Supporting Information

A Transition-metal-free Synthesis of Arylcarboxyamides from Aryl Diazonium Salts and Isocyanides

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1. General Information

All reactions involving air sensitive reagents or intermediates were carried out in pre-heated glassware under an argon atmosphere using standard *Schlenk* techniques. All isocyanides were purchased from Sigma Aldrich. All other reagents were purchased without further purification unless otherwise noted. Acetone was purified according to the literature.[1] Ferrocene was purchased in 98.8% purity from Sigma Aldrich. Water was distilled before use. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF254). Visualization of the developed plates was performed under UV light (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal tetramethylsilane standard or the DMSO-d_6 residual peak (δ 2.50) for ^{1}H NMR. Chemical shifts of ^{13}C NMR are reported relative to $CDCl_3$ (δ 77.0) or DMSO-d_6 (δ 39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, *J*, were reported in Hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS Spectrometer.

2. General Procedures

1. Preparation of Aryl Diazonium Tetrafluoroborates

The appropriate aniline (10 mmole) was dissolved in a mixture of 4 mL of distilled water and 3.4 mL of 50% hydrofluoroboric acid. After cooling the reaction mixture to 0°C using ice bath, sodium nitrite (0.69 g in 1.5 mL of distilled water) was added dropwise in 5 min. The resulting mixture was stirred for 30 min and the precipitate was collected by filtration and re-dissolved in minimum amount of acetone. Diethyl ether was added until precipitation of diazonium tetrafluoroborate, which is filtered, washed several times with diethyl ether and dried under vacuum. [2]

2. Synthesis of Arylcarboxyamides

General Procedure A

A *Schlenk*-tube containing diazoniumtetrafluoroborate (0.2 mmol, 1.0 equiv) was degassed by three evacuation/Ar backfill cycles, then it was cooled to 0 ℃ (water/ice bath), 0.5 mL of acetone, isocyanide (equiv as noted in the text) in 0.5 mL of acetone, Cs_2CO_3 (71.7 mg, 1.1 equiv) in 0.4 mL of H_2O were added successively and slowly by syringe. The mixture was stirred at 0 ℃ for 20 minutes. After addition of water, the reaction mixture was extracted with ethyl acetate (3×10 mL), and the organic layers were combined, dried over anhydrous

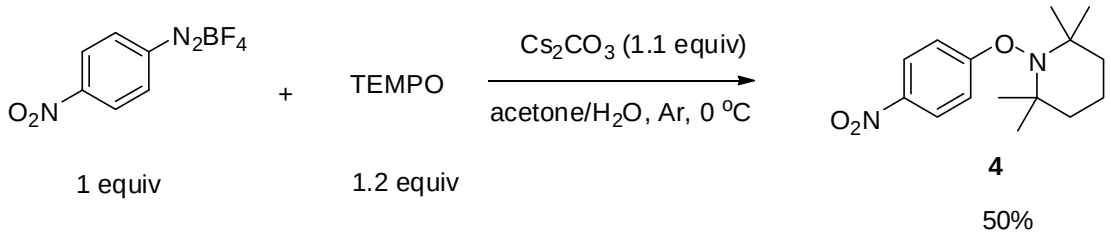
Na_2SO_4, concentrated under reduced pressure. The residue was separated by column chromatography (petroleum ether/EtOAc 10:2) to give the pure product.

General Procedure B

A *Schlenk*-tube containing diazoniumtetrafluoroborate (0.6 mmol, 3.0 equiv) was degassed by three evacuation/Ar backfill cycles, then it was cooled to 0 ℃ (water/ice bath), 0.5 mL of acetone, isocyanide (0.2 mmol, 1.0 equiv) in 0.5 mL of acetone, Cs_2CO_3 (71.7 mg, 1.1 equiv) in 0.4 mL of H_2O were added successively and slowly by syringe. The mixture was stirred at 0 ℃ for 20 minutes. After addition of water, the reaction mixture was extracted with ethyl acetate (3×10 mL), and the organic layers were combined, dried over anhydrous Na_2SO_4, concentrated under reduced pressure. The residue was separated by column chromatography (petroleum ether/EtOAc 10:2) to give the pure product.

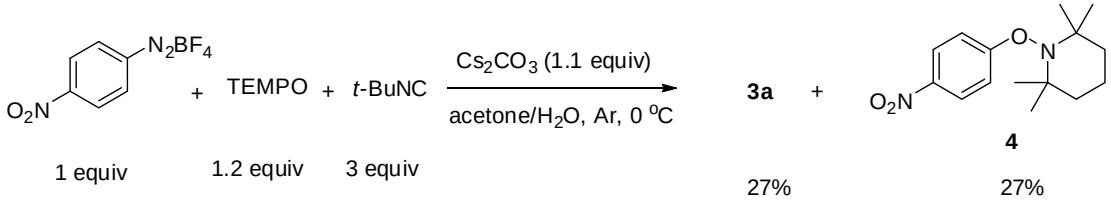
3. Radical Capturing Experiments with TEMPO

A: without **2a** [3]



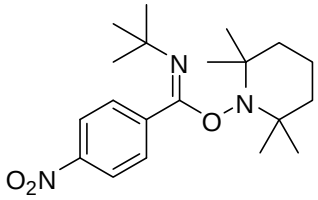
A *Schlenk*-tube containing 4-nitrobenzenediazonium tetrafluoroborate (0.2 mmol, 47.4 mg) was degassed by three evacuation/Ar backfill cycles, then it was cooled to 0 ℃ (water/ice bath), 0.5 mL of acetone, TEMPO (0.24 mmol, 37.5 mg) in 0.5 mL of acetone , Cs_2CO_3 (0.22 mmol, 71.7 mg) in 0.4 mL of H_2O were added successively and slowly by syringe. The mixture was stirred at 0 ℃ for 20 minutes. After addition of water, the reaction mixture was extracted with ethyl acetate (3×10 mL), and the organic layers were combined, dried over anhydrous Na_2SO_4, concentrated under reduced pressure. The residue was separated by column chromatography (petroleum ether/EtOAc 10:1) to give the pure product.

B: with 3 equiv of **2a**



A *Schlenk*-tube containing 4-nitrobenzenediazonium tetrafluoroborate (0.2 mmol, 47.4 mg) was degassed by three evacuation/Ar backfill cycles, then it was cooled to 0 ℃ (water/ice bath), 0.4 mL of acetone, *tert*-butyl-isocyanide (0.6 mmol, 68 μL) in 0.3 mL of acetone, TEMPO (0.24 mmol, 37.5 mg) in 0.4 mL of acetone, Cs_2CO_3(0.22 mmol, 71.7 mg) in 0.4 mL of H_2O were added successively by syringe. The mixture was stirred at 0 ℃ for 20 minutes.

After addition of water, the reaction mixture was extracted with ethyl acetate (3×10 mL), and the organic layers were combined, dried over anhydrous Na_2SO_4, concentrated under reduced pressure. The residue was separated by column chromatography (petroleum ether/EtOAc 240:1) to give the pure product.

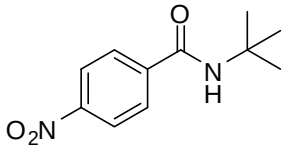


5

The adduct **5** between the imidoyl radical intermediate **B** and TEMPO was not isolated.

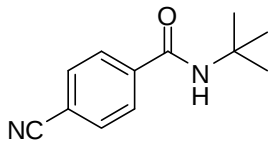
4. Analytical Data for 3a-3t and 4

***N*-(*tert*-butyl)-4-nitrobenzamide (3a)**



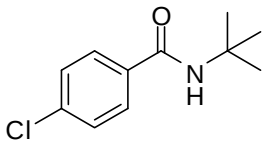
Following the general procedure A, **2a** (3.0 equiv). 35.5 mg (80%) of **3a** (yellow solid) were isolated. mp 159-161 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H), 6.03 (br s, 1H), 1.48 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.9, 149.3, 141.5, 127.9, 123.6, 52.2, 28.7; IR (KBr): 3311, 3072, 2972, 2929, 1642, 1599, 1546, 1460, 1396, 1348, 1311, 1225, 866, 721 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{15}N_2O_3$ $[M+H]^+$ 223.1077, found 223.1079.

