

Supplementary Information

Copper-Mediated C(sp²)-H Amination Using TMSN₃ as a Nitrogen Source: Redox-neutral Access to Primary Anilines

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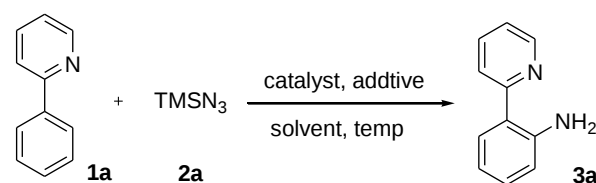
Table of Contents

I. General information.....	2
II. Screening of reaction conditions for the synthesis of 3a	2
III. Mechanistic studies	3
IV. General procedures and characterization of the products.....	4
(1) General procedure for the synthesis of 3	4
(2) Characterization of the products.....	4
V. Copies of ¹ H NMR and ¹³ C NMR Spectra.....	11

I. General information

All reagents were purchased without further purification unless otherwise noted. DCE was distilled under reduced pressure before use. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF254). Visualization of the developed plates was performed under UV light (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^1H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. ^1H and ^{13}C NMR spectra are recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) are reported in ppm referenced to an internal tetramethylsilane standard or the DMSO- d_6 residual peak (δ 2.50) for ^1H NMR. Chemical shifts of ^{13}C NMR are reported relative to CDCl_3 (δ 77.0) or DMSO- d_6 (δ 39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, J , were reported in Hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS Spectrometer.

II. Screening of reaction conditions for the synthesis of 3a

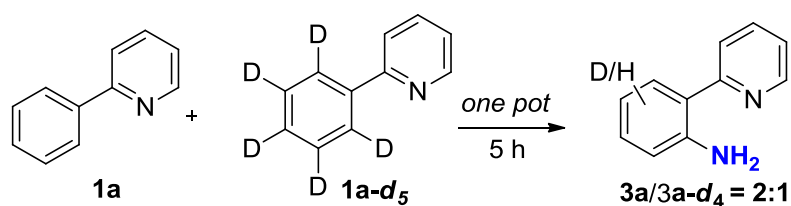


entry	catalyst (equiv)	additive (equiv)	solvent	time (h)	tem. ($^{\circ}\text{C}$)	yield (%)
1	CuTc (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	120	24
2	CuCl (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	120	nr
3	CuBr (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	120	nr
4	CuI (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	120	nr
5	CuOAc (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	120	nr
6	$\text{Cu}(\text{TFA})_2$ (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	115	26
7	$\text{Cu}(\text{OTf})_2$ (0.3)	$\text{CF}_3\text{SO}_3\text{H}$ (1.0)	DCB	24	115	nr

8	CuOTf (0.3)	CF ₃ SO ₃ H (1.0)	DCB	24	115	nr
9	CuTc (0.3)	TFA (1.0)	DCB	24	115	34
10	CuTc (0.3)	HBF ₄ (1.0)	DCB	24	115	nr
11 ^e	CuTc (0.3)	HFIP (1.0)	DCB	24	115	trace
12	CuTc (0.3)	<i>p</i> -TsOH (1.0)	DCB	24	115	16
13	CuTc (0.3)	3,4,5-trifluorobenzoic acid (1.0)	DCB	24	115	24
14	Cu(TFA) ₂ (1.0)	TFA (1.0)	DCB	12	115	88
15	Cu(OAc) ₂ (1.0)	TFA (1.0)	DCB	12	115	nd
16	CuSO ₄ (1.0)	TFA (1.0)	DCB	12	115	nr
17	CuCl ₂ (1.0)	TFA (1.0)	DCB	12	115	nd
18	Cu(acac) ₂ (1.0)	TFA (1.0)	DCB	12	115	nr
19	Cu(hfacac) ₂ (1.0)	TFA (1.0)	DCB	12	115	48
20	Cu(OTf) ₂ (1.0)	TFA (1.0)	DCB	12	115	nr
21	Cu(ClO ₄) ₂ (1.0)	TFA (1.0)	DCB	12	115	nr
22	Cu(TFA) ₂ (1.0)	TFA (1.0)	DCE	12	115	nd
23	Cu(TFA) ₂ (1.0)	TFA (1.0)	mesitylene	12	115	messy
24	Cu(TFA) ₂ (1.0)	TFA (1.0)	toluene	12	115	52%
25	Cu(TFA) ₂ (1.0)	TFA (1.0)	DMSO	12	115	nd
26	Cu(TFA) ₂ (1.0)	TFA (1.0)	DMF	12	115	nd
27	Cu(TFA) ₂ (1.0)	TFA (1.0)	GDE	12	115	trace
28	Cu(TFA) ₂ (1.0)	TFA (0.5)	DCB	12	115	68
29	Cu(TFA) ₂ (1.0)	TFA (1.2)	DCB	12	115	86
30	Cu(TFA) ₂ (1.0)	TFA (1.5)	DCB	24	115	75
31	Cu(TFA) ₂ (1.0)	TFA (2.0)	DCB	24	115	nd

Reaction conditions: **1a** (0.20 mmol), **2** (0.40 mol), solvent (1.0 mL), sealed tube, Ar, T/ °C, isolated yields, nd = not detected, nr = no reaction. DCB = 1, 2-dichlorobenzene.

III. Mechanistic studies



A mixture of substrate **1a** (0.1 mmol), **1a-d₅** (0.1 mmol), TMSN₃ (0.4 mmol, 2.0 equiv), Cu(TFA)₂ (57.8 mg, 0.2 mmol, 1.0 equiv), TFA (0.2 mmol, 1.0 equiv) in 1, 2-dichlorobenzene (1.0 mL) was stirred at 115 °C in sealed tube under Ar atmosphere. The reaction was cooled down to room temperature after 10h. Saturated aqueous NaCl (10 mL), NH₄OH (1 mL) and EtOAc were added to the reaction mixture successively. The organic phase was further extracted with EtOAc (2 × 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated. The residue was purified by flash chromatography to provide the mixture of **3a** and **3a-d₄**.

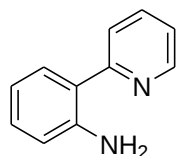
IV. General procedures and characterization of the products

(1) General procedure for the synthesis of **3**

A mixture of substrate **1** (0.2 mmol), TMSN₃ (0.4 mmol, 2.0 equiv), Cu(TFA)₂ (57.8 mg, 0.2 mmol, 1.0 equiv), TFA (0.2 mmol, 1.0 equiv) in 1, 2-dichlorobenzene (1.0 mL) was stirred at 115 °C in sealed tube under Ar atmosphere. The reaction was cooled down to room temperature after complete consumption of the starting material as being monitored by TLC. Saturated aqueous NaCl (10 mL), NH₄OH (1 mL) and EtOAc (10 mL) were added to the reaction mixture successively. The organic phase was separated, and the aqueous phase was further extracted with EtOAc (2 × 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated. The residue was purified by flash chromatography to provide the desired product **3**.

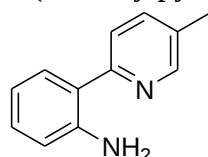
(2) Characterization of the products

2-(pyridin-2-yl)benzenamine (**3a**)



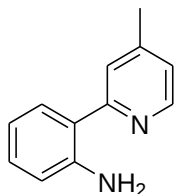
¹H NMR (400 MHz, CDCl₃) δ 8.61 (d, *J* = 4.0 Hz, 1H), 7.74-7.78 (m, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.16-7.19 (m, 2H), 6.75-6.80 (m, 2H), 5.71 (br s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 159.4, 147.8, 146.5, 136.9, 129.9, 129.4, 122.2, 122.2, 120.9, 117.6, 117.2; HRMS (ESI): Exact mass calcd for C₁₁H₁₁N₂ [M+H]⁺ 171.0922, Found 171.0917.

2-(5-methylpyridin-2-yl)benzenamine (**3b**)



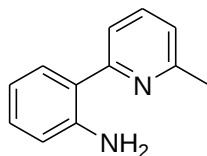
^1H NMR (400 MHz, CDCl_3) δ 8.44 (s, 1H), 7.54-7.56 (m, 2H), 7.48 (d, J = 8.0 Hz, 1H), 7.13-7.17 (m, 1H), 6.74-6.79 (m, 2H), 5.65 (br s, 2H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 156.6, 148.1, 146.3, 137.5, 130.3, 129.5, 129.2, 122.4, 121.7, 117.6, 117.1, 18.1; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$ 185.1079, Found 185.1072.

2-(4-methylpyridin-2-yl)benzenamine (**3c**)



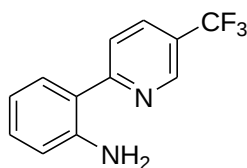
^1H NMR (400 MHz, CDCl_3) δ 8.46 (d, J = 4.0 Hz, 1H), 7.49 (d, J = 8.0 Hz, 1H), 7.46 (s, 1H), 7.14-7.18 (m, 1H), 7.01 (d, J = 5.0 Hz, 1H), 6.74-6.80 (m, 2H), 5.65 (br s, 2H), 2.40 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.4, 147.7, 147.7, 146.7, 129.7, 129.3, 123.0, 122.6, 121.9, 117.5, 117.1, 21.1; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$ 185.1079, Found 185.1075.

2-(6-methylpyridin-2-yl)benzenamine (**3d**)



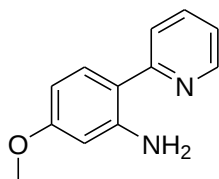
^1H NMR (400 MHz, CDCl_3) δ 7.63-7.67 (m, 1H), 7.50 (d, J = 8.0 Hz, 1H), 7.45 (d, J = 8.0 Hz, 1H), 7.14-7.18 (m, 1H), 7.04 (d, J = 7.6 Hz, 1H), 6.74-6.79 (m, 2H), 5.66 (br s, 2H), 2.59 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.8, 156.5, 146.4, 137.2, 129.7, 129.4, 122.5, 120.4, 119.2, 117.6, 117.2, 24.5; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$ 185.1079, Found 185.1074.

2-(5-(trifluoromethyl)pyridin-2-yl)benzenamine (**3e**)



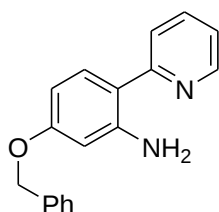
^1H NMR (400 MHz, CDCl_3) δ 8.87 (s, 1H), 7.97 (d, J = 8.5 Hz, 1H), 7.79 (d, J = 8.5 Hz, 1H), 7.56 (d, J = 7.8 Hz, 1H), 7.20-7.24 (m, 1H), 6.76-6.82 (m, 2H), 5.83 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 162.7, 147.2, 144.8 (q, J = 4.1 Hz), 133.7 (q, J = 3.5 Hz), 131.0, 129.5, 123.7 (q, J = 271.9 Hz), 123.4 (q, J = 33.0 Hz), 121.4, 120.2, 117.6, 117.5; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{10}\text{F}_3\text{N}_2$ $[\text{M}+\text{H}]^+$ 239.0796, Found 239.0791.

5-methoxy-2-(pyridin-2-yl)benzenamine (**3f**)



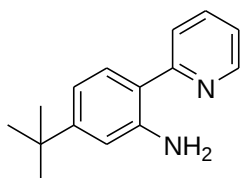
^1H NMR (400 MHz, CDCl_3) δ 8.55 (d, J = 4.0 Hz, 1H), 7.69-7.73 (m, 1H), 7.59 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 8.0 Hz, 1H), 7.09-7.12 (m, 1H), 6.37 (d, J = 8.0 Hz, 1H), 6.27 (s, 1H), 5.93 (br s, 2H), 3.80 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 161.1, 159.3, 148.3, 147.6, 136.7, 130.5, 121.3, 120.1, 115.4, 104.2, 101.4, 55.1; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ 201.1028, Found 201.1023.

5-(benzyloxy)-2-(pyridin-2-yl)benzenamine (**3g**)



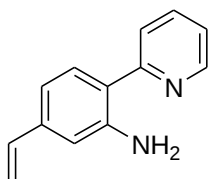
^1H NMR (400 MHz, CDCl_3) δ 8.55 (d, J = 4.0 Hz, 1H), 7.69-7.73 (m, 1H), 7.59 (d, J = 8.0 Hz, 1H), 7.33-7.48 (m, 6H), 7.10-7.12 (m, 1H), 6.44 (d, J = 8.0 Hz, 1H), 6.35 (s, 1H), 5.92 (br s, 2H), 5.08 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 160.3, 159.2, 148.3, 147.6, 137.0, 136.7, 130.5, 128.5, 127.9, 127.4, 121.4, 120.2, 115.6, 104.9, 102.4, 69.8; HRMS (ESI): Exact mass calcd for $\text{C}_{18}\text{H}_{17}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ 277.1341, Found 277.1341.

5-tert-butyl-2-(pyridin-2-yl)benzenamine (**3h**)



^1H NMR (400 MHz, CDCl_3) δ 8.58 (d, J = 4.0 Hz, 1H), 7.71-7.73 (m, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 8.0 Hz, 1H), 7.14-7.15 (m, 1H), 6.83 (d, J = 8.0 Hz, 1H), 6.78 (s, 1H), 5.77 (br s, 2H), 1.32 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.4, 153.2, 147.8, 146.3, 136.7, 129.0, 121.8, 120.5, 119.5, 115.1, 114.3, 34.5, 31.1; HRMS (ESI): Exact mass calcd for $\text{C}_{15}\text{H}_{19}\text{N}_2$ $[\text{M}+\text{H}]^+$ 227.1548, Found 227.1545.

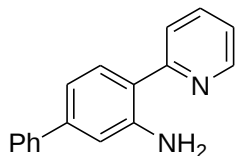
2-(pyridin-2-yl)-5-vinylbenzenamine (**3i**)



^1H NMR (400 MHz, CDCl_3) δ 8.60 (d, J = 4.0 Hz, 1H), 7.73-7.77 (m, 1H), 7.66 (d, J

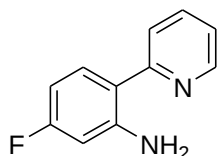
= 8.0 Hz, 1H), 7.50 (d, J = 8.0 Hz, 1H), 7.15-7.18 (m, 1H), 6.87 (d, J = 8.0 Hz, 1H), 6.79 (s, 1H), 6.65 (dd, J = 17.6, 10.8 Hz, 1H), 5.79 (br s, 2H), 5.76 (d, J = 17.6 Hz, 1H), 5.26 (d, J = 10.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.1, 147.8, 146.7, 139.0, 136.8, 136.6, 129.5, 121.9, 121.5, 120.8, 115.6, 115.0, 114.2; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$ 197.1079, Found 197.1075.

4-(pyridin-2-yl)-[1,1'-biphenyl]-3-amine (**3j**)



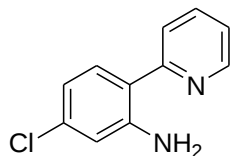
^1H NMR (400 MHz, CDCl_3) δ 8.62 (d, J = 4.0 Hz, 1H), 7.70-7.79 (m, 2H), 7.60-7.64 (m, 3H), 7.42-7.46 (m, 2H), 7.34-7.37 (m, 1H), 7.17-7.20 (m, 1H), 7.03 (d, J = 8.0 Hz, 1H), 7.00 (s, 1H), 5.89 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.4, 147.8, 147.1, 142.9, 141.0, 136.6, 129.6, 128.6, 127.3, 127.0, 121.8, 121.2, 120.6, 116.6, 115.7; HRMS (ESI): Exact mass calcd for $\text{C}_{17}\text{H}_{15}\text{N}_2$ $[\text{M}+\text{H}]^+$ 247.1235, Found 247.1230.

5-fluoro-2-(pyridin-2-yl)benzenamine (**3k**)



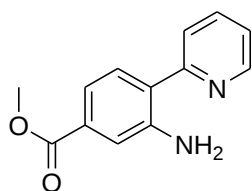
^1H NMR (400 MHz, CDCl_3) δ 8.58 (d, J = 4.0 Hz, 1H), 7.73-7.77 (m, 1H), 7.59 (d, J = 8.0 Hz, 1H), 7.46-7.50 (m, 1H), 7.15-7.18 (m, 1H), 6.43-6.49 (m, 2H), 5.94 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 164.0 (d, J = 246.2 Hz), 158.7, 148.5 (d, J = 11.3 Hz), 147.8, 136.9, 130.9 (d, J = 10.5 Hz), 121.8, 120.8, 118.2 (d, J = 2.3 Hz), 104.5 (d, J = 21.9 Hz), 103.0 (d, J = 24 Hz); HRMS (ESI): Exact mass calcd for $\text{C}_{11}\text{H}_{10}\text{FN}_2$ $[\text{M}+\text{H}]^+$ 189.0828, Found 189.0825.

5-chloro-2-(pyridin-2-yl)benzenamine (**3l**)



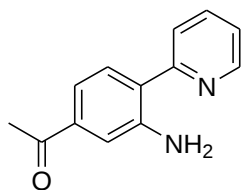
^1H NMR (400 MHz, CDCl_3) δ 8.59 (d, J = 4.0 Hz, 1H), 7.73-7.77 (m, 1H), 7.61 (d, J = 8.0 Hz, 1H), 7.44 (d, J = 8.0 Hz, 1H), 7.17-7.20 (m, 1H), 6.72-6.74 (m, 2H), 5.91 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.5, 147.8, 147.7, 137.0, 135.4, 130.4, 121.9, 121.1, 120.3, 117.5, 116.5; HRMS (ESI): Exact mass calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_2$ $[\text{M}+\text{H}]^+$ 205.0533, Found 205.0530.

Methyl 3-amino-4-(pyridin-2-yl)benzoate (**3m**)



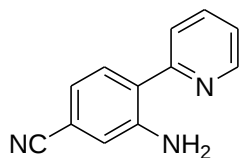
^1H NMR (400 MHz, CDCl_3) δ 8.63 (d, J = 4.0 Hz, 1H), 7.77-7.81 (m, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H), 7.40-7.44 (m, 2H), 7.21-7.24 (m, 1H), 5.85 (br s, 2H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 167.0, 158.4, 148.0, 146.5, 137.0, 131.0, 129.3, 125.7, 122.6, 121.6, 118.3, 118.2, 52.0; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{13}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 229.0977, Found 229.0970.

1-(3-amino-4-(pyridin-2-yl)phenyl)ethanone (**3n**)



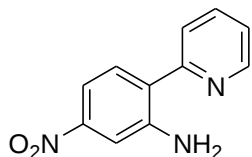
^1H NMR (400 MHz, CDCl_3) δ 8.64 (d, J = 4.0 Hz, 1H), 7.78-7.82 (m, 1H), 7.70 (d, J = 8.0 Hz, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.33-7.34 (m, 2H), 7.22-7.24 (m, 1H), 5.98 (br s, 2H), 2.60 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.1, 158.3, 148.0, 146.7, 138.0, 137.0, 129.5, 125.9, 122.6, 121.7, 117.4, 116.6, 26.7; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{13}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$ 213.1028, Found 213.1028.

3-amino-4-(pyridin-2-yl)benzonitrile (**3o**)

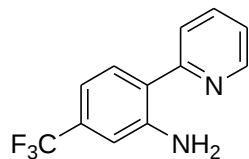


^1H NMR (400 MHz, CDCl_3) δ 8.64 (d, J = 4.0 Hz, 1H), 7.80-7.84 (m, 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H), 7.26-7.28 (m, 1H), 7.00-7.03 (m, 2H), 6.01 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.9, 148.0, 147.1, 137.0, 129.8, 125.5, 122.3, 121.9, 120.3, 120.0, 118.7, 113.3; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{10}\text{N}_3$ $[\text{M}+\text{H}]^+$ 196.0875, Found 196.0871.

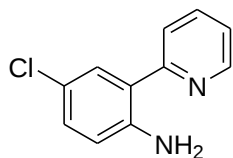
5-nitro-2-(pyridin-2-yl)benzenamine (**3p**)



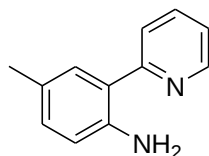
^1H NMR (400 MHz, CDCl_3) δ 8.66 (d, J = 4.0 Hz, 1H), 7.82-7.86 (m, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.56-7.58 (m, 2H), 7.27-7.30 (m, 1H), 6.14 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.4, 148.7, 148.1, 147.4, 137.3, 130.0, 126.8, 122.8, 122.3, 111.7, 111.3; HRMS (ESI): Exact mass calcd for $\text{C}_{11}\text{H}_{10}\text{N}_3\text{O}_2$ $[\text{M}+\text{H}]^+$ 216.0773, Found 216.0768.

5-(trifluoromethyl)-2-(pyridin-2-yl)benzenamine (**3q**)

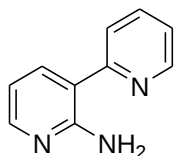
^1H NMR (400 MHz, CDCl_3) δ 8.64 (d, $J = 4.0$ Hz, 1H), 7.79-7.82 (m, 1H), 7.68 (d, $J = 8.0$ Hz, 1H), 7.60 (d, $J = 8.0$ Hz, 1H), 7.23-7.26 (m, 1H), 6.98-7.00 (m, 2H), 5.95 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.2, 148.0, 146.7, 137.1, 131.6 (q, $J = 32$ Hz), 129.7, 124.5, 124.0 (q, $J = 270$ Hz), 122.4, 121.7, 113.7 (q, $J = 3.8$ Hz), 113.6 (q, $J = 3.8$ Hz); HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{10}\text{F}_3\text{N}_2$ $[\text{M}+\text{H}]^+$ 239.0796, Found 239.0791.

4-chloro-2-(pyridin-2-yl)benzenamine (**3r**)

^1H NMR (400 MHz, CDCl_3) δ 8.59 (d, $J = 4.0$ Hz, 1H), 7.73-7.77 (m, 1H), 7.61 (d, $J = 8.0$ Hz, 1H), 7.44 (d, $J = 8.0$ Hz, 1H), 7.17-7.20 (m, 1H), 6.72-6.74 (m, 2H), 5.91 (br s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.5, 147.8, 147.7, 137.0, 135.4, 130.4, 121.9, 121.1, 120.3, 117.5, 116.5; HRMS (ESI): Exact mass calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_2$ $[\text{M}+\text{H}]^+$ 205.0533, Found 205.0529.

