Electronic Supplementary Material (ESI) for Green Chemistry.
This journal is © The Royal Society of Chemistry 2024

Supporting Information

Cobalt-nanoparticles catalysed *N*-alkylation of amides with alcohols

Rui Ma,[a] Jie Gao,[a] Lan Zhang,[b] Ning Wang,[b] Yue Hu,[a] Stephan Bartling,[a] Henrik Lund,[a] Sebastian Wohlrab,*[a] Rajenahally V. Jagadeesh,*[a,c] and Matthias Beller*[a]

[a] Leibniz-Institut für Katalyse e.V., Albert-Einstein-Str. 29a, 18059 Rostock, Germany.

[b] Faculty of Environment and Life, Beijing University of Technology, 100124 Beijing, China.

[c] Nanotechnology Centre, Centre for Energy and Environmental Technologies, VŠB-Technical University of Ostrava, Ostrava-Poruba, Czech Republic.

Table of Contents

1. Materials and methods

2. Preparation of catalysts

3. Screening of bases

4. Procedure for catalytic reactions

5. Characterization of Co-materials

6. NMR data

7. NMR spectra

8. References

1. Materials and methods

All the alcohols and amides were purchased from various chemical companies. Cobalt(II) nitrate hexahydrate (cat no.139267-100G) was provided by Sigma-Aldrich. Toluene solvent (analytic reagent grade) was obtained from Fischer scientific chemicals. Carbon powder, VULCAN® XC72R with Code XVC72R and CAS No. 1333-86-4 was obtained from Cabot Corporation Prod.

XRD powder pattern were recorded on a Panalytical X'Pert θ/2θ -diffractometer equipped with Xcelerator detector using automatic divergence slits and Cu kα1/α2 radiation (40 kV, 40 mA; λ= 0.15406 nm, 0.154443 nm). Cu beta-radiation was excluded using a nickel filter foil. Measurements were performed with $0.005°s^{-1}$. Finely crushed samples were mounted on silicon zero background holders. After data collection obtained intensities were converted from automatic to fixed divergence slits (0.25°) for further analysis. Peak positions and profile were fitted with Pseudo-Voigt function using the HighScore Plus software package (Panalytical). Phase identification was done by using the PDF-2 database of the International Center of Diffraction Data (ICDD).

XPS (X-ray Photoelectron Spectroscopy) measurements were performed on an ESCALAB 220iXL (Thermo Fisher Scientific) with monochromated Al Kα radiation (E = 1486.6 eV). Samples are prepared on a stainless-steel holder with conductive double-sided adhesive carbon tape. The electron binding energies were obtained with charge compensation using a flood electron source and referenced to the C 1s core level of adventitious carbon at 284.8 eV (C-C and C-H bonds). For quantitative analysis the peaks were deconvoluted with Gaussian-Lorentzian curves using the software Unifit 2021. The peak areas were normalized by the transmission function of the spectrometer and the element specific sensitivity factor of Scofield.

The surface area and porosity were carried out by the N_2 adsorption isotherm using the Brunauer-Emmett-Teller (BET) method on an ASAP 2020 Micromeritics instrument. Before analysis, all samples were degassed at 200 °C for 6 h to desorb moisture and impurities from their surfaces. The pore size distributions were calculated using the Barrett-Joyner-Halenda (BJH) model from the desorption branch.

TEM measurements were performed on a JEM-2100 operating at an accelerated voltage of 200 kV. The sample was ultrasonicated in ethanol solution, and a drop was deposited on a copper grid covered with a holey carbon membrane for observation.

GC analyses was performed on an Agilent 7890A instrument (Column: Agilent 19091J-413: 30 m × 320 μm × 0.25 μm, carrier gas: H2, FID detection.

All catalytic reactions were carried out in pressure tubes by placing them into a heated aluminum block.

2. Preparation of catalysts

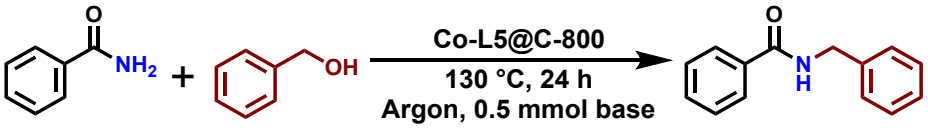
All catalysts mentioned in this work were prepared via the same procedure. A typical procedure for the preparation of the optimal catalyst (Co-**L5**@C-800) is as follows: In a round bottomed flask, 0.3 mmol of $Co(NO_3)_2 \cdot 6H_2O$, and 1 mmol of 1,2-phenylenediamine were stirred in methanol (100 mL) for 10 min at room temperature. Then, the carbon (Vulcan XC72R powder) support (2.0 g) without any treatment was added and the mixture was stirred overnight at RT (room temperature). After removing methanol by rotary evaporation at 45 °C, the resulting black solid material was grinded to give a fine powder, which was pyrolyzed under argon at 800 °C for 2 h. The cobalt mass loading in Co-**L5**@C-800 is found to be 0.937 wt% by ICP-OES analysis (Table 1). A series of catalysts were prepared through the same procedure by changing ligand, support, and calcination temperature. Different catalytic materials such as Co-**L1**@ZSM-5-800, Co-**L1**@TiO_2-800, Co-**L1**@TiO_2, Co-**L1**@Al_2O_3, Co-**L1**@MgO-Al_2O_3-800, Co @C-800, Co-**L1**@C-800, Co-**L2**@C-800, Co-**L3**@C-800, Co-**L4**@C-800, Co-**L5**@C-400, Co-**L5**@C-600 and Co-**L5**@C-1000, were prepared using the described protocol. The procedure, molar amounts or grams of reagents/components used in preparing these catalysts are as follows: 0.3 mmol of $Co(NO_3)_2 \cdot 6H_2O$, and 1 mmol of ligand were stirred in methanol (100 mL) for 10 min at room temperature. Then, the support (2.0 g) without any treatment was added and the mixture was stirred overnight at room temperature. After removing methanol by rotary evaporation at 45 °C, the resulting black solid material was grinded to give a fine powder, which was pyrolyzed under argon at a certain temperature for 2 h. Further, these obtained materials are used as catalysts.

3. Screening of bases and solvents

We screened many bases to optimize the reaction conditions (**Table S1**). It was clearly found that catalytic system without base is inactive toward the model reaction (**Table S1**, entry 1). The Na_2CO_3, K_2CO_3 and $NaHCO_3$ involved systems did not show any reactivity, too (**Table S1**, entries 2-4). The bases Cs_2CO_3, K_3PO_4, and NaOH exhibited lower to moderate product yields (**Table S1**, entries 5-7). KOH and t-BuOK showed similar yield of 96-97% (**Table S1**, entries 8-9). Due to the lower price of KOH, it was selected as the optimal base.

Next, we investigated the effect of KOH loading on the catalytic performance (**Figure S1**). The yield was gradually increased from 30% (0.05 mmol KOH) to 97% (0.3 mmol KOH). However, the yield could not be improved by further increasing the KOH loading. Thus, the optimal KOH loading is selected as 0.3 mmol.

Table S1. Screening the base for the *N*-alkylation of benzamide with benzyl alcohol.



Entry	Base type	Conversion, %	Yield, %
1	without base	0	0
2	Na_2CO_3	0	0
3	K_2CO_3	0	0
4	$NaHCO_3$	0	0
5	$CsCO_3$	88	87
6	K_3PO_4	68	67
7	NaOH	89	45
8	t-BuOK	97	96
9	KOH	98	97

Reaction conditions: 0.5 mmol benzamide, 0.55 mmol benzyl alcohol, 60 mg catalyst, 0.5 mmol base, 1 atm argon, 3 mL toluene, 130 ℃, 24 h. Yields were determined by GC based on benzamide using n-hexadecane as standard.

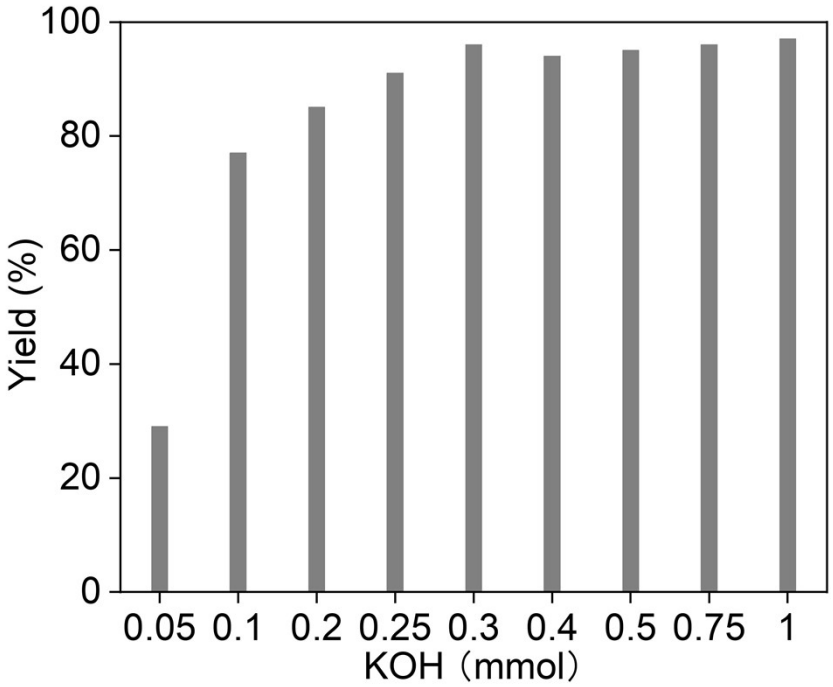
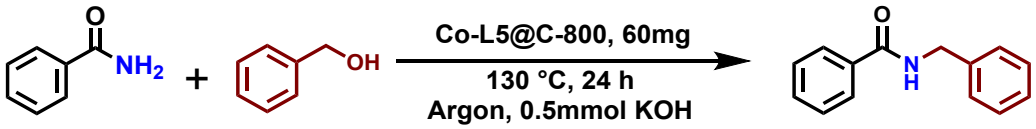


Figure S1. Screening the KOH loading for the *N*-alkylation of benzamide with benzyl alcohol. Reaction conditions: 0.5 mmol benzamide, 0.55 mmol benzyl alcohol, 60 mg catalyst, 1 atm argon, 3 mL toluene, 130 °C, 24 h. Yields were determined by GC based on benzamide using n-hexadecane as standard.

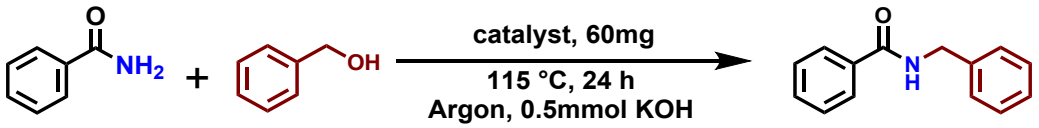
Table S2. Screening the solvents for the *N*-alkylation of benzamide with benzyl alcohol.



Entry	Solvent	Conversion, %	Yield, %
1	Tetrahydrofuran	16	15
2	Methanol	0	0
3	Ethanol	0	0
4	Toluene	98	97
5	n-hexane	35	34

Reaction conditions: 0.5 mmol benzamide, 0.55 mmol benzyl alcohol, 60 mg catalyst, 0.5 mmol KOH, 1 atm argon, 3 mL solvent, 130 °C, 24 h. Yields were determined by GC based on benzamide using n-hexadecane as standard.

Table S3. Screening the metal and ligand content.



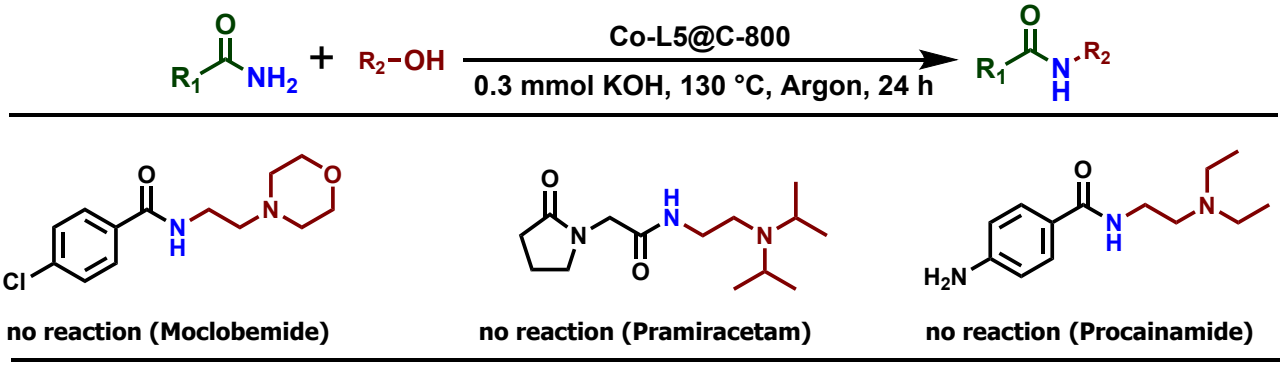
Entry	catalyst	Conversion, %	Yield, %
1	0.5Co-**L5**@C-800	82	82
2	Co-**L5**@C-800	87	87
3	1.5Co-**L5**@C-800	88	88
4	Co-2**L5**@C-800	86	86
5	Co-3.5**L5**@C-800	90	90

Reaction conditions: 0.5 mmol benzamide, 0.55 mmol benzyl alcohol, 60 mg catalyst, 0.5 mmol KOH, 1 atm argon, 3 mL solvent, 115 °C, 24 h. Yields were determined by GC based on benzamide using n-hexadecane as standard.

4. General procedure for catalytic reactions

A 25 mL pressure tube was charged with magnetic stirring bar, 0.5 mmol benzamide, 0.55 mmol benzyl alcohol. Then, 3 mL of toluene was added followed by the addition of catalyst (60 mg). Then, the pressure tube was flushed with argon gas three times and fitted with septum and cap. The pressure tube was placed into an aluminum block preheated at 135 °C (placed 30 minutes before counting the

reaction time in order to attain reaction temperature) and the reactions were stirred for required time. During the reaction the inside temperature of the pressure tube was measured to be 130 °C and this temperature was used as the reaction temperature. After the completion of the reactions, the pressure tubes were taken out and products were analyzed by GC and GCMS. The corresponding products were purified by column chromatography (silica, n-hexane, ethyl acetate, methanol). The purified products were analyzed by NMR (^{1}H, ^{13}C) and HRMS.



Scheme S1. Synthesis of selected drug molecules. Reaction conditions: 0.5mmol amide, 0.55 mmol alcohol, 60 mg Co-**L5**@C-800 (1.9 mol% Co), 0.3 mmol KOH, 3 mL toluene, 130 °C, 24 h, in 1 atm argon

Catalyst recycling. The procedure of catalyst recycling is as following: after the completion of each reaction, the catalyst was separated by centrifugation and washed with methanol followed by drying at 60 °C. Then, the catalyst was reused for the next run without reactivation.

5. Characterization of Co-materials

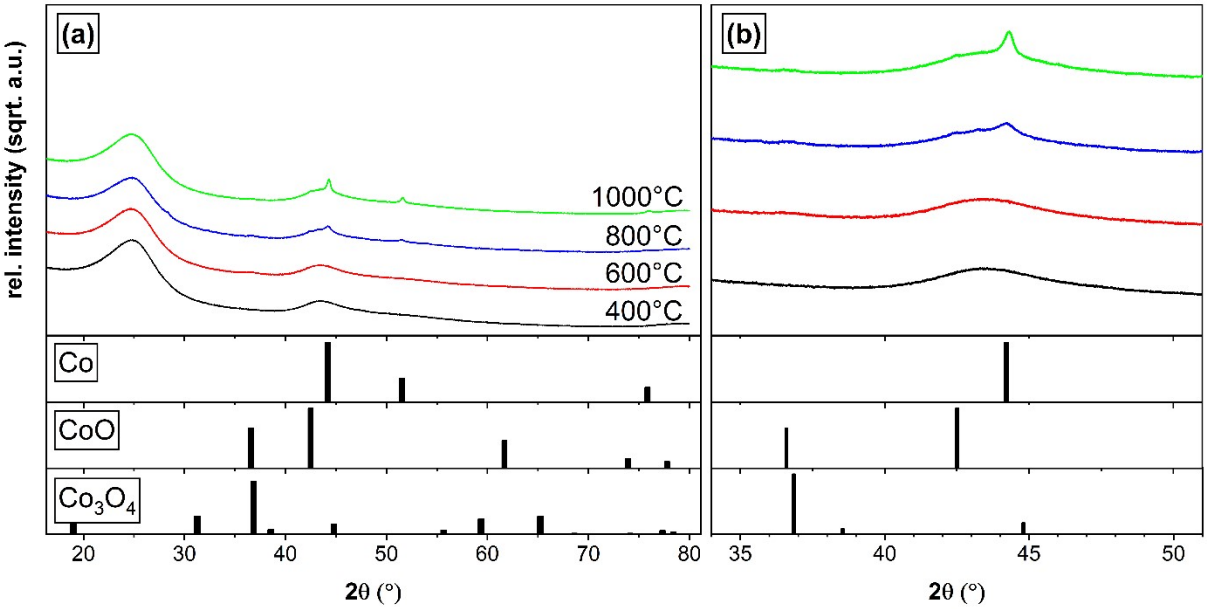


Figure S2. (a) Powder diffraction pattern of Co-**L5**@C-400, 600, 800 and 1000 and references, respectively (Co ICDD pdf 00-015-0806, CoO ICDD pdf 01-074-2391, Co_3O_4 ICDD pdf 00-024-1467); (b) magnification of angular region $34° \leq 2\theta \leq 51°$.

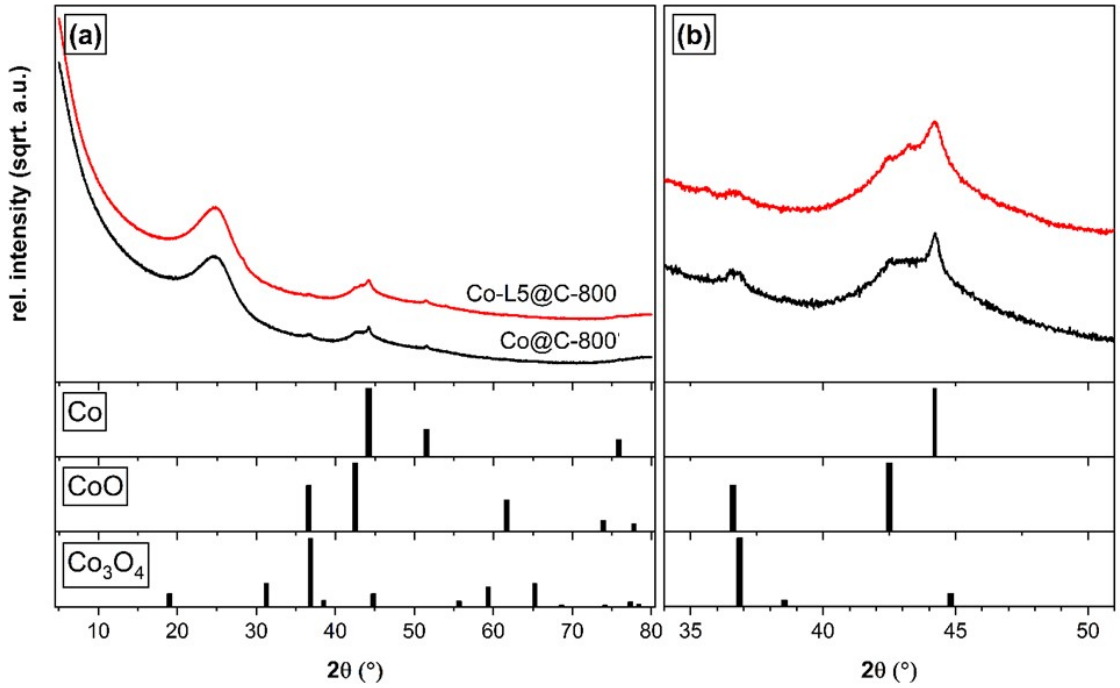


Figure S3. (a) Comparison of X-ray powder diffraction pattern of Co-**L5**@C-800, Co@C-800 and references, respectively (Co ICDD pdf 00-015-0806, CoO ICDD pdf 01-074-2391, Co_3O_4 ICDD pdf 00-024-1467); (b) magnification of angular region $32° \leq 2\theta \leq 53°$.

Table S4. Elemental distribution of the Co@C-800 derived by EDX.

Element	Family	Atomic Fraction (%)	Mass Fraction (%)
C	k	98.9	97.5
N	k	0.0	0.0
O	k	0.9	1.1
Co	k	0.3	1.4

Table S5. Elemental distribution of the Co-**L5**@C-800 derived by EDX.

Element	Family	Atomic Fraction (%)	Mass Fraction (%)
C	k	97.6	93.7
N	k	0.0	0.0
O	k	1.5	1.9
Co	k	0.9	4.4

Table S6. Elemental composition of the near-surface region (at %) derived by XPS.

Element	Co-L5@C-400	Co-L5@C-600	Co-L5@C-800	Co-L5@C-1000	Co@C-800
C	96.8	97.9	97.3	98.8	98.3
N	1.2	0.7	0.4	0.1	0.0
Co	0.3	0.2	0.1	0.1	0.1
O	1.2	0.8	1.6	0.6	1.0
S	0.2	0.1	0.2	0.2	0.2
Si	0.2	0.3	0.3	0.4	0.4

Table S7. The proportion of different forms of cobalt on the near-surface region (%) derived by XPS.

Co form	Co-L5@C-400	Co-L5@C-600	Co-L5@C-800	Co-L5@C-1000	Co@C-800
Co^0	0	0	12.0	22.7	0
Co_xO_y	100	100	88.0	77.3	100

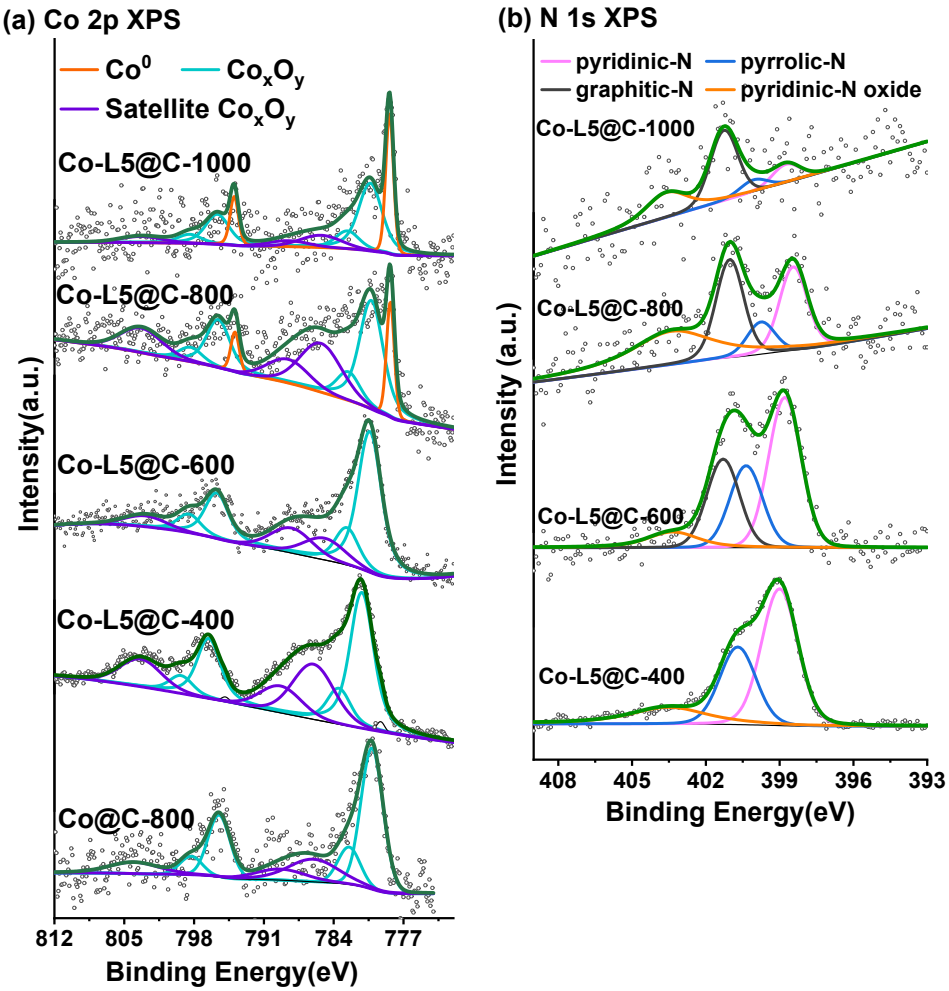


Figure S4. (a) Co 2p XPS and (b) N 1s XPS spectra of selected materials.

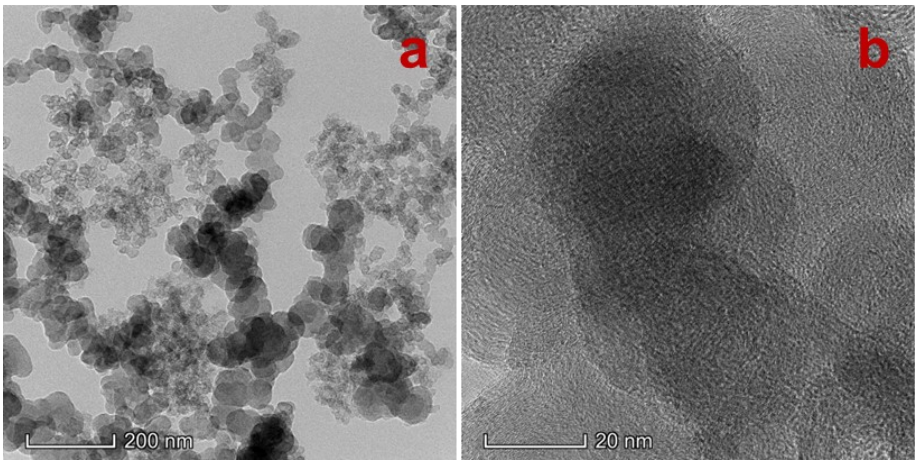


Figure S5. Structure characterization of Co-L5@C-400. (a-b) TEM images, (a) shows the metal complexes were uniformly distributed on the carbon support, (b) magnification TEM image at 20 nm.

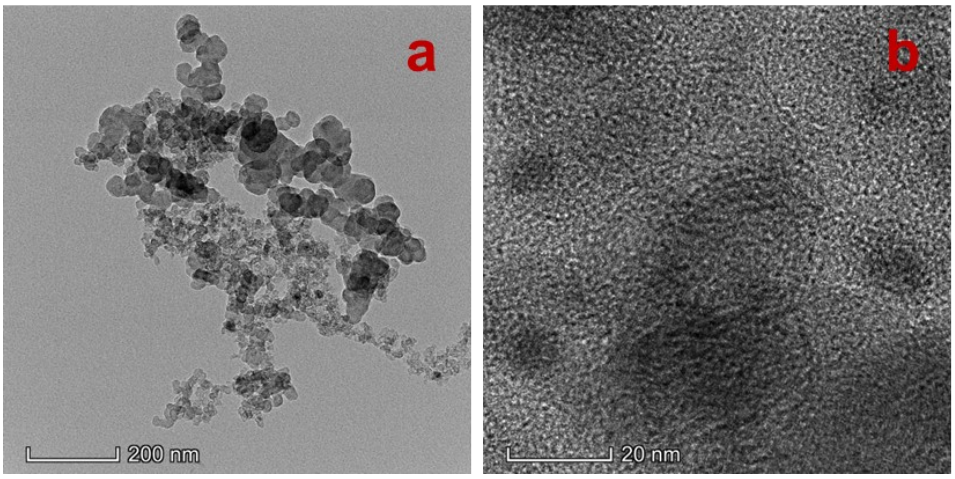


Figure S6. Structure characterization of Co-**L5**@C-600. (a-b) TEM images show (a) the metal complexes were uniformly distributed on the carbon support, (b) magnification TEM image at 20 nm.

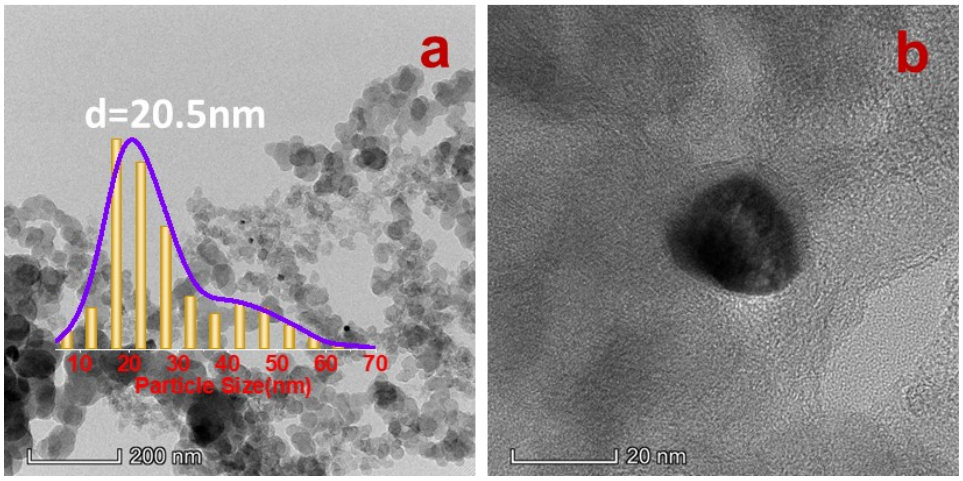


Figure S7. Structure characterization of Co-**L5**@C-1000. (a-b) TEM images, (a) shows the Co nanoparticles were distributed on the carbon support with an average diameter of 20.5 nm, (b) magnification TEM image at 20nm.

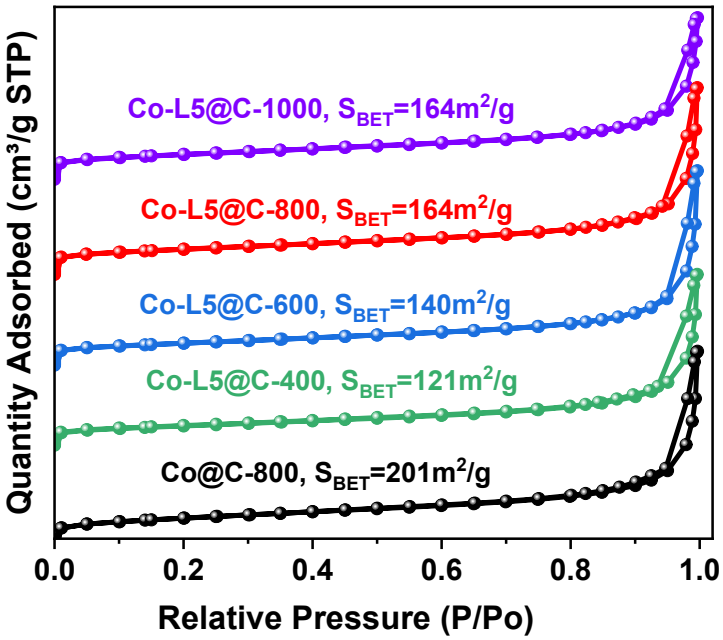


Figure S8. BET spectra of selected materials.

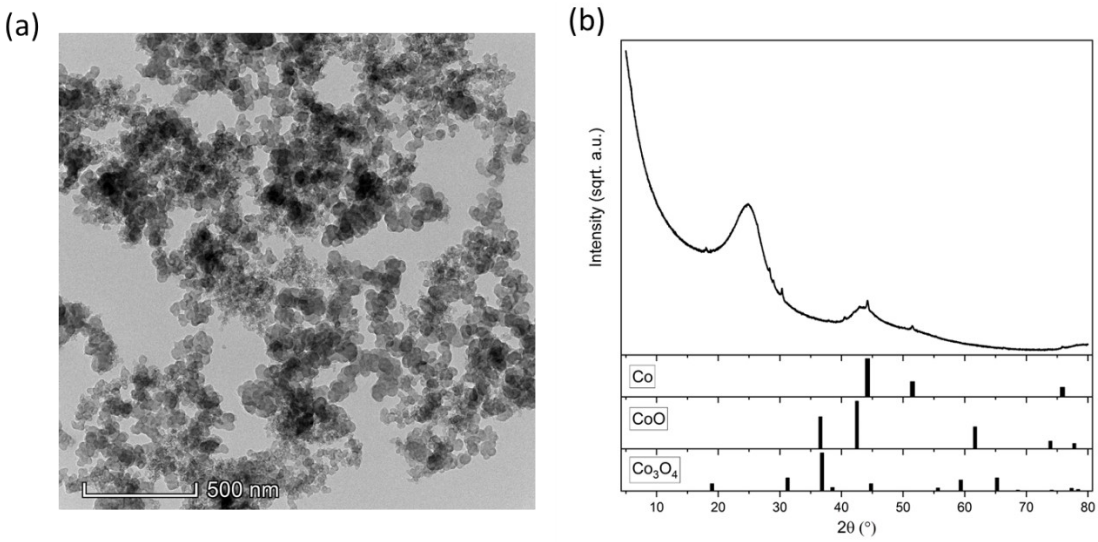


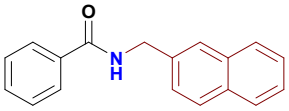
Figure S9. Characterization of catalyst Co-**L5**@C-800 after recycling. (a) TEM image, (b) Comparison of X-ray powder diffraction pattern of Co-**L5**@C-800, Co@C-800 and references, respectively (Co ICDD pdf 00-015-0806, CoO ICDD pdf 01-074-2391, Co_3O_4 ICDD pdf 00-024-1467).

6. NMR data

NMR spectra were recorded on Bruker AV 300 and 400 spectrometers. All chemical shifts (δ) are reported in parts per million (ppm) and coupling constants (*J*) in Hz. Mass spectra were recorded on an AMD 402/3 or a HP 5973 mass selective detector.
HRMS data were recorded on (1) ESI-HRMS: HPLC System 1200 /ESI-TOF-MS 6210 (Agilent) and (2) EI-HRMS: Mass Spectrometer MAT 95XP (Thermo Electron), 70eV. GC-MS was performed on an ISQ Trace 1300 in the electron ionization (EI) mode.

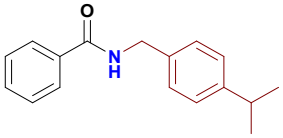
Isolated yields are reported. In the following, chemical shifts of the products are given.

1a: N-(naphthalen-2-ylmethyl)benzamide[1]



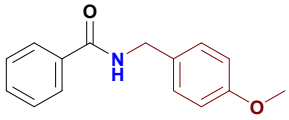
^{1}H NMR (300 MHz, $CDCl_3$) *δ* 7.87 – 7.77 (m, 6H), 7.54 – 7.39 (m, 6H), 6.75 (s, 1H), 4.79 (dd, *J* = 5.8, 0.8 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** *δ* 167.50, 135.70, 134.37, 133.40, 132.82, 131.60, 128.62, 127.78, 127.73, 127.06, 126.52, 126.33, 126.00, 77.52, 77.10, 76.67, 44.25.

1b: N-(4-isopropylbenzyl)benzamide[2]



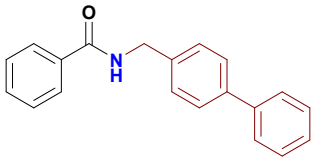
^{1}H NMR (300 MHz, DMSO) δ 9.03 (t, *J* = 5.9 Hz, 1H), 7.99 – 7.88 (m, 2H), 7.58 – 7.42 (m, 3H), 7.29 – 7.23 (m, 2H), 7.22 – 7.17 (m, 2H), 4.48 (dd, *J* = 5.8, 1.7 Hz, 2H), 2.86 (pd, *J* = 7.0, 2.1 Hz, 1H), 1.20 (d, *J* = 2.6 Hz, 3H), 1.18 (d, *J* = 2.6 Hz, 3H**). ^{13}C NMR (75 MHz, DMSO)** *δ* 166.61, 147.36, 147.24, 137.57, 134.89, 131.64, 128.74, 127.80, 127.73, 127.07, 126.62, 126.38, 42.90, 40.83, 40.55, 40.27, 39.99, 39.71, 39.44, 39.16, 33.62, 24.45, 24.40.