***N*-(*tert*-butyl)-4-cyanobenzamide (3b)**



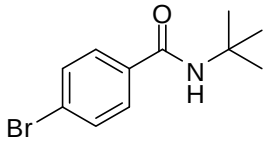
Following the general procedure A, **2a** (3.0 equiv). 29.1 mg (72%) of **3b** (yellow solid) were isolated. mp 151-153 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 5.94 (br s, 1H), 1.48 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.1, 139.9, 132.3, 127.5, 118.1, 114.6, 52.2, 28.7; IR (KBr): 3363, 3065, 2977, 2930, 2237, 1650, 1544, 1500, 1455, 1394, 1361, 1310, 1283, 1224, 864, 761 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{15}N_2O$ $[M+H]^+$ 203.1179, found 203.1177.

***N*-(*tert*-butyl)-4-chlorobenzamide (3c)**



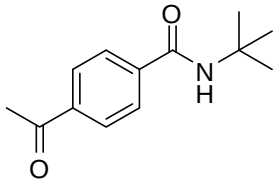
Following the general procedure A, **2a** (3.0 equiv). 28.7 mg (68%) of **3c** (yellow solid) were isolated. mp 138-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.6 Hz, 2H), 7.38 (d, *J* = 8.6 Hz, 2H), 5.88 (br s, 1H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.8, 137.0, 134.2, 128.4, 128.1, 51.7, 28.7; IR (KBr): 3324, 3063, 2972, 2927, 1638, 1596, 1538, 1483, 1451, 1393, 1361, 1315, 1220, 1013, 960, 846, 761 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{15}ClNO$ $[M+H]^+$ 212.0837, found 212.0836.

***N*-(*tert*-butyl)-4-bromobenzamide (3d)**



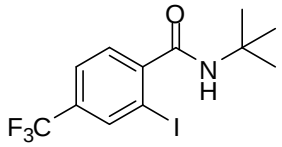
Following the general procedure A, **2a** (3.0 equiv). 33.2 mg (65%) of **3d** (yellow solid) were isolated. mp 135 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 8.6 Hz, 2H), 5.89 (br s, 1H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.9, 134.7, 131.5, 128.3, 125.5, 51.7, 28.7; IR (KBr): 3611, 3525, 3445, 3351, 2981, 2962, 1636, 1588, 1539, 1484, 1454, 1399, 1362, 1314, 1222, 1010, 845, 756 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{15}BrNO$ $[M+H]^+$ 256.0332, found 256.0331.

***N*-(*tert*-butyl)-4-acetylbenzamide (3e)**



Following the general procedure A, **2a** (3.0 equiv). 26.3 mg (60%) of **3e** (white solid) were isolated. mp 138 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 2H), 6.03 (br s, 1H), 2.61 (s, 3H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.5, 165.9, 139.8, 138.7, 128.3, 127.0, 51.9, 28.7, 26.7; IR (KBr): 3298, 3060, 2974, 2926, 1681, 1641, 1537, 1451, 1396, 1359, 1314, 1266, 1231, 959, 854, 767, 661 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{18}NO_2$ $[M+H]^+$ 220.1332, found 220.1332.

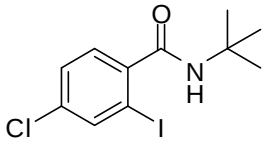
***N*-(*tert*-butyl)-2-iodo-4-(trifluoromethyl)benzamide (3f)**



Following the general procedure A, **2a** (3.0 equiv). 48.2 mg (65%) of **3f** (white solid) were isolated. mp 178-180 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.62 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 5.53 (br s, 1H), 1.49 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ

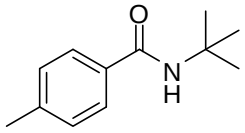
167.6, 146.5 , 136.4 (q, J_{C-F} = 3.8 Hz), 132.4 (q, J_{C-F} = 32.9 Hz),128.1, 125.4 (q, J_{C-F} = 28.3 Hz), 122.3 (q, J_{CF3} = 271.3 Hz), 92.2, 52.5, 28.6; IR (KBr): 3610, 3525, 3255, 1635, 1563, 1322, 1175, 1117, 1075, 835 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{14}F_3INO$ $[M+H]^+$ 372.0067, found 372.0069.

***N*-(*tert*-butyl)-4-chloro-2-iodobenzamide (3g)**



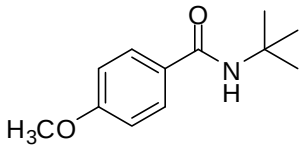
Following the general procedure A, **2a** (5.0 equiv). 39.1 mg (58%) of **3g** (white solid) were isolated. mp 142-143 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 1.6 Hz, 1H), 7.33 (m, 2H), 5.53 (br s, 1H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.7, 141.5, 138.9, 135.5, 128.7, 128.3, 92.5, 52.3, 28.6; IR (KBr): 3611, 3525, 3257, 3075, 2965, 1638, 1581, 1554, 1460, 1365, 1323, 1225, 822 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{14}ClINO$ $[M+H]^+$ 337.9803, found 337.9805.

***N*-(*tert*-butyl)-4-methylbenzamide (3h)**



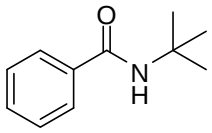
Following the general procedure A, **2a** (5.0 equiv). 16.8 mg (44%) of **3h** (white solid) were isolated. mp 114-116 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.2 Hz, 2H), 5.90 (br s, 1H), 2.38 (s, 3H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.8, 141.3, 133.1, 129.0, 126.6, 51.4, 28.9, 21.3; IR (KBr): 3355, 3034, 2979, 2927, 1643, 1544, 1453, 1360, 1391, 1360, 1312, 1226, 957, 875, 838, 752 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{18}NO$ $[M+H]^+$ 192.1383, found 192.1382.

***N*-(*tert*-butyl)-4-methoxybenzamide (3i)**



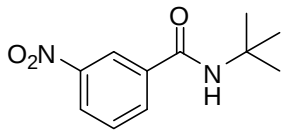
Following the general procedure A, **2a** (5.0 equiv). 17.8 mg (43%) of **3i** (white solid) were isolated. mp 120 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, J = 8.8 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 5.86 (br s, 1H), 3.83 (s, 3H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4, 161.8, 128.4, 128.2, 113.6, 55.3, 51.4, 28.9; IR (KBr): 3312, 3069, 2967, 2845, 1635, 1546, 1508, 1452, 1320, 1254, 1218, 1180, 878, 844, 769 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_{18}NO_2$ $[M+H]^+$ 208.1332, found 208.1334.

***N*-(*tert*-butyl)benzamide (3j)**



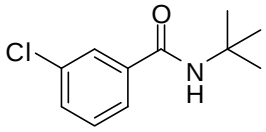
Following the general procedure A, **2a** (5.0 equiv). 17.0 mg (48%) of **3j** (white solid) were isolated. mp 134-136 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 6.8 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.41 (dd, *J* = 7.6, 6.8 Hz, 2H), 5.94 (br s, 1H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.9, 135.9, 131.0, 128.4, 126.7, 51.5, 28.8; IR (KBr): 3323, 3061, 2971, 1638, 1538, 1488, 1449, 1362, 1311, 1221, 935, 875, 804, 718 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{16}NO$ $[M+H]^+$ 178.1226, found 178.1228.

