

## Supporting information

# Stable and Reusable Ni-based Nanoparticles for General and Selective Hydrogenation of Nitriles to Amines

Zhuang Ma,<sup>a</sup> Vishwas G. Chandrashekhar,<sup>a</sup> Bei Zhou,<sup>a</sup> Asma M. Alenad,<sup>b</sup> Nils Rockstroh,<sup>a</sup> Stephan Bartling,<sup>a</sup> Matthias Beller\*,<sup>a</sup> and Rajenahally V. Jagadeesh\*<sup>a</sup>

<sup>a</sup> Leibniz-Institut für Katalyse e.V., Albert-Einstein-Str. 29a, Rostock, D-18059, Germany

<sup>b</sup> Chemistry Department, College of Science, Jouf University, P.O. Box: 2014, Sakaka, Saudi Arabia

\*E-Mails: matthias.beller@catalysis.de; jagadeesh.rajenahally@catalysis.de

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## **S1. Materials and methods**

All nitriles were obtained commercially from various chemical companies. Unless otherwise stated all reagents were used directly without purification. Nickel (II) nitrate hexahydrate (cat no. 139267-100G), cobalt (II) nitrate hexahydrate (cat no. 239267-100G), copper (II) nitrate trihydrate (cat no. 61194-100G), iron (III) nitrate nonahydrate (cat no. 216828-100G) and manganese (II) nitrate tetrahydrate (cat no. 63547-100G) were purchased from Sigma Aldrich. Silica suspension (Silica LUDOX® AS-40 colloidal silica, cat no. 420840-1L) was purchased from Sigma Aldrich.

Preparation of catalytic materials was performed in a tube furnace (Lenton Thermal Design Ltd). All catalytic reactions were carried out in 300 mL autoclave (PARR Instrument Company). To avoid unspecific reactions, all catalytic reactions were carried out either in glass vials, which were placed inside the autoclave, or glass/Teflon vessel fitted autoclaves.

GC and GC-MS were recorded on Agilent 6890N instrument. GC conversions and yields were determined by GC-FID, HP6890 chromatograph with FID detector, column HP530 m x 250 mm x 0.25  $\mu\text{m}$ .

NMR spectra were recorded on Bruker 300 Fourier, Bruker AV 300 and Bruker AV 400 spectrometers. Chemical shifts are reported in ppm relative to the deuterated solvent. Coupling constants are expressed in Hertz (Hz). The following abbreviations are used: s = singlet, d = doublet, t = triplet and m = multiple. The residual solvent signals were used as references for  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra ( $\text{CDCl}_3$ :  $\delta\text{H} = 7.26$  ppm,  $\delta\text{C} = 77.12$  ppm;  $\text{DMSO-d}_6$ :  $\delta\text{H} = 2.50$  ppm,  $\delta\text{C} = 39.52$  ppm).

High resolution mass spectra (HRMS) were obtained either from a MAT 95 XP from Thermo (EI) or from an HPLC system 1200 and downstream ESI-TOF-MS 6210 from Agilent (ESI).

XRD powder patterns were recorded on a Panalytical X'Pert diffractometer equipped with a Xcelerator detector or on a Panalytical Empyrean diffractometer equipped with a PIXcel 3D detector system, both used with automatic divergence slits and  $\text{Cu K}\alpha$  radiation (40 kV, 40 mA).  $\text{Cu } \beta$ -radiation was excluded by using nickel filter foil. Peak positions and profile were fitted with Pseudo-Voigt function using the Panalytical HighScore Plus software package. Phase identification was done by using the PDF-2 database of the International Center of Diffraction Data (ICDD)

Scanning transmission electron microscopy (STEM) measurements were performed with an aberration-corrected JEM-ARM200F (JEOL, Corrector: CEOS) at 200 kV. The microscope was equipped with an Enfinium ER (GATAN) electron energy-loss spectrometer for chemical analysis. High-Angle Annular Dark Field (HAADF) and Annular Bright Field (ABF) detectors were used for general imaging, the Annular Dark Field (ADF) detector was used for position control for EELS acquisition. EELS was done at a camera length of 4 cm, an illumination semi angle of 27.8 mrad and a filter entrance aperture semi angle of 41.3 mrad. The solid samples were deposited without any

pretreatment on a holey carbon supported Cu-grid (mesh 300) and transferred to the microscope.

The X-ray Photoelectron Spectroscopy (XPS) measurements were performed on an ESCALAB 220iXL (Thermo Fisher Scientific) with monochromated Al K $\alpha$  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.

## **S2. Procedure for the preparation of catalysts**

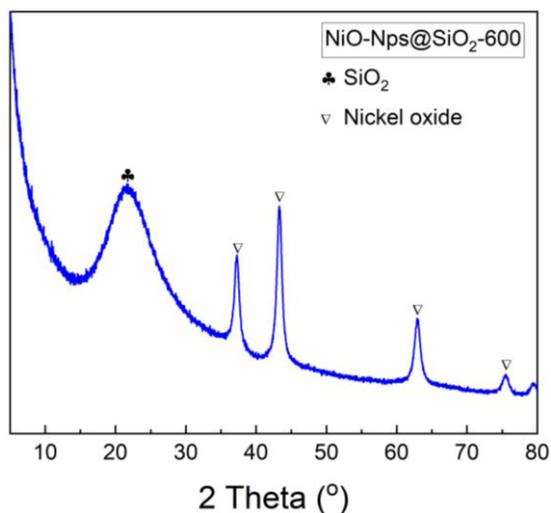
In a 250 mL dried round bottom flask, 280 mg Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O was dissolved in 50 mL distilled water and then 1.25 g silica suspension (Silica LUDOX® AS-40 colloidal silica) was added. The round bottom flask containing reaction mixture was stirred at 65 °C for 5 h. Then, the water was removed by rotary evaporator and the obtained solid was dried overnight in an oven at 110 °C. The dried solid material was grinded to a fine powder and calcined in a tube furnace at 600 °C for 6 h under air atmosphere. Then, the calcined material was reduced under 10% H<sub>2</sub>/N<sub>2</sub> flow at different temperature (300 °C, 400 °C, 500 °C, 600 °C and 700 °C) for 5 h. The resulting materials are represented as M-NPs@SiO<sub>2</sub>-T, where M and T denotes metal and reduction temperature.

Elemental analysis of the optimal catalyst, Ni-NPs@SiO<sub>2</sub>-500 (wt%: Si=33.4%, C=43.5%, H=9.7%, Ni=8.9% by ICP-OES).

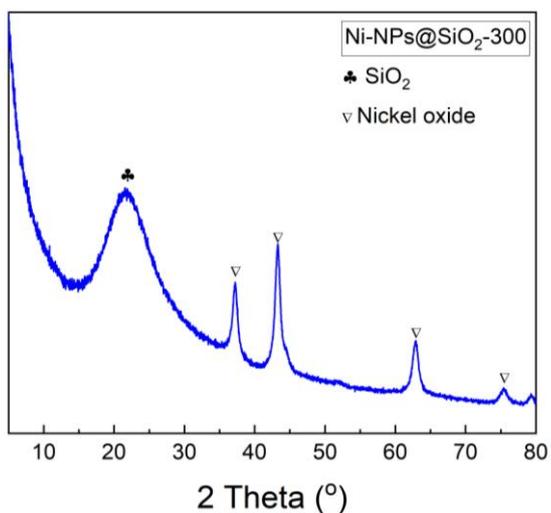
Same procedure has been applied for the preparation of other catalytic materials such as, Fe-NPs@SiO<sub>2</sub>-500, Mn-NPs@SiO<sub>2</sub>-500, Co-NPs@SiO<sub>2</sub>-500 and Cu-NPs@SiO<sub>2</sub>-500 using Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, Mn(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, and Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O, respectively.

### S3. Characterization of catalysts

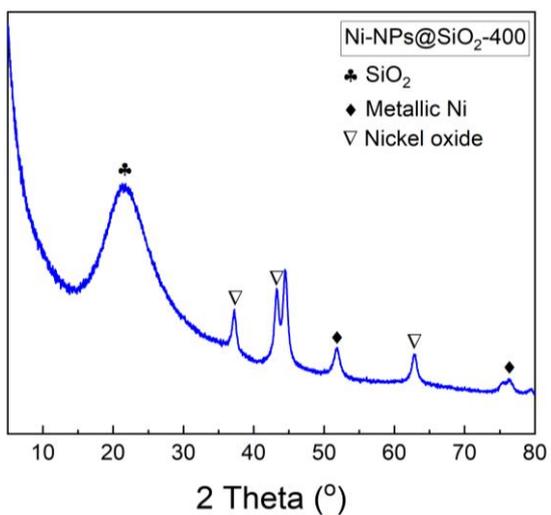
#### XRD



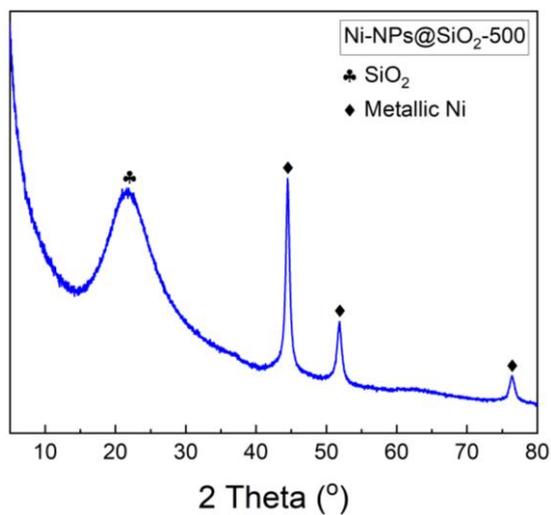
**Figure S1.** XRD patterns of NiO@SiO<sub>2</sub> calcined at 600 °C without reduction catalyst.



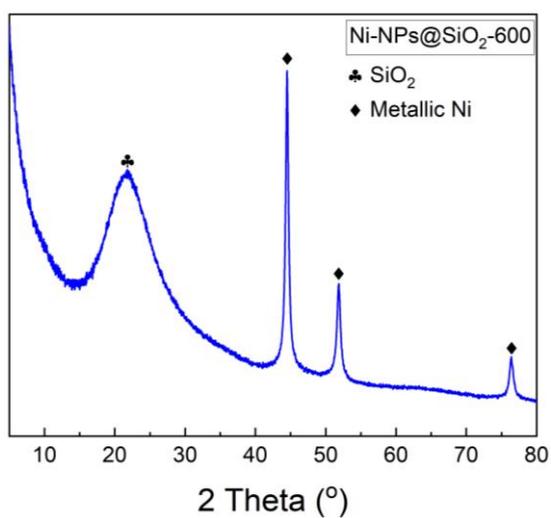
**Figure S2.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-300 catalyst.



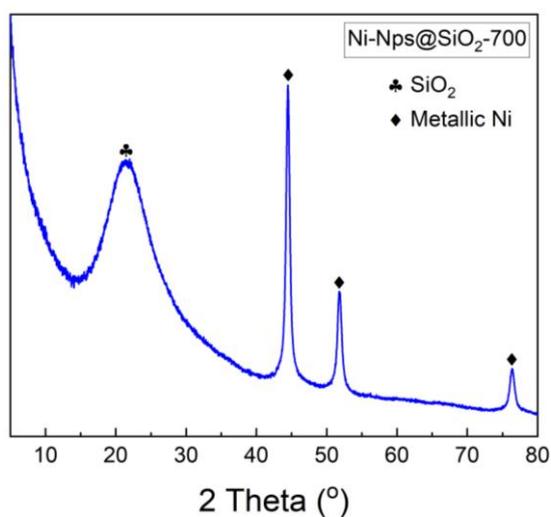
**Figure S3.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-400 catalyst.



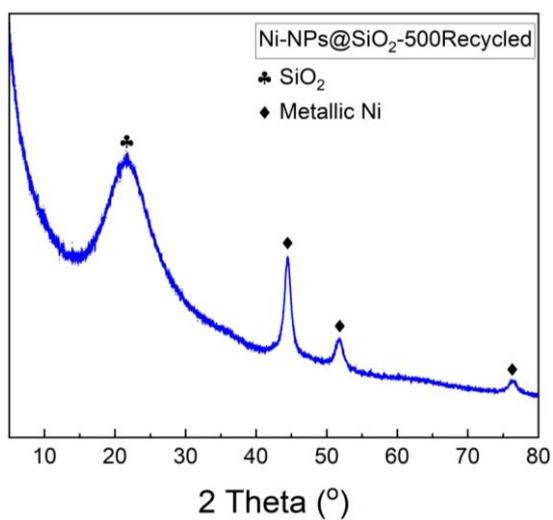
**Figure S4.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-500 catalyst.



**Figure S5.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-600 catalyst.

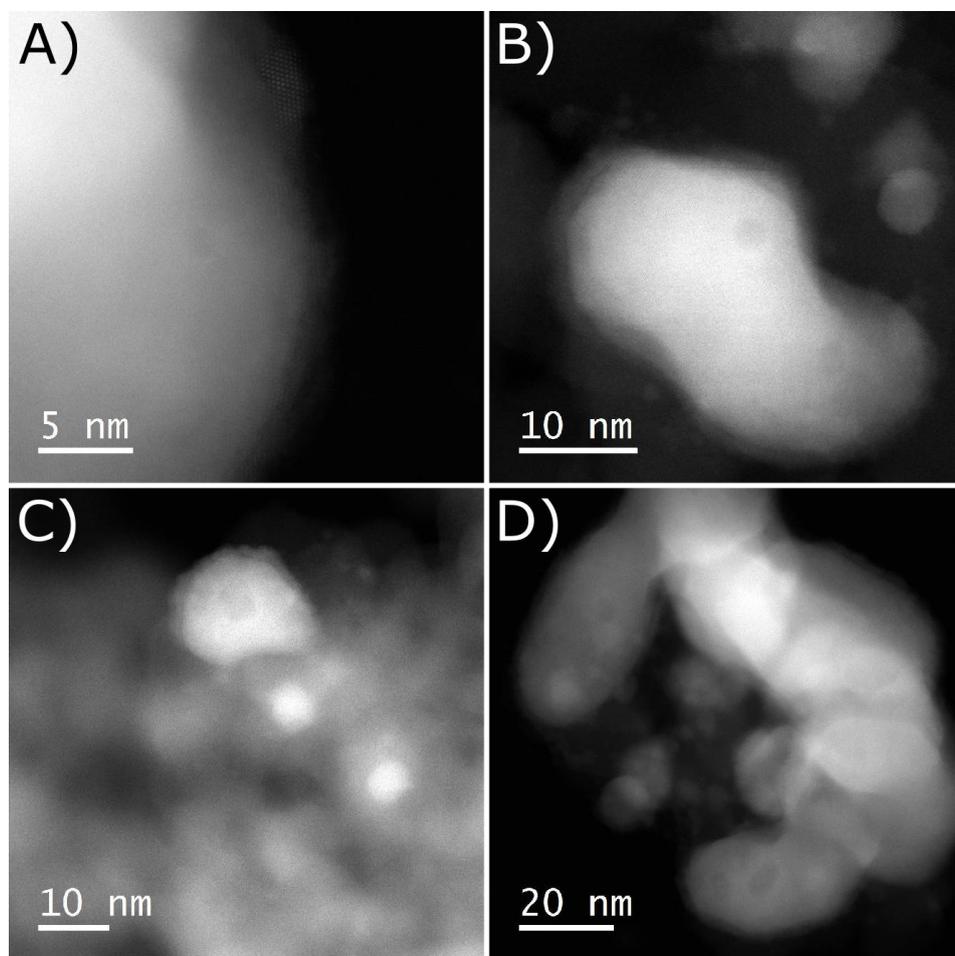


**Figure S6.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-700 catalyst.

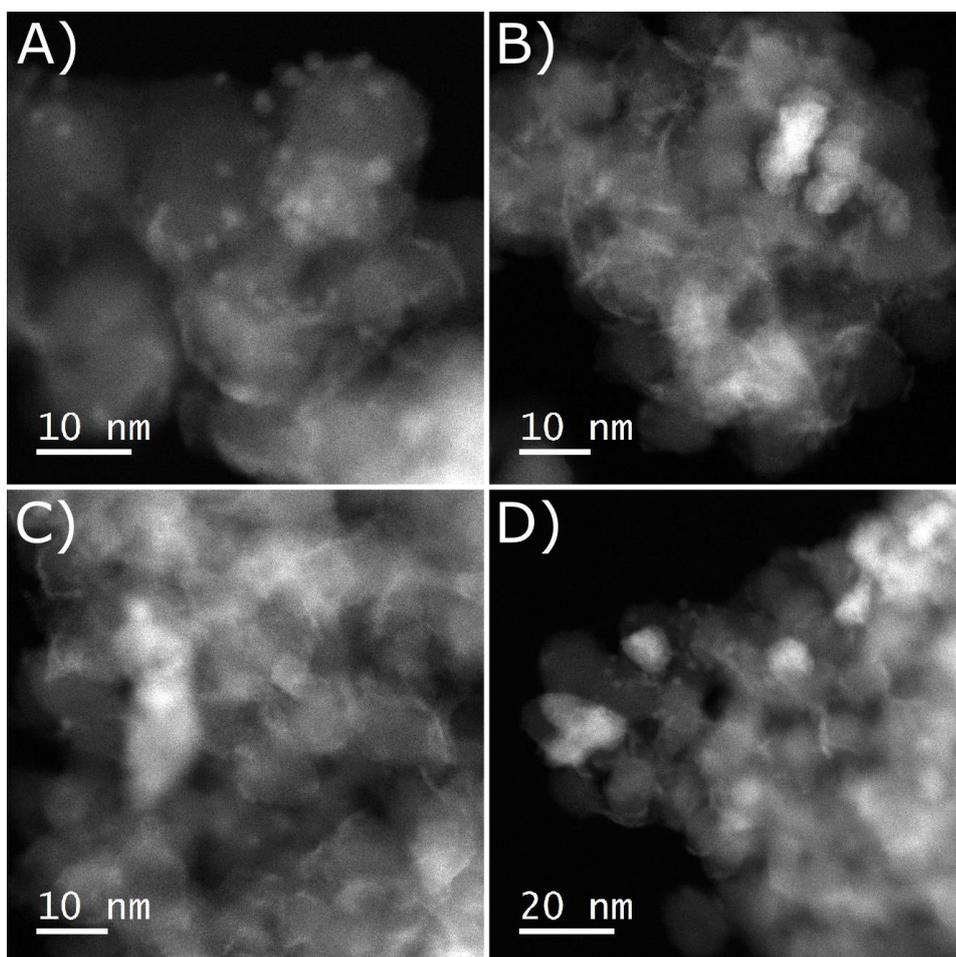


**Figure S7.** XRD patterns of Ni-NPs@SiO<sub>2</sub>-500 recycled catalyst (after one run).

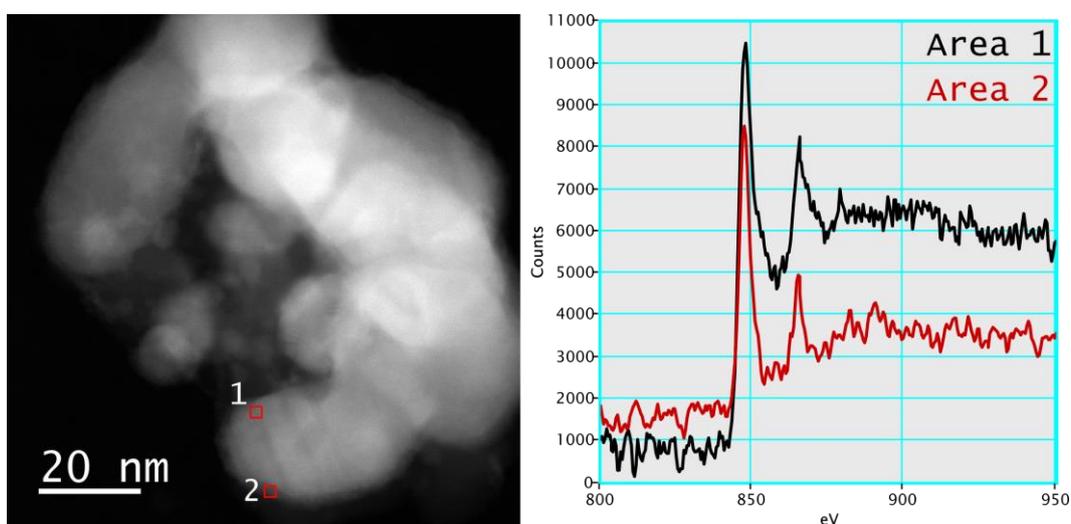
STEM



**Figure S8.** Selected STEM-HAADF images of Ni@SiO<sub>2</sub>-500 showing the (partial) presence of an oxidic layer/shell around a metallic Ni core.



**Figure S9.** Selected STEM-HAADF images of Ni-NPs@SiO<sub>2</sub>-500R (after one run) showing the presence of Ni (oxide) at the interfaces of the support particles.



**Figure S10.** STEM-ADF image of Ni-NPs@SiO<sub>2</sub>-500 (left) and corresponding Ni L edge electron-energy loss (EEL) spectra of the highlighted areas (right). In area 1, the L3/L2 ratio implies the presence of metallic Ni, while the edge shape of spectrum 2 points towards the occurrence of NiO<sup>S1</sup>. The EEL spectra are background subtracted and deconvoluted.

#### S4. General procedure for the hydrogenation of nitriles to primary amines

The magnetic stirring bar and 0.5 mmol of corresponding nitrile were transferred to 8 mL glass vial and then 2 mL MeOH were added. Next, 15-30 mg of catalyst (Ni-NPs@SiO<sub>2</sub>-500; 4.5-9.0 mol% Ni) was added and the vial was fitted with septum, cap, and needle. The reaction vials (8 vials with different substrates at a time) were placed into a 300 mL autoclave. The autoclave was flushed with hydrogen twice at 20 bar pressure and then it was pressurized with 5-7 bar ammonia gas and 35 bar hydrogen. The autoclave was placed into an aluminium block preheated at 60 and 80 °C and the reactions were stirred for required time. After the completion of the reactions, the autoclave was cooled to room temperature. The remaining ammonia and hydrogen were discharged and the vials containing reaction products were removed from the autoclave. The solid catalyst was filtered off and washed thoroughly with ethyl acetate. The reaction products were analyzed by GC-MS. The corresponding primary amines were purified by column chromatography. Then, the resulted free amines were converted to their respective hydrochloride salt and characterized by NMR and HR-MS analysis. For converting into hydrochloride salt of amine, 1-2 mL methanolic HCl (1.5M HCl in methanol) was added to the ether solution of respective amine and stirred at room temperature for 4-5 h. Then, solvent was removed by rotary evaporation, and the resulted hydrochloride salt of amine is dried under high vacuum. The yields were determined by GC for the selected amines: After completion of the reaction, n-hexadecane (50 µL) as standard was added to the reaction vials and the reaction products were diluted with ethyl acetate followed by filtration using plug of silica and then analyzed by GC.

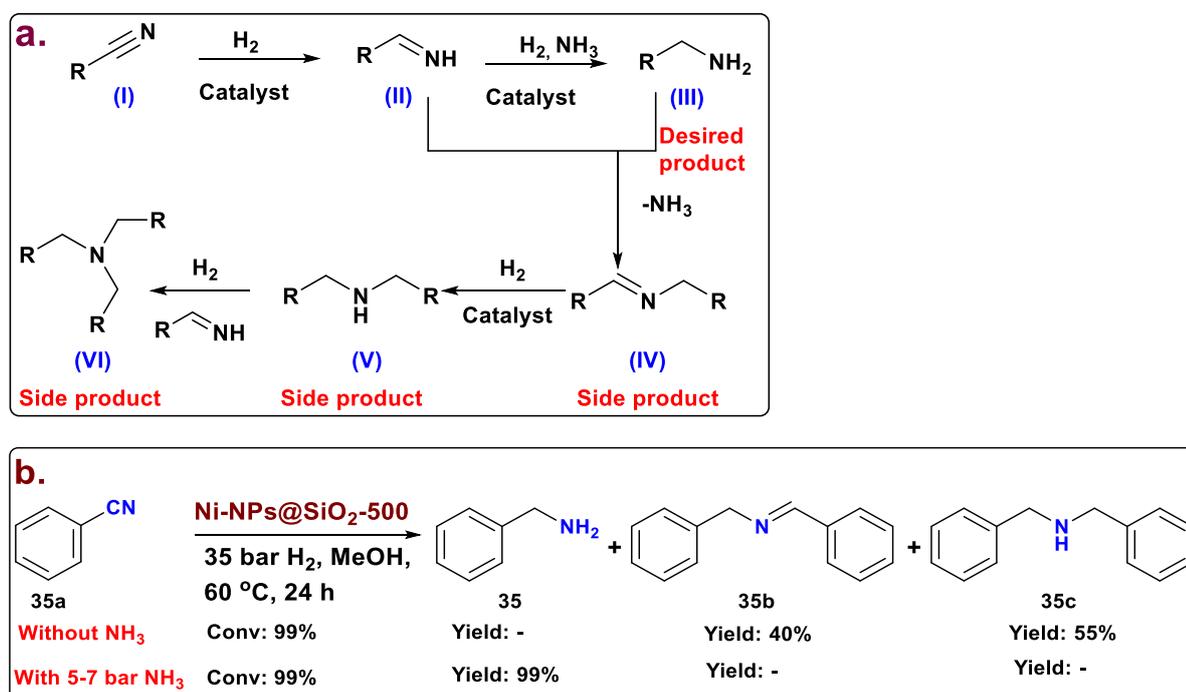
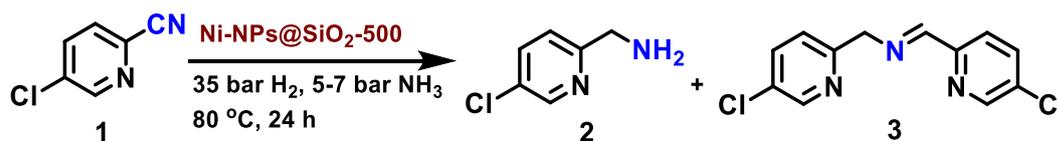


Figure S11. a: Reaction pathway for the catalytic hydrogenation of nitriles. b: control experiments.

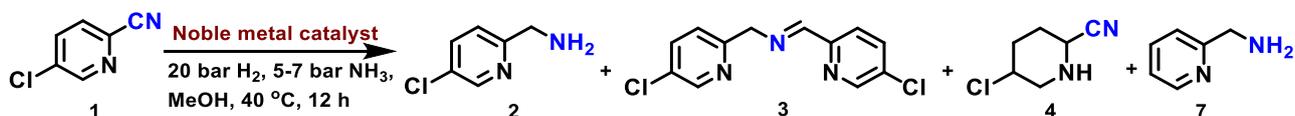
**Table S1.** Hydrogenation of 5-chloropicolinonitrile to (5-chloropyridin-2-yl)methanamine: Testing of different solvent<sup>a</sup>.



Entry	Solvent	Conv (%)	Yield (%) of	
			2	3
1	MeOH	>99	88	11
2	EtOH	90	79	10
3	t-BuOH	76	43	33
4	i-PrOH	91	80	11
5	Toluene	40	21	19
6	THF	54	29	25
7	MeCN	-	-	-
8	H <sub>2</sub> O	-	-	-
9 <sup>b</sup>	MeOH	>99	85	13

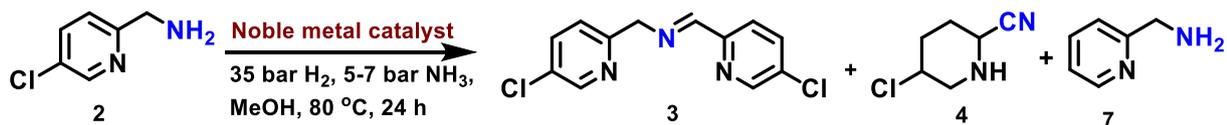
**Reaction conditions<sup>a</sup>:** 0.5 mmol 5-chloropicolinonitrile, 30 mg Ni-NPs@SiO<sub>2</sub>-500 (9.0 mol% Ni), 35 bar H<sub>2</sub>, 5-7 bar NH<sub>3</sub>, 2 mL solvent, 80 °C, 24 h. <sup>b</sup>: same as “a” using NiCl<sub>2</sub> as nickel precursor. Conversions and yields were determined by GC using n-hexadecane standard.

**Table S2.** Hydrogenation of 5-chloropicolinonitrile to (5-chloropyridin-2-yl)methanamine: Testing of different noble metal catalyst.



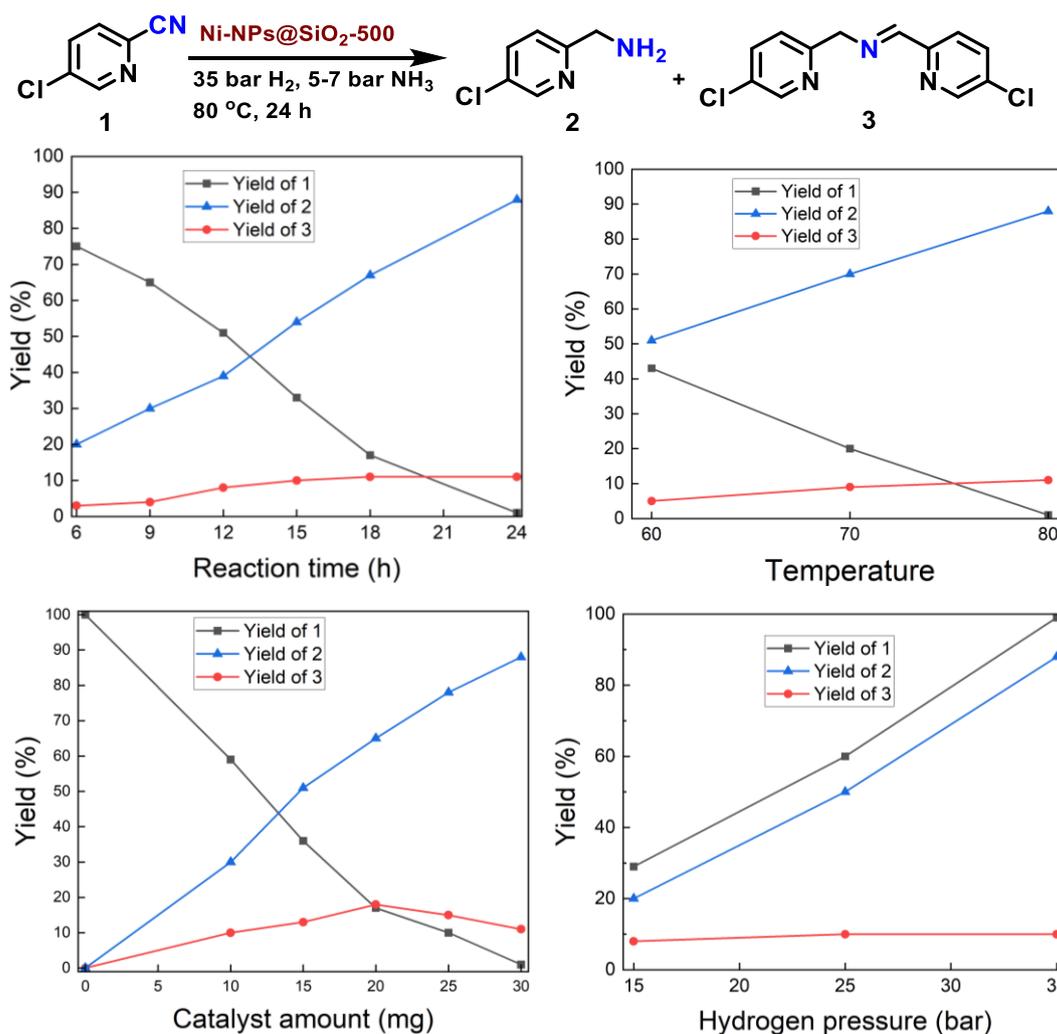
Catalyst	Conv (%)	Yield of 2 (%)	Yield of 3 (%)	Yield of 4 (%)	Yield of 7 (%)
5% Ru/C	42	11	<5	<5	17
5% Rh/C	35	9	<5	<5	11
10% Pd/C	50	19	<5	<5	21
5% Pt/C	20	8	-	-	10

**Reaction conditions:** 0.5 mmol nitrile, 4 mol% catalyst, 20 bar H<sub>2</sub>, 5-7 bar NH<sub>3</sub>, 2 mL MeOH, 40 °C, 12 h.

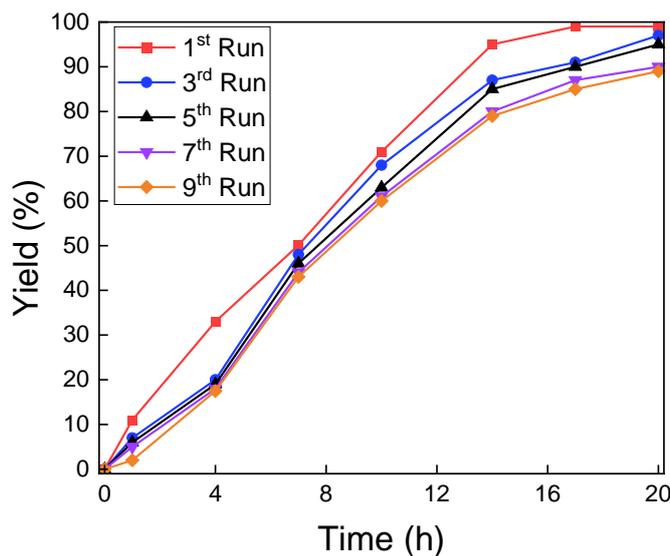
**Table S3.** Hydrogenation of (5-chloropyridin-2-yl)methanamine to product 3 and 4.

Catalyst	Conv (%)	Yield of 3 (%)	Yield of 4 (%)	Yield of 7 (%)
5% Ru/C	37	-	-	35
5% Rh/C	30	-	-	27
10% Pd/C	41	-	-	40
5% Pt/C	13	-	-	12

**Reaction conditions:** 0.5 mmol (5-chloropyridin-2-yl)methanamine, 4 mol% catalyst, 35 bar H<sub>2</sub>, 5-7 bar NH<sub>3</sub>, 2 mL MeOH, 40 °C, 24 h.

**Figure S11.** Hydrogenation of 5-chloropicolinonitrile to (5-chloropyridin-2-yl)methanamine: Testing different reaction time, temperature, catalyst amount and H<sub>2</sub> pressure. Reaction conditions: 0.5 mmol 5-chloropicolinonitrile, 10-30 mg Ni-NPs@SiO<sub>2</sub>-500 (3.0-9.0 mol% Ni), 15-35 bar H<sub>2</sub>, 5-7 bar NH<sub>3</sub>, 2 mL MeOH, 60-80 °C, 6-24 h. Conversions and yields are determined by GC using n-hexadecane

standard.



**Figure S12.** Hydrogenation of 5-chloropicolinonitrile to (5-chloropyridin-2-yl)methanamine: Kinetic profiles for recycled experiments.

### **S5. Catalyst recycling**

The magnetic stirring bar, 10 mmol benzonitrile, 320 mg Ni-NPs@SiO<sub>2</sub>-500 (4.8 mol% Ni) were transferred to 300 mL glass fitted autoclave and 40 mL of dry MeOH was added. The autoclave was flushed with hydrogen twice at 20 bar pressure and then it was pressurized with 5-7 bar ammonia gas and 35 bar hydrogen. The autoclave was placed into an aluminum block heated at 60 °C and stirred for 20 h. After the completion of the reaction, the autoclave was cooled to room temperature. The remaining ammonia and hydrogen were discharged, and reaction products were removed from the autoclave. To the reaction products, 500 μL n-hexadecane as standard was added. The catalyst was separated by centrifugation and the centrifugate containing reaction products were subjected to GC analysis. The separated catalyst was washed with water, methanol and ethyl acetate and then dried under vacuum. The dried catalyst was used for the next run without further purification or reactivation.

### **S6. Procedure of reaction upscaling**

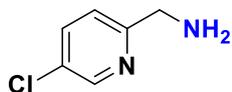
To a glass fitted 300 mL autoclave, the magnetic stirring bar and corresponding nitrile were transferred and then 30-70 mL of dry MeOH were added. Next, the required amount of catalyst (Ni-NPs@SiO<sub>2</sub>-500, 4.5-9.0 mol%; 30 mg for each 0.5 mmol substrate **27**; 15 mg for each 0.5 mmol substrate **35**, **39** and **72**; 20 mg for each 0.5 mmol substrate **83** and **103**) was added. The autoclave was closed and flushed with hydrogen twice at 40 bar pressure and then it was pressurized with 5-7 bar ammonia gas and 35 bar hydrogen. The autoclave was placed into an aluminium block preheated at 60 and 80 °C and the reactions were stirred for required time. After completion of the reaction, the

autoclave was cooled to room temperature. The remaining ammonia and hydrogen were discharged, and the reaction products were removed from the autoclave. The solid catalyst was filtered off and washed thoroughly with ethyl acetate. The reaction products were analyzed by GC-MS and the corresponding primary amines were purified by column chromatography (silica; n-hexane-ethyl acetate mixture).

## S7. References

S1. Jeangros, Q.; Hansen, T. W.; Wagner, J. B.; Dunin-Borkowski, R. E.; Hébert, C.; Hessler-Wyser, A. Oxidation mechanism of nickel particles studied in an environmental transmission electron microscope. *Acta. Mater* 2014, 67, 362-372.

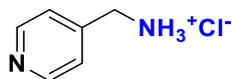
## S8. NMR data



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.35 (dd, *J* = 2.5, 0.8 Hz, 1H), 7.85 – 7.78 (m, 1H), 7.45 (d, *J* = 8.2 Hz, 1H), 3.73 (s, 2H), 2.87 (s, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 149.19, 148.54, 139.42, 139.18, 124.17, 42.70.

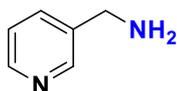
**HRMS (ESI-TOF):** calcd for C<sub>6</sub>H<sub>7</sub>ClN<sub>2</sub> [M+H] 143.0376; found 143.0375.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.83 (s, 5H), 7.60 (d, *J* = 4.7 Hz, 2H), 4.07 (s, 2H).

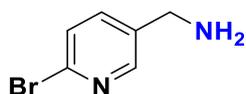
**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 150.16, 143.41, 124.53, 41.41.

**HRMS (ESI-TOF):** calcd for C<sub>6</sub>H<sub>8</sub>N<sub>2</sub> [M+H] 108.0687; found 109.0765.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.52 (d, *J* = 2.2 Hz, 1H), 8.41 (dd, *J* = 4.8, 1.7 Hz, 1H), 7.74 (dddt, *J* = 7.8, 2.4, 1.6, 0.7 Hz, 1H), 7.32 (ddd, *J* = 7.8, 4.8, 0.9 Hz, 1H), 3.73 (s, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 149.17, 147.94, 139.83, 135.23, 123.73, 43.59.

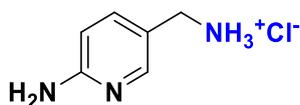


**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.32 (dq, *J* = 2.4, 0.8 Hz, 1H), 7.75 – 7.69 (m, 1H), 7.57 (d, *J* =

8.2 Hz, 1H), 3.70 (d,  $J = 0.7$  Hz, 2H), 1.95 (s, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  149.81, 139.91, 139.41, 139.02, 127.91, 42.77.

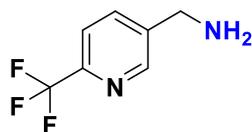
HRMS (ESI-TOF): calcd for  $\text{C}_6\text{H}_7\text{N}_2\text{Br}$  [M+H] 186.9871; found 186.9870.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.70 (s, 3H), 8.39 (s, 2H), 8.15 – 8.06 (m, 2H), 7.11 – 7.05 (m, 1H), 3.94 (d,  $J = 5.2$  Hz, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  154.21, 145.59, 136.75, 118.66, 113.61, 38.85.

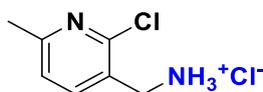
HRMS (ESI-TOF): calcd for  $\text{C}_6\text{H}_9\text{N}_3$  [M+H] 124.0797; found 124.0874.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.71 (d,  $J = 2.2$  Hz, 1H), 8.06 – 7.96 (m, 1H), 7.80 (pent,  $J = 3.6$  Hz, 1H), 3.86 (s, 2H), 2.28 (d,  $J = 5.2$  Hz, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  149.67, 145.28, 145.24, 144.82, 143.88, 136.94, 136.91, 127.74, 124.12, 120.56, 120.50, 116.88, 43.17.

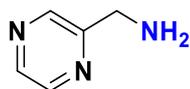
HRMS (ESI-TOF): calcd for  $\text{C}_7\text{H}_7\text{F}_3\text{N}_2$  [M+H] 177.0639; found 177.0641.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  9.05 – 8.96 (m, 3H), 8.69 (dd,  $J = 8.3, 2.0$  Hz, 1H), 7.98 (d,  $J = 8.3$  Hz, 1H), 4.24 (q,  $J = 5.7$  Hz, 2H), 2.78 (s, 3H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  153.67, 146.91, 141.82, 131.63, 127.76, 38.99, 19.37.

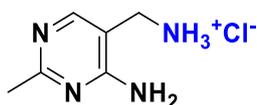
HRMS (ESI-TOF): calcd for  $\text{C}_7\text{H}_9\text{ClN}_2$  [M+H] 156.0640; found 157.0643.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.72 – 8.69 (m, 1H), 8.54 (dd,  $J = 2.7, 1.5$  Hz, 1H), 8.48 (d,  $J = 2.6$  Hz, 1H), 3.86 (d,  $J = 0.7$  Hz, 2H), 2.01 (s, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  158.82, 144.03, 143.86, 143.07, 45.87.

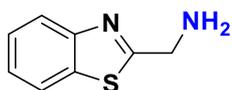
HRMS (ESI-TOF): calcd for  $\text{C}_5\text{H}_7\text{N}_3$  [M+H] 109.0640; found 110.0718.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 9.10 (d, *J* = 47.0 Hz, 2H), 8.72 (s, 3H), 8.36 (s, 1H), 4.05 – 3.95 (m, 2H), 2.51–2.44 (m, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 163.38, 161.30, 144.54, 108.62, 35.38, 21.42.

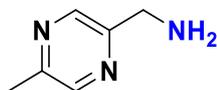
**HRMS (ESI-TOF):** calcd for C<sub>6</sub>H<sub>9</sub>N<sub>3</sub> [M+H] 138.0905; found 138.0983.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.05 (ddd, *J* = 7.9, 1.4, 0.7 Hz, 1H), 7.90 (ddd, *J* = 8.0, 1.3, 0.6 Hz, 1H), 7.46 (ddd, *J* = 8.1, 7.2, 1.4 Hz, 1H), 7.38 (ddd, *J* = 7.7, 7.2, 1.3 Hz, 1H), 4.14 (s, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 178.94, 153.86, 134.99, 126.22, 124.83, 122.57, 122.53, 44.38.

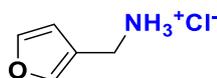
**HRMS (ESI-TOF):** calcd for C<sub>8</sub>H<sub>8</sub>N<sub>2</sub>S [M+H] 164.2213; found 164.2215.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.55 (d, *J* = 1.5 Hz, 1H), 8.42 (d, *J* = 1.6 Hz, 1H), 3.81 (s, 2H), 2.45 (s, 3H).

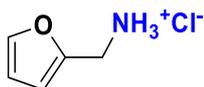
**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 155.46, 151.49, 143.37, 142.47, 45.50, 21.13.

**HRMS (ESI-TOF):** calcd for C<sub>6</sub>H<sub>9</sub>N<sub>3</sub> [M+H] 124.0875; found 124.0877.



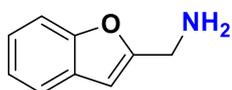
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.28 (s, 3H), 7.73 (d, *J* = 31.7 Hz, 2H), 6.73 (s, 1H), 3.90 (s, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 144.16, 142.38, 119.35, 111.60, 33.83.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.70 (s, 3H), 7.72 (dd, *J* = 1.9, 0.9 Hz, 1H), 6.65 – 6.39 (m, 2H), 4.04 – 4.02 (m, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 148.15, 143.97, 111.37, 110.72, 35.35.

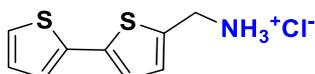


**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 7.62 – 7.48 (m, 2H), 7.30 – 7.18 (m, 2H), 6.77 (q, *J* = 0.9 Hz, 1H),

3.89 (d,  $J = 0.9$  Hz, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  157.85, 154.67, 128.71, 124.15, 123.13, 121.20, 111.32, 104.06, 45.48.

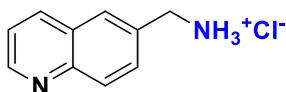
HRMS (ESI-TOF): calcd for  $\text{C}_9\text{H}_9\text{NO}$  [M+H] 147.1771; found 147.1776.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.55 (s, 3H), 7.53 (d,  $J = 4.8$  Hz, 1H), 7.30 (s, 1H), 7.22 (s, 2H), 7.10 (s, 1H), 4.20 (s, 2H).

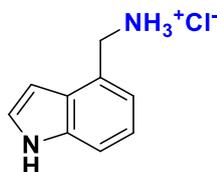
$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  141.10, 134.82, 134.26, 130.77, 128.90, 126.31, 124.81, 124.22, 39.29.

HRMS (ESI-TOF): calcd for  $\text{C}_9\text{H}_{10}\text{NS}_2$  [M+H] 195.0176; found 195.0180.



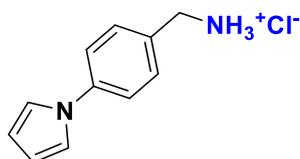
$^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  9.28 (dd,  $J = 5.1, 1.5$  Hz, 1H), 9.09 – 9.04 (m, 1H), 8.94 (s, 3H), 8.48 – 8.41 (m, 2H), 8.28 (dd,  $J = 8.8, 1.9$  Hz, 1H), 8.04 (dd,  $J = 8.4, 5.1$  Hz, 1H), 4.31 (q,  $J = 5.8$  Hz, 2H).

$^{13}\text{C}$  NMR (101 MHz, DMSO- $d_6$ )  $\delta$  146.86, 144.90, 139.50, 135.83, 134.80, 129.50, 128.48, 123.11, 122.86, 42.17.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  11.50 (s, 1H), 8.66 (s, 3H), 7.51 – 7.35 (m, 2H), 7.27 – 7.02 (m, 2H), 6.68 (ddd,  $J = 3.1, 1.9, 1.0$  Hz, 1H), 4.24 (q,  $J = 5.8$  Hz, 2H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  136.29, 127.05, 126.17, 125.43, 121.24, 119.38, 112.29, 99.75, 40.44.



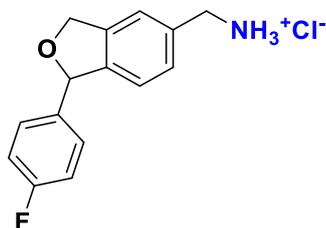
$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.55 (s, 3H), 7.61 (q,  $J = 8.5$  Hz, 4H), 7.41 (d,  $J = 2.1$  Hz, 2H), 6.28 (t,  $J = 2.0$  Hz, 2H), 4.02 (s, 2H).



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.65 (s, 3H), 7.54 – 7.38 (m, 2H), 7.09 – 6.94 (m, 2H), 4.49 (t, *J* = 5.0 Hz, 2H), 4.00 – 3.86 (m, 6H), 3.60 – 3.43 (m, 4H), 3.23 (d, *J* = 11.2 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 157.99, 131.13, 127.39, 115.16, 63.56, 62.94, 55.12, 52.08, 41.98.

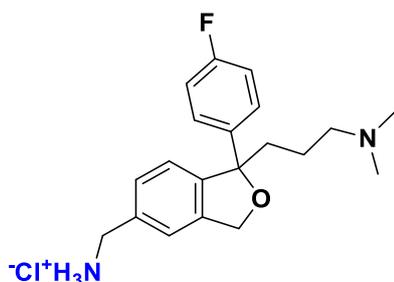
**HRMS (ESI-TOF):** calcd for C<sub>13</sub>H<sub>20</sub>N<sub>2</sub>O<sub>2</sub> [M+H] 236.3151; found 236.3155.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.67 (s, 3H), 7.54 (d, *J* = 1.6 Hz, 1H), 7.45 – 7.34 (m, 3H), 7.23 – 7.14 (m, 2H), 7.08 (dt, *J* = 7.8, 0.8 Hz, 1H), 6.19 (t, *J* = 2.3 Hz, 1H), 5.51 – 4.96 (m, 2H), 4.33 – 3.88 (m, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 163.79, 160.56, 142.56, 139.51, 139.29, 139.24, 134.30, 129.06, 128.98, 128.87, 122.62, 122.47, 115.88, 115.60, 84.48, 72.85, 42.38.

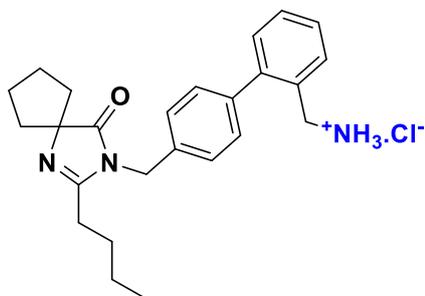
**HRMS (ESI-TOF):** calcd for C<sub>15</sub>H<sub>15</sub>ClFNO [M+H] 279.7390; found 279.7411.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 10.73 (s, 1H), 8.67 (bs, 3H), 7.64 – 7.52 (m, 3H), 7.50 – 7.42 (m, 2H), 7.20 – 7.06 (m, 2H), 5.14 (q, *J* = 13.0 Hz, 2H), 3.97 (q, *J* = 5.8 Hz, 2H), 3.00 (q, *J* = 7.1 Hz, 2H), 2.63 – 2.57 (m, 6H), 2.24 (t, *J* = 8.1 Hz, 2H), 1.49 (ddt, *J* = 21.8, 14.0, 7.1 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 161.82 (d, *J* = 341.1 Hz), 143.99, 141.11, 138.79, 133.91, 128.73, 126.87 (d, *J* = 7.7 Hz), 122.09 (d, *J* = 8.0 Hz), 115.04 (d, *J* = 20.7 Hz), 90.04, 71.38, 56.34, 41.93, 41.83, 37.37, 19.10. White solid.

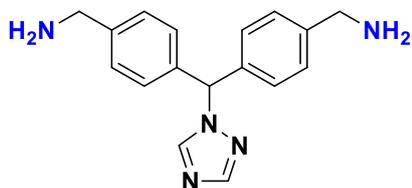
**HRMS (ESI-TOF):** calcd for C<sub>20</sub>H<sub>25</sub>FN<sub>2</sub>O [M+H] 329.2001; found 329.2004.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.74 (bs, 3H), 7.82 – 7.77 (m, 1H), 7.50 – 7.37 (m, 6H), 7.28 – 7.23 (m, 1H), 5.01 (s, 2H), 3.90 (q, *J* = 5.8 Hz, 2H), 2.95 – 2.88 (m, 2H), 2.23 – 1.78 (m, 8H), 1.66 – 1.54 (m, 2H), 1.39 – 1.21 (m, 2H), 0.82 (t, *J* = 7.3 Hz, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 179.10, 172.81, 140.81, 139.32, 134.16, 131.43, 130.04, 129.73, 128.74, 128.35, 127.97, 127.24, 71.52, 43.60, 36.78, 27.00, 26.45, 25.23, 21.48, 13.44. White solid.

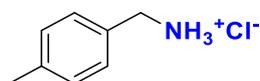
**HRMS (ESI-TOF):** calcd for C<sub>25</sub>H<sub>31</sub>N<sub>3</sub>O [M+H] 390.2561; found 390.2565.



**<sup>1</sup>H NMR (300 MHz, MeOD)** δ 8.36 (s, 1H), 8.03 (s, 1H), 7.38 – 7.08 (m, 8H), 6.97 – 6.90 (m, 1H), 3.85 – 3.67 (m, 4H).

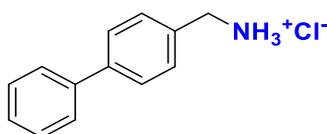
**<sup>13</sup>C NMR (75 MHz, MeOD)** δ 152.51, 141.15, 129.96, 129.50, 129.40, 128.91, 68.15, 53.19. Off white solid.

**HRMS (ESI-TOF):** calcd for C<sub>17</sub>H<sub>19</sub>N<sub>5</sub> [M+H] 294.1655; found 294.1601.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.62 (s, 3H), 7.62 – 7.33 (m, 2H), 7.20 (d, *J* = 7.7 Hz, 2H), 3.94 (s, 2H), 2.31 (s, 3H).

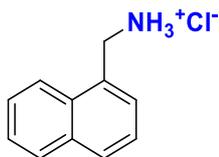
**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 138.12, 131.56, 129.49, 129.45, 42.31, 21.23.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.78 (s, 3H), 7.74 – 7.57 (m, 6H), 7.53 – 7.31 (m, 3H), 4.06 (s, 2H).

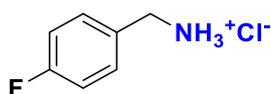
**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 140.60, 140.03, 133.79, 130.14, 129.46, 128.13, 127.20, 127.16,

42.25.



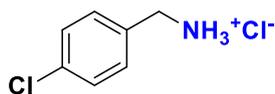
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  9.97 – 8.20 (m, 3H), 8.18 – 8.14 (m, 1H), 8.02 – 7.96 (m, 2H), 7.70 (dd,  $J = 7.1, 1.2$  Hz, 1H), 7.63 – 7.55 (m, 3H), 4.50 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  133.66, 131.14, 130.96, 129.31, 129.09, 127.57, 127.15, 126.64, 125.83, 123.97, 39.75.



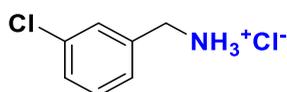
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  = 9.01 – 8.51 (s, 3H), 7.84 – 7.51 (m, 2H), 7.40 – 7.07 (m, 2H), 4.14 – 3.84 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  162.50 (d,  $J = 244.5$  Hz), 131.90 (d,  $J = 8.5$  Hz), 130.88 (d,  $J = 3.0$  Hz), 115.74 (d,  $J = 21.4$  Hz), 41.79.



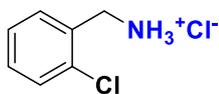
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.72 (s, 3H), 7.67 – 7.52 (m, 2H), 7.48 (dt,  $J = 8.6, 2.3$  Hz, 2H), 4.01 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  133.62, 131.51, 131.49, 128.93, 41.83.



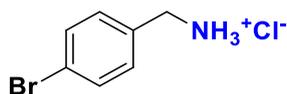
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.60 (s, 3H), 7.82 – 7.74 (m, 2H), 7.37 – 7.29 (m, 2H), 3.97 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  137.73, 134.32, 131.78, 129.43, 129.01, 95.31, 42.04.



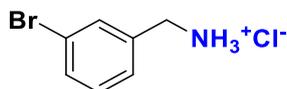
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.67 (s, 3H), 7.66 (ddd,  $J = 6.2, 4.0, 2.2$ , 1H), 7.59 – 7.48 (m, 1H), 7.48 – 7.26 (m, 2H), 4.12 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  132.41, 130.76, 129.32, 129.25, 128.35, 127.55, 43.65.



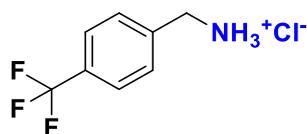
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.73 (s, 3H), 7.64 – 7.55 (m, 2H), 7.55 – 7.46 (m, 2H), 3.99 (q,  $J$  = 5.6 Hz, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  134.00, 131.83, 122.13, 41.88.



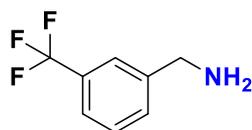
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.71 (s, 3H), 7.94 – 7.20 (m, 4H), 4.02 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  137.26, 132.27, 131.63, 131.11, 128.65, 122.07, 41.84.



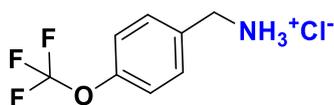
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.81 (s, 3H), 7.77 (s, 4H), 4.12 (q,  $J$  = 5.8 Hz, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  139.33, 130.28, 129.92, 129.49, 129.07, 128.65, 126.40, 125.84, 125.78, 125.73, 125.68, 122.80, 42.00.



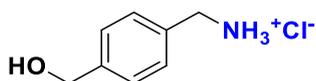
$^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (dq,  $J$  = 2.0, 1.0 Hz, 1H), 7.51 – 7.38 (m, 3H), 3.91 (d,  $J$  = 1.9 Hz, 2H), 1.61 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{Chloroform-}d$ )  $\delta$  144.02, 130.98, 130.55, 130.49, 130.47, 128.89, 126.03, 123.82, 123.77, 123.61, 123.56, 122.42, 45.93.



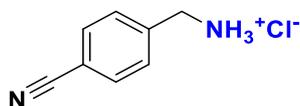
$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.72 (s, 3H), 7.67 (d,  $J$  = 7.7 Hz, 2H), 7.39 (d,  $J$  = 7.5 Hz, 2H), 4.04 (s, 2H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  148.69, 134.12, 131.70, 121.54, 120.49 (q,  $J$  = 256.4 Hz), 41.78.



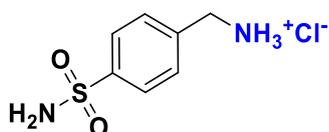
$^1\text{H NMR}$  (300 MHz, DMSO- $d_6$ )  $\delta$  8.57 (s, 3H), 7.48 – 7.26 (m, 4H), 4.48 (s, 2H), 3.95 (t,  $J$  = 5.7 Hz, 2H).

$^{13}\text{C NMR}$  (75 MHz, DMSO- $d_6$ )  $\delta$  143.31, 132.73, 129.22, 126.93, 62.91, 42.39.



$^1\text{H NMR}$  (300 MHz, DMSO- $d_6$ )  $\delta$  8.72 (s, 3H), 8.00 – 7.87 (m, 2H), 7.81 – 7.62 (m, 2H), 4.12 (s, 2H).

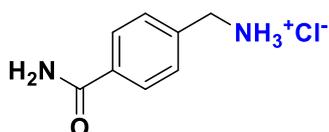
$^{13}\text{C NMR}$  (75 MHz, DMSO- $d_6$ )  $\delta$  140.15, 132.86, 130.31, 119.06, 111.54, 42.11.



$^1\text{H NMR}$  (300 MHz, DMSO- $d_6$ )  $\delta$  8.63 (s, 3H), 7.87 – 7.82 (m, 2H), 7.69 (d,  $J$  = 8.0 Hz, 2H), 7.44 (s, 2H), 4.09 (d,  $J$  = 5.7 Hz, 2H).

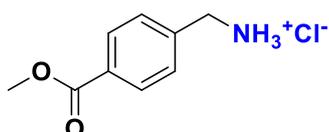
$^{13}\text{C NMR}$  (75 MHz, DMSO- $d_6$ )  $\delta$  144.54, 138.32, 129.90, 126.20, 42.07.

**HRMS (ESI-TOF):** calcd for  $\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2\text{S}$  [M+H] 187.0541; found 187.0543.



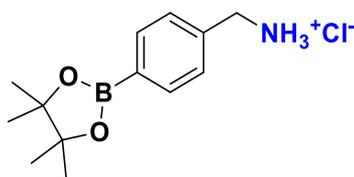
$^1\text{H NMR}$  (300 MHz, DMSO- $d_6$ )  $\delta$  8.68 (s, 3H), 8.12 – 8.04 (m, 1H), 7.93 – 7.89 (m, 2H), 7.62 – 7.56 (m, 2H), 7.43 (s, 1H), 4.07 (d,  $J$  = 5.5 Hz, 2H).

$^{13}\text{C NMR}$  (75 MHz, DMSO- $d_6$ )  $\delta$  167.87, 137.66, 134.58, 129.18, 128.11, 42.20.



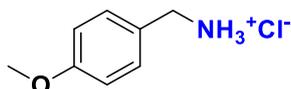
$^1\text{H NMR}$  (300 MHz, DMSO- $d_6$ )  $\delta$  8.82 (s, 3H), 8.00 – 7.94 (m, 2H), 7.74 – 7.64 (m, 2H), 4.10 (q,  $J$  = 5.6 Hz, 2H), 3.85 (s, 3H).

$^{13}\text{C NMR}$  (75 MHz, DMSO- $d_6$ )  $\delta$  166.37, 139.93, 129.87, 129.72, 129.66, 52.71, 42.13.



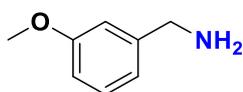
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.83 – 8.50 (s, 3H), 7.67 – 7.60 (d, *J* = 7.5 Hz, 2H), 7.51 – 7.45 (d, *J* = 7.6 Hz, 2H), 4.01 – 3.93 (s, 2H), 1.30 – 1.20 (s, 12H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 137.78, 134.92, 129.44, 128.79, 84.18, 42.49, 25.12.



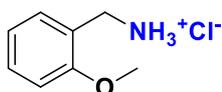
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.65 (s, 3H), 7.67 – 7.26 (m, 2H), 7.10 – 6.71 (m, 2H), 4.11 – 3.81 (m, 2H), 3.74 (s, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 159.74, 131.08, 126.47, 114.31, 55.67, 42.05.



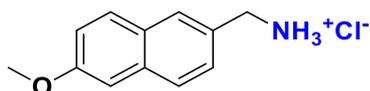
**<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)** δ 7.23 (td, *J* = 7.9, 1.3 Hz, 1H), 6.95 – 6.83 (m, 2H), 6.77 (dq, *J* = 8.3, 1.6 Hz, 1H), 3.82 – 3.79 (m, 2H), 3.78 (dd, *J* = 2.1, 1.0 Hz, 3H).

**<sup>13</sup>C NMR (75 MHz, Chloroform-*d*)** δ 159.85, 144.93, 129.56, 119.35, 112.63, 112.24, 55.17, 46.42.



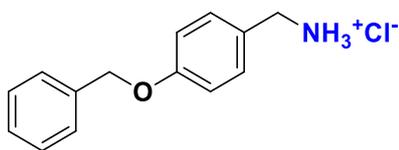
**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)** δ 8.48 (s, 3H), 7.58 – 6.87 (m, 4H), 3.93 (s, 2H), 3.83 (s, 3H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 157.61, 130.71, 130.58, 122.29, 120.71, 111.34, 55.99, 37.86.



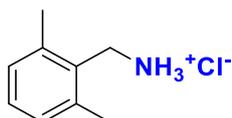
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.61 (s, 3H), 7.93 (s, 1H), 7.84 (dd, *J* = 13.4, 8.6 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 2.5 Hz, 1H), 7.24 – 7.17 (m, 1H), 4.13 (d, *J* = 5.7 Hz, 2H), 3.88 (d, *J* = 1.2 Hz, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 158.12, 134.49, 129.75, 129.64, 128.45, 128.39, 127.54, 119.61, 106.35, 55.71, 42.76, 31.17.



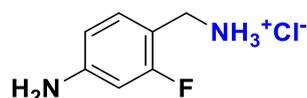
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.59 (s, 3H), 7.48 – 7.30 (m, 7H), 7.06 – 7.00 (m, 2H), 5.13 (s, 2H), 3.99 – 3.84 (m, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 158.77, 137.44, 131.07, 128.92, 128.30, 128.10, 126.73, 115.25, 69.62, 42.04.



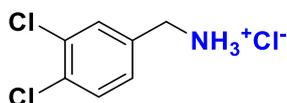
**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)** δ 8.40 (s, 3H), 7.18 (dd, *J* = 8.2, 6.7 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 2H), 4.00 (q, *J* = 5.7 Hz, 2H), 2.41 (s, 6H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 138.53, 131.28, 129.25, 128.78, 36.72, 19.94.



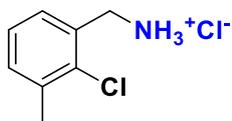
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.72 (s, 3H), 7.61 (dd, *J* = 6.3, 2.5 Hz, 1H), 7.52 – 7.36 (m, 2H), 4.17 – 3.93 (m, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 161.30, 158.03, 129.38, 129.34, 126.32, 126.27, 126.18, 126.06, 123.01, 122.79, 117.41, 117.10, 35.90, 35.85.



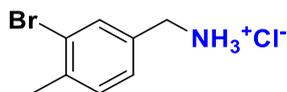
**<sup>1</sup>H NMR (400 MHz, DMSO)** δ 8.75 (s, 3H), 7.88 (d, *J* = 2.0 Hz, 1H), 7.68 (d, *J* = 8.3 Hz, 1H), 7.54 (dd, *J* = 8.3, 2.1 Hz, 1H), 4.03 (q, *J* = 5.6 Hz, 2H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 135.70, 131.72, 131.49, 131.41, 131.07, 130.02, 41.33.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.82 (s, 3H), 7.51 (ddq, *J* = 7.5, 1.9, 0.6 Hz, 1H), 7.41 – 7.28 (m, 2H), 4.11 (s, 2H), 2.36 (d, *J* = 0.7 Hz, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 136.69, 133.41, 132.31, 131.65, 128.45, 127.26, 40.34, 20.59.



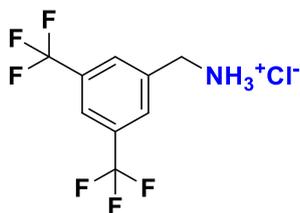
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.67 (s, 3H), 7.79 (d,  $J = 2.2$  Hz, 1H), 7.59 – 7.03 (m, 2H), 3.98 (s, 2H), 2.34 (d,  $J = 4.2$  Hz, 3H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  137.80, 134.40, 133.03, 131.51, 128.93, 124.44, 41.53, 22.58.



$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.76 (s, 3H), 7.98 (d,  $J = 2.1$  Hz, 1H), 7.84 (d,  $J = 8.5$  Hz, 1H), 7.71 (ddd,  $J = 8.5, 2.1, 0.9$  Hz, 1H), 4.19 (s, 2H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  138.16, 135.05, 131.03, 129.45, 128.71, 128.66, 126.52, 126.22, 38.73.



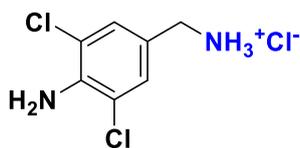
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.91 (s, 3H), 8.36 (s, 2H), 8.11 (s, 1H), 4.26 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  137.97, 131.29, 130.86, 130.41, 129.98, 125.52, 122.42, 121.90, 41.51.



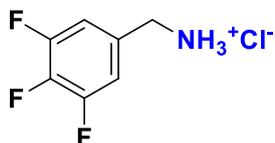
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.42 (s, 3H), 7.20 (d,  $J = 8.6$  Hz, 1H), 6.84 (d,  $J = 8.6$  Hz, 1H), 3.98 – 3.65 (m, 11H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  154.42, 151.84, 141.81, 125.21, 119.87, 108.14, 61.51, 60.87, 56.44, 37.25.



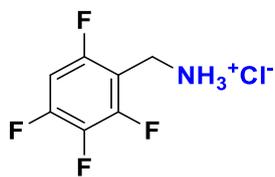
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.52 (s, 3H), 7.43 (s, 2H), 6.36 (s, 2H), 4.06 – 3.73 (m, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  141.63, 130.10, 129.59, 123.92, 122.94, 117.99, 41.29.



$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.80 (s, 3H), 7.95 – 7.01 (m, 2H), 4.05 (s, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  152.06, 152.01, 151.93, 151.88, 148.78, 148.73, 148.65, 148.60, 140.87, 140.66, 140.46, 137.56, 137.35, 137.15, 132.07, 132.00, 131.96, 131.89, 131.84, 131.78, 114.72, 114.64, 114.53, 114.44, 41.36.



$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.91 (s, 3H), 7.69 – 7.58 (m, 1H), 4.03 (t,  $J = 1.5$  Hz, 2H).

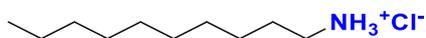
$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  157.91, 157.87, 157.75, 157.62, 154.63, 154.51, 154.45, 154.40, 154.33, 152.73, 152.59, 152.52, 152.30, 151.78, 151.64, 151.56, 149.42, 149.34, 149.20, 149.13, 149.07, 148.99, 148.45, 148.38, 148.31, 148.24, 148.17, 148.09, 138.58, 138.45, 138.38, 138.24, 135.33, 135.19, 135.12, 134.98, 134.91, 108.96, 108.90, 108.73, 108.67, 108.61, 108.44, 108.39, 102.65, 102.60, 102.36, 102.31, 102.27, 102.22, 101.98, 101.93, 30.09.

**HRMS (ESI-TOF):** calcd for  $\text{C}_7\text{H}_5\text{NF}_4$  [ $\text{M}+\text{H}$ ] 180.0436; found 180.0441.



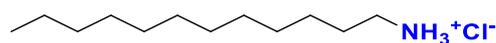
$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.37 – 7.57 (m, 3H), 2.73 (d,  $J = 8.1$  Hz, 2H), 1.53 (pent,  $J = 6.2$  Hz, 2H), 1.27 (t,  $J = 9.4$  Hz, 6H), 0.90 – 0.83 (m, 3H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  39.19, 31.20, 27.36, 25.99, 22.37, 14.33.



$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  5.66 (s, 3H), 2.62 (t,  $J = 7.3$  Hz, 2H), 1.45 (q,  $J = 7.1$  Hz, 2H), 1.25 (s, 14H), 0.89 – 0.82 (m, 3H).

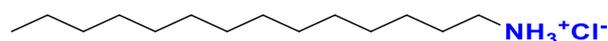
$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  40.44, 31.80, 30.25, 29.48, 29.28, 29.22, 26.67, 22.58, 14.36.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.11 (s, 3H), 2.87 – 2.60 (m, 2H), 1.55 (pent,  $J = 7.2$  Hz, 2H), 1.24 (s, 18H), 0.90 – 0.82 (m, 3H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  39.14, 31.78, 29.53, 29.50, 29.43, 29.34, 29.20, 29.04, 27.38, 26.36, 22.58, 14.42.

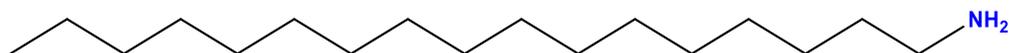
HRMS (ESI-TOF): calcd for  $\text{C}_{12}\text{H}_{27}\text{N}$  [M+H] 186.2222; found 186.2226.



$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.05 (s, 3H), 2.69 (s, 2H), 1.51 (s, 2H), 1.21 (s, 22H), 0.86 – 0.80 (m, 3H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  39.15, 31.74, 29.49, 29.40, 29.30, 29.16, 29.01, 27.37, 26.33, 22.54, 14.41.

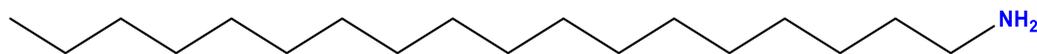
HRMS (ESI-TOF): calcd for  $\text{C}_{14}\text{H}_{31}\text{N}$  [M+H] 214.2534; found 214.2537.



$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.70 (t,  $J = 7.0$  Hz, 2H), 1.50 – 1.42 (m, 2H), 1.26 (d,  $J = 7.0$  Hz, 30H), 0.87 (t,  $J = 6.5$  Hz, 3H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  42.06, 31.94, 29.71, 29.68, 29.64, 29.48, 29.38, 26.88, 22.71, 14.14.

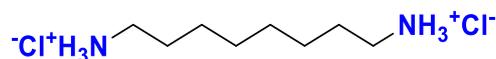
HRMS (ESI-TOF): calcd for  $\text{C}_{17}\text{H}_{37}\text{N}$  [M+H] 256.3004; found 256.3004.



$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.61 (t,  $J = 6.9$  Hz, 2H), 1.36 (pent,  $J = 6.6$  Hz, 4H), 1.19 (s, 30H), 0.84 – 0.78 (m, 3H).

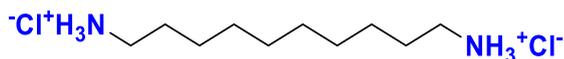
$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  42.19, 33.79, 31.94, 29.71, 29.67, 29.53, 29.38, 26.92, 22.71, 14.13.

HRMS (ESI-TOF): calcd for  $\text{C}_{18}\text{H}_{39}\text{N}$  [M+H] 270.3161; found 270.3162.



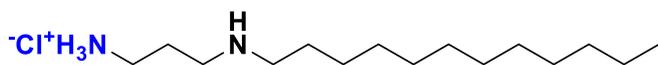
$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  8.07 (s, 6H), 2.73 (q,  $J = 6.3$  Hz, 4H), 1.55 (q,  $J = 7.3$  Hz, 4H), 1.30 (d,  $J = 13.3$  Hz, 8H).

$^{13}\text{C}$  NMR (75 MHz, DMSO- $d_6$ )  $\delta$  39.12, 28.71, 27.31, 26.16.



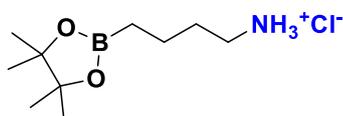
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.04 (s, 6H), 2.73 (q,  $J = 6.7$  Hz, 4H), 1.54 (q,  $J = 7.4$  Hz, 4H), 1.27 (s, 12H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  39.44, 29.13, 28.95, 27.37, 26.29.



