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Brønsted acid mediated N–O bond cleavage for α-amination of ketones through aromatic nitroso aldol reaction

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General Procedures

All non-aqueous reactions were carried out under an atmosphere of nitrogen in flame-dried glassware and were stirred using a magnetic stir plate. All reactions were carried out using anhydrous solvent unless otherwise noted. CH_3CN, and CH_2Cl_2 were dried over calcium hydride. Dry THF was prepared by distilling over sodium ketyl.

All reactions were monitored by thin layer chromatography (TLC) on WhatmanPartisil® K6F TLC plates (silica gel 60 Å, 0.25 mm thickness) and visualized using a UV lamp (366 or 254 nm) or by use of one of the following visualization reagents: PMA: 10 g phosphomolybdic acid/ 100 mL ethanol; $KMnO_4$: 0.75 g potassium permanganate, 5 g K_2CO_3, / 100mL water. Products were isolated by column chromatography (Merck silica gel 100-200μm). Yields refer to chromatographically and spectroscopically homogenous materials unless noted otherwise. ^{13}C and ^{1}H NMR spectra were recorded on a Bruker400 or Bruker 500 MHz spectrometers. Chemical shift values (δ) are reported in ppm and calibrated to the residual solvent peak $CDCl_3$ $\delta = 7.2600$ ppm for ^{1}H, $\delta = 77.16$ for ^{13}C; or calibrated to tetramethylsilane ($\delta = 0.00$). All NMR spectra were recorded at ambient temperature (290 K) unless otherwise noted. ^{1}H NMR spectra are reported as follows: chemical shift (multiplicity, coupling constant, integration). The following abbreviations are used to indicate multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; sept, septet; m, multiplet; dd, doublet of doublet; dt, doublet of triplet; dq, doublet of quartet; td, triplet of doublet; tt, triplet of triplet; dq, doublet of quartet; br, broad; app, apparent.

Mass spectra were recorded by electrospray ionization (ESI) method on a Q-TOF Micro with lock spray source. The crystal data were collected and integrated using a BrukerAxs kappa apex2 CCD diffractometer, with graphite monochromated Mo-Kα radiation.

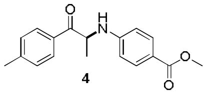
The silyl enol ethers were synthesized following literature procedures published previously and were purified by silica gel column chromatography or distillation (*Angew. Chem. Int. Ed.* 2008, **47**, 3795; *Angew. Chem. Int. Ed.* 2012, **51**, 1942). The parent nitrosobenzene was purchased from Sigma-Aldrich company; other nitrosobezene derivatives were synthesized following literature procedures published previously (*J. Org. Chem*. 2005, **70**, 2350).

Synthesis of silyl enol ethers of 1,7-diphenylheptane-1,7-dione: To a 100mL round bottom flask (primary flask) equipped with a magnetic stir bar and rubber septum was added CH_2Cl_2 (20mL) under nitrogen atmosphere. The flask was cooled to 0ºC and hexamethyldisilane (5.3 mmol, 1.5

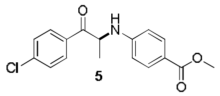
equiv.) followed by triflic acid (5.3 mmol, 1.5 equiv.) was added drop-wise. The mixture was warmed to room temperature and stirred for one hour. A secondary flask was equipped with a rubber septum was charged with CH_2Cl_2 (20mL), triethylamine (7.2 mmol, 2 equiv.), and 1,7-diphenylhepatane-1,7-dione (3.5 mmol, 1equiv.) were added. The secondary flask was cooled to 0ºC and the content of the primary flask was transferred into it via syringe. The reaction was allowed to warm to room temperature and stirred for additional 3 h. Reaction was quenched with saturated aqueous $NaHCO_3$ and extracted with hexanes. Combined organic layers dried over magnesium sulfate and concentrated under reduced pressure to give crude product as a mixture of mono- and di-silylated silyl enol ethers. The pure silyl enol ethers were isolated after purification via silica gel (neutralized with triehtylamine) column chromatography to provide mono-silylated silyl enol ether in 40% yield ($R_f = 0.2$ in hexane). The di-silylated silyl enol ether was isolated only in 10% yield ($R_f = 0.7$ in hexane). However, better yield of di-silylated silyl enol ether (55% yield) was achieved when excess hexamethyldisilane (3.5 equiv.) and triflic acid (3.5 equiv.) were used.

General procedure for the α-amination of ketones using phenylboronic acid promoted N–O bond cleavage:

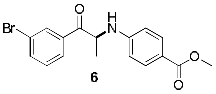
Nitroso compound (1.2 equiv.) was taken in a 16x100 mm oven dried test tube equipped with a magnetic stir. The test tube was capped with a septum and kept under vacuum for 10 min and then purged with nitrogen. Dry CH_3CN (3 mL) and silyl enol ether (0.15 mmol, 1.0 equiv.) were added and the mixture was allowed to stir for 5 h at room temperature. Afterward, phenylboronic acid (3.0 equiv.) was added in one portion and the stirring was continued for additional 17 h at room temperature. The reaction mixture was then transfer to a pear shape flask after dilution with CH_2Cl_2. The solvent was evaporated to dryness. The crude reaction mixture was loaded directly onto silica gel column and purified to provide pure nitroso aldol products **4-16**.


4

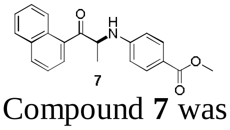
4: Compound **4** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (42 mg, 94%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.86 (m, 4H), 7.31 (d, *J* = 7.96 Hz, 2H), 6.62 (d, *J* = 8.80 Hz, 2H), 5.30 (d, *J* = 6.44 Hz, 1H), 5.17-5.10 (m, 1H), 3.84 (s, 3H), 2.43 (s, 3H), 1.48 (d, *J* = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.1, 167.3, 150.3, 145.1, 131.8 (2×C), 129.8, 128.8, 118.8, 112.1, 52.7, 51.7, 20.8, 19.7; HRMS (TOF MS ES$^+$) $C_{18}H_{19}NO_3H^+$ m/z (%) = 298.1624 ([M+H]$^+$, 100%).


5

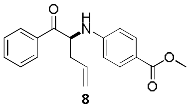
5: Compound **5** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (44 mg, 93%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.92 (m, 2H), 7.89-7.86 (m, 2H), 7.51-7.47 (m, 2H), 6.71-6.69 (m, 2H), 5.12 (q, *J* = 6.92 Hz, 1H), 3.85 (s, 3H), 1.51 (d, *J* = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.1, 167.2, 149.3, 140.7, 132.5, 131.8, 130.1, 129.5, 120.0, 113.1, 53.6, 51.8, 19.3; HRMS (TOF MS ES$^+$) $C_{17}H_{16}ClNO_3Na^+$ m/z (%) = 340.0781 ([M+Na]$^+$, 100%).


6

6: Compound **6** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (48 mg, 90 %) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$):δ = 8.12 (t, *J* = 1.72 Hz, 1H), 7.93-7.91(m, 1H), 7.89-7.86 (m, 2H), 7.75 (dq, J^1 = 0.96 Hz, J^2 = 7.96 Hz, 1H), 7.40 (t, *J* = 7.89 Hz, 1H), 6.66-6.64 (m, 2H), 5.11 (q, *J* = 6.96 Hz, 1H), 3.84 (s, 3H), 1.50 (d, *J* = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.3, 167.3, 149.7, 137.0, 136.1, 131.8, 131.7, 130.7, 127.1, 123.5, 119.5, 112.5, 53.3, 51.8, 19.4; HRMS (TOF MS ES$^+$) $C_{17}H_{17}NO_3BrH^+$ m/z (%) = 362.0392 ([M+H]$^+$, 100%).

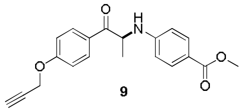

7

7: Compound **7** was prepared following general *α*-amination of ketones through aromatic nitroso aldol reaction protocol described above (24 h reaction time) and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (37 mg, 75.0%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.42 (d, *J* = 8.28 Hz, 1H), 8.05 (d, *J* = 8.24 1H), 7.92-7.87 (m, 4H),7.63-7.52 (m , 3H), 6.75-6.73 (m, 2H), 5.18 (q, *J* = 7.00 Hz, 1H), 3.86 (s, 3H), 1.44 (d, *J* = 7.04 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 202.8, 167.3, 149.8, 134.1, 133.6, 133.4, 131.8, 130.7, 128.7, 128.4, 127.1, 127.0, 125.4, 124.5, 119.5, 112.7, 55.9, 51.7, 18.6; HRMS (TOF MS ES$^+$) $C_{21}H_{19}NO_3K^+$ m/z (%) = 372.1015 ([M+K]$^+$, 100%).



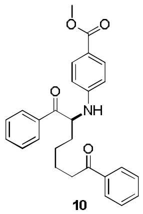
8

8: Compound **8** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (1→10% EtOAc : hexane) to provide pure compound (37 mg, 81%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.00-7.98 (m, 2H), 7.86 (dt, J^1 = 9.44 Hz, J^2 = 2.48 Hz, 2H), 7.65-7.61 (m, 1H),7.54-7.50 (m , 2H), 6.56 (d, *J* = 8.72 Hz, 2H), 5.69-5.63 (m, 1H), 5.23 (t, *J* = 5.36 Hz, 1H), 5.08-5.05 (m, 1H), 5.01-4.96 (m, 1H), 3.84 (s, 3H), 2.82-2.77 (m,1H), 2.55-2.50 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 198.6, 167.3, 150.3, 134.9, 134.1, 131.9, 131.8, 129.1, 128.6, 119.5, 119.3, 112.5, 56.9, 51.7, 36.9; HRMS (TOF MS ES$^+$) $C_{19}H_{19}NO_3H^+$ m/z (%) = 310.1559 ([M+H]$^+$, 100%).



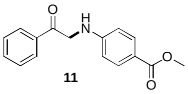
9

9: Compound **9** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (36 mg,72%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.01-8.00 (m, 2H), 7.87-7.84 (m, 2H), 7.07-7.06 (m, 2H), 6.63-6.60 (m , 2H), 5.29 (s, 1H), 5.11 (s, 1H), 4.78-4.77 (m, 2H), 3.83 (s, 3H), 2.58-2.57 (m,1H), 1.48 (d, *J* = 5.52 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 197.9, 167.4, 162.0, 150.3, 131.8, 130.9, 127.8, 118.8, 115.1, 112.1, 77.7, 76.5, 56.0, 52.5, 51.7, 19.7; HRMS (TOF MS ES$^+$) $C_{20}H_{19}NO_4H^+$ m/z (%) = 338.1372 ([M+H]$^+$, 100%).



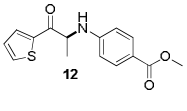
10

10: Compound **10** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→15% EtOAc : hexane) to provide pure compound (63 mg, 98%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.91 (dd, J^1 = 8.48 Hz, J^2 = 1.28 Hz, 2H), 7.79-7.77 (m, 4H), 7.57-7.53 (m, 1H), 7.47-7.42 (m, 3H), 7.34 (t, J = 7.84 Hz, 2H) 6.56 (d, J = 8.80 Hz, 2H), 5.17-5.15 (m, 1H) 5.12-5.10 (m, 1H), 3.76 (s, 3H), 2.79 (td, , J^1 = 7.08 Hz, J^2 = 1.32 Hz, 2H), 2.02-1.98 (m, 1H), 1.67-1.61 (m, 1H), 1.60-1.55 (m, 1H), 1.35-1.20 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 200.0, 199.5, 167.3, 150.7, 136.9, 134.7, 134.1, 133.1, 131.8, 129.1, 128.7, 128.5, 128.1, 119.0, 122.2, 57.0, 51.7, 38.2, 32.8, 24.6, 24.0; HRMS (TOF MS ES$^+$) $C_{27}H_{27}NO_4H^+$ m/z (%) = 430.2129 ([M+H]$^+$, 100%).



11

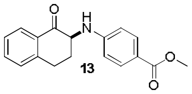
11: Compound **11** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (28 mg, 70%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.03-8.01 (m, 2H), 7.92-7.90 (m, 2H), 7.66-7.63 (m, 1H), 7.55-7.51 (m, 2H), 6.68-6.66 (m, 2H), 4.65 (s, 2H), 3.85 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 194.2, 167.4, 150.7, 134.7, 134.3, 131.8, 129.1, 128.0, 119.1, 112.1, 51.7, 49.6; HRMS (TOF MS ES$^+$) $C_{16}H_{15}NO_3H^+$ m/z (%) = 270.1130 ([M+H]$^+$, 100%).



12

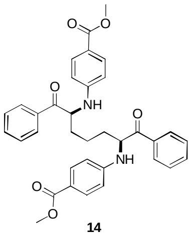
12: Compound **12** was prepared following general *α*-amination of ketones through aromatic nitroso aldol reaction protocol described above and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (37 mg, 86%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.87-7.85 (m, 3H), 7.72-7.71 (m, 1H), 7.18 (dd, J^1 = 3.84

Hz, J^2 = 3.08 Hz, 1H), 6.71-6.67 (m, 2H), 4.95 (q, J = 5.52 Hz, 1H), 3.84 (s, 3H), 1.59 (d, J = 5.52 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 192.6, 167.3, 149.5, 140.8, 135.2, 133.0, 131.8, 128.7, 113.1, 52.2, 51.9, 20.1; HRMS (TOF MS ES$^+$) $C_{15}H_{15}NO_3SH^+$ m/z (%) = 290.0934 ([M+H]$^+$).



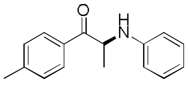
13

13: Compound **13** was prepared following general *α*-amination of ketones through aromatic nitroso aldol reaction protocol described above and purified by silica gel column chromatography (1-15% EtOAc : hexane) to provide pure compound (25 mg, 55%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.06 (dd, J^1 = 6.64 Hz , J^2 = 1.2 Hz 1H), 7.90 dd, J^1 = 5.00 Hz , J^2 = 1.8 Hz 2H), 7.54 (td, J^1 = 7.48 Hz , J^2 = 1.4 Hz 1H), 7.36 (t, J = 7.60 Hz 1H), 7.30 (d, J = 7.68 Hz, 1H), 6.67 (dd, J^1 = 6.96 Hz , J^2 = 1.92 Hz 2H), 5.60 (d, J = 3.84 Hz, 1H), 4.28(dt, J^1 = 13.52. Hz , J^2 = 4.28 Hz, 1H), 3.86 (s, 3H), 3.35-3.26 (m, 1H), 3.10 (ddd, J^1 = 17.16 Hz , J^2 = 3.88 Hz, J^3 = 2.72 Hz 1H), 2.77-2.70 (m, 1H), 1.98 (qd, J^1 = 26.56 Hz , J^2 = 4.36 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.0, 167.4, 150.5, 143.5, 134.2, 131.8, 131.6, 129.1, 127.9, 127.1, 119.2, 112.2, 59.4, 51.8, 30.6, 28.4.



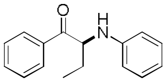
14

14: Compound **14** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (10→35% EtOAc : hexane) to provide pure compound (80 mg, 92%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$):δ = 7.87-7.85 (m, 4H), 7.83-7.80 (m, 4H), 7.62-7.57 (m, 2H), 7.48-7.43 (m, 4H), 6.54-6.52 (m, 4H), 5.07-5.02 (m, 4H) 3.84 (s, 6H), 2.04-1.90 (m, 2H), 1.67-1.59 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 199.2 (2×C), 167.3, 150.5 (2×C), 134.7 (2×C), 134.1, 131.8, 129.2,128.4, 119.3 (2×C), 112.3, 56.9 (2×C), 51.7, 32.7, 32.5, 20.5, 20.2; HRMS (TOF MS ES$^+$) $C_{35}H_{34}N_2O_6H^+$ m/z (%) = 579.2536 ([M+H]$^+$, 100%).



15

15: Compound **15** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (33 mg, 92%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 2H), 7.31-7.29 (m, 2H), 7.19-7.14 (m, 2H), 6.73-6.65 (m, 3H), 5.10 (q, *J* = 6.88 Hz, 1H), 4.71 (s, 1H), 2.43 (s, 3H), 1.47 (d, *J* = 6.88 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 146.8, 144.7, 132.3, 129.7, 129.5, 128.7, 118.0, 113.6, 53.4, 21.8, 19.8; HRMS (TOF MS ES$^+$) $C_{16}H_{17}NO_3H^+$ m/z (%) = 240.1434 ([M+H]$^+$, 100%).

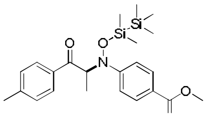


16

16: Compound **16** was prepared following the general procedure for *α*-amination of ketones described above and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (36 mg, 96%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00-7.98 (m, 2H), 7.60 (tt, J^1 = 7.36 Hz, J^2 = 1.28 Hz, 1H), 7.52-7.48 (m, 2H), 7.19-7.15 (m, 2H), 6.72-6.68 (m, 3H), 5.08-5.06 (m, 1H) 4.71 (s,1H), 2.09-2.04 (m, 1H), 1.77-1.70 (m,1H), 0.90 (t, *J* = 7.44 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 147.2, 135.4, 133.7, 129.5, 129.0, 128.5, 118.0, 113.7, 58.9, 26.0, 9.3; HRMS (TOF MS ES$^+$) $C_{17}H_{17}NO_3Na^+$ m/z (%) = 240.1477 ([M+Na]$^+$, 100%).

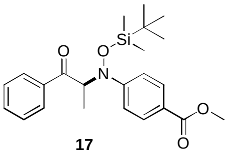
Isolation of the intermediates 3 and 17:

Nitroso compound (1.2 equiv.) was taken in a 16x100 mm oven dried test tube equipped with a magnetic stir. The test tube was capped with a septum and kept under vacuum for 10 min and then purged with nitrogen. Dry CH_3CN (3 mL) and the corresponding silyl enol ether (0.15, mmol, 1.0 equiv.) were added and the mixture was allowed to stir at room temperature until sillyl enol ether was consumed (for TMS-SiMe$_2$-silyl enol ether, 5 h; for TBS-silyl enol ether, 16 h). The reaction mixture was transfer to a pear shape flask after dilution with CH_2Cl_2. The solvent was evaporated to dryness. The crude reaction mixture was loaded directly onto silica gel column and purified to provide pure nitroso aldol intermediates **3** or **17**.



3

3: Compound **3** was prepared following the above procedure and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (65 mg, 98%) as colorless liquid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.98-7.92 (m, 4H), 7.24-7.22 (m, 2H), 7.20-7.18 (m, 2H), 4.96 (q, *J* = 6.64 Hz, 1H), 3.89 (s, 3H), 2.40 (s, 3H) 1.28 (d, *J* = 6.64 Hz, 3H), – 0.01 (s, 3H), – 0.02 (s, 3H), – 0.04 (s, 9H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 196.3, 167.0, 156.8, 143.9, 134.4, 130.5, 129.3, 129.2, 124.8, 118.4, 68.0, 52.0, 21.8, 11.4, – 0.5, – 2.1.

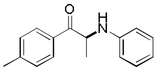


17

17: Compound **17** was prepared following the above procedure and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (58 mg, 95%) as colourless liquid. ^{1}H NMR (500 MHz, CDCl$_3$): δ = 7.99 (d, *J* =7.55 Hz, 2H), 7.95-7.93 (m, 2H), 7.54-7.51 (m, 1H), 7.24 (t, *J* = 7.65, 2H), 7.23-7.21 (m, 2H), 4.94 (q, *J* = 6.7 Hz, 1H), 3.88 (s, 3H), 1.31 (d, *J* = 6.7 Hz, 3H), 0.87(s, 9H), – 0.12 (s, 3H), – 0.29 (s, 3H); ^{13}C NMR (125 MHz, CDCl$_3$): δ = 196.9, 166.9, 157.0, 137.2, 133.1, 130.3, 129.0, 128.5, 125.3, 119.1, 68.4, 52.0, 26.1, 18.1, 11.8, – 4.70 (2×C).

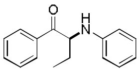
General procedure for the α-amination of ketones using silica gel promoted N–O bond cleavage:

Nitroso compound (1.2 equiv.) was taken in a 16x100 mm oven dried test tube equipped with a magnetic stir. The test tube was capped with a septum and kept under vacuum for 10 min and then purged with nitrogen. Dry CH$_3$CN (3 mL) and silyl enol ether (0.15, mmol, 1.0 equiv.) were added and the mixture was allowed to stir for 24 h at room temperature. The reaction mixture was transfer to a pear shape flask after dilution with CH$_2$Cl$_2$. The solvent was evaporated to dryness. The crude reaction mixture was loaded directly onto silica gel column (30 g of Merck 100-200 mesh silica gel was used and height of the column was 30 cm) and purified to provide pure nitroso aldol products **15-41**.



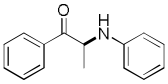
15

15: Compound **15** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (31 mg, 90%) as white solid. Details of NMR-spectra were described in the preceding paragraph.



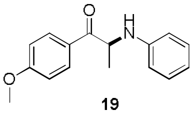
16

16: Compound **16** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (36 mg, 96%) as white solid. Details of NMR-spectra were described in the preceding paragraph.



18

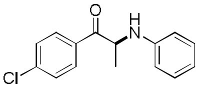
18: Compound **18** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (33 mg, 96%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.02-8.01 (m, 2H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.85 Hz, 2H), 7.19-7.16 (m, 2H), 6.72 (t, *J* = 7.35 Hz, 1H), 6.68 (d, *J* = 7.75 Hz, 2H), 5.13 (q, *J* = 6.85 Hz, 1H) 4.71 (s,1H), 1.48 (d, *J* = 6.95 Hz, 3H) ; ^{13}C NMR (100 MHz, CDCl$_3$): δ = 200.9, 146.8, 134.9, 133.9, 129.6, 129.1, 128.7, 118.1, 113.7, 53.6, 19.8; HRMS (TOF MS ES$^+$) $C_{15}H_{15}NOH^+$ m/z (%) = 226.1373 ([M+H]$^+$, 100%).



19

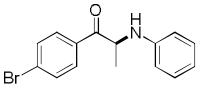
19: Compound **19** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→2% EtOAc : hexane) to provide pure compound (32 mg, 84%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.01 (dt, J^1 = 8.92 Hz, J^2 = 2.84 Hz, 2H), 7.19-7.14 (m, 2H), 6.99-6.96 (m, 2H), 6.72-6.66 (m, 3H), 5.09-5.06

(m, 1H), 4.72 (s, 1H), 3.89 (s, 3H) 1.47 (d, *J* = 6.88 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.2, 164.1, 146.8, 130.9, 129.5, 127.7, 117.9, 114.2, 113.6, 55.7, 53.1, 19.9.



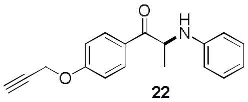
20

20: Compound **20** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (34 mg, 86%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$):δ = 7.96 (dt, J^1 =8.64 Hz, J^2 = 2.4 Hz, 2H), 7.48 (dt, J^1 =8.64 Hz, J^2 = 2.4 Hz, 2H), 7.20-7.15 (m, 2H), 6.73 (tt, J^1 =8.32 Hz, J^2 = 1 Hz, 1H), 6.67-6.64 (m, 2H), 5.07 (m,1H), 4.64-4.63 (m, 1H), 1.47 (d, *J* = 6.92 Hz, 3H);^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.7, 146.5, 140.3, 133.2, 130.0, 129.6, 129.4, 118.2, 113.6, 53.6, 19.6; HRMS (TOF MS ES$^+$) $C_{15}H_{14}ClNOH^+$ m/z (%) = 304.0379 ([M+H]$^+$, 100%).



21

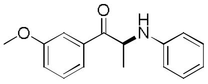
21: Compound **21** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (37 mg, 80%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89-7.86 (m, 2H), 7.67-7.63 (m, 2H), 7.20-7.15 (m, 2H), 6.75-6.71 (m, 1H), 6.67-6.65 (m, 2H), 5.07-5.05 (s,1H), 4.64 (m, 1H), 1.47 (d, *J* = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 146.5, 133.5, 132.4, 130.1, 129.6, 129.0, 118.2, 113.6, 53.6, 19.6; HRMS (TOF MS ES$^+$) $C_{17}H_{15}BrNOH^+$ m/z (%) = 304.0379 ([M+H]$^+$, 100%).



22

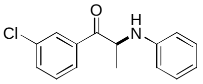
22: Compound **22** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (33 mg, 79%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.04-8.01 (m, 2H), 7.19-7.16 (m, 2H), 7.08-7.05 (m, 2H), 6.73-6.70 (m, 1H), 6.68-6.66 (m, 2H), 5.08 (q, *J* =

6.90 Hz, 1H), 4.77 (d, J = 2.35 Hz, 2H), 4.72 (brs, 1H), 2.57 (t, J = 2.40 Hz, 1H), 1.47 (d, J = 6.90 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 199.2, 161.8, 146.7, 130.8, 129.5, 128.4, 117.9, 115.0, 113.6, 77.7, 76.5, 56.0 53.1, 19.8.



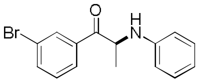
23

22: Compound **22** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (33 mg, 87%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.61-7.59 (m, 1H), 7.54-7.53 (m, 1H), 7.42 (t, J = 7.96 Hz, 1H), 7.20-7.14 (m, 3H), 6.74-6.67 (m, 3H), 5.11 (q, J = 6.88 Hz, 1H), 4.70 (s, 1H), 3.86 (s, 3H), 1.48 (d, J = 6.88 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.7, 160.2, 146.7, 136.2, 130.0, 129.5, 121.0, 120.1, 118.0, 113.6, 113.1, 55.6, 53.6, 19.8.



24

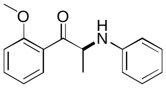
24: Compound **24** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (33 mg, 84%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98 (t, J = 1.54 Hz, 1H), 7.88 (dt, J^1 = 7.80 Hz, J^2 = 1.4 Hz, 1H), 7.60-7.57 (m, 1H), 7.45 (t, J = 7.80 Hz, 1H), 7.21-7.16 (m, 2H), 6.75-6.71 (m, 1H), 6.67 (dd, J^1 = 8.6 Hz, J^2 = 1 Hz, 2H), 5.07 (q, J = 6.90 Hz, 1H), 4.63 (s, 1H), 1.48 (d, J = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.7, 146.5, 136.4, 135.4, 133.7, 130.3, 129.6, 128.7, 126.6, 118.3, 113.7, 53.8, 19.6; HRMS (TOF MS ES$^+$) $C_{15}H_{14}ClNOH^+$ m/z (%) = 260.0900 ([M+H]$^+$, 100%).



25

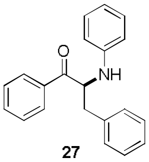
25: Compound **25** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (38 mg, 84%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$):δ = 8.14 (t, J = 1.7

Hz, 1H), 7.94-7.92 (m, 1H), 7.74 (ddd, J^1 = 8.00 Hz, J^2 = 1.96 Hz, J^3 = 1 Hz, 1H), 7.39 (t, J = 7.88 Hz, 1H), 7.21-7.16 (m, 2H), 6.75-6.72 (m, 1H), 6.67 (dd, J^1 = 8.64 Hz, J^2 = 1Hz, 2H), 5.06 (q, J = 6.96 Hz, 1H), 4.64 (s, 1H), 1.48 (d, J = 6.90 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 146.4, 136.6, 131.6, 130.6, 129.5, 127.0, 123.4, 121.8, 118.3, 113.7, 53.7, 19.5.



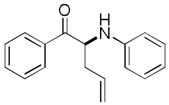
26

26: Compound **26** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (31 mg, 80%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$):δ = 7.72 (dd, J^1 = 7.72 Hz, J^2 = 1.8 Hz, 1H), 7.52-7.47 (m, 1H),7.20-7.15 (m, 2H), 7.04-6.98 (m, 2H), 6.78-6.73 (m, 3H), 5.23 (q, J = 6.96 Hz, 1H), 3.94 (s, 3H), 1.42 (d, J = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.3, 158.5, 146.1, 134.3, 131.3, 129.4, 126.0, 121.2, 118.7, 114.7, 111.7, 58.3, 55.8, 18.4.



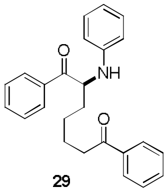
27

27: Compound **27** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (40 mg, 89%) as yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96 (d, J = 7.92 Hz, 2H), 7.60 (t, J = 7.36 Hz, 1H),7.49 (t, J = 7.64 Hz, 2H), 7.24-7.16 (m, 5H), 7.07-7.05 (m, 2H), 6.75-6.72 (m, 2H), 6.68-6.66 (m, 2H), 5.34(s,1H), 4.64 (s, 1H), 3.30 (dd, J^1 = 13.76 Hz, J^2 = 5.68 Hz, 1H), 3.05 (dd, J^1 = 13.76 Hz, J^2 = 5.68 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 146.6, 136.6, 135.5, 133.7, 129.6, 129.5, 129.0, 128.6, 128.5, 127.0, 118.2, 113.8, 59.1, 38.8; HRMS (TOF MS ES^+) $C_{21}H_{19}NOH^+$ m/z (%) = 302.1518 ($[M+H]^+$, 100%).



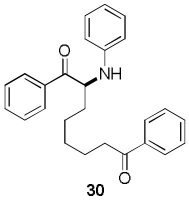
28

28: Compound **28** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (29 mg, 76%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.00-7.98 (m, 2H), 7.61 (tt, J^1 = 7.6 Hz, J^2 = 1.16 Hz, 1H), 7.53-7.49 (m, 2H), 7.20-7.15 (m, 2H), 6.77-7.72 (m, 3H), 5.79-5.68 (m, 1H), 5.17 (t, *J* = 5.56 Hz, 1H), 5.09-5.06 (m,1H), 5.02 (qd, J^1 = 16.96 Hz, J^2 = 1.32 Hz, 1H), 2.82-2.75 (m, 1H), 2.54-2.48 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 199.6, 146.3, 135.3, 133.8, 132.6, 129.5, 129.0, 128.5, 119.0, 118.6, 114.2, 58.0, 37.1.



29

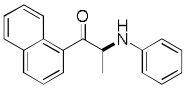
29: Compound **29** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (33 mg, 61%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$):δ = 8.00-7.98 (m, 2H), 7.90-7.87 (m, 2H), 7.60 (tt, , J^1 = 7.4 Hz, J^2 = 1.20 Hz, 1H), 7.56-7.48 (m, 3H), 7.45-7.41(m, 2H), 7.19-7.14 (m, 2H), 6.73-6.68 (m, 3H), 5.10(dd, J^1 = 6.6 Hz, J^2 = 4.68 Hz, 1H) 4.68 (s, 1H), 2.89 (td, J^1 = 8.20 Hz, J^2 = 1.76 Hz, 2H), 2.09-2.01(m, 1H), 1.78-1.63 (m, 3H), 1.60-1.39 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 200.7, 200.1, 147.2, 137.0, 135.3, 133.7, 133.1, 129.5, 129.0, 128.7, 128.5, 128.1, 118.1, 113.8, 57.9, 38.4, 33.1, 25.0, 24.1; HRMS (TOF MS ES$^+$) $C_{25}H_{24}NO_2H^+$ m/z (%) = 372.2196 ([M+H]$^+$, 100%).



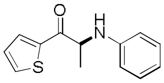
30

30: Compound **30** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→10% EtOAc : hexane) to provide pure compound (52 mg, 90%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$):δ = 8.00 (d, *J* = 7.23

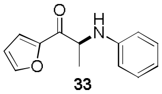
Hz, 2H), 7.91(d, *J* = 7.32 Hz, 2H), 7.62-7.58 (m, 1H), 7.56-7.48 (m, 3H), 7.45-7.42 (m, 2H), 7.18-7.14 (m, 2H), 6.72-6.68 (m, 3H), 5.09-5.07 (m, 1H), 4.67 (s, 1H), 2.89 (t, *J* = 7.20 Hz, 2H), 2.03-1.97 (m, 1H), 1.73-1.70 (m, 4H), 1.61-1.31 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.8, 200.3, 147.3, 137.1, 135.4, 133.7, 133.0, 129.5, 129.0, 128.7, 128.5, 128.1, 118.1, 113.7, 58.0, 38.4, 33.1, 29.2, 25.1, 24.1.


31

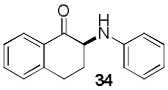
31: Compound **31** was prepared following the above general procedure for *α*-amination of ketones (36 h) and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (31 mg, 75%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.32 (d, *J* = 8.00 Hz, 1H), 7.94 (d, *J* = 8.04, 1H), 7.80 (t, *J* = 8.32 Hz, 2H), 7.52-7.42 (m , 3H), 7.16-7.11 (m, 2H), 6.66-6.65 (m, 3H), 5.02 (q, *J* = 6.52 Hz, 1H), 4.80 (brs, 1H), 1.34 (d, *J* = 6.72 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.4, 146.7, 134.5, 134.1, 133.0, 130.7, 129.6, 128.6, 128.2, 126.8, 125.6, 124.5, 118.8, 118.1, 113.7, 56.5, 19.0; HRMS (TOF MS ES$^+$) $C_{19}H_{17}NOH^+$ m/z (%) = 276.1396 ([M+H]$^+$, 100%).


32

32: Compound **32** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (30 mg, 86%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 3.2 Hz, 1H), 7.68 (d, *J* = 4.8, 1H), 7.19-7.15 (m, 3H), 6.72 (t, *J* = 7.32 Hz, 1H), 6.66 (d, *J* = 7.8 Hz, 2H), 4.87-4.85 (m, 1H), 4.51 (s, 1H), 1.55 (d, *J* = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 194.3, 146.6, 141.1, 134.5, 132.7, 129.5, 128.3, 118.3, 113.7, 55.5, 20.2; HRMS (TOF MS ES$^+$) $C_{13}H_{13}NOSH^+$ m/z (%) = 232.0879 ([M+H]$^+$, 100%).

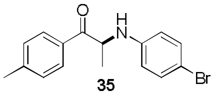

33

33: Compound **33** was prepared following general *α*-amination of ketones through aromatic nitroso aldol reaction protocol described above and purified by silica gel column chromatography (1→7% EtOAc : hexane) to provide pure compound (26 mg, 81%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63 (dd, J^1 = 1.64 Hz, J^2 = 0.64 Hz, 1H), 7.33 (dd, J^1 = 4.68 Hz, J^2 = 0.68 Hz,1H), 7.19-7.14 (m, 2H), 6.73-6.69 (m, 1H), 6.66 (dd J^1 = 8.64 Hz, J^2 = 1.00 Hz, 2H), 6.58 (dd J^1 = 3.60 Hz, J^2 = 1.68 Hz, 1H), 4.91 (q, J = 6.80 Hz, 1H), 4.55 (s, 1H), 1.50 (d, J = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.2, 151.2, 146.9, 146.6, 129.5, 118.4, 118.2, 113.7, 112.6, 54.2, 19.2; HRMS (TOF MS ES$^+$)$C_{13}H_{13}NOH^+$ m/z (%) = 216.1084 ($[M+H]^+$, 100%).



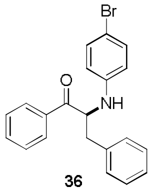
34

34:Compound **34** was prepared following general α-amination of ketones through aromatic nitroso aldol reaction protocol described above and purified by silica gel column chromatography (1-5% EtOAc : hexane) to provide pure compound (20 mg, 54%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.07 (dd, J^1 = 6.76 Hz , J^2 = 1.16 Hz 1H), 7.52 (td, J^1 = 7.52 Hz , J^2 = 1.44 Hz 2H), 7.35 (t, J = 7.60 Hz), 7.29 (d, J = 7.68 Hz 1H), 7.24-7.20 (m, 2H), 6.78-6.72 (m, 3H), 5.24 (brs, 1H), 4.20 (dd, J^1 = 13.40 Hz , J^2 = 4.36 Hz, 1H), 3.33-3.24 (m, 1H), 3.08(ddd, J^1 = 17.12 Hz , J^2 = 3.96 Hz, J^3 = 2.80 Hz 1H), 2.77-2.71 (m, 1H), 1.98 (qd, J^1 = 26.48 Hz , J^2 = 4.32 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 146.9, 143.7, 134.0, 131.8, 129.6, 129.0, 127.9, 127.0, 118.1, 112.3, 60.2, 31.1, 28.4; HRMS (TOF MS ES$^+$) $C_{16}H_{15}NOH^+$ m/z (%) = 230.1249 ($[M+H]^+$, 100%).



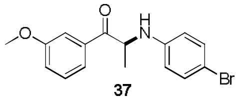
35

35: Compound **35** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→3% EtOAc : hexane) to provide pure compound (36 mg, 77%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81 (d, J = 8.24 Hz, 2H), 7.23 (d, J = 7.96 Hz, 2H), 7.19-7.17 (m, 2H), 6.54 (d, J = 8.4 Hz, 2H), 4.98 (q, J = 6.92 Hz, 1H) 2.36 (s, 3H), 1.40 (d, J = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.9, 145.7, 145.0, 132.2, 132.1, 129.8, 128.7, 115.1, 109.5, 53.3, 21.9, 19.7; HRMS (TOF MS ES$^+$) $C_{16}H_{16}BrNOH^+$ m/z (%) = 318.0488 ($[M+H]^+$, 100%).



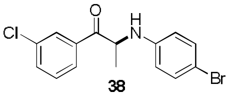
36

36: Compound **36** was prepared following the above general procedure for α-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (45 mg, 80%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$):δ = 7.94 (dd, , J^1 = 7.16 Hz, J^2 = 1.36 Hz, 2H), 7.60 (tt, J^1 = 7.40 Hz, J^2 = 1.16 Hz, 1H), 7.50 (t, *J* = 7.88 Hz, 2H), 7.24-7.17 (m, 5H), 7.02-6.99 (m, 2H), 6.49 (d, *J* = 8.44 Hz, 2H), 5.27-5.23 (m, 1H), 4.66 (d, *J* = 8.24 Hz, 1H), 3.27 (dd, J^1 = 13.84 Hz, J^2 = 5.48 Hz, 1H), 3.00 (dd, J^1 = 13.84 Hz, J^2 = 5.92 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.2, 145.7, 136.3, 135.3, 133.9, 132.2, 129.6, 129.1, 128.6(2×C), 127.1, 115.4, 109.8, 59.2, 38.7 ; HRMS (TOF MS ES^+)$C_{21}H_{18}NOH^+$ m/z (%) = 380.0660 ($[M+H]^+$, 100%).