1c: N-(4-methoxybenzyl)benzamide[2]



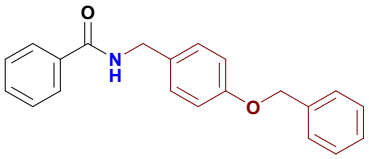
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 – 7.64 (m, 2H), 7.42 – 7.34 (m, 1H), 7.18 – 7.13 (m, 2H), 6.79 – 6.74 (m, 2H), 6.59 (s, 1H), 4.44 (d, J = 5.6 Hz, 2H), 3.69 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.37, 159.06, 134.46, 131.48, 130.38, 129.27, 128.62, 128.54, 127.02, 114.12, 113.90, 77.54, 77.12, 76.69, 55.32, 43.57.

1d: N-(4-phenylbenzyl)benzamide[3]



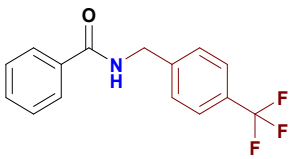
^{1}H NMR (300 MHz, DMSO) δ 9.12 (t, J = 6.0 Hz, 1H), 8.01 – 7.89 (m, 2H), 7.68 – 7.61 (m, 4H), 7.59 – 7.29 (m, 8H), 4.55 (d, J = 6.2 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.71, 140.48, 139.44, 139.19, 139.03, 134.80, 131.74, 129.38, 128.81, 128.33, 127.78, 127.74, 127.51, 127.10, 127.05, 127.02, 126.86, 42.85, 40.82, 40.54, 40.26, 39.98, 39.70, 39.43, 39.15.

1e: N-(4-(benzyloxy)benzyl)benzamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 – 7.75 (m, 1H), 7.58 – 7.21 (m, 9H), 7.02 – 6.94 (m, 2H), 6.57 (s, 1H), 5.09 (d, J = 2.7 Hz, 2H), 4.66 – 4.54 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.40, 158.37, 158.31, 137.00, 136.93, 134.42, 131.54, 130.59, 129.34, 128.67, 128.64, 128.59, 128.03, 128.00, 127.48, 127.01, 115.38, 115.14, 114.93, 77.53, 77.11, 76.68, 70.07, 70.05, 43.63.
HRMS (ESI-TOF): *m/z* calcd. for $C_{21}H_{19}NO_2$+H^+ ([M+H^+]) 318.1494, found 318.1502.

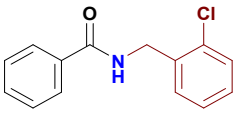
1f: N-(4-(trifluoromethyl)benzyl)benzamide[3]



^{1}H NMR (300 MHz, DMSO) δ 8.94 (t, J = 5.8 Hz, 1H), 8.01 – 7.87 (m, 2H), 7.58 – 7.43 (m, 3H), 7.30 – 7.23 (m, 1H), 7.21 – 7.12 (m, 3H), 4.49 (d, J = 5.9 Hz, 2H**). ^{13}C NMR (75 MHz, DMSO)** δ

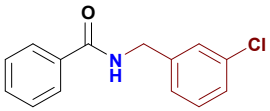
166.69, 137.65, 135.95, 134.87, 131.67, 130.35, 128.77, 127.78, 127.75, 127.20, 126.19, 41.17, 40.83, 40.55, 40.28, 40.00, 39.72, 39.44, 39.16.

1g: N-(2-chlorobenzyl)benzamide[4]



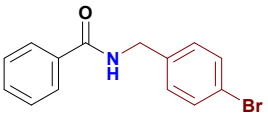
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 2H), 7.53 – 7.29 (m, 6H), 7.25 – 7.22 (m, 1H), 6.88 (d, J = 59.5 Hz, 1H), 4.67 (dd, J = 34.3, 5.8 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 167.51, 167.49, 138.30, 135.66, 134.38, 134.25, 133.62, 131.61, 131.53, 130.16, 129.55, 128.95, 128.75, 128.59, 128.56, 127.87, 127.55, 127.14, 127.07, 127.05, 77.44, 77.32, 77.12, 76.80, 44.07, 41.99.

1h: N-(3-chlorobenzyl)benzamide[5]



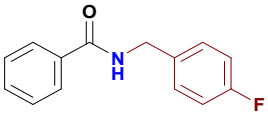
^{1}H NMR (300 MHz, DMSO) δ 9.05 (t, J = 6.2 Hz, 1H), 7.94 – 7.87 (m, 2H), 7.63 – 7.44 (m, 4H), 7.34 – 7.31 (m, 3H), 4.58 – 4.45 (m, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 140.17, 131.71, 128.79, 128.75, 127.70, 127.65, 127.19, 43.05, 40.81, 40.53, 40.25, 39.97, 39.70, 39.42, 39.14.

1j: N-(4-bromobenzyl)benzamide[1]



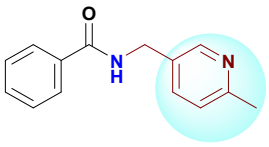
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 – 7.47 (m, 4H), 7.37 (dddd, J = 8.3, 3.8, 2.8, 0.9 Hz, 4H), 7.31 – 7.12 (m, 1H), 4.68 (d, J = 7.1 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 140.68, 139.89, 128.81, 127.49, 127.35, 127.12, 77.46, 77.04, 76.62, 65.43, 65.14, 29.73.

1k: N-(4-fluorobenzyl)benzamide[6]



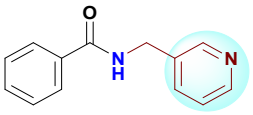
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.77 (m, 2H), 7.54 – 7.48 (m, 1H), 7.45 – 7.40 (m, 2H), 7.28 (s, 2H), 7.11 – 6.94 (m, 2H), 4.62 – 4.54 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 167.48, 167.40, 163.74, 163.42, 161.29, 160.98, 134.41, 134.26, 134.17, 134.13, 131.63, 131.54, 129.57, 129.49, 129.37, 129.29, 128.60, 128.58, 127.03, 126.99, 115.66, 115.63, 115.45, 115.42, 115.09, 77.41, 77.09, 76.77, 43.32.

1m: N-((6-methylpyridin-3-yl)methyl)benzamide[8]



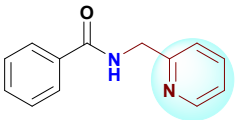
^{1}H NMR (300 MHz, DMSO) δ 9.08 (t, J = 5.9 Hz, 1H), 8.43 (dd, J = 2.3, 0.8 Hz, 1H), 7.93 – 7.86 (m, 2H), 7.64 – 7.60 (m, 1H), 7.56 – 7.43 (m, 3H), 7.20 (d, J = 7.9 Hz, 1H), 4.47 (d, J = 5.9 Hz, 2H), 2.44 (d, J = 1.9 Hz, 3H). **^{13}C NMR (75 MHz, DMSO)** δ 166.77, 156.78, 156.69, 148.61, 136.00, 134.66, 132.50, 131.76, 128.79, 127.70, 123.16, 122.99, 40.81, 40.66, 40.53, 40.25, 39.97, 39.70, 39.42, 39.14, 24.15, 24.11.

1n: N-(pyridin-3-ylmethyl)benzamide[9]



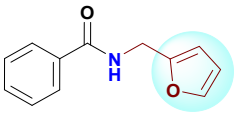
^{1}H NMR (300 MHz, DMSO) δ 9.26 – 9.07 (m, 1H), 8.60 – 8.44 (m, 2H), 8.06 – 7.86 (m, 2H), 7.81 – 7.67 (m, 1H), 7.57 – 7.31 (m, 4H), 4.58 – 4.48 (m, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.85, 151.17, 151.12, 149.31, 148.58, 148.51, 148.48, 137.07, 135.62, 134.58, 131.80, 129.75, 128.80, 128.50, 127.75, 123.94, 40.90, 40.78, 40.50, 40.22, 39.94, 39.66, 39.39, 39.11.

1o: N-(pyridin-2-ylmethyl)benzamide[8]



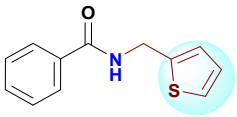
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.58 – 8.49 (m, 1H), 7.92 – 7.38 (m, 6H), 7.21 (s, 2H), 4.82 – 4.70 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.45, 156.37, 148.92, 148.48, 136.92, 136.73, 134.33, 131.49, 128.56, 128.52, 128.50, 127.13, 122.45, 122.28, 122.24, 120.58, 77.54, 77.11, 76.69, 44.76.

1p: N-(furan-2-ylmethyl)benzamide[6]



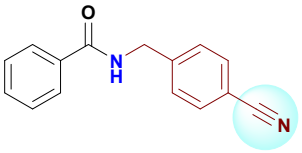
^{1}H NMR (300 MHz, DMSO) δ 8.97 (t, J = 5.7 Hz, 1H), 8.05 – 7.79 (m, 2H), 7.61 – 7.40 (m, 4H), 6.40 (ddd, J = 3.8, 3.2, 1.8 Hz, 1H), 6.28 (dp, J = 2.8, 0.9 Hz, 1H), 4.48 (dd, J = 5.8, 0.9 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.53, 152.92, 142.56, 142.46, 134.58, 131.77, 128.77, 127.75, 110.95, 110.75, 107.31, 107.29, 40.82, 40.54, 40.26, 39.99, 39.71, 39.43, 39.15, 36.51.

1q: N-(thiophen-2-ylmethyl)benzamide[6]



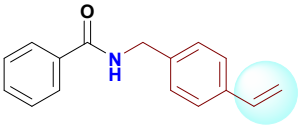
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 – 7.78 (m, 2H), 7.55 – 7.49 (m, 1H), 7.47 – 7.40 (m, 2H), 7.26 (s, 1H), 7.05 (dq, J = 3.4, 0.9 Hz, 1H), 6.99 (dd, J = 5.1, 3.5 Hz, 1H), 6.67 (d, J = 17.0 Hz, 1H), 4.82 (dd, J = 5.6, 0.8 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 167.20, 140.83, 134.16, 131.66, 128.61, 128.59, 127.04, 126.98, 126.90, 126.25, 125.36, 124.97, 77.50, 77.07, 76.65, 39.11, 38.84.

1r: N-(4-cyanobenzyl)benzamide[2]



^{1}H NMR (300 MHz, DMSO) δ 9.09 (t, J = 6.0 Hz, 1H), 7.94 – 7.88 (m, 2H), 7.86 – 7.80 (m, 2H), 7.58 – 7.45 (m, 2H), 7.42 – 7.25 (m, 3H), 4.54 (t, J = 5.8 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 168.29, 146.37, 133.09, 129.04, 128.83, 128.01, 127.81, 127.35, 126.40, 62.94, 40.80, 40.53, 40.25, 39.97, 39.69, 39.41, 39.13.

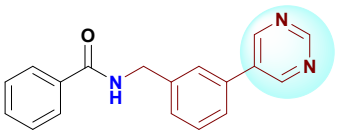
1s: N-(4-vinylbenzyl)benzamide



^{1}H NMR (300 MHz, DMSO) δ 9.04 (t, J = 6.0 Hz, 1H), 7.94 – 7.86 (m, 2H), 7.59 – 7.40 (m, 5H), 7.34 – 7.26 (m, 2H), 6.72 (dd, J = 17.7, 10.9 Hz, 1H), 5.80 (dd, J = 17.7, 1.1 Hz, 1H), 5.27 – 5.19 (m, 1H), 4.48 (d, J = 6.0 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.66, 136.87, 131.72, 128.79, 127.95, 127.70, 126.55, 114.29, 42.87, 40.83, 40.55, 40.28, 40.00, 39.72, 39.44, 39.16.

HRMS (ESI-TOF): *m/z* calcd. for $C_{16}H_{15}NO+H^+$ ([M+H$^+$]) 238.1232, found 238.1229.

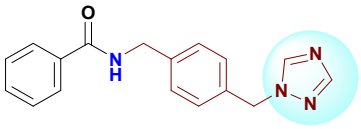
1t: N-(3-(pyrimidin-5-yl)benzyl)benzamide



^{1}H NMR (300 MHz, DMSO) δ 9.17 (d, J = 0.9 Hz, 1H), 9.09 (d, J = 1.7 Hz, 3H), 7.97 – 7.82 (m, 2H), 7.76 – 7.70 (m, 1H), 7.66 (ddd, J = 7.5, 1.9, 1.4 Hz, 1H), 7.53 – 7.39 (m, 5H), 4.57 (dd, J = 5.9, 3.8 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.83, 157.78, 157.71, 155.16, 155.14, 141.35, 134.76, 134.23, 133.72, 131.75, 129.85, 129.60, 128.80, 128.21, 127.76, 127.46, 126.46, 125.90, 125.75, 125.38, 43.08, 40.80, 40.52, 40.25, 39.97, 39.69, 39.41, 39.13.

HRMS (ESI-TOF): *m/z* calcd. for $C_{18}H_{15}N_3O+Na^+$ ([M+Na$^+$]) 312.1107, found 312.1110.

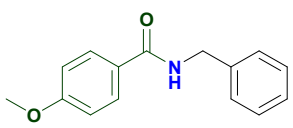
1u: N-(4-((1H-1,2,4-triazol-1-yl) methyl)benzyl)benzamide



^{1}H NMR (300 MHz, DMSO) δ 9.08 (t, J = 6.0 Hz, 1H), 8.68 (s, 1H), 8.00 (s, 1H), 7.96 – 7.90 (m, 2H), 7.55 – 7.43 (m, 3H), 7.37 – 7.23 (m, 4H), 5.41 (s, 2H), 4.50 (d, J = 6.0 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 166.76, 152.18, 144.60, 139.99, 135.23, 134.80, 131.72, 128.78, 128.41, 128.01, 127.74, 52.37, 42.87, 40.80, 40.53, 40.25, 39.97, 39.69, 39.41, 39.13.

HRMS (ESI-TOF): *m/z* calcd. for $C_{17}H_{16}N_4O+H^+$ ([M+H$^+$]) 293.1402, found 293.1403.

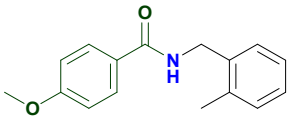
2a: N-benzyl-4-methoxybenzamide[11]



^{1}H NMR (400 MHz, DMSO) δ 8.92 (t, J = 6.0 Hz, 1H), 7.95 – 7.87 (m, 2H), 7.33 (d, J = 4.6 Hz, 4H), 7.28 – 7.21 (m, 1H), 7.05 – 6.98 (m, 2H), 4.49 (d, J = 6.0 Hz, 2H), 3.81 (s, 3H). **^{13}C NMR (101**

MHz, DMSO) δ 166.19, 162.07, 140.39, 129.56, 128.72, 127.65, 127.14, 127.03, 113.98, 55.80, 43.01, 40.60, 40.39, 40.18, 39.97, 39.76, 39.55, 39.34.

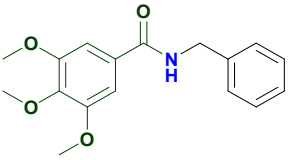
2b: 4-methoxy-N-(2-methylbenzyl)benzamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 – 7.72 (m, 2H), 7.41 – 7.09 (m, 4H), 6.98 – 6.87 (m, 2H), 6.36 (s, 1H), 4.62 (d, J = 5.3 Hz, 2H), 3.85 (s, 3H), 2.37 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 166.82, 162.21, 136.60, 136.01, 130.59, 128.81, 128.63, 127.81, 126.61, 126.26, 113.76, 77.52, 77.09, 76.67, 55.42, 42.26, 19.08.

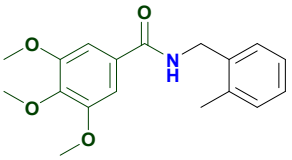
HRMS (ESI-TOF): *m/z* calcd. for $C_{16}H_{17}NO_2+H^+$ ([M+H$^+$]) 256.1337, found 256.1340.

2d: N-benzyl-3,4,5-trimethoxybenzamide[13]



^{1}H NMR (400 MHz, DMSO) δ 9.01 (t, J = 6.0 Hz, 1H), 7.35 – 7.32 (m, 4H), 7.28 – 7.23 (m, 3H), 4.50 (d, J = 5.9 Hz, 2H), 3.84 (s, 6H), 3.71 (s, 3H). **^{13}C NMR (101 MHz, DMSO)** δ 166.03, 153.07, 140.43, 140.17, 129.88, 128.77, 128.51, 127.72, 127.24, 127.09, 126.88, 105.28, 60.54, 56.45, 43.17, 40.59, 40.38, 40.17, 39.96, 39.76, 39.55, 39.34.

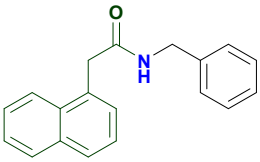
2e: 3,4,5-trimethoxy-N-(2-methylbenzyl)benzamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 1H), 7.28 – 7.20 (m, 3H), 7.03 (s, 2H), 6.17 (s, 1H), 4.67 (d, J = 5.3 Hz, 2H), 3.91 (d, J = 5.4 Hz, 9H), 2.41 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 153.26, 130.72, 128.87, 128.01, 126.32, 104.40, 77.45, 77.03, 76.61, 60.93, 56.41, 42.51, 19.13.

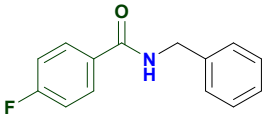
HRMS (ESI-TOF): *m/z* calcd. for $C_{18}H_{21}NO_4+H^+$ ([M+H$^+$]) 316.1549 found 316.1553.

2f: N-benzyl-2-(naphthalen-1-yl)acetamide[14]



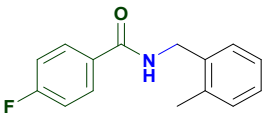
^{1}H NMR (300 MHz, DMSO) δ 8.65 (t, J = 5.9 Hz, 1H), 8.16 – 8.08 (m, 1H), 7.96 – 7.90 (m, 1H), 7.86 – 7.79 (m, 1H), 7.55 – 7.50 (m, 2H), 7.48 – 7.43 (m, 2H), 7.32 – 7.21 (m, 5H), 4.30 (d, J = 5.9 Hz, 2H), 3.98 (s, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 170.55, 139.97, 133.83, 133.25, 132.44, 128.84, 128.71, 128.33, 127.70, 127.54, 127.23, 126.37, 126.11, 125.99, 124.78, 42.74, 40.83, 40.55, 40.27, 39.99, 39.72, 39.44, 39.16.

2g: N-benzyl-4-fluorobenzamide[6]



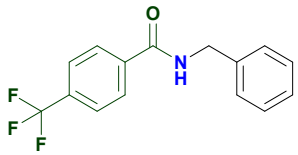
^{1}H NMR (400 MHz, DMSO) δ 9.07 (dt, J = 10.9, 5.9 Hz, 1H), 8.04 – 7.85 (m, 2H), 7.52 – 7.45 (m, 1H), 7.40 – 7.28 (m, 5H), 7.25 (dddd, J = 7.1, 4.3, 3.2, 1.7 Hz, 1H), 4.49 (dd, J = 6.0, 3.1 Hz, 2H). **^{13}C NMR (101 MHz, DMSO)** δ 166.69, 165.64, 165.60, 140.16, 140.04, 134.80, 131.72, 131.27, 131.24, 130.41, 130.32, 128.80, 128.76, 128.51, 127.71, 127.68, 127.66, 127.24, 127.20, 126.88, 115.83, 115.61, 43.13, 43.07, 40.59, 40.38, 40.17, 39.96, 39.75, 39.54, 39.33.

2h: 4-fluoro-N-(2-methylbenzyl)benzamide[15]



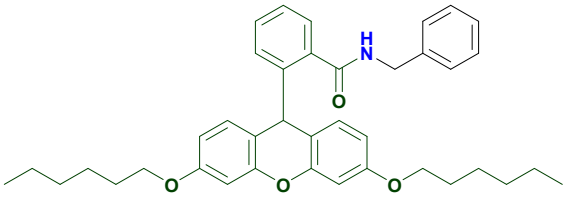
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 – 7.74 (m, 2H), 7.52 – 7.40 (m, 1H), 7.34 – 7.28 (m, 1H), 7.28 – 7.18 (m, 3H), 7.13 – 7.07 (m, 1H), 6.38 (s, 1H), 4.64 (dd, J = 5.4, 4.1 Hz, 2H), 2.38 (d, J = 1.9 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.30, 166.42, 166.23, 163.08, 136.63, 136.60, 135.76, 135.67, 134.35, 131.57, 130.67, 130.54, 129.38, 129.27, 128.70, 128.62, 127.97, 127.94, 126.96, 126.32, 115.76, 115.47, 77.49, 77.07, 76.65, 42.40, 42.36, 19.08.

2i: N-benzyl-4-(trifluoromethyl)benzamide[7]



^{1}H NMR (300 MHz, DMSO) δ 9.29 (t, J = 5.9 Hz, 1H), 8.14 – 8.06 (m, 2H), 7.90 – 7.82 (m, 2H), 7.36 – 7.21 (m, 5H), 4.52 (d, J = 6.0 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 165.55, 139.76, 138.56, 131.86, 131.44, 128.79, 128.65, 127.73, 127.31, 126.22, 125.91, 125.86, 125.81, 125.75, 122.61, 43.24, 40.81, 40.53, 40.25, 39.98, 39.70, 39.42, 39.14.

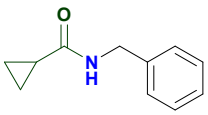
2j: N-benzyl-2-(3,6-bis(hexyloxy)-9H-xanthen-9-yl)benzamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 6H), 7.25 (ddd, J = 8.1, 6.9, 1.5 Hz, 1H), 7.10 (ddd, J = 22.9, 8.0, 0.9 Hz, 4H), 6.70 (d, J = 2.5 Hz, 2H), 6.61 (t, J = 5.8 Hz, 1H), 6.52 (dd, J = 8.5, 2.6 Hz, 2H), 5.83 (s, 1H), 4.62 (d, J = 5.8 Hz, 2H), 3.95 (t, J = 6.5 Hz, 4H), 1.88 – 1.76 (m, 4H), 1.56 – 1.34 (m, 12H), 1.03 – 0.92 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.97, 158.83, 151.53, 151.49, 146.16, 138.23, 135.14, 131.25, 130.80, 130.74, 128.85, 128.29, 127.94, 127.67, 126.40, 126.17, 116.83, 110.73, 101.71, 77.63, 77.20, 76.78, 68.17, 44.12, 38.54, 31.66, 29.26, 25.81, 22.74, 22.69, 14.15.

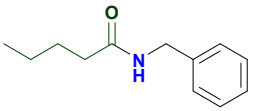
HRMS (ESI-TOF): *m/z* calcd. for $C_{39}H_{45}NO_4$+H^+ ([M+H^+]) 592.3427, found 592.3438.

2k: N-benzylcyclopropanecarboxamide[16]



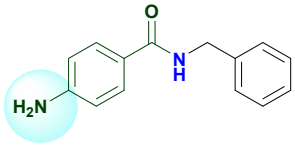
^{1}H NMR (300 MHz, DMSO) δ 8.55 (s, 1H), 7.36 – 7.21 (m, 5H), 4.29 (d, J = 5.9 Hz, 2H), 1.66 – 1.55 (m, 1H), 0.94 – 0.55 (m, 4H). **^{13}C NMR (75 MHz, DMSO)** δ 173.03, 140.16, 128.76, 128.71, 127.75, 127.62, 127.21, 127.14, 42.71, 42.43, 40.82, 40.55, 40.27, 39.99, 39.72, 39.44, 39.16, 14.13, 14.03, 6.71.

2l: N-benzylpentanamide[17]



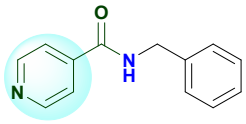
^{1}H NMR (300 MHz, DMSO) δ 8.29 (s, 1H), 7.52 – 6.90 (m, 5H), 4.26 (d, J = 5.9 Hz, 2H), 2.19 – 2.10 (m, 2H), 1.58 – 1.45 (m, 2H), 1.35 – 1.20 (m, 2H), 0.92 – 0.82 (m, 3H). **^{13}C NMR (75 MHz, DMSO)** δ 172.58, 140.24, 128.71, 127.61, 127.13, 42.43, 40.83, 40.55, 40.27, 39.99, 39.72, 39.44, 39.16, 35.54, 27.95, 22.30, 14.19.

2m: 4-amino-N-benzylbenzamide[18]



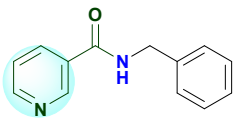
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 – 7.59 (m, 2H), 7.42 – 7.29 (m, 5H), 6.73 – 6.58 (m, 2H), 6.34 (s, 1H), 4.63 (d, J = 5.7 Hz, 2H), 4.32 – 3.55 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.11, 149.68, 138.66, 128.76, 128.73, 127.91, 127.48, 123.88, 114.16, 77.48, 77.06, 76.63, 43.98.

2n: N-benzylisonicotinamide[8]



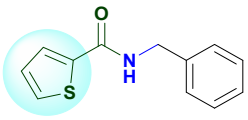
^{1}H NMR (300 MHz, DMSO) δ 9.35 (t, J = 6.0 Hz, 1H), 8.77 – 8.71 (m, 2H), 7.84 – 7.79 (m, 2H), 7.37 – 7.23 (m, 5H), 4.52 (d, J = 6.0 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 165.17, 150.75, 141.71, 139.57, 128.82, 127.75, 127.37, 121.74, 43.19, 40.80, 40.52, 40.24, 39.97, 39.69, 39.41, 39.13.

2o: N-benzylnicotinamide[19]



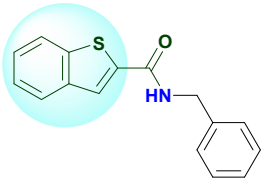
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.83 (d, J = 89.3 Hz, 2H), 8.15 (d, J = 7.9 Hz, 1H), 6.97 (s, 1H), 4.65 (d, J = 5.6 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.52, 152.07, 147.83, 137.79, 135.37, 128.85, 127.97, 127.77, 77.49, 77.06, 76.64, 44.19.

2p: N-benzylthiophene-3-carboxamide[19]



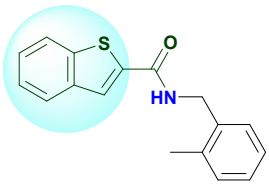
^{1}H NMR (400 MHz, DMSO) δ 8.88 (t, J = 6.1 Hz, 1H), 8.18 (dd, J = 3.0, 1.3 Hz, 1H), 7.61 – 7.54 (m, 2H), 7.36 – 7.31 (m, 4H), 7.25 (ddd, J = 8.7, 3.7, 2.5 Hz, 1H), 4.46 (d, J = 6.0 Hz, 2H). **^{13}C NMR (101 MHz, DMSO)** δ 162.52, 140.14, 138.15, 129.26, 128.76, 128.51, 127.69, 127.30, 127.23, 42.74, 40.60, 40.39, 40.18, 40.08, 39.97, 39.76, 39.55, 39.34.

2q: N-benzylbenzo[b]thiophene-2-carboxamide[20]



^{1}H NMR (400 MHz, DMSO) δ 9.35 (t, J = 6.0 Hz, 1H), 8.16 (d, J = 0.8 Hz, 1H), 8.06 – 8.00 (m, 1H), 7.97 – 7.92 (m, 1H), 7.49 – 7.42 (m, 2H), 7.38 – 7.32 (m, 4H), 7.29 – 7.23 (m, 1H), 4.52 (d, J = 6.0 Hz, 2H)**. ^{13}C NMR (101 MHz, DMSO)** δ 162.03, 140.70, 140.33, 139.71, 139.66, 128.84, 128.59, 128.51, 127.83, 127.39, 126.89, 126.69, 125.67, 125.39, 125.34, 123.27, 43.19, 40.58, 40.37, 40.17, 40.09, 39.96, 39.75, 39.54, 39.33.

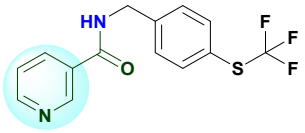
2r: N-(2-methylbenzyl) benzo[b]thiophene-2-carboxamide



^{1}H NMR (300 MHz, DMSO) δ 9.19 (t, J = 5.7 Hz, 1H), 8.19 (d, J = 0.8 Hz, 1H), 8.09 – 7.99 (m, 1H), 7.97 – 7.91 (m, 1H), 7.51 – 7.41 (m, 2H), 7.33 – 7.26 (m, 1H), 7.21 – 7.15 (m, 3H), 4.49 (d, J = 5.7 Hz, 2H), 2.34 (s, 3H). **^{13}C NMR (75 MHz, DMSO)** δ 161.94, 140.68, 140.34, 139.69, 137.20, 136.15, 130.45, 128.14, 127.45, 126.66, 126.26, 125.65, 125.37, 123.27, 41.33, 40.82, 40.54, 40.26, 39.98, 39.71, 39.43, 39.15, 19.21.

HRMS (ESI-TOF): *m/z* calcd. for $C_{17}H_{15}NOS+H^+$ ([M+H$^+$]) 282.0952, found 282.0952.

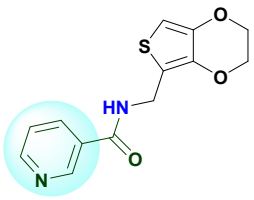
2s: N-(4-((trifluoromethyl)thio)benzyl)nicotinamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 9.07 (d, J = 2.3 Hz, 1H), 8.73 (dd, J = 4.9, 1.7 Hz, 1H), 8.21 (ddd, J = 7.9, 2.3, 1.7 Hz, 1H), 7.68 – 7.63 (m, 2H), 7.46 – 7.41 (m, 3H), 6.97 (s, 1H), 4.77 – 4.68 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.47, 152.08, 147.60, 141.02, 136.78, 136.53, 135.74, 129.98, 128.86, 127.65, 123.78, 77.46, 77.03, 76.61, 43.56.

HRMS (ESI-TOF): *m/z* calcd. for $C_{14}H_{11}N_2OSF_3+H^+$ ([M+H$^+$]) 313.0622, found 313.0622.

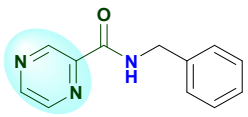
2t: N-((2,3-dihydrothieno[3,4-b][1,4]dioxin-5-yl)methyl)nicotinamide



^{1}H NMR (300 MHz, $CDCl_3$) δ 9.02 (ddd, J = 16.1, 2.3, 0.9 Hz, 1H), 8.76 (ddd, J = 12.2, 4.9, 1.7 Hz, 1H), 8.20 – 8.12 (m, 1H), 7.45 – 7.38 (m, 1H), 6.59 (d, J = 4.9 Hz, 1H), 6.29 (s, 1H), 4.68 (d, J = 5.4 Hz, 2H), 4.28 – 4.20 (m, 4H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 152.29, 147.83, 135.32, 123.57, 98.30, 77.45, 77.03, 76.61, 64.82, 64.60, 35.44.

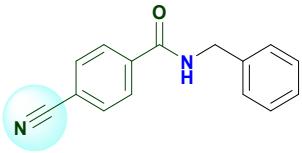
HRMS (ESI-TOF): *m/z* calcd. for $C_{13}H_{12}N_2O_3S+H^+$ ([M+H$^+$]) 277.0647, found 277.0654.

2u: N-benzylpyrazine-2-carboxamide[21]



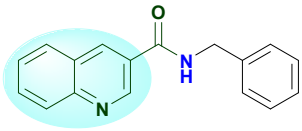
^{1}H NMR (300 MHz, DMSO) δ 9.49 (s, 1H), 9.21 (dd, J = 1.5, 0.3 Hz, 1H), 8.88 (dd, J = 2.5, 0.3 Hz, 1H), 8.75 (dd, J = 2.5, 1.5 Hz, 1H), 7.35 – 7.21 (m, 5H), 4.52 (d, J = 6.4 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 148.02, 144.07, 139.71, 128.75, 127.84, 127.30, 42.85, 40.83, 40.55, 40.27, 40.00, 39.72, 39.44, 39.16.

2v: N-benzyl-4-cyanobenzamide[22]



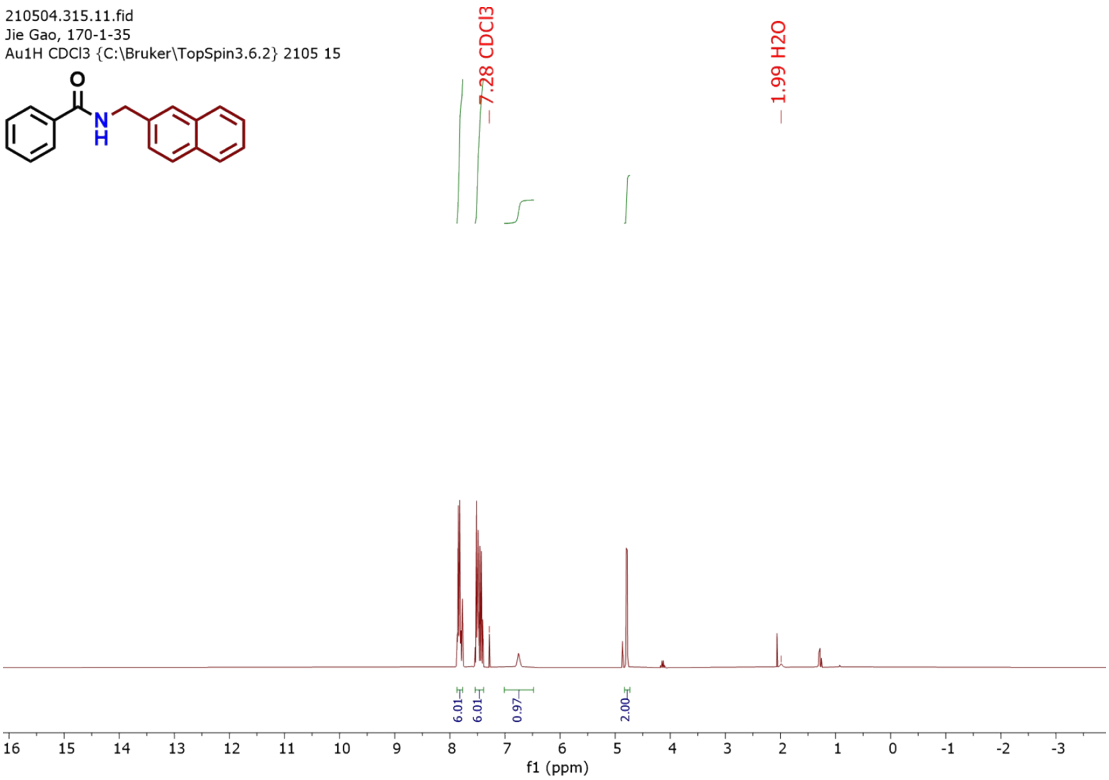
^{1}H NMR (300 MHz, DMSO) δ 9.30 (t, J = 6.0 Hz, 1H), 8.08 – 8.02 (m, 2H), 8.01 – 7.94 (m, 2H), 7.35 – 7.25 (m, 5H), 4.50 (dd, J = 5.8, 2.1 Hz, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 165.32, 139.66, 138.77, 132.95, 128.81, 128.59, 127.74, 127.34, 118.82, 114.12, 43.27, 40.83, 40.55, 40.27, 39.99, 39.72, 39.44, 39.16.

