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Rhodium(III)-catalyzed *ortho*-C–H amidation of 2-arylindazoles with dioxazolone as an amidating reagent

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1. General Information:

All reagents were purchased from commercial sources and used without further purification. ^{1}H NMR spectra were determined on 400 MHz spectrometer as solutions in $CDCl_3$. Chemical shifts were expressed in parts per million (δ) and the signals were reported as s (singlet), br s (broad singlet), d (doublet), dd (doublet of doublet), t (triplet), q (quartet), m (multiplet), and coupling constants (J) were given in Hz. $^{13}C\{^{1}H\}$ NMR spectra were recorded at 100 MHz in $CDCl_3$ solution. Chemical shifts as internal standard were referenced to $CDCl_3$ (δ = 7.26 for ^{1}H and δ = 77.16 for $^{13}C\{^{1}H\}$ NMR) as internal standard. TLC was done on silica gel coated glass slide. All solvents were dried and distilled before use. Commercially available solvents were freshly distilled before the reaction. All reactions involving moisture sensitive reactants were executed using oven dried glassware. All the 2*H*-indazoles and dioxazolone cerivatives were prepared by the reported method.[1,2]

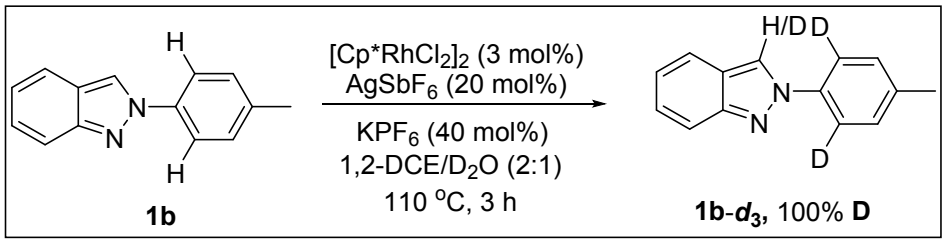
2. Typical experimental procedure for the compound *N*-(2-(2*H*-indazol-2-yl)phenyl)acetamide (3aa):

A mixture of 2-phenyl-2*H*-indazole (**1a**) (0.2 mmol, 38.8 mg), $[Cp^*RhCl_2]_2$ (3 mol%, 3.7 mg), $AgSbF_6$ (20 mol%, 13.7 mg) and KPF_6 (40 mol%, 14.7 mg) was taken in an oven dried screw-capped reaction tube. Then 1,2-DCE (2 mL) was added to the mixture and stirred for 5 min at room temperature under open atmosphere. After that, 3-methyl-1,4,2-dioxazol-5-one (**2a)** (0.3 mmol, 30 mg) was added, and the resultant mixture was stirred at 110 ^{o}C for 3 h. After completion of the reaction (TLC), the reaction was cooled to room temperature and extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4. The crude residue was obtained after evaporating the solvent in vacuum and was purified by column

chromatography on silica gel using a mixture of petroleum ether and ethyl acetate (83:17) as an eluting solvent to afford the pure product **3aa** (38 mg, 76%) as a white solid.

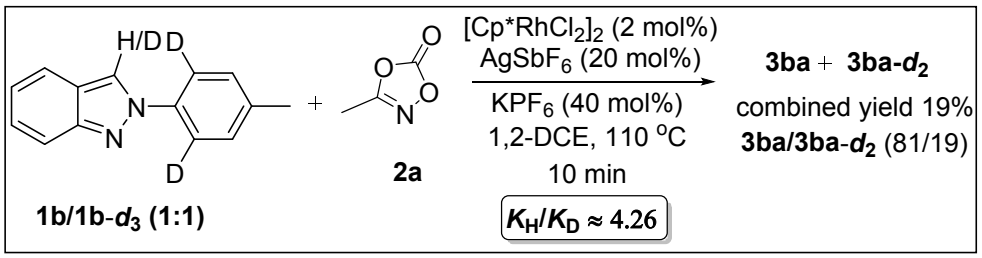
3. Mechanistic investigations:

3.1. Preparation of 2-(4-methylphenyl-2,6-*d*$_2$)-2*H*-indazole-3-*d* (1b-*d*$_3$):[1]



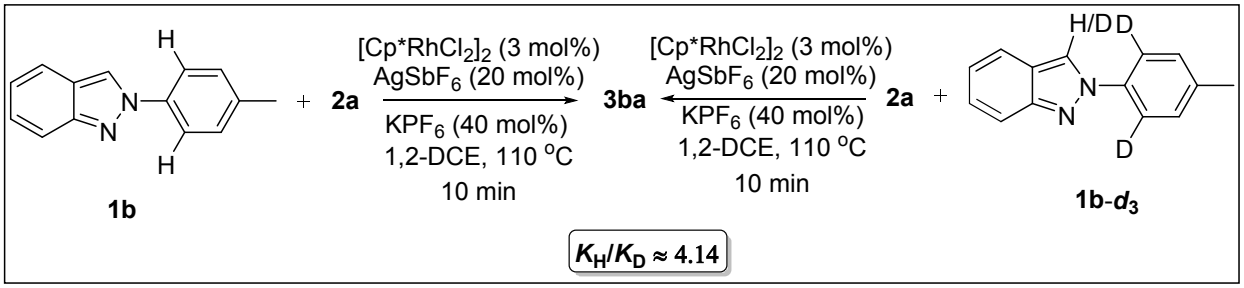
A mixture of 2-(*p*-tolyl)-2*H*-indazole (0.2 mmol, 41 mg) (**1b**), $[Cp^*RhCl_2]_2$ (3 mol%, 3.7 mg), $AgSbF_6$ (20 mol%, 13.7 mg), and KPF_6 (40 mol%, 14.7 mg) was taken in an oven dried screw-capped reaction tube. Then 1,2-DCE (2 mL) was added to the mixture and stirred for 5 min at room temperature under open atmosphere. After that, D_2O (1 mL) was added, and the resultant mixture was stirred at 110 ^oC for 3 h. Then, the reaction was cooled to room temperature and extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4. The crude residue was obtained after evaporating the solvent in vacuum and was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate (75:25) as an eluting solvent to afford the pure product **1b-*d*$_3$** as a white solid. The deuterium incorporation was determined using 400 MHz ^1H NMR as 100%.

3.2. Intermolecular kinetic isotope effect study:[2]



3-Methyl-1,4,2-dioxazol-5-one (**2a**) (0.3 mmol, 30 mg) was reacted with 2-(*p*-tolyl)-2*H*-indazole (**1b**) (0.1 mmol, 20.8 mg) and 2-(4-methylphenyl-2,6-d_2)-2*H*-indazole-3-*d* (**1b-*d*$_3$**) (0.1 mmol, 21.1 mg) for 10 min under standard reaction condition. The resulting solution was then diluted with dichloromethane (3 x 10 mL) and washed with brine (2 x 5 mL) and water (5 mL). The organic phase was dried over anhydrous Na_2SO_4. The crude residue was obtained after evaporating the solvent in vacuum and was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate (80:20) as an eluting solvent to afford **3ba** and a mixture of unreacted **1b** and **1b-*d*$_3$** as a white solid. The intermolecular K_H/K_D was found to be 4.26 after 10 min at 19% conversion, based on 400 MHz ^{1}H NMR of the product **3ba** and **3ba-*d*$_2$**.

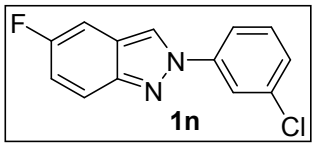
3.3. Independent kinetic isotope effect study:[2]



In a set of two experiments: in first set, 3-methyl-1,4,2-dioxazol-5-one (**2a**) (0.15 mmol, 15 mg) was reacted with 2-(*p*-tolyl)-2*H*-indazole (**1b**) (0.1 mmol, 20.8 mg) under standard reaction conditions. Whereas in another set, 2-(4-methylphenyl-2,6-d_2)-2*H*-indazole-3-*d* (**1b-*d*$_3$**) (0.1 mmol, 21.1 mg) was used instead of **1b** in the reaction with 3-methyl-1,4,2-dioxazol-5-one (**2a**) (0.15 mmol, 15 mg) under the standard reaction conditions. The two reactions were allowed to stir at 110 °C for 10 min. The resulting solution was then diluted with dichloromethane (3 x 10 mL) and washed with brine (2 x 5 mL) and water (5 mL). The organic phase was dried over anhydrous Na_2SO_4. The crude residue was obtained after evaporating the solvent in vacuum and

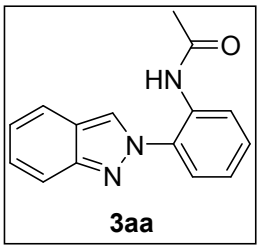
was purified by column chromatography on silica gel using a mixture petroleum ether and ethyl acetate (80:20) as an eluting solvent to afford **3ba**. The yield of **3ba** was obtained as 29% and 7% yields, respectively. The KIE value of 4.14 was determined by the ratio of obtained yield of **3ba** (KIE = 29%/7%/100% = 4.14).

3. Characterization data for the synthesized products:



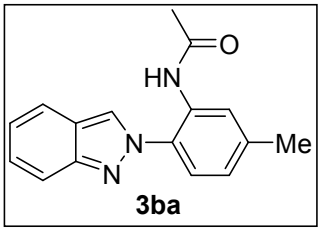
1n

2-(4-Chlorophenyl)-5-fluoro-2*H*-indazole (1n): Yellow solid (86%, 634.75 mg, 3.0 mmol); R_f = 0.55 (PE:EA = 95:05); M.p. 98-99 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 8.04-8.03 (m, 1H), 7.86-7.83 (m, 2H), 7.56-7.52 (m, 1H), 7.48-7.46 (m, 1H), 7.36-7.33 (m, 1H), 7.26-7.21 (m, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 158.9 (d, J_{C-F} = 240.0 Hz), 147.4, 141.3, 135.5, 130.7, 128.1, 122.2 (d, J_{C-F} = 12.0 Hz), 121.2, 120.5 (d, J_{C-F} = 9.0 Hz), 120.2 (d, J_{C-F} = 10.0 Hz), 119.2, 118.5 (d, J_{C-F} = 29.0 Hz), 102.8 (d, J_{C-F} = 23.0 Hz); Anal. Calcd for $C_{13}H_8ClFN_2$: C, 63.30; H, 3.27; N, 11.36%; Found: C, 63.45; H, 3.33; N, 11.25%.



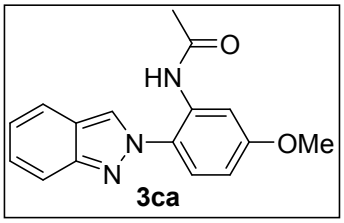
3aa

***N*-(2-(2*H*-Indazol-2-yl)phenyl)acetamide (3aa)**: White solid (76%, 38.1 mg); R_f = 0.50 (PE:EA = 83:17); M.p. 180-181 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.61 (s, 1H), 8.54 (d, J = 8.4 Hz, 1H), 8.28 (s, 1H), 7.67 (t, J = 9.2 Hz, 2H), 7.46-7.37 (m, 3H), 7.18 (q, J = 8.4 Hz, 2H), 2.12 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 149.8, 132.2, 129.2, 129.1, 127.6, 124.3, 124.0, 123.6, 123.0, 122.8, 121.9, 120.6, 117.2, 25.1; HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{15}H_{14}N_3O]^+$: 252.1131; found: 252.1135.



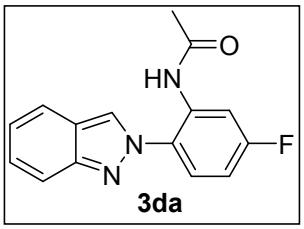
3ba

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)acetamide (3ba)**: White solid (90%, 47.7 mg); R_f = 0.5 (PE:EA = 82:18); M.p. 183-184 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.52 (s, 1H), 8.36 (s, 1H), 8.26 (s, 1H), 7.76 (t, J = 8.4 Hz, 2H), 7.40-7.33 (m, 2H), 7.19-7.15 (m, 1H), 7.01 (d, J = 9.6 Hz, 1H), 2.43 (s, 3H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 149.7, 139.6, 131.9, 127.5, 126.9, 124.8, 124.2, 123.4, 123.3, 122.7, 121.9, 120.6, 117.3, 25.2, 21.6; Anal. Calcd for $C_{16}H_{15}N_3O$: C, 72.43; H, 5.70; N, 15.84%; Found: C, 72.65; H, 5.65; N, 15.90%.



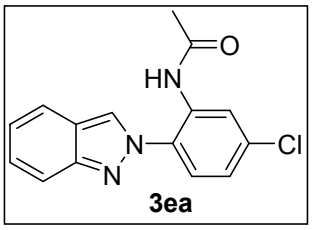
3ca

***N*-(2-(2*H*-Indazol-2-yl)-5-methoxyphenyl)acetamide (3ca)**: White solid (81%, 45.5 mg); R_f = 0.45 (PE:EA = 80:20); M.p. 188-189 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.56 (s, 1H), 8.22 (s, 2H), 7.75 (t, J = 8.0 Hz, 2H), 7.40-7.35 (m, 2H), 7.18-7.14 (m, 1H), 6.73 (dd, J = 8.8 Hz, 2.8 Hz, 1H), 3.88 (s, 3H), 2.12 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.8, 160.1, 149.7, 133.5, 127.4, 124.6, 124.1, 122.7, 122.5, 121.9, 120.5, 117.2, 110.3, 107.1, 55.7, 25.3; Anal. Calcd for $C_{16}H_{15}N_3O_2$: C, 68.31; H, 5.37; N, 14.94%; Found: C, 68.12; H, 5.33; N, 15.03%.



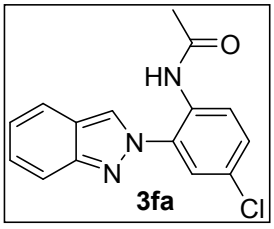
3da

***N*-(5-Fluoro-2-(2*H*-indazol-2-yl)phenyl)acetamide (3da)**: White solid (85%, 45.7 mg); R_f = 0.55 (PE:EA = 85:15); M.p. 179-180 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.66 (s, 1H), 8.42 (dd, J = 11.2 Hz, 2.8 Hz, 1H), 8.25 (s, 1H), 7.78-7.75 (m, 2H), 7.44-7.38 (m, 2H), 7.21-7.17 (m, 1H), 6.92-6.87 (m, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 162.5 (d, J_{C-F} = 246.0 Hz), 149.9, 134.0 (d, J_{C-F} = 11.0 Hz), 127.8, 125.2, 124.9 (d, J_{C-F} = 10.0 Hz), 124.4, 123.0, 122.0, 120.6, 117.3, 110.6 (d, J_{C-F} = 23.0 Hz), 109.9 (d, J_{C-F} = 29.0 Hz), 25.2; Anal. Calcd for $C_{15}H_{12}FN_3O$: C, 66.91; H, 4.49; N, 15.60%; Found: C, 67.08; H, 4.56; N, 15.49%.



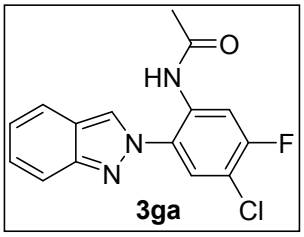
3ea

***N*-(5-Chloro-2-(2*H*-indazol-2-yl)phenyl)acetamide (3ea)**: White solid (88%, 50.2 mg); R_f = 0.5 (PE:EA = 84:16); M.p. 192-193 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.75 (s, 1H), 8.67 (s, 1H), 8.27 (s, 1H), 7.77-7.74 (m, 2H), 7.42-7.38 (m, 2H), 7.20-7.16 (m, 2H), 2.14 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 149.9, 135.0, 133.3, 127.9, 127.3, 124.4, 124.3, 123.9, 123.1, 122.7, 122.0, 120.6, 117.3, 25.2; Anal. Calcd for $C_{15}H_{12}ClN_3O$: C, 63.05; H, 4.23; N, 14.71%; Found: C, 62.87; H, 4.17; N, 14.79%.

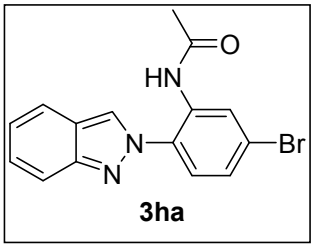


3fa

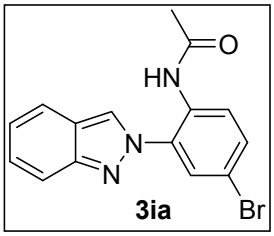
***N*-(4-Chloro-2-(2*H*-indazol-2-yl)phenyl)acetamide (3fa)**: White solid (91%, 52.0 mg); R_f = 0.50 (PE:EA = 84:16); M.p. 190-191 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.68 (s, 1H), 8.54 (d, J = 8.8 Hz, 1H), 8.30 (s, 1H), 7.77-7.74 (m, 2H), 7.48 (d, J = 2.4 Hz, 1H), 7.42-7.38 (m, 2H), 7.21-7.17 (m, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 150.0, 131.0, 129.6, 129.0, 128.8, 128.0, 124.3, 124.1, 123.5, 123.2, 122.0, 120.7, 117.3, 25.2; Anal. Calcd for $C_{15}H_{12}ClN_3O$: C, 63.05; H, 4.23; N, 14.71%; Found: C, 63.20; H, 4.27; N, 14.61%.



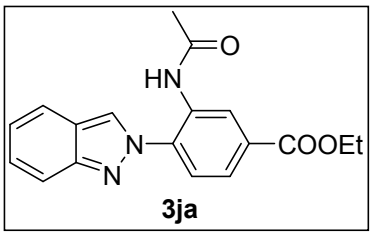
***N*-(4-Chloro-5-fluoro-2-(2*H*-indazol-2-yl)phenyl)acetamide (3ga)**: White solid (82%, 49.8 mg); R_f = 0.55 (PE:EA = 85:15); M.p. 194-195 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.78 (s, 1H), 8.56 (d, J = 11.2 Hz, 1H), 8.26 (m, 1H), 7.76-7.73 (m, 2H), 7.51 (d, J = 6.8 Hz, 1H), 7.43-7.38 (m, 1H), 7.21-7.17 (m, 1H), 2.14 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 157.6 (d, J_{C-F} = 248.0 Hz), 149.9, 132.4 (d, J_{C-F} = 11.0 Hz), 128.1, 125.3 (d, J_{C-F} = 3.0 Hz), 124.9, 124.3, 123.3, 122.0, 120.6, 117.2, 115.2 (d, J_{C-F} = 20.0 Hz), 110.7 (d, J_{C-F} = 28.0 Hz), 25.2; Anal. Calcd for $C_{15}H_{11}ClFN_3O$: C, 59.32; H, 3.65; N, 13.84%; Found: C, 59.11; H, 3.70; N, 13.89%.



***N*-(5-Bromo-2-(2*H*-indazol-2-yl)phenyl)acetamide (3ha)**: White solid (89%, 58.7 mg); R_f = 0.5 (PE:EA = 82:18); M.p. 187-188 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.78 (s, 1H), 8.81 (s, 1H), 8.27 (s, 1H), 7.77-7.73 (m, 2H), 7.42-7.38 (m, 1H), 7.31 (s, 2H), 7.20-7.16 (m, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 149.9, 133.4, 127.9, 127.7, 126.9, 125.5, 124.6, 124.2, 123.2, 122.9, 122.0, 120.6, 117.3, 25.3; Anal. Calcd for $C_{15}H_{12}BrN_3O$: C, 54.56; H, 3.66; N, 12.73%; Found: C, 54.42; H, 3.61; N, 12.66%.

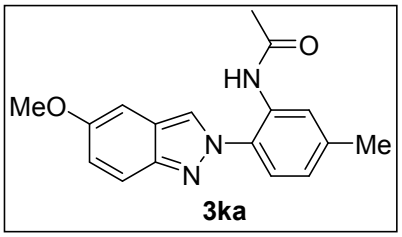


***N*-(4-Bromo-2-(2*H*-indazol-2-yl)phenyl)acetamide (3ia)**: White solid (79%, 52.1 mg); R_f = 0.55 (PE:EA = 80:20); M.p. 184-185 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.68 (s, 1H), 8.49 (d, J = 9.2 Hz, 1H), 8.30 (s, 1H), 7.77-7.74 (m, 2H), 7.63 (d, J = 2.4 Hz, 1H), 7.53 (dd, J = 9.2 Hz, 2.4 Hz, 1H) 7.43-7.39 (m, 1H), 7.21-7.17 (m, 1H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 150.0, 132.0, 131.5, 129.8, 128.0, 127.0, 126.3, 124.3, 123.2, 122.0, 120.7, 117.3, 115.9, 25.2; Anal. Calcd for $C_{15}H_{12}BrN_3O$: C, 54.56; H, 3.66; N, 12.73%; Found: C, 54.75; H, 3.69; N, 12.81%.



3ja

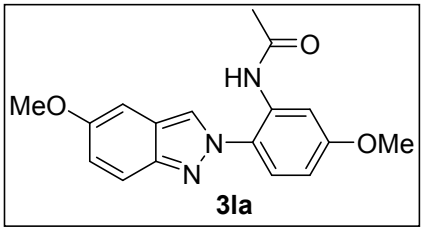
Ethyl 3-acetamido-4-(2*H*-indazol-2-yl)benzoate (3ja): White solid (87%, 56.2 mg); R_f = 0.45 (PE:EA = 75:25); M.p. 198-199 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.88 (s, 1H), 9.19 (s, 1H), 8.36 (s, 1H), 7.91-7.89 (m, 1H), 7.78-7.75 (m, 2H), 7.55 (d, J = 8.4 Hz, 1H), 7.43-7.39 (m, 1H), 7.21-7.17 (m, 1H), 4.41 (q, J = 7.6 Hz, 2H), 2.16 (s, 3H), 1.42 (t, J = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 165.6, 150.0, 132.2, 131.9, 131.1, 128.1, 125.3, 124.47, 124.42, 123.3, 122.1, 120.7, 117.4, 61.5, 25.2, 14.4; HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{18}H_{18}N_3O_3]^+$: 324.1343; found: 324.1342.



3ka

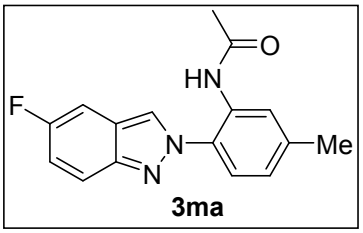
***N*-(2-(5-Methoxy-2*H*-indazol-2-yl)-5-methylphenyl)acetamide (3ka)**: White solid (83%, 49.0 mg); R_f = 0.45 (PE:EA = 77:23); M.p. 196-197 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.47 (s, 1H), 8.35 (s, 1H), 8.10 (s, 1H), 7.65 (d, J = 9.2 Hz, 1H), 7.32 (d, J = 8.4 Hz, 1H), 7.09 (dd, J = 9.6 Hz, 2.4 Hz, 1H), 7.00-6.98 (m, 1H), 6.93 (d, J = 2.0 Hz, 1H), 3.86 (s, 3H), 2.42 (s, 3H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 155.8, 146.6, 139.3, 131.9, 127.0, 124.7, 123.3, 123.0, 122.8, 122.6, 122.0, 118.6, 96.5,

55.5, 25.2, 21.6; Anal.Calcd for $C_{17}H_{17}N_3O_2$: C, 69.14; H, 5.80; N, 14.23%; Found: C, 68.98; H, 5.73; N, 14.18%.



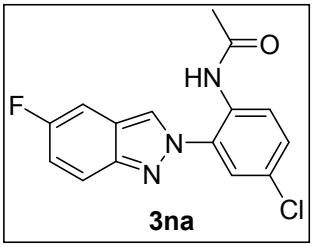
3la

***N*-(5-Methoxy-2-(5-methoxy-2*H*-indazol-2-yl)phenyl)acetamide (3la)**: White solid (84%, 52.3 mg); R_f = 0.5 (PE:EA = 75:25); M.p. 201-202 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.51 (s, 1H), 8.20 (s, 1H), 8.05 (s, 1H), 7.64 (d, J = 9.2 Hz, 1H), 7.31 (d, J = 8.4 Hz, 1H), 7.07 (dd, J = 9.6 Hz, 2.4 Hz, 1H), 6.92 (s, 1H), 6.71-6.68 (m, 1H), 3.86 (s, 3H), 3.85 (s, 3H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.7, 159.8, 155.7, 146.5, 133.4, 124.4, 123.0, 122.6, 122.4, 122.0, 118.5, 110.1, 107.0, 96.5, 55.7, 55.5, 25.2; Anal. Calcd for $C_{17}H_{17}N_3O_3$: C, 65.58; H, 5.50; N, 13.50%; Found: C, 65.45; H, 5.53; N, 13.39%.



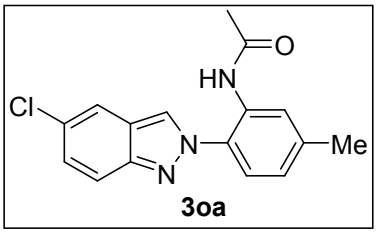
3ma

***N*-(2-(5-Fluoro-2*H*-indazol-2-yl)-5-methylphenyl)acetamide (3ma)**: White solid (78%, 44.1 mg); R_f = 0.55 (PE:EA = 84:16); M.p. 168-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.36 (s, 1H), 8.33 (s, 1H), 8.20 (s, 1H), 7.72 (q, J = 4.8 Hz, 1H), 7.32-7.29 (m, 2H), 7.20-7.14 (m, 1H), 7.00-6.98 (m, 1H), 2.41 (s, 3H), 2.10 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 158.8, (d, J_{C-F} = 241.0 Hz), 147.0, 139.7, 131.8, 126.8, 124.8, 124.2 (d, J_{C-F} = 8.0 Hz), 123.4, 121.3, 121.2, 119.2 (d, J_{C-F} = 9.0 Hz), 119.1 (d, J_{C-F} = 29.0 Hz), 102.9 (d, J_{C-F} = 25.0 Hz), 25.1, 21.5; Anal. Calcd for $C_{16}H_{14}FN_3O$: C, 67.83; H, 4.98; N, 14.83%; Found: C, 67.98; H, 4.92; N, 14.93%.



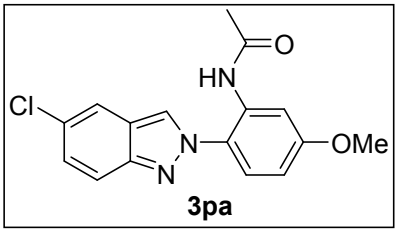
3na

***N*-(4-Chloro-2-(5-fluoro-2*H*-indazol-2-yl)phenyl)acetamide (3na)**: White solid (92%, 55.8 mg); R_f = 0.55 (PE:EA = 85:15); M.p. 205-206 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.50 (s, 1H), 8.51 (d, J = 8.8 Hz, 1H), 8.25 (s, 1H), 7.73 (q, J = 4.8 Hz, 1H), 7.45 (d, J = 2.4 Hz, 1H), 7.37 (dd, J = 9.2 Hz, 2.4 Hz, 1H), 7.31 (dd, J = 8.8 Hz, 2.4 Hz, 1H), 7.22-7.17 (m, 1H), 2.12 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 159.0 (d, $J_{C–F}$ = 240.0 Hz), 147.3, 130.8, 129.3 (d, $J_{C–F}$ = 31.0 Hz), 128.8, 124.3 (d, $J_{C–F}$ = 9.0 Hz), 124.2, 123.4, 121.4 (d, $J_{C–F}$ = 12.0 Hz), 119.9, 119.6, 119.4 (d, $J_{C–F}$ = 10.0 Hz), 103.0 (d, $J_{C–F}$ = 25.0 Hz), 25.1; Anal. Calcd for $C_{15}H_{11}ClFN_3O$: C, 59.32; H, 3.65; N, 13.84%; Found: C, 59.15; H, 3.58; N, 13.72%.



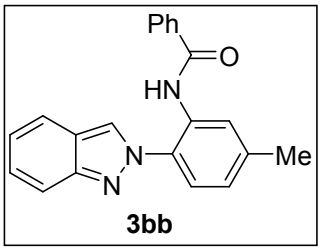
3oa

***N*-(2-(5-Chloro-2*H*-indazol-2-yl)-5-methylphenyl)acetamide (3oa)**: White solid (86%, 51.5 mg); R_f = 0.50 (PE:EA = 85:15); M.p. 192-193 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.31 (s, 1H), 8.33 (s, 1H), 8.20 (s, 1H), 7.72-7.69 (m, 2H), 7.34-7.30 (m, 2H), 7.01 (d, J = 8.0 Hz, 1H), 2.43 (s, 3H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.6, 148.0, 140.0, 131.9, 128.9, 128.5, 126.7, 124.9, 123.8, 123.57, 123.52, 122.3, 119.2, 118.8, 25.2, 21.6; HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{15}ClN_3O$: 300.0898; found: 300.0905.



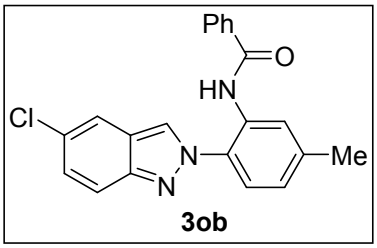
3pa

***N*-(2-(5-Chloro-2*H*-indazol-2-yl)-5-methoxyphenyl)acetamide (3pa)**: White solid (89%, 56.2 mg); R_f = 0.5 (PE:EA = 80:20); M.p. 198-199 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.36 (s, 1H), 8.20-8.17 (m, 2H), 7.72-7.68 (m, 2H), 7.36-7.30 (m, 2H), 6.74-6.72 (m, 1H), 3.87 (s, 3H), 2.12 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 168.8, 160.3, 148.0, 133.5, 128.8, 128.4, 124.6, 123.7, 122.3, 122.2, 119.2, 118.7, 110.4, 107.2, 55.8, 25.3; Anal. Calcd for $C_{16}H_{14}ClN_3O_2$: C, 60.86; H, 4.47; N, 13.31%; Found: C, 61.04; H, 4.49; N, 13.25%.



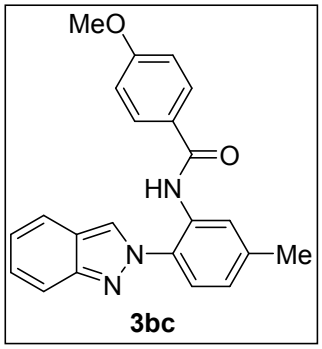
3bb

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)benzamide (3bb)**: White solid (90%, 58.9 mg); R_f = 0.5 (PE:EA = 81:19); M.p. 178-179 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.96 (s, 1H), 8.65 (s, 1H), 8.31 (s, 1H), 8.01-7.98 (m, 2H), 7.81-7.72 (m, 2H), 7.54-7.45 (m, 3H), 7.43-7.38 (m, 2H), 7.18-7.14 (m, 1H), 7.04-7.01 (m, 1H), 2.46 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 165.4, 149.6, 139.7, 134.9, 132.1, 131.9, 128.7, 127.7, 127.4, 126.9, 124.9, 124.1, 123.2, 122.9, 122.8, 121.9, 120.7, 116.9, 21.7; Anal. Calcd for $C_{21}H_{17}N_3O$: C, 77.04; H, 5.23; N, 12.84%; Found: C, 77.17; H, 5.28; N, 12.75%.



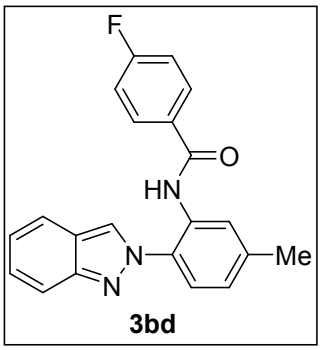
3ob

***N*-(2-(5-Chloro-2*H*-indazol-2-yl)-5-methylphenyl)benzamide (3ob)**: White solid (80%, 57.7 mg); R_f = 0.55 (PE:EA = 82:18); M.p. 184-185 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.67 (s, 1H), 8.61 (s, 1H), 8.25 (s, 1H), 7.95-7.93 (m, 2H), 7.73-7.70 (m, 2H), 7.53-7.45 (m, 3H), 7.40 (d, J = 8.0 Hz, 1H), 7.33 (dd, J = 9.6 Hz, 2.0 Hz, 1H), 7.04 (d, J = 8.4 Hz, 1H), 2.47 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 165.4, 147.9, 140.0, 134.8, 132.0, 129.2, 128.8, 128.6, 127.3, 126.7, 125.0, 123.7, 123.4, 123.0, 122.3, 120.8, 119.3, 118.4, 21.7; Anal. Calcd for $C_{21}H_{16}ClN_3O$: C, 69.71; H, 4.46; N, 11.61%; Found: C, 69.87; H, 4.42; N, 11.67%.