4-methyl-2-(pyridin-2-yl)benzenamine (**3s**)

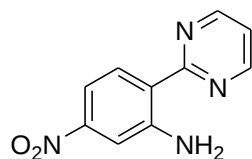
^1H NMR (400 MHz, CDCl_3) δ 8.60 (d, $J = 4.0$ Hz, 1H), 7.73-7.77 (m, 1H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.34 (s, 1H), 7.15-7.18 (m, 1H), 7.00 (d, $J = 8.0$ Hz, 1H), 6.69 (d, $J = 8.0$ Hz, 1H), 5.47 (br s, 2H), 2.30 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 159.7, 147.9, 144.2, 136.5, 130.6, 129.7, 126.6, 122.6, 122.1, 120.7, 117.3, 20.3; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$ 185.1079, Found 185.1075.

3-(pyridin-2-yl)pyridin-2-amine (**3t**)

^1H NMR (400 MHz, CDCl_3) δ 8.62 (d, $J = 4.0$ Hz, 1H), 8.11 (d, $J = 4.0$ Hz, 1H), 7.84 (d, $J = 8.0$ Hz, 1H), 7.75-7.77 (m, 1H), 7.69 (d, $J = 8.0$ Hz, 1H), 7.20-7.23 (m, 1H), 6.75 (br s, 2H), 6.69-6.72 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.7, 157.5, 149.1, 147.9, 136.7, 136.3, 121.4, 121.2, 116.3, 113.2; HRMS (ESI): Exact mass

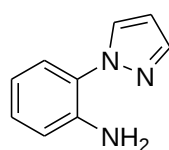
calcd for $C_{10}H_{10}N_3$ $[M+H]^+$ 172.0875, Found 172.0870.

5-nitro-2-(pyrimidin-2-yl)benzenamine (**3u**)



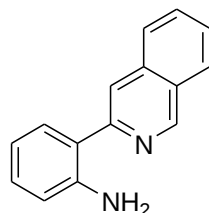
1H NMR (400 MHz, $CDCl_3$) δ 8.84 (d, J = 4.0 Hz, 2H), 8.64 (d, J = 8.8 Hz, 1H), 7.58 (s, 1H), 7.54 (d, J = 8.8 Hz, 1H), 7.21-7.24 (m, 1H), 6.63 (br s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.5, 156.4, 149.9, 149.0, 132.2, 123.1, 118.5, 111.5, 110.7; HRMS (ESI): Exact mass calcd for $C_{10}H_9N_4O_2$ $[M+H]^+$ 217.0726, Found 217.0721.

2-(1H-pyrazol-1-yl)benzenamine (**3v**)



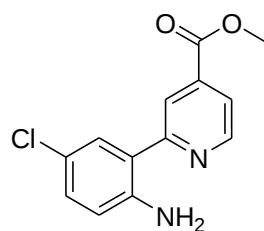
1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 12.0 Hz, 2H), 7.13-7.19 (m, 2H), 6.76-6.84 (m, 2H), 6.44 (s, 1H), 4.67 (br s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.2, 140.3, 129.6, 128.4, 126.7, 124.1, 117.9, 117.3, 106.2; HRMS (ESI): Exact mass calcd for $C_9H_{10}N_3$ $[M+H]^+$ 160.0875, Found 160.0870.

2-(isoquinolin-3-yl)benzenamine (**3w**)



1H NMR (400 MHz, $CDCl_3$) δ 9.28 (s, 1H), 7.98 (d, J = 8.0 Hz, 1H), 7.95 (s, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.67-7.71 (m, 1H), 7.57-7.60 (m, 2H), 7.18-7.22 (m, 1H), 6.79-6.87 (m, 2H), 5.22 (br s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.3, 150.9, 146.2, 136.9, 130.5, 129.9, 129.6, 127.5, 127.0, 126.9, 126.8, 123.6, 118.6, 118.1, 117.1; HRMS (ESI): Exact mass calcd for $C_{15}H_{13}N_2$ $[M+H]^+$ 221.1079, Found 221.1074.

Methyl 2-(2-amino-5-chlorophenyl)pyridine-4-carboxylate (**3x**)



1H NMR (400 MHz, $CDCl_3$) δ 8.75 (d, J = 4.0 Hz, 1H), 8.21 (s, 1H), 7.75 (d, J = 4.0

Hz, 1H), 7.58 (s, 1H), 7.14 (d, $J = 8.0$ Hz, 1H), 6.70 (d, $J = 8.0$ Hz, 1H), 5.81 (br s, 2H), 4.00 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 165.5, 159.1, 148.7, 145.4, 138.4, 130.2, 128.8, 122.1, 122.0, 121.3, 120.3, 118.5, 52.8; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 263.0587, Found 263.0584.

V. Copies of ^1H NMR and ^{13}C NMR Spectra

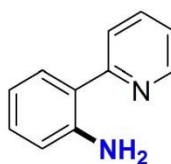
114233

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7.175
7.158
6.807
6.788
6.773
6.753

5.711

1.613

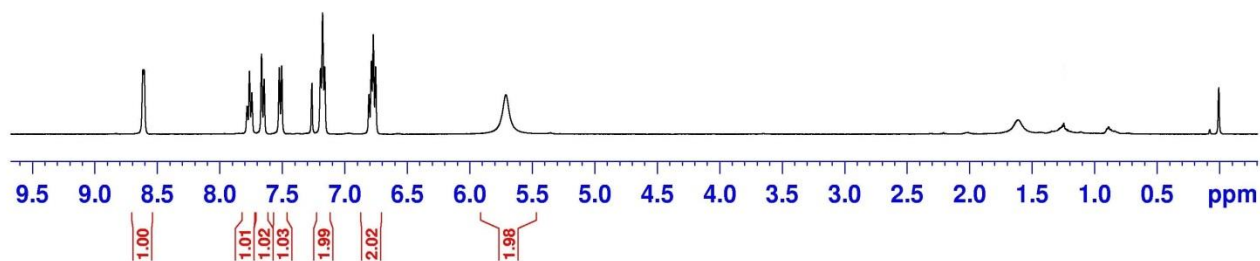
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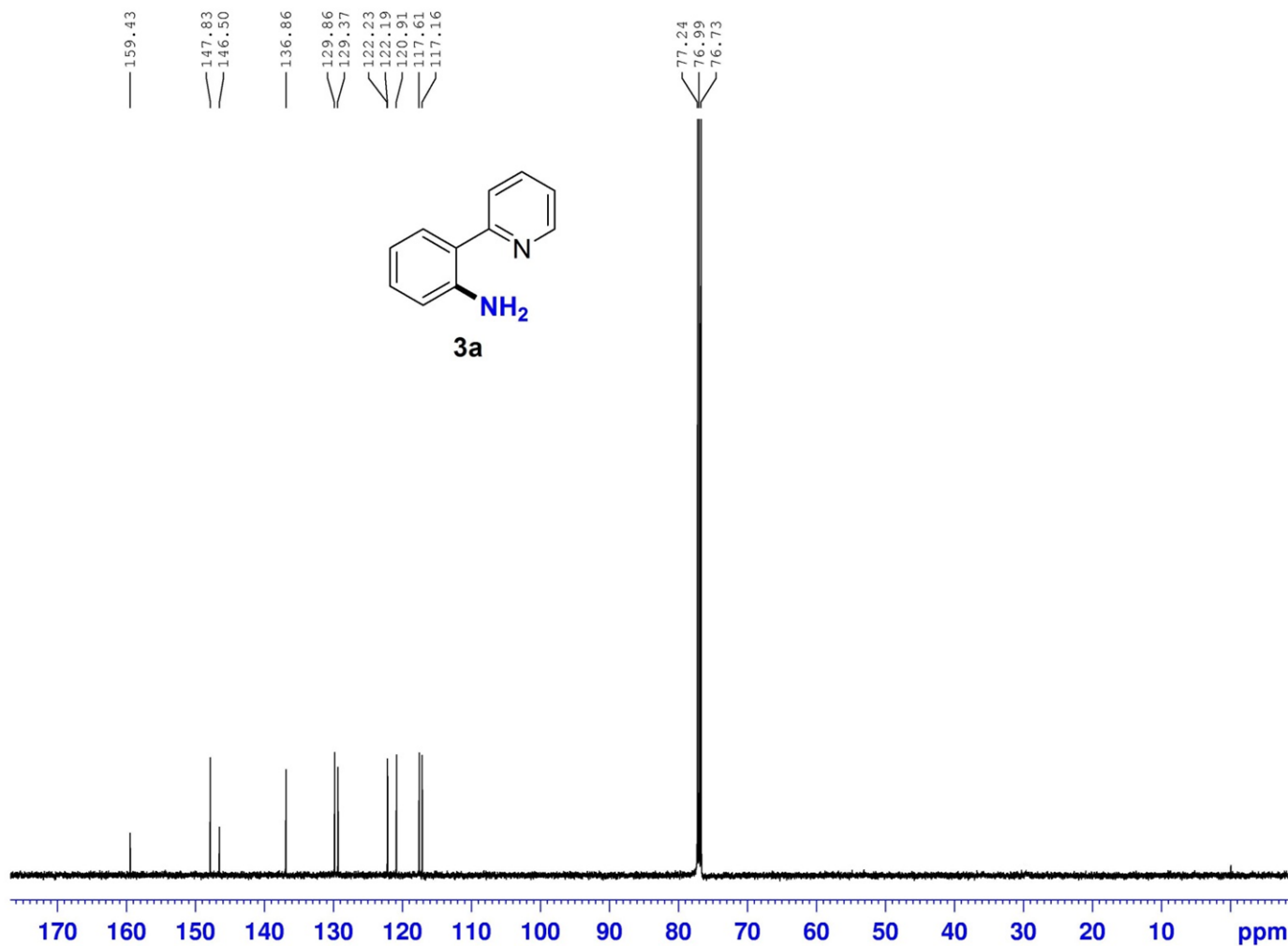
3a

NAME NH2 H-1
EXPNO 114233
PROCNO 1
Date_ 20130923
Time 21.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114283

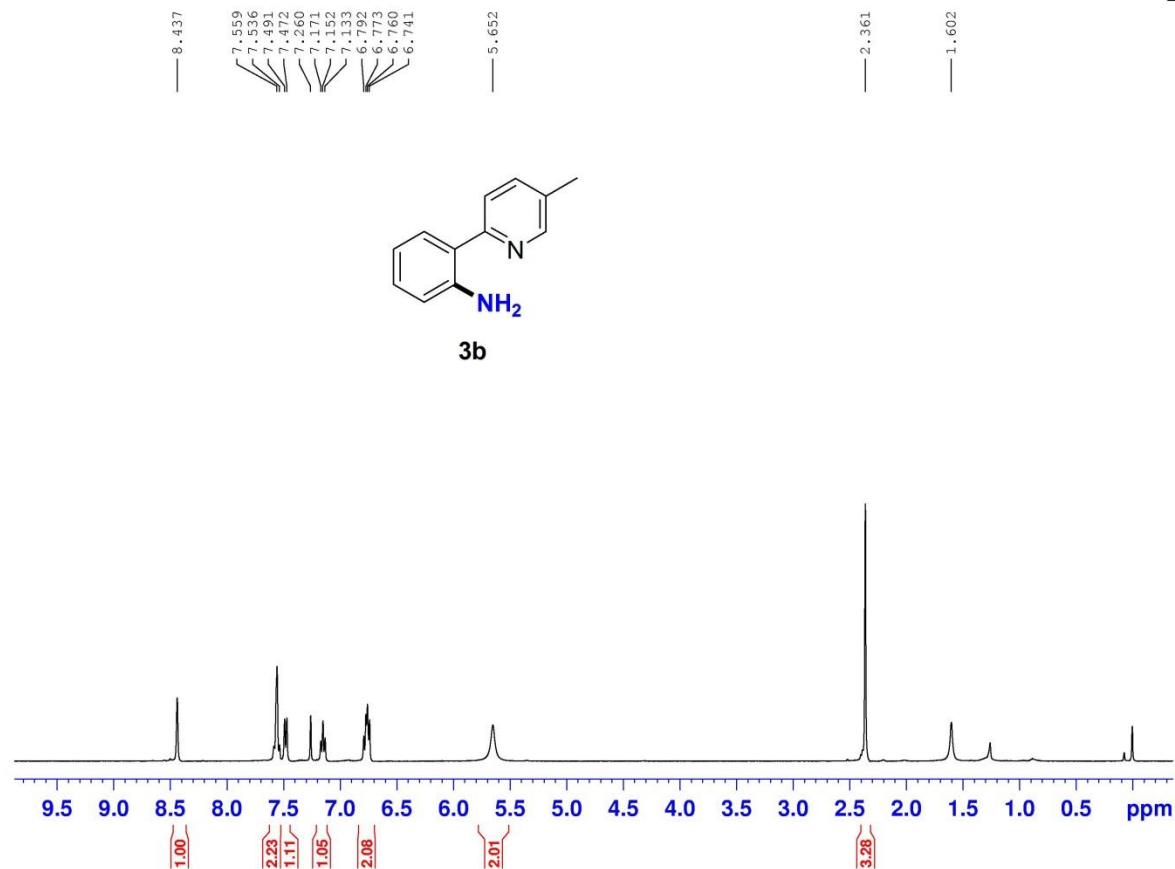


NAME NH2 C13
EXPNO 114283
PROCNO 1
Date_ 20130925
Time 16.08
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 932
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

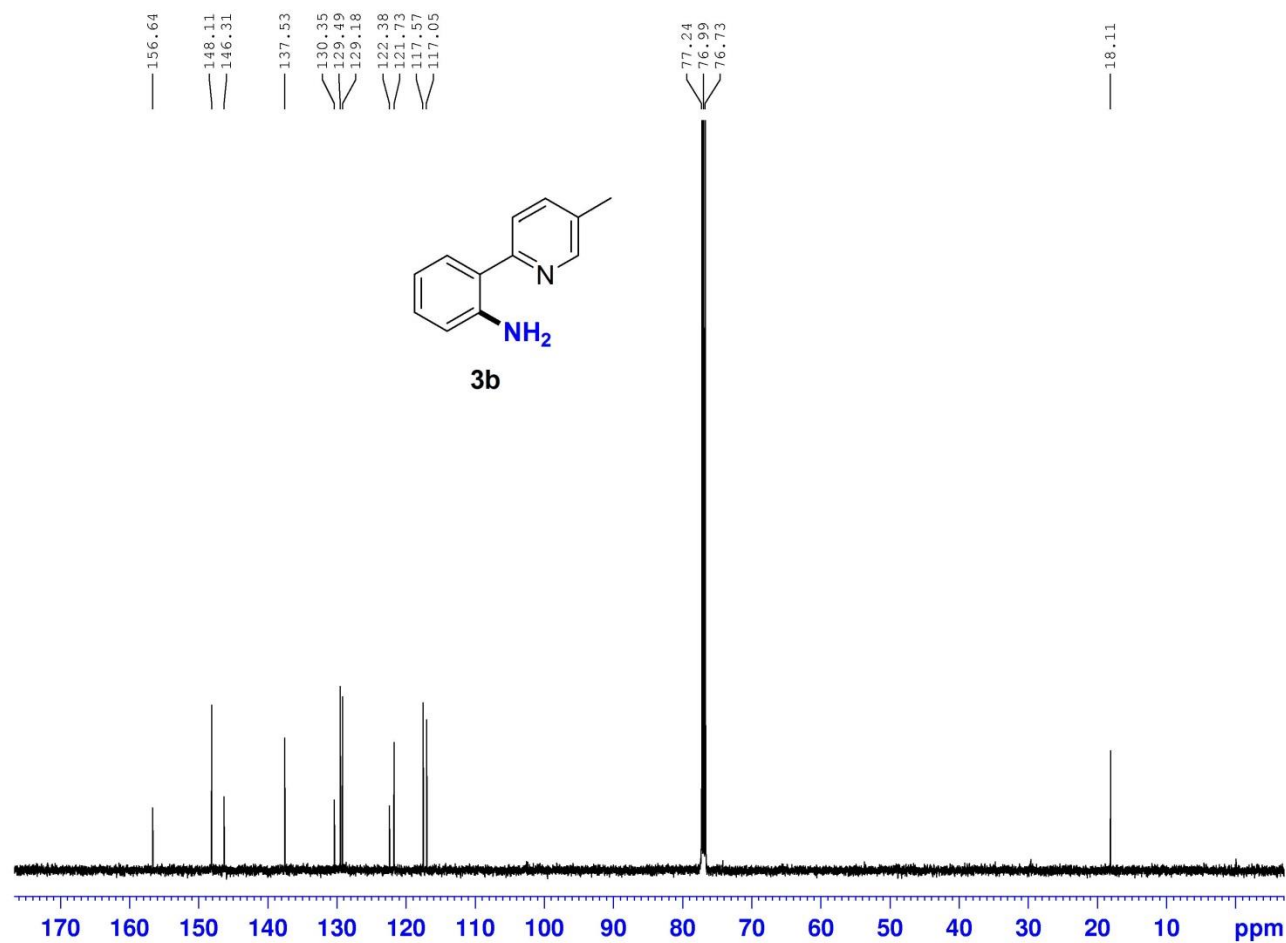
114274



NAME NH2 H-1
EXPNO 114274
PROCNO 1
Date_ 20131009
Time 15.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

114274

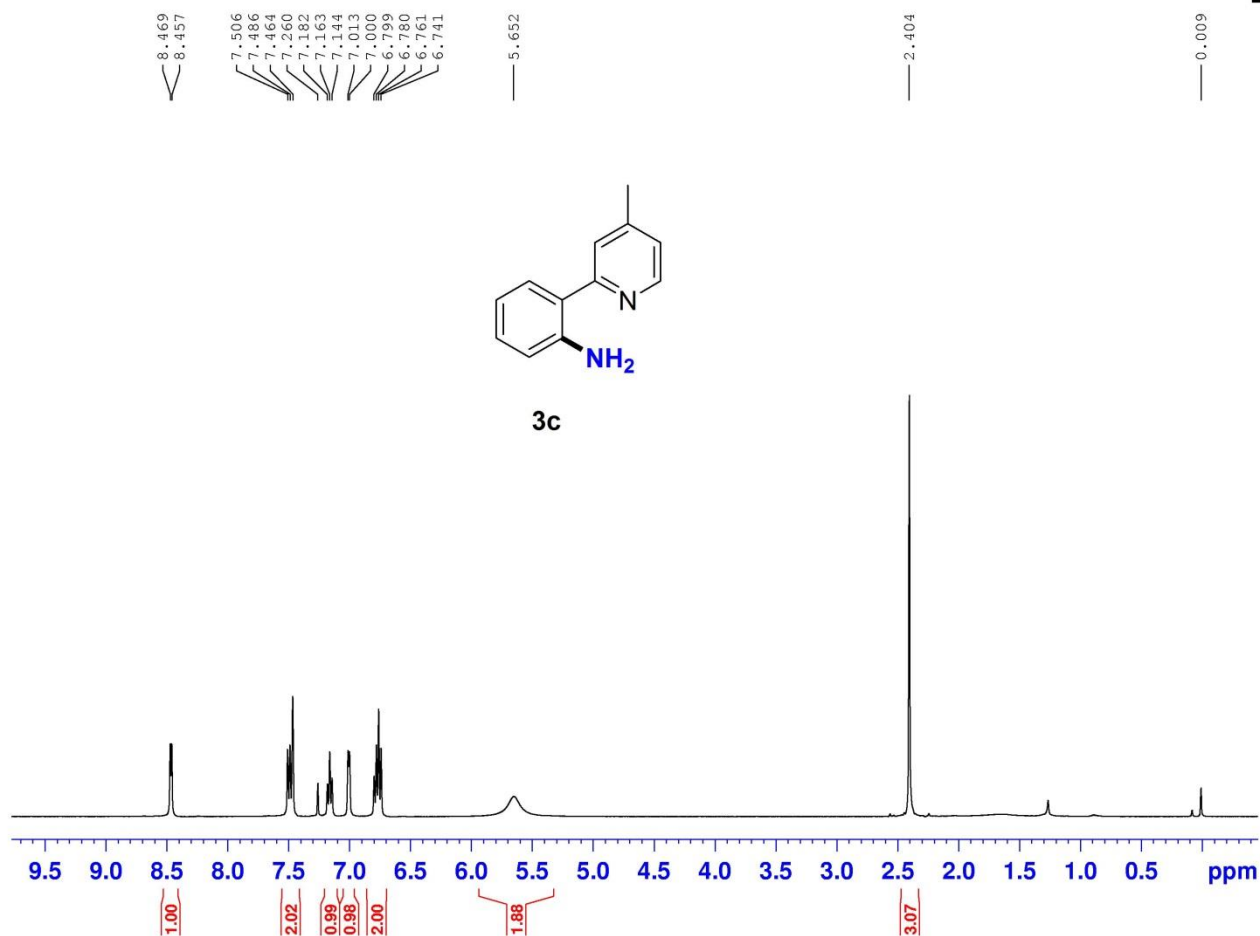


NAME NH2 C13
EXPNO 114274
PROCNO 1
Date_ 20131011
Time 20.15
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 655
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
S1 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