37

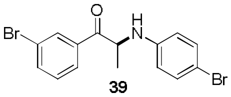
37: Compound **37** was prepared following the above general procedure for α-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (31 mg, 62%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57 (d, *J* = 7.68 Hz, 1H), 7.51-7.50 (m, 1H), 7.42 (t, *J* = 8.04 Hz, 1H), 7.26-7.22 (m, 2H), 7.16 (ddd, J^1 = 8.24 Hz, J^2 = 2.60 Hz, J^3 = 0.72 Hz, 1H), 6.54 (d, *J* = 8.84 Hz, 2H), 5.04 (q, *J* = 6.60 Hz, 1H), 4.72 (s, 1H), 3.87 (s, 3H), 1.46 (d, *J* = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.2, 160.2, 145.6, 136.0, 132.2, 130.0, 120.9, 120.3, 115.2, 113.1, 109.6, 55.7, 53.6, 19.6.



38

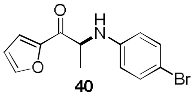
38: Compound **38** was prepared following the above general procedure for α-amination of ketones and purified by silica gel column chromatography (1→3% EtOAc : hexane) to provide pure compound (38 mg, 75%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96 (t, *J* = 1.72 Hz, 1H), 7.86 (d, *J* = 7.76 Hz, 1H) 7.59 (ddd, J^1 = 8.00 Hz, J^2 = 2.00 Hz, J^3 = 1.00 Hz, 1H), 7.45 (t, *J* = 7.84 Hz, 1H), 7.27-7.23 (m, 2H), 6.53 (d, *J* = 8.84 Hz, 2H), 5.02-4.89 (m, 1H), 4.68

(s, 1H), 1.46 (d, *J* = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 199.2, 145.4, 136.2, 135.5, 133.9, 132.3, 130.4, 128.7, 126.6, 115.2, 109.9, 53.7, 19.4; $C_{15}H_{13}ClBrNOH^+$ m/z (%) = 337.9942 ([M+H]$^+$, 100%).



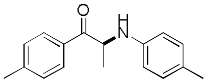
39

39: Compound **39** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→5% EtOAc : hexane) to provide pure compound (44 mg, 78%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.11 (t, *J* = 1.68 Hz, 1H), 7.90 (d, *J* = 7.80 Hz, 1H) 7.74 (ddd, J^1 = 8.00 Hz, J^2 = 2.00 Hz, J^3 = 1.00 Hz, 1H), 7.39 (t, *J* = 7.88 Hz, 1H), 7.24 (d, *J* = 8.84 Hz, 2H), 6.53 (d, *J* = 8.84 Hz, 2H), 5.00-4.99 (m, 1H), 4.68 (s, 1H), 1.45 (d, *J* = 6.92 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 199.1, 145.4, 136.8, 136.4, 132.3, 131.6, 130.6, 127.0, 123.4, 115.2, 109.8, 53.7, 19.4.



40

40: Compound **40** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→4% EtOAc : hexane) to provide pure compound (29 mg, 65%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$):δ = 7.64 (dd, J^1 = 1.56 Hz, J^2 = 0.60 Hz, 1H), 7.32 (dd, J^1 = 3.6 Hz, J^2 = 0.52 Hz,1H), 7.25-7.21 (m, 2H), 6.59 (dd, J^1 = 3.6 Hz, J^2 = 1.72 Hz, 1H), 6.54-6.50 (m, 2H), 4.88-4.82 (m, 1H), 4.58 (s, 1H), 1.49 (d, *J* = 6.88 Hz, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 189.7, 151.2, 147.0, 145.6, 132.2, 118.5, 115.2, 112.8, 109.8, 54.2, 19.2; HRMS (TOF MS ES$^+$) $C_{13}H_{12}BrNO_2H^+$ m/z (%) = 294.0327 ([M+H]$^+$, 100%).



41

41: Compound **41** was prepared following the above general procedure for *α*-amination of ketones and purified by silica gel column chromatography (1→3% EtOAc : hexane) to provide pure compound (27 mg, 71%) as white solid. ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.92 (d, *J* = 8.16 Hz, 2H), 7.30 (d, *J* = 8.00 Hz, 2H), 6.98 (d, *J* = 8.12 Hz, 2H), 6.60 (d, *J* = 8.24 Hz, 2H), 5.08 (q, *J* = 6.84 Hz, 1H), 2.43 (s, 3H), 2.23 (s, 3H), 1.46 (d, *J* = 6.88 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.7, 144.6, 144.5, 132.4, 130.0, 129.7, 128.7, 127.2, 113.9, 53.7, 21.8, 20.5, 19.8.

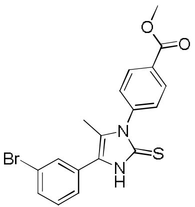
Gram scale synthesis of compounds 6 and 15:

Nitroso compound (1.2 equiv.) was taken in a 250 ml oven dried round bottom flask equipped with a magnetic stir. The flask was capped with a septum and kept under vacuum for 10 min and then purged with nitrogen. Dry CH_3CN (80 mL) and corresponding silyl enol ether (6.1 mmol, 1.7 g, 1.0 equiv.) were added and the mixture was allowed to stir at room temperature until sillyl enol ether was consumed. Afterward, phenylboronic acid (3.0 equiv.) was added in one portion and stirring was continued for additional 24 h at room temperature. The solvent was evaporated to dryness. The crude reaction mixture was loaded directly onto silica gel column and purified to provide pure nitroso aldol products **6** (2.01 g, 91%) or **15** (1.34 g, 92%). Details of NMR-spectra were described in the preceding paragraph.

Synthesis of fully substituted imidazoles:

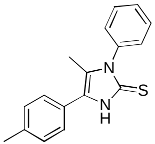
STEP-I: Nitroso aldol product **6** or **15** (0.15 mmol, 1 equiv.), p-TSOH (5 equiv.), and NH_4SCN (5 equiv.) were taken into a schlenk tube under nitrogen and the mixture was heated at 80 ^{o}C for 10 min. After cooling to room temperature, the reaction was quenched with water, extracted with ethyl acetate, dried over $MgSO_4$ and concentrated under reduced pressure to give yellow solid. The crude product was further purified by recrystallization from ethanol to give pure compound **43** (53 mg, 87% yield) or **44** (36 mg, 85% yield) as pale yellow solid.

STEP-II: The recrystallized product **43** or **44** (0.15 mmol, 1 equiv.) and 2-bromo acetophenone (1 equiv.) were taken into a schlenk tube. EtOH (3 mL) and Et_3N (3 drops) were added and the reaction mixture was allowed to stir at 80 ^{o}C for 6 h. The solvent was evaporated to dryness. The crude reaction mixture was loaded directly onto silica gel column and purified (1→15% EtOAc : hexane) to provide fully substituted imidazoles **45** (66 mg, 85% yield) or **46** (48 mg, 82% yield) as pale yellow solid.



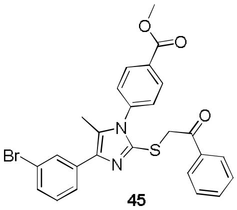
43

43: ^{1}H NMR (400 MHz, CDCl$_3$): δ = 12.02 (s, 1H), 8.15 (d, *J* = 6.8 Hz, 2H), 7.52 (t, *J* = 1.36 Hz, 1H), 7.40 (d, *J* = 6.76 Hz, 2H), 7.38-7.33 (m, 2H), 7.20 (t, *J* = 6.32 Hz, 1H), 3.89 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 166.2, 162.0, 139.8, 131.3, 131.1 (2×C), 130.8, 130.3, 130.1, 128.7, 125.9, 124.7, 123.7, 123.1, 52.6, 11.2.



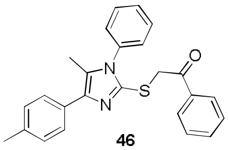
44

44: ^{1}H NMR (400 MHz, CDCl$_3$): δ = 12.01(s, 1H), 7.56-7.51 (m, 2H), 7.50-7.46 (m, 1H), 7.38-7.33 (m, 4H), 7.19 (d, *J* = 7.92 Hz, 2H), 2.33 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 161.0, 138.0, 136.3, 129.8, 129.6, 129.3, 128.5, 127.1, 125.7, 122.8, 21.3, 11.1; HRMS (TOF MS ES$^+$) $C_{17}H_{16}N_2SNa^+$ m/z (%) = 303.0938 ([M+Na]$^+$, 100%).



45

45: ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.21 (d, *J* = 8.52 Hz, 2H), 8.03-8.01 (m, 2H), 7.84 (t, *J* = 1.72 Hz, 1H), 7.64-7.60 (m, 1H), 7.58-7.56 (m, 1H), 7.52-7.48 (m, 2H), 7.43-7.37 (m, 3H), 7.29-7.27 (m, 1H) 4.68 (s, 2H), 4.00 (s, 3H), 2.24 (s, 3H);^{13}C NMR (100 MHz, CDCl$_3$): δ = 194.2, 166.1, 140.7, 139.3, 137.5, 136.6, 135.7, 133.8, 131.1, 130.0(2×C), 129.6, 128.8(3×C), 128.0, 127.0, 125.5, 122.8, 52.6, 41.0, 11.4.

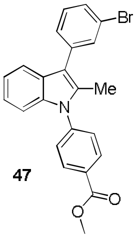


46

46: ^{1}H NMR (400 MHz, CDCl$_3$): δ = 7.99-7.97 (m, 2H), 7.59-7.54 (m, 3H), 7.50-7.42 (m, 5H), 7.24-7.20 (m, 4H), 4.64 (s, 2H), 2.37 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$): δ = 194.5, 139.9, 138.5, 136.2, 135.8, 135.7, 133.6, 132.0, 129.6, 129.2 (2×C), 128.8, 128.7, 127.9, 127.0, 126.4, 41.2, 21.3, 11.3; HRMS (TOF MS ES$^+$) $C_{25}H_{22}N_2OH^+$ m/z (%) = 399.1526 ([M+H]$^+$, 100%).

Synthesis of substituted indoles:

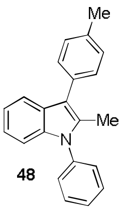
To a solution of aldol product **6** or **15** (0.15 mmol, 1.0 equiv.) and 18-crown-6 (0.17 mmol, 1.1 equiv.) in 1.5 ml CH_3CN /DCE (1/4, v/v) mixture was added silyl aryl triflate (0.21 mmol, 1.4 equiv.) under nitrogen. Then, anhydrous CsF (0.45 mmol, 3.0 equiv.) and anhydrous Cs_2CO_3 (0.30 mmol, 2.0 equiv.) were added. The resulting reaction mixture was stirred at room temperature until complete consumption of the aldol product (6 h). After completion, ethyl acetate and water were added and the aqueous phase was extracted with ethyl acetate. The organic layer was washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. The crude-residue was re-dissolved in a mixture of MeOH/DCM/AcOH (1.5 ml, 1:1:1) and silica gel was added to get a slurry. The mixture was heated at 40 °C for 4 h (TLC monitored). After cooling, the crude mixture was loaded directly onto silica gel column and purified (1→2% EtOAc : hexane) to provide fully substituted indoles **47** (40 mg, 64% yield) or **48** (31 mg, 70% yield) as yellow waxy solid.



47

47: ^{1}H NMR (400 MHz, CDCl$_3$): δ = 8.26 (d, *J* =8.28 Hz, 2H), 7.71-7.67 (m, 2H), 7.51-7.46 (m, 4H), 7.36 (t, *J* = 7.80 Hz, 1H), 7.20-7.18 (m, 3H), 3.99 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100

MHz, $CDCl_3$): δ = 166.5, 141.9, 137.4, 137.2, 133.5, 132.5, 131.1, 130.2, 129.2, 128.4, 127.9, 127.4, 122.7, 122.5, 121.2, 120.6, 118.8, 115.2, 110.2, 52.5, 12.3.



48

48: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.75-7.73 (m, 1H), 7.58 (t, *J* = 7.32 Hz, 2H), 7.51-7.43 (m, 5H), 7.34 (d, *J* = 7.76 Hz, 2H), 7.18-7.15 (m, 3H), 2.46 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.1, 137.6, 135.6, 133.5, 132.5, 129.7, 129.6, 129.4, 128.3, 127.9, 127.5, 121.7, 120.4, 118.9, 115.3, 110.2, 21.4, 12.2; HRMS (TOF MS ES^+)$C_{22}H_{19}NH^+$ m/z (%) = 298.1592 ($[M+H]^+$, 100%).

Crystallographic experimental section:

Crystal structure of compound **4 (CCDC number: 1440804)**:

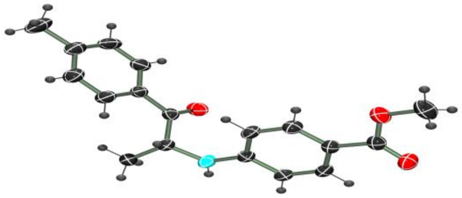


Table 1. Crystal data and structure refinement for **4**.

Identification code	4	
Empirical formula	C18 H19 N O3	
Formula weight	297.34	
Temperature	296(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Monoclinic, P2(1)/n	
Unit cell dimensions	a = 9.6614(2) A	alpha = 90 deg.
	b = 9.0050(2) A	beta = 91.9684(13) deg.
	c = 18.1004(4) A	gamma = 90 deg.
Volume	1573.82(6) A^3	
Z, Calculated density	4, 1.255 Mg/m^3	
Absorption coefficient	0.085 mm^-1	

F(000)	632
Crystal size	0.280 x 0.220 x 0.160 mm
Theta range for data collection	2.252 to 27.150 deg.
Limiting indices	-12<=h<=12, -11<=k<=11, -17<=l<=23
Reflections collected / unique	12074 / 3472 [R(int) = 0.0196]
Completeness to theta	= 25.242 99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3472 / 0 / 207
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0408, wR2 = 0.1077
R indices (all data)	R1 = 0.0559, wR2 = 0.1188
Extinction coefficient	0.0107(17)
Largest diff. peak and hole	0.185 and -0.144 e.A^-3

Crystal structure of compound **18 (CCDC number: 1440807)**:

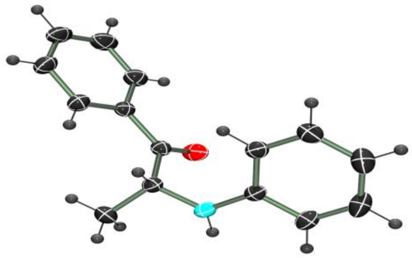


Table 2. Crystal data and structure refinement for **18**.

Identification code	17
Empirical formula	C15 H15 N O
Formula weight	225.28
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 8.9551(4) A alpha = 90 deg.
	b = 9.4163(6) A beta = 95.037(2) deg.
	c = 14.8711(9) A gamma = 90 deg.
Volume	1249.15(12) A^3
Z, Calculated density	4, 1.198 Mg/m^3

Absorption coefficient	0.075 mm^-1
F(000)	480
Crystal size	0.250 x 0.160 x 0.100 mm
Theta range for data collection	2.560 to 24.999 deg.
Limiting indices	-10<=h<=8, -10<=k<=11, -17<=l<=17
Reflections collected / unique	9305 / 2188 [R(int) = 0.0497]
Completeness to theta	= 24.999 99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2188 / 0 / 160
Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0394, wR2 = 0.0992
R indices (all data)	R1 = 0.0624, wR2 = 0.1100
Extinction coefficient	0.014(3)
Largest diff. peak and hole	0.118 and -0.133 e.A^-3

Crystal structure of compound **35 (CCDC number: 1440806):**

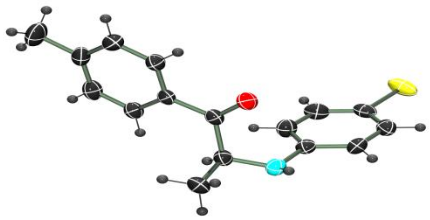


Table 3. Crystal data and structure refinement for **35**.

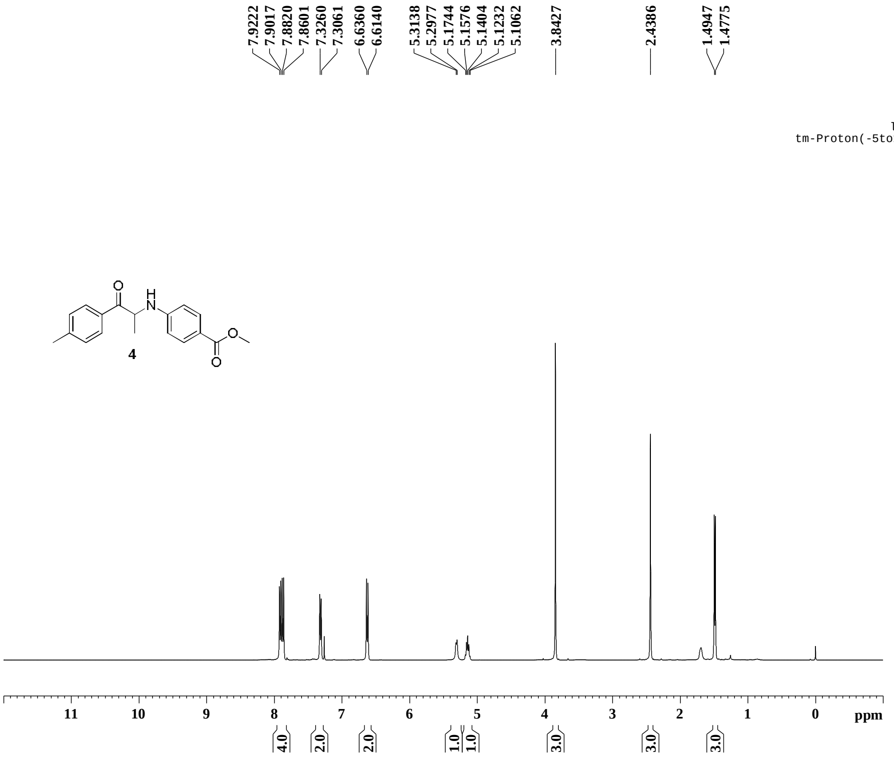
Identification code	33
Empirical formula	C16 H16 Br N O
Formula weight	318.21
Temperature	296(2) K
Wavelength	0.71073 A

Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 12.4037(4) A alpha = 90 deg.
	b = 9.4552(3) A beta = 100.5942(17) deg.
	c = 13.0281(5) A gamma = 90 deg.
Volume	1501.88(9) A^3
Z, Calculated density	4, 1.407 Mg/m^3
Absorption coefficient	2.729 mm^-1
F(000)	648
Crystal size	0.350 x 0.250 x 0.250 mm
Theta range for data collection	2.084 to 24.999 deg.
Limiting indices	-13<=h<=14, -8<=k<=11, -15<=l<=14
Reflections collected / unique	10970 / 2651 [R(int) = 0.0237]
Completeness to theta = 24.999	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2651 / 0 / 178
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	R1 = 0.0420, wR2 = 0.0952
R indices (all data)	R1 = 0.0663, wR2 = 0.1063
Extinction coefficient	n/a
Largest diff. peak and hole	0.631 and -0.912 e.A^-3

Crystal structure of compound **44 (CCDC number: 1440805):**

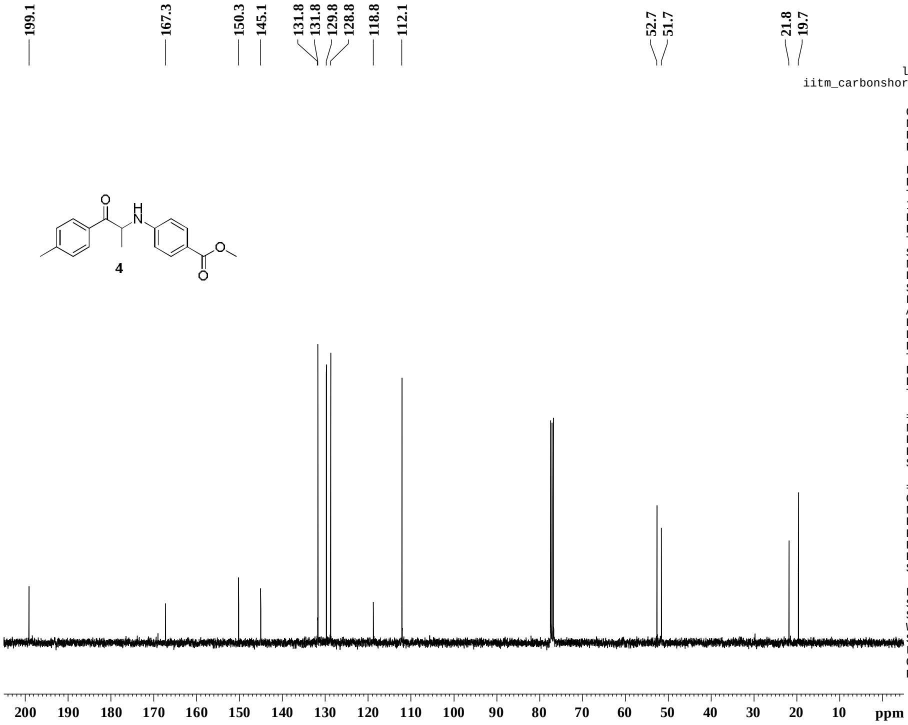
Table 4. Crystal data and structure refinement for **44**.

Identification code	327
Empirical formula	C17 H16 N2 S
Formula weight	280.38
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 9.0979(2) A alpha = 90 deg.
	b = 9.8360(2) A beta = 102.3206(11) deg.
	c = 17.0069(4) A gamma = 90 deg.
Volume	1486.84(6) A^3
Z, Calculated density	4, 1.253 Mg/m^3
Absorption coefficient	0.209 mm^-1
F(000)	592
Crystal size	0.250 x 0.250 x 0.200 mm
Theta range for data collection	2.291 to 24.998 deg.
Limiting indices	-9<=h<=10, -11<=k<=11, -19<=l<=20
Reflections collected / unique	11081 / 2614 [R(int) = 0.0175]
Completeness to theta	= 24.998 100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2614 / 0 / 187
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0343, wR2 = 0.0897
R indices (all data)	R1 = 0.0418, wR2 = 0.0969
Extinction coefficient	n/a
Largest diff. peak and hole	0.187 and -0.194 e.A^-3



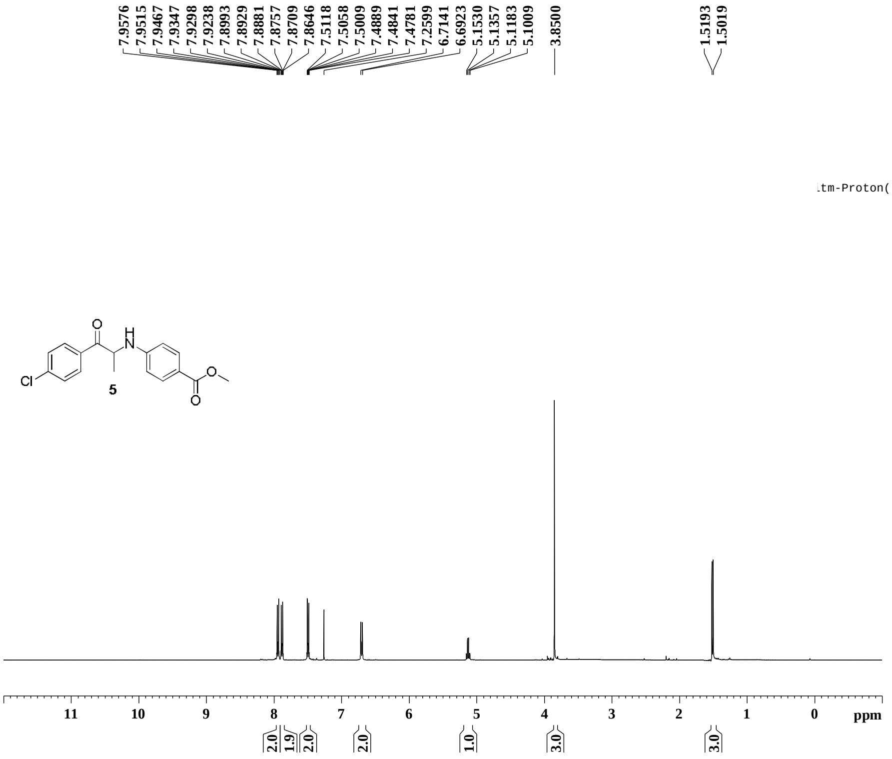
```
        lab mmbram-276
tm-Proton(-5to15) CDCl3 /opt/topspin nmr
```

```
Current Data Parameters
NAME           mmb40915
EXPNO               182
PROCNO                1

F2 - Acquisition Parameters
Date_          20150928
Time              22.38
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               124.58
DW               62.400 use
DE                 6.50 use
TE                298.0 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300094 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



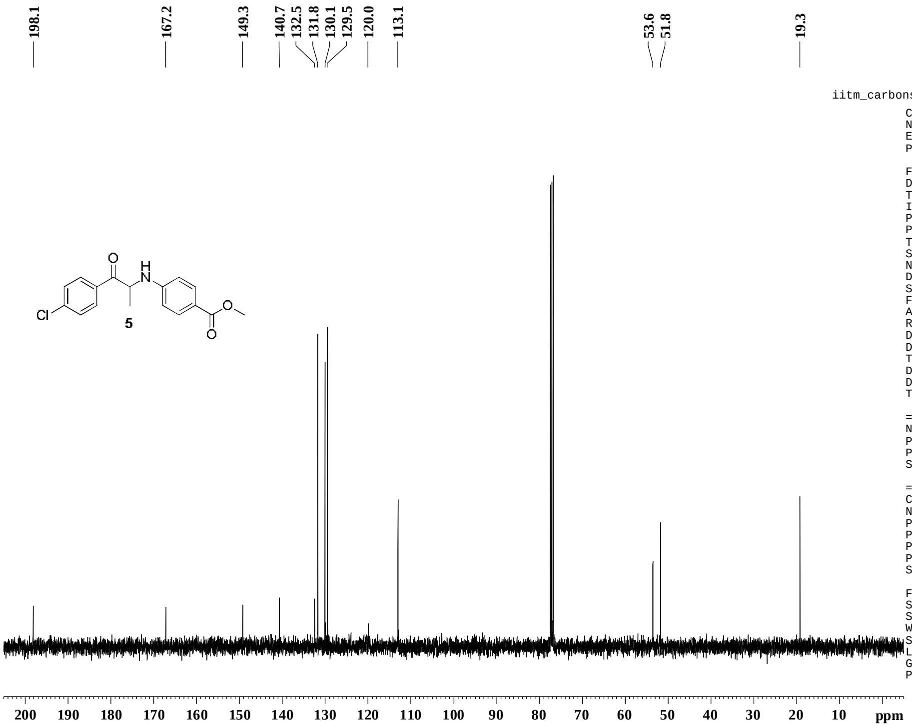
```
        lab mmbram-276
iitm_carbonshort CDCl3 /opt/topspin nmr 15
```

```
Current Data Parameters
NAME           mmb40915
EXPNO               183
PROCNO                1

F2 - Acquisition Parameters
Date_          20150928
Time              22.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                298.8 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127574 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



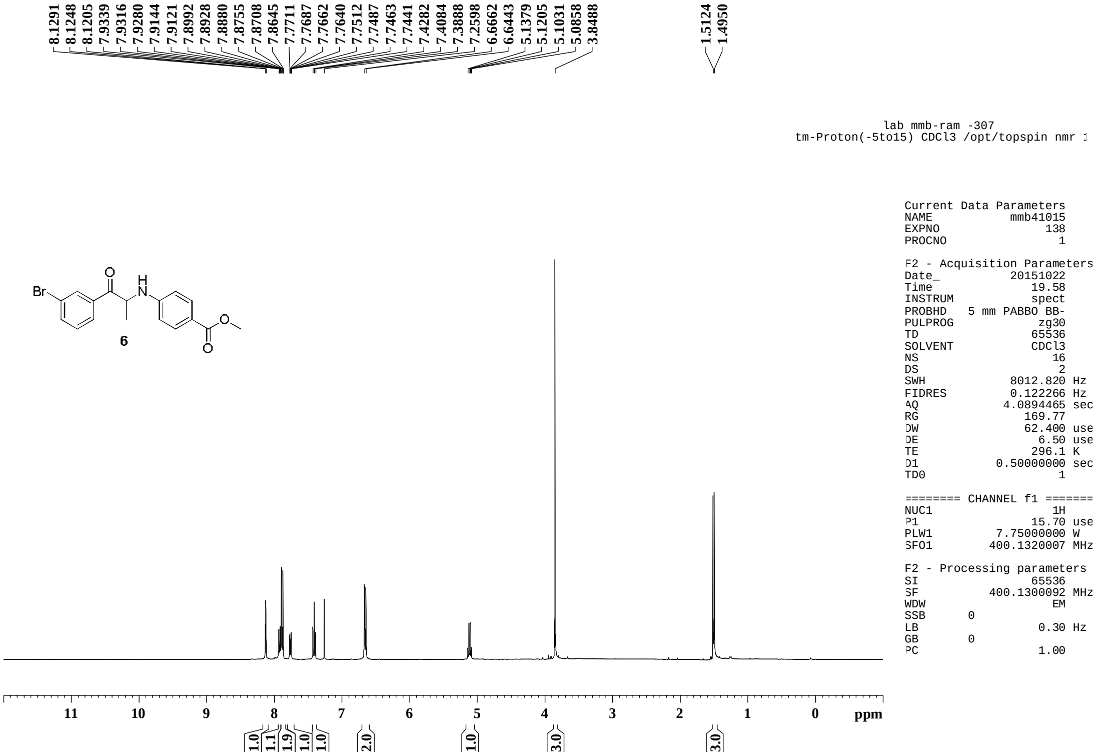
5

```
                lab mmb-ram-309
tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                178
PROCNO                 1

=2 - Acquisition Parameters
Date_           20151027
Time               20.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                200.34
DW                62.400 use
DE                  6.50 use
TE                 296.2 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300095 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

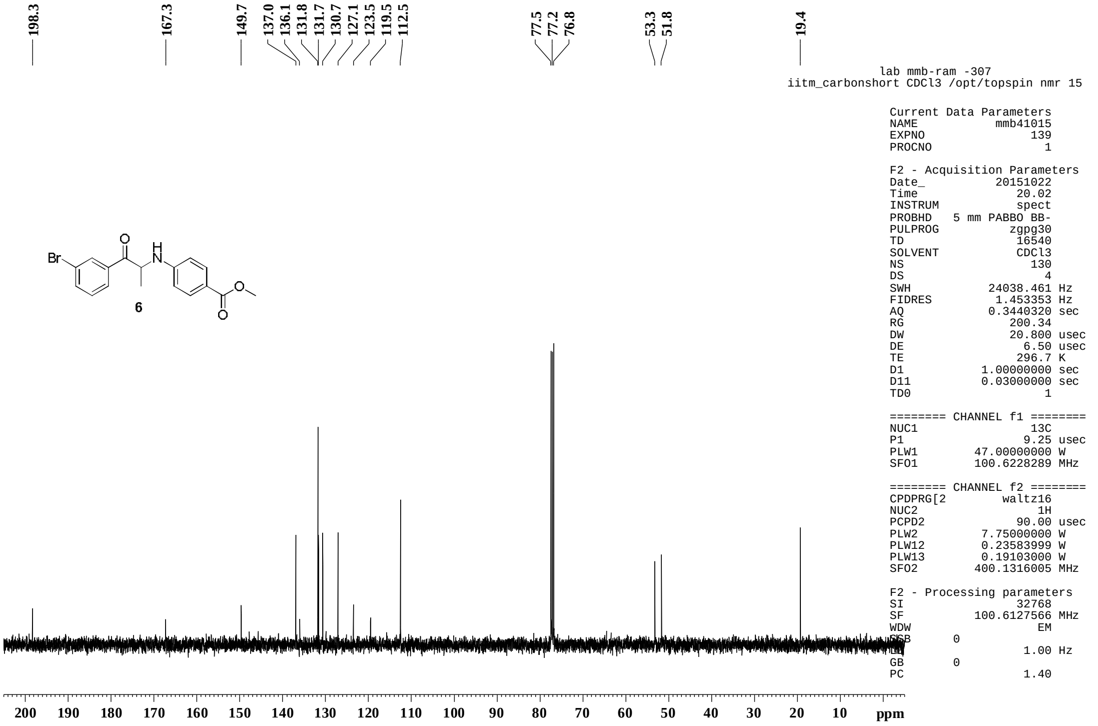


5

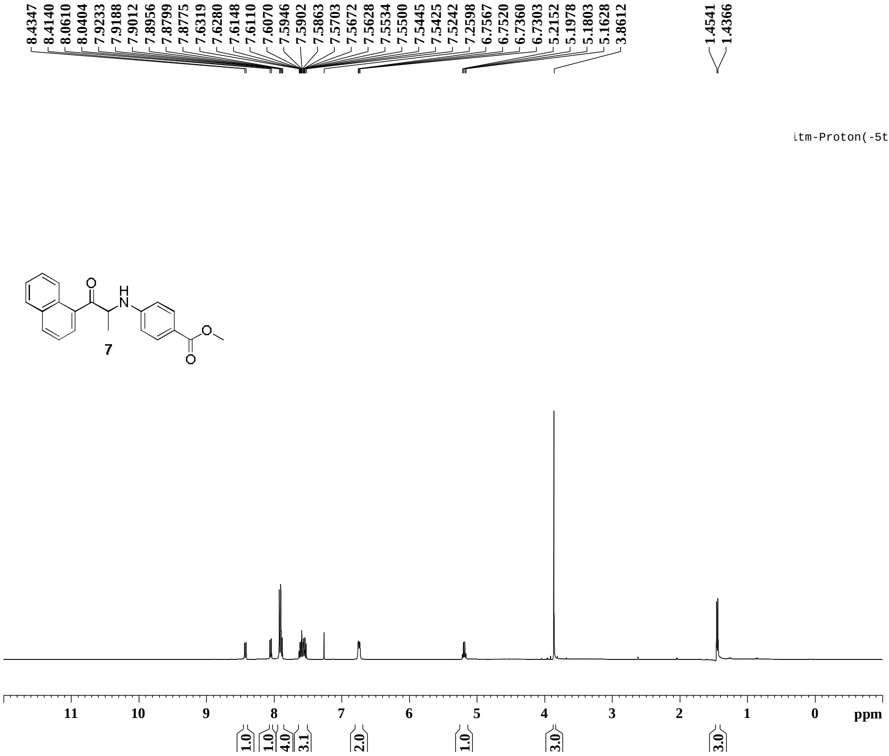
```
                  lab mmb-ram-309
iitm_carbonshort CDCl3 /opt/topspin nmr 9

Current Data Parameters
NAME            mmb41015
EXPNO                179
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151027
Time               20.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 297.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127560 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



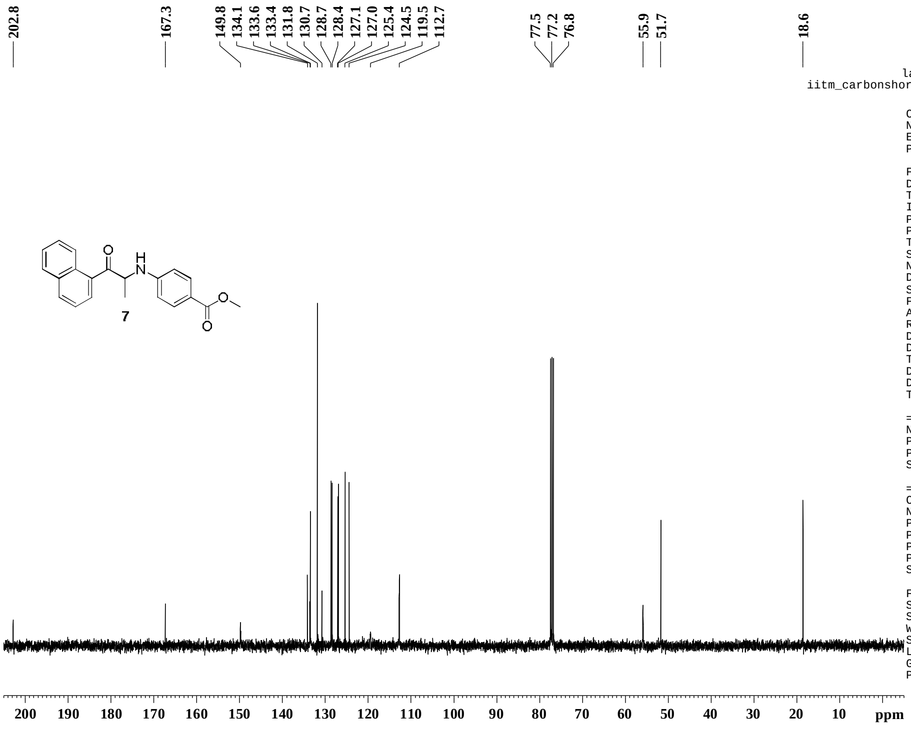
```
lab mmb-ram -307
tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                138
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151022
Time               19.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                169.77
DW                62.400 use
DE                  6.50 use
TE                 296.1 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300092 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



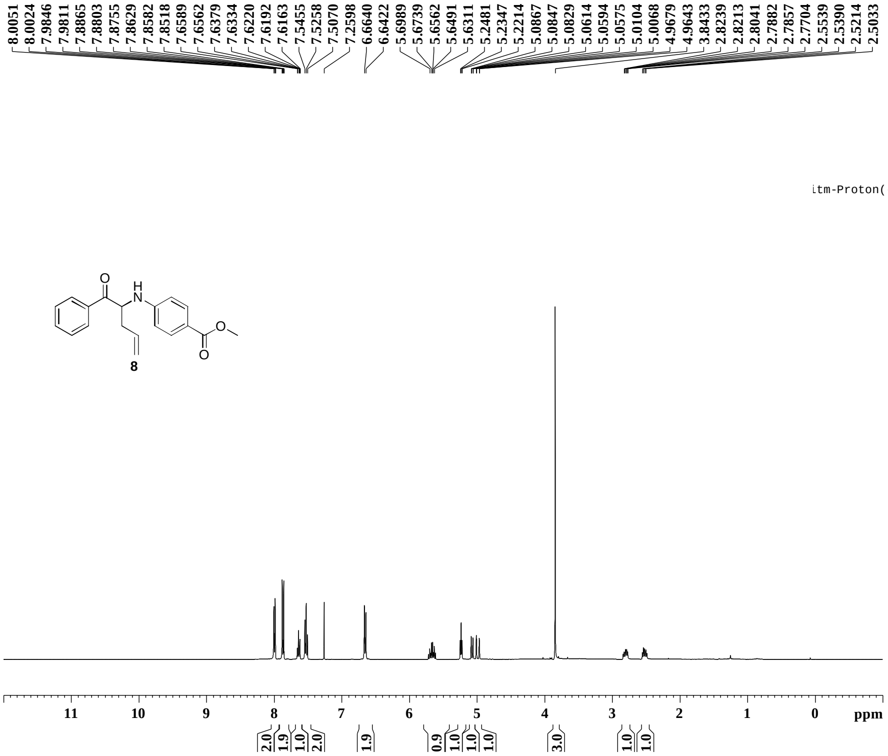
```
lab mmb-ram -307
iitm_carbonshort CDCl3 /opt/topspin nmr 15