***N*-(*tert*-butyl)-3-nitrobenzamide (3k)**



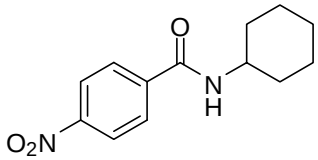
Following the general procedure A, **2a** (3.0 equiv). 32.0 mg (72%) of **3k** (yellow solid) were isolated. mp 126-128 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 1.4 Hz, 1H), 8.27 (dd, *J* = 8.0, 1.4 Hz, 1H), 8.08 (d, *J* = 7.6 Hz, 1H), 7.58 (t, *J* = 8.0 Hz, 1H), 6.21 (br s, 1H), 1.48 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.4, 148.0, 137.5, 133.0, 129.7, 125.6, 121.5, 52.2, 28.7; IR (KBr): 3316, 3081, 2971, 2929, 1651, 1530, 1457, 1394, 1354, 1315, 1222, 919, 717 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{15}N_2O_3$ $[M+H]^+$ 223.1077, found 223.1078.

***N*-(*tert*-butyl)-3-chlorobenzamide (3l)**



Following the general procedure A, **2a** (3.0 equiv). 25.8 mg (61%) of **3l** (white solid) were isolated. mp 99-101 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.34 (dd, *J* = 7.6, 8.0 Hz, 1H), 5.91 (br s, 1H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.5, 137.7, 134.6, 131.1, 129.8, 127.1, 124.8, 51.9, 28.8; IR (KBr): 3609, 3520, 3284, 2967, 1639, 1544, 1453, 1364, 1317, 1217, 898 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_{15}ClNO$ $[M+H]^+$ 212.0837, found 212.0838.

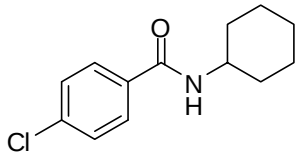
***N*-cyclohexyl-4-nitrobenzamide (3m)**



Following the general procedure A, cyclohexyl isocyanide (3.0 equiv). 39.2 mg (79%) of **3m** (yellow solid) were isolated. mp 159 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.6 Hz, 2H), 7.91 (d, *J* = 8.6 Hz, 2H), 6.03 (br d, NH, 1H), 3.98 (m, 1H), 2.06-1.20 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 149.4, 140.7, 128.0, 123.7, 49.2, 33.1, 25.4, 24.8; IR (KBr):

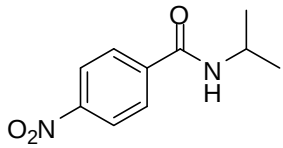
3320, 2929, 2860, 1625, 1611, 1541, 1506, 1353, 1252 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{17}N_2O_3$ $[M+H]^+$ 249.1234, found 249.1240.

_N_-cyclohexyl-4-chlorobenzamide (3n)



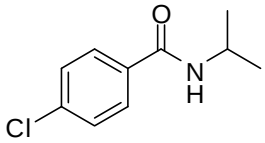
Following the general procedure A, cyclohexyl isocyanide (5.0 equiv). 27.0 mg (57%) of **3n** (yellow solid) were isolated. mp 184-185 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 6.03 (br s, NH, 1H), 3.94 (m, 1H), 2.02-1.17 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.5, 137.4, 133.5, 128.7, 128.3, 48.8, 33.2, 25.5, 24.9; IR (KBr): 3288, 2929, 1629, 1541, 1486, 1454, 1332, 1154, 1018 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{17}ClNO$ $[M+H]^+$ 238.0993, found 238.1000.

_N_-isopropyl-4-nitrobenzamide (3o)



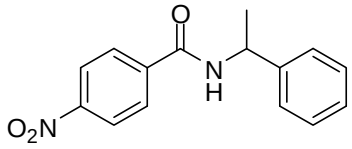
Following the general procedure A, isopropyl isocyanide (3.0 equiv). 29.1 mg (70%) of **3o** (yellow solid) were isolated. mp 151-153 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, J = 8.8 Hz, 2H), 7.91 (d, J = 8.8 Hz, 2H), 5.99 (br s, NH, 1H), 4.30 (m, 1H), 1.29 (d, J = 6.8 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 149.5, 140.5, 128.0, 123.7, 42.4, 22.7; IR (KBr): 3611, 3525, 3446, 3304, 2980, 1639, 1602, 1543, 1520, 1345, 1318, 1290, 871, 827 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{13}N_2O_3$ $[M+H]^+$ 209.0921, found 209.0923.

_N_-isopropyl-4-chlorobenzamide (3p)



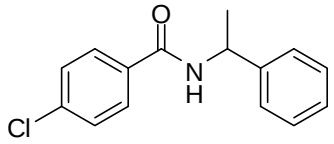
Following the general procedure A, isopropyl isocyanide (5.0 equiv). 24.8 mg (63%) of **3p** (yellow solid) were isolated. mp 142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 8.2 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 6.00 (br s, NH, 1H), 4.26 (m, 1H), 1.25 (d, J = 6.4 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.6, 137.4, 133.4, 128.7, 128.2, 42.0, 22.8; IR (KBr): 3746, 3610, 3525, 3446, 3310, 2977, 1630, 1596, 1538, 1487, 1016, 846, 764 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{10}H_{13}ClNO$ $[M+H]^+$ 198.0680, found 198.0681.

4-nitro-_N_-(1-phenylethyl)benzamide (3q)



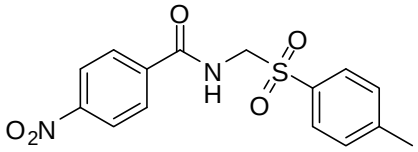
Following the general procedure B. 40.5 mg (75%) of **3q** (yellow solid) were isolated. mp 120-121 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.8 Hz, 2H), 7.92 (d, *J* = 8.8 Hz, 2H),7.40-7.29 (m, 5H), 6.43 (br d, NH, 1H), 5.34 (m, 1H), 1.64 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 149.6, 142.4, 140.1, 128.9, 128.1, 127.8, 126.3, 123.8, 49.8, 21.5; IR (KBr): 3746, 3610, 3526, 3446, 3334, 1710, 1596, 1519, 1349, 1168, 853, 747 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{15}N_2O_3$ $[M+H]^+$ 271.1077, found 271.1085.

4-chloro-*N*-(1-phenylethyl)benzamide (3r)



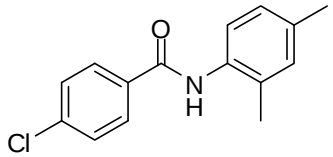
Following the general procedure B. 27.0 mg (52%) of **3r** (yellow solid) were isolated. mp 137-138 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.8 Hz, 2H), 7.40-7.27 (m, 7H), 6.32 (br s, NH, 1H), 5.32 (m, 1H), 1.61 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.5, 142.9, 137.7, 133.0, 128.8, 128.4, 127.6, 126.2, 49.4, 21.6; IR (KBr): 3609, 3526, 3445, 3276, 1647, 1630, 1595, 1536, 1486, 1331, 1091, 1013, 847, 760, 700 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{15}ClNO$ $[M+H]^+$ 260.0837, found 260.0838.

4-nitro-*N*-(1-tosylmethyl)benzamide (3s)



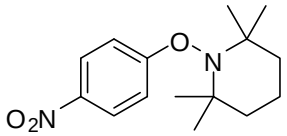
Following the general procedure B. 42.8 mg (64%) of **3s** (yellow solid) were isolated. mp 206-207 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.82 (br s, NH, 1H), 8.31 (d, *J* = 8.2 Hz, 2H), 7.95 (d, *J* = 8.2 Hz, 2H), 7.75 (d, *J* = 7.8 Hz, 2H), 7.42 (d, *J* = 7.8 Hz, 2H), 4.88 (d, *J* = 6.4 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.9, 149.4, 144.8, 138.6, 134.7, 129.9, 129.0, 128.5, 123.7, 61.1, 21.1; IR (KBr): 3611, 3526, 3446, 3334, 1652, 1520, 1329, 1142, 719 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{15}N_2O_5S$ $[M+H]^+$ 335.0696, found 335.0708.