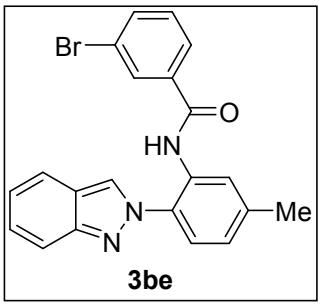
3bc

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)-4-methoxybenzamide (3bc)**: White solid (75%, 53.6 mg); R_f = 0.50 (PE:EA = 83:17); M.p. 188-189 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.76 (s, 1H), 8.61 (s, 1H), 8.32 (s, 1H), 7.96-7.92 (m, 2H), 7.80 (d, J = 8.8 Hz, 1H), 7.76 (d, J = 8.4 Hz, 1H), 7.44-7.39 (m, 2H), 7.17 (t, J = 8.0 Hz, 1H), 7.04 (d, J = 8.4 Hz, 1H), 6.97-6.94 (m, 2H), 3.86 (m, 3H), 2.47 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 165.0, 162.6, 149.6, 139.7, 132.3, 129.3, 127.7, 127.2, 126.9, 124.6, 124.1, 123.2, 123.0, 122.8, 121.9, 120.7, 116.9, 113.9, 55.5, 21.7; Anal. Calcd for $C_{22}H_{19}N_3O_2$: C, 73.93; H, 5.36; N, 11.76%; Found: C, 73.75; H, 5.33; N, 11.81%.



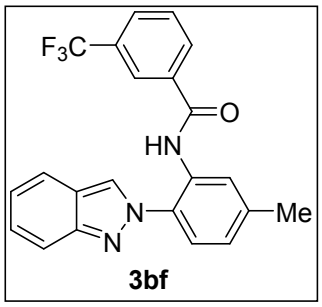
3bd

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)-4-fluorobenzamide (3bd)**: White solid (84%, 58.0 mg); R_f = 0.55 (PE:EA = 85:15); M.p. 189-190 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.94 (s, 1H), 8.60 (s, 1H), 8.32 (s, 1H), 8.00-7.96 (m, 2H), 7.75 (d, J = 9.6 Hz, 2H), 7.44-7.39 (m, 2H), 7.19-7.12 (m, 3H), 7.05-7.03 (m, 1H), 2.46 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 166.3, 164.0 (d, J_{C-F} = 48.0 Hz), 149.5, 139.6, 131.9, 131.1, 129.8, 129.7, 127.9, 126.8, 124.9, 124.1, 123.1, 122.8 (d, J_{C-F} = 7.0 Hz), 121.9, 120.7, 116.7, 115.7 (d, J_{C-F} = 22.0 Hz), 21.6; Anal. Calcd for $C_{21}H_{16}FN_3O$: C, 73.03; H, 4.67; N, 12.17%; Found: C, 72.81; H, 4.72; N, 12.25%.



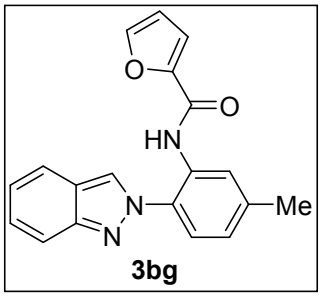
3be

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)-3-bromobenzamide (3be)**: White solid (88%, 71.5 mg); R_f = 0.5 (PE:EA = 83:17); M.p. 193-194 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.17 (s, 1H), 8.62 (s, 1H), 8.33 (s, 1H), 8.17-8.16 (m, 1H), 7.95-7.88 (m, 2H), 7.74 (d, J = 8.4 Hz, 1H), 7.65-7.63 (m, 1H), 7.45-7.40 (m, 2H), 7.35 (t, J = 8.0 Hz, 1H), 7.19-7.15 (m, 1H), 7.05-7.03 (m, 1H), 2.46 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 163.6, 149.5, 139.6, 136.8, 134.8, 131.7, 130.3, 127.8, 126.7, 126.3, 125.0, 124.0, 123.0, 122.99, 122.91, 122.8, 122.6, 121.8, 120.6, 117.1, 21.6; Anal. Calcd for $C_{21}H_{16}BrN_3O$: C, 62.08; H, 3.97; N, 10.34%; Found: C, 62.22; H, 4.04; N, 10.25%.



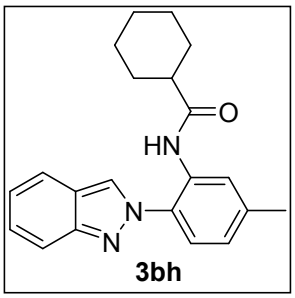
3bf

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)-3-(trifluoromethyl)benzamide (3bf)**: White solid (72%, 56.9 mg); R_f = 0.55 (PE:EA = 90:10); M.p. 172-173 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.19 (s, 1H), 8.63 (s, 1H), 8.36 (s, 1H), 8.27 (s, 1H), 8.21 (d, J = 7.6 Hz, 1H), 7.81-7.74 (m, 3H), 7.62 (t, J = 8.0 Hz, 1H), 7.47-7.40 (m, 2H), 7.21-7.16 (m, 1H), 7.08 (d, J = 8.4 Hz, 1H), 2.48 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 163.7, 149.7, 139.7, 135.7, 131.5 (d, J_{C-F} = 24.0 Hz), 131.1, 129.7, 128.5 (q, J_{C-F} = 4.0 Hz), 127.9, 126.9, 125.3, 124.1, 123.2, 123.0, 122.8, 122.5, 122.0 (q, J_{C-F} = 270.0 Hz), 121.9, 121.2, 120.6, 116.9; Anal. Calcd for $C_{22}H_{16}F_3N_3O$: C, 66.83; H, 4.08; N, 10.63%; Found: C, 66.98; H, 4.12; N, 10.56%.



3bg

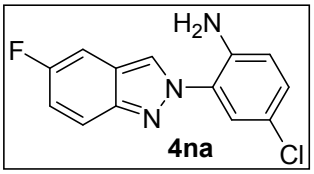
***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)furan-2-carboxamide (3bg)**: White solid (86%, 54.5 mg); R_f = 0.45 (PE:EA = 80:20); M.p. 140-141 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.74 (s, 1H), 8.55 (s, 1H), 8.30 (s, 1H), 7.84 (d, J = 8.8 Hz, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.50 (s, 1H), 7.43-7.38 (m, 2H), 7.19-7.15 (m, 2H), 7.06-7.04 (m, 1H), 6.50 (q, J = 2.0 Hz, 1H), 2.46 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 156.8, 149.7, 148.3, 144.6, 139.7, 131.7, 127.5, 127.1, 124.9, 123.9, 123.29, 123.22, 122.8, 122.0, 120.6, 117.3, 114.9, 112.3, 21.6; Anal. Calcd for $C_{19}H_{15}N_3O_2$: C, 71.91; H, 4.76; N, 13.24%; Found: C, 71.78; H, 4.73; N, 13.35%.



3bh

***N*-(2-(2*H*-Indazol-2-yl)-5-methylphenyl)cyclohexanecarboxamide (3bh)**: White solid (87%, 58.0 mg); R_f = 0.5 (PE:EA = 85:15); M.p. 187-188 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.71 (s, 1H), 8.42 (s, 1H),

8.26 (s, 1H), 7.76 (d, J = 9.2 Hz, 2H), 7.41-7.35 (m, 2H), 7.19-7.15 (m, 1H), 7.01-6.99 (m, 1H), 2.42 (s, 3H), 2.25-2.17 (m, 1H), 1.93 (d, J = 12.8 Hz, 2H), 1.79-1.75 (m, 2H), 1.50-1.41 (m, 2H), 1.32-1.21 (m, 4H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 174.9, 149.6, 139.6, 132.2, 127.5, 127.0, 124.6, 124.1, 123.4, 123.2, 122.7, 121.9, 120.6, 117.1, 46.8, 29.6, 25.9, 25.8, 21.6; Anal. Calcd for $C_{21}H_{23}N_3O$: C, 75.65; H, 6.95; N, 12.60%; Found: C, 75.45; H, 6.91; N, 12.54%.



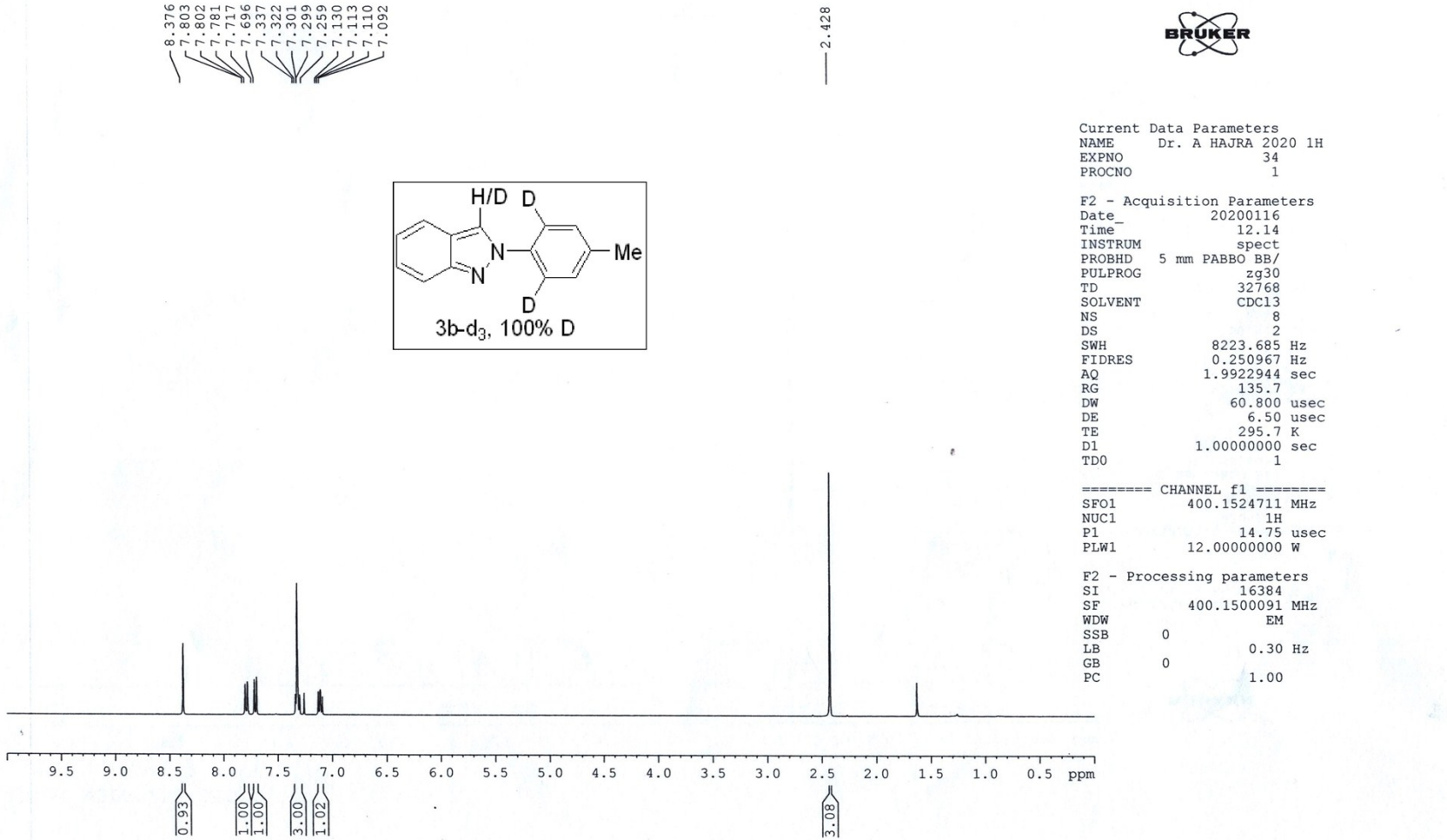
4na

4-Chloro-2-(5-fluoro-2*H*-indazol-2-yl)aniline (**4na**): White solid (89%, 46.5 mg); R_f = 0.55 (PE:EA = 90:10); M.p. 218-219 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.71 (q, J = 4.8 Hz, 1H), 7.33-7.27 (m, 2H), 7.18-7.12 (m, 2H), 6.79 (d, J = 8.8 Hz, 1H), 4.93 (s, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 158.8 (d, J_{C-F} = 239.0 Hz), 147.0, 140.0, 129.4, 126.6, 124.5, 123.7 (d, J_{C-F} = 8.0 Hz), 122.4, 121.2 (d, J_{C-F} = 12.0 Hz), 119.7 (d, J_{C-F} = 9.0 Hz), 118.9, 118.6 (d, J_{C-F} = 7.0 Hz), 102.8 (d, J_{C-F} = 24.0 Hz); Anal. Calcd for $C_{13}H_9ClFN_3$: C, 59.67; H, 3.47; N, 16.06%; Found: C, 59.55; H, 3.54; N, 16.16%.

4. References:

1. M. R. Kumar, A. Park, N. Park and S. Lee, *Org. Lett*., 2011, **13**, 3542−3545.
2. S. Samanta, S. Mondal, D. Ghosh and A. Hajra, *Org. Lett.,* 2019, **21**, 4905-4909.
3. (*a*) U. Dutta, S. Maiti, S. Pimparkar, S. Maiti, L. R. Gahan, E. H. Krenske, D. W. Lupton and D. Maiti, *Chem. Sci.,* 2019, **10**, 7426-7432; (*b*) A. Modi, P. Sau, N. Chakraborty and B. K. Patel, *Adv. Synth. Catal*., 2019, **361**, 1368-1375.

5. NMR spectra for the synthesized products

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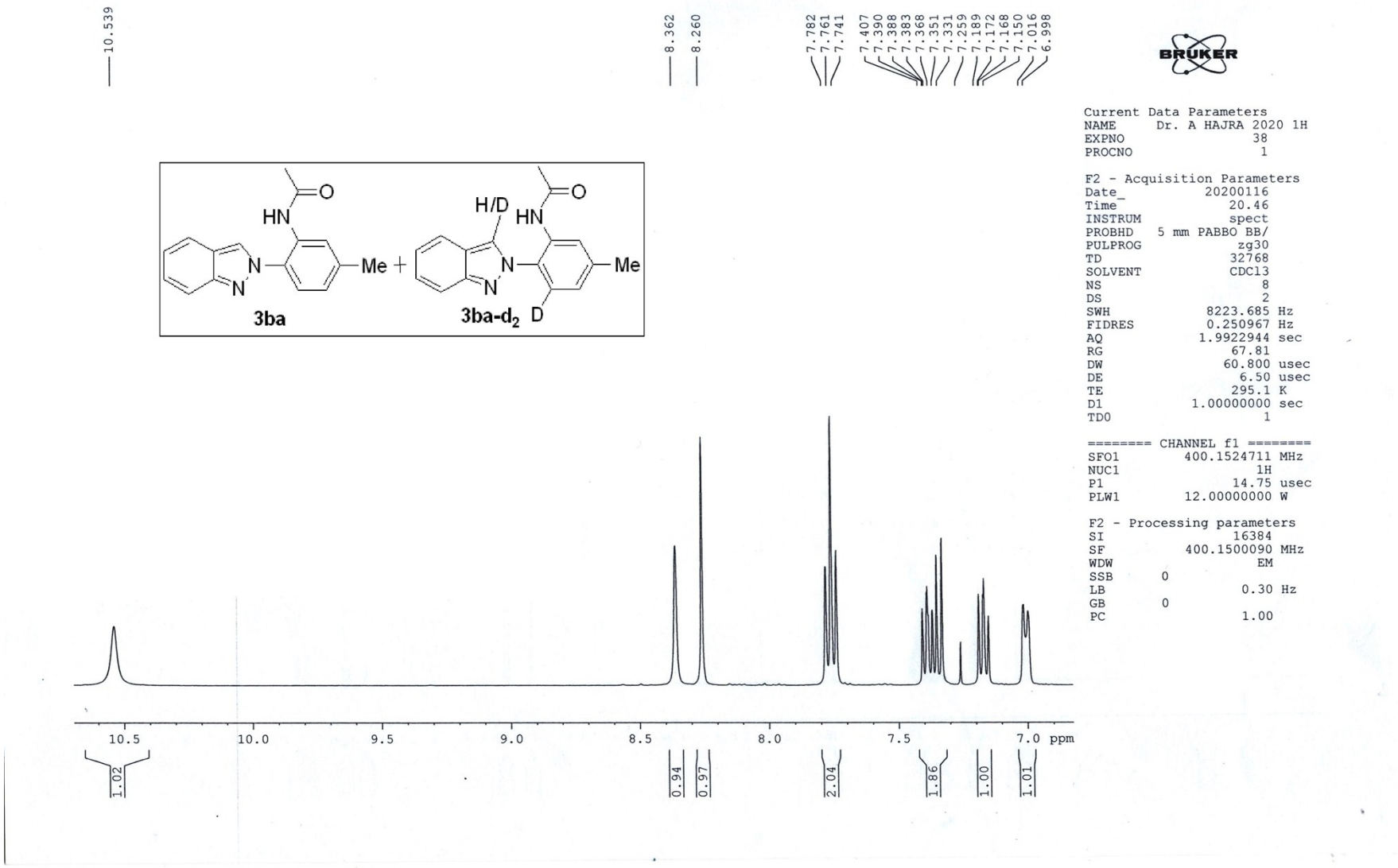
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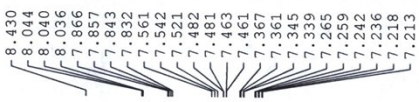
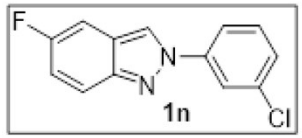
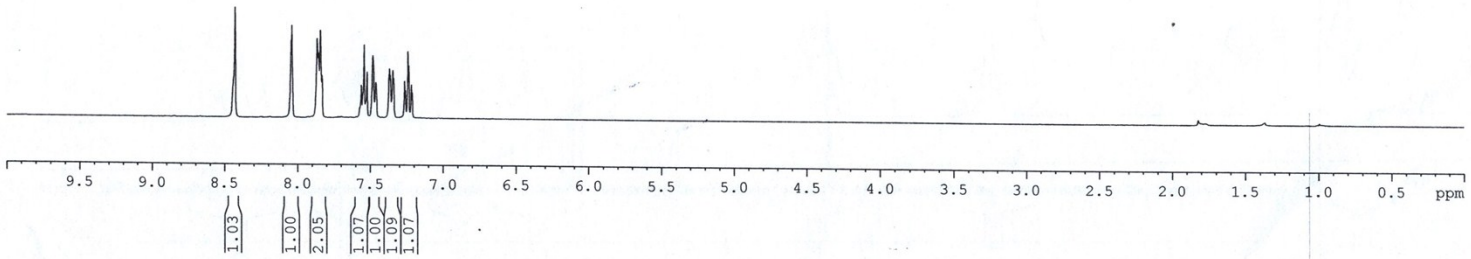
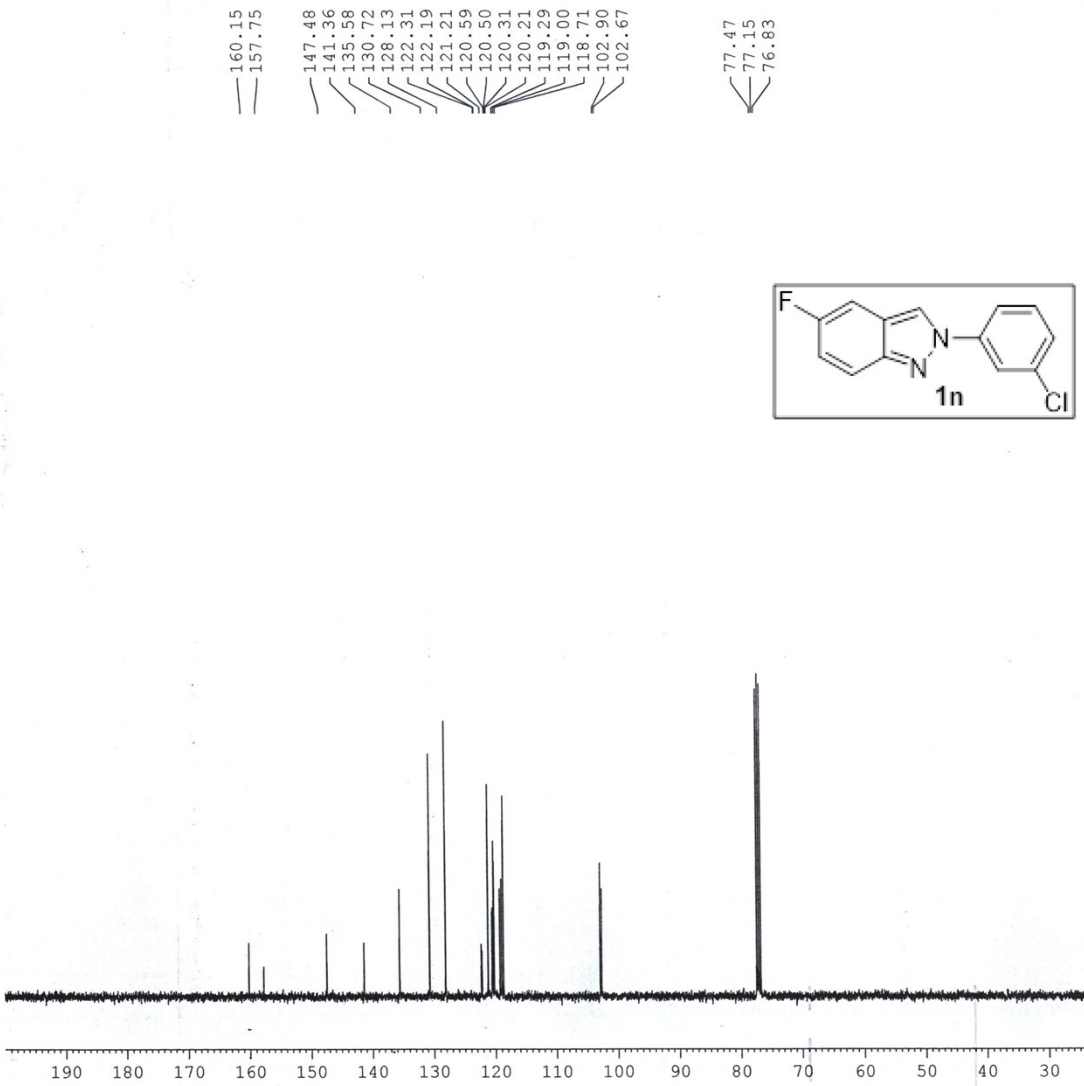
3ba-d2
Current Data Parameters
NAME Dr. A HAJRA 2020 1H
EXPNO 38
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200116
Time 20.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9922944 sec
RG 67.81
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1524711 MHz
NUC1 1H
P1 14.75 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 16384
SF 400.1500090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0 ppm
1.02
0.94
0.97
2.04
1.86
1.00
1.01


8.430
8.044
8.040
8.036
7.866
7.857
7.843
7.832
7.561
7.542
7.521
7.482
7.481
7.463
7.461
7.367
7.361
7.345
7.339
7.265
7.259
7.242
7.236
7.218
7.213


F
N
N
1n
Cl


9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.03
1.00
2.05
1.07
1.00
1.09
1.07



BRUKER

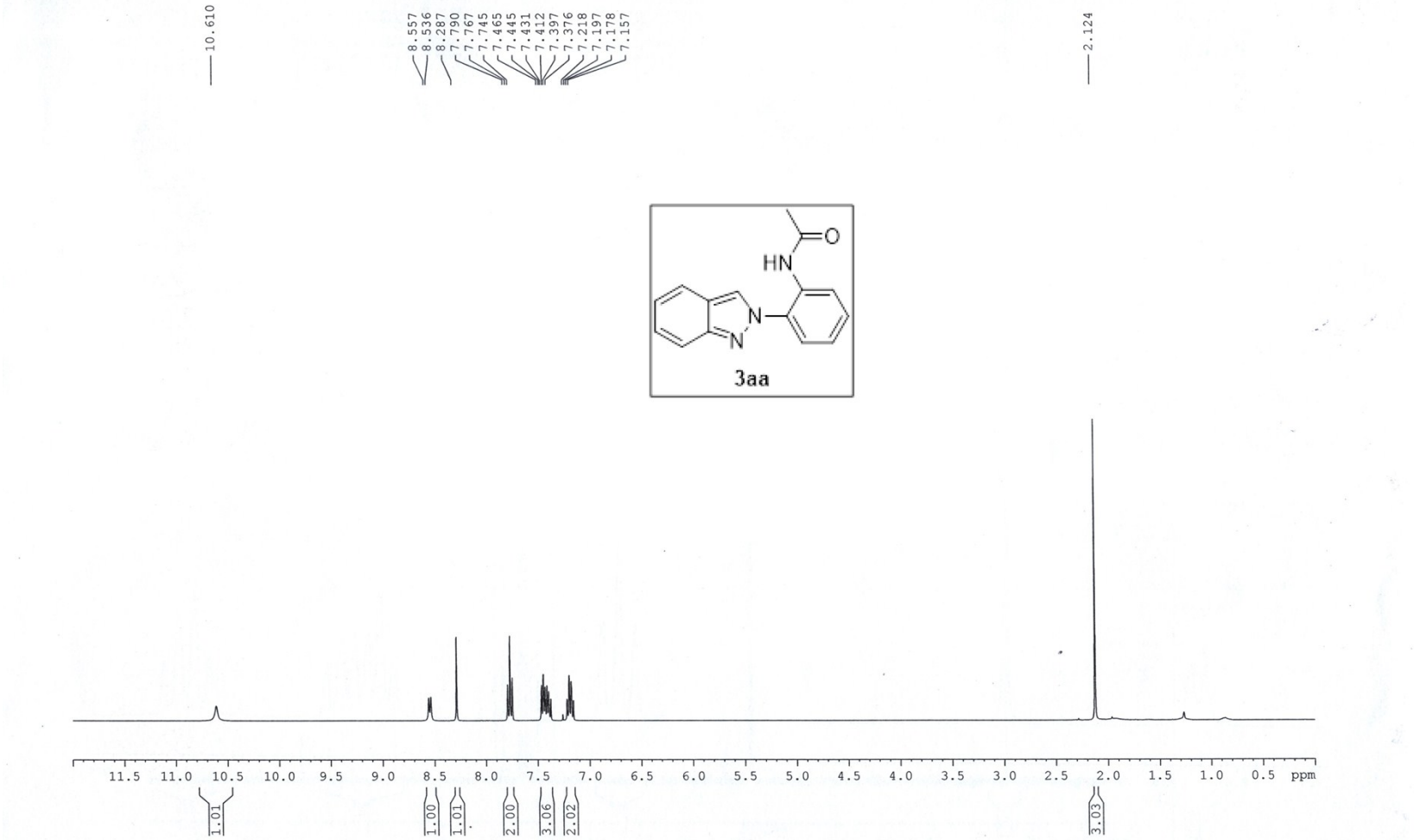
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 488
PROCNO 1

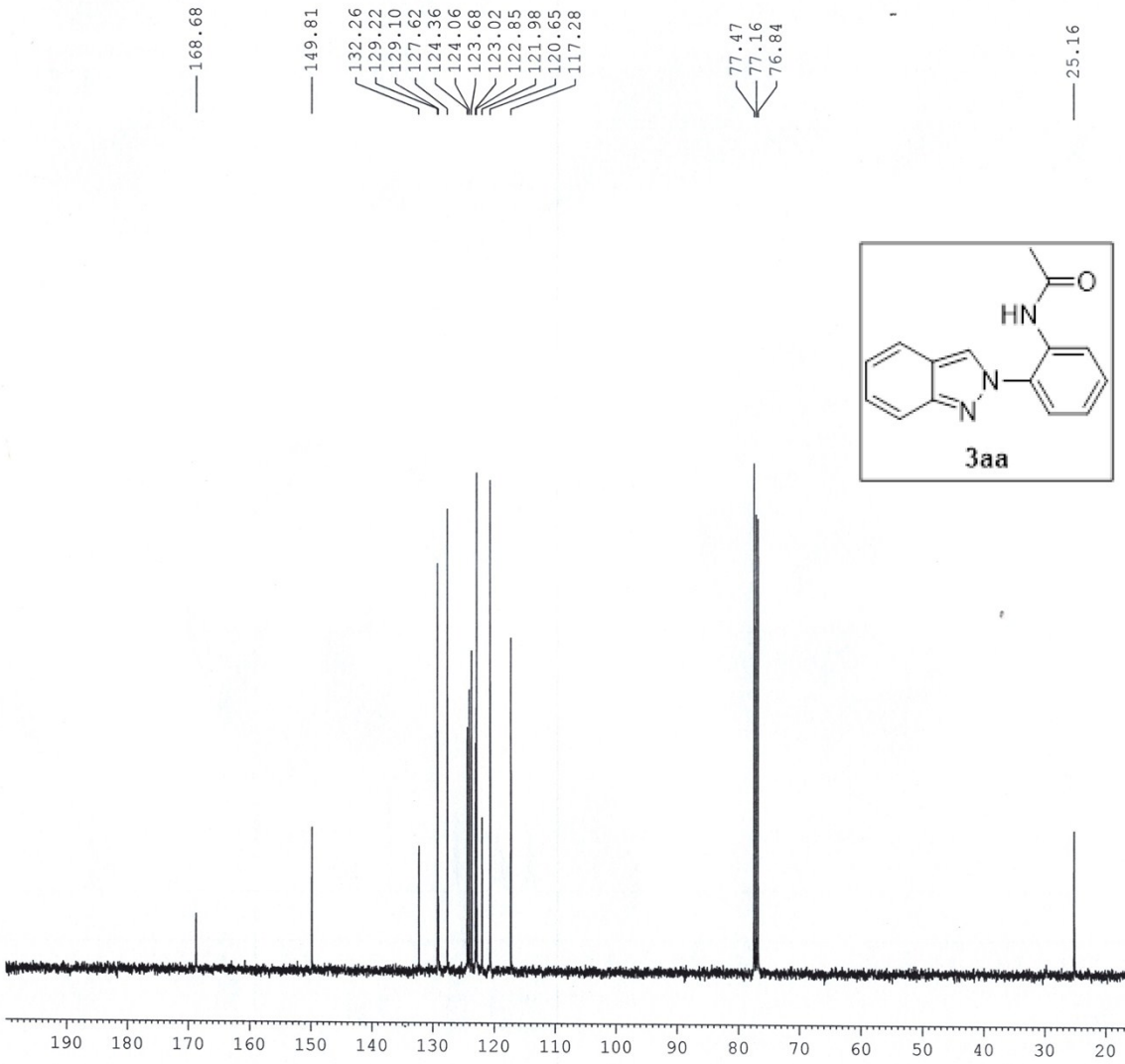
F2 - Acquisition Parameters
Date_ 20191101
Time 13.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 156
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