Current Data Parameters
NAME            mmb41015
EXPNO                139
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151022
Time               20.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   130
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 296.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127566 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



```
          lab mmbram-308
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                159
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151026
Time               11.24
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                124.58
DW                62.400 use
DE                  6.50 use
TE                 294.9 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300094 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

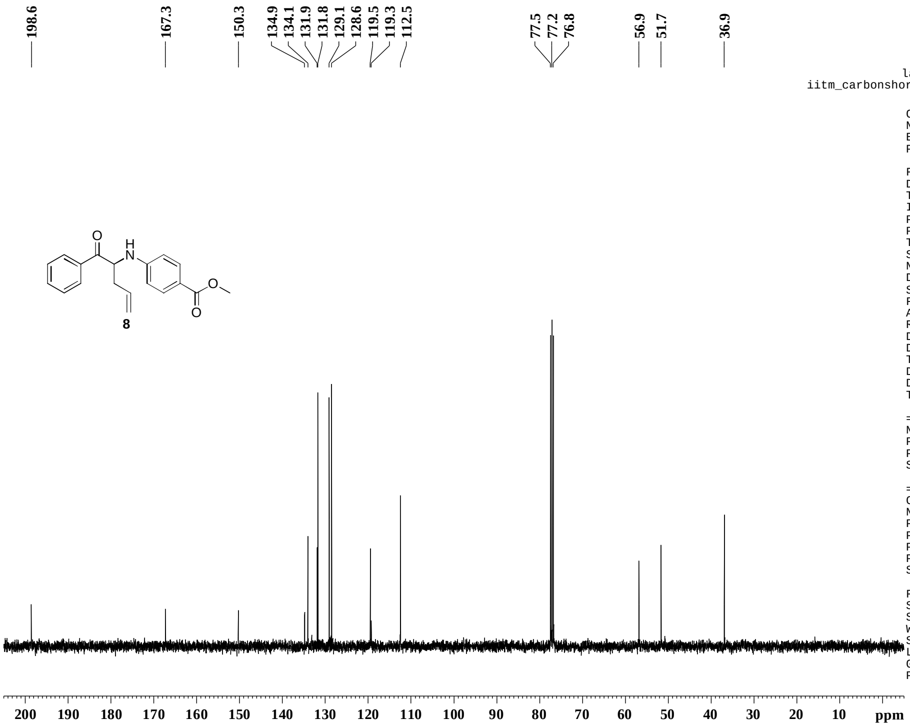


```
           lab mmbram-308
iitm_carbonshort CDCl3 /opt/topspin nmr 4

Current Data Parameters
NAME            mmb41015
EXPNO                160
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151026
Time               11.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 295.7 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127586 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



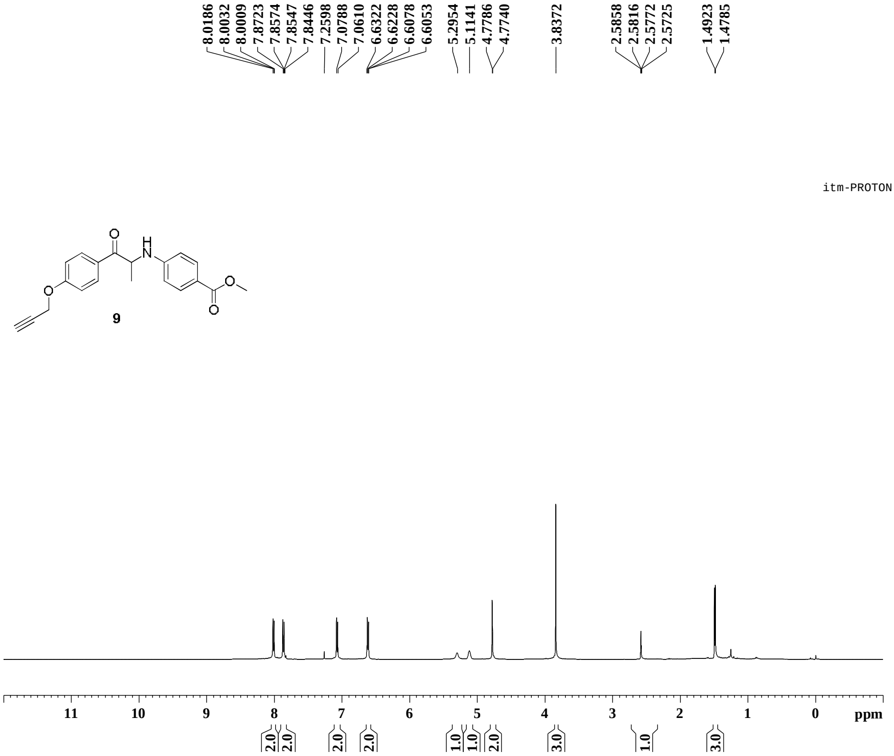
8

```
 lab mmbram-285
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                110
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151020
Time               14.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                153.13
DW                62.400 use
DE                  6.50 use
TE                 295.0 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300093 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



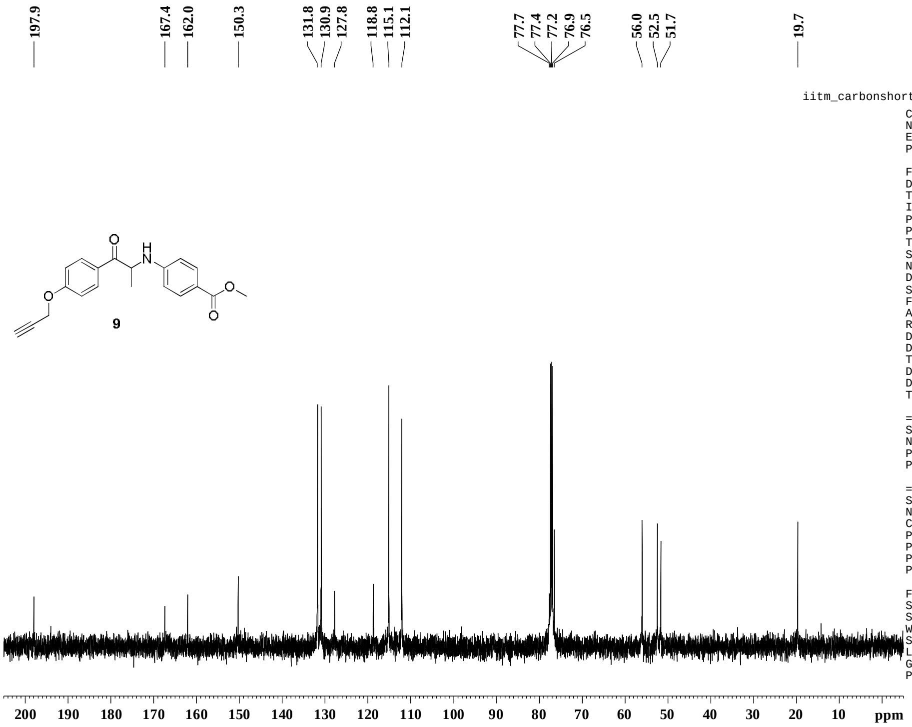
8

```
 lab mmbram-285
iitm_carbonshort CDCl3 /opt/topspin nmr 6

Current Data Parameters
NAME            mmb41015
EXPNO                111
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151020
Time               14.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 295.9 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127570 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



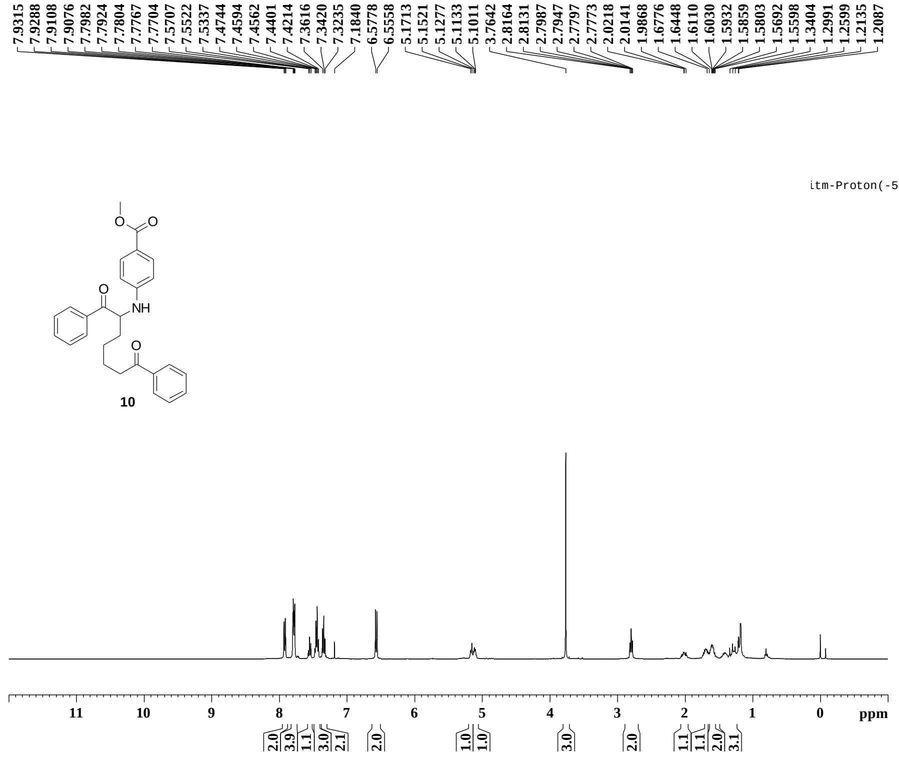
9

```
          Lab mmb
          ram-317
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 9

Current Data Parameters
NAME            mmb51115
EXPNO                 43
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151105
Time               18.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             1.6384000 sec
RG                 79.04
DW                50.000 use
DE                  6.50 use
TE                 297.1 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
SFO1         500.1525008 MHz
NUC1                  1H
P1                 11.75 use
PLW1         15.30000019 W

F2 - Processing parameters
SI                 65536
SF           500.1500230 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

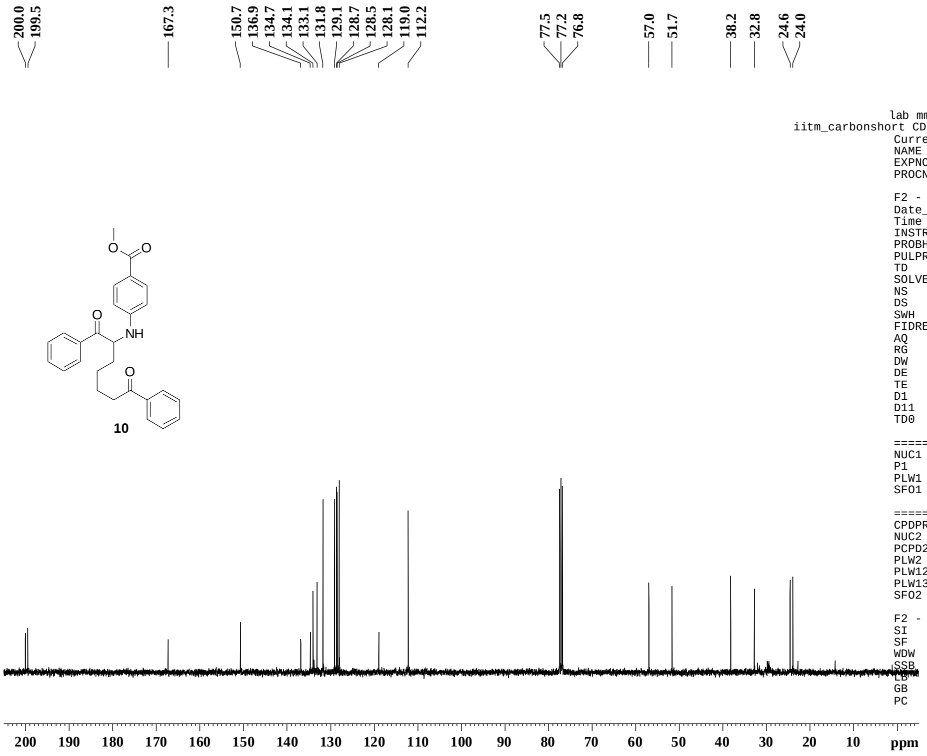


9

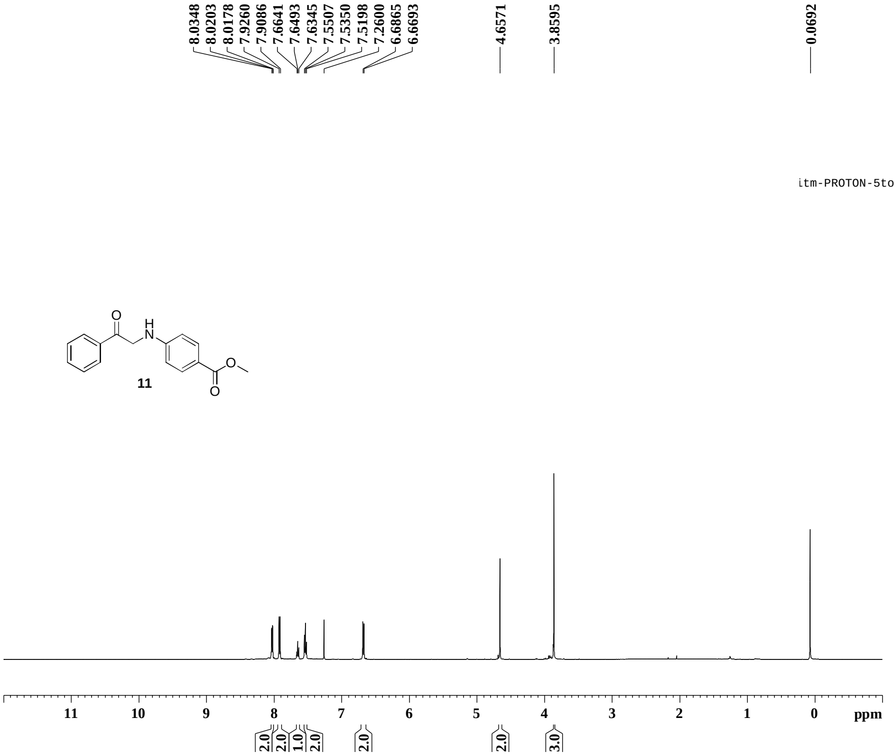
```
              Lab mmb
              ram-317
iitm_carbonshort CDCl3 /opt/topspin iitm 4

Current Data Parameters
NAME            mmb51115
EXPNO                 28
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151104
Time               19.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 20480
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            29761.904 Hz
FIDRES          1.453218 Hz
AQ             0.3440640 sec
RG                202.34
DW                16.800 usec
DE                  6.50 usec
TE                 298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7753932 MHz
NUC1                 13C
P1                  9.63 usec
PLW1        103.00000000 W

======== CHANNEL f2 ========
SFO2         500.1520006 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         15.30000019 W
PLW12         0.33006001 W
PLW13         0.21123999 W

F2 - Processing parameters
SI                 32768
SF           125.7628083 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



```
                lab mmb-ram-340
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME           mmb41115
EXPNO               225
PROCNO                1

F2 - Acquisition Parameters
Date_          20151130
Time              15.56
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                95.73
DW               62.400 use
DE                 6.50 use
TE                292.6 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300397 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



```
                 lab mmb-ram-340
iitm_carbonshort CDCl3 /opt/topspin nmr 13
Current Data Parameters
NAME           mmb41115
EXPNO               228
PROCNO                1

F2 - Acquisition Parameters
Date_          20151130
Time              16.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                292.9 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127606 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



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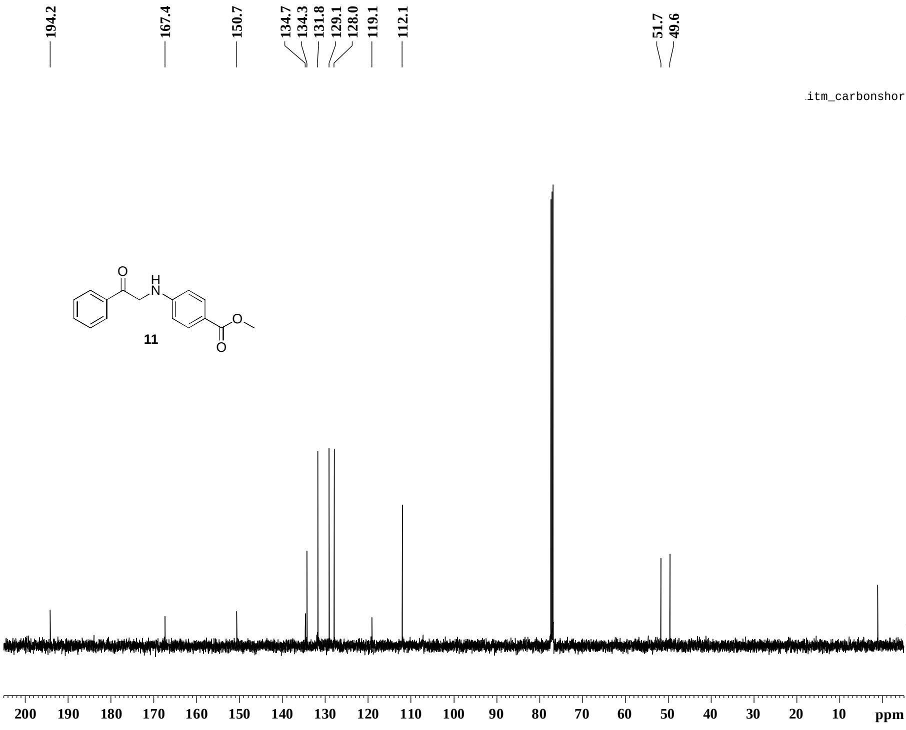
```
      Lab mmb
      ram-301
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 1

Current Data Parameters
NAME           mmb51015
EXPNO                21
PROCNO                1

F2 - Acquisition Parameters
Date_          20151016
Time              15.39
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            1.6384000 sec
RG               138.53
DW               50.000 use
DE                 6.50 use
TE                296.7 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
SFO1       500.1525008 MHz
NUC1                 1H
P1                11.75 use
PLW1       15.30000019 W

F2 - Processing parameters
SI                65536
SF          500.1500221 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



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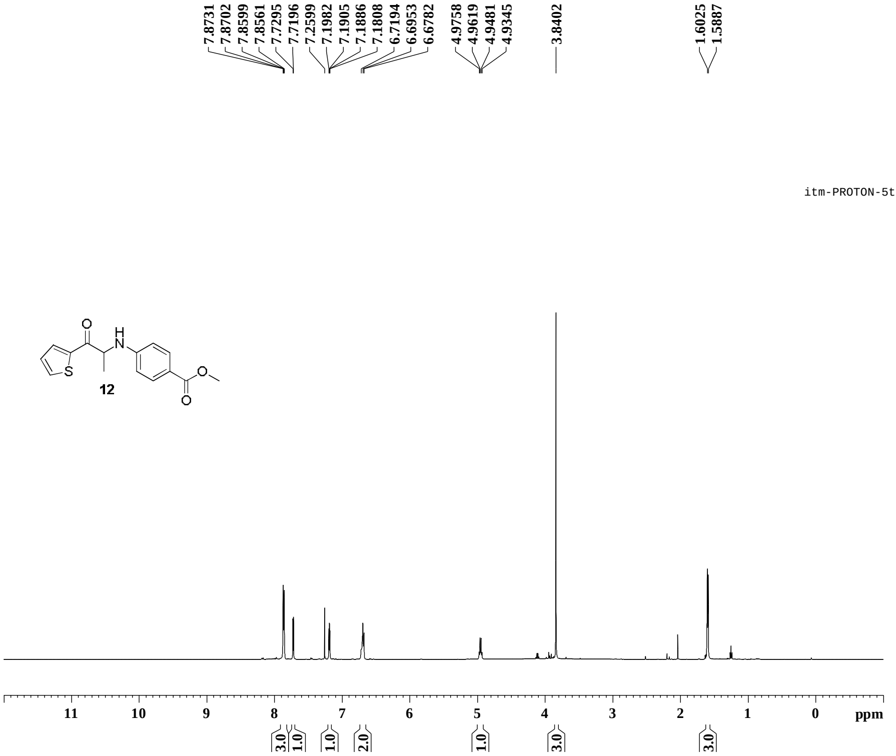
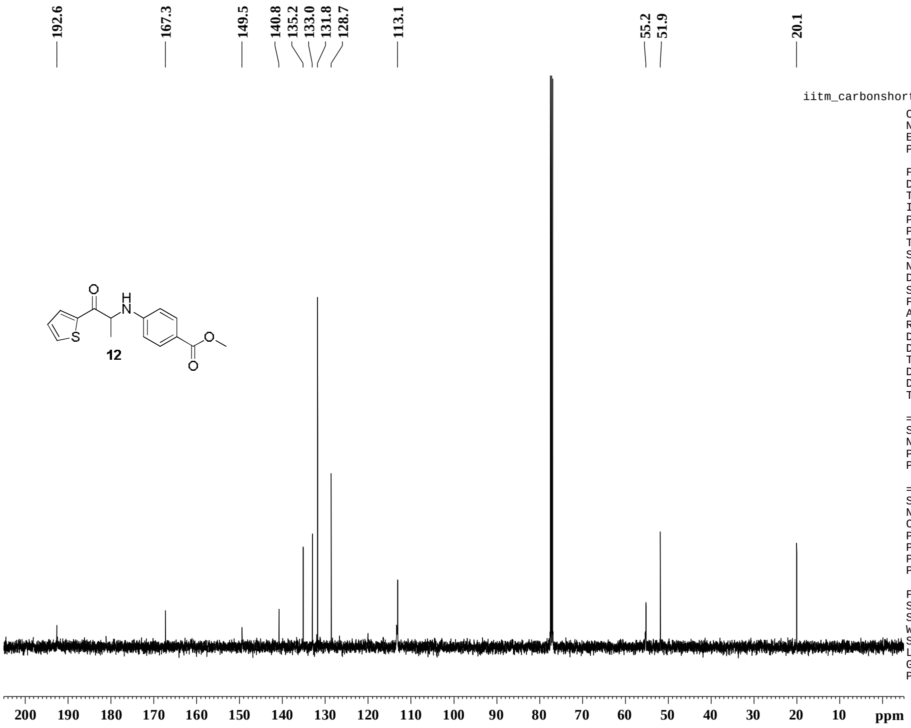
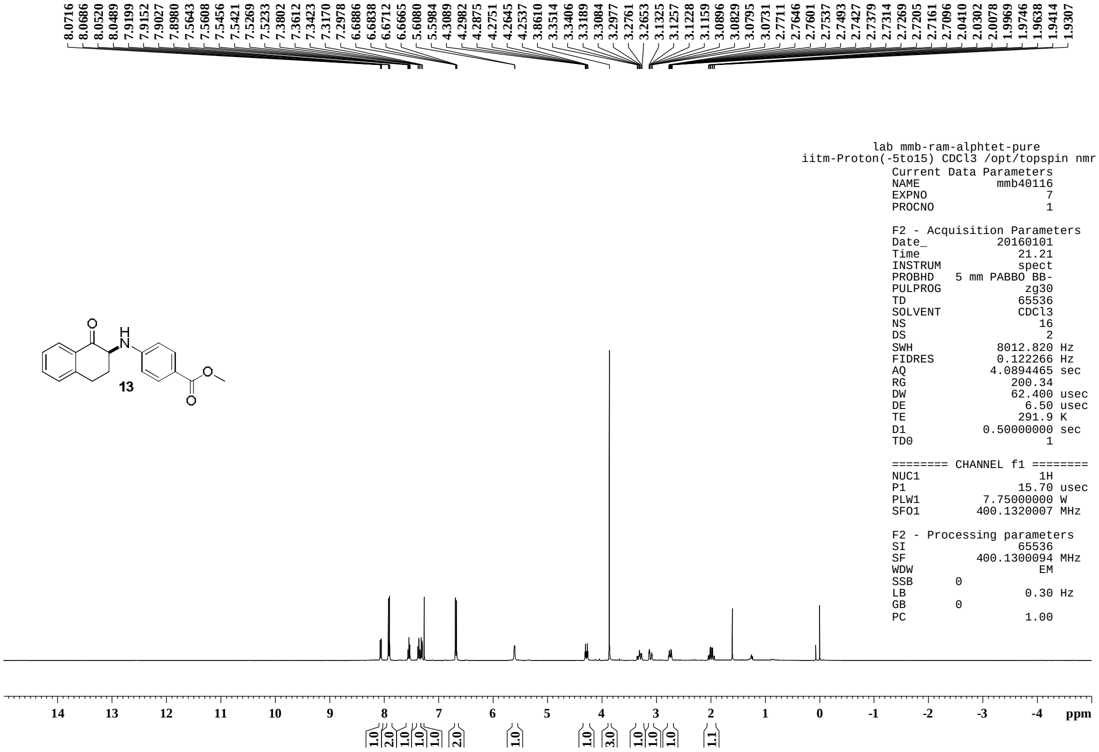
```
      Lab mmb
      ram-301
itm_carbonshort CDCl3 /opt/topspin iitm 11

Current Data Parameters
NAME           mmb51015
EXPNO                22
PROCNO                1

F2 - Acquisition Parameters
Date_          20151016
Time              15.46
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                20480
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           29761.904 Hz
FIDRES         1.453218 Hz
AQ            0.3440640 sec
RG               202.34
DW               16.800 usec
DE                 6.50 usec
TE                297.3 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1       125.7753932 MHz
NUC1                13C
P1                 9.63 usec
PLW1      103.00000000 W

======== CHANNEL f2 ========
SFO2       500.1520006 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        15.30000019 W
PLW12        0.33006001 W
PLW13        0.21123999 W

F2 - Processing parameters
SI                32768
SF          125.7628043 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



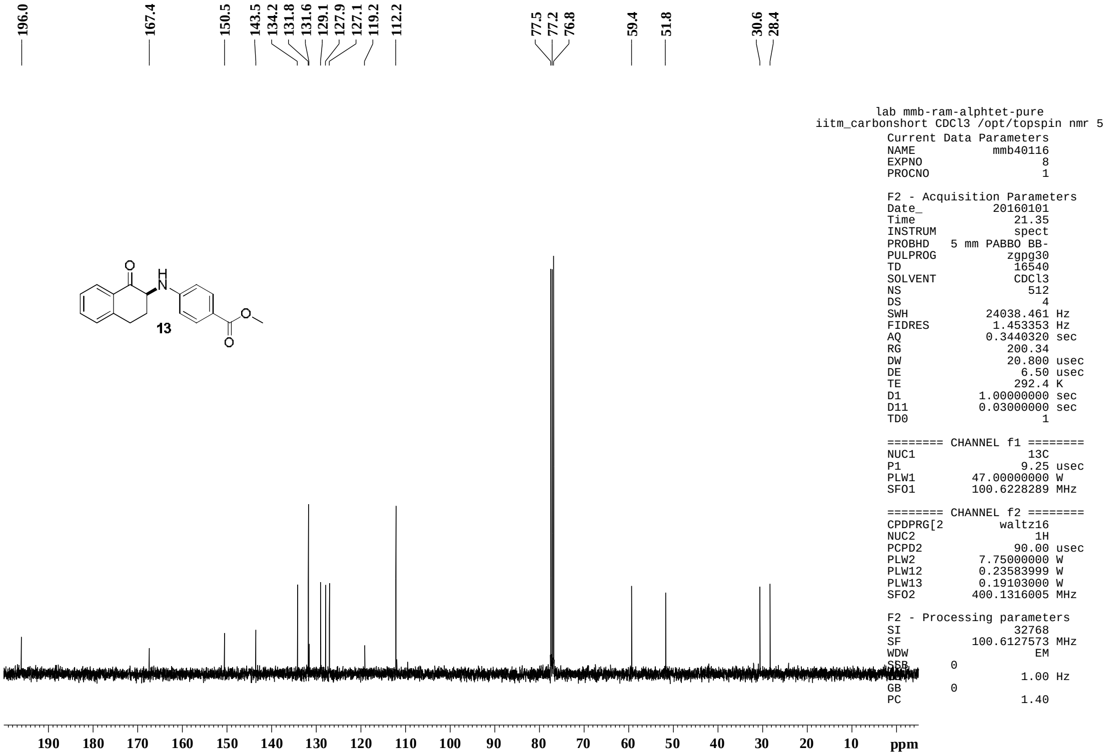
```
   Lab mmb
   ram-302
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 1
Current Data Parameters
NAME          mmb51015
EXPNO               24
PROCNO               1

F2 - Acquisition Parameters
Date_         20151016
Time             20.31
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ           1.6384000 sec
RG              114.76
DW              50.000 use
DE                6.50 use
TE               294.7 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
SFO1       500.1525008 MHz
NUC1                1H
P1               11.75 use
PLW1       15.30000019 W

F2 - Processing parameters
SI               65536
SF         500.1500221 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



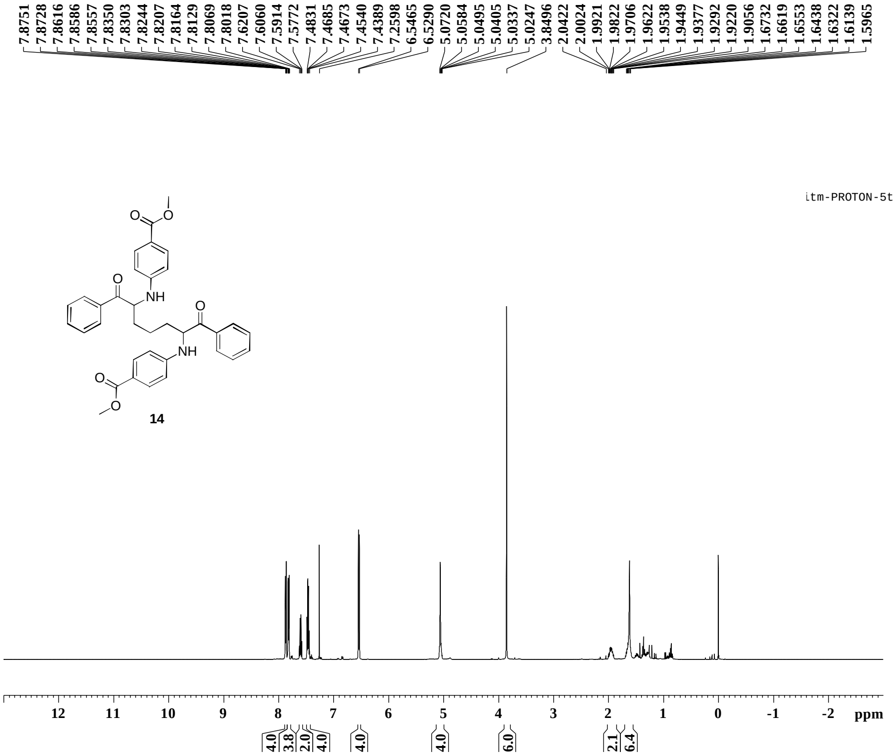
```
   Lab mmb
   ram-302
iitm_carbonshort CDCl3 /opt/topspin iitm 1

Current Data Parameters
NAME          mmb51015
EXPNO               25
PROCNO               1

F2 - Acquisition Parameters
Date_         20151016
Time             20.38
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD               20480
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          29761.904 Hz
FIDRES        1.453218 Hz
AQ           0.3440640 sec
RG              202.34
DW              16.800 usec
DE                6.50 usec
TE               295.3 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       125.7753932 MHz
NUC1               13C
P1                9.63 usec
PLW1      103.00000000 W

======== CHANNEL f2 ========
SFO2       500.1520006 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2            80.00 usec
PLW2       15.30000019 W
PLW12       0.33006001 W
PLW13       0.21123999 W

F2 - Processing parameters
SI               32768
SF         125.7627948 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



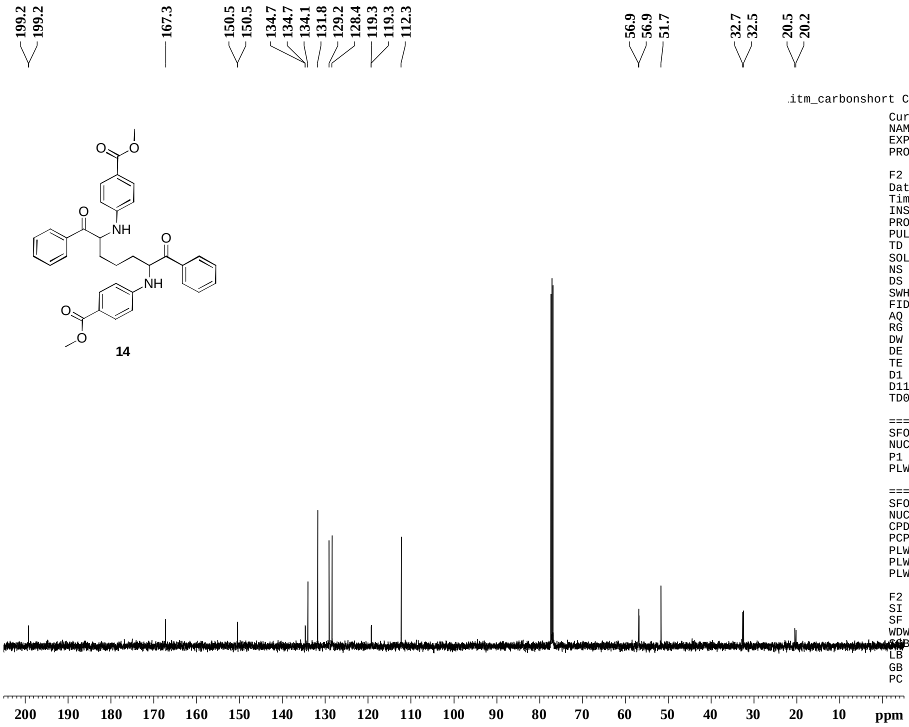
```
            lab mmb-ram-alphtet-pure
iitm-Proton(-5to15) CDCl3 /opt/topspin nmr 5
              Current Data Parameters
              NAME            mmb40116
              EXPNO                  7
              PROCNO                 1

              F2 - Acquisition Parameters
              Date_           20160101
              Time               21.21
              INSTRUM            spect
              PROBHD   5 mm PABBO BB-
              PULPROG             zg30
              TD                 65536
              SOLVENT            CDCl3
              NS                    16
              DS                     2
              SWH             8012.820 Hz
              FIDRES          0.122266 Hz
              AQ             4.0894465 sec
              RG                200.34
              DW                62.400 usec
              DE                  6.50 usec
              TE                 291.9 K
              D1            0.50000000 sec
              TD0                    1

              ======== CHANNEL f1 ========
              NUC1                  1H
              P1                 15.70 usec
              PLW1          7.75000000 W
              SFO1         400.1320007 MHz

              F2 - Processing parameters
              SI                 65536
              SF           400.1300094 MHz
              WDW                   EM
              SSB      0
              LB                  0.30 Hz
              GB       0
              PC                  1.00
```



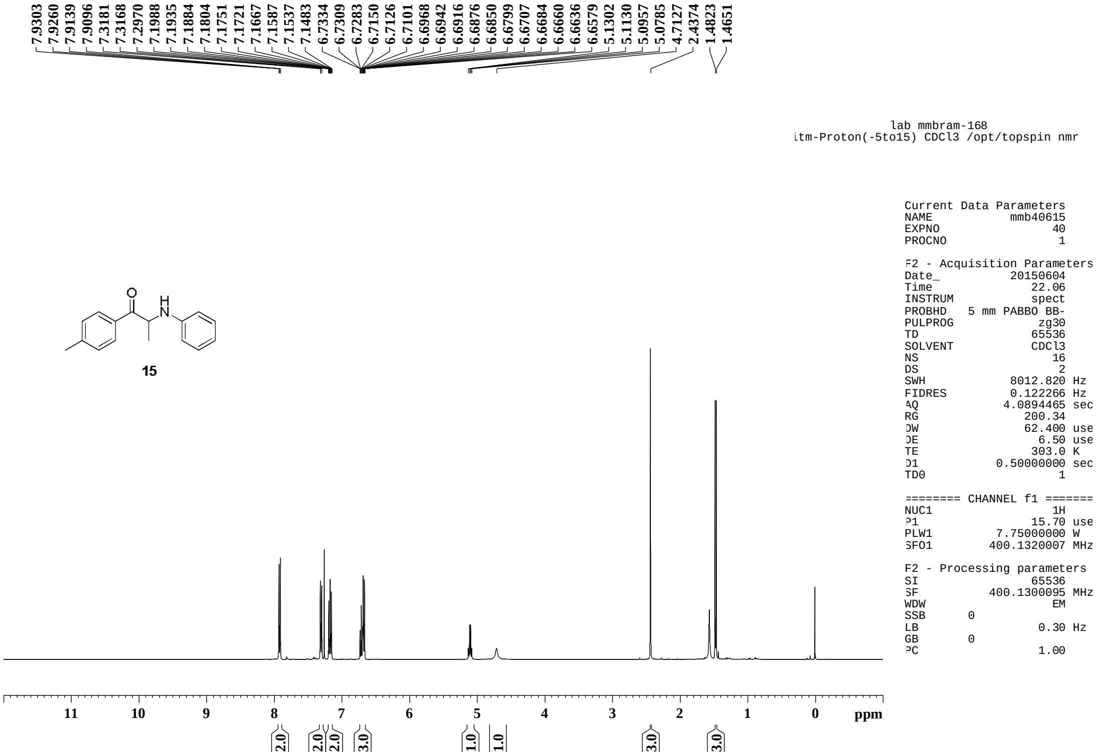
```
            lab mmb-ram-alphtet-pure
iitm_carbonshort CDCl3 /opt/topspin nmr 5
              Current Data Parameters
              NAME            mmb40116
              EXPNO                  8
              PROCNO                 1

              F2 - Acquisition Parameters
              Date_           20160101
              Time               21.35
              INSTRUM            spect
              PROBHD   5 mm PABBO BB-
              PULPROG           zgpg30
              TD                 16540
              SOLVENT            CDCl3
              NS                   512
              DS                     4
              SWH            24038.461 Hz
              FIDRES          1.453353 Hz
              AQ             0.3440320 sec
              RG                200.34
              DW                20.800 usec
              DE                  6.50 usec
              TE                 292.4 K
              D1            1.00000000 sec
              D11           0.03000000 sec
              TD0                    1

              ======== CHANNEL f1 ========
              NUC1                 13C
              P1                  9.25 usec
              PLW1         47.00000000 W
              SFO1         100.6228289 MHz

              ======== CHANNEL f2 ========
              CPDPRG[2         waltz16
              NUC2                  1H
              PCPD2              90.00 usec
              PLW2          7.75000000 W
              PLW12         0.23583999 W
              PLW13         0.19103000 W
              SFO2         400.1316005 MHz

              F2 - Processing parameters
              SI                 32768
              SF           100.6127573 MHz
              WDW                   EM
              SSB      0
              LB                  1.00 Hz
              GB       0
              PC                  1.40
```