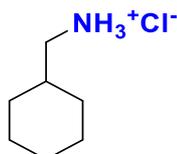
$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.01 (s, 3H), 3.04 – 2.54 (m, 4H), 1.24 (s, 20H), 0.86 (d,  $J = 6.3$  Hz, 3H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  39.16, 31.76, 29.48, 29.40, 29.31, 29.18, 29.01, 27.39, 26.49, 26.32, 22.56, 14.43.



$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.06 (s, 3H), 2.69 (s, 2H), 1.51 (q,  $J = 7.6$  Hz, 2H), 1.33 (pent,  $J = 8.1$  Hz, 2H), 1.16 (s, 12H), 0.66 (t,  $J = 7.5$  Hz, 2H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{DMSO-}d_6$ )  $\delta$  83.15, 38.99, 29.88, 25.43, 25.13, 21.15.



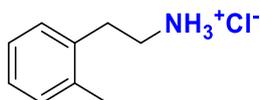
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.20 (s, 3H), 2.57 (d,  $J = 6.5$  Hz, 2H), 1.79 – 1.48 (m, 6H), 1.15 (t,  $J = 10.9$  Hz, 3H), 0.90 (t,  $J = 11.7$  Hz, 2H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  44.80, 35.76, 30.30, 26.13, 25.54.



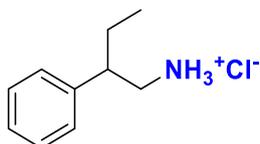
$^1\text{H NMR}$  (300 MHz,  $\text{DMSO-}d_6$ )  $\delta$  5.33 (s, 3H), 2.31 (s, 2H), 1.93 (pent,  $J = 3.1$  Hz, 3H), 1.69 – 1.56 (m, 6H), 1.47 (d,  $J = 3.0$  Hz, 6H).

$^{13}\text{C NMR}$  (75 MHz,  $\text{DMSO-}d_6$ )  $\delta$  52.58, 39.75, 36.95, 33.05, 28.12.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.26 (s, 3H), 7.06 (t, *J*=7.2, 1H), 6.90 (d, *J*=8.7, 3H), 2.93 – 2.71 (m, 4H), 2.13 (s, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 137.99, 137.76, 129.62, 128.87, 127.65, 126.02, 40.44, 33.22, 21.39.



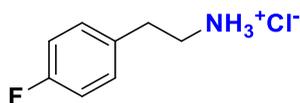
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.24 (s, 3H), 7.51 – 7.04 (m, 5H), 3.04 (dd, *J* = 12.0, 7.0 Hz, 1H), 2.99 – 2.79 (m, 2H), 1.95 – 1.66 (m, 1H), 1.52 (ddq, *J* = 14.2, 9.0, 7.2 Hz, 1H), 0.67 (t, *J* = 7.3 Hz, 3H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 141.50, 129.07, 128.37, 127.38, 45.26, 44.17, 26.28, 11.87.



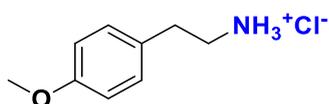
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.49 (s, 3H), 8.20 (d, *J* = 8.0 Hz, 1H), 7.95 – 7.87 (m, 1H), 7.80 (dd, *J* = 6.1, 3.3 Hz, 1H), 7.57 – 7.48 (m, 2H), 7.41 (q, *J* = 3.0 Hz, 2H), 3.44 (dd, *J* = 10.1, 5.9 Hz, 2H), 3.06 (dt, *J* = 11.0, 5.6 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 134.06, 133.93, 131.75, 129.16, 127.85, 127.35, 126.84, 126.29, 126.14, 124.03, 30.60.



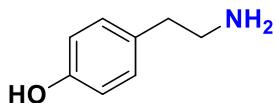
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.29 (s, 3H), 7.22 (dd, *J* = 8.2, 5.1 Hz, 2H), 7.05 (t, *J* = 8.4 Hz, 2H), 3.00 – 2.75 (m, 4H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 163.09, 159.88, 134.07, 134.04, 131.04, 130.93, 115.84, 115.56, 40.39, 32.42.



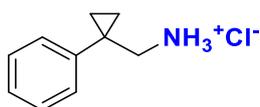
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.26 (s, 3H), 7.09 (d, *J* = 8.1 Hz, 2H), 6.79 (d, *J* = 8.1 Hz, 2H), 3.63 (s, 3H), 2.92 – 2.74 (m, 4H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 158.42, 130.07, 129.71, 114.41, 55.52, 40.63, 32.46.



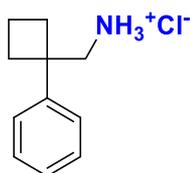
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 9.68 – 9.16 (s, 1H), 8.48 – 7.83 (s, 3H), 7.07 – 6.99 (m, 2H), 6.77 – 6.70 (m, 2H), 2.98 – 2.86 (ddt, *J* = 14.2, 9.1, 5.0 Hz, 2H), 2.83 – 2.74 (dd, *J* = 9.3, 5.8 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 156.66, 129.98, 127.76, 115.85, 40.71, 32.61.



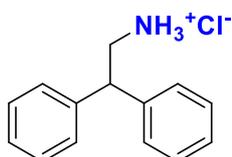
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 7.89 (s, 3H), 7.38 – 7.13 (m, 5H), 2.96 (s, 2H), 0.97 – 0.90 (m, 2H), 0.80 (s, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 141.52, 129.00, 128.97, 127.26, 47.59, 23.94, 13.55.



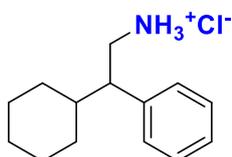
**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)** δ 8.05 (s, 3H), 7.33 (d, *J* = 7.1 Hz, 2H), 7.22 (d, *J* = 7.2 Hz, 3H), 3.39 (s, 2H), 2.31 (dq, *J* = 47.0, 9.5 Hz, 4H), 1.89 (dq, *J* = 114.7, 9.0 Hz, 2H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 146.12, 128.92, 126.84, 126.53, 48.34, 45.07, 30.99, 15.57.



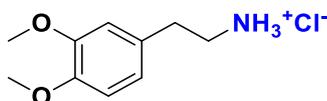
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.19 (s, 3H), 7.40 – 7.21 (m, 10H), 4.46 (t, *J* = 7.8 Hz, 1H), 3.52 (d, *J* = 7.9 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 141.70, 129.21, 128.34, 127.41, 49.03, 42.94.



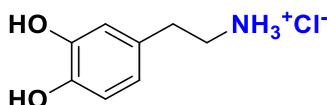
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.10 (s, 3H), 7.50 – 6.85 (m, 5H), 3.05 (dd, *J* = 12.8, 9.5 Hz, 1H), 1.86 – 1.47 (m, 5H), 1.45 – 1.30 (m, 1H), 1.30 – 0.53 (m, 5H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 139.97, 129.22, 128.77, 127.31, 49.25, 41.35, 40.31, 31.26, 29.72, 26.04.



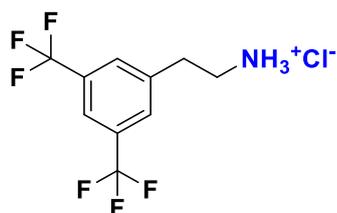
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.20 (s, 3H), 6.80 – 6.73 (m, 2H), 6.65 (d, *J* = 7.9 Hz, 1H), 3.62 (d, *J* = 10.3 Hz, 6H), 2.88 (s, 2H), 2.76 (t, *J* = 7.4 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 149.13, 147.95, 130.20, 120.93, 112.92, 112.39, 56.01, 55.93, 40.57, 32.89.



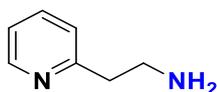
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 9.10 – 8.66 (d, *J* = 22.4 Hz, 2H), 8.47 – 7.80 (s, 3H), 6.74 – 6.61 (m, 2H), 6.50 – 6.44 (m, 1H), 2.97 – 2.83 (t, *J* = 7.9 Hz, 2H), 2.78 – 2.64 (dd, *J* = 9.6, 6.2 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 32.80, 40.78, 116.28, 116.54, 119.67, 128.45, 144.52, 145.76.



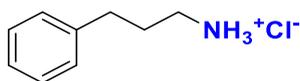
**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)** δ 8.30 (s, 3H), 8.04 (d, *J* = 1.7 Hz, 2H), 7.96 (dt, *J* = 1.9, 1.0 Hz, 1H), 3.15 (s, 4H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 141.49, 131.22, 130.90, 130.57, 130.49, 130.45, 130.25, 127.93, 125.22, 122.51, 121.02, 120.98, 120.94, 119.80, 39.60, 32.65.



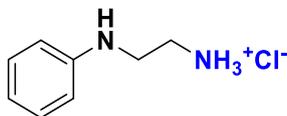
**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)** δ 8.47 (ddd, *J* = 4.9, 1.9, 1.0 Hz, 1H), 7.68 (td, *J* = 7.6, 1.9 Hz, 1H), 7.29 – 7.07 (m, 2H), 2.92 – 2.85 (m, 2H), 2.79 (td, *J* = 6.8, 1.2 Hz, 2H), 1.45 (s, 2H).

**<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)** δ 160.74, 149.42, 136.73, 123.62, 121.63, 42.39, 42.37.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.27 (s, 3H), 7.32 – 7.24 (m, 2H), 7.23 – 7.16 (m, 3H), 2.79 – 2.69 (m, 2H), 2.68 – 2.60 (m, 2H), 1.94 – 1.83 (m, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 141.38, 128.85, 128.74, 126.45, 38.72, 32.36, 29.13.



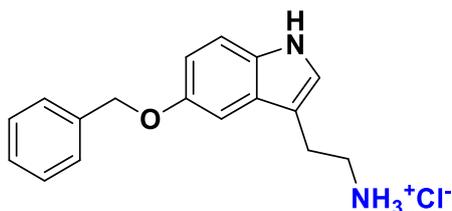
**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 8.23 (s, 3H), 7.17 (t, *J* = 7.6 Hz, 2H), 6.88 (d, *J* = 7.9 Hz, 2H), 6.77 (t, *J* = 7.3 Hz, 1H), 3.15 (t, *J* = 7.0 Hz, 2H), 2.86 (s, 2H), 1.90 (pent, *J* = 7.2 Hz, 2H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 145.21, 129.62, 120.19, 115.97, 42.77, 37.10, 25.99.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 7.66 (s, 3H), 7.13 (t, *J* = 7.7 Hz, 2H), 6.82 – 6.44 (m, 3H), 3.32 (t, *J* = 7.2 Hz, 4H), 2.80 (d, *J* = 7.5 Hz, 2H), 1.83 (pent, *J* = 7.3 Hz, 2H), 1.04 (t, *J* = 6.9 Hz, 3H).

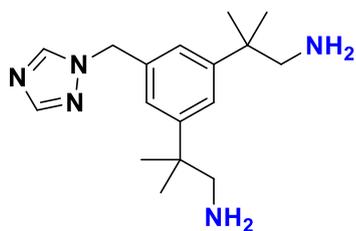
**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 147.89, 129.57, 115.69, 112.28, 47.17, 44.56, 37.30, 26.00, 12.49.



**<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)** δ 10.89 (s, 1H), 8.21 (s, 3H), 7.53 – 7.47 (m, 2H), 7.40 (ddt, *J* = 7.9, 6.3, 1.1 Hz, 2H), 7.35 – 7.30 (m, 1H), 7.30 – 7.23 (m, 2H), 7.20 (d, *J* = 2.4 Hz, 1H), 6.82 (dd, *J* = 8.7, 2.4 Hz, 1H), 5.12 (s, 2H), 3.06 (s, 4H).

**<sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)** δ 152.58, 138.26, 132.09, 128.81, 128.20, 128.09, 127.66, 124.53, 112.64, 112.32, 109.81, 102.27, 70.36, 39.16, 23.59.

**HRMS (ESI-TOF):** calcd for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O [M+H] 267.1497; found 267.1503.



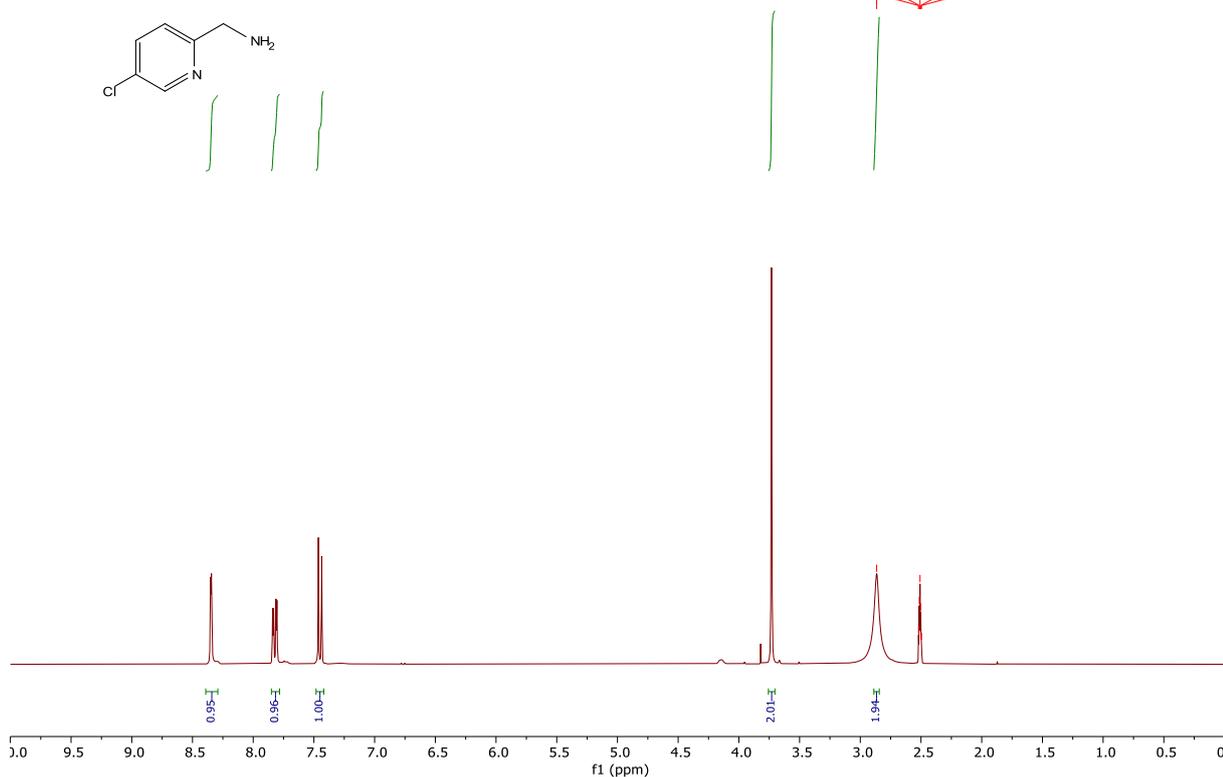
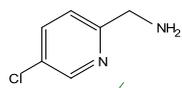
**<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)** δ 8.06 (s, 1H), 7.91 (s, 1H), 7.27 – 7.26 (m, 1H), 7.05 – 7.01 (m, 2H), 5.30 (s, 2H), 2.73 (s, 4H), 1.24 (s, 16H).

**<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)** δ 152.02, 148.14, 143.23, 134.49, 124.37, 123.66, 54.39, 54.02, 39.83, 26.41. Colorless oil.

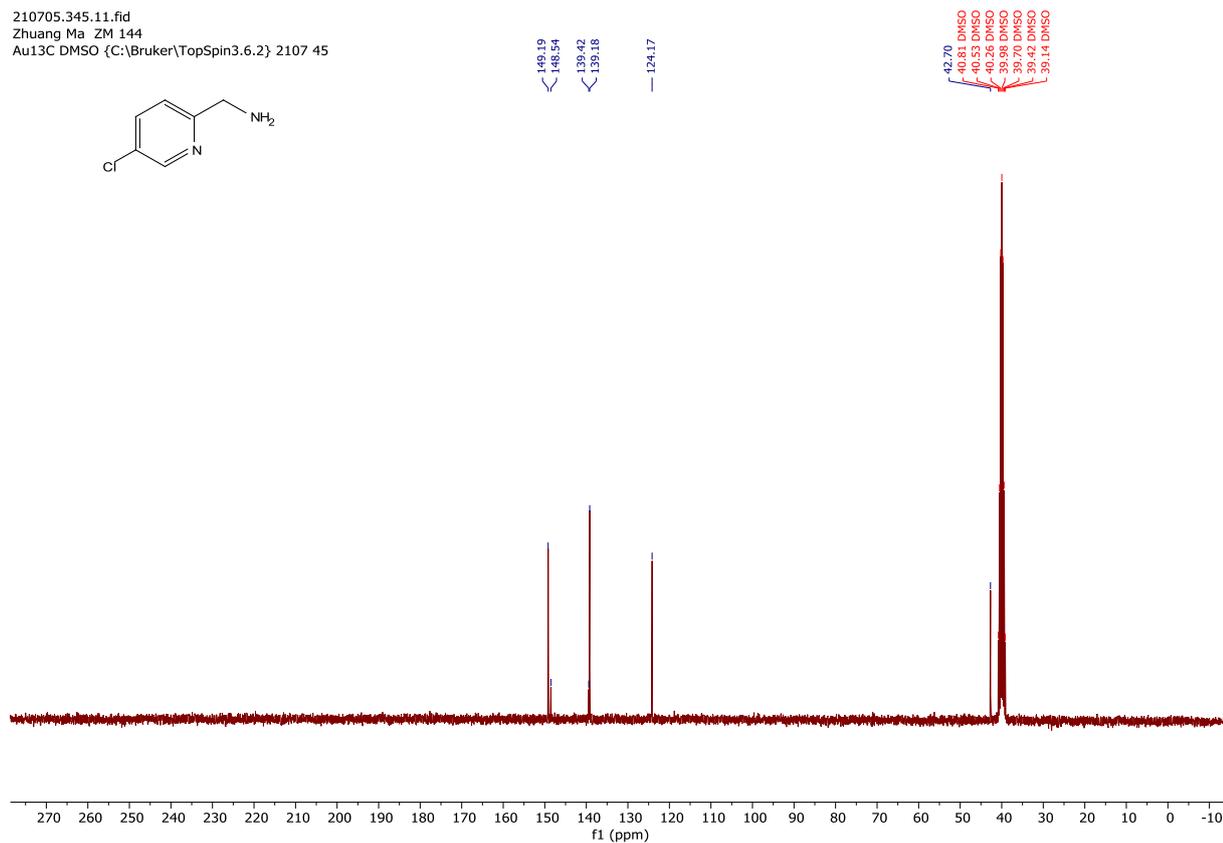
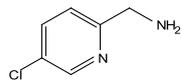
**HRMS (ESI-TOF):** calcd for C<sub>17</sub>H<sub>27</sub>N<sub>5</sub> [M+H] 302.2271; found 302.2279.

# S9. NMR spectra

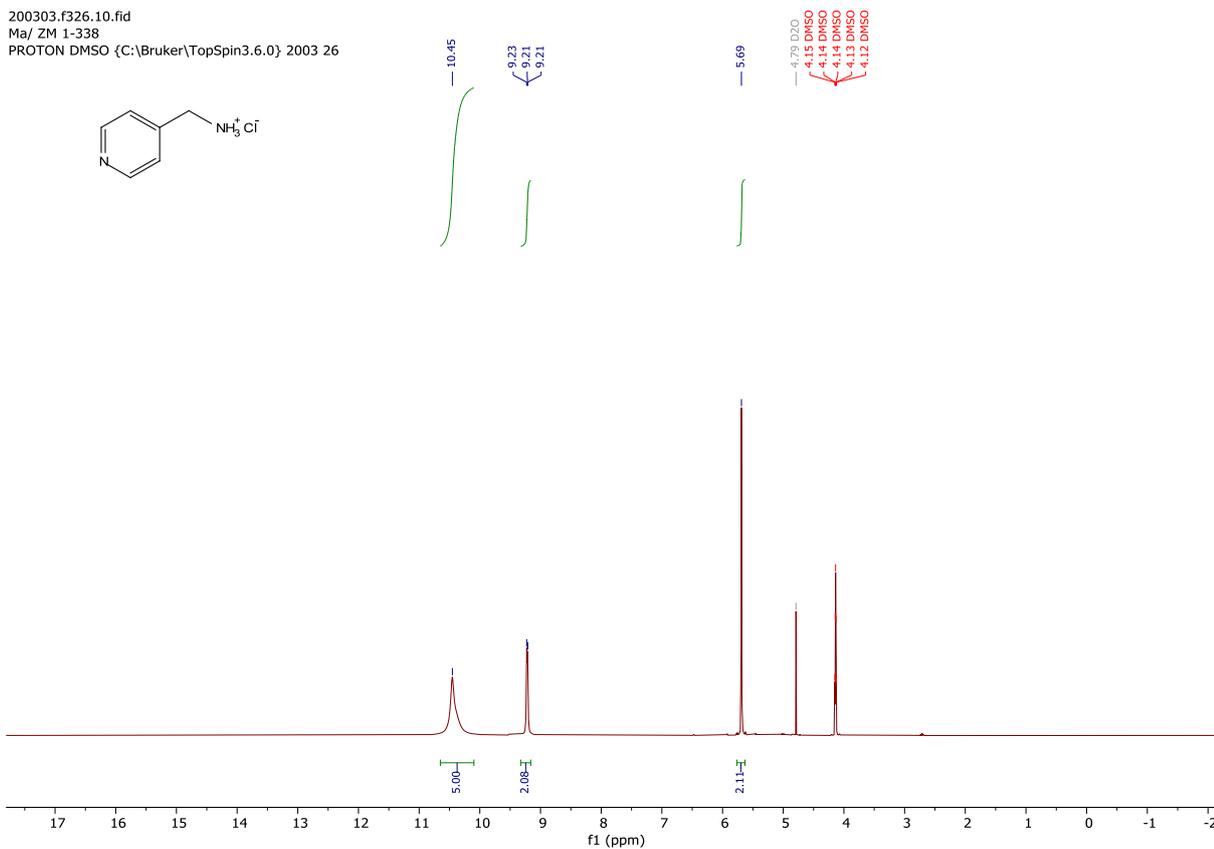
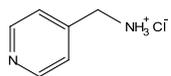
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 Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2107 45



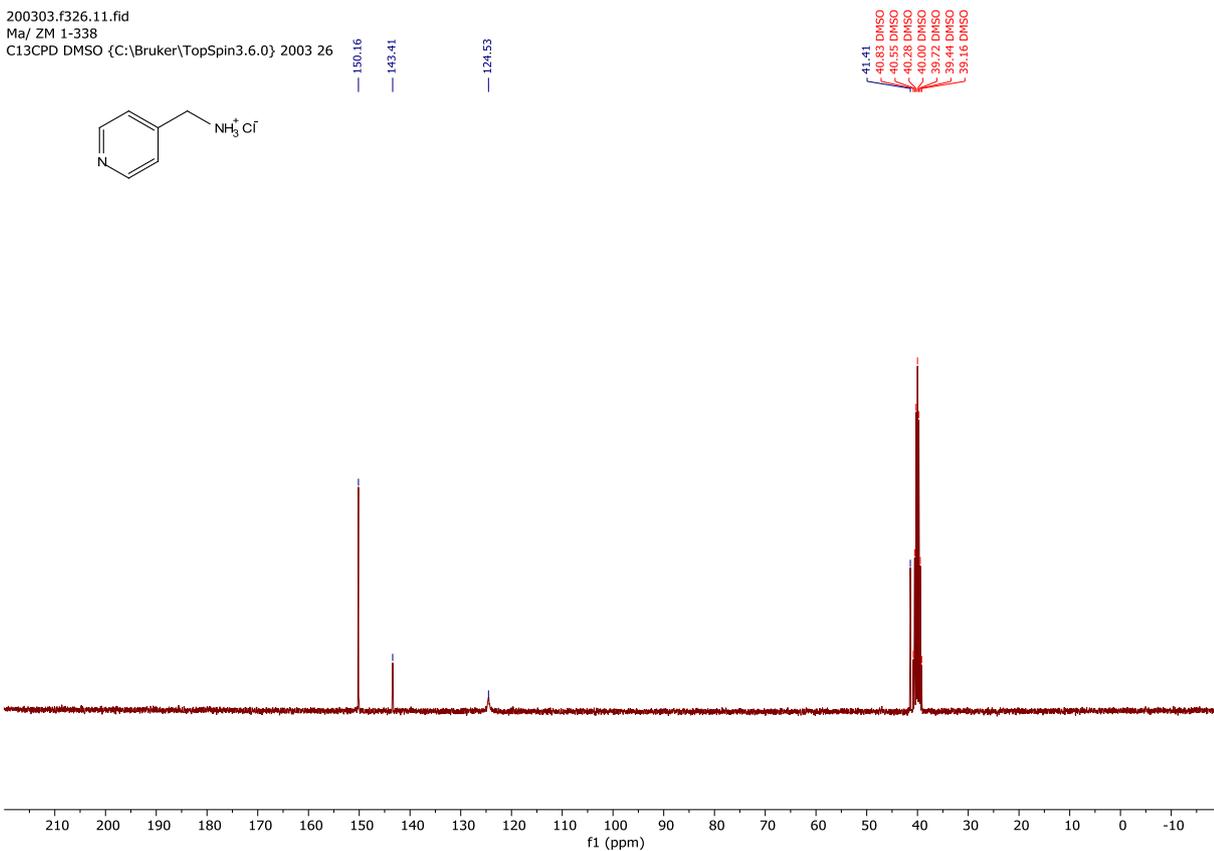
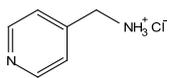
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 Zhuang Ma ZM 144  
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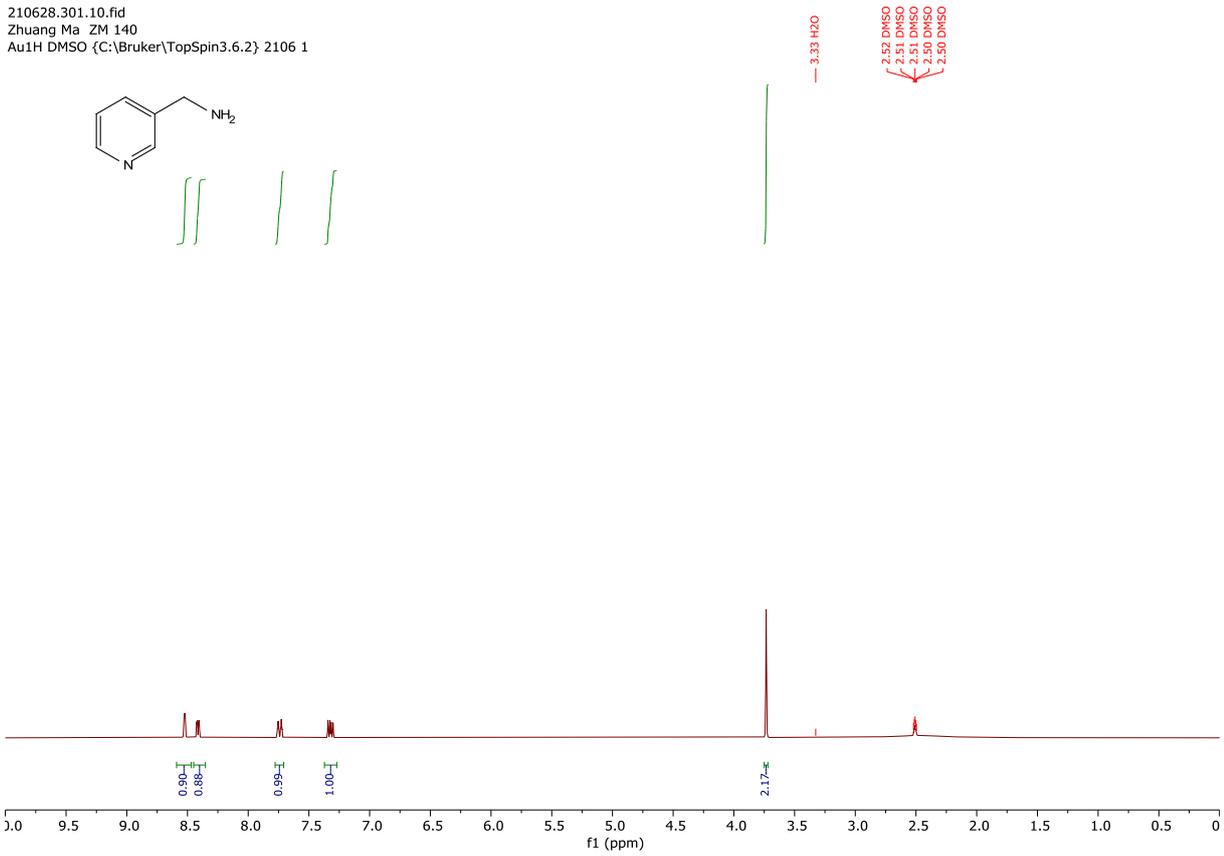
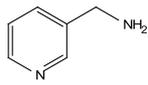
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Ma/ ZM 1-338  
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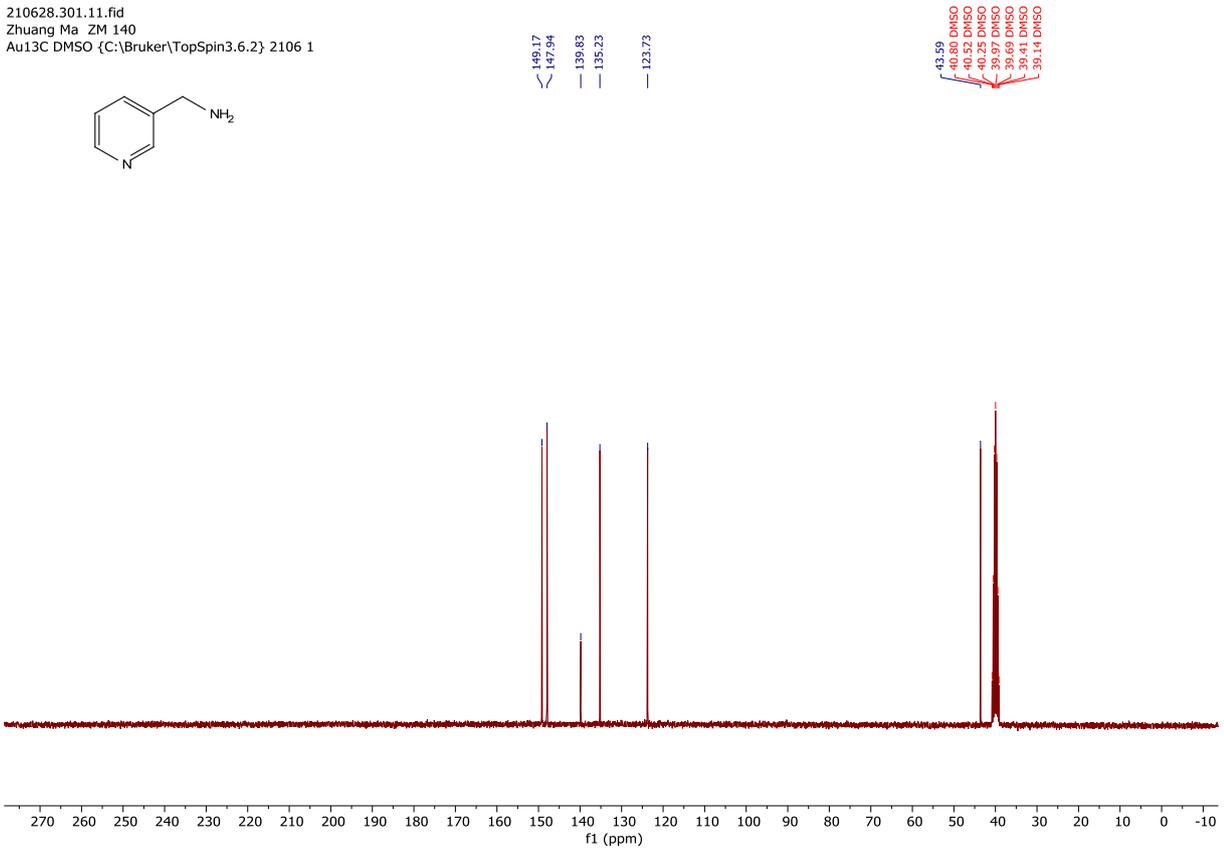
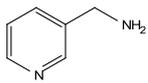
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 26



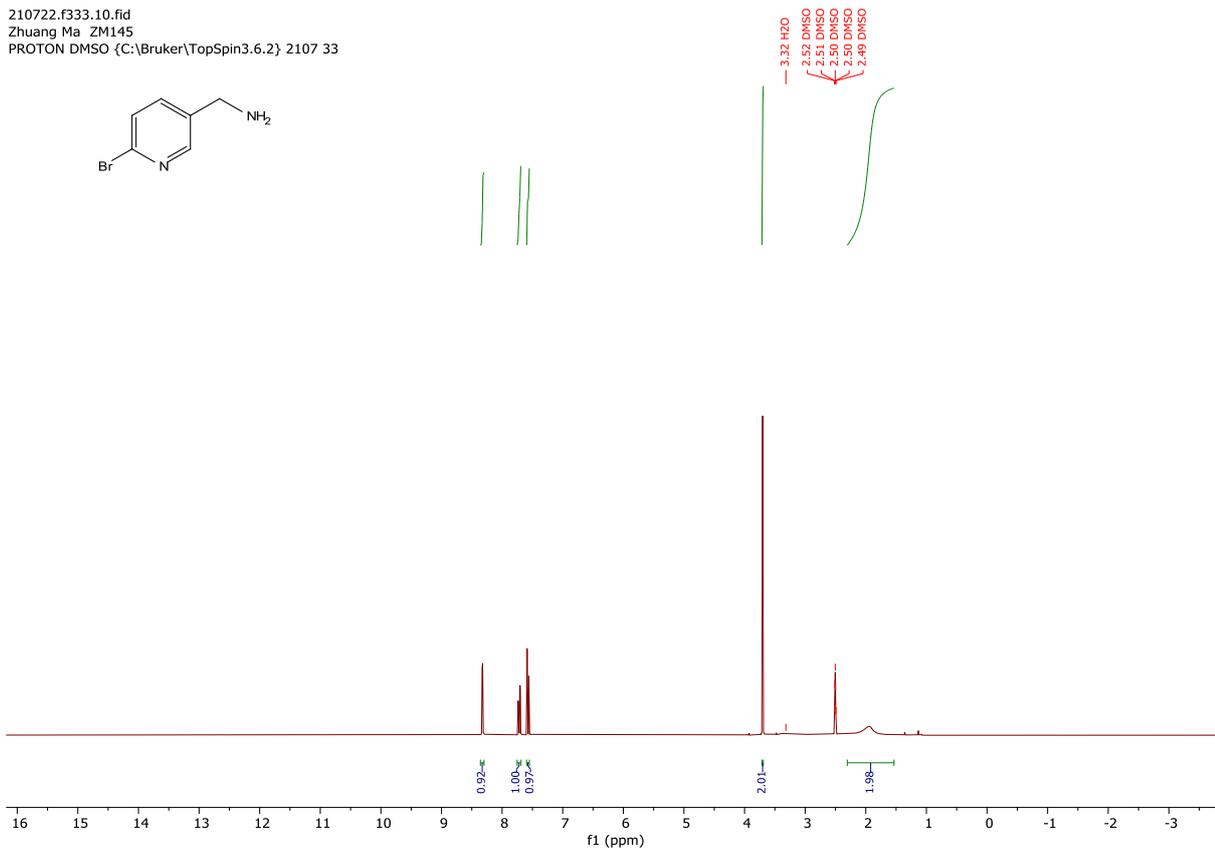
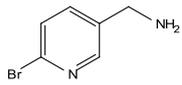
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2106 1



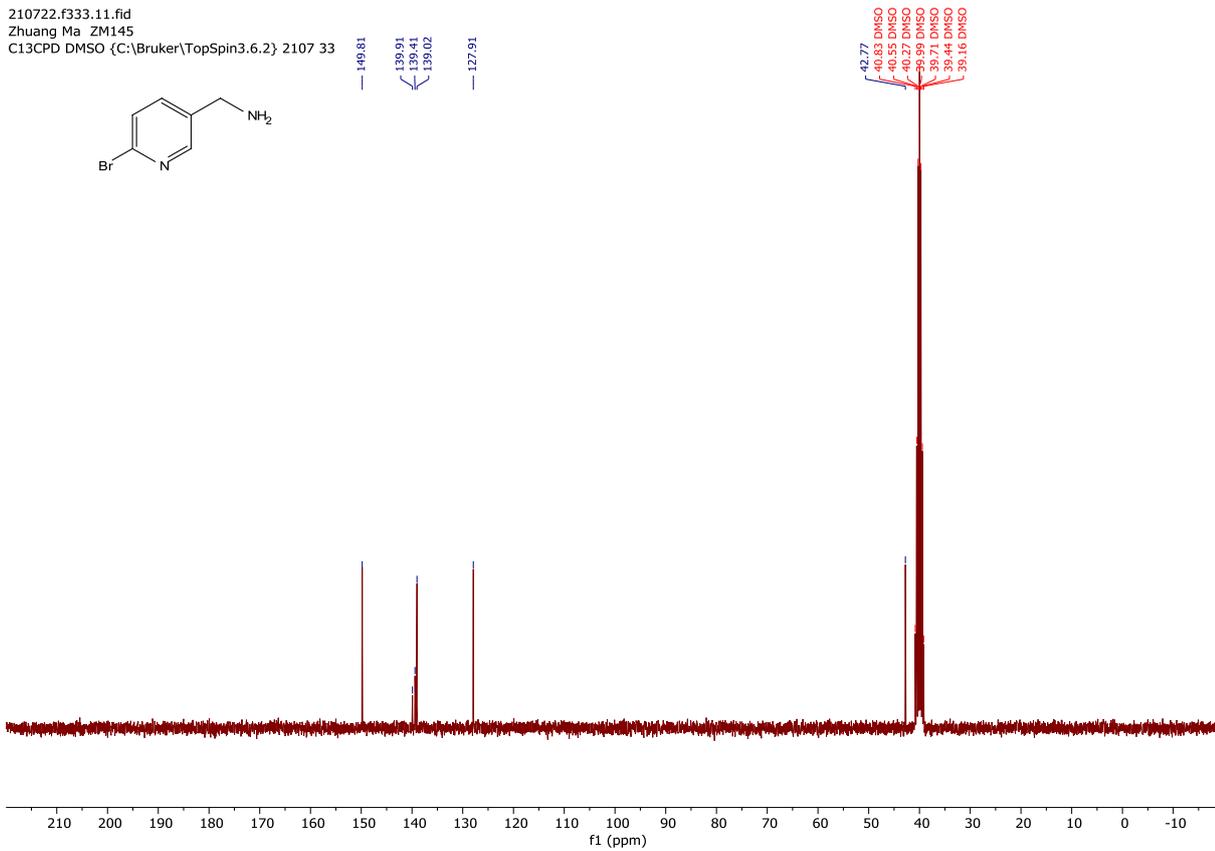
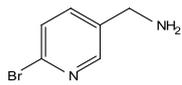
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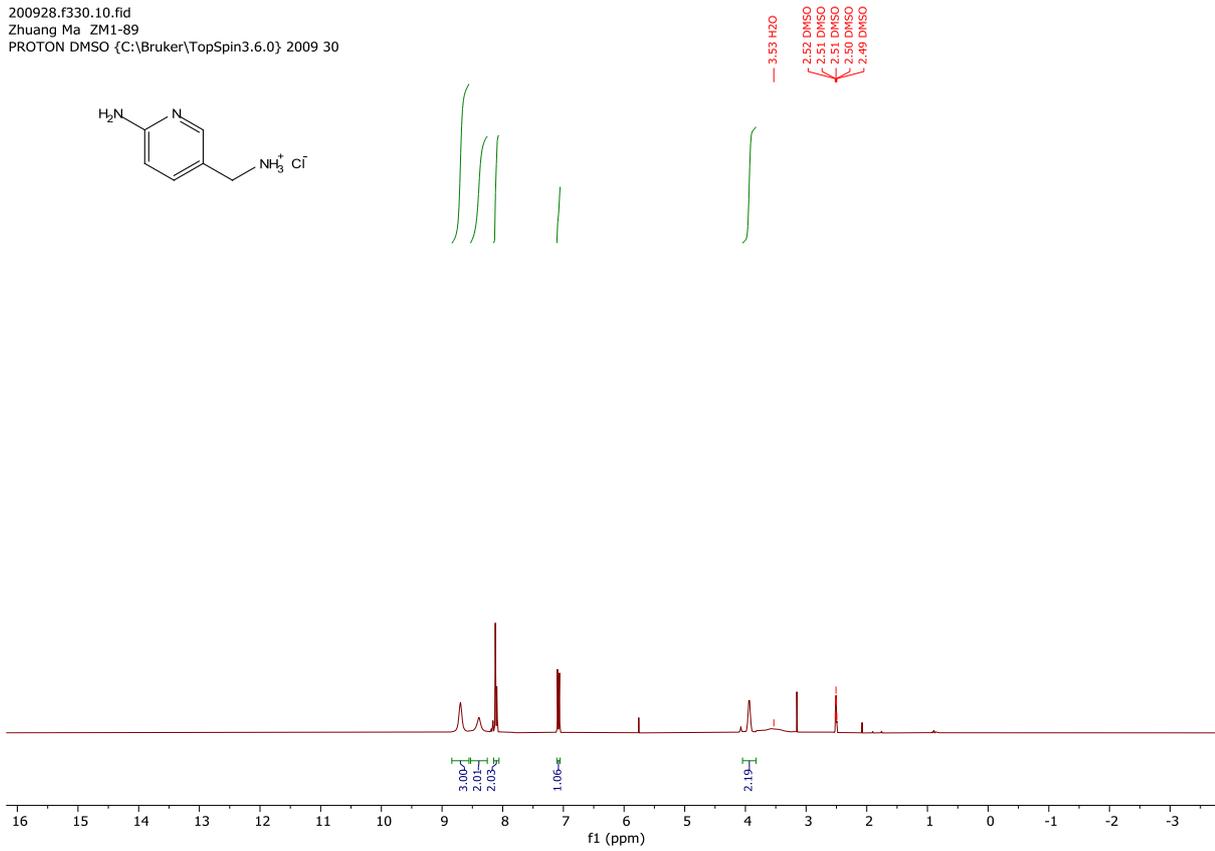
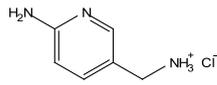
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PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2107 33



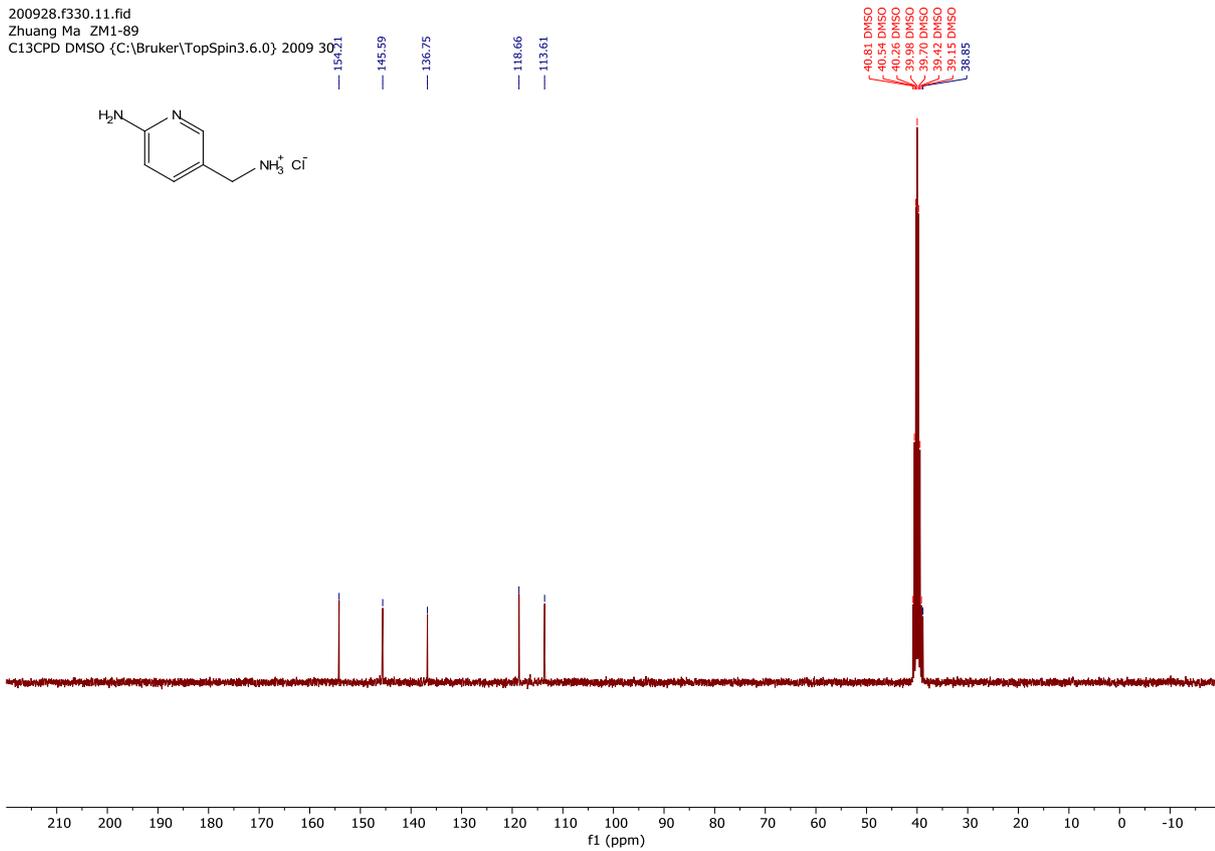
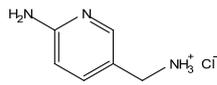
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C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2107 33



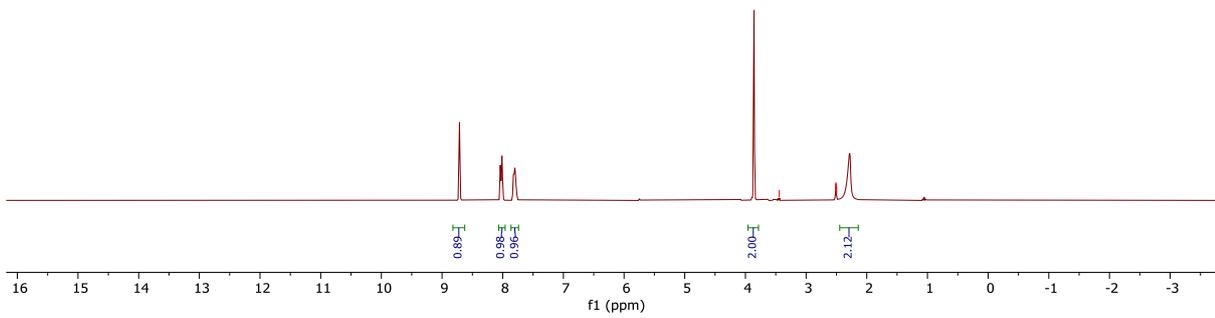
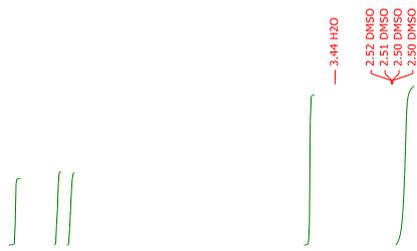
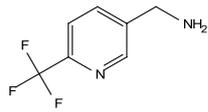
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PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2009 30



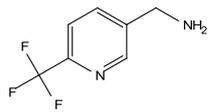
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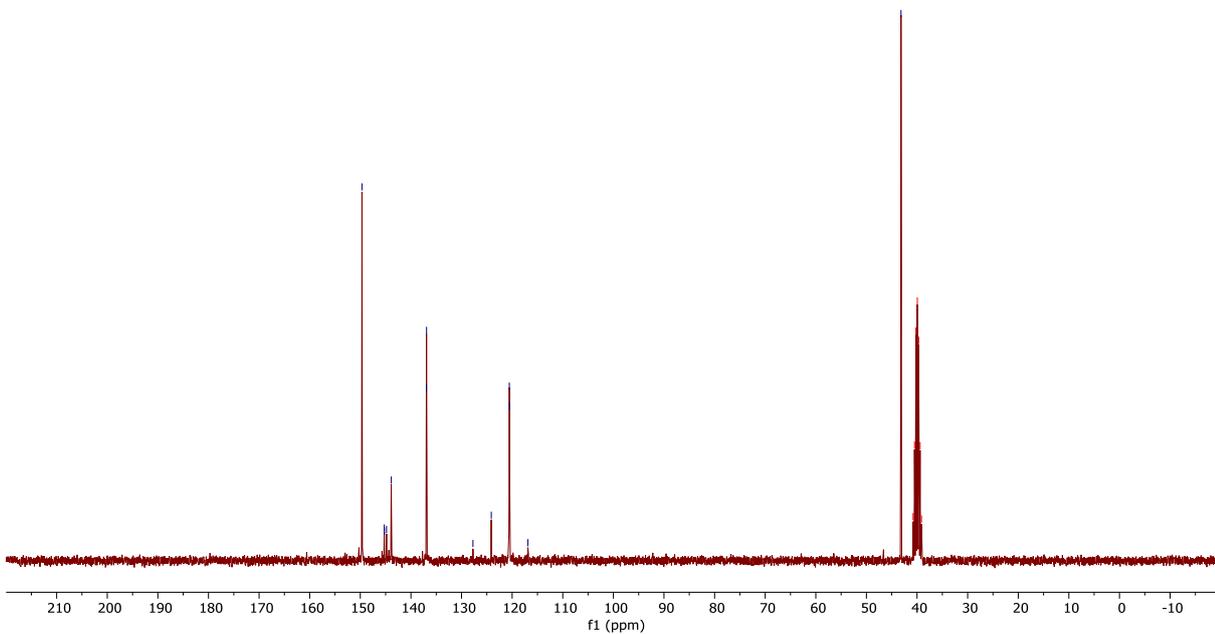
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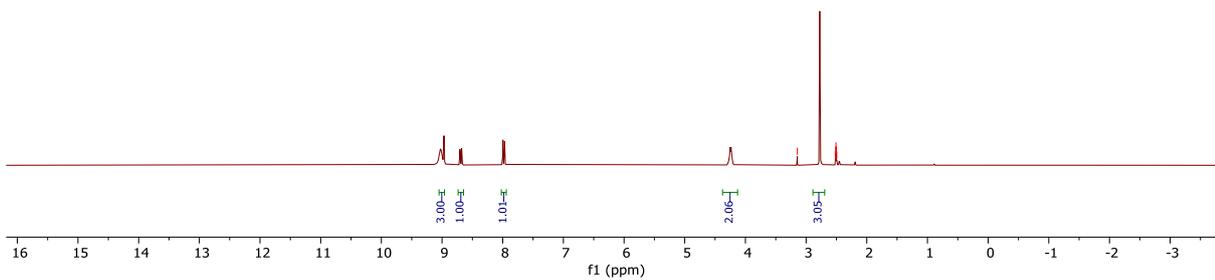
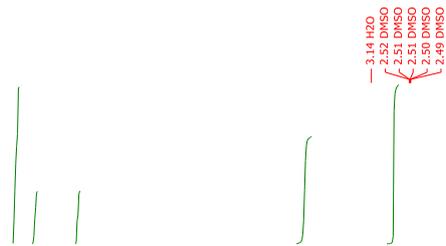
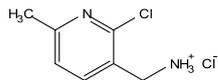
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Ma/ ZM139  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2106 50



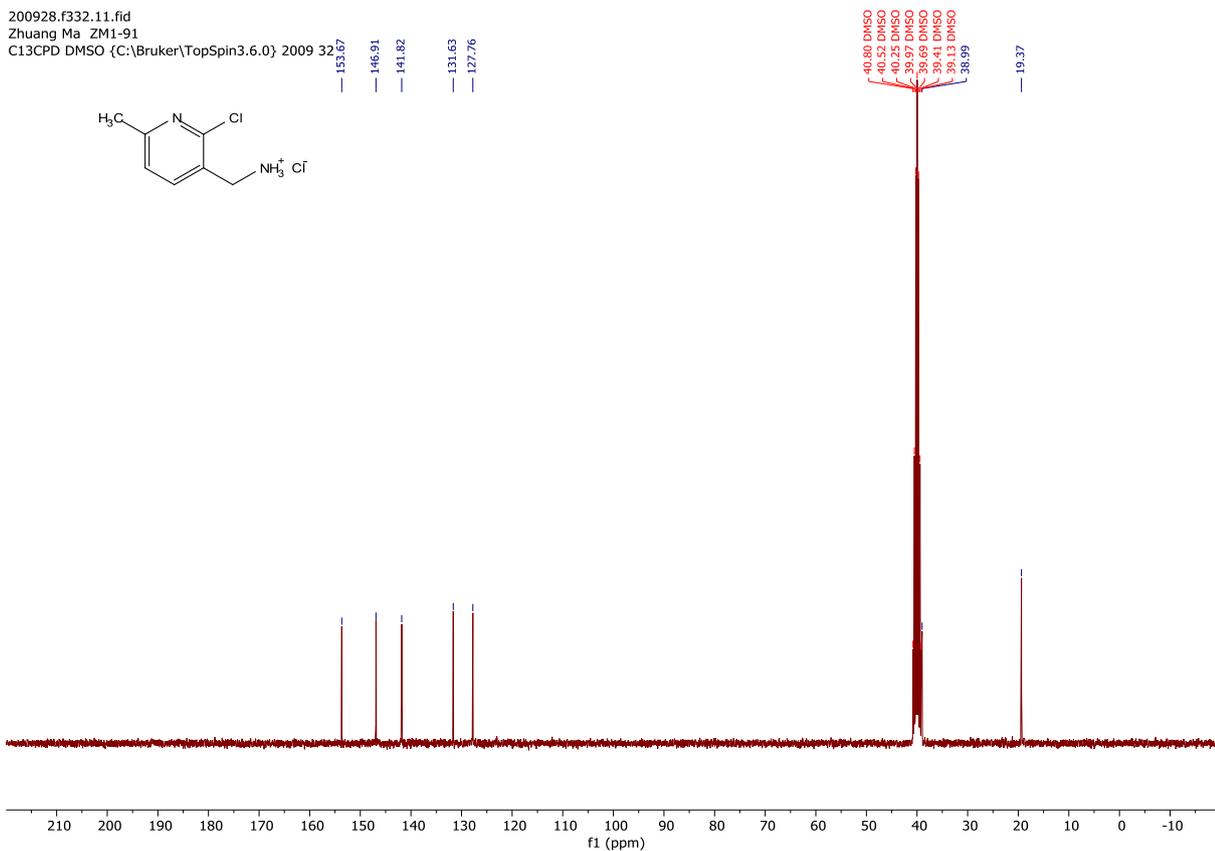
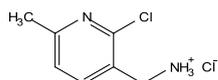
149.67  
145.28  
145.24  
144.82  
143.88  
136.94  
136.91  
127.74  
124.12  
120.56  
120.50  
116.88  
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40.76 DMSO  
40.50 DMSO  
40.27 DMSO  
39.94 DMSO  
39.67 DMSO  
38.39 DMSO  
38.11 DMSO



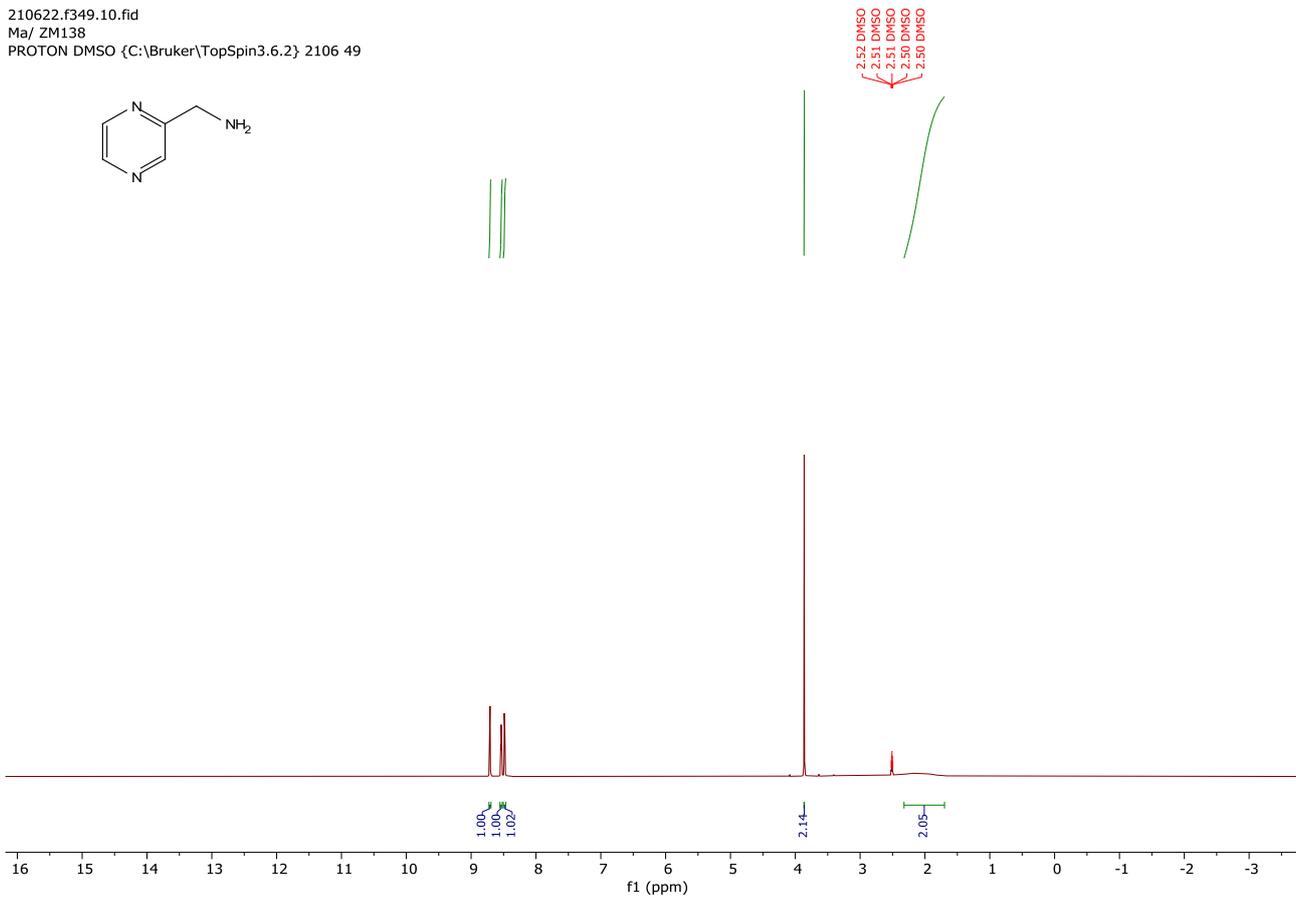
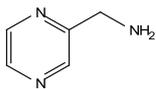
200928.f332.10.fid  
Zhuang Ma ZM1-91  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2009 32



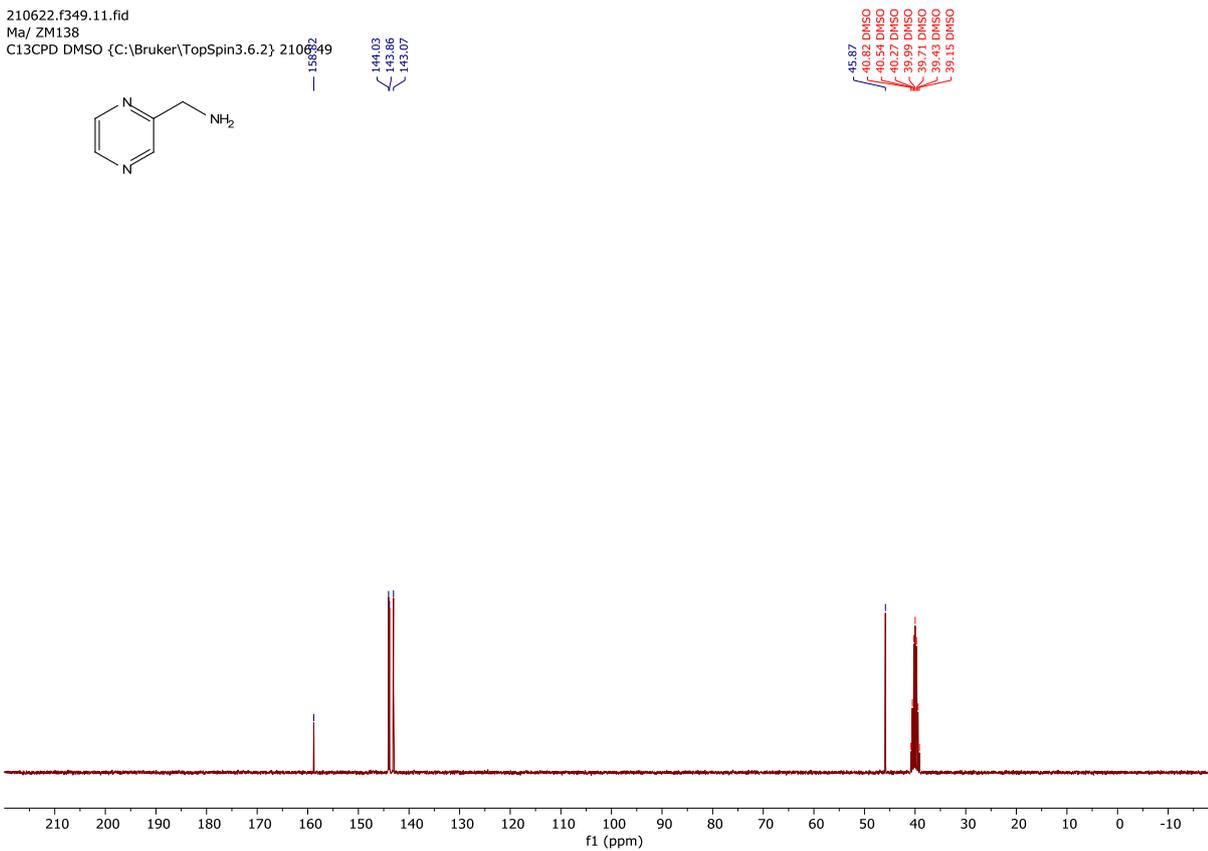
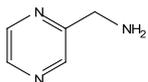
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Zhuang Ma ZM1-91  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2009 32



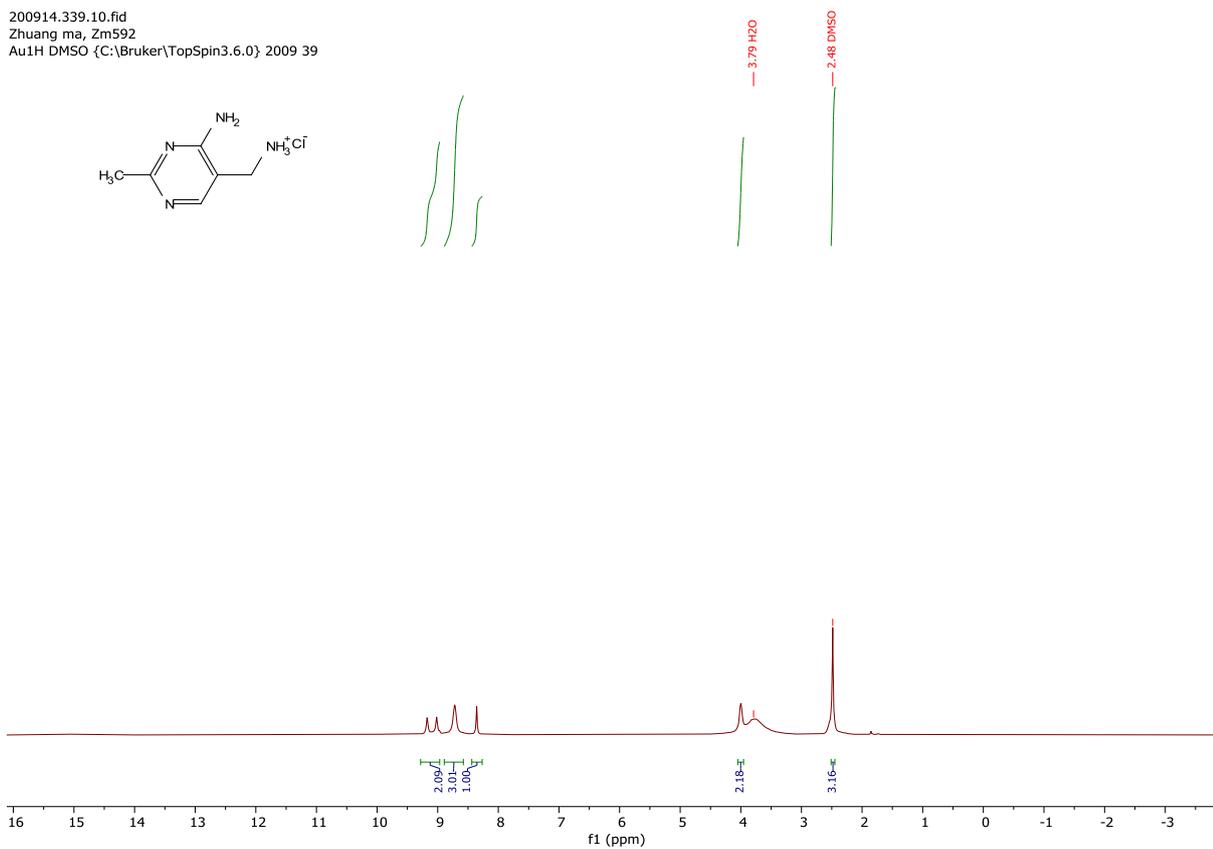
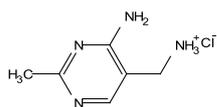
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Ma/ ZM138  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2106 49



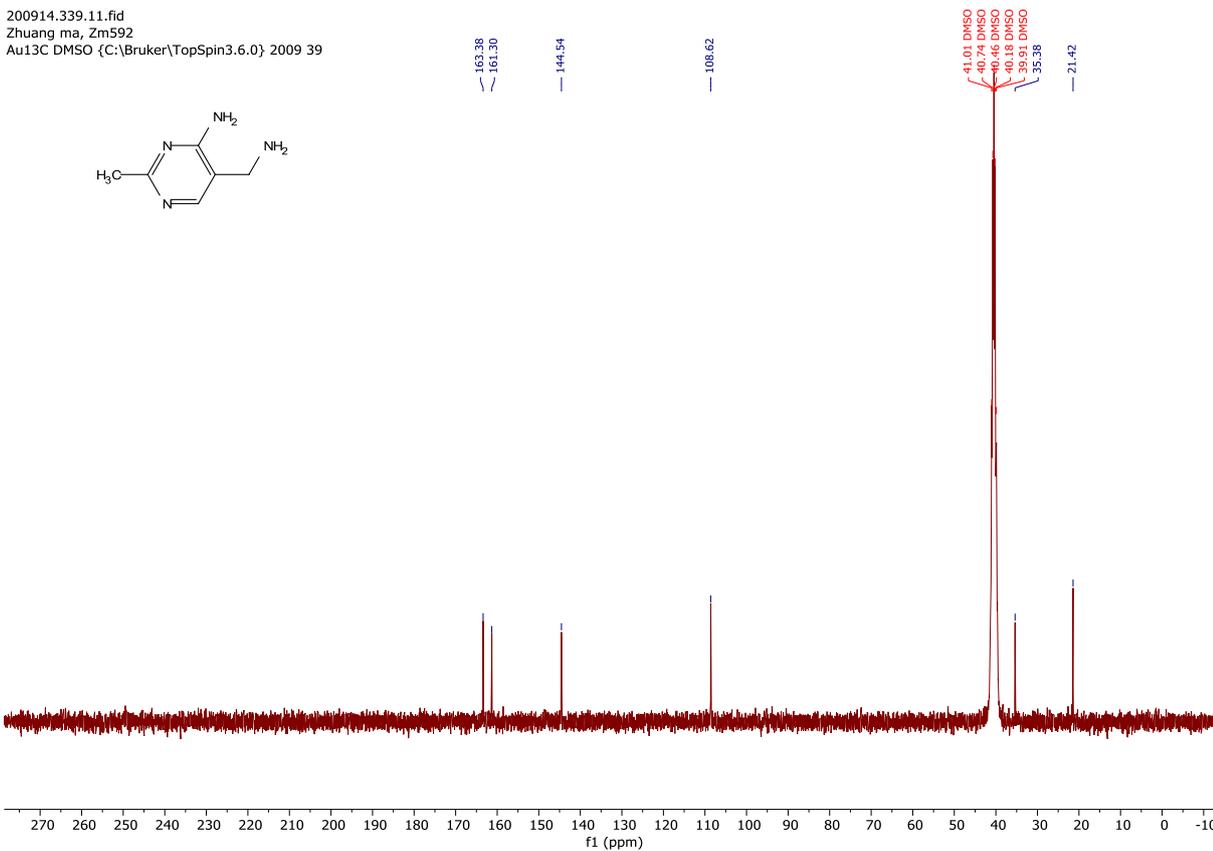
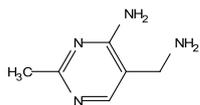
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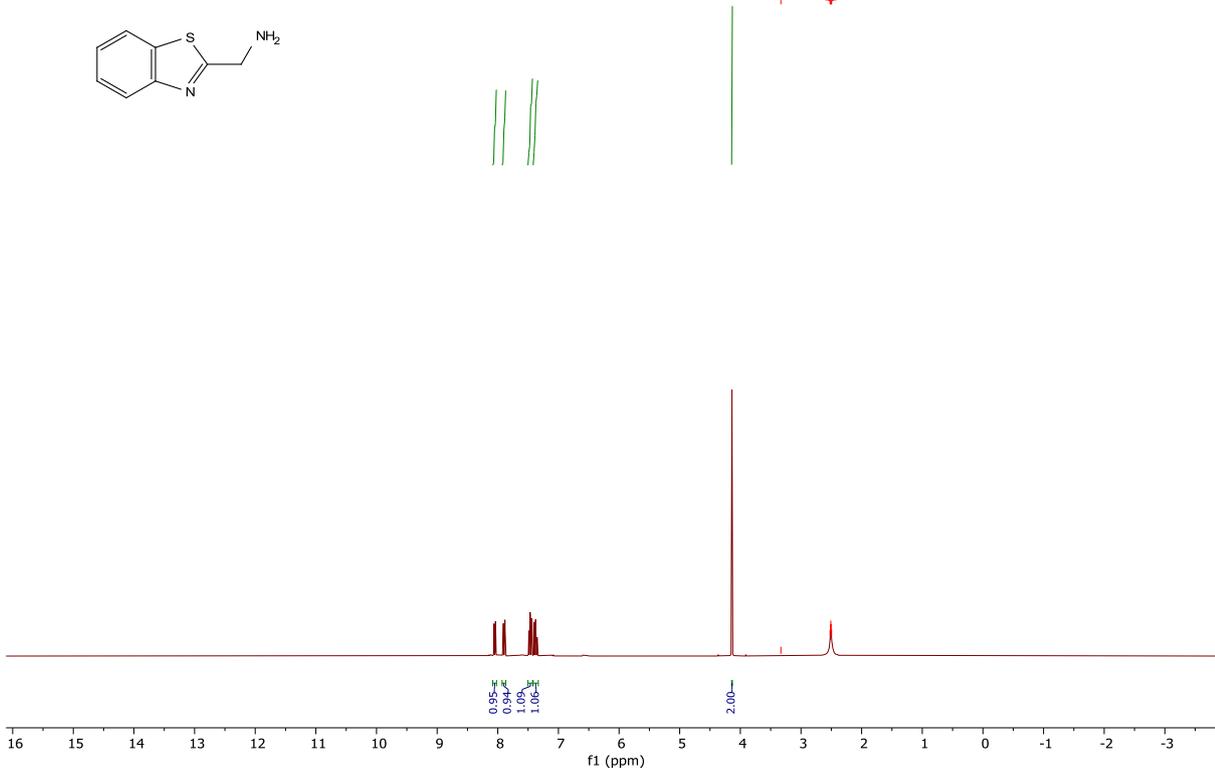
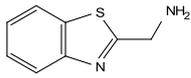
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Zhuang ma, Zm592  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2009 39