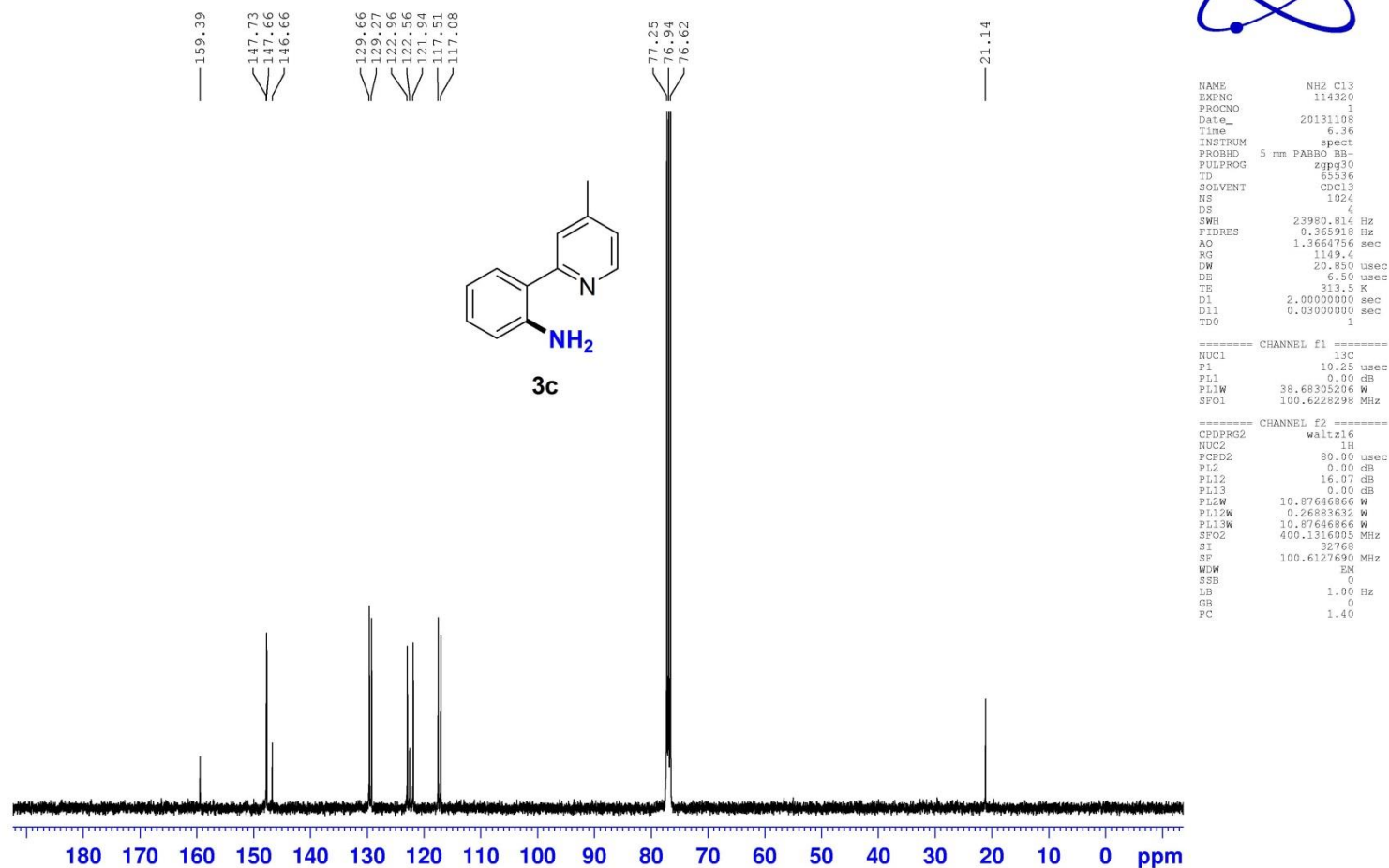
114320



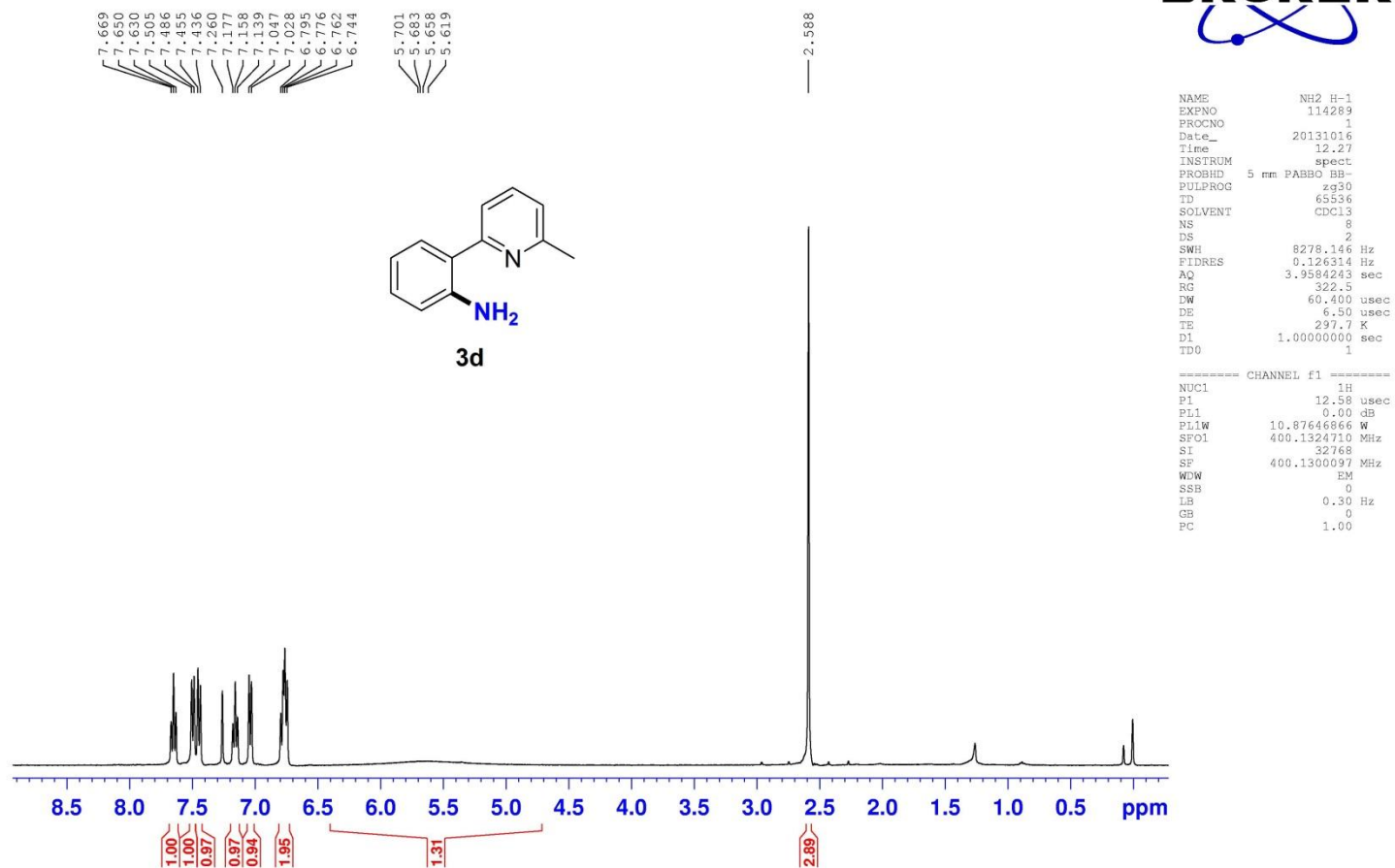
NAME NH2 H-1
EXPNO 114320
PROCNO 1
Date_ 20131106
Time 11.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646966 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

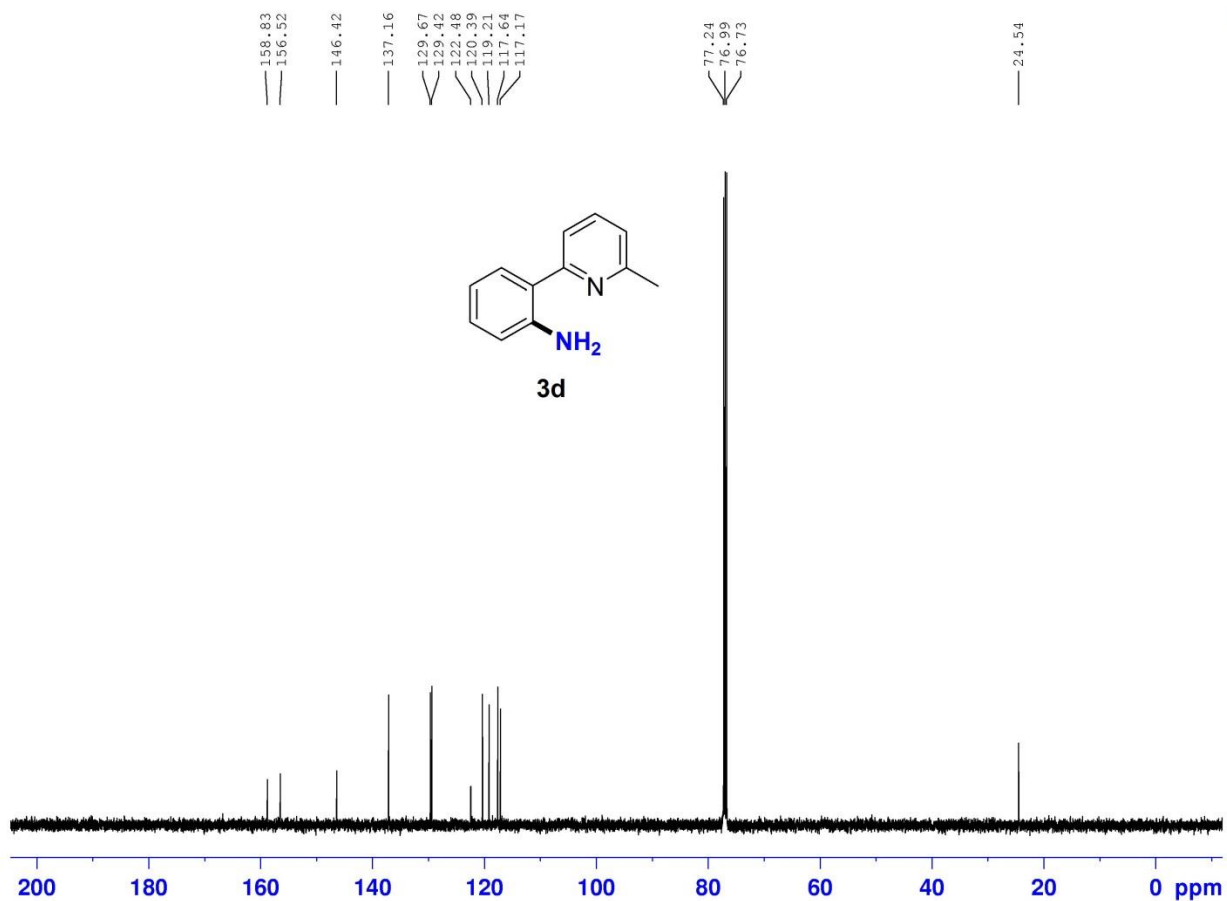
114320



114289



114289



NAME NH2 C13
EXPNO 114289
PROCNO 1
Date_ 20131023
Time 15.10
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 238
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

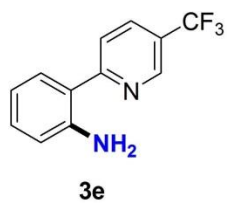
----- CHANNEL f1 -----
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SF01 125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SF02 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

114286

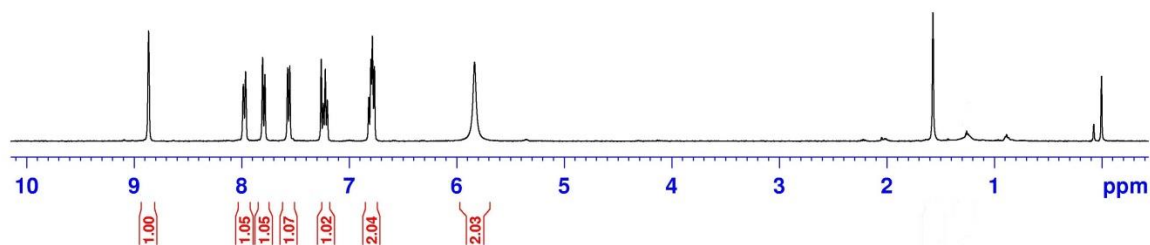


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1.570
1.259

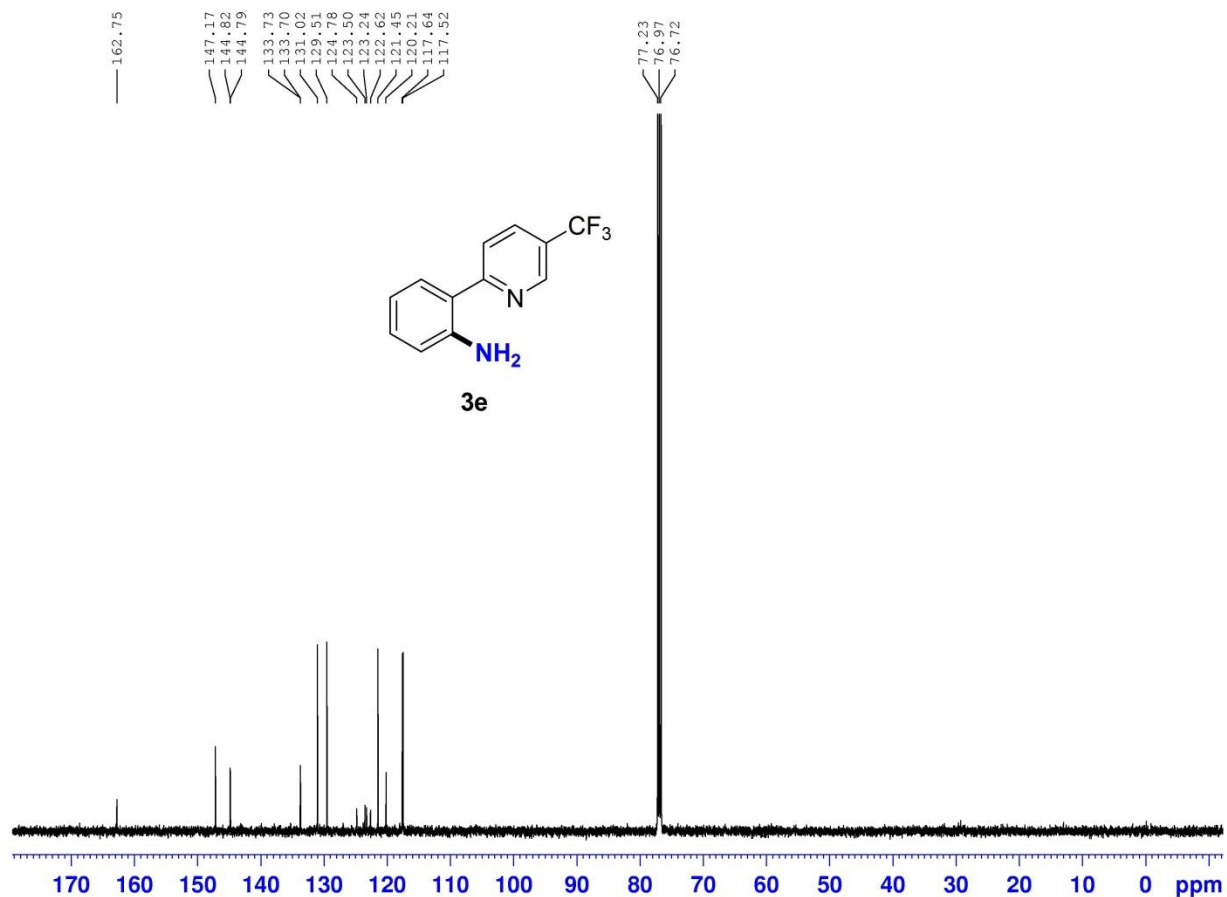


NAME NH2 H-1
EXPNO 114286
PROCNO 1
Date_ 20131016
Time 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WEW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114286

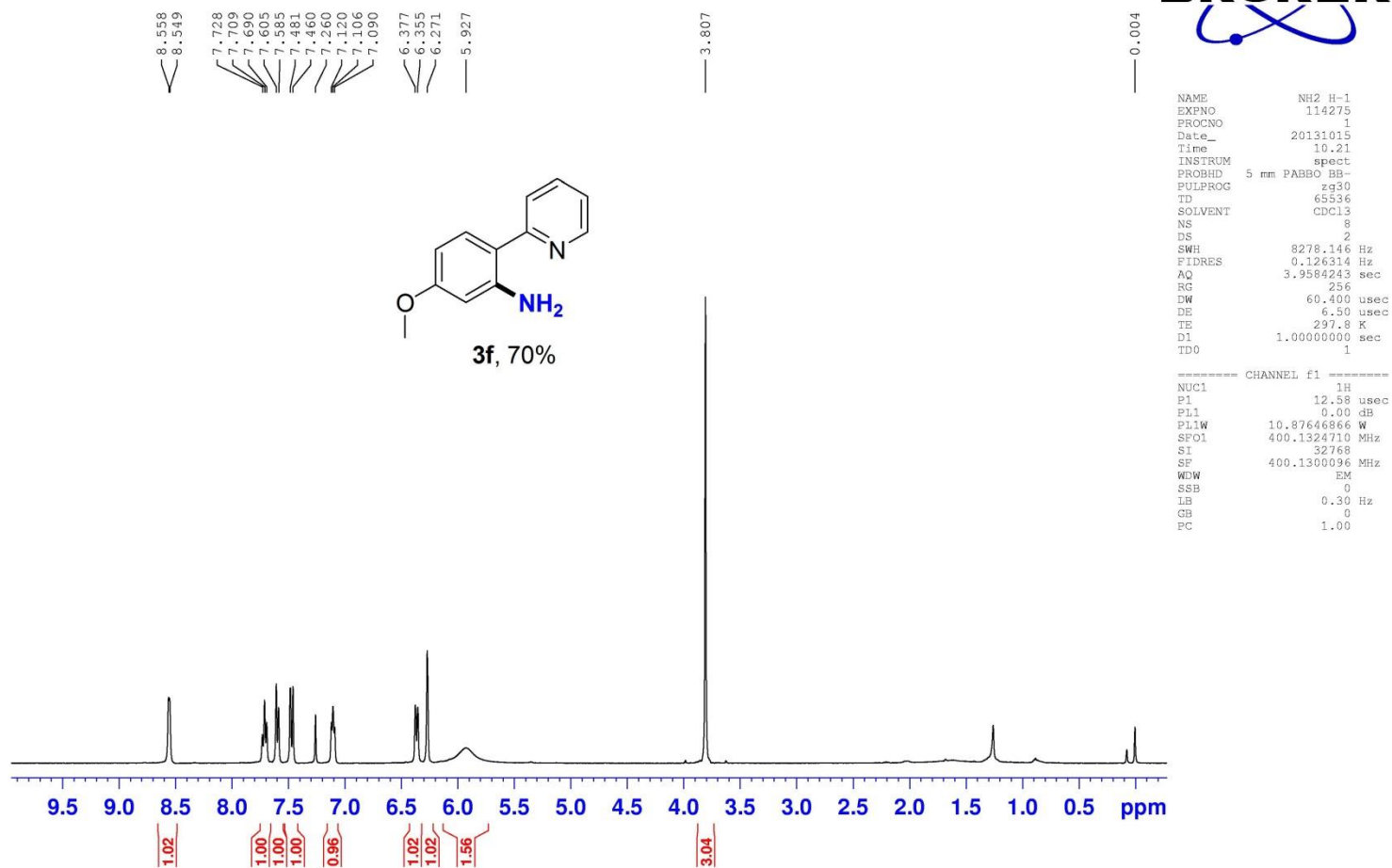


NAME NH2 C13
EXPNO 114286
PROCNO 1
Date_ 20131023
Time 15.32
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 951
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

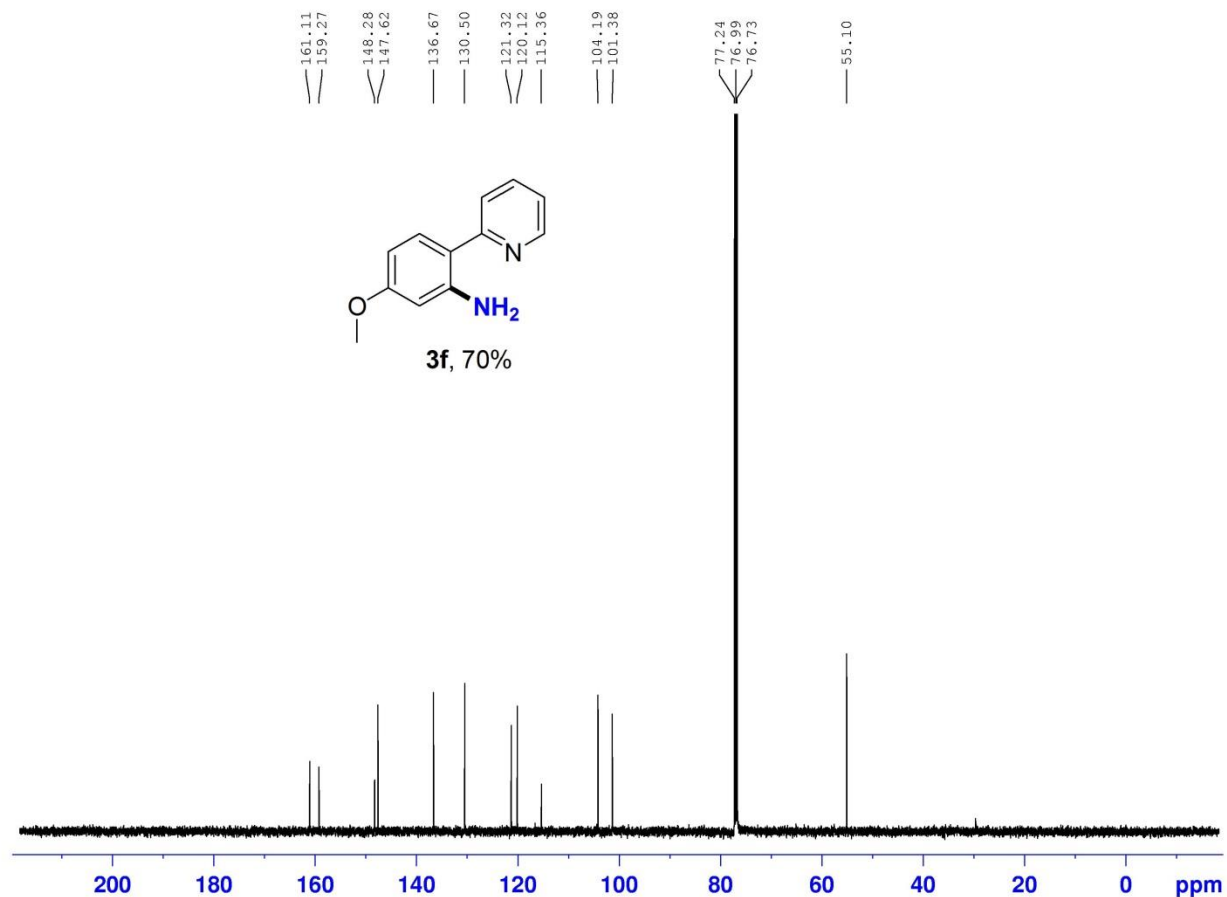
----- CHANNEL f1 -----
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2 Waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
S1 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