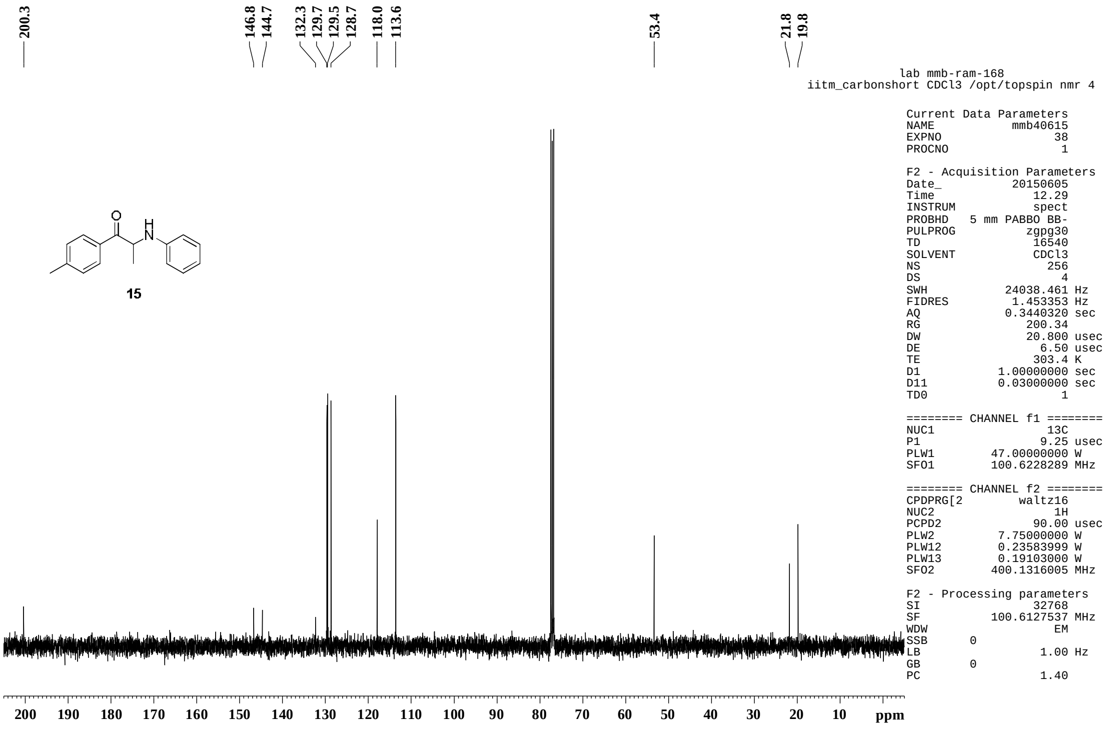
```
      Lab mmb
      ram-343
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 1
Current Data Parameters
NAME             mmb51115
EXPNO                 106
PROCNO                  1

F2 - Acquisition Parameters
Date_            20151125
Time                18.05
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             10000.000 Hz
FIDRES           0.305176 Hz
AQ              1.6384000 sec
RG                 138.53
DW                 50.000 use
DE                   6.50 use
TE                  301.1 K
D1             0.50000000 sec
TD0                     1

======== CHANNEL f1 =======
SFO1          500.1525008 MHz
NUC1                   1H
P1                  11.75 use
PLW1          15.30000019 W

F2 - Processing parameters
SI                  65536
SF            500.1500218 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



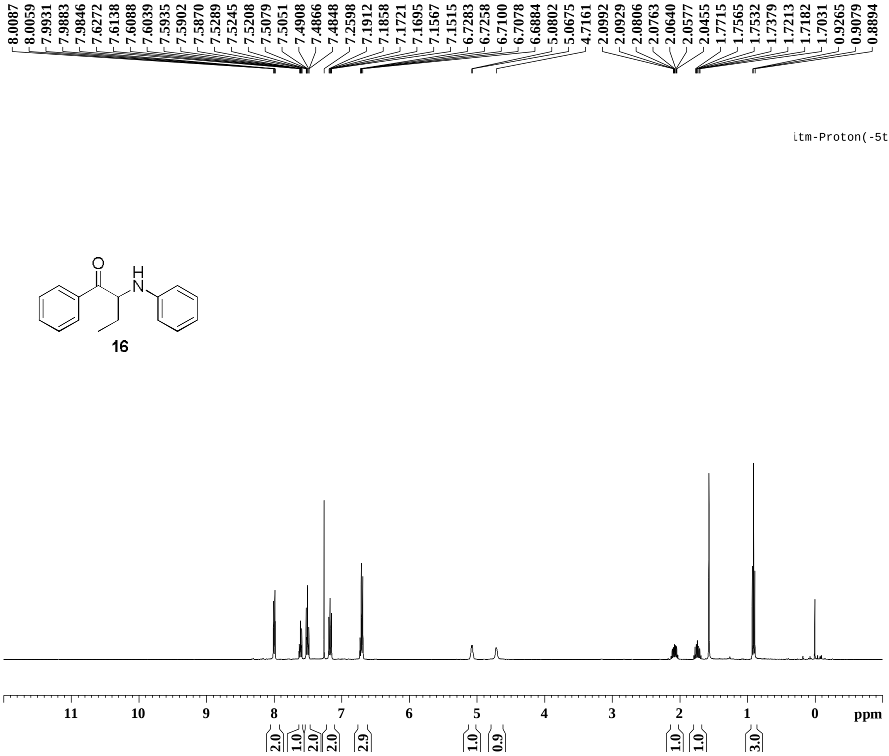
```
      Lab mmb
      ram-343
itm_carbonshort CDCl3 /opt/topspin iitm 10

Current Data Parameters
NAME             mmb51115
EXPNO                 107
PROCNO                  1

F2 - Acquisition Parameters
Date_            20151125
Time                18.12
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                  20480
SOLVENT             CDCl3
NS                    256
DS                      4
SWH             29761.904 Hz
FIDRES           1.453218 Hz
AQ              0.3440640 sec
RG                 202.34
DW                 16.800 usec
DE                   6.50 usec
TE                  301.5 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          125.7753932 MHz
NUC1                  13C
P1                   9.63 usec
PLW1         103.00000000 W

======== CHANNEL f2 ========
SFO2          500.1520006 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2          15.30000019 W
PLW12          0.33006001 W
PLW13          0.21123999 W

F2 - Processing parameters
SI                  32768
SF            125.7628030 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



```
lab mmbram-168
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb40615
EXPNO               40
PROCNO               1

F2 - Acquisition Parameters
Date_         20150604
Time             22.06
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              200.34
DW              62.400 use
DE                6.50 use
TE               303.0 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300095 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

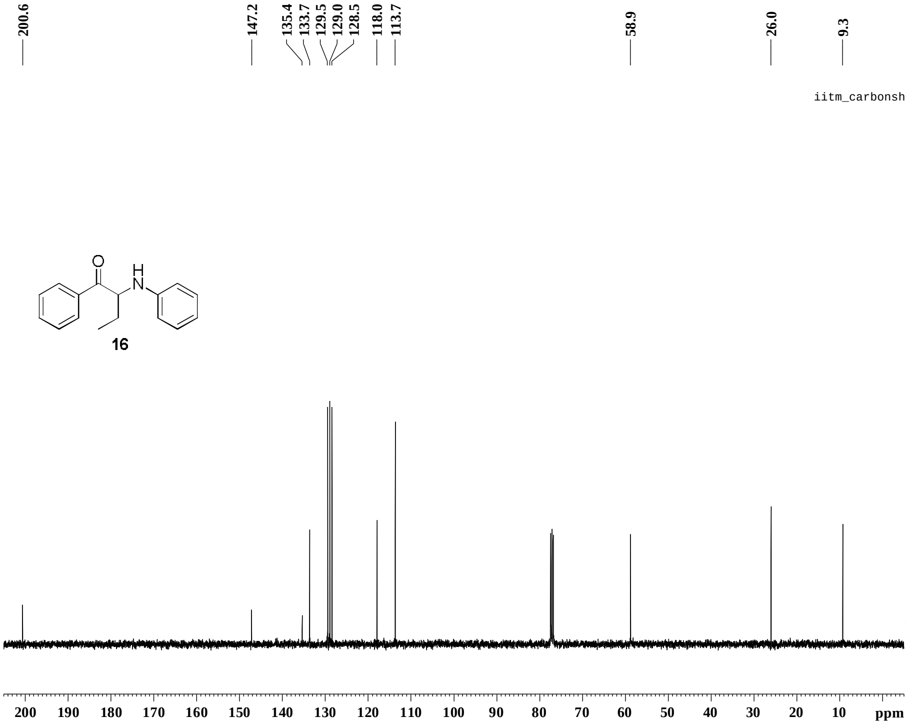


```
lab mmb-ram-168
iitm_carbonshort CDCl3 /opt/topspin nmr 4

Current Data Parameters
NAME          mmb40615
EXPNO               38
PROCNO               1

F2 - Acquisition Parameters
Date_         20150605
Time             12.29
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               303.4 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127537 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```

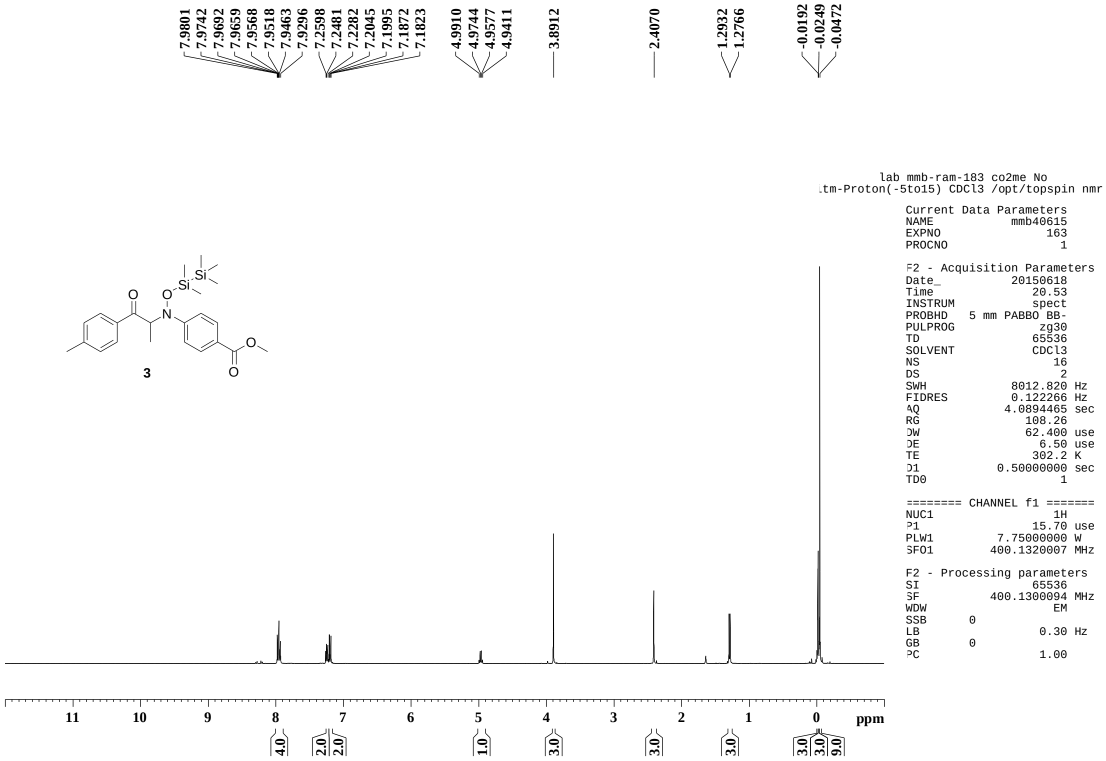


```
                 lab mmbram-176
itm-Proton(-5to15) CDCl3 /opt/topspin nmr
```

```
Current Data Parameters
NAME           mmb40615
EXPNO                90
PROCNO                1

F2 - Acquisition Parameters
Date_          20150612
Time               5.45
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               200.34
DW               62.400 use
DE                 6.50 use
TE                299.5 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300095 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

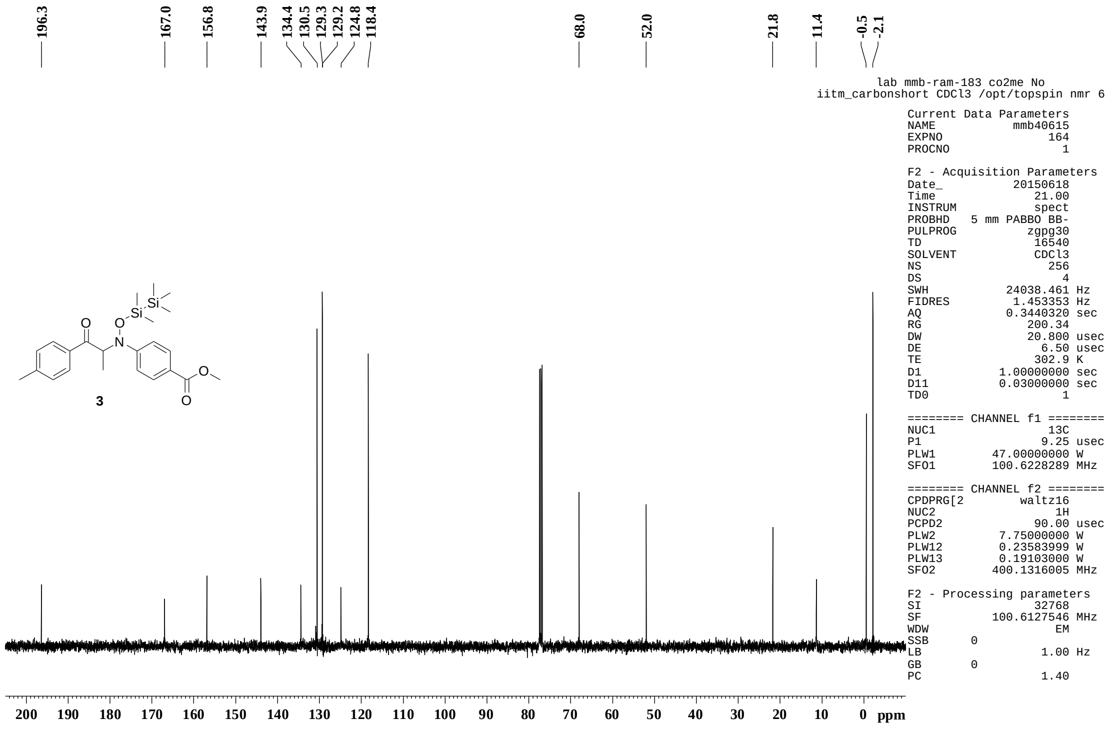


```
                 lab mmb-ram-176
iitm_carbonshort CDCl3 /opt/topspin nmr 3
Current Data Parameters
NAME           mmb40615
EXPNO                83
PROCNO                1

F2 - Acquisition Parameters
Date_          20150611
Time              17.05
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                299.5 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127563 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



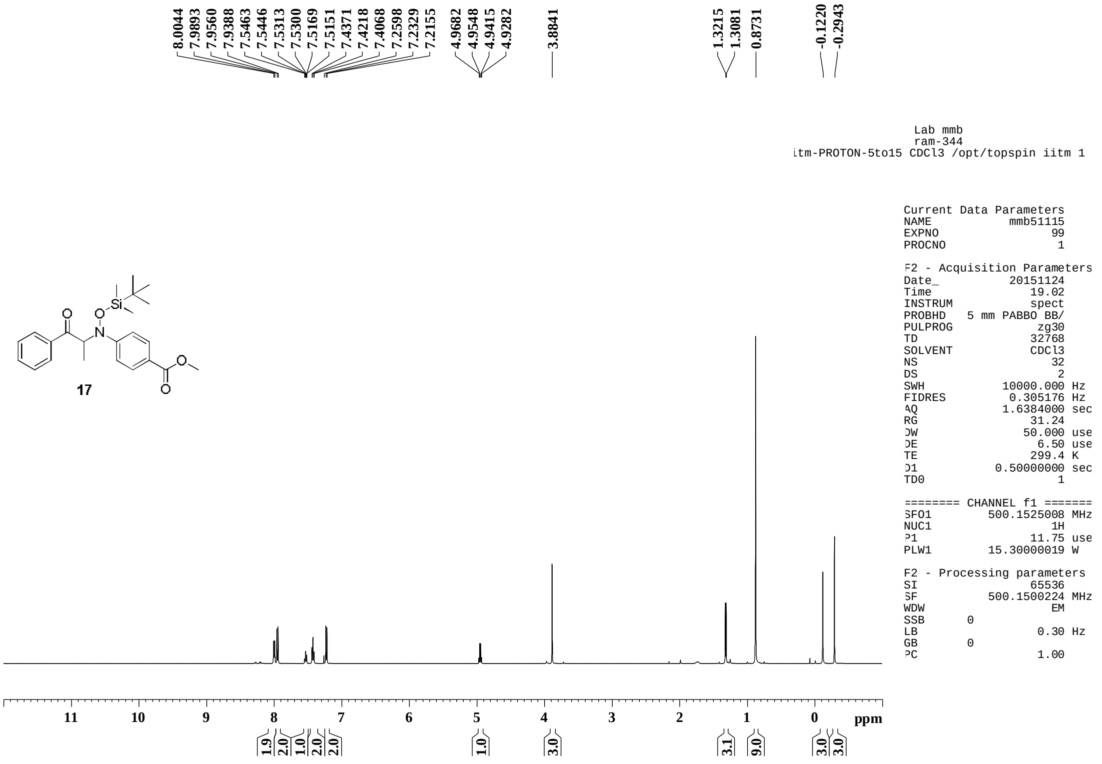
3

```
          lab mmb-ram-183 co2me No
.tm-Proton(-5to15) CDCl3 /opt/topspin nmr

               Current Data Parameters
               NAME            mmb40615
               EXPNO                163
               PROCNO                 1

               F2 - Acquisition Parameters
               Date_           20150618
               Time               20.53
               INSTRUM            spect
               PROBHD   5 mm PABBO BB-
               PULPROG             zg30
               TD                 65536
               SOLVENT            CDCl3
               NS                    16
               DS                     2
               SWH             8012.820 Hz
               FIDRES          0.122266 Hz
               AQ             4.0894465 sec
               RG                108.26
               DW                62.400 use
               DE                  6.50 use
               TE                 302.2 K
               D1            0.50000000 sec
               TD0                    1

               ======== CHANNEL f1 =======
               NUC1                  1H
               P1                 15.70 use
               PLW1          7.75000000 W
               SFO1         400.1320007 MHz

               F2 - Processing parameters
               SI                 65536
               SF           400.1300094 MHz
               WDW                   EM
               SSB      0
               LB                  0.30 Hz
               GB       0
               PC                  1.00
```

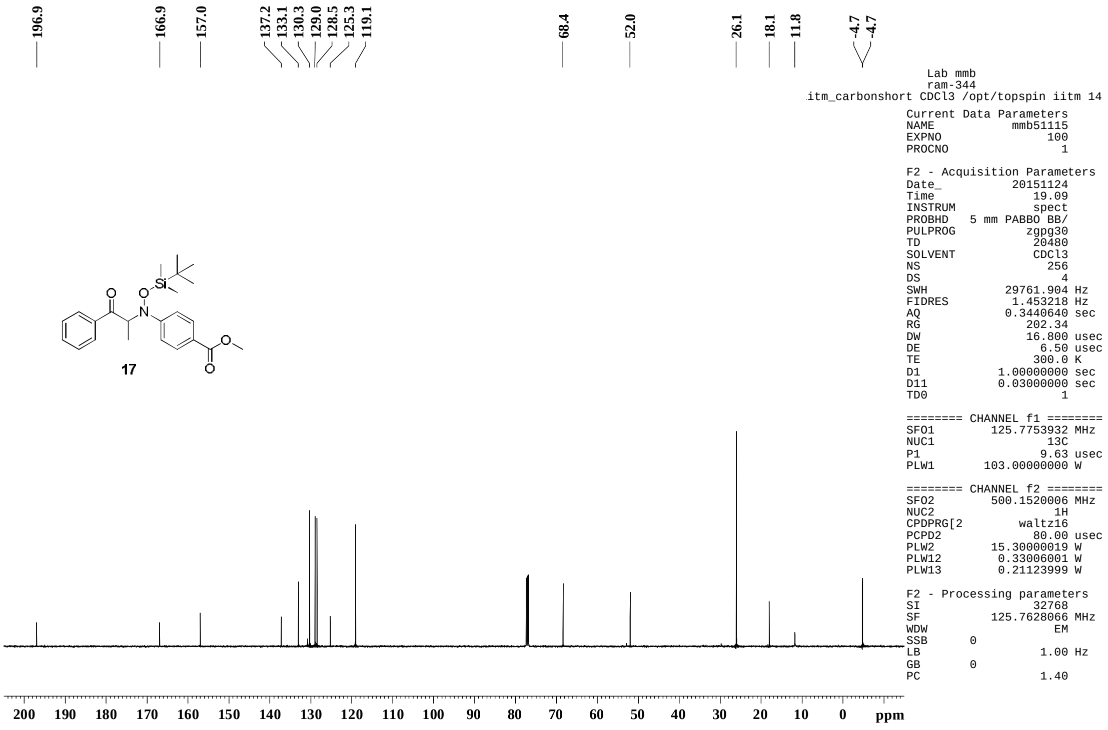


3

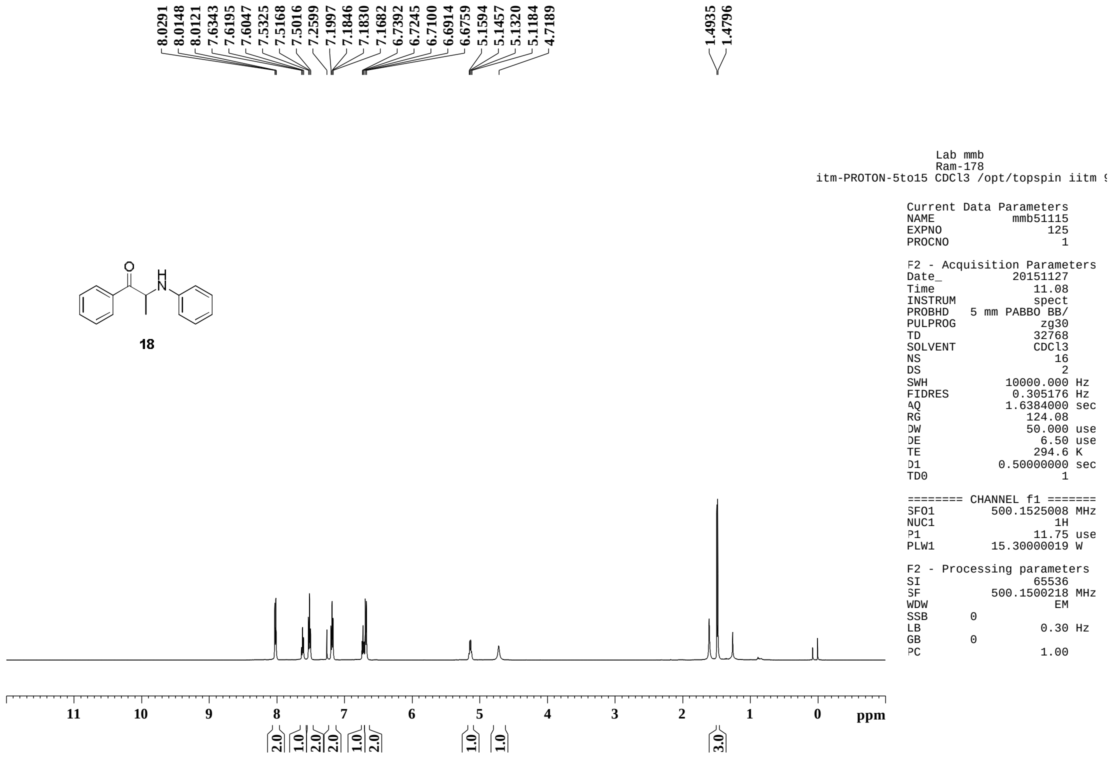
```
          lab mmb-ram-183 co2me No
iitm_carbonshort CDCl3 /opt/topspin nmr 6

               Current Data Parameters
               NAME            mmb40615
               EXPNO                164
               PROCNO                 1

               F2 - Acquisition Parameters
               Date_           20150618
               Time               21.00
               INSTRUM            spect
               PROBHD   5 mm PABBO BB-
               PULPROG           zgpg30
               TD                 16540
               SOLVENT            CDCl3
               NS                   256
               DS                     4
               SWH            24038.461 Hz
               FIDRES          1.453353 Hz
               AQ             0.3440320 sec
               RG                200.34
               DW                20.800 usec
               DE                  6.50 usec
               TE                 302.9 K
               D1            1.00000000 sec
               D11           0.03000000 sec
               TD0                    1

               ======== CHANNEL f1 ========
               NUC1                   13C
               P1                   9.25 usec
               PLW1         47.00000000 W
               SFO1         100.6228289 MHz

               ======== CHANNEL f2 ========
               CPDPRG[2           waltz16
               NUC2                    1H
               PCPD2               90.00 usec
               PLW2           7.75000000 W
               PLW12          0.23583999 W
               PLW13          0.19103000 W
               SFO2         400.1316005 MHz

               F2 - Processing parameters
               SI                  32768
               SF            100.6127546 MHz
               WDW                    EM
               SSB      0
               LB                   1.00 Hz
               GB       0
               PC                   1.40
```



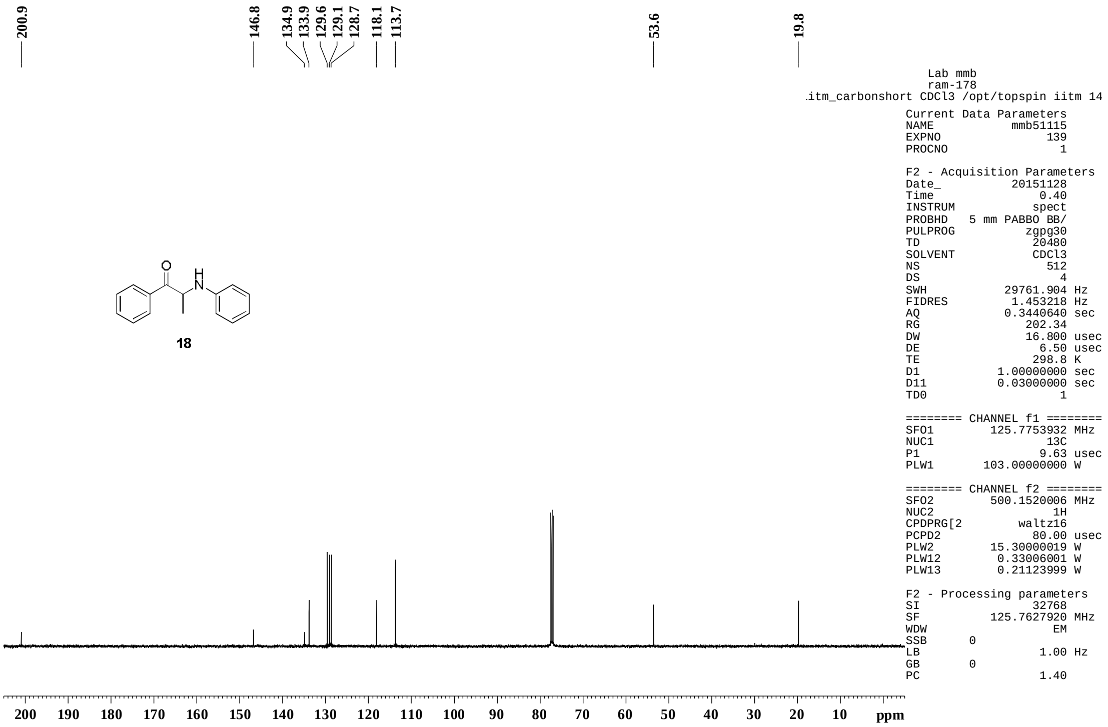
```
        Lab mmb
        ram-344
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 1

Current Data Parameters
NAME            mmb51115
EXPNO                 99
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151124
Time               19.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             1.6384000 sec
RG                 31.24
DW                50.000 use
DE                  6.50 use
TE                 299.4 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
SFO1         500.1525008 MHz
NUC1                  1H
P1                 11.75 use
PLW1         15.30000019 W

F2 - Processing parameters
SI                 65536
SF           500.1500224 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



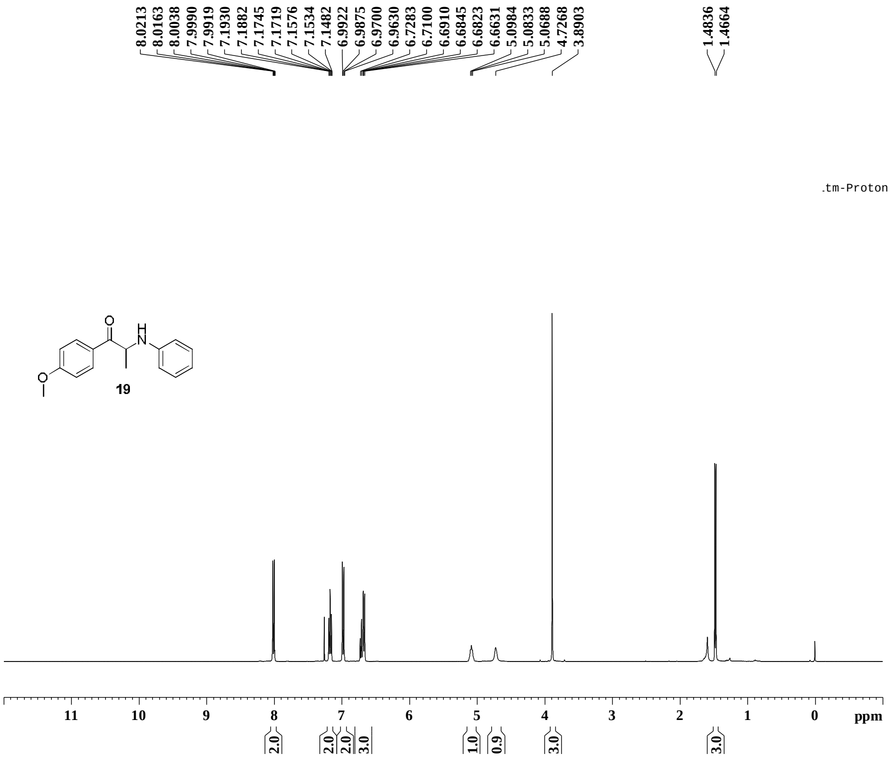
```
        Lab mmb
        ram-344
itm_carbonshort CDCl3 /opt/topspin iitm 14
Current Data Parameters
NAME            mmb51115
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151124
Time               19.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 20480
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            29761.904 Hz
FIDRES          1.453218 Hz
AQ             0.3440640 sec
RG                202.34
DW                16.800 usec
DE                  6.50 usec
TE                 300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7753932 MHz
NUC1                 13C
P1                  9.63 usec
PLW1        103.00000000 W

======== CHANNEL f2 ========
SFO2         500.1520006 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         15.30000019 W
PLW12         0.33006001 W
PLW13         0.21123999 W

F2 - Processing parameters
SI                 32768
SF           125.7628066 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



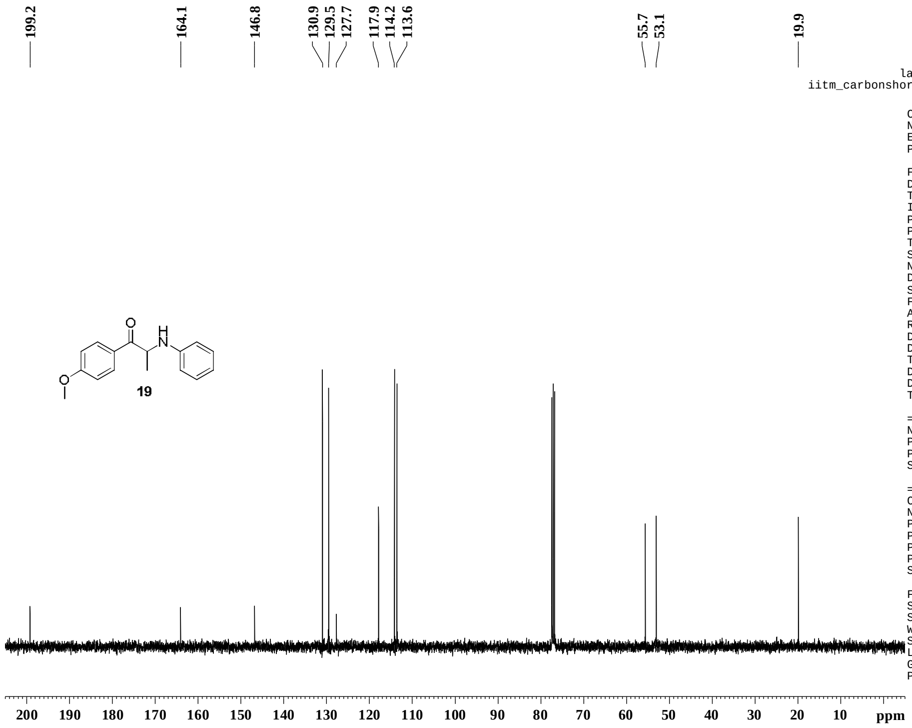
```
       Lab mmb
       Ram-178
itm-PROTON-5to15 CDCl3 /opt/topspin iitm

Current Data Parameters
NAME           mmb51115
EXPNO               125
PROCNO                1

F2 - Acquisition Parameters
Date_          20151127
Time              11.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ            1.6384000 sec
RG               124.08
DW               50.000 use
DE                 6.50 use
TE                294.6 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
SFO1        500.1525008 MHz
NUC1                 1H
P1                11.75 use
PLW1        15.30000019 W

F2 - Processing parameters
SI                65536
SF          500.1500218 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



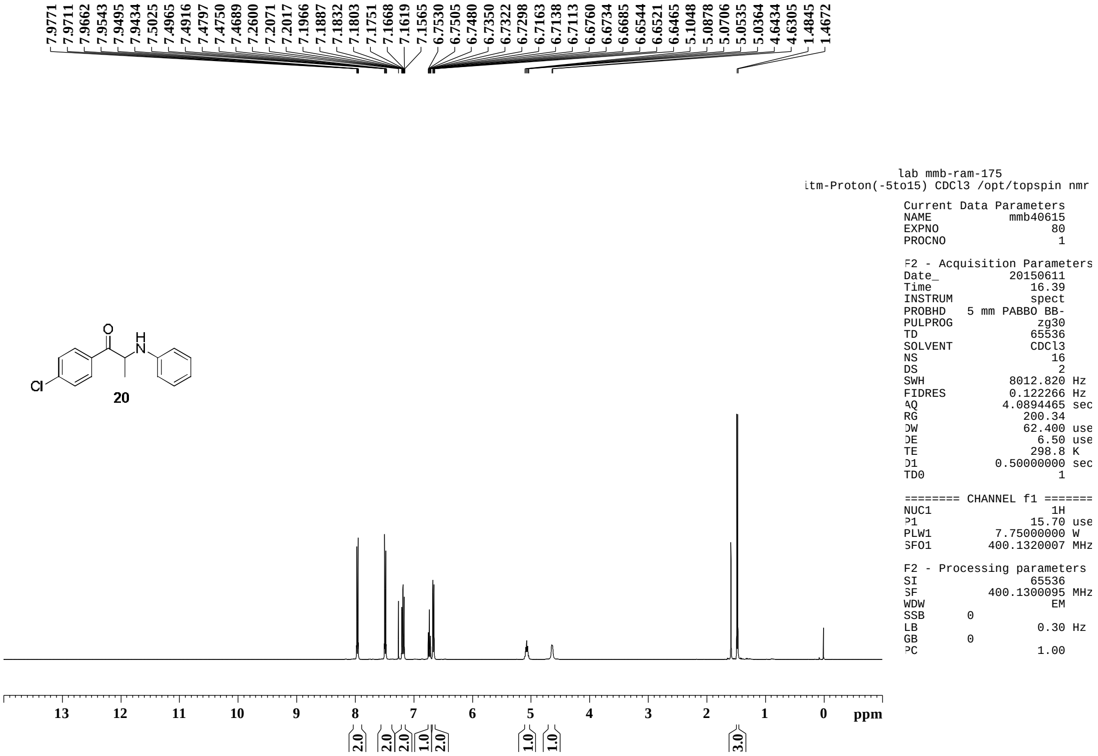
```
       Lab mmb
       ram-178
itm_carbonshort CDCl3 /opt/topspin iitm 14
Current Data Parameters
NAME           mmb51115
EXPNO               139
PROCNO                1

F2 - Acquisition Parameters
Date_          20151128
Time               0.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                20480
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES         1.453218 Hz
AQ            0.3440640 sec
RG               202.34
DW               16.800 usec
DE                 6.50 usec
TE                298.8 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7753932 MHz
NUC1                13C
P1                 9.63 usec
PLW1       103.00000000 W

======== CHANNEL f2 ========
SFO2        500.1520006 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             80.00 usec
PLW2        15.30000019 W
PLW12        0.33006001 W
PLW13        0.21123999 W