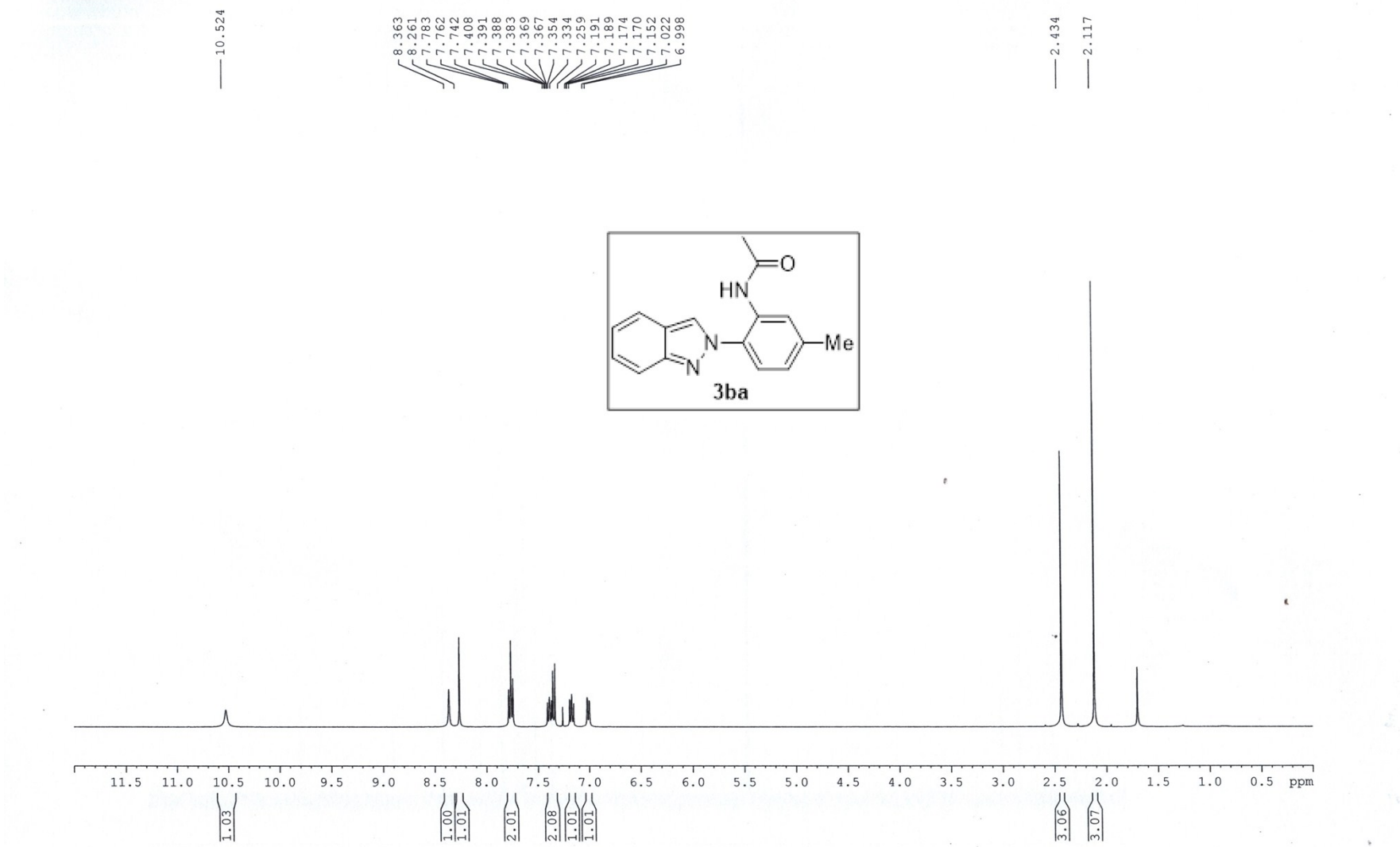
F2 - Processing parameters
SI 16384
SF 100.6177874 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

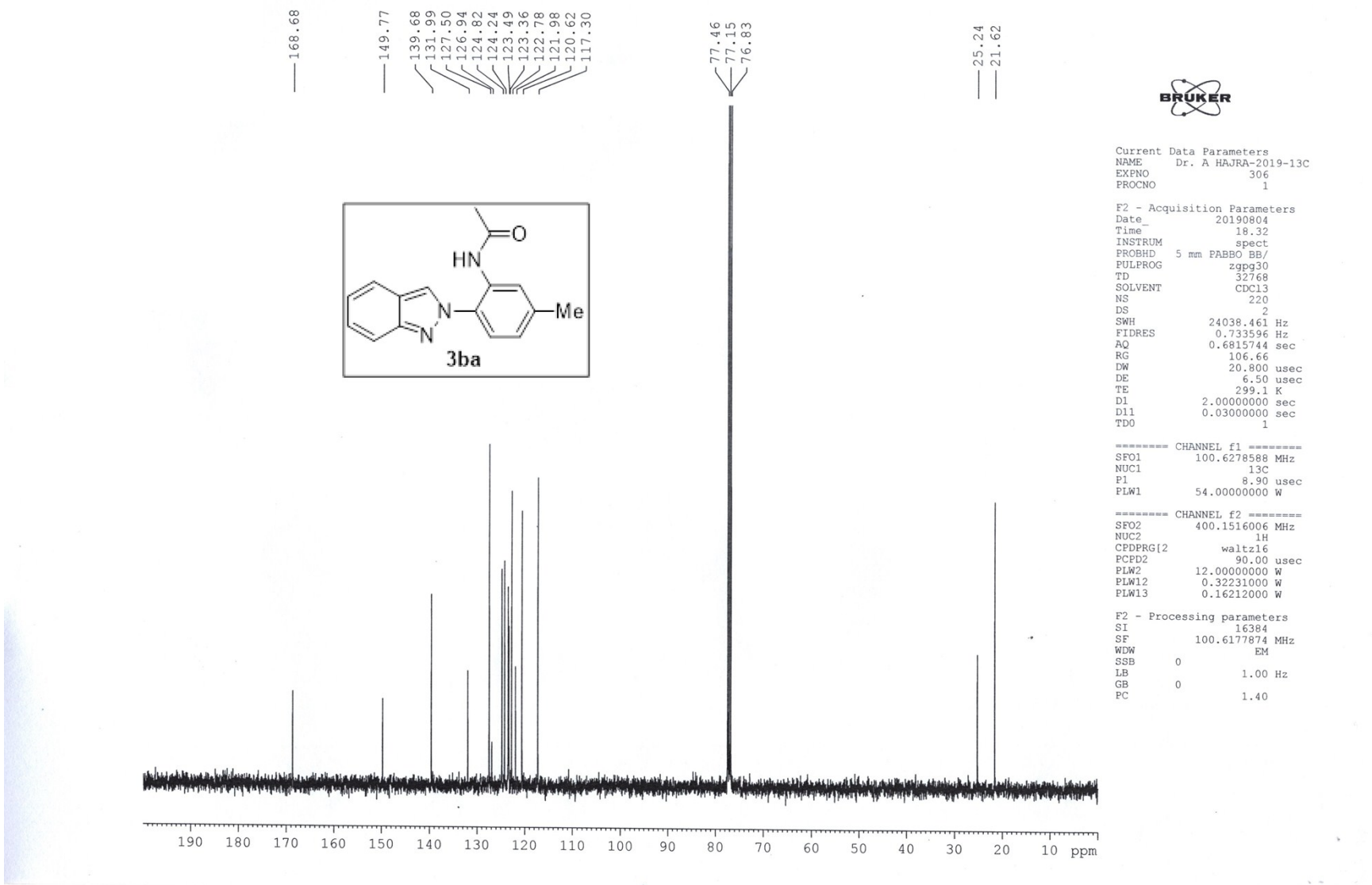

10.610
8.557
8.536
8.287
7.790
7.767
7.745
7.465
7.445
7.431
7.412
7.397
7.376
7.218
7.197
7.178
7.157
2.124
HN
O
N
N
3aa
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01
1.00
1.01
2.00
3.06
2.02
3.03



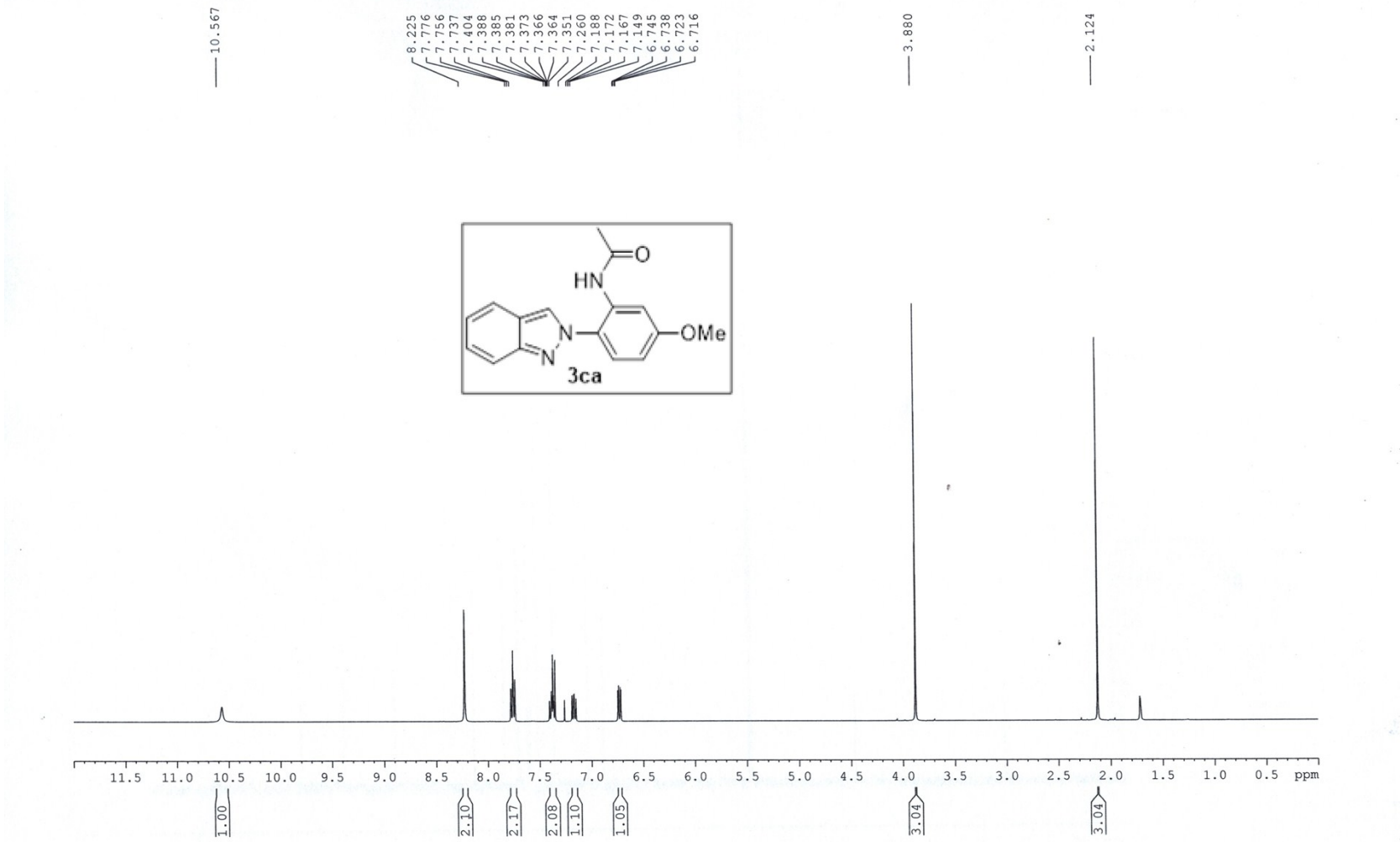
BRUKER

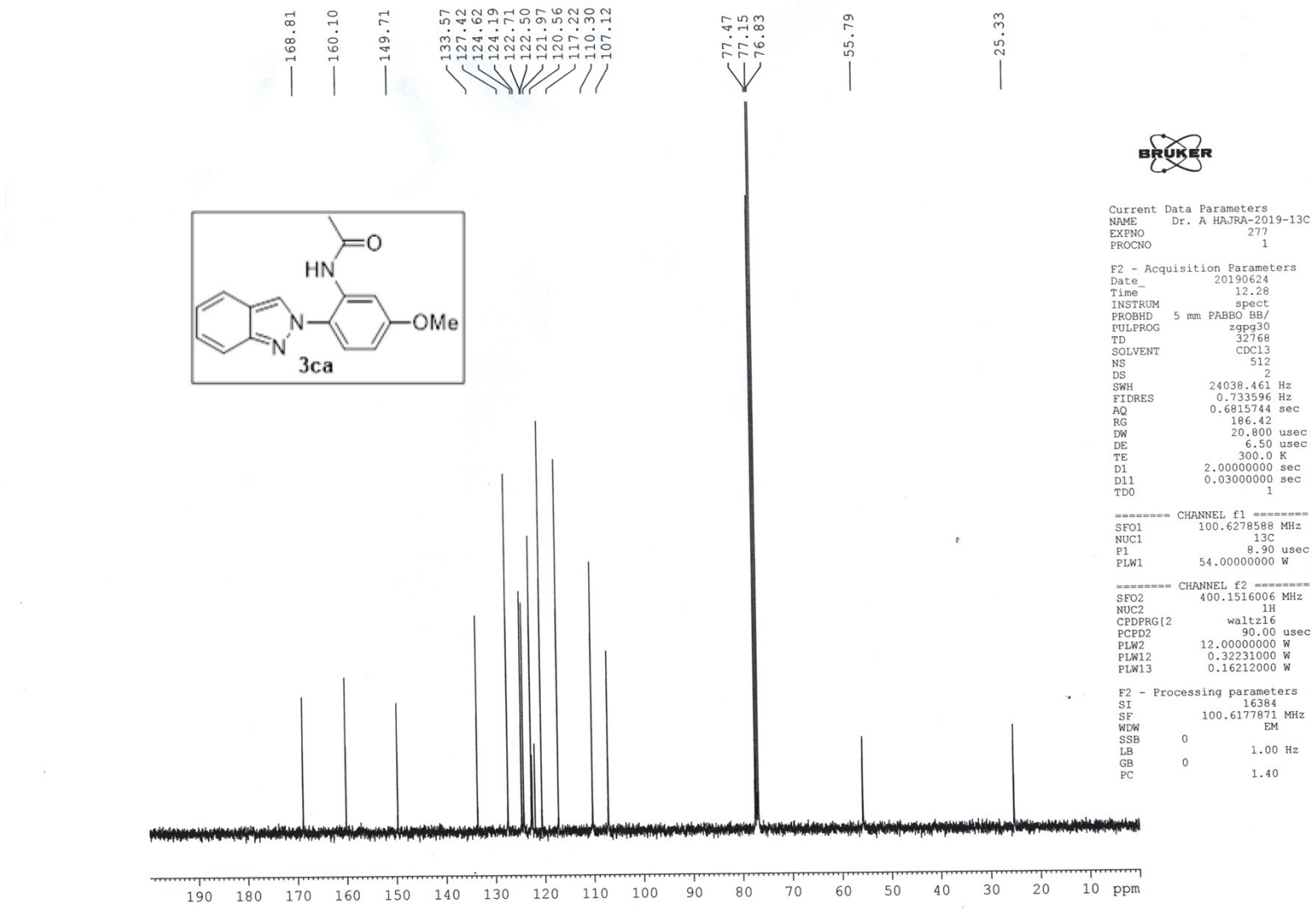
```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  362
PROCNO                   1

F2 - Acquisition Parameters
Date_             20190911
Time                 7.34
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   160
DS                     2
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 57.28
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6278588 MHz
NUC1                 13C
P1                  8.90 usec
PLW1          54.00000000 W

======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          12.00000000 W
PLW12          0.32231000 W
PLW13          0.16212000 W

F2 - Processing parameters
SI                   16384
SF            100.6177933 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```


10.524
8.363
8.261
7.783
7.762
7.742
7.408
7.391
7.388
7.383
7.369
7.367
7.354
7.334
7.259
7.191
7.189
7.174
7.170
7.152
7.022
6.998
2.434
2.117
HN
O
N
N
Me
3ba
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.00
1.01
2.01
2.08
1.01
1.01
3.06
3.07




```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  306
PROCNO                   1

F2 - Acquisition Parameters
Date_           20190804
Time              18.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  220
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                106.66
DW               20.800 usec
DE                6.50 usec
TE                299.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                 8.90 usec
PLW1         54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.32231000 W
PLW13         0.16212000 W

F2 - Processing parameters
SI                 16384
SF           100.6177874 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```


10.567
8.225
7.776
7.756
7.737
7.404
7.388
7.385
7.381
7.373
7.366
7.364
7.351
7.260
7.188
7.172
7.167
7.149
6.745
6.738
6.723
6.716
3.880
2.124
HN
O
N
N
OMe
3ca
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
2.10
2.17
2.08
1.10
1.05
3.04
3.04



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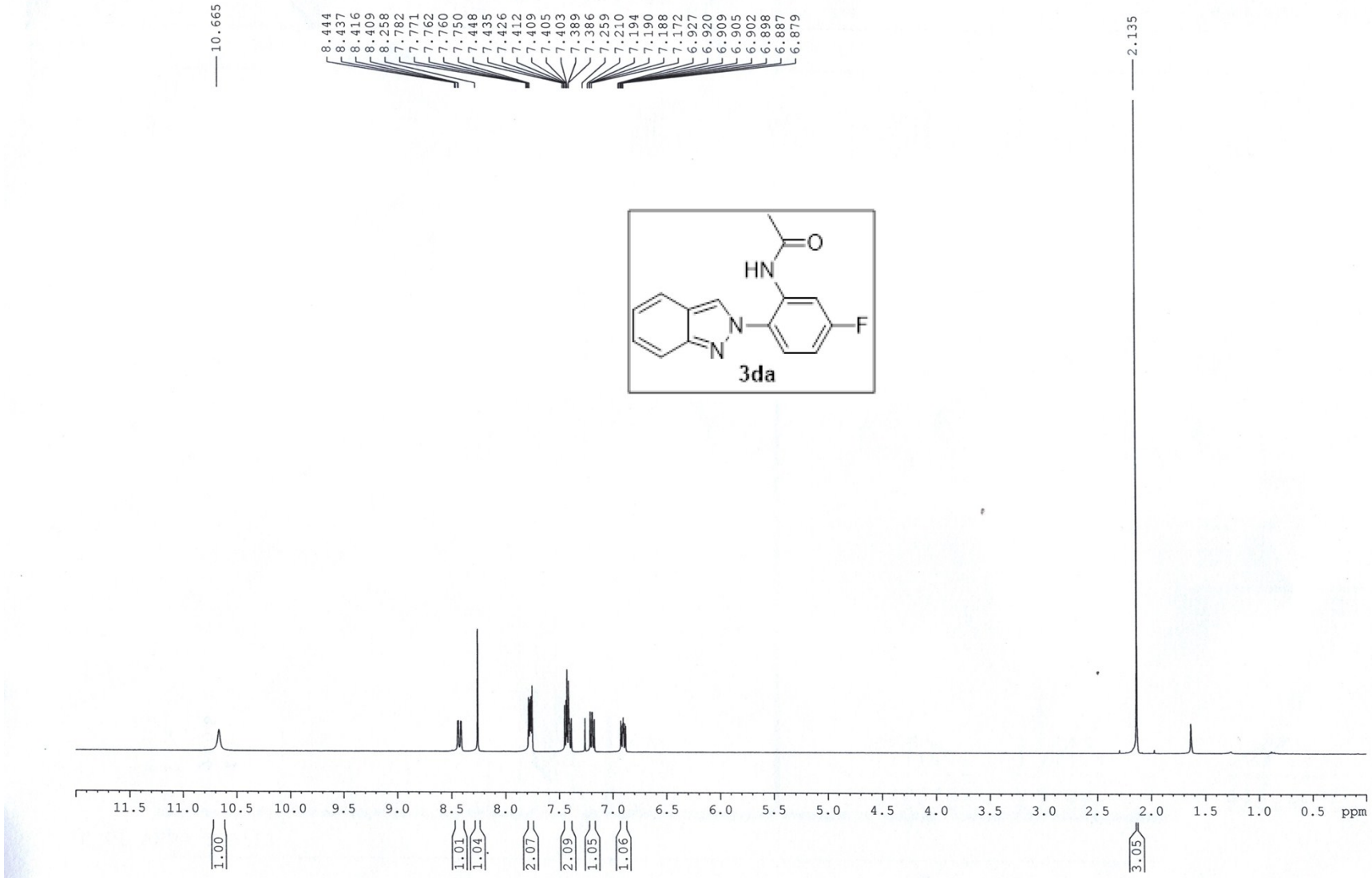
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 277
PROCNO 1

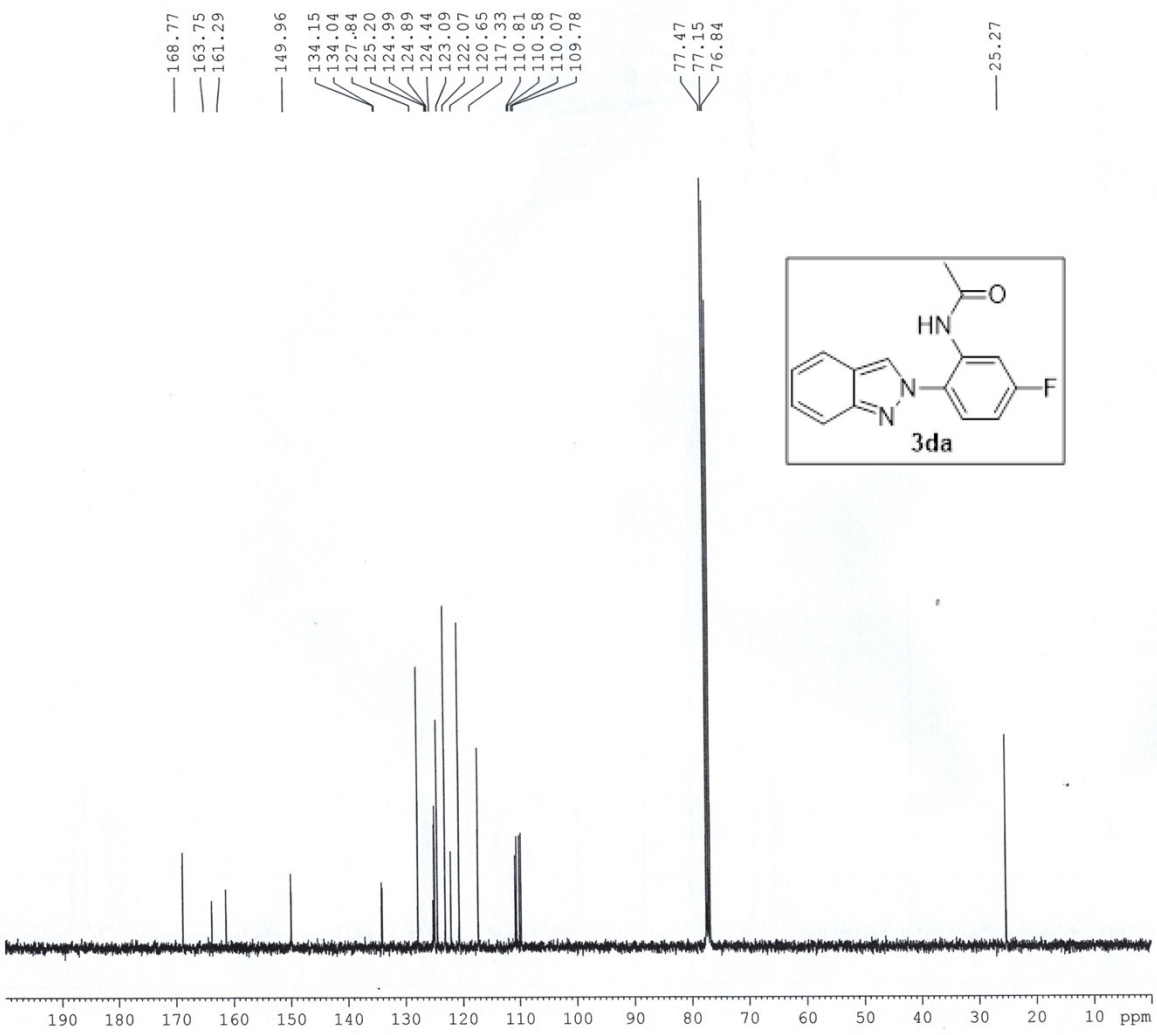
F2 - Acquisition Parameters
Date_ 20190624
Time 12.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177871 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.665
8.444
8.437
8.416
8.409
8.258
7.782
7.771
7.762
7.760
7.750
7.448
7.435
7.426
7.412
7.409
7.405
7.403
7.389
7.386
7.259
7.210
7.194
7.190
7.188
7.172
6.927
6.920
6.909
6.905
6.902
6.898
6.887
6.879
2.135
HN
O
N
N
F
3da
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.01
1.04
2.07
2.09
1.05
1.06
3.05



BRUKER

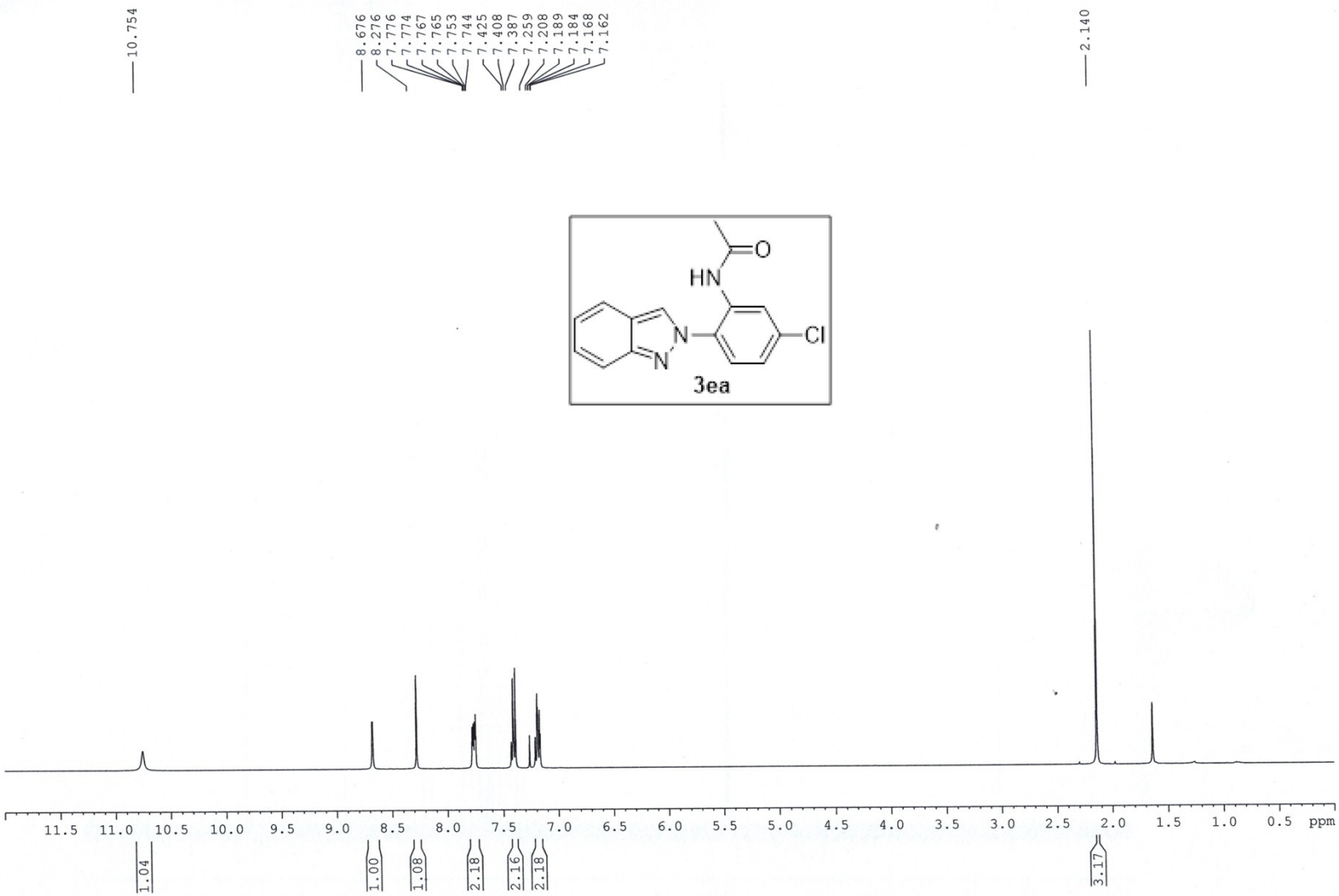
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 319
PROCNO 1

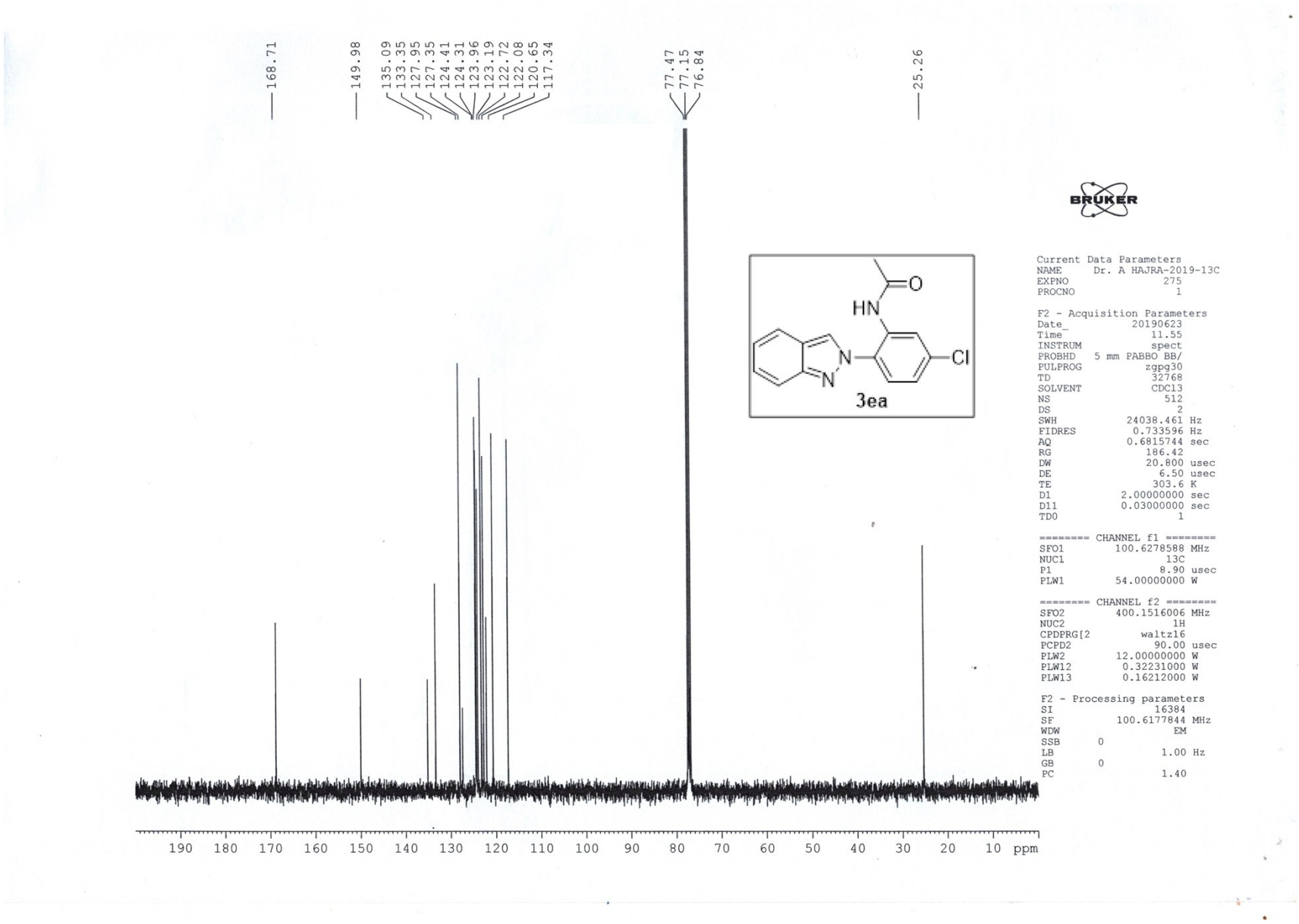
F2 - Acquisition Parameters
Date_ 20190814
Time 12.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 87.66
DW 20.800 usec
DE 6.50 usec
TE 302.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177844 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.754
8.676
8.276
7.776
7.774
7.767
7.765
7.753
7.744
7.425
7.408
7.387
7.259
7.208
7.189
7.184
7.168
7.162
2.140
HN
O
N
N
Cl
3ea
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.04
1.00
1.08
2.18
2.16
2.18
3.17