114275



114275

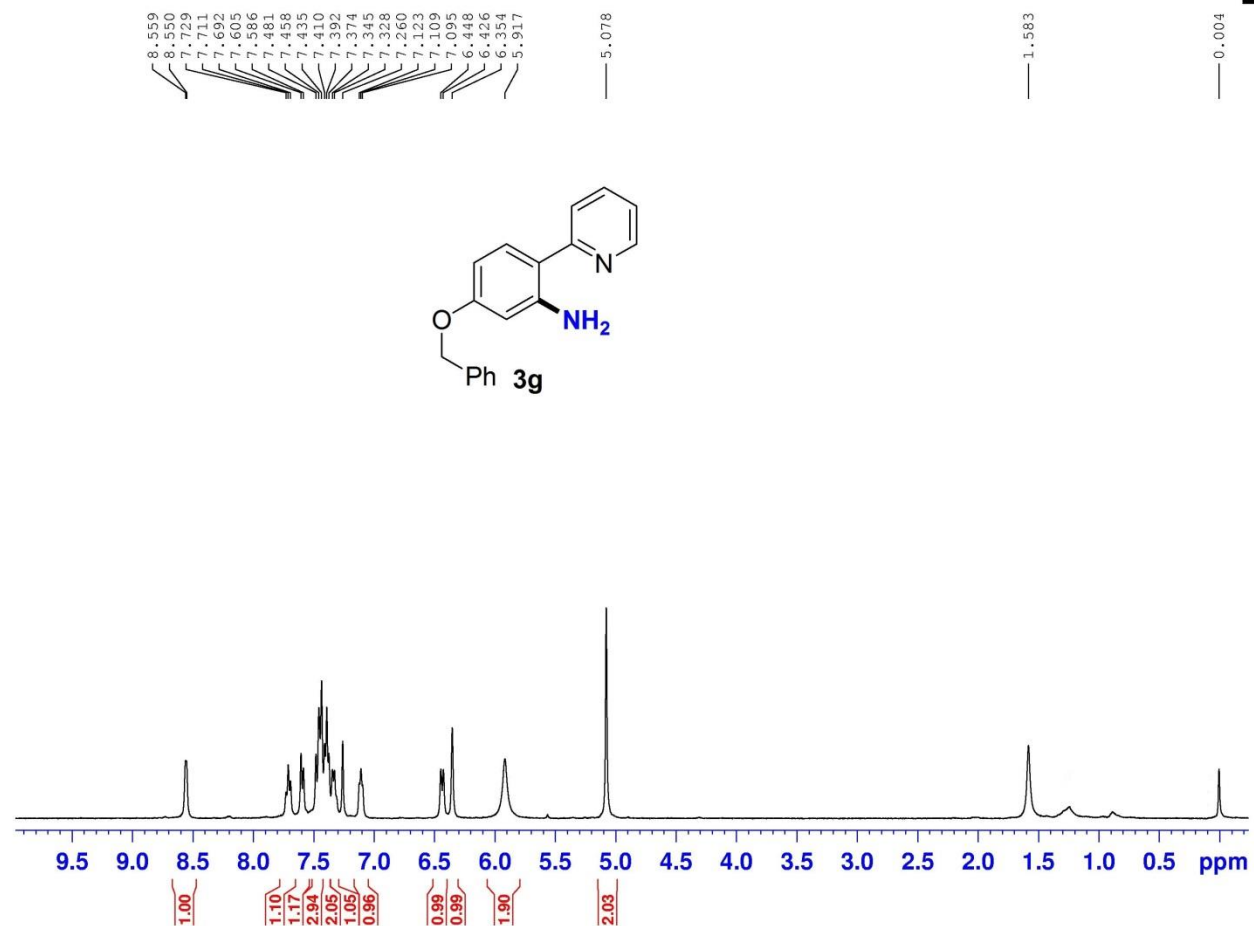


NAME NH2 C13
EXPNO 114275
PROCNO 1
Date_ 20131018
Time 21.17
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 469
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2 Waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
S1 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

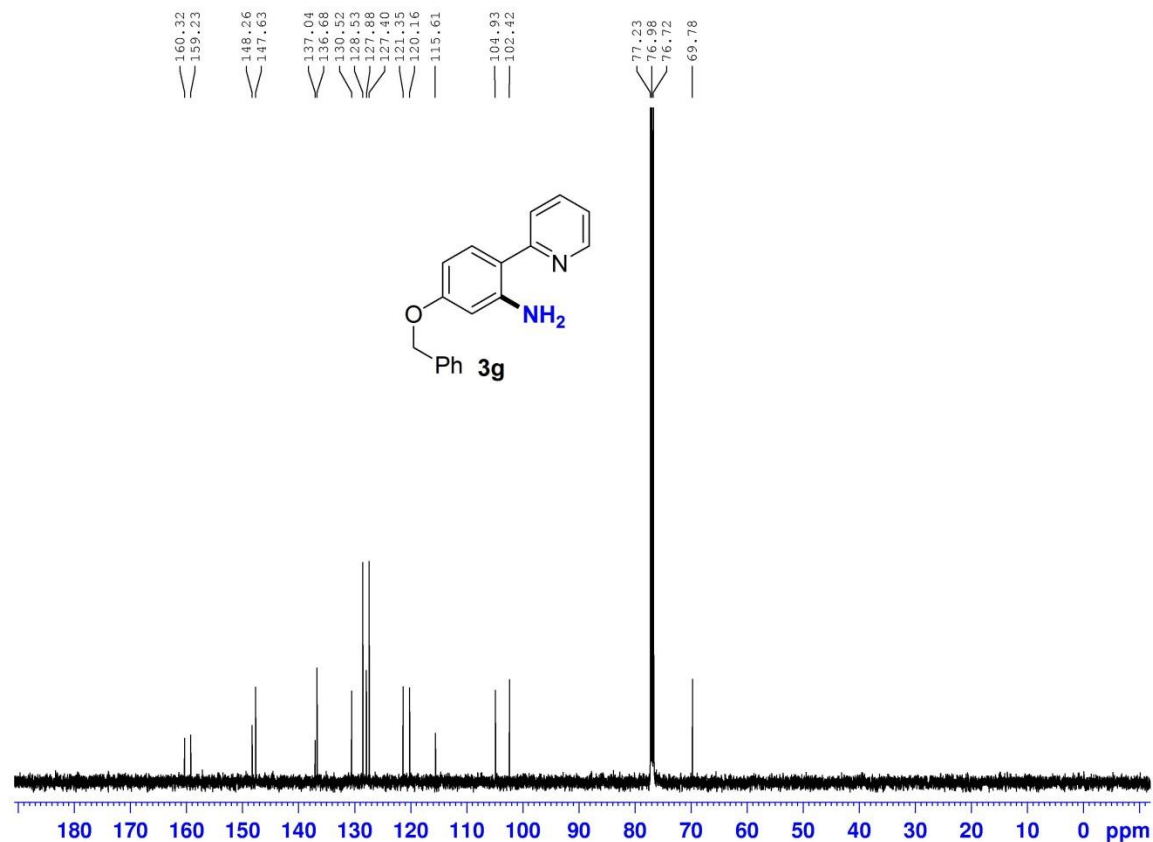
114276



NAME NH2 H-1
EXPNO 114276
PROCNO 1
Date_ 20131011
Time 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

114276

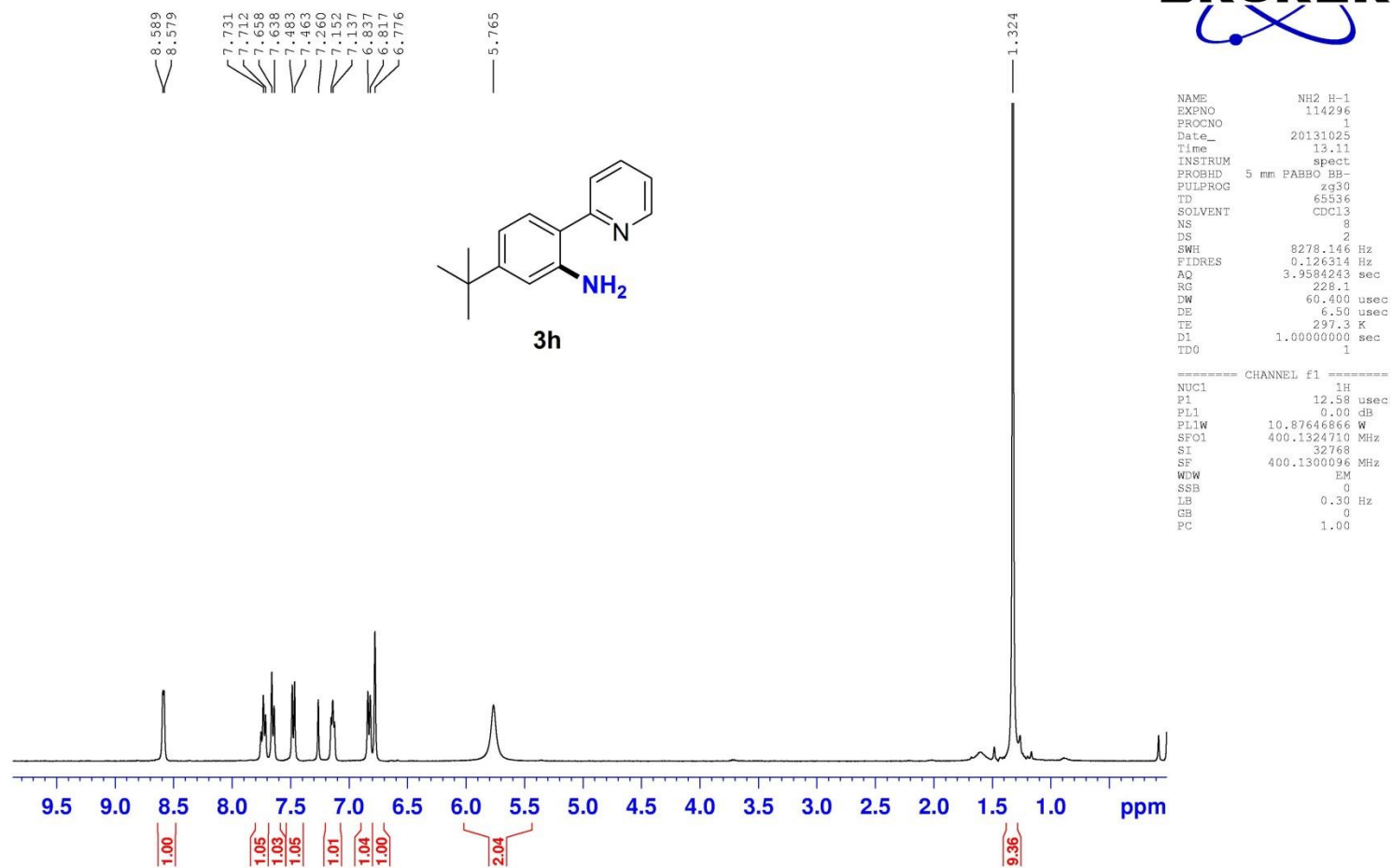


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NAME      NH2 C13
EXPNO     114276
PROCNO    1
Date_     20131012
Time      20.44
INSTRUM    Spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         32768
SOLVENT    CDCl3
NS         463
DS         4
SWH        29761.904 Hz
FIDRES     0.908261 Hz
AQ         0.5505524 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         297.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
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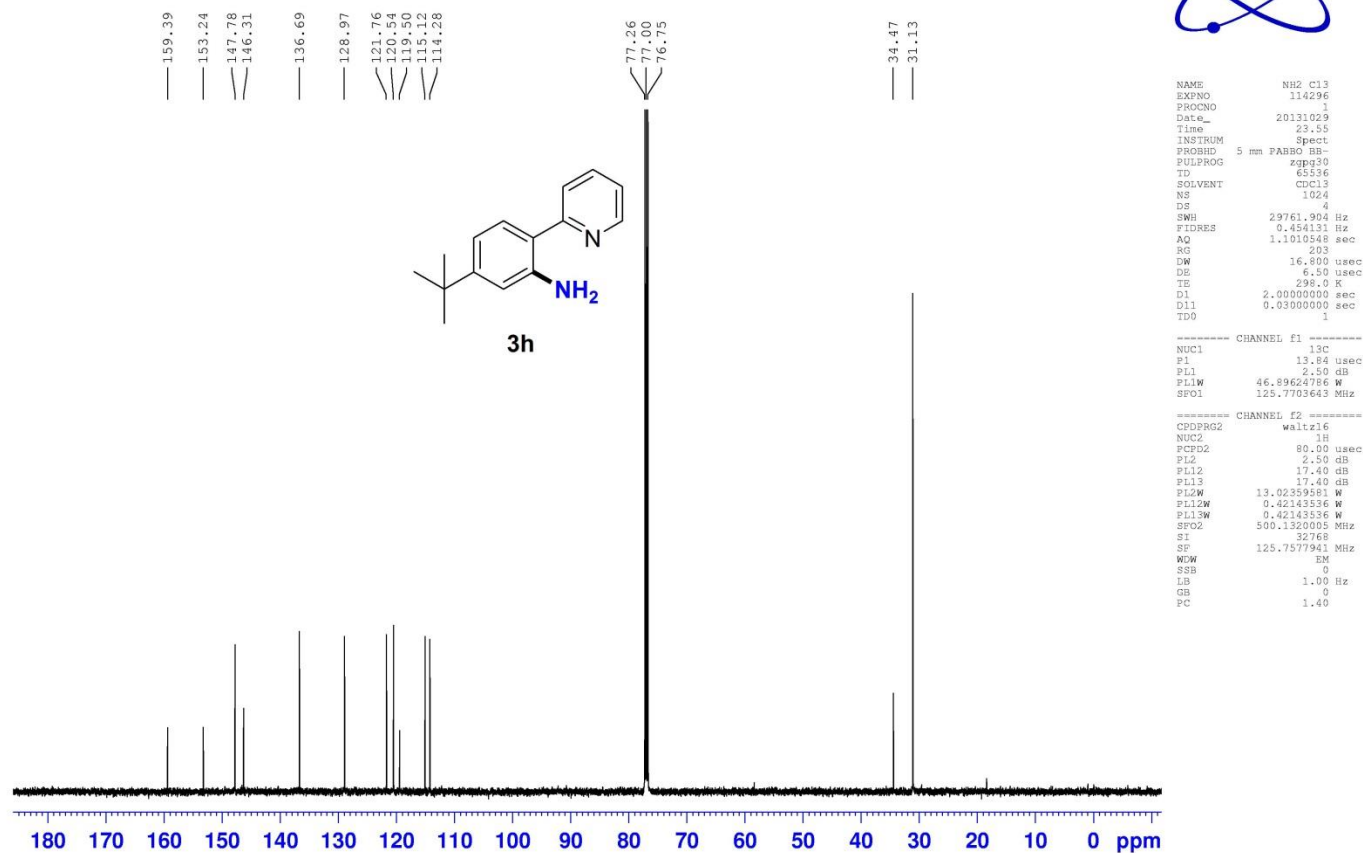
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===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      17.40 dB
PL2W      13.02359581 W
PL12W     0.42143536 W
PL13W     0.42143536 W
SFO2      500.1320005 MHz
SI         32768
SF        125.7577966 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

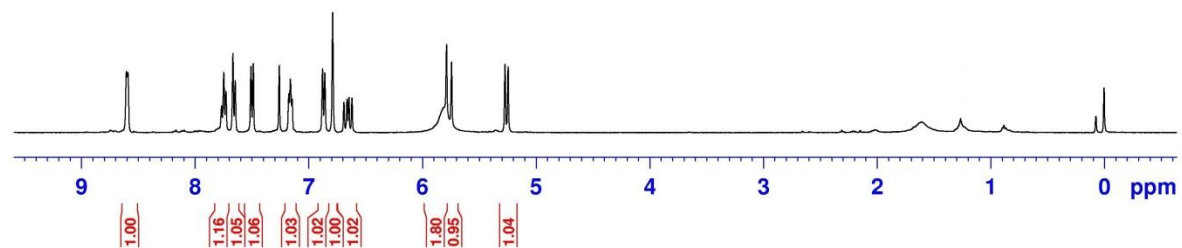
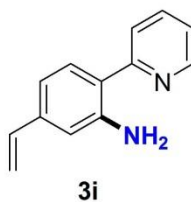
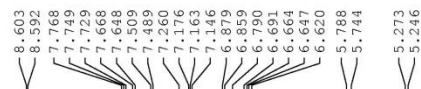
114296



114296



114343

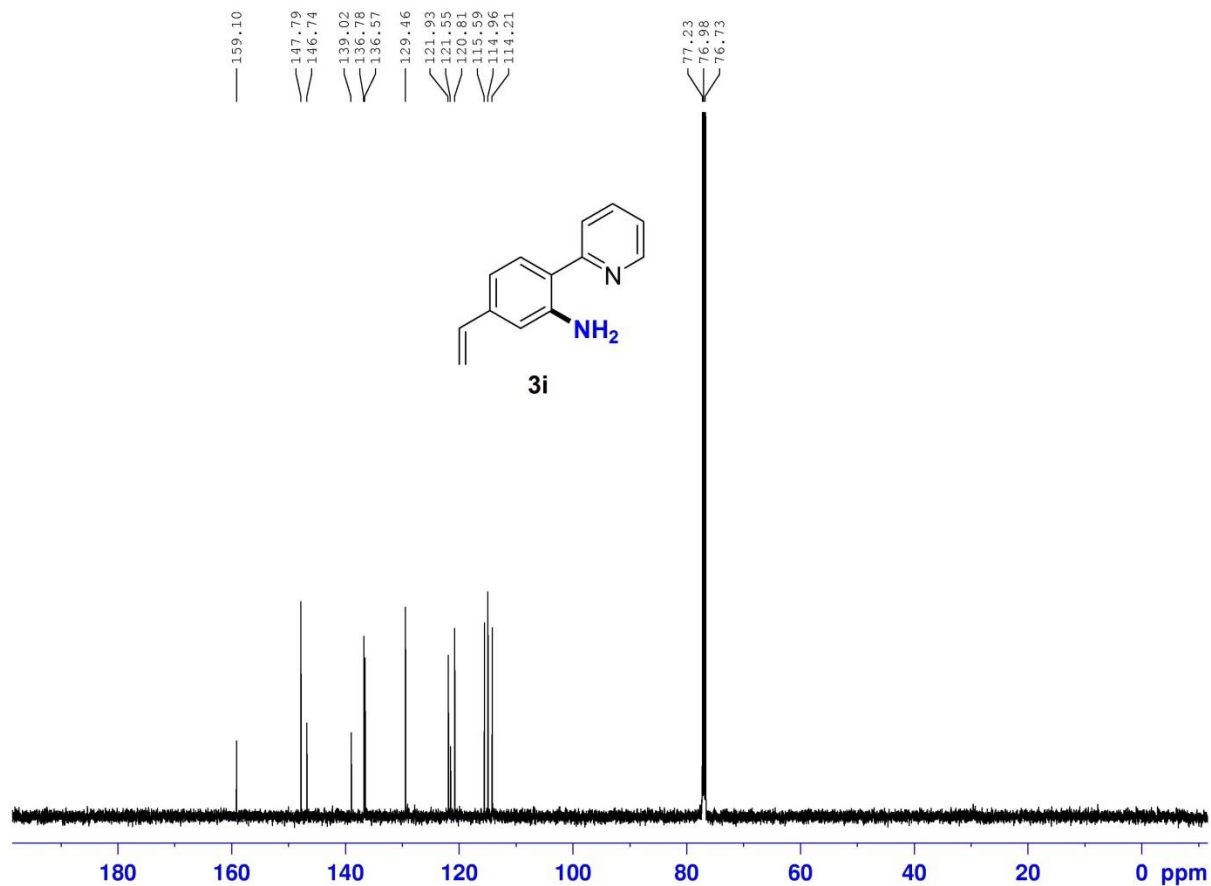


— 0.003

NAME NH2 H-1
EXPNO 114343
PROCNO 1
Date_ 20131115
Time 13.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

114343

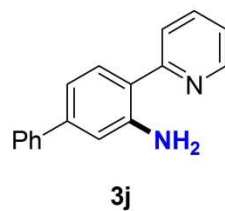


```
NAME      NH2 C13
EXPNO     114343
PROCNO    1
Date_     20131121
Time      17.01
INSTRUM   Spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         663
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         13.84 usec
PL1        2.50 dB
PL1W       46.89624786 W
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.50 dB
PL12       17.40 dB
PL13       17.40 dB
PL1W       13.02359581 W
PL12W      0.42143536 W
PL13W      0.42143536 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7571972 MHz
WDW        EM
SFB        0
LB         1.00 Hz
GB         0
PC         1.40
```