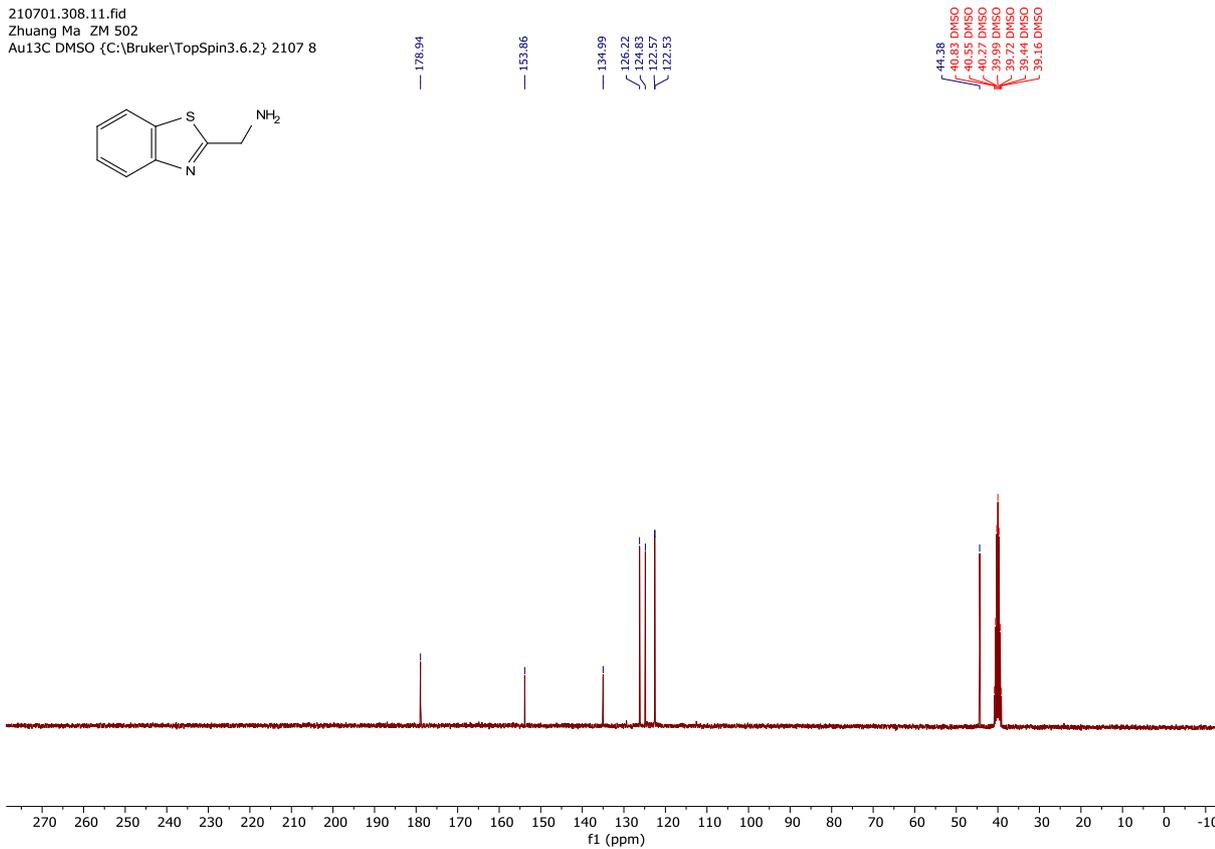
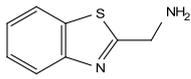
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Zhuang ma, Zm592  
Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2009 39



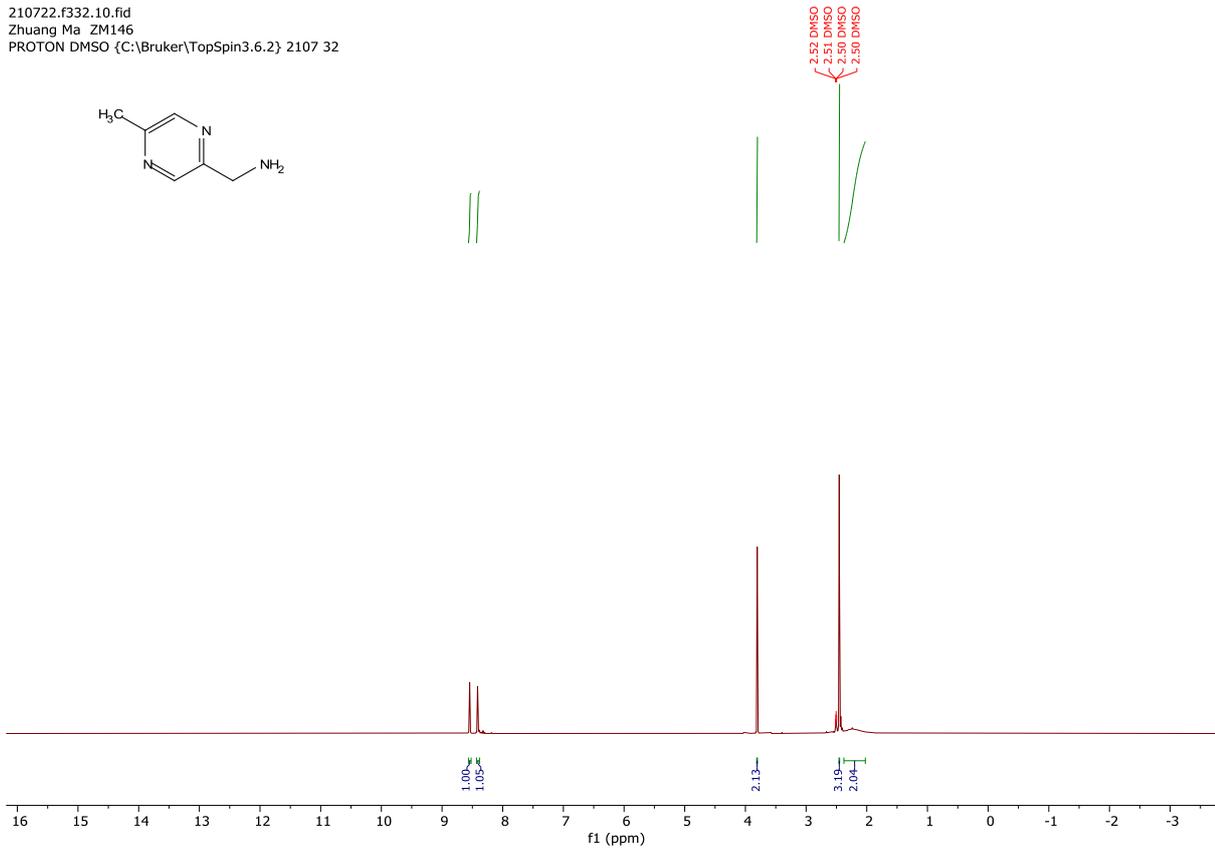
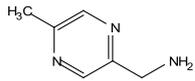
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2107 8



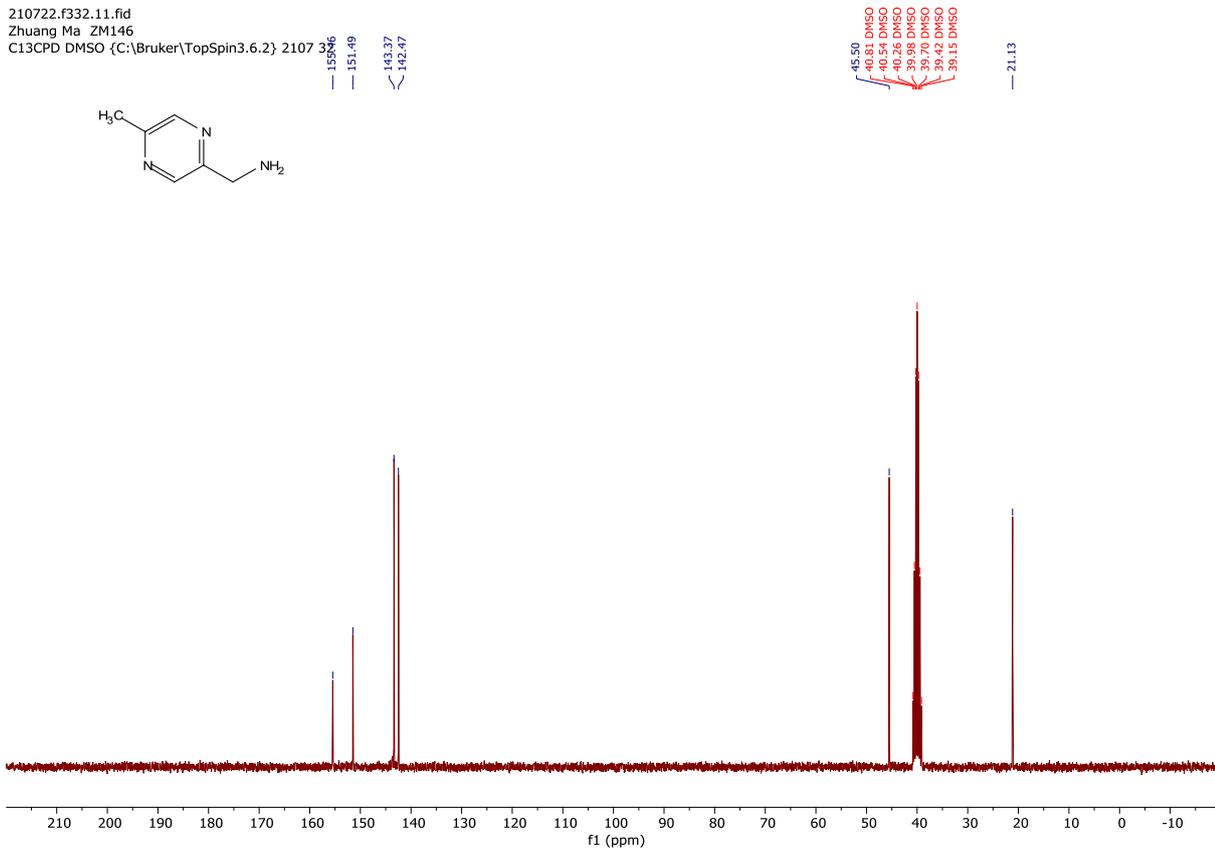
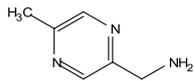
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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2107 8



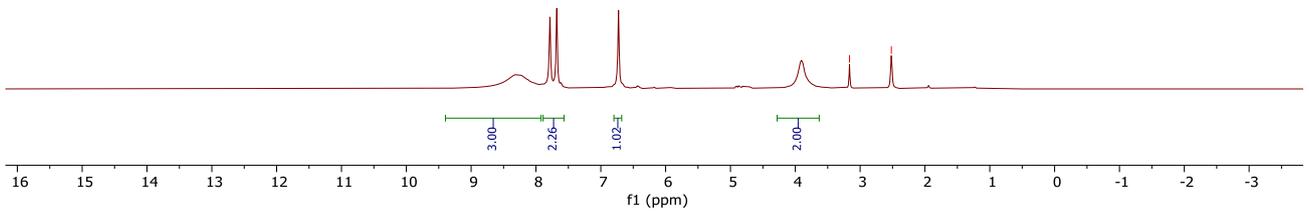
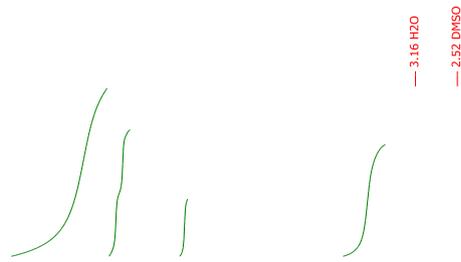
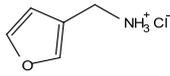
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PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2107 32



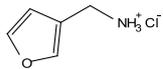
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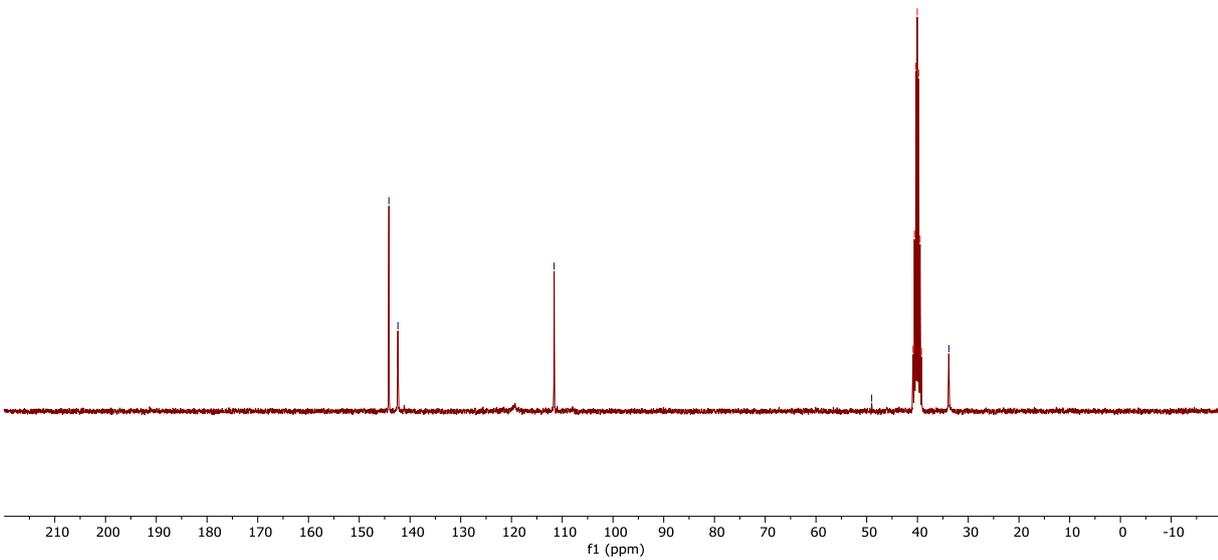
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Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2005 47



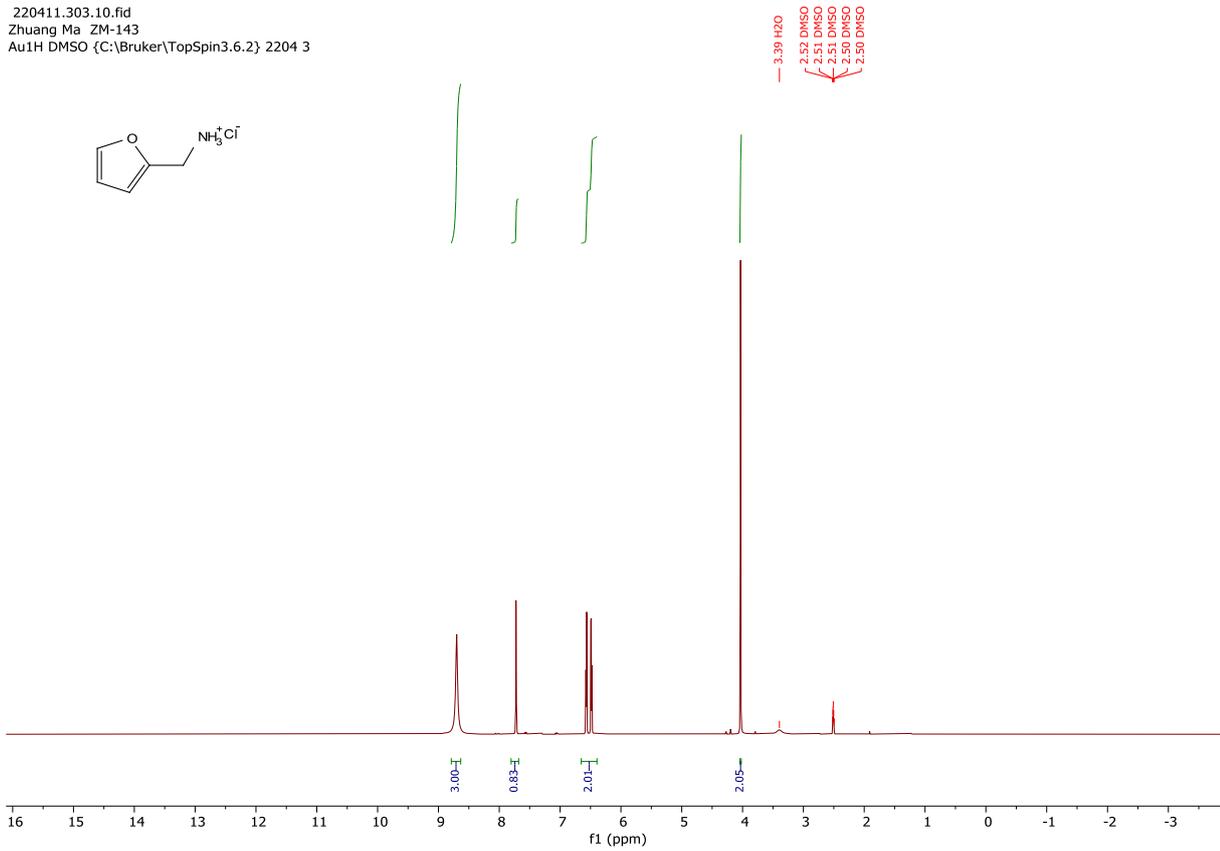
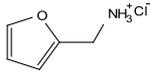
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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2005 47



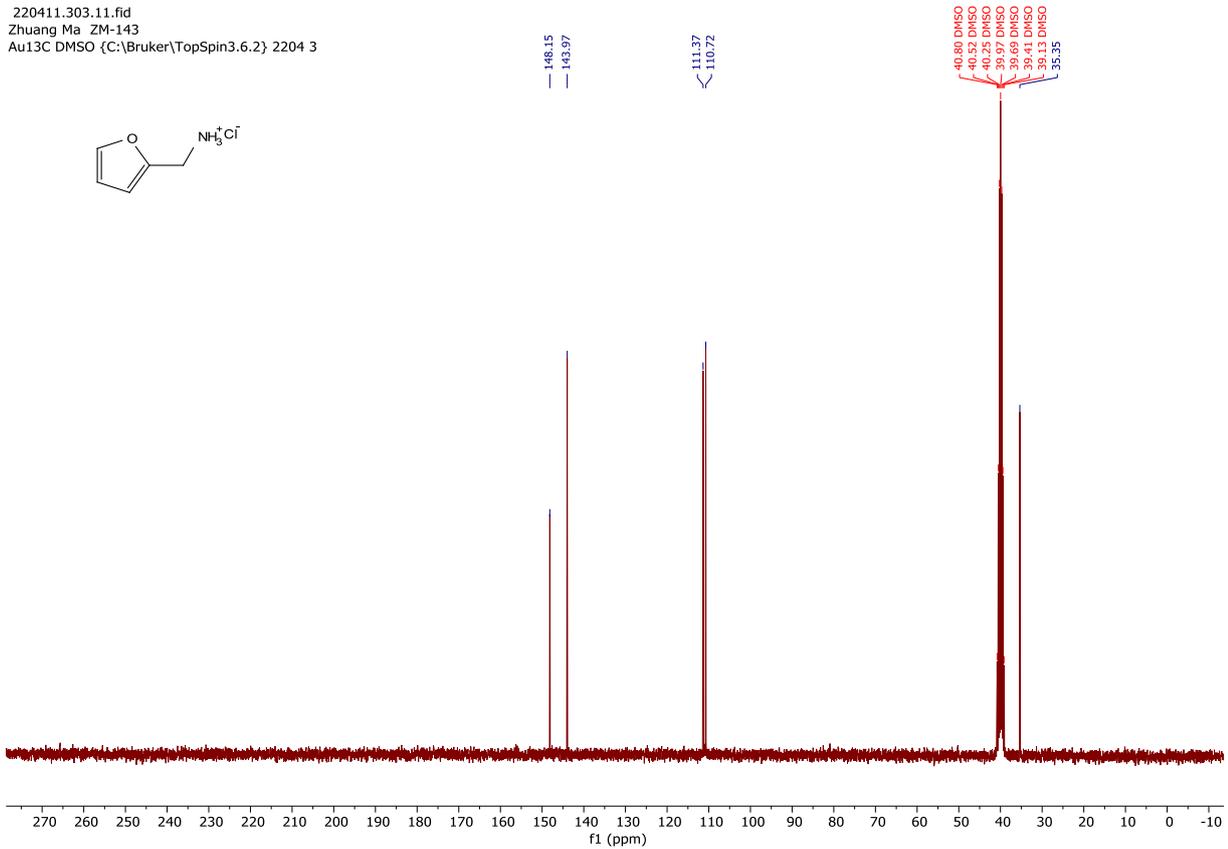
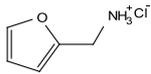
144.16  
142.38  
111.60  
49.02  
40.87 DMSO  
40.60 DMSO  
40.32 DMSO  
40.04 DMSO  
38.76 DMSO  
38.50 DMSO  
38.21 DMSO  
33.83



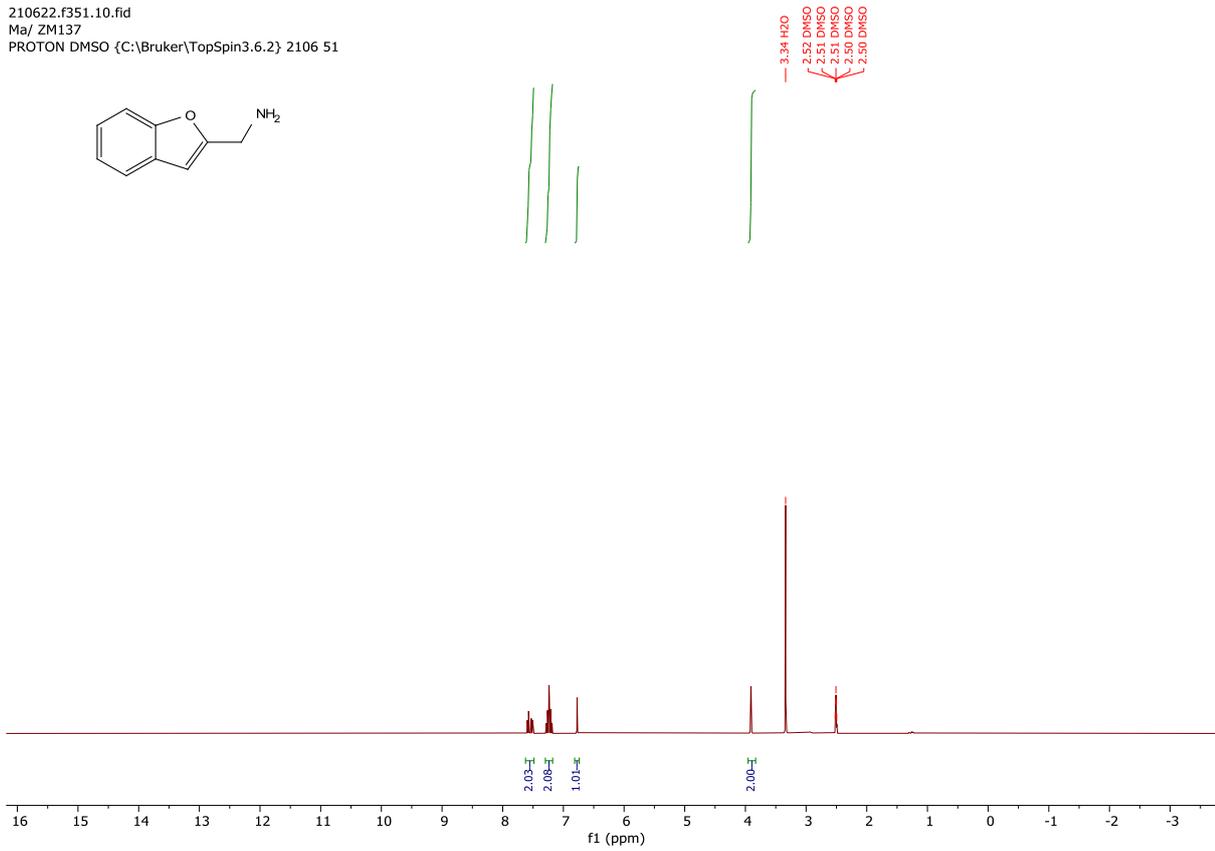
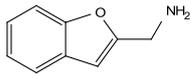
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2204 3



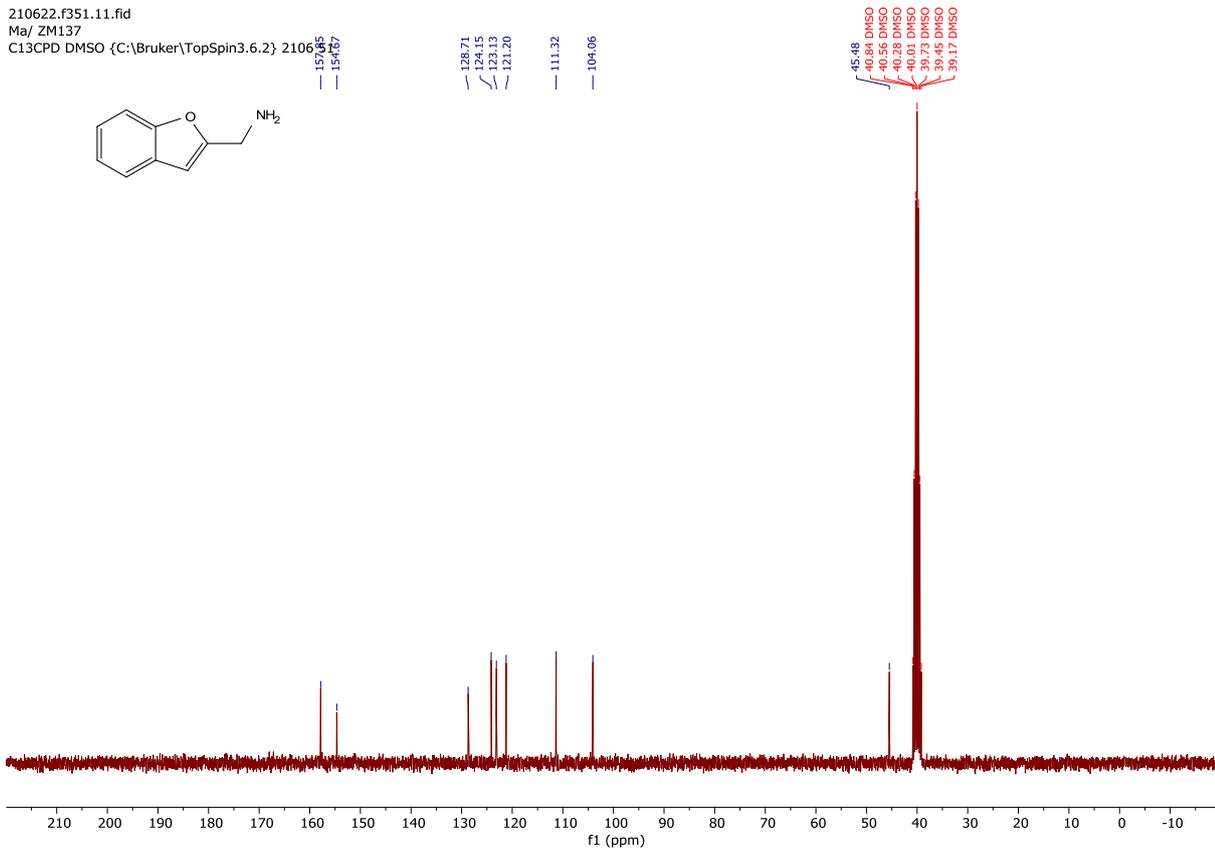
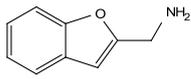
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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2204 3



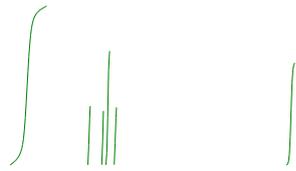
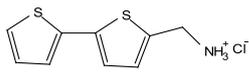
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Ma/ ZM137  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2106 51



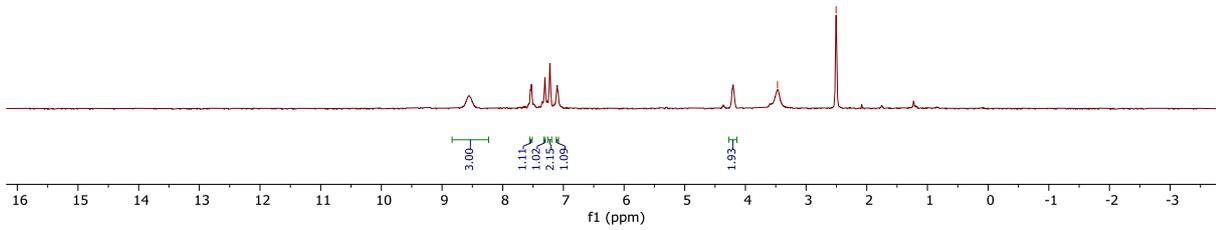
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Ma/ ZM137  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2106



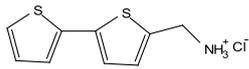
200316.f337.10.fid  
Ma/ ZM 1-524  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 37



3.47 H<sub>2</sub>O  
2.50 DMSO

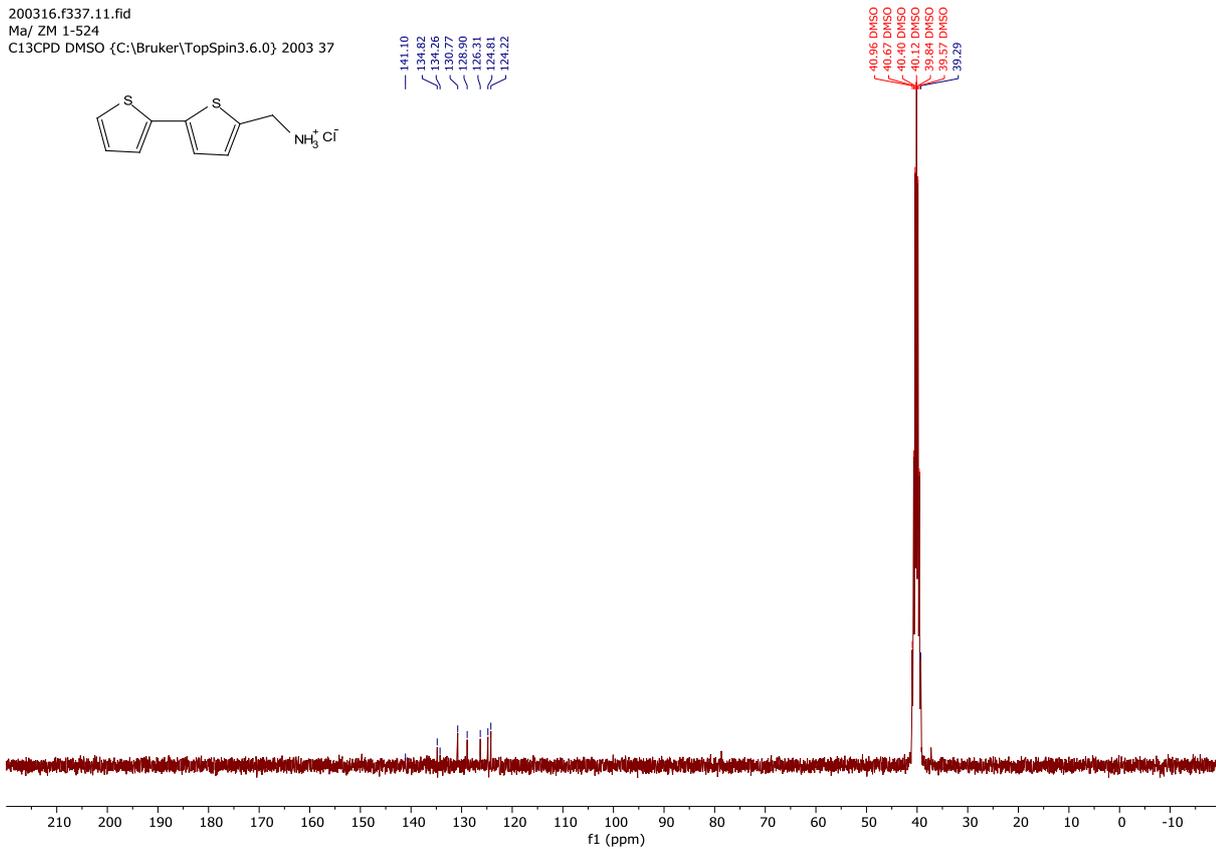


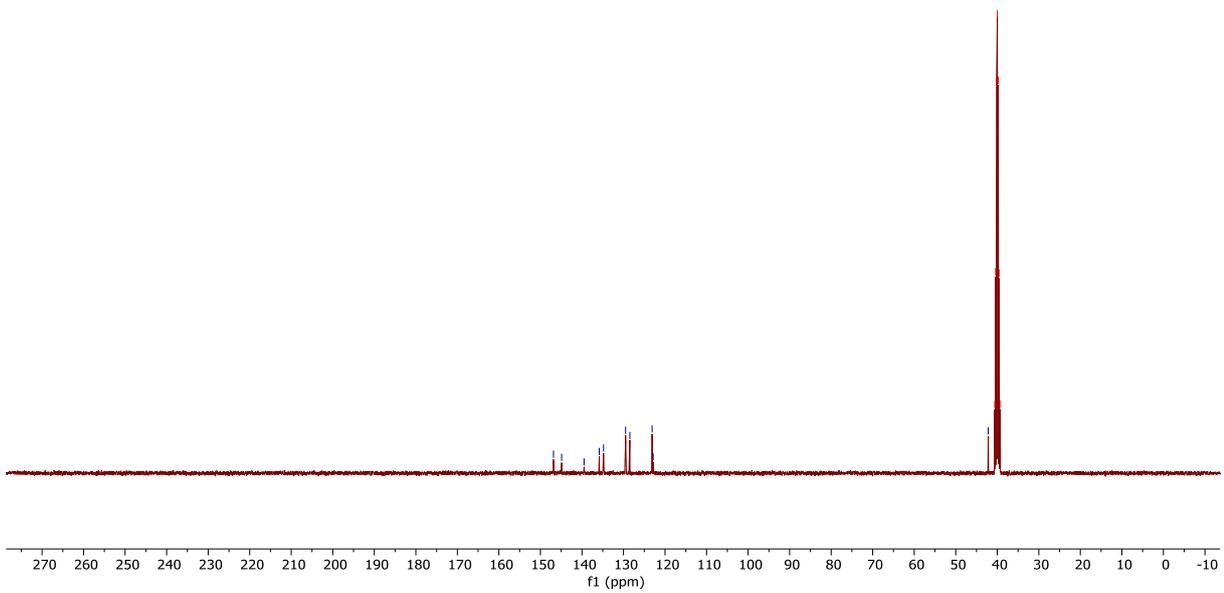
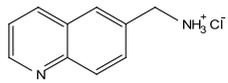
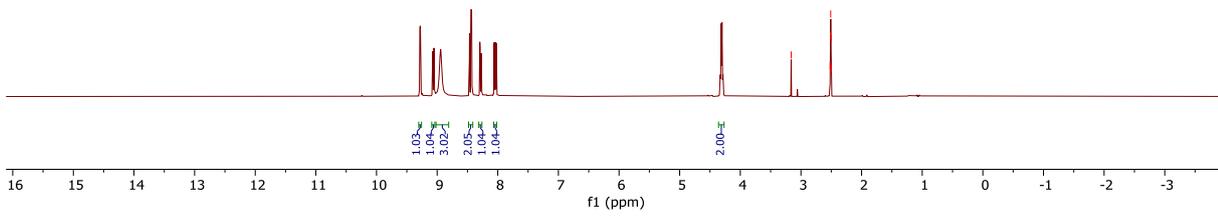
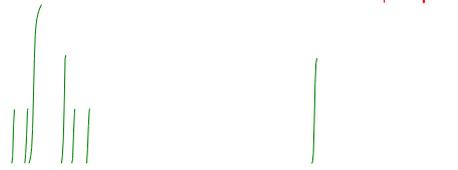
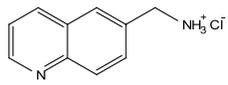
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Ma/ ZM 1-524  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 37



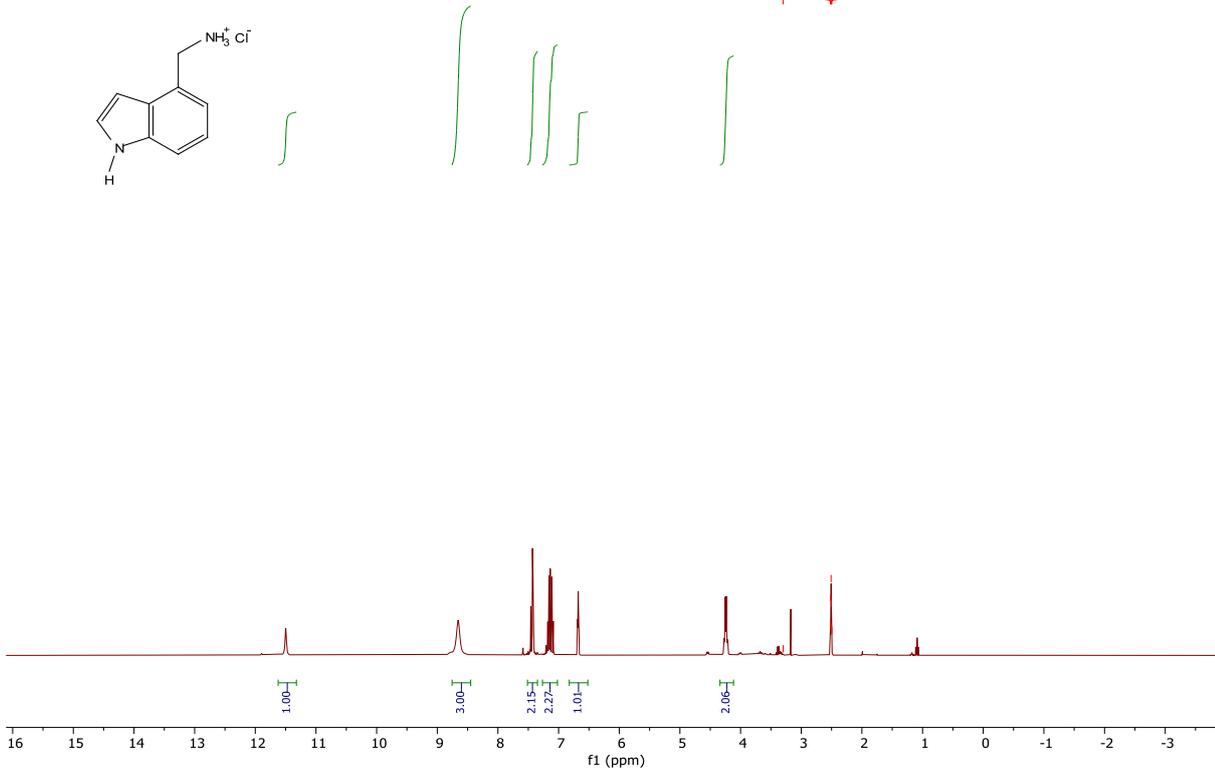
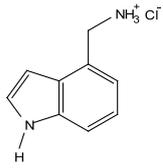
141.10  
134.82  
134.26  
130.77  
128.90  
124.81  
124.22

40.96 DMSO  
40.67 DMSO  
40.40 DMSO  
40.12 DMSO  
39.84 DMSO  
39.57 DMSO  
39.29

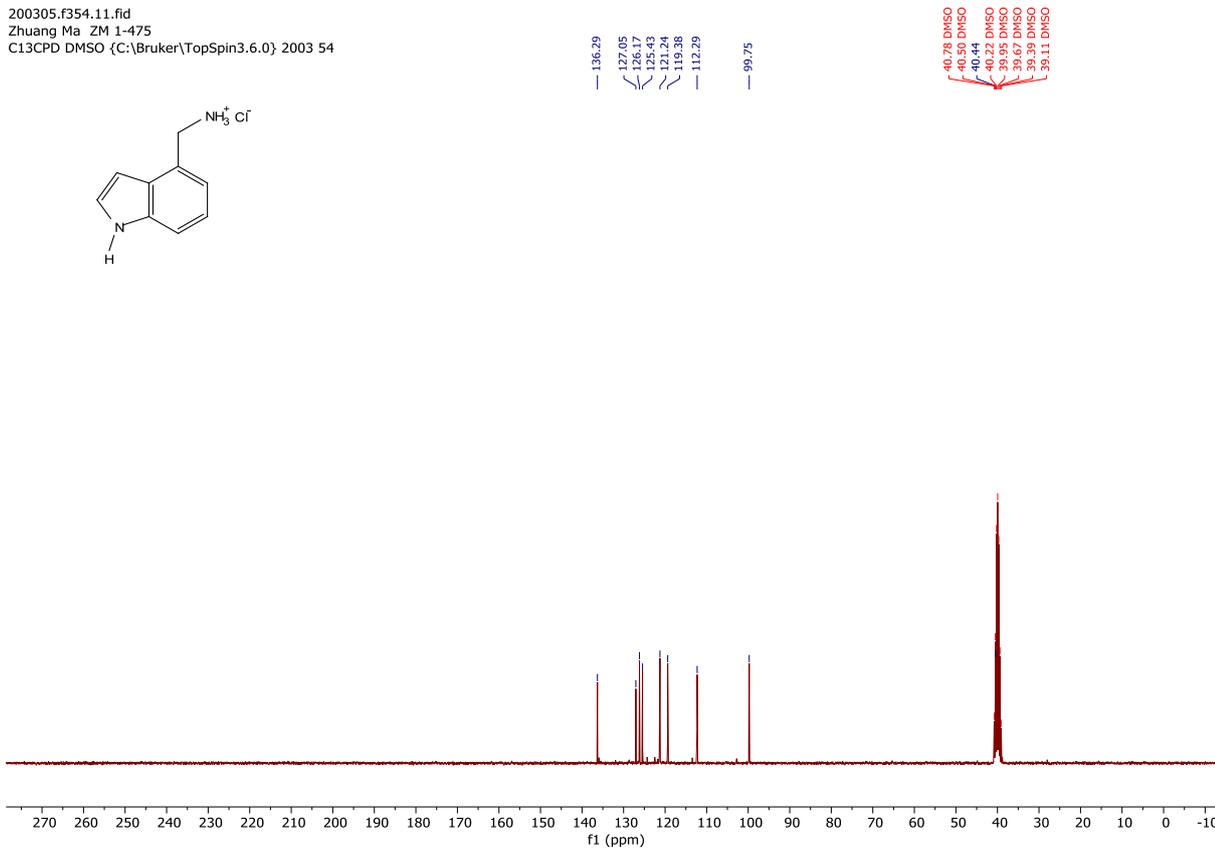
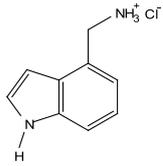




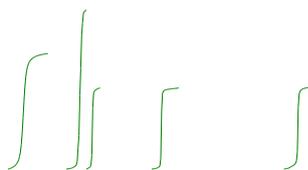
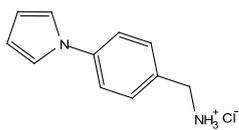
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Zhuang Ma\_ZM 1-475  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 54



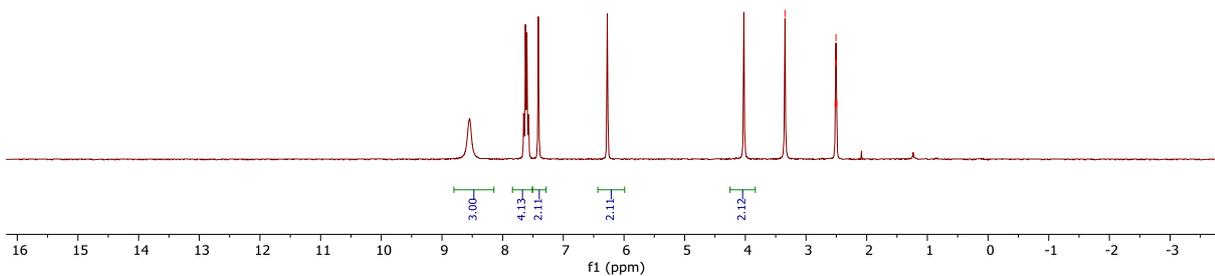
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Zhuang Ma\_ZM 1-475  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 54



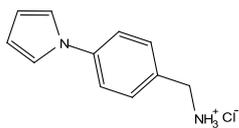
200303.f332.10.fid  
Ma/ ZM 1-509  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 32



3.35 H2O  
2.52 DMSO  
2.51 DMSO  
2.51 DMSO  
2.50 DMSO  
2.48 DMSO

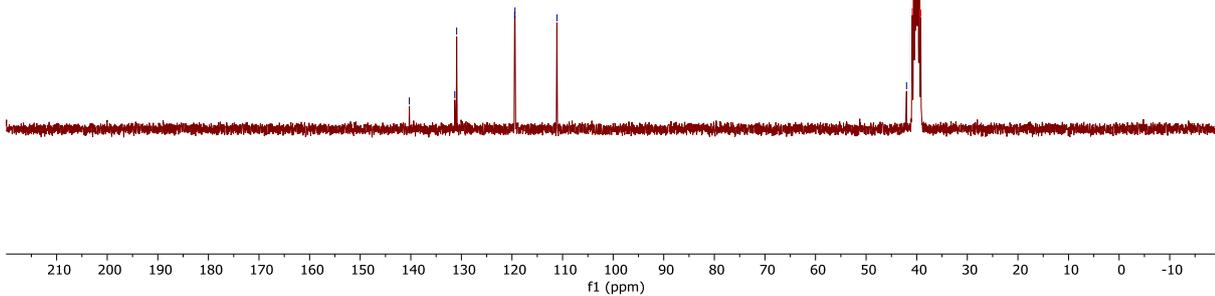


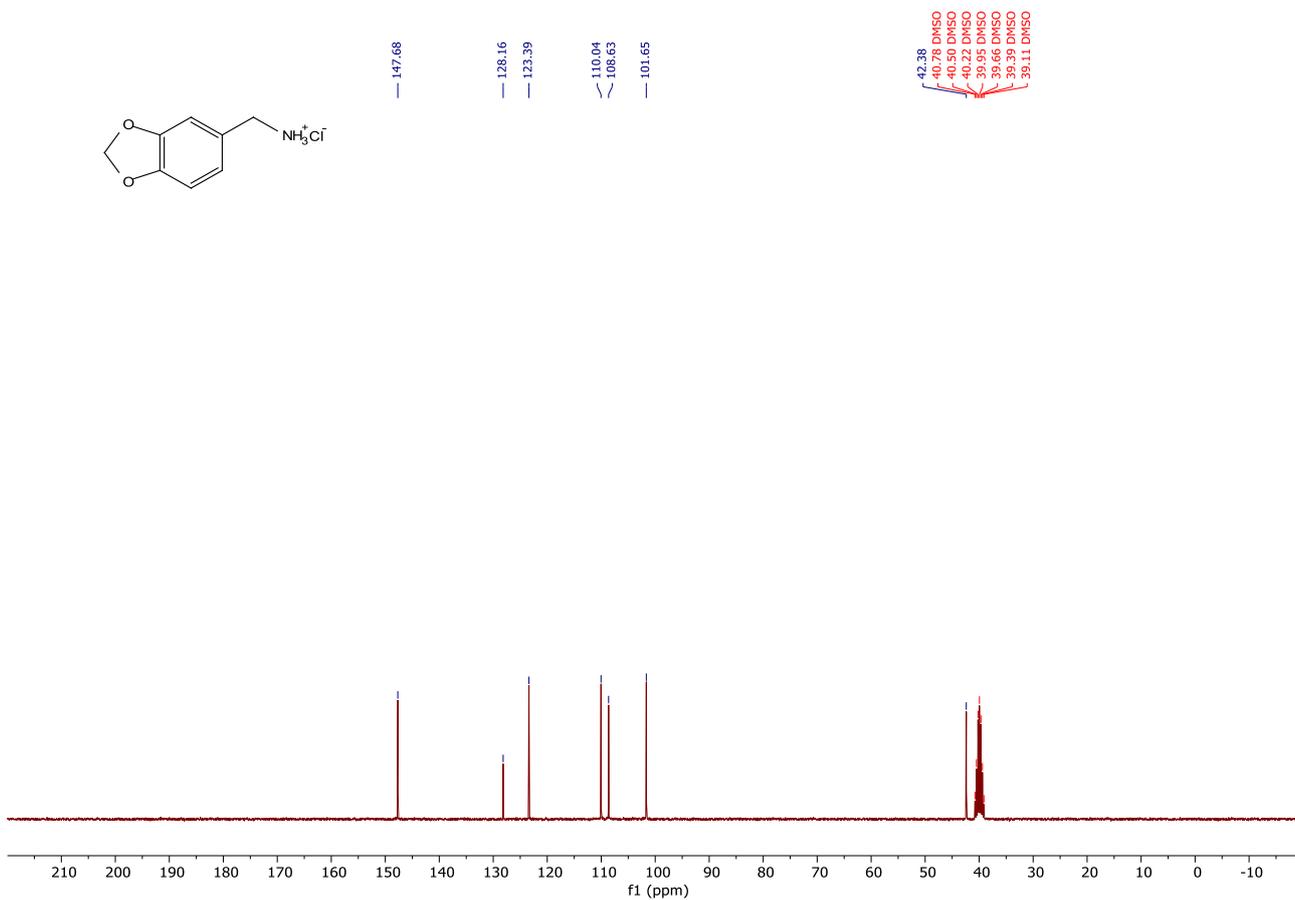
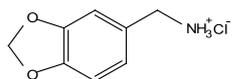
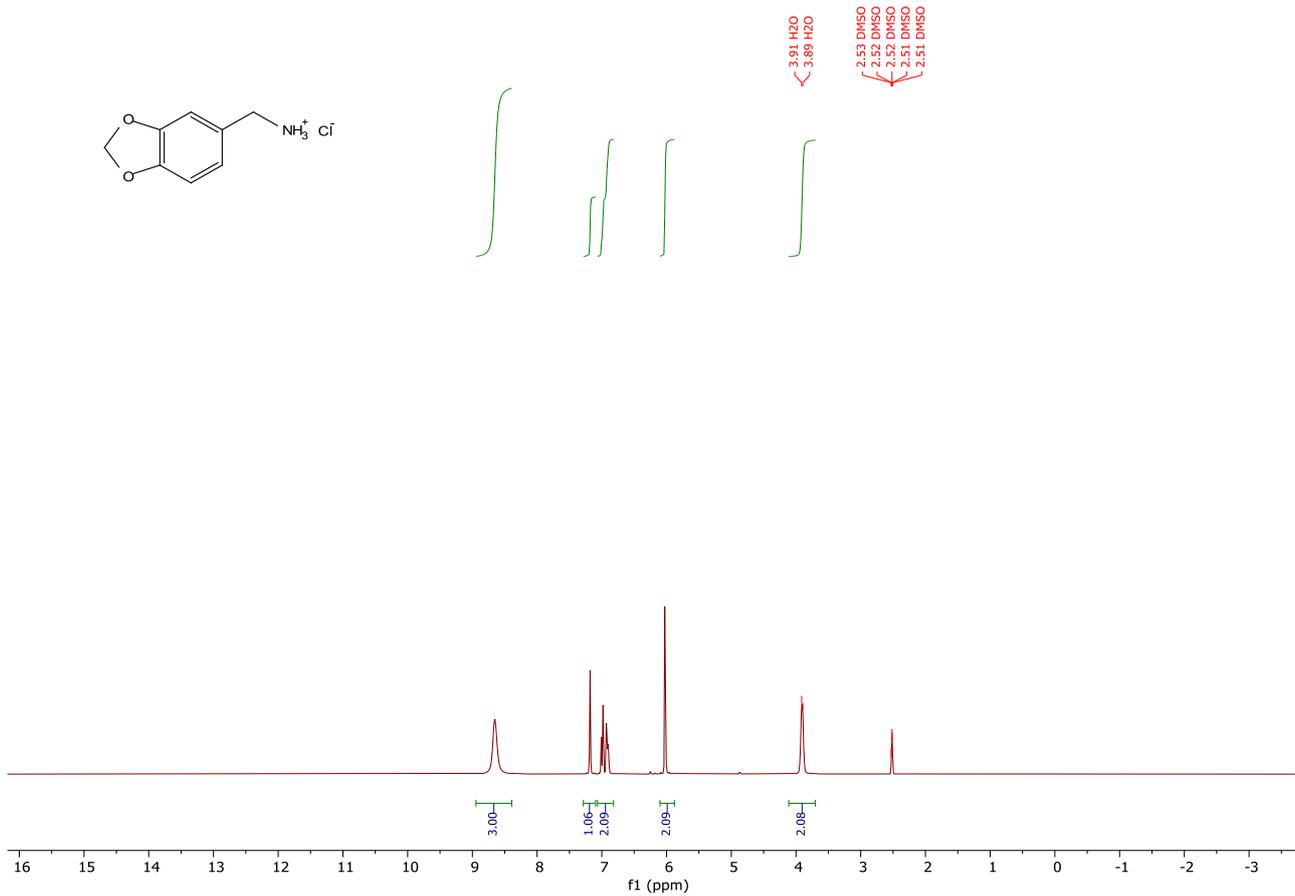
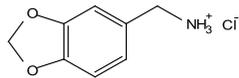
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Ma/ ZM 1-509  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 32



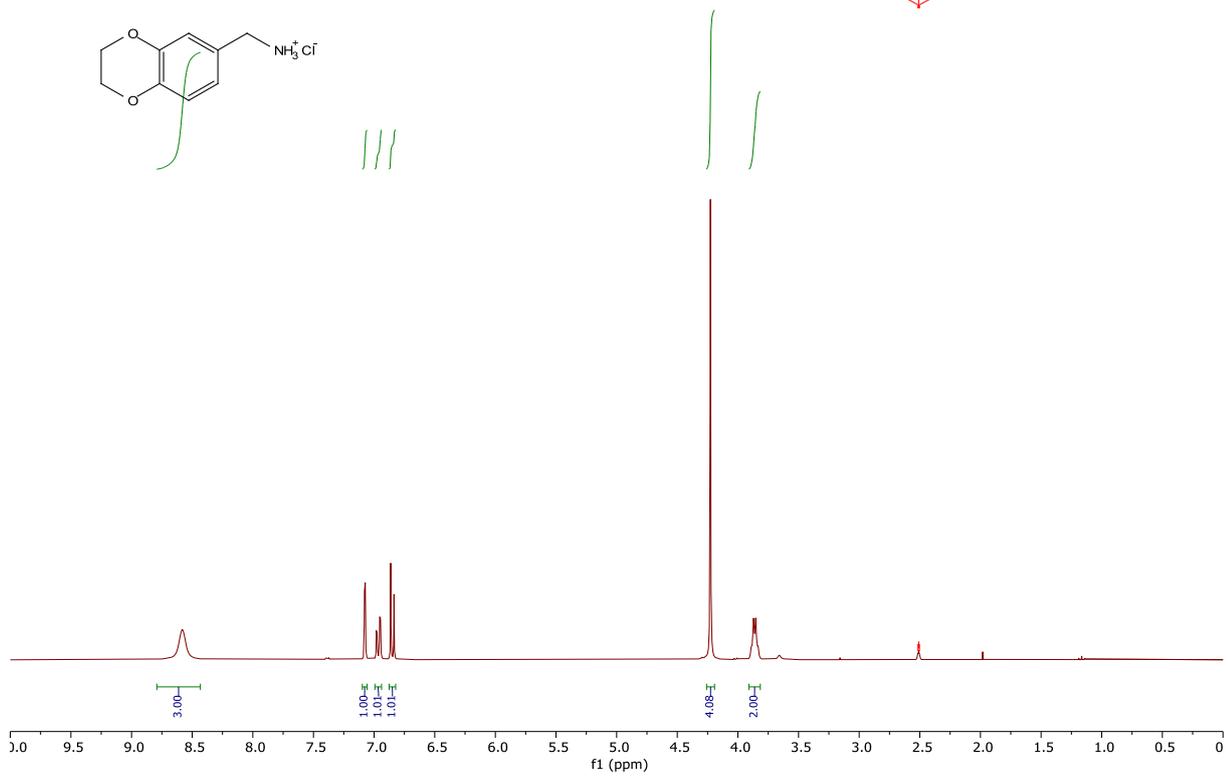
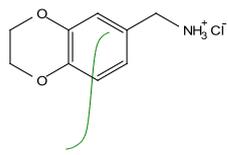
140.28  
131.31  
130.94  
119.49  
119.40  
111.12

42.02  
40.87 DMSO  
40.87 DMSO  
40.32 DMSO  
40.32 DMSO  
39.75 DMSO  
39.48 DMSO  
39.21 DMSO

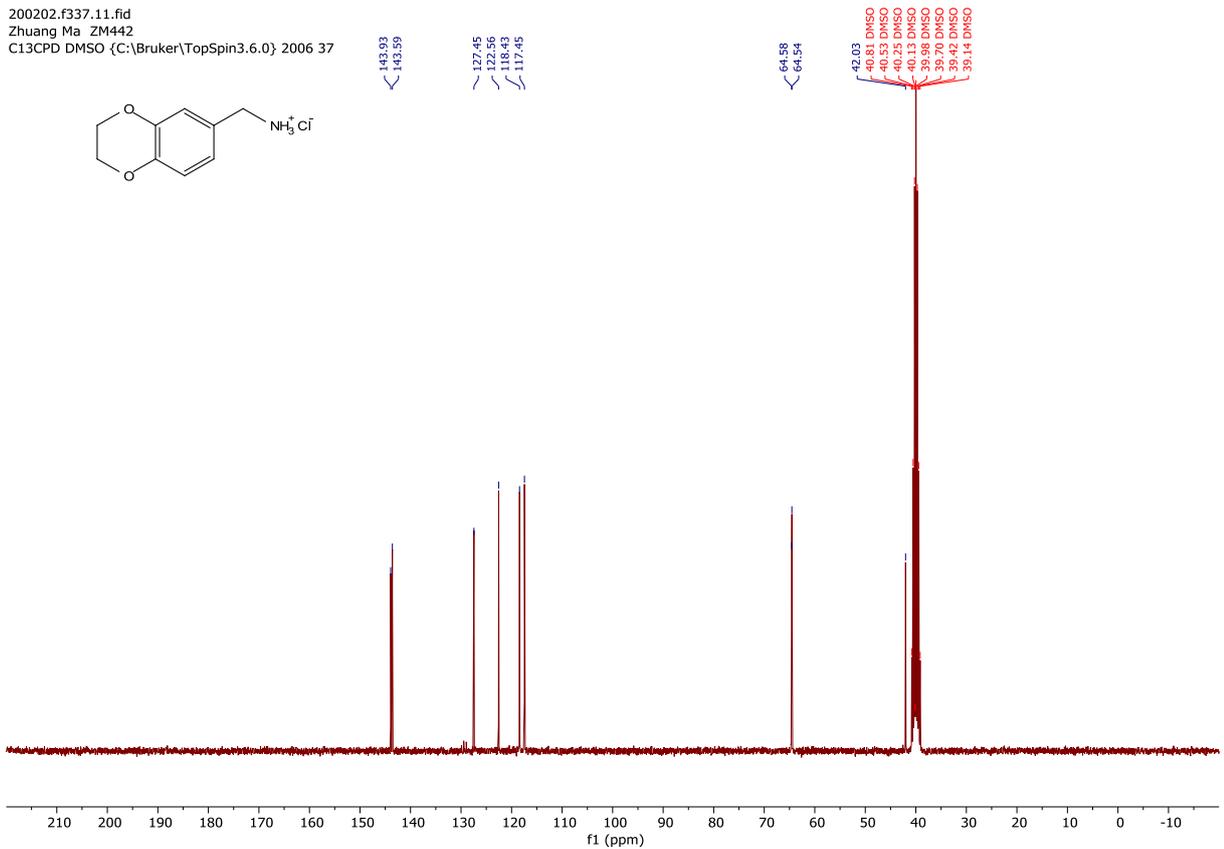
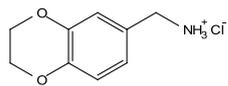




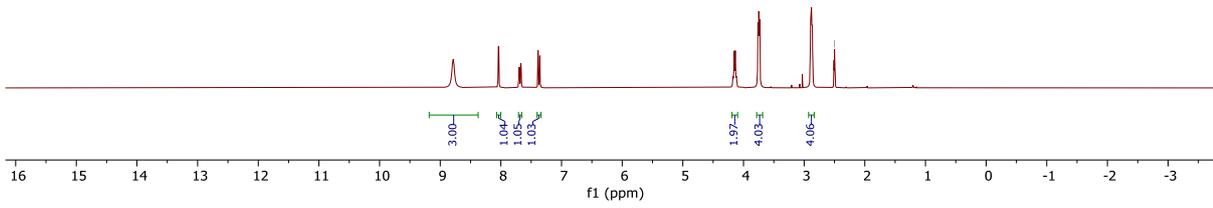
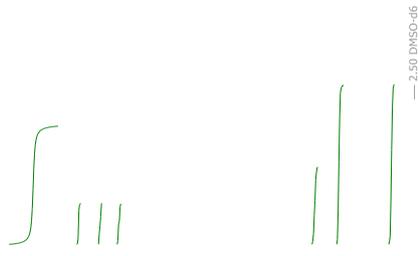
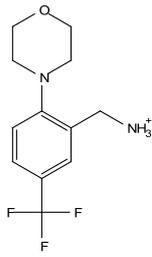
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PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2006 37



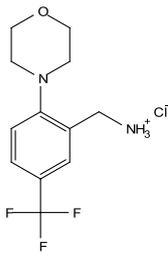
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Zhuang Ma\_ZM442  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2006 37



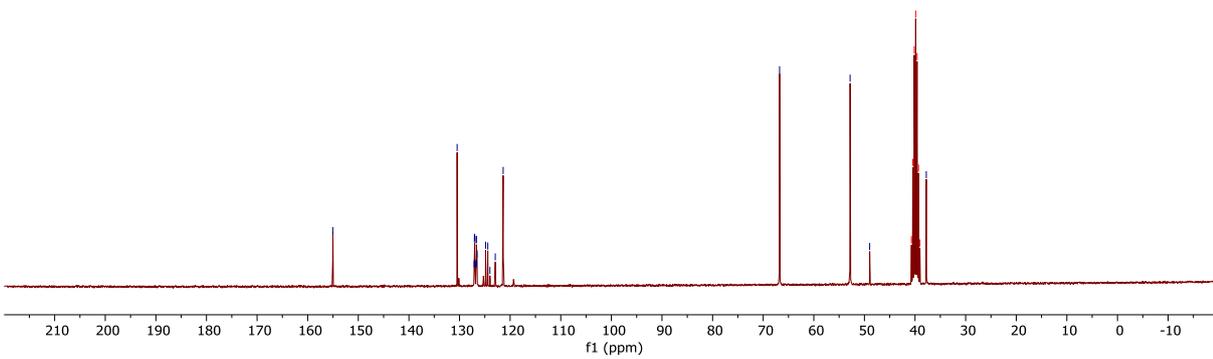
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 Zhuang Ma ZM-549  
 PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 24



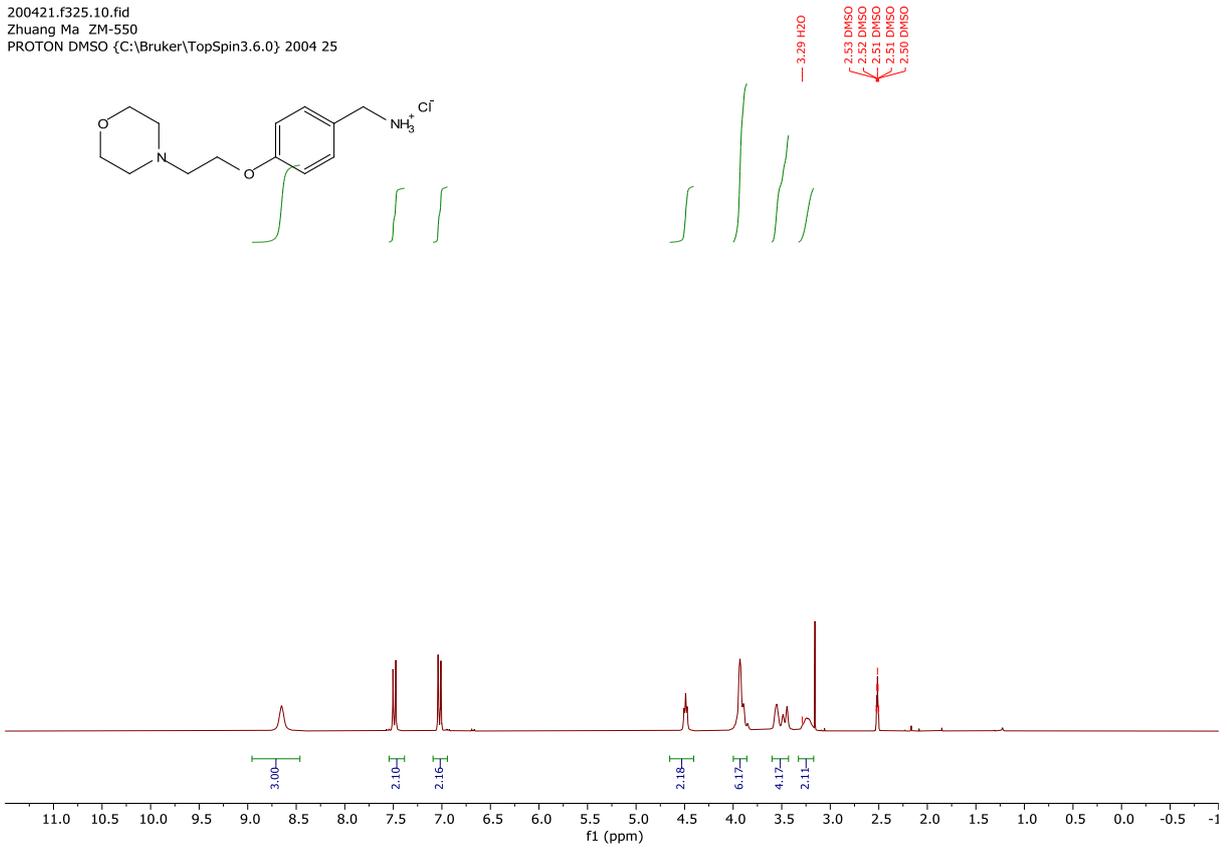
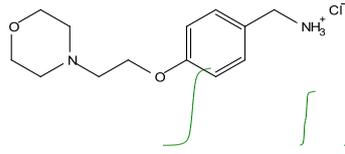
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 Zhuang Ma ZM-549  
 C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 24



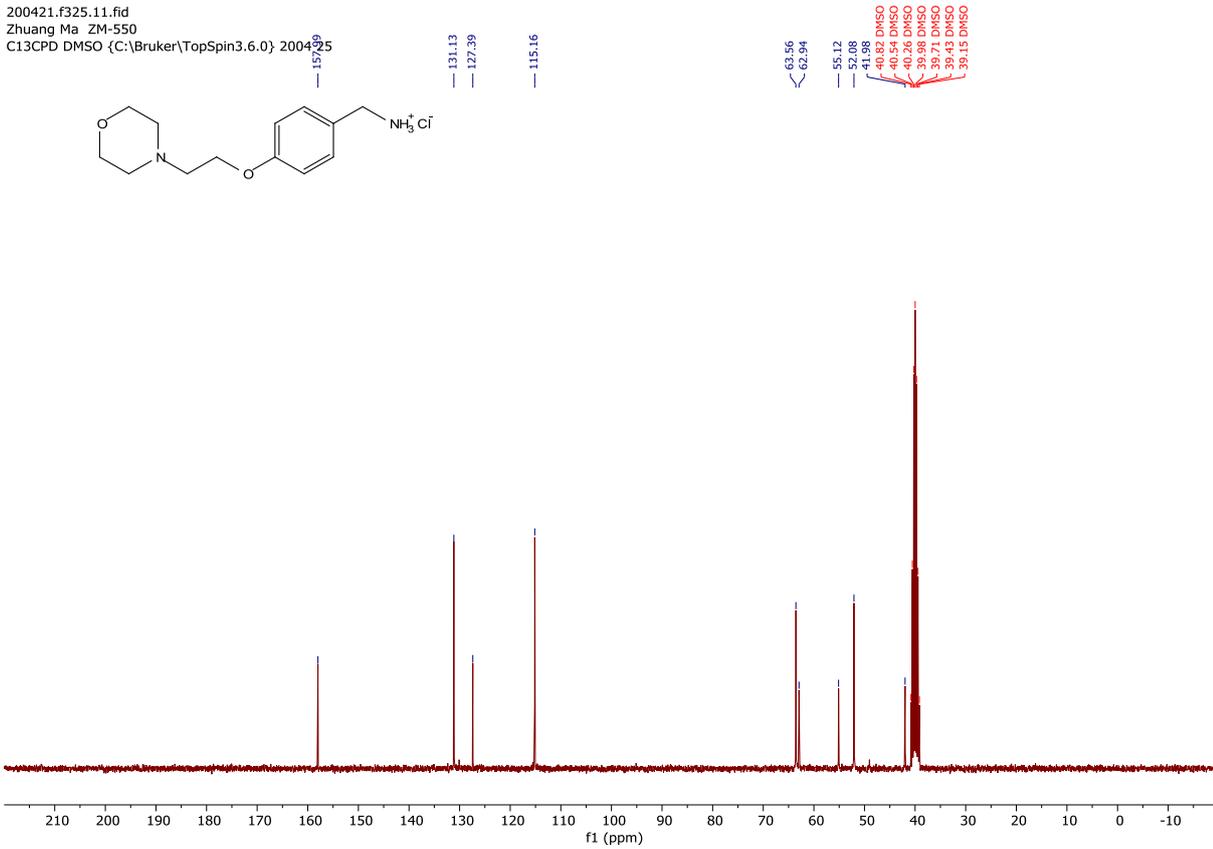
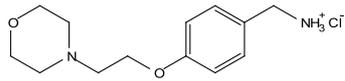
155.04  
 130.47  
 127.71  
 127.06  
 127.01  
 126.96  
 126.76  
 126.71  
 126.66  
 126.61  
 126.56  
 124.84  
 124.42  
 123.99  
 122.94  
 121.38  
 66.76  
 52.83  
 48.96  
 40.71 DMSO  
 40.43 DMSO  
 40.15 DMSO  
 39.87 DMSO  
 39.59 DMSO  
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 39.04 DMSO  
 37.77



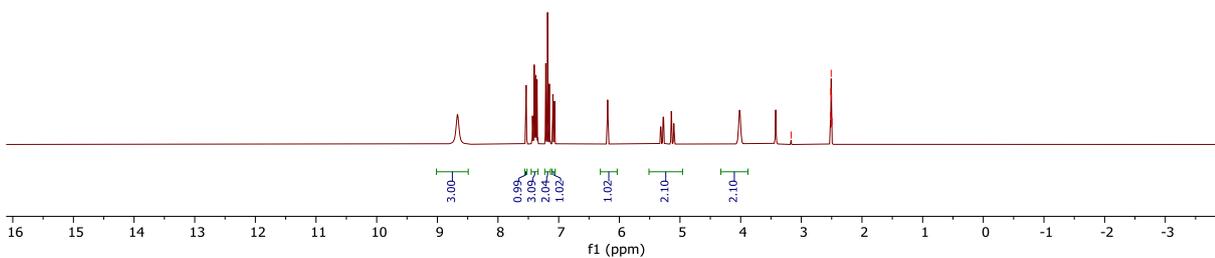
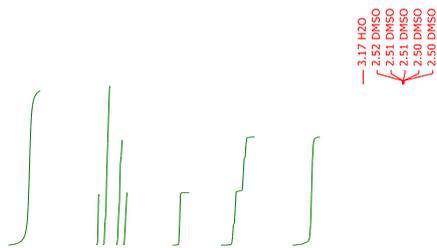
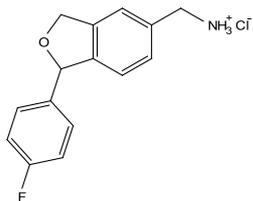
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Zhuang Ma ZM-550  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 25



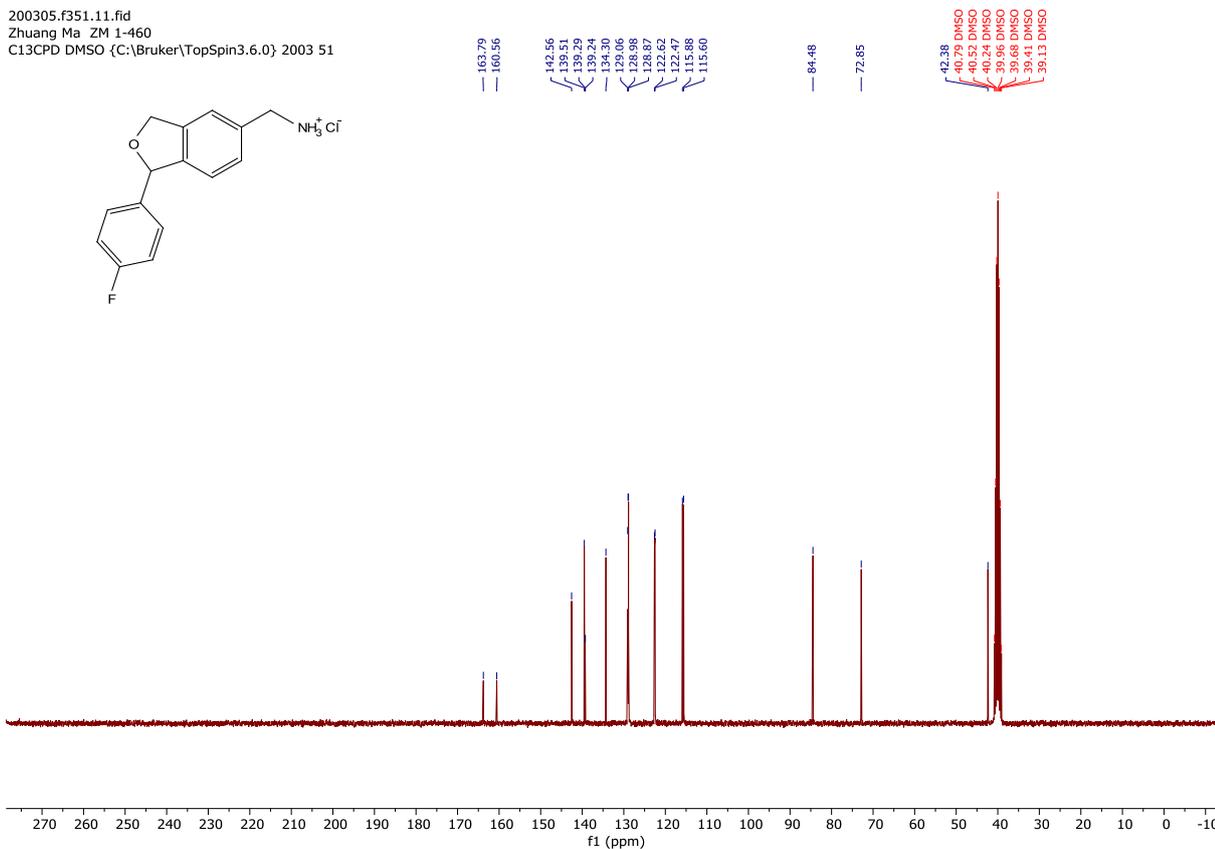
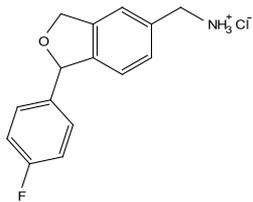
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Zhuang Ma ZM-550  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 25