BRUKER

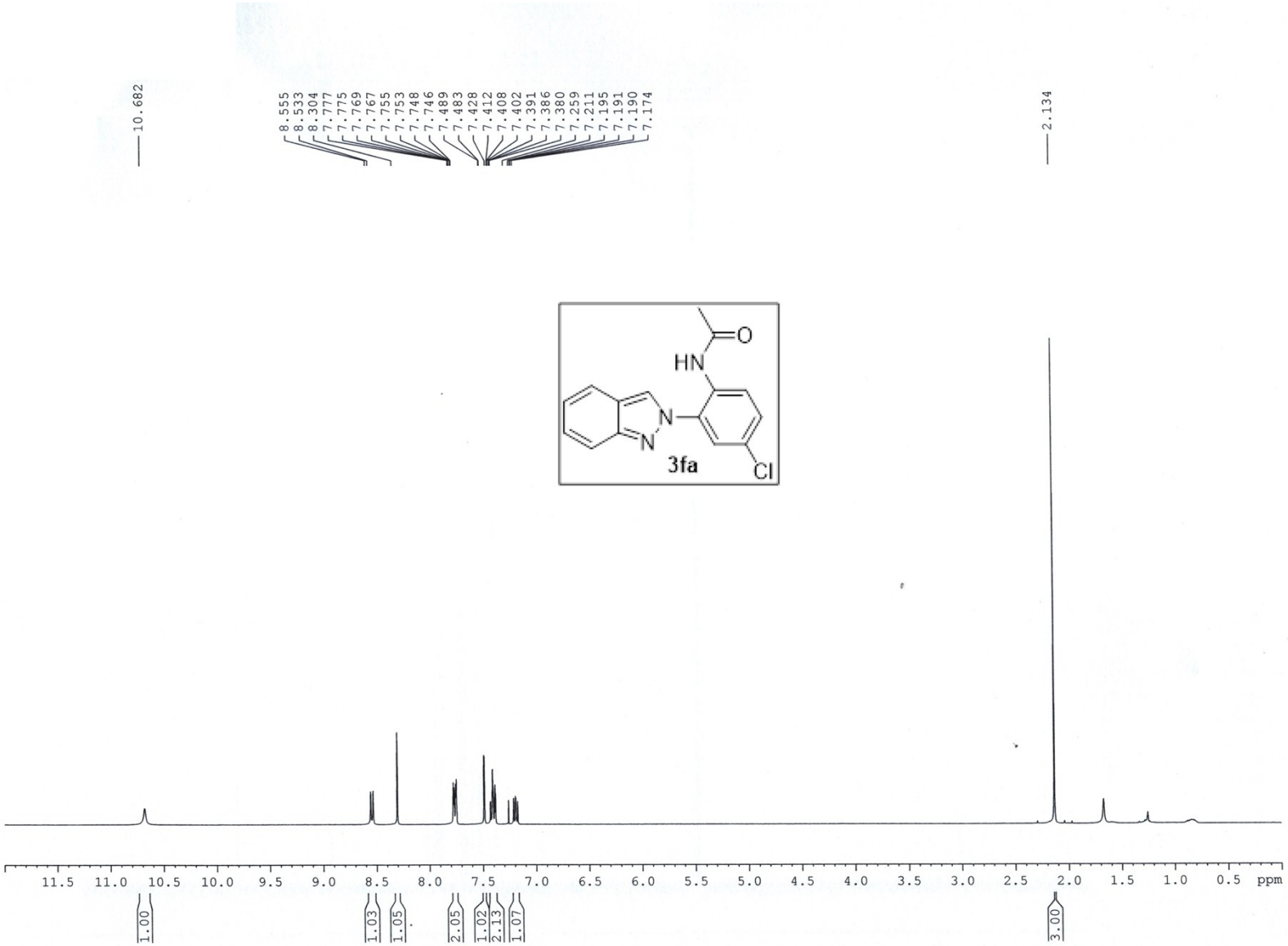
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 275
PROCNO 1

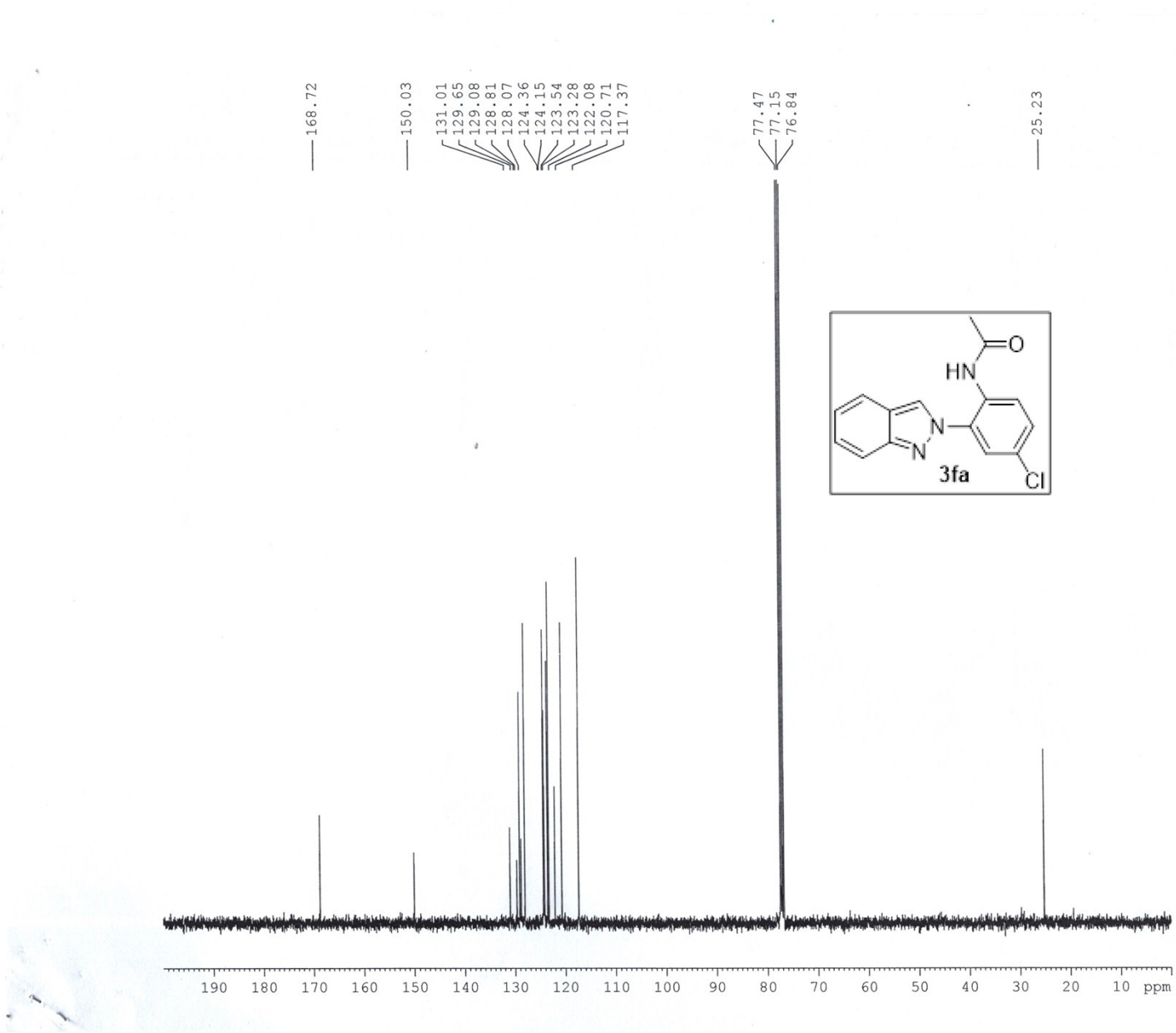
F2 - Acquisition Parameters
Date_ 20190623
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 303.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177844 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.682
8.555
8.533
8.304
7.777
7.775
7.769
7.767
7.755
7.753
7.748
7.746
7.489
7.483
7.428
7.412
7.408
7.402
7.391
7.386
7.380
7.259
7.211
7.195
7.191
7.190
7.174
2.134
HN
O
N
N
Cl
3fa
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.03
1.05
2.05
1.02
2.13
1.07
3.00



BRUKER

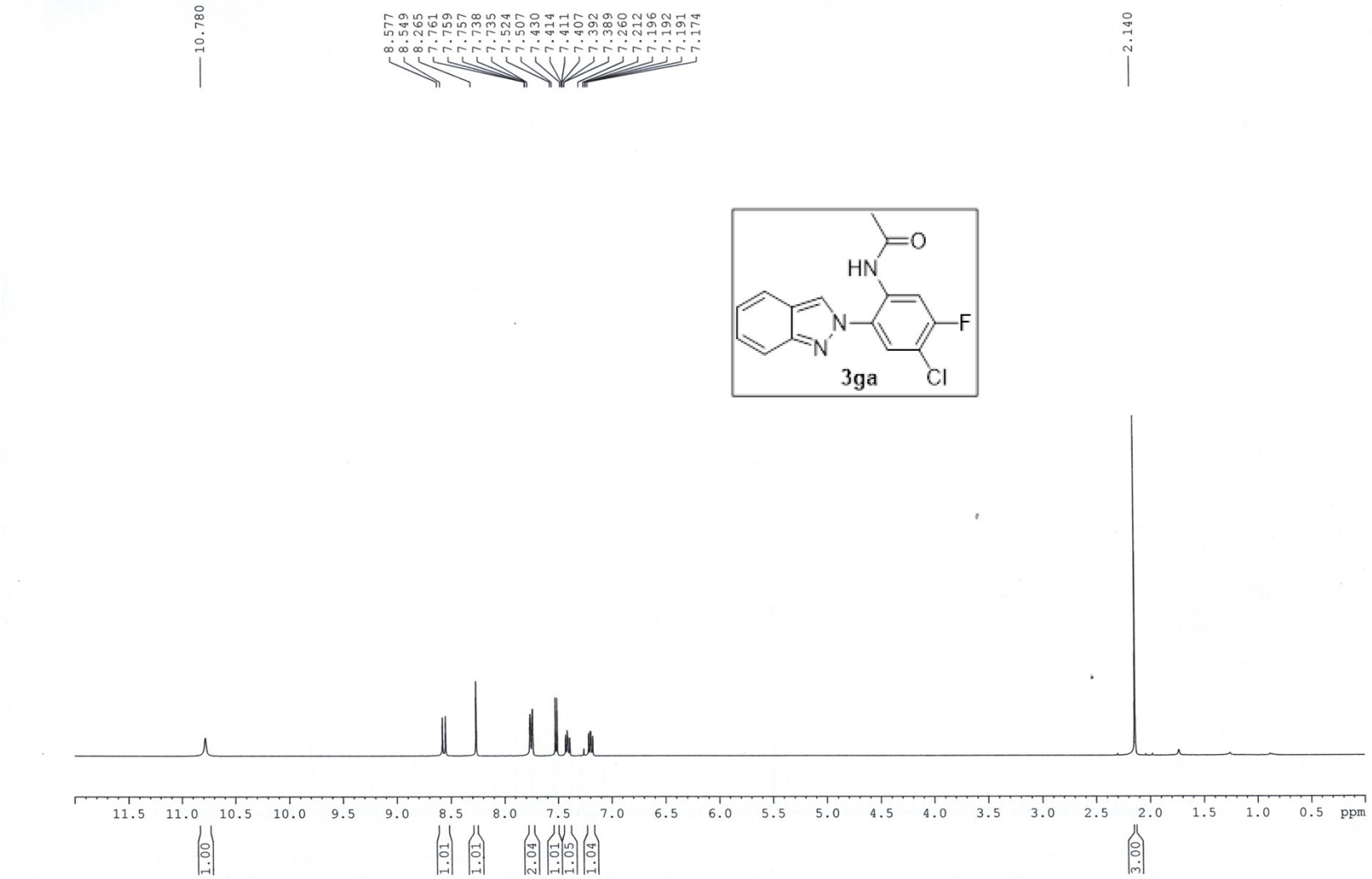
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 303
PROCNO 1

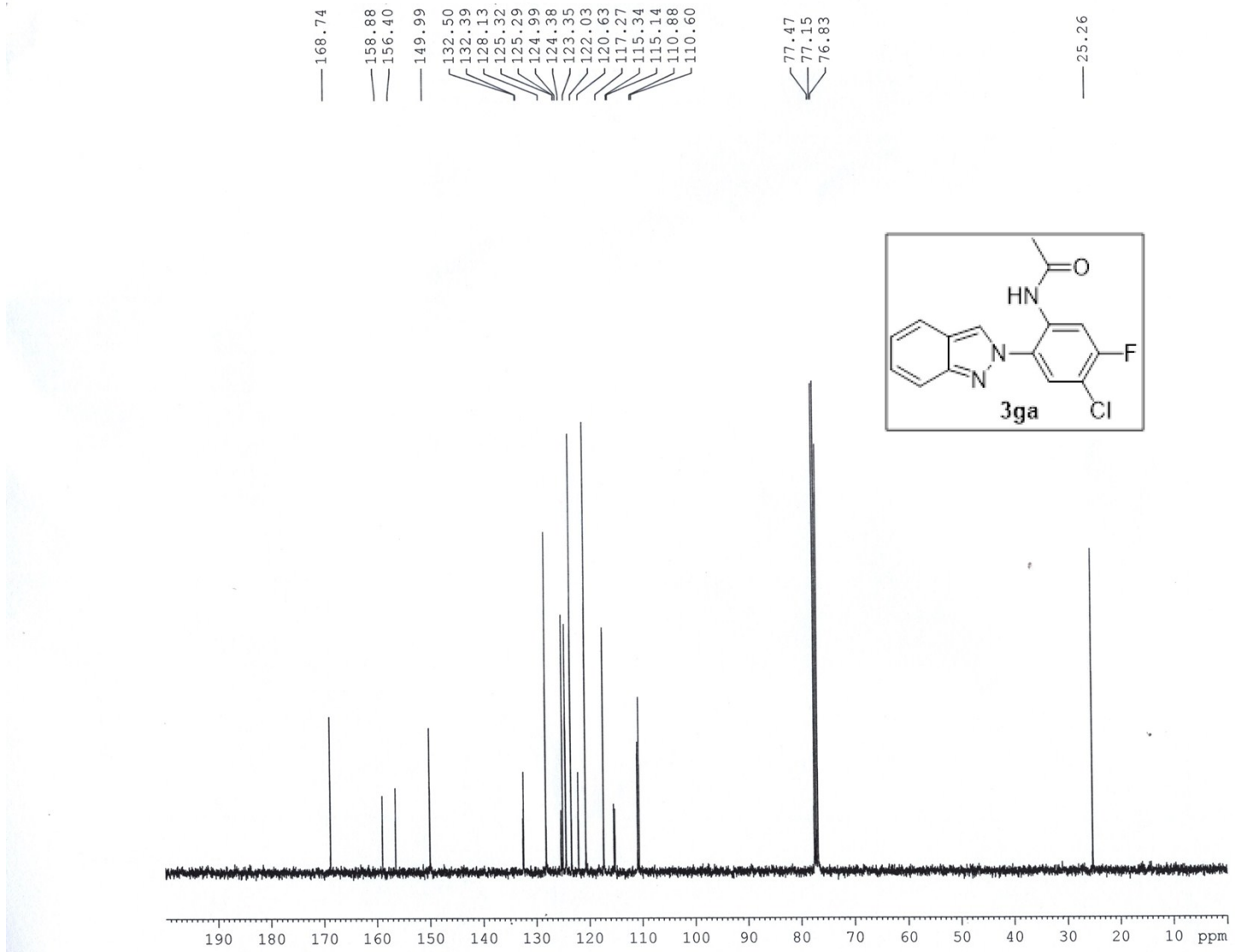
F2 - Acquisition Parameters
Date_ 20190803
Time 18.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 470
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177858 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.780
8.577
8.549
8.265
7.761
7.759
7.757
7.738
7.735
7.524
7.507
7.430
7.414
7.411
7.407
7.392
7.389
7.260
7.212
7.196
7.192
7.191
7.174
2.140
HN
O
N
N
F
Cl
3ga
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.01
1.01
2.04
1.01
1.05
1.04
3.00



BRUKER

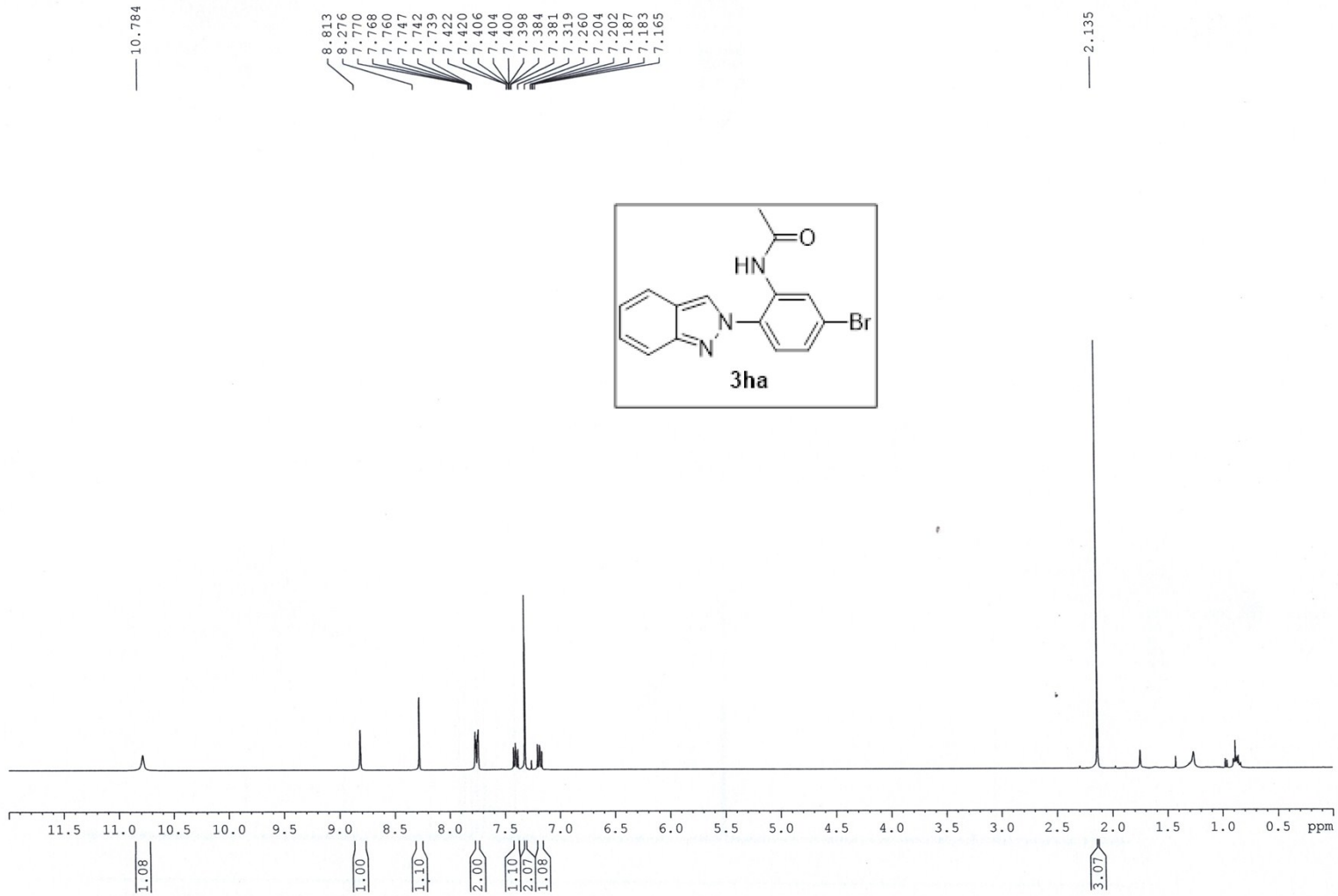
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 346
PROCNO 1

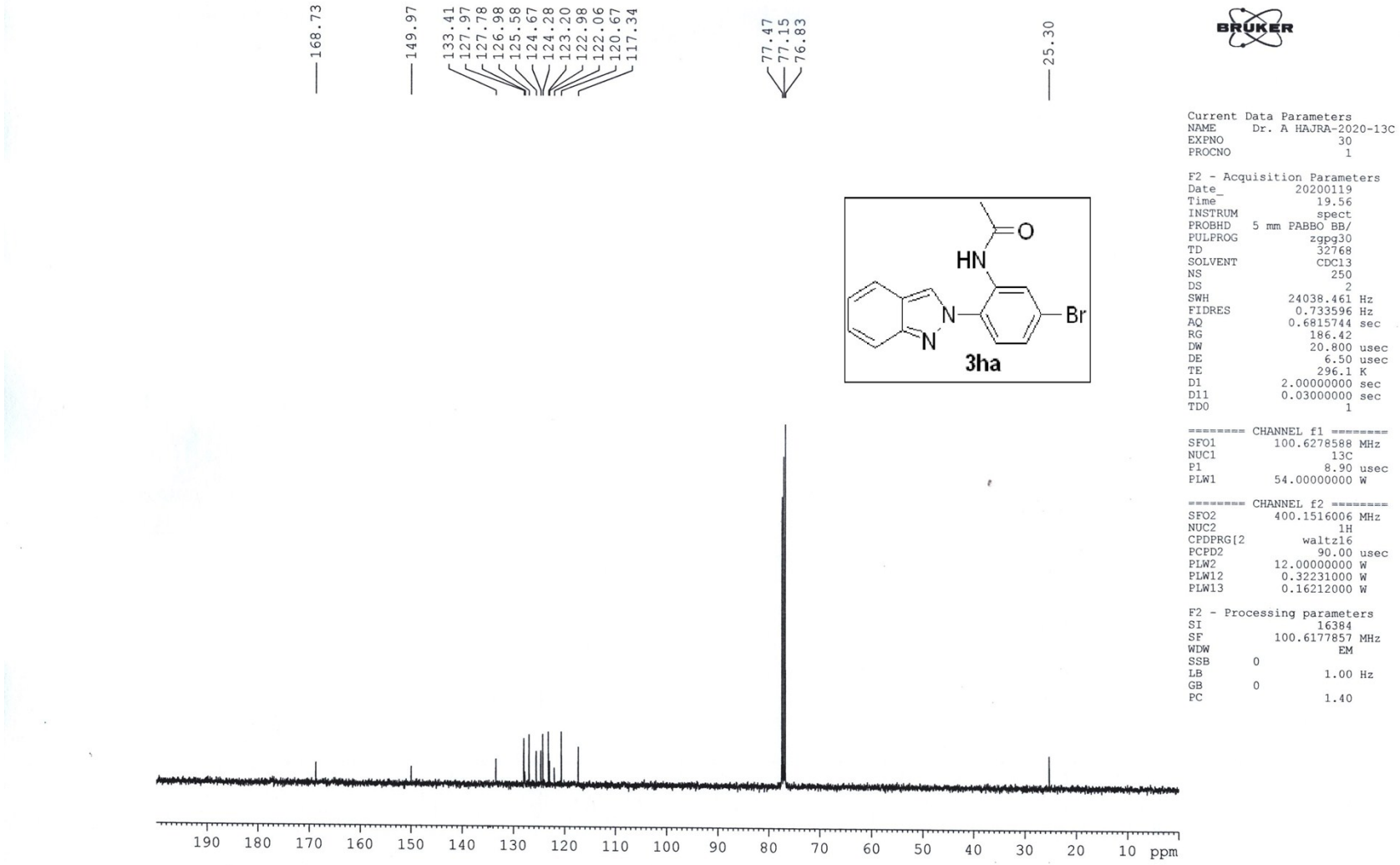
F2 - Acquisition Parameters
Date_ 20190828
Time 20.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 250
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

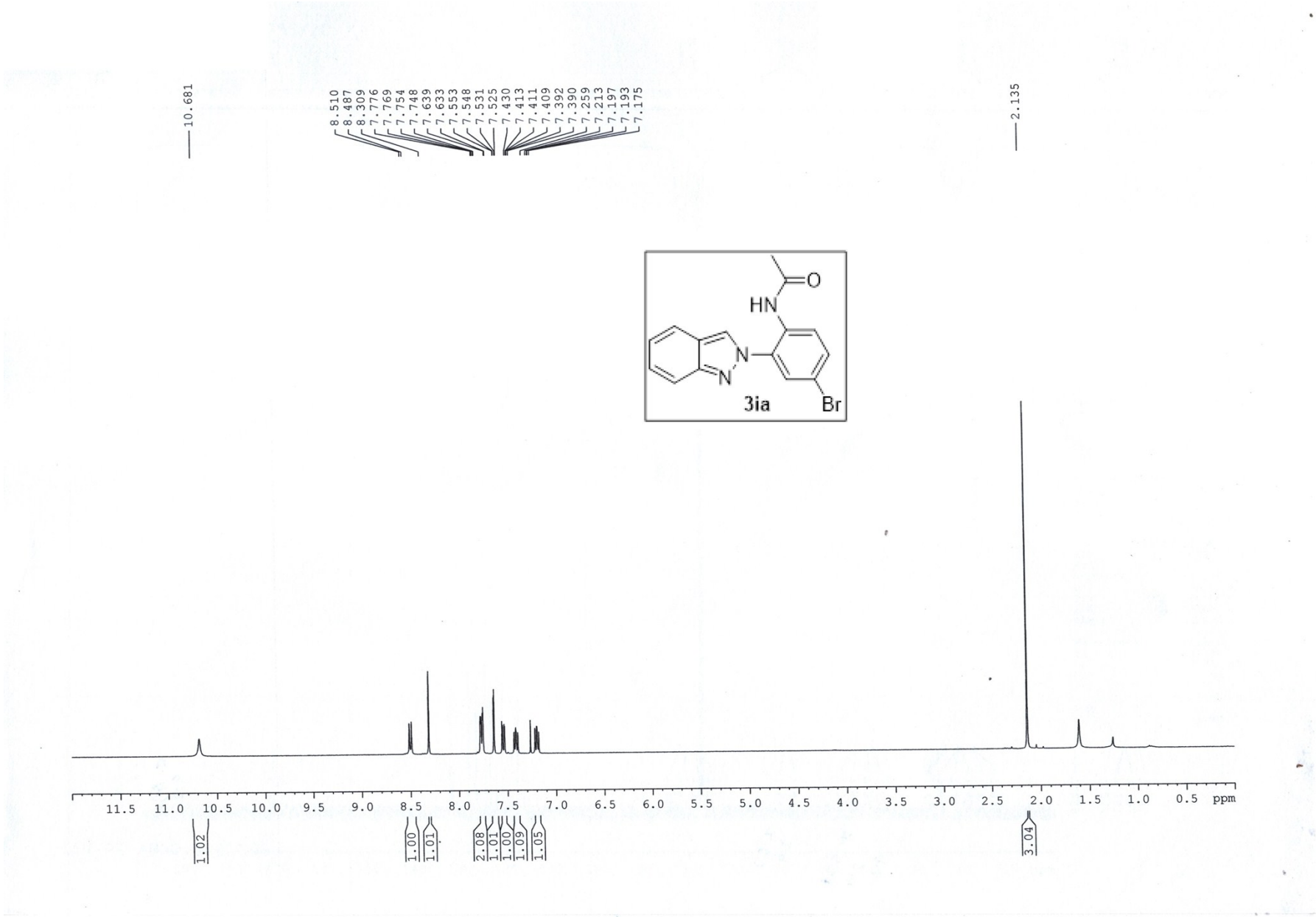
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

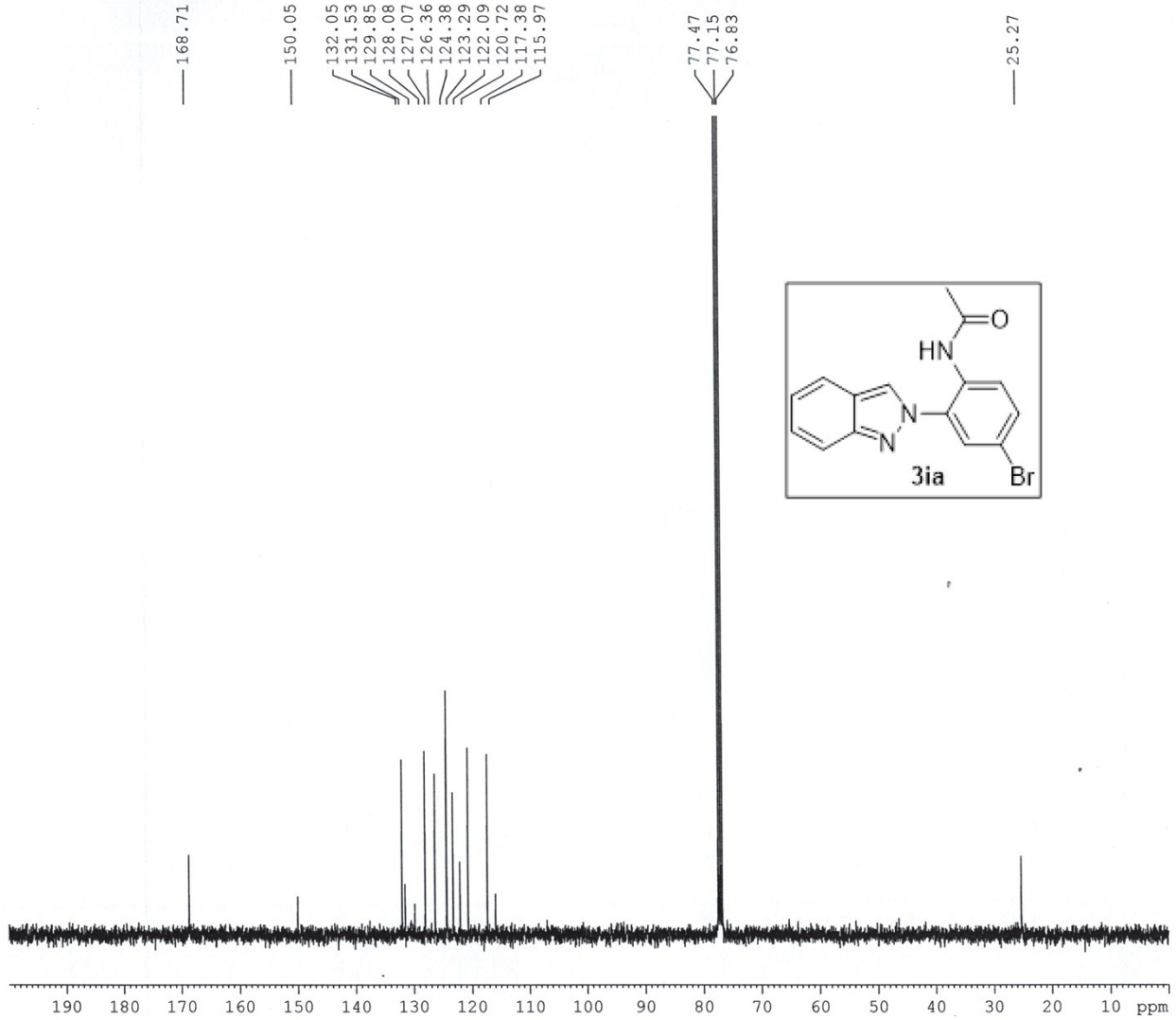
F2 - Processing parameters
SI 16384
SF 100.6177889 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.784
8.813
8.276
7.770
7.768
7.760
7.747
7.742
7.739
7.422
7.420
7.406
7.404
7.400
7.398
7.384
7.381
7.319
7.260
7.204
7.202
7.187
7.183
7.165
2.135
HN
O
N
N
Br
3ha
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.08
1.00
1.10
2.00
1.10
2.07
1.08
3.07