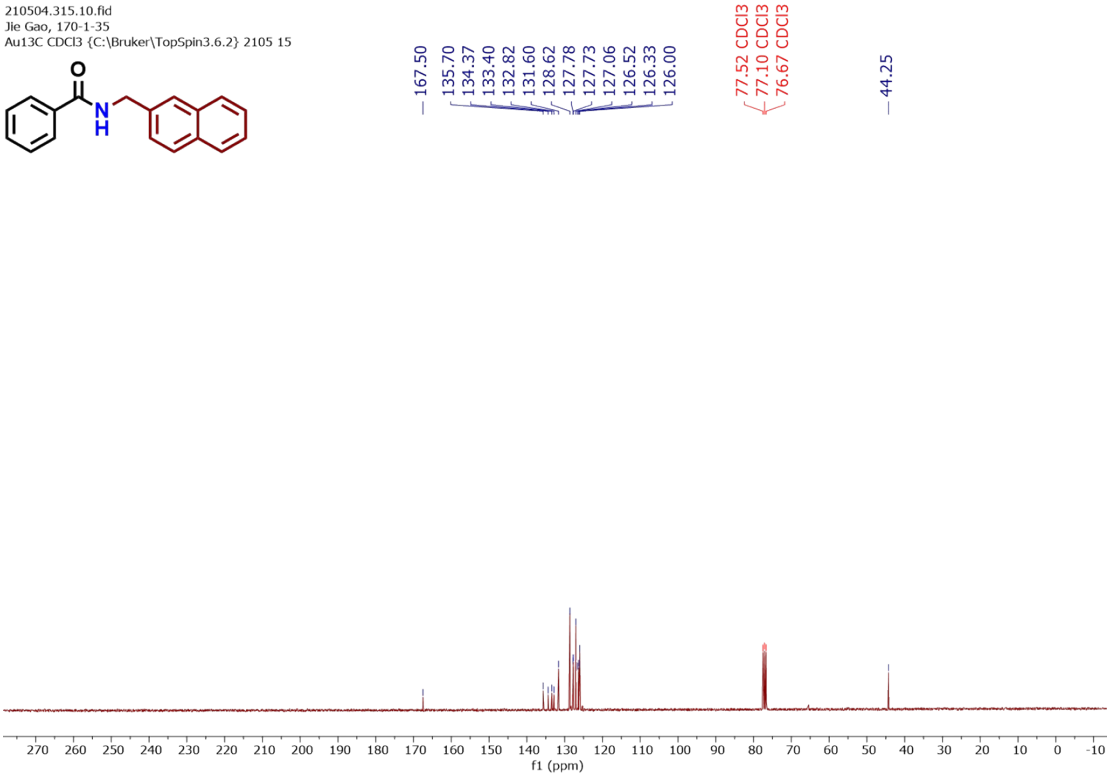
2w: N-benzylquinoline-3-carboxamide[23]



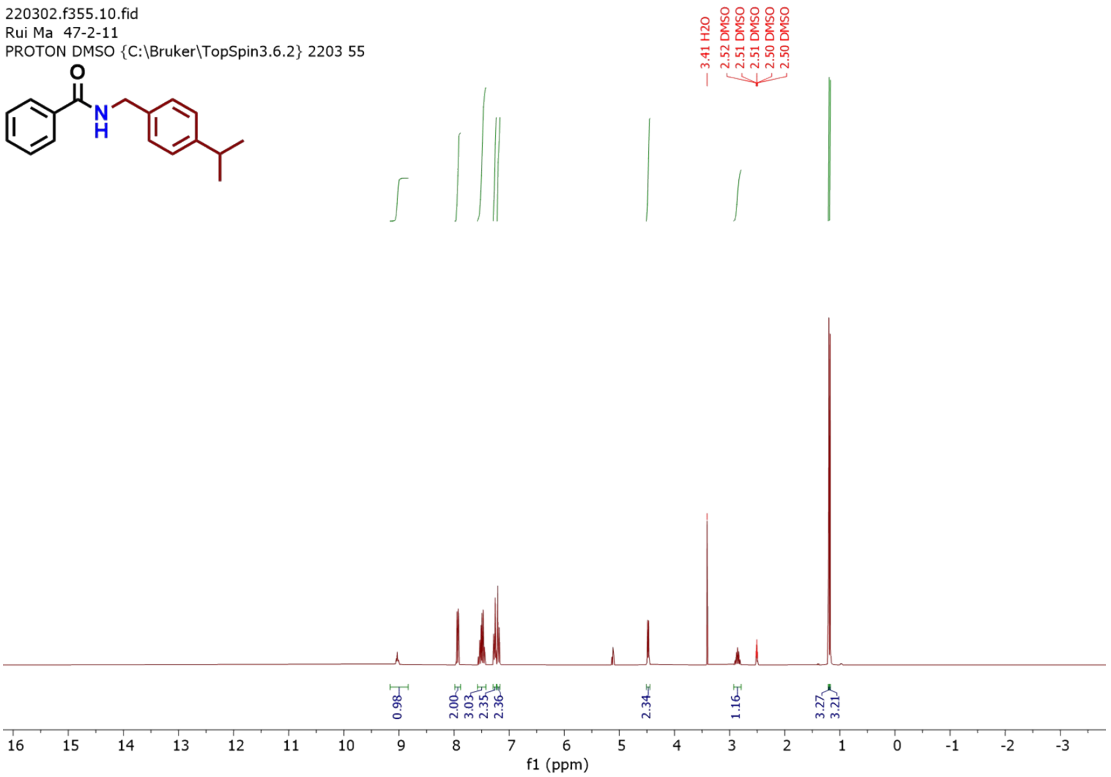
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.76 (dd, J = 4.3, 1.7 Hz, 1H), 8.19 – 8.13 (m, 1H), 7.98 – 7.85 (m, 3H), 7.41 (d, J = 5.5 Hz, 1H), 7.28 – 7.09 (m, 6H), 4.52 (d, J = 5.7 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.01, 151.84, 149.18, 138.19, 136.98, 132.35, 129.71, 128.71, 127.81, 127.54, 127.48, 127.35, 121.82, 77.58, 44.19.

7. NMR spectra

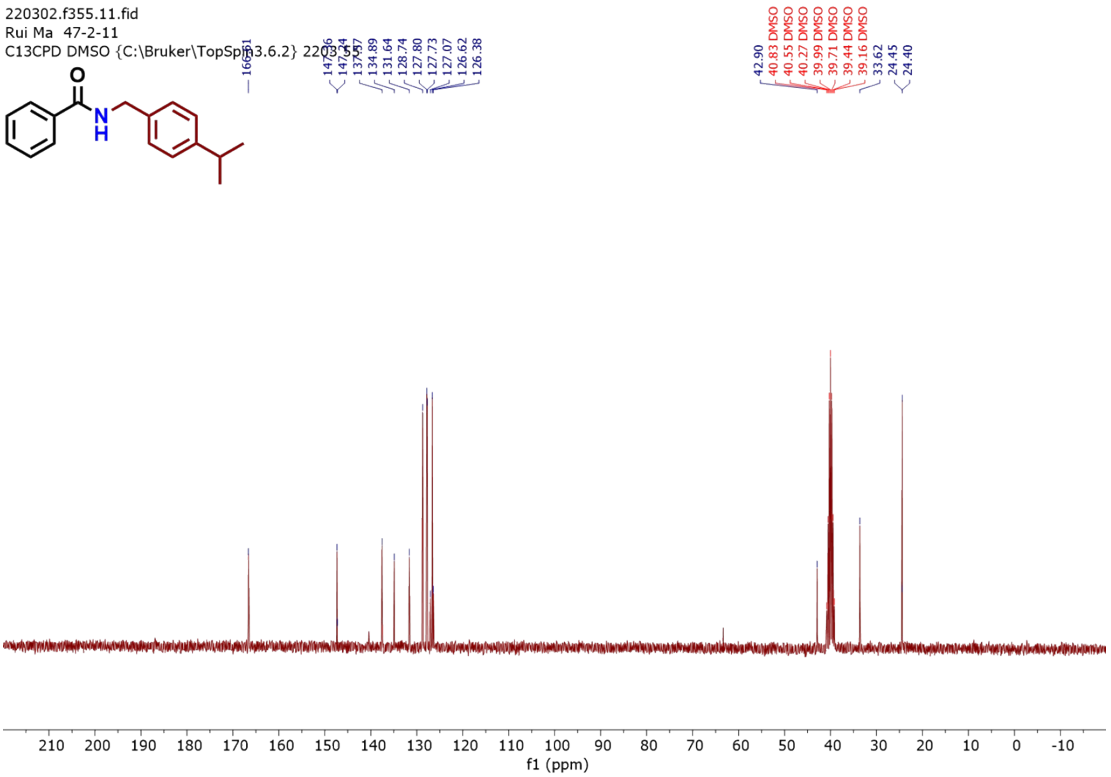




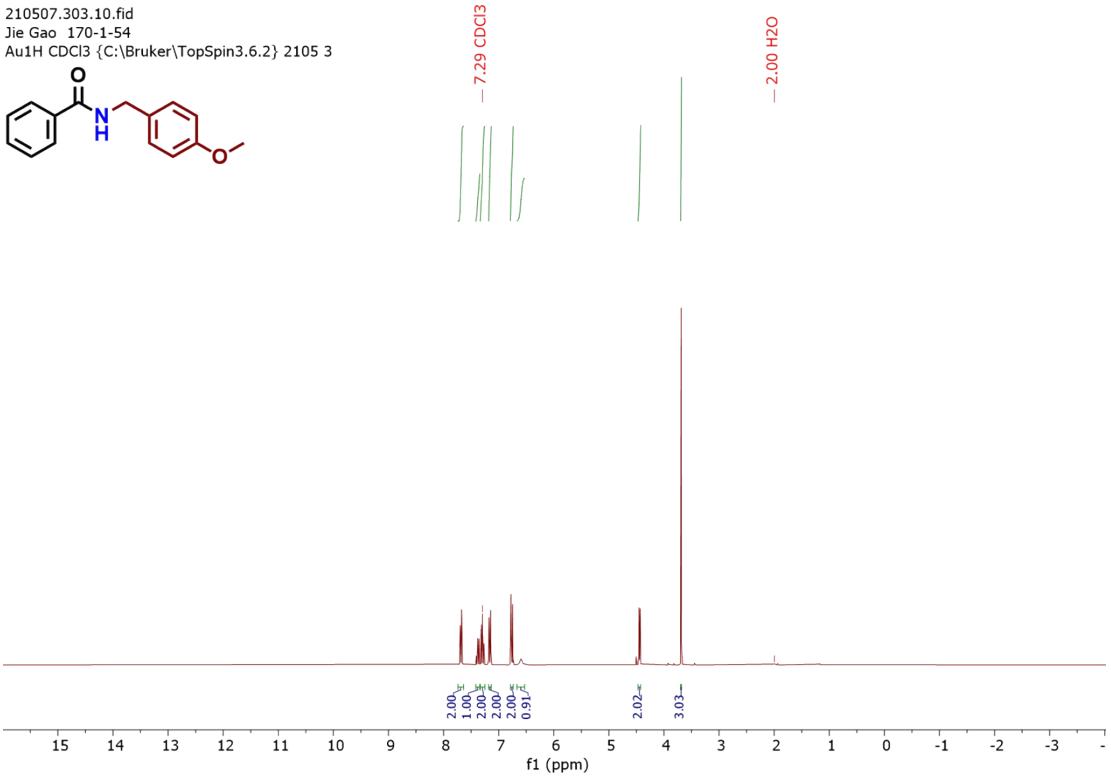
220302.f355.10.fid
Rui Ma 47-2-11
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2203 55



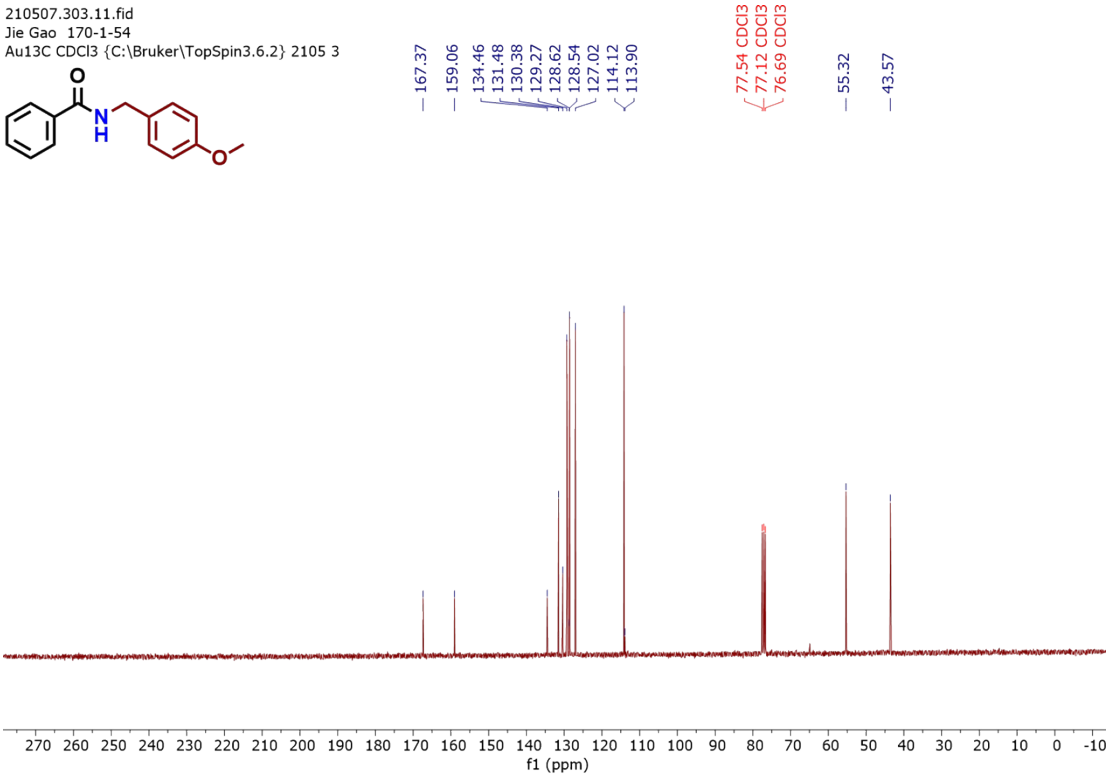
220302.f355.11.fid
Rui Ma 47-2-11
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2203 55



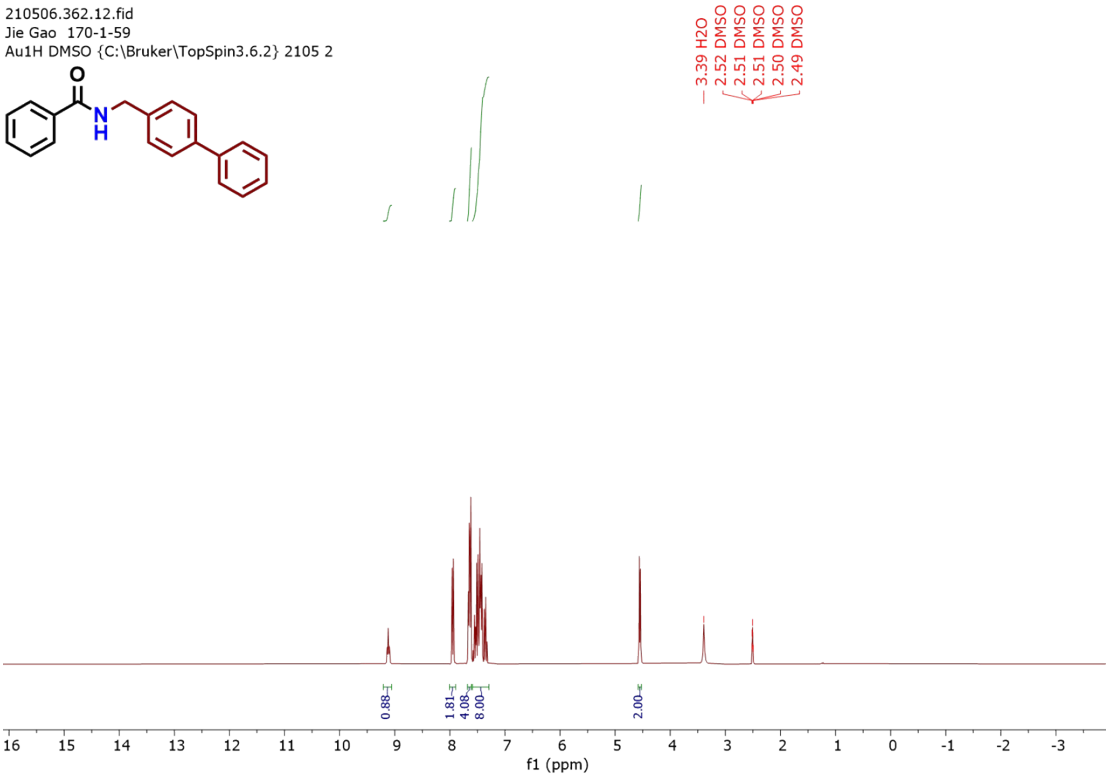
210507.303.10.fid
Jie Gao 170-1-54
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 3



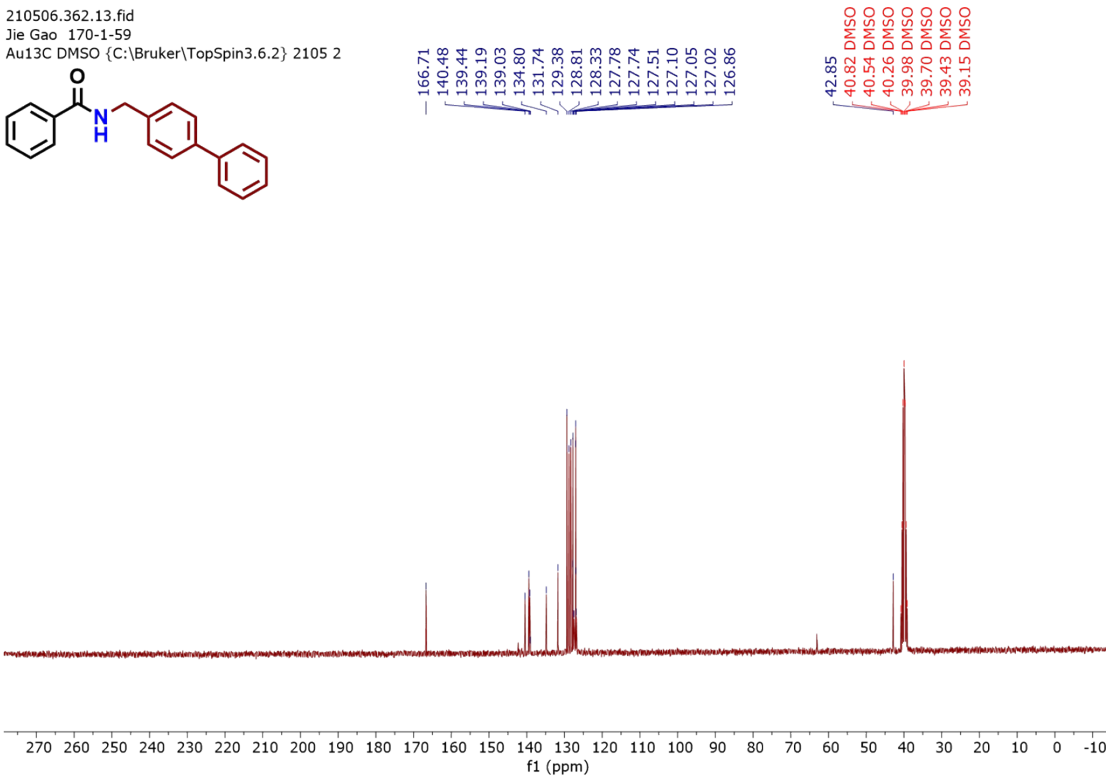
210507.303.11.fid
Jie Gao 170-1-54
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 3



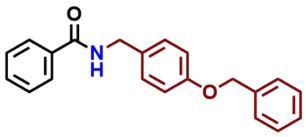
210506.362.12.fid
Jie Gao 170-1-59
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 2

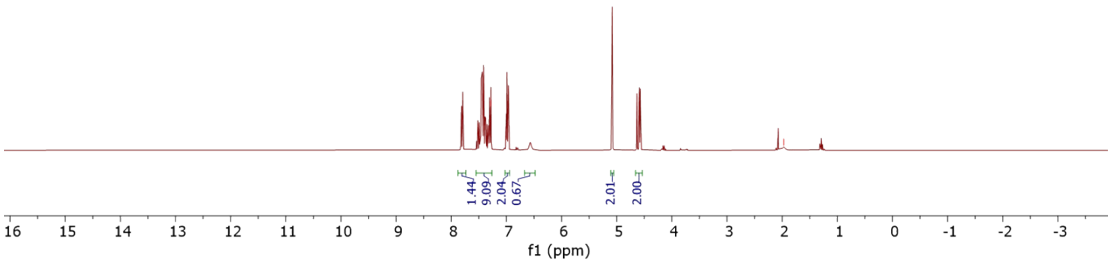


210506.362.13.fid
Jie Gao 170-1-59
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 2

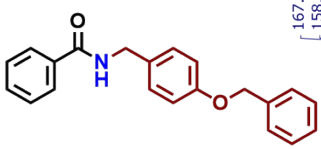


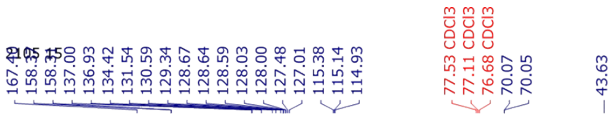
210507.315.10.fid
Jie Gao 170-1-58
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 15

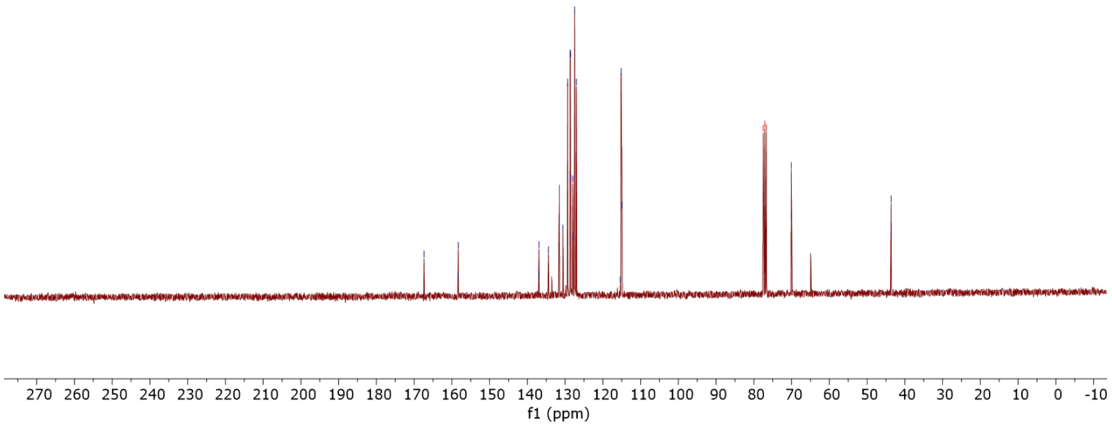




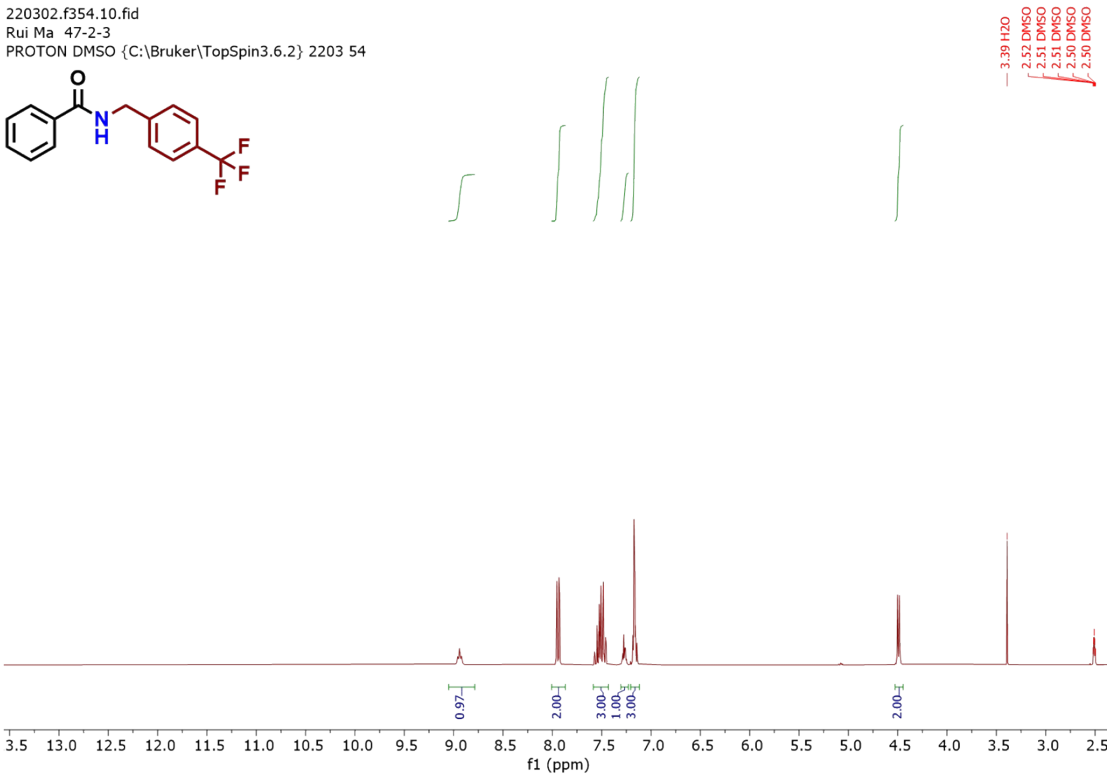
210507.315.11.fid
Jie Gao 170-1-58
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 1



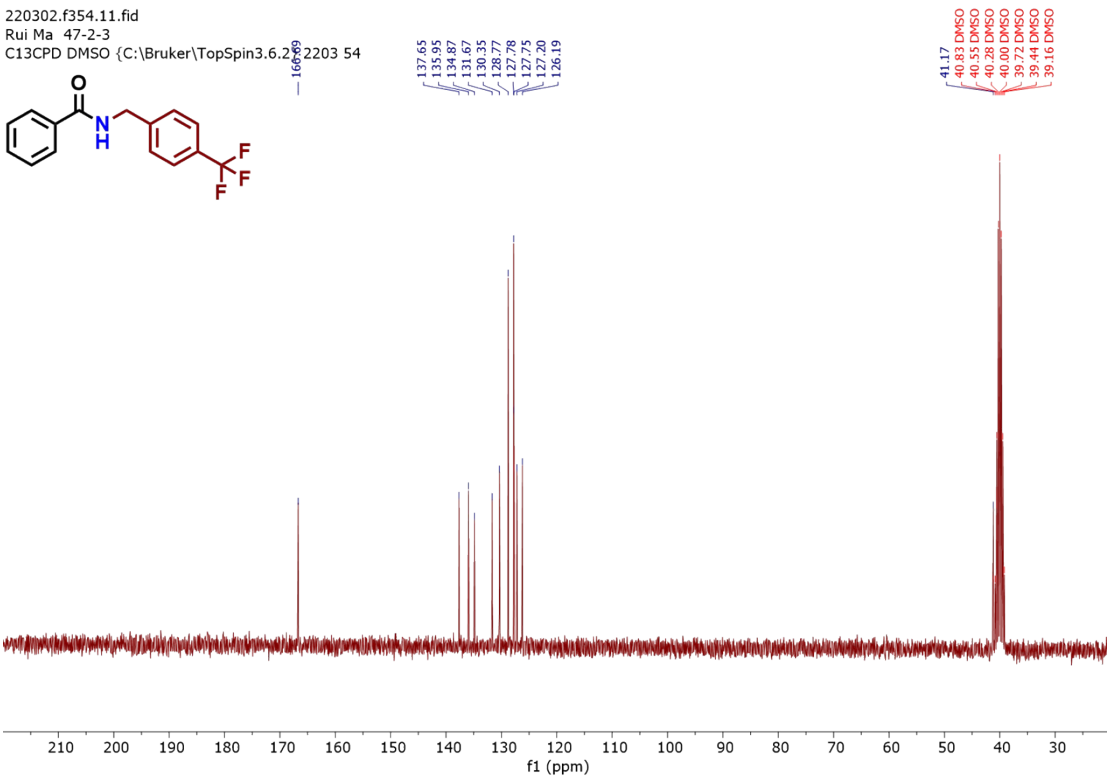




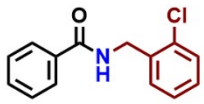
220302.f354.10.fid
Rui Ma 47-2-3
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2203 54

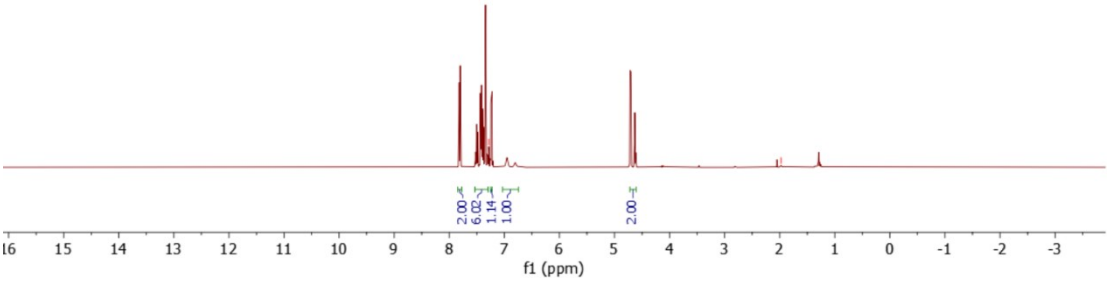


220302.f354.11.fid
Rui Ma 47-2-3
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2203 54

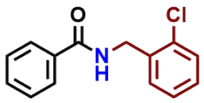


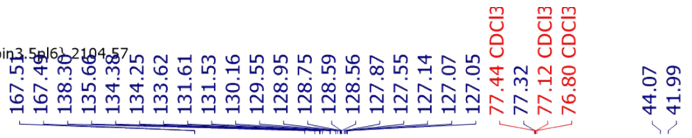
210429.457.10.fid
Jie Gao 170-1-3a
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2104 57

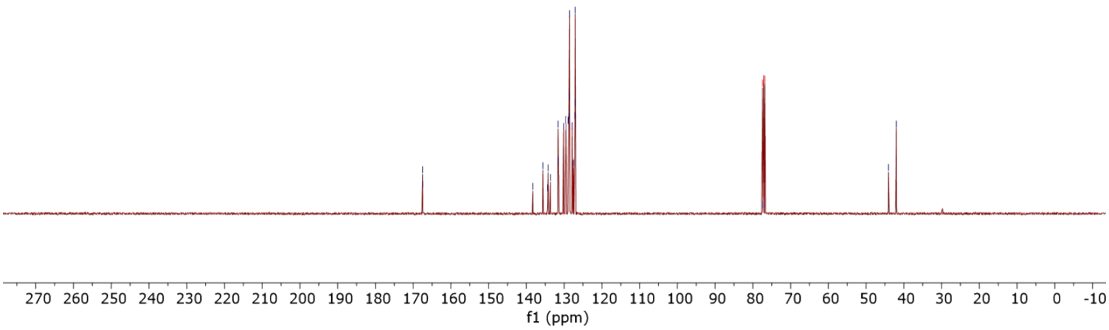




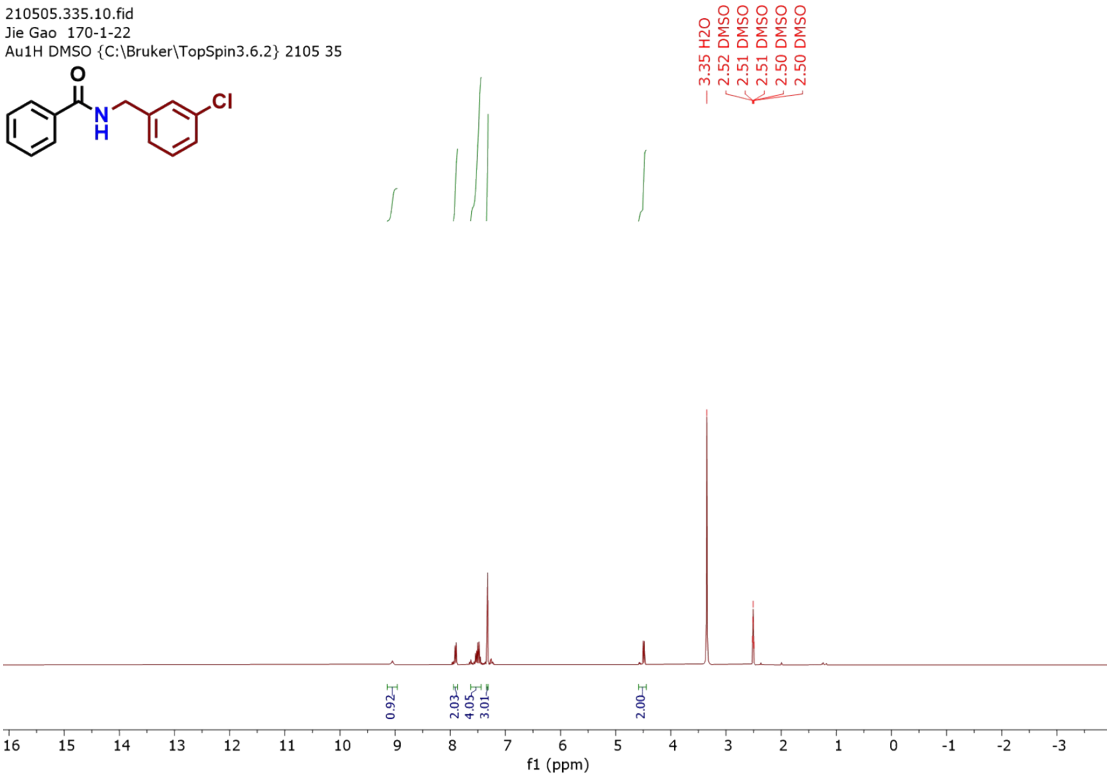
210429.457.11.fid
Jie Gao 170-1-3a
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2104 57