4-chloro-*N*-(2,4-dimethylphenyl)benzamide (3t)



Following the general procedure A, 2,4-dimethylphenyl isocyanide (3.0 equiv). 10.4 mg (20%) of **3t** (yellow solid) were isolated. mp 160-163 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 6.8 Hz, 1H), 7.58 (s, 1H), 7.45 (d, *J* = 8.0 Hz, 2H), 7.06-7.05 (m, 2H), 2.32 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.7, 138.0, 135.5, 133.3, 132.8, 131.3, 130.0, 129.0, 128.5, 127.4, 123.7, 20.9, 17.8; IR (KBr): 3747, 3610, 3527, 3445, 3332, 3063, 1649, 1596, 1527, 1330, 1277, 1142, 963, 849, 819 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{15}ClNO$ $[M+H]^+$ 260.0837, found 260.0834.

2,2,6,6-tetramethyl-1-(4-nitrophenoxy)piperidine (4)



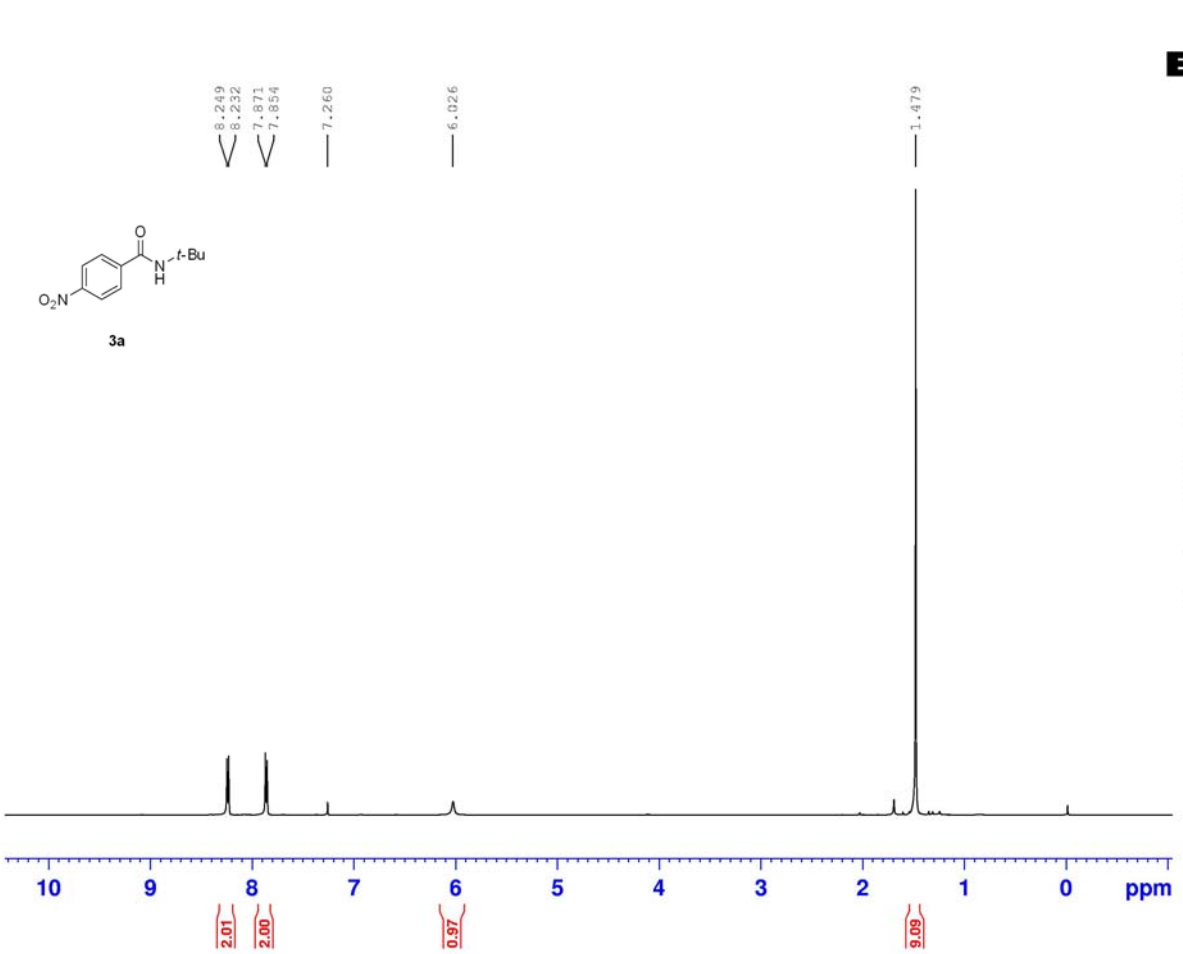
4

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 9.6 Hz, 2H), 7.4-7.2 (m, 2H), 1.66-1.60 (m, 5H), 1.46-1.42 (m, 1H), 1.24 (s, 6H), 0.99 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.7, 141.2, 125.5, 114.1, 60.9, 39.7, 32.3, 20.5, 16.9; ESI-MS: 279.1 (100, M+1); HRMS (ESI): Exact mass calcd for $C_{15}H_{23}N_2O_3$ $[M+H]^+$ 279.1703, found 279.1686.

5. References

(1) Burfield, D. R.; Smithers, R. H. *J. Org. Chem.* **1978**, *43*, 3966.
(2) Hanson, P.; Jones, J. R.; Taylor, A. B.; Walton, P. H.; Timms, A. W. *J. Chem. Soc., Perkin Trans. 2*. **2002**, 1135.
(3) (a) Hering, T.; Hari D. P.; König, B. *J. Org. Chem.* **2012**, *77*, 10347. (b) Hari D. P.; Schroll, P.; König, B. *J. Am. Chem.Soc.* **2012**, *134*, 2958.