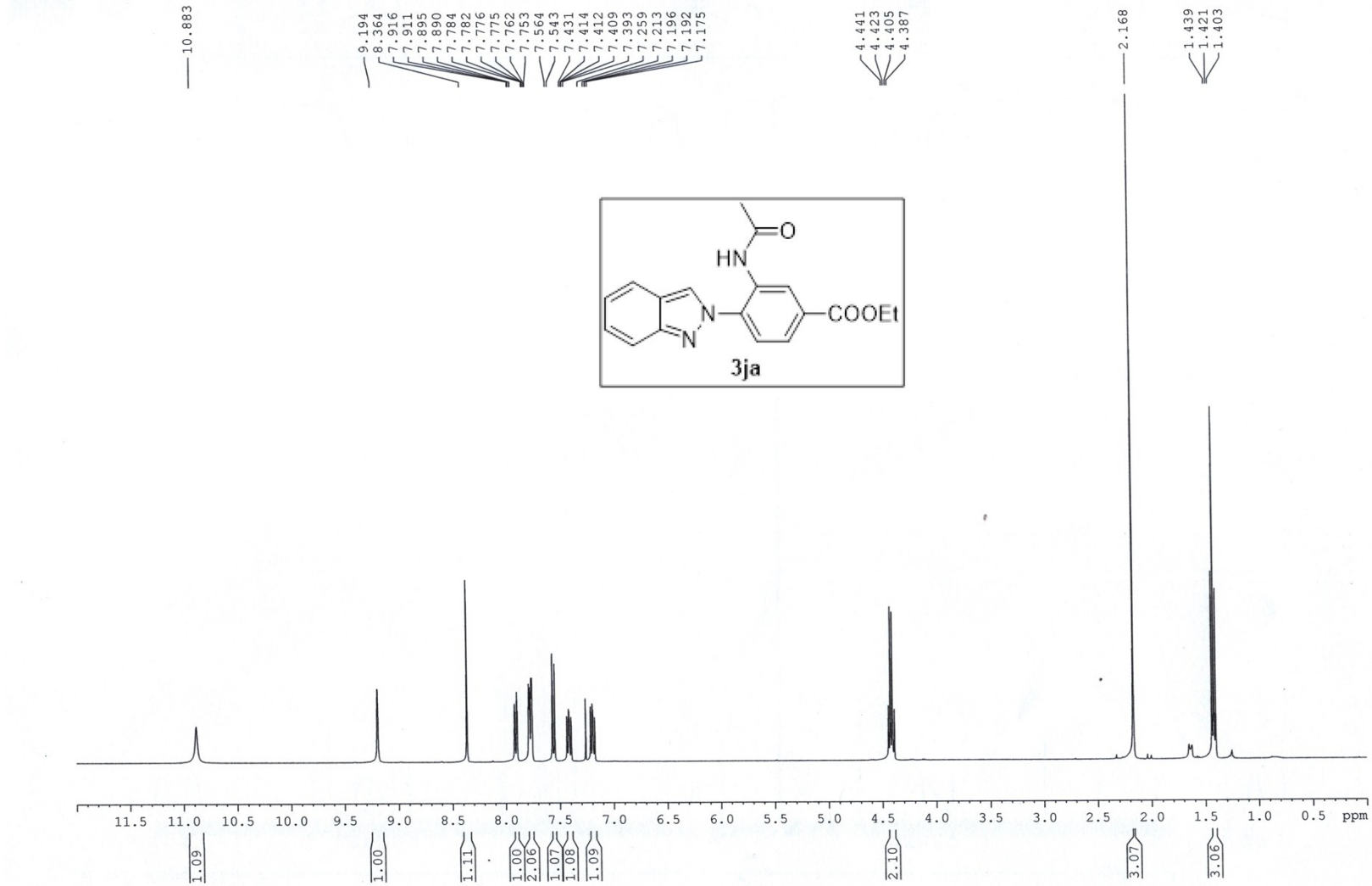


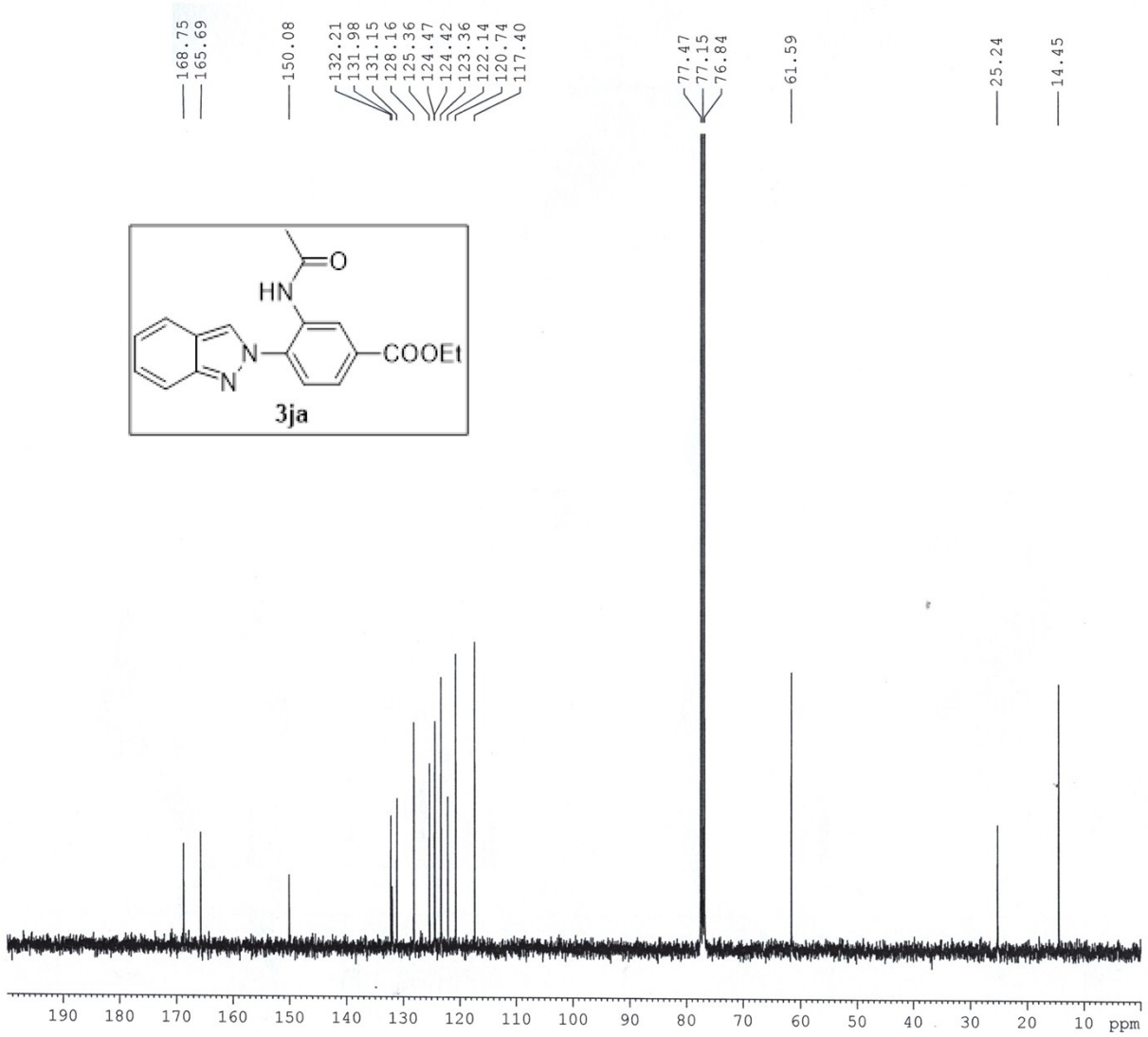

```
Current Data Parameters
NAME      Dr. A HAJRA-2020-13C
EXPNO                       30
PROCNO                       1

F2 - Acquisition Parameters
Date_              20200119
Time                   19.56
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                      32768
SOLVENT               CDCl3
NS                        250
DS                          2
SWH                24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                    186.42
DW                    20.800 usec
DE                     6.50 usec
TE                    296.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1           100.6278588 MHz
NUC1                     13C
P1                      8.90 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2           400.1516006 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2           12.00000000 W
PLW12           0.32231000 W
PLW13           0.16212000 W

F2 - Processing parameters
SI                       16384
SF             100.6177857 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```


10.681
8.510
8.487
8.309
7.776
7.769
7.754
7.748
7.639
7.633
7.553
7.548
7.531
7.525
7.430
7.413
7.411
7.409
7.392
7.390
7.259
7.213
7.197
7.193
7.175
2.135
HN
O
N
N
3ia
Br
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02
1.00
1.01
2.08
1.01
1.00
1.09
1.05
3.04




```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  369
PROCNO                   1

F2 - Acquisition Parameters
Date_           20190918
Time                8.52
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  300
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                148.91
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                 8.90 usec
PLW1        54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.00000000 W
PLW12        0.32231000 W
PLW13        0.16212000 W

F2 - Processing parameters
SI                  16384
SF           100.6177846 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```


10.883
9.194
8.364
7.916
7.911
7.895
7.890
7.784
7.782
7.776
7.775
7.762
7.753
7.564
7.543
7.431
7.414
7.412
7.409
7.393
7.259
7.213
7.196
7.192
7.175
4.441
4.423
4.405
4.387
2.168
1.439
1.421
1.403
HN
O
N
N
COOEt
3ja
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.09
1.00
1.11
1.00
2.06
1.07
1.08
1.09
2.10
3.07
3.06



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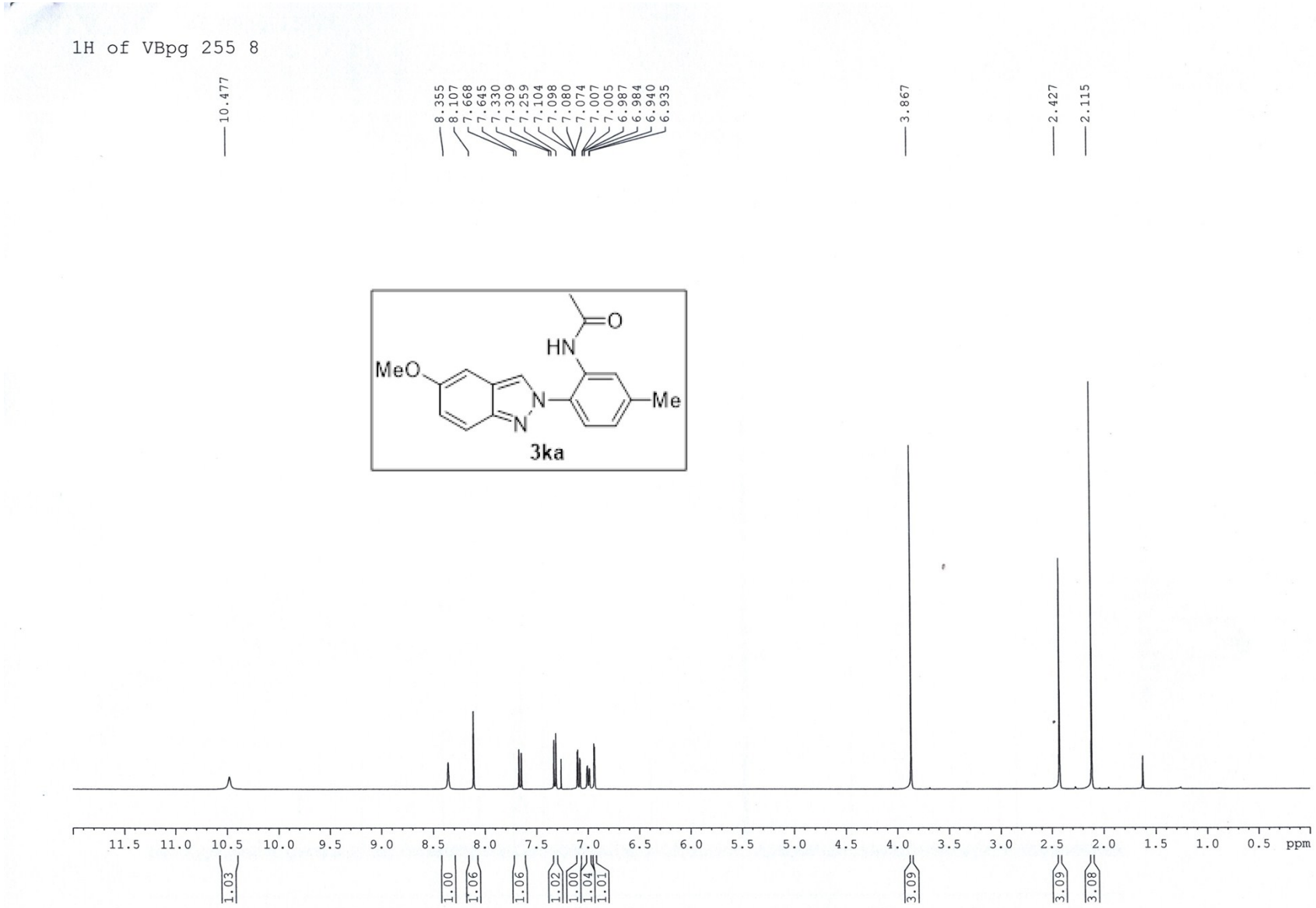
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 312
PROCNO 1

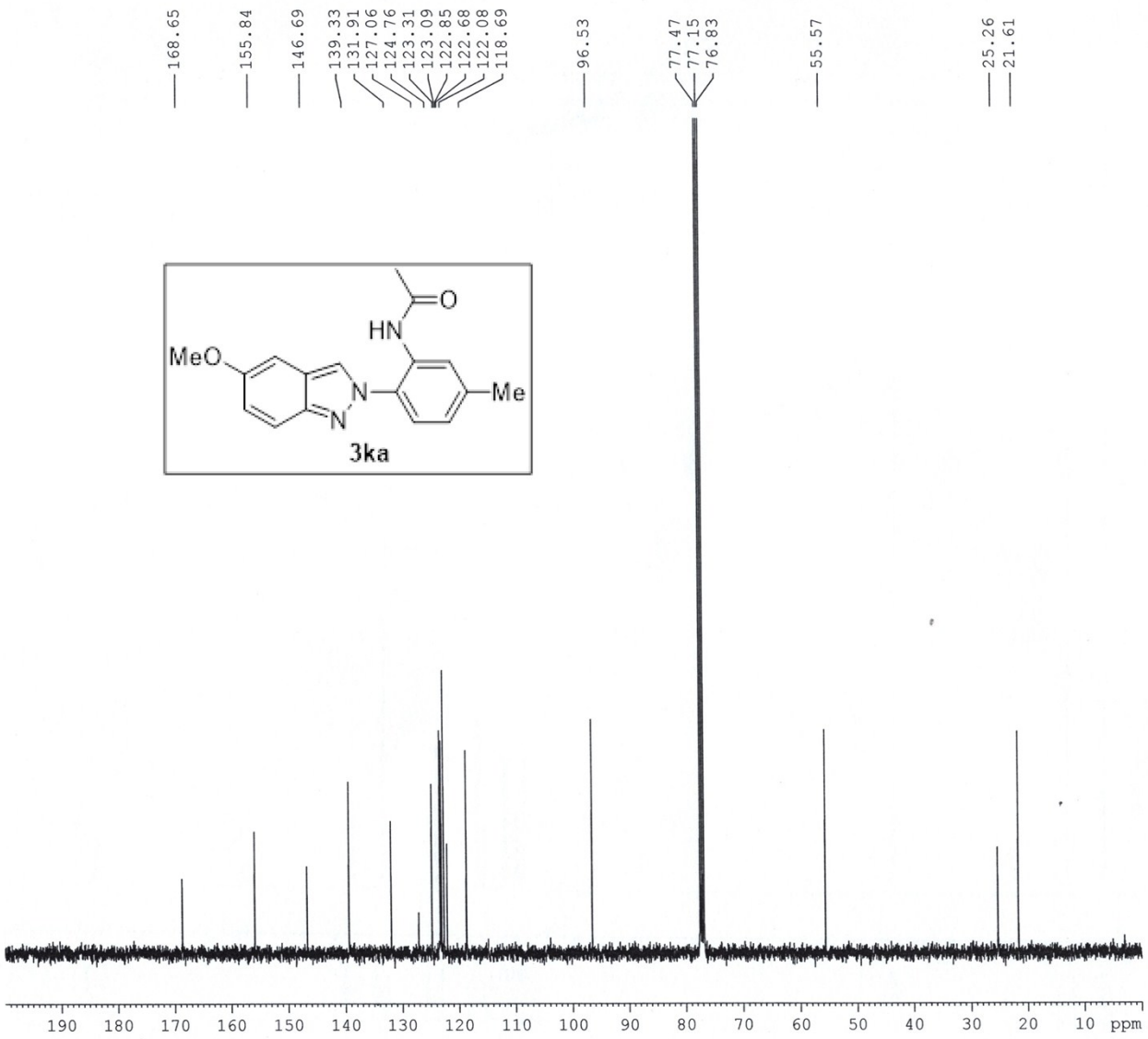
F2 - Acquisition Parameters
Date_ 20190807
Time 13.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177844 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


1H of VBpg 255 8
10.477
8.355
8.107
7.668
7.645
7.330
7.309
7.259
7.104
7.098
7.080
7.074
7.007
7.005
6.987
6.984
6.940
6.935
3.867
2.427
2.115
MeO
HN
O
N
N
Me
3ka
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.00
1.06
1.06
1.02
1.00
1.04
1.01
3.09
3.09
3.08



BRUKER

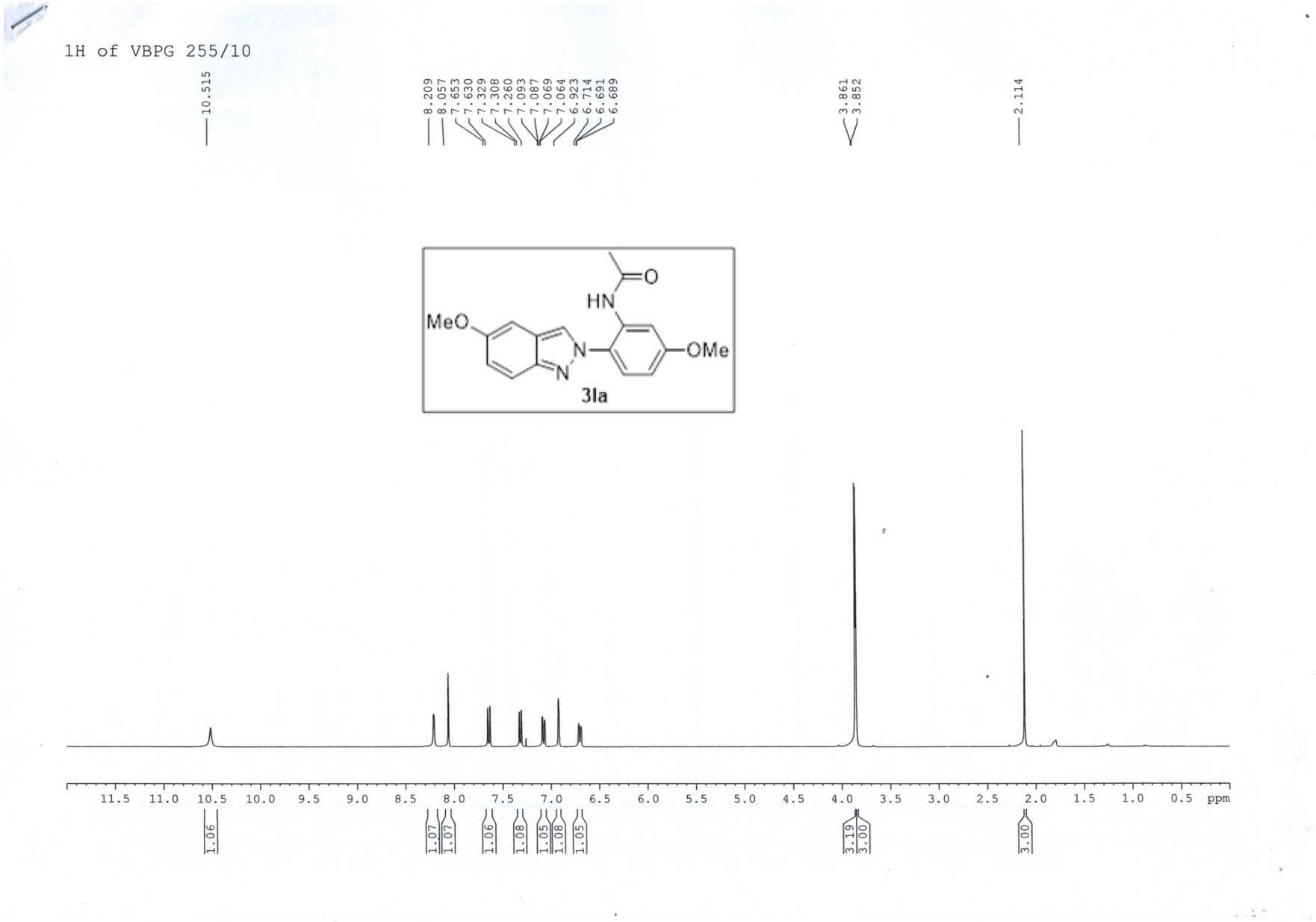
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 314
PROCNO 1

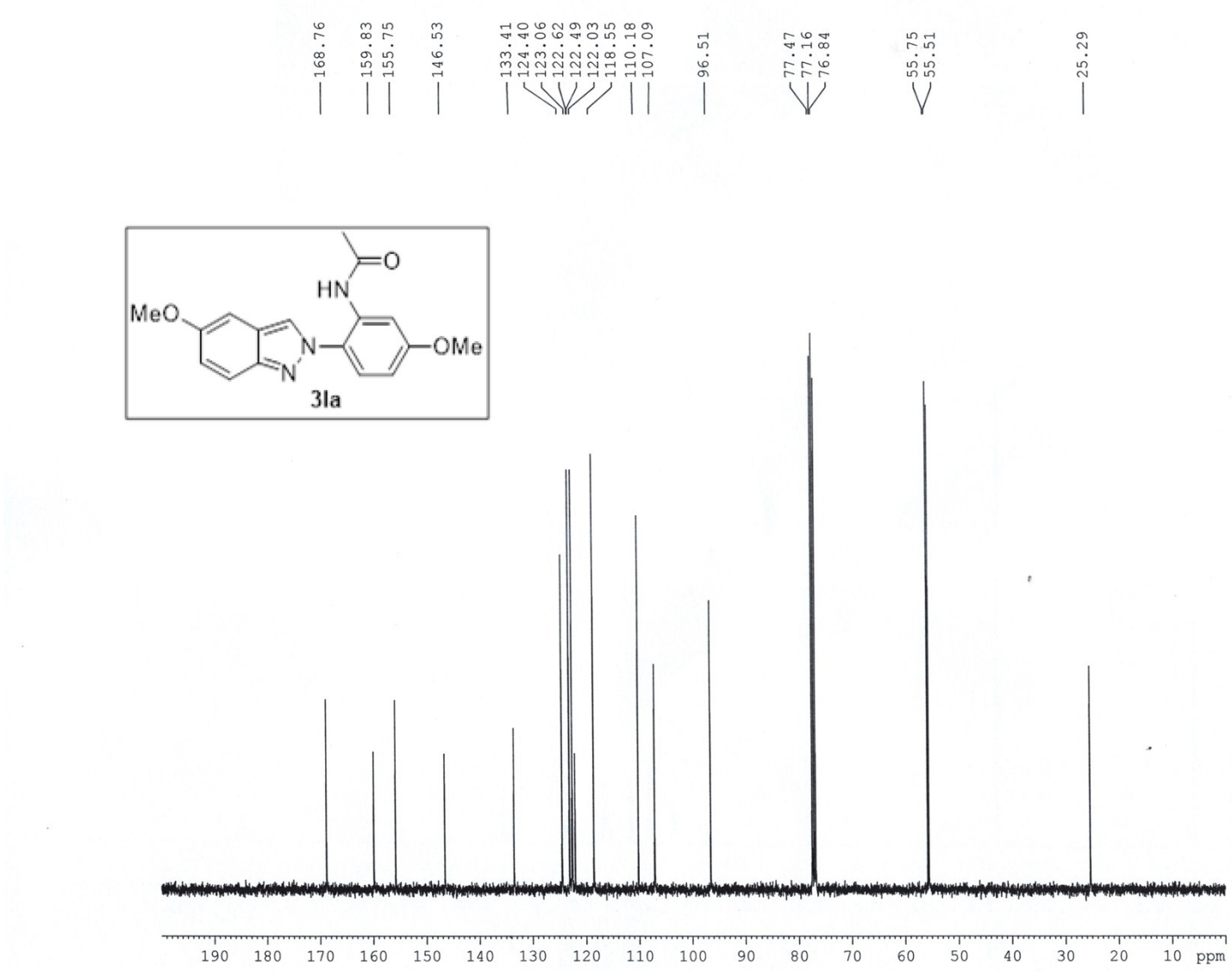
F2 - Acquisition Parameters
Date_ 20190809
Time 12.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 460
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 106.66
DW 20.800 usec
DE 6.50 usec
TE 302.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


1H of VBPG 255/10
10.515
8.209
8.057
7.653
7.630
7.329
7.308
7.260
7.093
7.087
7.069
7.064
6.923
6.714
6.691
6.689
3.861
3.852
2.114
MeO
HN
O
N
N
OMe
3Ia
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.06
1.07
1.07
1.06
1.08
1.05
1.08
1.05
3.19
3.00
3.00



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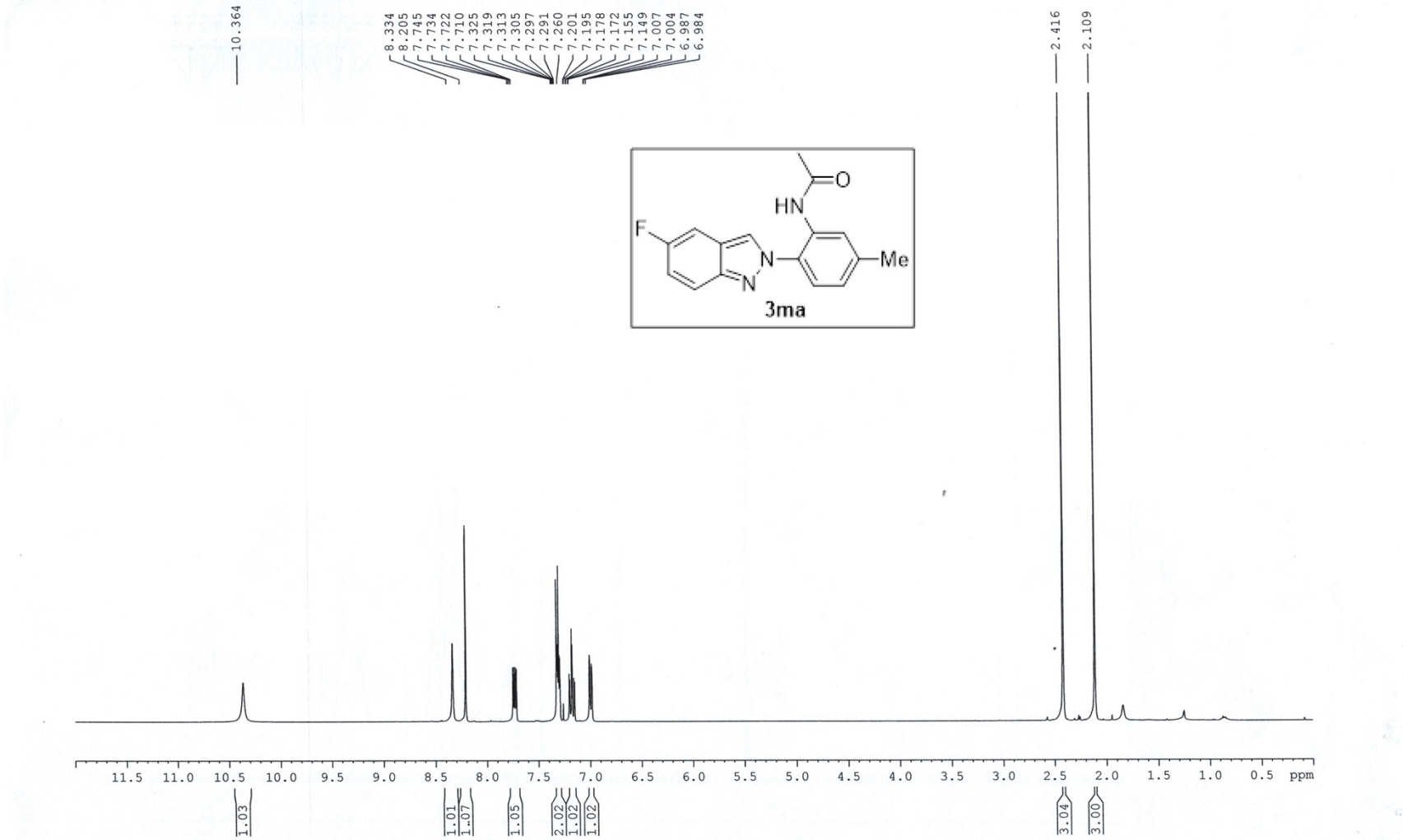
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 333
PROCNO 1

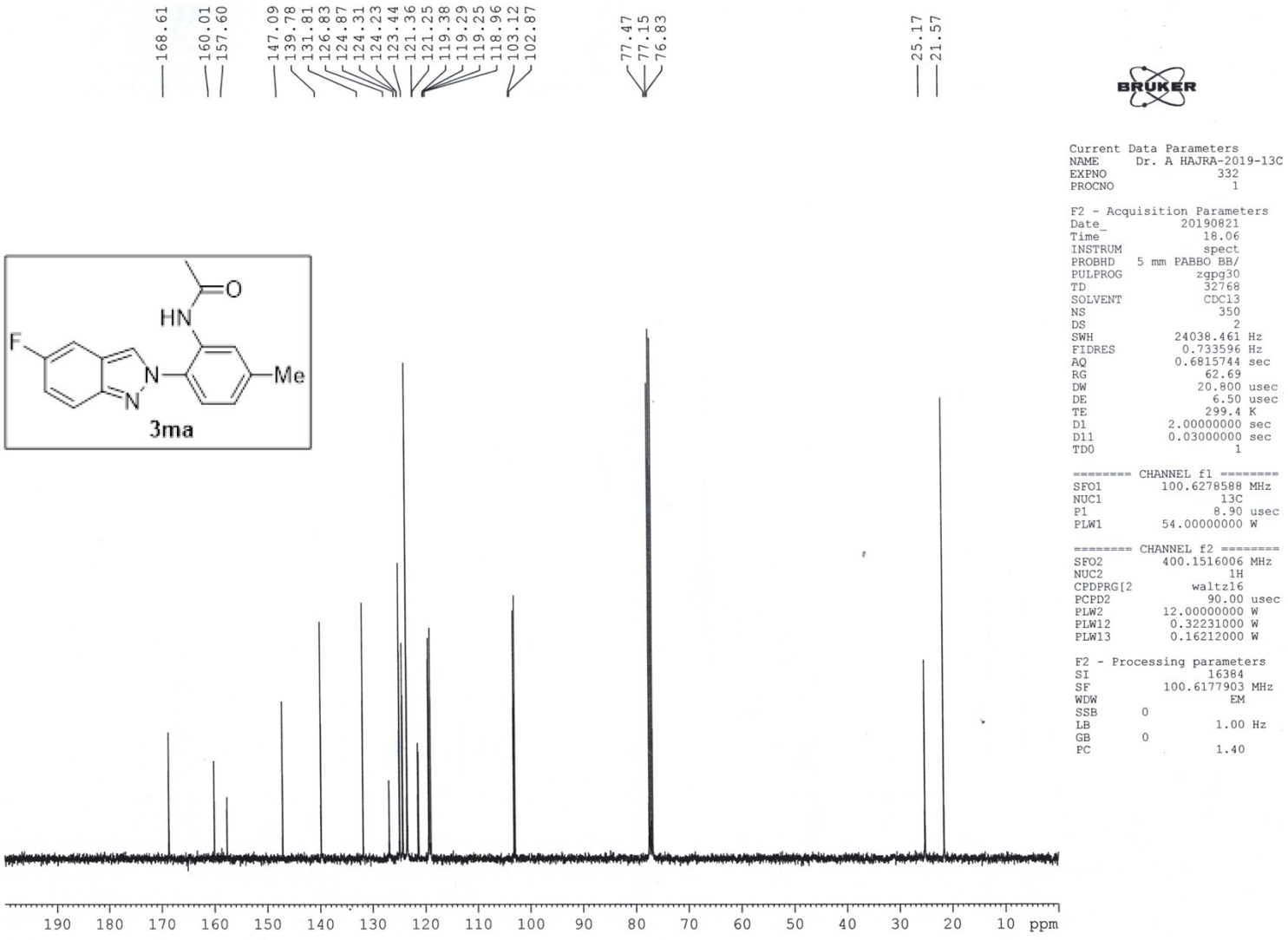
F2 - Acquisition Parameters
Date_ 20190822
Time 11.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 300
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 301.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177886 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.364
8.334
8.205
7.745
7.734
7.722
7.710
7.325
7.319
7.313
7.305
7.297
7.291
7.260
7.201
7.195
7.178
7.172
7.155
7.149
7.007
7.004
6.987
6.984
2.416
2.109
O
HN
F
N
N
Me
3ma
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.01
1.07
1.05
2.02
1.02
1.02
3.04
3.00