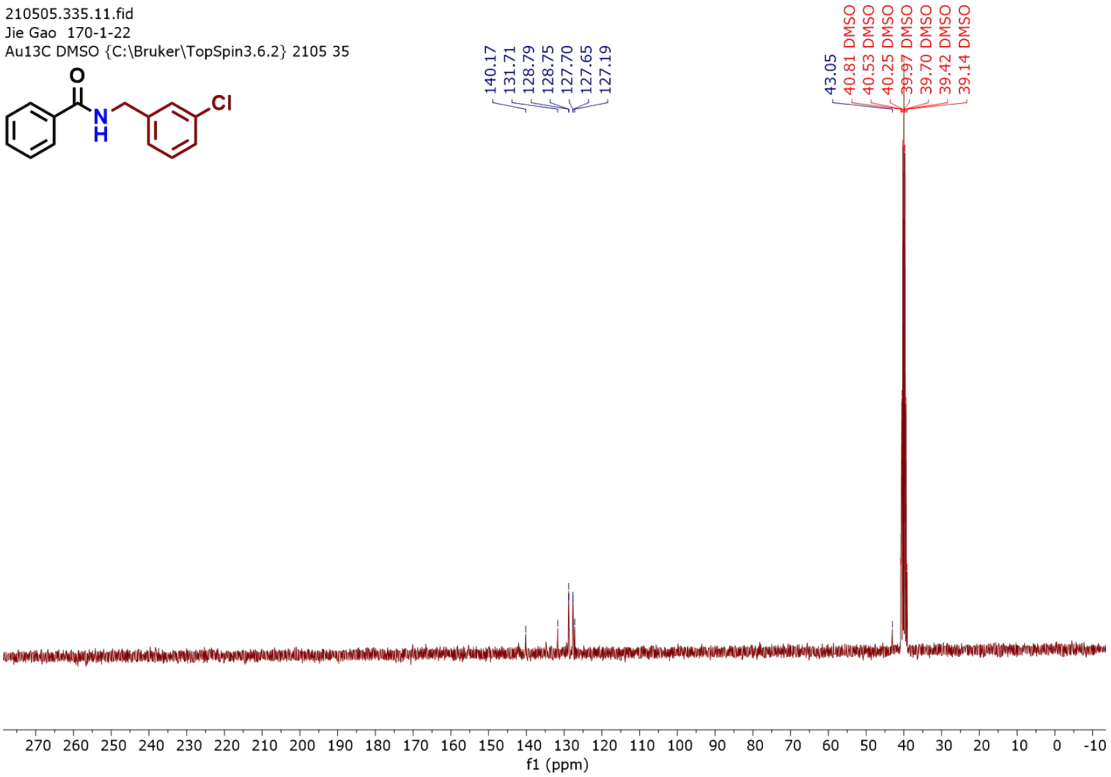




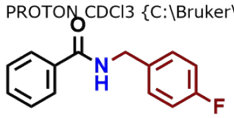
210505.335.10.fid
Jie Gao 170-1-22
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 35

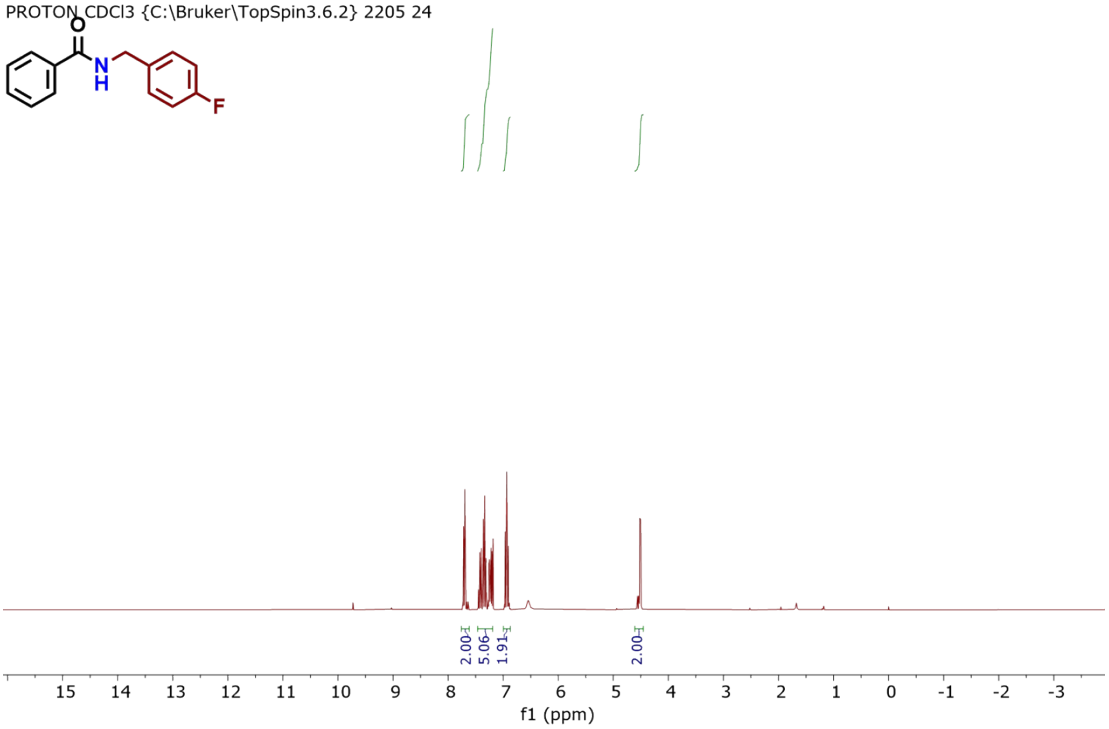


210505.335.11.fid
Jie Gao 170-1-22
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 35

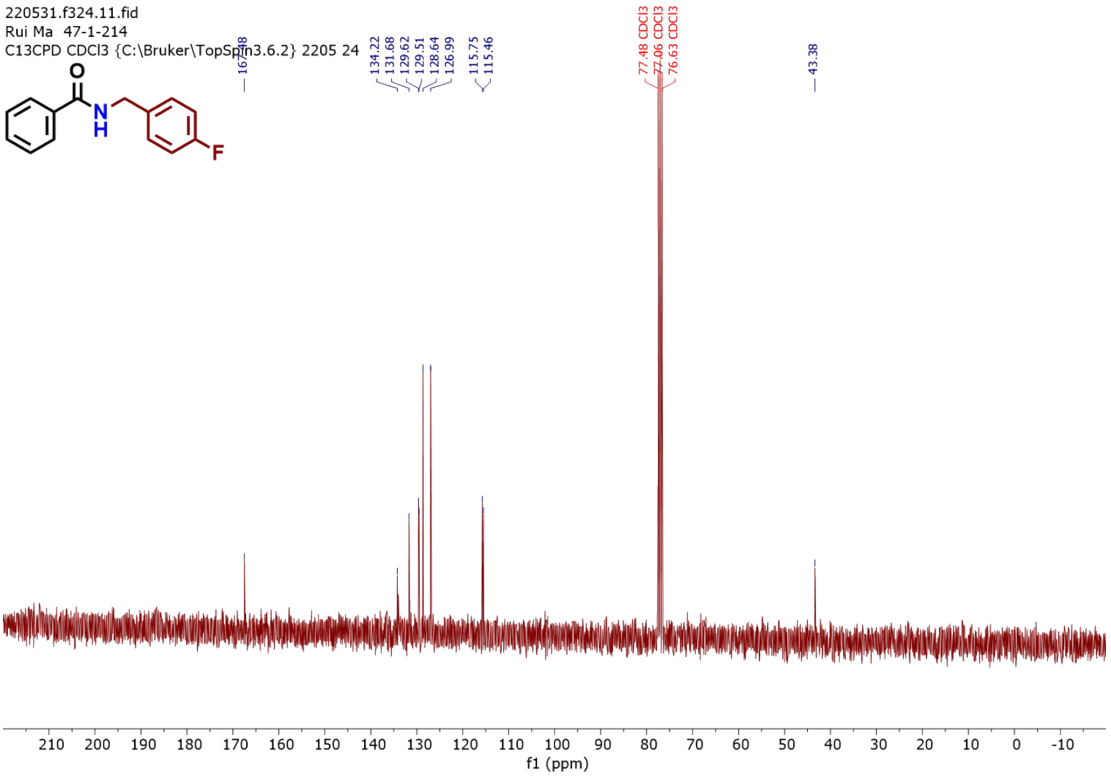


220531.f324.10.fid
Rui Ma 47-1-214
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2205 24

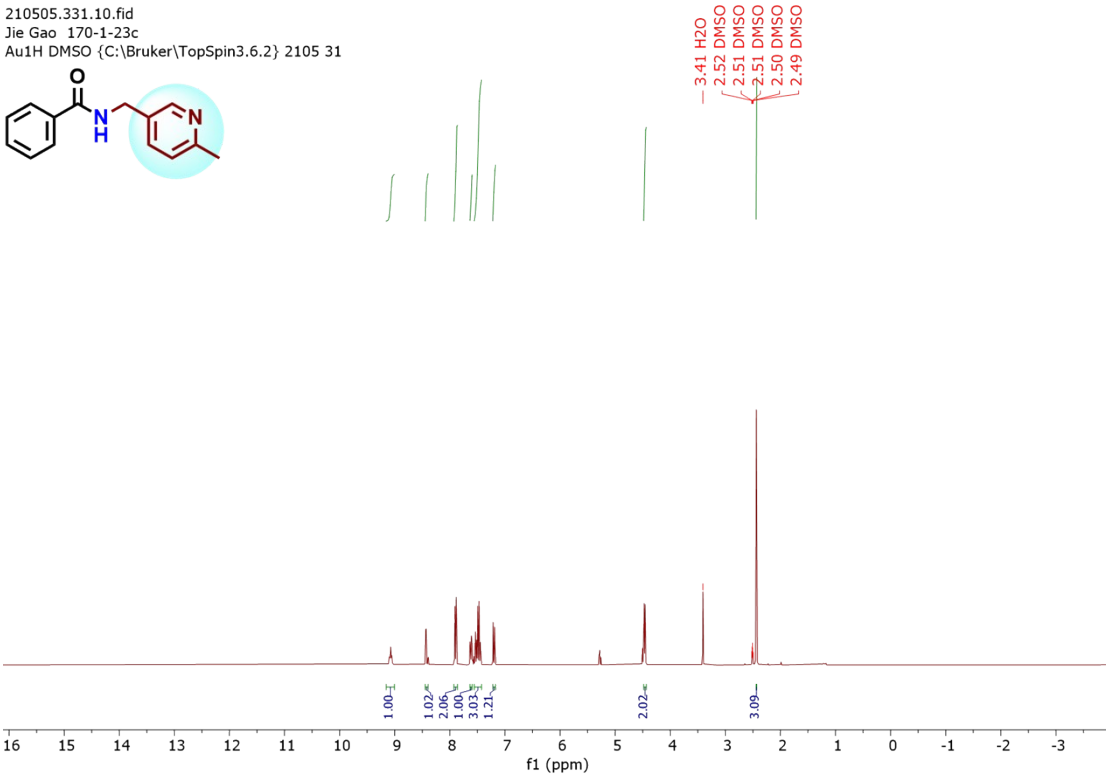




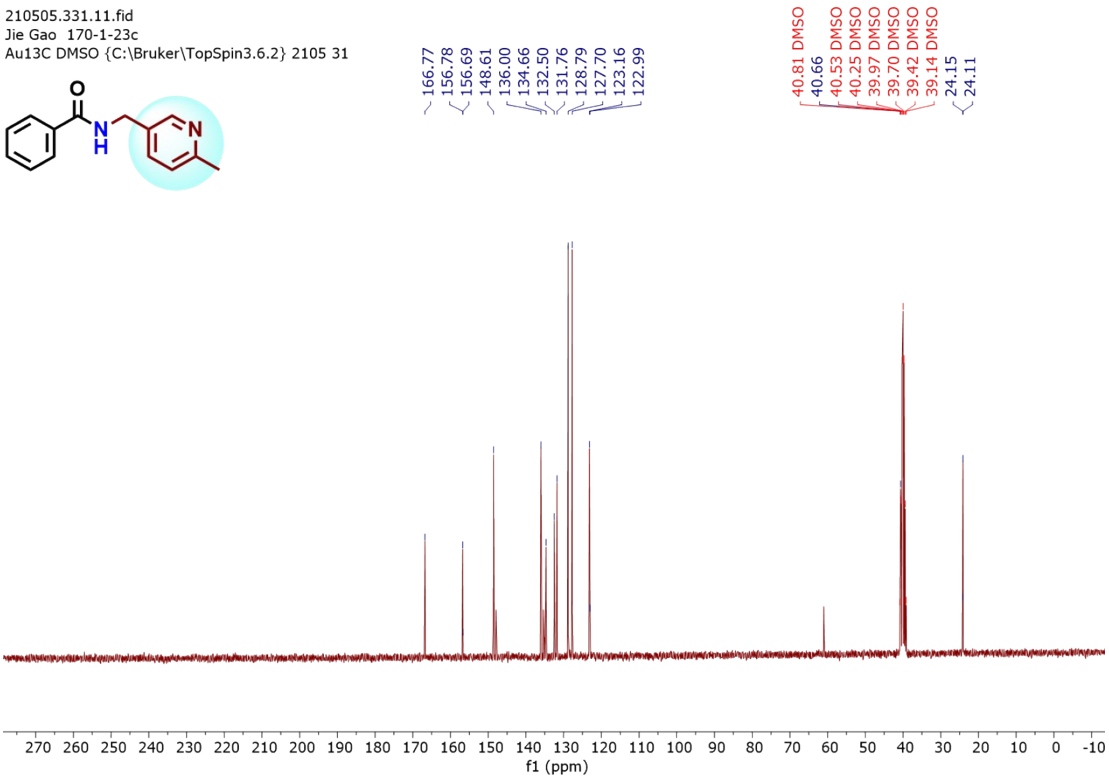
220531.f324.11.fid
Rui Ma 47-1-214
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2205 24



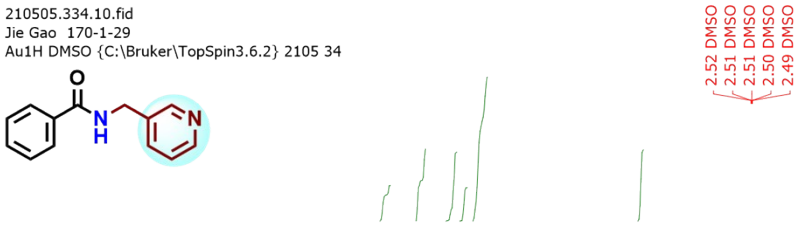
210505.331.10.fid
Jie Gao 170-1-23c
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 31

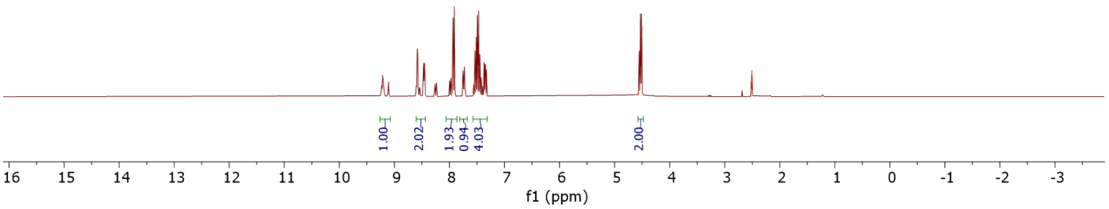


210505.331.11.fid
Jie Gao 170-1-23c
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 31

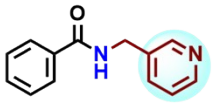


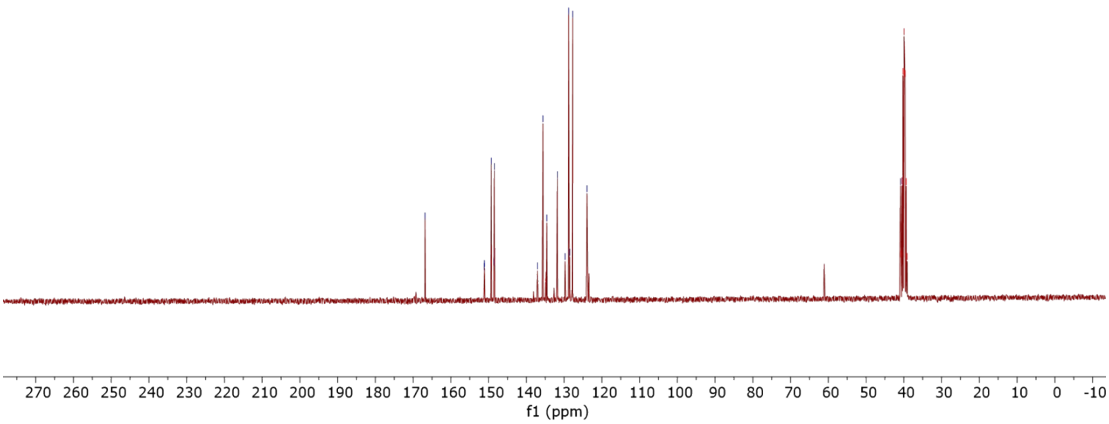
210505.334.10.fid
Jie Gao 170-1-29
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 34



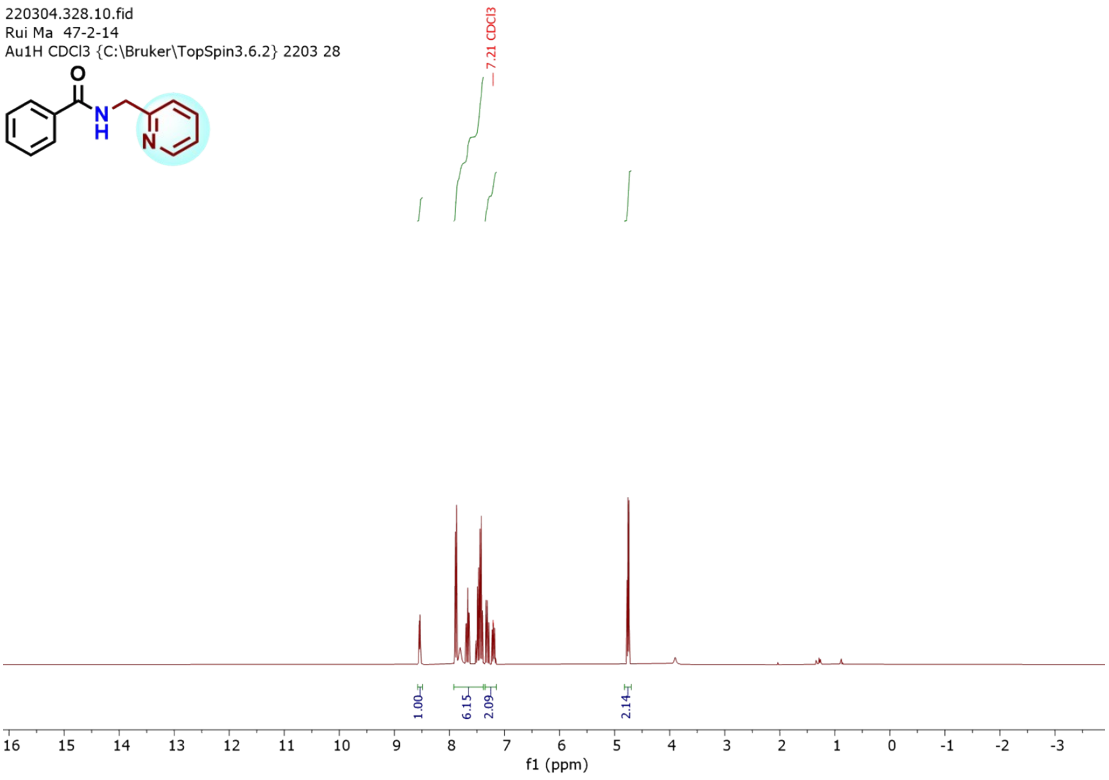


210505.334.11.fid
Jie Gao 170-1-29
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 34

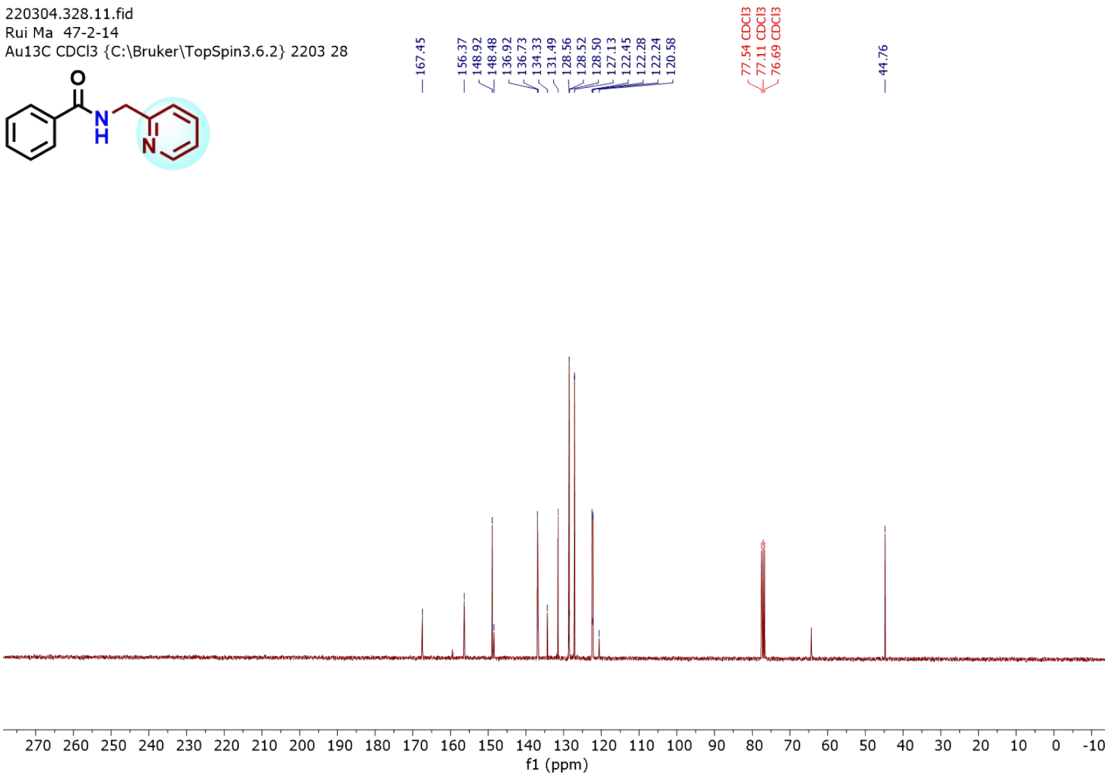




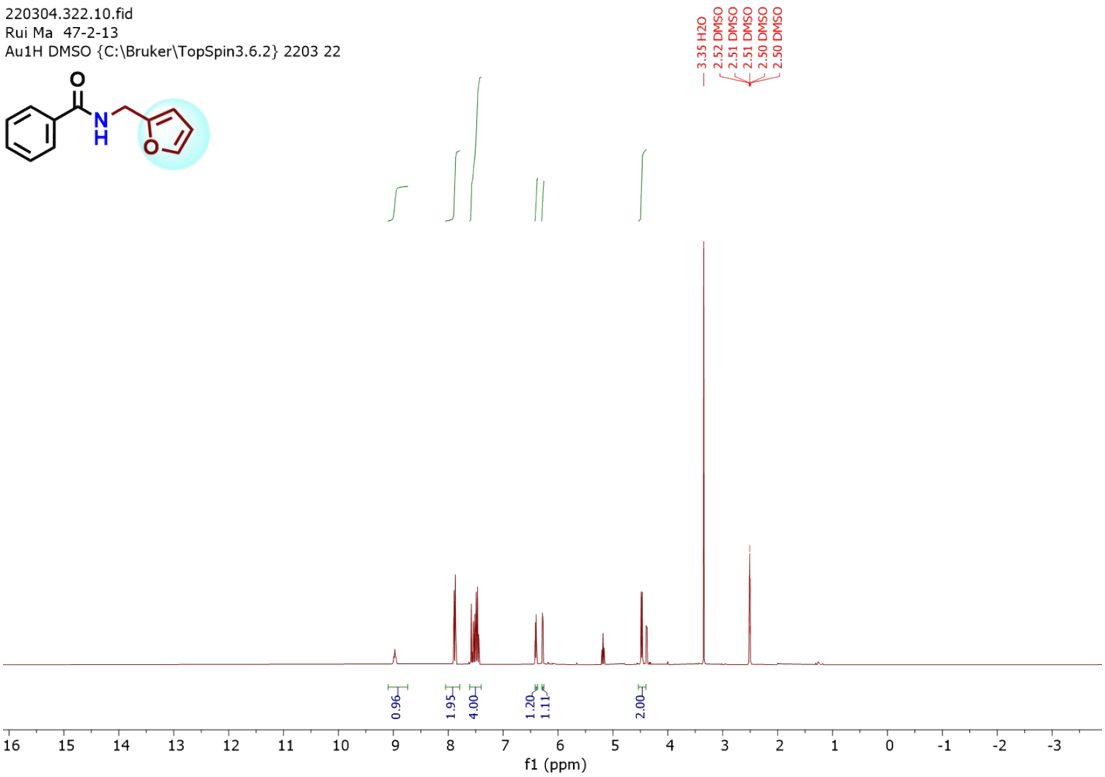
220304.328.10.fid
Rui Ma 47-2-14
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 28



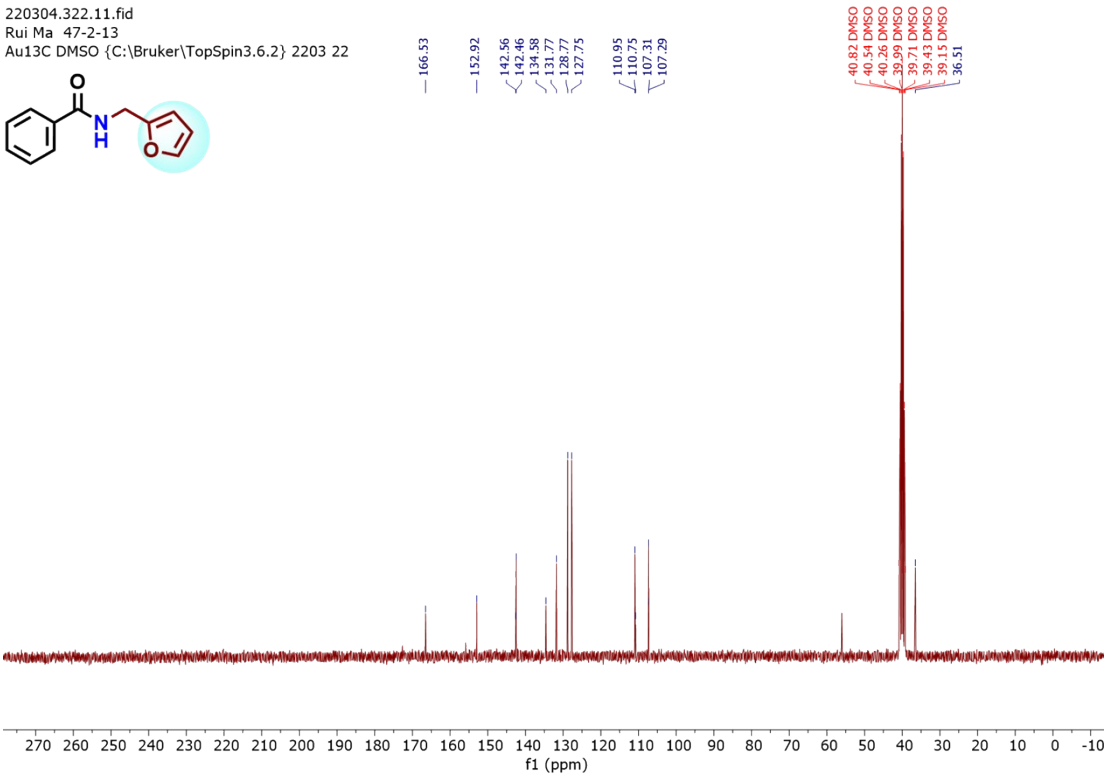
220304.328.11.fid
Rui Ma 47-2-14
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 28



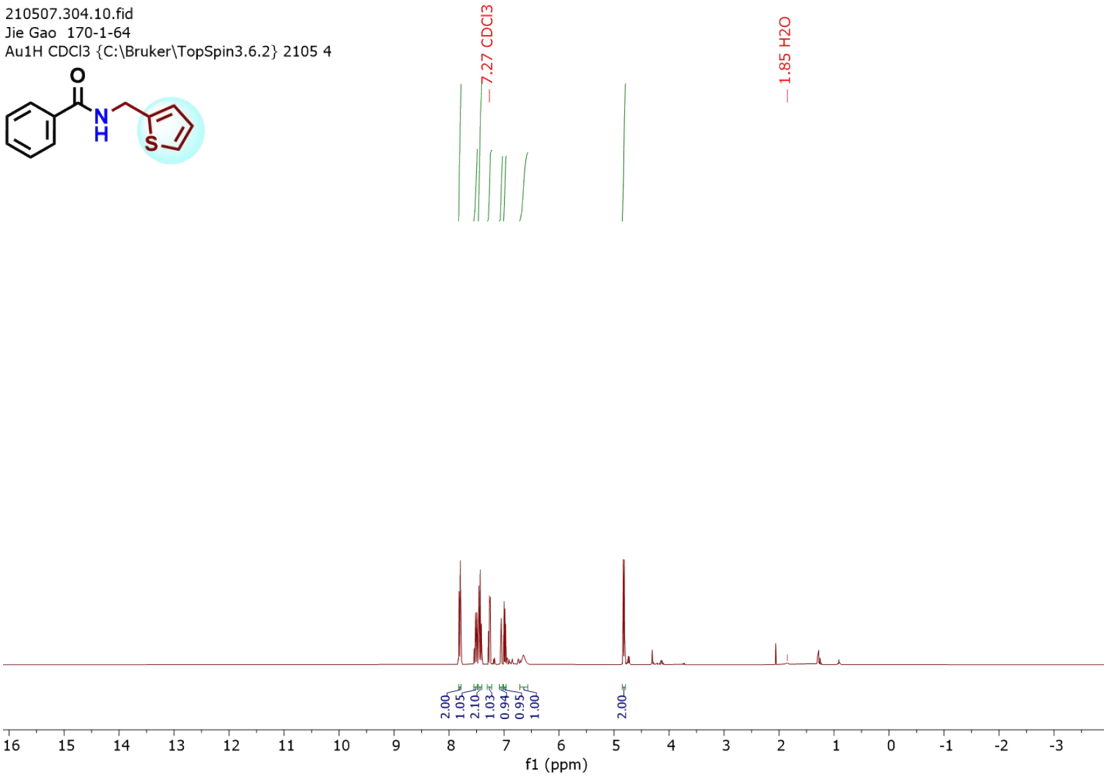
220304.322.10.fid
Rui Ma 47-2-13
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2203 22



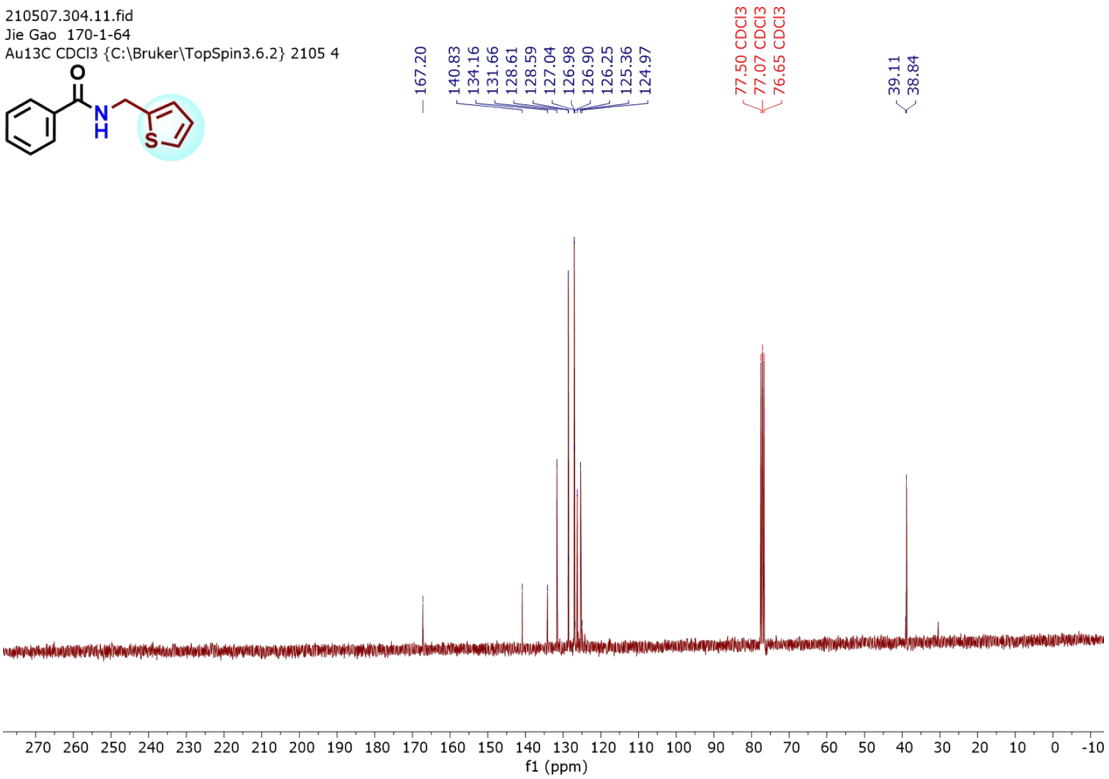
220304.322.11.fid
Rui Ma 47-2-13
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2203 22



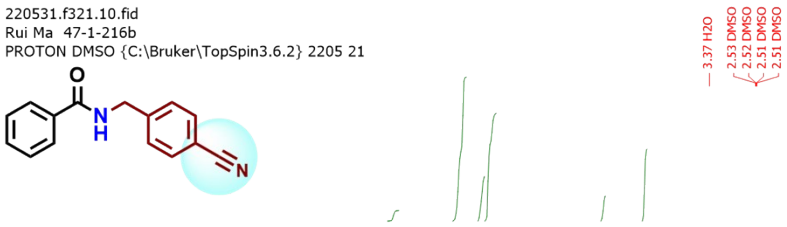
210507.304.10.fid
Jie Gao 170-1-64
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 4

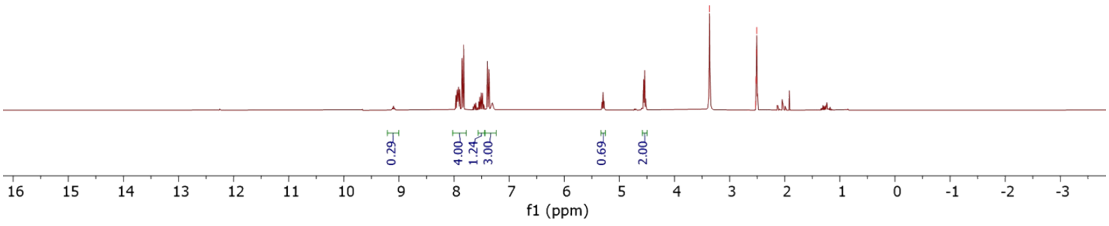


210507.304.11.fid
Jie Gao 170-1-64
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 4