114311



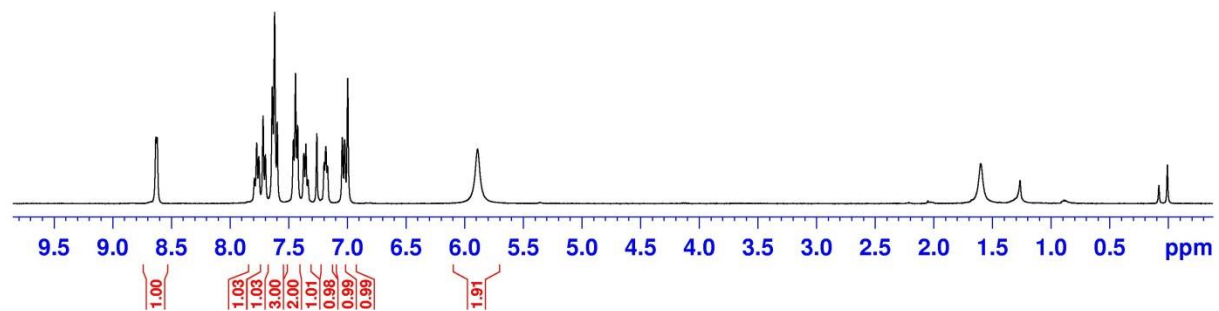
— 1.599

— 0.008

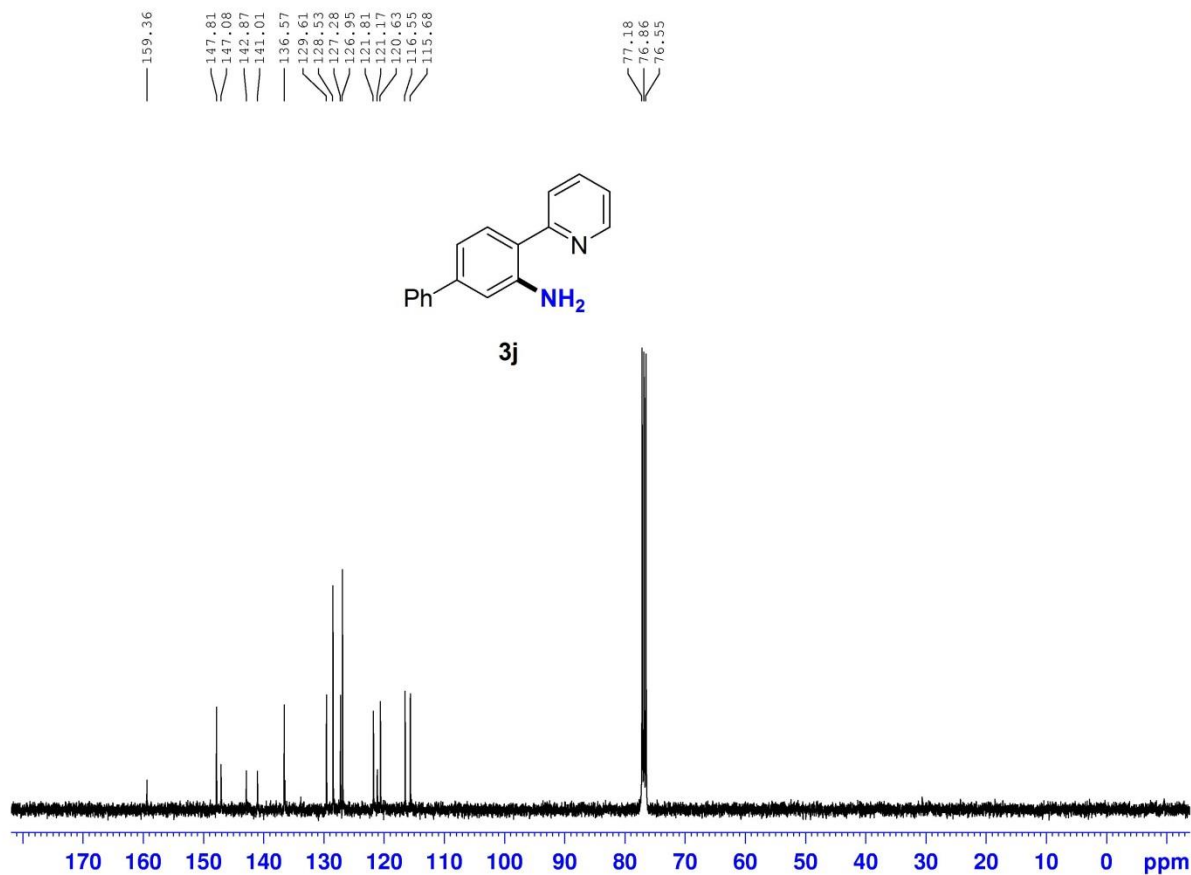


NAME NH2 H-1
EXPNO 114311
PROCNO 1
Date_ 20131031
Time 13.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.56 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114311



NAME NH2 C13
EXPNO 114311
PROCNO 1
Date_ 20131106
Time 5.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1149.4
DW 20.850 usec
DE 6.50 usec
TE 313.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WCM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

114293

8.588
8.576
7.770
7.751
7.732
7.607
7.587
7.496
7.478
7.458
7.260
7.183
7.170
7.153
6.492
6.488
6.471
6.467
6.452
6.425
5.944

**3k**

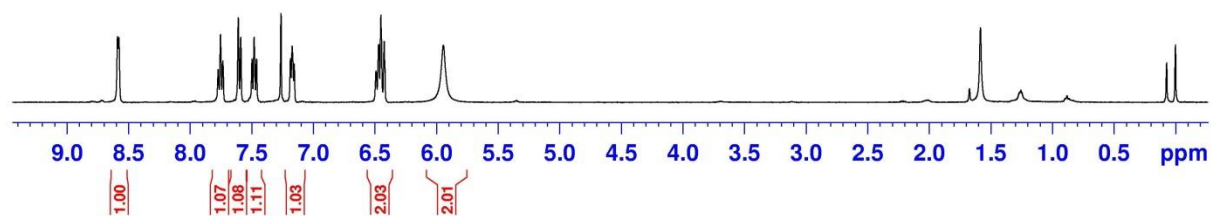
1.585

0.001

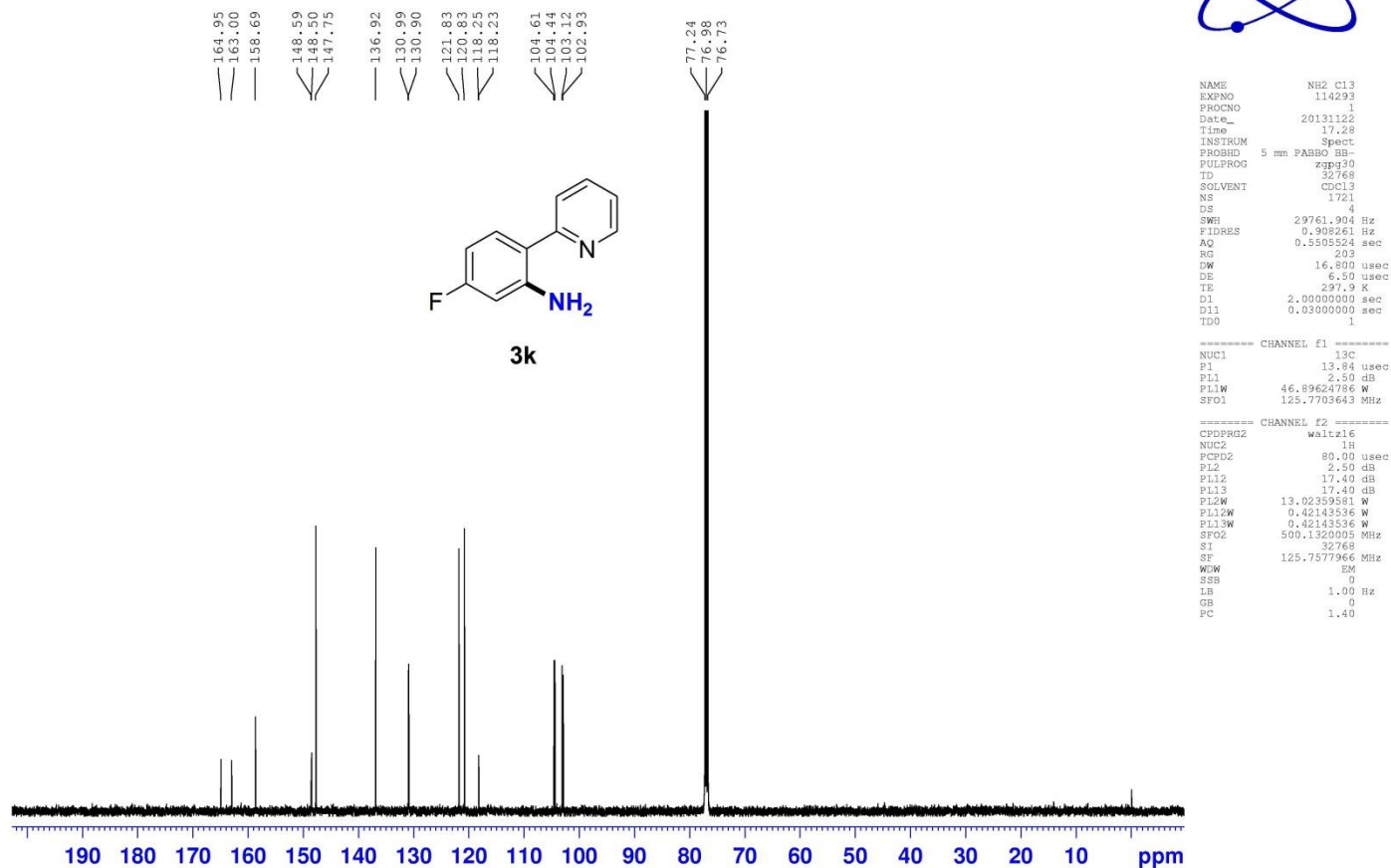


NAME NH2 H-1
EXPNO 114293
PROCNO 1
Date_ 20131029
Time 16.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1

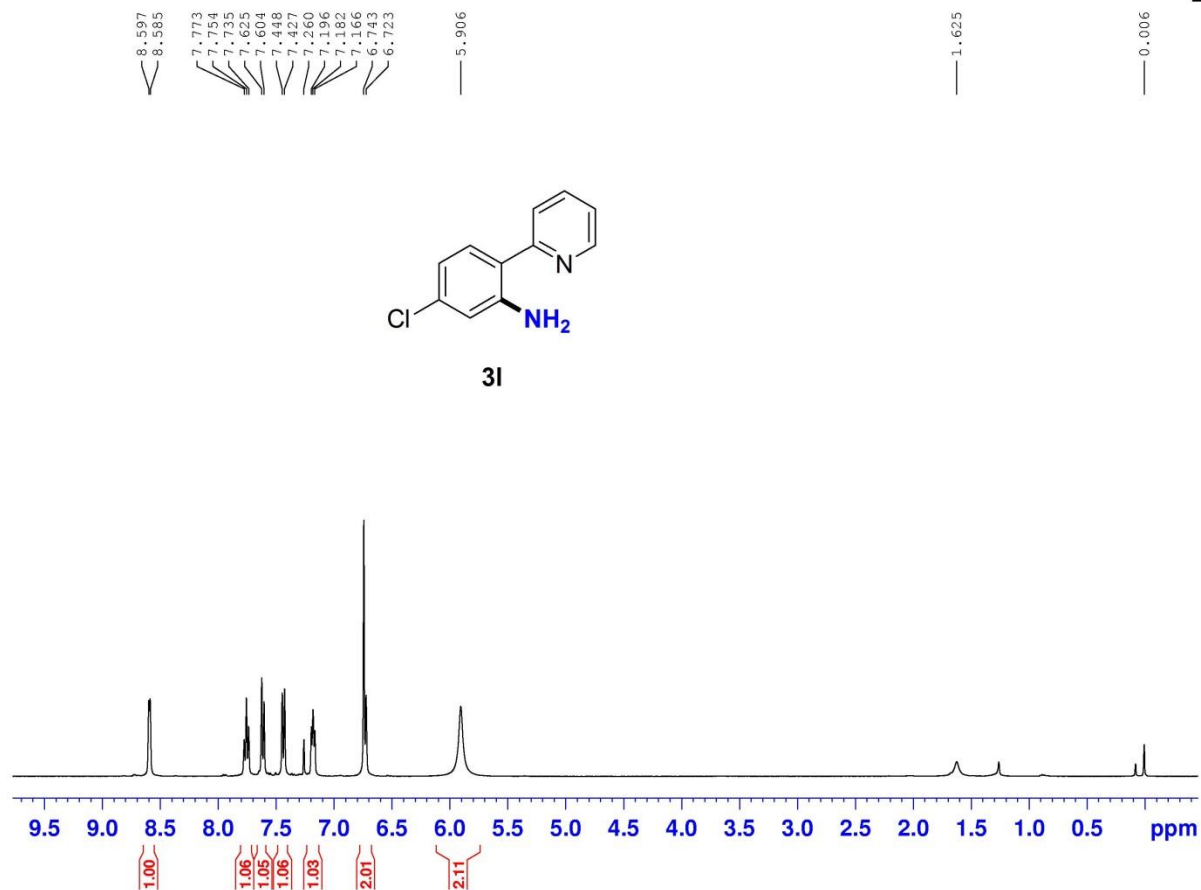
===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SE 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114293



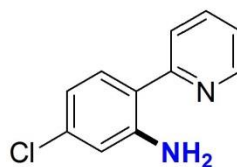
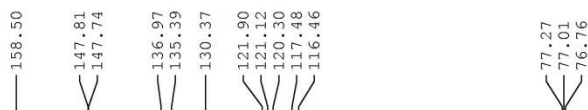
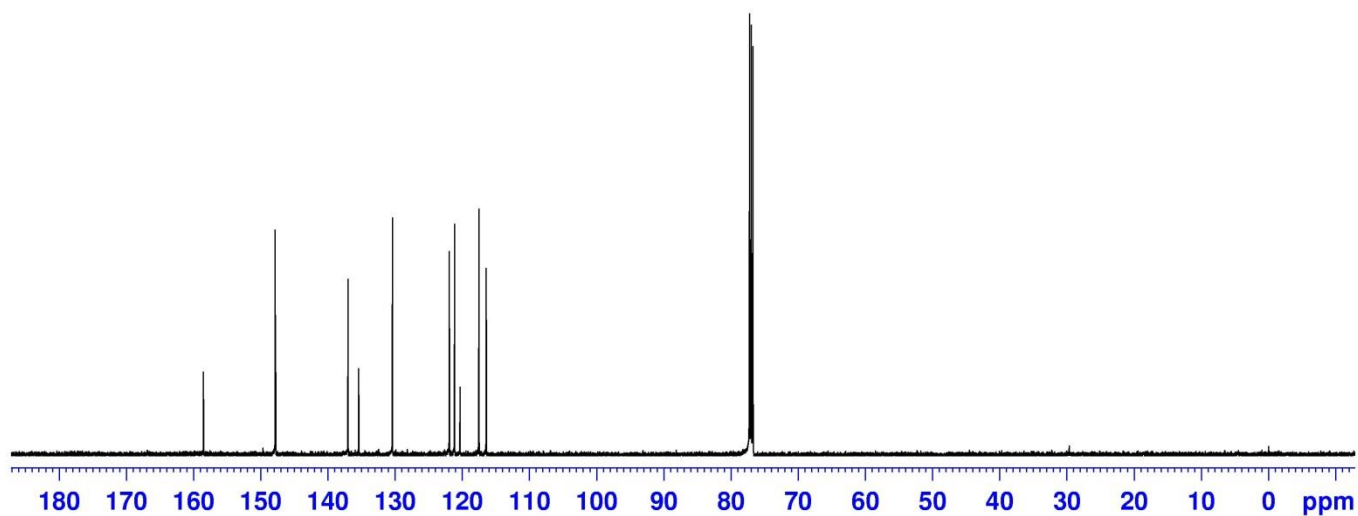
114312



NAME NH2 H-1
EXPNO 114312
PROCNO 1
Date_ 20131031
Time 16.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

114312

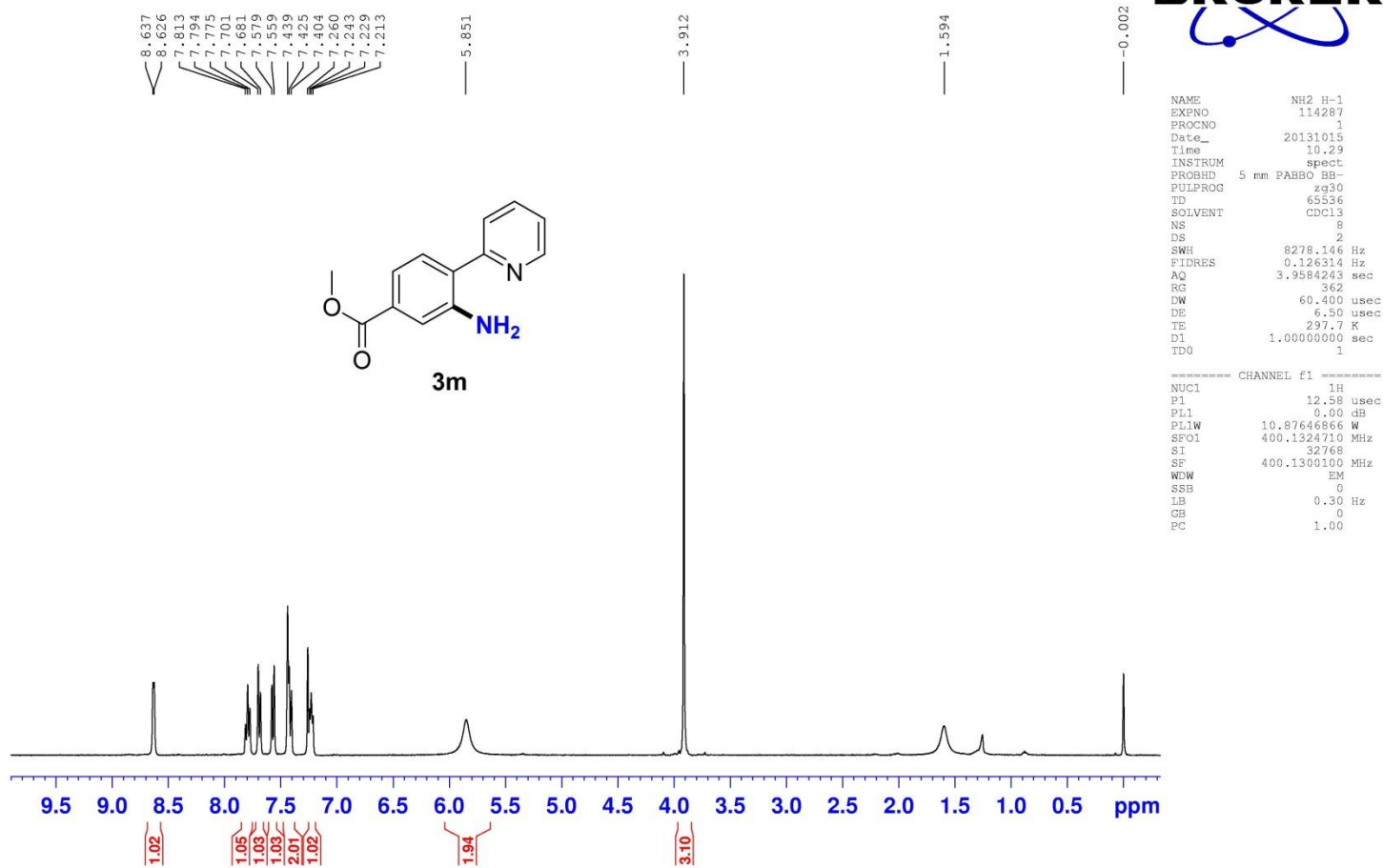
**31**

```
NAME      NH2 C13
EXPNO     114312
PROCNO    1
Date_     20131115
Time      18.33
INSTRUM   Spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        845
DS        4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010548 sec
RG        203
DW        16.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
```