BRUKER

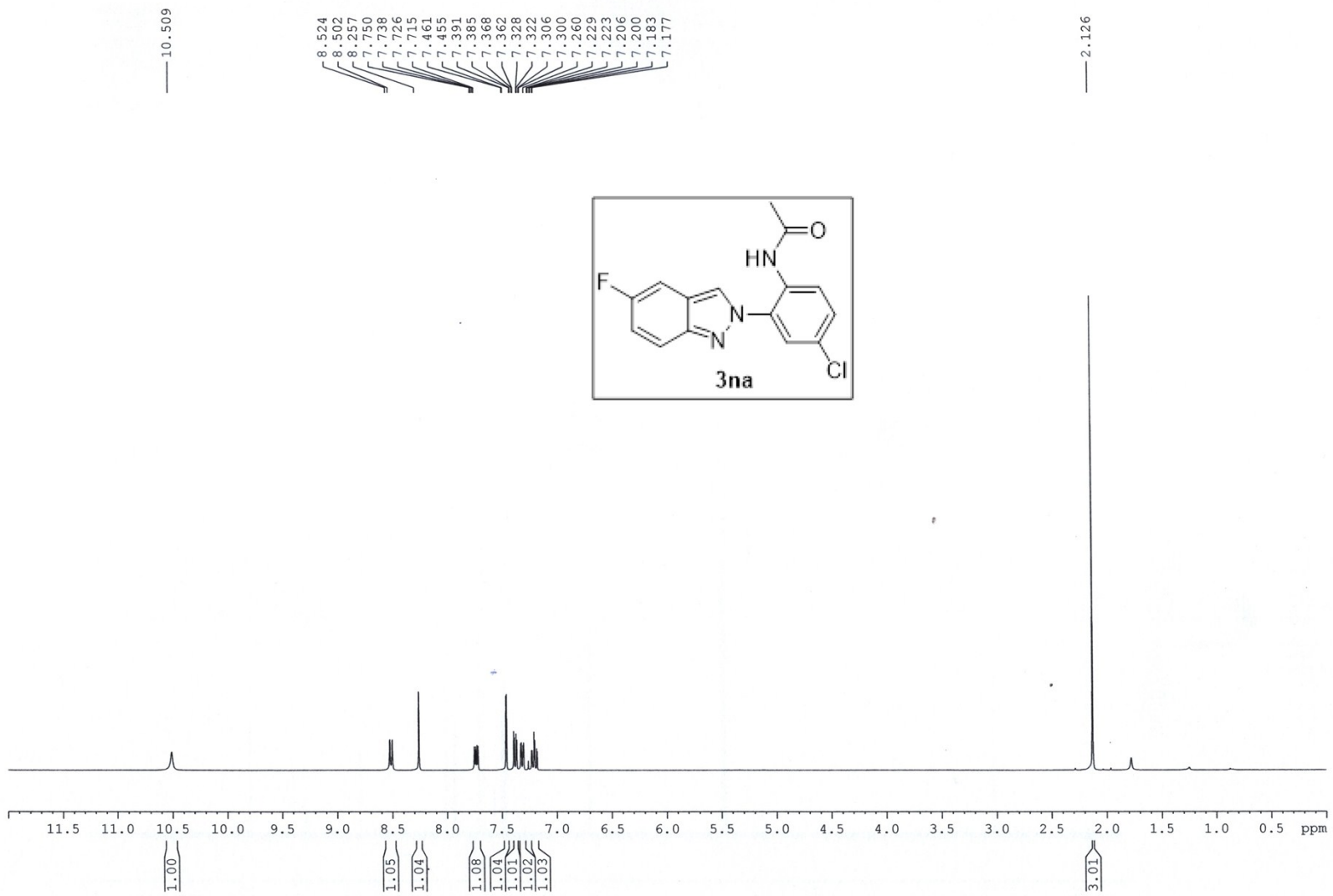
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 332
PROCNO 1

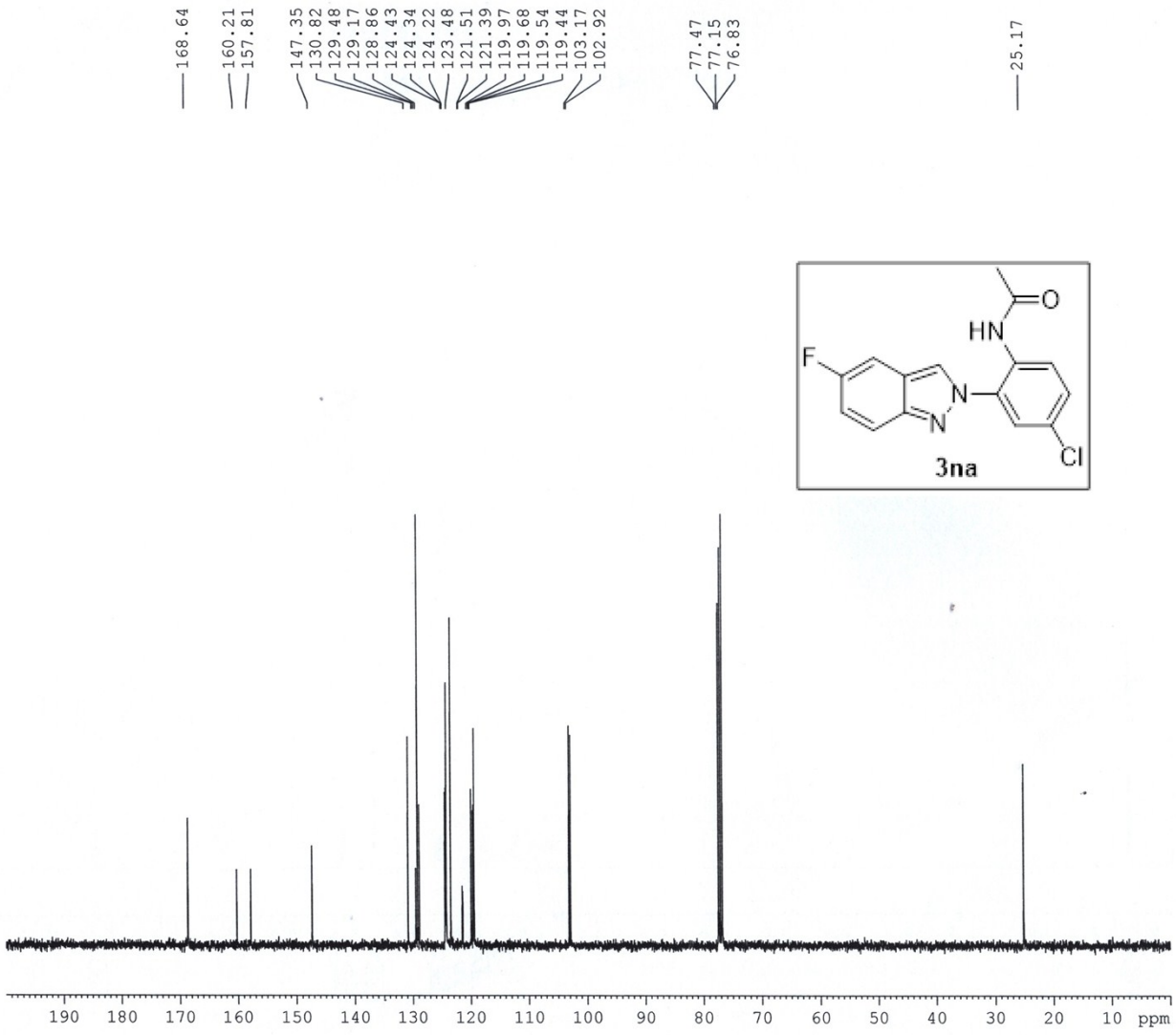
F2 - Acquisition Parameters
Date_ 20190821
Time 18.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 350
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177903 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.509
8.524
8.502
8.257
7.750
7.738
7.726
7.715
7.461
7.455
7.391
7.385
7.368
7.362
7.328
7.322
7.306
7.300
7.260
7.229
7.223
7.206
7.200
7.183
7.177
2.126
O
HN
F
N
N
Cl
3na
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.05
1.04
1.08
1.04
1.01
1.02
1.03
3.01



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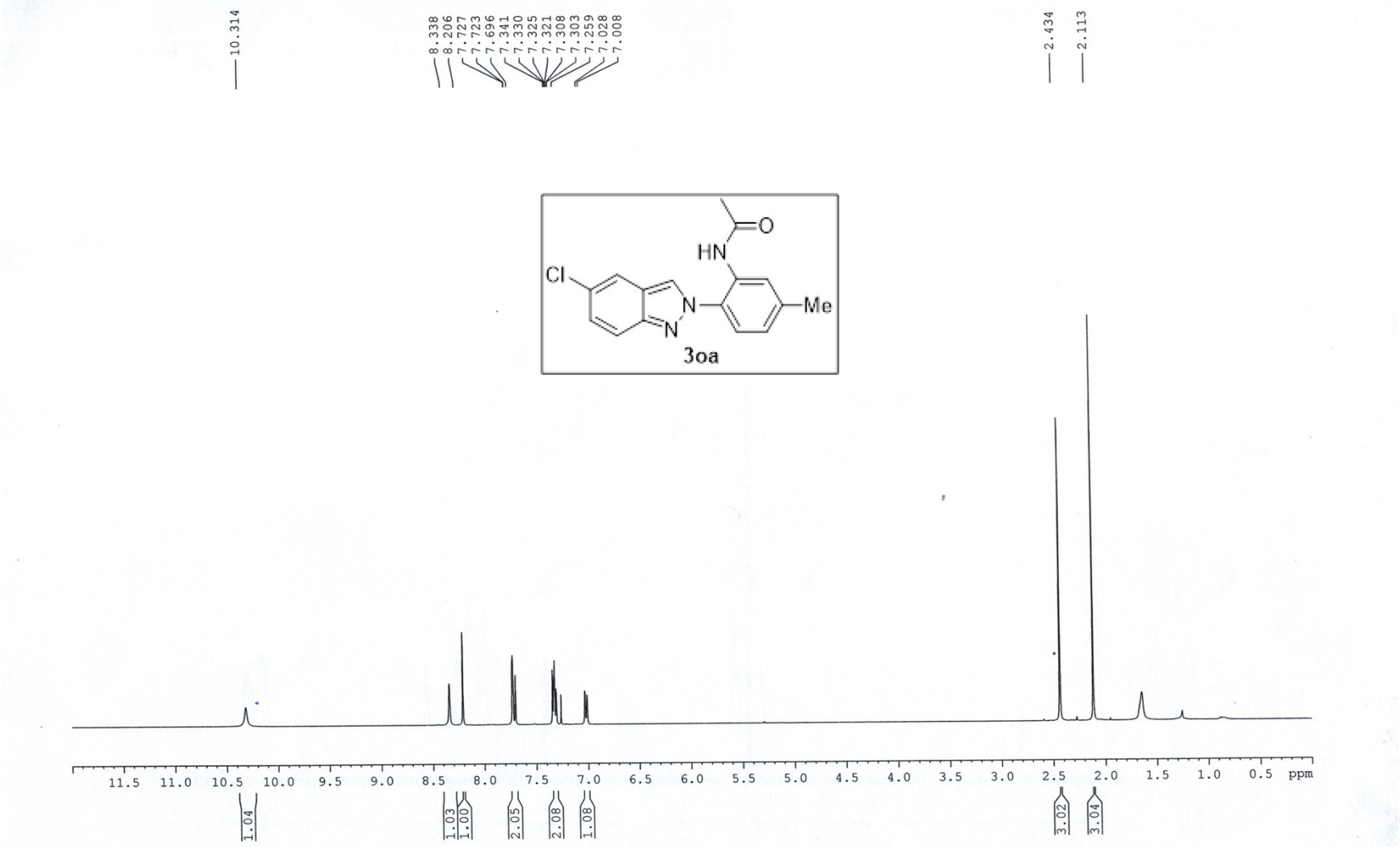
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 354
PROCNO 1

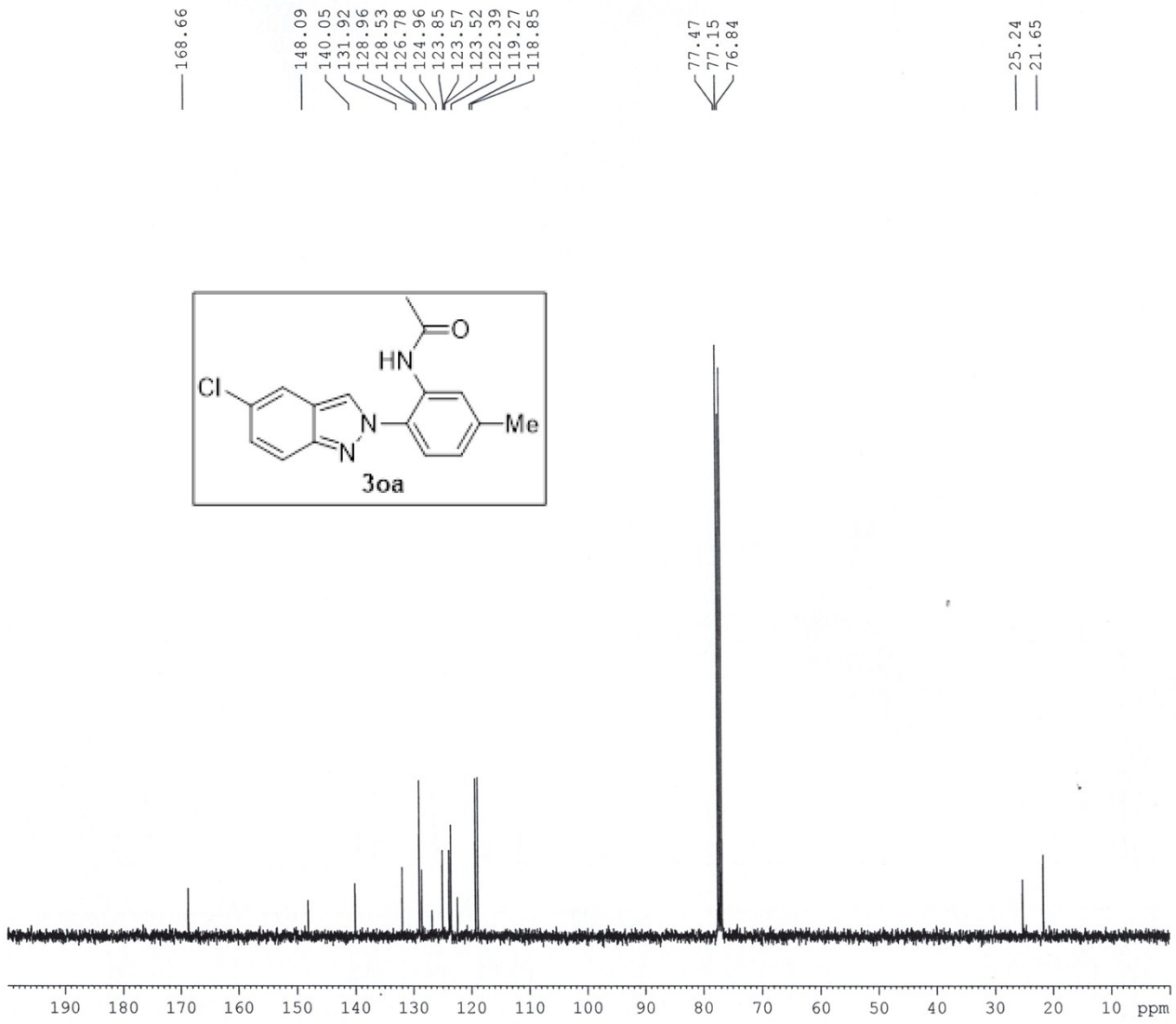
F2 - Acquisition Parameters
Date_ 20190909
Time 9.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

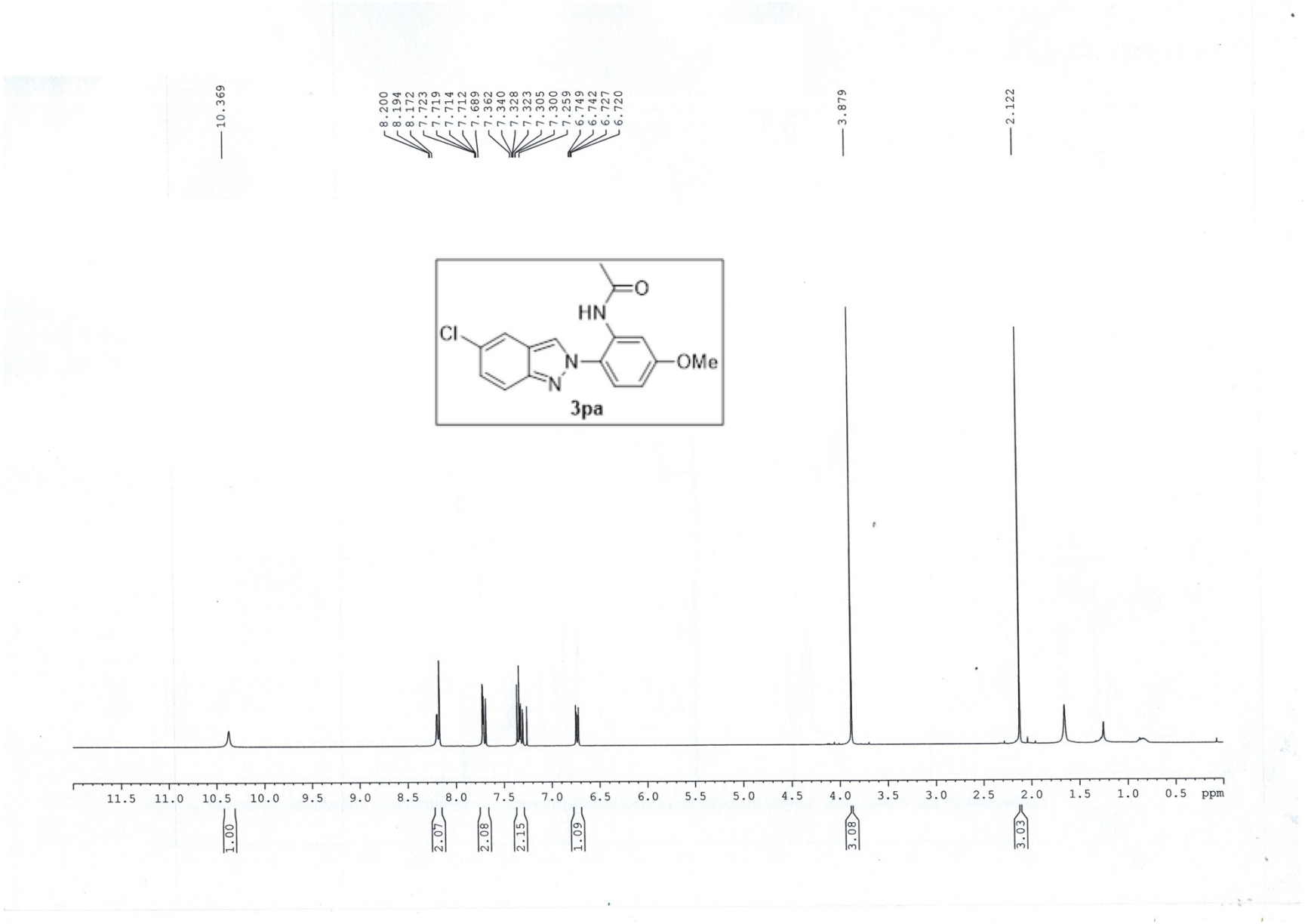
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

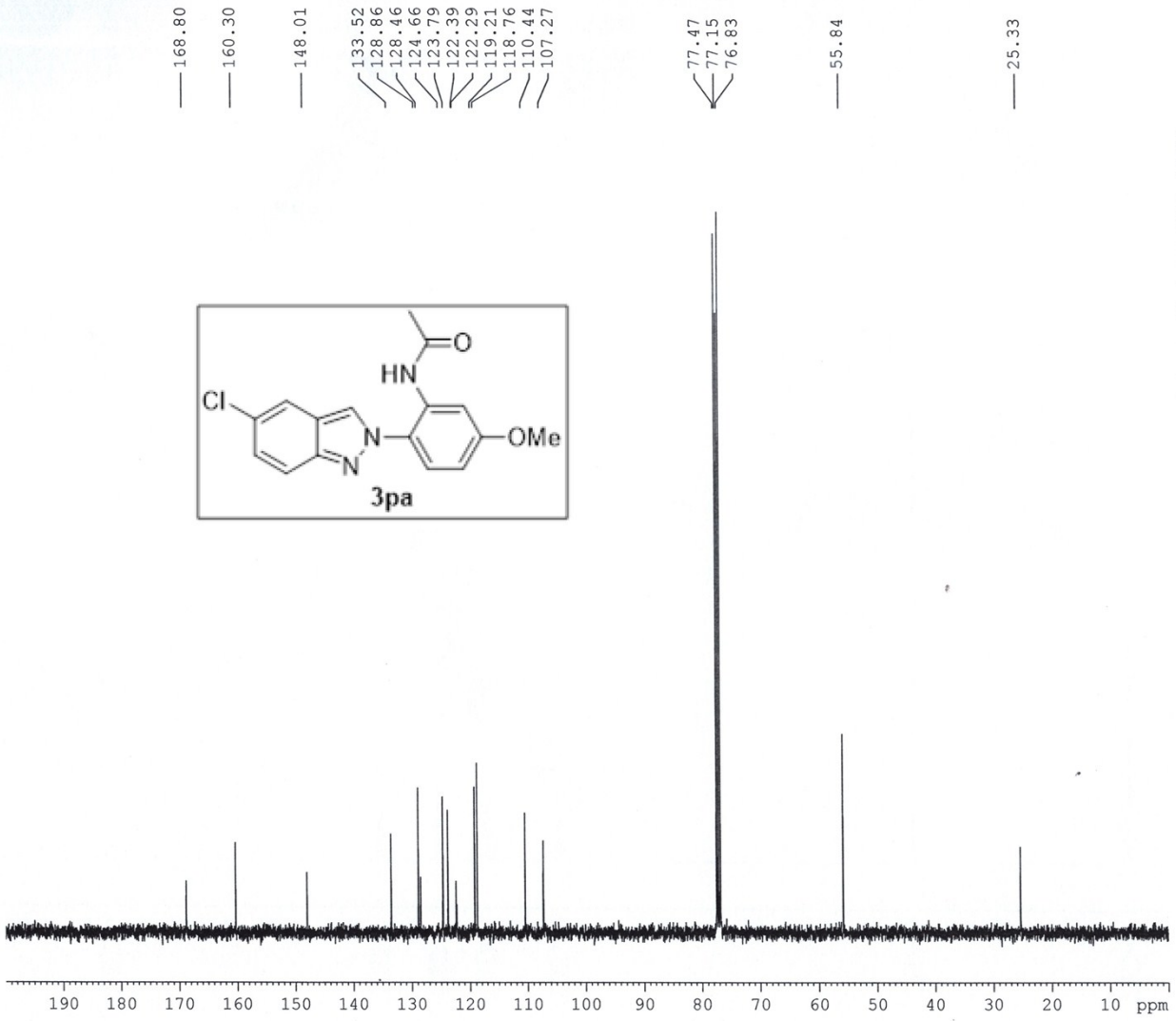
F2 - Processing parameters
SI 16384
SF 100.6177886 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.314
8.338
8.206
7.727
7.723
7.696
7.341
7.330
7.325
7.321
7.308
7.303
7.259
7.028
7.008
2.434
2.113
HN
O
Cl
N
N
Me
3oa
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.04
1.03
1.00
2.05
2.08
1.08
3.02
3.04



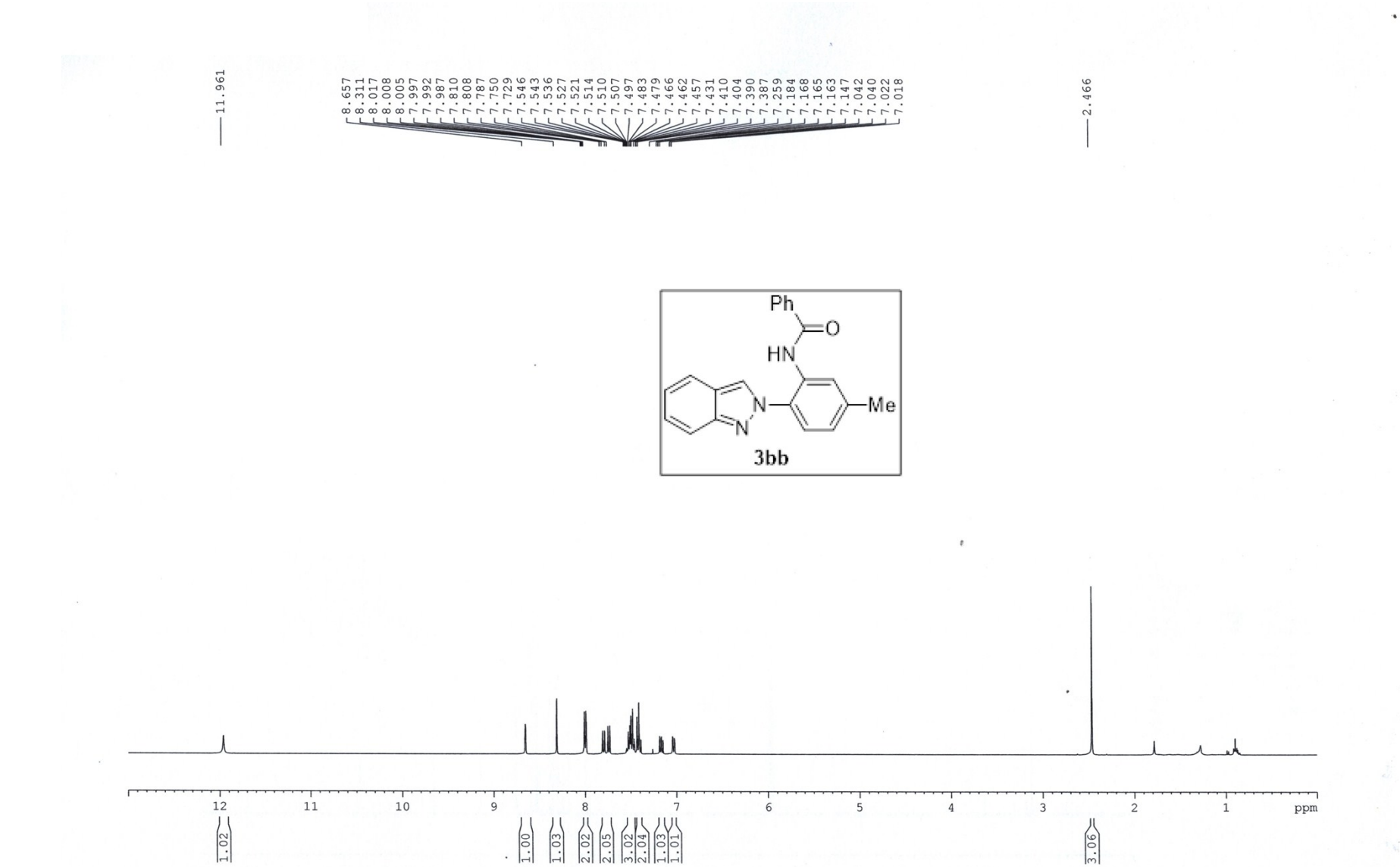

```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  359
PROCNO                   1

F2 - Acquisition Parameters
Date_           20190910
Time                7.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  256
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                106.66
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                 8.90 usec
PLW1         54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.32231000 W
PLW13         0.16212000 W

F2 - Processing parameters
SI                 16384
SF           100.6177848 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

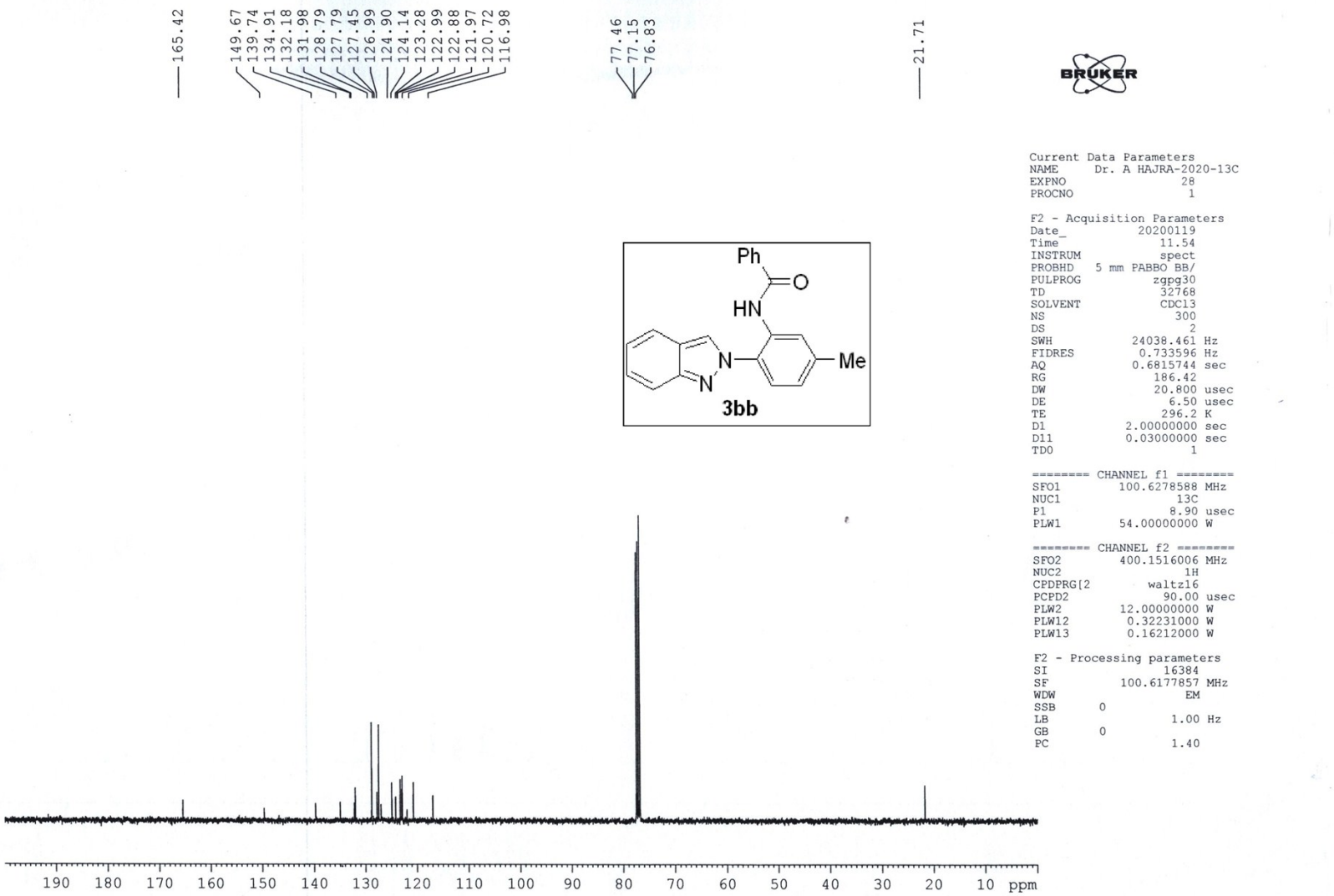

10.369
8.200
8.194
8.172
7.723
7.719
7.714
7.712
7.689
7.362
7.340
7.328
7.323
7.305
7.300
7.259
6.749
6.742
6.727
6.720
3.879
2.122
HN
O
Cl
N
N
OMe
3pa
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
2.07
2.08
2.15
1.09
3.08
3.03



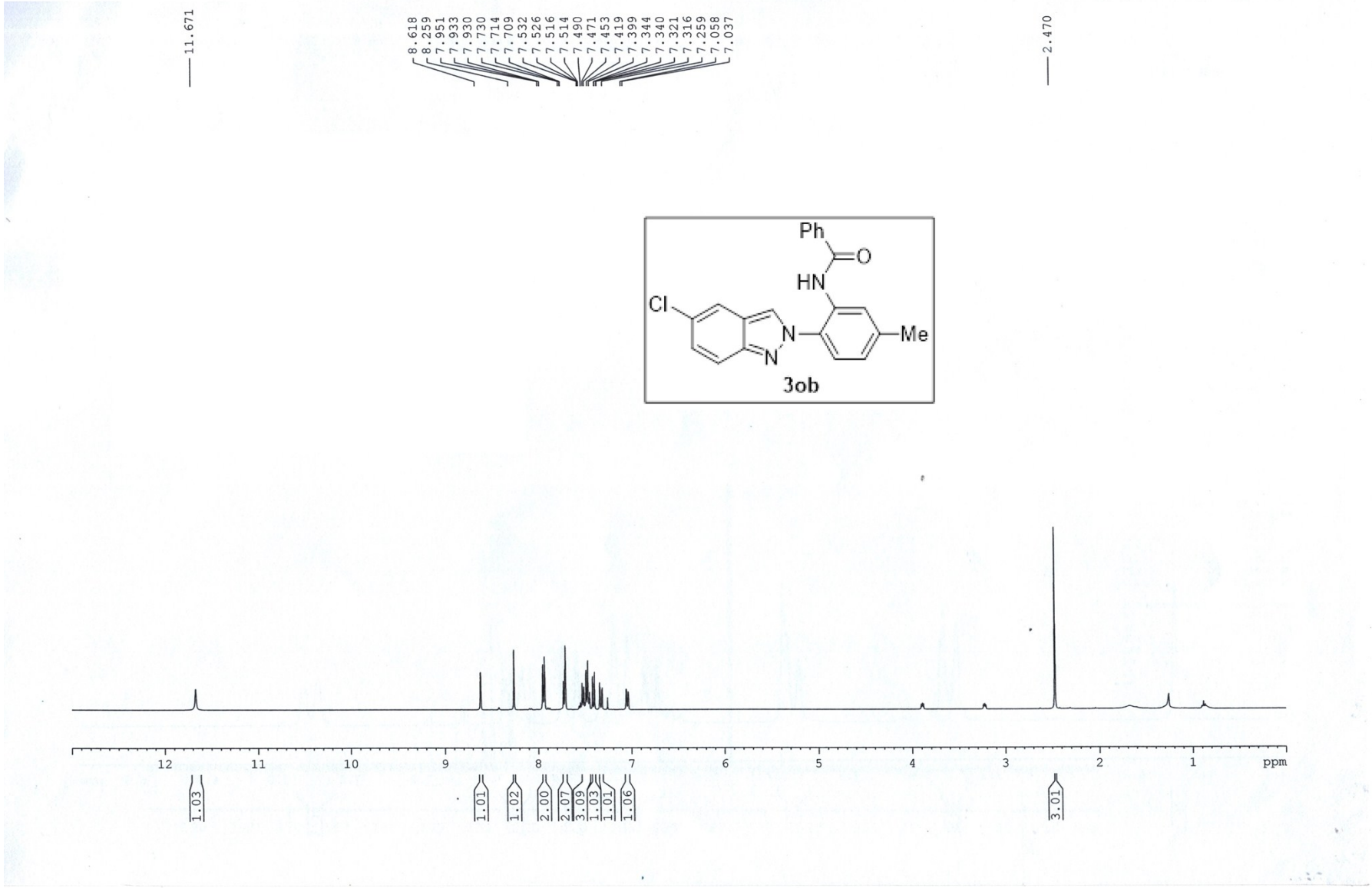
BRUKER

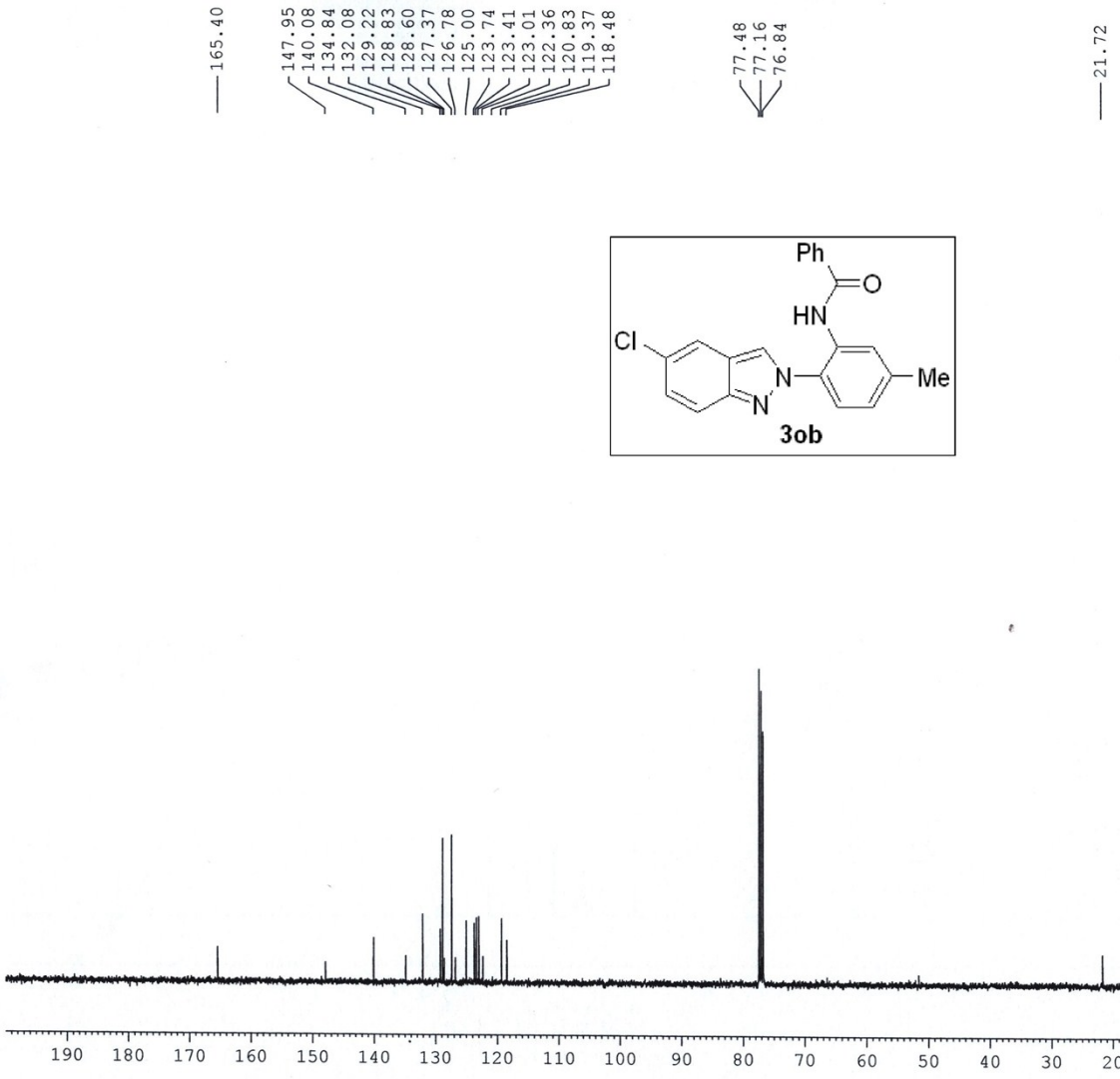
```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  355
PROCNO                   1