F2 - Processing parameters
SI                32768
SF          125.7627920 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



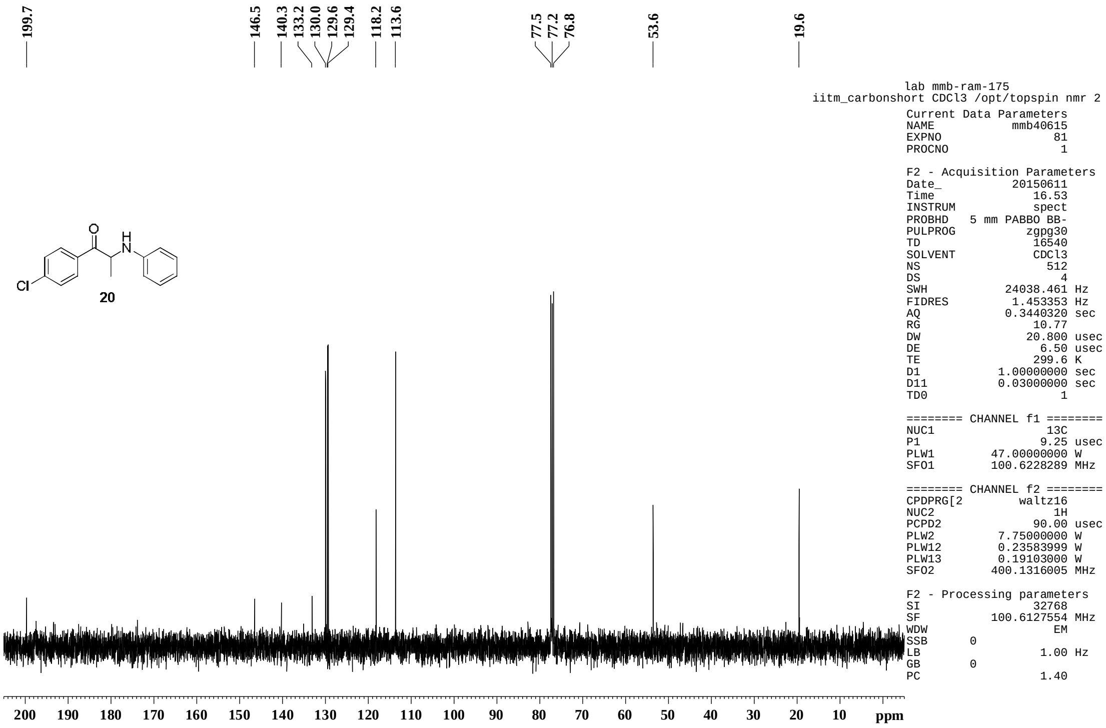
```
           lab mmb-ram-174
.tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME           mmb40615
EXPNO                78
PROCNO                1

F2 - Acquisition Parameters
Date_          20150611
Time              16.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               169.77
DW               62.400 use
DE                 6.50 use
TE                298.8 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300096 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



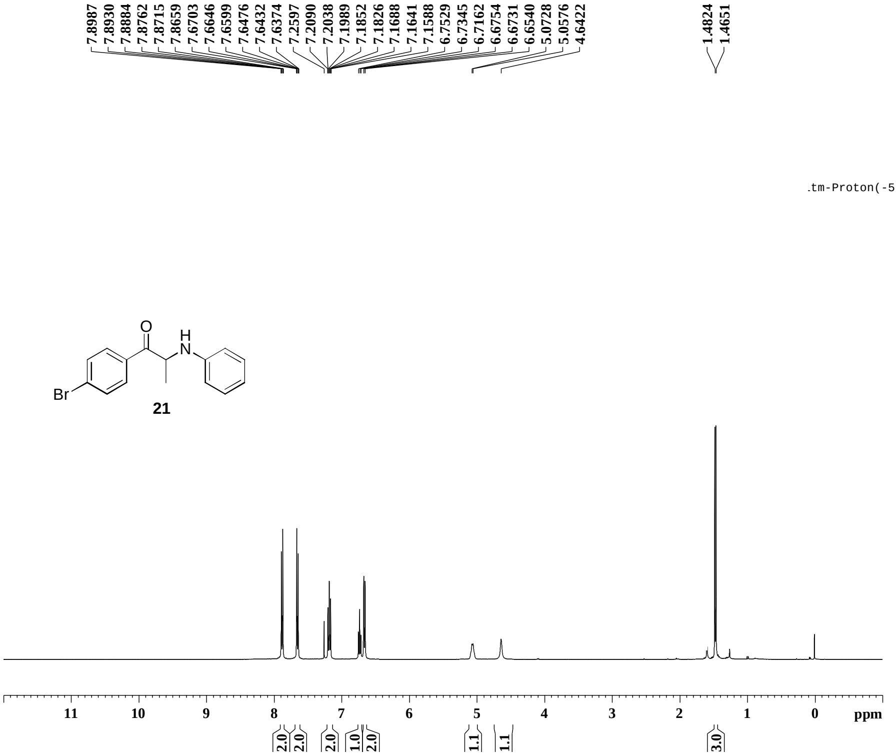
```
           lab mmb-ram-174
iitm_carbonshort CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME           mmb40615
EXPNO                79
PROCNO                1

F2 - Acquisition Parameters
Date_          20150611
Time              16.34
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                299.5 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127554 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



```
   lab mmb-ram-175
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb40615
EXPNO               80
PROCNO               1

F2 - Acquisition Parameters
Date_         20150611
Time             16.39
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              200.34
DW              62.400 use
DE                6.50 use
TE               298.8 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300095 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

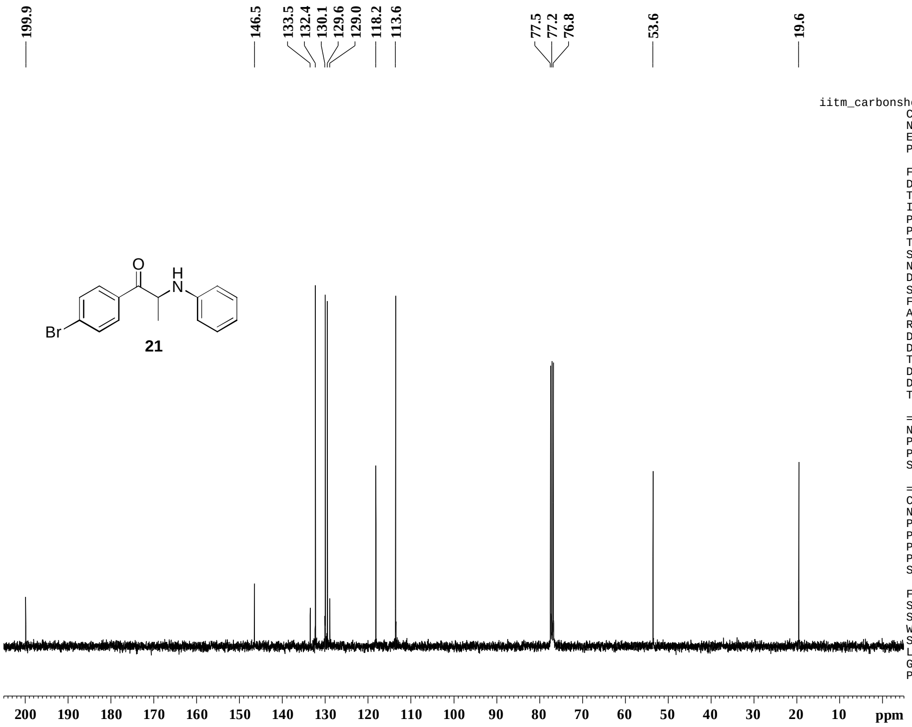


```
   lab mmb-ram-175
iitm_carbonshort CDCl3 /opt/topspin nmr 2

Current Data Parameters
NAME          mmb40615
EXPNO               81
PROCNO               1

F2 - Acquisition Parameters
Date_         20150611
Time             16.53
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG               10.77
DW              20.800 usec
DE                6.50 usec
TE               299.6 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2       waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127554 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



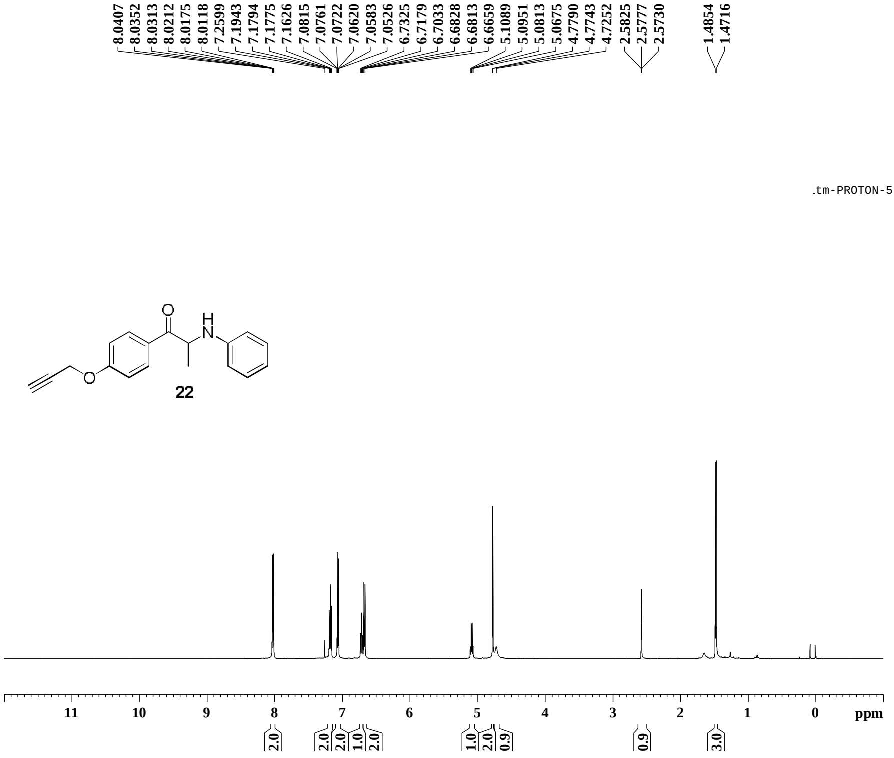
21

```
              lab mmb-ram-194
.tm-Proton(-5to15) CDCl3 /opt/topspin nmr
             Current Data Parameters
             NAME           mmb40615
             EXPNO                38
             PROCNO                1

             F2 - Acquisition Parameters
             Date_          20150705
             Time              16.05
             INSTRUM           spect
             PROBHD   5 mm PABBO BB-
             PULPROG            zg30
             TD                65536
             SOLVENT           CDCl3
             NS                   16
             DS                    2
             SWH            8012.820 Hz
             FIDRES         0.122266 Hz
             AQ            4.0894465 sec
             RG               153.13
             DW               62.400 use
             DE                 6.50 use
             TE                296.5 K
             D1           0.50000000 sec
             TD0                   1

             ======== CHANNEL f1 =======
             NUC1                 1H
             P1                15.70 use
             PLW1         7.75000000 W
             SFO1        400.1320007 MHz

             F2 - Processing parameters
             SI                65536
             SF          400.1300094 MHz
             WDW                  EM
             SSB      0
             LB                 0.30 Hz
             GB       0
             PC                 1.00
```

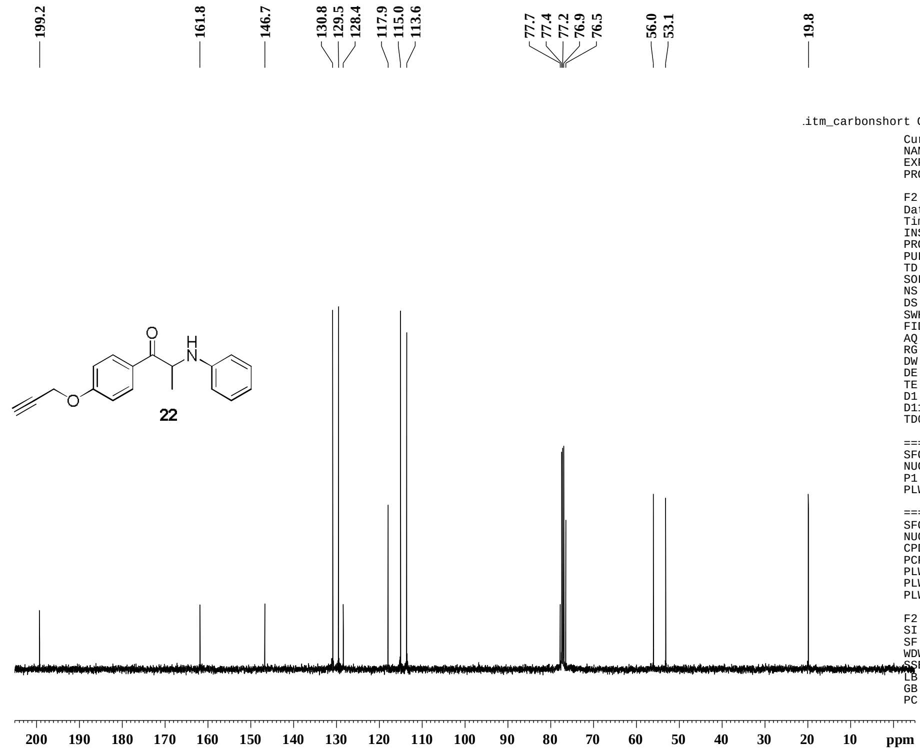


21

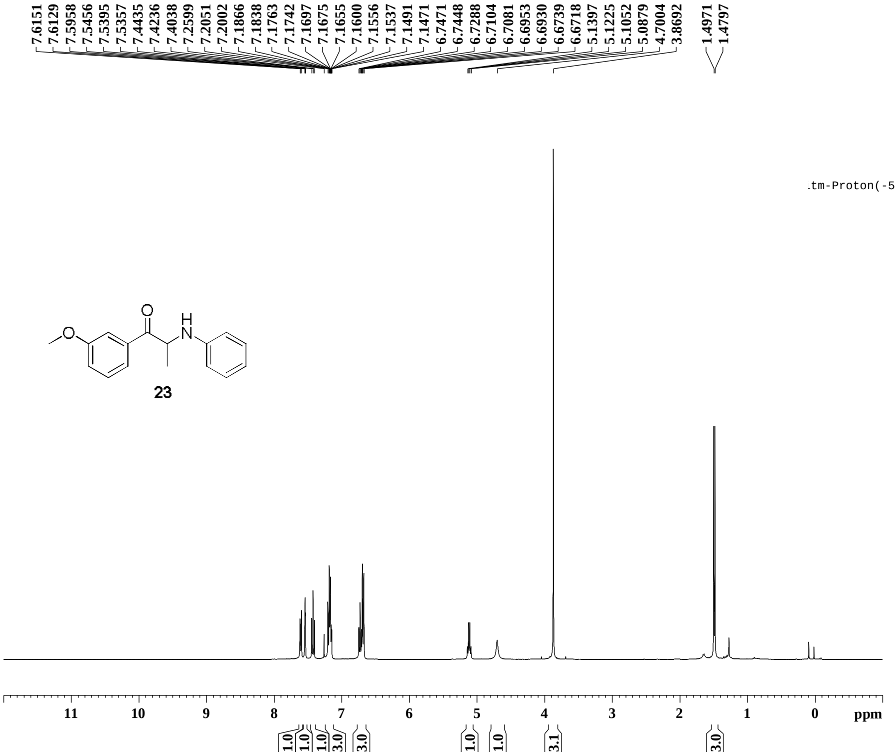
```
              lab mmb-ram-194
iitm_carbonshort CDCl3 /opt/topspin nmr 1
          Current Data Parameters
          NAME           mmb40615
          EXPNO                39
          PROCNO                1

          F2 - Acquisition Parameters
          Date_          20150705
          Time              16.10
          INSTRUM           spect
          PROBHD   5 mm PABBO BB-
          PULPROG          zgpg30
          TD                16540
          SOLVENT           CDCl3
          NS                  512
          DS                    4
          SWH           24038.461 Hz
          FIDRES         1.453353 Hz
          AQ            0.3440320 sec
          RG               200.34
          DW               20.800 usec
          DE                 6.50 usec
          TE                297.3 K
          D1           1.00000000 sec
          D11          0.03000000 sec
          TD0                   1

          ======== CHANNEL f1 ========
          NUC1                13C
          P1                 9.25 usec
          PLW1        47.00000000 W
          SFO1        100.6228289 MHz

          ======== CHANNEL f2 ========
          CPDPRG[2        waltz16
          NUC2                 1H
          PCPD2             90.00 usec
          PLW2         7.75000000 W
          PLW12        0.23583999 W
          PLW13        0.19103000 W
          SFO2        400.1316005 MHz

          F2 - Processing parameters
          SI                32768
          SF          100.6127570 MHz
          WDW                  EM
          SSB      0
          LB                 1.00 Hz
          GB       0
          PC                 1.40
```



```
     Lab mmb
     ram-316
.tm-PROTON-5to15 CDCl3 /opt/topspin iitm 1
             Current Data Parameters
             NAME           mmb51115
             EXPNO                35
             PROCNO                1

             F2 - Acquisition Parameters
             Date_          20151104
             Time              21.09
             INSTRUM           spect
             PROBHD   5 mm PABBO BB/
             PULPROG            zg30
             TD                32768
             SOLVENT           CDCl3
             NS                   16
             DS                    2
             SWH           10000.000 Hz
             FIDRES         0.305176 Hz
             AQ            1.6384000 sec
             RG                79.04
             DW               50.000 use
             DE                 6.50 use
             TE                295.7 K
             D1           0.50000000 sec
             TD0                   1

             ======== CHANNEL f1 =======
             SFO1        500.1525008 MHz
             NUC1                 1H
             P1                11.75 use
             PLW1        15.30000019 W

             F2 - Processing parameters
             SI                65536
             SF          500.1500231 MHz
             WDW                  EM
             SSB      0
             LB                 0.30 Hz
             GB       0
             PC                 1.00
```

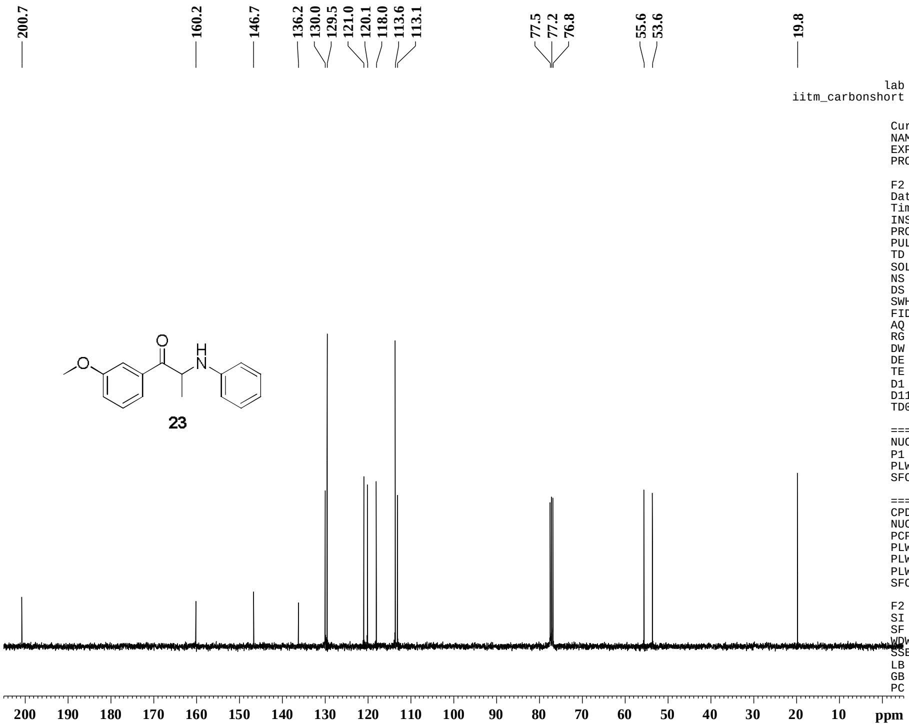


```
     Lab mmb
     ram-316
.itm_carbonshort CDCl3 /opt/topspin iitm 11
             Current Data Parameters
             NAME           mmb51115
             EXPNO                36
             PROCNO                1

             F2 - Acquisition Parameters
             Date_          20151104
             Time              21.16
             INSTRUM           spect
             PROBHD   5 mm PABBO BB/
             PULPROG          zgpg30
             TD                20480
             SOLVENT           CDCl3
             NS                  256
             DS                    4
             SWH           29761.904 Hz
             FIDRES         1.453218 Hz
             AQ            0.3440640 sec
             RG               202.34
             DW               16.800 usec
             DE                 6.50 usec
             TE                296.3 K
             D1           1.00000000 sec
             D11          0.03000000 sec
             TD0                   1

             ======== CHANNEL f1 ========
             SFO1        125.7753932 MHz
             NUC1                13C
             P1                 9.63 usec
             PLW1       103.00000000 W

             ======== CHANNEL f2 ========
             SFO2        500.1520006 MHz
             NUC2                 1H
             CPDPRG[2        waltz16
             PCPD2             80.00 usec
             PLW2        15.30000019 W
             PLW12        0.33006001 W
             PLW13        0.21123999 W

             F2 - Processing parameters
             SI                32768
             SF          125.7628095 MHz
             WDW                  EM
             SSB      0
             LB                 1.00 Hz
             GB       0
             PC                 1.40
```



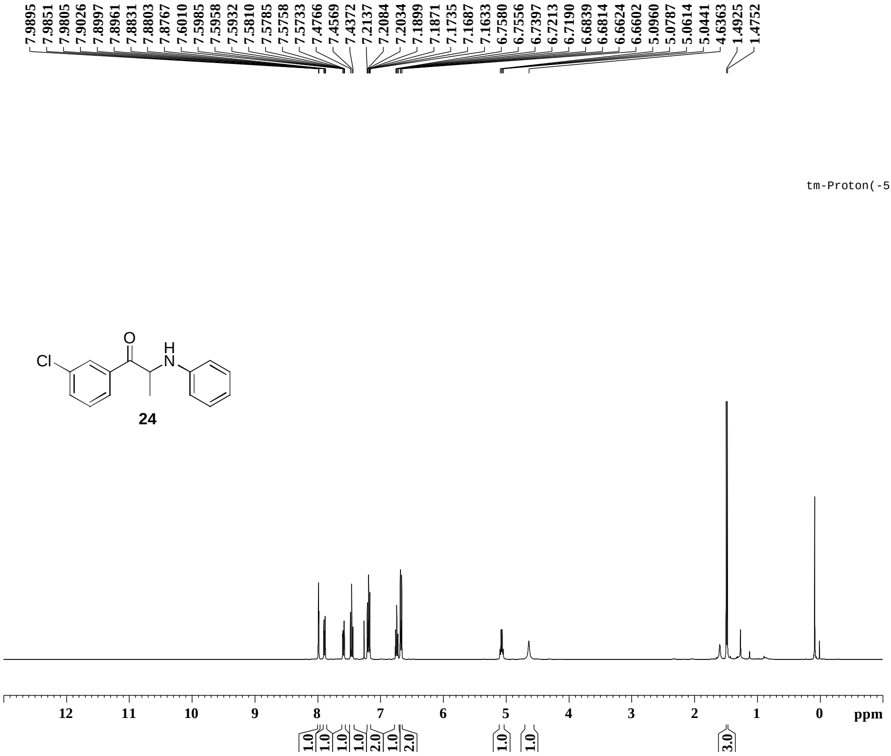
23

```
          lab mmb-ram-253
.tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb40915
EXPNO               85
PROCNO               1

F2 - Acquisition Parameters
Date_         20150915
Time             12.13
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              108.26
DW              62.400 use
DE                6.50 use
TE               298.6 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300095 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

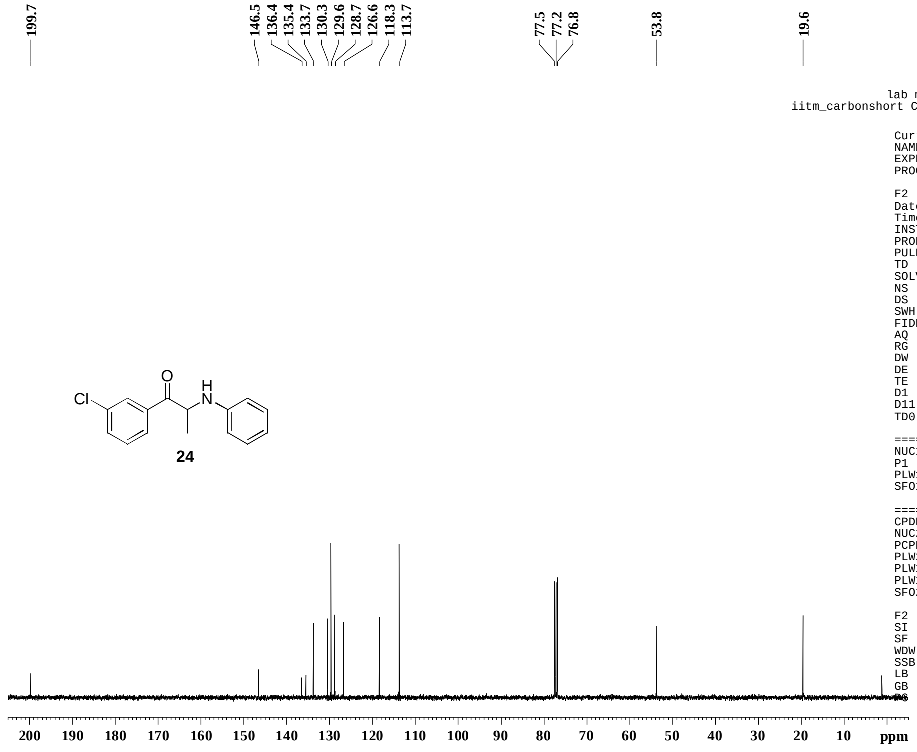


23

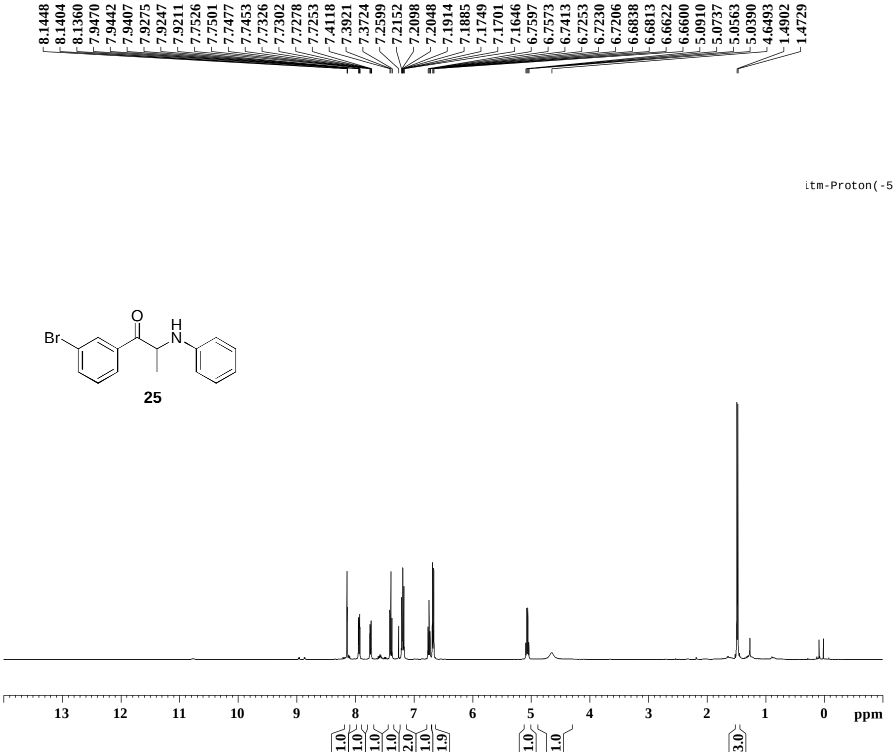
```
          lab mmb-ram-253
iitm_carbonshort CDCl3 /opt/topspin nmr 8

Current Data Parameters
NAME          mmb40915
EXPNO               86
PROCNO               1

F2 - Acquisition Parameters
Date_         20150915
Time             12.21
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               299.2 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127590 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



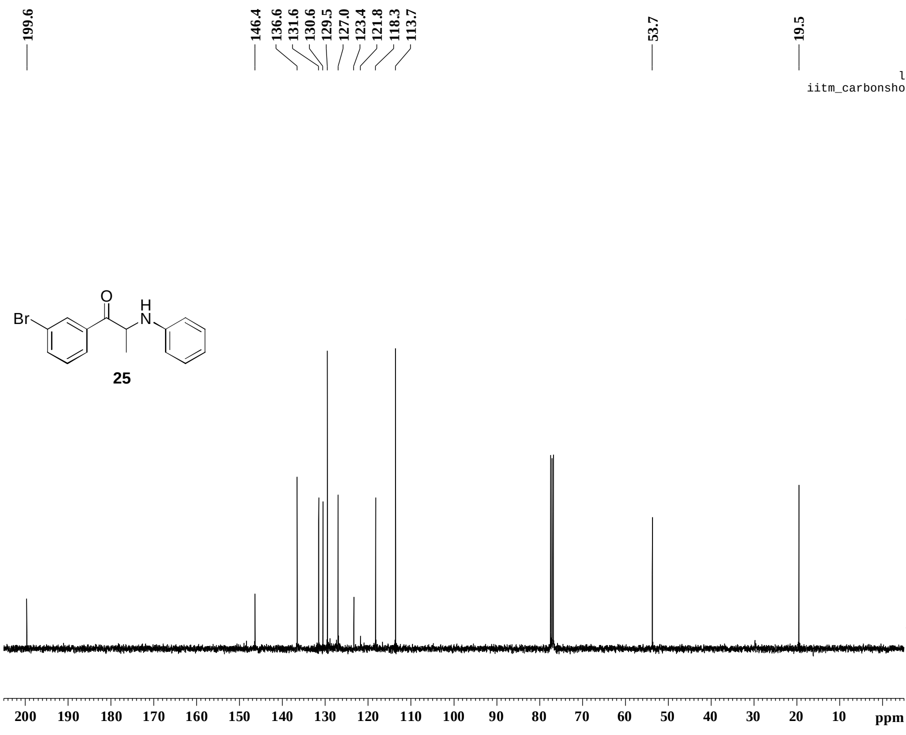
```
          lab mmb-ram-260
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME            mmb40915
EXPNO                154
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150918
Time               17.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                153.13
DW                62.400 use
DE                  6.50 use
TE                 298.1 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300094 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



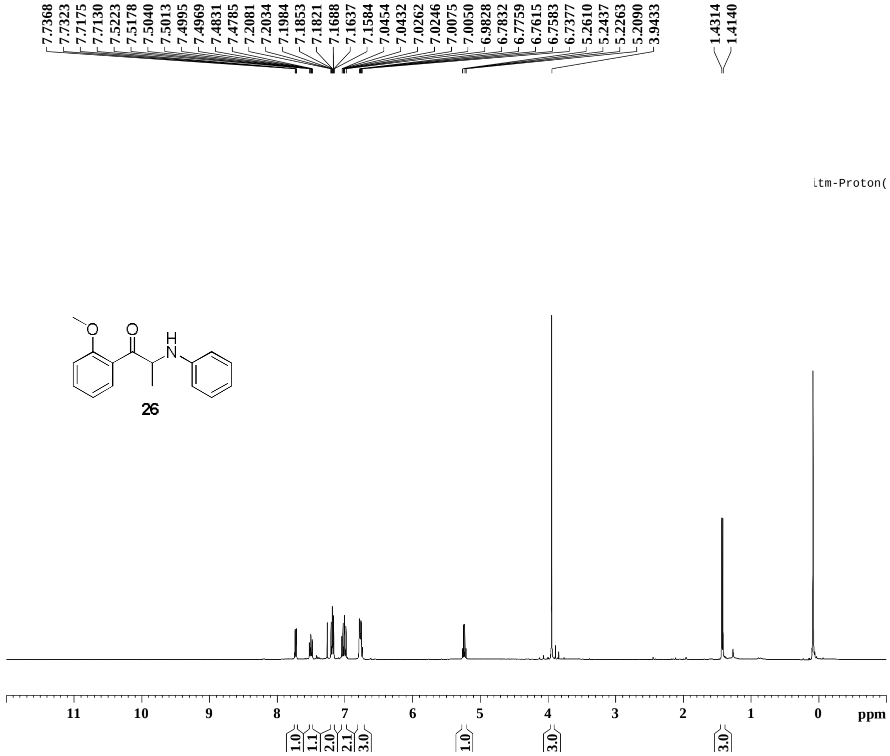
```
          lab mmb-ram-260
iitm_carbonshort CDCl3 /opt/topspin nmr 16

Current Data Parameters
NAME            mmb40915
EXPNO                155
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150918
Time               17.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 298.8 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127567 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



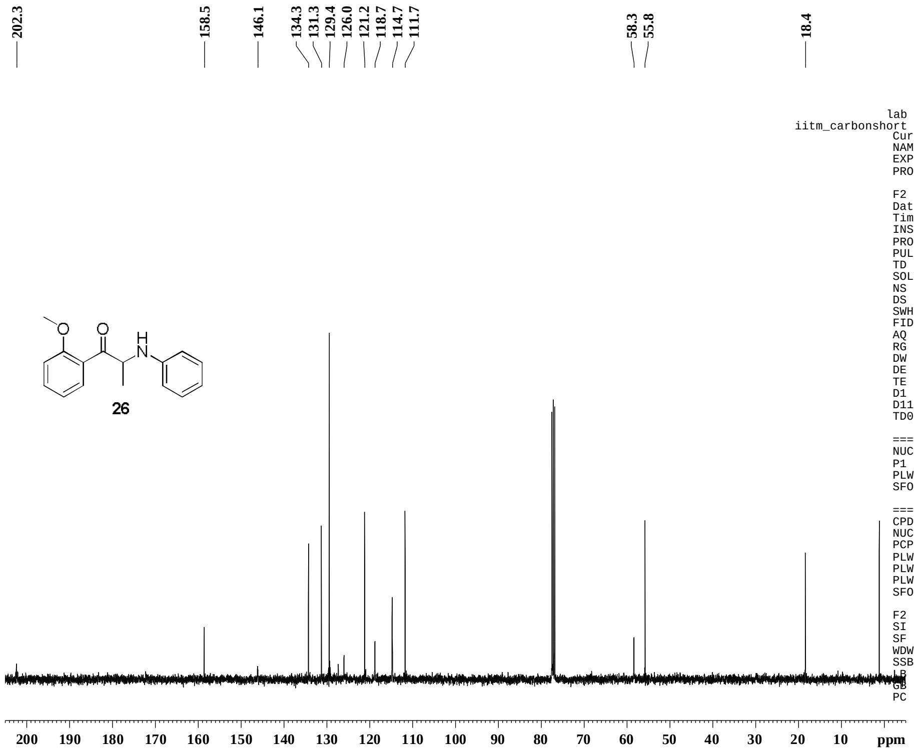
```
lab mmb-ram-248
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME           mmb40815
EXPNO               276
PROCNO                1

F2 - Acquisition Parameters
Date_          20150831
Time              18.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               124.58
DW               62.400 use
DE                 6.50 use
TE                296.8 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300094 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



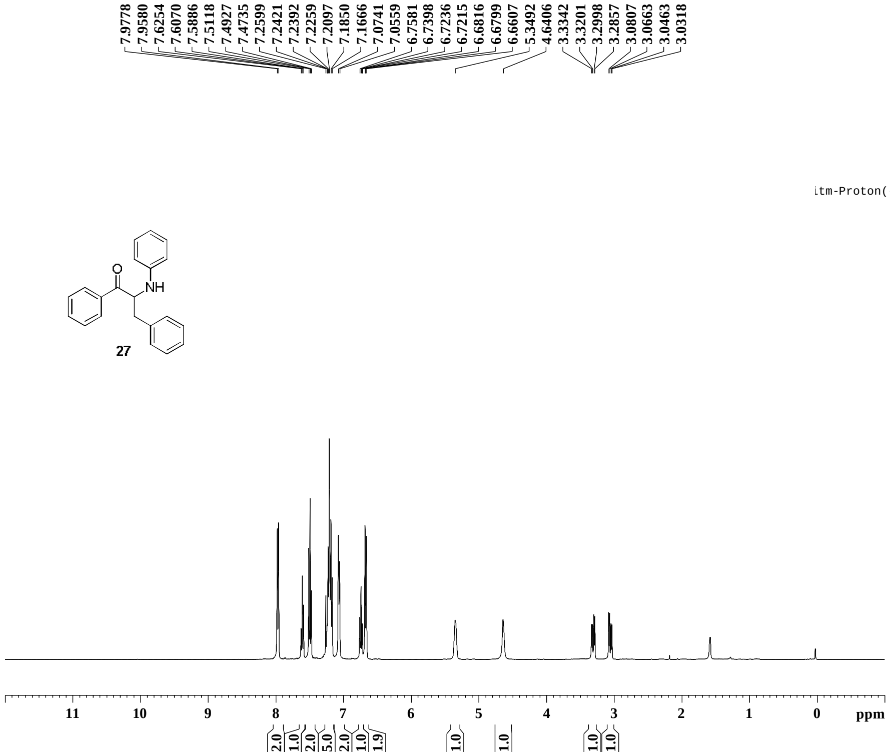
```
lab mmb-ram-248
iitm_carbonshort CDCl3 /opt/topspin nmr 9

Current Data Parameters
NAME           mmb40815
EXPNO               277
PROCNO                1

F2 - Acquisition Parameters
Date_          20150831
Time              18.37
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                297.6 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127588 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