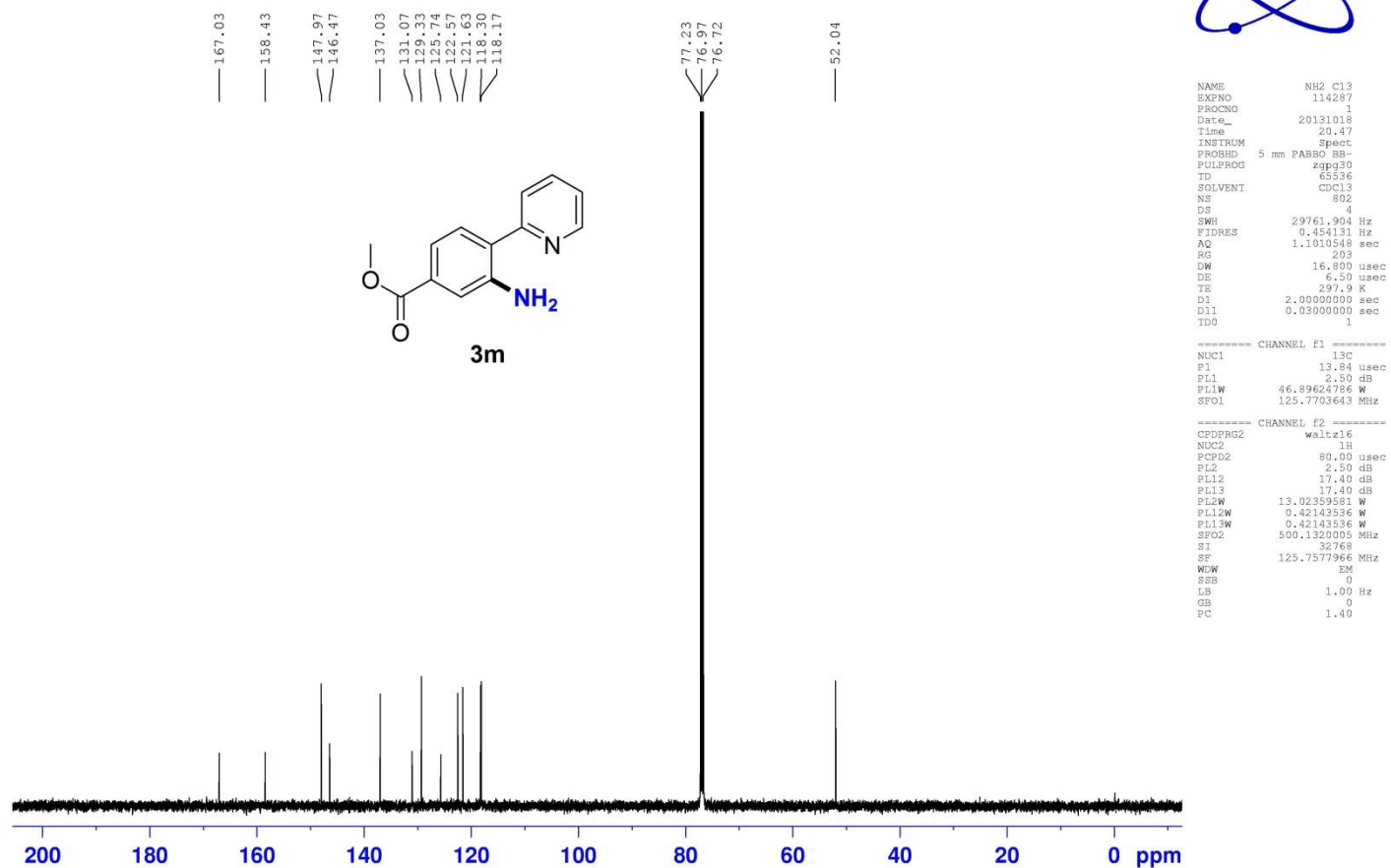
```
===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      17.40 dB
PL2W      13.02359581 W
PL12W     0.42143536 W
PL13W     0.42143536 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7577966 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

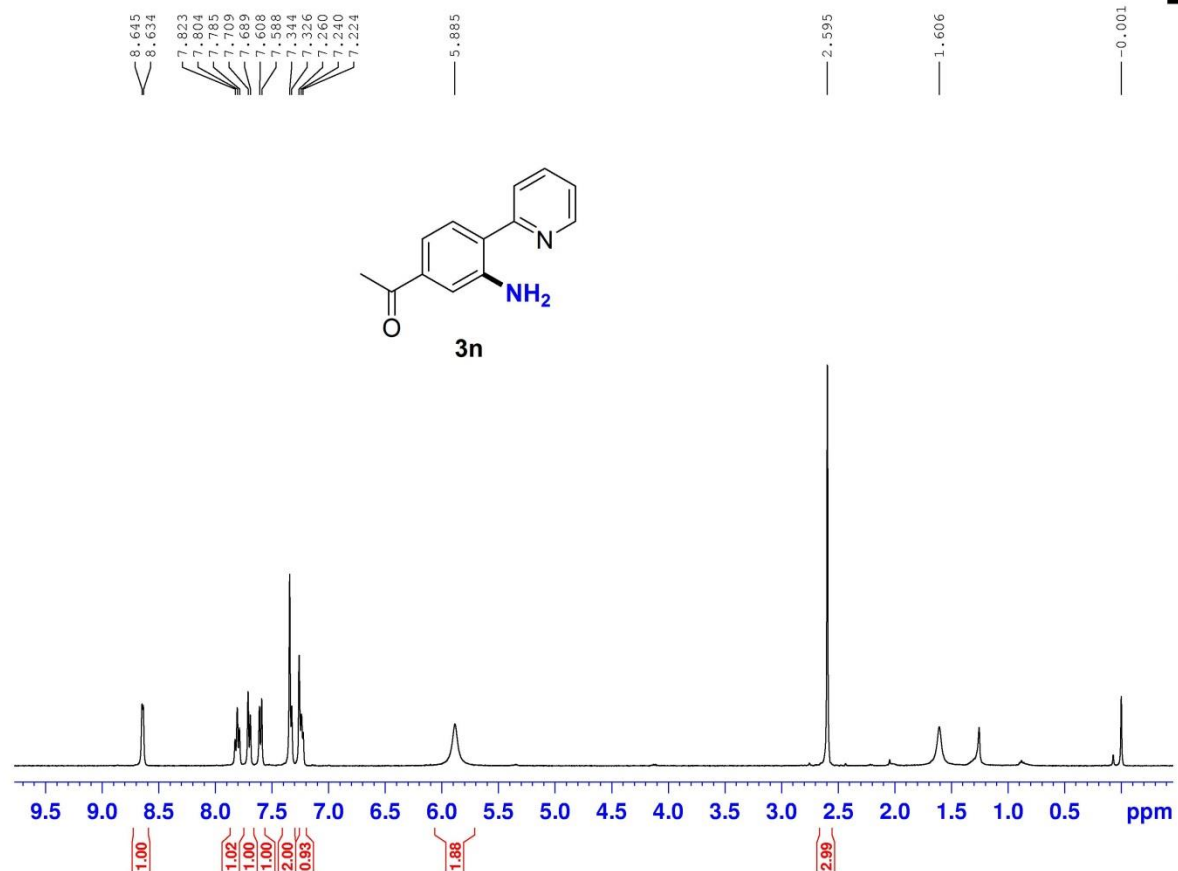
114287



114287



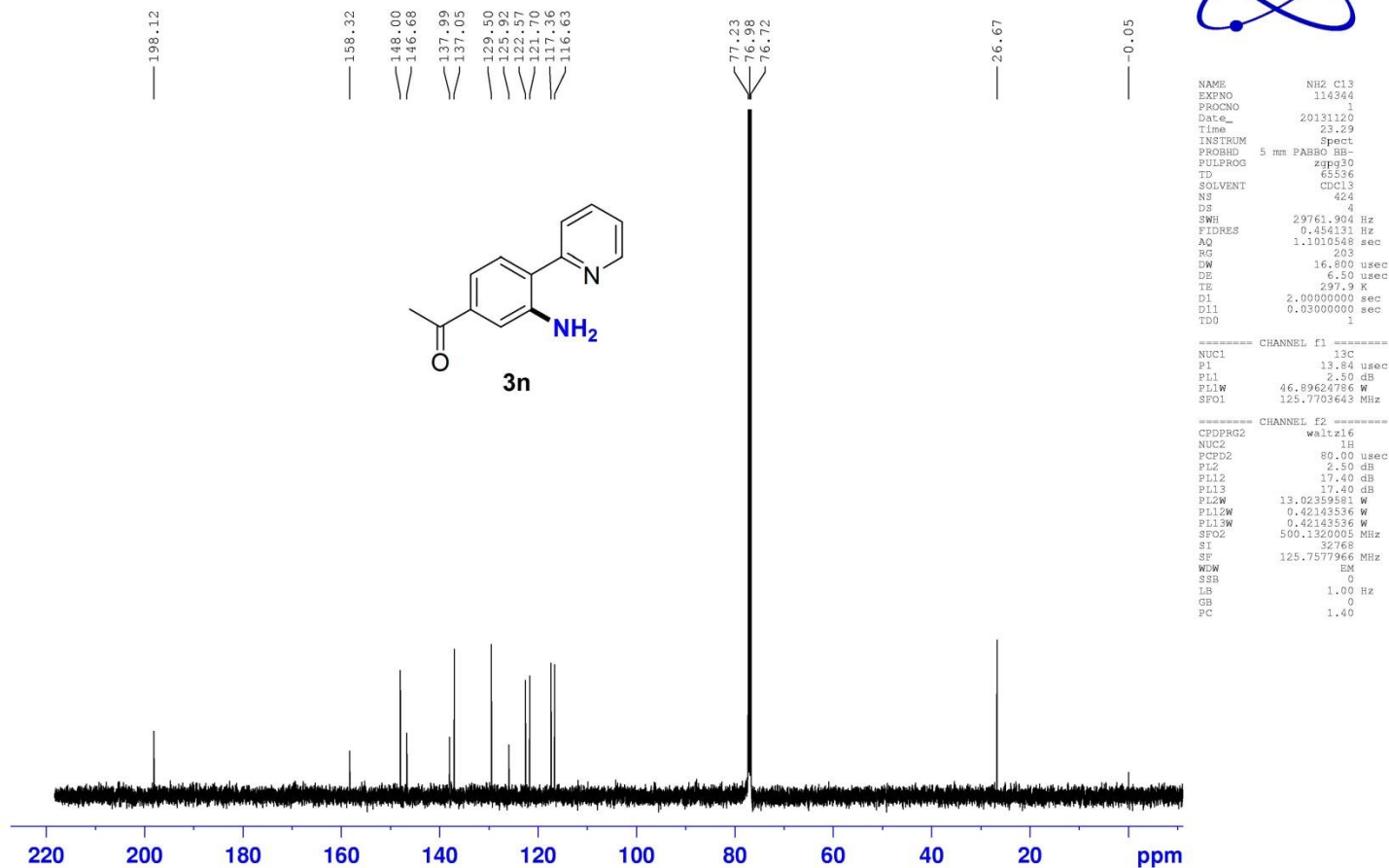
114344



NAME NH2 H-1
EXPNO 114344
PROCNO 1
Date_ 20131115
Time 13.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

114344



114281

8.643
8.632
7.839
7.829
7.819
7.809
7.679
7.659
7.585
7.565
7.280
7.260
7.030
7.009
6.998

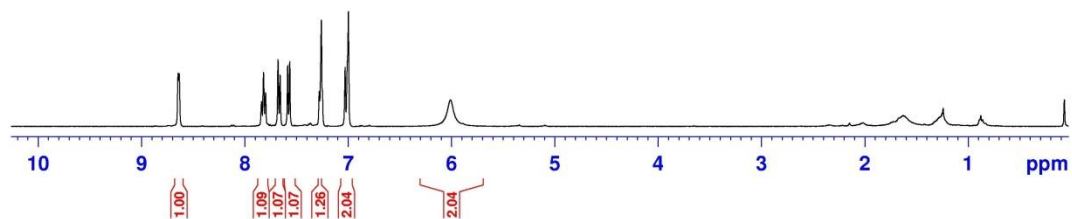
6.009

1.673
1.630
1.613

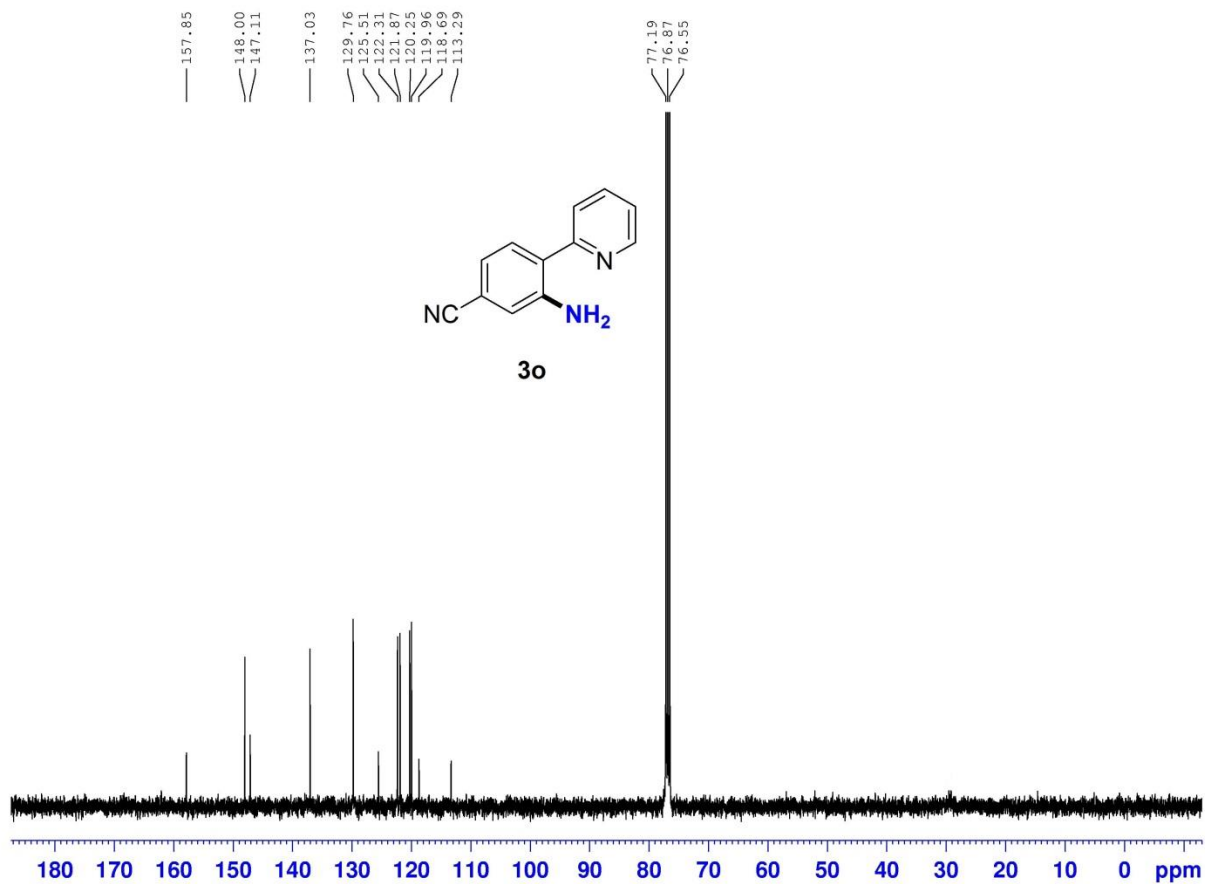
**3o**

NAME NH2 H-1
EXPNO 114281
PROCNO 1
Date_ 20131107
Time 16.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TDO 1

CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11428