6. ^{1}H and ^{13}C Spectra for 3a-3t and 4

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PROCNO                     1
Date_               20121127
Time                   21.42
INSTRUM                Spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                      144
DW                 48.400 usec
DE                  6.50 usec
TE                  295.7 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                    1H
P1                 14.00 usec
PL1                 2.50 dB
PL1W          13.02359581 W
SFO1          500.1330885 MHz
SI                   32768
SF            500.1300224 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
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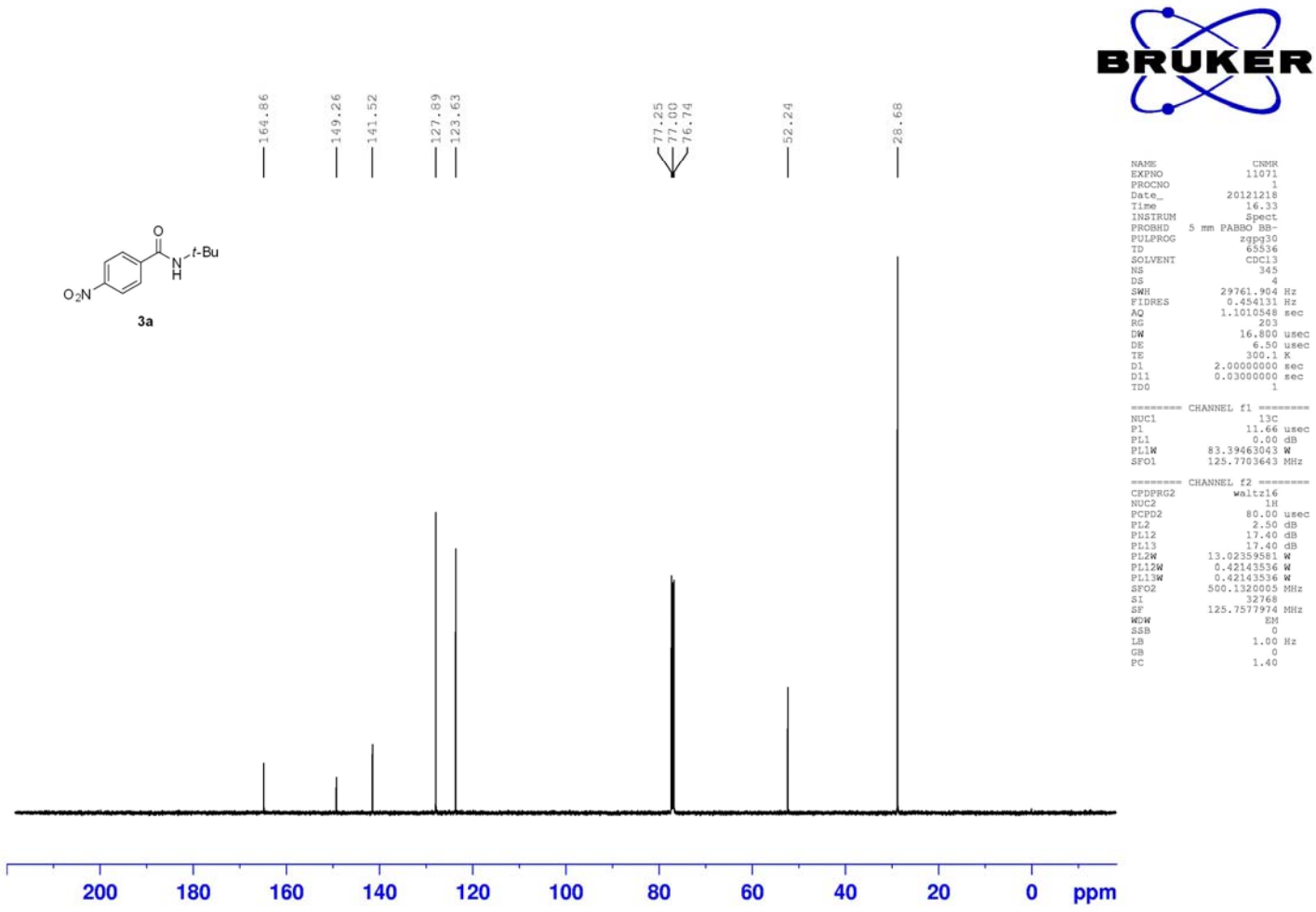


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NAME                  CNMR
EXPNO                11071
PROCNO                   1
Date_             20121218
Time                 16.33
INSTRUM              Spect
PROBHD  5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   345
DS                     4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                   203
DW                16.800 usec
DE                 6.50 usec
TE                300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.66 usec
PL1                 0.00 dB
PL1W        83.39463043 W
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 2.50 dB
PL12               17.40 dB
PL13               17.40 dB
PL2W        13.02359581 W
PL12W        0.42143536 W
PL13W        0.42143536 W
SFO2       500.1320005 MHz
SI                 32768
SF         125.7577974 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
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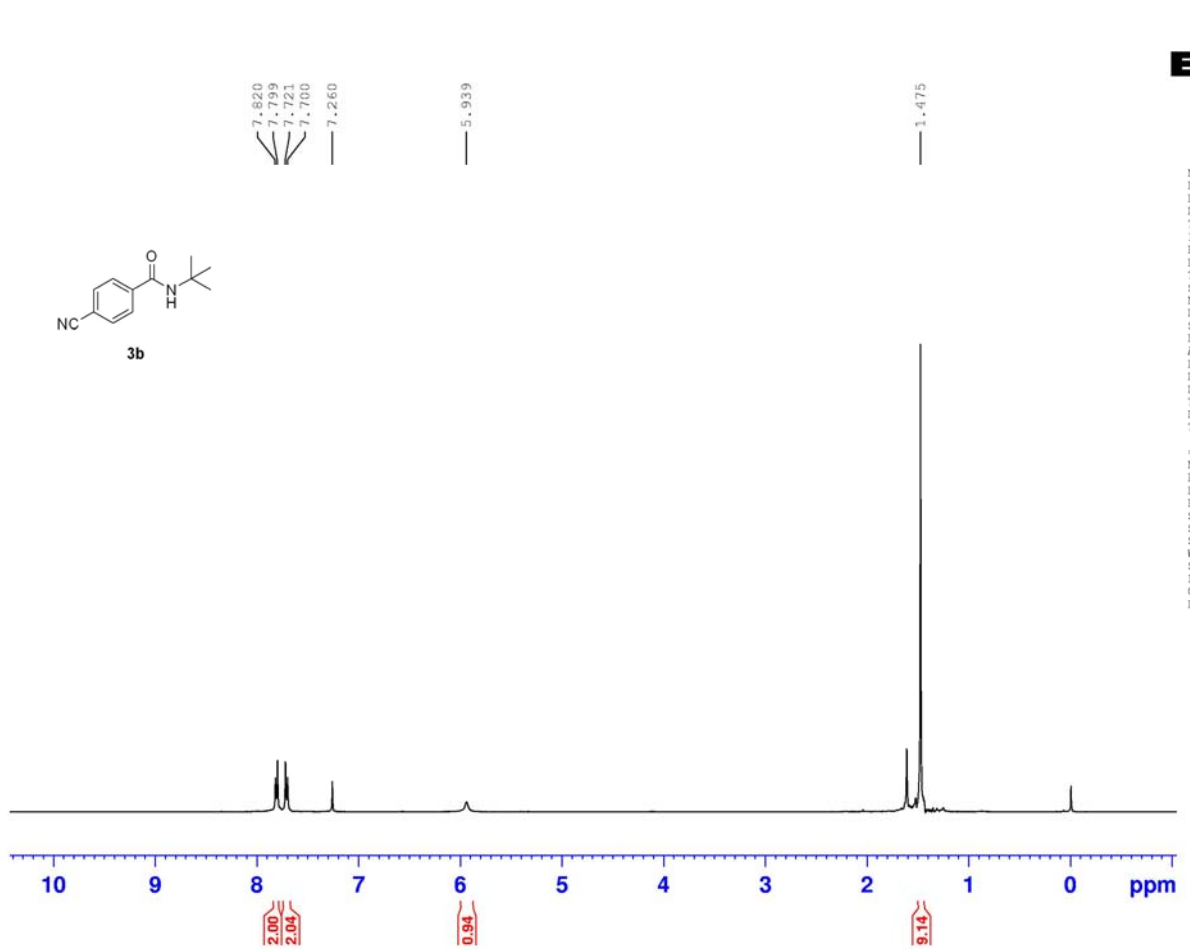