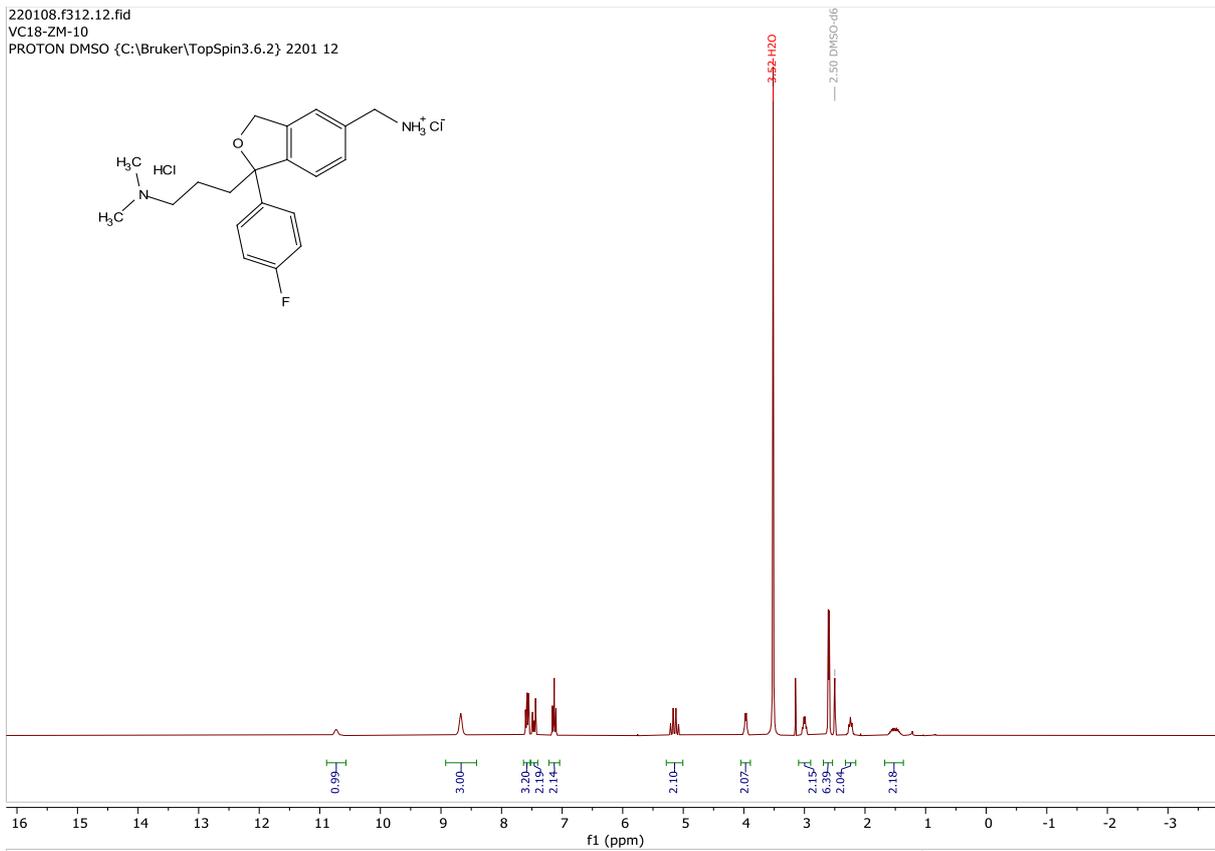
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Zhuang Ma\_ZM 1-460  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 51



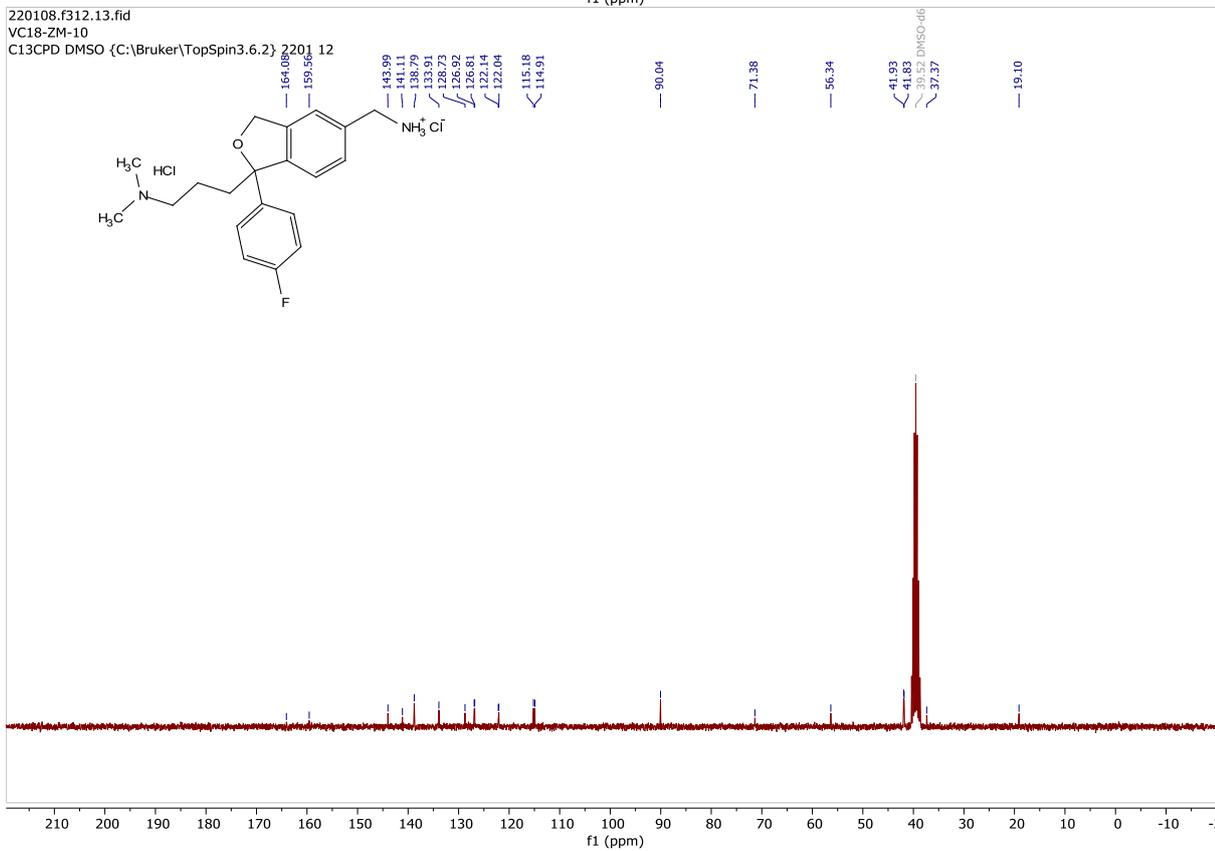
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 51



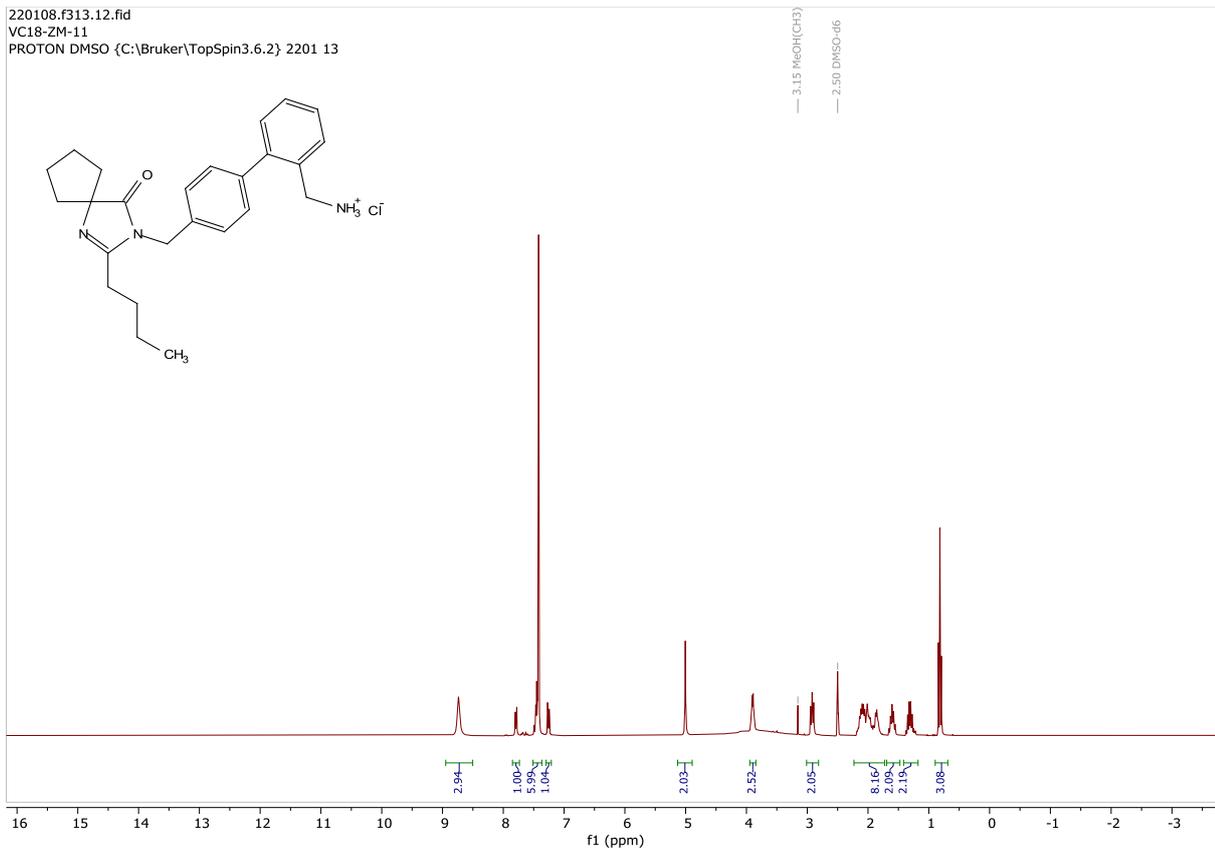
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PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2201 12



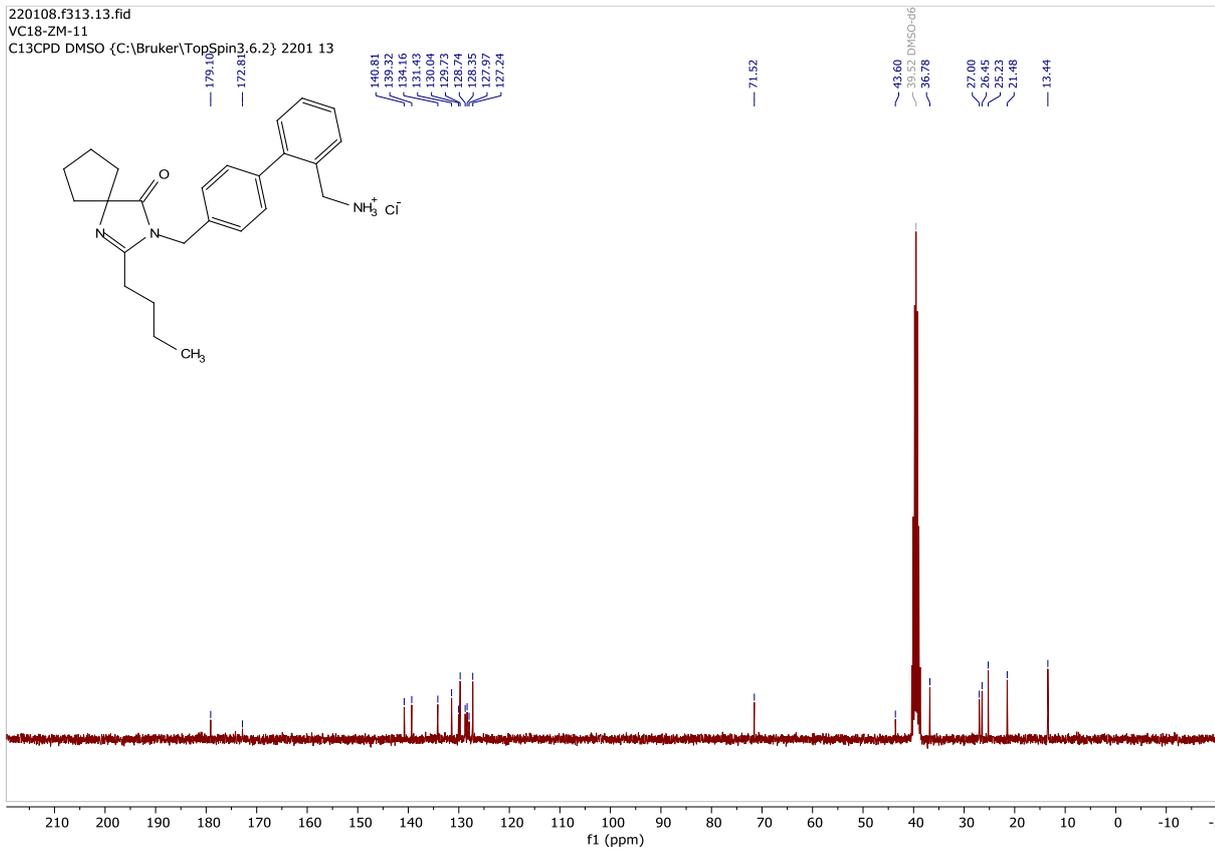
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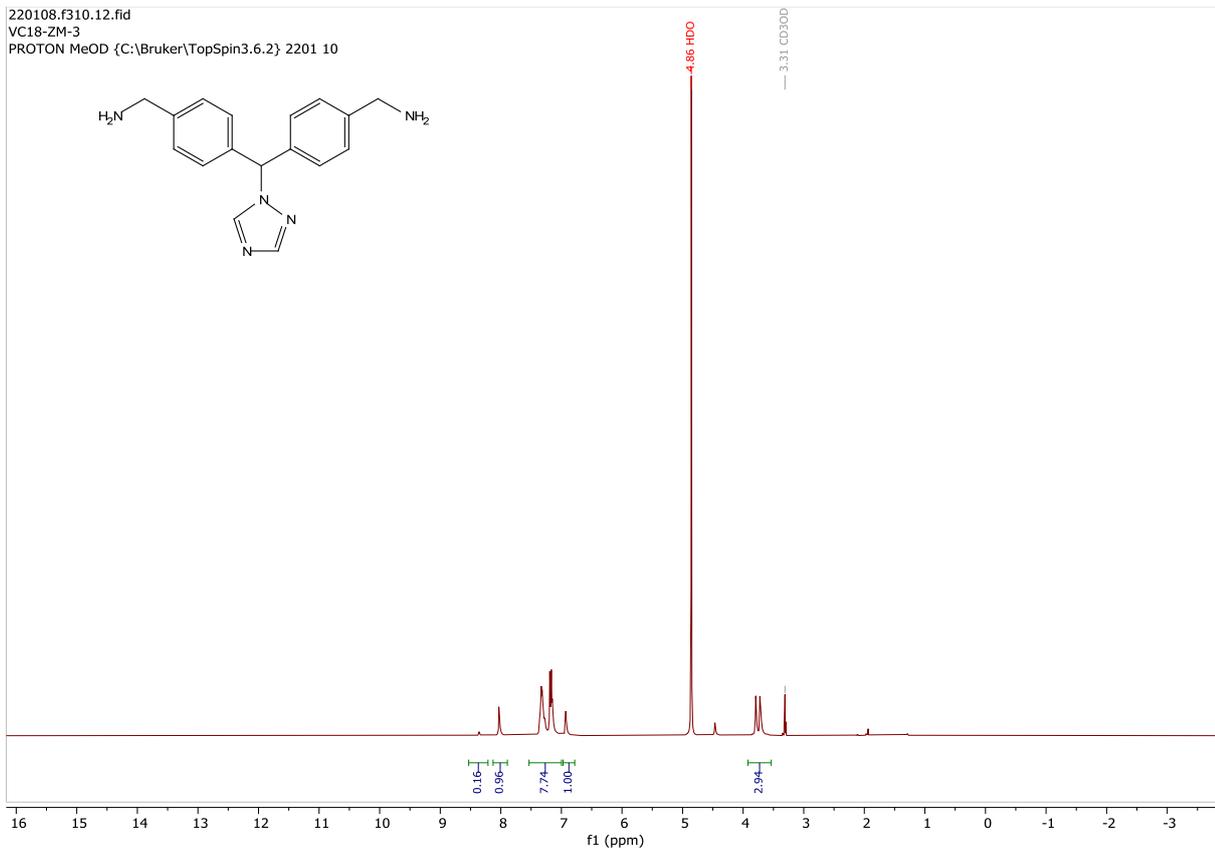
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VC18-ZM-11  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2201 13



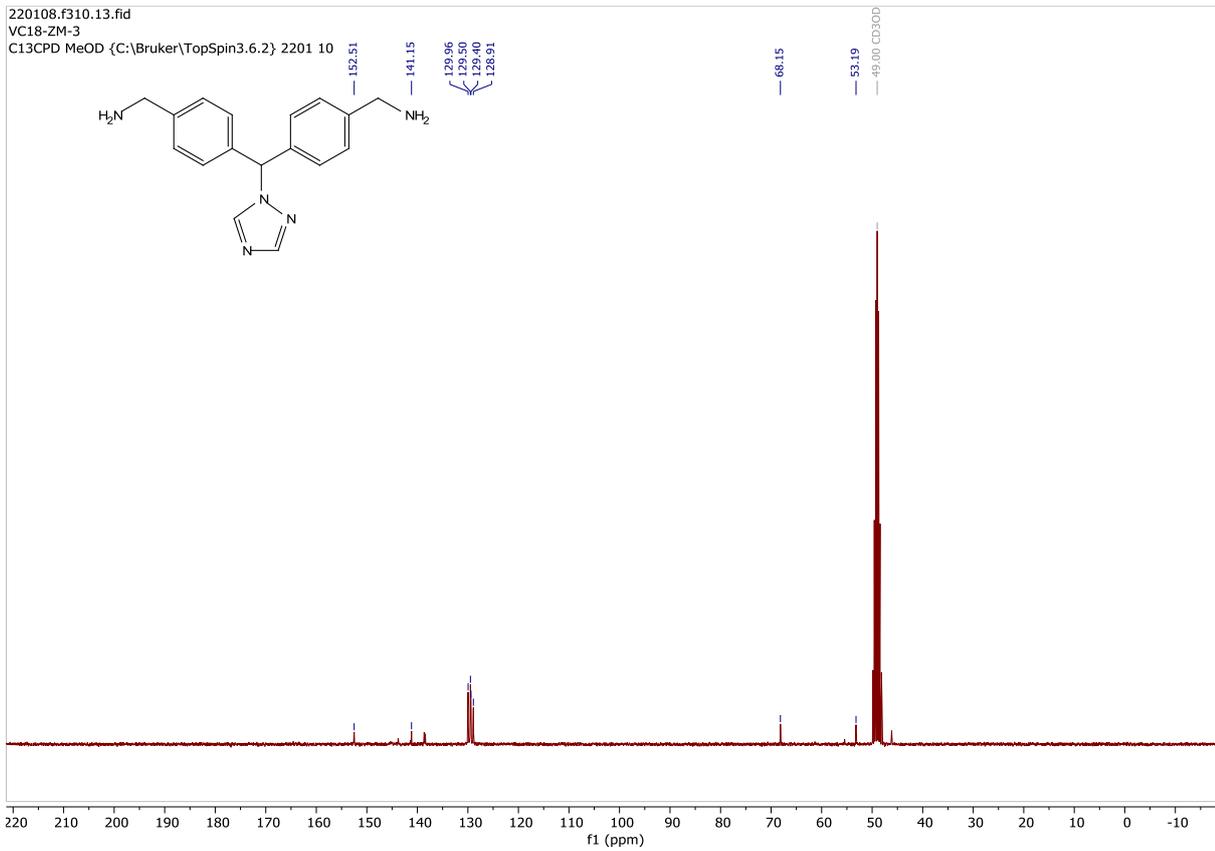
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VC18-ZM-11  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2201 13



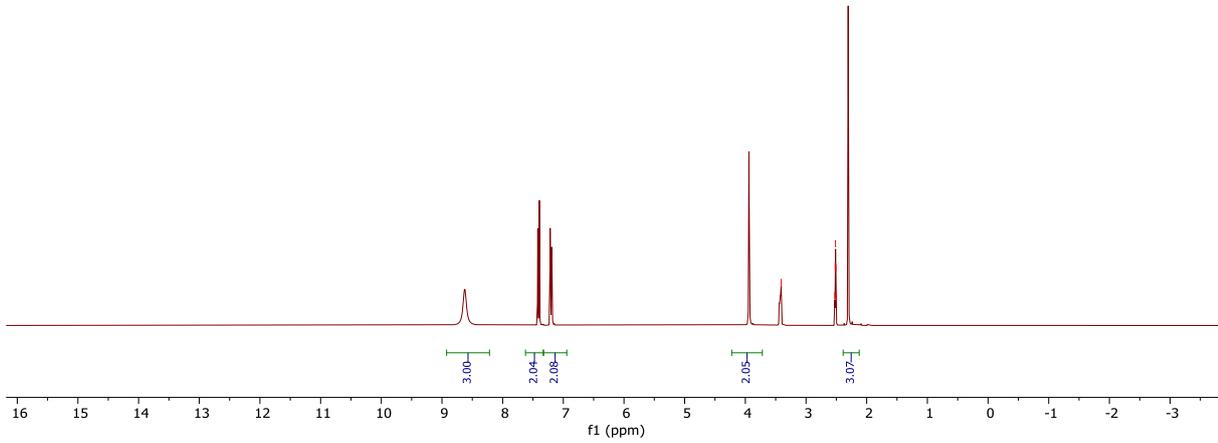
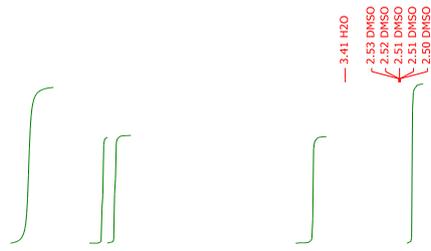
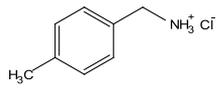
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VC18-ZM-3  
PROTON MeOD {C:\Bruker\TopSpin3.6.2} 2201 10



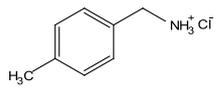
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C13CPD MeOD {C:\Bruker\TopSpin3.6.2} 2201 10



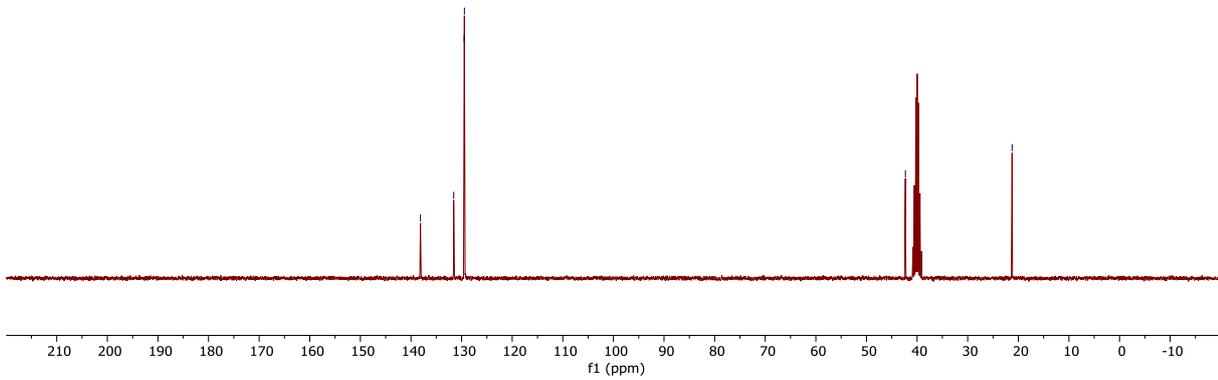
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Zhuang Ma ZM 4-489  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2003 3



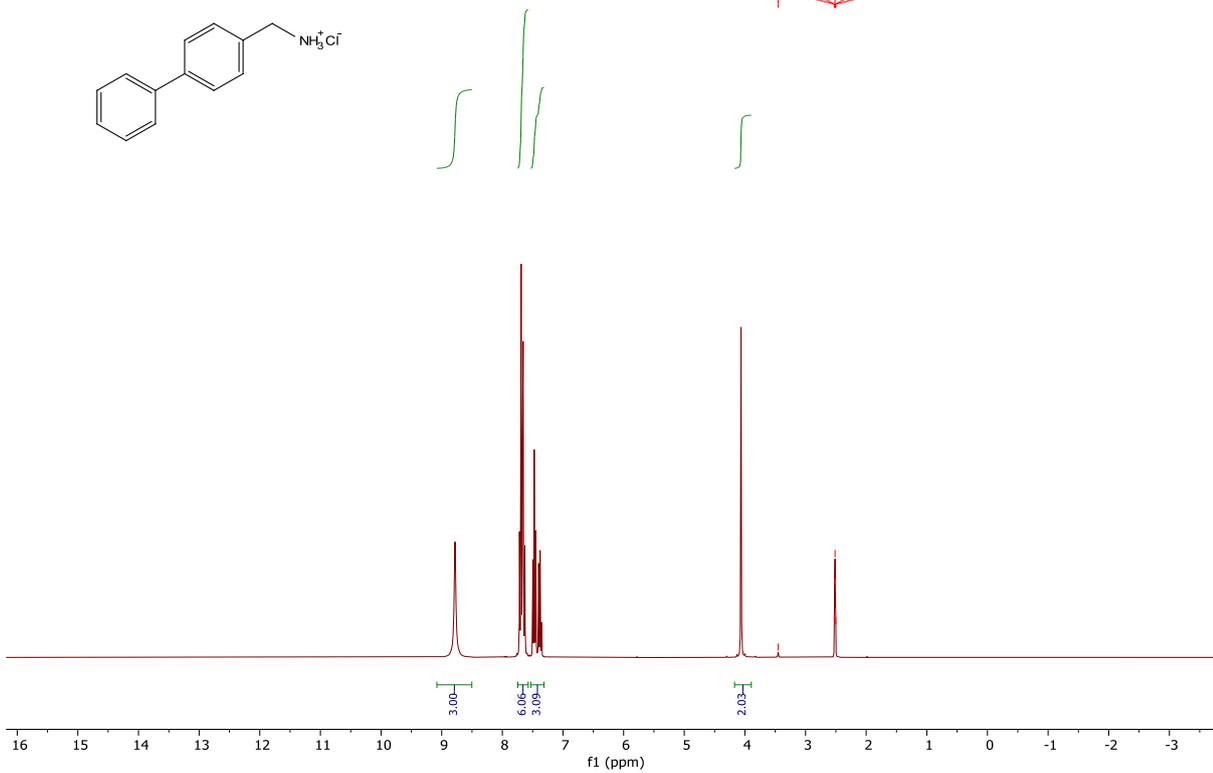
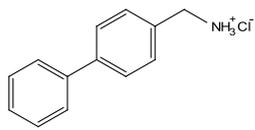
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Zhuang Ma ZM 4-489  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2003 3



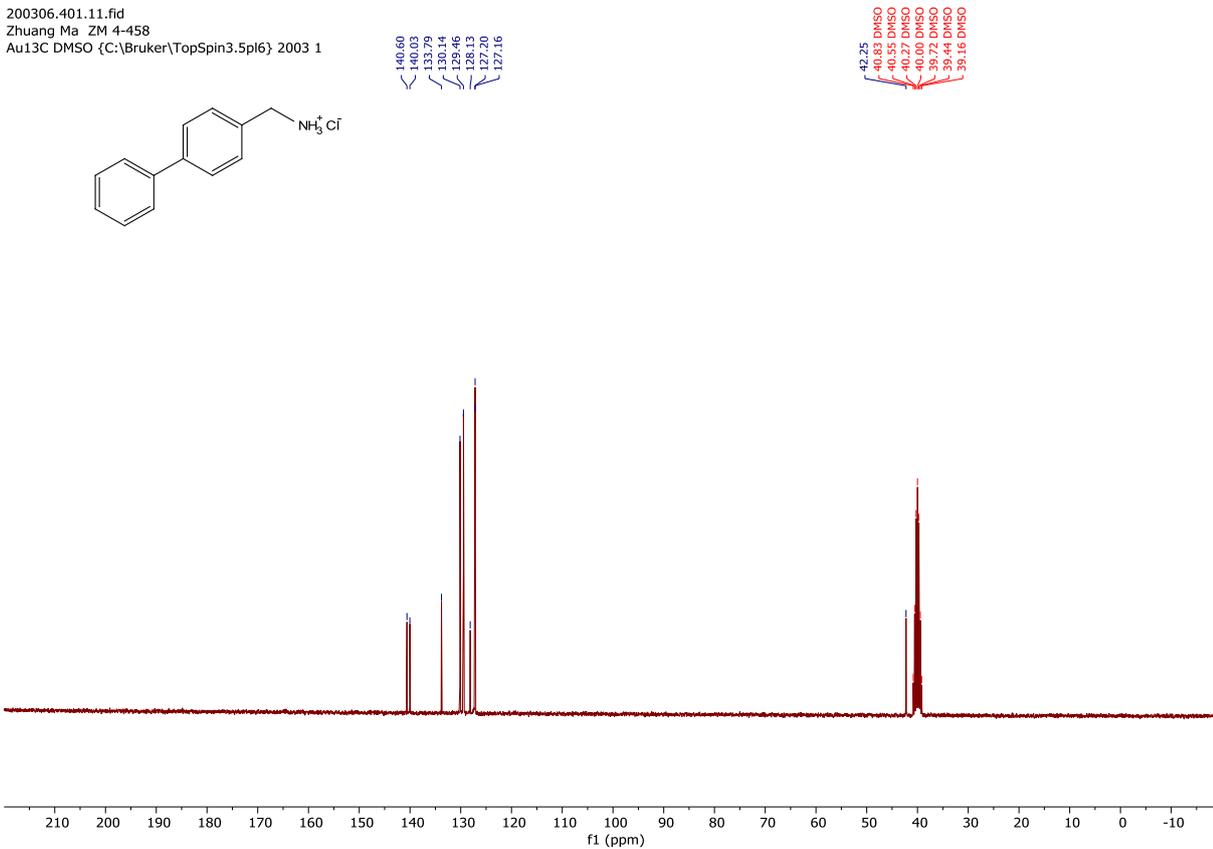
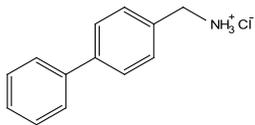
138.12  
131.56  
129.49  
129.45  
42.31  
21.23



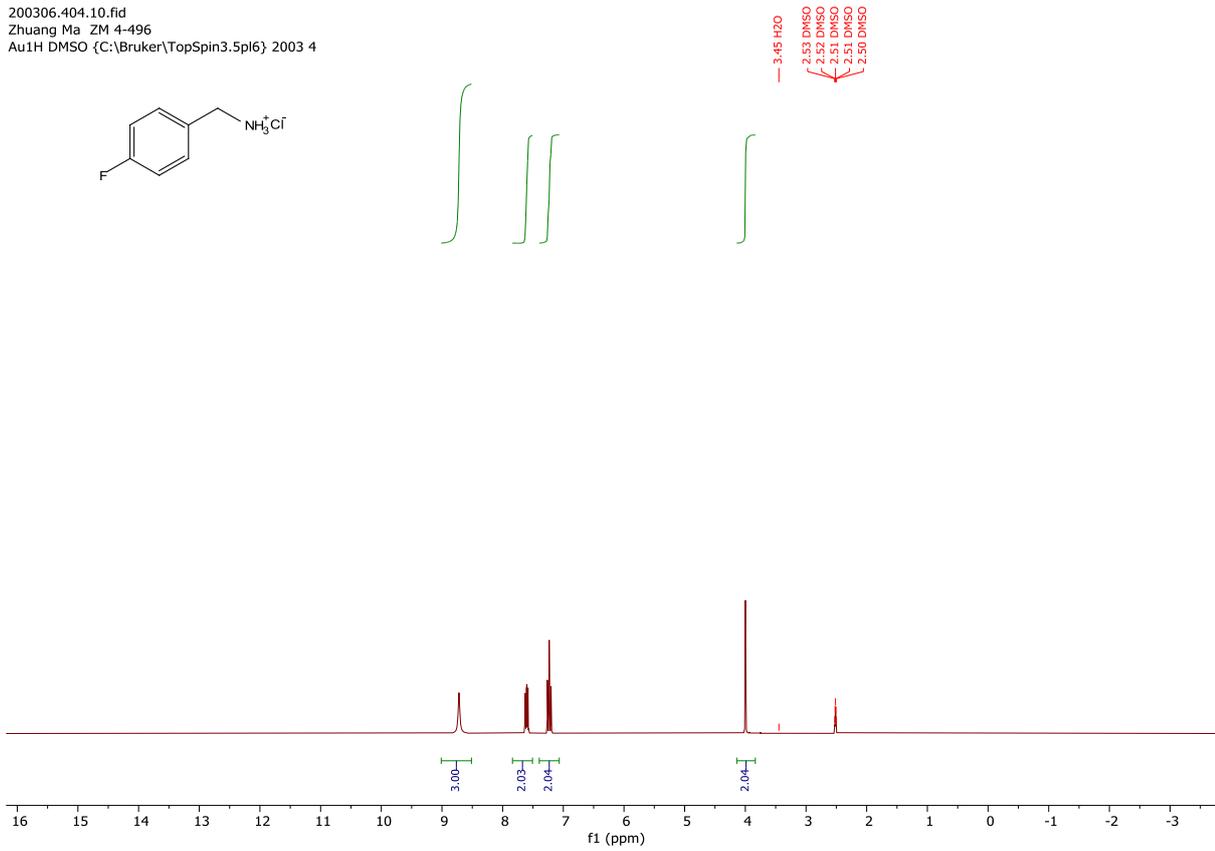
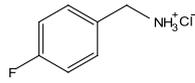
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Zhuang Ma ZM 4-458  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2003 1



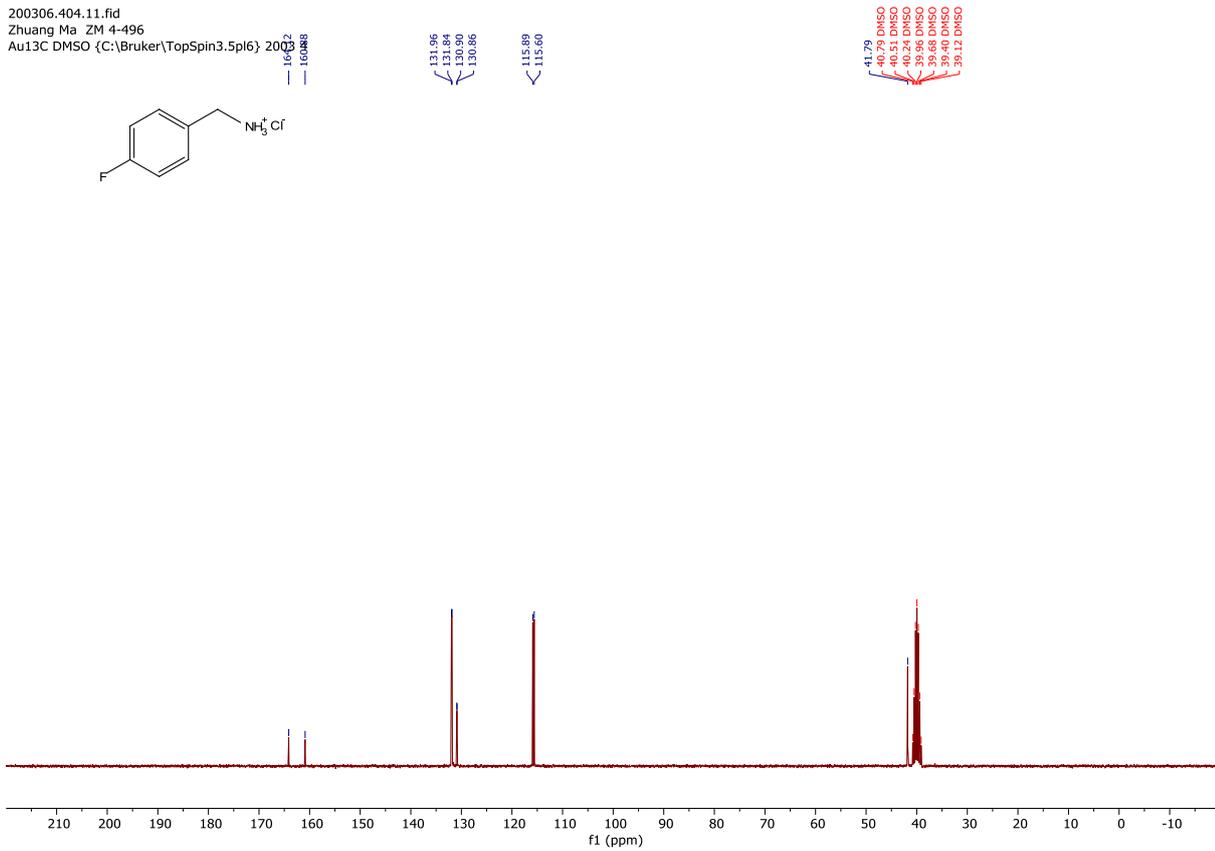
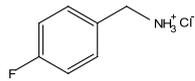
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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2003 1



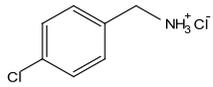
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Zhuang Ma ZM 4-496  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2003 4



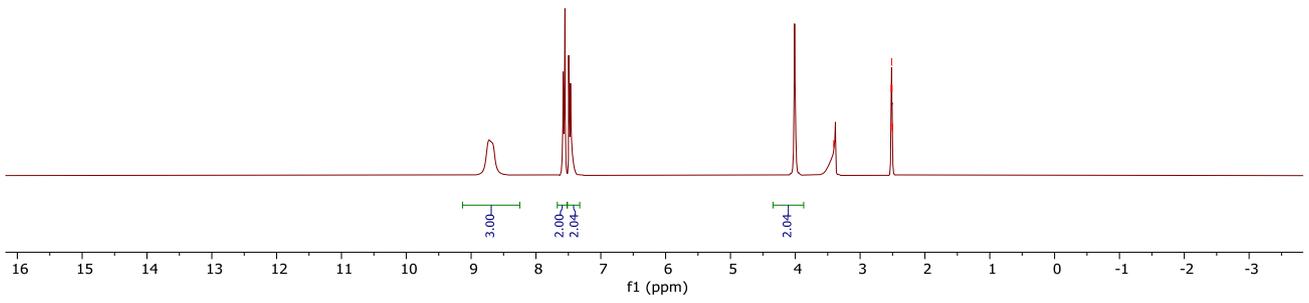
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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2003 4



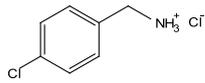
200305.f352.11.fid  
Zhuang Ma ZM 1-303  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 52



3.41 H2O  
2.53 DMSO  
2.52 DMSO  
2.11 DMSO  
2.11 DMSO  
2.50 DMSO

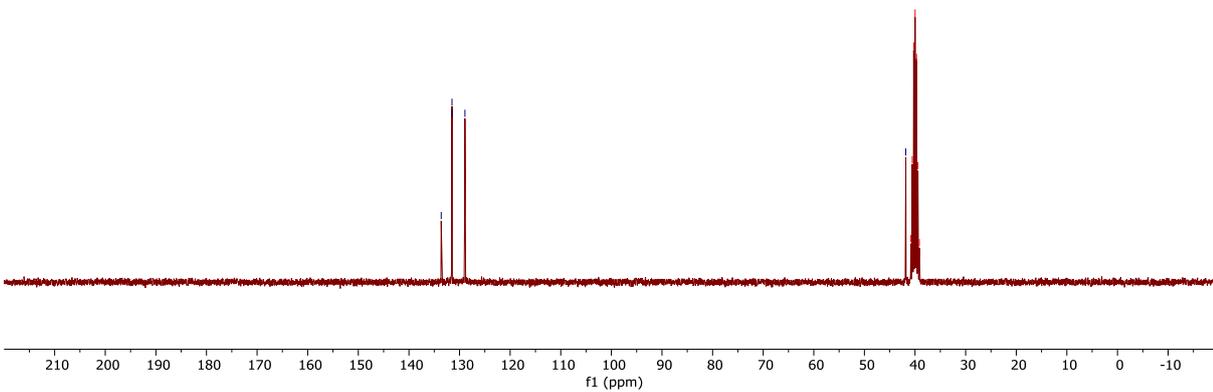


200305.f352.11.fid  
Zhuang Ma ZM 1-303  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 52

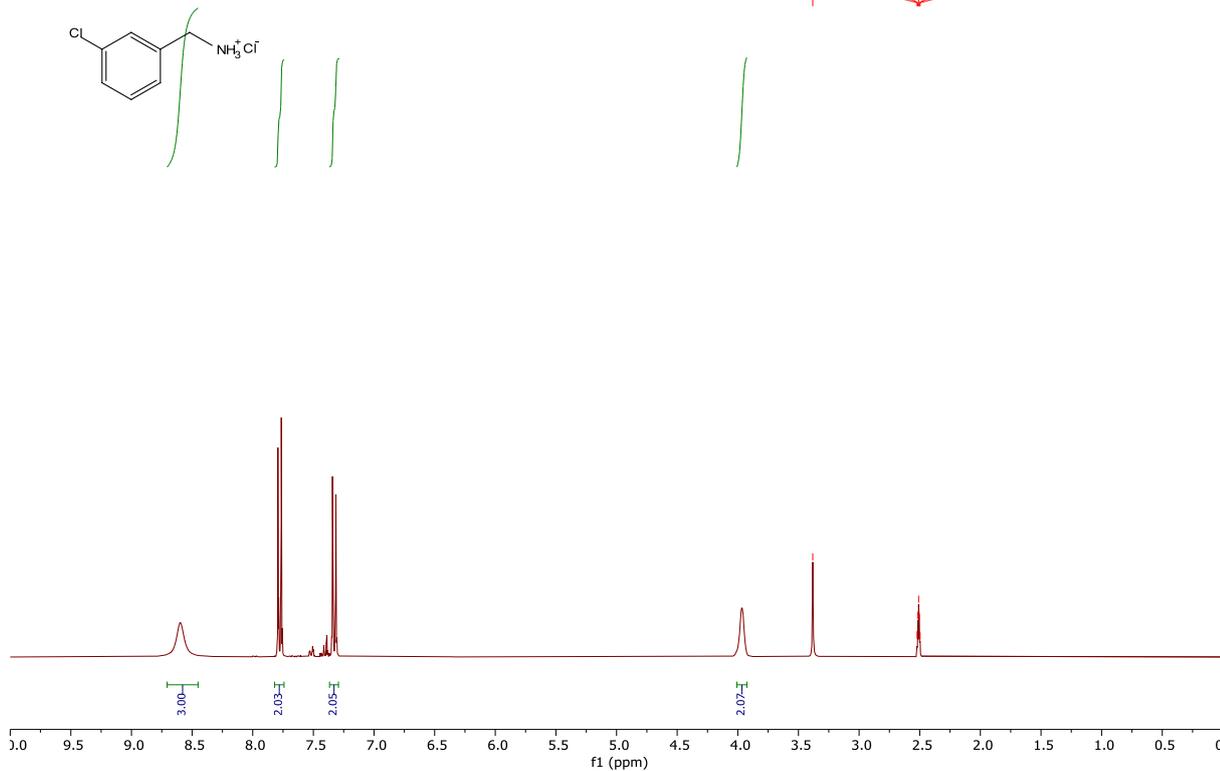
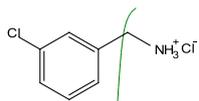


133.62  
131.51  
128.93

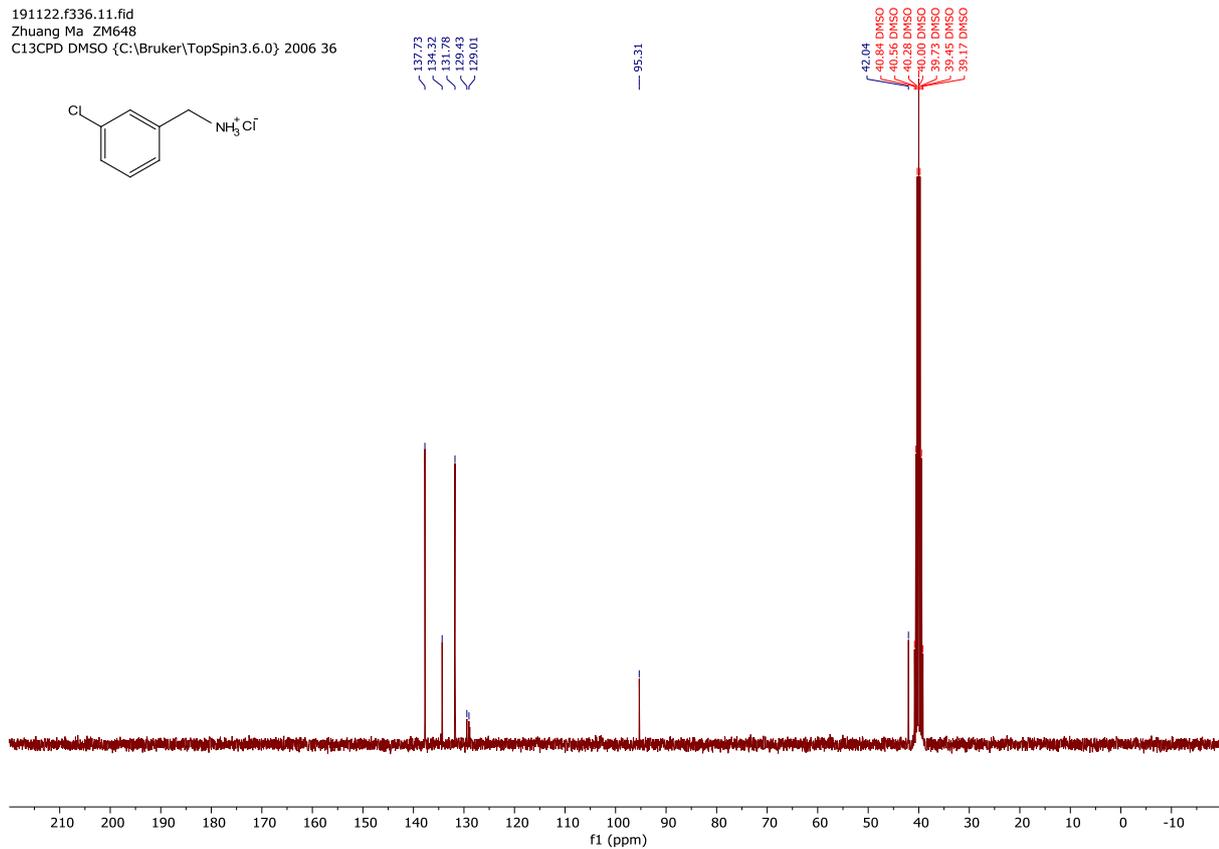
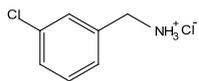
41.83  
40.82 DMSO  
40.78 DMSO  
40.38 DMSO  
39.99 DMSO  
39.71 DMSO  
39.43 DMSO  
39.15 DMSO



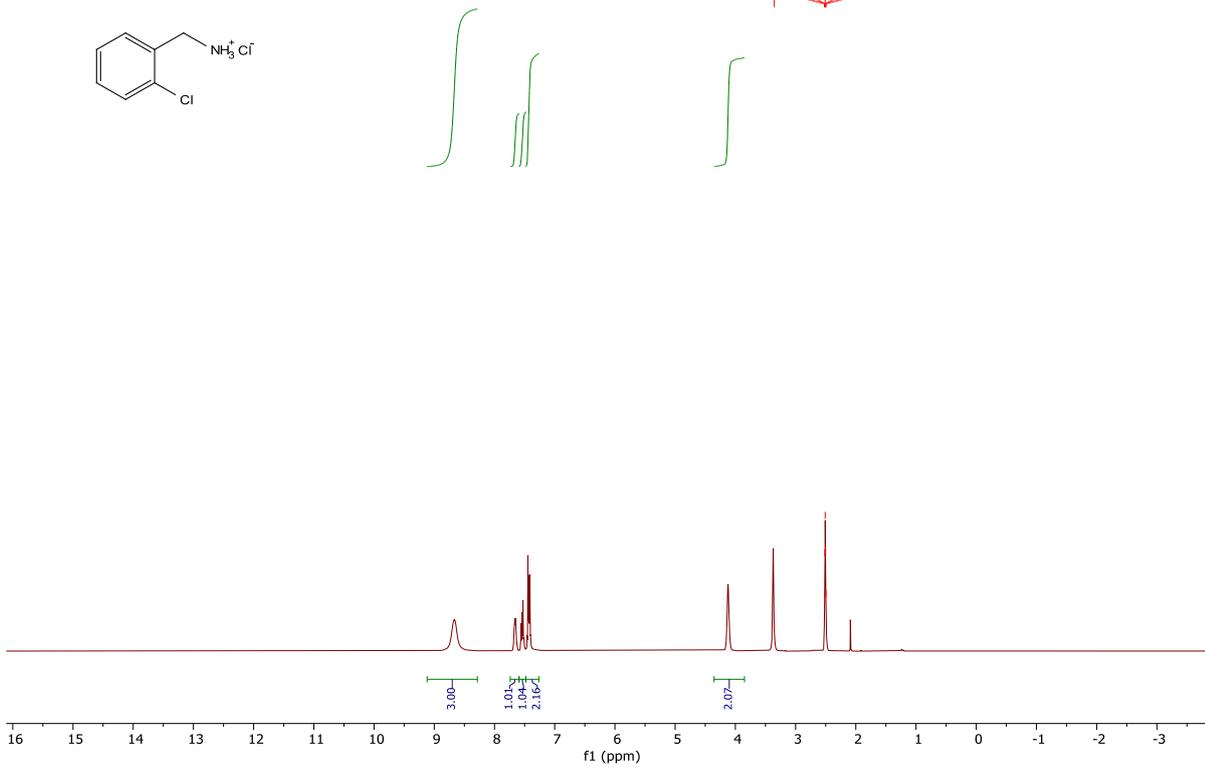
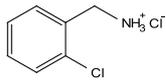
191122.f336.10.fid  
Zhuang ZM648  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2006 36



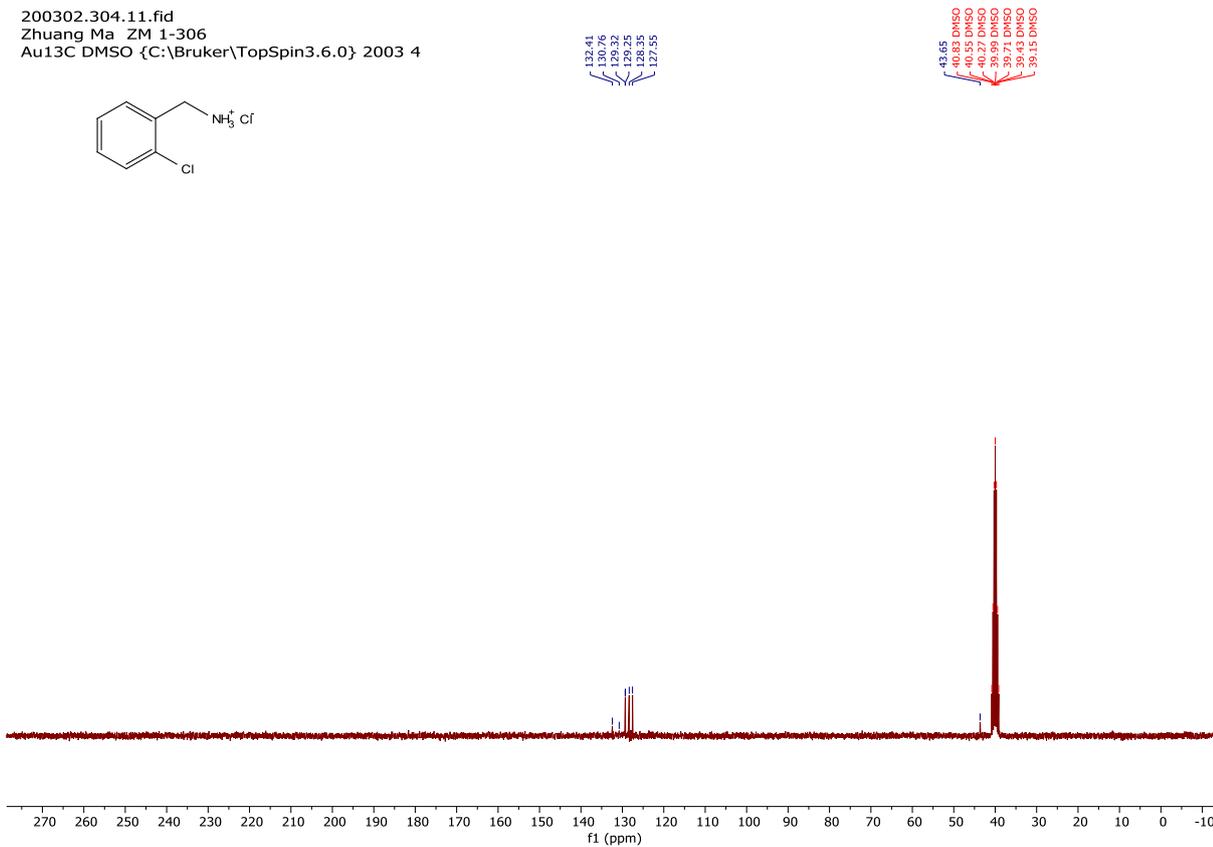
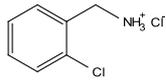
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Zhuang Ma ZM648  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2006 36



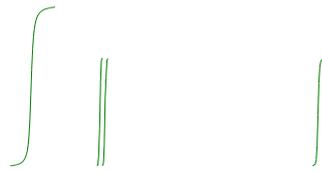
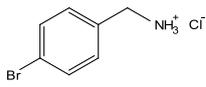
200302.304.10.fid  
Zhuang Ma ZM 1-306  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2003 4



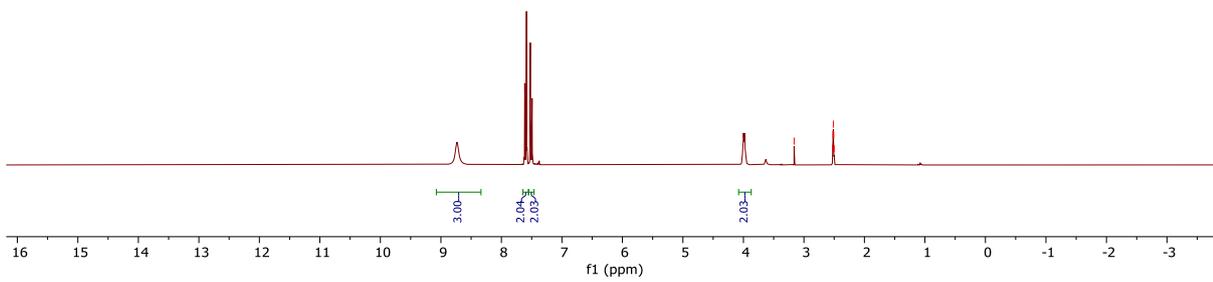
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Zhuang Ma ZM 1-306  
Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2003 4



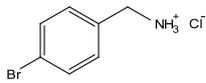
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Zhuang Ma ZM 1-304  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2003 3



3.16 H2O  
2.53 DMSO  
2.52 DMSO  
2.51 DMSO  
2.51 DMSO  
2.50 DMSO



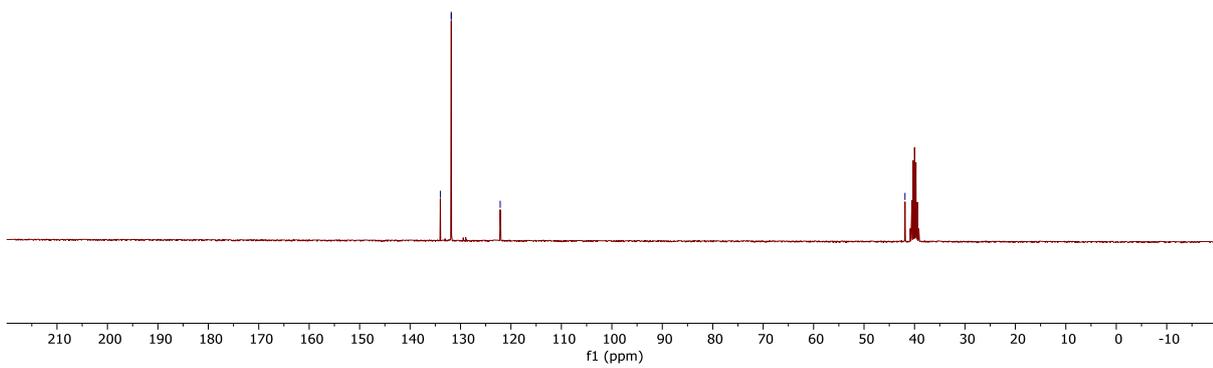
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Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2003 3



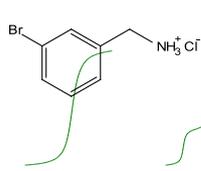
134.00  
131.83  
131.83

122.13

41.88

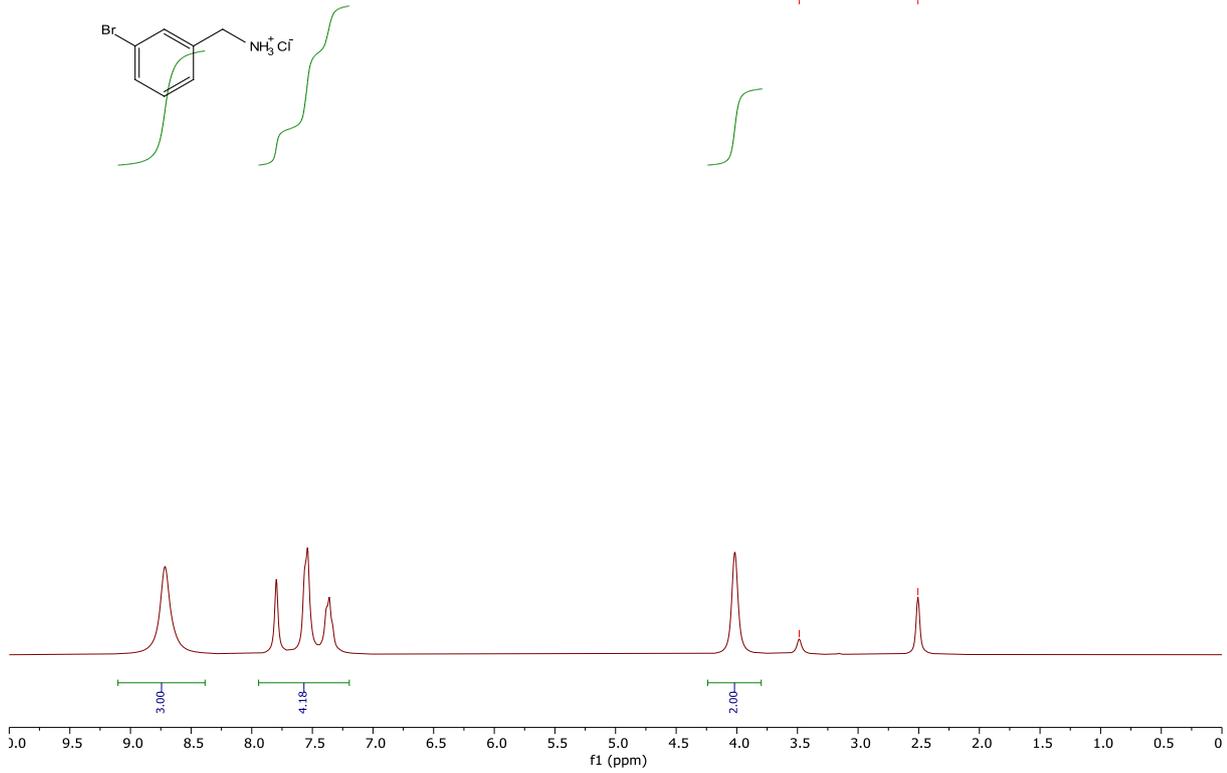


200119.321.10.fid  
Zhuang Ma ZM118  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2010 21

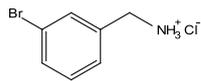


3.49 H<sub>2</sub>O

2.51 DMSO

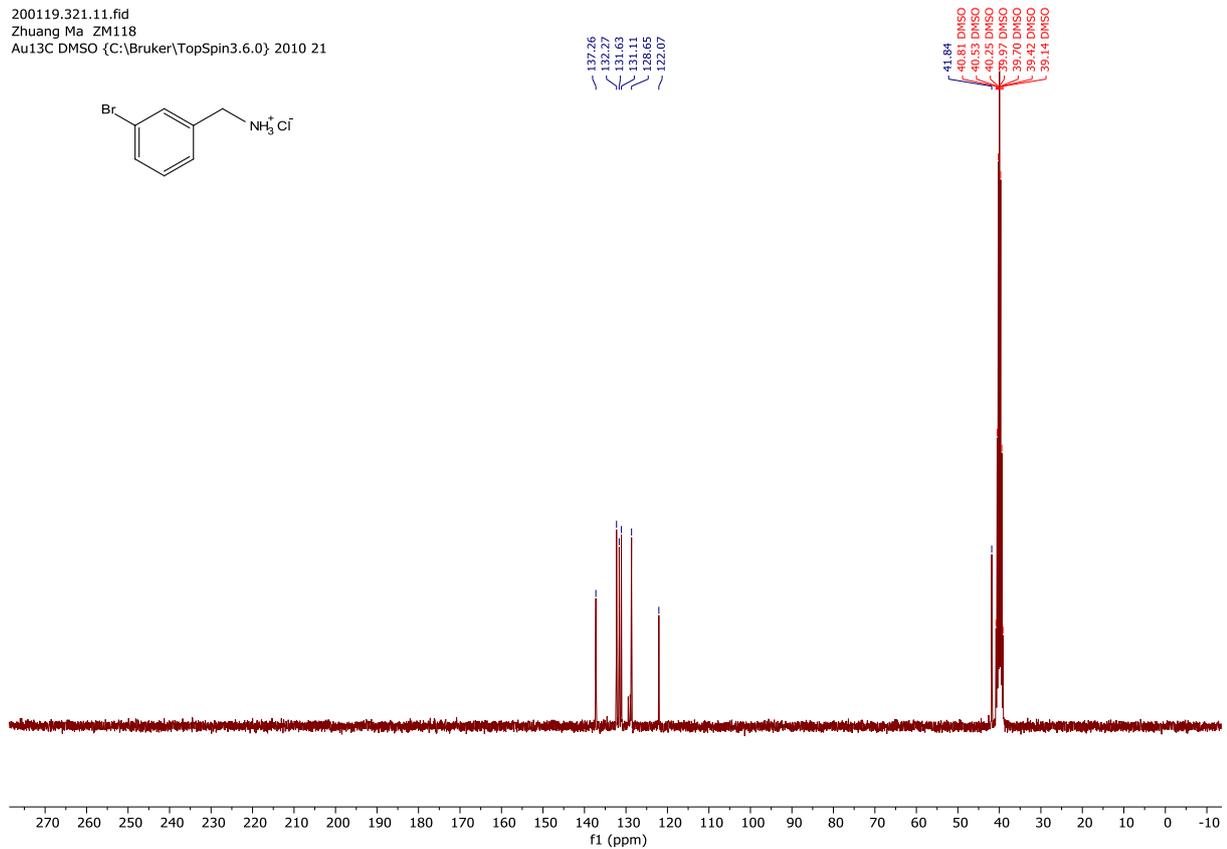


200119.321.11.fid  
Zhuang Ma ZM118  
Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2010 21

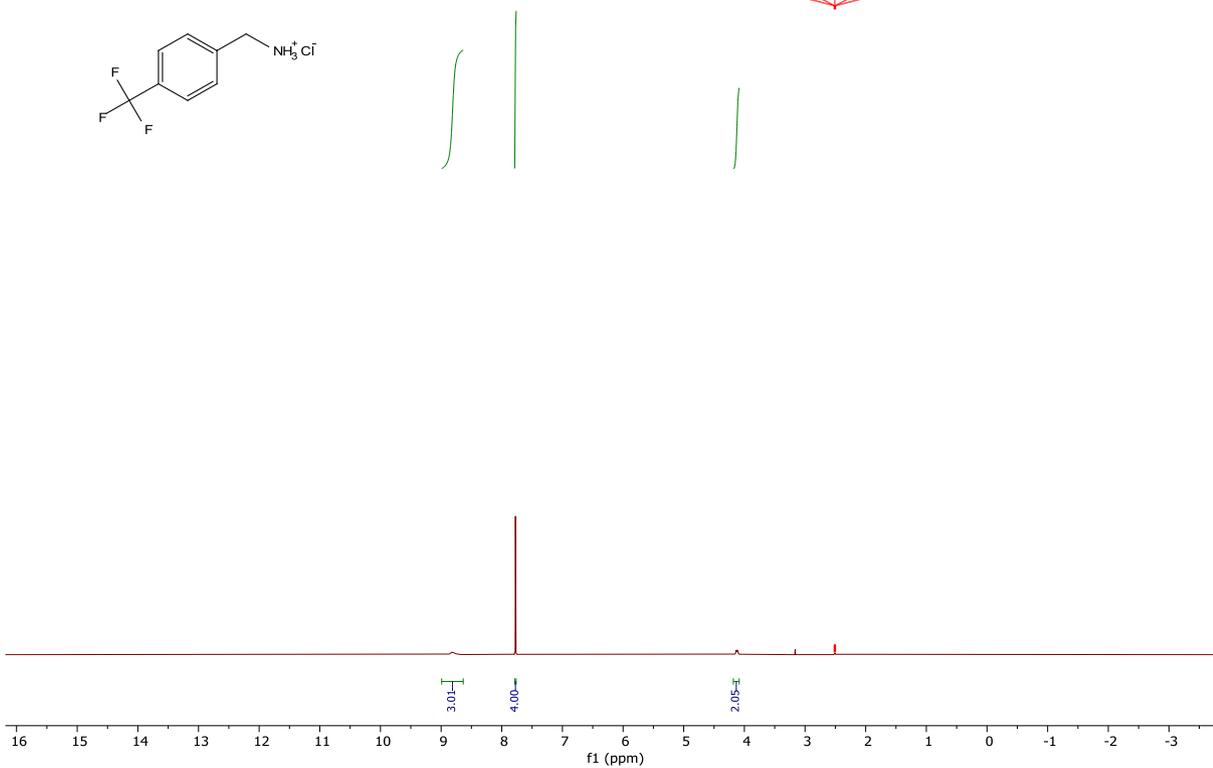
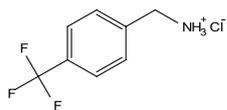


137.26  
132.27  
131.63  
131.11  
128.65  
122.07

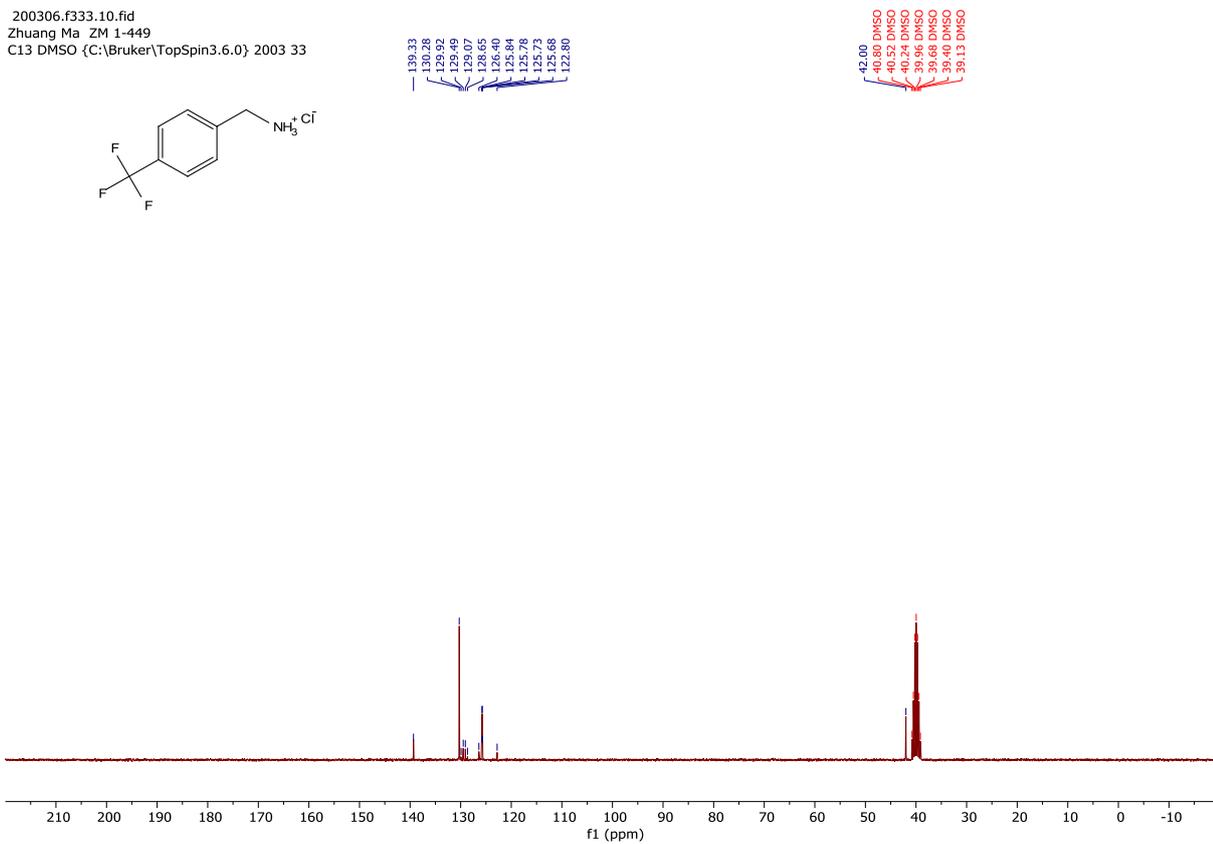
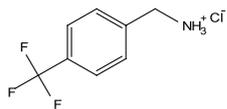
41.84  
40.81 DMSO  
40.72 DMSO  
40.72 DMSO  
39.97 DMSO  
39.70 DMSO  
39.42 DMSO  
39.14 DMSO