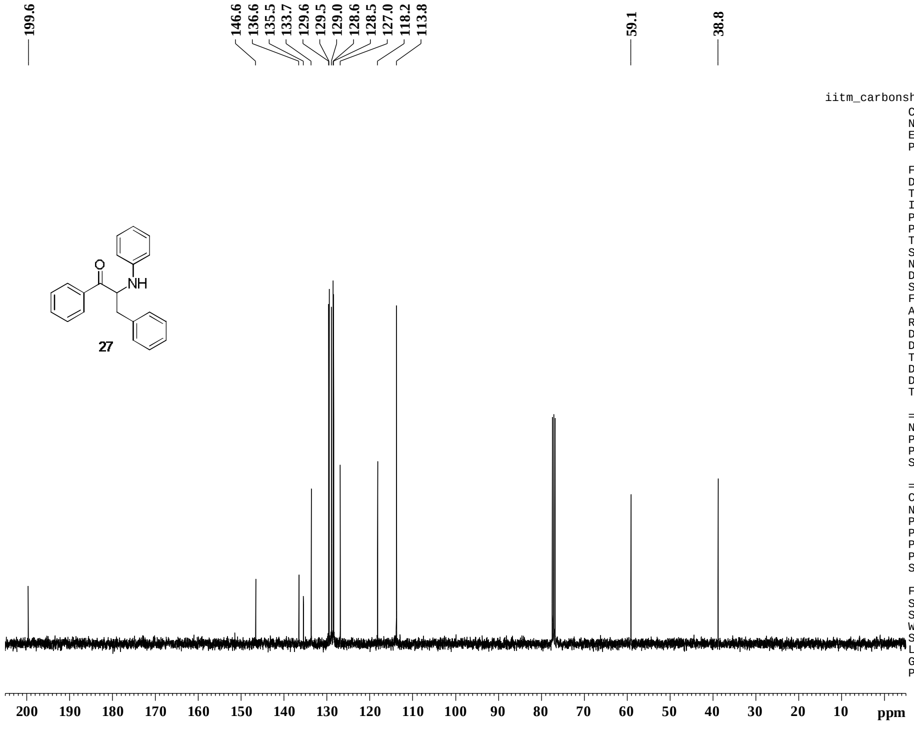
```
          lab mmb-ram-264
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                166
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151026
Time               18.30
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH          8012.820 Hz
FIDRES       0.122266 Hz
AQ          4.0894465 sec
RG                 95.73
DW            62.400 use
DE              6.50 use
TE             296.0 K
D1        0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1             15.70 use
PLW1        7.75000000 W
SFO1      400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF        400.1300095 MHz
WDW                   EM
SSB      0
LB               0.30 Hz
GB       0
PC               1.00
```



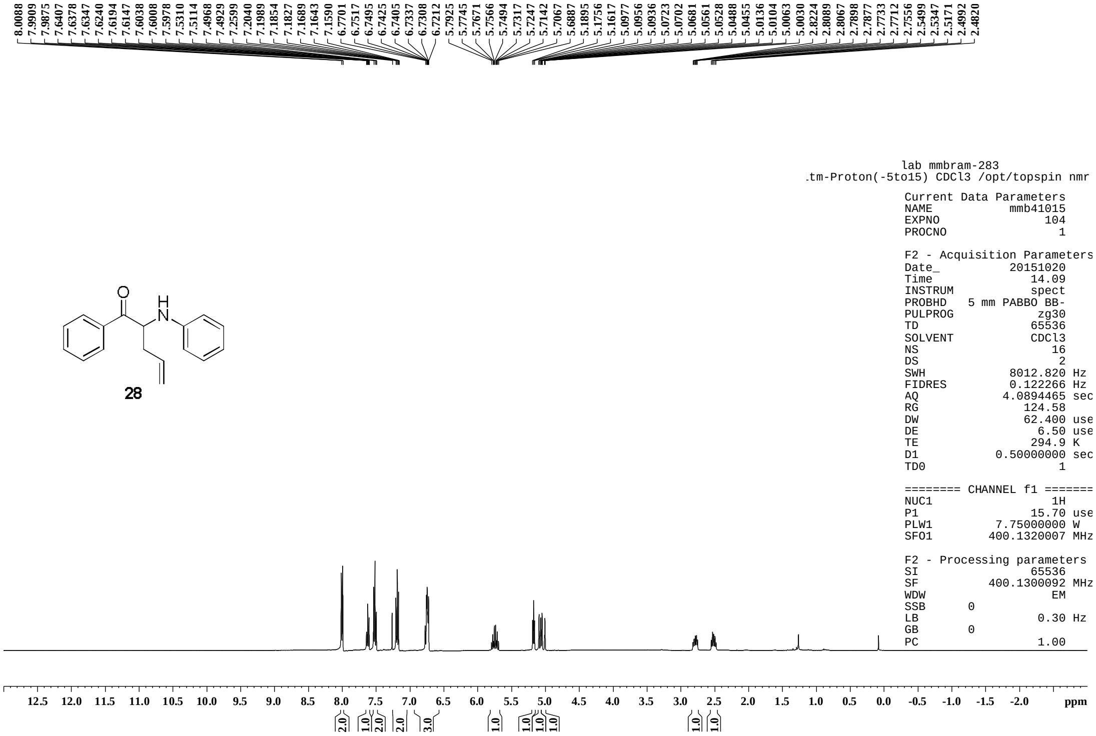
```
              lab mmb-ram-264
iitm_carbonshort CDCl3 /opt/topspin nmr 2
         Current Data Parameters
         NAME            mmb41015
         EXPNO                167
         PROCNO                 1

         F2 - Acquisition Parameters
         Date_           20151026
         Time               18.38
         INSTRUM            spect
         PROBHD   5 mm PABBO BB-
         PULPROG           zgpg30
         TD                 16540
         SOLVENT            CDCl3
         NS                   256
         DS                     4
         SWH         24038.461 Hz
         FIDRES       1.453353 Hz
         AQ          0.3440320 sec
         RG                200.34
         DW             20.800 usec
         DE               6.50 usec
         TE              296.8 K
         D1         1.00000000 sec
         D11        0.03000000 sec
         TD0                    1

         ======== CHANNEL f1 ========
         NUC1                 13C
         P1                  9.25 usec
         PLW1       47.00000000 W
         SFO1       100.6228289 MHz

         ======== CHANNEL f2 ========
         CPDPRG[2         waltz16
         NUC2                  1H
         PCPD2              90.00 usec
         PLW2         7.75000000 W
         PLW12        0.23583999 W
         PLW13        0.19103000 W
         SFO2       400.1316005 MHz

         F2 - Processing parameters
         SI                 32768
         SF         100.6127592 MHz
         WDW                   EM
         SSB      0
         LB                 1.00 Hz
         GB       0
         PC                 1.40
```



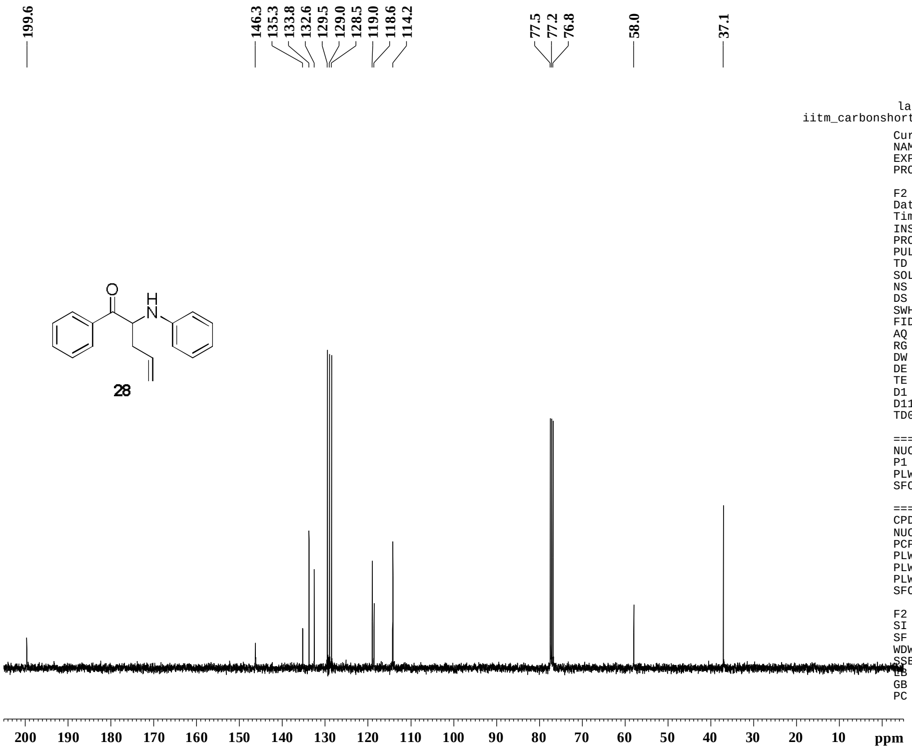
```
           lab mmbram-278
itm-Proton(-5to15) CDCl3 /opt/topspin nmr
Current Data Parameters
NAME           mmb40915
EXPNO               196
PROCNO                1

F2 - Acquisition Parameters
Date_          20150930
Time              13.21
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG               124.58
DW               62.400 use
DE                 6.50 use
TE                297.8 K
D1          0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300092 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```



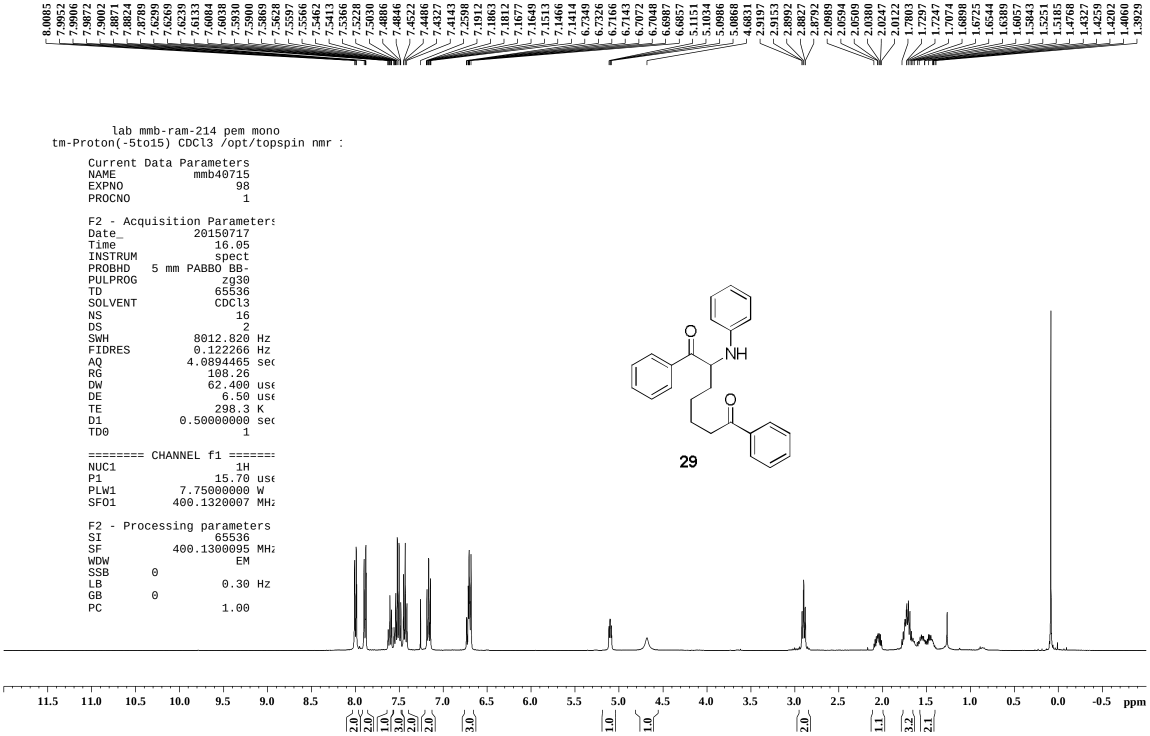
```
            lab mmbram-278
iitm_carbonshort CDCl3 /opt/topspin nmr 6
Current Data Parameters
NAME           mmb40915
EXPNO               197
PROCNO                1

F2 - Acquisition Parameters
Date_          20150930
Time              13.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                298.6 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127588 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



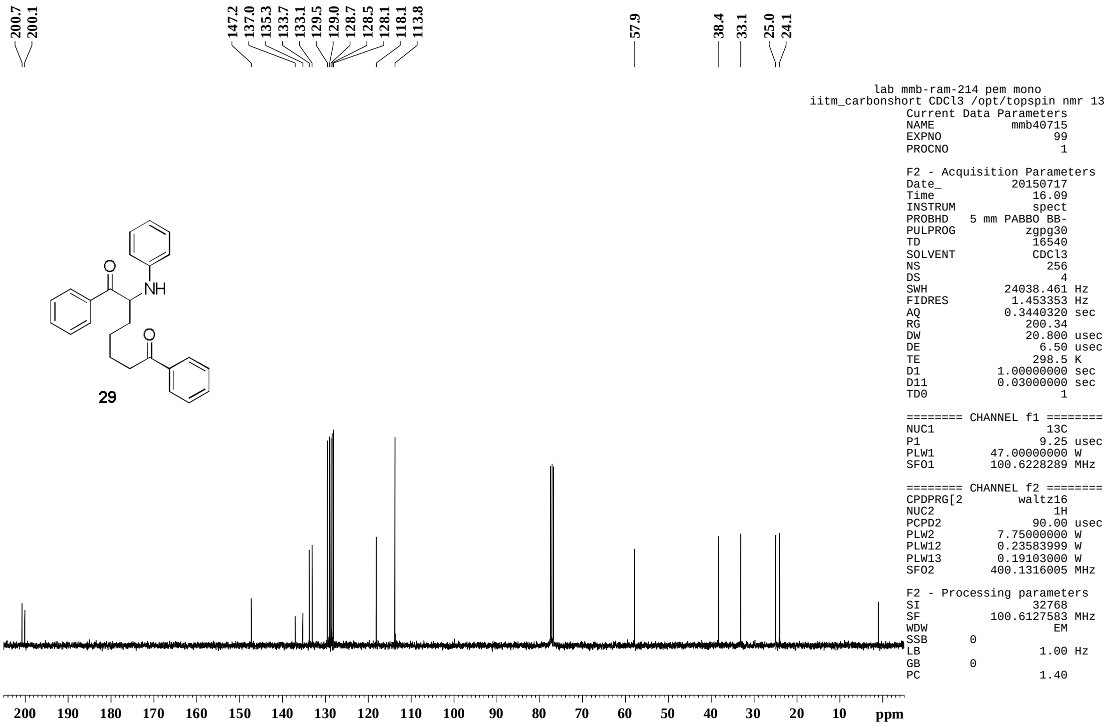
```
lab mmbram-283
.tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                104
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151020
Time               14.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                124.58
DW                62.400 use
DE                  6.50 use
TE                 294.9 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1         400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300092 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



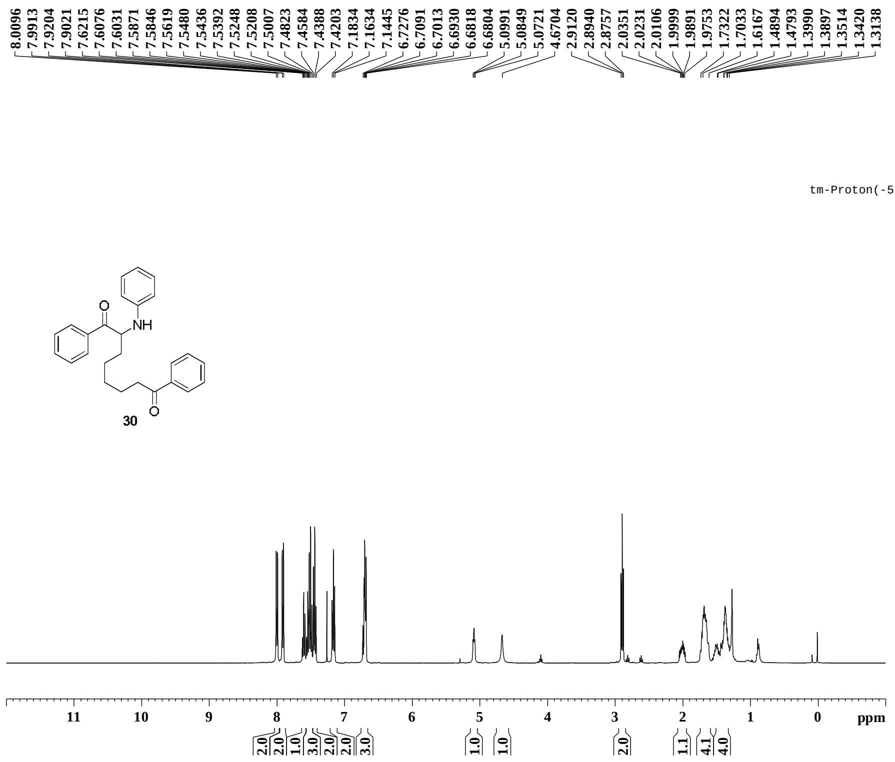
```
lab mmbram-283
iitm_carbonshort CDCl3 /opt/topspin nmr 4

Current Data Parameters
NAME            mmb41015
EXPNO                105
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151020
Time               14.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 295.8 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127587 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



```
      lab mmb-ram-214 pem mono
tm-Proton(-5to15) CDCl3 /opt/topspin nmr :

     Current Data Parameters
     NAME           mmb40715
     EXPNO                98
     PROCNO                1

     F2 - Acquisition Parameters
     Date_          20150717
     Time              16.05
     INSTRUM           spect
     PROBHD   5 mm PABBO BB-
     PULPROG            zg30
     TD                65536
     SOLVENT           CDCl3
     NS                   16
     DS                    2
     SWH            8012.820 Hz
     FIDRES         0.122266 Hz
     AQ            4.0894465 sec
     RG               108.26
     DW               62.400 use
     DE                 6.50 use
     TE                298.3 K
     D1           0.50000000 sec
     TD0                   1

     ======== CHANNEL f1 ======
     NUC1                 1H
     P1                15.70 use
     PLW1        7.75000000 W
     SFO1        400.1320007 MHz

     F2 - Processing parameters
     SI                65536
     SF          400.1300095 MHz
     WDW                  EM
     SSB      0
     LB                 0.30 Hz
     GB       0
     PC                 1.00
```

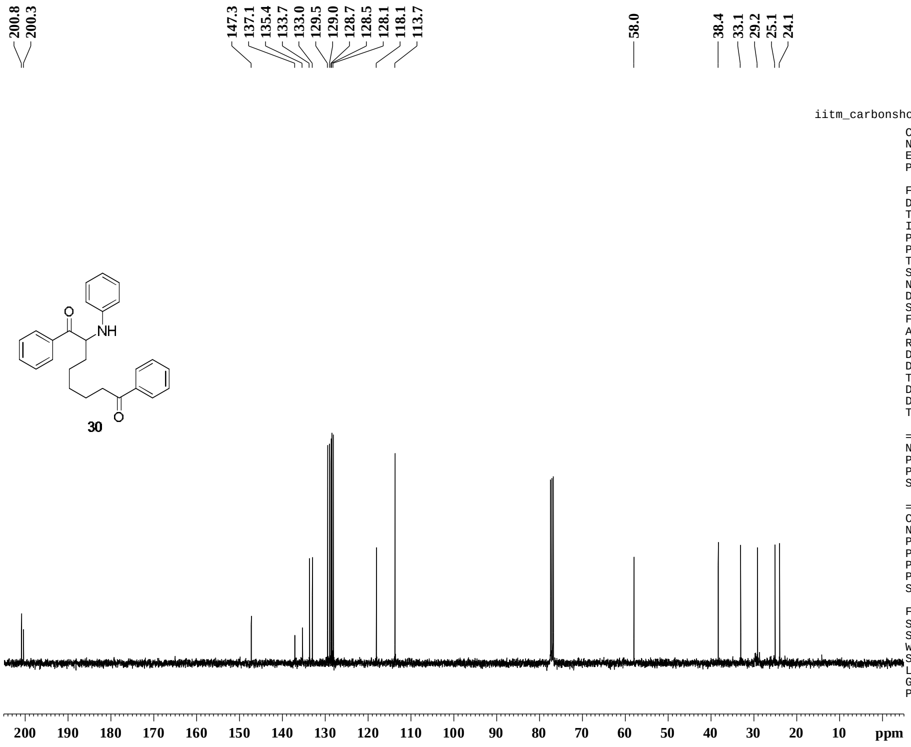


```
          lab mmb-ram-214 pem mono
iitm_carbonshort CDCl3 /opt/topspin nmr 13
             Current Data Parameters
             NAME           mmb40715
             EXPNO                99
             PROCNO                1

             F2 - Acquisition Parameters
             Date_          20150717
             Time              16.09
             INSTRUM           spect
             PROBHD   5 mm PABBO BB-
             PULPROG          zgpg30
             TD                16540
             SOLVENT           CDCl3
             NS                  256
             DS                    4
             SWH           24038.461 Hz
             FIDRES         1.453353 Hz
             AQ            0.3440320 sec
             RG               200.34
             DW               20.800 usec
             DE                 6.50 usec
             TE                298.5 K
             D1           1.00000000 sec
             D11          0.03000000 sec
             TD0                   1

             ======== CHANNEL f1 ========
             NUC1                13C
             P1                 9.25 usec
             PLW1        47.00000000 W
             SFO1        100.6228289 MHz

             ======== CHANNEL f2 ========
             CPDPRG[2        waltz16
             NUC2                 1H
             PCPD2             90.00 usec
             PLW2         7.75000000 W
             PLW12        0.23583999 W
             PLW13        0.19103000 W
             SFO2        400.1316005 MHz

             F2 - Processing parameters
             SI                32768
             SF          100.6127583 MHz
             WDW                  EM
             SSB      0
             LB                 1.00 Hz
             GB       0
             PC                 1.40
```



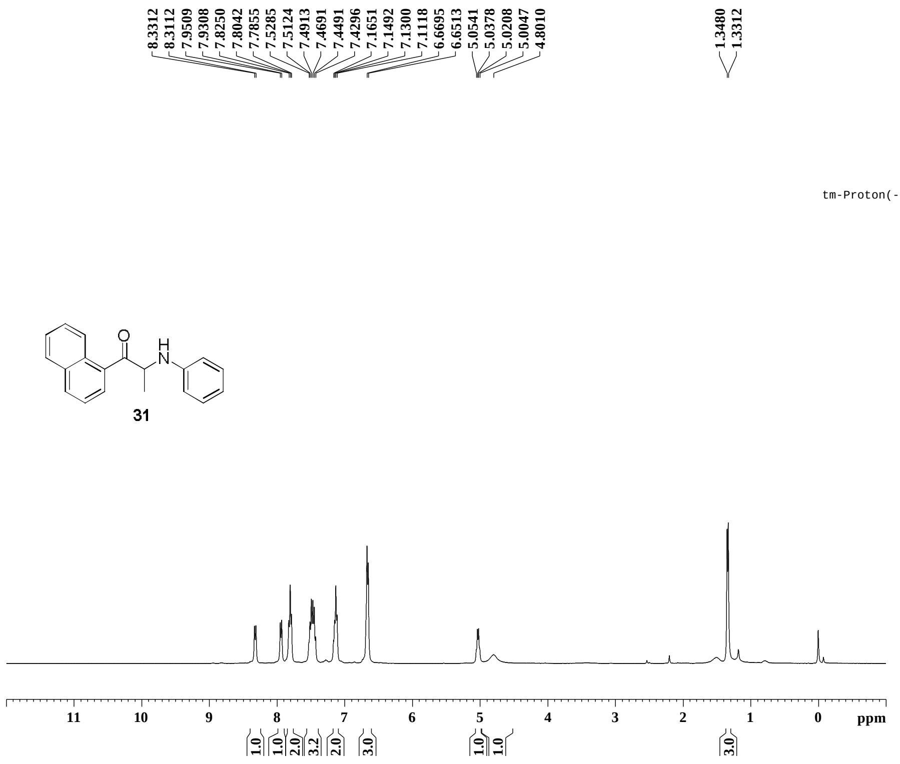
30

```
lab mmb-ram-239
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME     ram 239 and 240 up
EXPNO               145
PROCNO                1

F2 - Acquisition Parameters
Date_          20150822
Time              17.36
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               124.58
DW               62.400 use
DE                 6.50 use
TE                299.6 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300094 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

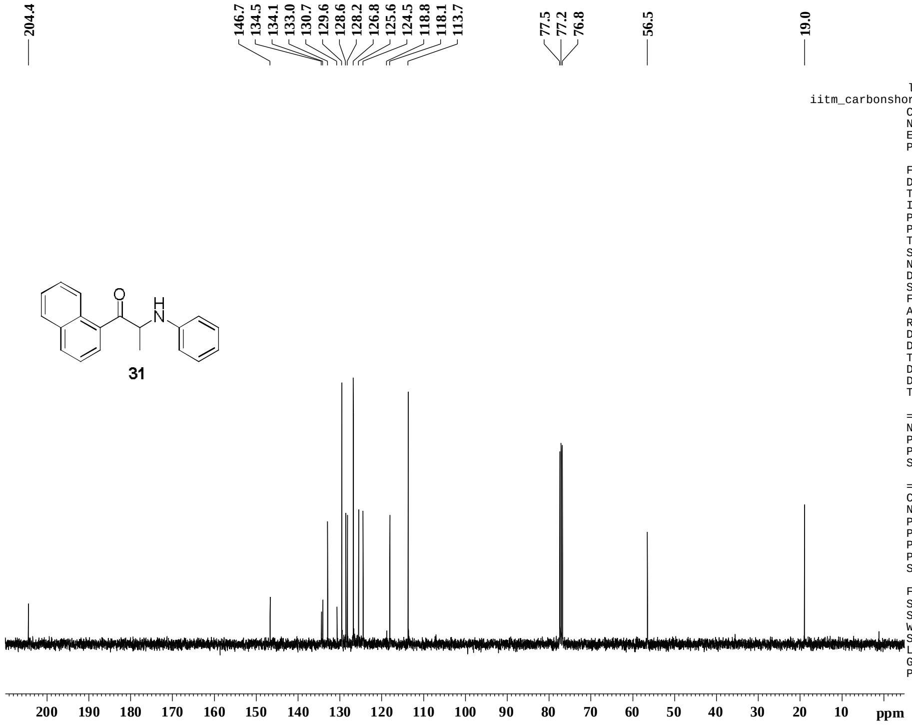


30

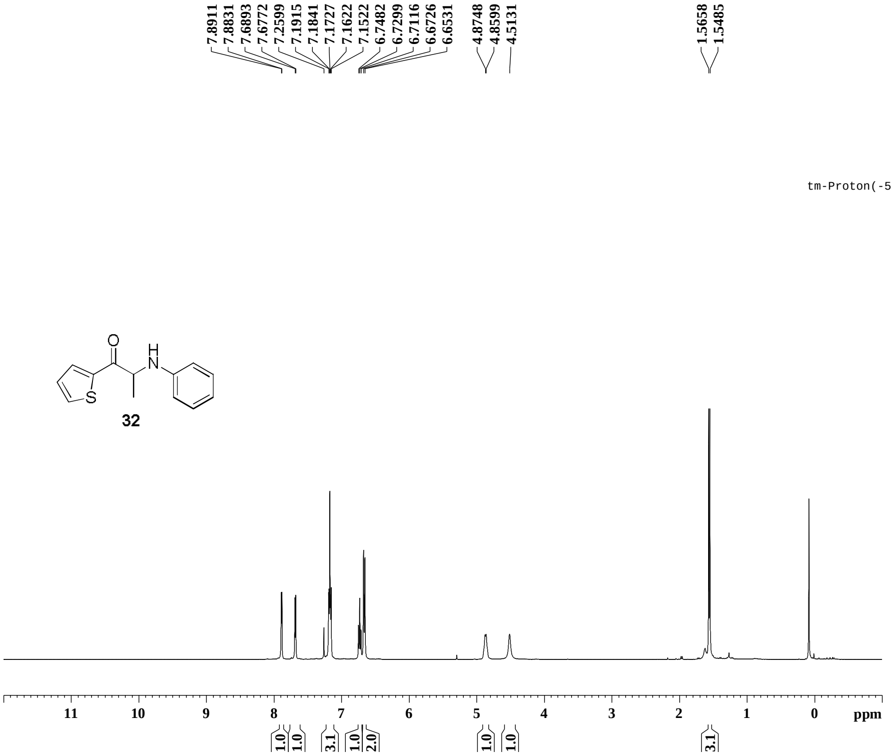
```
lab mmb-ram-239
iitm_carbonshort CDCl3 /opt/topspin nmr 13

Current Data Parameters
NAME     ram 239 and 240 up
EXPNO               146
PROCNO                1

F2 - Acquisition Parameters
Date_          20150822
Time              17.44
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                300.2 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127572 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



```
      lab mmbram=269
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1
Current Data Parameters
NAME          mmb40915
EXPNO              180
PROCNO               1

F2 - Acquisition Parameters
Date_         20150928
Time             22.25
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              138.85
DW              62.400 use
DE                6.50 use
TE               298.0 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300469 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

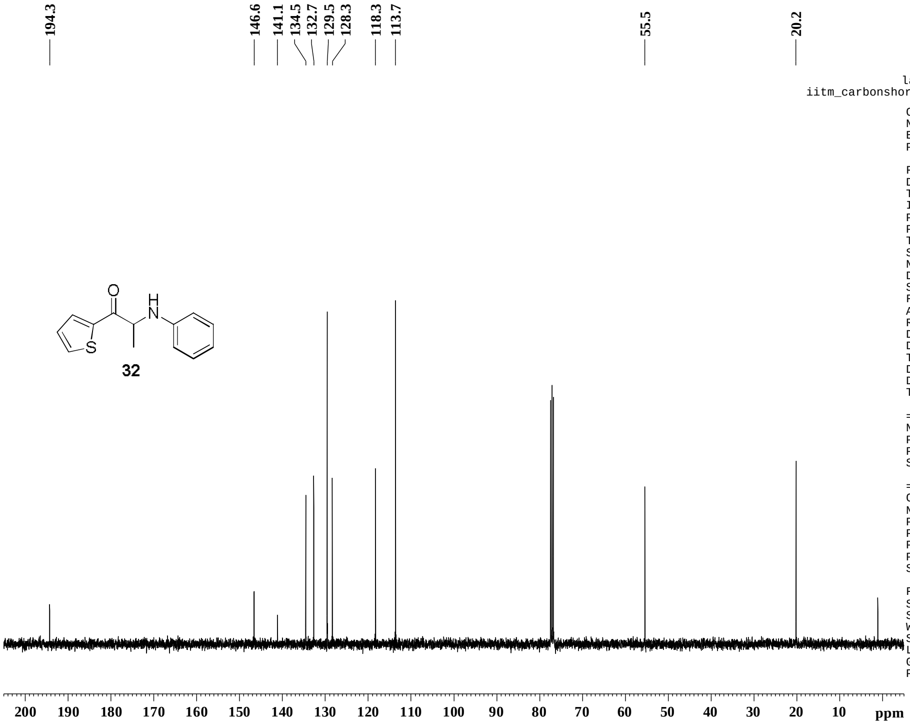


```
          lab mmbram=269
iitm_carbonshort CDCl3 /opt/topspin nmr 14
Current Data Parameters
NAME          mmb40915
EXPNO              181
PROCNO               1

F2 - Acquisition Parameters
Date_         20150928
Time             22.33
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               298.8 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127573 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



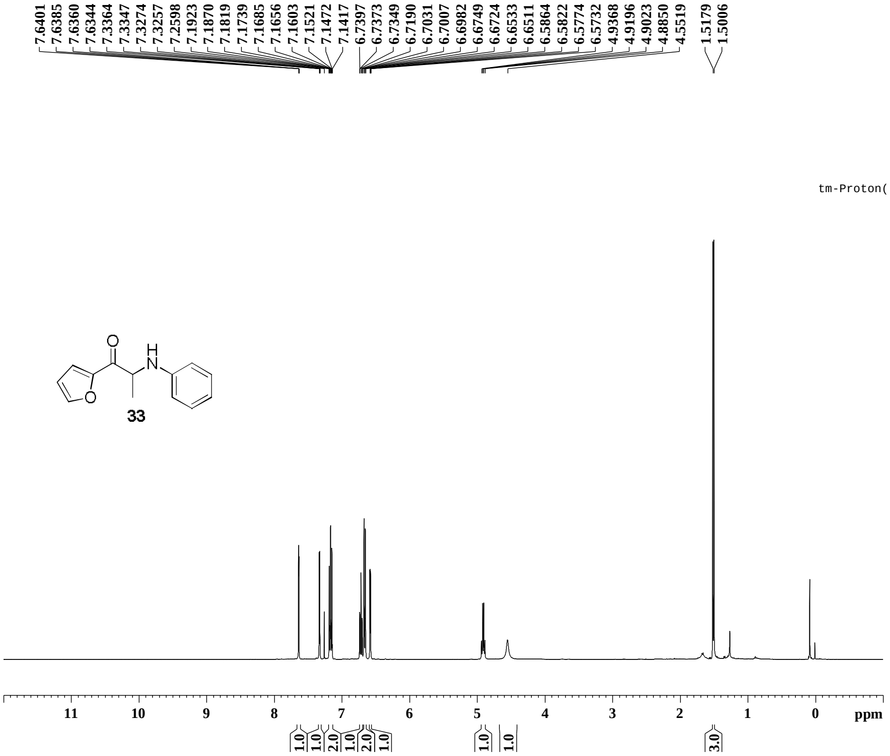
32

```
        lab mmb-ram-207
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME            mmb40715
EXPNO                103
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150717
Time               16.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                153.13
DW                62.400 use
DE                  6.50 use
TE                 298.2 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300094 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

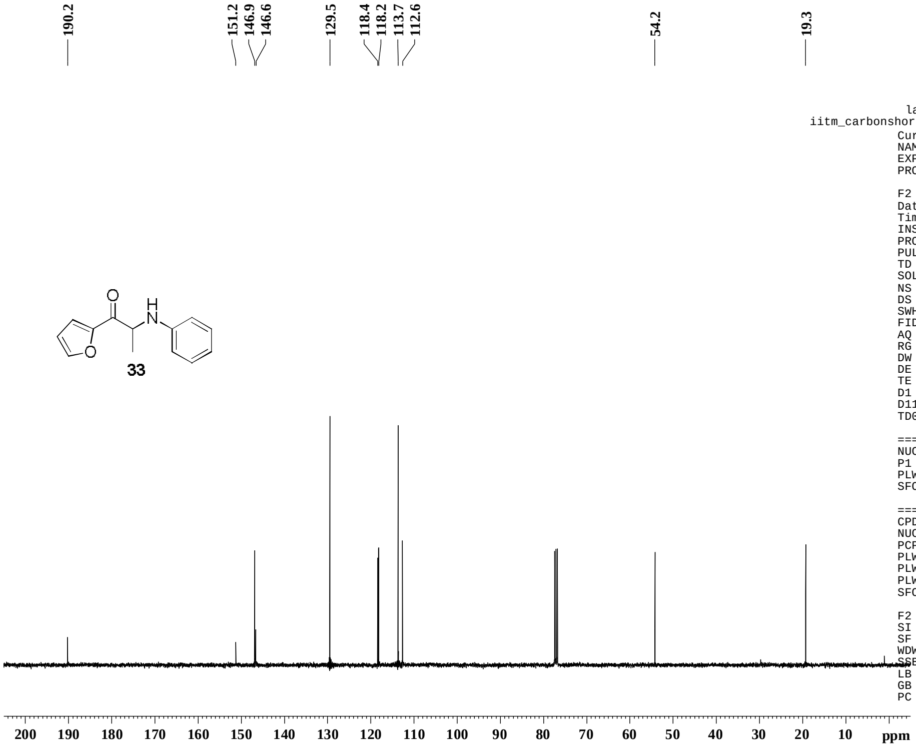


32

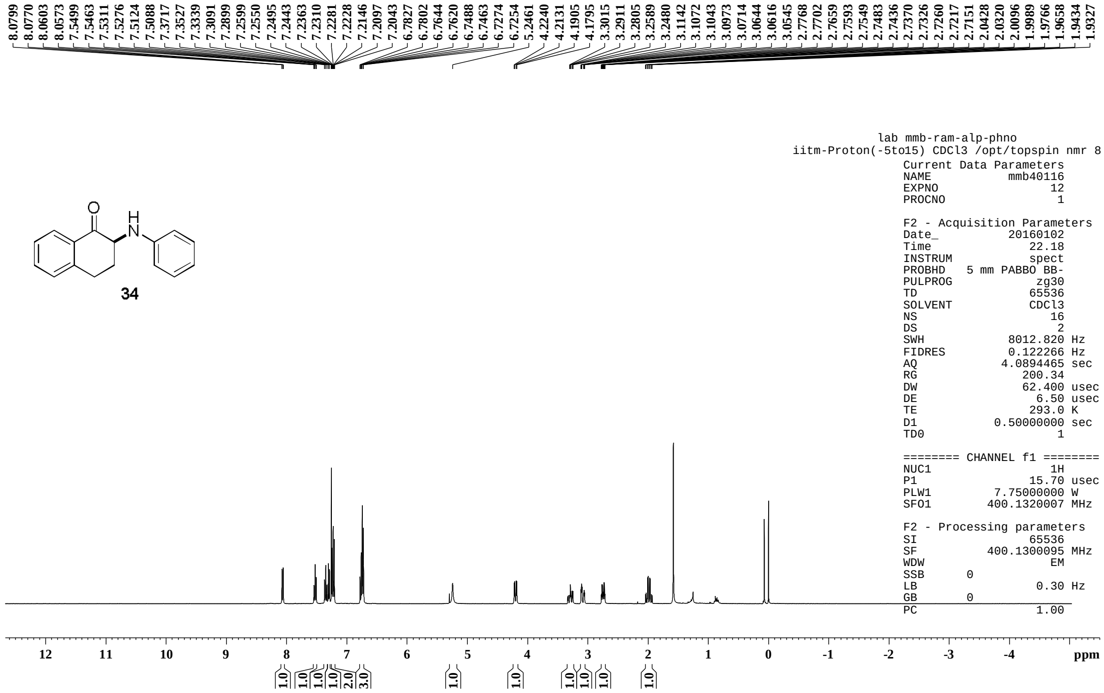
```
        lab mmb-ram-207
iitm_carbonshort CDCl3 /opt/topspin nmr 14

Current Data Parameters
NAME            mmb40715
EXPNO                104
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150717
Time               16.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   231
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 298.6 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127576 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



