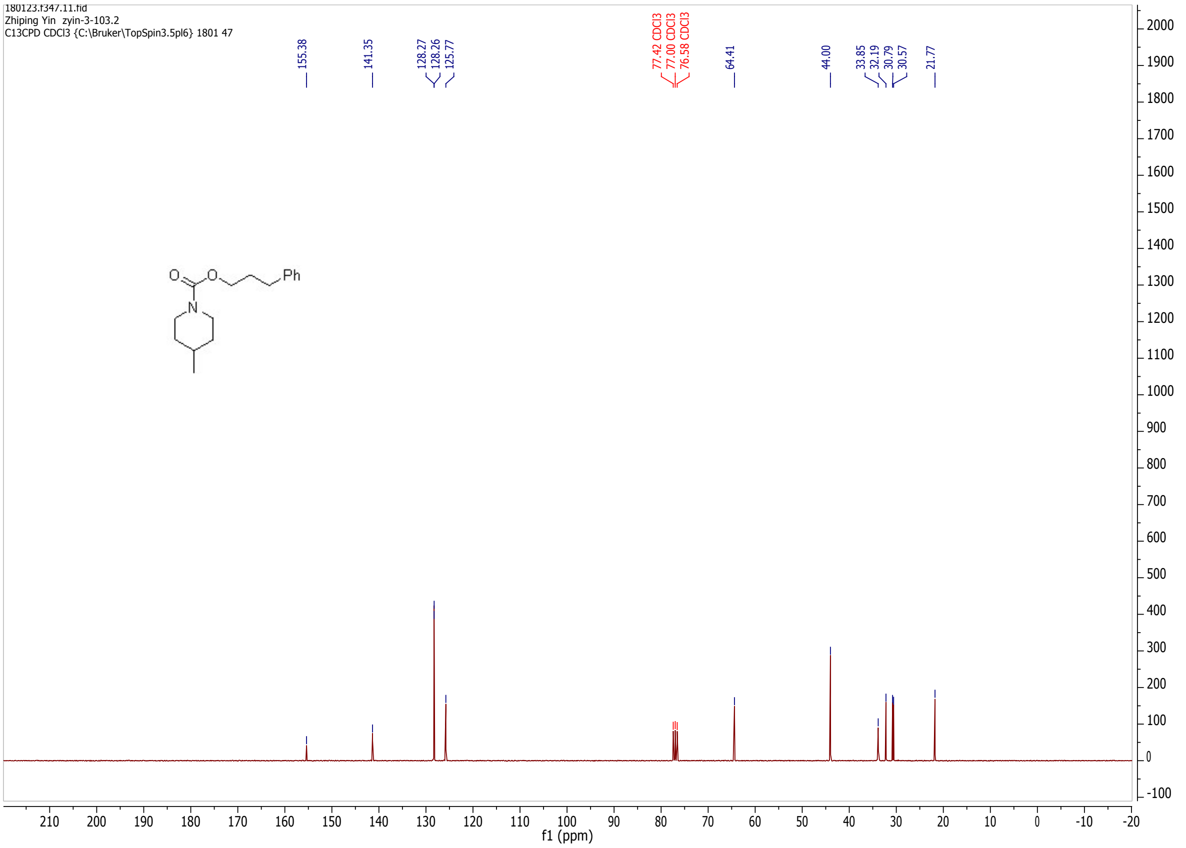

171011.f310.10.fid
Zhiping Yin zyin-2-192.2
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1710 10
7.28
7.26
7.26
7.25
7.24
7.23
7.23
7.21
7.20
7.19
7.18
7.18
7.17
7.16
7.16
4.39
3.67
2.80
2.75
Ph
N
O
O
5.08
2.07
3.00
3.05
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
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)