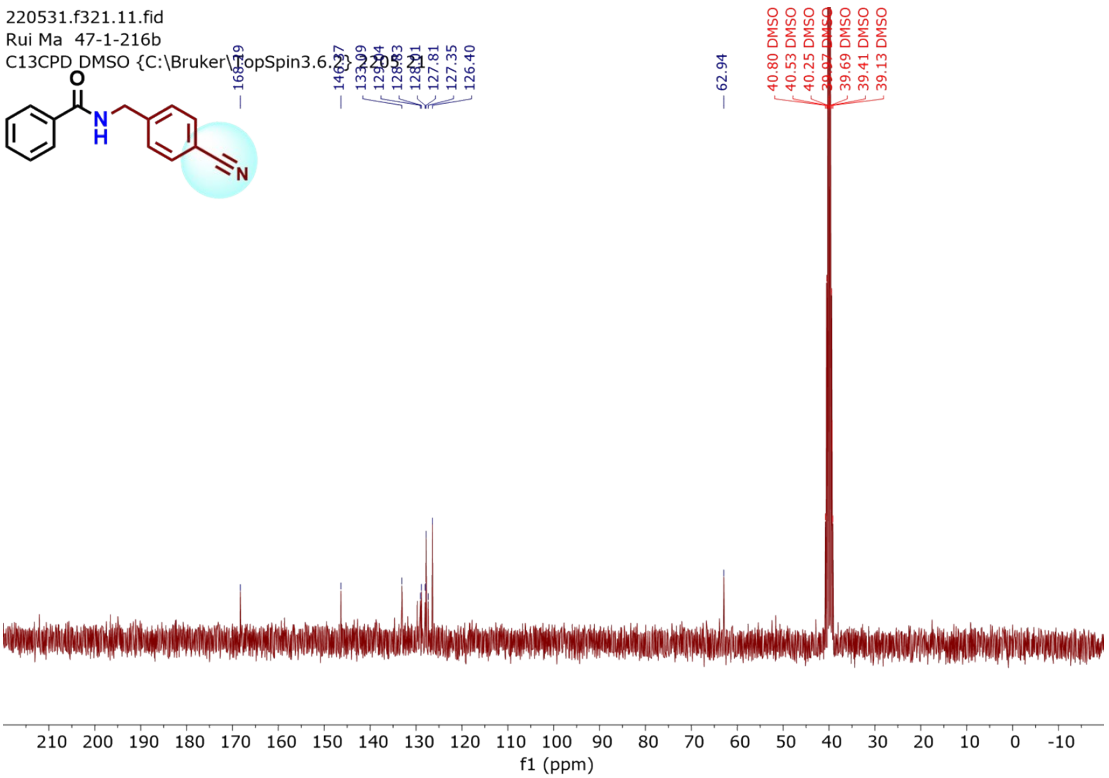


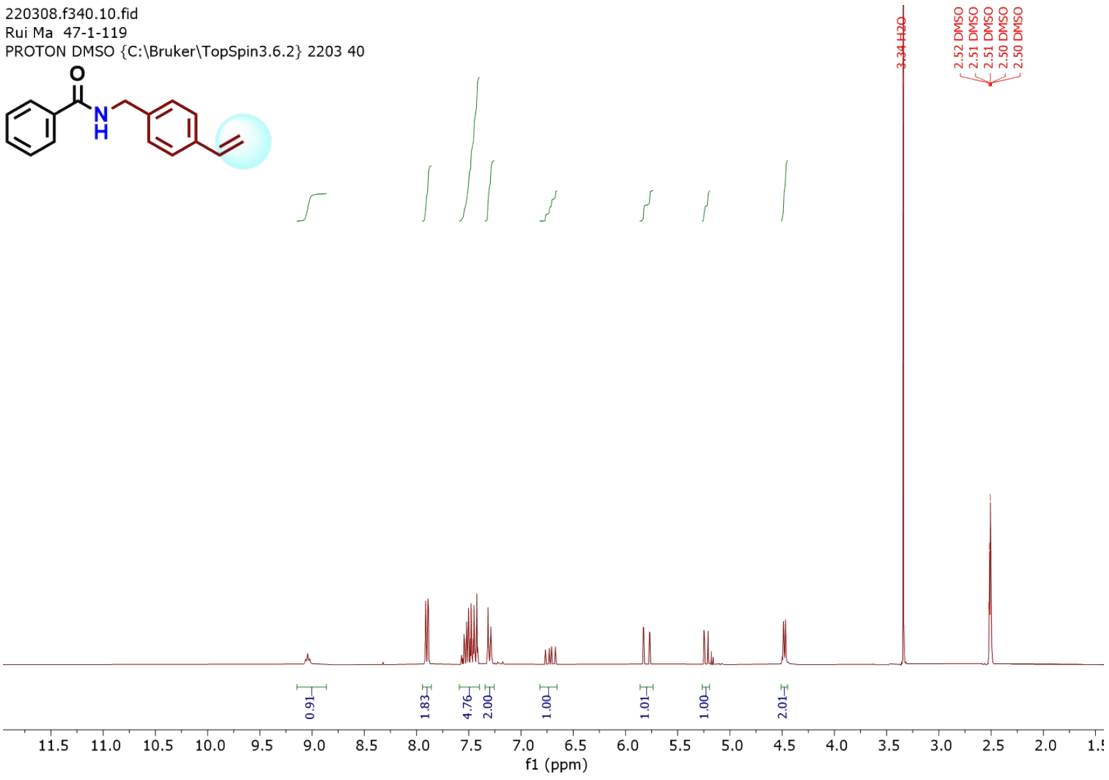
220531.f321.10.fid
Rui Ma 47-1-216b
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2205 21

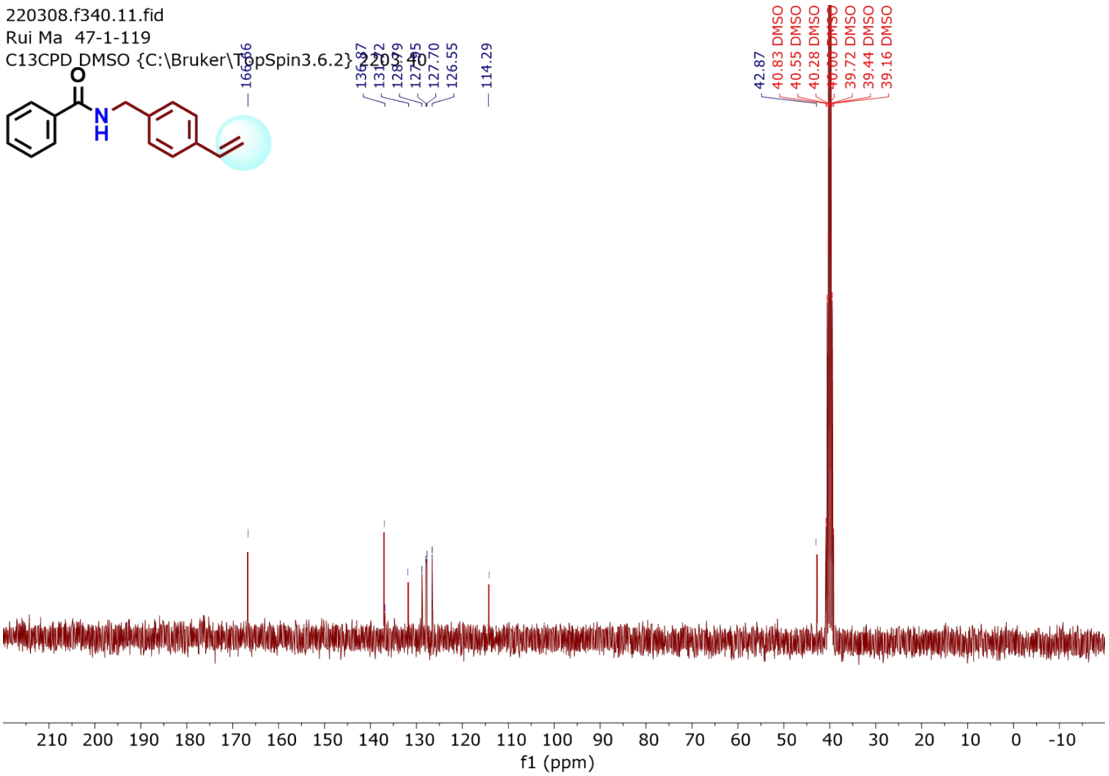




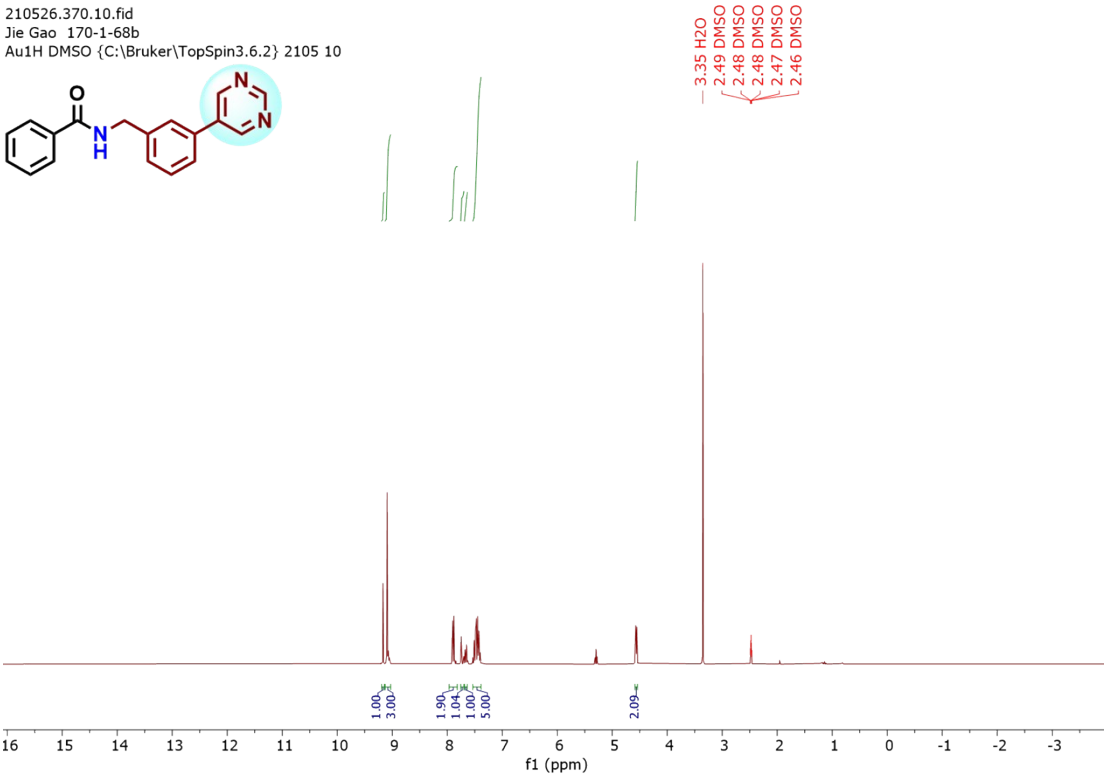
220531.f321.11.fid
Rui Ma 47-1-216b
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2205 21



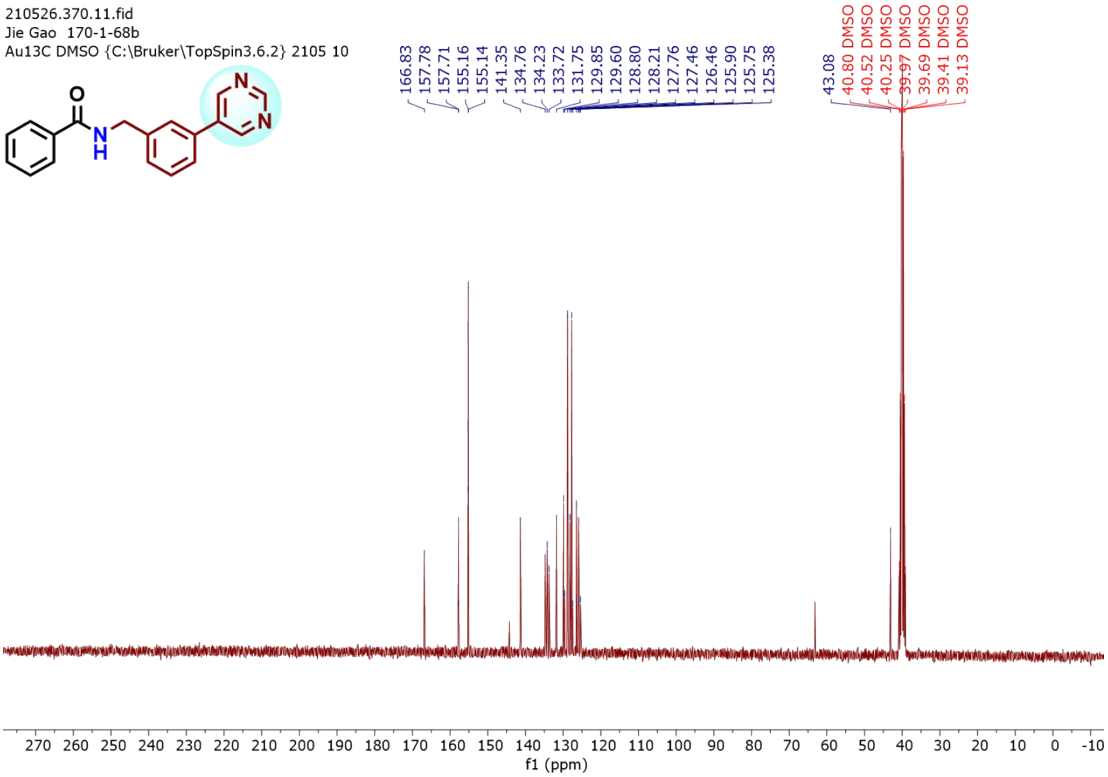

220308.f340.10.fid
Rui Ma 47-1-119
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2203 40
3.34 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
0.91
1.83
4.76
2.00
1.00
1.01
1.00
2.01
f1 (ppm)


220308.f340.11.fid
Rui Ma 47-1-119
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2203 40
166.66
136.87
131.92
128.79
127.75
127.70
126.55
114.29
42.87
40.83 DMSO
40.55 DMSO
40.28 DMSO
40.00 DMSO
39.72 DMSO
39.44 DMSO
39.16 DMSO
f1 (ppm)

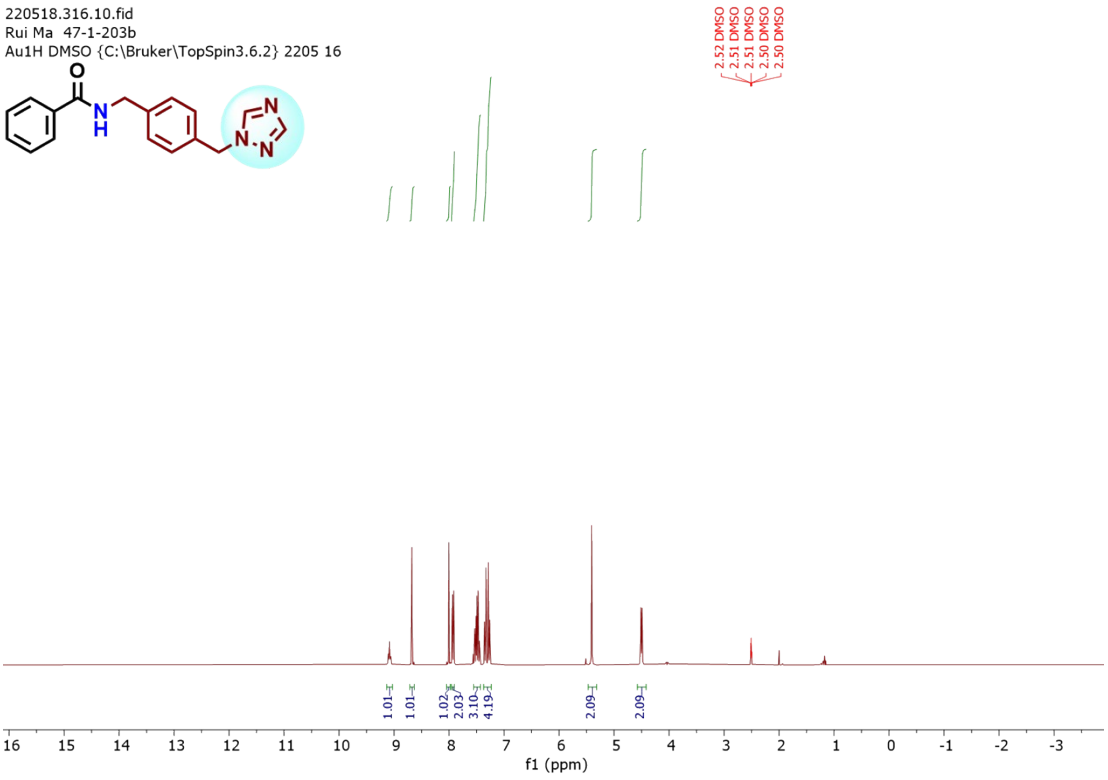
210526.370.10.fid
Jie Gao 170-1-68b
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 10



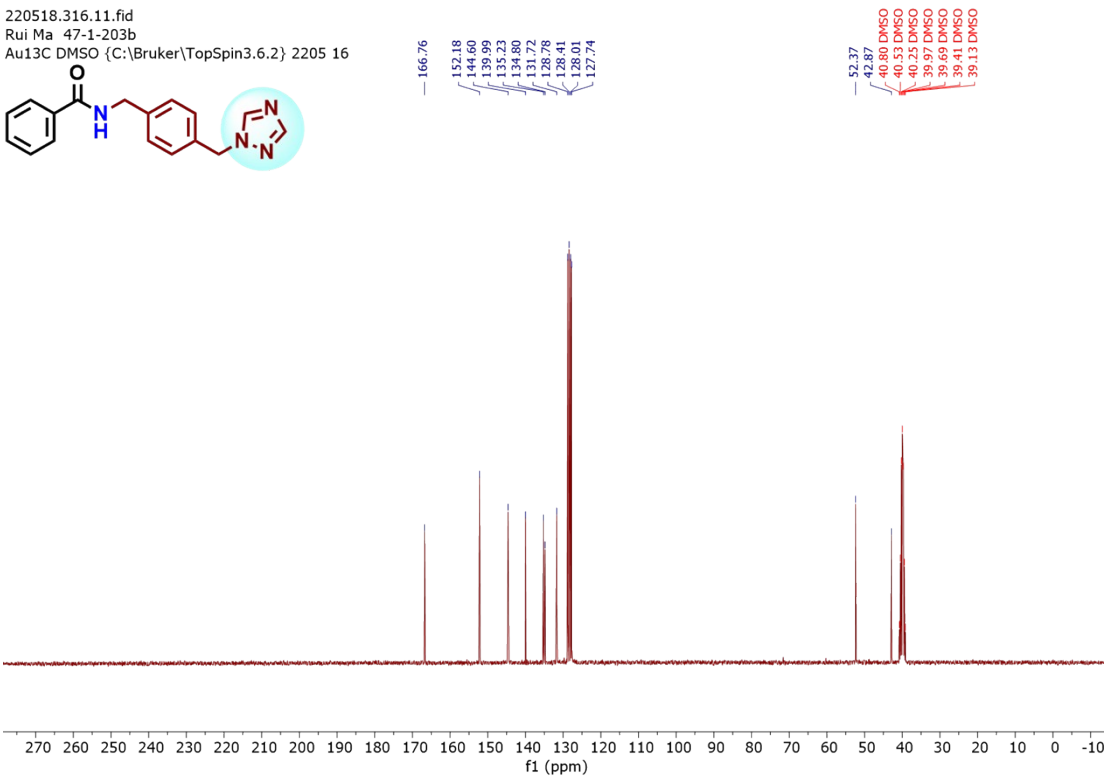
210526.370.11.fid
Jie Gao 170-1-68b
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 10



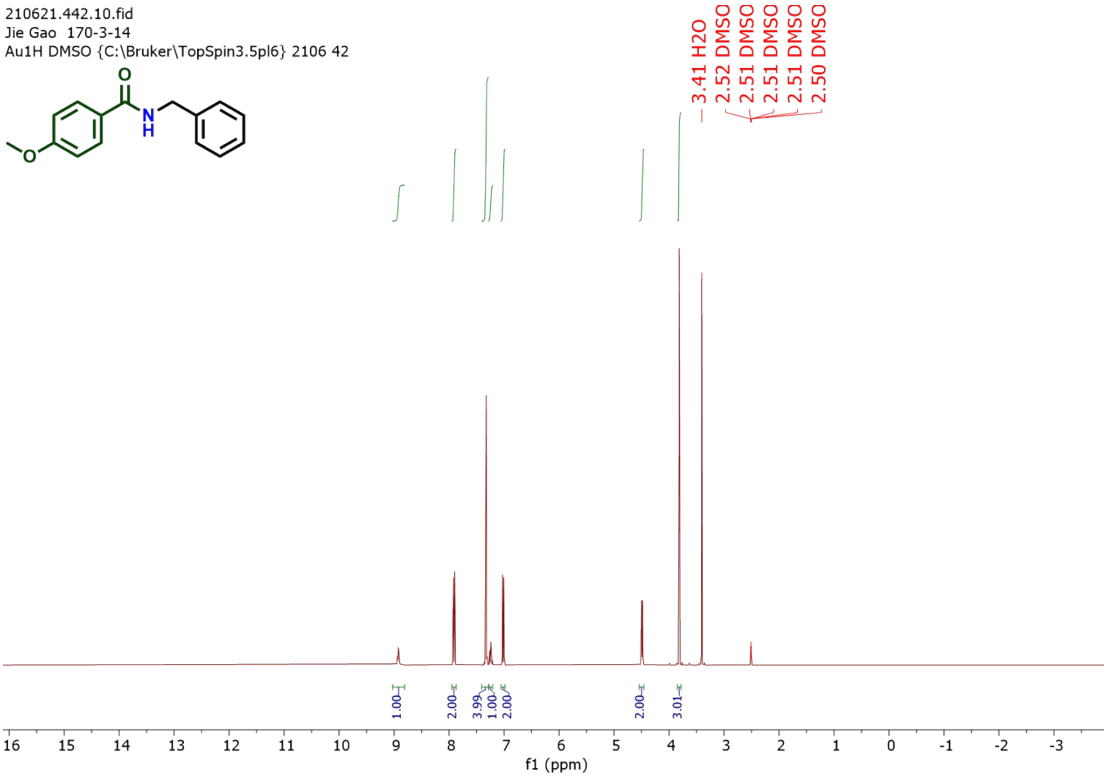
220518.316.10.fid
Rui Ma 47-1-203b
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2205 16



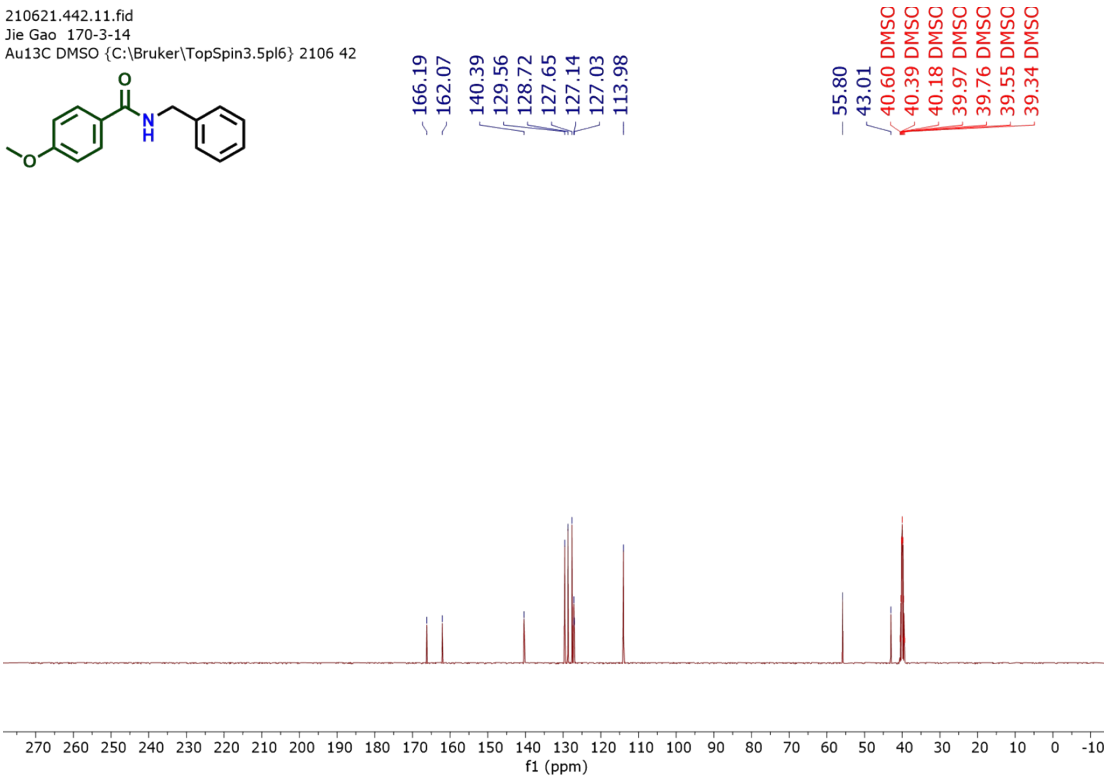
220518.316.11.fid
Rui Ma 47-1-203b
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2205 16



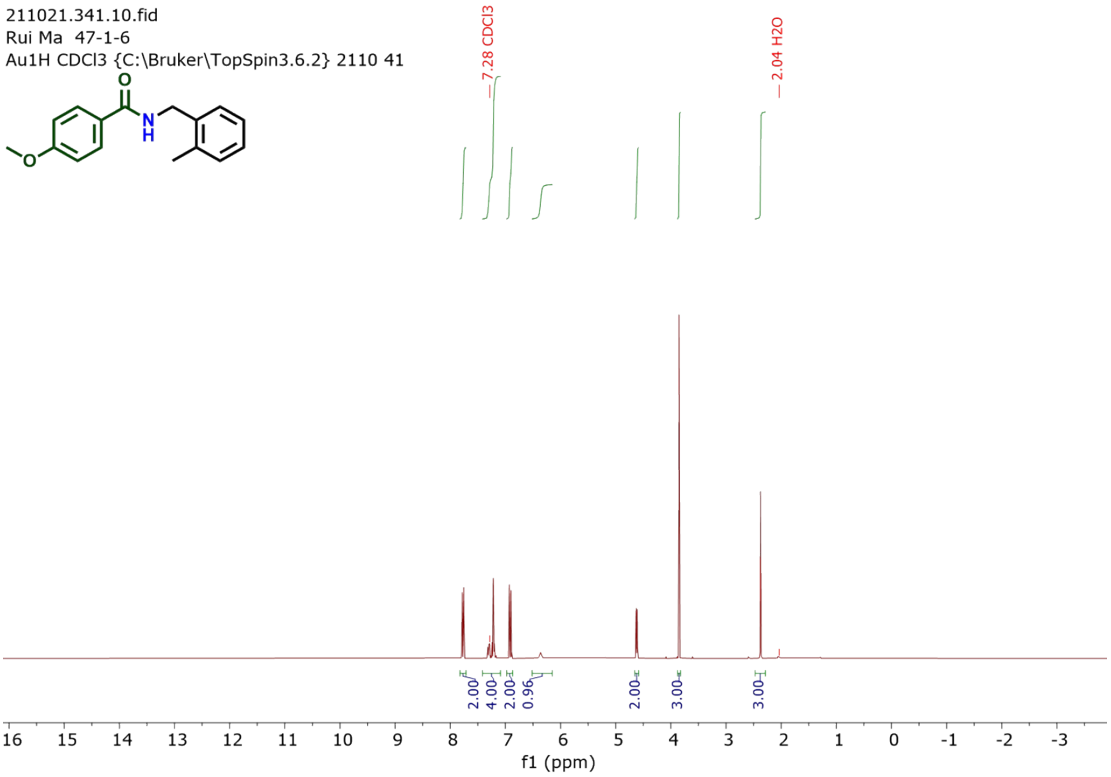
210621.442.10.fid
Jie Gao 170-3-14
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2106 42



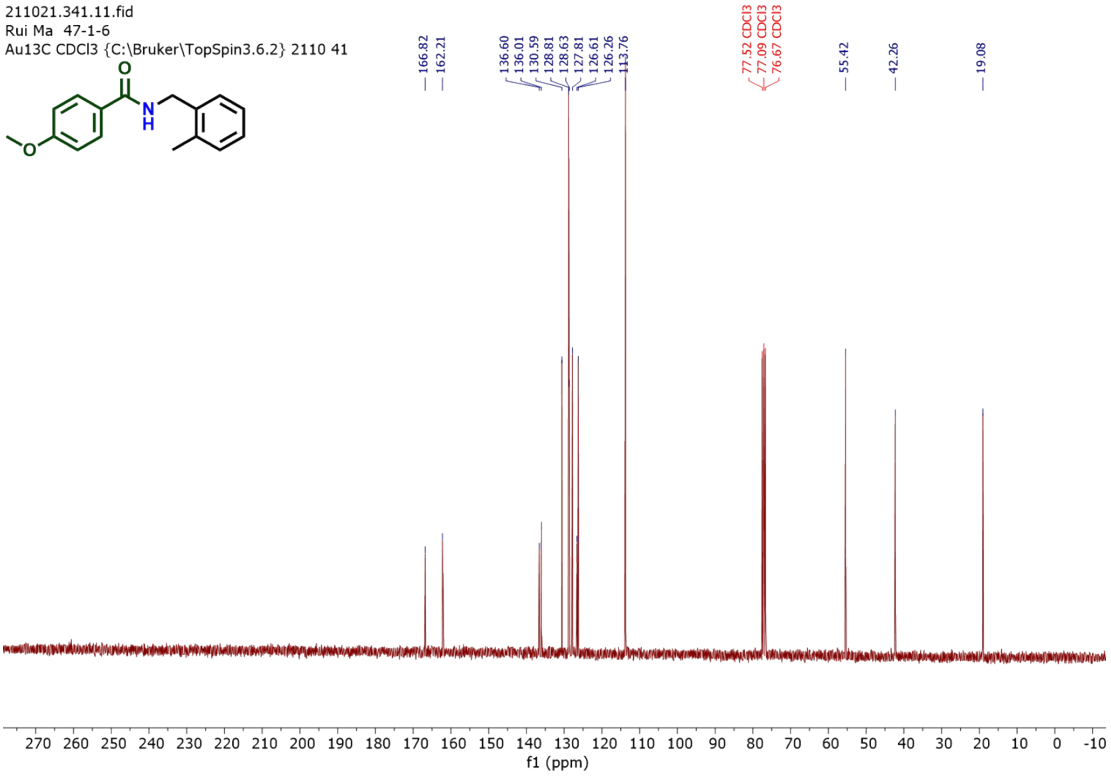
210621.442.11.fid
Jie Gao 170-3-14
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2106 42



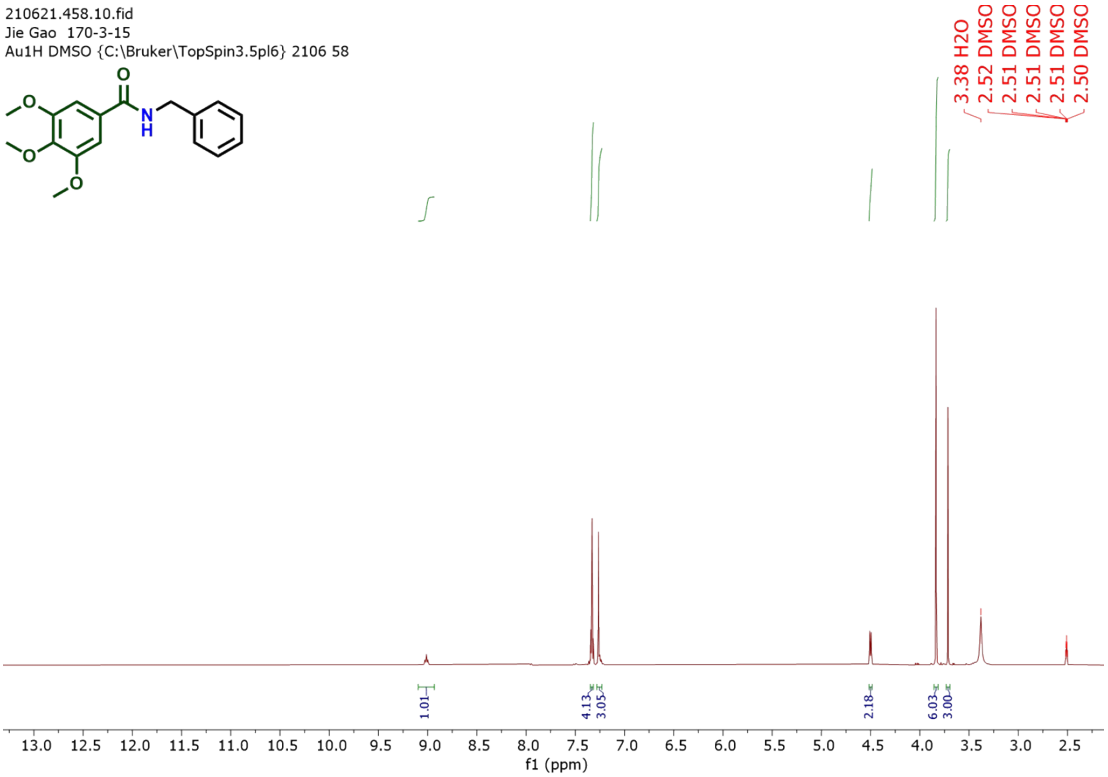
211021.341.10.fid
Rui Ma 47-1-6
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 41



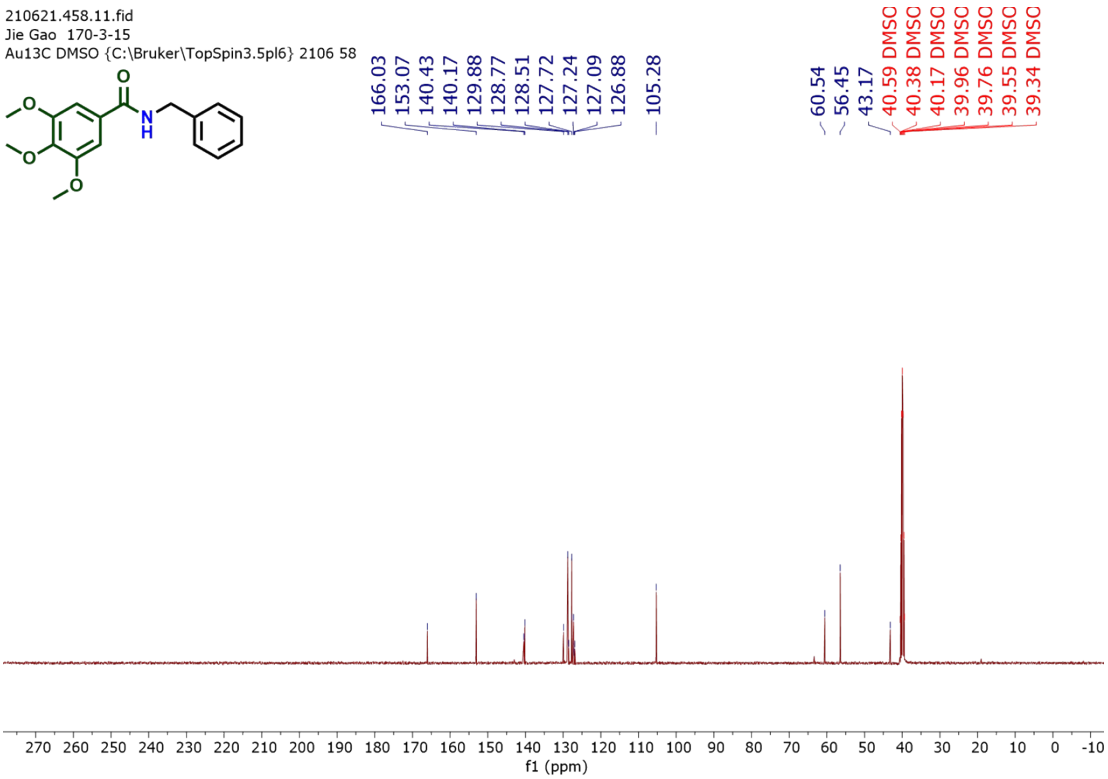
211021.341.11.fid
Rui Ma 47-1-6
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 41