```
NAME      NH2 C13
EXPNO     114281
PROCNO    1
Date_     20131112
Time      9.37
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        1024
DS        4
SWH       23980.814 Hz
FIDRES    0.365918 Hz
AQ        1.3664756 sec
RG        1149.4
DW        20.850 usec
DE        6.50 usec
TE        313.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        10.25 usec
PL1       0.00 dB
PL1W      38.68305206 W
SFO1      100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      16.07 dB
PL13      0.00 dB
PL2W      10.87646866 W
PL12W     0.26883632 W
PL13W     10.87646866 W
SFO2      400.1316009 MHz
SI        32768
SF        100.6127690 MHz
WOW       EM
SFB       0
LB        1.00 Hz
GB        0
PC        1.40
```

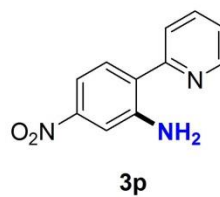
114346

8.662
8.651
7.858
7.838
7.819
7.715
7.695
7.650
7.629
7.581
7.557
7.303
7.290
7.272
7.260

6.138

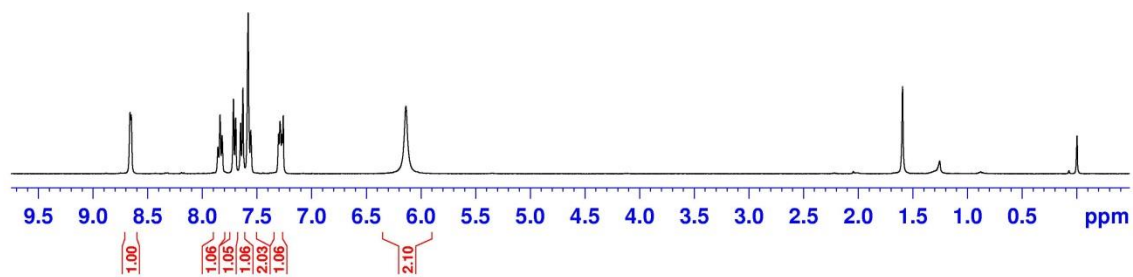
1.593

-0.003

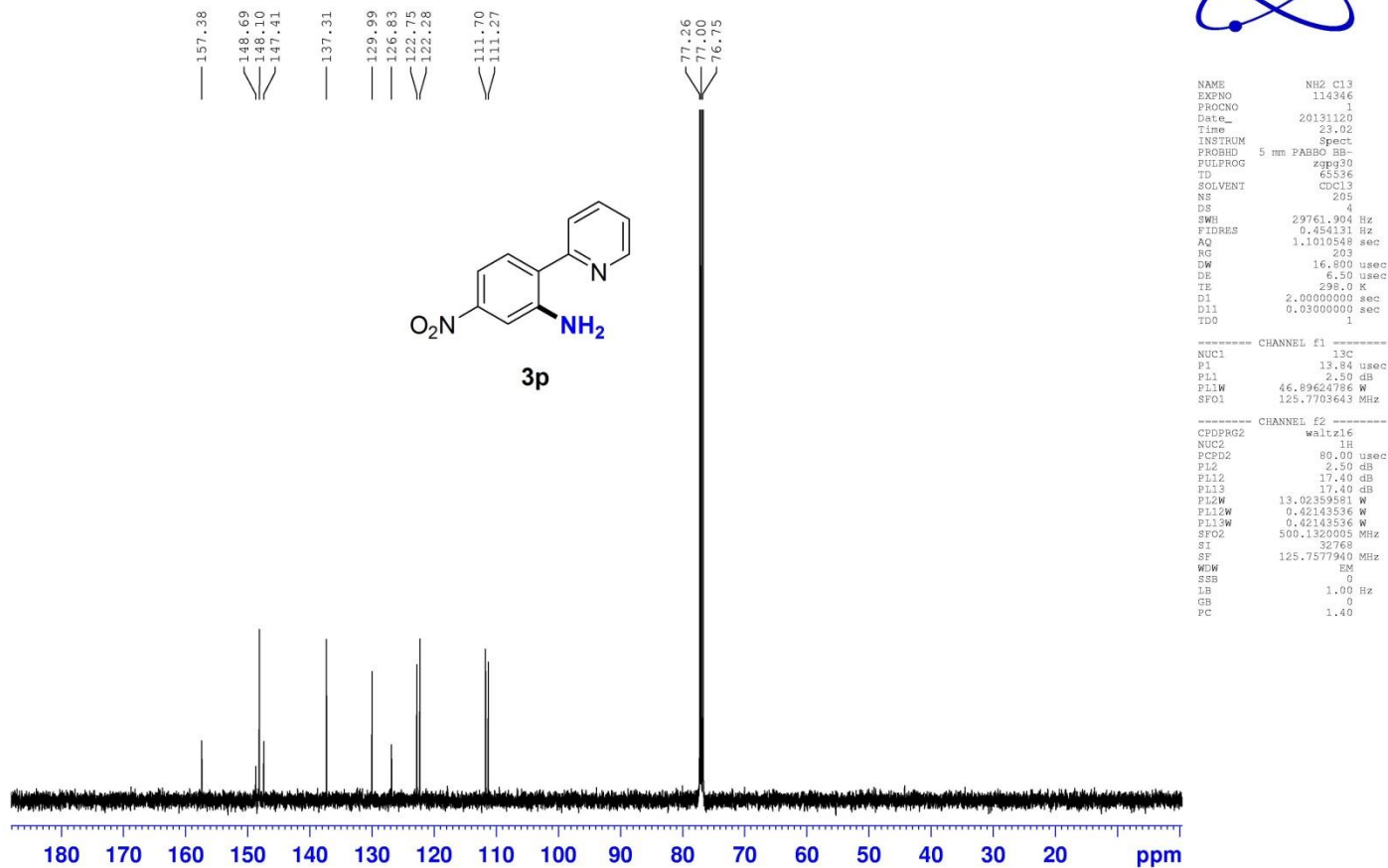


NAME NH2 H-1
EXPNO 114346
PROCNO 1
Date_ 20131115
Time 13.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1

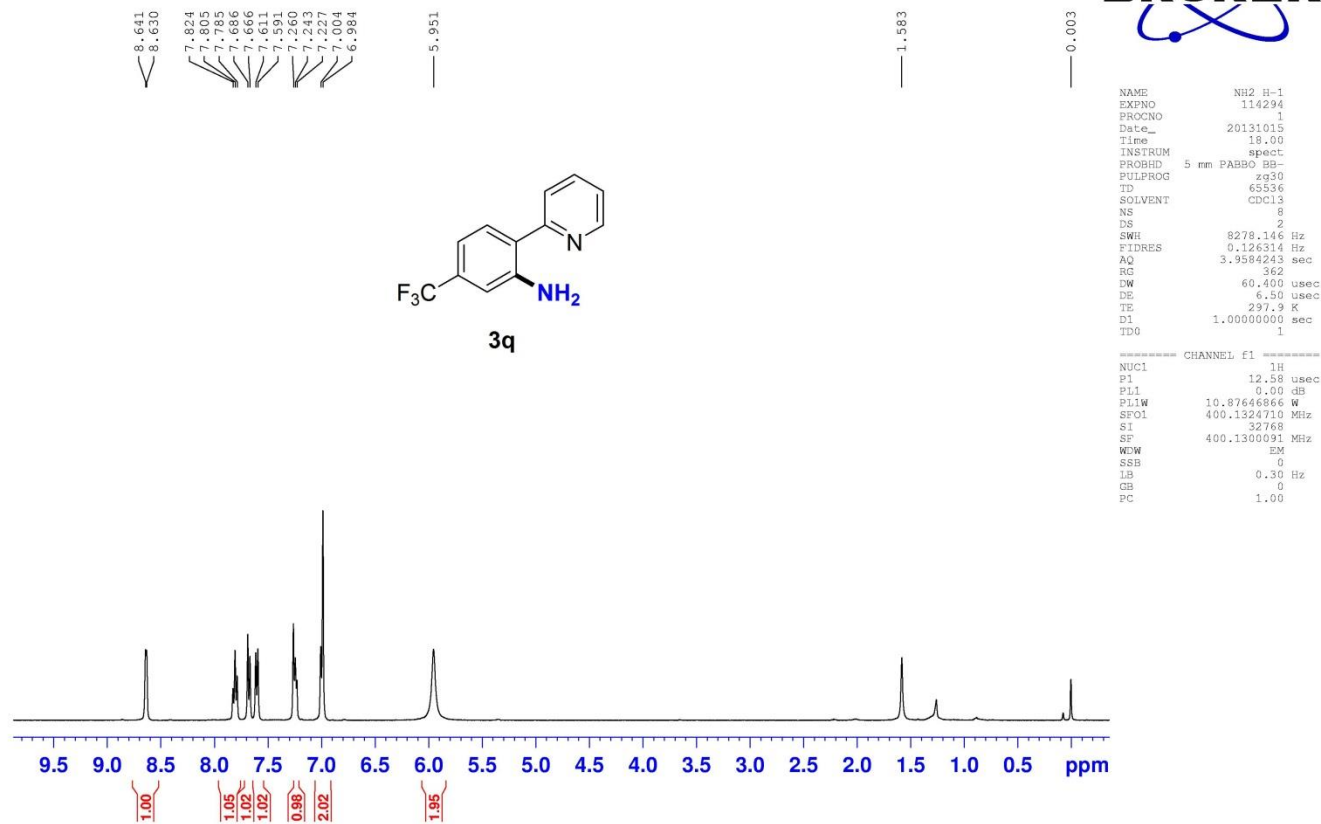
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



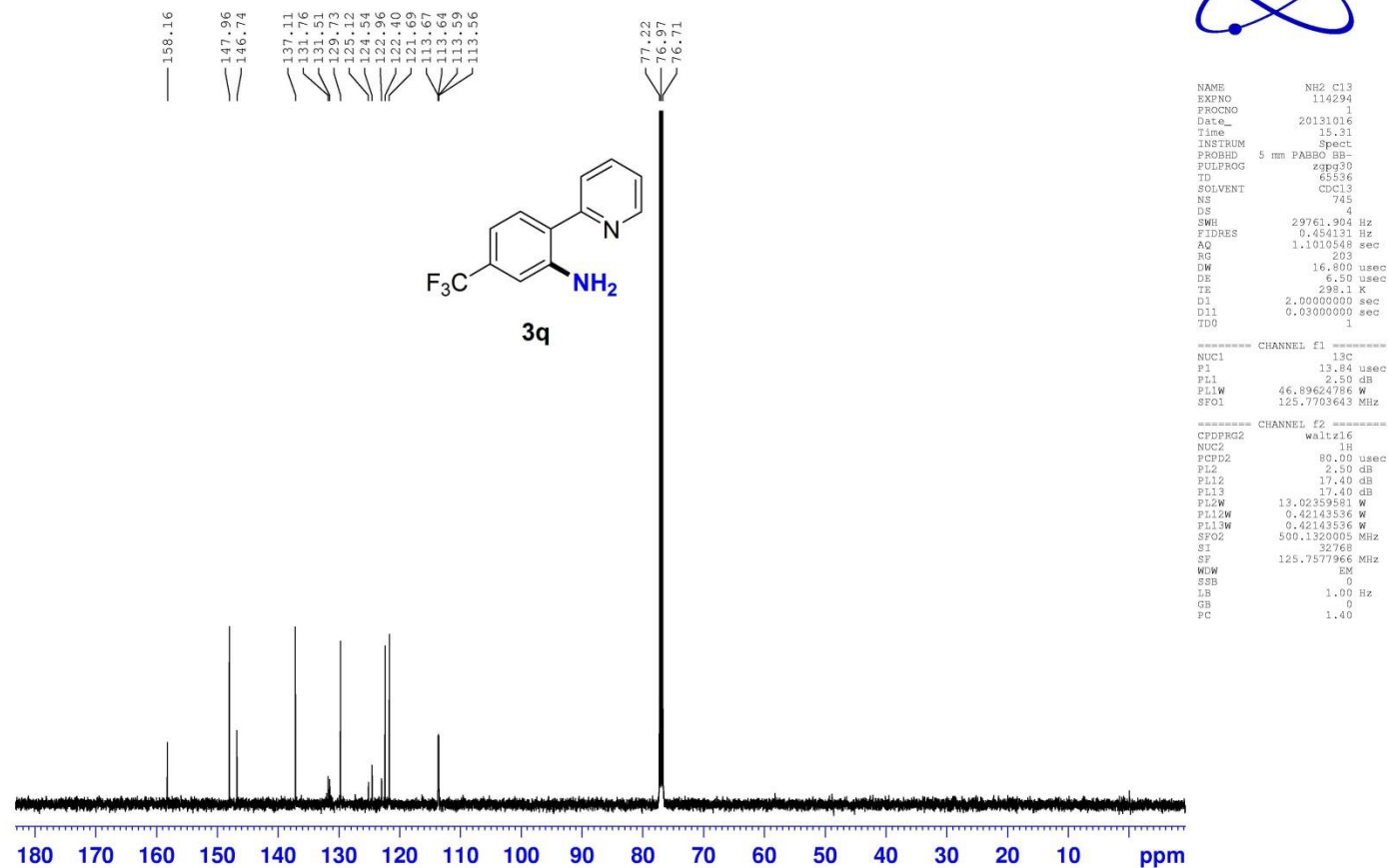
114346



114294



114294



114362

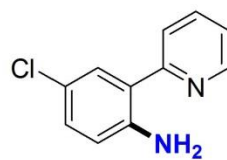
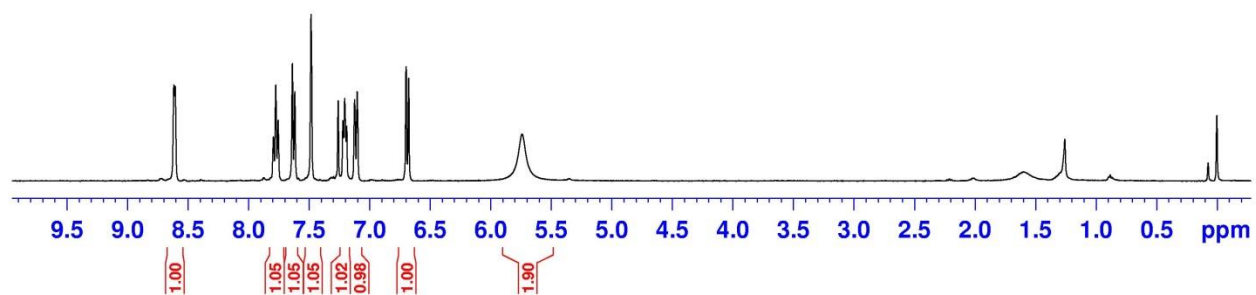
8.614
8.603
7.795
7.776
7.756
7.638
7.618
7.484
7.260
7.220
7.206
7.190
7.124
7.103
6.699
6.678

5.741

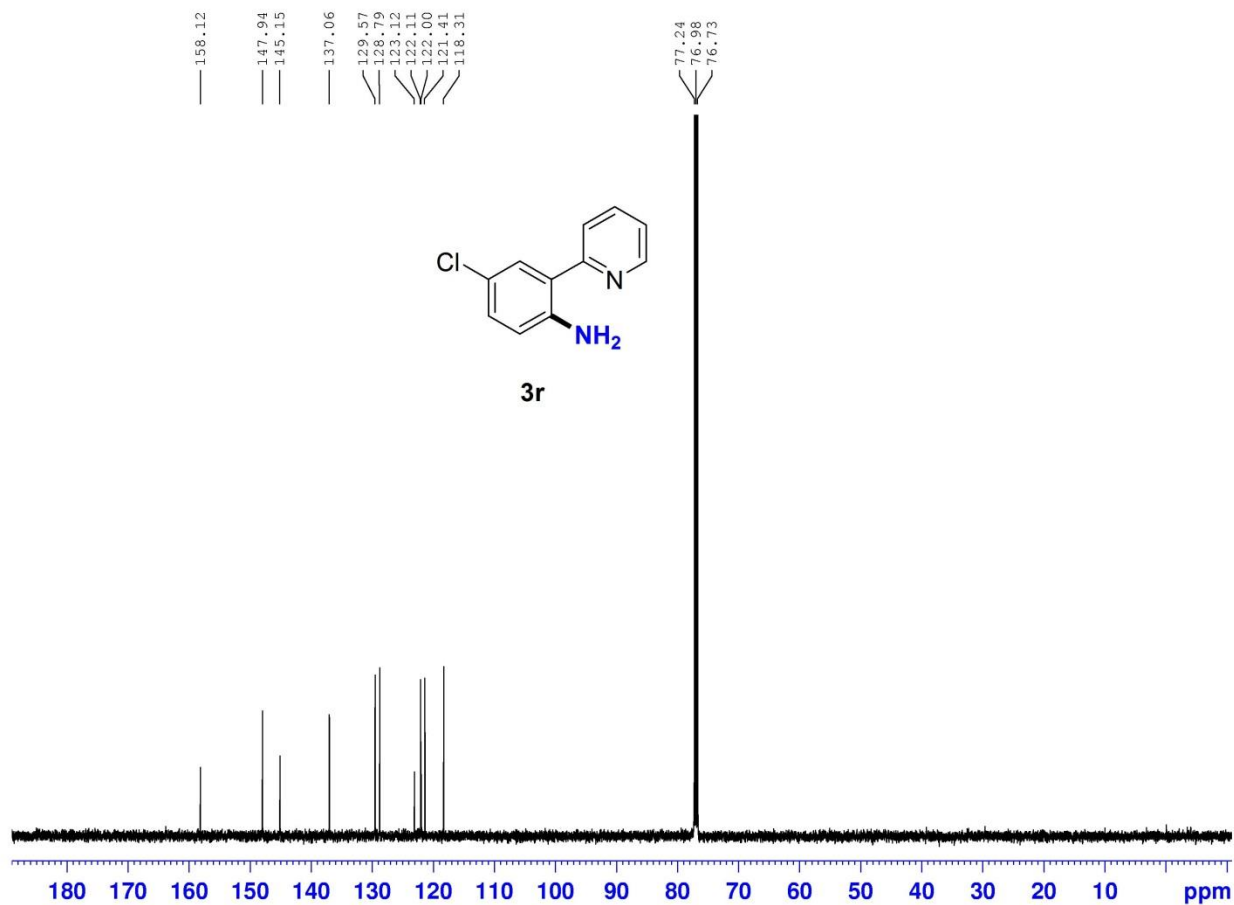


NAME NH2 H-1
EXPNO 114362
PROCNO 1
Date_ 20131120
Time 17.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

**3r**

114362

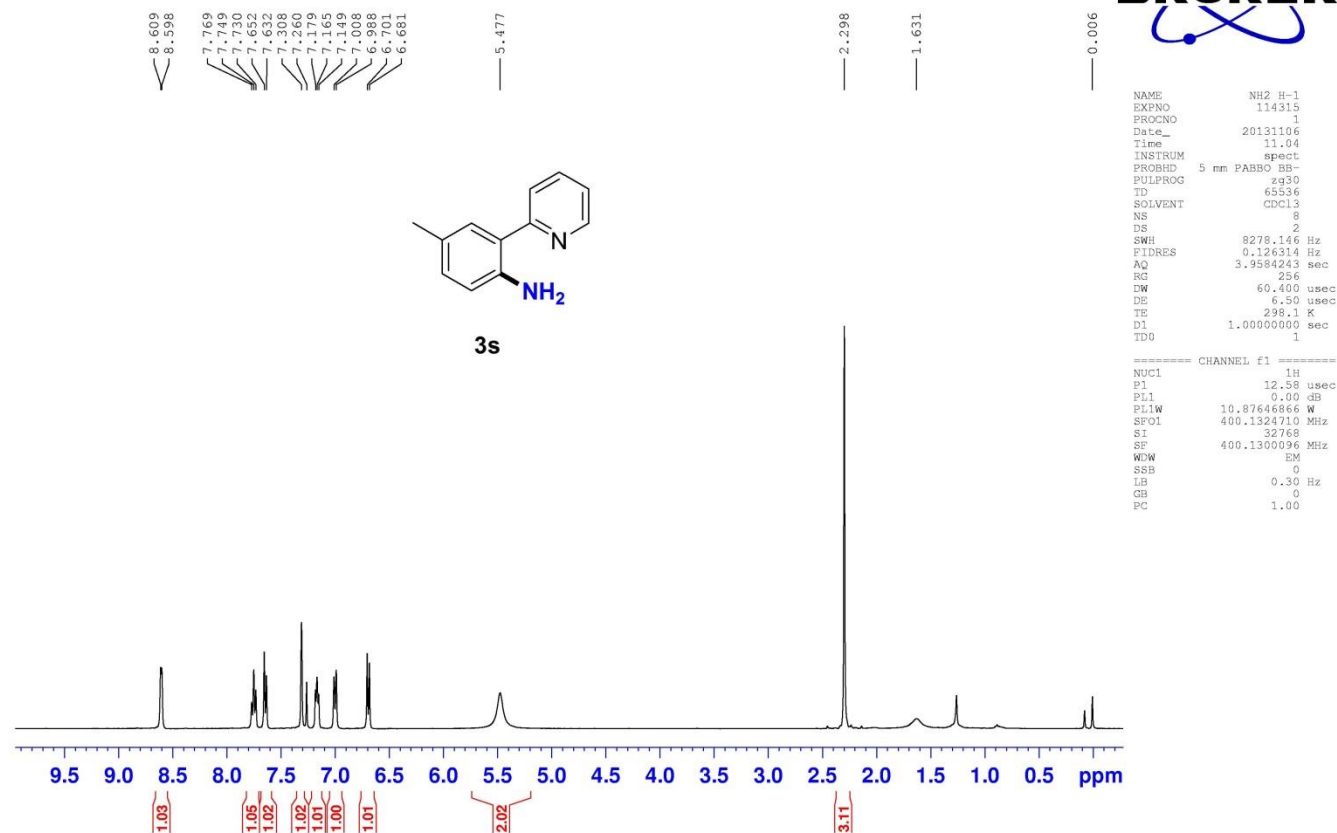


```
NAME      NH2 C13
EXPNO     114362
PROCNO     1
Date_     20131125
Time      16.29
INSTRUM    Spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         411
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         203
RW         16.600 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
```

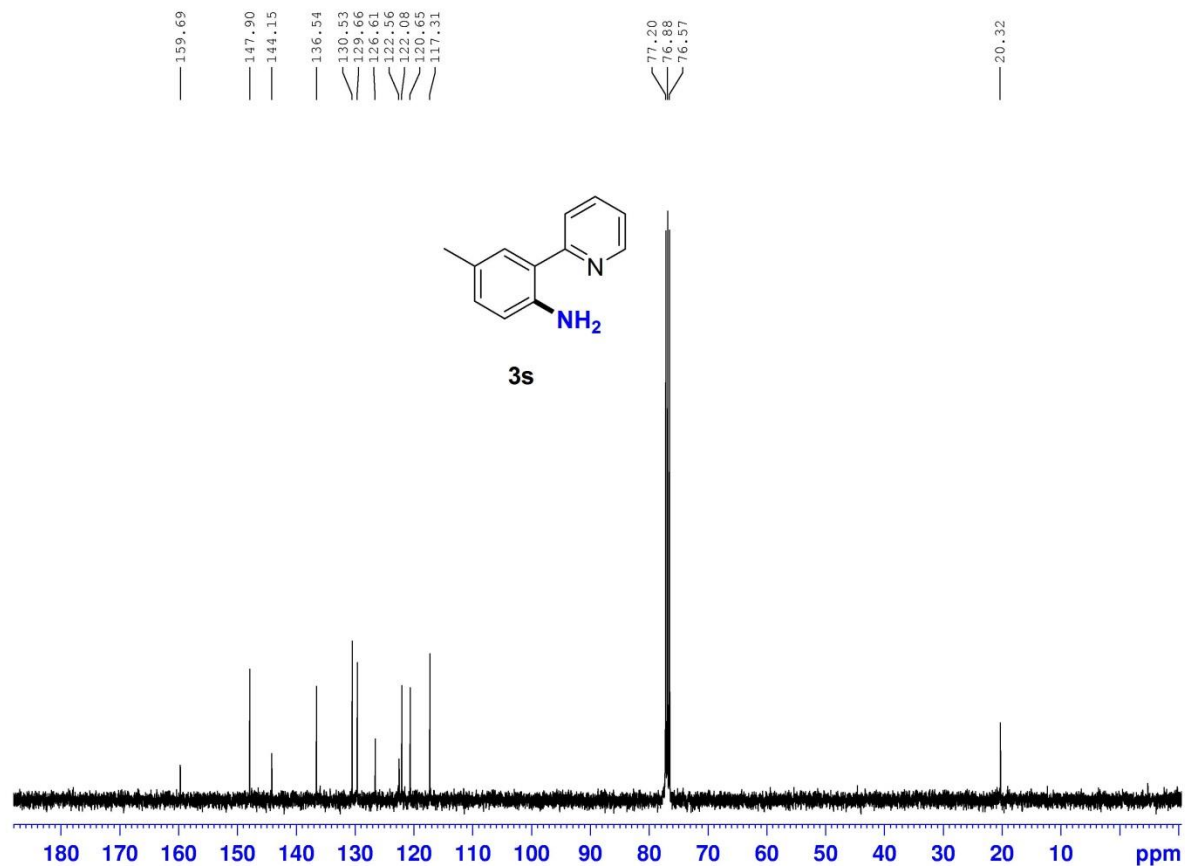
```
===== CHANNEL f1 =====
NUC1       13C
P1         13.84 usec
PL1        2.50 dB
PL1W       46.89624786 W
SFO1       125.7703643 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.50 dB
PL12       17.40 dB
PL13       17.40 dB
PL2W       13.02359581 W
PL12W      0.42143536 W
PL13W      0.42143536 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7577966 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