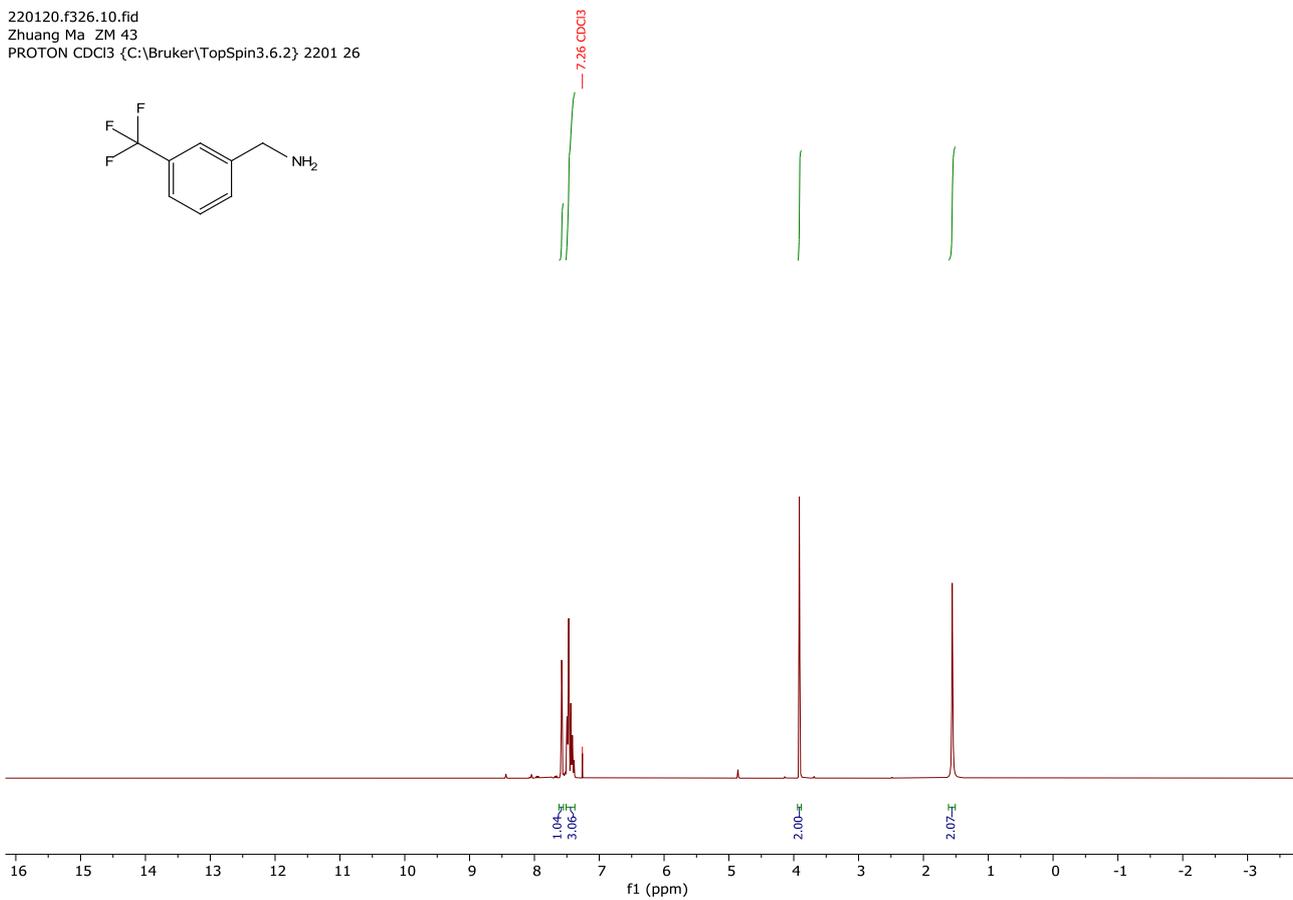
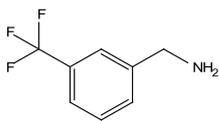
200306.f333.10.fid  
Zhuang Ma ZM 1-449  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 33



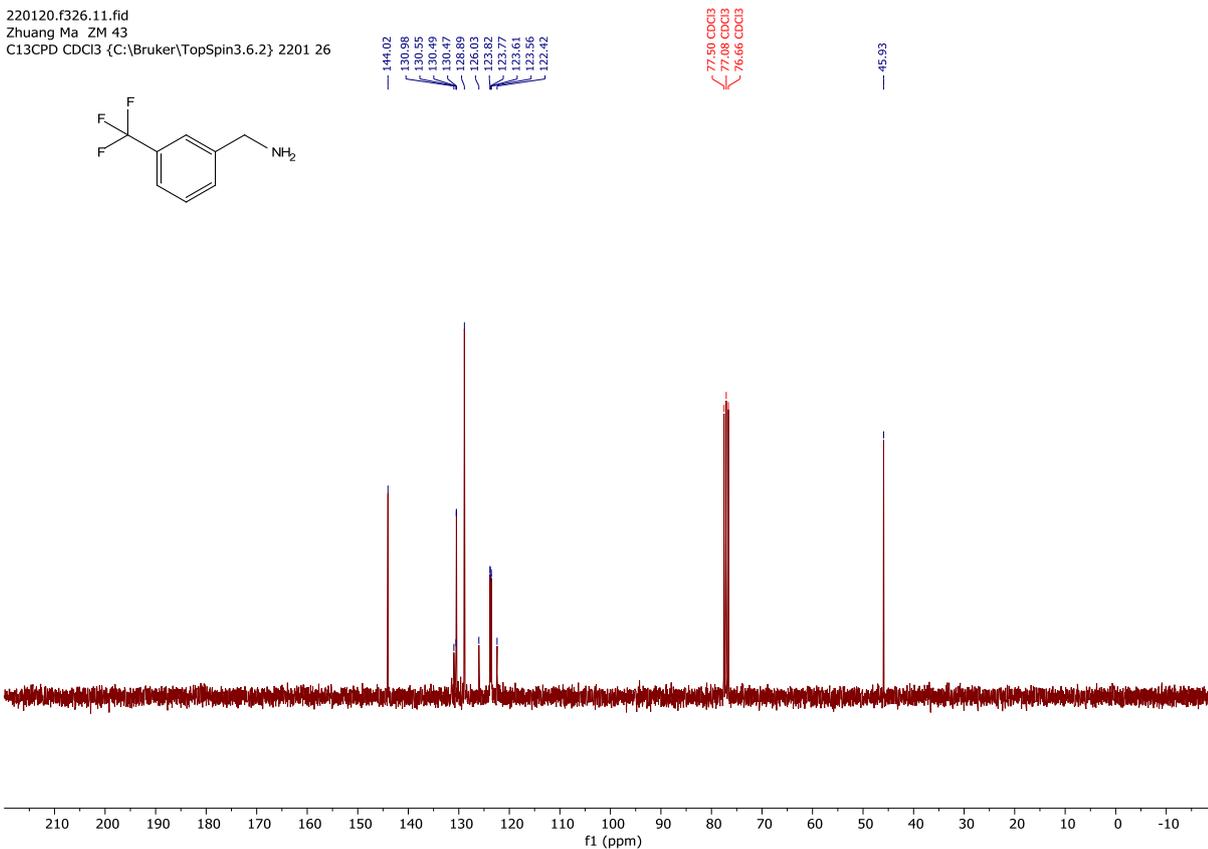
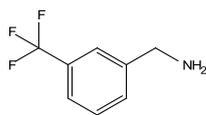
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Zhuang Ma ZM 1-449  
C13 DMSO {C:\Bruker\TopSpin3.6.0} 2003 33



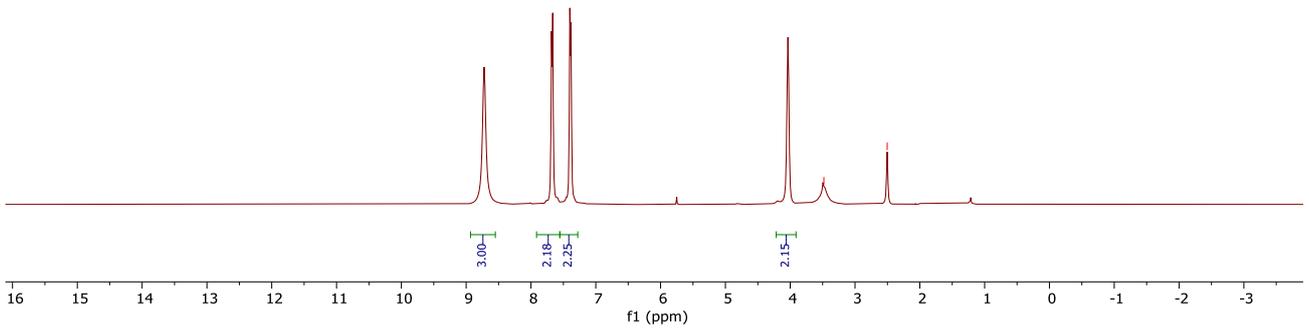
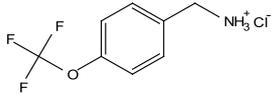
220120.f326.10.fid  
Zhuang Ma ZM 43  
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2201 26



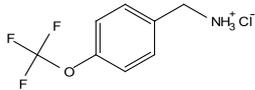
220120.f326.11.fid  
Zhuang Ma ZM 43  
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2201 26



200427.460.10.fid  
Zhuang Ma ZM-559  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 60

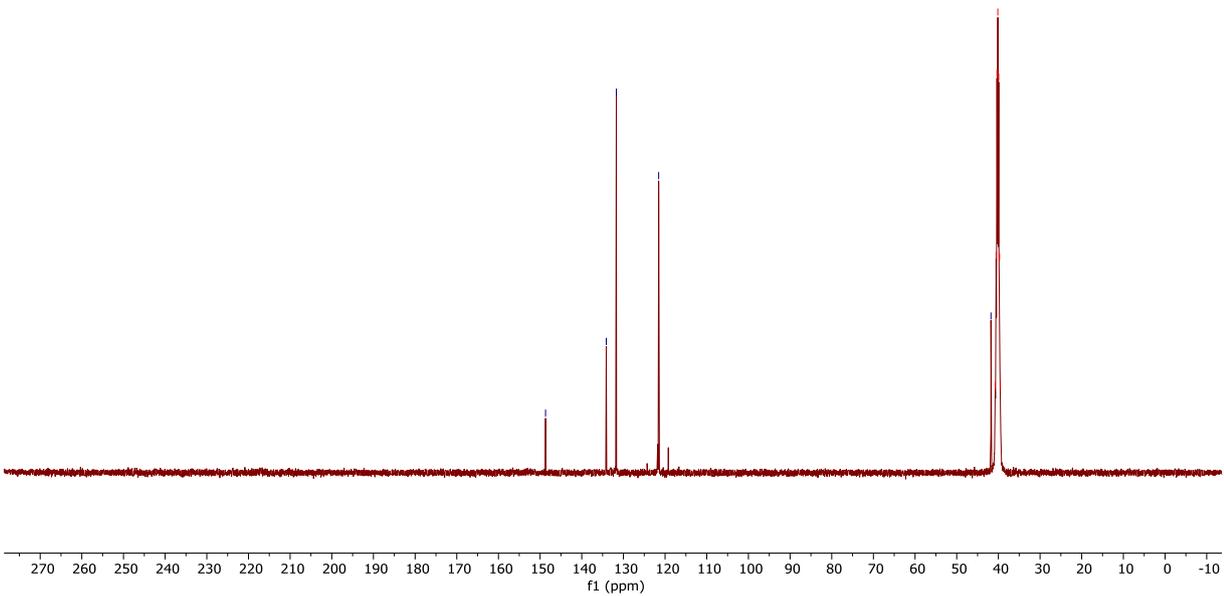


200427.460.11.fid  
Zhuang Ma ZM-559  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2004 60

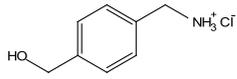


148.69  
134.12  
131.70  
121.54

41.78  
40.77 DMSO  
40.55 DMSO  
40.35 DMSO  
39.83 DMSO  
39.72 DMSO  
39.51 DMSO

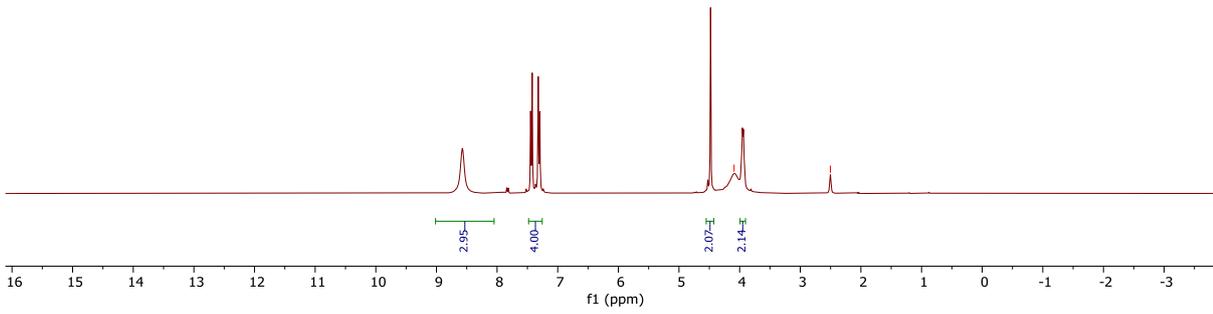


200914.337.10.fid  
Zhuang ma, Zm590  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2009 37

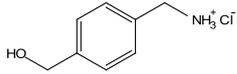


4.09 H2O

2.50 DMSO



200914.337.11.fid  
Zhuang ma, Zm590  
Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2009 37



143.31

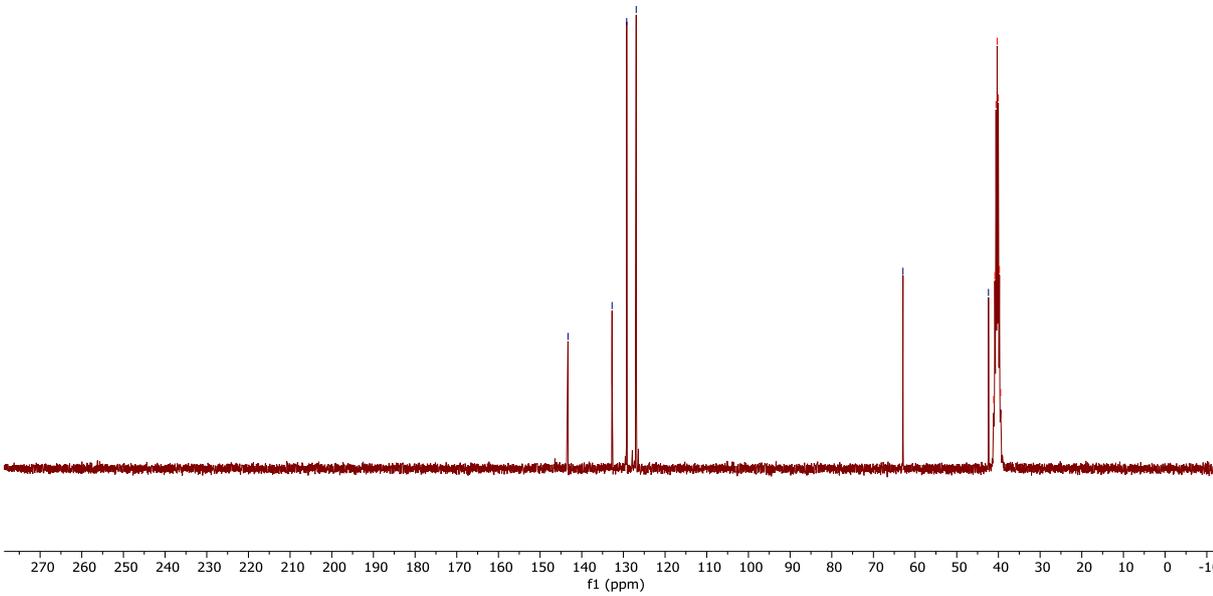
132.73

129.22

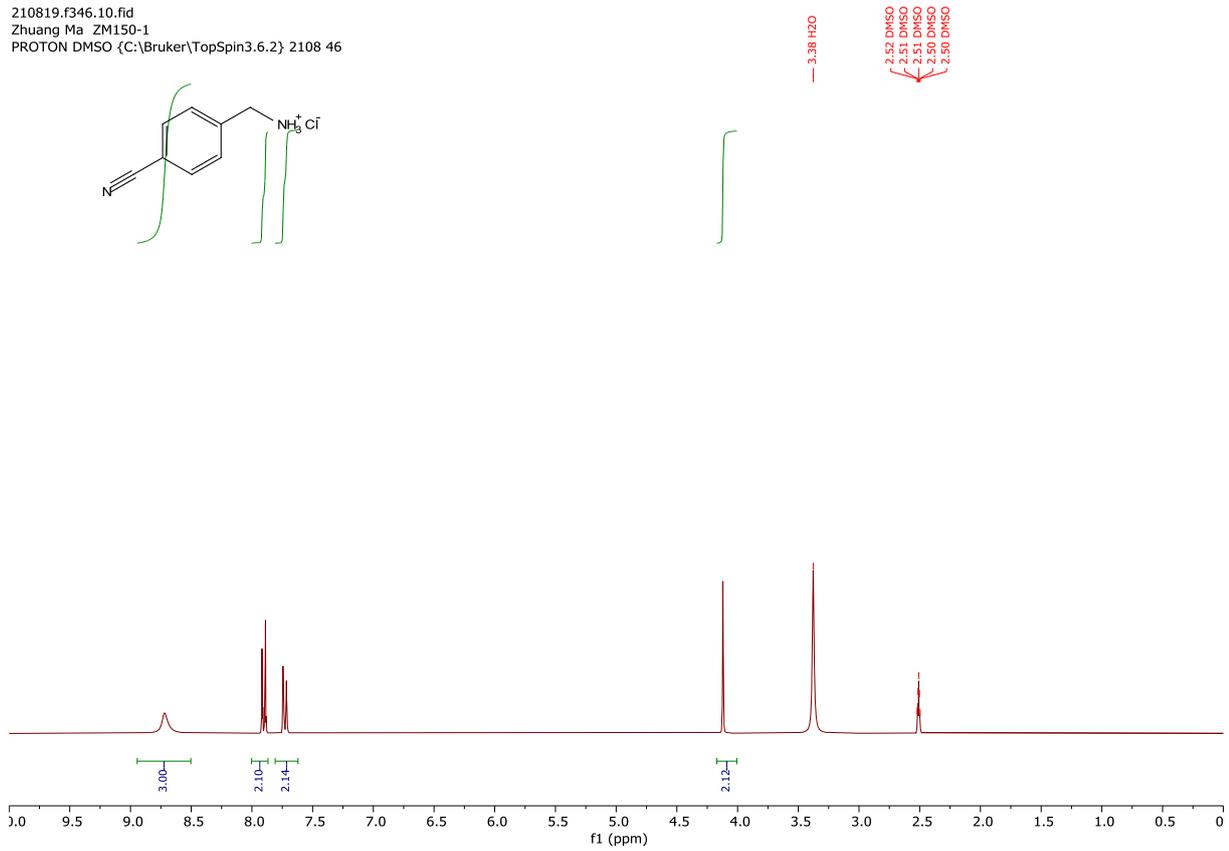
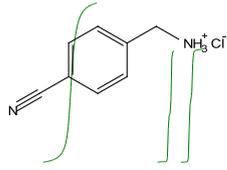
126.93

62.91

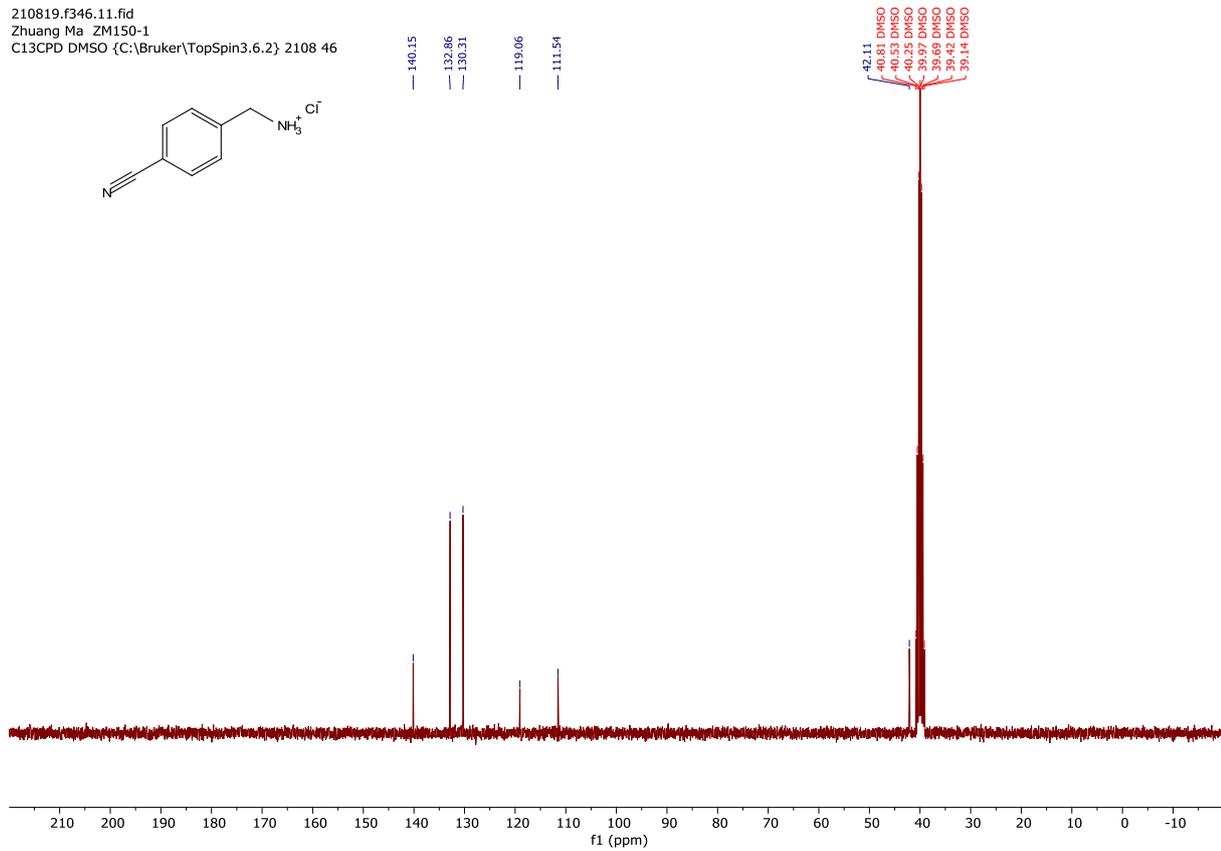
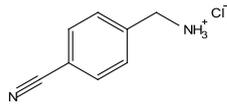
42.39  
41.14 DMSO  
40.86 DMSO  
40.33 DMSO  
40.03 DMSO  
39.75 DMSO  
39.47 DMSO



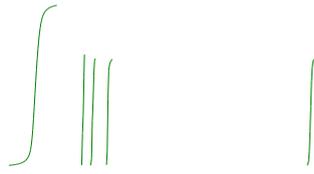
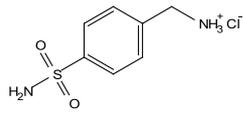
210819.f346.10.fid  
Zhuang Ma\_ZM150-1  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2108 46



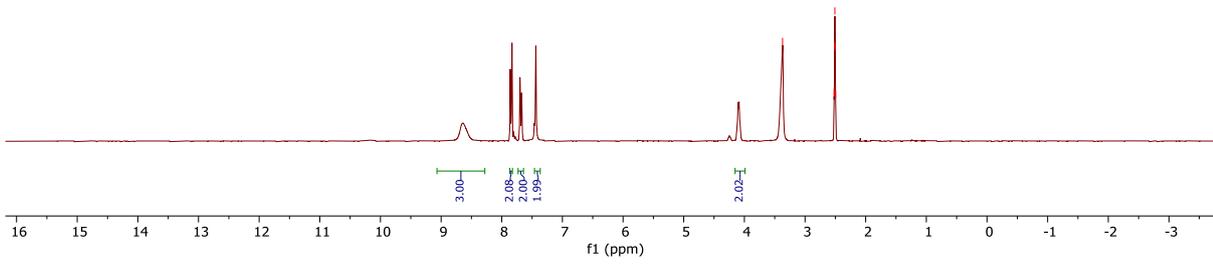
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Zhuang Ma\_ZM150-1  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2108 46



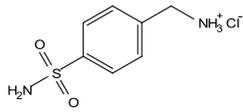
200511.f321.10.fid  
Zhuang Ma ZM-537  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2005 21



3.37 H2O  
2.52 DMSO  
2.51 DMSO  
2.51 DMSO  
2.50 DMSO  
2.50 DMSO

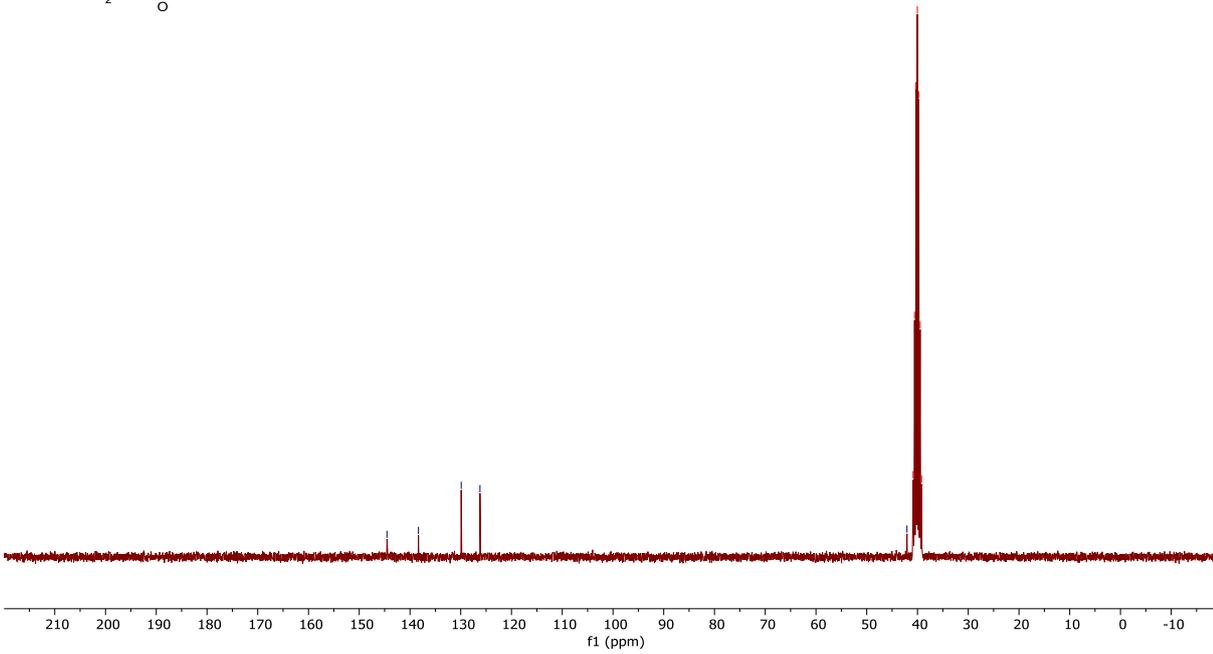


200511.f321.11.fid  
Zhuang Ma ZM-537  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2005 21

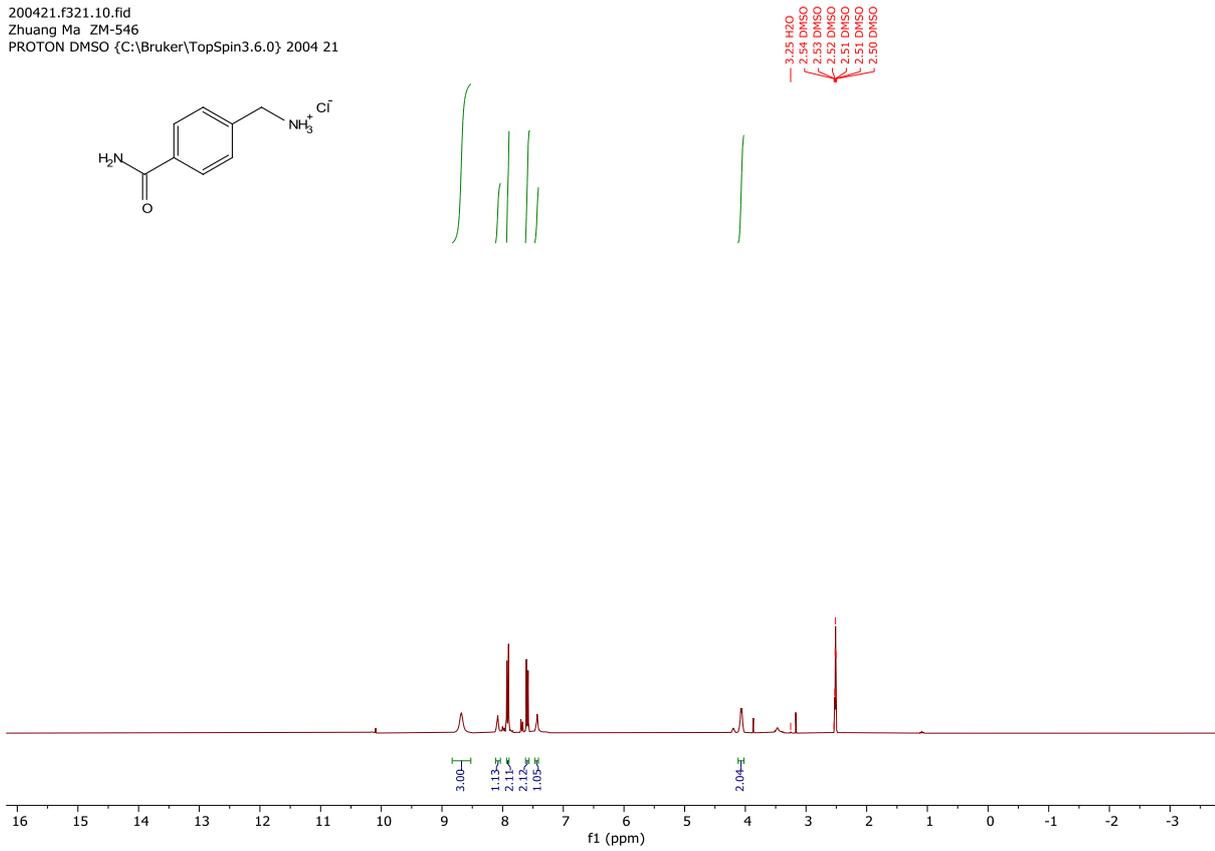
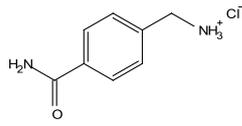


144.54  
138.32  
129.90  
126.20

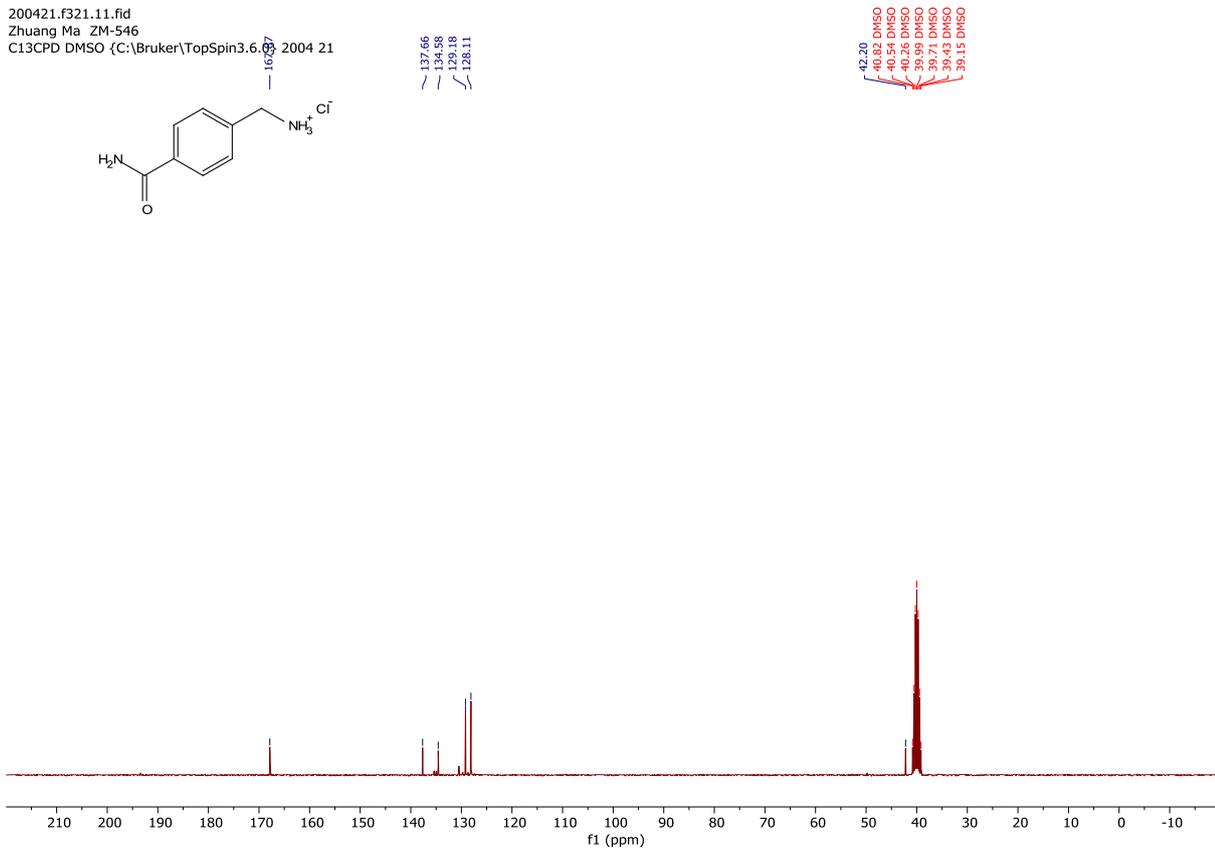
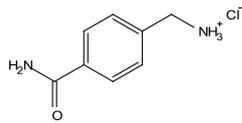
42.07  
40.86 DMSO  
40.58 DMSO  
40.58 DMSO  
40.02 DMSO  
39.74 DMSO  
39.47 DMSO  
39.19 DMSO



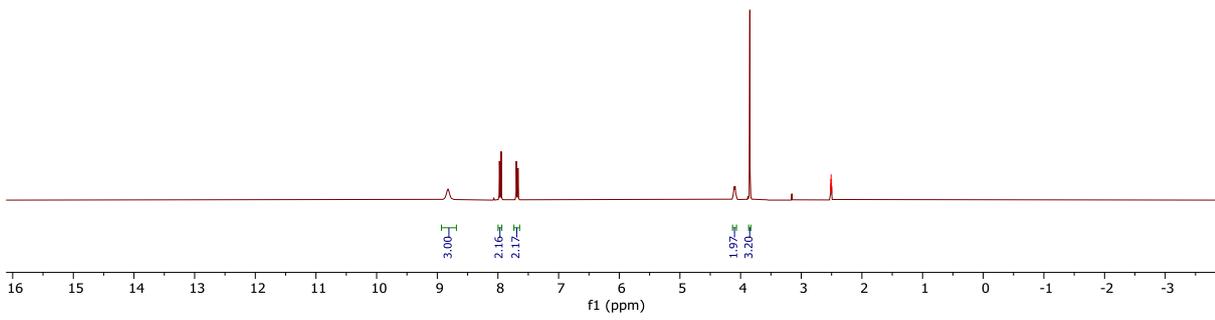
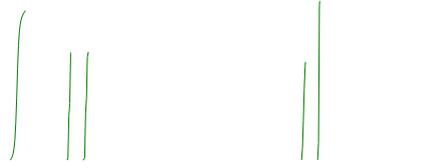
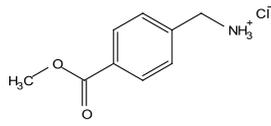
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Zhuang Ma\_ZM-546  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 21



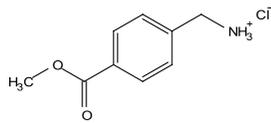
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Zhuang Ma\_ZM-546  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 21



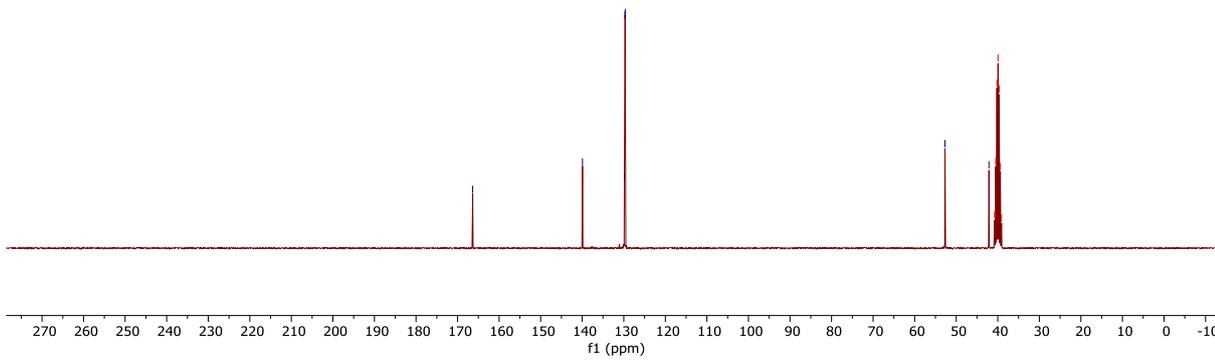
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Zhuang Ma ZM-551  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 26



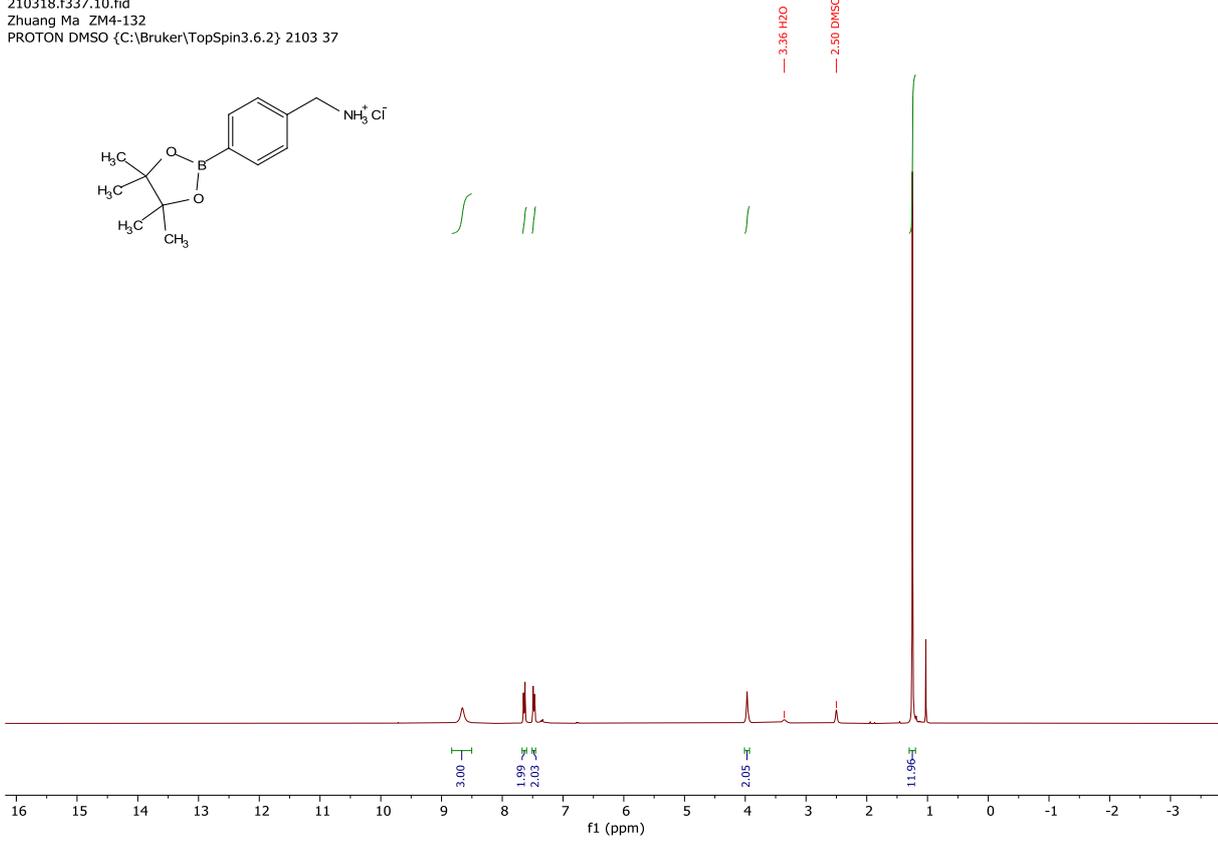
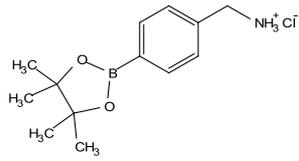
200421.f326.11.fid  
Zhuang Ma ZM-551  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 26



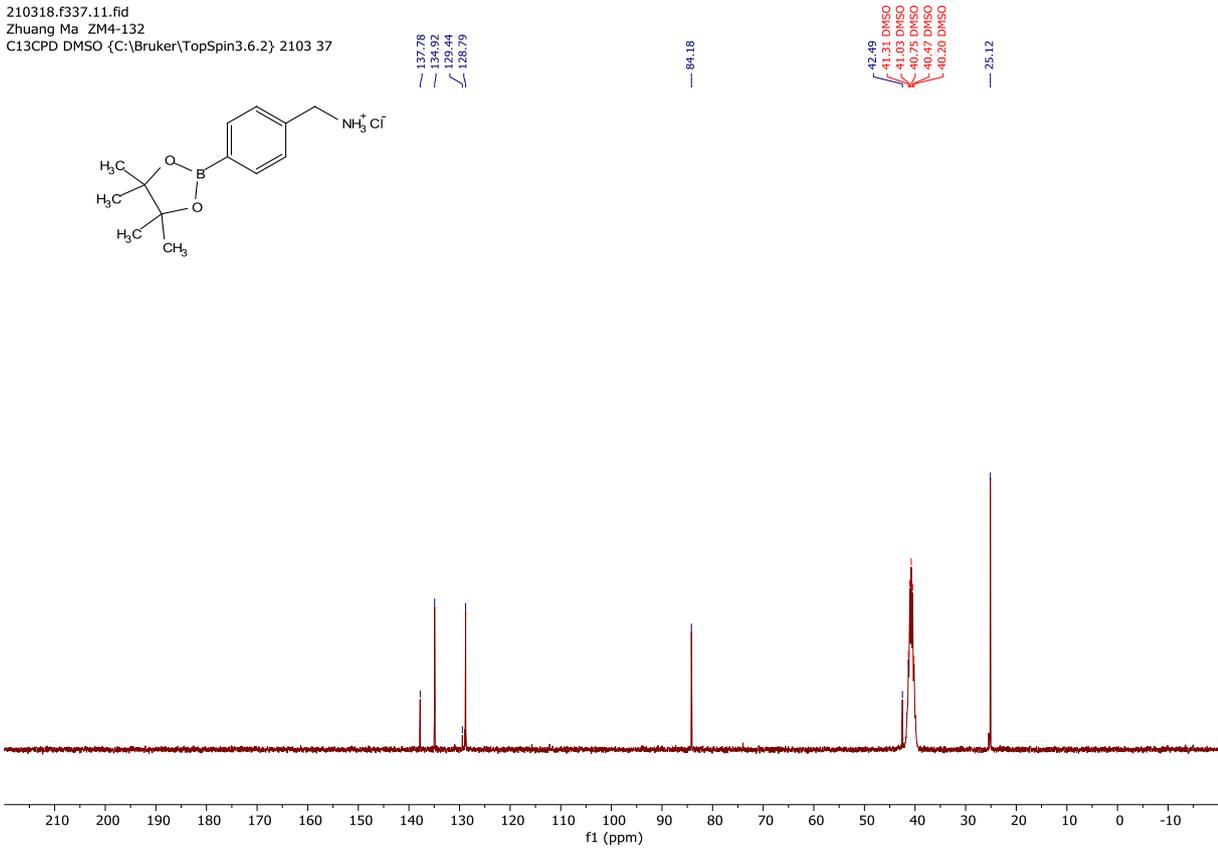
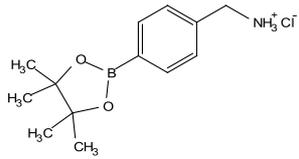
166.37  
139.93  
129.87  
129.72  
129.66  
52.71  
42.19  
40.46  
40.22  
39.94  
39.66  
39.38  
39.11



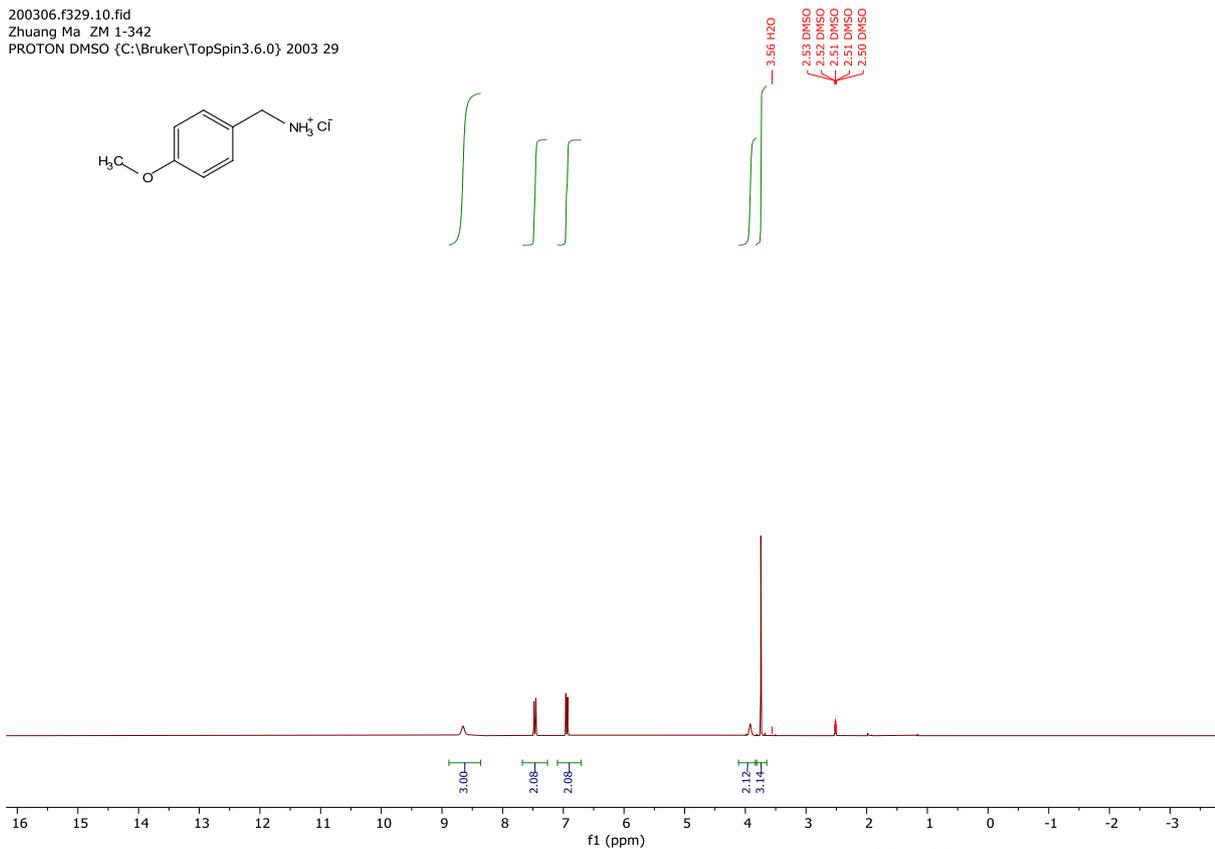
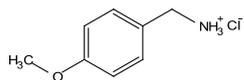
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Zhuang Ma ZM4-132  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2103 37



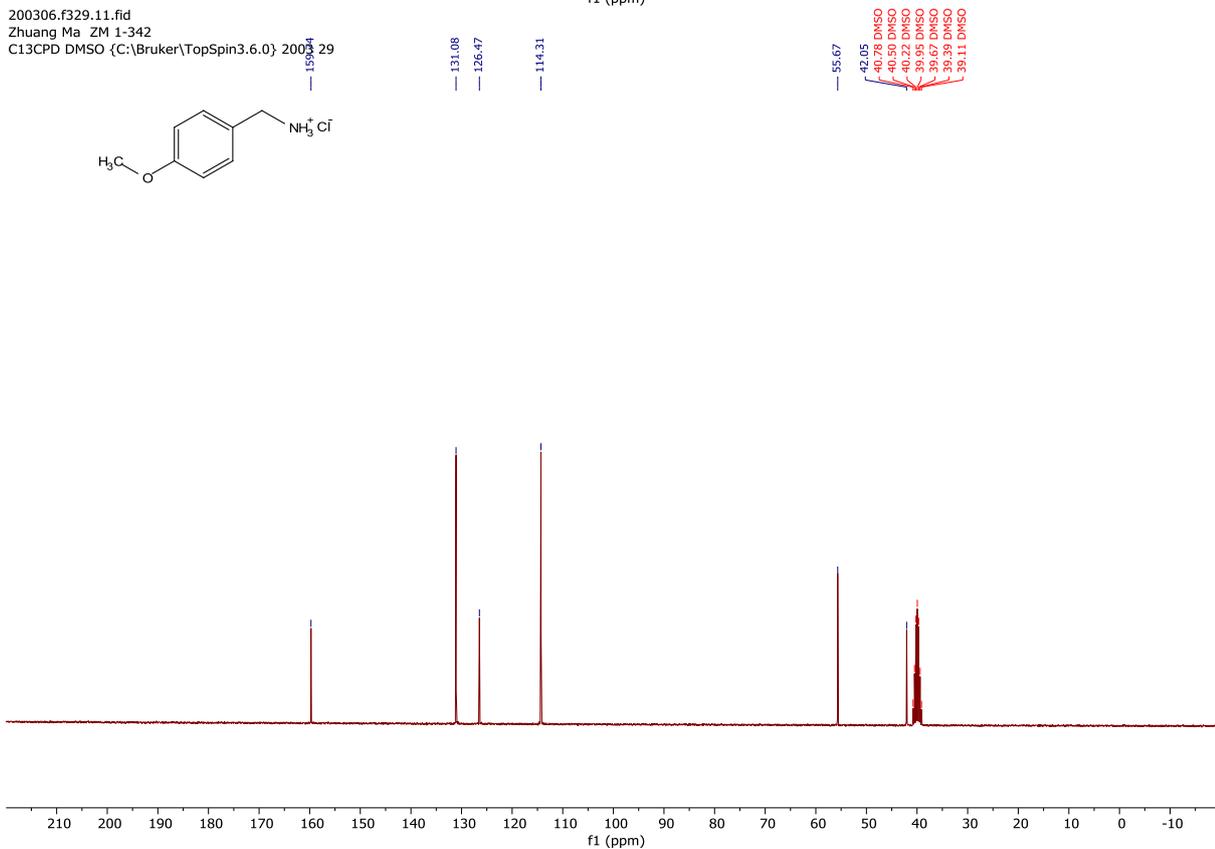
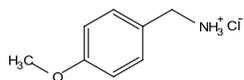
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C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2103 37



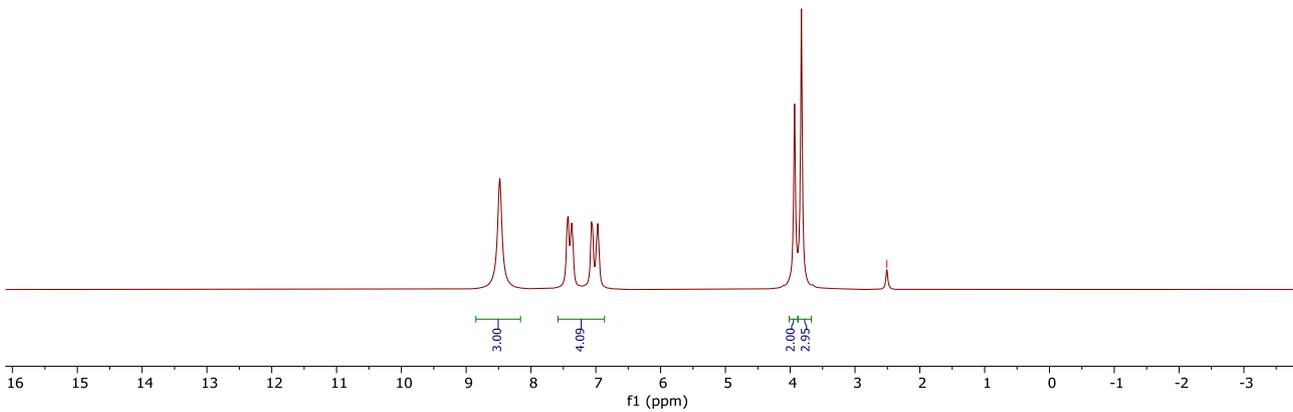
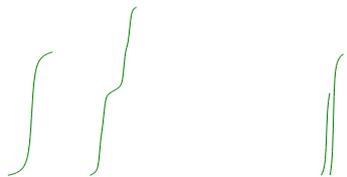
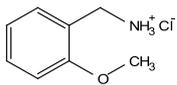
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Zhuang Ma\_ZM 1-342  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 29



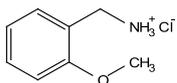
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Zhuang Ma\_ZM 1-342  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 29



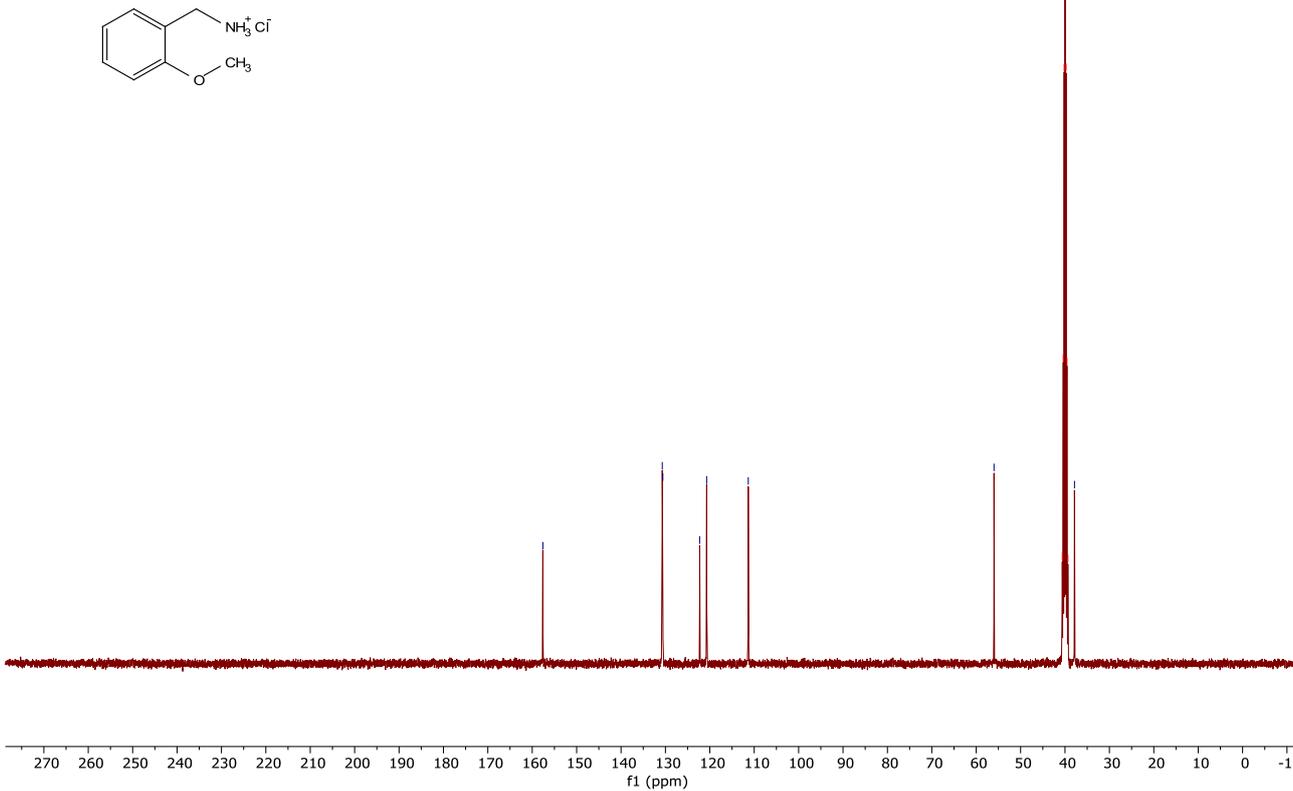
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Zhuang ZM-395  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 1911 14



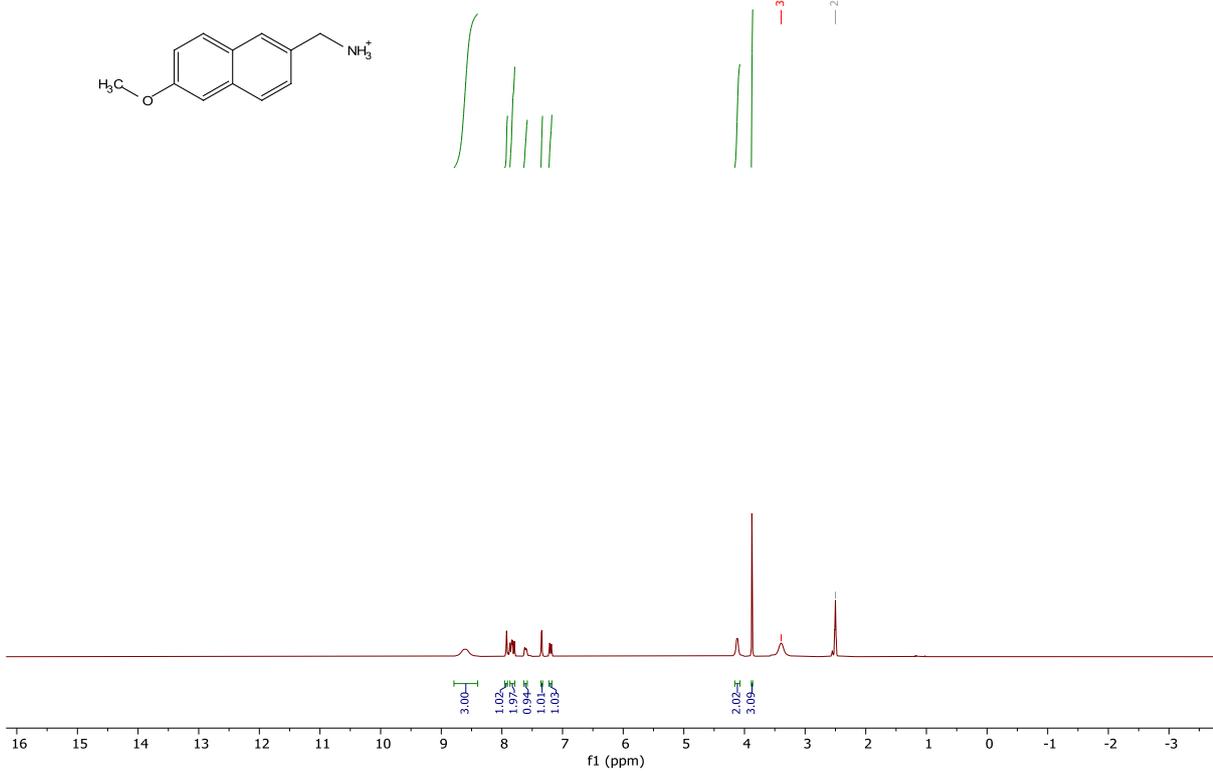
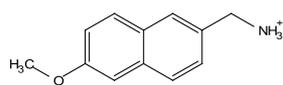
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Zhuang ZM-395  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 1911 14



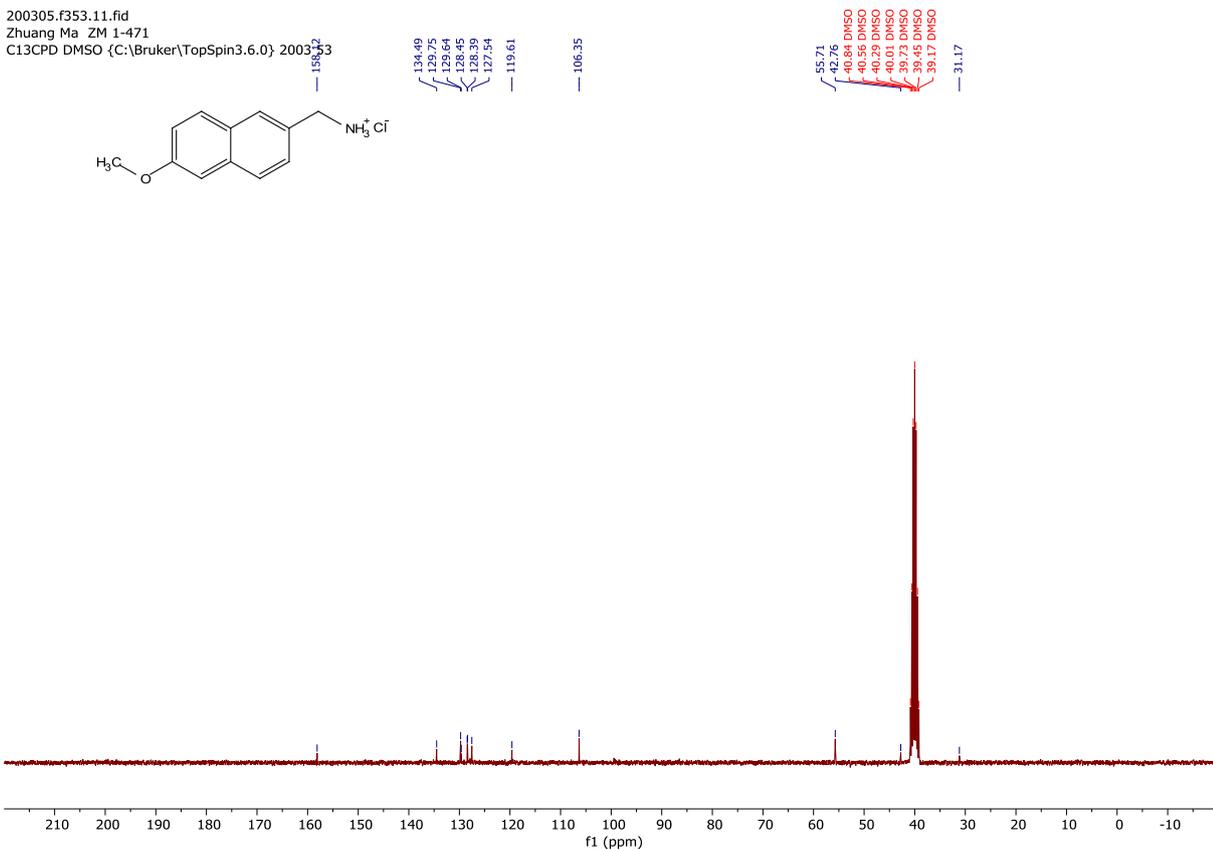
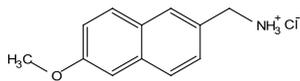
157.61  
130.71  
130.58  
122.29  
120.71  
111.34  
55.99  
40.60 DMSO  
40.39 DMSO  
40.18 DMSO  
39.98 DMSO  
39.77 DMSO  
39.56 DMSO  
39.35 DMSO  
37.86



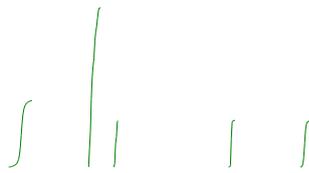
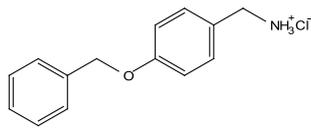
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Zhuang Ma ZM-549  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 24



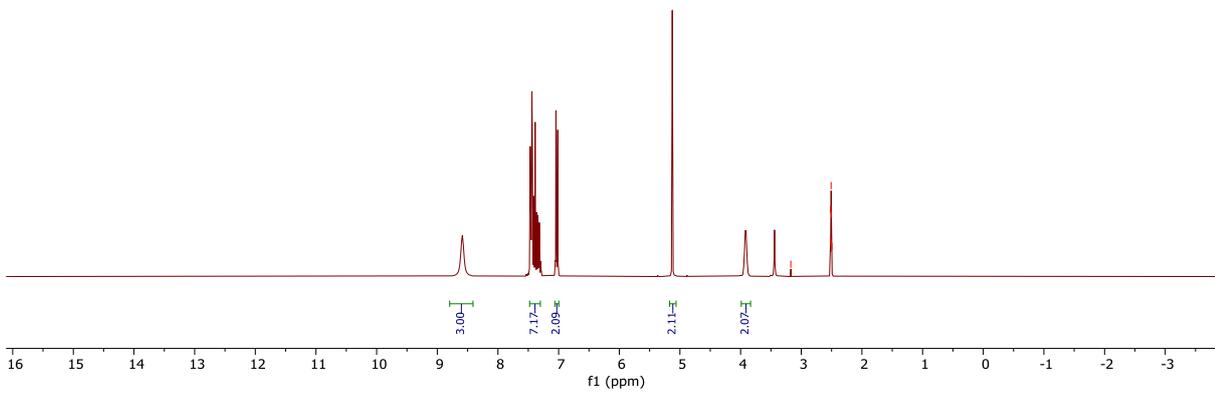
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 53



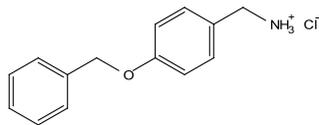
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Ma/ ZM 1-503  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 31



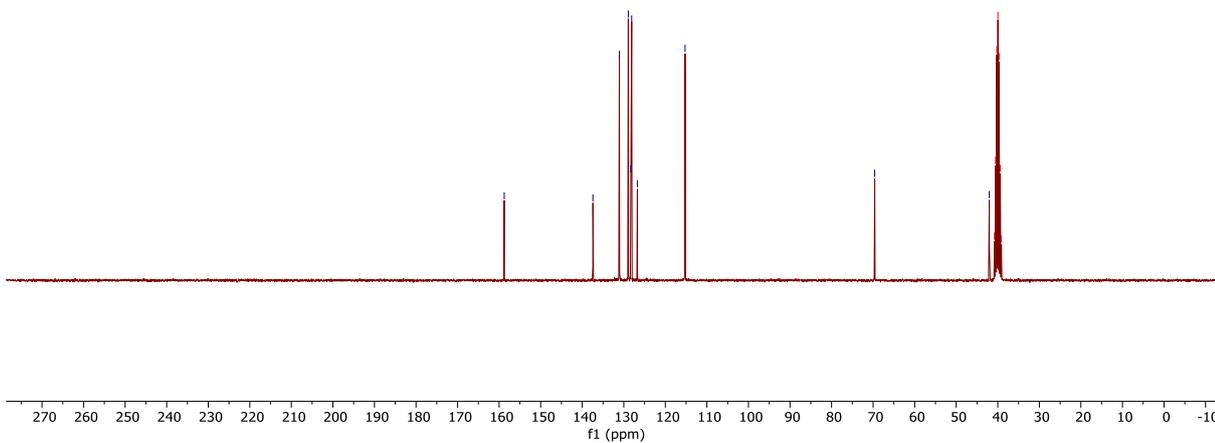
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2.52 DMSO  
2.51 DMSO  
2.51 DMSO  
2.50 DMSO  
2.38 DMSO



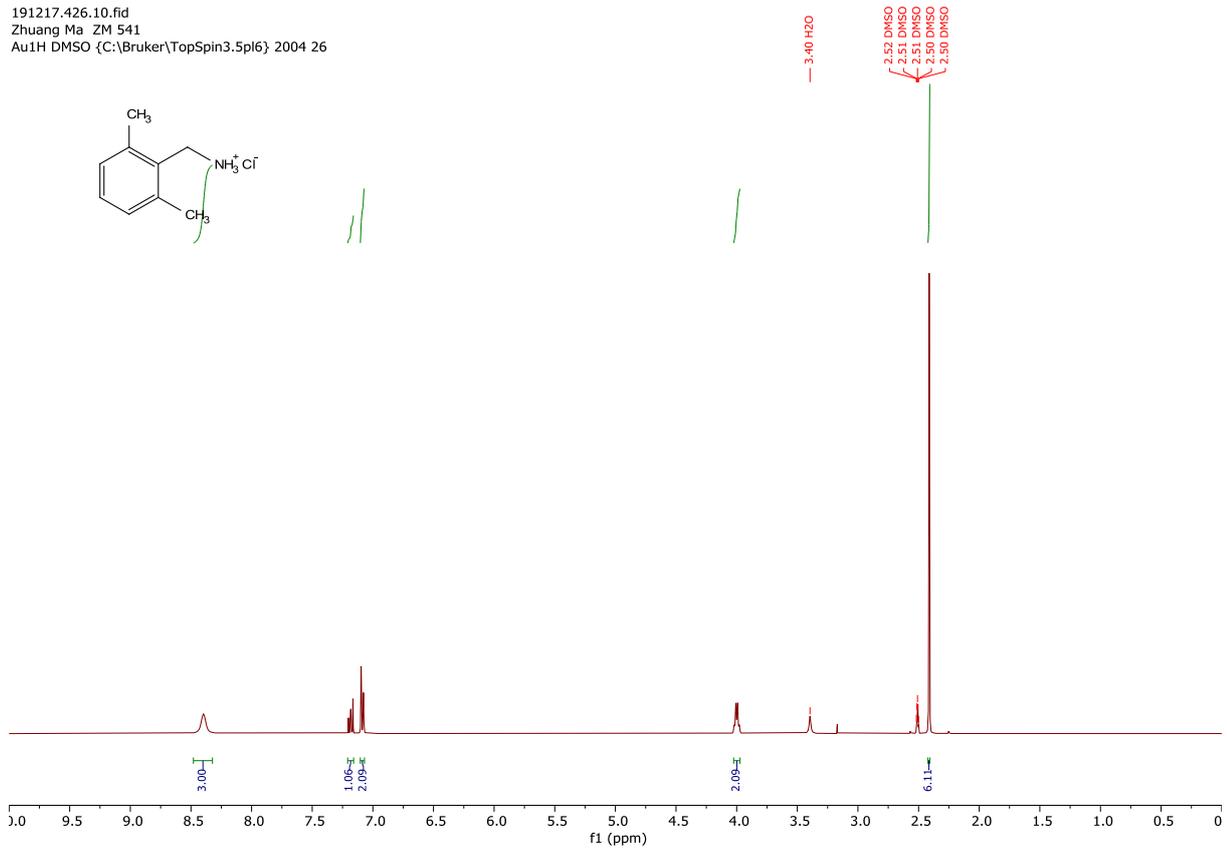
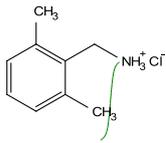
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Ma/ ZM 1-503  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 31



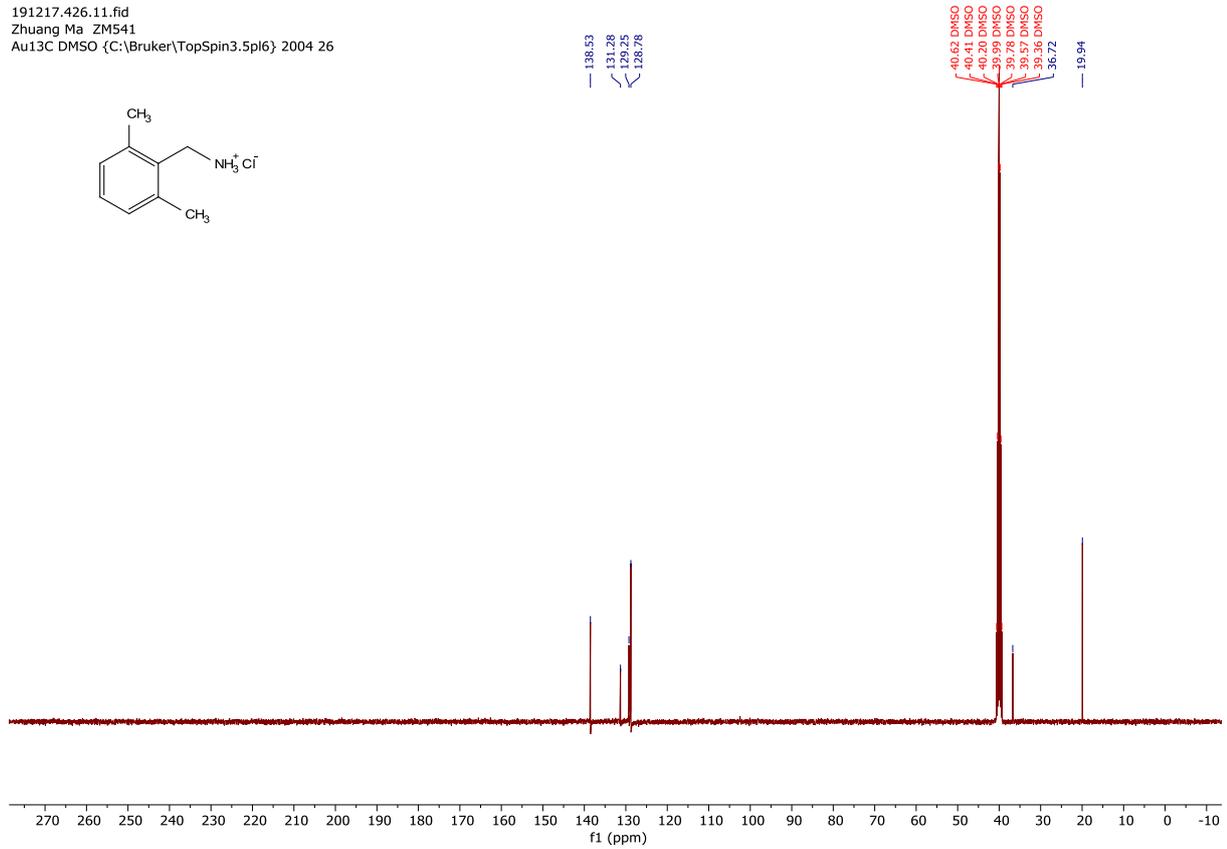
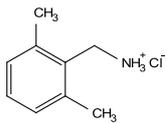
158.77  
137.44  
131.07  
128.92  
128.30  
126.73  
115.25  
68.62  
42.04  
40.83 DMSO  
40.52 DMSO  
39.97 DMSO  
39.69 DMSO  
39.41 DMSO  
39.14 DMSO