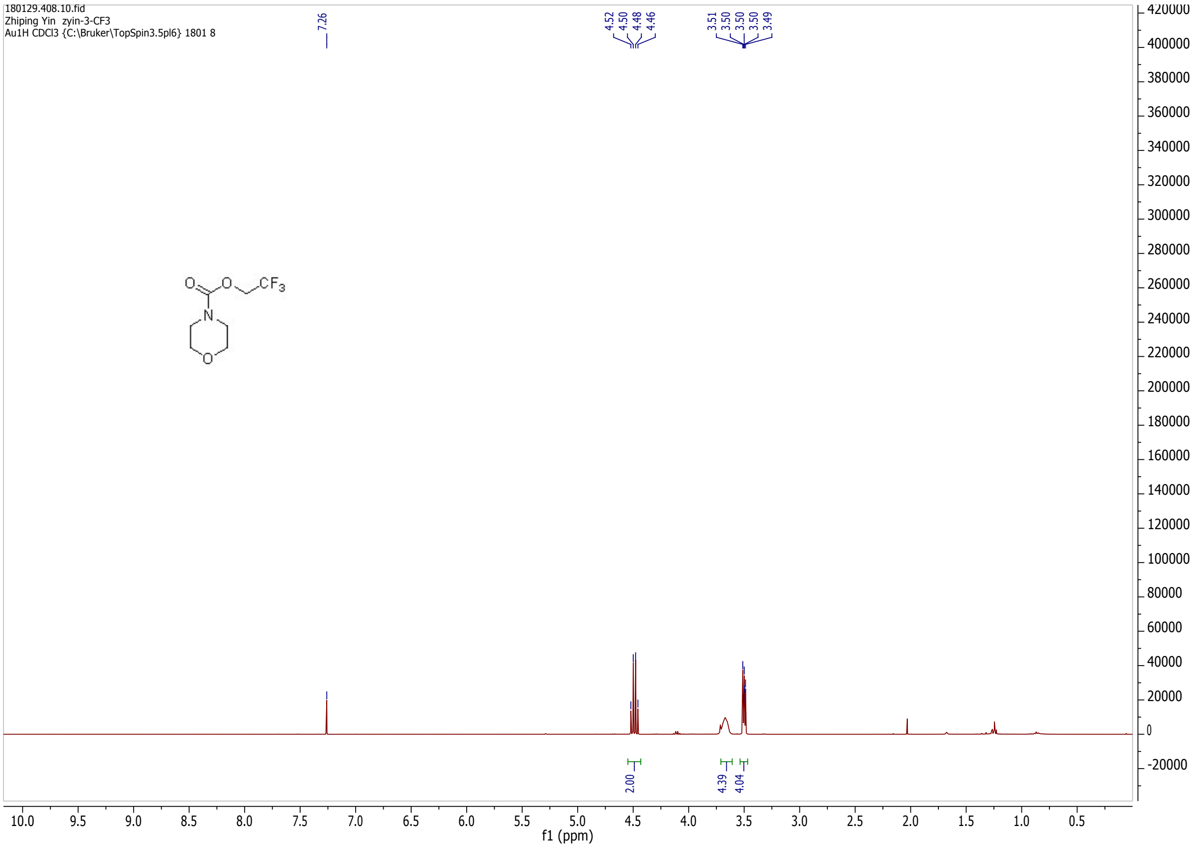

171011.f310.11.fid
Zhiping Yin zyin-2-192.2
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1710 10
137.42
128.52
127.75
127.29
127.18
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
52.69
52.48
52.22
Ph
N
O
O
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