BRUKER

```
NAME            pnmr-digt
EXPNO               11271
PROCNO                  1
Date_            20130326
Time                17.46
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    362
DW                60.400 usec
DE                 6.50 usec
TE                297.5 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.58 usec
PL1                 0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300090 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
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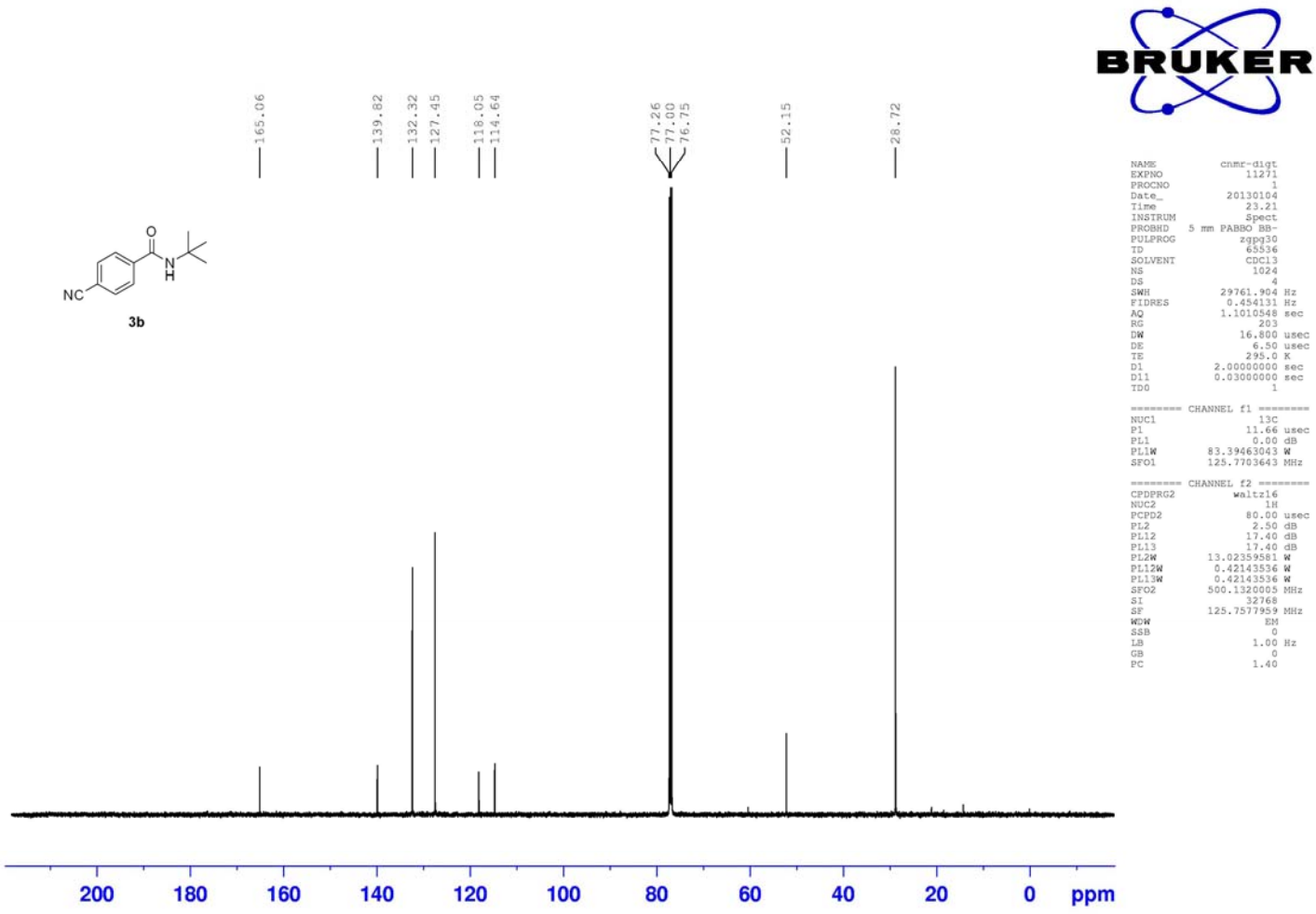
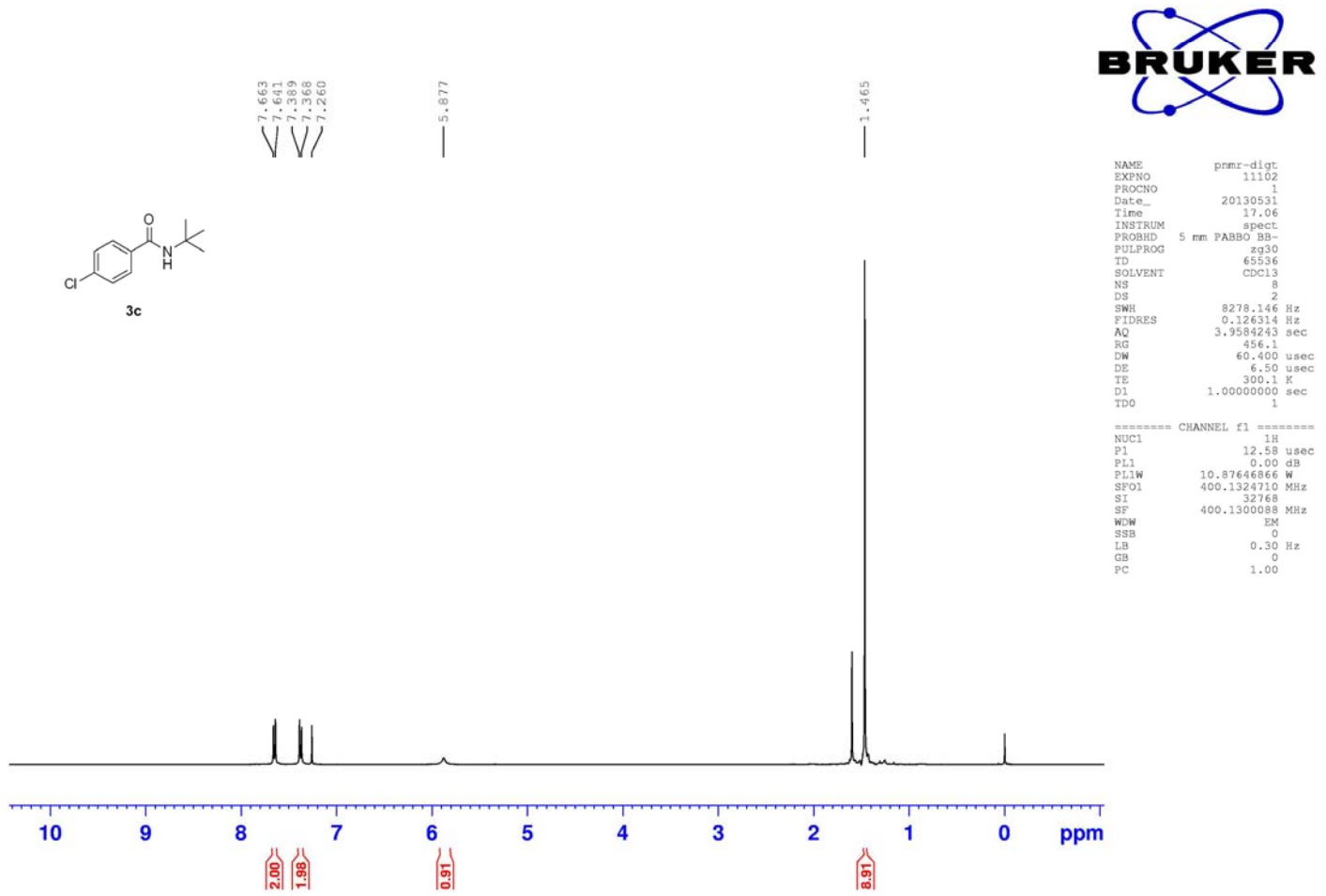


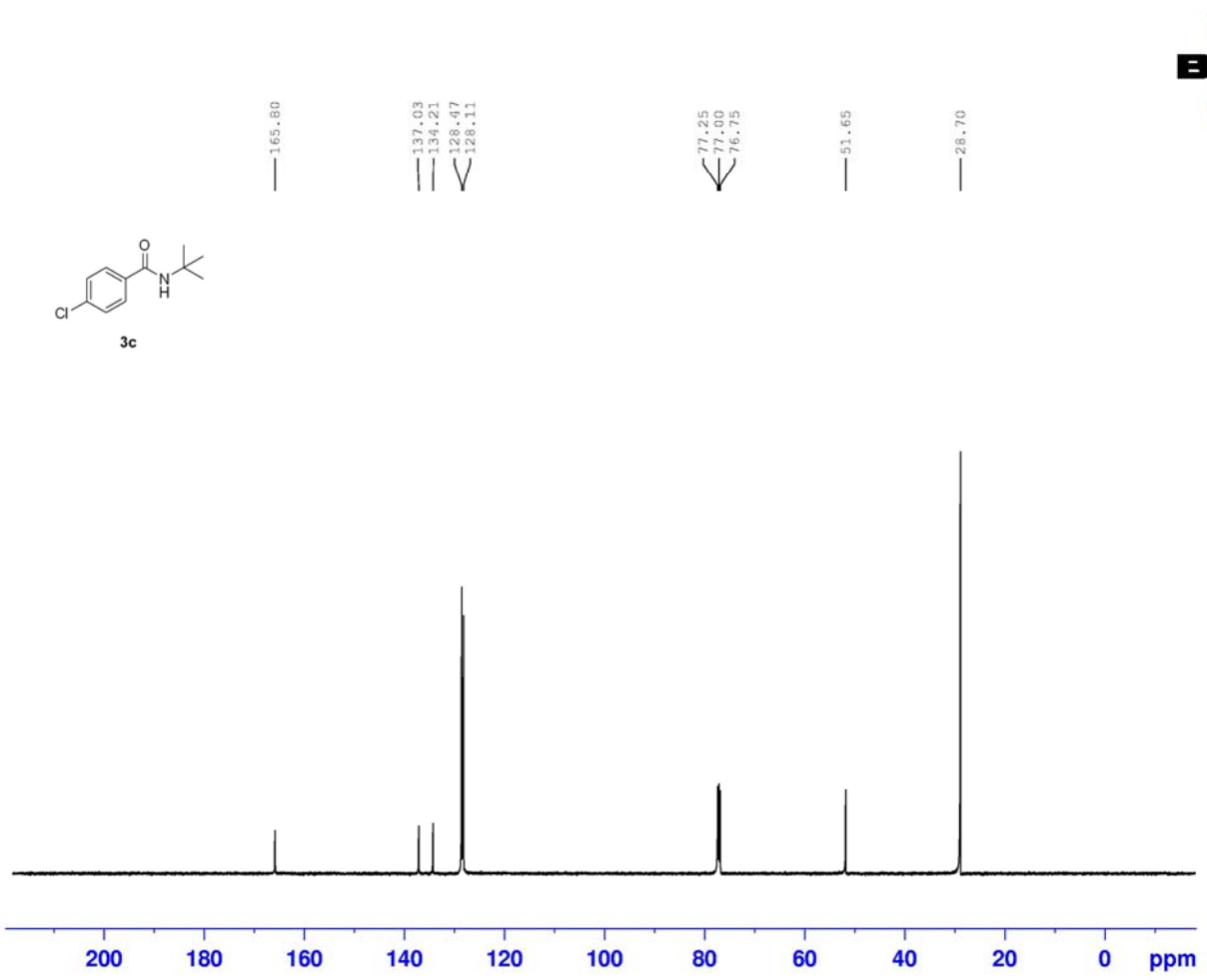
BRUKER

NAME	cnmr-dlgt
EXPNO	11271
PROCNO	1
Date_	20130104
Time	23.21