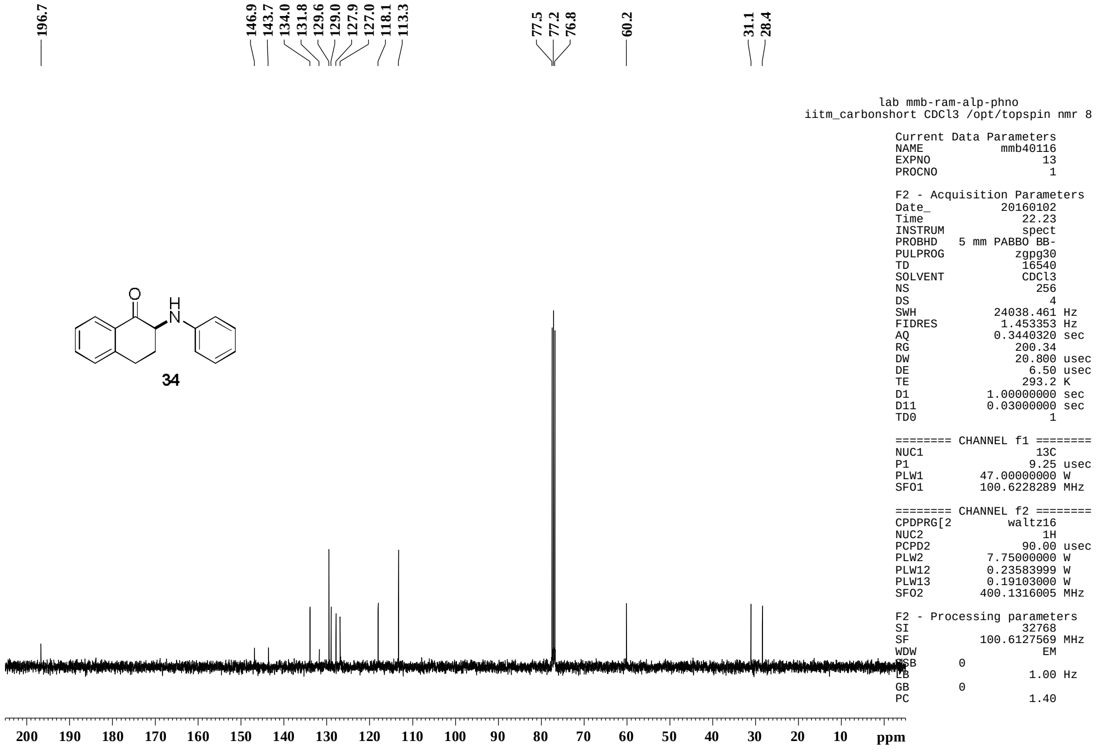
```
          lab mmb-ram-236
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1
         Current Data Parameters
         NAME            mmb40815
         EXPNO                103
         PROCNO                 1

         F2 - Acquisition Parameters
         Date_           20150819
         Time               22.01
         INSTRUM            spect
         PROBHD   5 mm PABBO BB-
         PULPROG             zg30
         TD                 65536
         SOLVENT            CDCl3
         NS                    16
         DS                     2
         SWH             8012.820 Hz
         FIDRES          0.122266 Hz
         AQ             4.0894465 sec
         RG                108.26
         DW                62.400 use
         DE                  6.50 use
         TE                 298.7 K
         D1            0.50000000 sec
         TD0                    1

         ======== CHANNEL f1 =======
         NUC1                  1H
         P1                 15.70 use
         PLW1          7.75000000 W
         SFO1         400.1320007 MHz

         F2 - Processing parameters
         SI                 65536
         SF           400.1300094 MHz
         WDW                   EM
         SSB      0
         LB                  0.30 Hz
         GB       0
         PC                  1.00
```



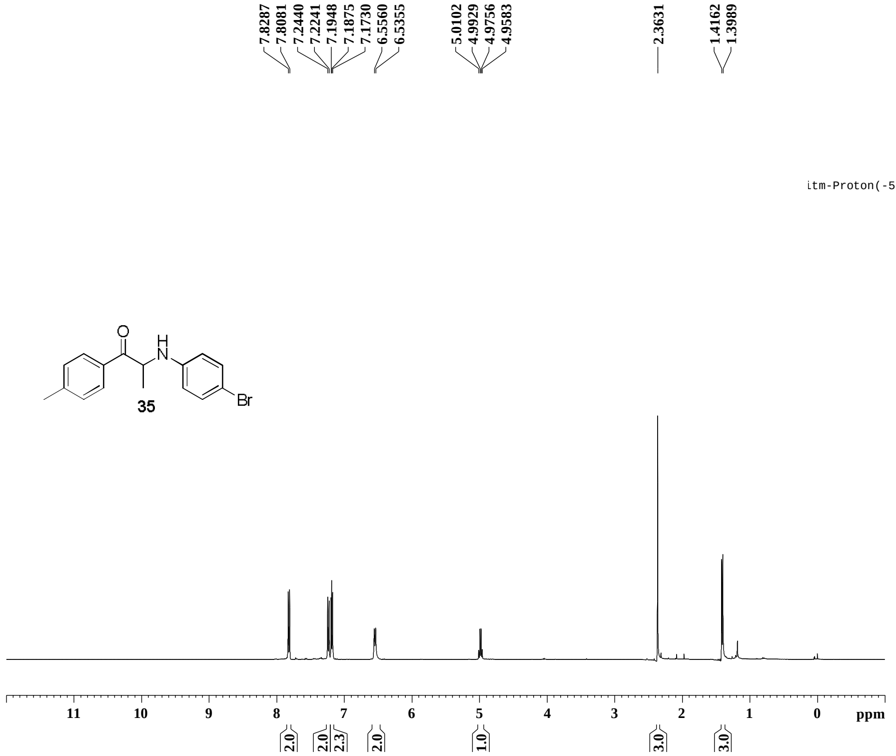
```
          lab mmb-ram-236
iitm_carbonshort CDCl3 /opt/topspin nmr 16
         Current Data Parameters
         NAME            mmb40815
         EXPNO                104
         PROCNO                 1

         F2 - Acquisition Parameters
         Date_           20150819
         Time               22.11
         INSTRUM            spect
         PROBHD   5 mm PABBO BB-
         PULPROG          zgpg30
         TD                 16540
         SOLVENT            CDCl3
         NS                   256
         DS                     4
         SWH            24038.461 Hz
         FIDRES          1.453353 Hz
         AQ             0.3440320 sec
         RG                200.34
         DW                20.800 usec
         DE                  6.50 usec
         TE                 299.4 K
         D1            1.00000000 sec
         D11           0.03000000 sec
         TD0                    1

         ======== CHANNEL f1 ========
         NUC1                 13C
         P1                  9.25 usec
         PLW1         47.00000000 W
         SFO1         100.6228289 MHz

         ======== CHANNEL f2 ========
         CPDPRG[2         waltz16
         NUC2                  1H
         PCPD2              90.00 usec
         PLW2          7.75000000 W
         PLW12         0.23583999 W
         PLW13         0.19103000 W
         SFO2         400.1316005 MHz

         F2 - Processing parameters
         SI                 32768
         SF           100.6127590 MHz
         WDW                   EM
         SSB      0
         LB                  1.00 Hz
         GB       0
         PC                  1.40
```



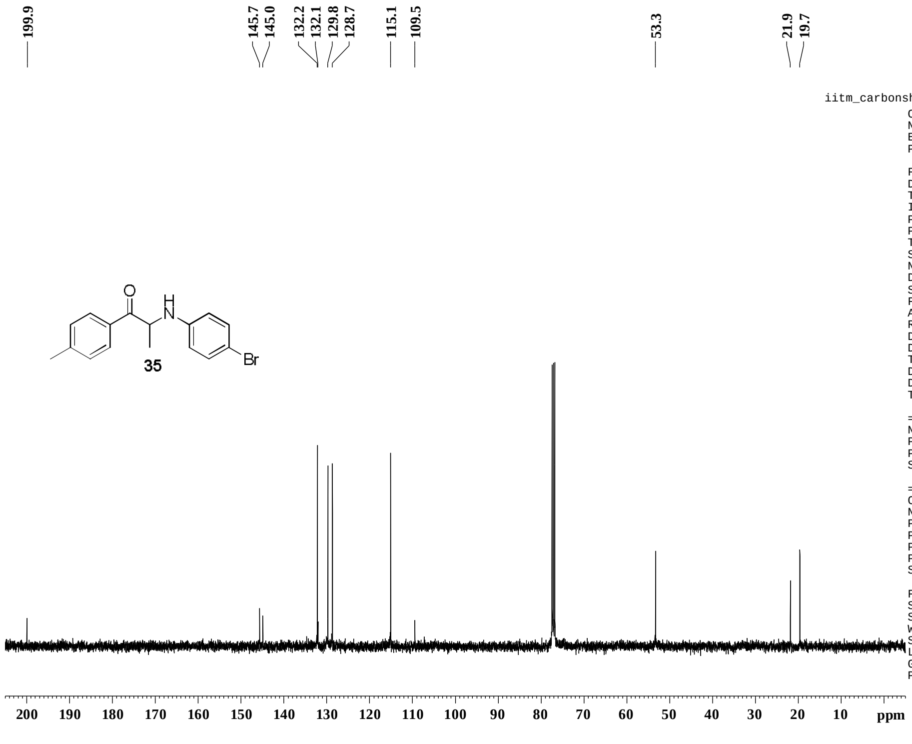
```
                  lab mmb-ram-alp-phno
iitm-Proton(-5to15) CDCl3 /opt/topspin nmr 8
                Current Data Parameters
                NAME           mmb40116
                EXPNO                12
                PROCNO                1

                F2 - Acquisition Parameters
                Date_          20160102
                Time              22.18
                INSTRUM           spect
                PROBHD   5 mm PABBO BB-
                PULPROG            zg30
                TD                65536
                SOLVENT           CDCl3
                NS                   16
                DS                    2
                SWH            8012.820 Hz
                FIDRES         0.122266 Hz
                AQ            4.0894465 sec
                RG               200.34
                DW               62.400 usec
                DE                 6.50 usec
                TE                293.0 K
                D1           0.50000000 sec
                TD0                   1

                ======== CHANNEL f1 ========
                NUC1                 1H
                P1                15.70 usec
                PLW1         7.75000000 W
                SFO1        400.1320007 MHz

                F2 - Processing parameters
                SI                65536
                SF          400.1300095 MHz
                WDW                  EM
                SSB      0
                LB                 0.30 Hz
                GB       0
                PC                 1.00
```



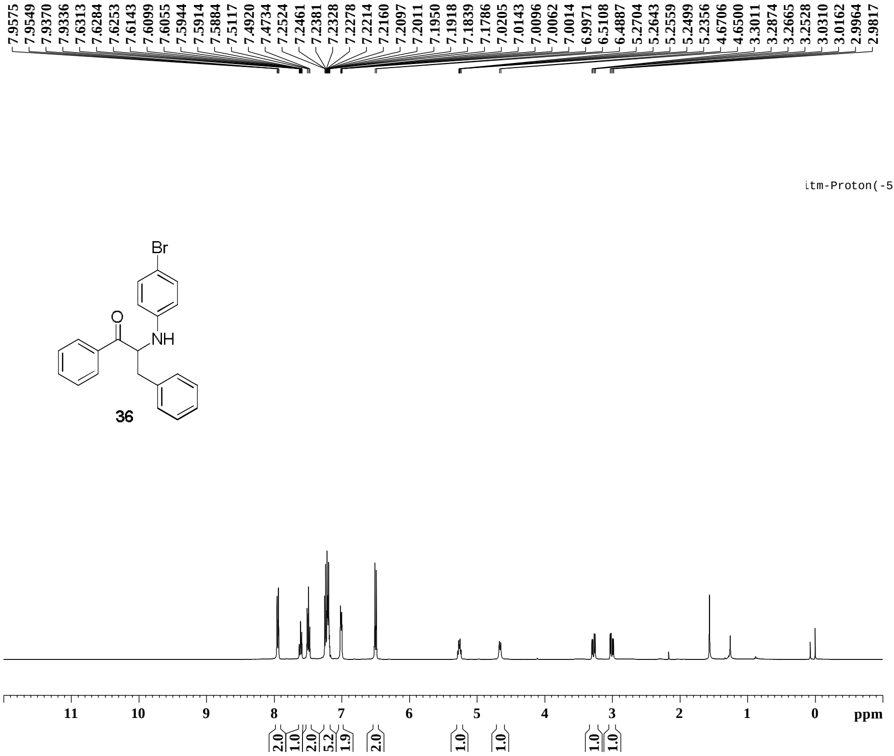
```
                  lab mmb-ram-alp-phno
     iitm_carbonshort CDCl3 /opt/topspin nmr 8

                Current Data Parameters
                NAME           mmb40116
                EXPNO                13
                PROCNO                1

                F2 - Acquisition Parameters
                Date_          20160102
                Time              22.23
                INSTRUM           spect
                PROBHD   5 mm PABBO BB-
                PULPROG          zgpg30
                TD                16540
                SOLVENT           CDCl3
                NS                  256
                DS                    4
                SWH           24038.461 Hz
                FIDRES         1.453353 Hz
                AQ            0.3440320 sec
                RG               200.34
                DW               20.800 usec
                DE                 6.50 usec
                TE                293.2 K
                D1           1.00000000 sec
                D11          0.03000000 sec
                TD0                   1

                ======== CHANNEL f1 ========
                NUC1                13C
                P1                 9.25 usec
                PLW1        47.00000000 W
                SFO1        100.6228289 MHz

                ======== CHANNEL f2 ========
                CPDPRG[2        waltz16
                NUC2                 1H
                PCPD2             90.00 usec
                PLW2         7.75000000 W
                PLW12        0.23583999 W
                PLW13        0.19103000 W
                SFO2        400.1316005 MHz

                F2 - Processing parameters
                SI                32768
                SF          100.6127569 MHz
                WDW                  EM
                SSB      0
                LB                 1.00 Hz
                GB       0
                PC                 1.40
```



```
          lab mmbram-289
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb41015
EXPNO                161
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151026
Time               12.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                169.77
DW                62.400 use
DE                  6.50 use
TE                 295.0 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300385 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```

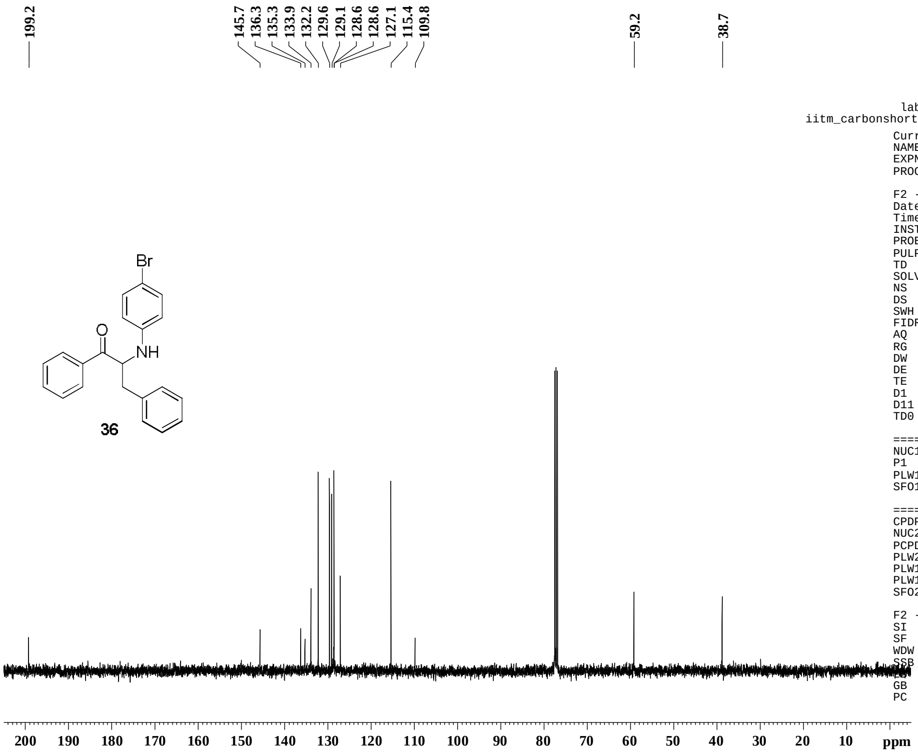


```
          lab mmb-ram-202
iitm_carbonshort CDCl3 /opt/topspin nmr 2

Current Data Parameters
NAME          New Folder
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150705
Time               16.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 16540
SOLVENT            CDCl3
NS                  1000
DS                     4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1         100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127551 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



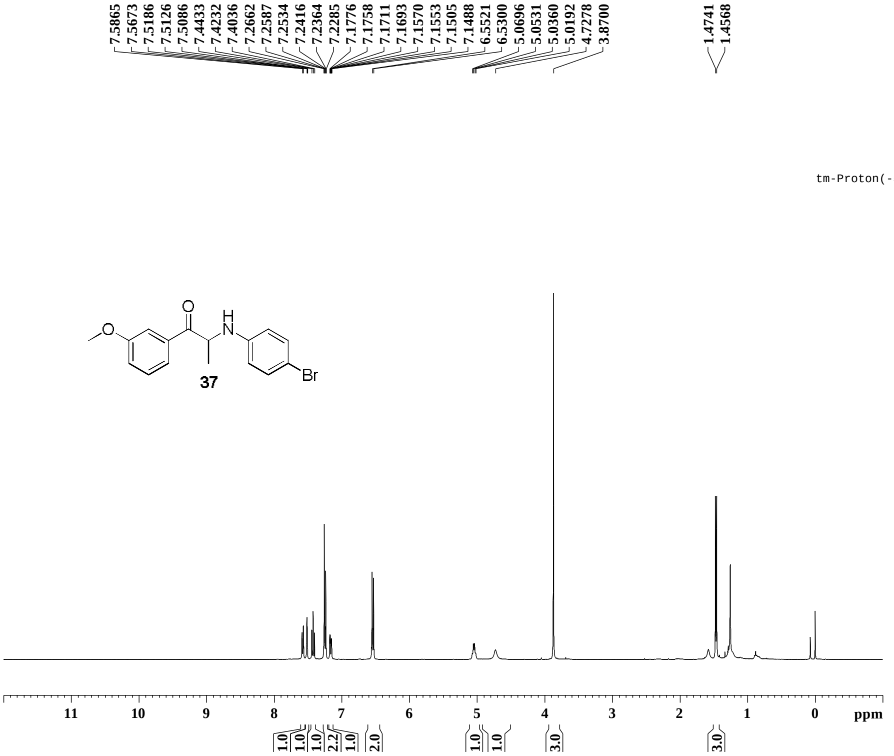
36

```
          lab mmbram-279
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME            mmb40915
EXPNO                204
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150930
Time               14.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                169.77
DW                62.400 use
DE                  6.50 use
TE                 298.3 K
D1            0.50000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 15.70 use
PLW1          7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300125 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



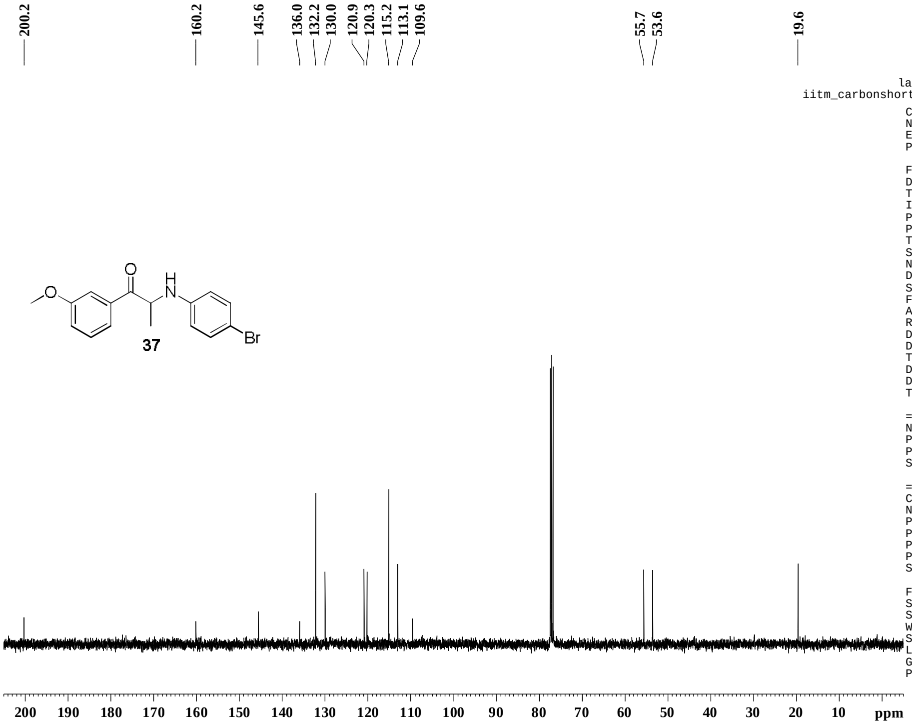
36

```
          lab mmbram-279
iitm_carbonshort CDCl3 /opt/topspin nmr 9

Current Data Parameters
NAME            mmb40915
EXPNO                205
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150930
Time               14.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 16540
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            24038.461 Hz
FIDRES          1.453353 Hz
AQ             0.3440320 sec
RG                200.34
DW                20.800 usec
DE                  6.50 usec
TE                 299.1 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PLW1         47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          7.75000000 W
PLW12         0.23583999 W
PLW13         0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127555 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



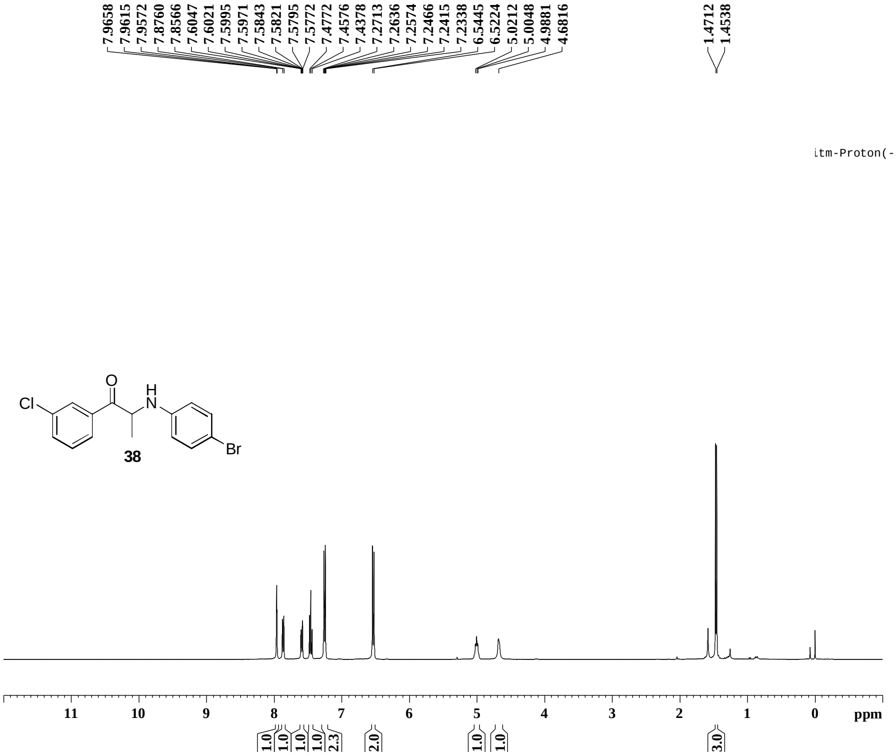
37

```
         lab mmb-ram-254
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME          mmb41115
EXPNO              211
PROCNO               1

F2 - Acquisition Parameters
Date_          20151128
Time              14.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               200.34
DW               62.400 use
DE                 6.50 use
TE                297.3 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300099 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

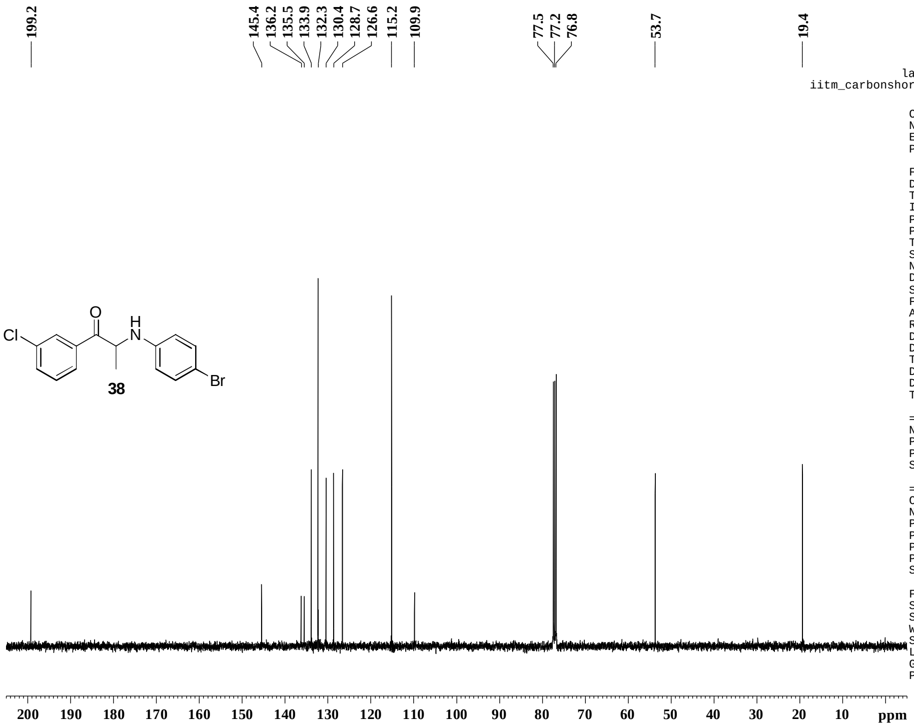


37

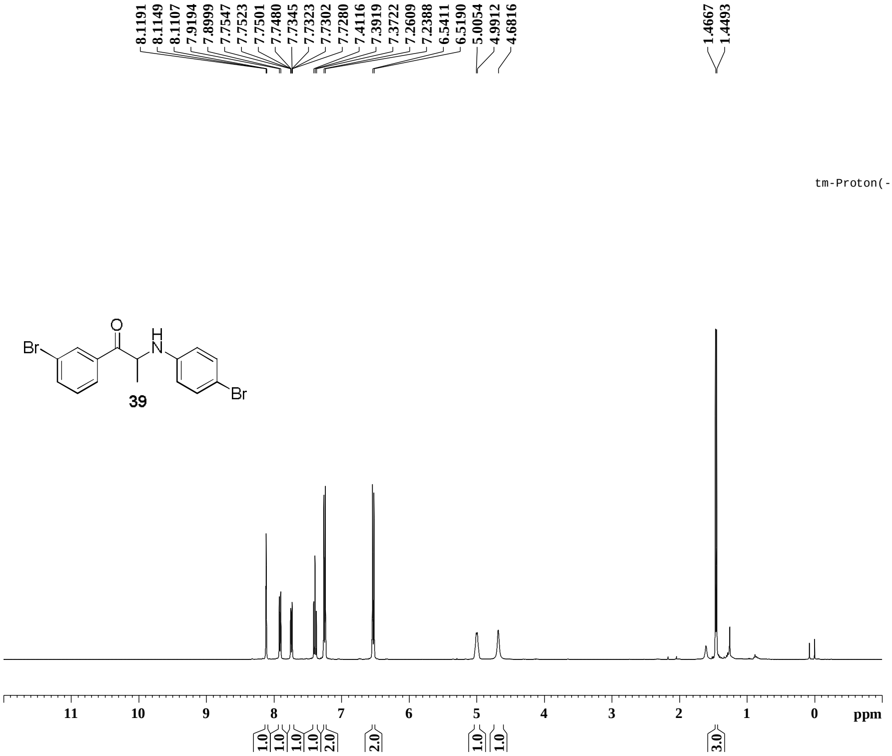
```
         lab mmb-ram-254
iitm_carbonshort CDCl3 /opt/topspin nmr 10

Current Data Parameters
NAME          mmb41115
EXPNO              212
PROCNO               1

F2 - Acquisition Parameters
Date_          20151128
Time              15.00
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                297.8 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127560 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



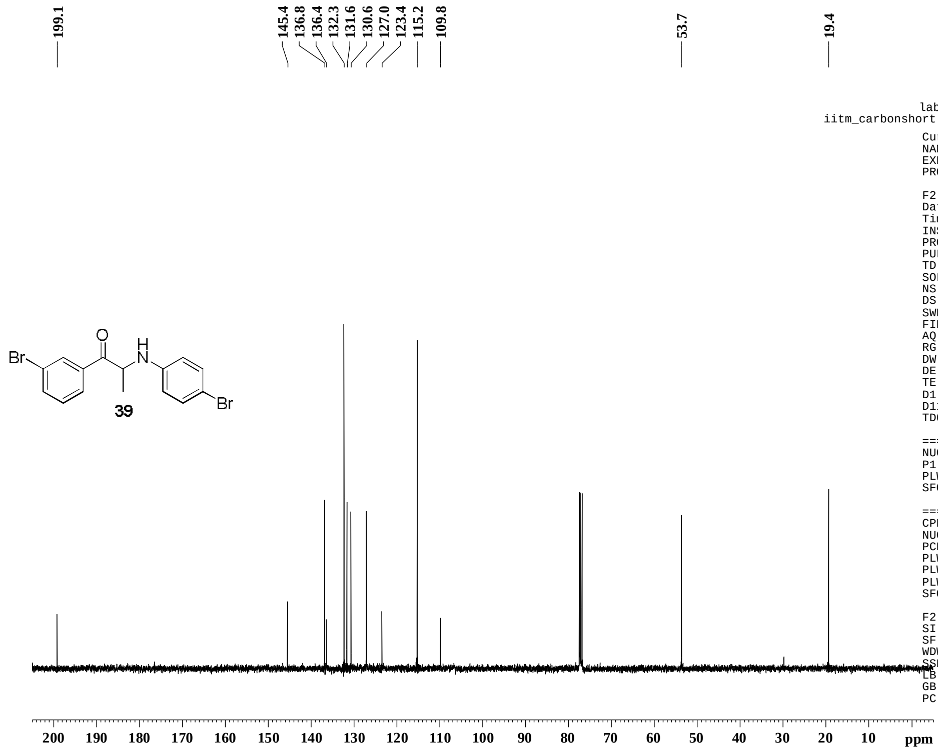
```
           lab mmb-ram-261
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb40915
EXPNO               88
PROCNO               1

F2 - Acquisition Parameters
Date_         20150915
Time             12.29
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              169.77
DW              62.400 use
DE                6.50 use
TE               298.6 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300107 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



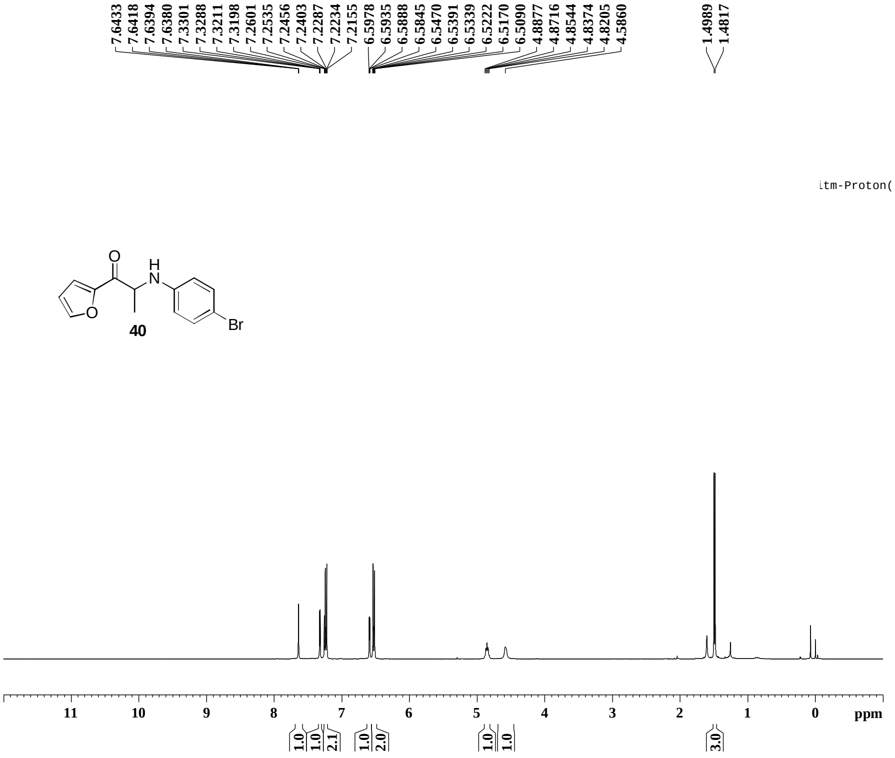
```
           lab mmb-ram-261
iitm_carbonshort CDCl3 /opt/topspin nmr 9

Current Data Parameters
NAME          mmb40915
EXPNO               89
PROCNO               1

F2 - Acquisition Parameters
Date_         20150915
Time             12.35
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               299.2 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127561 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



```
     lab mmb-ram-249
tm-Proton(-5to15) CDCl3 /opt/topspin nmr 1

Current Data Parameters
NAME          mmb40815
EXPNO              280
PROCNO               1

F2 - Acquisition Parameters
Date_         20150831
Time             18.45
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              124.58
DW              62.400 use
DE                6.50 use
TE               296.8 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300107 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

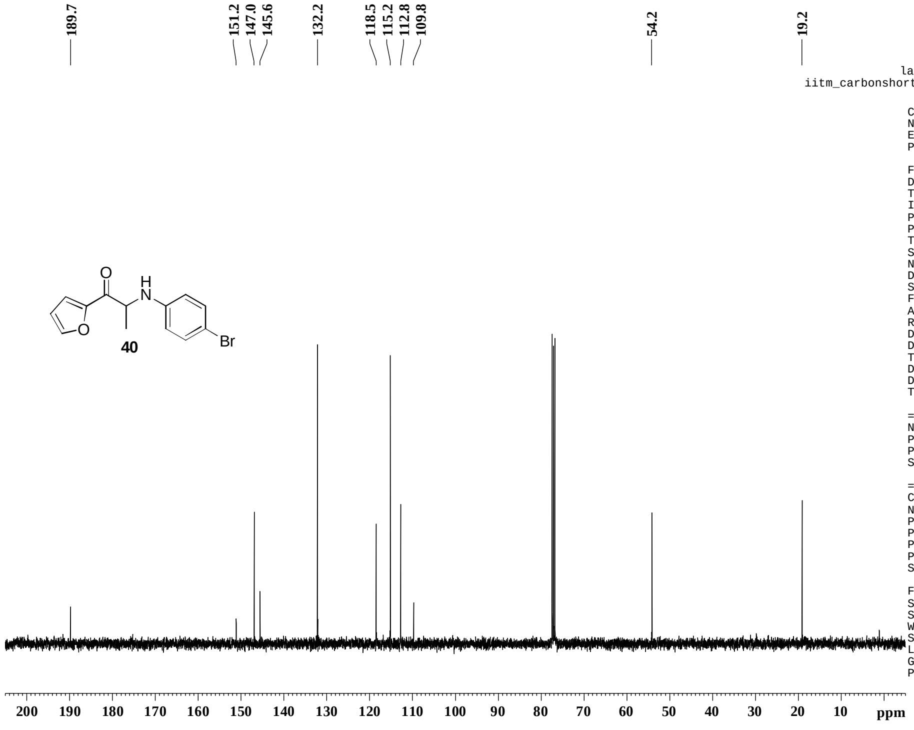


```
     lab mmb-ram-249
iitm_carbonshort CDCl3 /opt/topspin nmr 10
Current Data Parameters
NAME          mmb40815
EXPNO              281
PROCNO               1

F2 - Acquisition Parameters
Date_         20150831
Time             18.52
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               297.6 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2       waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127583 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