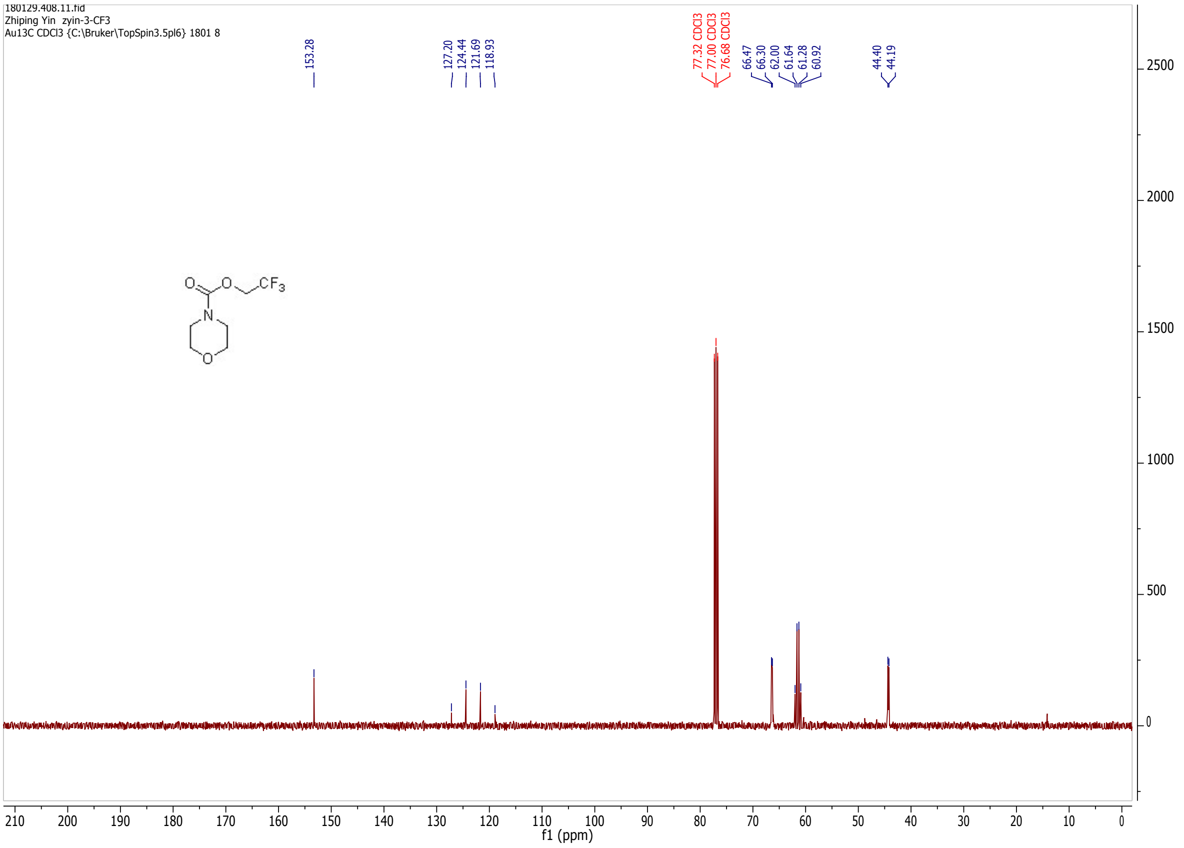

180117.411.10.fid
Zhiping Yin zyin-3-101.7
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 11
7.33 7.33 7.31 7.31 7.31 7.31 7.29 7.29 7.28 7.23 7.23 7.22 7.21 7.21 7.21 7.21 7.20 7.19
4.15 4.13 4.12
3.45 3.43 3.42
2.74 2.73 2.71
2.01 2.00 1.99 1.99 1.98
1.62 1.61 1.60 1.59 1.58 1.57 1.57 1.56 1.56 1.55 1.55 1.54 1.54 1.53
2.03 2.94 2.00 3.92 2.07 2.17 5.98
O
O
Ph
N
f1 (ppm)
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
150000 140000 130000 120000 110000 100000 90000 80000 70000 60000 50000 40000 30000 20000 10000 0 -10000