114315



114315



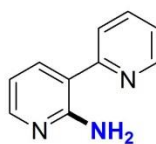
```
NAME      NH2 C13
EXPNO     114315
PROCNO    1
Date_     20131108
Time      9.20
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         400
DS         4
SWH       23980.814 Hz
FIDRES    0.365918 Hz
AQ        1.3664756 sec
RG         2048
DW        20.850 usec
DE         6.50 usec
TE        313.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
```

```
===== CHANNEL f1 =====
NUC1       13C
P1         10.25 usec
PL1        0.00 dB
PL1W       38.68305206 W
SFO1       100.6228298 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        0.00 dB
PL12       16.07 dB
PL13       0.00 dB
PL2W       10.87646866 W
PL12W      0.26883632 W
PL13W      10.87646866 W
SFO2       400.1316005 MHz
SI         32768
SF         100.6127690 MHz
WCM        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

114301

8.624
8.614
8.113
8.103
7.846
7.827
7.793
7.774
7.755
7.697
7.677
7.260
7.213
7.198
6.748
6.725
6.710
6.694

**3t**

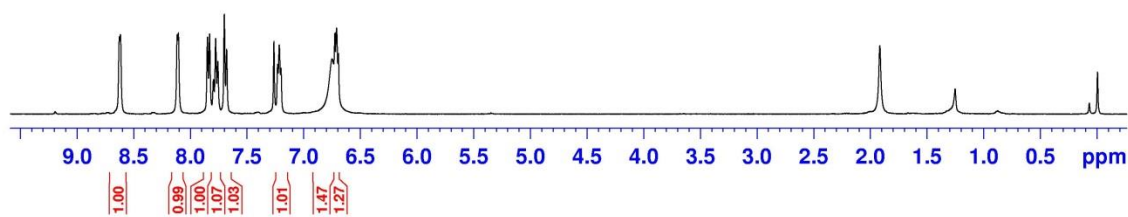
1.916

-0.004

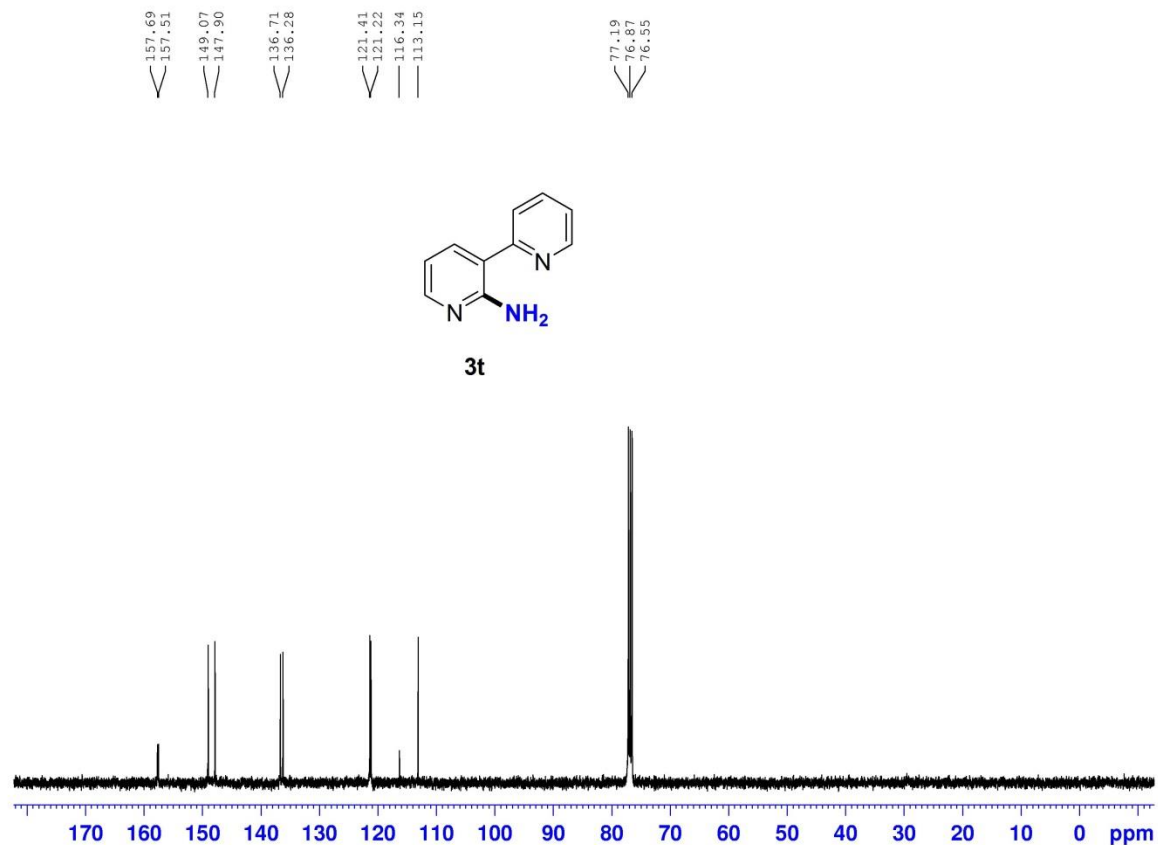


NAME NH2 H-1
EXPNO 114301
PROCNO 1
Date_ 20131029
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114301



NAME NH2 C13
EXPNO 114301
PROCNO 1
Date_ 20131106
Time 5.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1290.2
DW 20.850 usec
DE 6.50 usec
TE 313.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz

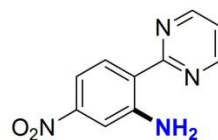
===== CHANNEL f2 =====
PCPD2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL1W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
F1 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

114361

8.847
8.836
8.649
8.627

7.582
7.548
7.526
7.260
7.237
7.226
7.214

6.630



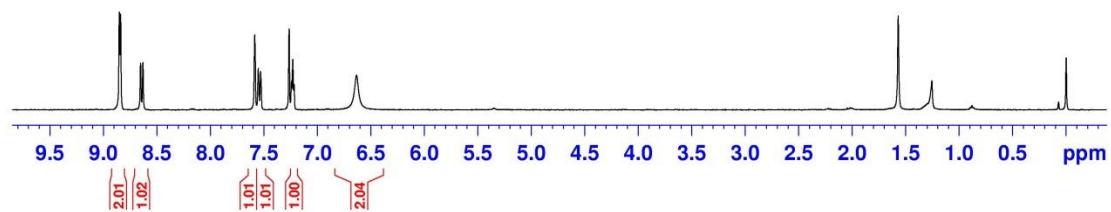
1.569

-0.002

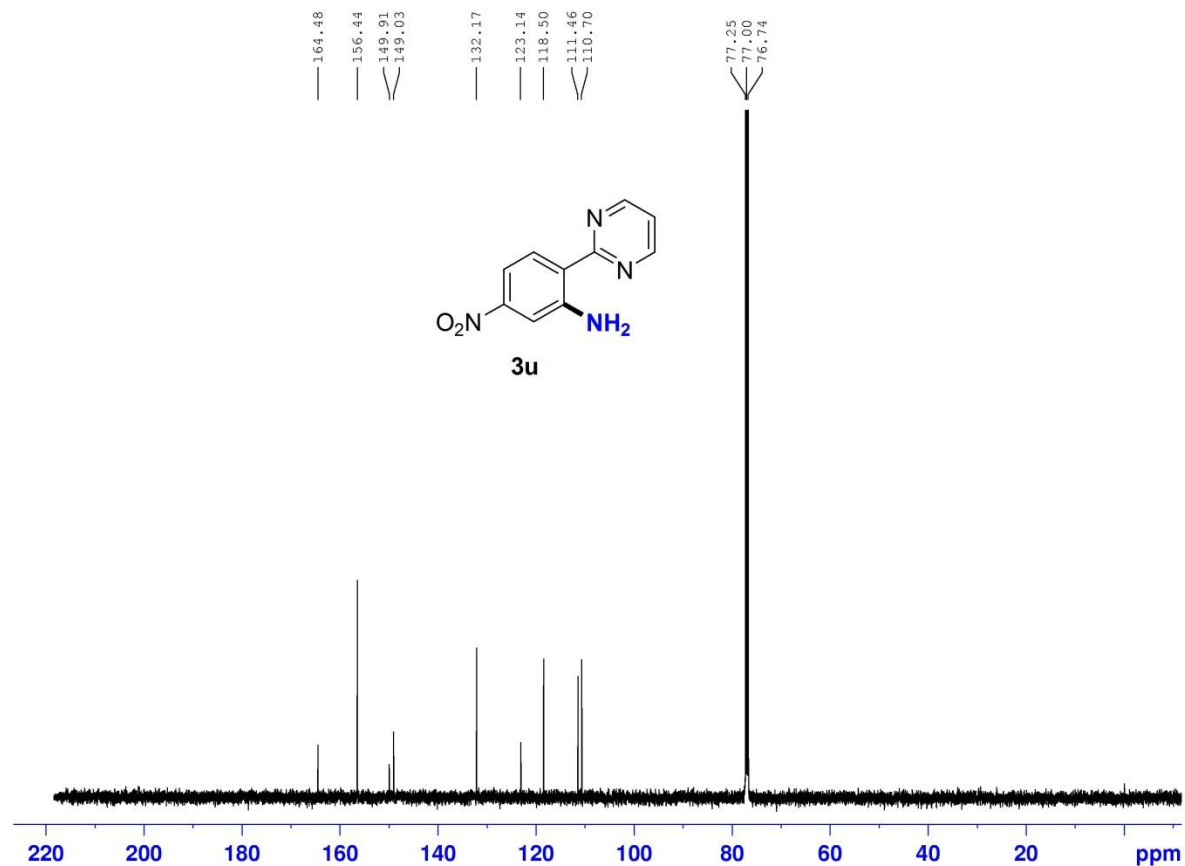


NAME NH2 H-1
EXPNO 114361
PROCNO 1
Date_ 20131205
Time 14.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9594243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114361



NAME NH2 C13
EXPNO 114361
PROCNO 1
Date_ 20131211
Time 23.53
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 980
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577931 MHz
WCM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

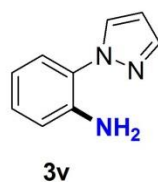
114308



7.746
7.717
7.260
7.193
7.173
7.151
7.132
6.841
6.821
6.803
6.784
6.765
6.444

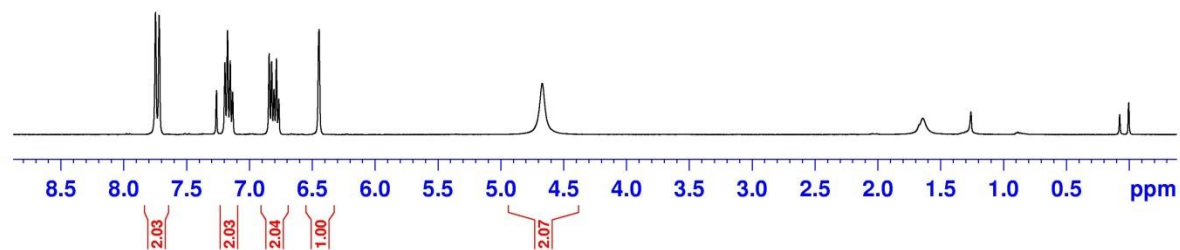
4.671

1.643

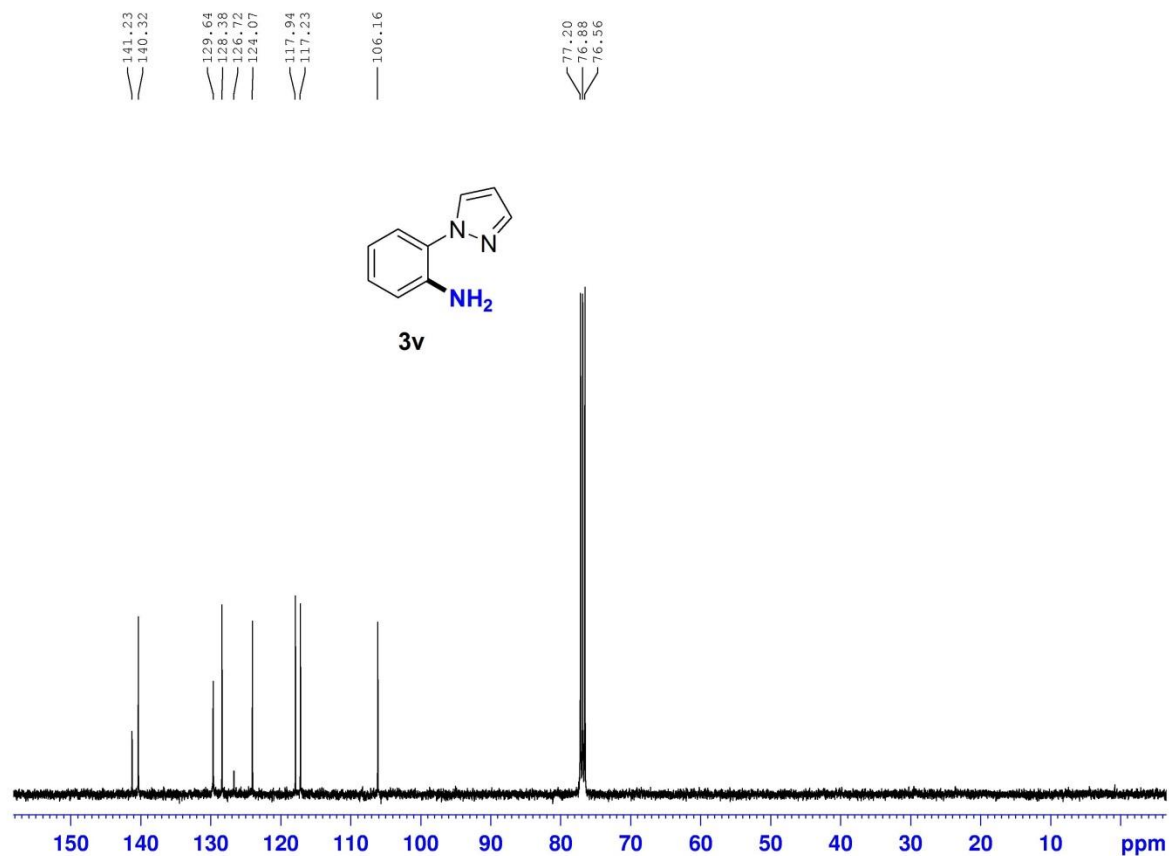


NAME NH2 H-1
EXPNO 114308
PROCNO 1
Date_ 20131031
Time 13.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



114308

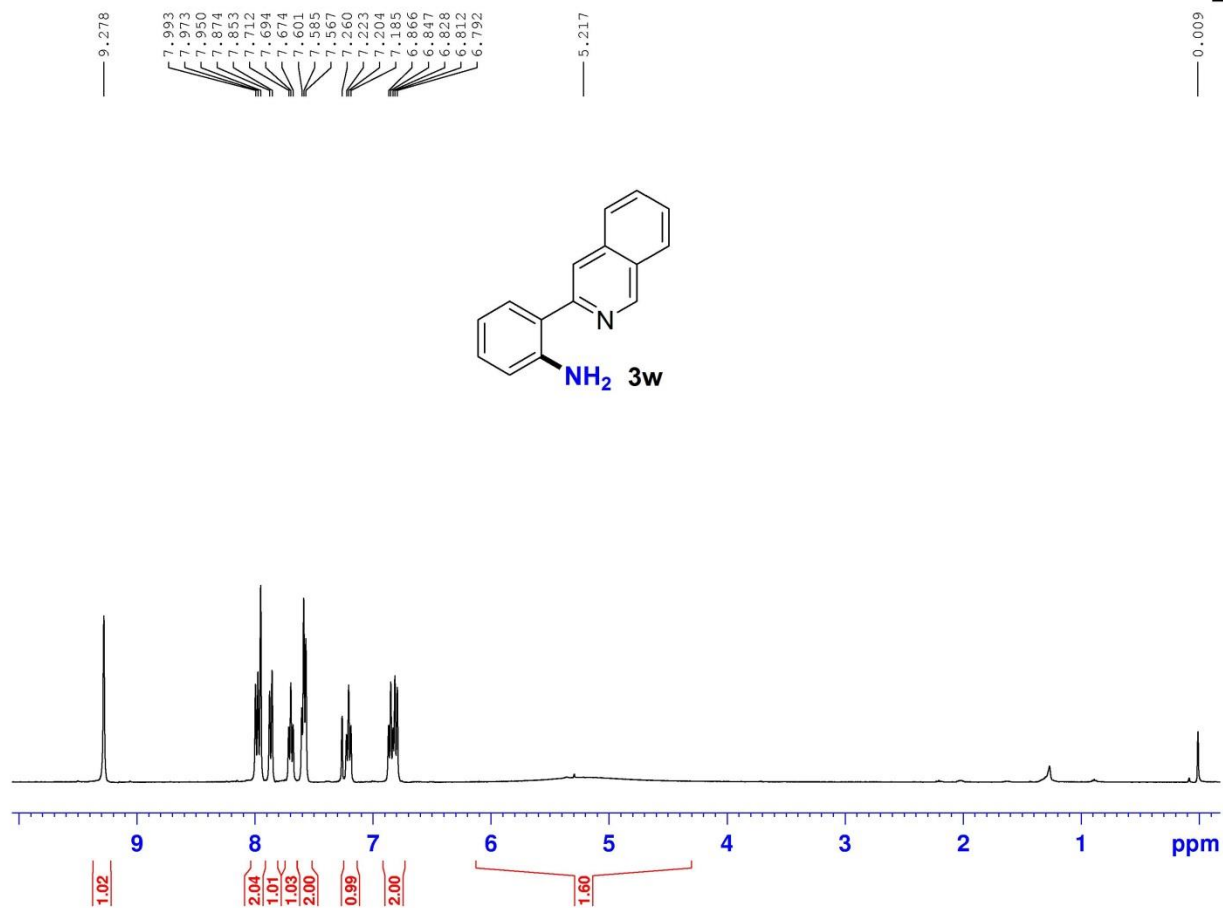


NAME NH2 C13
EXPNO 114308
PROCNO 1
Date_ 20131106
Time 4.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2048
DW 20.850 usec
DE 6.50 usec
TE 313.3 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.25 usec
PL1 0.00 dB
PL1W 38.68305206 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
PCPD2 waitz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.07 dB
PL13 0.00 dB
PL2W 10.87646866 W
PL12W 0.26883632 W
PL13W 10.87646866 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

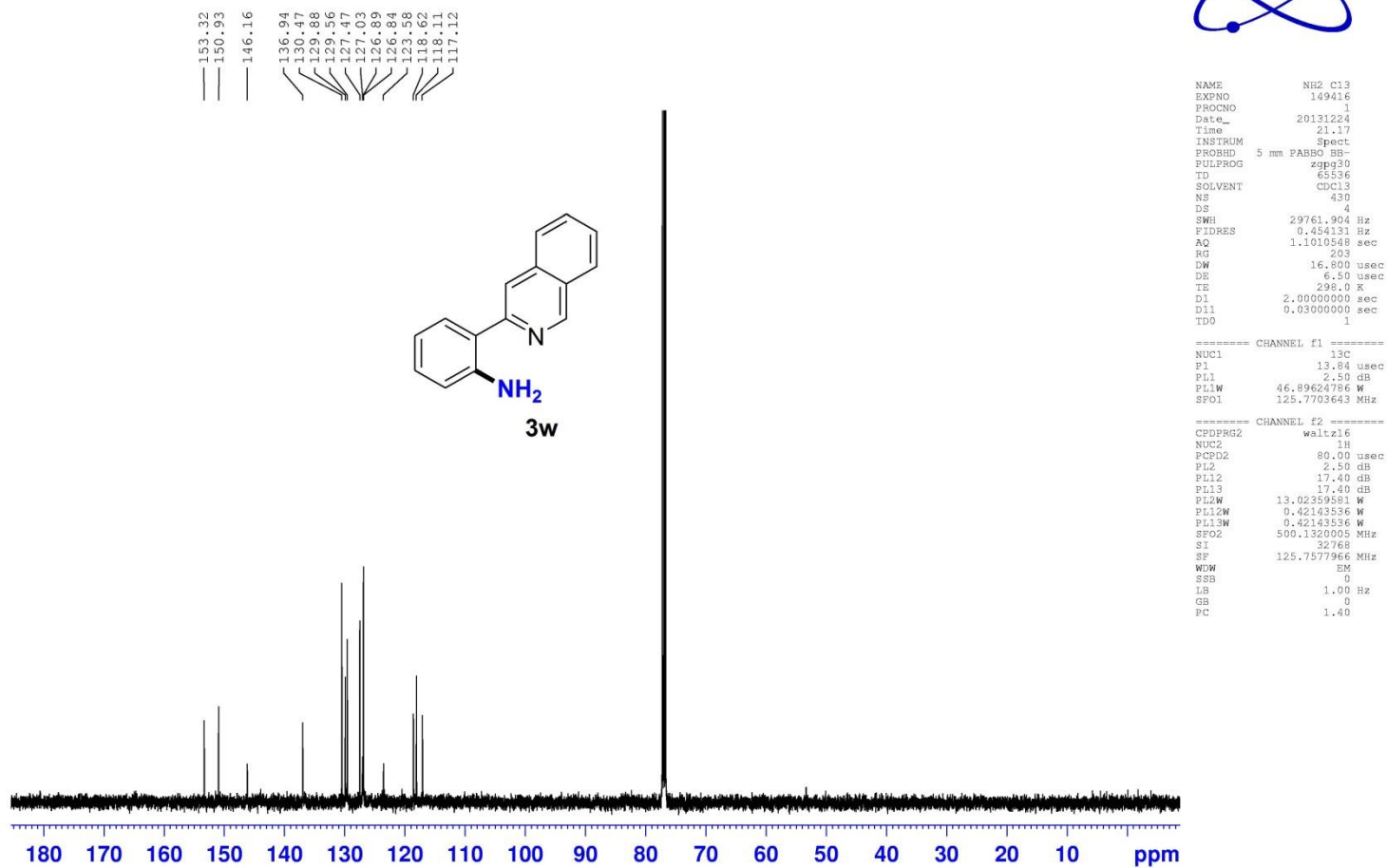
149416



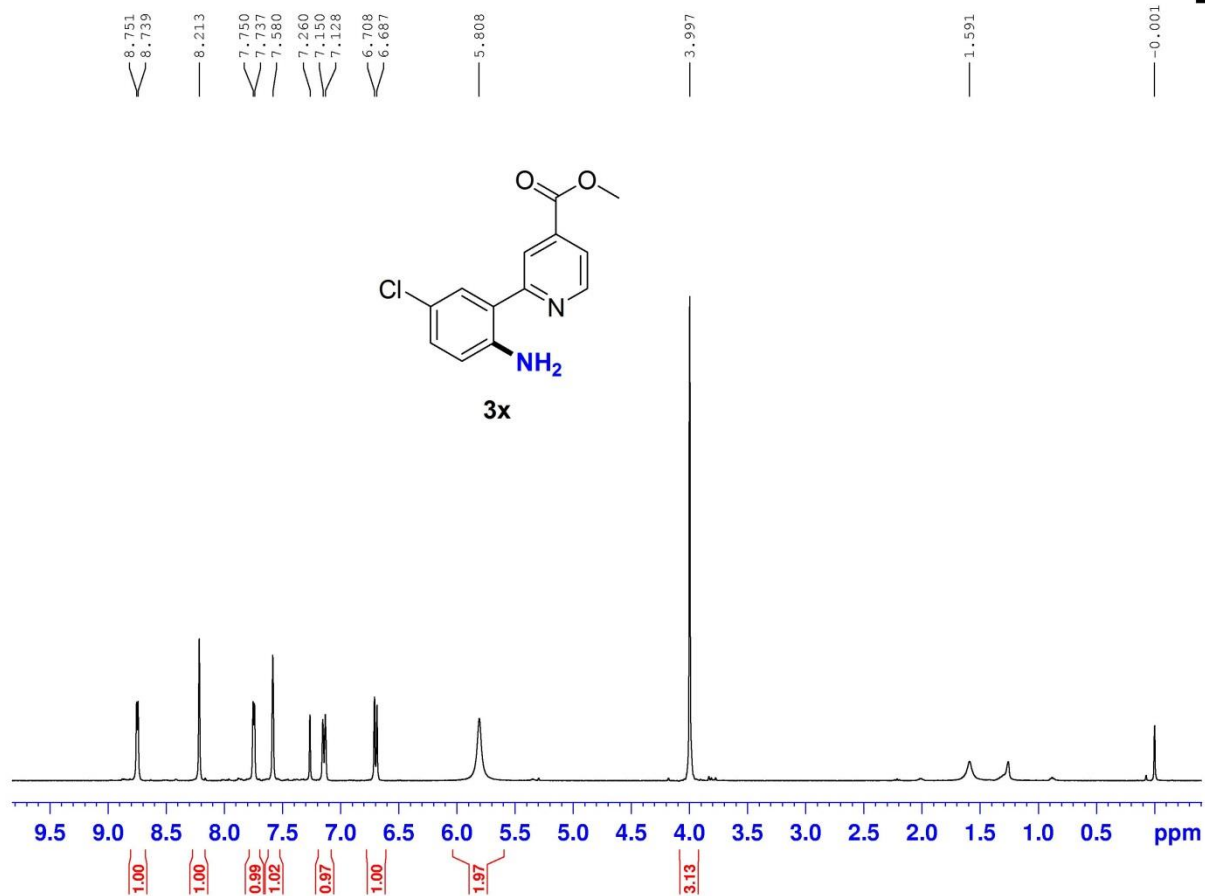
NAME NH2 H-1
EXPNO 149416
PROCNO 1
Date_ 20131224
Time 9.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

149416



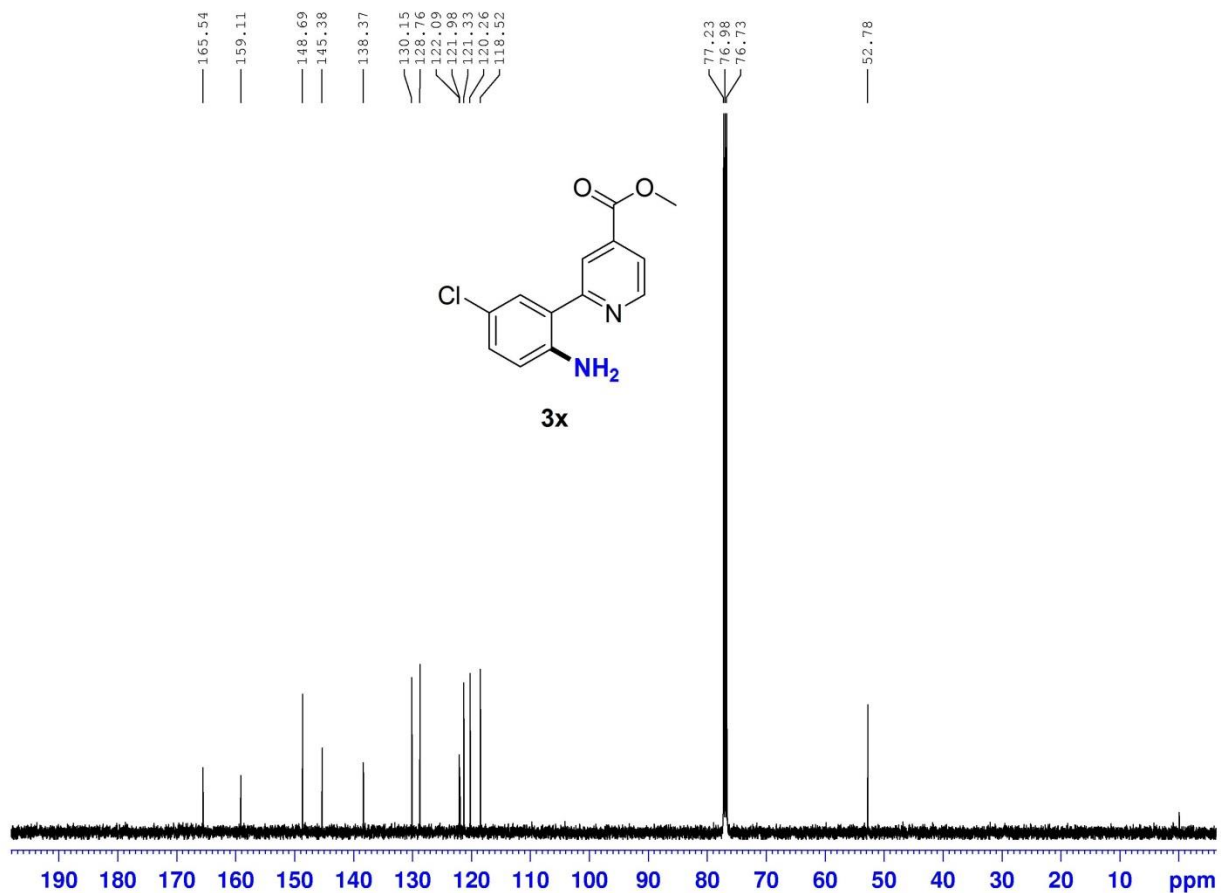
149421



NAME NH2 H-1
EXPNO 149421
PROCNO 1
Date_ 20131224
Time_ 9.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

149421



NAME NH2 C13
EXPNO 149421
PROCNO 1
Date_ 20131224
Time 18.11
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 514
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDZ 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40