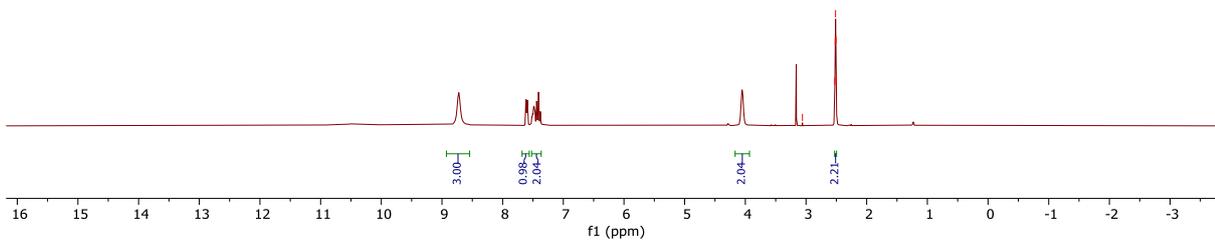
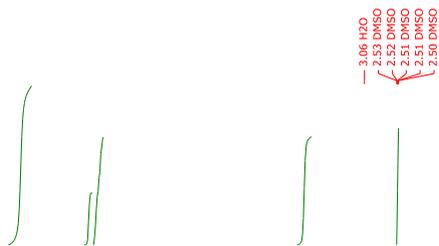
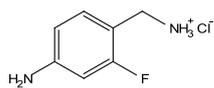
191217.426.10.fid  
Zhuang Ma ZM 541  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 26



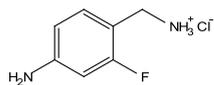
191217.426.11.fid  
Zhuang Ma ZM541  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2004 26



200421.f322.10.fid  
Zhuang Ma ZM-547  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 22

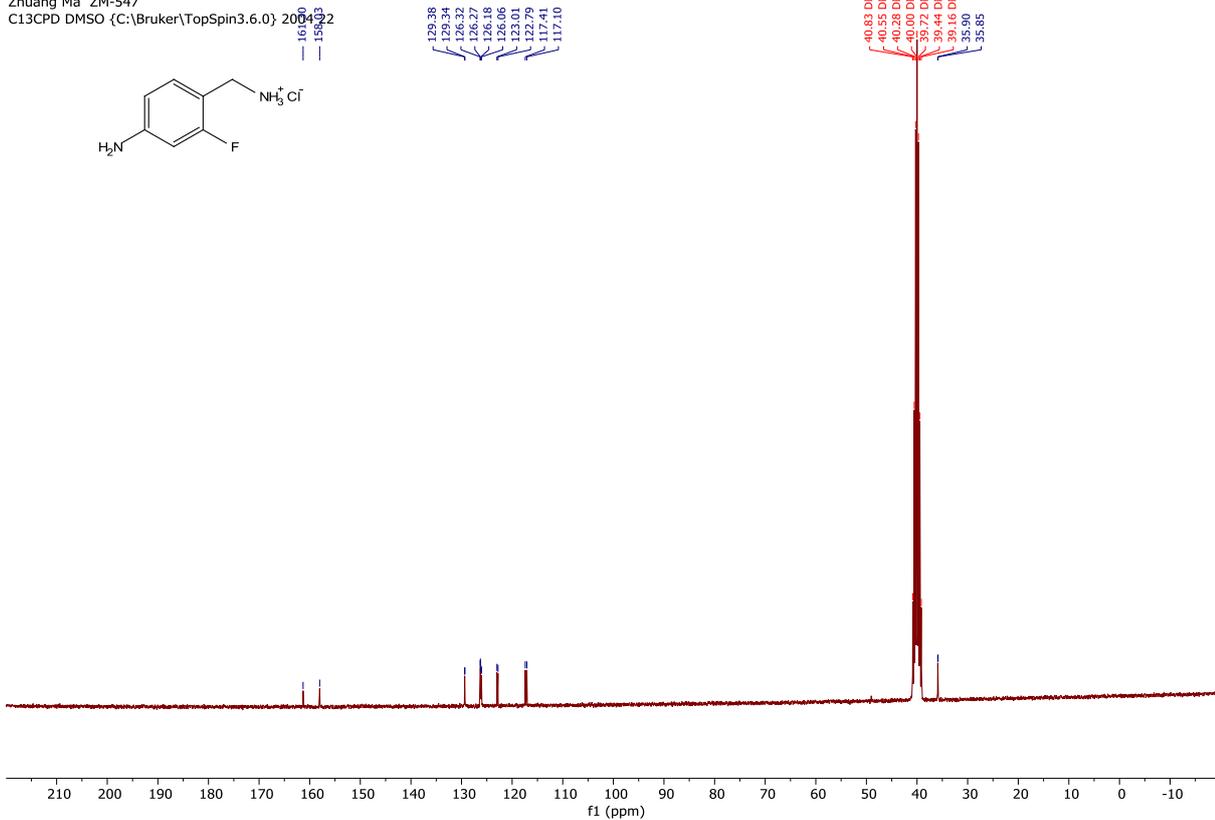


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Zhuang Ma ZM-547  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 22

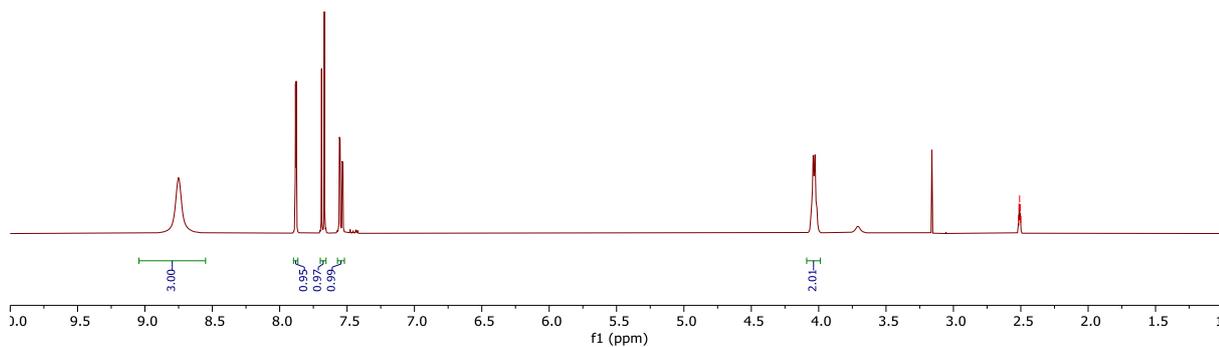
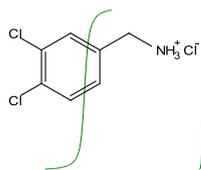


129.38  
128.74  
126.37  
126.27  
126.18  
126.06  
123.01  
122.79  
117.91  
117.10

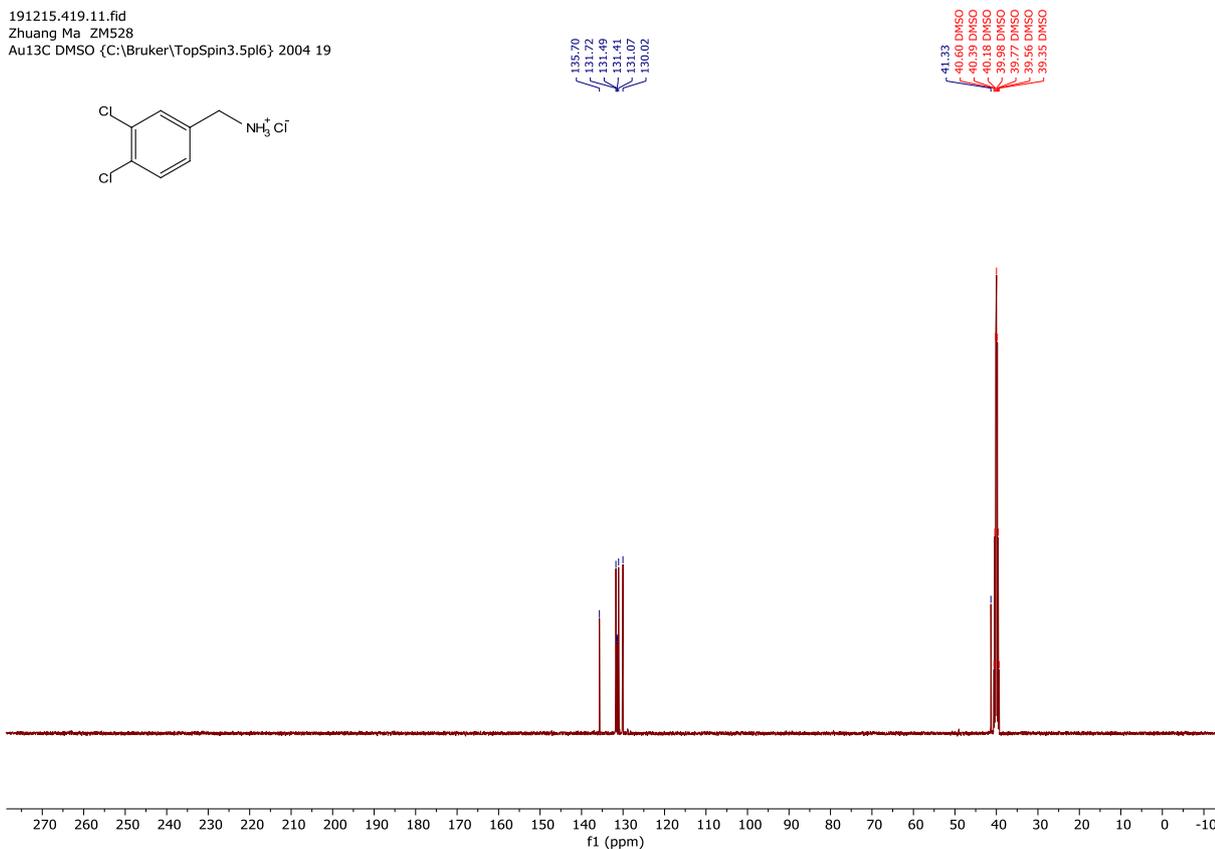
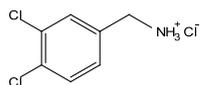
40.83 DMSO  
40.55 DMSO  
40.28 DMSO  
40.00 DMSO  
39.74 DMSO  
39.44 DMSO  
39.15 DMSO  
35.90  
35.85



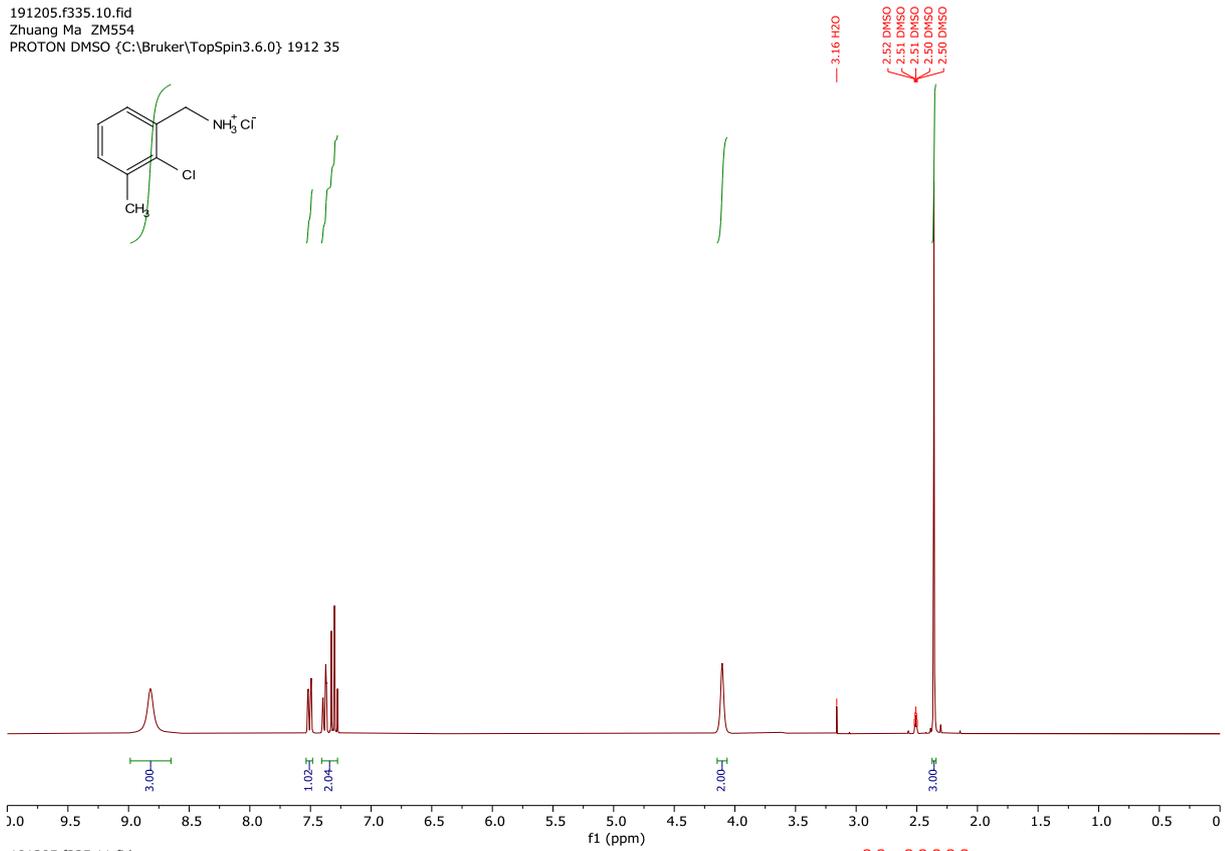
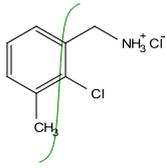
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Zhuang Ma ZM528  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 19



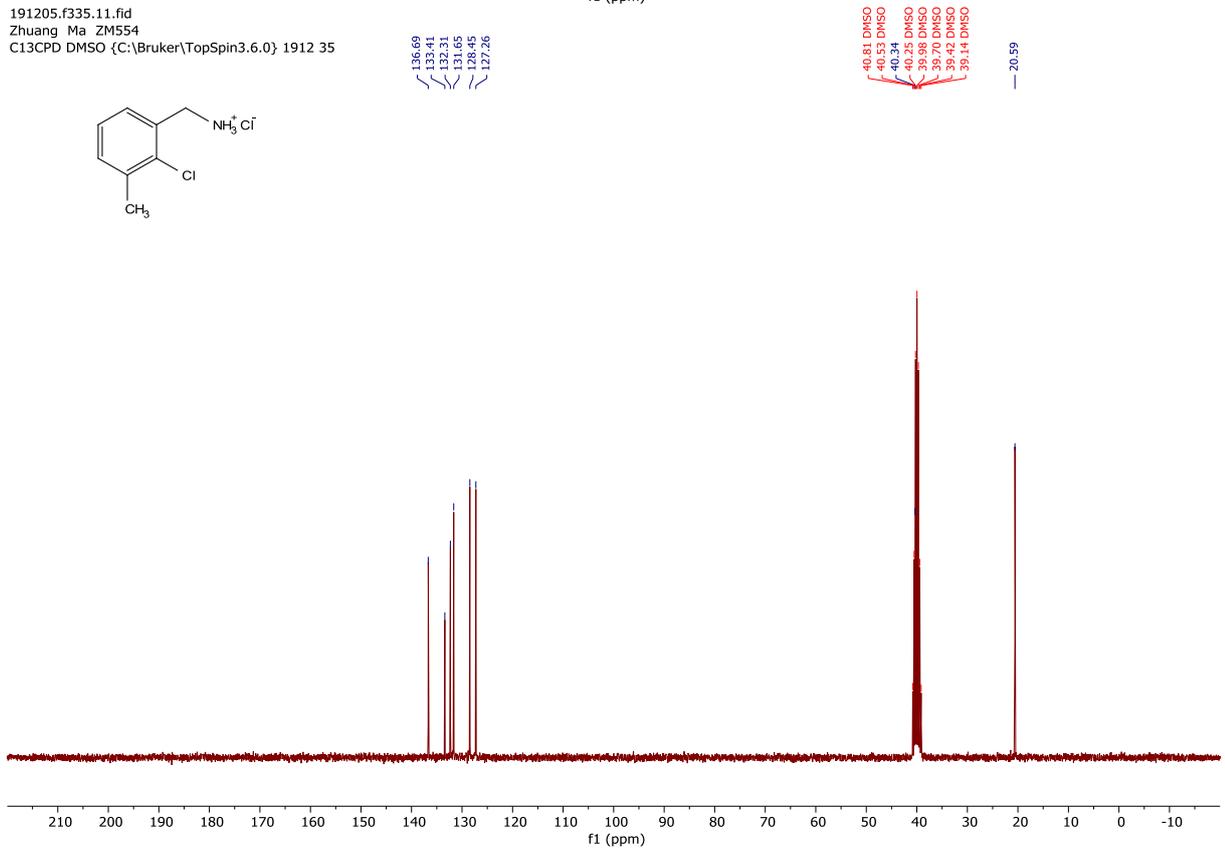
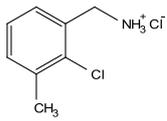
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Zhuang Ma ZM528  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2004 19



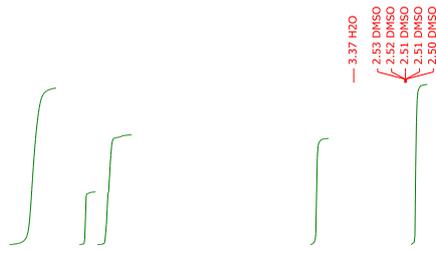
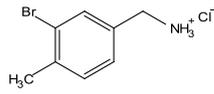
191205.f335.10.fid  
Zhuang Ma ZM554  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 1912 35



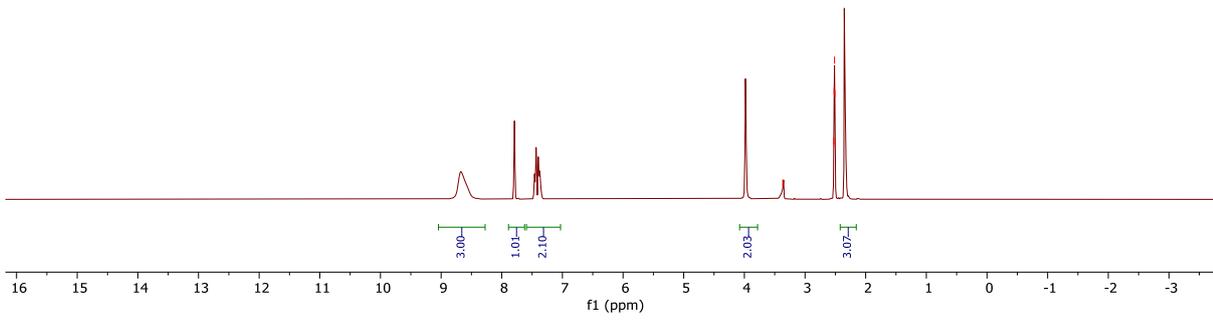
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 1912 35



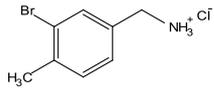
200305.f355.10.fid  
Zhuang Ma\_ZM 1-453  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 55



3.37 H2O  
2.53 DMSO  
2.52 DMSO  
2.51 DMSO  
2.50 DMSO



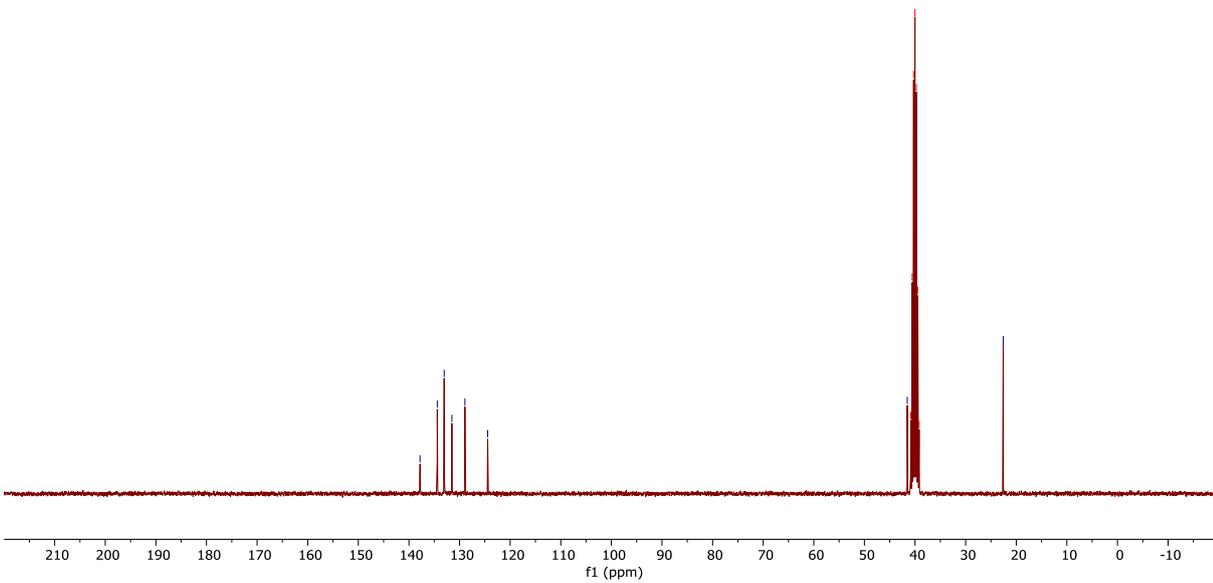
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Zhuang Ma\_ZM 1-453  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 55



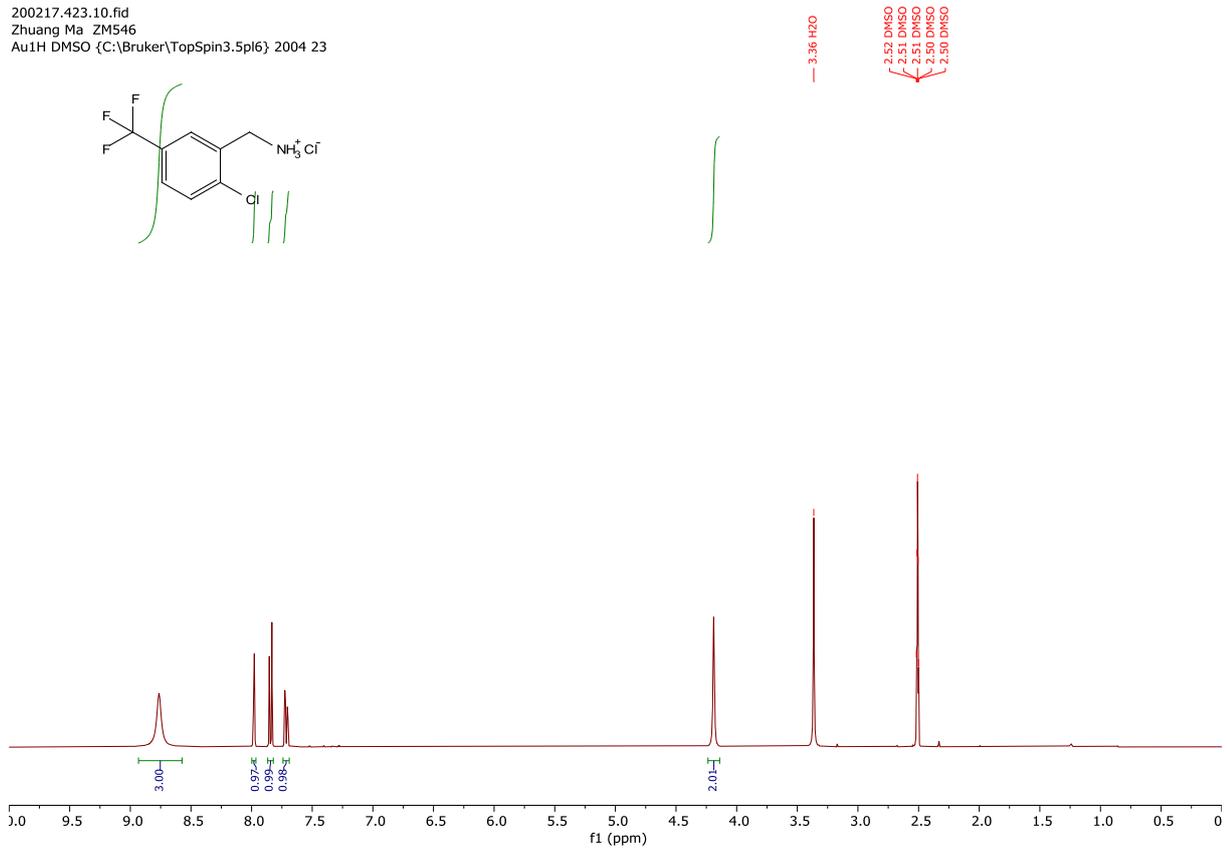
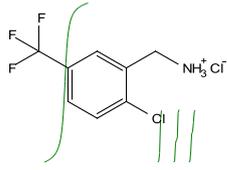
137.80  
134.40  
133.03  
128.91  
128.44

41.53  
40.84 DMSO  
40.57 DMSO  
40.32 DMSO  
40.01 DMSO  
39.73 DMSO  
39.46 DMSO  
39.18 DMSO

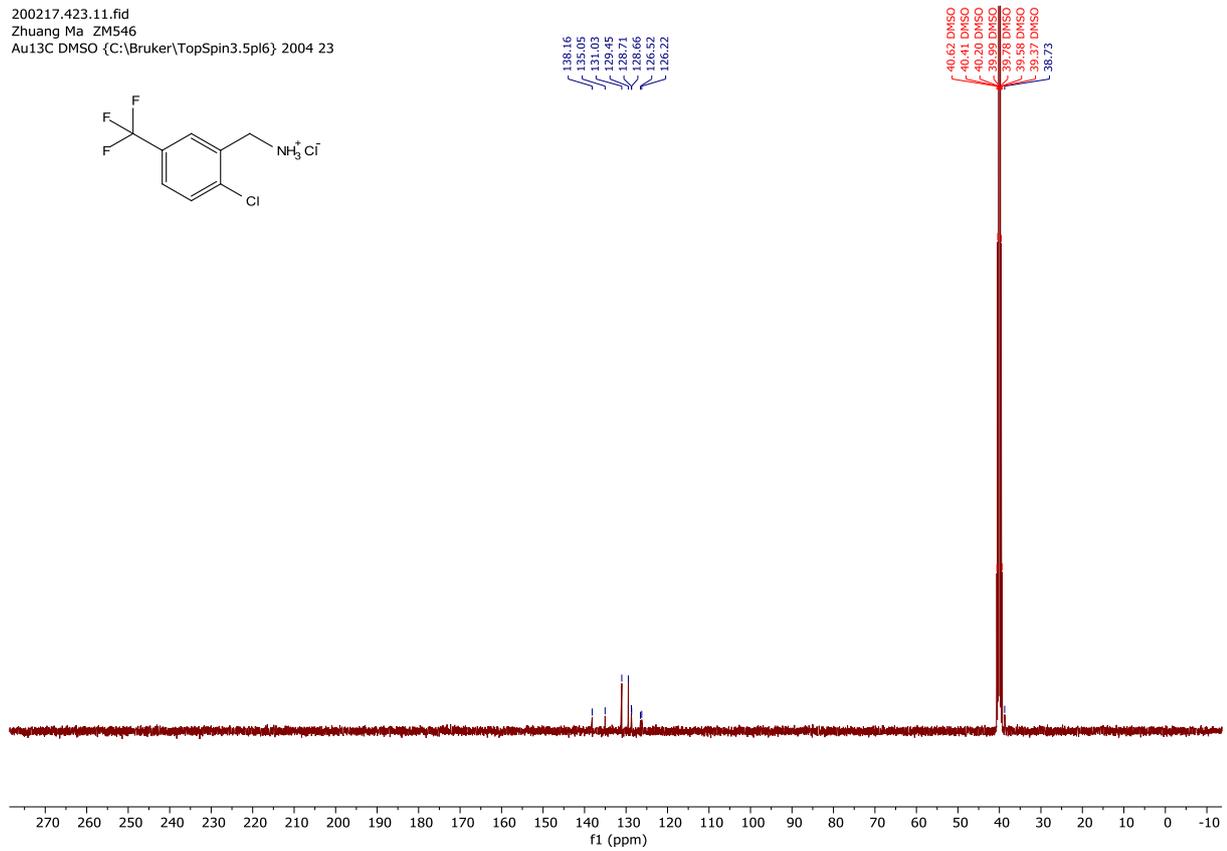
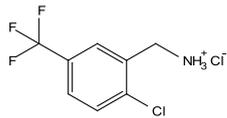
22.56



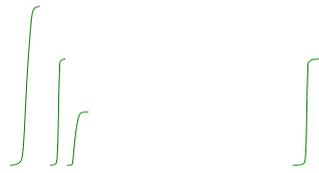
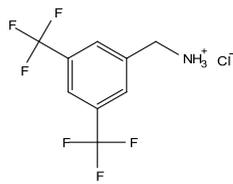
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Zhuang Ma ZM546  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 23



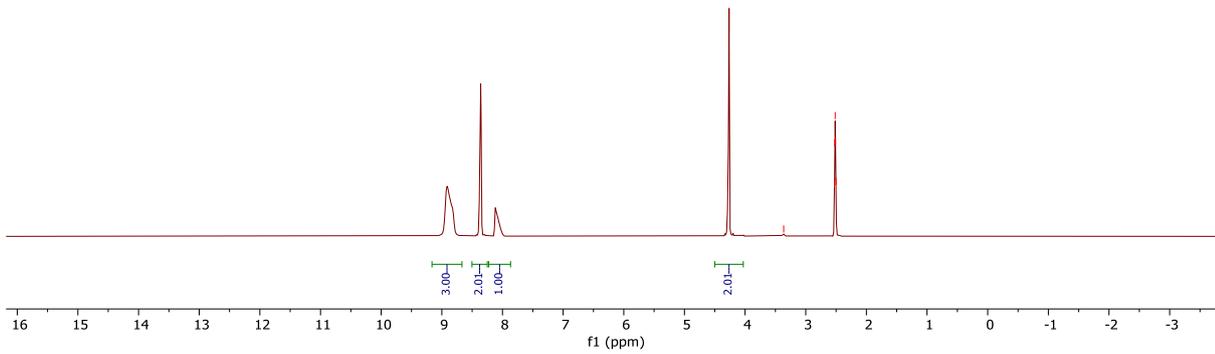
200217.423.11.fid  
Zhuang Ma ZM546  
Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2004 23



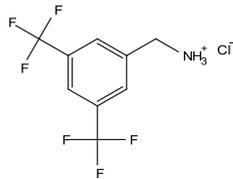
200303.f326.10.fid  
Ma/ ZM 1-311  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 26



3.36 H2O  
2.53 DMSO  
2.52 DMSO  
2.51 DMSO  
2.50 DMSO

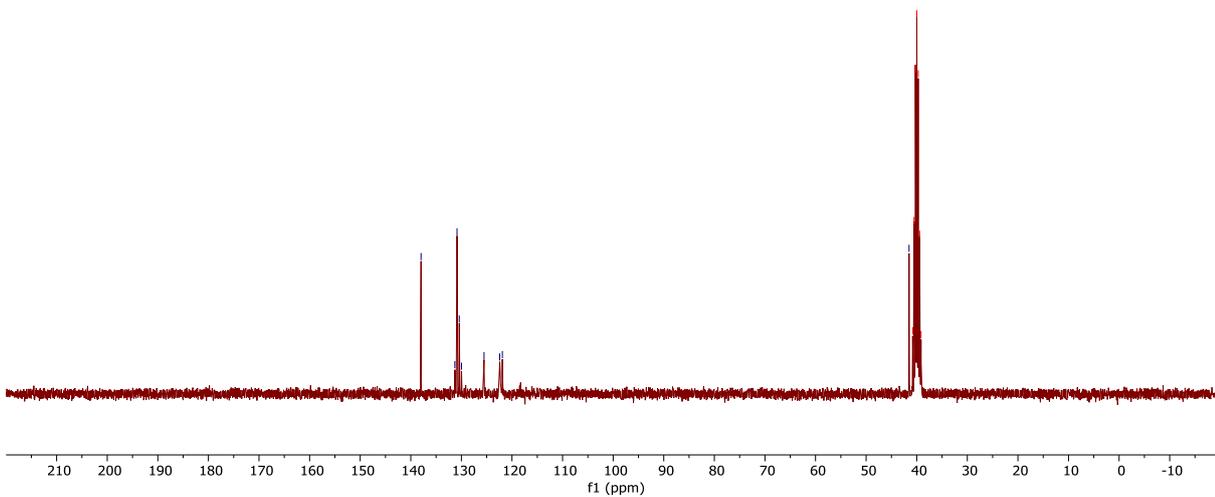


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Ma/ ZM 1-311  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 26

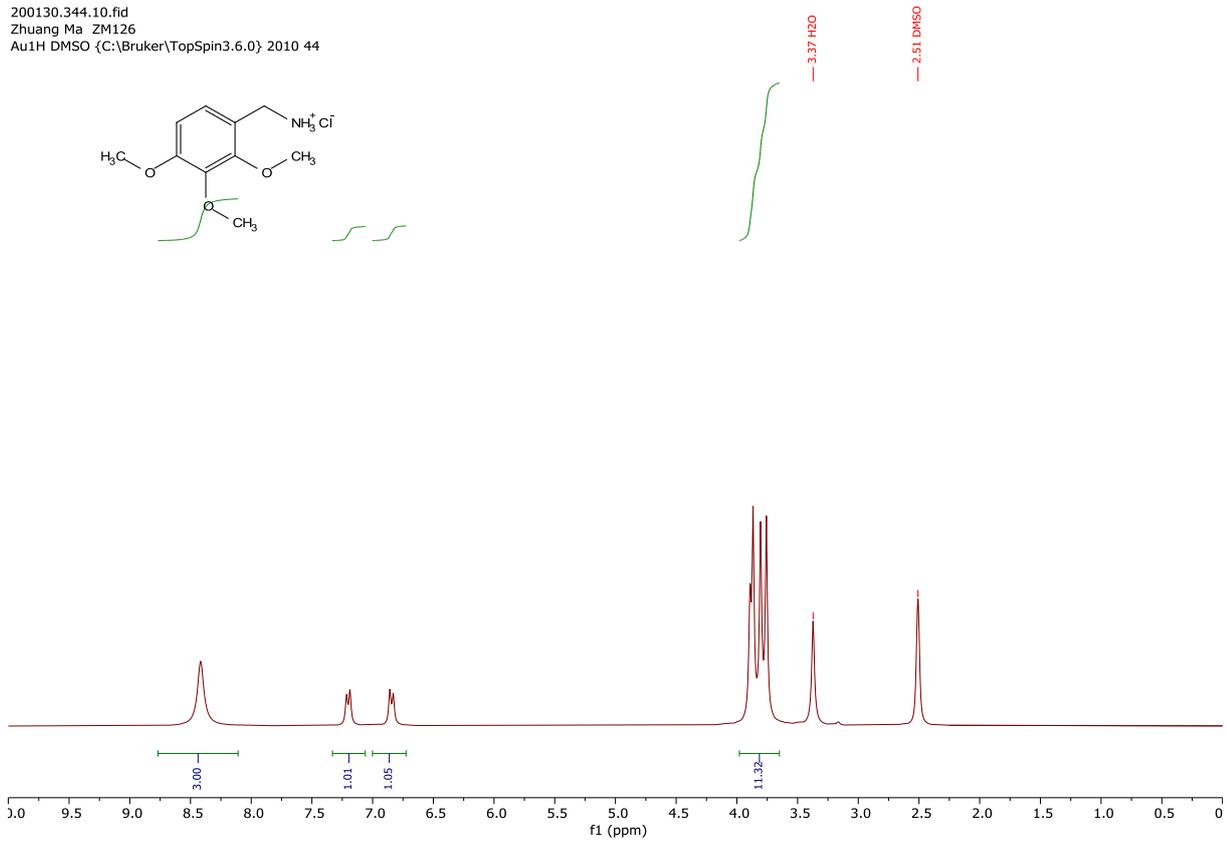
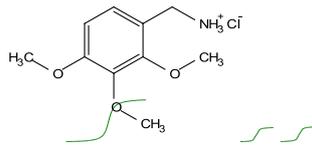


137.97  
131.29  
130.86  
130.41  
128.63  
125.62  
122.42  
121.90

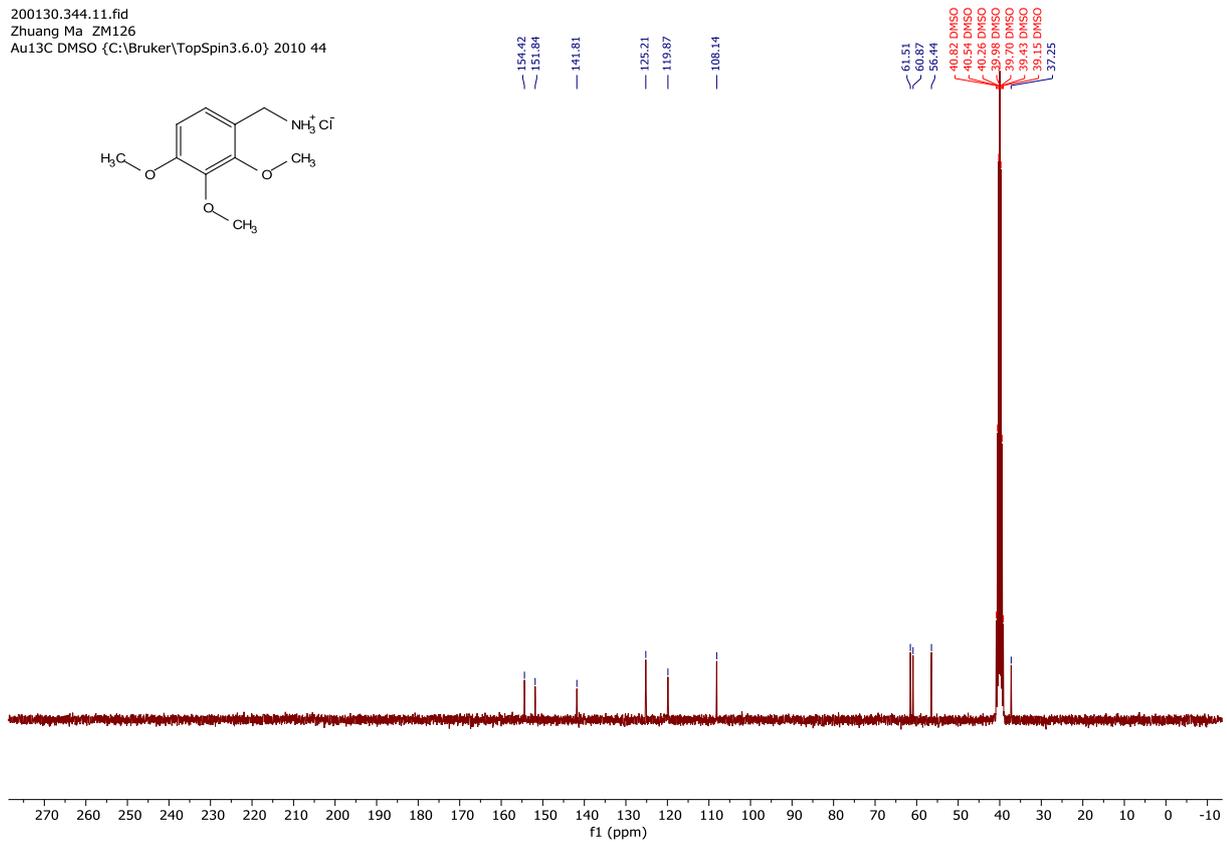
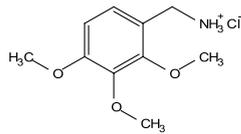
41.51  
40.81 DMSO  
40.52 DMSO  
40.32 DMSO  
39.98 DMSO  
39.70 DMSO  
39.42 DMSO  
39.15 DMSO



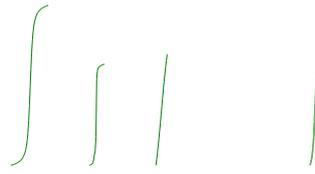
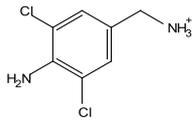
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Zhuang Ma ZM126  
Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2010 44



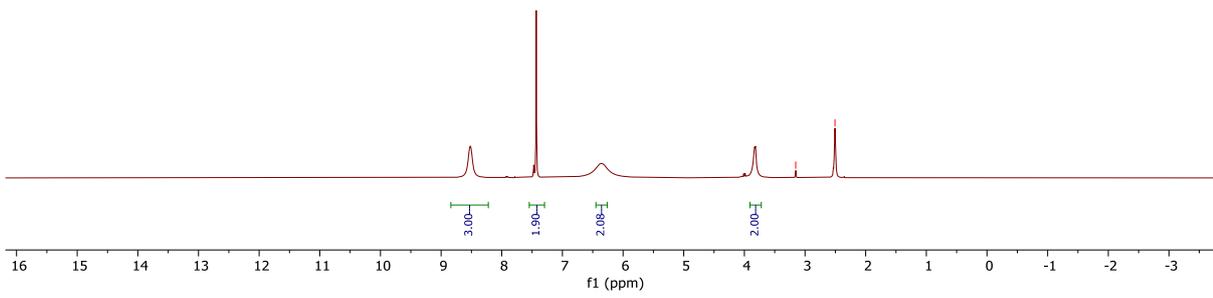
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Zhuang Ma ZM126  
Au13C DMSO {C:\Bruker\TopSpin3.6.0} 2010 44



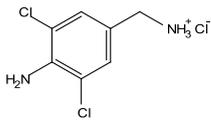
200421.f324.10.fid  
Zhuang Ma ZM-549  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 24



— 3.15 H<sub>2</sub>O  
— 2.51

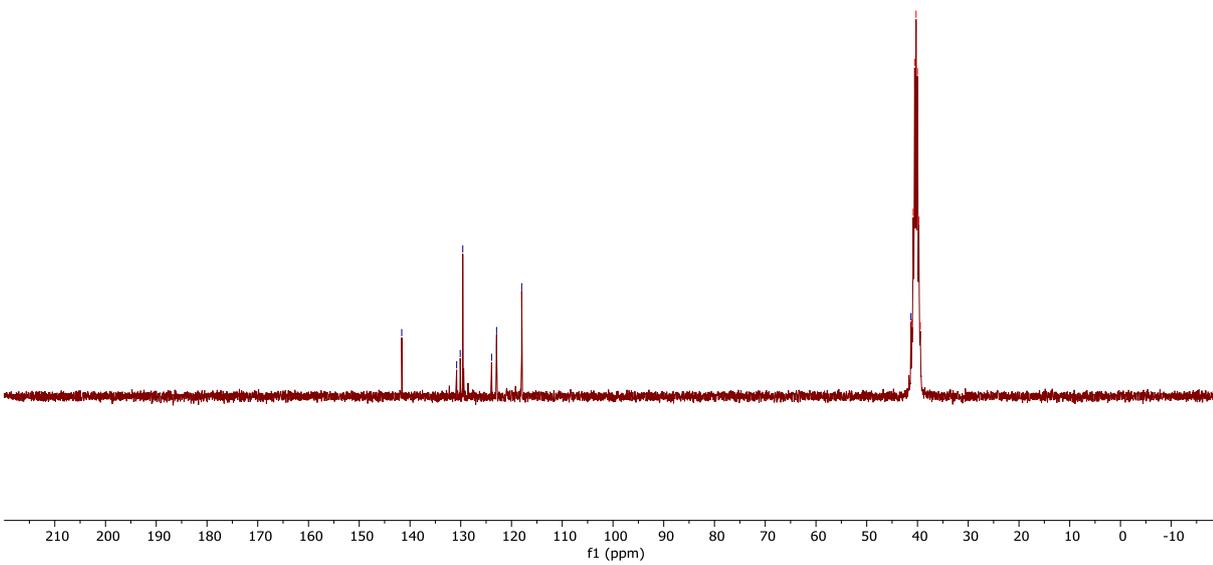


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Zhuang Ma ZM4-117  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2103 34

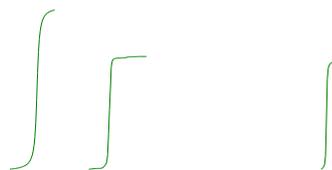
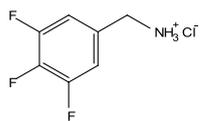


— 141.63  
— 130.80  
— 129.59  
— 123.02  
— 122.94  
— 117.99

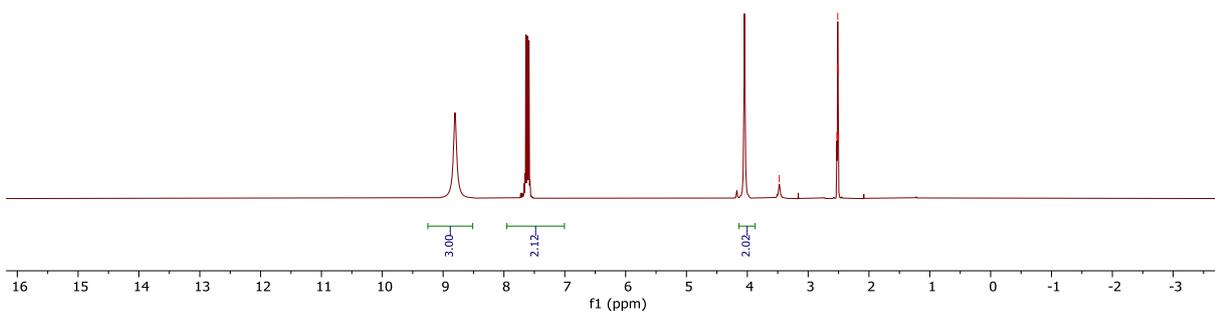
— 41.29  
— 41.10 DMSO  
— 40.83 DMSO  
— 40.55 DMSO  
— 40.27 DMSO  
— 40.00 DMSO  
— 39.72 DMSO  
— 39.44 DMSO



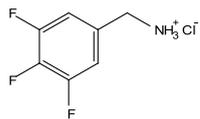
200305.f355.10.fid  
 Zhuang Ma\_ZM 1-308  
 PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 55



3.47 H2O  
 2.53 DMSO  
 2.52 DMSO  
 2.51 DMSO  
 2.50 DMSO

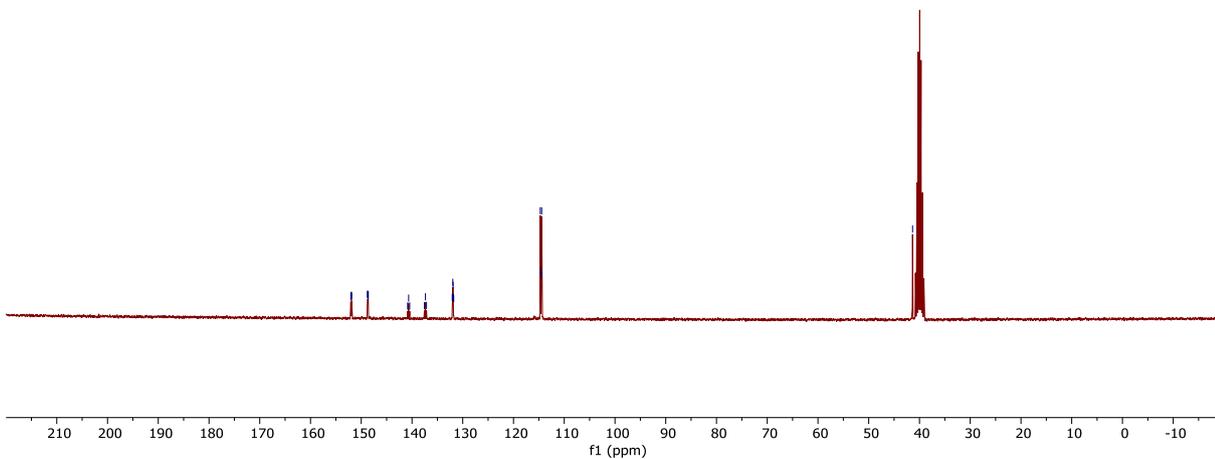


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 C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 55

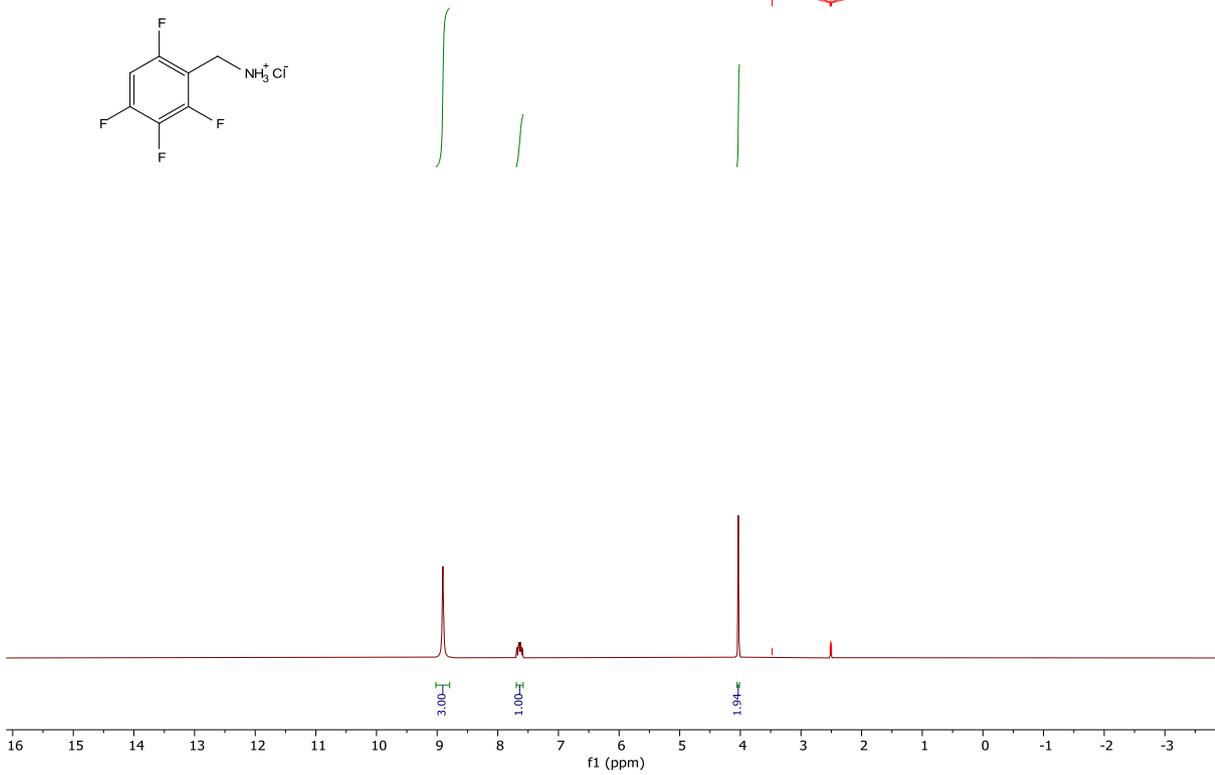
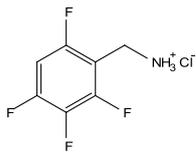


152.06  
 151.91  
 151.93  
 151.88  
 148.78  
 148.73  
 148.65  
 148.60  
 140.87  
 140.66  
 137.56  
 137.35  
 137.15  
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 131.84  
 131.78  
 114.72  
 114.64  
 114.53  
 114.44

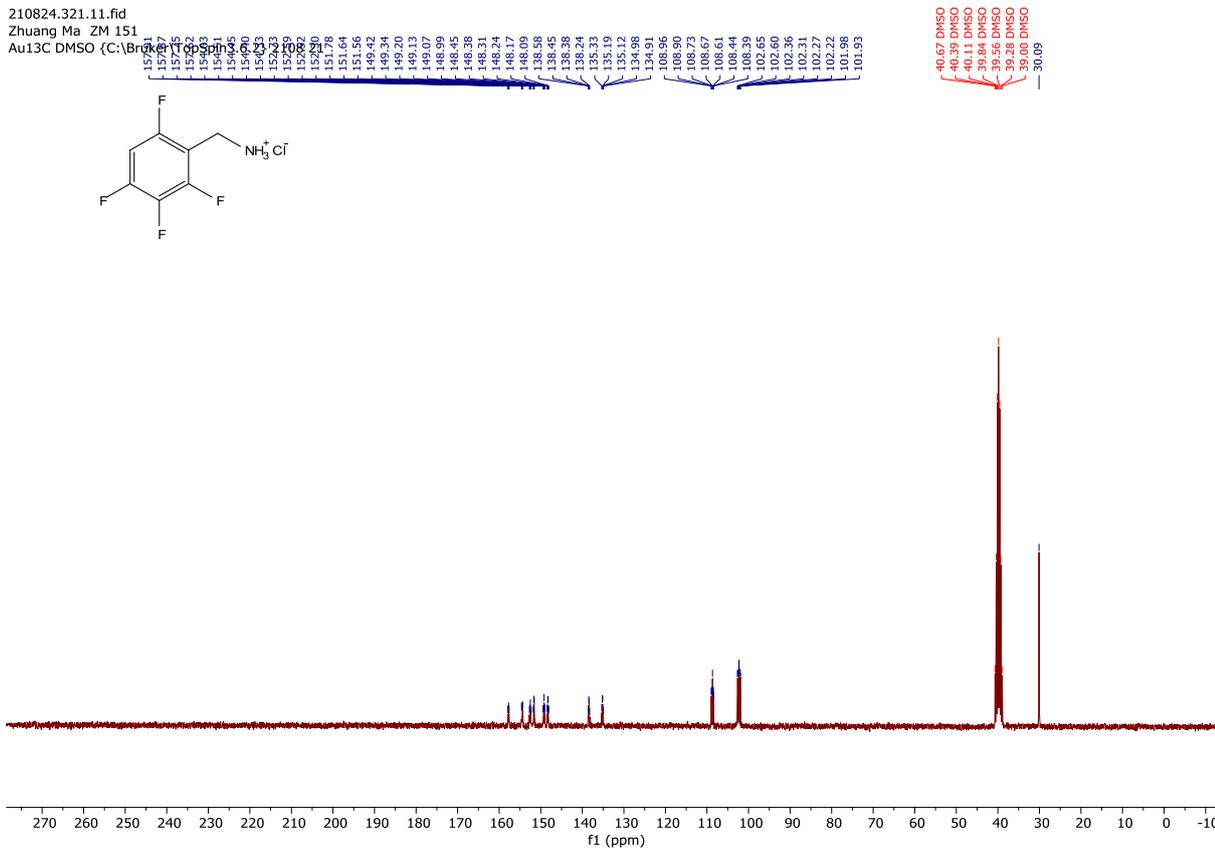
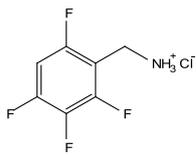
41.36



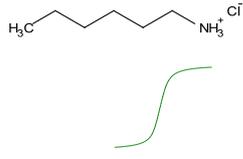
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 Zhuang Ma ZM 151  
 Au1H DMSO (C:\Bruker\TopSpin3.6.2} 2108 21



210824.321.11.fid  
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 Au13C DMSO (C:\Bruker\TopSpin3.6.2} 2108 21

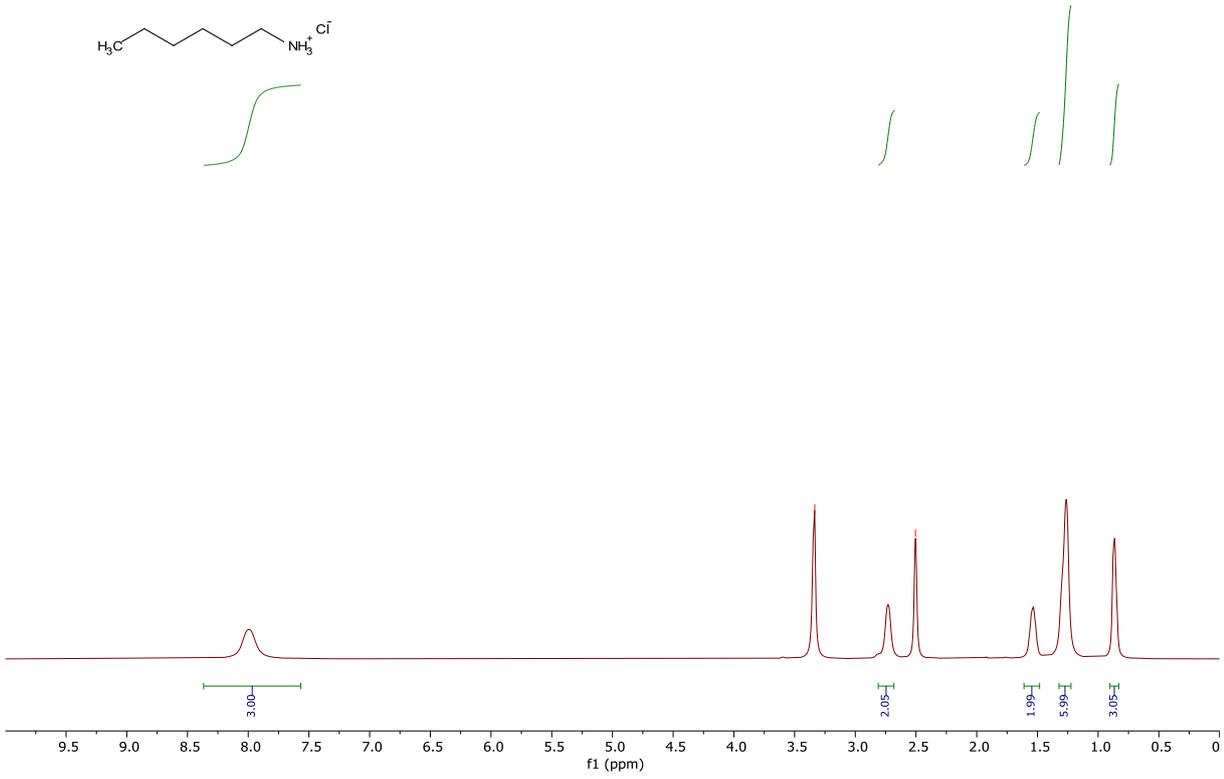


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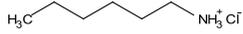


3.33 H<sub>2</sub>O

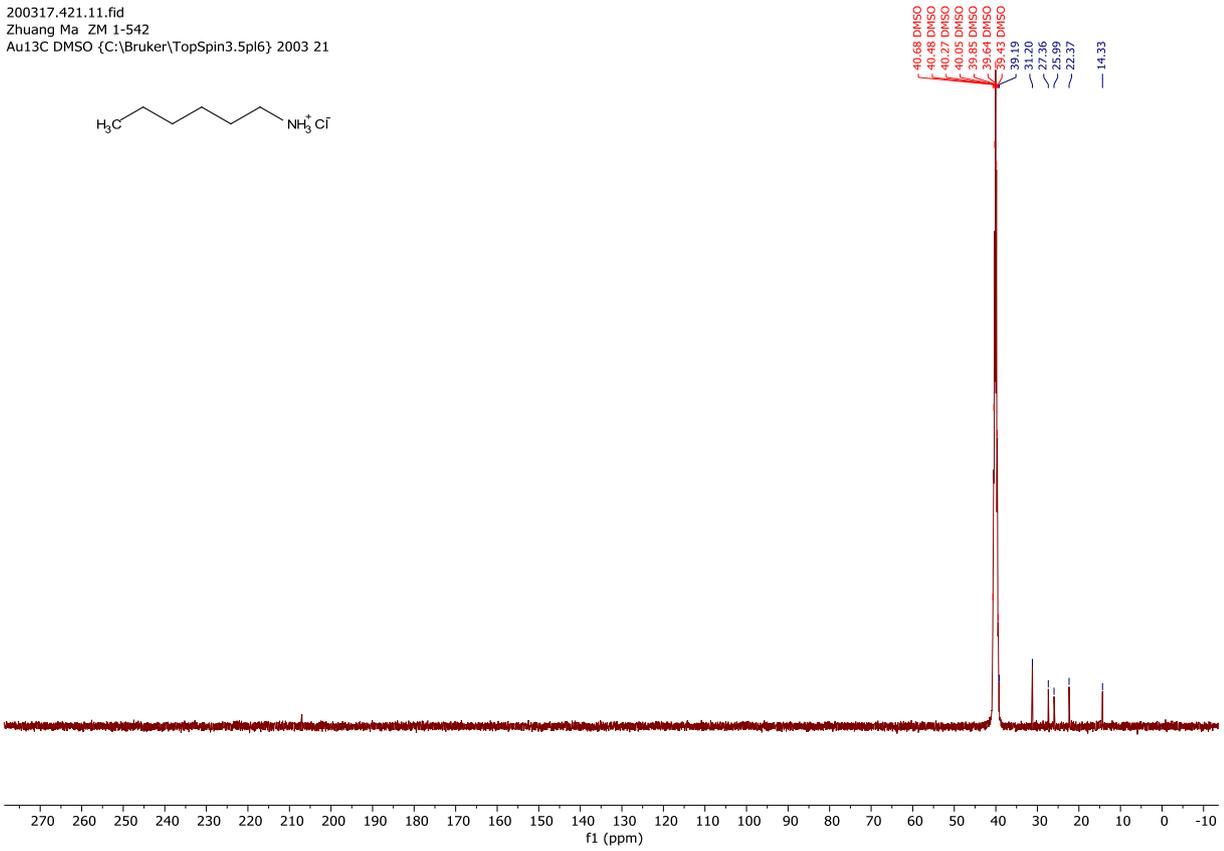
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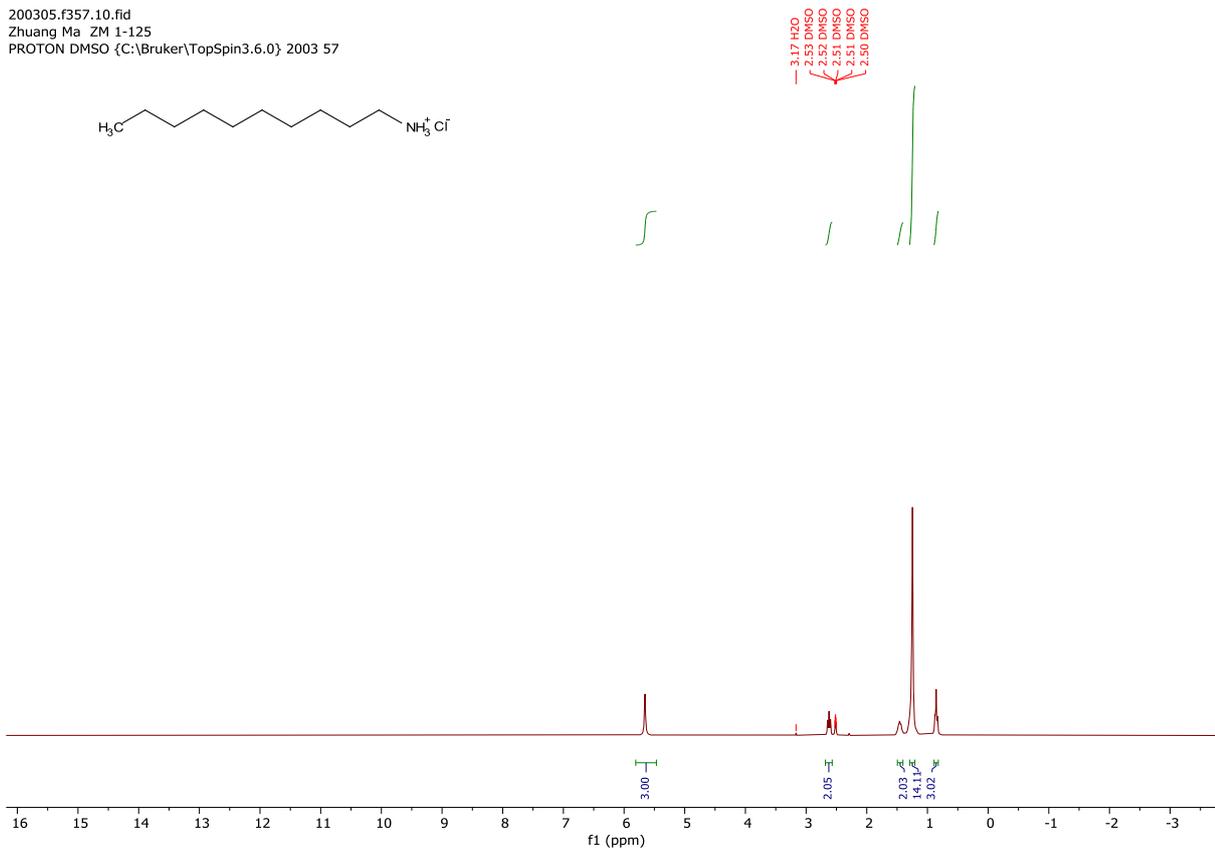
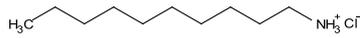
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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2003 21



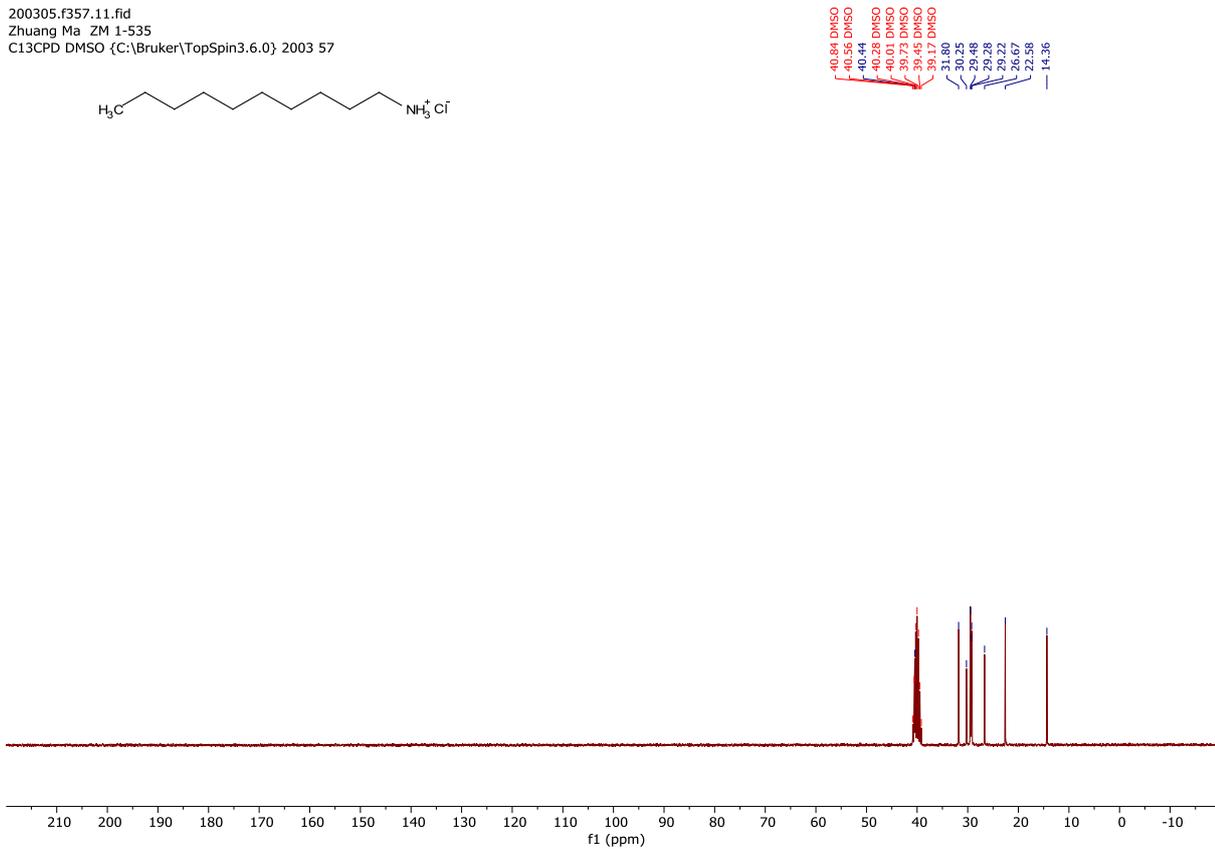
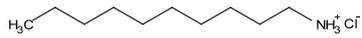
40.68 DMSO  
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40.27 DMSO  
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38.19  
31.20  
27.36  
25.99  
22.37  
14.33



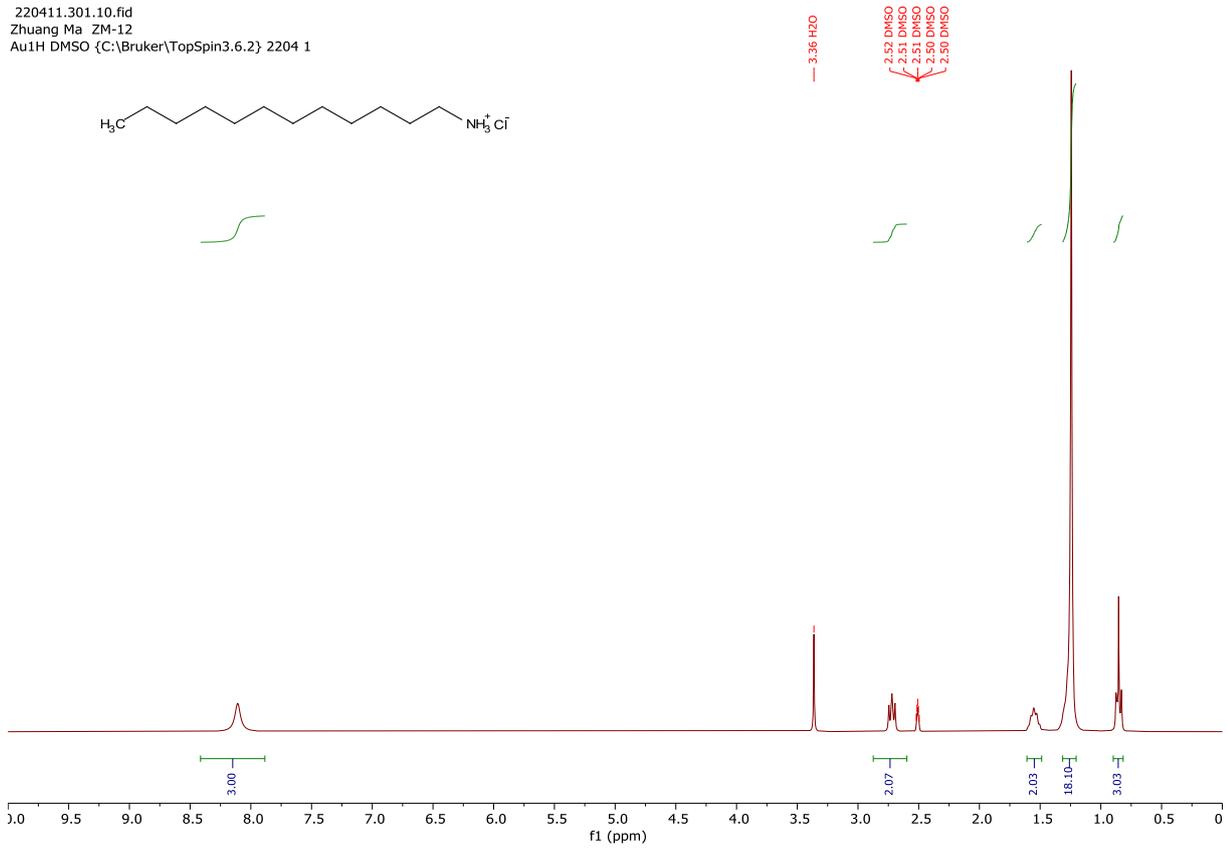
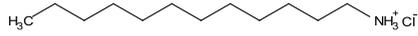
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PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 57