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	295.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577959 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

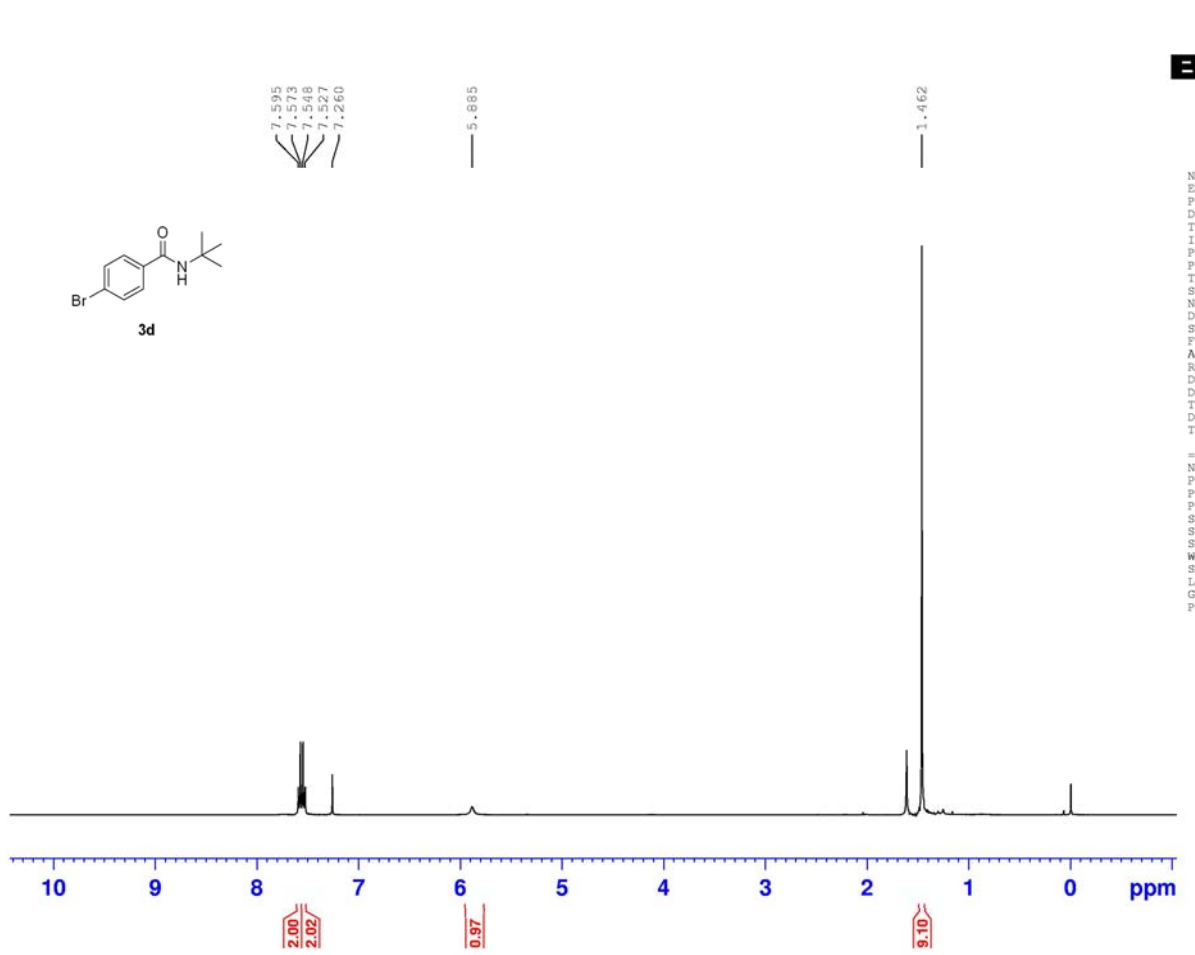


BRUKER

NAME	pnmr-digt
EXPNO	11102
PROCNO	1
Date_	20130531
Time	17.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	456.1
DW	60.400 usec
DE	6.50 usec
TE	300.1 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

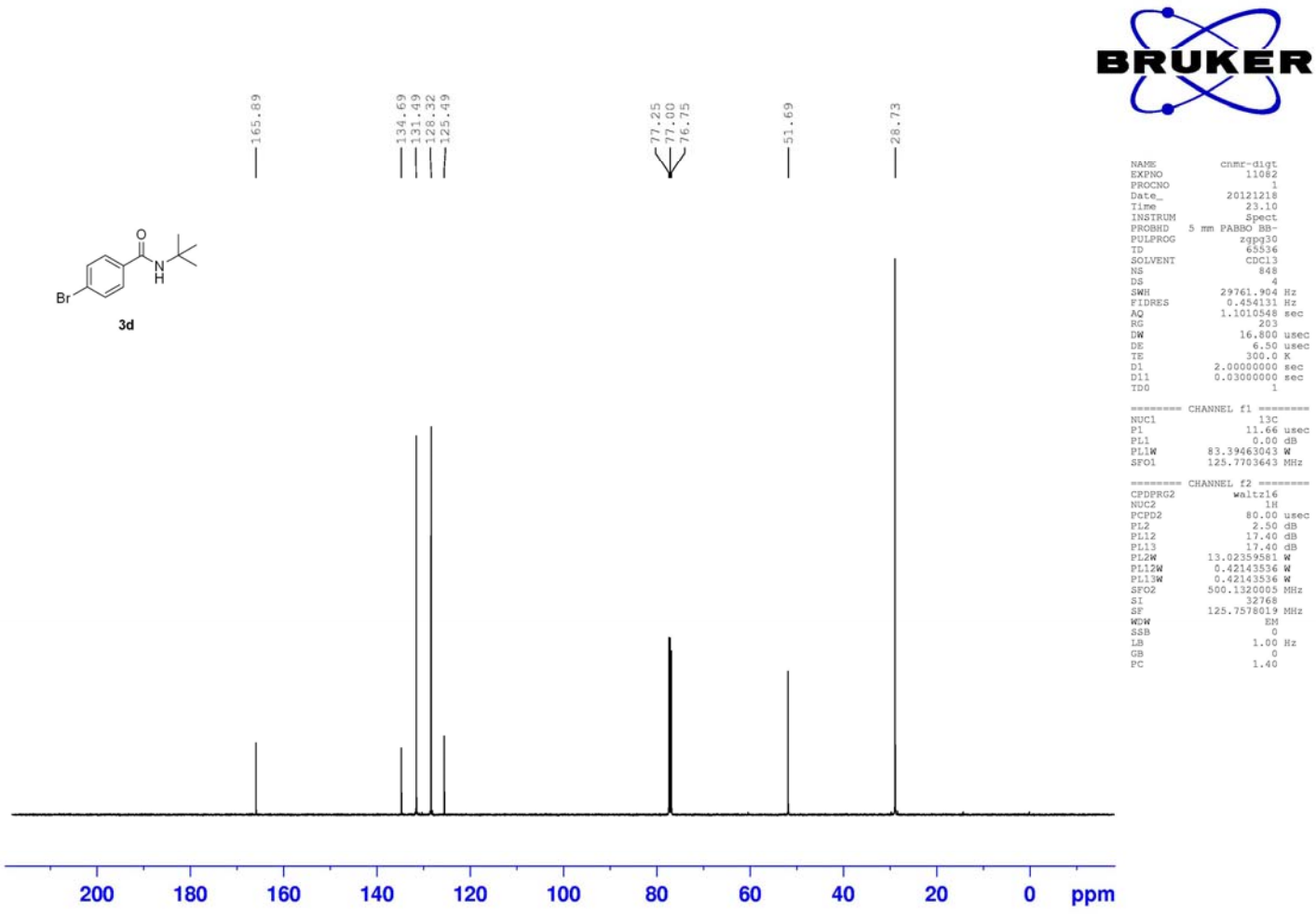


NAME	cnmr-dlgt
EXPNO	11102
PROCNO	1
Date_	20121217
Time	17.15
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	727
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578016 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	pnmr-digt
EXPNO	11082
PROCNO	1
Date_	20130326
Time	17.33
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