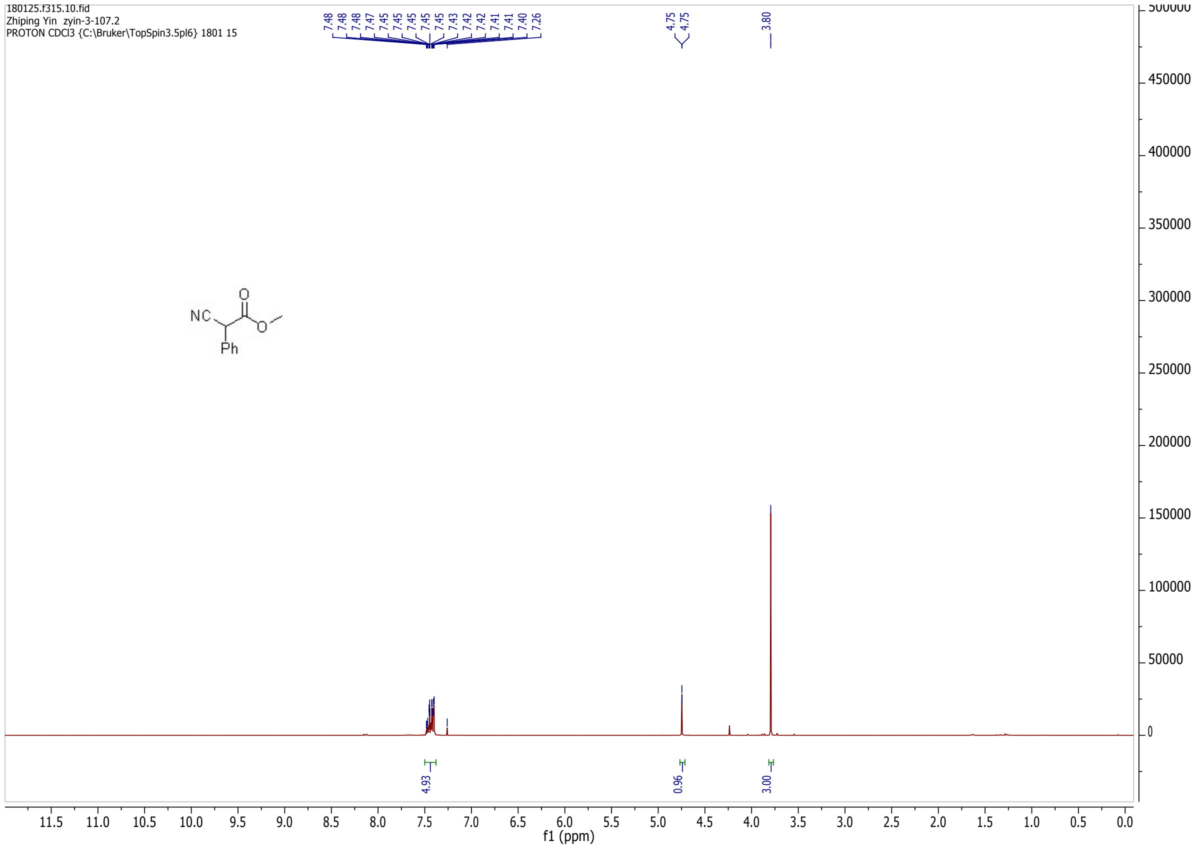

180117.411.11.fid
Zhiping Yin zyin-3-101.7
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 11
155.42
141.38
128.29 125.79
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3
64.41
44.64
32.22 30.59 25.60 24.32
O
O
Ph
N
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