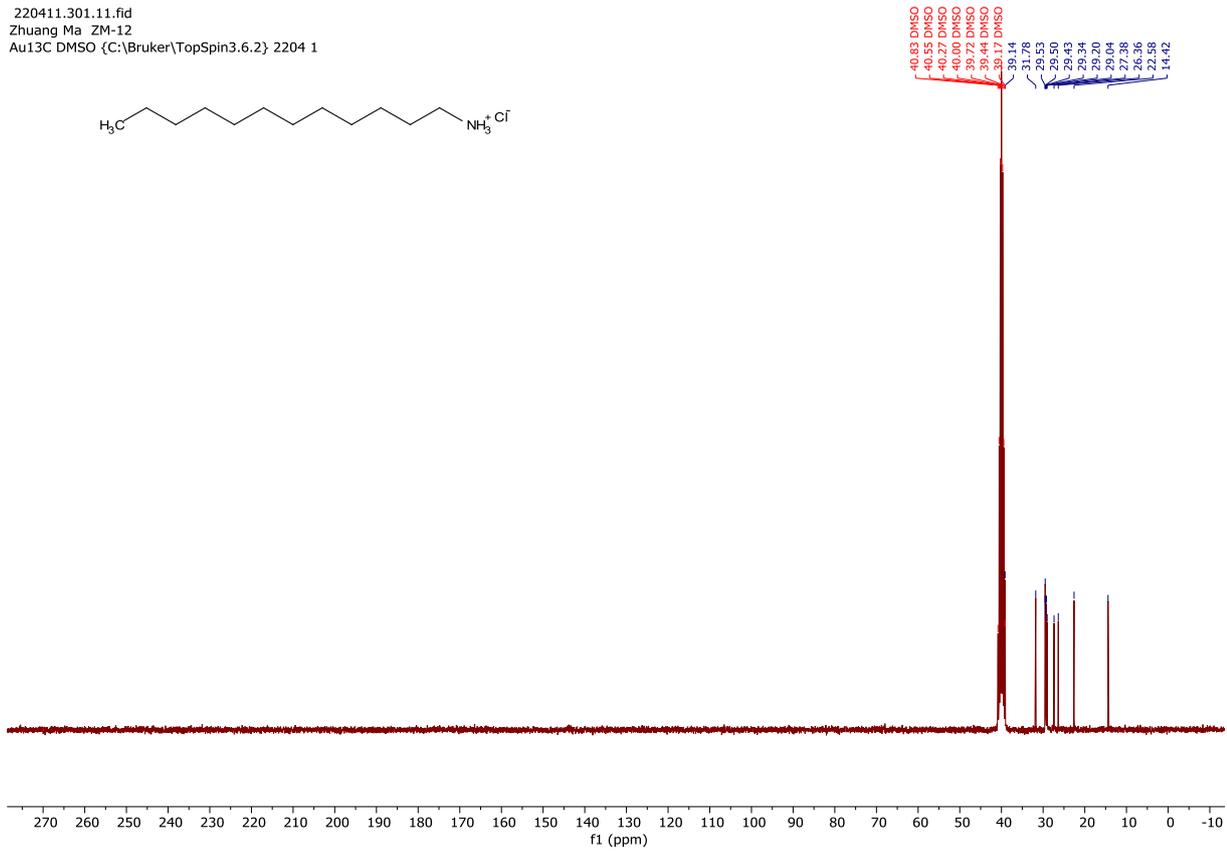
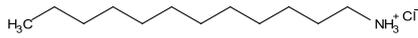
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 57



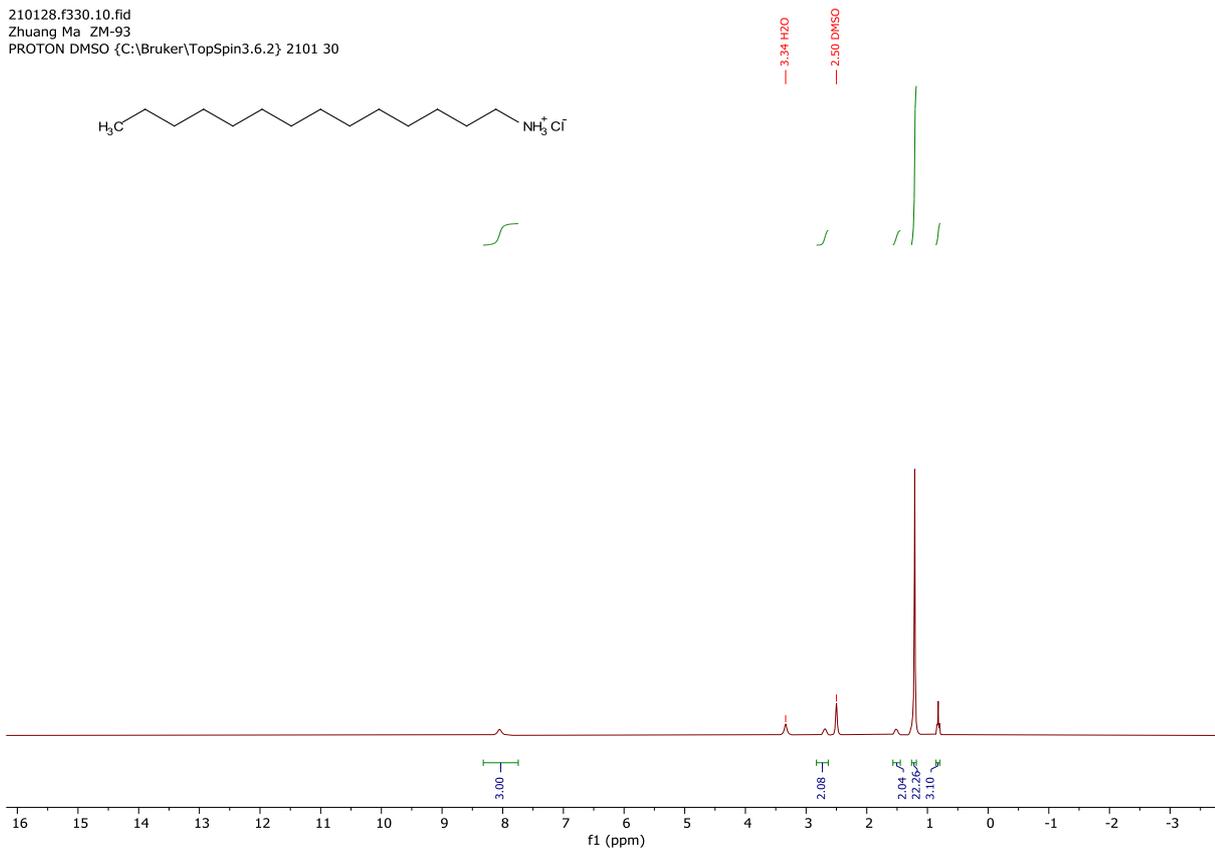
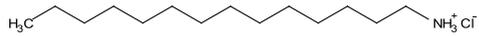
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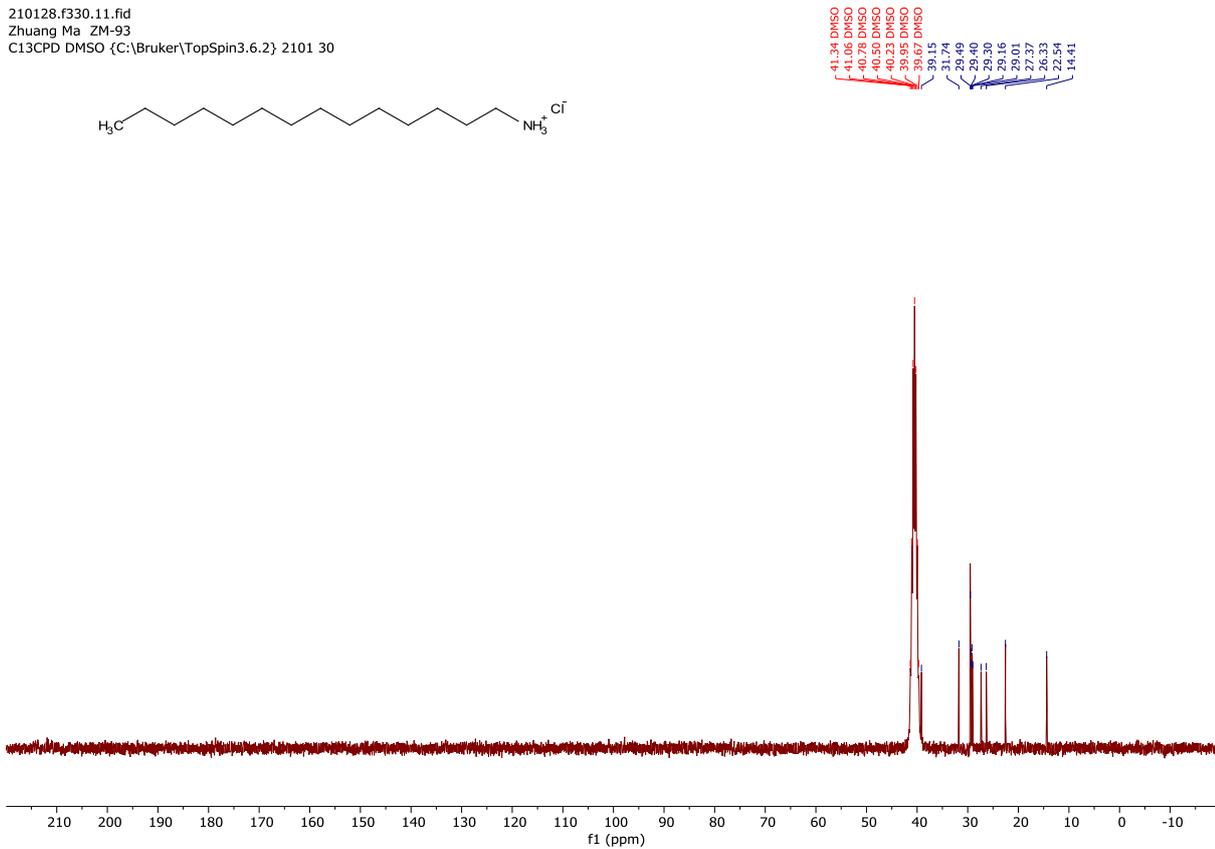
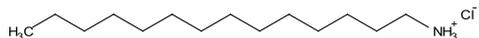
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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2204 1



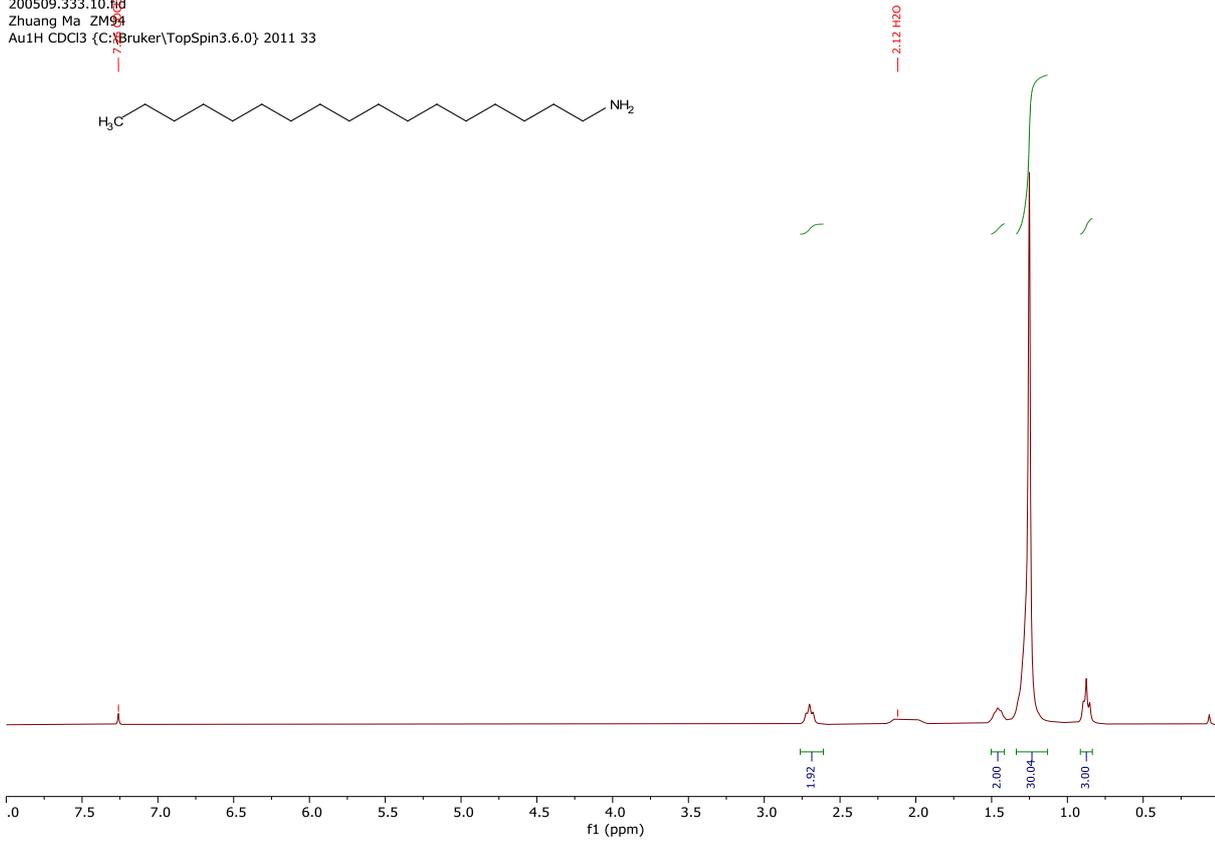
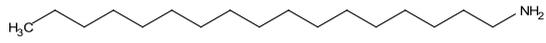
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PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2101 30



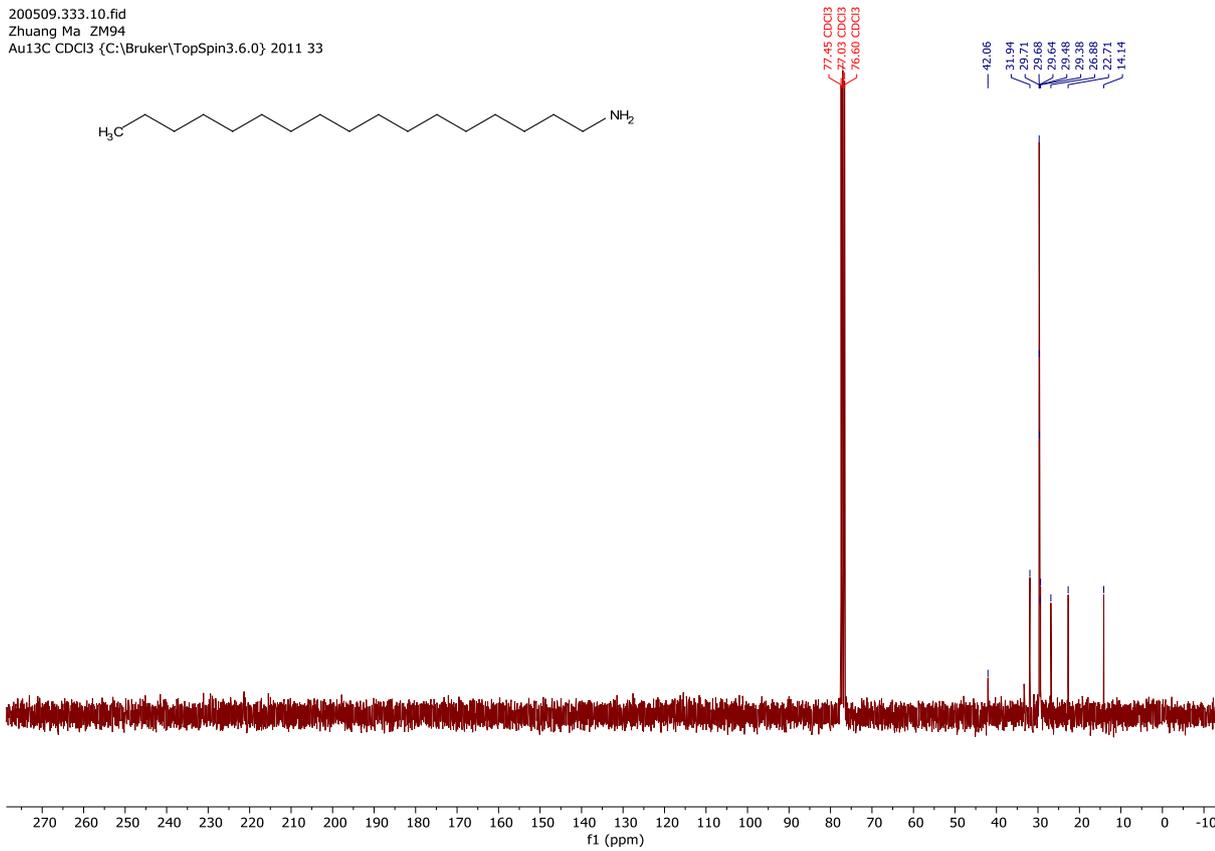
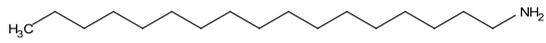
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C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2101 30



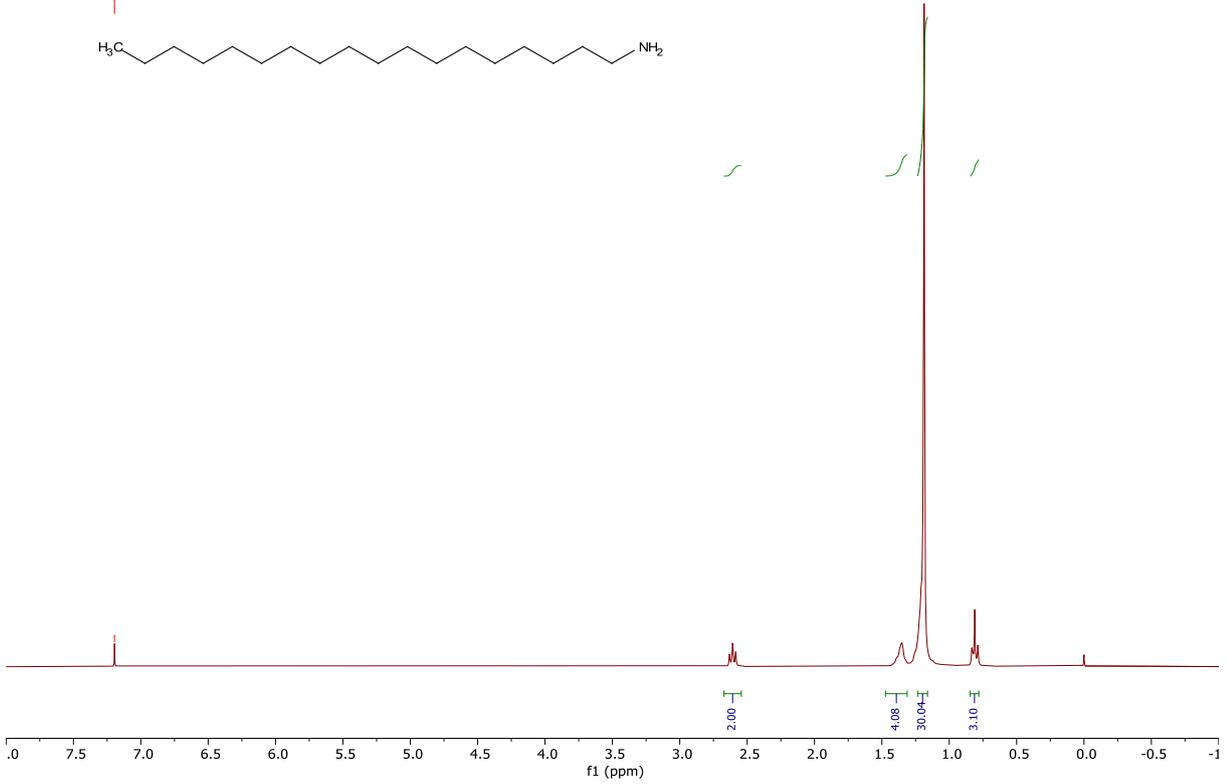
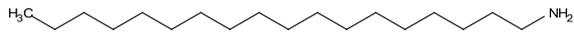
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Zhuang Ma\_ZM94  
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2011 33



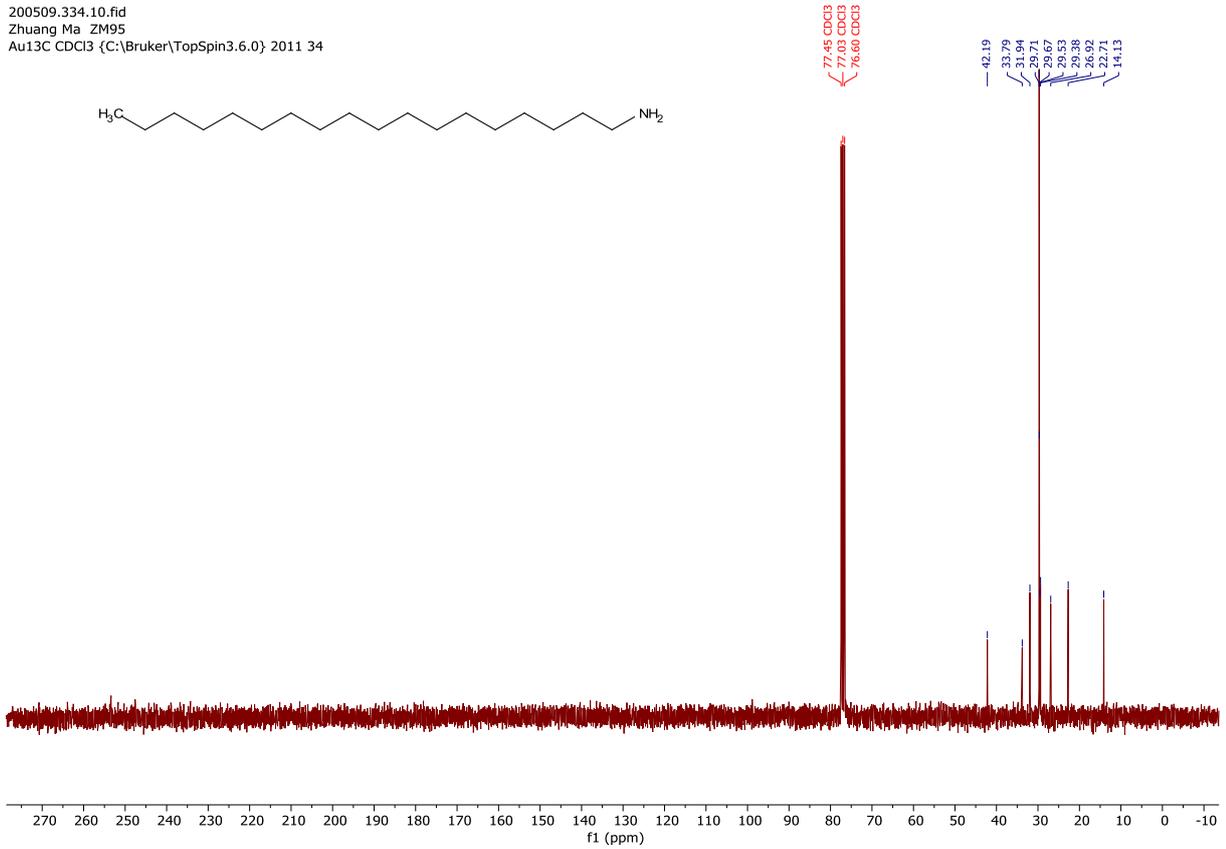
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Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2011 33



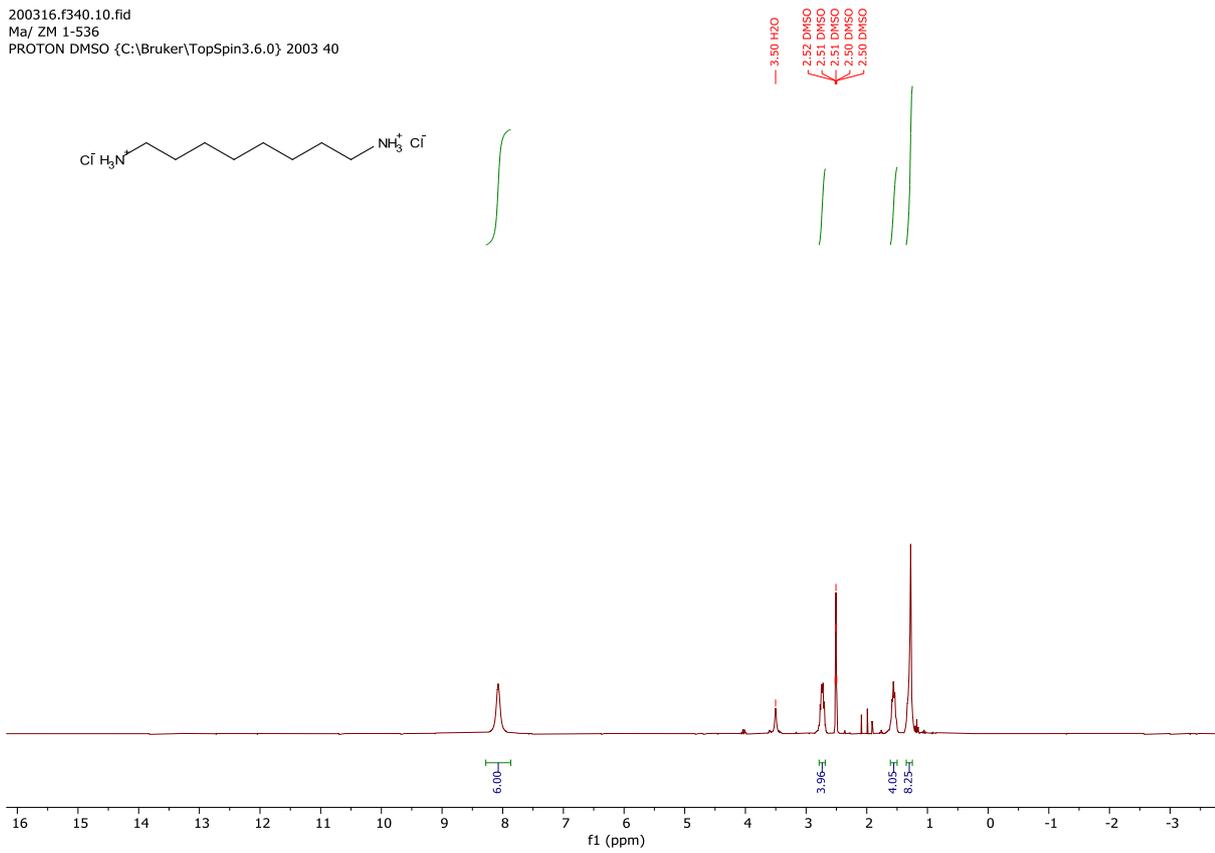
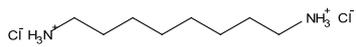
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Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2011 34



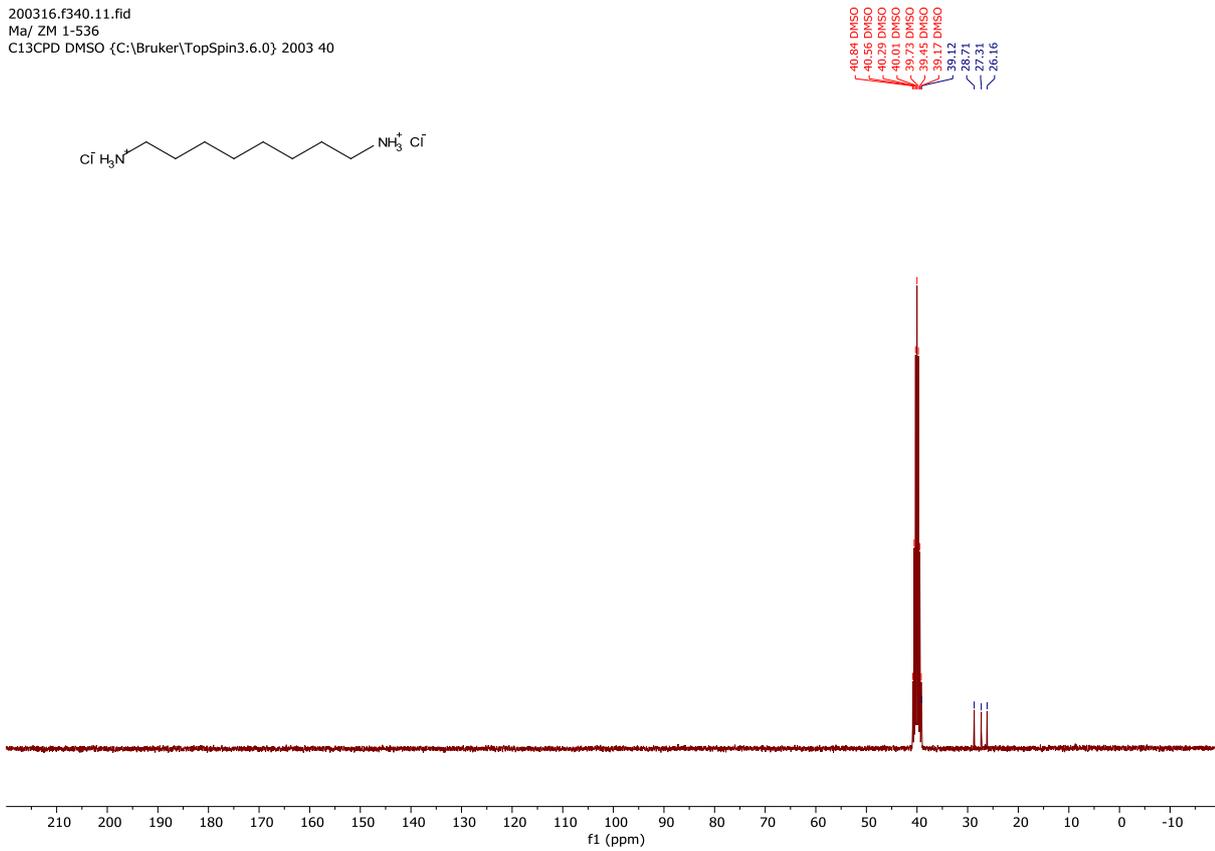
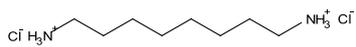
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Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2011 34



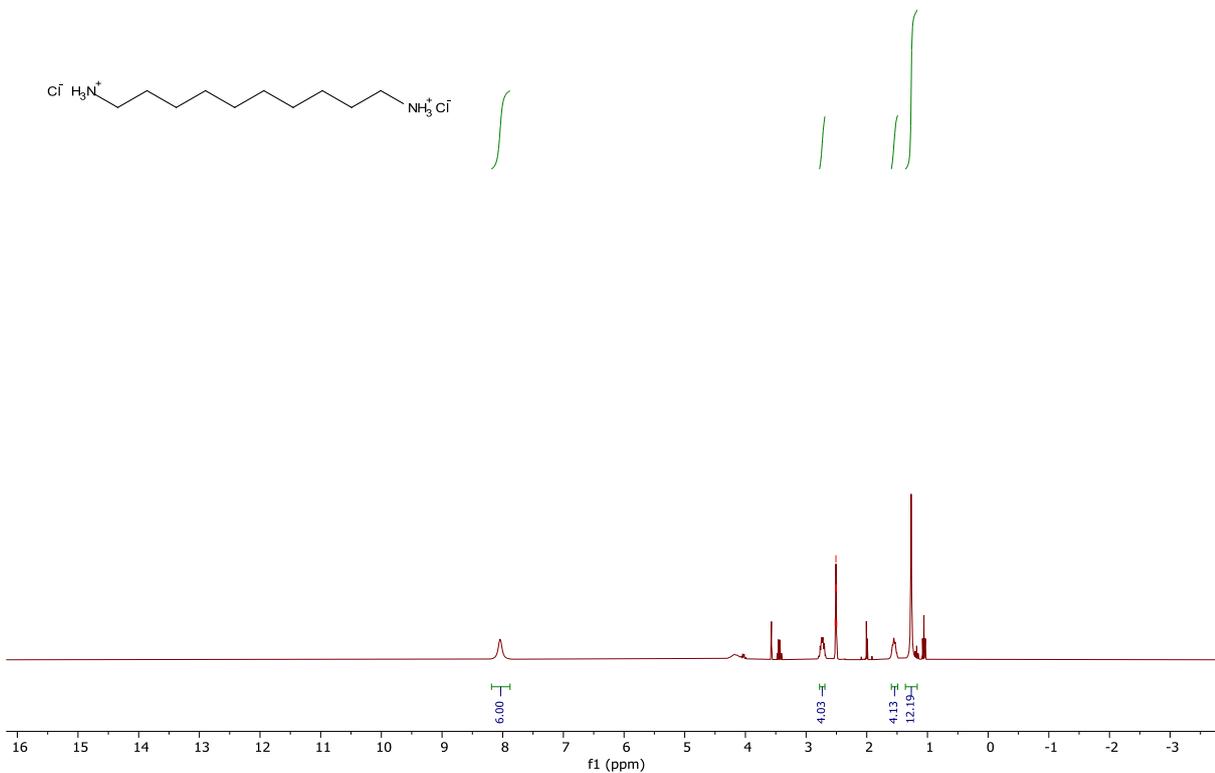
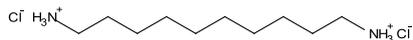
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Ma/ ZM 1-536  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 40



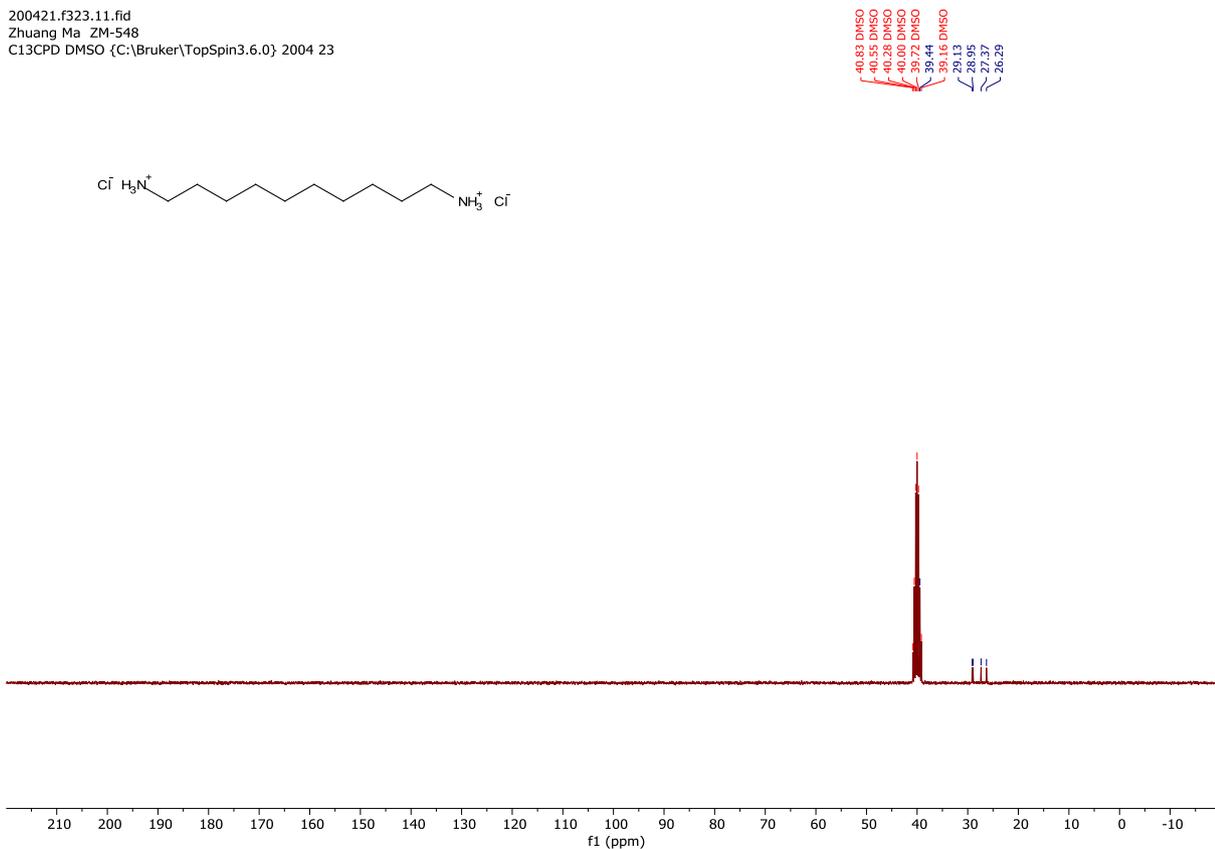
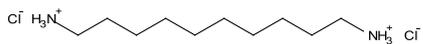
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Ma/ ZM 1-536  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 40



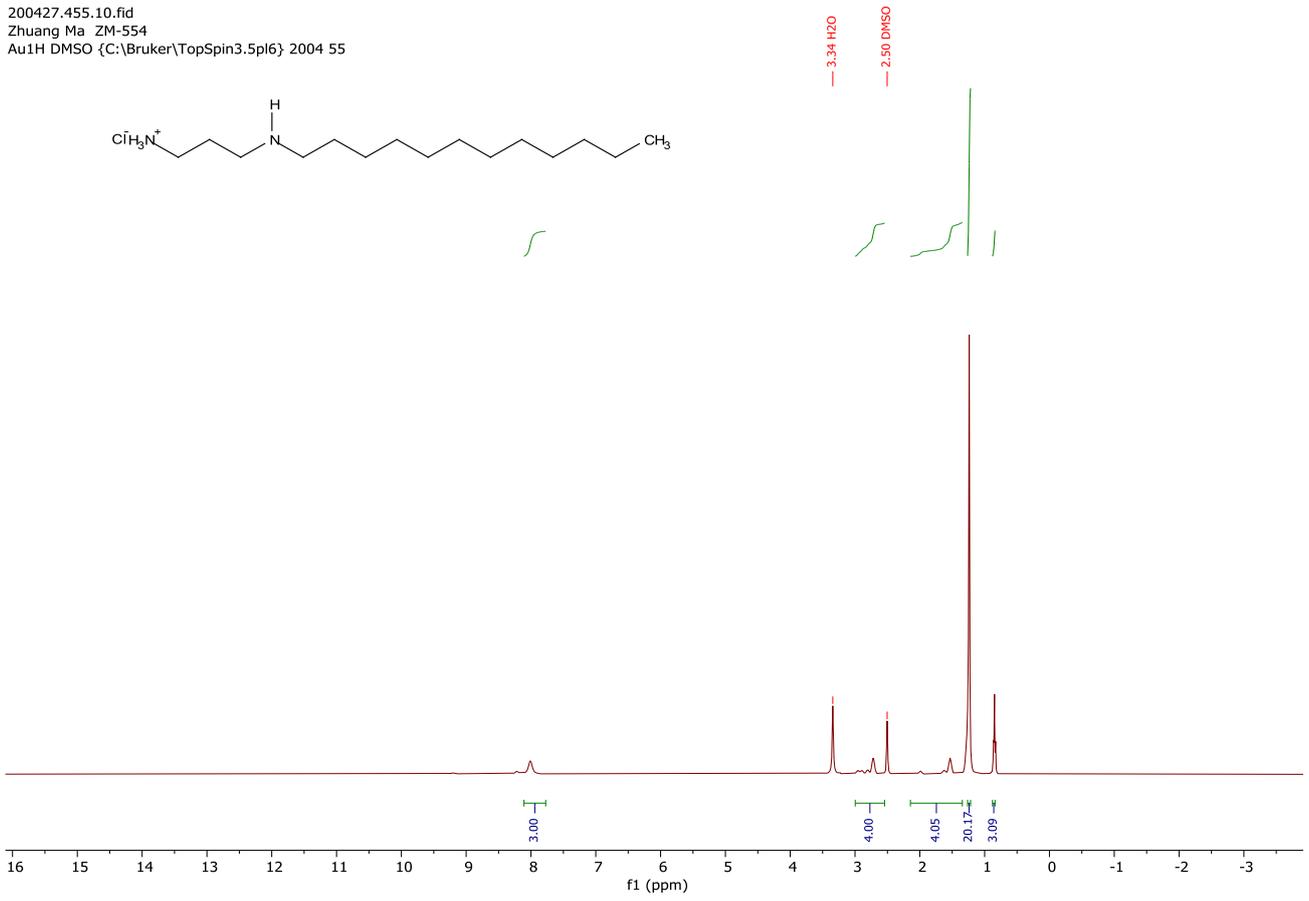
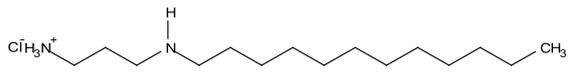
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H1CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 23



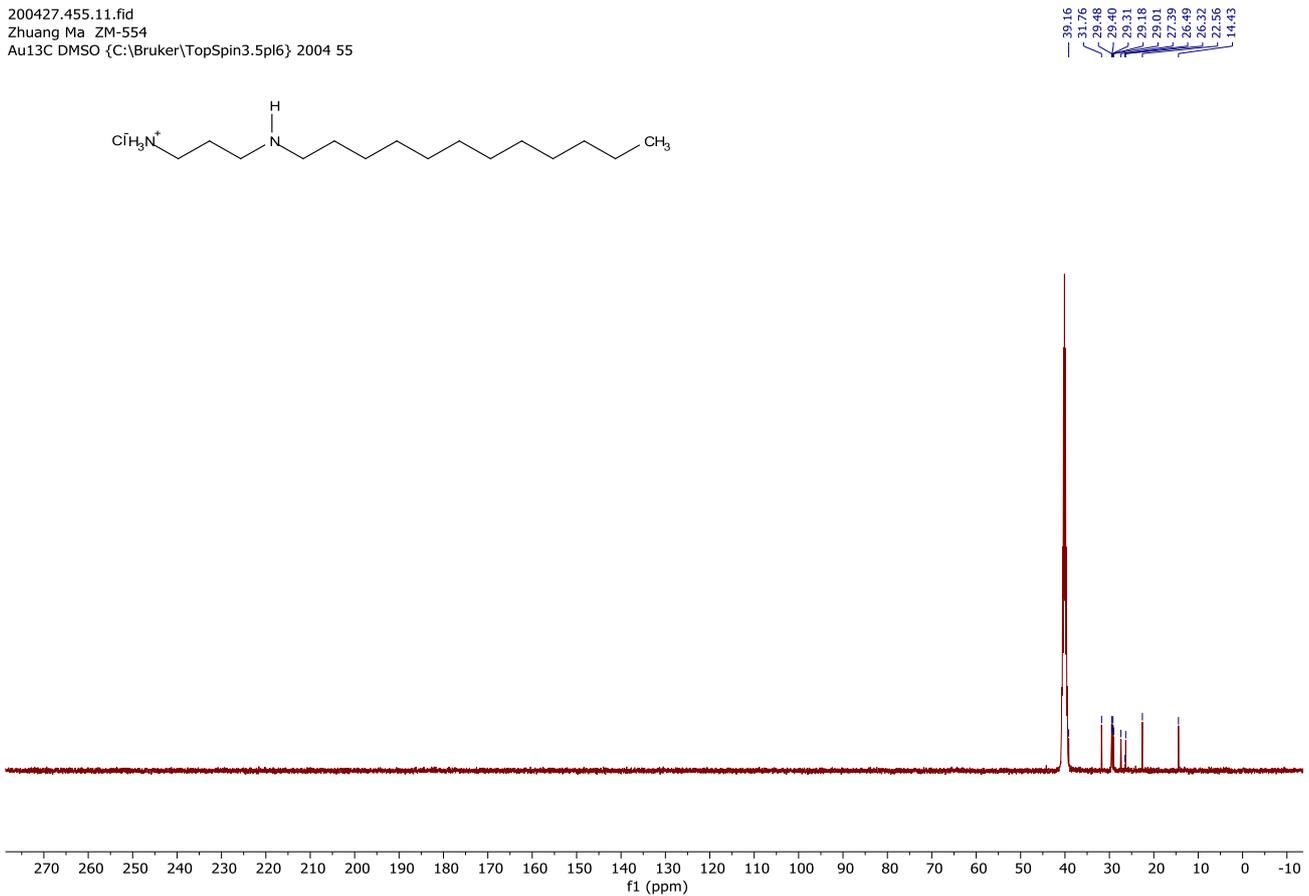
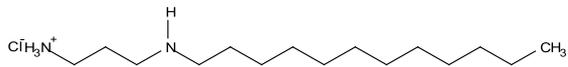
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 23



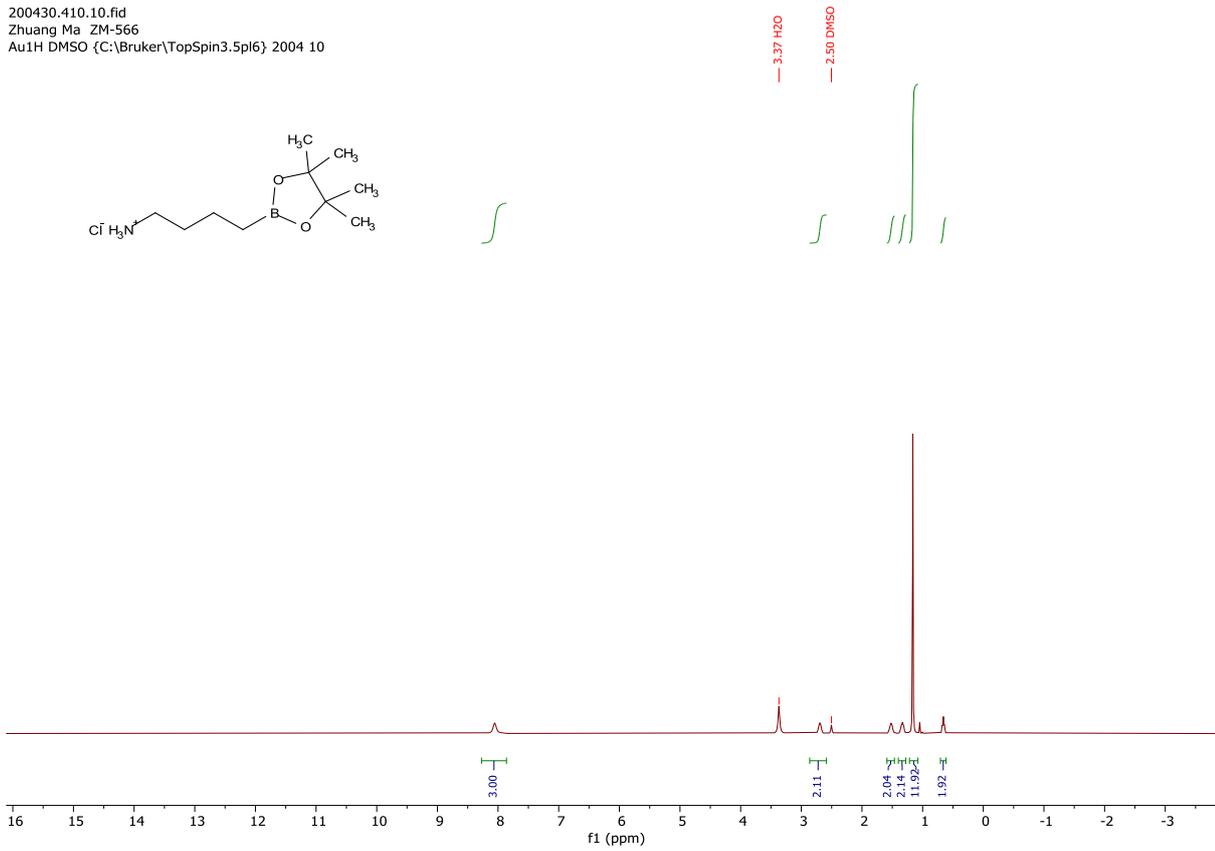
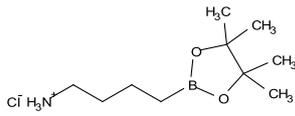
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Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 55



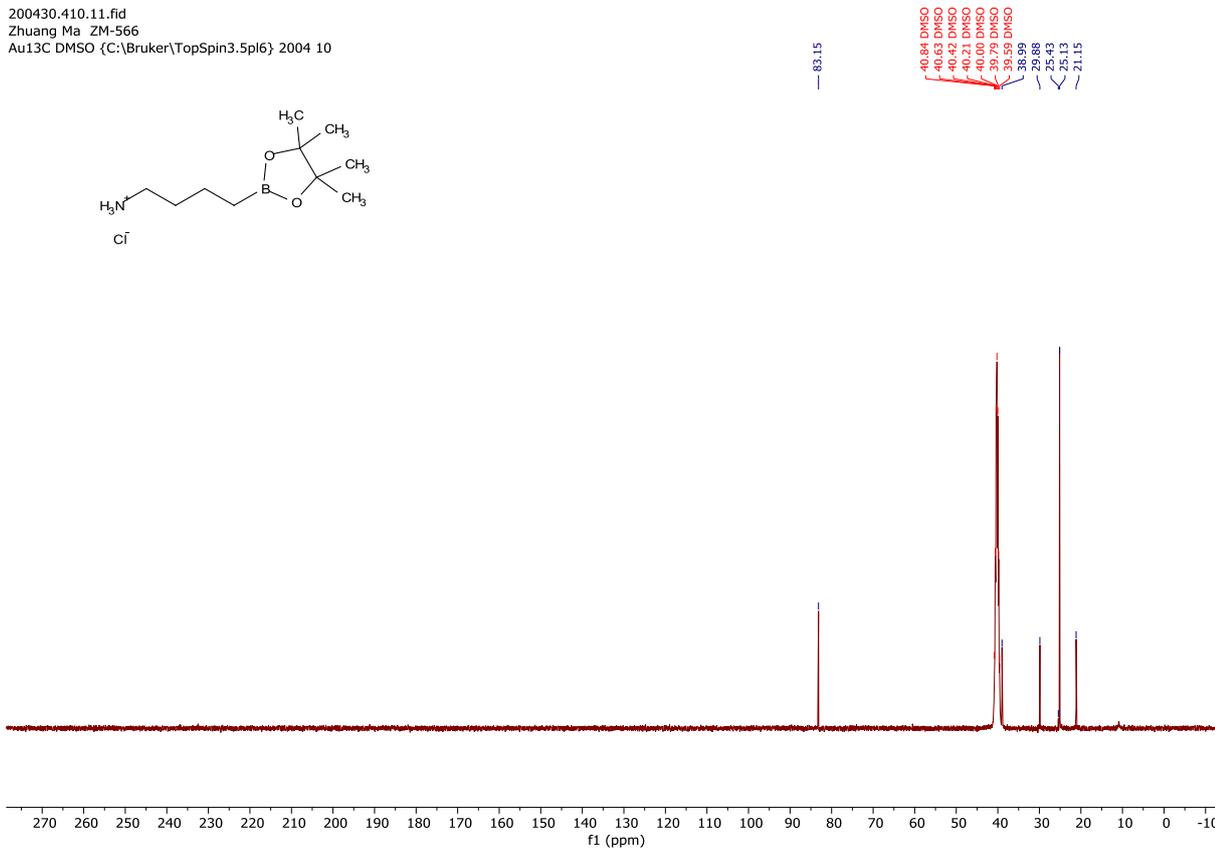
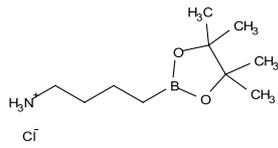
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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2004 55



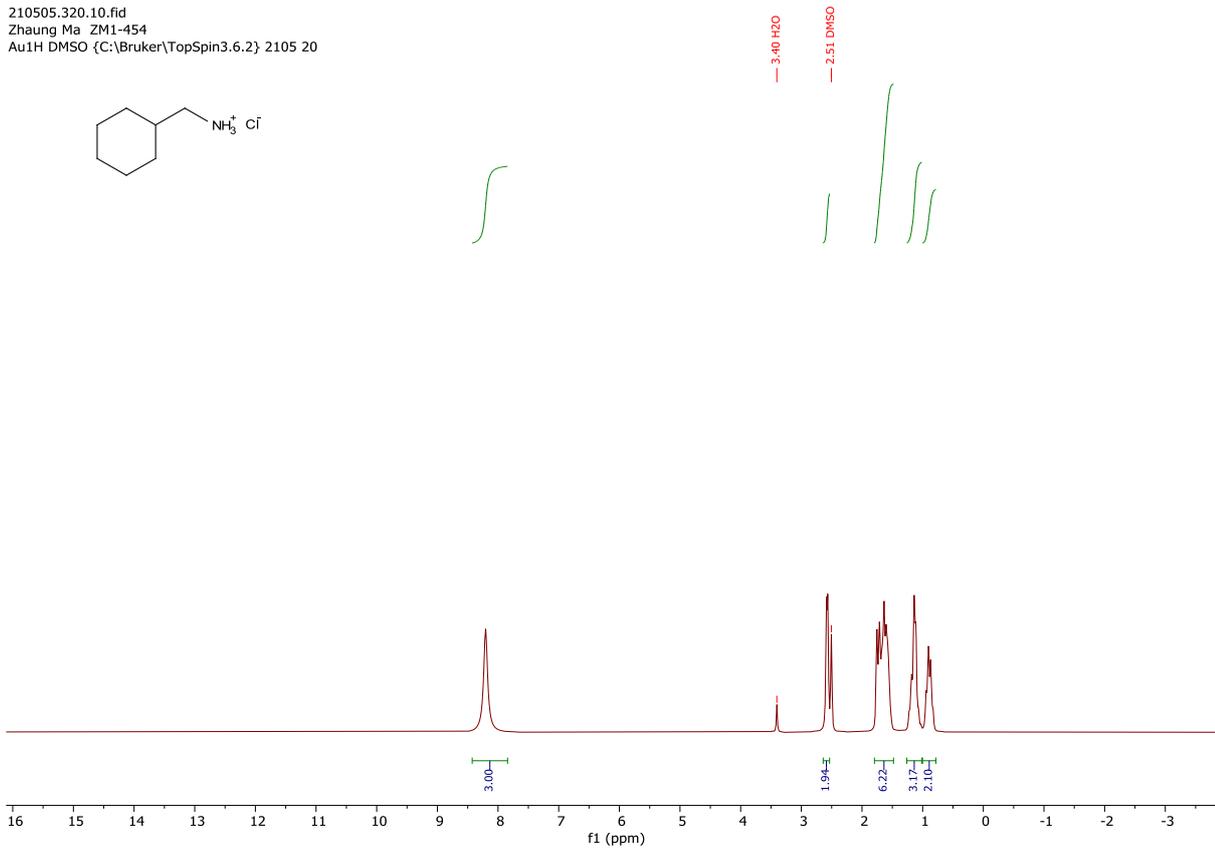
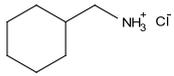
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Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2004 10



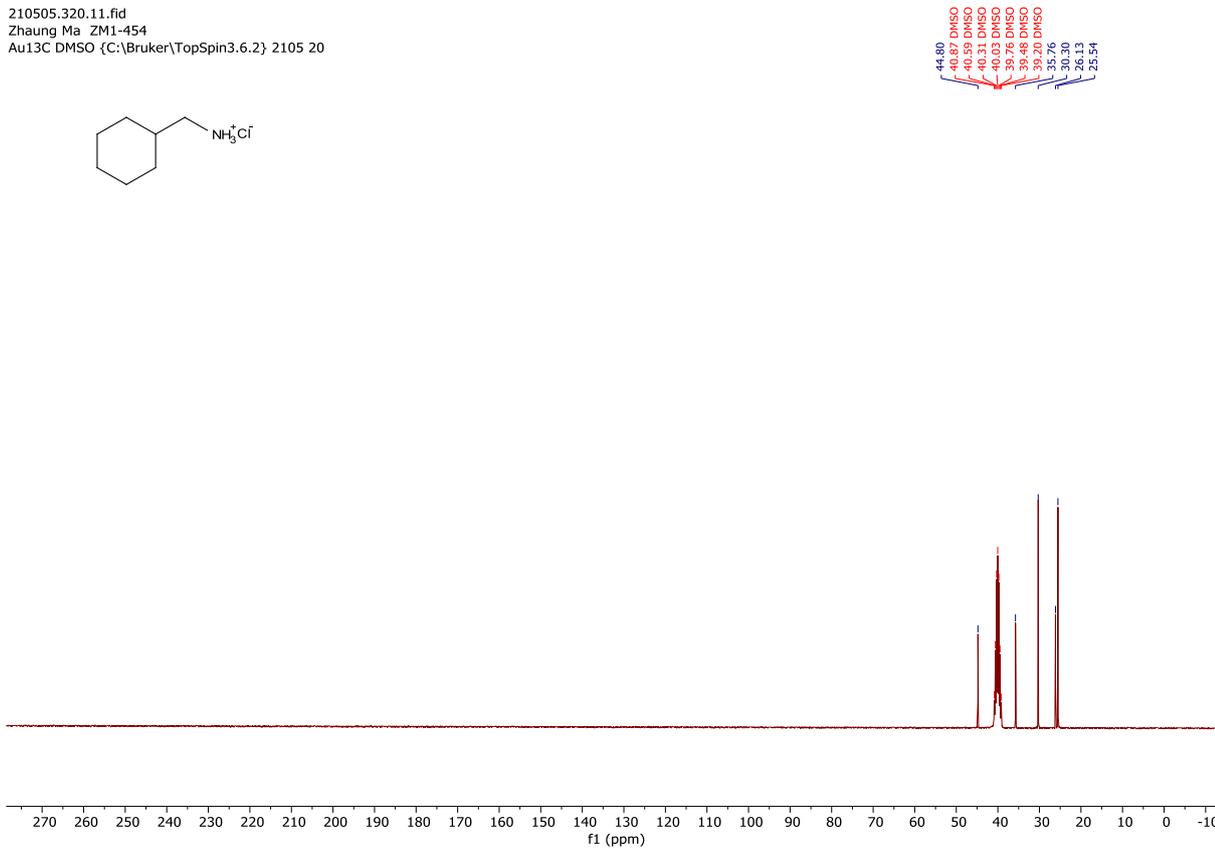
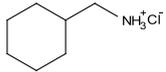
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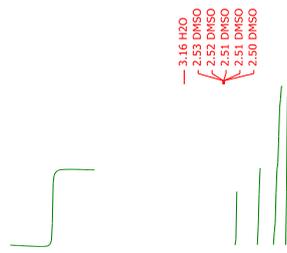
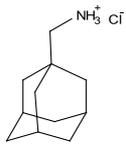
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 20



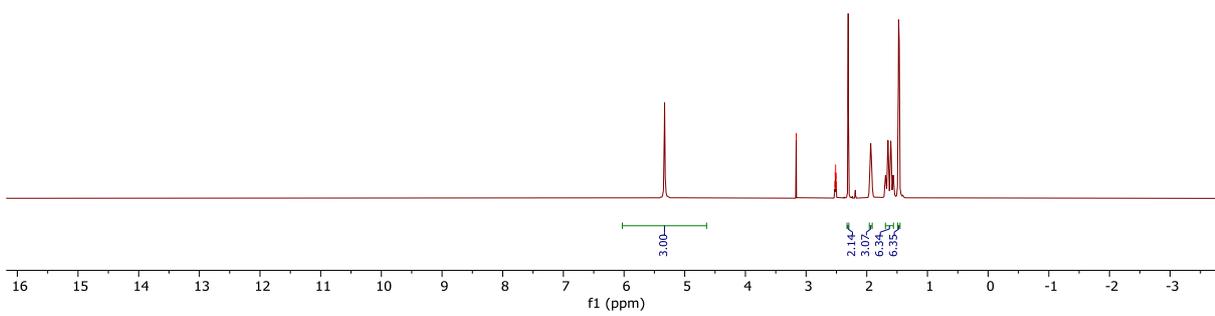
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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 20



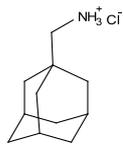
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Ma/ ZM 1-493  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 28



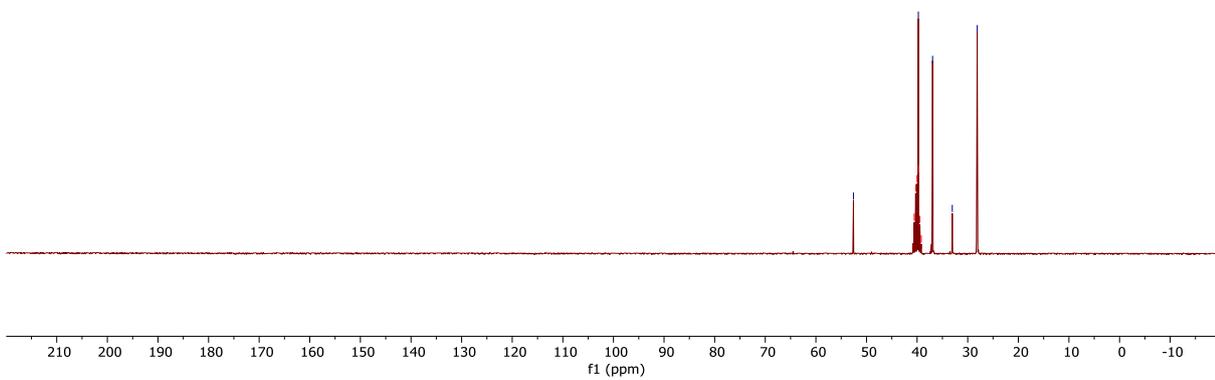
3.16 H2O  
2.53 DMSO  
2.52 DMSO  
2.51 DMSO  
2.50 DMSO



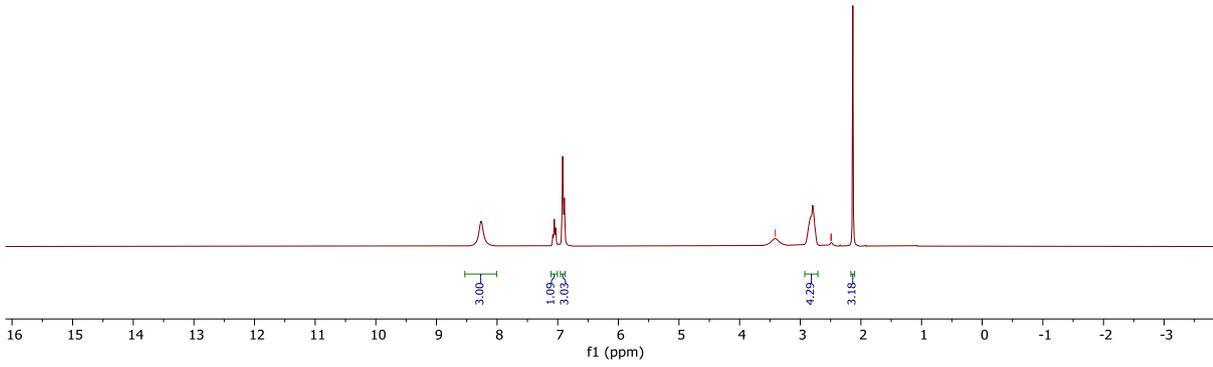
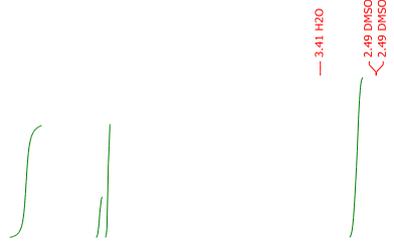
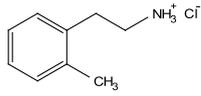
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Ma/ ZM 1-493  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 28



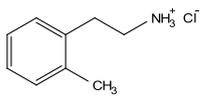
52.58  
40.56 DMSO  
40.28 DMSO  
40.01 DMSO  
39.73 DMSO  
39.45 DMSO  
39.18 DMSO  
36.95  
33.05  
28.12



210920.305.10.fid  
Ma/ ZM 158  
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2109 5

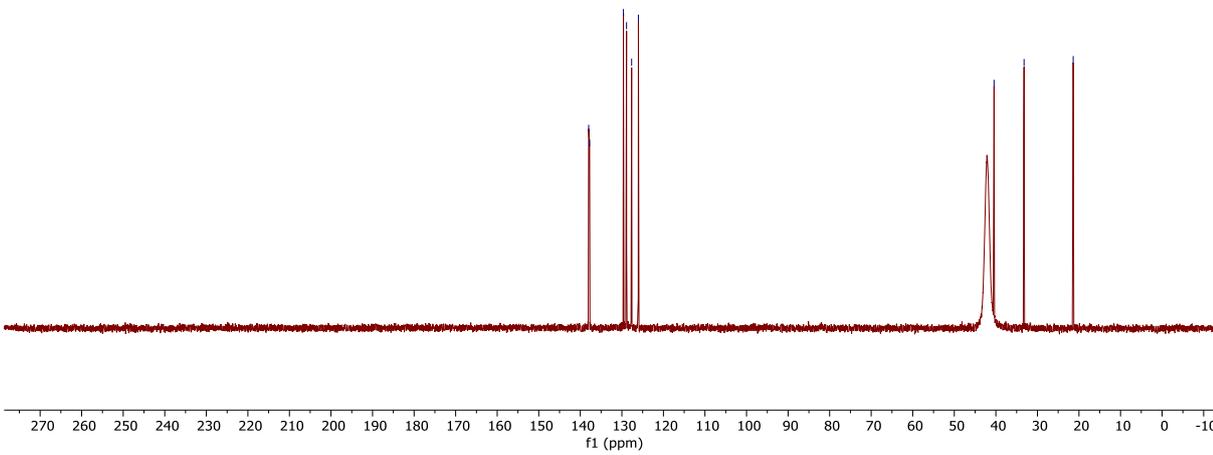


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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2109 5

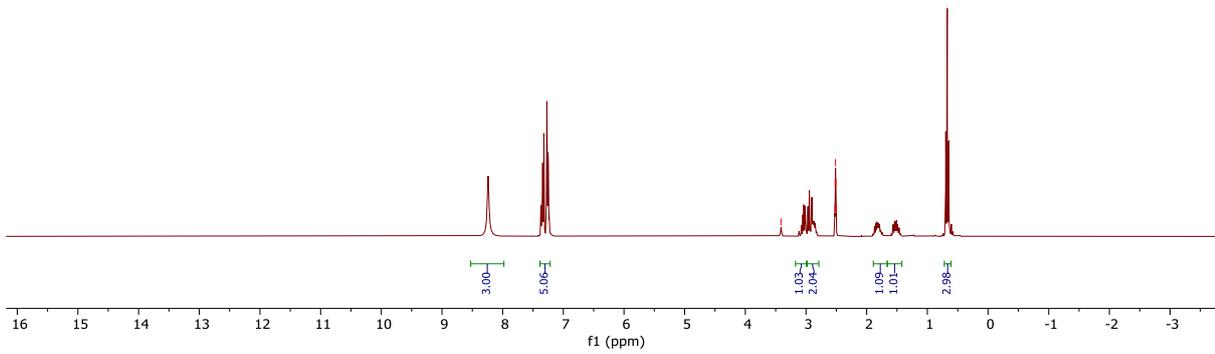
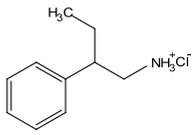


137.99  
137.76  
129.62  
128.87  
127.65  
126.02

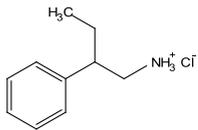
40.44  
33.22  
21.39



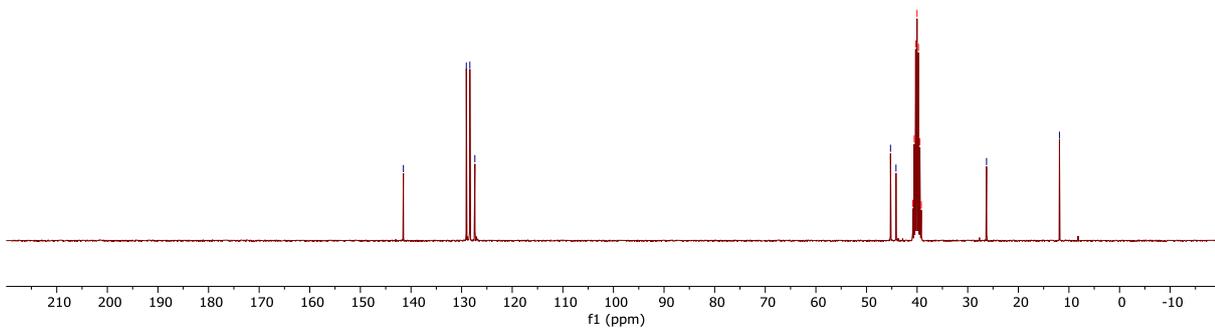
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Zhuang Ma\_ZM 1-463  
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 51



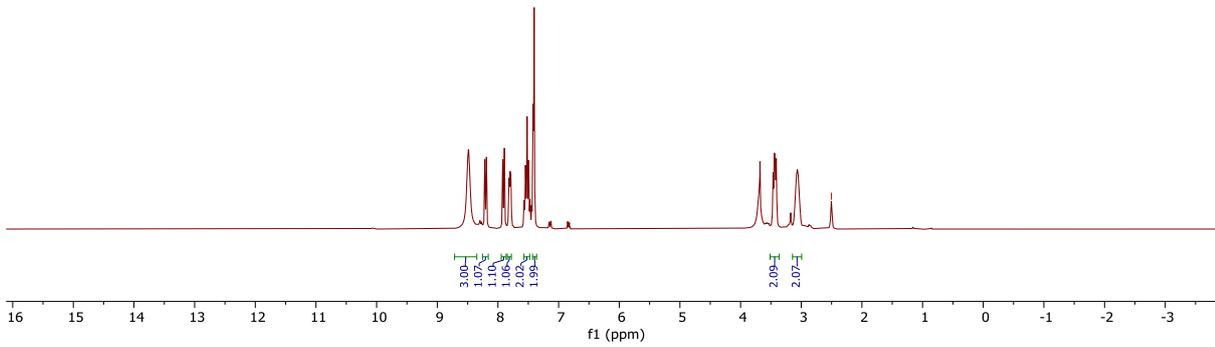
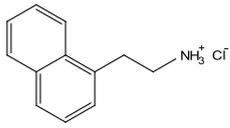
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 51



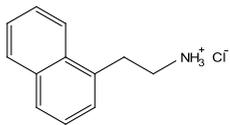
141.50  
129.07  
128.37  
127.38  
45.26  
44.17  
43.89 DMSO  
40.29 DMSO  
40.01 DMSO  
39.73 DMSO  
39.45 DMSO  
39.18 DMSO  
26.28  
11.87



210920.307.10.fid  
Ma/ ZM 160  
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2109 7

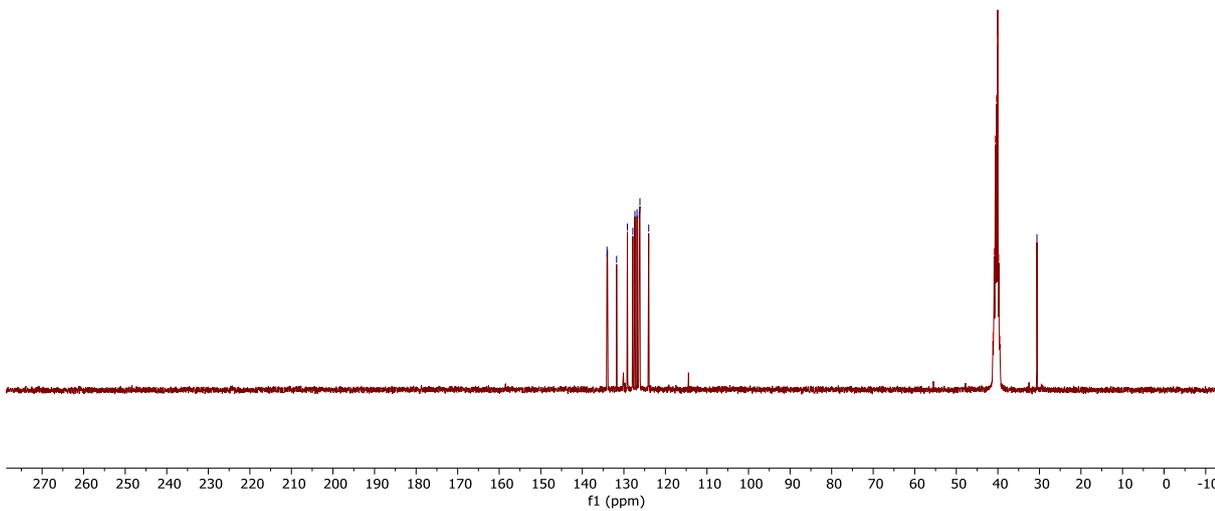


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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2109 7

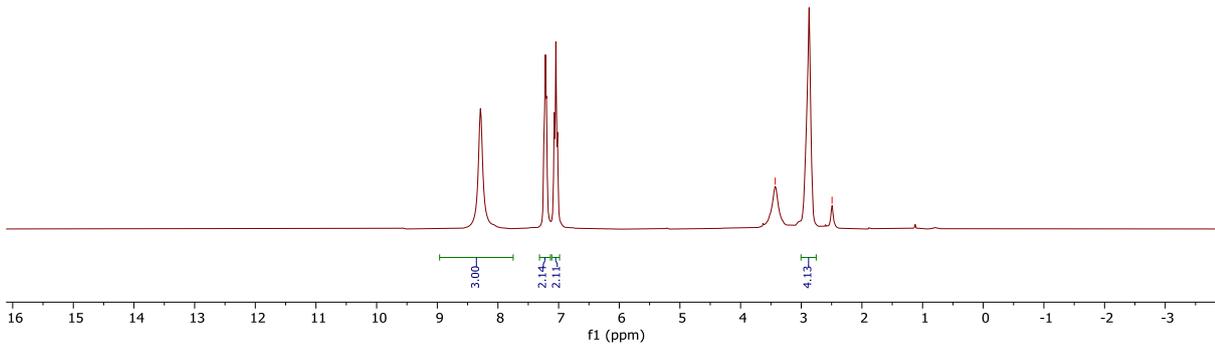
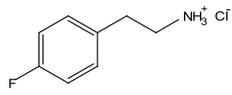


134.06  
133.93  
133.75  
129.16  
129.16  
127.35  
126.84  
126.29  
126.14  
124.03

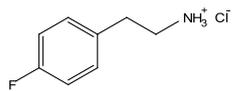
41.17 DMSO  
40.90 DMSO  
40.62 DMSO  
40.34 DMSO  
40.05 DMSO  
39.78 DMSO  
39.51 DMSO  
30.60