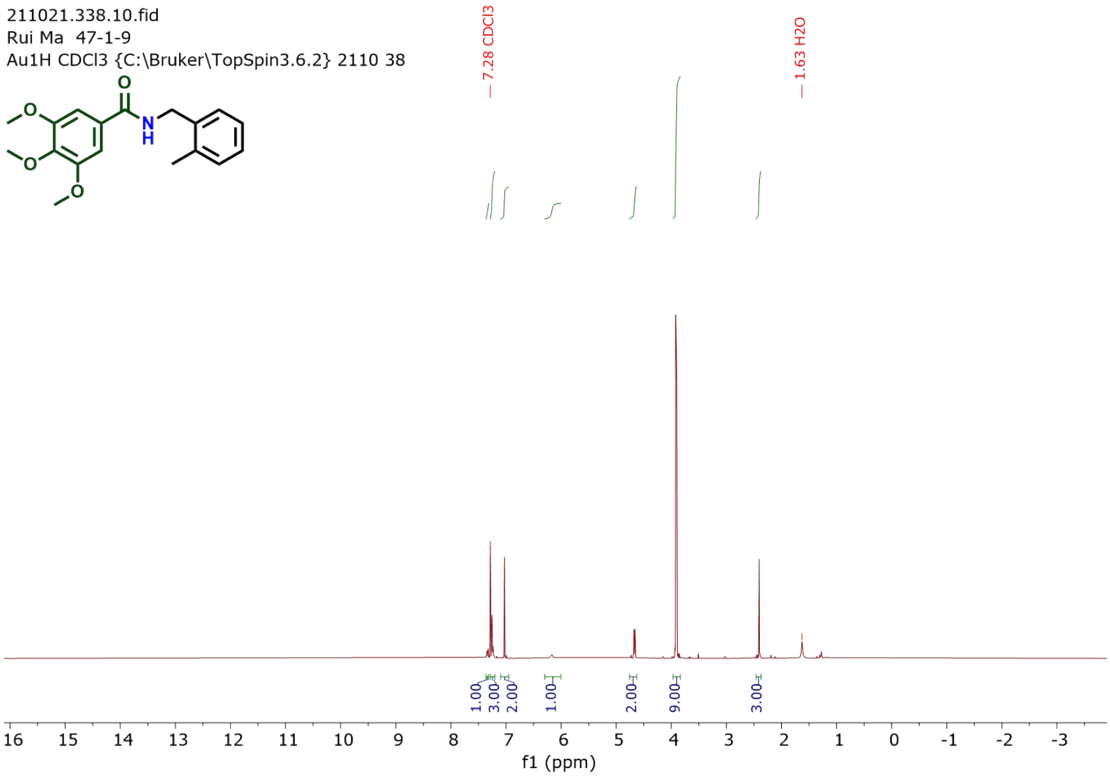
210621.458.10.fid
Jie Gao 170-3-15
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2106 58



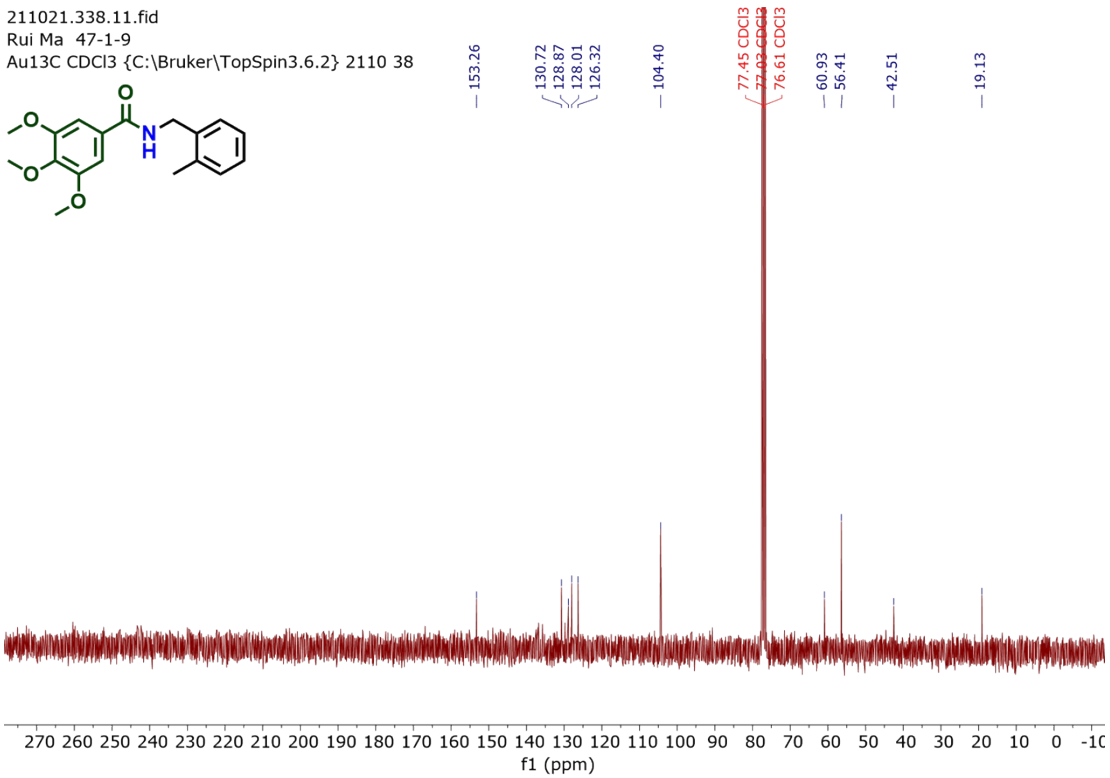
210621.458.11.fid
Jie Gao 170-3-15
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2106 58

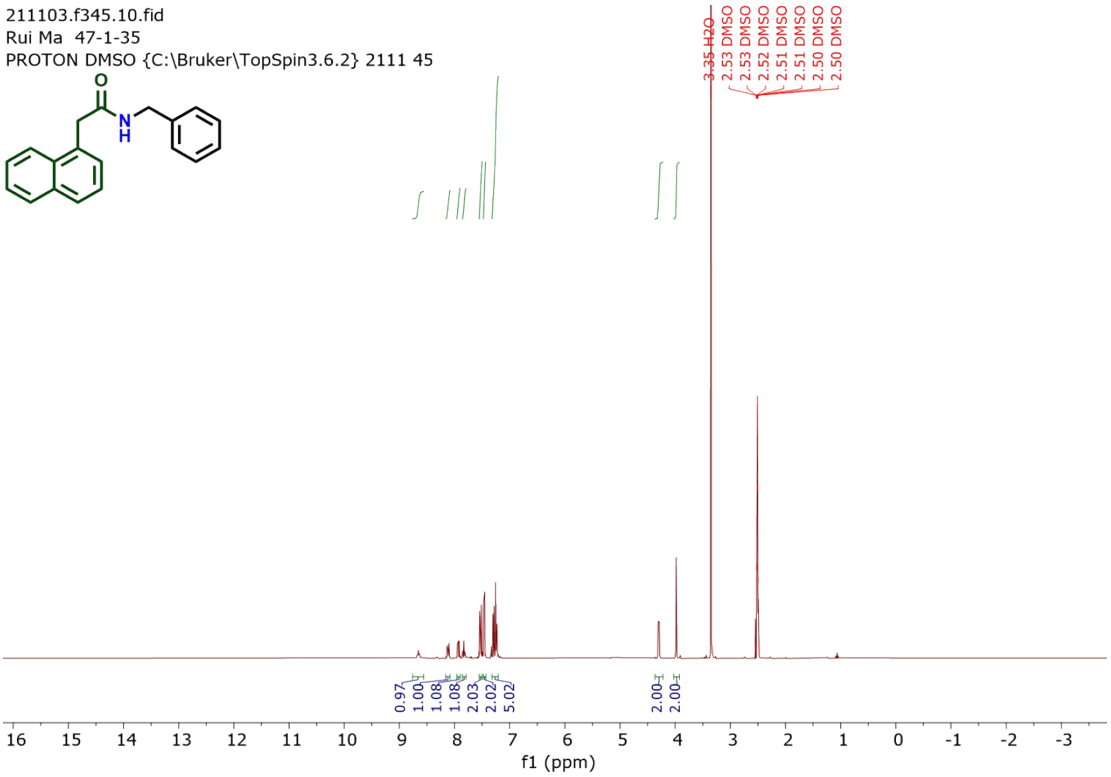


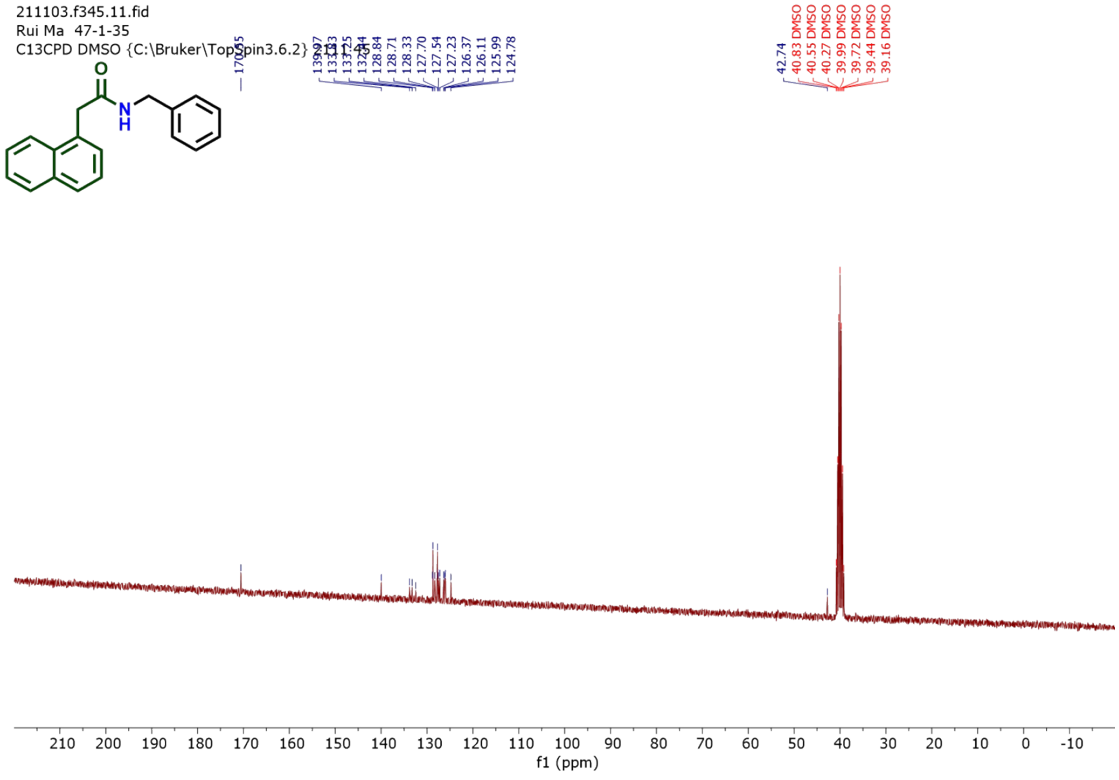
211021.338.10.fid
Rui Ma 47-1-9
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 38

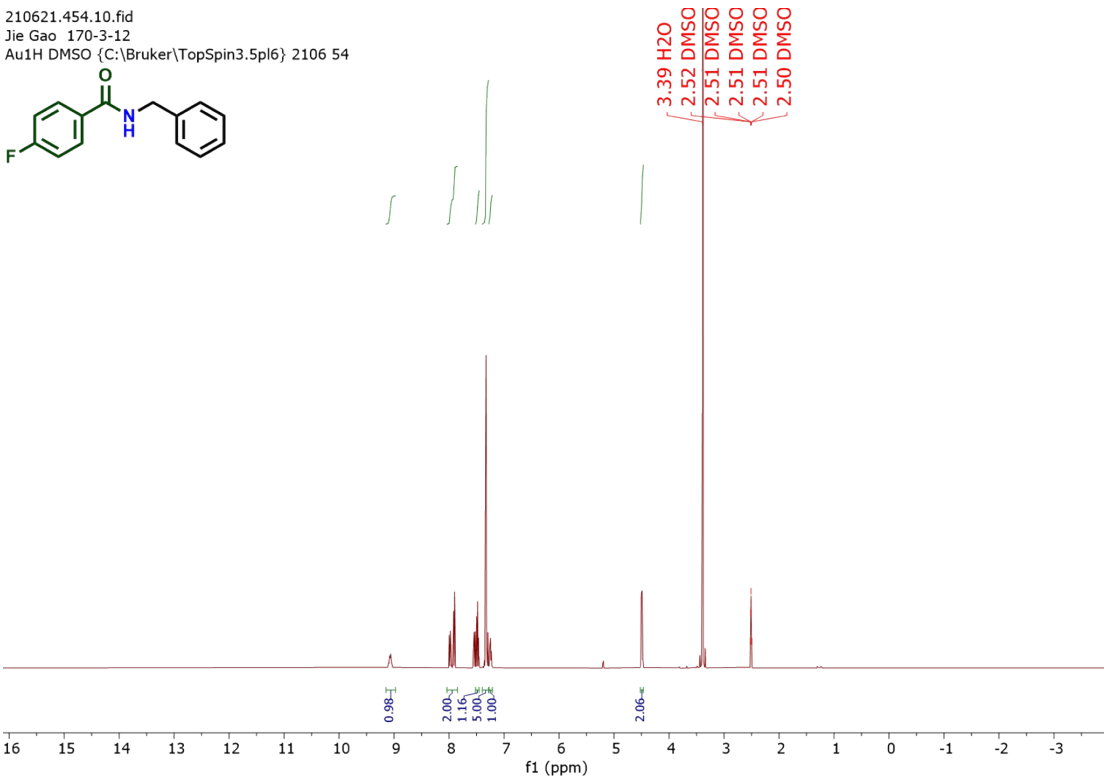


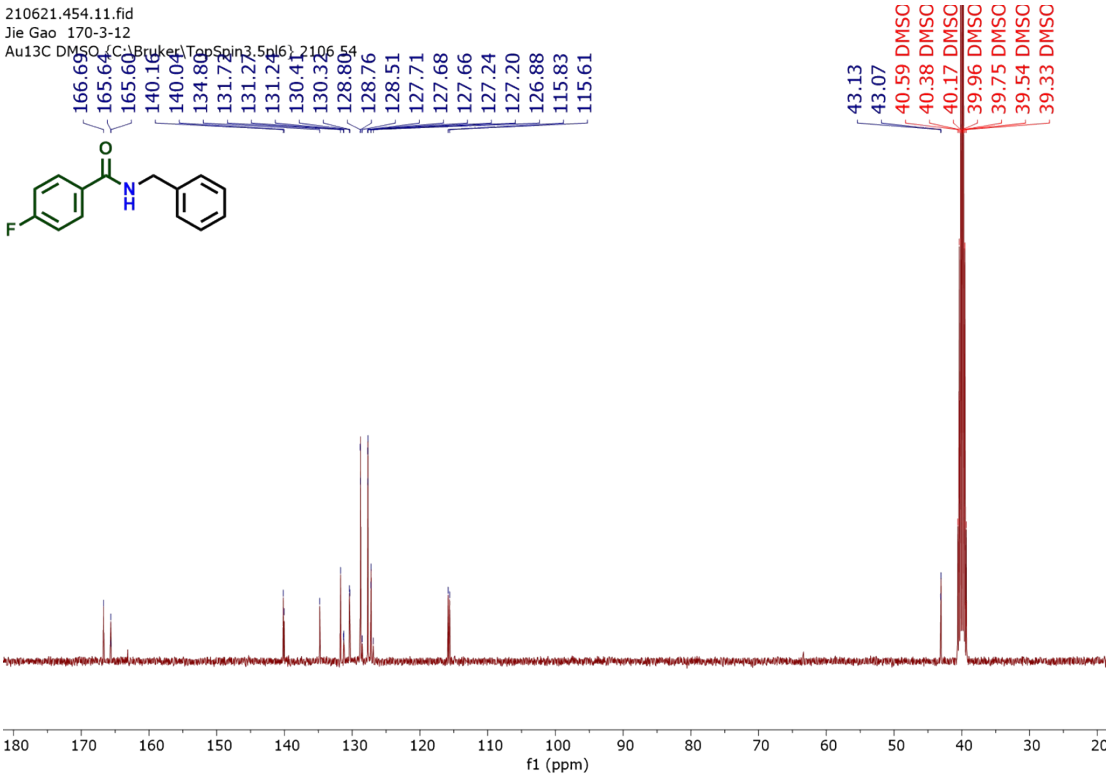
211021.338.11.fid
Rui Ma 47-1-9
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 38



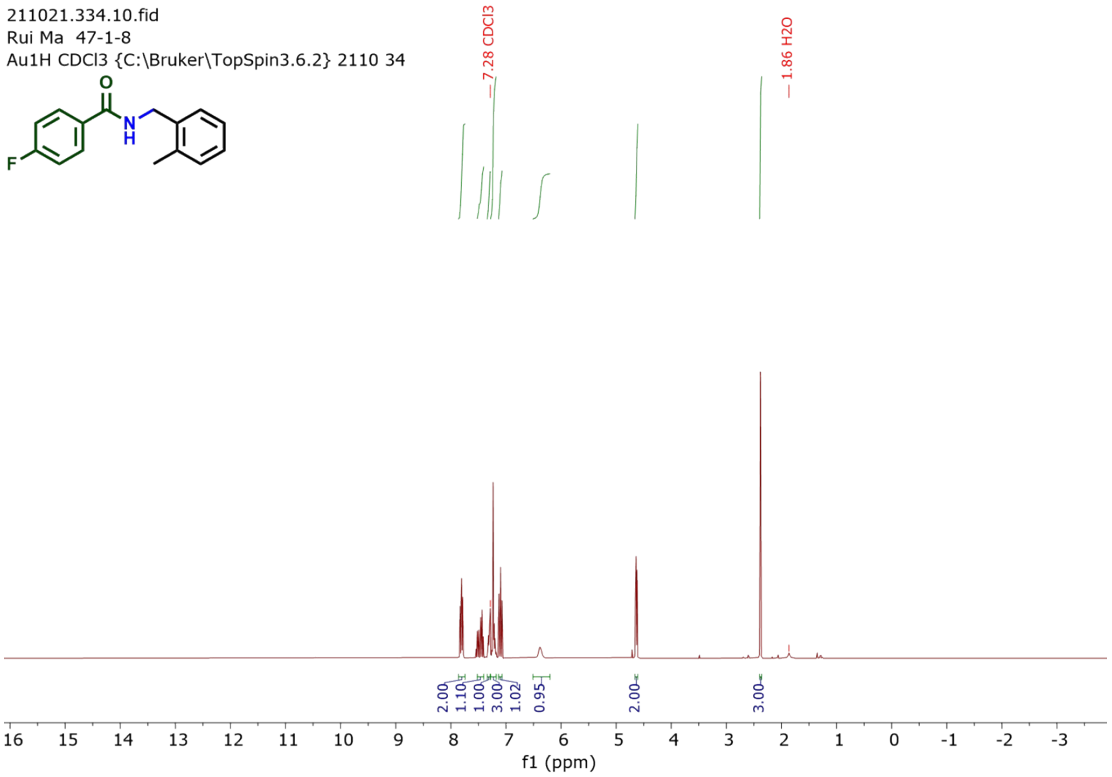

211103.f345.10.fid
Rui Ma 47-1-35
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2111 45
3.35 H2O
2.53 DMSO
2.53 DMSO
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
0.97
1.00
1.08
1.08
2.03
2.02
5.02
2.00
2.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


211103.f345.11.fid
Rui Ma 47-1-35
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2111 45
170.55
139.97
133.93
133.15
132.44
128.84
128.71
128.33
127.70
127.54
127.23
126.37
126.11
125.99
124.78
42.74
40.83 DMSO
40.55 DMSO
40.27 DMSO
39.99 DMSO
39.72 DMSO
39.44 DMSO
39.16 DMSO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

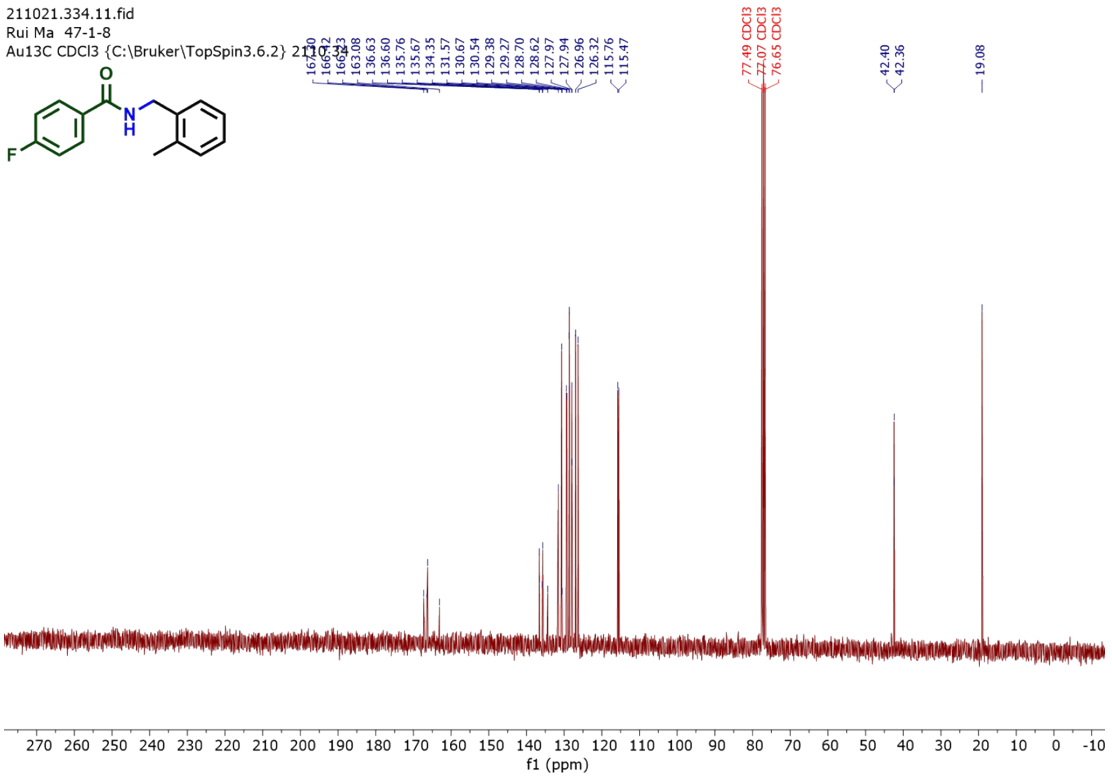

210621.454.10.fid
Jie Gao 170-3-12
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2106 54
3.39 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
0.98
2.00
1.16
5.00
1.00
2.06
f1 (ppm)

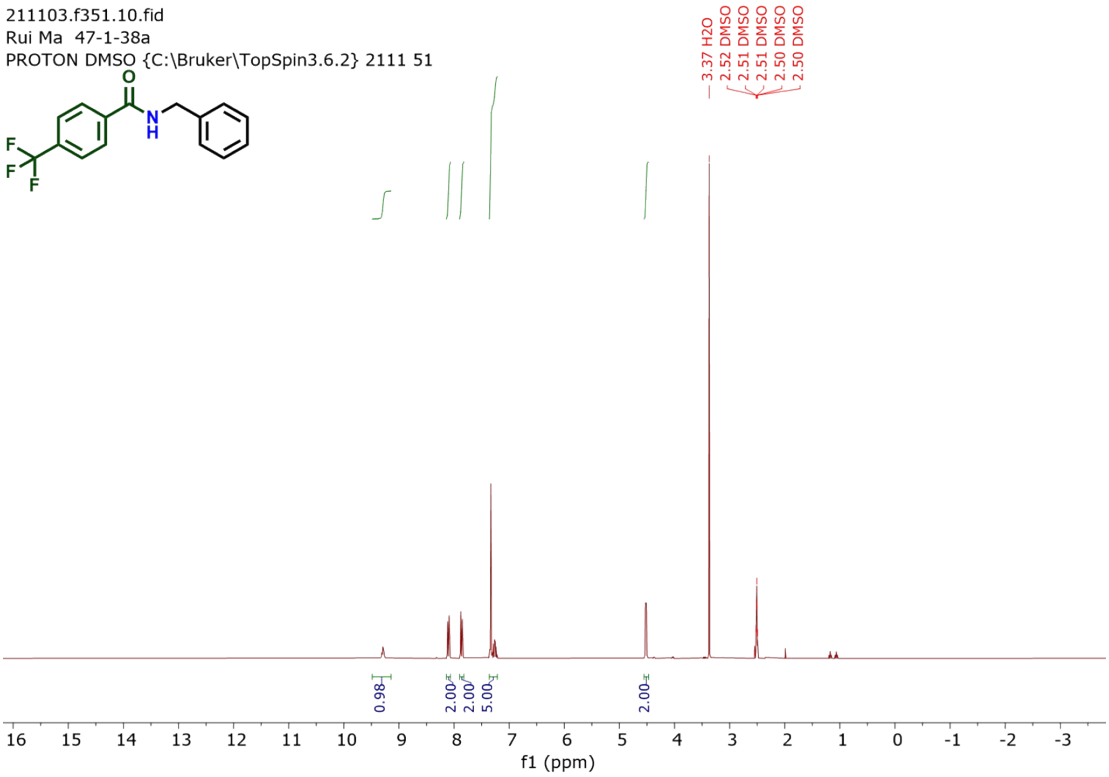

210621.454.11.fid
Jie Gao 170-3-12
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2106 54
166.69
165.64
165.60
140.16
140.04
134.80
131.72
131.27
131.24
130.41
130.32
128.80
128.76
128.51
127.71
127.68
127.66
127.24
127.20
126.88
115.83
115.61
43.13
43.07
40.59 DMSO
40.38 DMSO
40.17 DMSO
39.96 DMSO
39.75 DMSO
39.54 DMSO
39.33 DMSO
f1 (ppm)

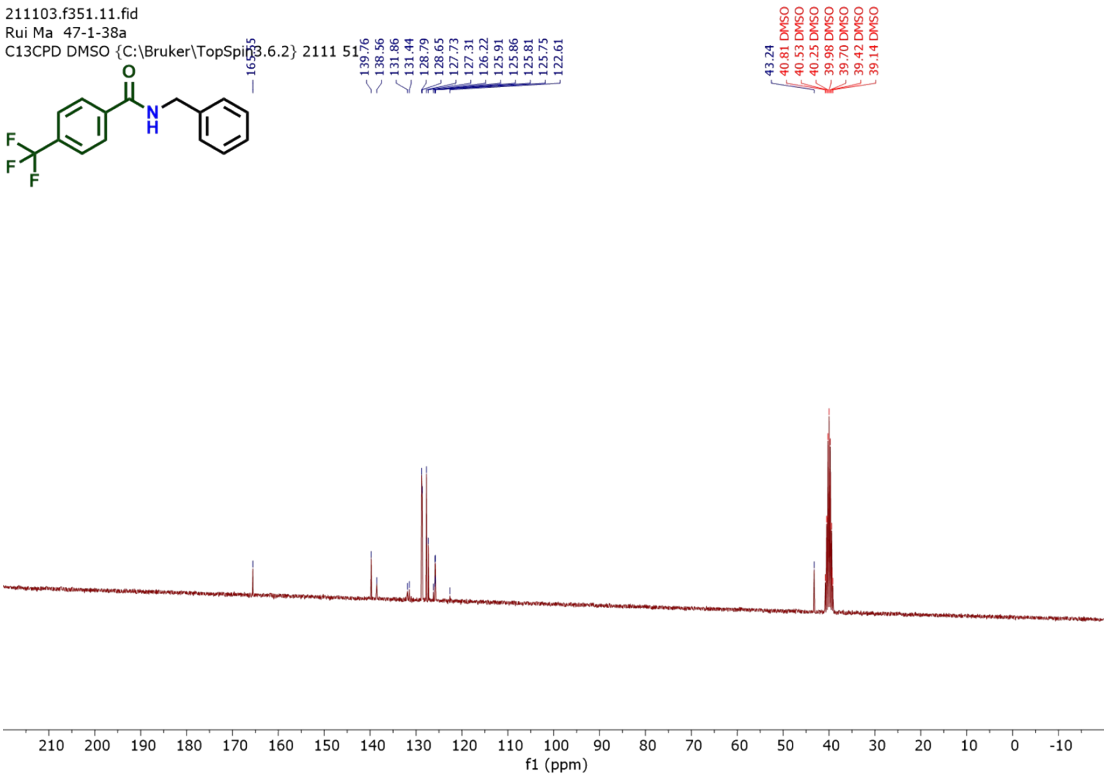
211021.334.10.fid
Rui Ma 47-1-8
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 34

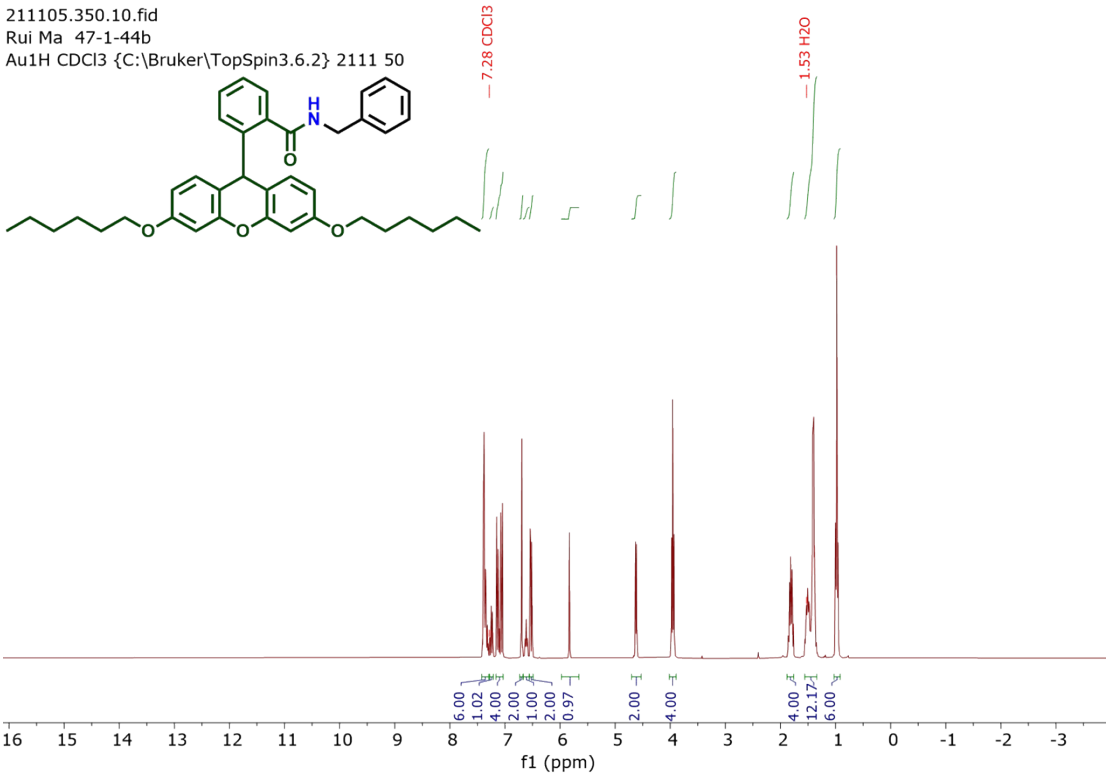


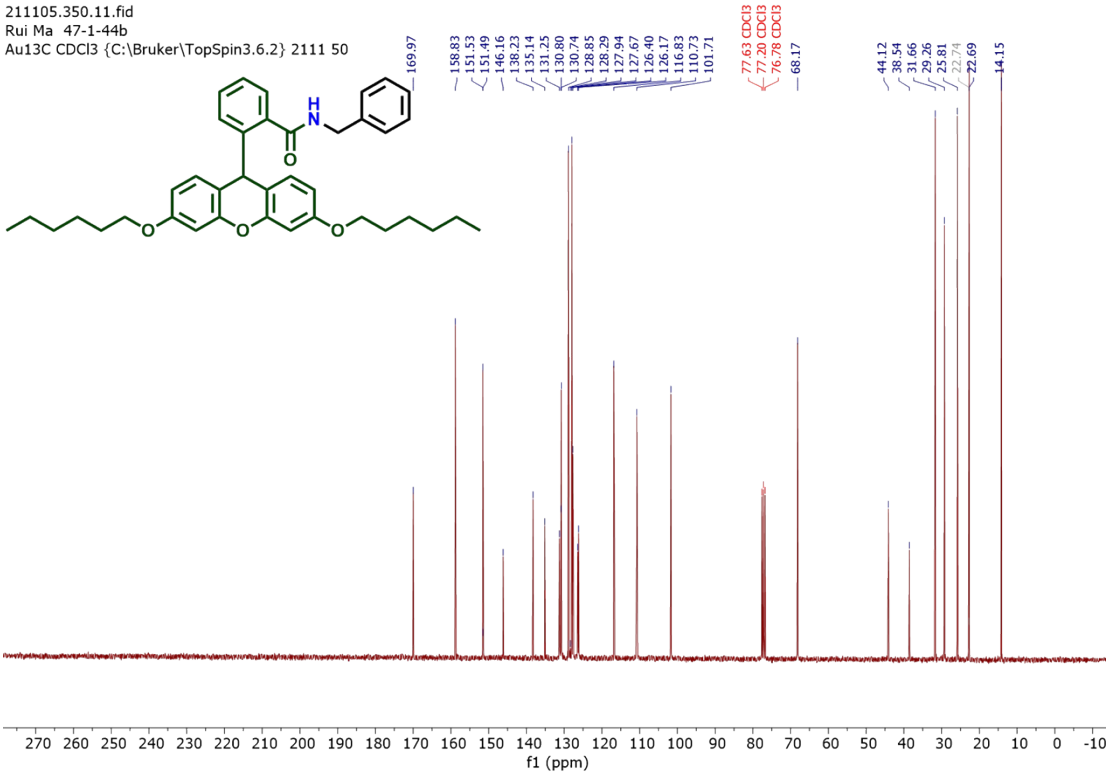
211021.334.11.fid
Rui Ma 47-1-8
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2110 34



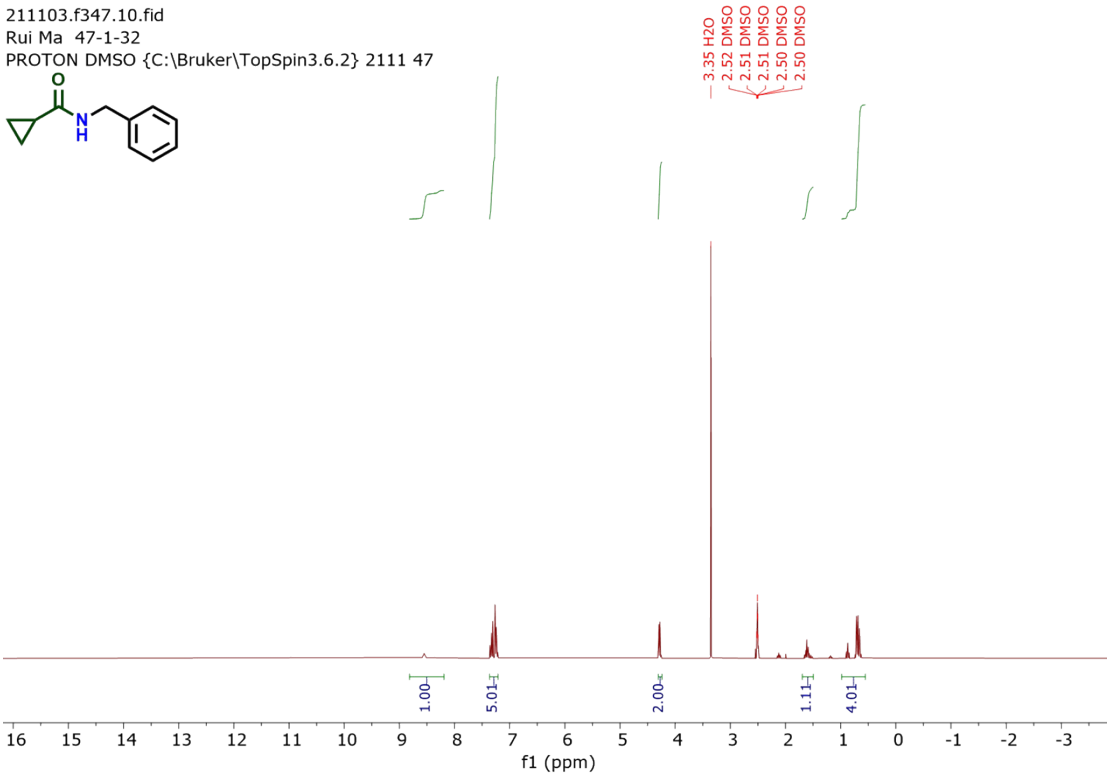

211103.f351.10.fid
Rui Ma 47-1-38a
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2111 51
3.37 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
0.98
2.00
2.00
5.00
2.00
f1 (ppm)