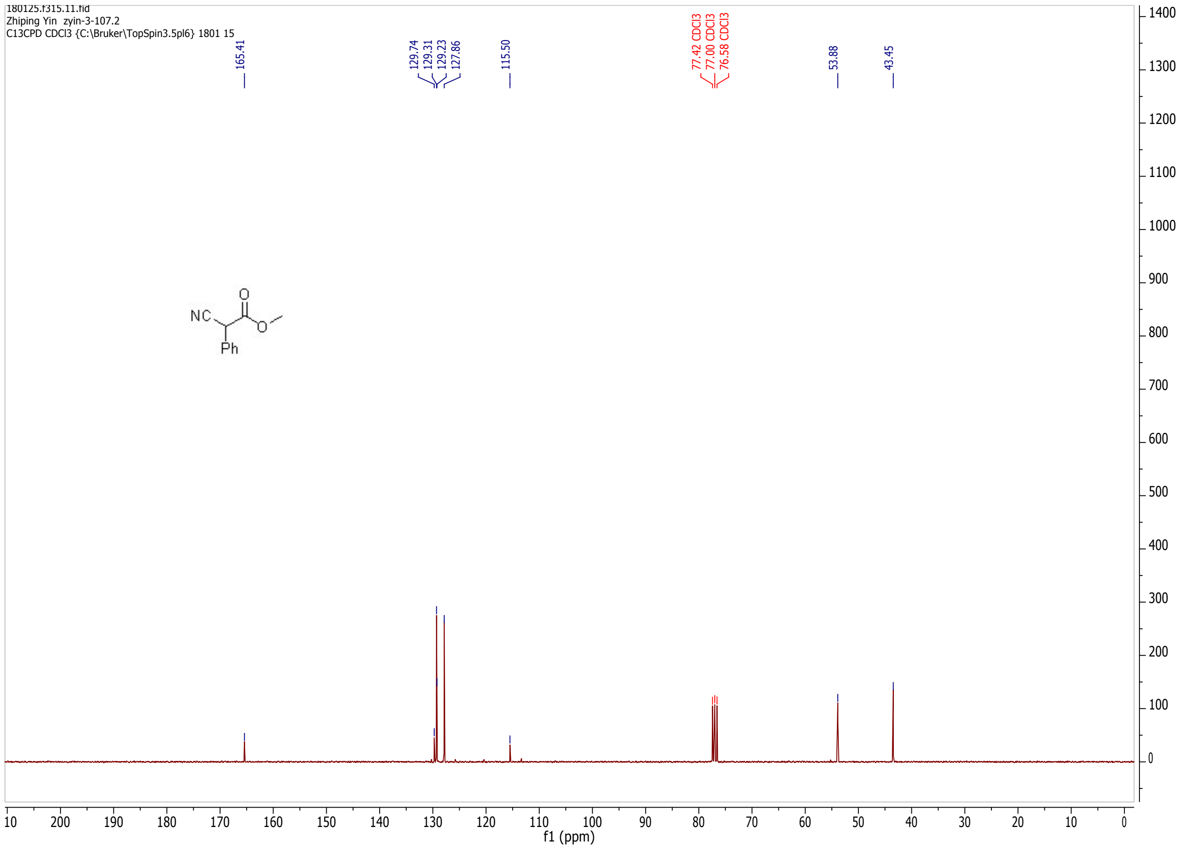

180123.f347.10.fid
Zhiping Yin zyin-3-103.2
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 47
7.22 7.22 7.20 7.20 7.20 7.19 7.19 7.18 7.17 7.17 7.16 7.12 7.11 7.11 7.10 7.10 7.09 7.09 7.09 7.09 7.08 7.07
4.04 4.02 3.99 3.99 3.97
2.69 2.68 2.64 2.61 2.61 2.60 2.58
1.90 1.88 1.88 1.87 1.85 1.50
1.03 1.02 1.02 0.99 0.98 0.98 0.87 0.84
O
O
Ph
N
2.09
3.03
3.82
4.06
2.14
3.05
2.07
3.00
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
12.5 12.0 11.5 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)


180123.f347.11.fid
Zhiping Yin zyin-3-103.2
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 47
155.38
141.35
128.27
128.26
125.77
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
64.41
44.00
33.85
32.19
30.79
30.57
21.77
O
O
Ph
N
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


180129.408.10.fid
Zhiping Yin zyin-3-CF3
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 8
7.26
4.52
4.50
4.48
4.46
3.51
3.50
3.50
3.50
3.49
O
O
CF3
N
O
2.00
4.39
4.04
420000
400000
380000
360000
340000
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
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
f1 (ppm)


180129.408.11.fid
Zhiping Yin zyin-3-CF3
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 8
153.28
127.20
124.44
121.69
118.93
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
66.47
66.30
62.00
61.64
61.28
60.92
44.40
44.19
O
O
CF3
N
O
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


180125.f315.10.fid
Zhiping Yin zyin-3-107.2
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 15
7.48
7.48
7.48
7.47
7.45
7.45
7.45
7.45
7.45
7.43
7.42
7.42
7.41
7.41
7.40
7.26
4.75
4.75
3.80
NC
O
O
Ph
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
4.93
0.96
3.00
11.5
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)


180125.f315.11.fid
Zhiping Yin zyin-3-107.2
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1801 15
165.41
129.74
129.31
129.23
127.86
115.50
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
53.88
43.45
NC
O
O
Ph
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)