F2 - Acquisition Parameters
Date_           20190909
Time               9.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              32768
SOLVENT          CDCl3
NS                 256
DS                   2
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               120.16
DW               20.800 usec
DE                6.50 usec
TE               298.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6278588 MHz
NUC1               13C
P1                8.90 usec
PLW1       54.00000000 W

======== CHANNEL f2 ========
SFO2        400.1516006 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2       12.00000000 W
PLW12       0.32231000 W
PLW13       0.16212000 W

F2 - Processing parameters
SI                16384
SF          100.6177854 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```


11.961
8.657
8.311
8.017
8.008
8.005
7.997
7.992
7.987
7.810
7.808
7.787
7.750
7.729
7.546
7.543
7.536
7.527
7.521
7.514
7.510
7.507
7.497
7.483
7.479
7.466
7.462
7.457
7.431
7.410
7.404
7.390
7.387
7.259
7.184
7.168
7.165
7.163
7.147
7.042
7.040
7.022
7.018
2.466
Ph
O
HN
N
N
Me
3bb
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.02
1.00
1.03
2.02
2.05
3.02
2.04
1.03
1.01
3.06

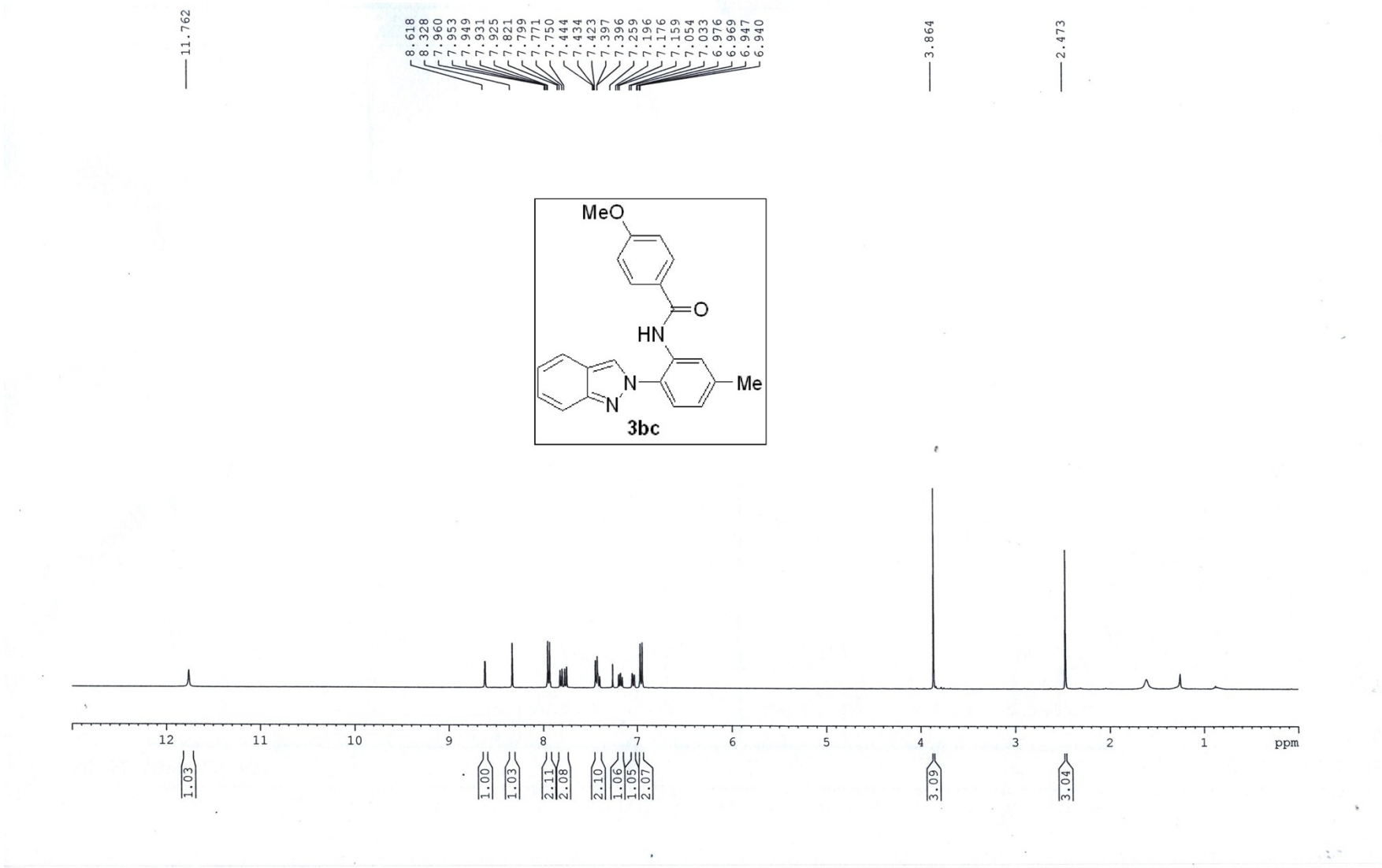


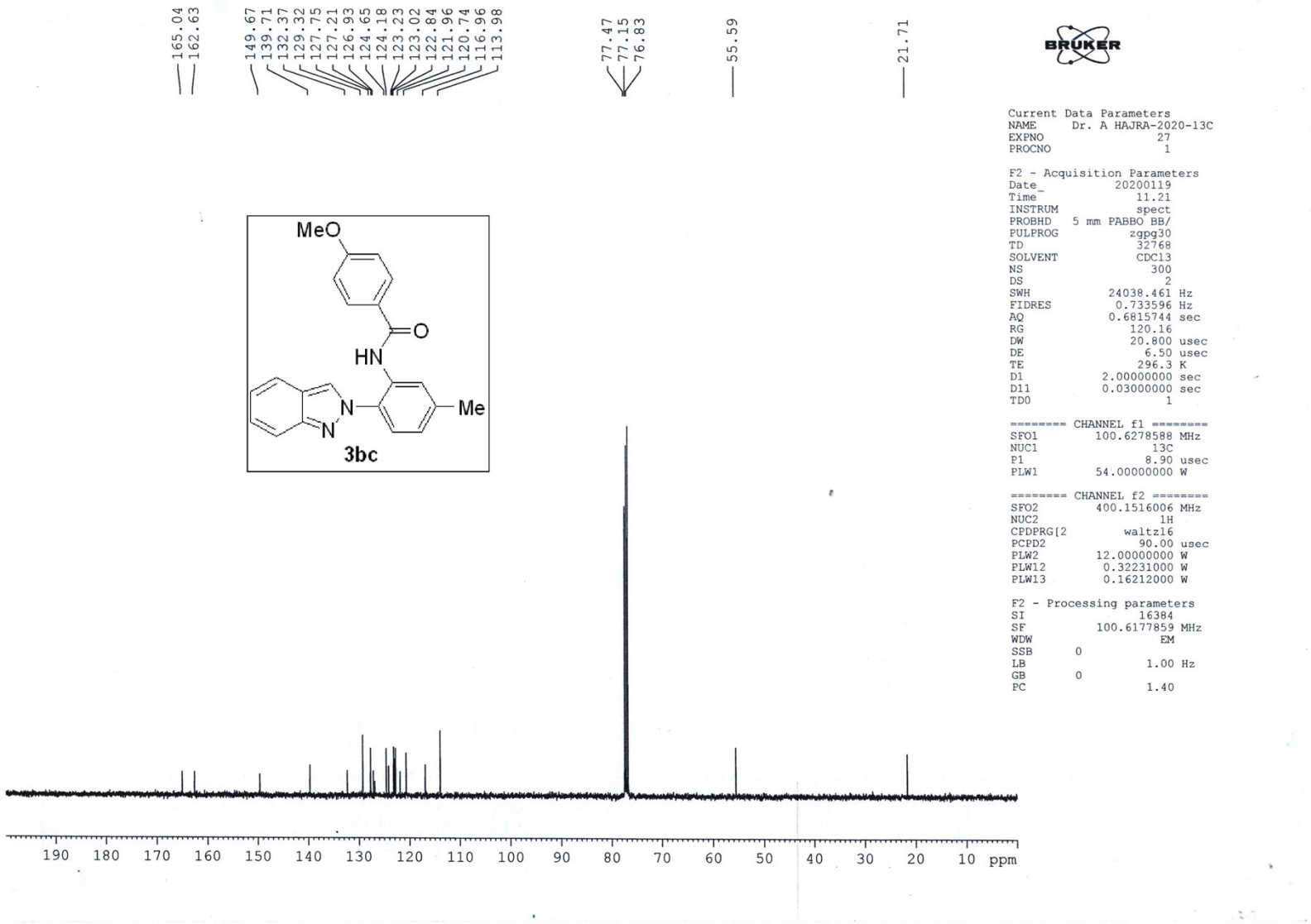

```
Current Data Parameters
NAME      Dr. A HAJRA-2020-13C
EXPNO                   28
PROCNO                   1

F2 - Acquisition Parameters
Date_           20200119
Time               11.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  300
DS                    2
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                186.42
DW                20.800 usec
DE                 6.50 usec
TE                296.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6278588 MHz
NUC1                13C
P1                 8.90 usec
PLW1         54.00000000 W

======== CHANNEL f2 ========
SFO2         400.1516006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.32231000 W
PLW13         0.16212000 W

F2 - Processing parameters
SI                 16384
SF           100.6177857 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```


11.671
8.618
8.259
7.951
7.933
7.930
7.730
7.714
7.709
7.532
7.526
7.516
7.514
7.490
7.471
7.453
7.419
7.399
7.344
7.340
7.321
7.316
7.259
7.058
7.037
2.470
Ph
O
HN
Cl
N
N
Me
3ob
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.01
1.02
2.00
2.07
3.09
1.03
1.01
1.06
3.01




```
Current Data Parameters
NAME      Dr. A HAJRA-2020-13C
EXPNO                   29
PROCNO                   1

F2 - Acquisition Parameters
Date_              20200119
Time                 19.30
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    165
DS                      2
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                  186.42
DW                  20.800 usec
DE                   6.50 usec
TE                  295.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6278588 MHz
NUC1                 13C
P1                  8.90 usec
PLW1          54.00000000 W

======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          12.00000000 W
PLW12          0.32231000 W
PLW13          0.16212000 W

F2 - Processing parameters
SI                   16384
SF            100.6177857 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```


11.762
8.618
8.328
7.960
7.953
7.949
7.931
7.925
7.821
7.799
7.771
7.750
7.444
7.434
7.423
7.397
7.396
7.259
7.196
7.176
7.159
7.054
7.033
6.976
6.969
6.947
6.940
3.864
2.473
MeO
O
HN
N
N
Me
3bc
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.00
1.03
2.11
2.08
2.10
1.06
1.05
2.07
3.09
3.04



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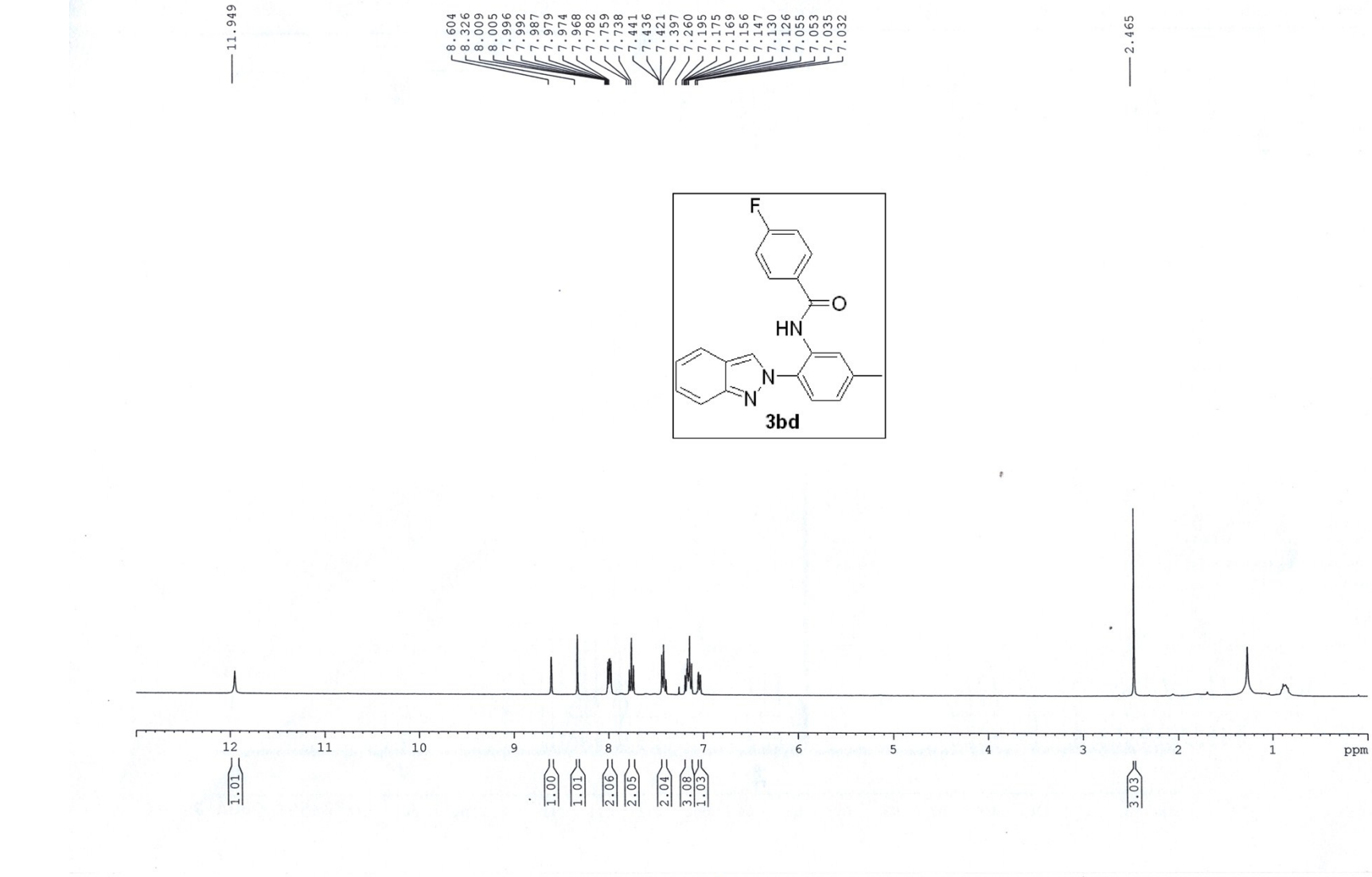
Current Data Parameters
NAME Dr. A HAJRA-2020-13C
EXPNO 27
PROCNO 1

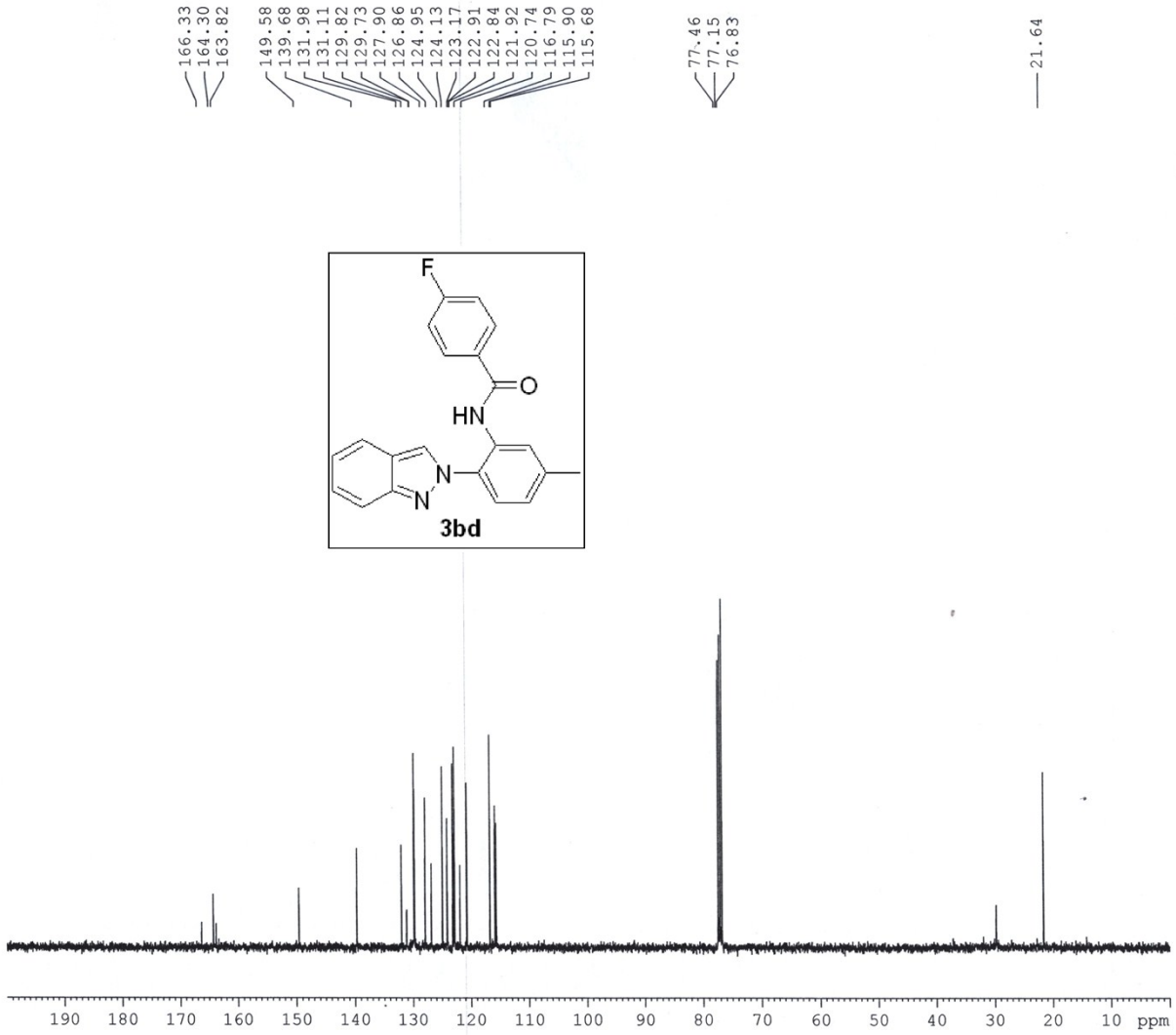
F2 - Acquisition Parameters
Date_ 20200119
Time 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 300
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 120.16
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177859 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


11.949
8.604
8.326
8.009
8.005
7.996
7.992
7.987
7.979
7.974
7.968
7.782
7.759
7.738
7.441
7.436
7.421
7.397
7.260
7.195
7.175
7.169
7.156
7.147
7.130
7.126
7.055
7.053
7.035
7.032
2.465
F
O
HN
N
N
3bd
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.01
1.00
1.01
2.06
2.05
2.04
3.08
1.03
3.03