211103.f351.11.fid
Rui Ma 47-1-38a
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2111 51
139.76
138.56
131.86
131.44
128.79
128.65
127.73
127.31
126.22
125.91
125.86
125.81
125.75
122.61
43.24
40.81 DMSO
40.53 DMSO
40.25 DMSO
39.98 DMSO
39.70 DMSO
39.42 DMSO
39.14 DMSO
f1 (ppm)

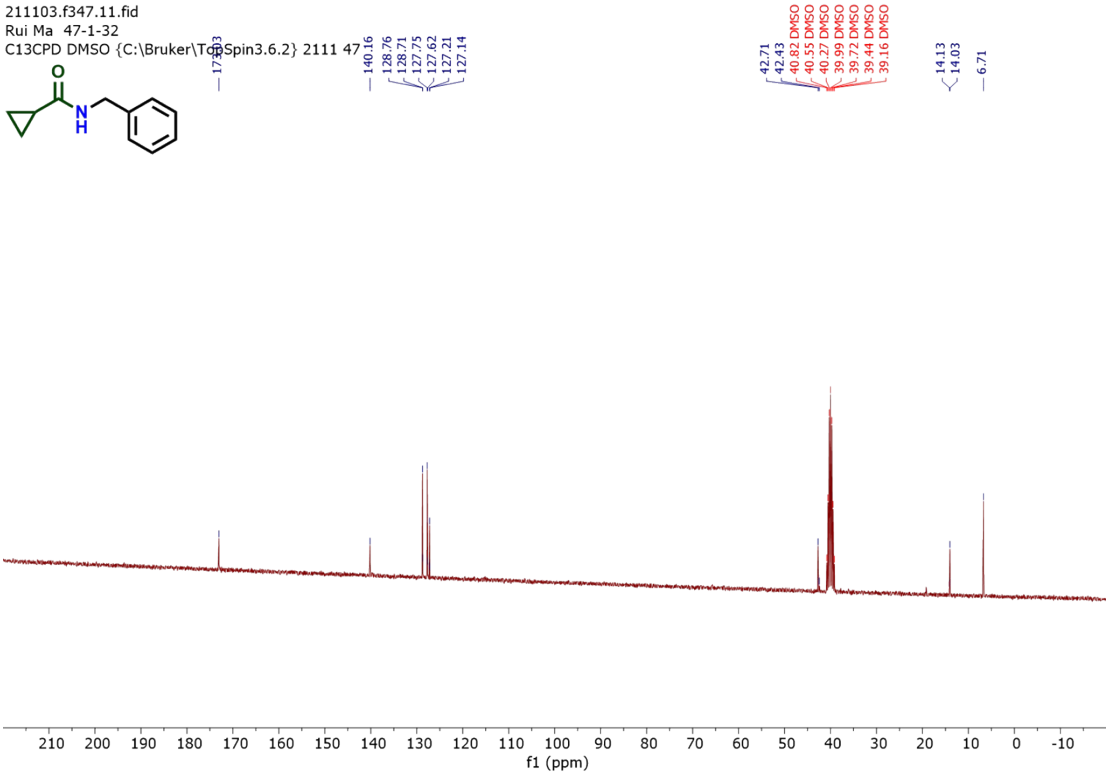

211105.350.10.fid
Rui Ma 47-1-44b
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 50
7.28 CDCl3
1.53 H2O
6.00
1.02
4.00
2.00
1.00
2.00
0.97
2.00
4.00
4.00
12.17
6.00
f1 (ppm)

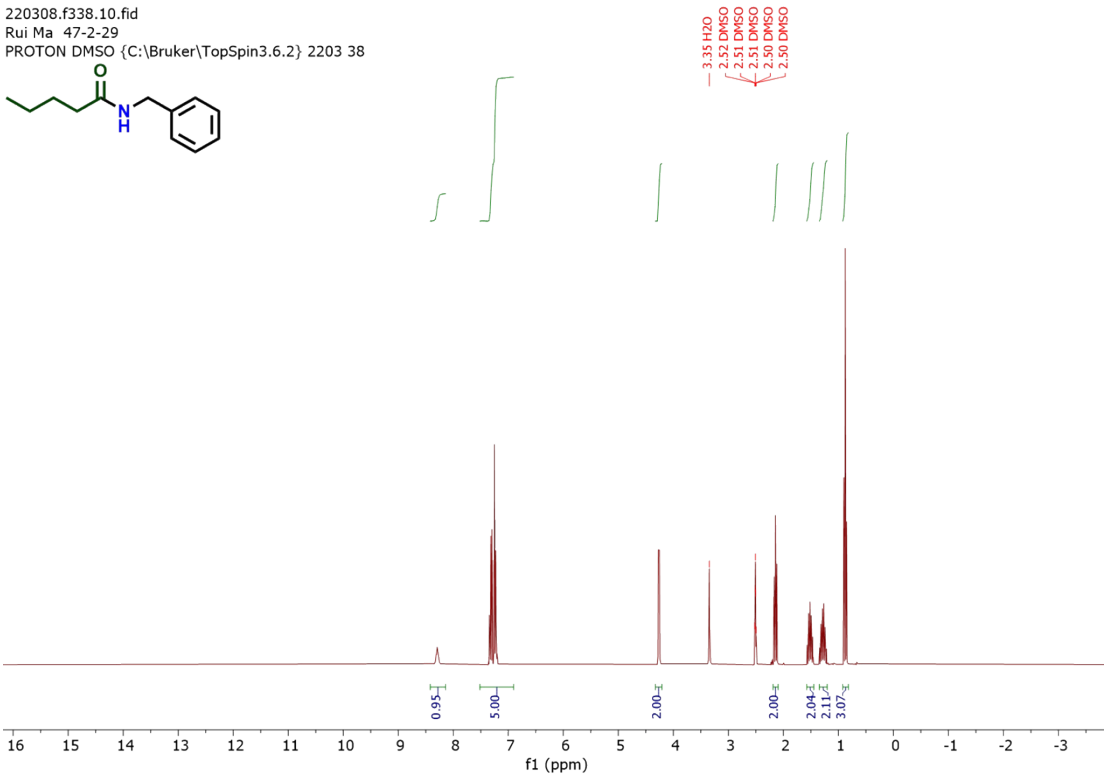

211105.350.11.fid
Rui Ma 47-1-44b
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 50
169.97
158.83
151.53
151.49
146.16
138.23
135.14
131.25
130.80
130.74
128.85
128.29
127.94
127.67
126.40
126.17
116.83
110.73
101.71
77.63 CDCl3
77.20 CDCl3
76.78 CDCl3
68.17
44.12
38.54
31.66
29.26
25.81
22.74
22.69
14.15
f1 (ppm)

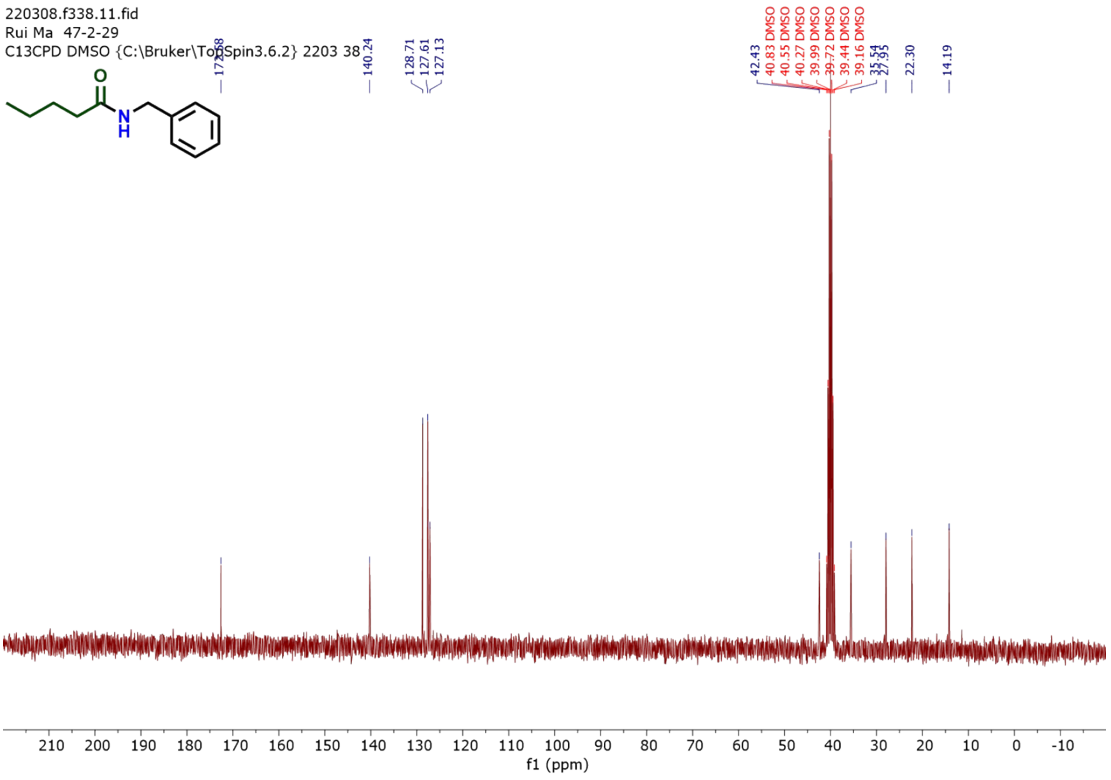
211103.f347.10.fid
Rui Ma 47-1-32
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2111 47



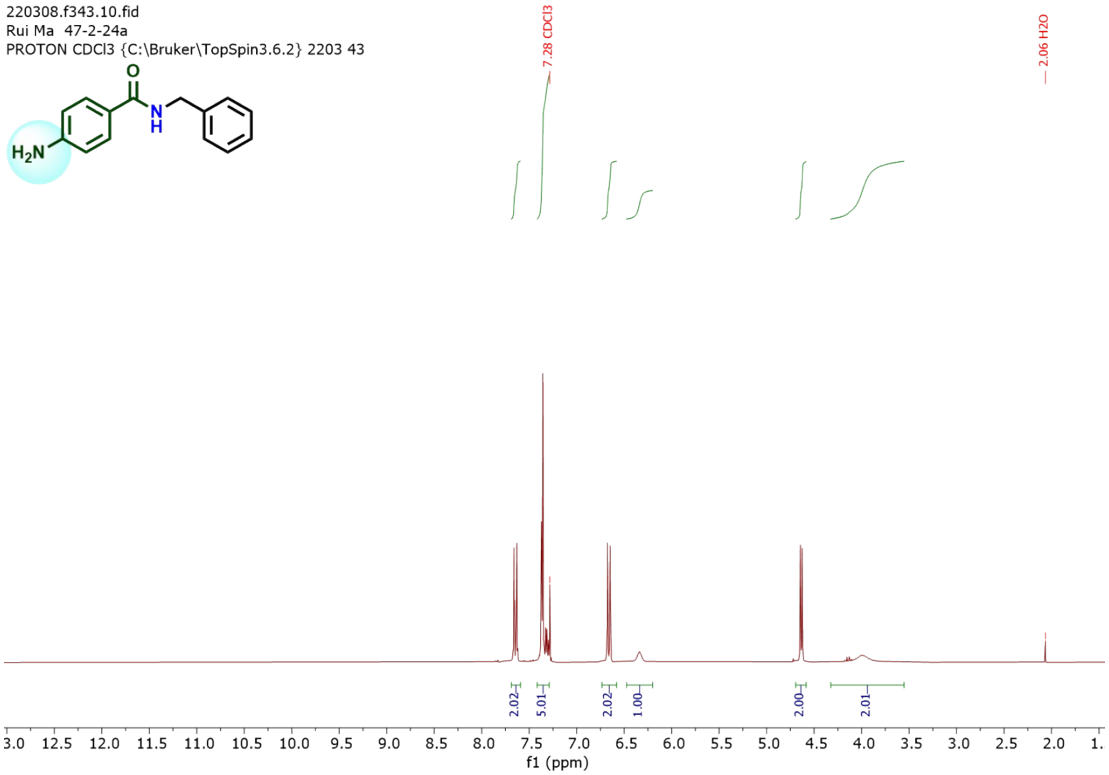
211103.f347.11.fid
Rui Ma 47-1-32
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2111 47



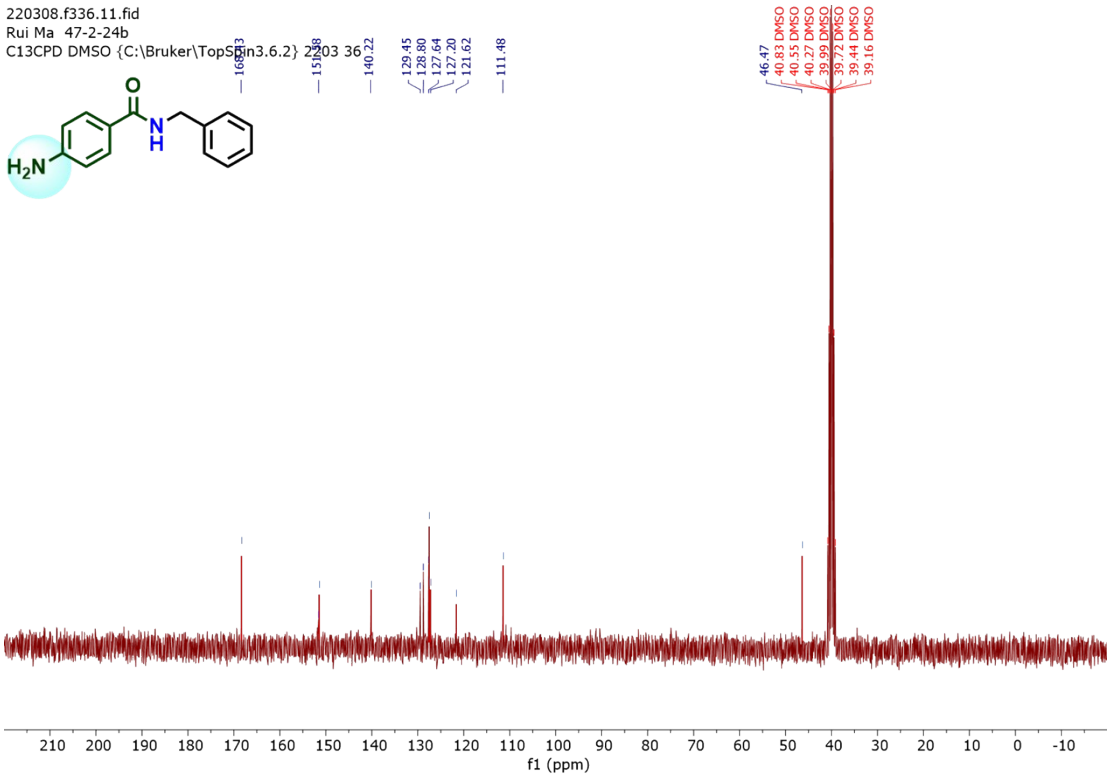

220308.f338.10.fid
Rui Ma 47-2-29
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2203 38
3.35 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
0.95
5.00
2.00
2.00
2.04
2.11
3.07
f1 (ppm)


220308.f338.11.fid
Rui Ma 47-2-29
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2203 38
140.24
128.71
127.61
127.13
42.43
40.83 DMSO
40.55 DMSO
40.27 DMSO
39.99 DMSO
39.72 DMSO
39.44 DMSO
39.16 DMSO
35.54
27.95
22.30
14.19
f1 (ppm)

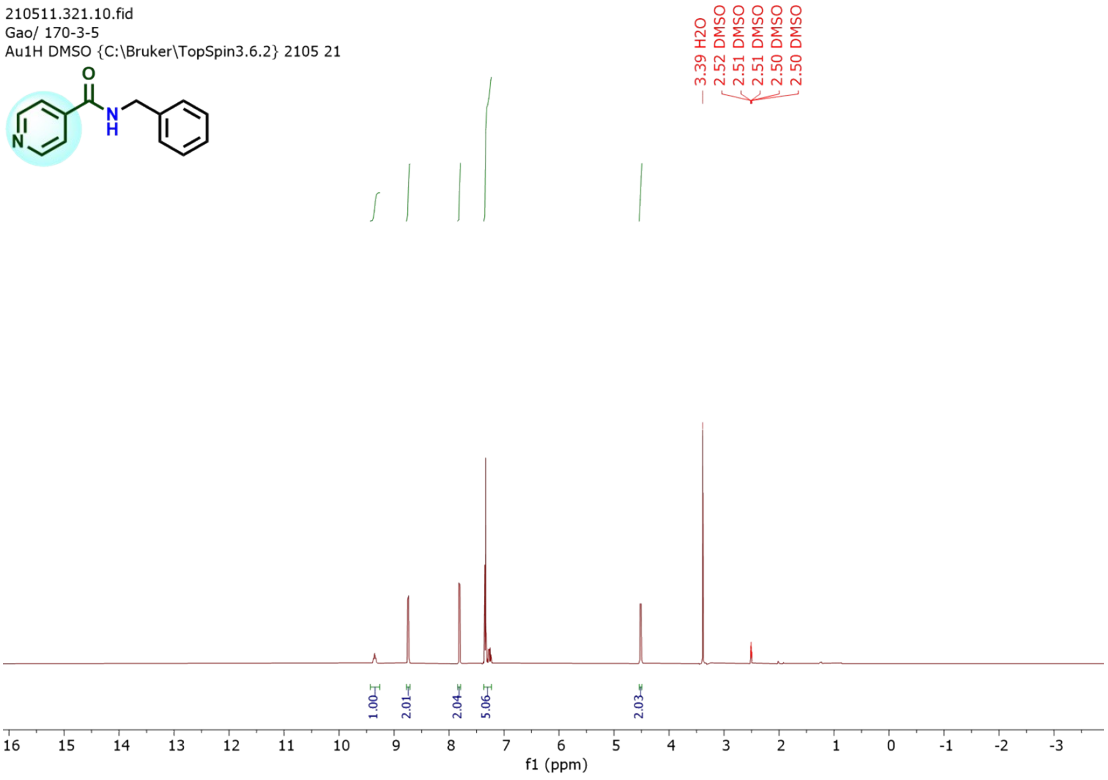
220308.f343.10.fid
Rui Ma 47-2-24a
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 43



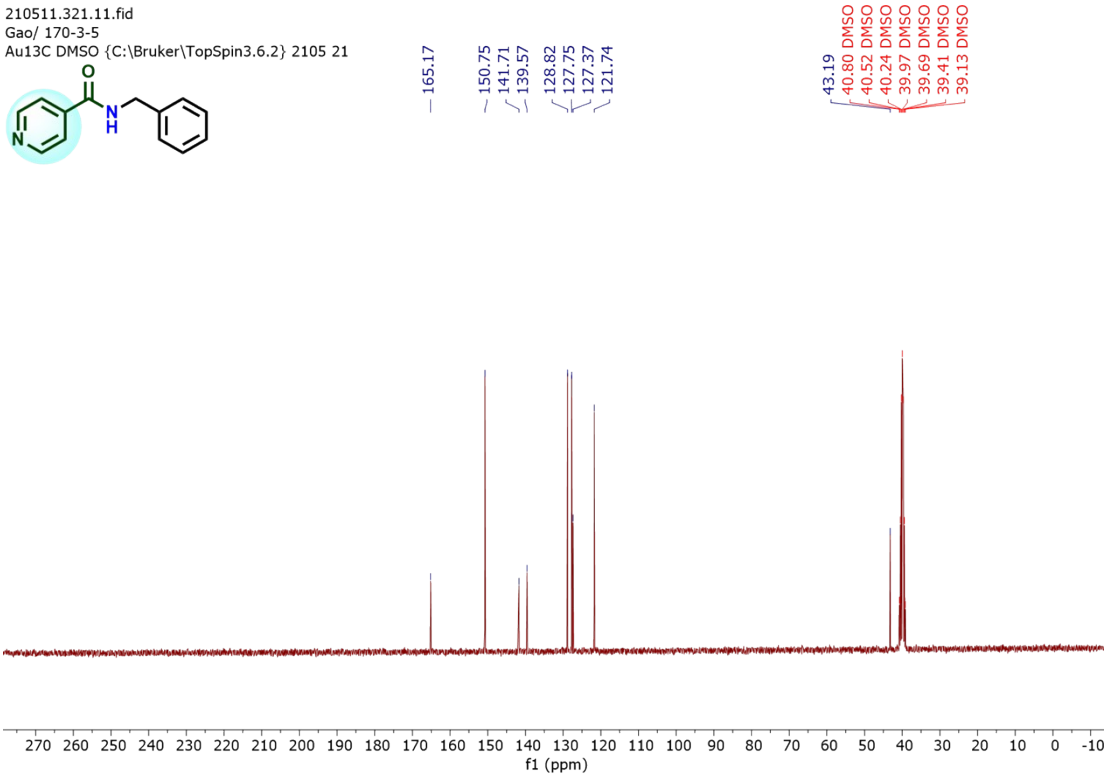
220308.f336.11.fid
Rui Ma 47-2-24b
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2203 36

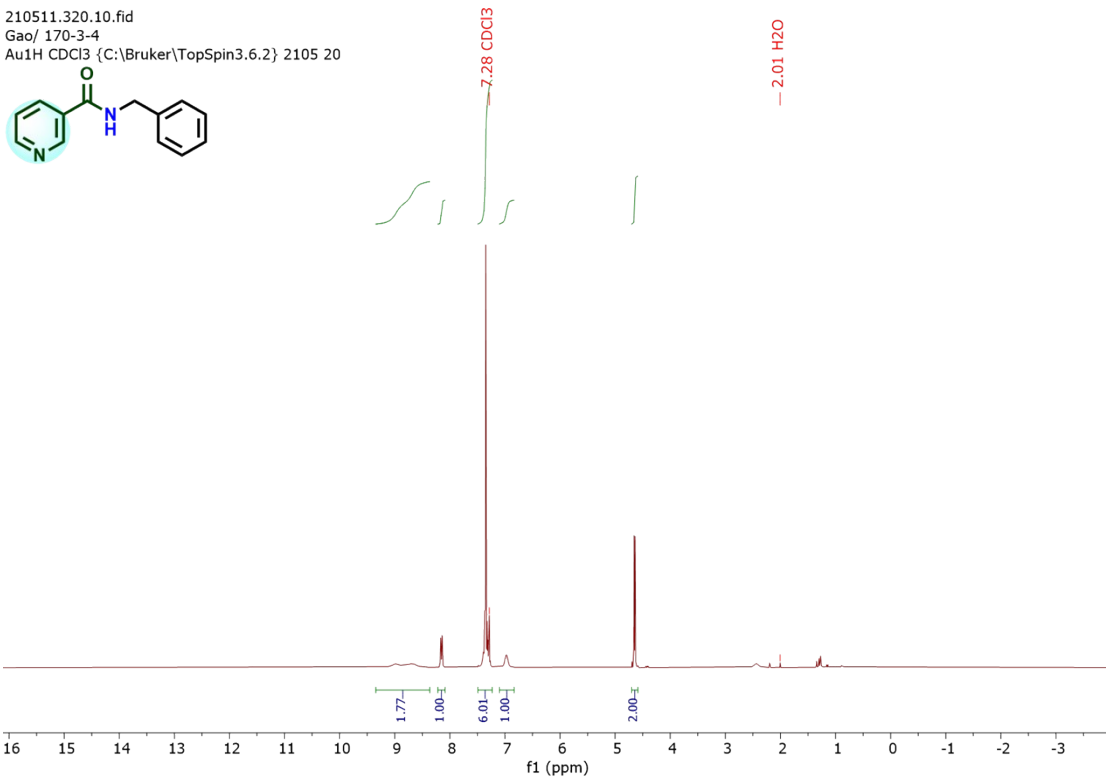


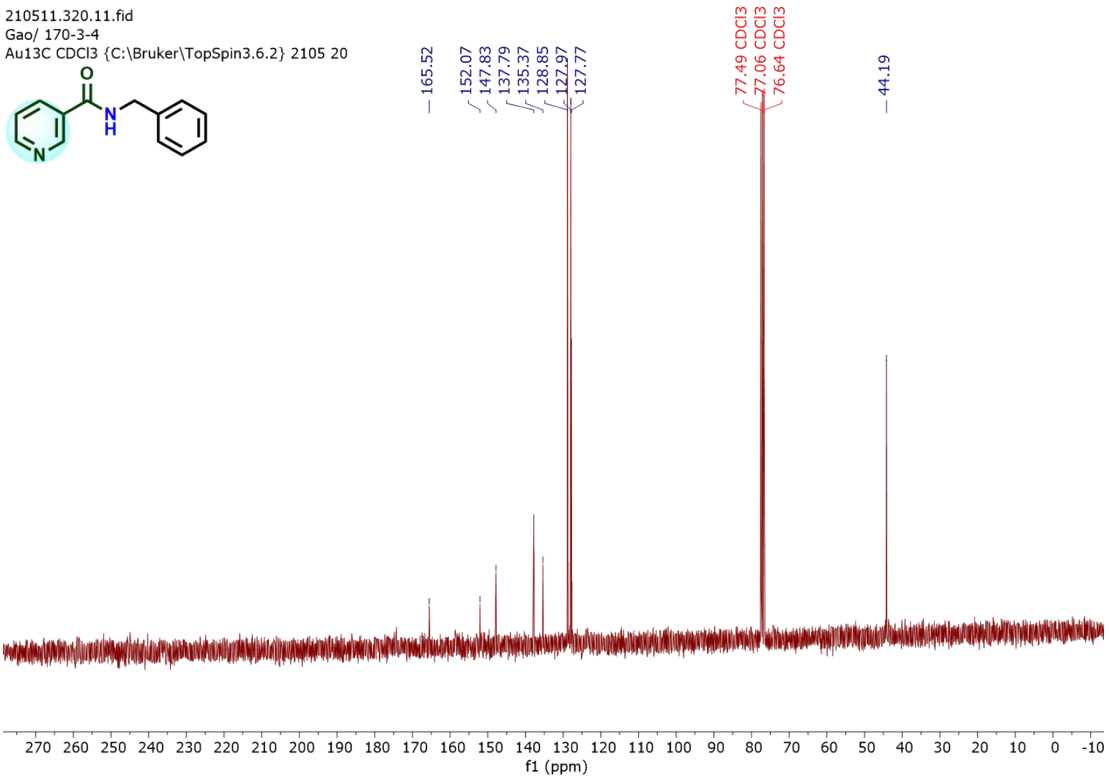
210511.321.10.fid
Gao/ 170-3-5
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 21



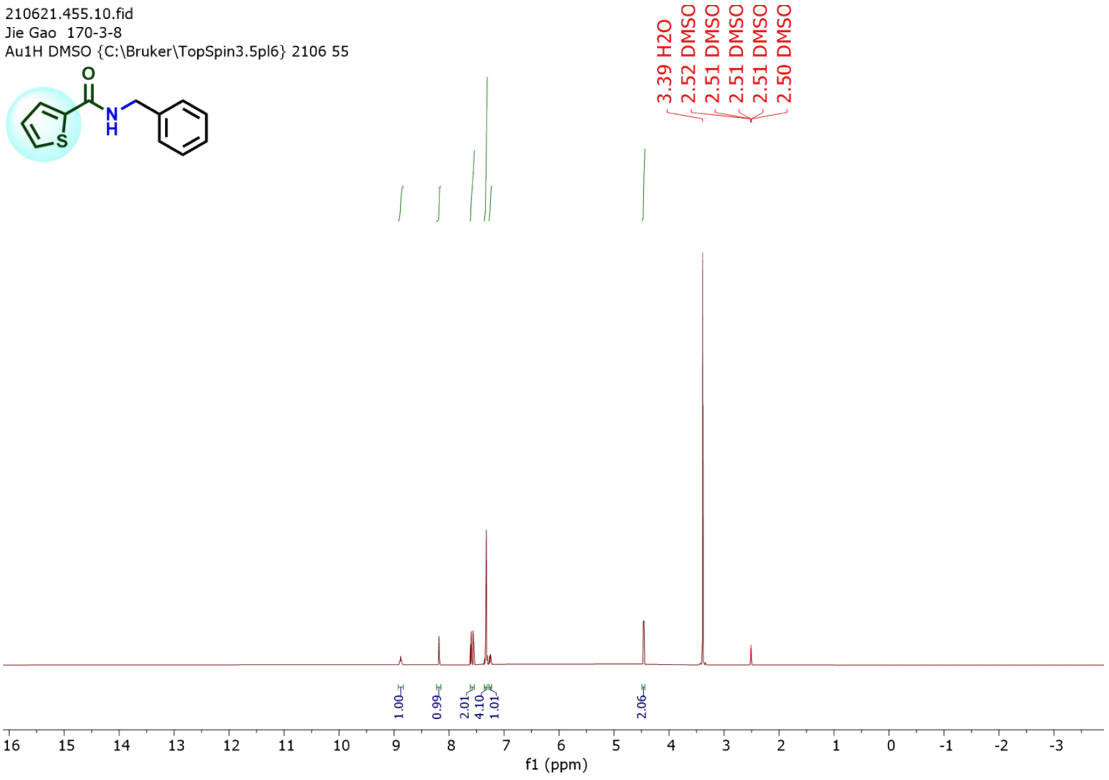
210511.321.11.fid
Gao/ 170-3-5
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 21



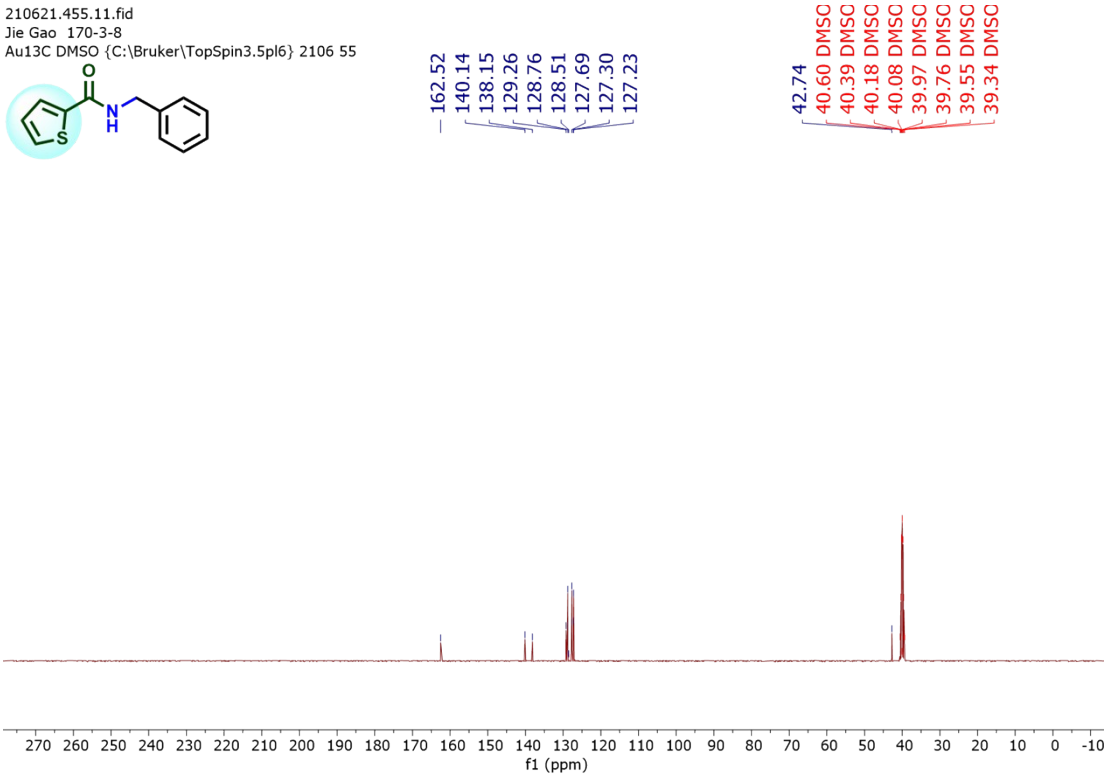

210511.320.10.fid
Gao/ 170-3-4
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 20
7.28 CDCl3
2.01 H2O
1.77
1.00
6.01
1.00
2.00
f1 (ppm)

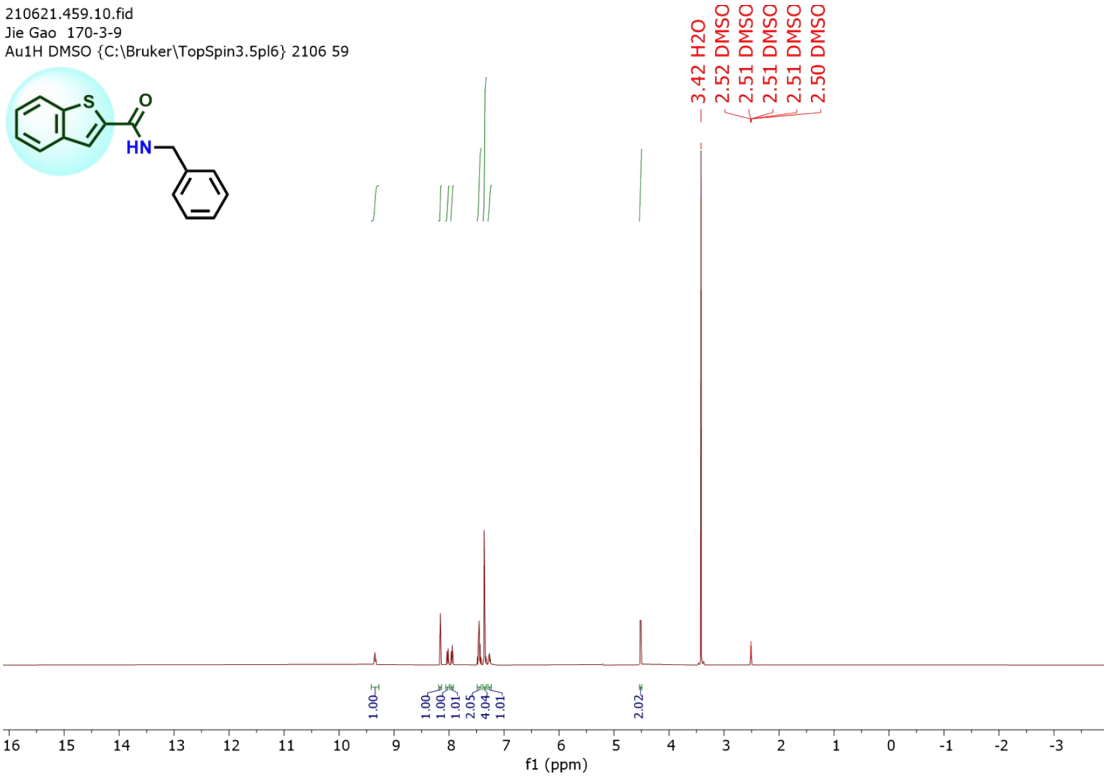

210511.320.11.fid
Gao/ 170-3-4
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2105 20
165.52
152.07
147.83
137.79
135.37
128.85
127.97
127.77
77.49 CDCl3
77.06 CDCl3
76.64 CDCl3
44.19
f1 (ppm)