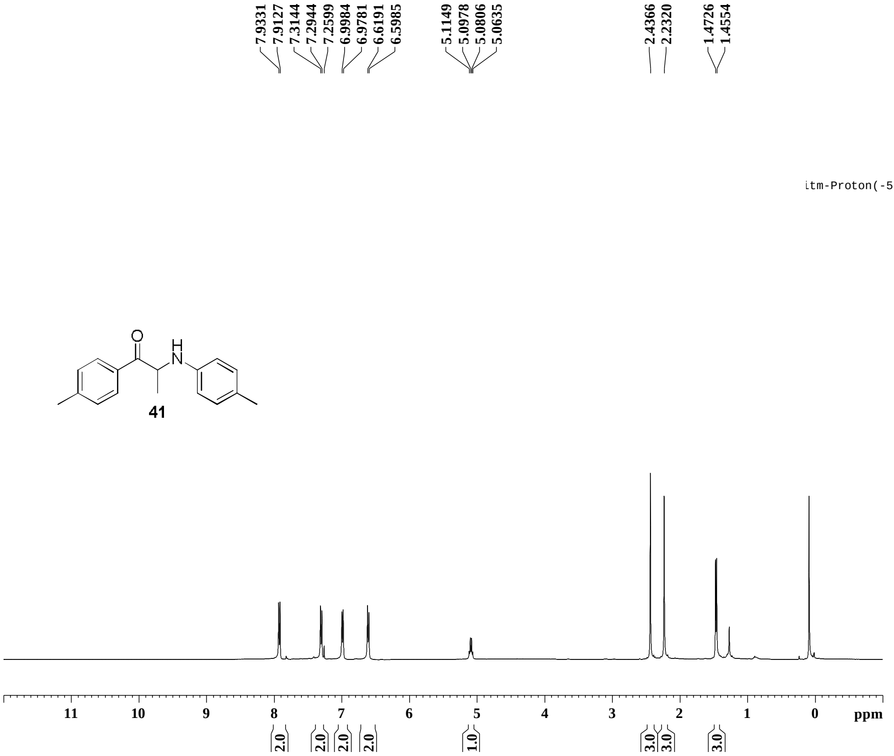
40

```
       lab mmb-ram-237
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME     ram-237 and 240
EXPNO              149
PROCNO               1

F2 - Acquisition Parameters
Date_          20150822
Time              22.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               200.34
DW               62.400 use
DE                 6.50 use
TE                299.4 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                15.70 use
PLW1         7.75000000 W
SFO1        400.1320007 MHz

F2 - Processing parameters
SI                65536
SF          400.1300092 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

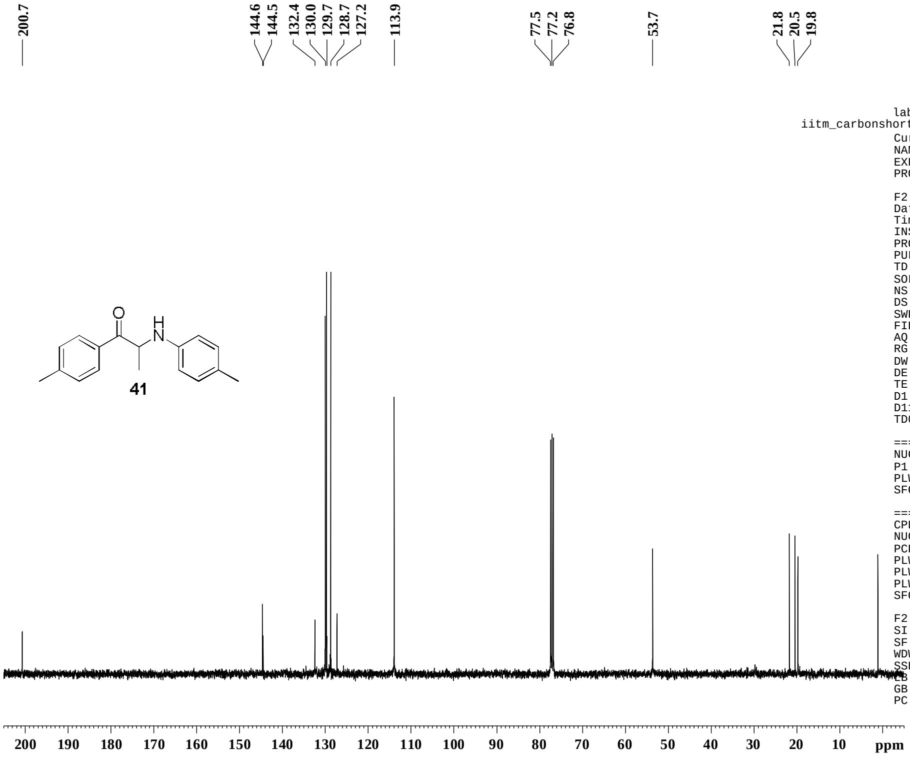


40

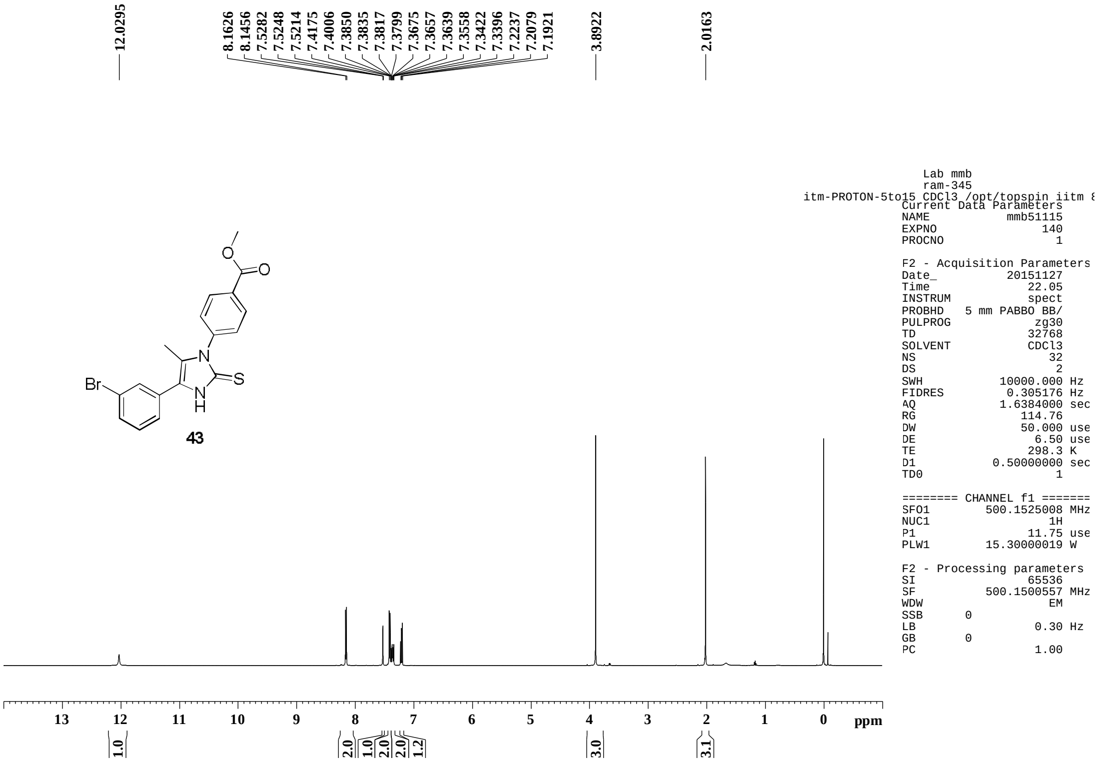
```
           lab mmb-ram-237
iitm_carbonshort CDCl3 /opt/topspin nmr 11

Current Data Parameters
NAME     ram-237 and 240
EXPNO              164
PROCNO               1

F2 - Acquisition Parameters
Date_          20150824
Time               0.04
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                16540
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         1.453353 Hz
AQ            0.3440320 sec
RG               200.34
DW               20.800 usec
DE                 6.50 usec
TE                300.1 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.25 usec
PLW1        47.00000000 W
SFO1        100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         7.75000000 W
PLW12        0.23583999 W
PLW13        0.19103000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127558 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```



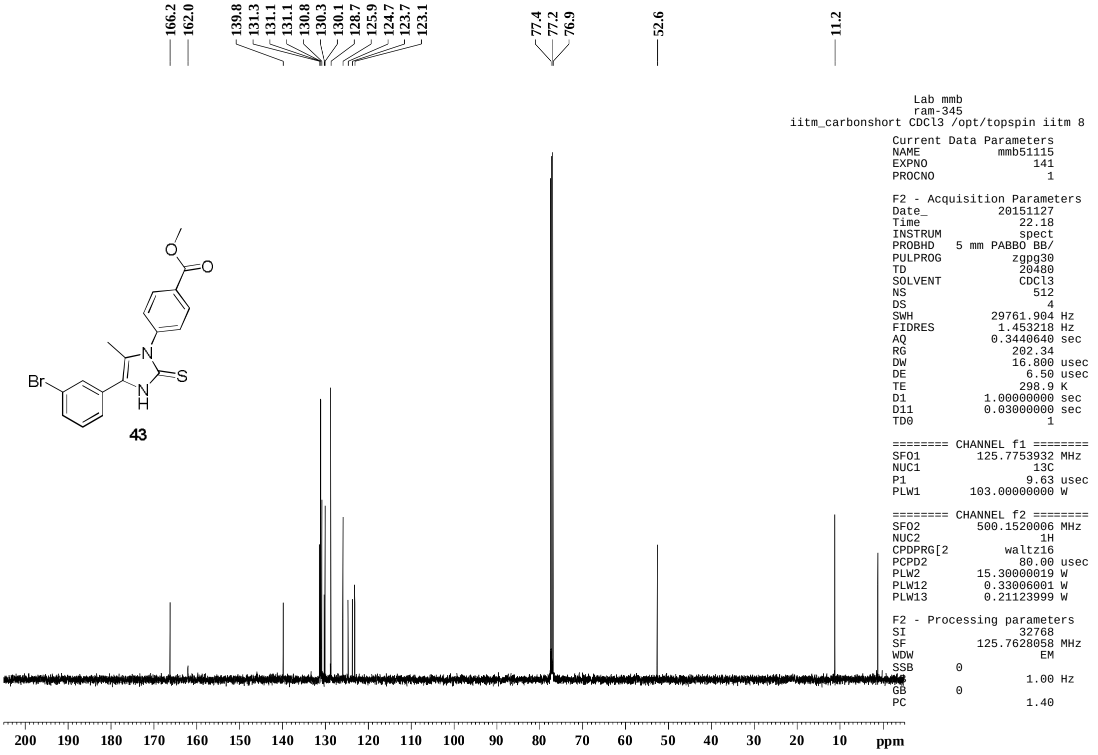
```
             lab mmb-ram-205
itm-Proton(-5to15) CDCl3 /opt/topspin nmr
Current Data Parameters
NAME              ram-205
EXPNO                  48
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150706
Time                23.36
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                  95.73
DW                 62.400 use
DE                   6.50 use
TE                  297.2 K
D1             0.50000000 sec
TD0                     1

======== CHANNEL f1 =======
NUC1                   1H
P1                  15.70 use
PLW1           7.75000000 W
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300092 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



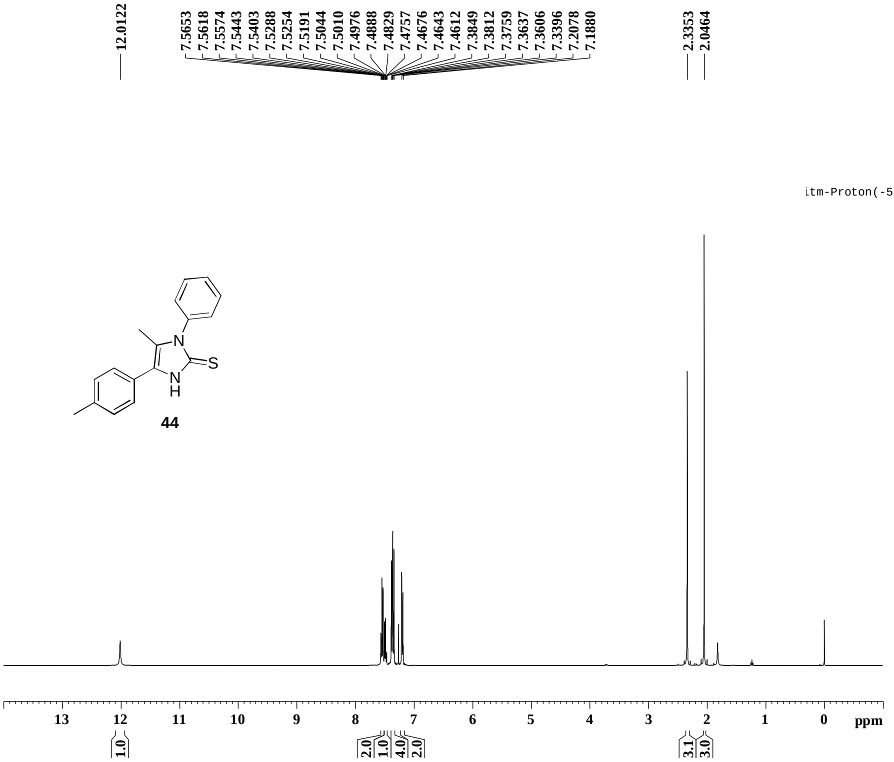
```
             lab mmb-ram-205
iitm_carbonshort CDCl3 /opt/topspin nmr 8
Current Data Parameters
NAME              ram-205
EXPNO                  49
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150706
Time                23.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  16540
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES           1.453353 Hz
AQ              0.3440320 sec
RG                 200.34
DW                 20.800 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.25 usec
PLW1          47.00000000 W
SFO1          100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2           7.75000000 W
PLW12          0.23583999 W
PLW13          0.19103000 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127577 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



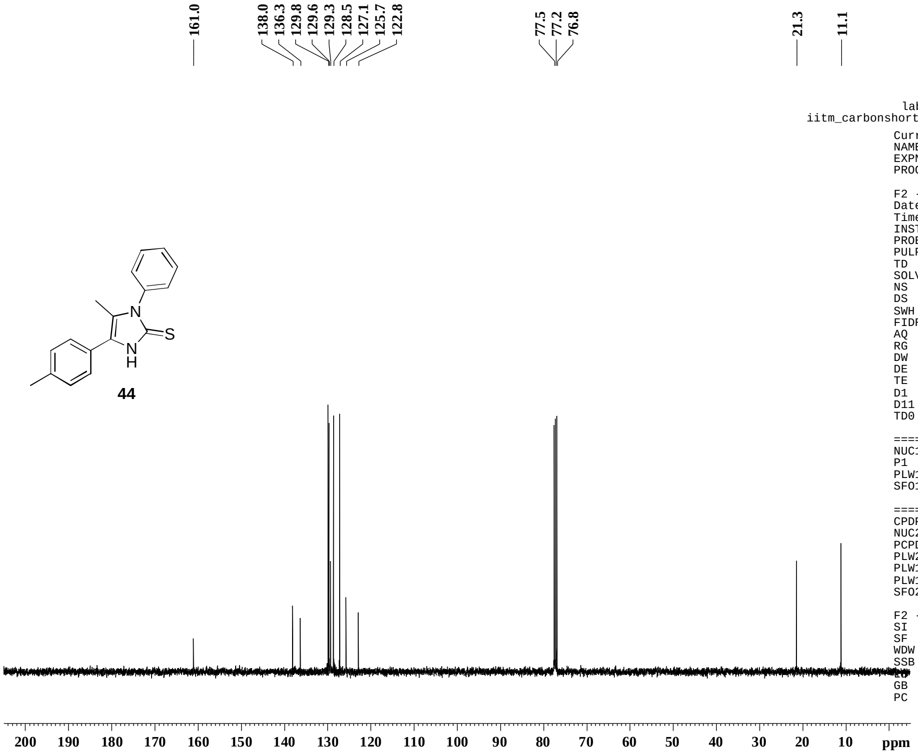
```
   Lab mmb
   ram-345
itm-PROTON-5to15 CDCl3 /opt/topspin iitm 8
Current Data Parameters
NAME          mmb51115
EXPNO              140
PROCNO               1

F2 - Acquisition Parameters
Date_         20151127
Time             22.05
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  32
DS                   2
SWH          10000.000 Hz
FIDRES        0.305176 Hz
AQ           1.6384000 sec
RG              114.76
DW              50.000 use
DE                6.50 use
TE               298.3 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
SFO1       500.1525008 MHz
NUC1                1H
P1               11.75 use
PLW1       15.30000019 W

F2 - Processing parameters
SI               65536
SF         500.1500557 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



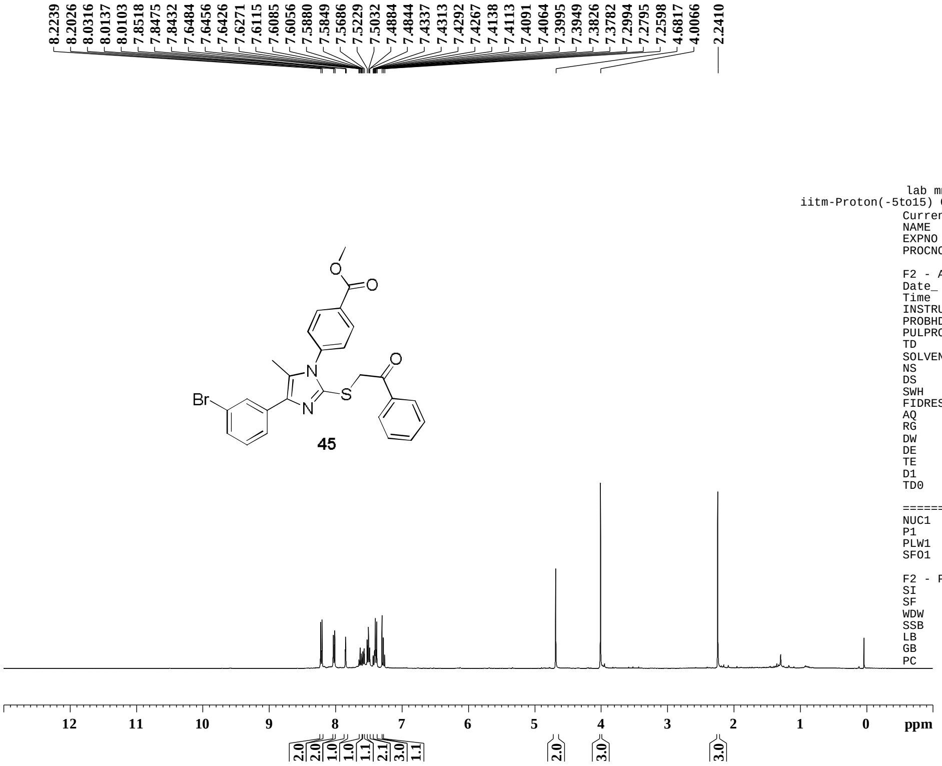
```
   Lab mmb
   ram-345
iitm_carbonshort CDCl3 /opt/topspin iitm 8

Current Data Parameters
NAME          mmb51115
EXPNO              141
PROCNO               1

F2 - Acquisition Parameters
Date_         20151127
Time             22.18
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD               20480
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          29761.904 Hz
FIDRES        1.453218 Hz
AQ           0.3440640 sec
RG              202.34
DW              16.800 usec
DE                6.50 usec
TE               298.9 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       125.7753932 MHz
NUC1               13C
P1                9.63 usec
PLW1      103.00000000 W

======== CHANNEL f2 ========
SFO2       500.1520006 MHz
NUC2                1H
CPDPRG[2       waltz16
PCPD2            80.00 usec
PLW2       15.30000019 W
PLW12       0.33006001 W
PLW13       0.21123999 W

F2 - Processing parameters
SI               32768
SF         125.7628058 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



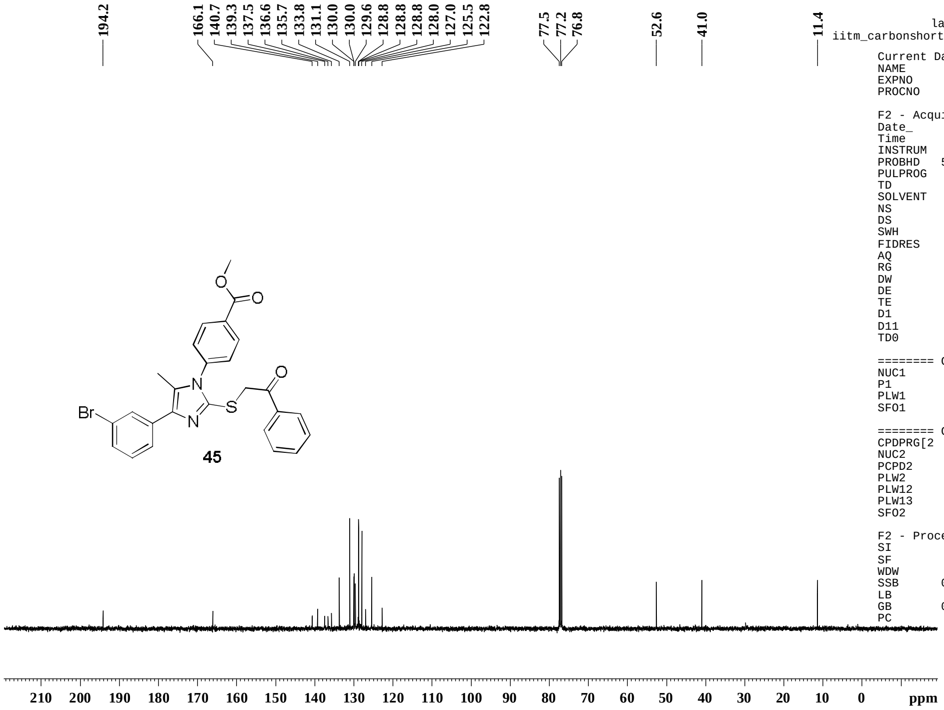
```
         lab mmbRam-327
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb41115
EXPNO              100
PROCNO               1

F2 - Acquisition Parameters
Date_         20151110
Time             11.25
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              138.85
DW              62.400 use
DE                6.50 use
TE               290.6 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300096 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



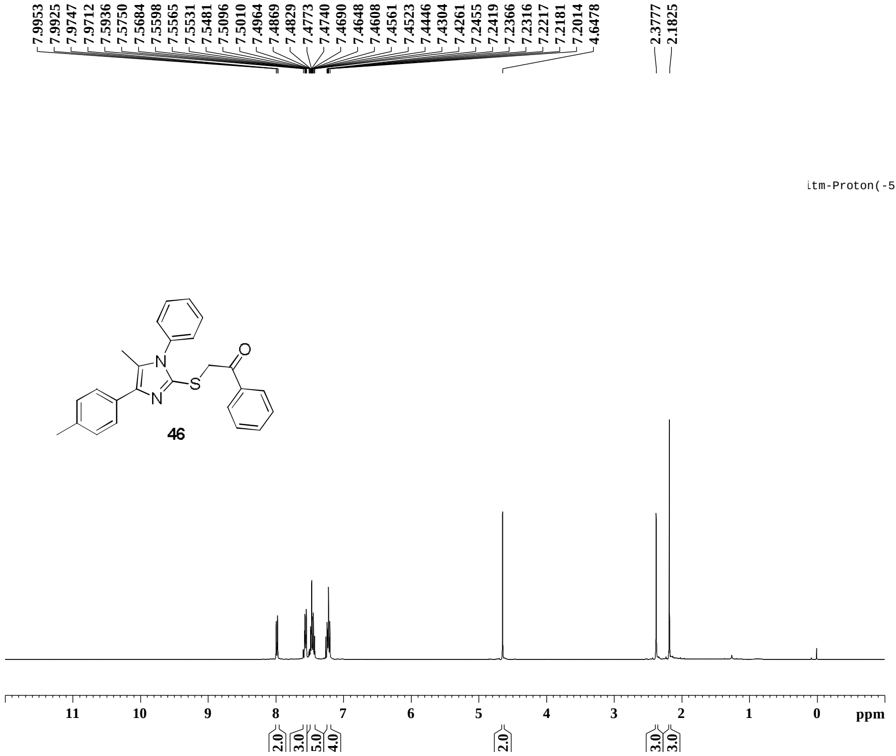
```
         lab mmbRam-327
iitm_carbonshort CDCl3 /opt/topspin nmr 2

Current Data Parameters
NAME          mmb41115
EXPNO              101
PROCNO               1

F2 - Acquisition Parameters
Date_         20151110
Time             11.32
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               291.4 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127622 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



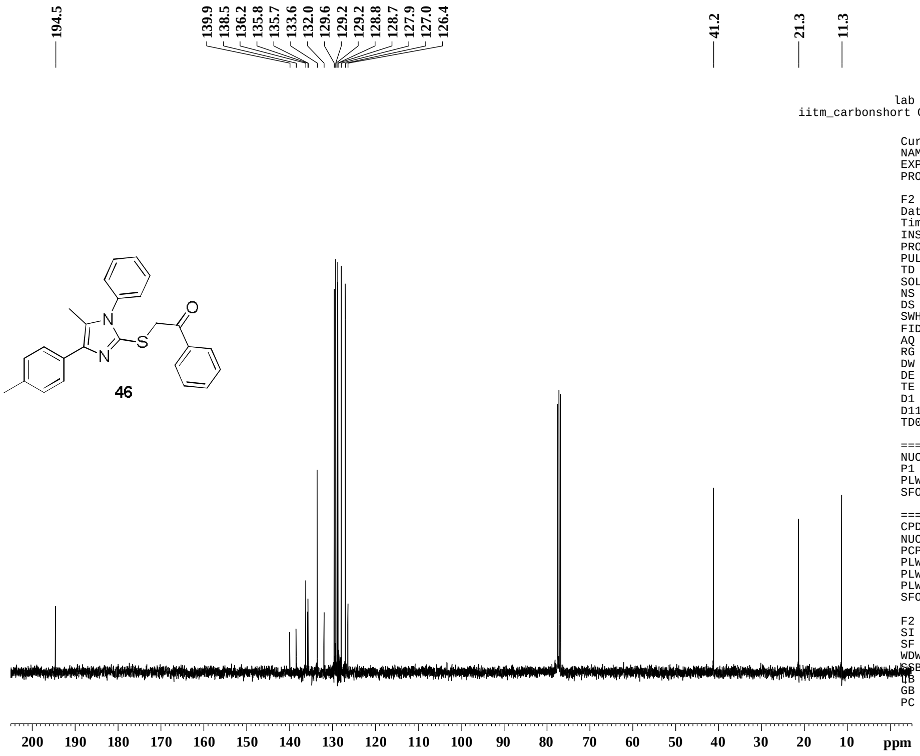
```
  lab mmbram-346
iitm-Proton(-5to15) CDCl3 /opt/topspin nmr 7
         Current Data Parameters
         NAME             ram-346
         EXPNO                 12
         PROCNO                 1

         F2 - Acquisition Parameters
         Date_           20151209
         Time               23.48
         INSTRUM            spect
         PROBHD   5 mm PABBO BB-
         PULPROG             zg30
         TD                 65536
         SOLVENT            CDCl3
         NS                    16
         DS                     2
         SWH             8012.820 Hz
         FIDRES          0.122266 Hz
         AQ             4.0894465 sec
         RG                169.77
         DW                62.400 usec
         DE                  6.50 usec
         TE                 295.2 K
         D1            0.50000000 sec
         TD0                    1

         ======== CHANNEL f1 ========
         NUC1                  1H
         P1                 15.70 usec
         PLW1          7.75000000 W
         SFO1        400.1320007 MHz

         F2 - Processing parameters
         SI                 65536
         SF           400.1299936 MHz
         WDW                   EM
         SSB      0
         LB                  0.30 Hz
         GB       0
         PC                  1.00
```



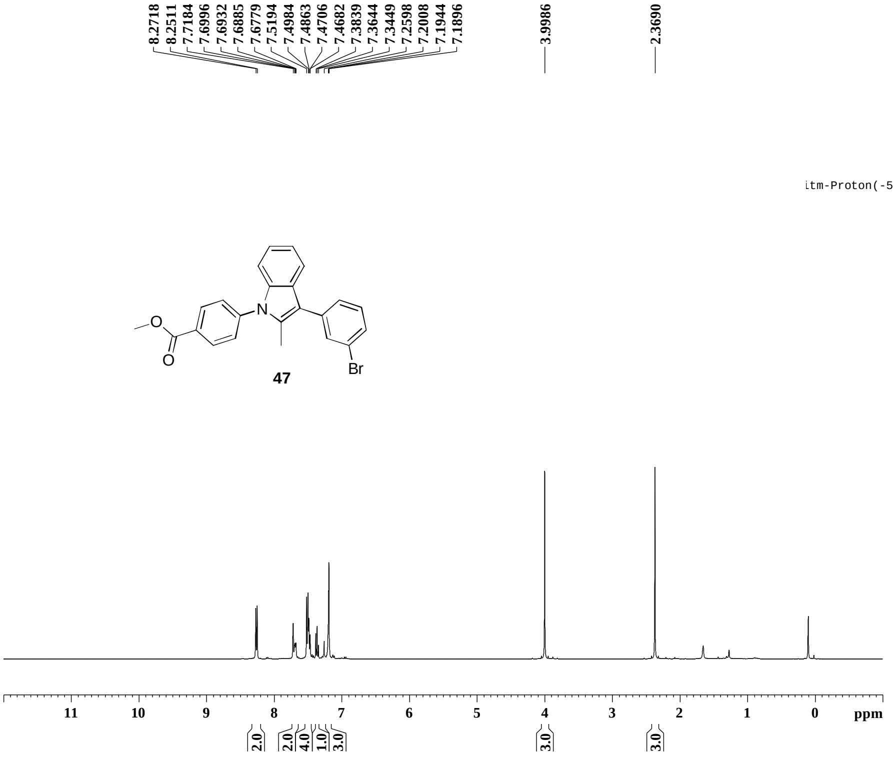
```
    lab mmbram-346
iitm_carbonshort CDCl3 /opt/topspin nmr 11

     Current Data Parameters
     NAME            mmb41215
     EXPNO                 24
     PROCNO                 1

     F2 - Acquisition Parameters
     Date_           20151210
     Time               17.58
     INSTRUM            spect
     PROBHD   5 mm PABBO BB-
     PULPROG           zgpg30
     TD                 16540
     SOLVENT            CDCl3
     NS                   700
     DS                     4
     SWH            24038.461 Hz
     FIDRES          1.453353 Hz
     AQ             0.3440320 sec
     RG                200.34
     DW                20.800 usec
     DE                  6.50 usec
     TE                 295.1 K
     D1            1.00000000 sec
     D11           0.03000000 sec
     TD0                    1

     ======== CHANNEL f1 ========
     NUC1                 13C
     P1                  9.25 usec
     PLW1         47.00000000 W
     SFO1        100.6228289 MHz

     ======== CHANNEL f2 ========
     CPDPRG[2         waltz16
     NUC2                  1H
     PCPD2              90.00 usec
     PLW2          7.75000000 W
     PLW12         0.23583999 W
     PLW13         0.19103000 W
     SFO2        400.1316005 MHz

     F2 - Processing parameters
     SI                 32768
     SF           100.6127569 MHz
     WDW                   EM
     SSB      0
     LB                  1.00 Hz
     GB       0
     PC                  1.40
```



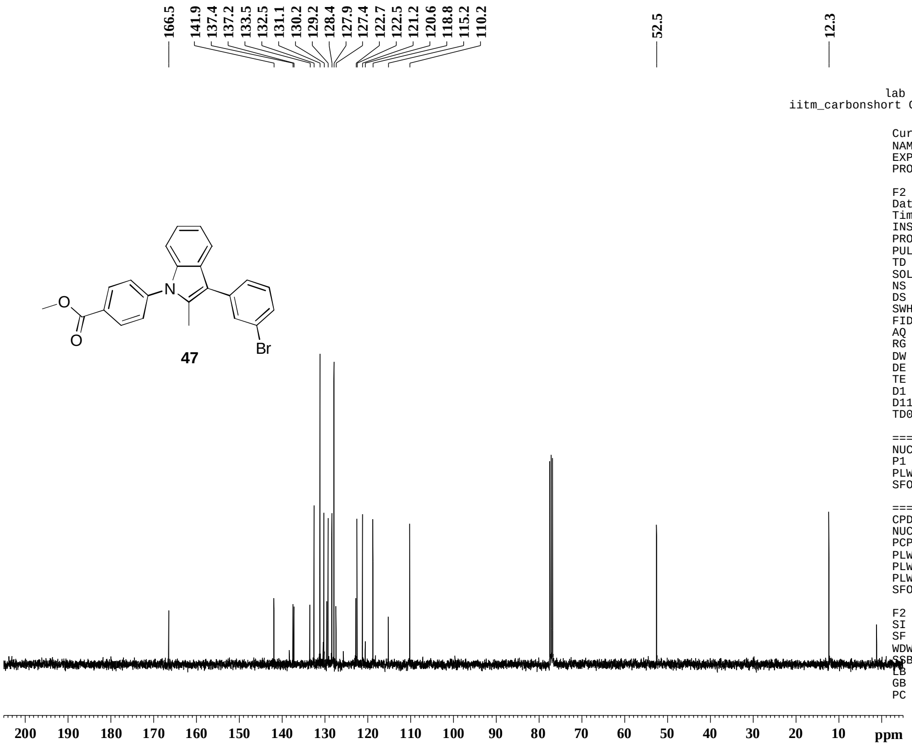
```
      lab mmb-ram-337
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb41115
EXPNO              197
PROCNO               1

F2 - Acquisition Parameters
Date_         20151120
Time              0.14
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              108.26
DW              62.400 use
DE                6.50 use
TE               291.9 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1      400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300096 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



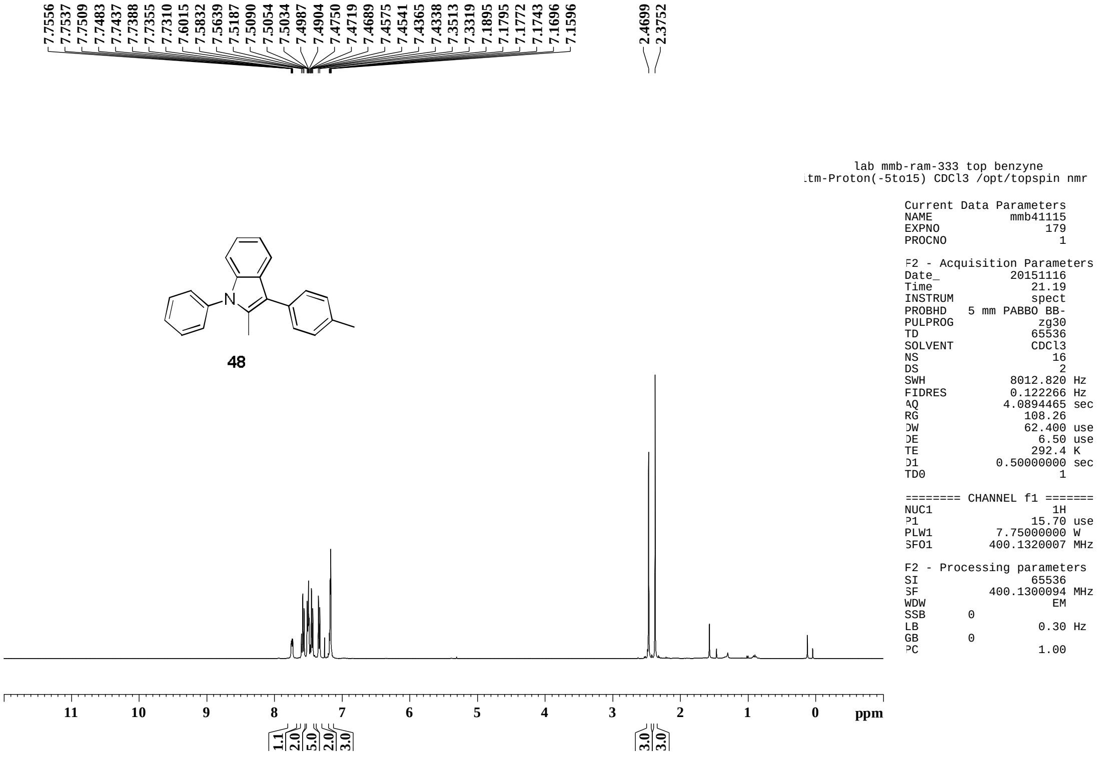
```
        lab mmb-ram-337
iitm_carbonshort CDCl3 /opt/topspin nmr 13

Current Data Parameters
NAME          mmb41115
EXPNO              207
PROCNO               1

F2 - Acquisition Parameters
Date_         20151120
Time              2.45
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               292.3 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1      100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2      400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127612 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



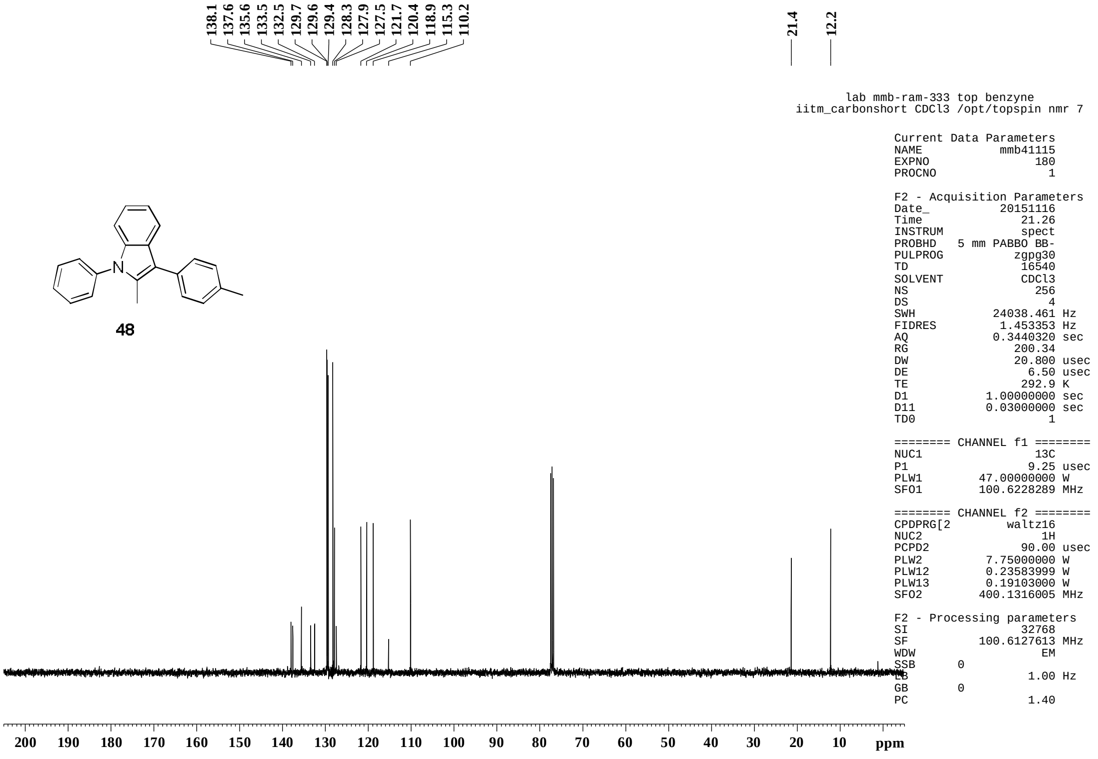
```
              lab mmb-ram-336
itm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb41115
EXPNO              166
PROCNO               1

F2 - Acquisition Parameters
Date_         20151120
Time              0.07
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              108.26
DW              62.400 use
DE                6.50 use
TE               291.9 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300093 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



```
              lab mmb-ram-336
iitm_carbonshort CDCl3 /opt/topspin nmr 12

Current Data Parameters
NAME          mmb41115
EXPNO              205
PROCNO               1

F2 - Acquisition Parameters
Date_         20151120
Time              2.32
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               292.3 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127618 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```



```
 lab mmb-ram-333 top benzyne
tm-Proton(-5to15) CDCl3 /opt/topspin nmr

Current Data Parameters
NAME          mmb41115
EXPNO              179
PROCNO               1

F2 - Acquisition Parameters
Date_         20151116
Time             21.19
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG              108.26
DW              62.400 use
DE                6.50 use
TE               292.4 K
D1          0.50000000 sec
TD0                  1

======== CHANNEL f1 =======
NUC1                1H
P1               15.70 use
PLW1        7.75000000 W
SFO1       400.1320007 MHz

F2 - Processing parameters
SI               65536
SF         400.1300094 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



```
 lab mmb-ram-333 top benzyne
iitm_carbonshort CDCl3 /opt/topspin nmr 7

Current Data Parameters
NAME          mmb41115
EXPNO              180
PROCNO               1

F2 - Acquisition Parameters
Date_         20151116
Time             21.26
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD               16540
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        1.453353 Hz
AQ           0.3440320 sec
RG              200.34
DW              20.800 usec
DE                6.50 usec
TE               292.9 K
D1          1.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.25 usec
PLW1       47.00000000 W
SFO1       100.6228289 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        7.75000000 W
PLW12       0.23583999 W
PLW13       0.19103000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI               32768
SF         100.6127613 MHz
WDW                 EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.40
```