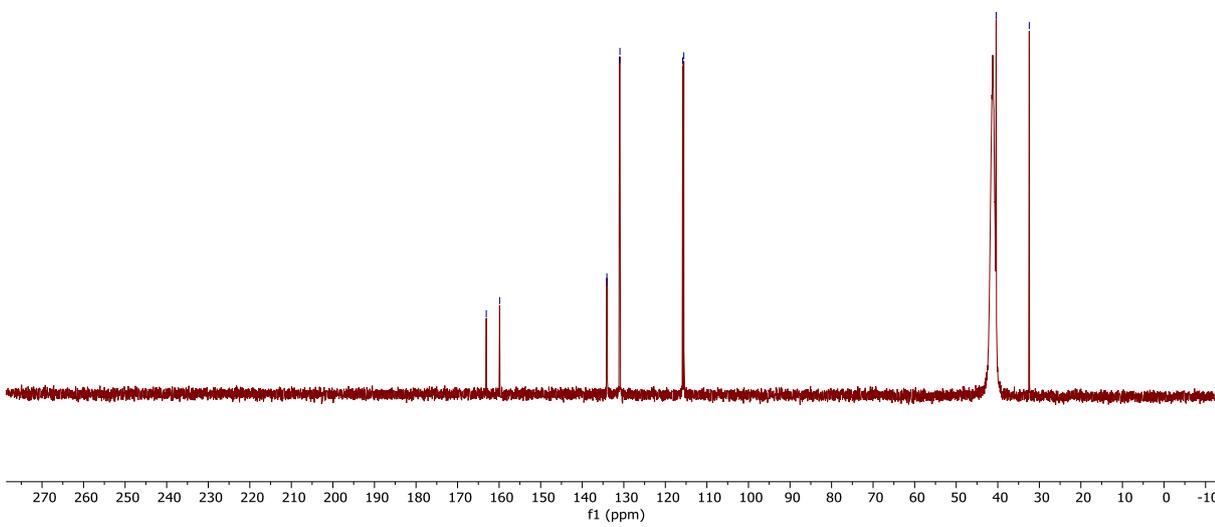
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Ma/ ZM 157  
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2109 4



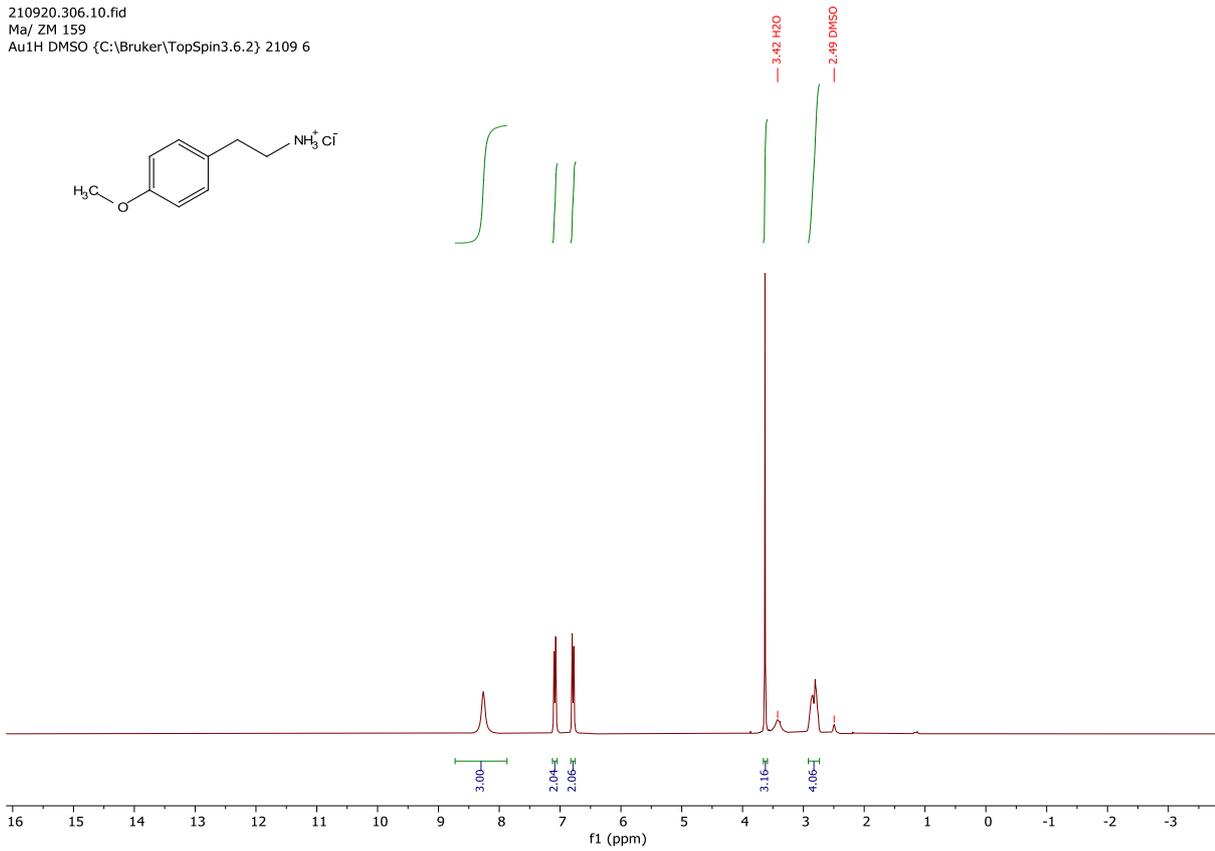
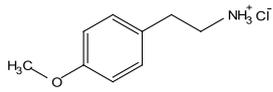
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Ma/ ZM 157  
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2109 4



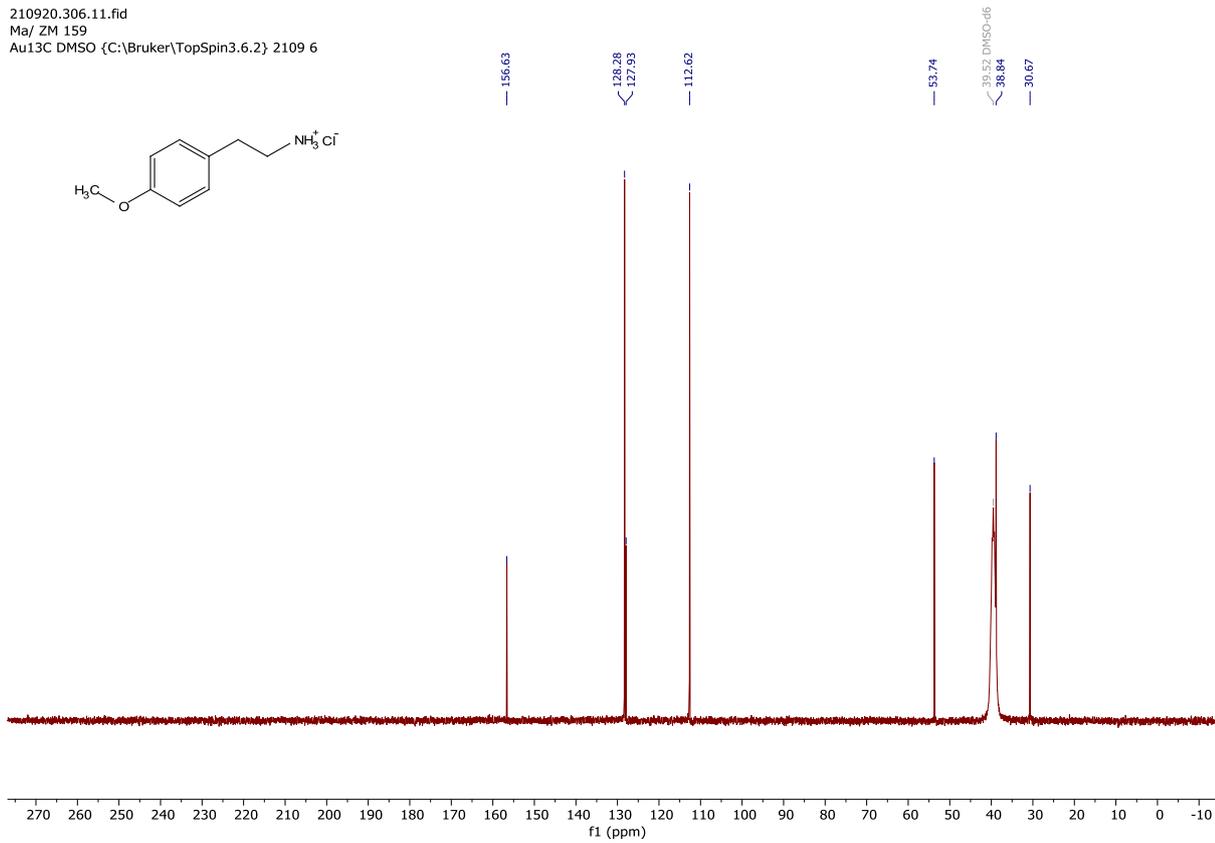
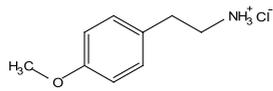
163.09  
159.88  
134.07  
133.04  
131.04  
130.93  
115.84  
115.56  
40.39  
32.42



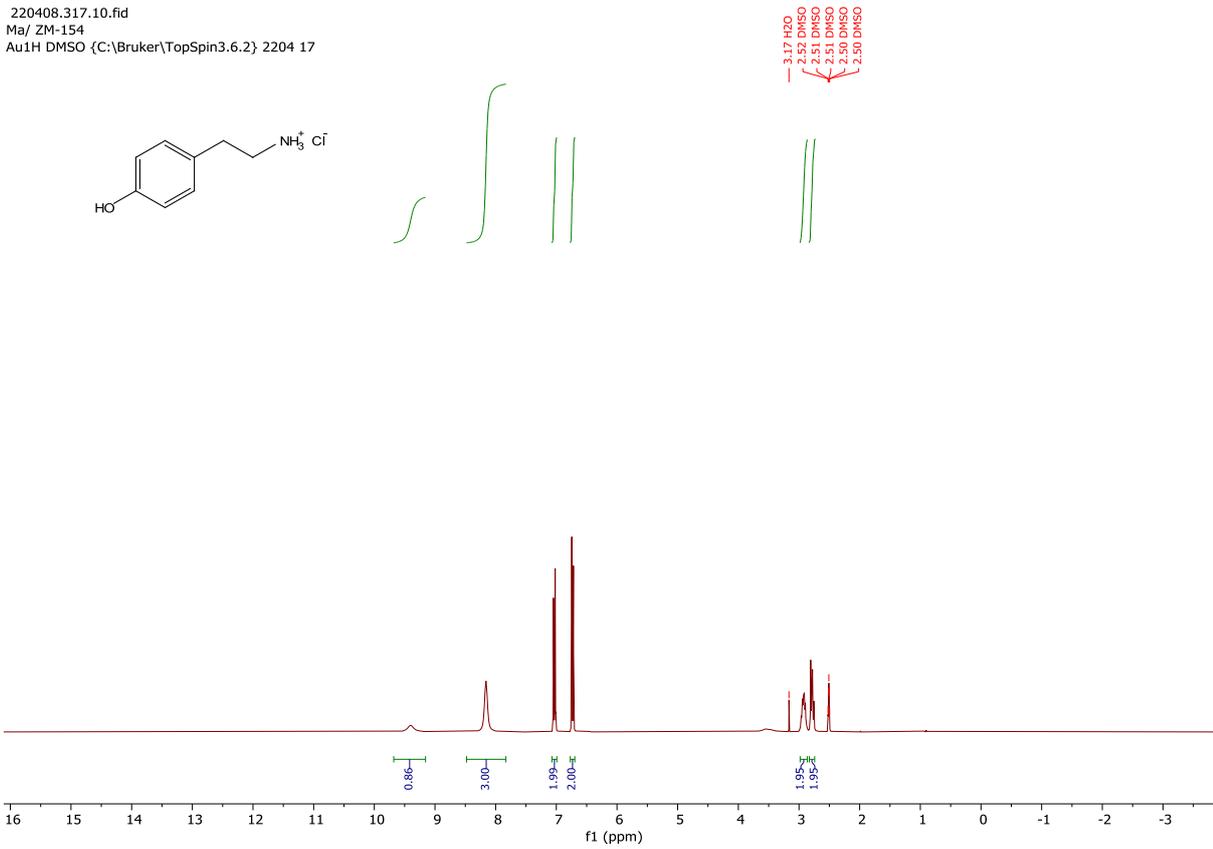
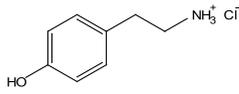
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Ma/ ZM 159  
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2109 6



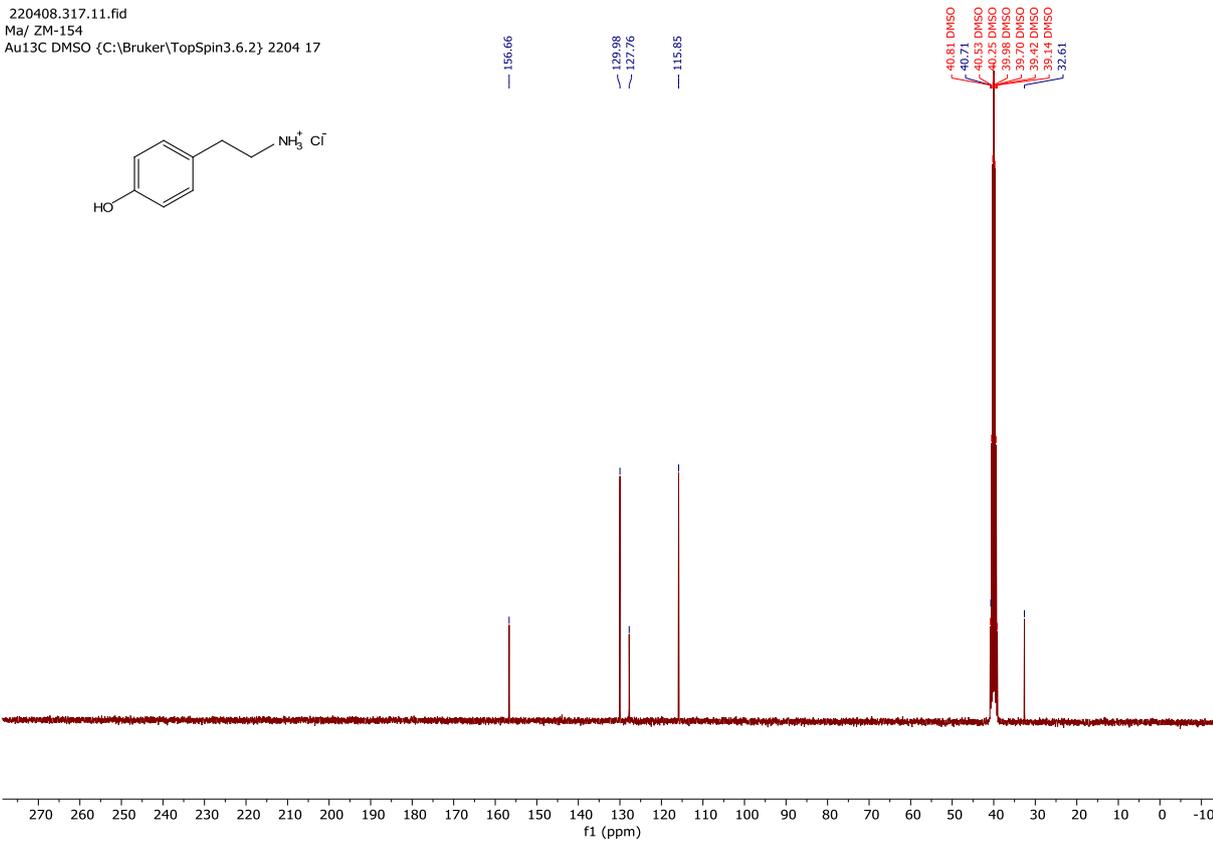
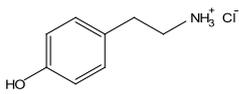
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Ma/ ZM 159  
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2109 6



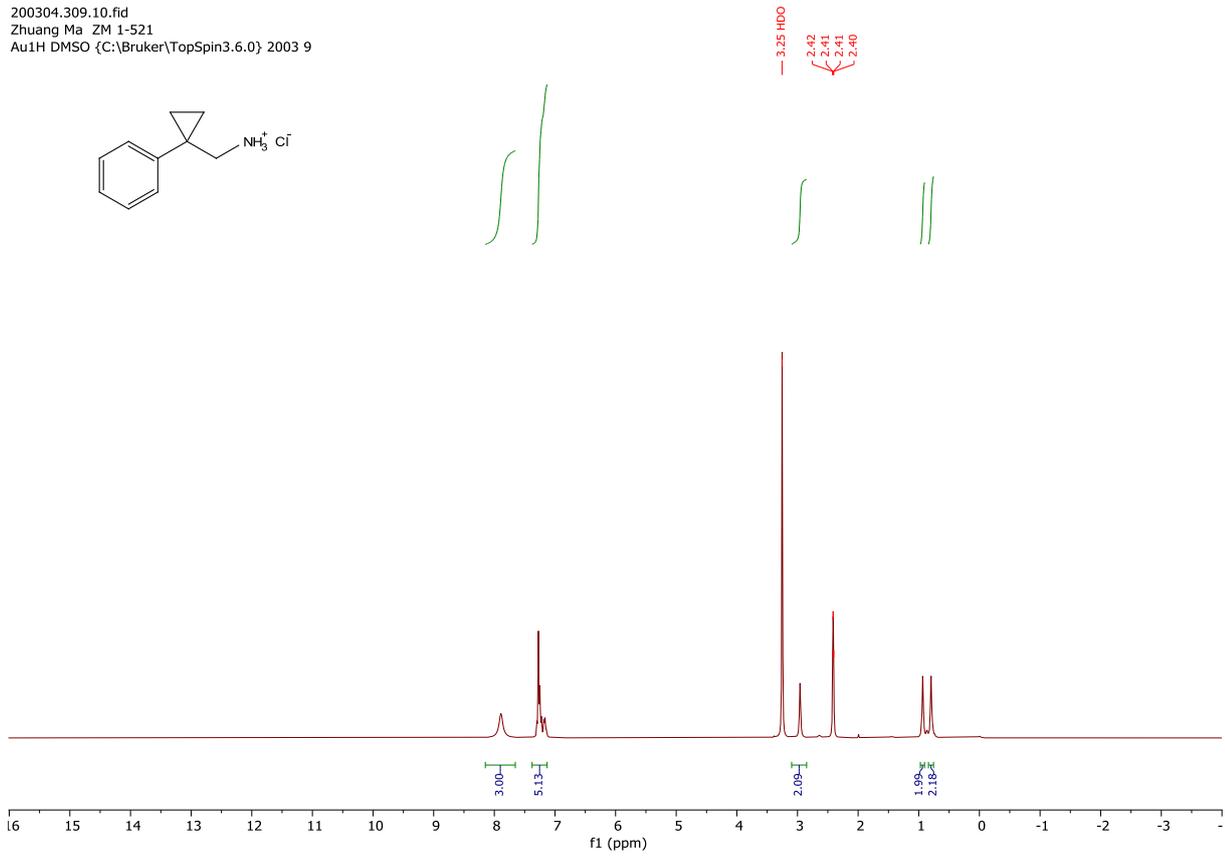
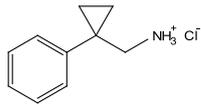
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2204 17



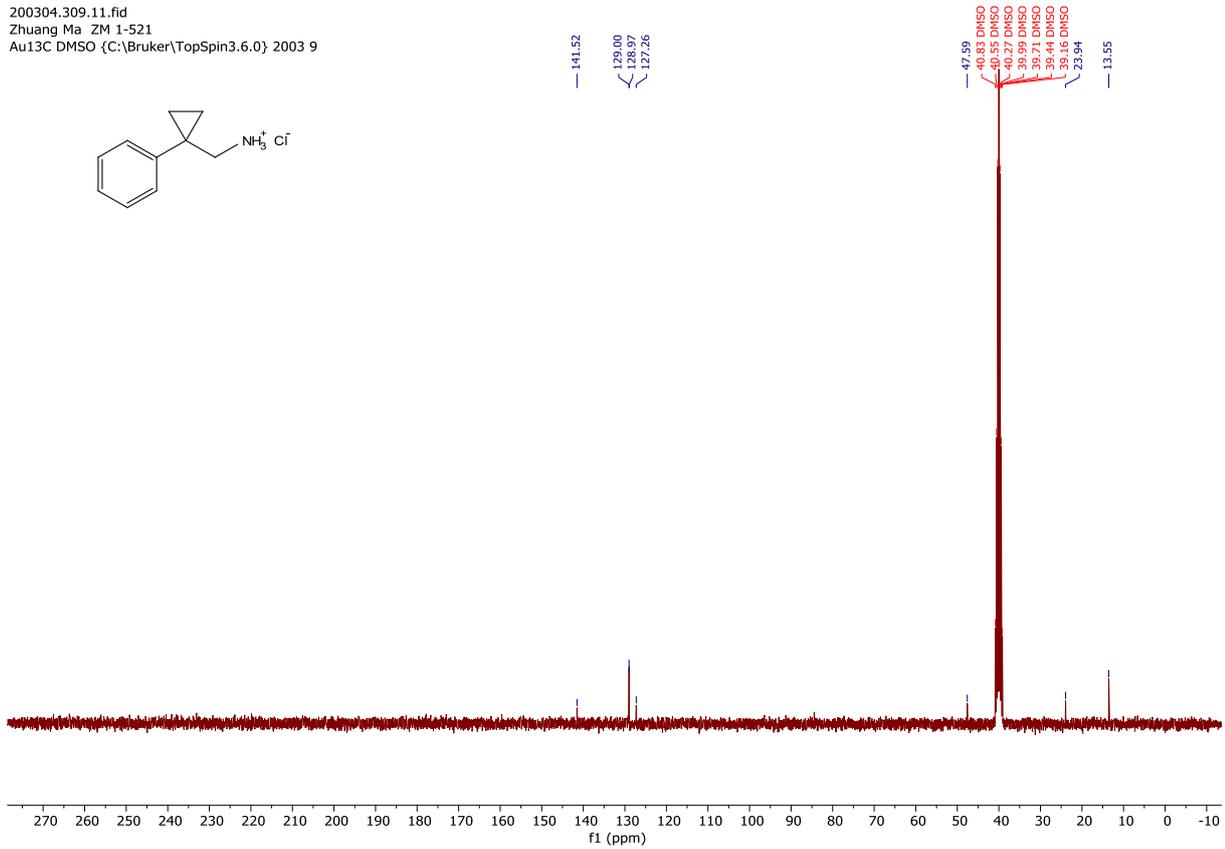
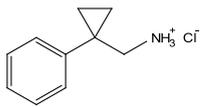
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Ma/ ZM-154  
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2204 17



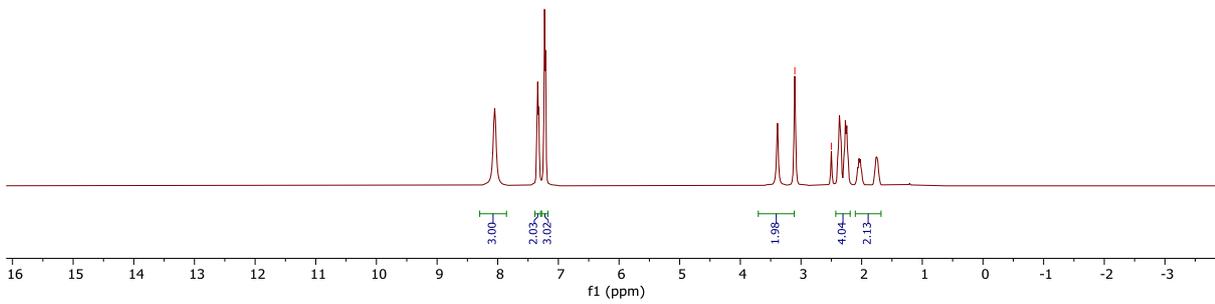
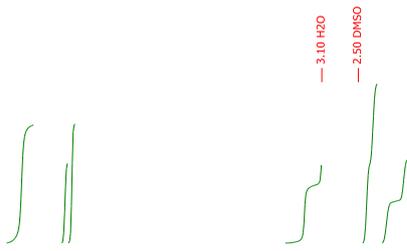
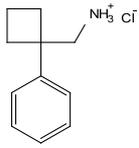
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Au1H DMSO {C:\Bruker\TopSpin3.6.0} 2003 9



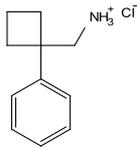
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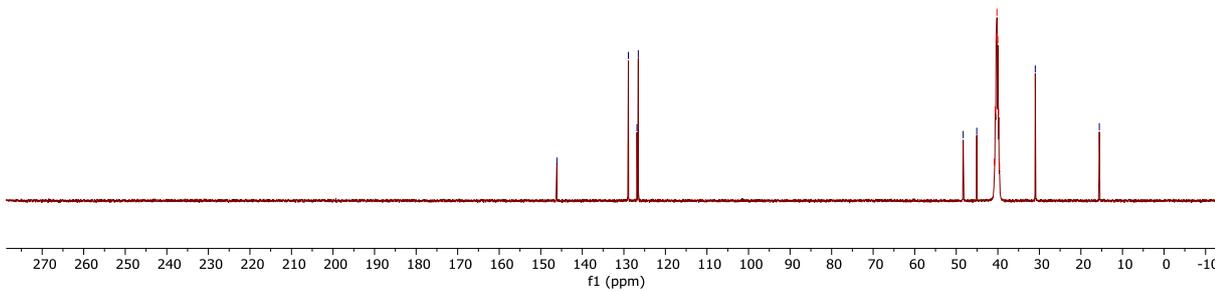
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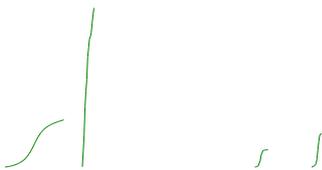
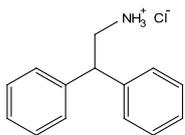
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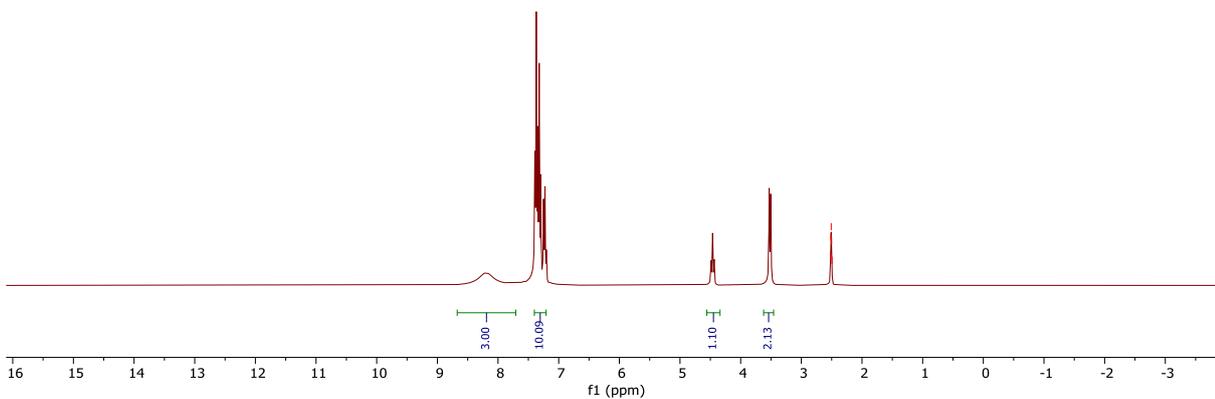
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128.92  
126.84  
126.53  
48.24  
48.07  
48.04 DMSO  
40.63 DMSO  
40.42 DMSO  
40.21 DMSO  
40.00 DMSO  
39.80 DMSO  
39.59 DMSO  
30.59  
15.57



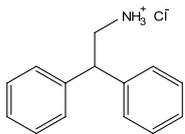
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PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 55



2.52 DMSO  
2.51 DMSO  
2.51 DMSO  
2.49 DMSO

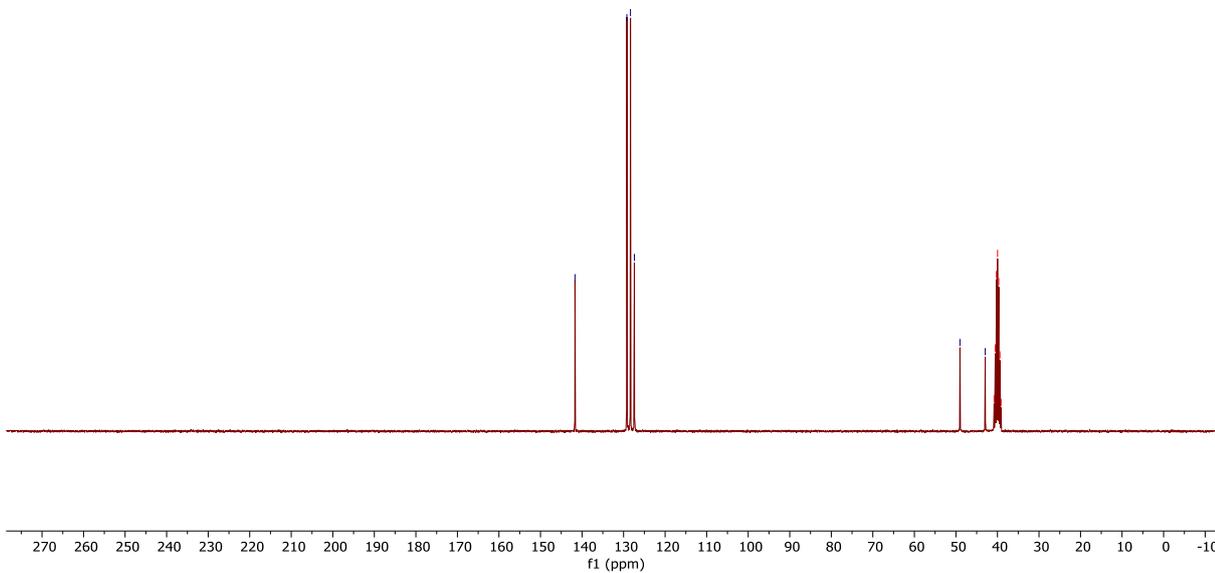


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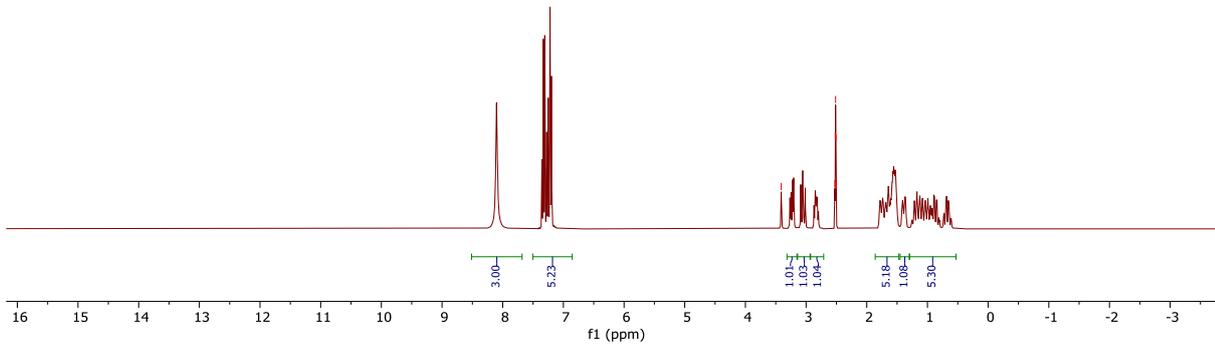
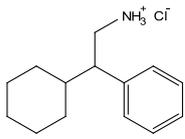


141.70  
129.21  
128.34  
127.41

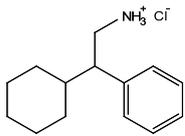
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42.94  
40.80 DMSO  
40.53 DMSO  
40.25 DMSO  
39.97 DMSO  
38.66 DMSO  
38.41 DMSO  
35.14 DMSO



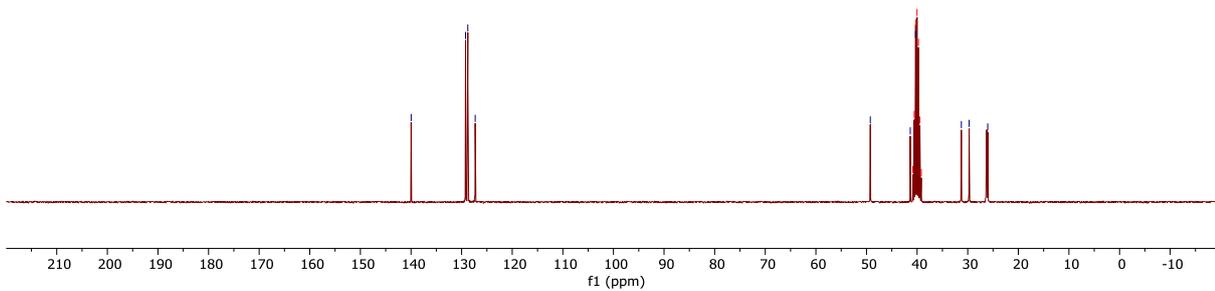
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Zhaung Ma ZM1-456  
Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2105 21



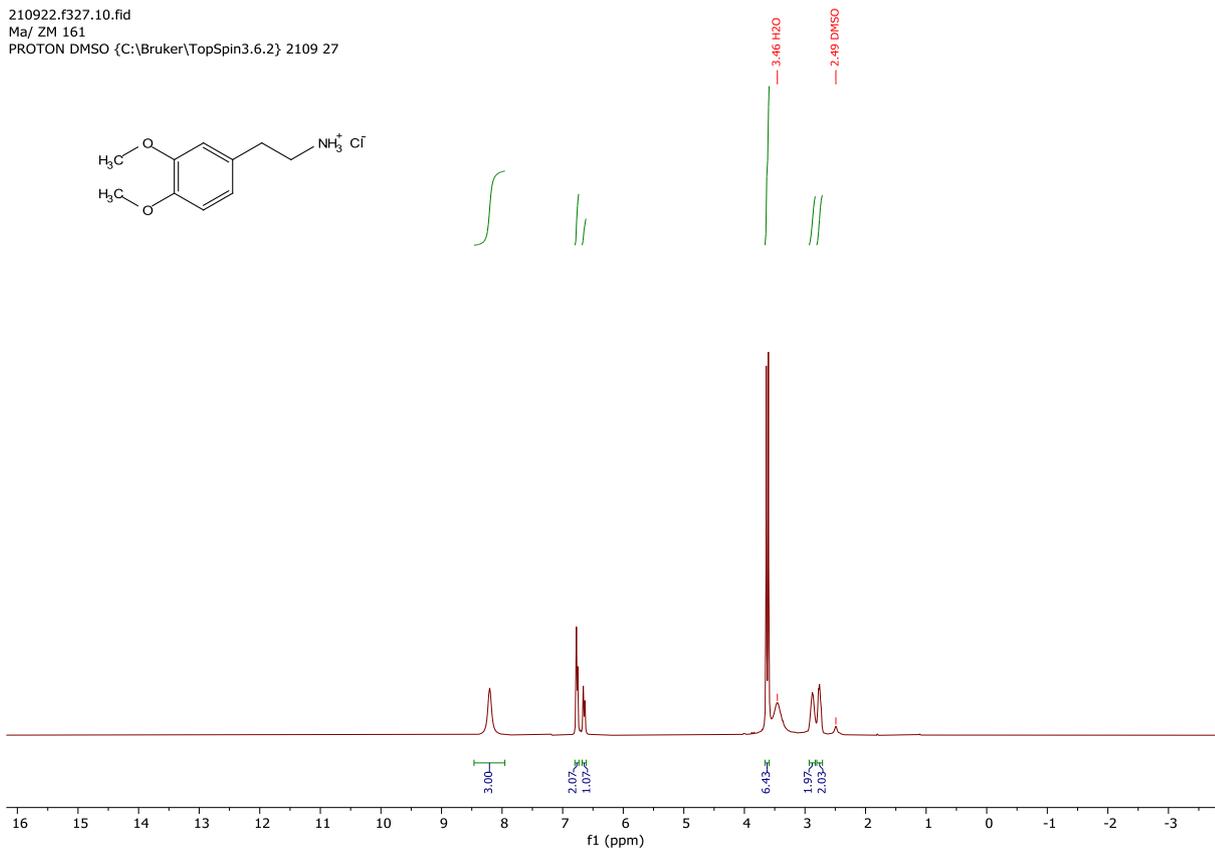
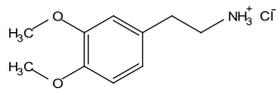
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Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2105 21



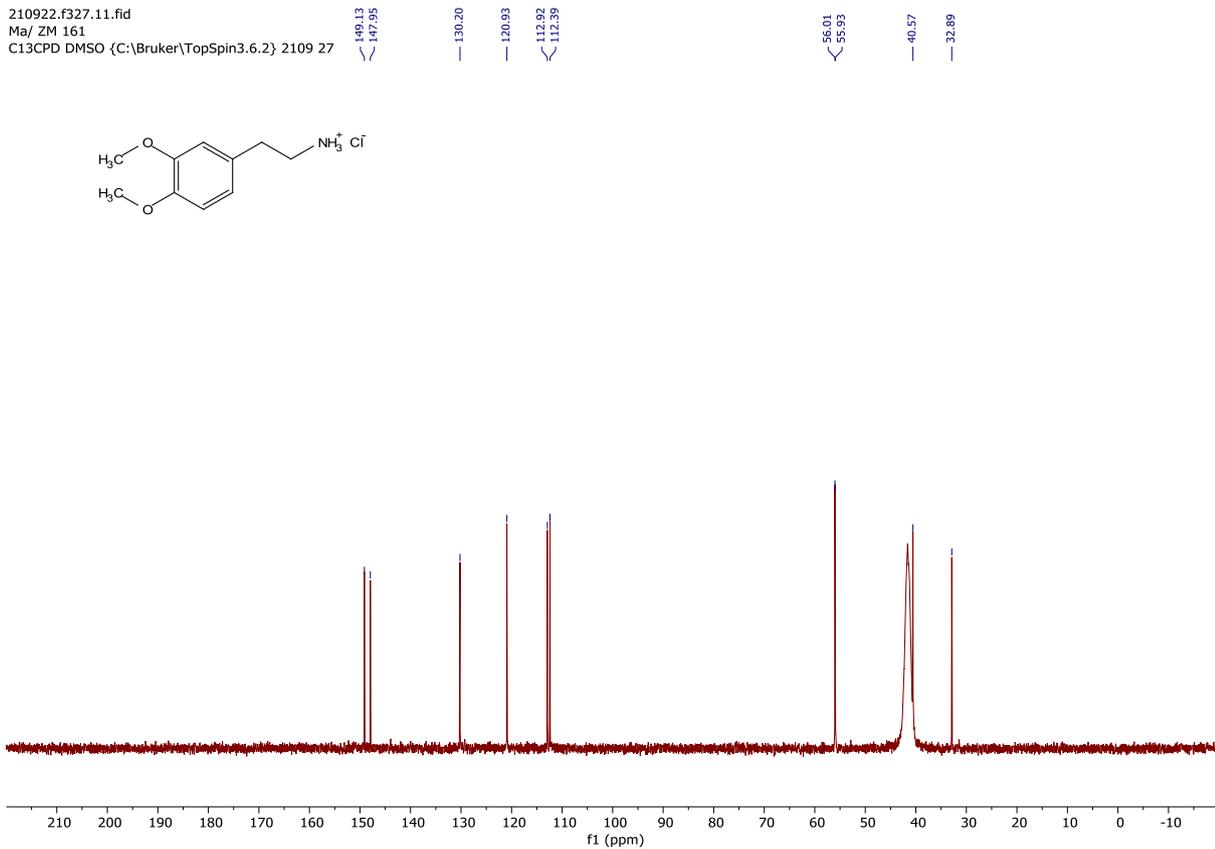
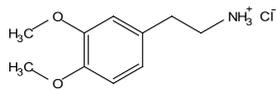
139.97  
129.22  
128.77  
127.31  
49.25  
41.35  
40.84 DMSO  
40.57 DMSO  
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39.46 DMSO  
39.18 DMSO  
31.26  
29.72  
26.04



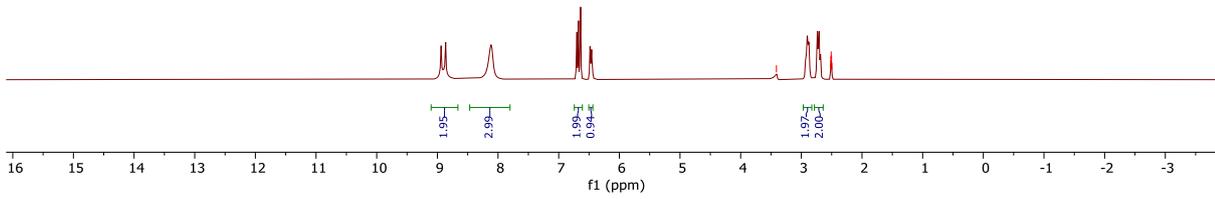
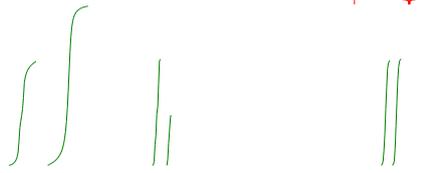
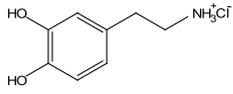
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Ma/ ZM 161  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2109 27



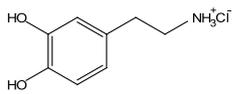
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C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2109 27



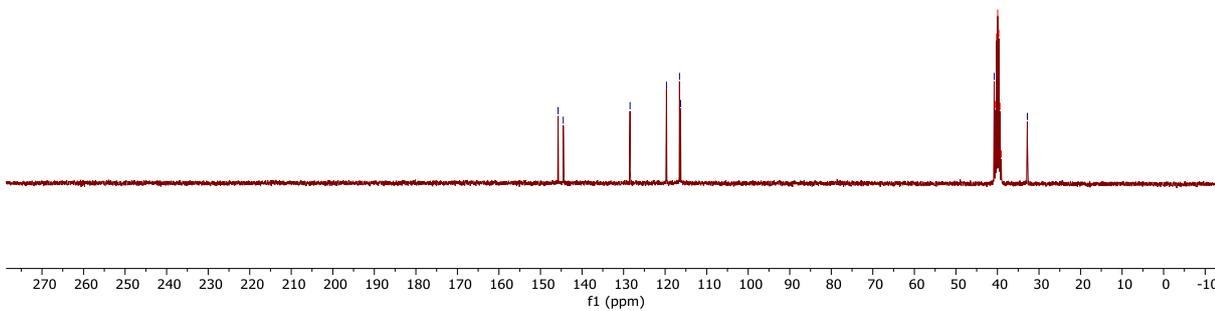
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Au1H DMSO {C:\Bruker\TopSpin3.6.2} 2108 17



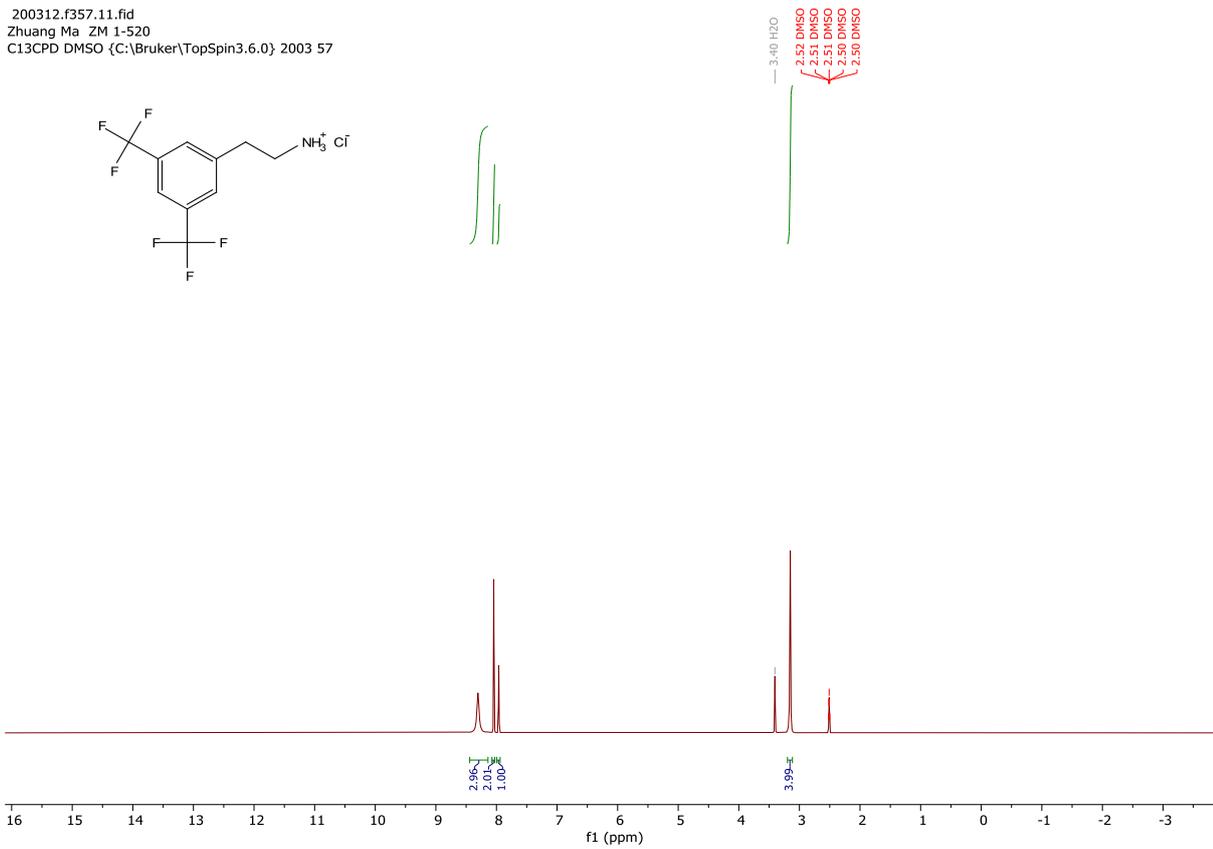
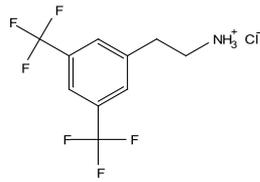
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Zhuang Ma ZM 153  
Au13C DMSO {C:\Bruker\TopSpin3.6.2} 2108 17



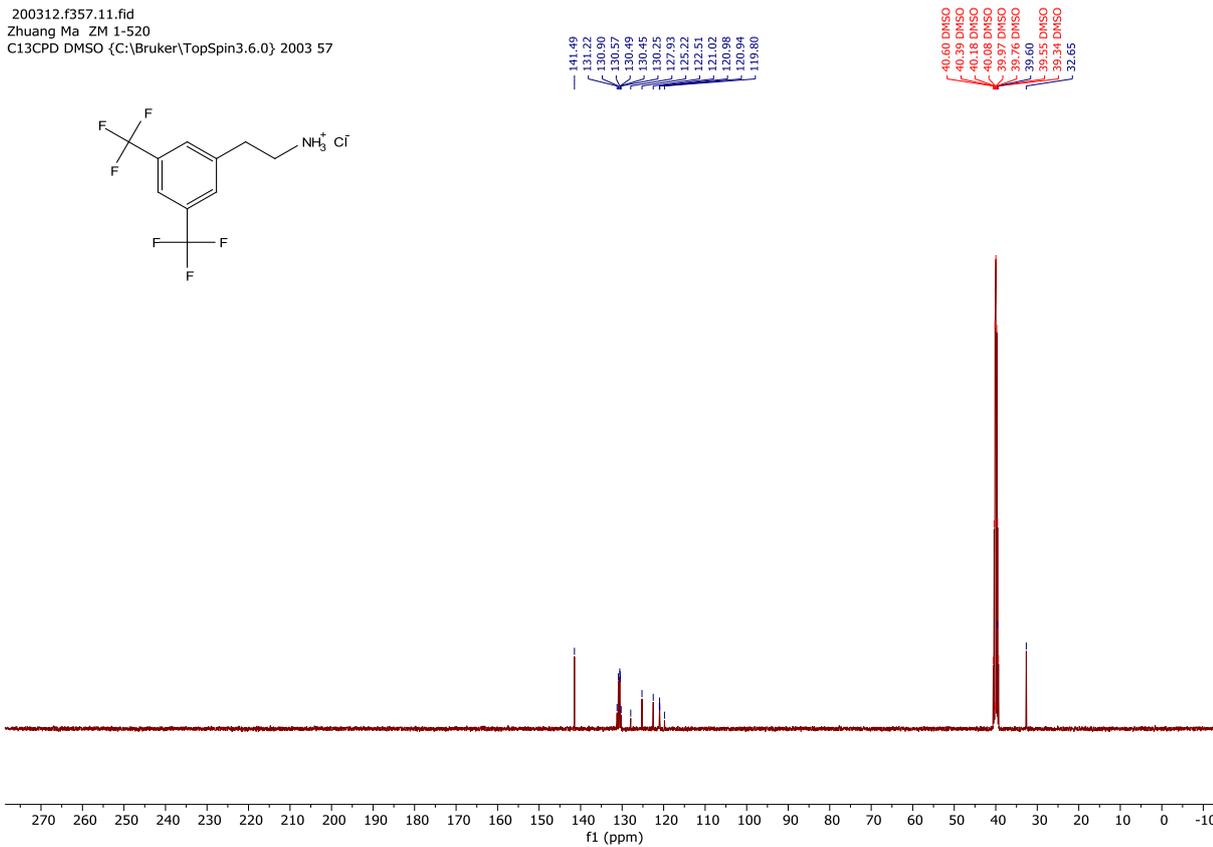
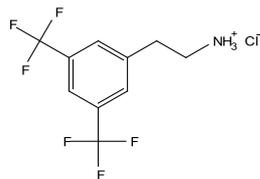
145.76  
144.52  
128.45  
119.67  
116.54  
116.28  
40.78  
40.50 DMSO  
40.22 DMSO  
39.66 DMSO  
39.39 DMSO  
39.11 DMSO  
32.80



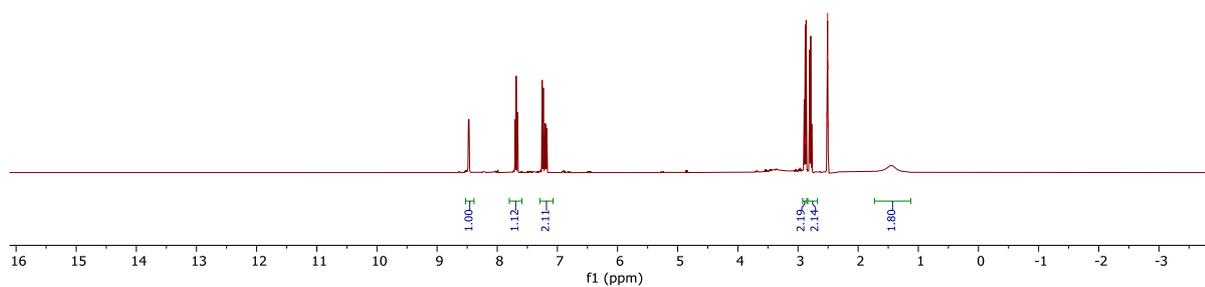
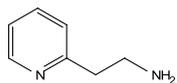
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Zhuang Ma ZM 1-520  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 57



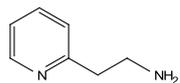
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Zhuang Ma ZM 1-520  
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 57



200228.412.10.fid  
Zhuang Ma ZM 1-336  
Au1H DMSO {C:\Bruker\TopSpin3.5pl6} 2002 12

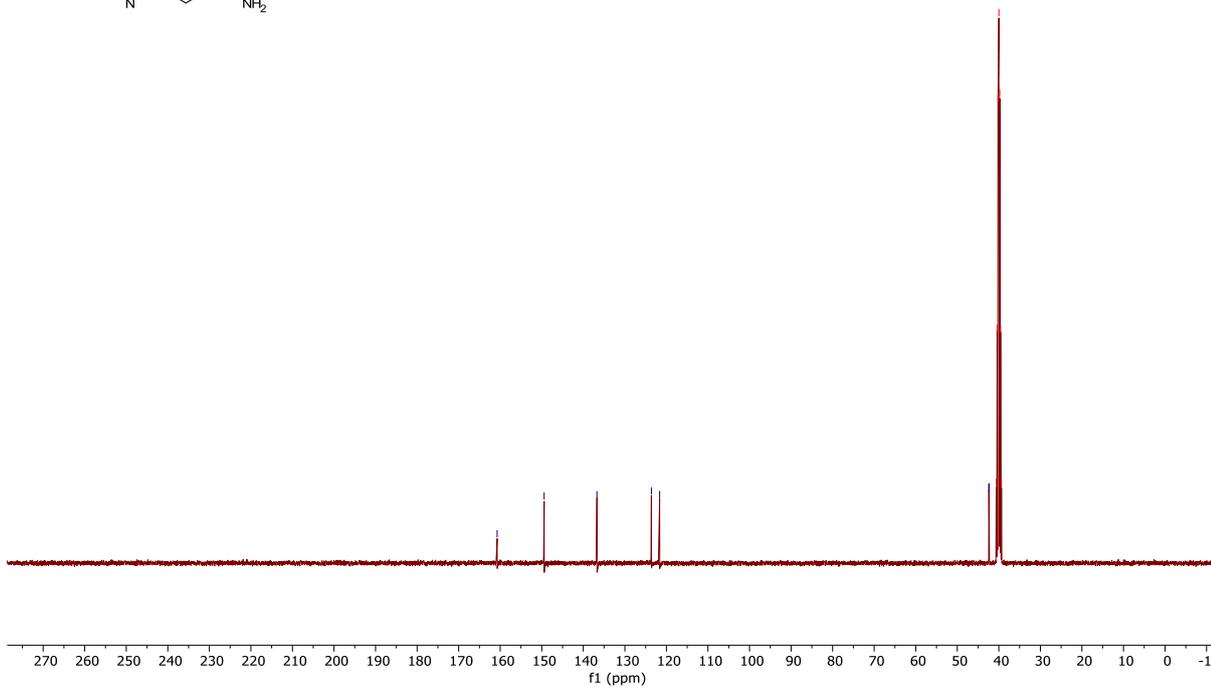


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Au13C DMSO {C:\Bruker\TopSpin3.5pl6} 2002 12

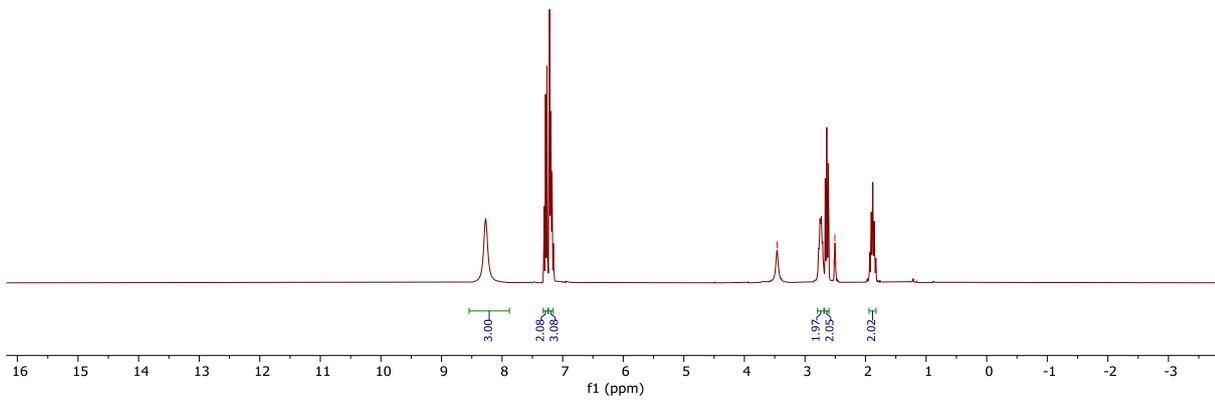
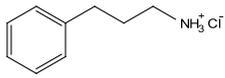


160.74  
149.42  
136.73  
123.62  
121.63

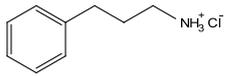
42.39  
42.37  
40.62 DMSO  
40.41 DMSO  
40.20 DMSO  
39.99 DMSO  
39.77 DMSO  
39.56 DMSO



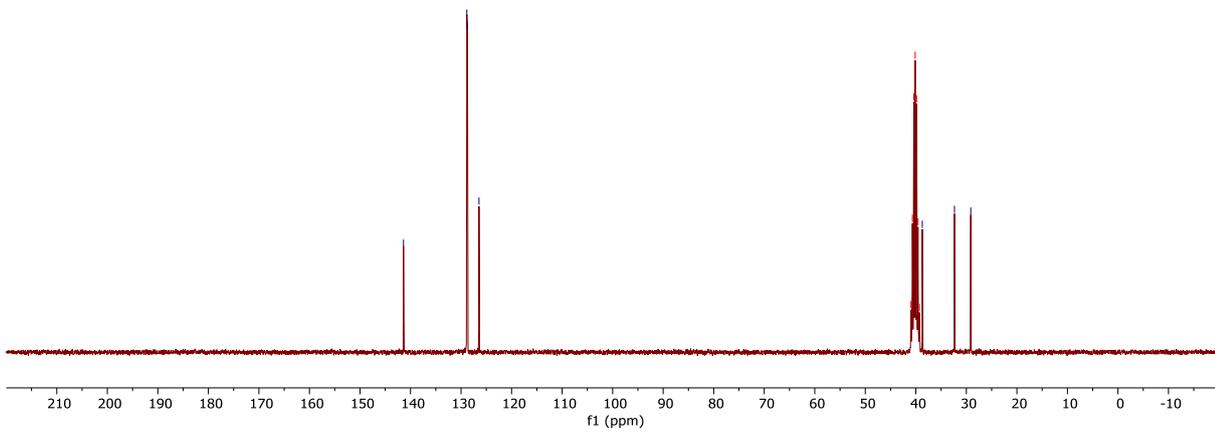
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Ma/ ZM 162  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2109 28



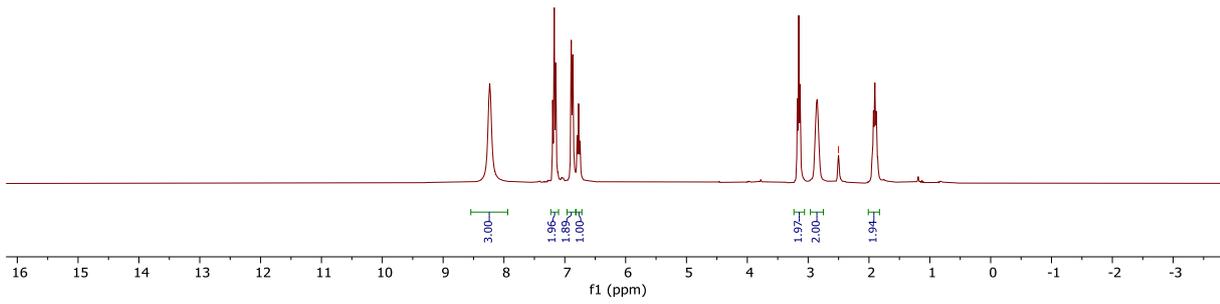
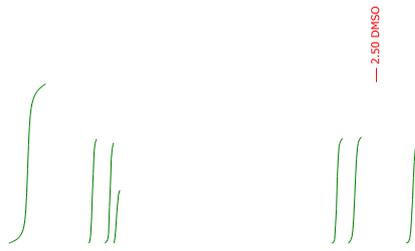
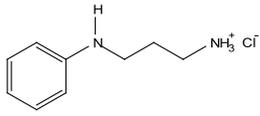
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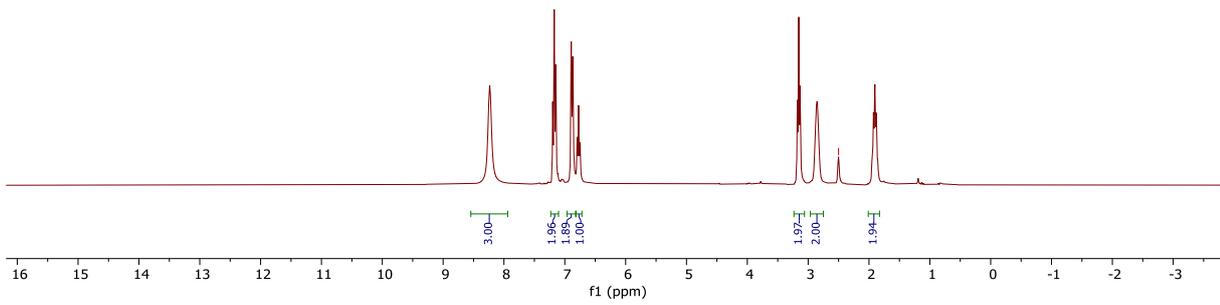
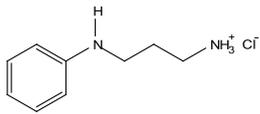
141.38  
128.85  
128.74  
126.45  
40.96 DMSO  
40.68 DMSO  
40.40 DMSO  
40.12 DMSO  
39.85 DMSO  
39.57 DMSO  
39.29 DMSO  
38.92  
38.46  
29.13



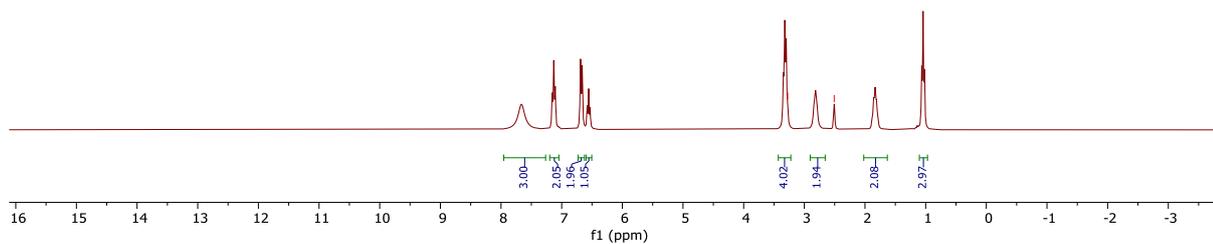
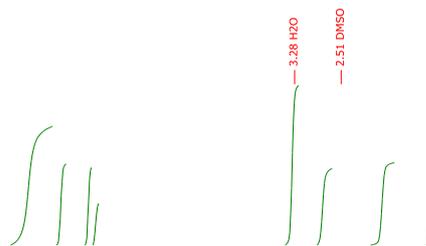
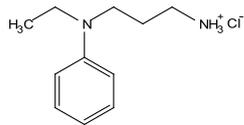
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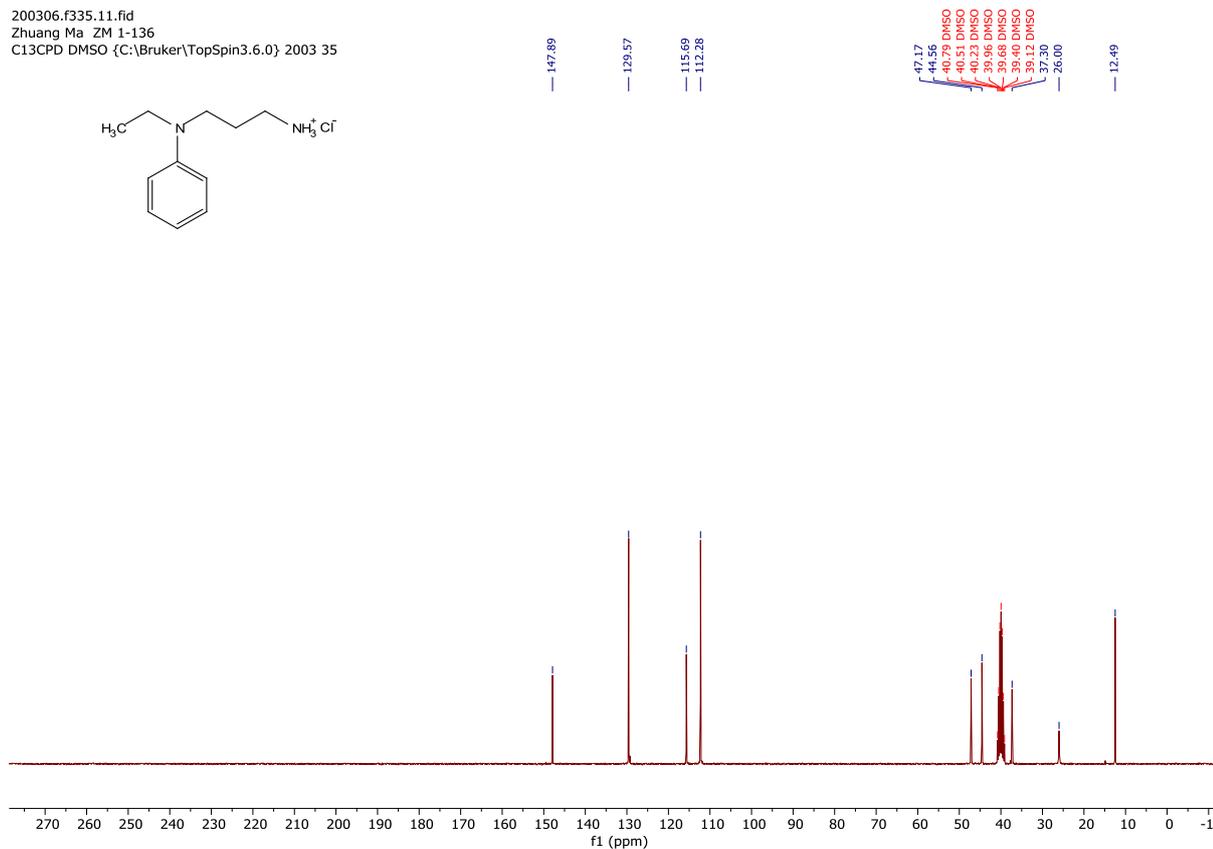
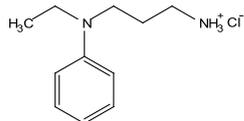
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Ma/ ZM 163  
PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2109 29



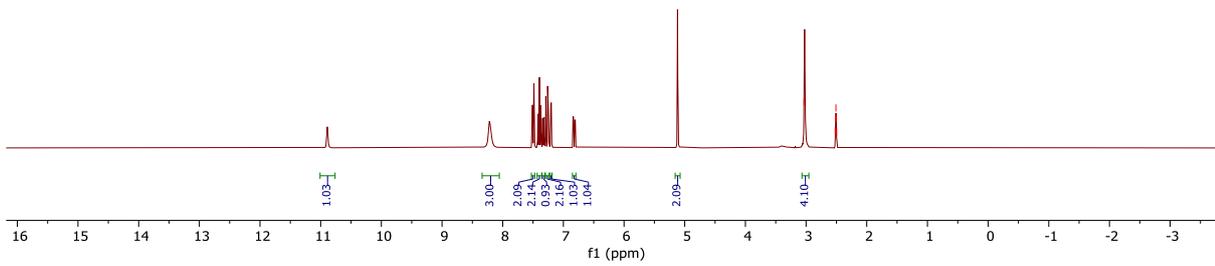
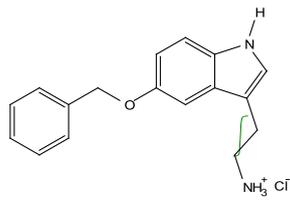
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PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 35



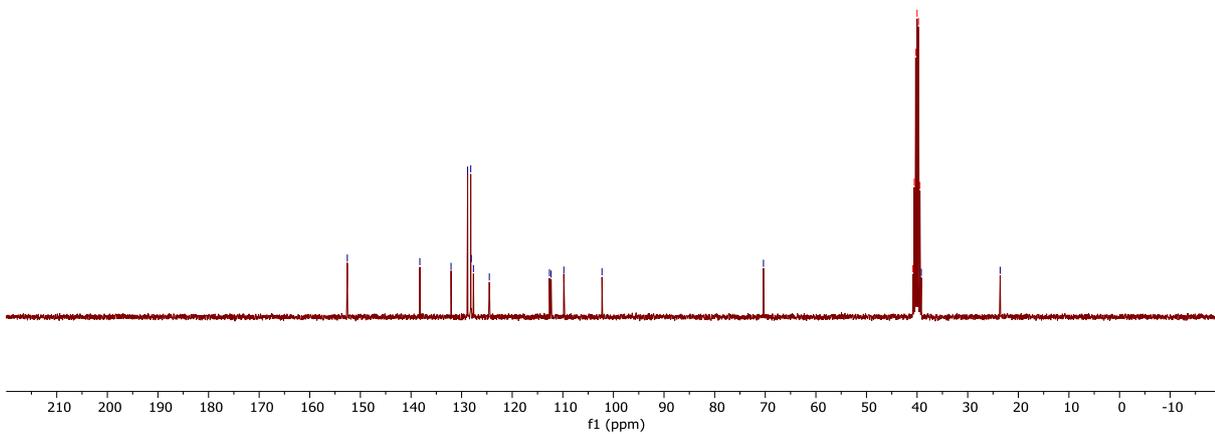
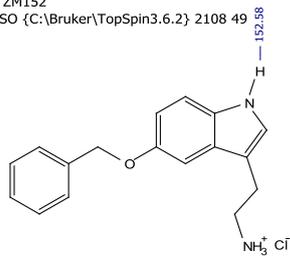
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C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 35



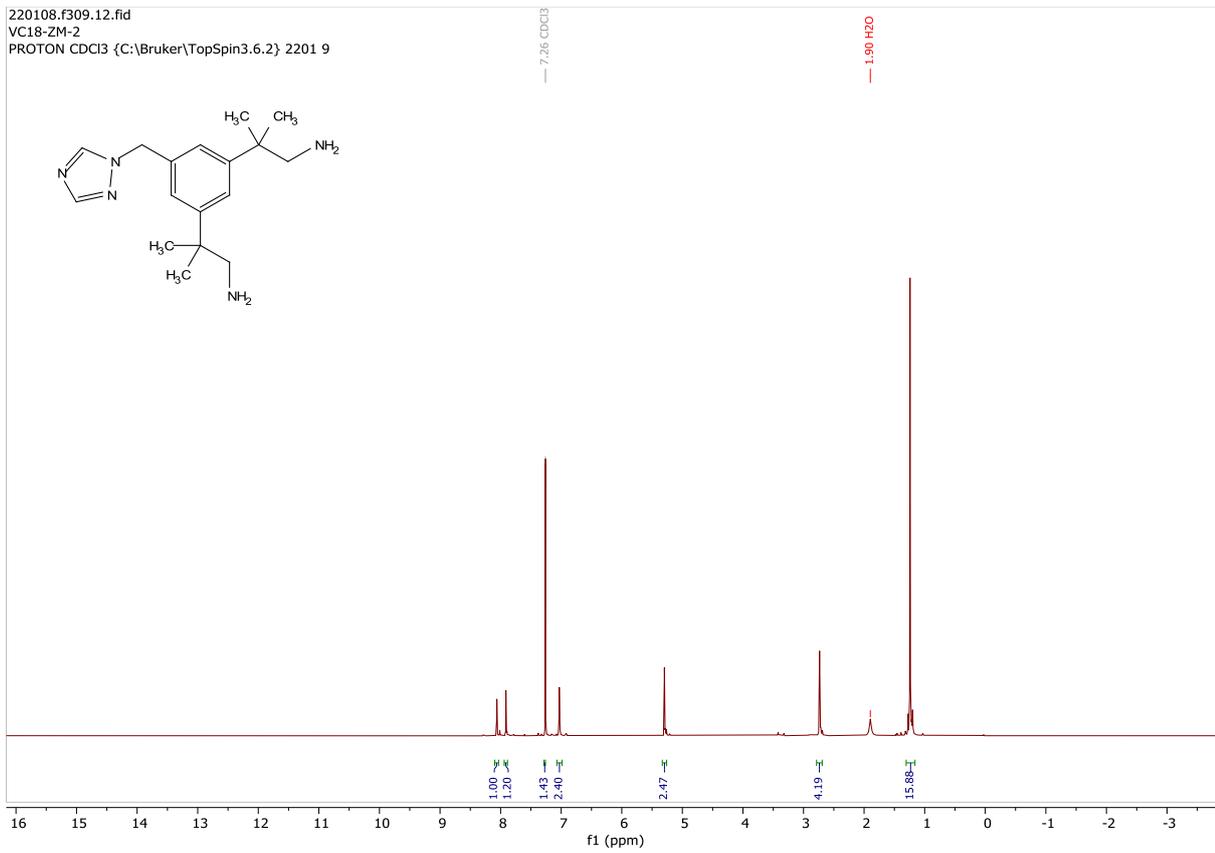
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PROTON DMSO {C:\Bruker\TopSpin3.6.2} 2108 49



210819.f349.11.fid  
Zhuang Ma\_ZM152  
C13CPD DMSO {C:\Bruker\TopSpin3.6.2} 2108 49



220108.f309.12.fid  
VC18-ZM-2  
PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2201 9



220108.f309.11.fid  
VC18-ZM-2  
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2201 9