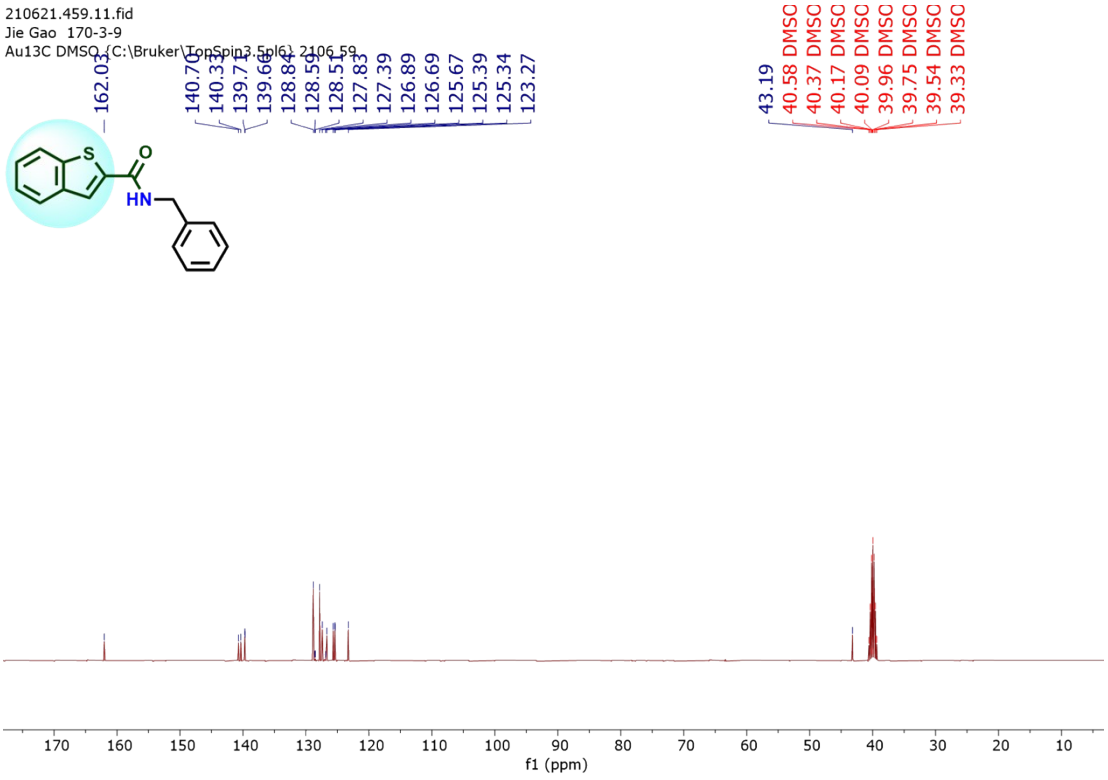
210621.455.10.fid
Jie Gao 170-3-8
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2106 55



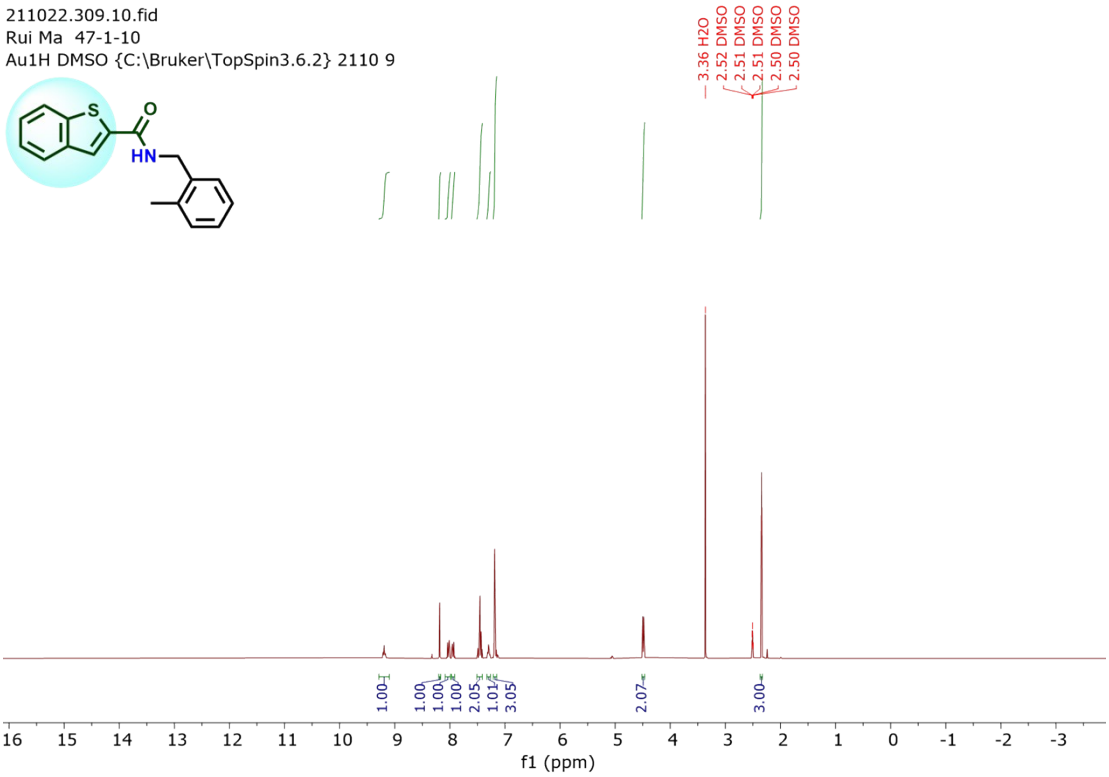
210621.455.11.fid
Jie Gao 170-3-8
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2106 55



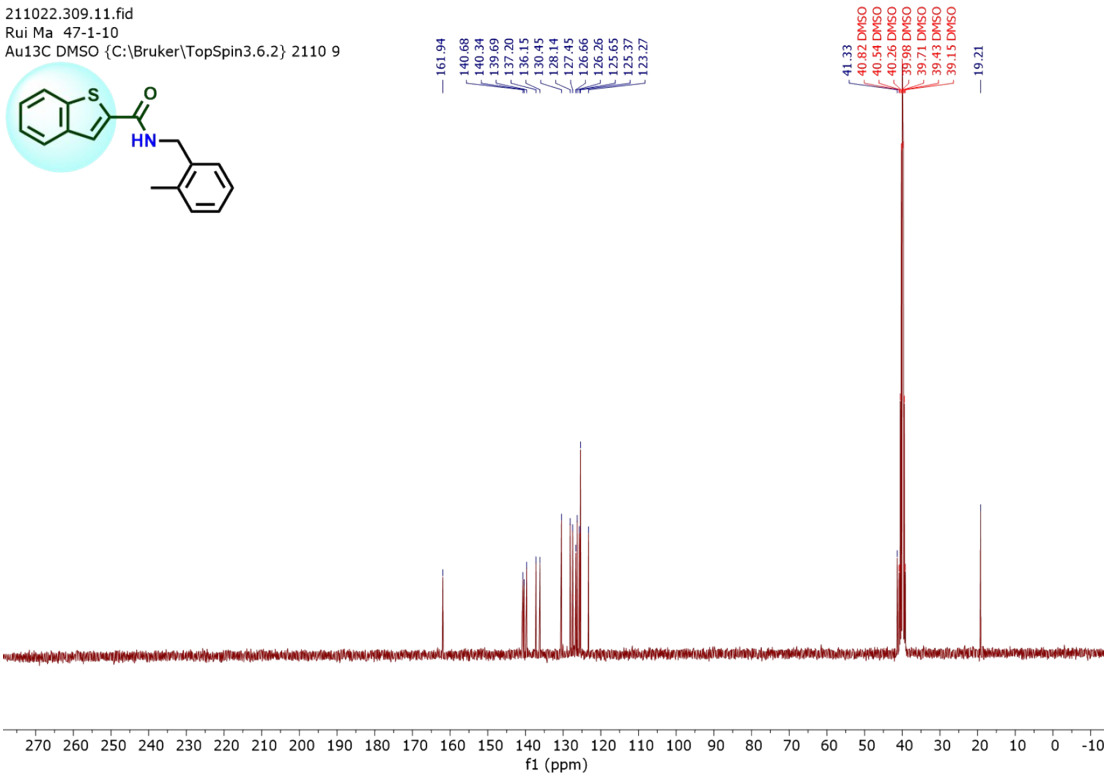

210621.459.10.fid
Jie Gao 170-3-9
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2106 59
3.42 H2O
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
1.00
1.00
1.00
1.01
2.05
4.04
1.01
2.02
f1 (ppm)


210621.459.11.fid
Jie Gao 170-3-9
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2106 59
162.03
140.70
140.33
139.71
139.66
128.84
128.59
128.51
127.83
127.39
126.89
126.69
125.67
125.39
125.34
123.27
43.19
40.58 DMSO
40.37 DMSO
40.17 DMSO
40.09 DMSO
39.96 DMSO
39.75 DMSO
39.54 DMSO
39.33 DMSO
f1 (ppm)

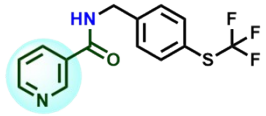
211022.309.10.fid
Rui Ma 47-1-10
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2110 9

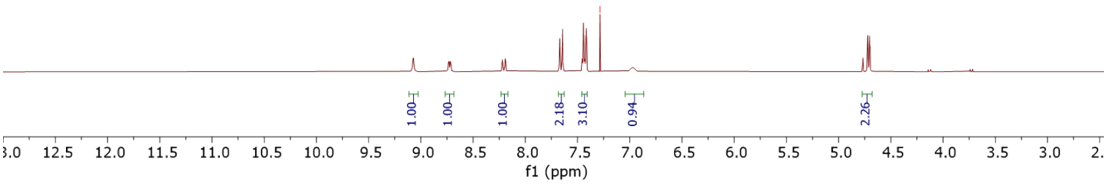


211022.309.11.fid
Rui Ma 47-1-10
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2110 9

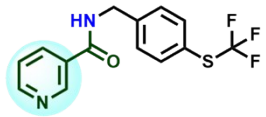


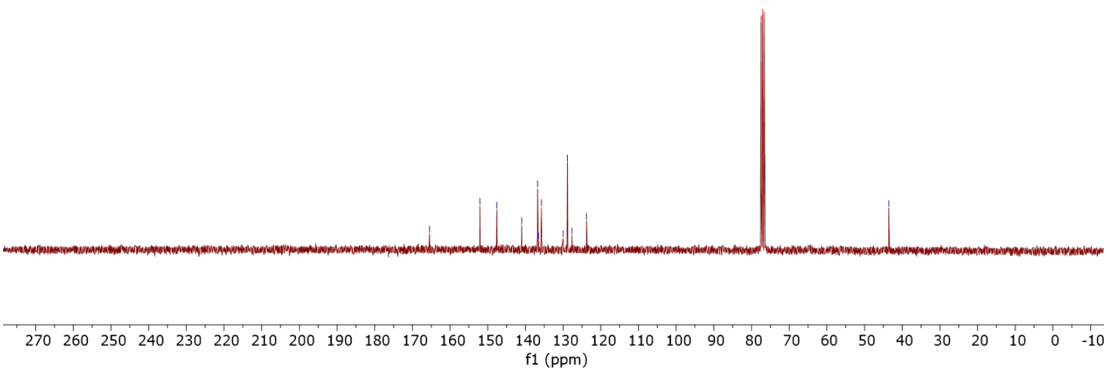
210629.323.10.fid
Jie Gao 170-3-26
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 23



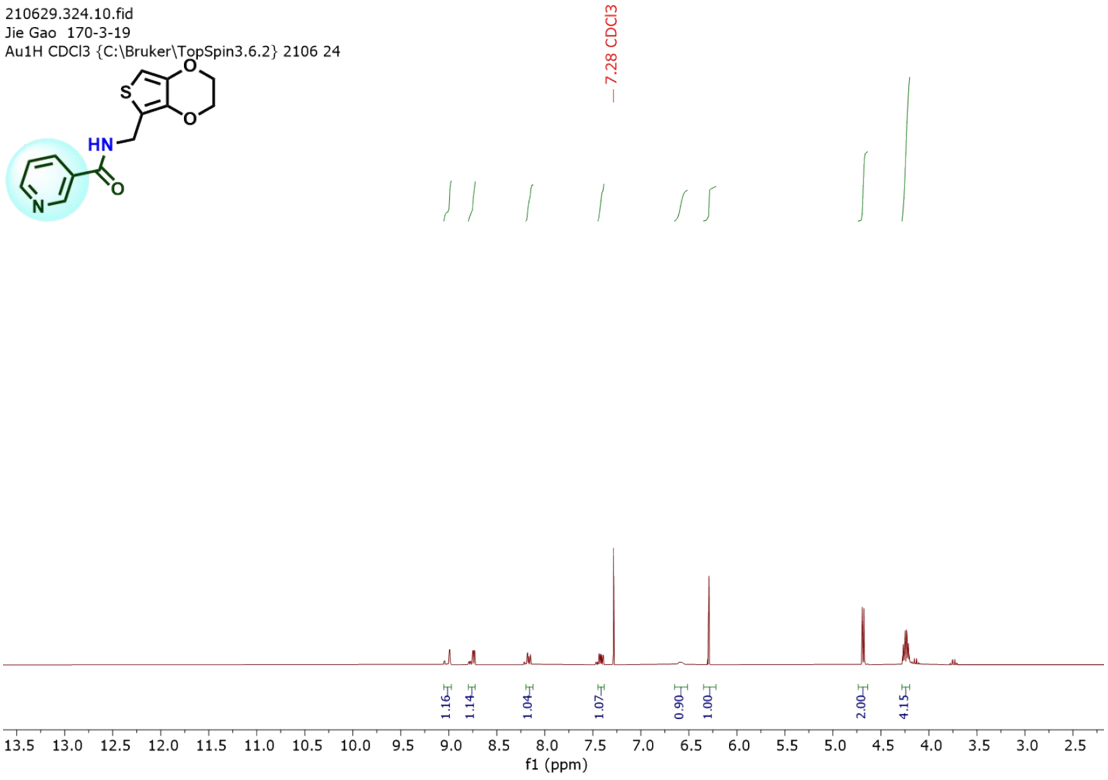


210629.323.11.fid
Jie Gao 170-3-26
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 23

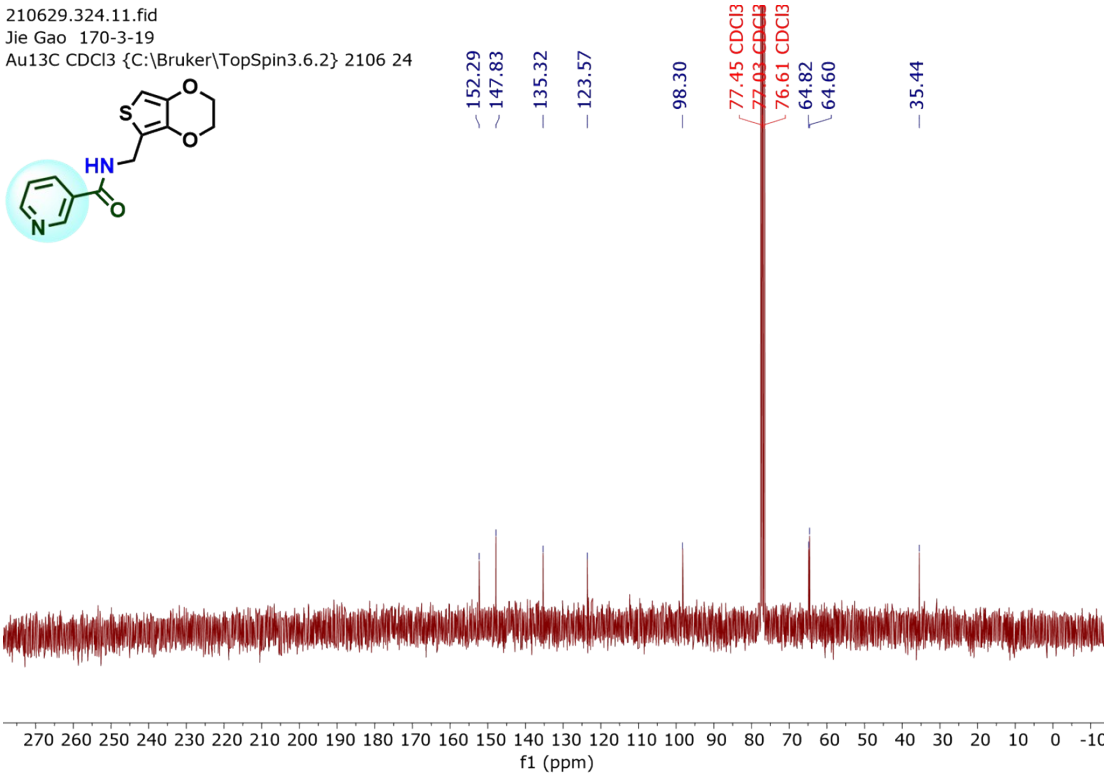


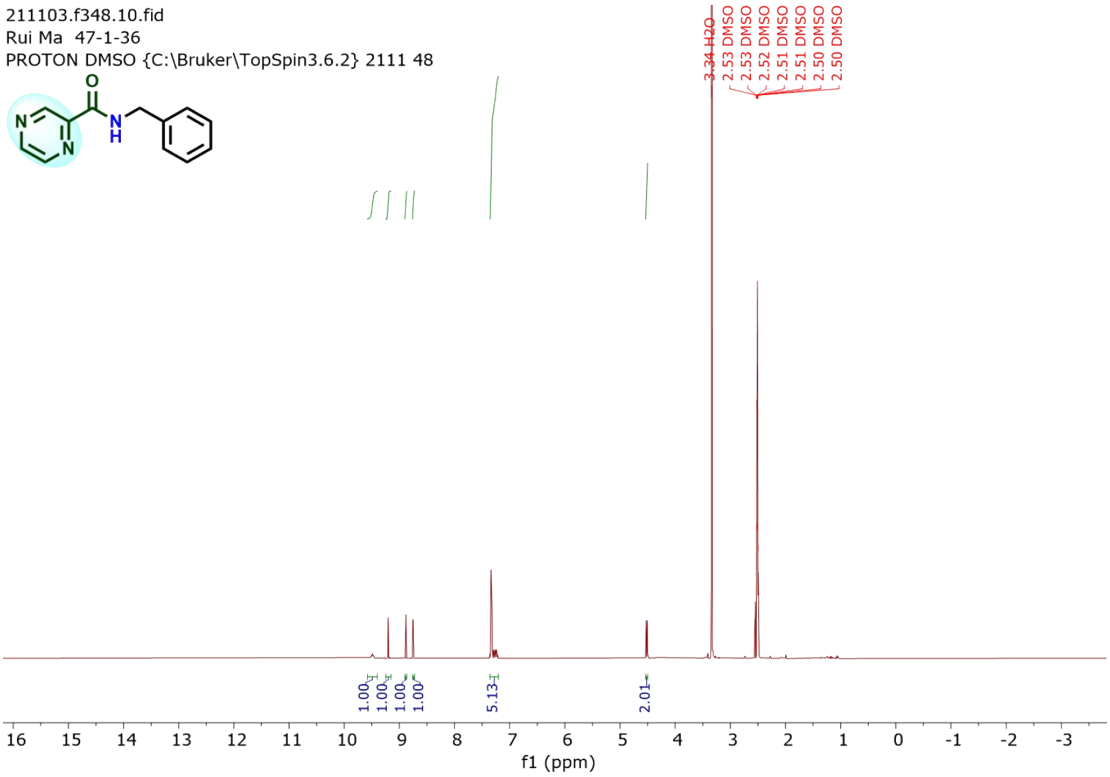


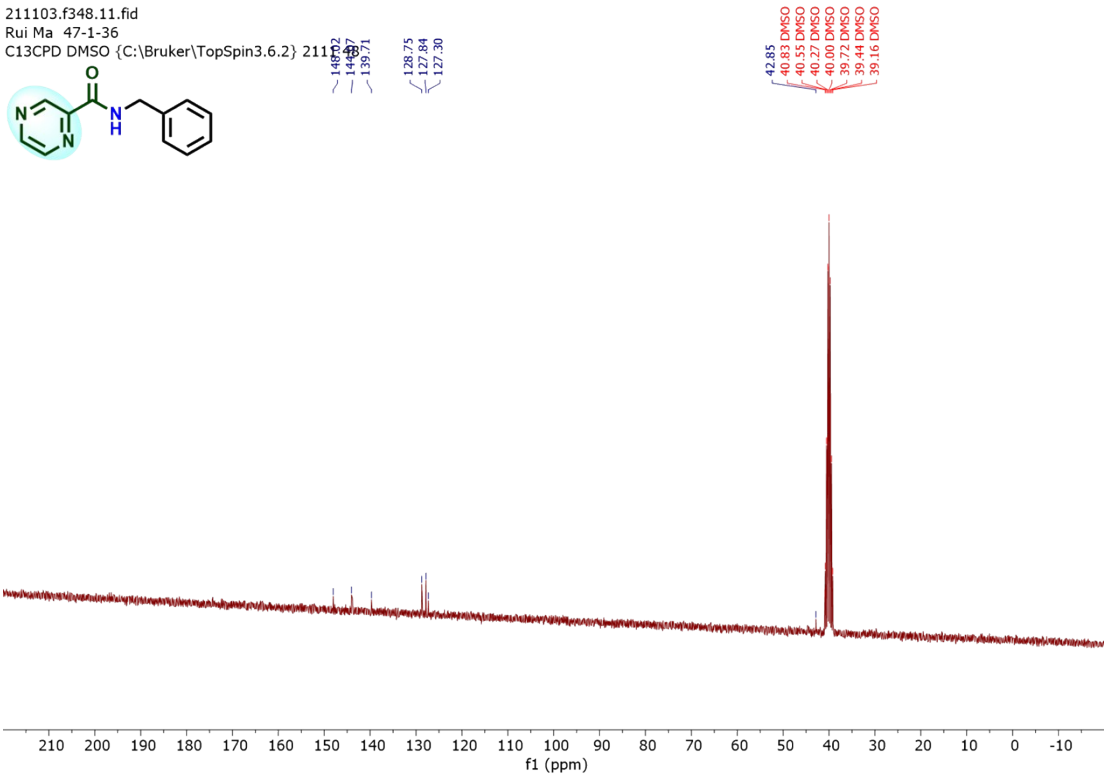
210629.324.10.fid
Jie Gao 170-3-19
Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 24

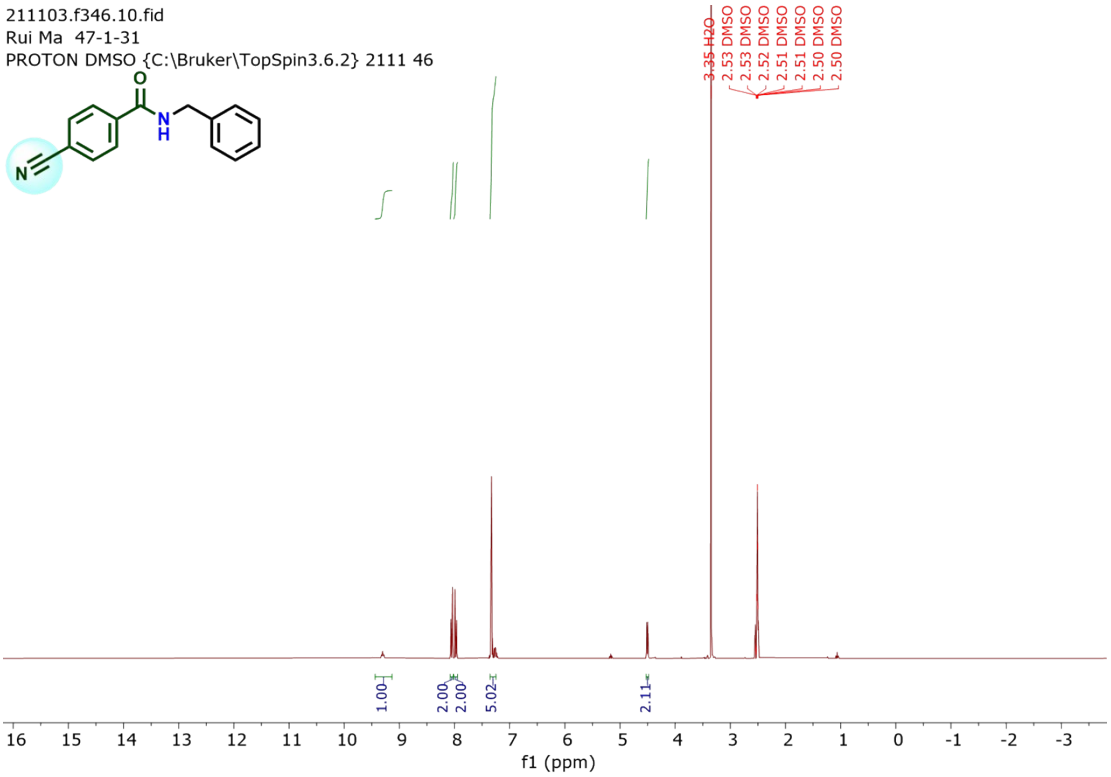


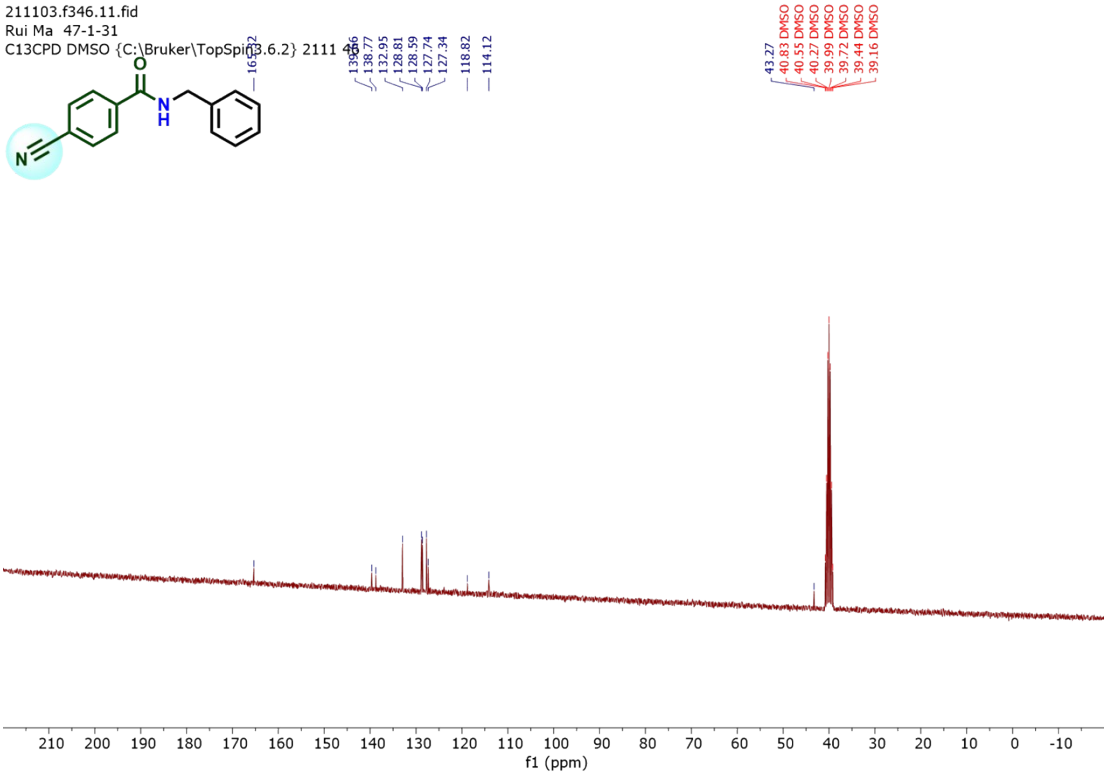
210629.324.11.fid
Jie Gao 170-3-19
Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 24



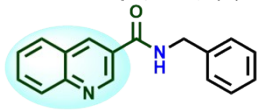

211103.f348.10.fid
Rui Ma 47-1-36
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2111 48
3.34 H2O
2.53 DMSO
2.53 DMSO
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
1.00
1.00
1.00
1.00
5.13
2.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

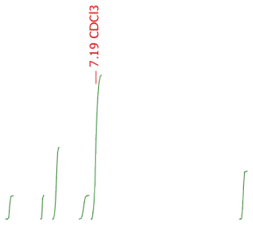

211103.f348.11.fid
Rui Ma 47-1-36
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2111 48
148.02
144.87
139.71
128.75
127.84
127.30
42.85
40.83 DMSO
40.55 DMSO
40.27 DMSO
40.00 DMSO
39.72 DMSO
39.44 DMSO
39.16 DMSO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

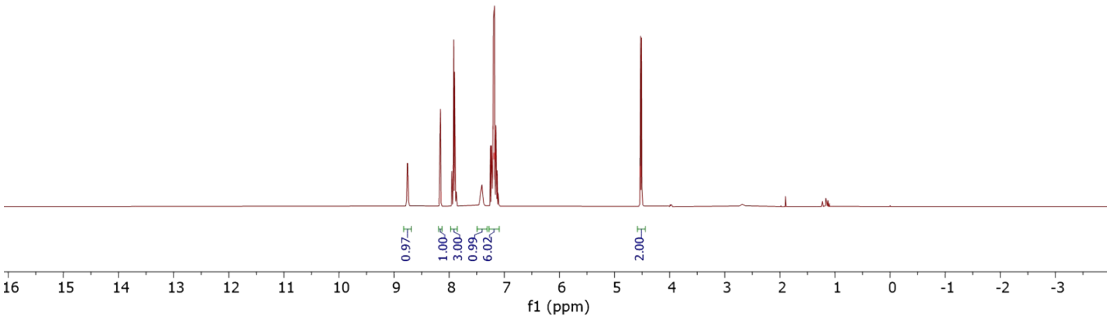

211103.f346.10.fid
Rui Ma 47-1-31
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2111 46
3.35 H2O
2.53 DMSO
2.53 DMSO
2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
1.00
2.00
2.00
5.02
2.11
f1 (ppm)


211103.f346.11.fid
Rui Ma 47-1-31
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2111 46
165.32
139.66
138.77
132.95
128.81
128.59
127.74
127.34
118.82
114.12
43.27
40.83 DMSO
40.55 DMSO
40.27 DMSO
39.99 DMSO
39.72 DMSO
39.44 DMSO
39.16 DMSO
f1 (ppm)

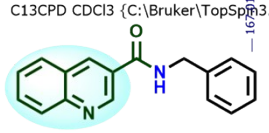
220624.f333.10.fid
Ma/ 47-1-314
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 33

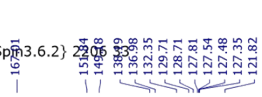


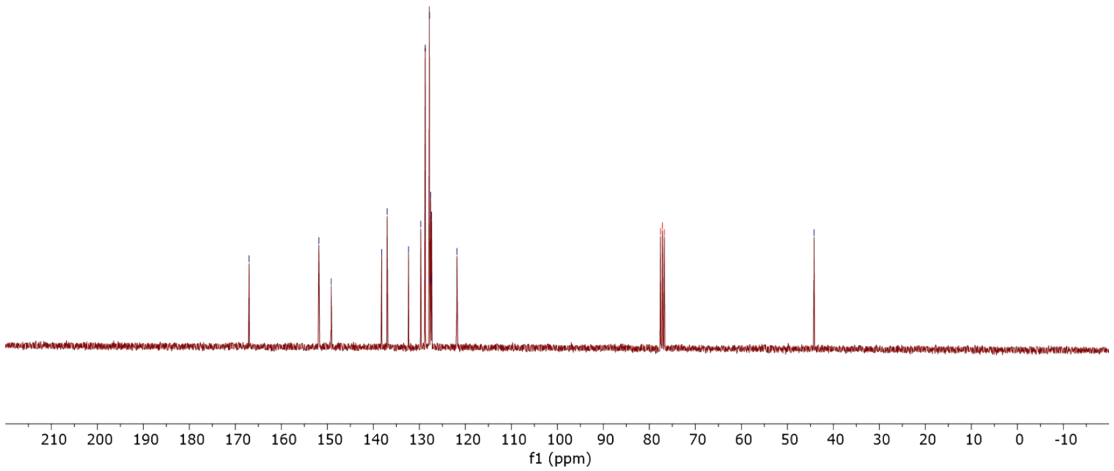




220624.f333.11.fid
Ma/ 47-1-314
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2206







8. References

[1] B. Sardar, R. Jamatia, A. Samanta, D. Srimani, *J Org Chem* **2022**, *87*, 5556-5567.
[2] J. Das, D. Banerjee, *J Org Chem* **2018**, *83*, 3378-3384.
[3] M. Trudell, T. Apsunde, *Synthesis* **2013**, *46*, 230-234.
[4] S. S. Kulkarni, X. Hu, R. Manetsch, *Chem Commun* **2013**, *49*, 1193-1195.
[5] J. Egly, W. Chen, A. Maisse-François, S. Bellemin-Laponnaz, T. Achard, *Eur. J. Ino. Che.,* **2022**, e202101033.
[6] N. Wang, X. Zou, J. Ma, F. Li, *Chem Commun* **2014**, *50*, 8303-8305.
[7] H. Cheng, M. Q. Xiong, C. X. Cheng, H. J. Wang, Q. Lu, H. F. Liu, F. B. Yao, C. Chen, F. Verpoort, *Chem Asian J* **2018**, *13*, 440-448.
[8] S. Kerdphon, X. Quan, V. S. Parihar, P. G. Andersson, *, J. Org. Chem* **2015**, *80*, 11529-11537.
[9] S. L. Zultanski, J. Zhao, S. S. Stahl, *J Am Chem Soc* **2016**, *138*, 6416-6419.
[10] J. A. Forni, N. Micic, T. U. Connell, G. Weragoda, A. Polyzos, *Angew Chem Int Edl* **2020**, *59*, 18646-18654.
[11] H. Lundberg, F. Tinnis, H. Adolfsson, *Chemistry* **2012**, *18*, 3822-3826.
[12] S. Thurow, E. J. Lenardao, X. Just-Baringo, D. J. Procter, *Org Lett* **2017**, *19*, 50-53.
[13] S. Azeez, P. Sureshbabu, S. Sabiah, J. Kandasamy, *Org Biomol Chem* **2022**, *20*, 2048-2053.
[14] P. V. Ramachandran, H. J. Hamann, *Org Lett* **2021**, *23*, 2938-2942.
[15] P. Xu, F.-S. Han, Y.-H. Wang, *Adv. Synth. Catal.,* **2015**, *357*, 3441-3446.
[16] Y. Liang, Z. Zhao, A. Taya, N. Shibata, *Org Lett* **2021**, *23*, 847-852.
[17] A. Wang, Y. Xie, J. Wang, D. Shi, H. Yu, *Chem Commun.,* **2022**, *58*, 1127-1130.
[18] V. Zubar, A. Dewanji, M. Rueping, *Org Lett* **2021**, *23*, 2742-2747.
[19] J.-W. Wu, Y.-D. Wu, J.-J. Dai, H.-J. Xu, *Adv. Synth. Catal.,* **2014**, *356*, 2429-2436.
[20] A. M. Martinez, N. Rodriguez, R. Gomez Arrayas, J. C. Carretero, *Chem Commun.,* **2014**, *50*, 6105-6107.
[21] N. Caldwell, C. Jamieson, I. Simpson, A. J. Watson, *Chem Commun.,* **2015**, *51*, 9495-9498.
[22] N. F. Nikitas, M. K. Apostolopoulou, E. Skolia, A. Tsoukaki, C. G. Kokotos, *Chemistry* **2021**, *27*, 7915-7922.
[23] W. J. Ang, L.-C. Lo, Y. Lam, *Tetrahedron* **2014**, *70*, 8545-8558.