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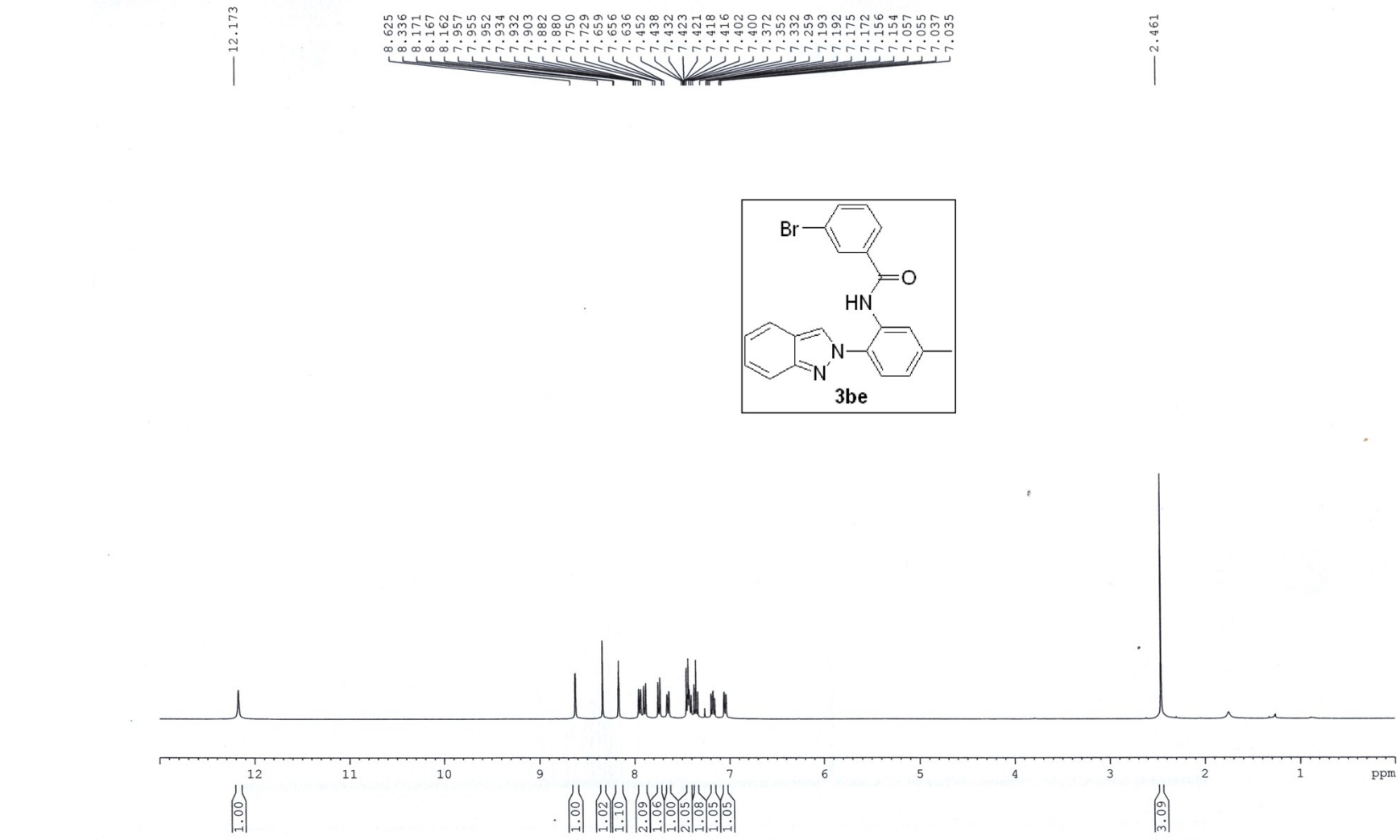
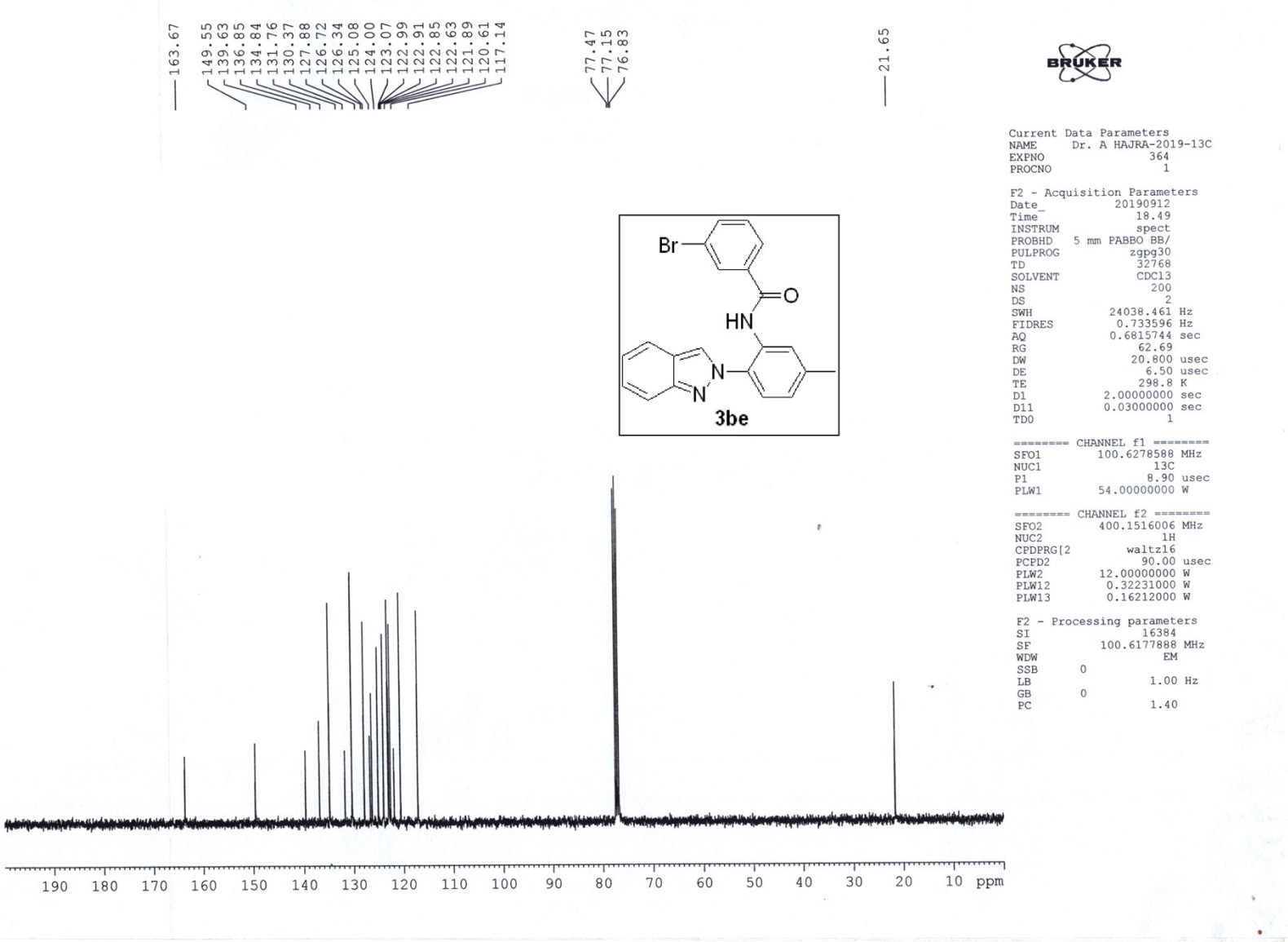
```
Current Data Parameters
NAME      Dr. A HAJRA-2019-13C
EXPNO                  361
PROCNO                   1

F2 - Acquisition Parameters
Date_             20190911
Time                 7.17
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     160
DS                       2
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   57.28
DW                  20.800 usec
DE                   6.50 usec
TE                  299.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6278588 MHz
NUC1                   13C
P1                    8.90 usec
PLW1          54.00000000 W

======== CHANNEL f2 ========
SFO2           400.1516006 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2          12.00000000 W
PLW12          0.32231000 W
PLW13          0.16212000 W

F2 - Processing parameters
SI                   16384
SF             100.6177887 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```


12.173
8.625
8.336
8.171
8.167
8.162
7.957
7.955
7.952
7.934
7.932
7.903
7.882
7.880
7.750
7.729
7.659
7.656
7.636
7.452
7.438
7.432
7.423
7.421
7.418
7.416
7.402
7.400
7.372
7.352
7.332
7.259
7.193
7.192
7.175
7.172
7.156
7.154
7.057
7.055
7.037
7.035
2.461
Br
O
HN
N
N
3be
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.00
1.02
1.10
2.09
1.06
1.00
2.05
1.08
1.05
1.05
3.09



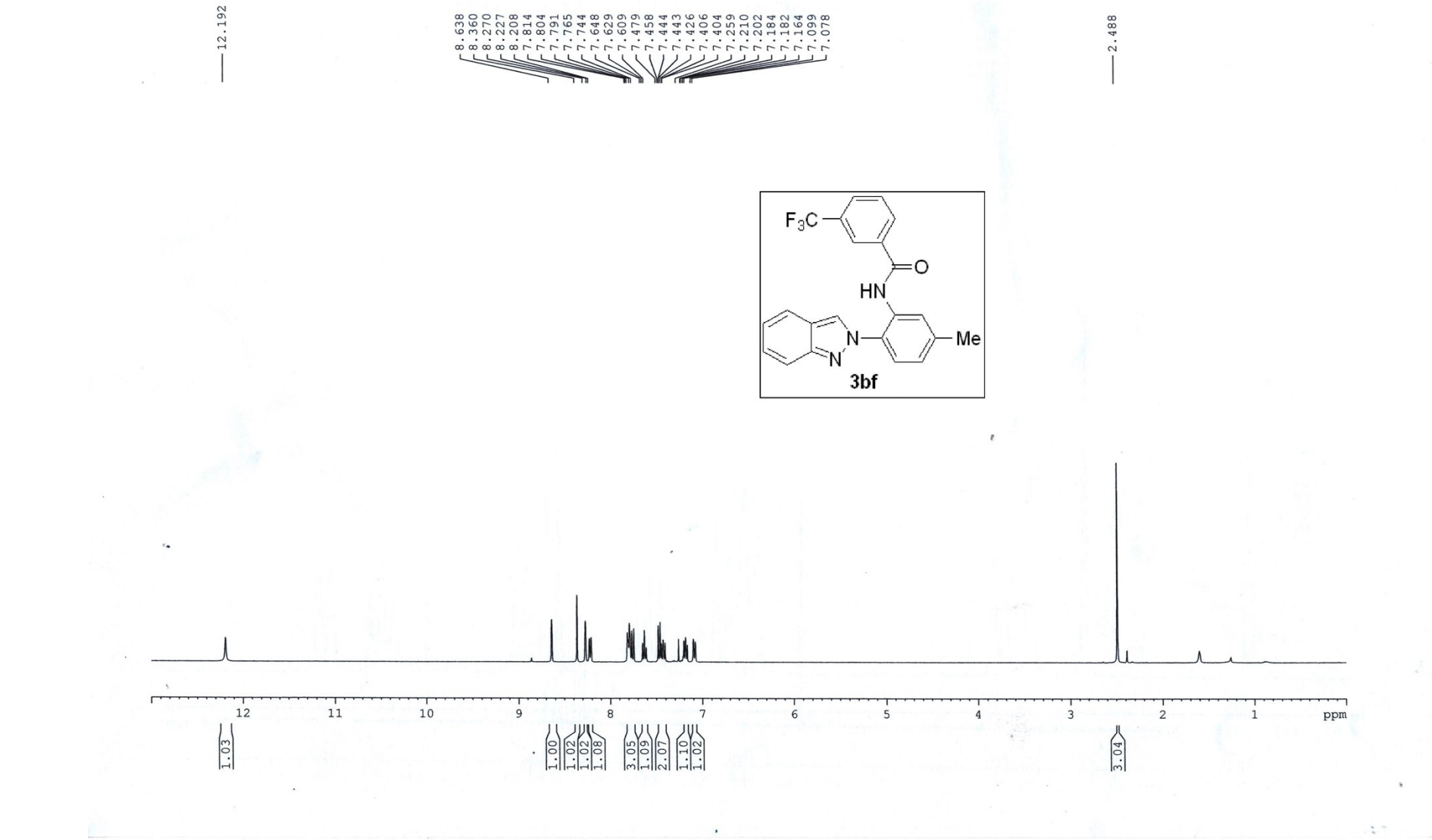
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 364
PROCNO 1

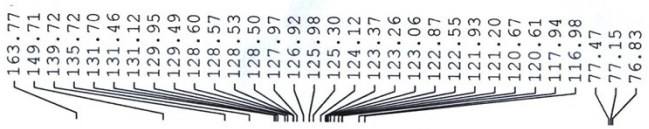
F2 - Acquisition Parameters
Date_ 20190912
Time 18.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 200
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 62.69
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

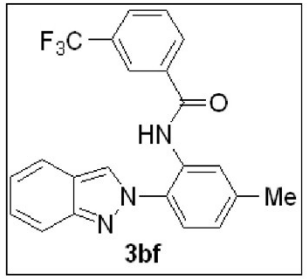
======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

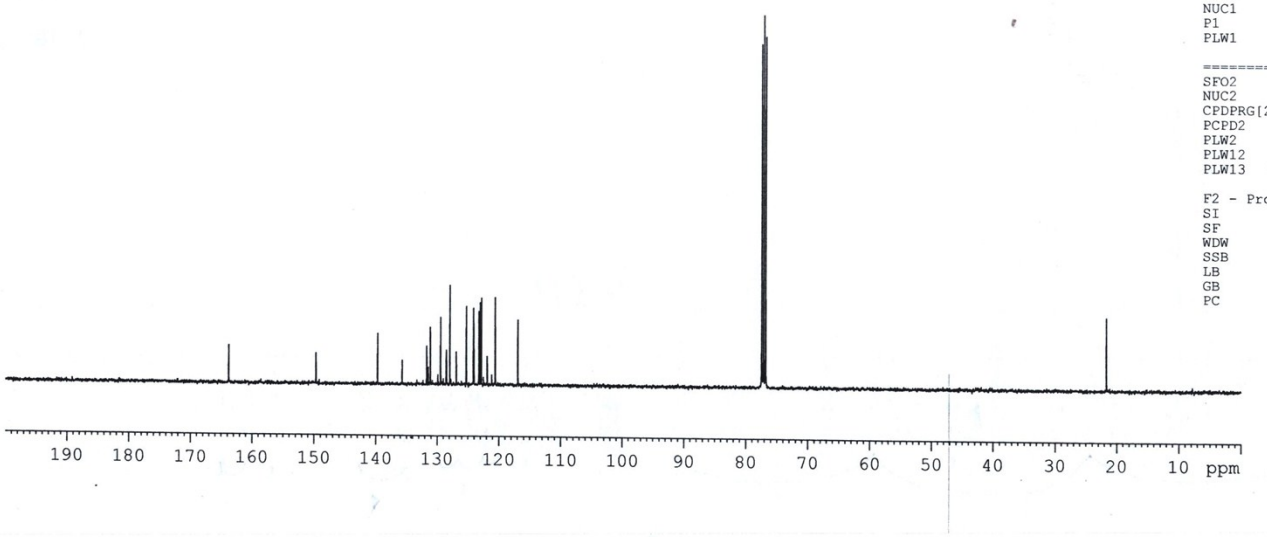
F2 - Processing parameters
SI 16384
SF 100.6177888 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

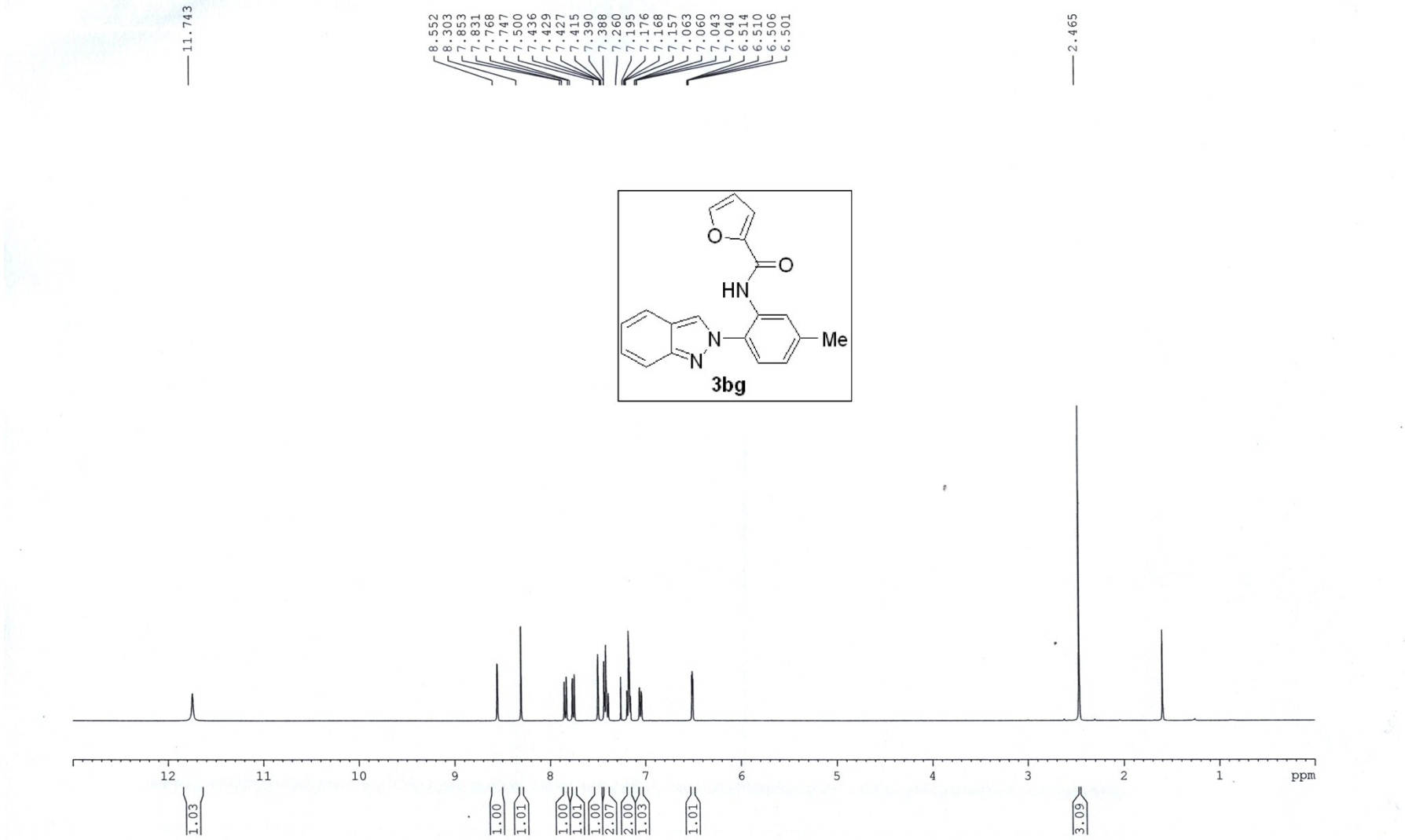

12.192
8.638
8.360
8.270
8.227
8.208
7.814
7.804
7.791
7.765
7.744
7.648
7.629
7.609
7.479
7.458
7.444
7.443
7.426
7.406
7.404
7.259
7.210
7.202
7.184
7.182
7.164
7.099
7.078
2.488
F3C
O
HN
N
N
Me
3bf
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.00
1.02
1.02
1.08
3.05
1.09
2.07
1.10
1.02
3.04

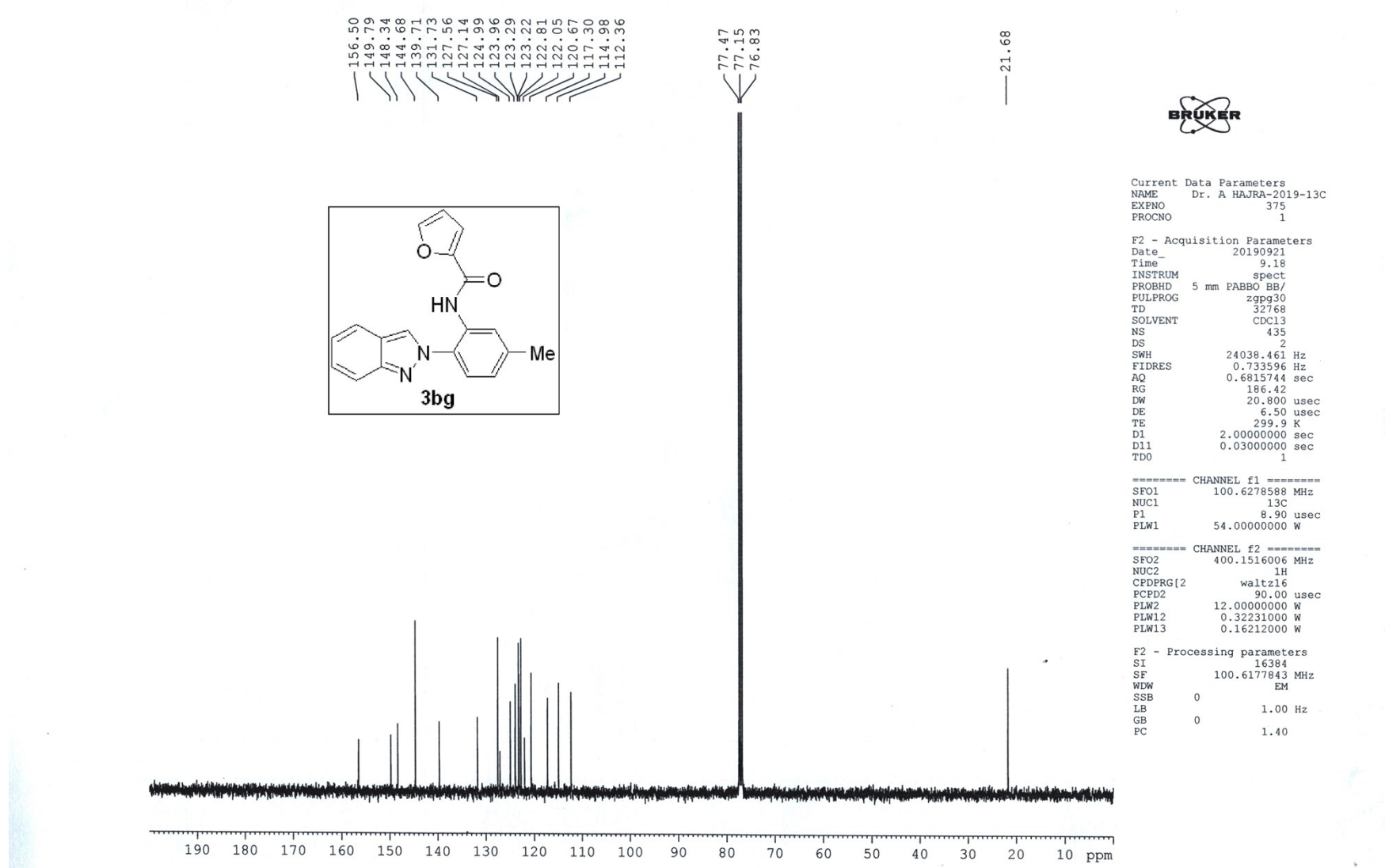





```
Current Data Parameters
NAME      Dr. A HAJRA-2020-13C
EXPNO                      26
PROCNO                      1

F2 - Acquisition Parameters
Date_               20200118
Time                   14.03
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                      32768
SOLVENT                 CDCl3
NS                       1024
DS                          2
SWH                24038.461 Hz
FIDRES             0.733596 Hz
AQ                 0.6815744 sec
RG                     135.7
DW                    20.800 usec
DE                     6.50 usec
TE                     295.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            100.6278588 MHz
NUC1                    13C
P1                     8.90 usec
PLW1           54.00000000 W

======== CHANNEL f2 ========
SFO2            400.1516006 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           12.00000000 W
PLW12           0.32231000 W
PLW13           0.16212000 W

F2 - Processing parameters
SI                      16384
SF              100.6177858 MHz
WDW                        EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```




11.743
8.552
8.303
7.853
7.831
7.768
7.747
7.500
7.436
7.429
7.427
7.415
7.390
7.388
7.260
7.195
7.176
7.168
7.157
7.063
7.060
7.043
7.040
6.514
6.510
6.506
6.501
2.465
O
O
HN
N
N
Me
3bg
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.00
1.01
1.00
1.01
1.00
2.07
2.00
1.03
1.01
3.09



BRUKER

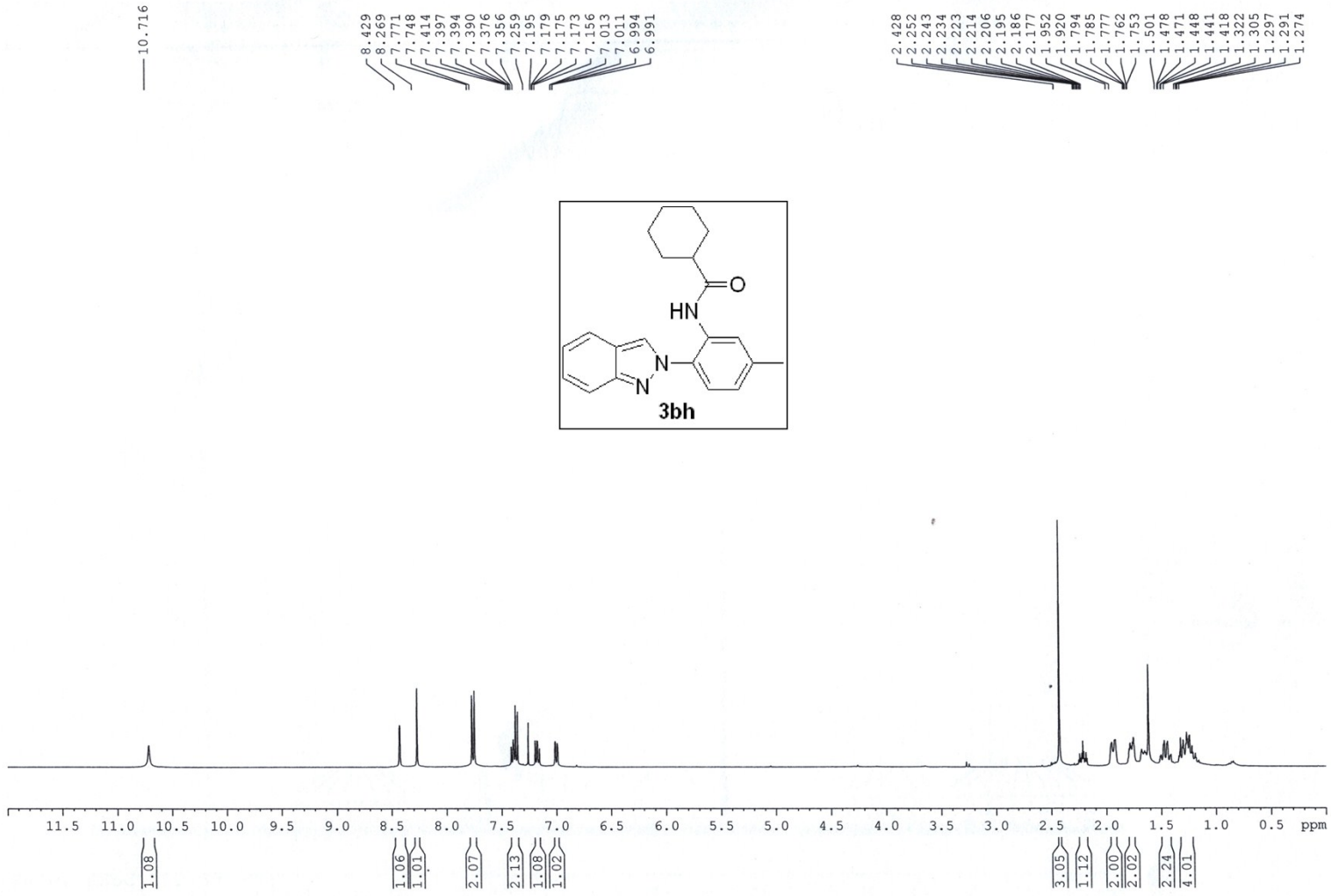
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 375
PROCNO 1

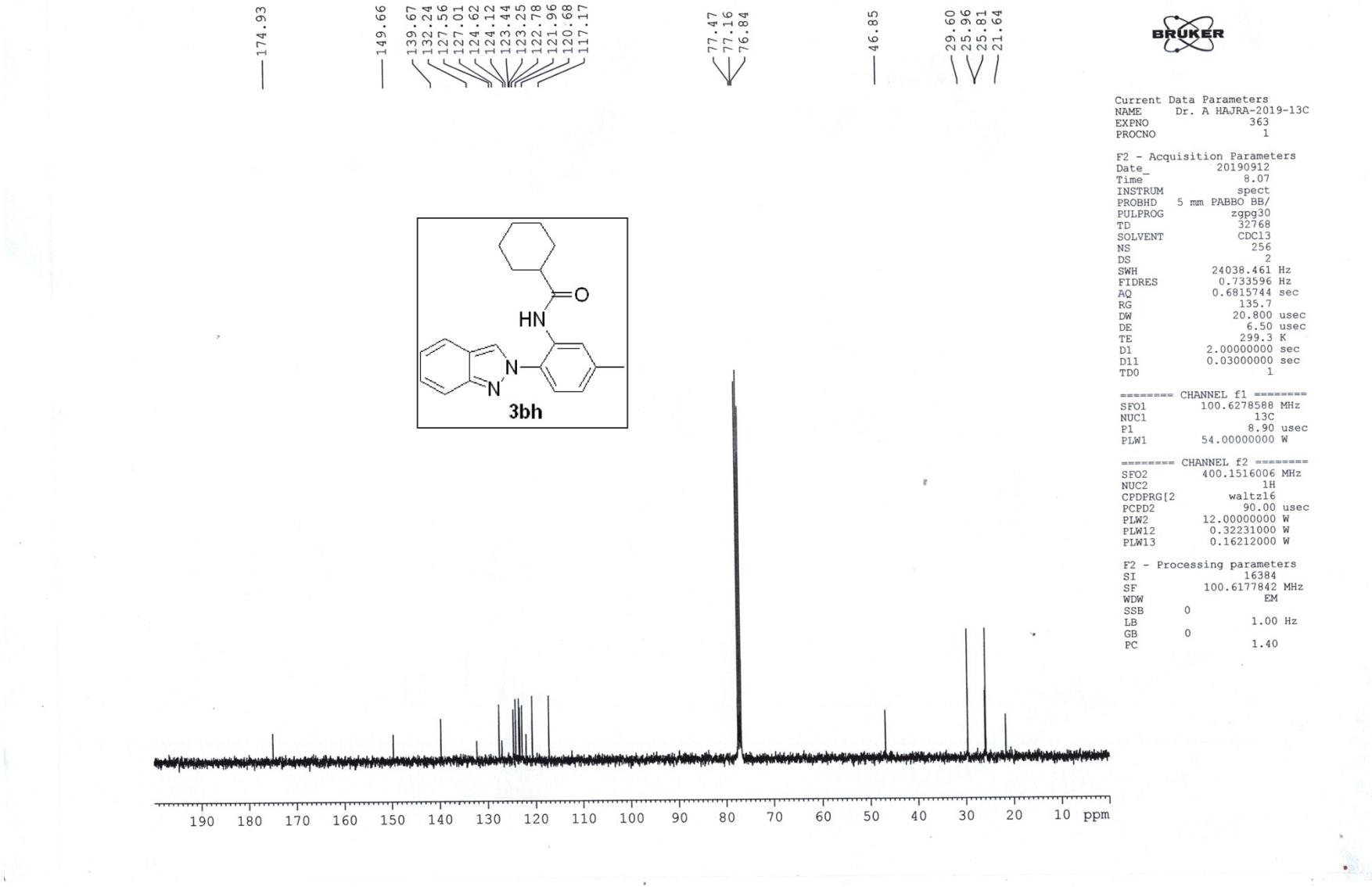
F2 - Acquisition Parameters
Date_ 20190921
Time 9.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 435
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177843 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10.716
8.429
8.269
7.771
7.748
7.414
7.397
7.394
7.390
7.376
7.356
7.259
7.195
7.179
7.175
7.173
7.156
7.013
7.011
6.994
6.991
2.428
2.252
2.243
2.234
2.223
2.214
2.206
2.195
2.186
2.177
1.952
1.920
1.794
1.785
1.777
1.762
1.753
1.501
1.478
1.471
1.448
1.441
1.418
1.322
1.305
1.297
1.291
1.274
O
HN
N
N
3bh
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.08
1.06
1.01
2.07
2.13
1.08
1.02
3.05
1.12
2.00
2.02
2.24
4.01



BRUKER

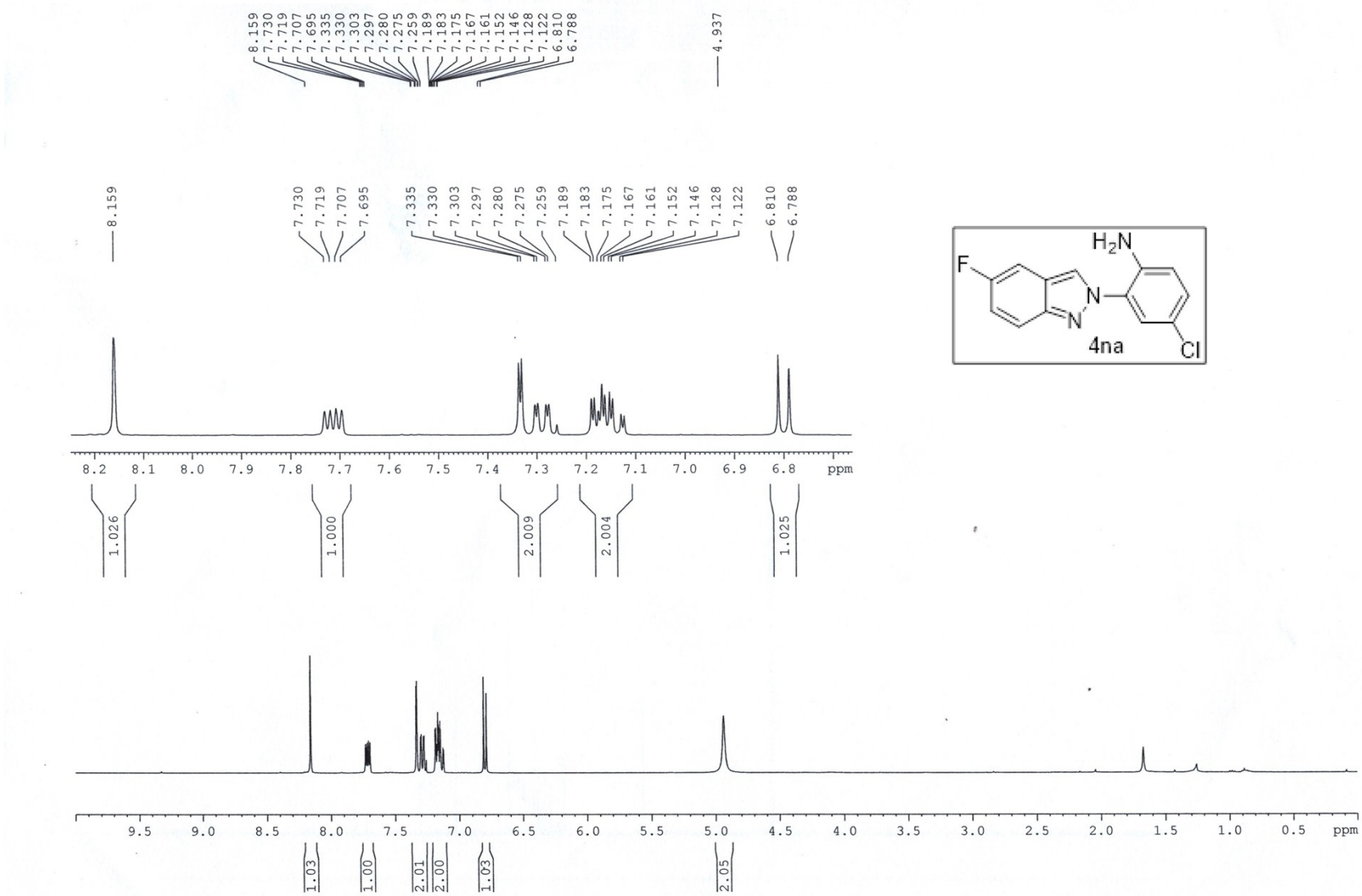
Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 363
PROCNO 1

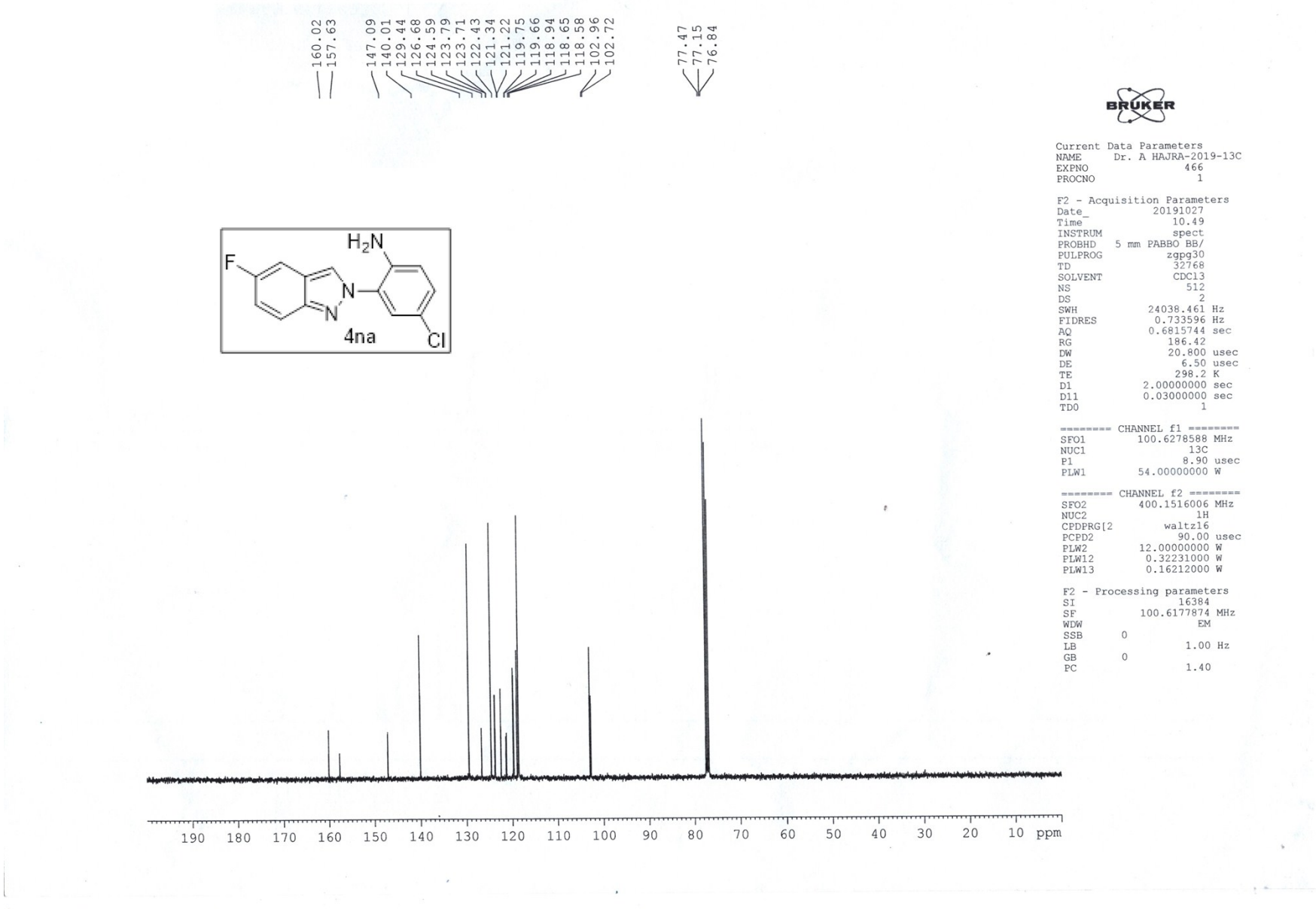
F2 - Acquisition Parameters
Date_ 20190912
Time 8.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 135.7
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.159
7.730
7.719
7.707
7.695
7.335
7.330
7.303
7.297
7.280
7.275
7.259
7.189
7.183
7.175
7.167
7.161
7.152
7.146
7.128
7.122
6.810
6.788
4.937
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
1.026
1.000
2.009
2.004
1.025
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.00
2.01
2.00
1.03
2.05
H2N
F
N
N
4na
Cl



BRUKER

Current Data Parameters
NAME Dr. A HAJRA-2019-13C
EXPNO 466
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191027
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 512
DS 2
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 186.42
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6278588 MHz
NUC1 13C
P1 8.90 usec
PLW1 54.00000000 W

======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.32231000 W
PLW13 0.16212000 W

F2 - Processing parameters
SI 16384
SF 100.6177874 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40