```
NAME                cnmr-digt
EXPNO                   11082
PROCNO                      1
Date_                20121218
Time                    23.10
INSTRUM                 Spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                 CDCl3
NS                        848
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        203
DW                     16.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.66 usec
PL1                     0.00 dB
PL1W               83.39463043 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     2.50 dB
PL12                   17.40 dB
PL13                   17.40 dB
PL2W               13.02359581 W
PL12W               0.42143536 W
PL13W               0.42143536 W
SFO2              500.1320005 MHz
SI                       32768
SF                125.7578019 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                        1.40
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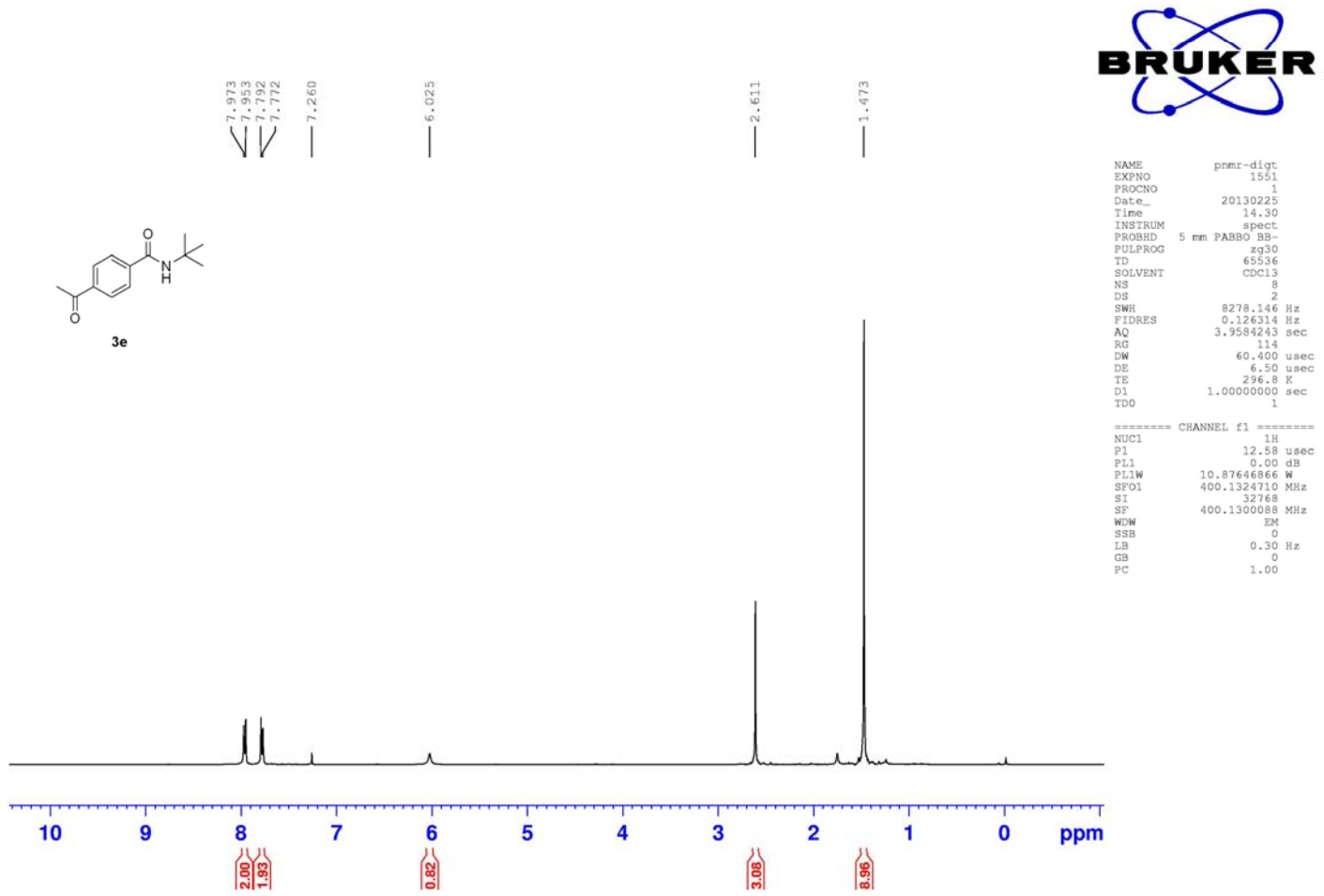

BRUKER

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NAME            pnmr-digt
EXPNO                1551
PROCNO                  1
Date_            20130225
Time                14.30
INSTRUM             spect
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PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    114
DW                 60.400 usec
DE                  6.50 usec
TE                 296.8 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.58 usec
PL1                  0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300088 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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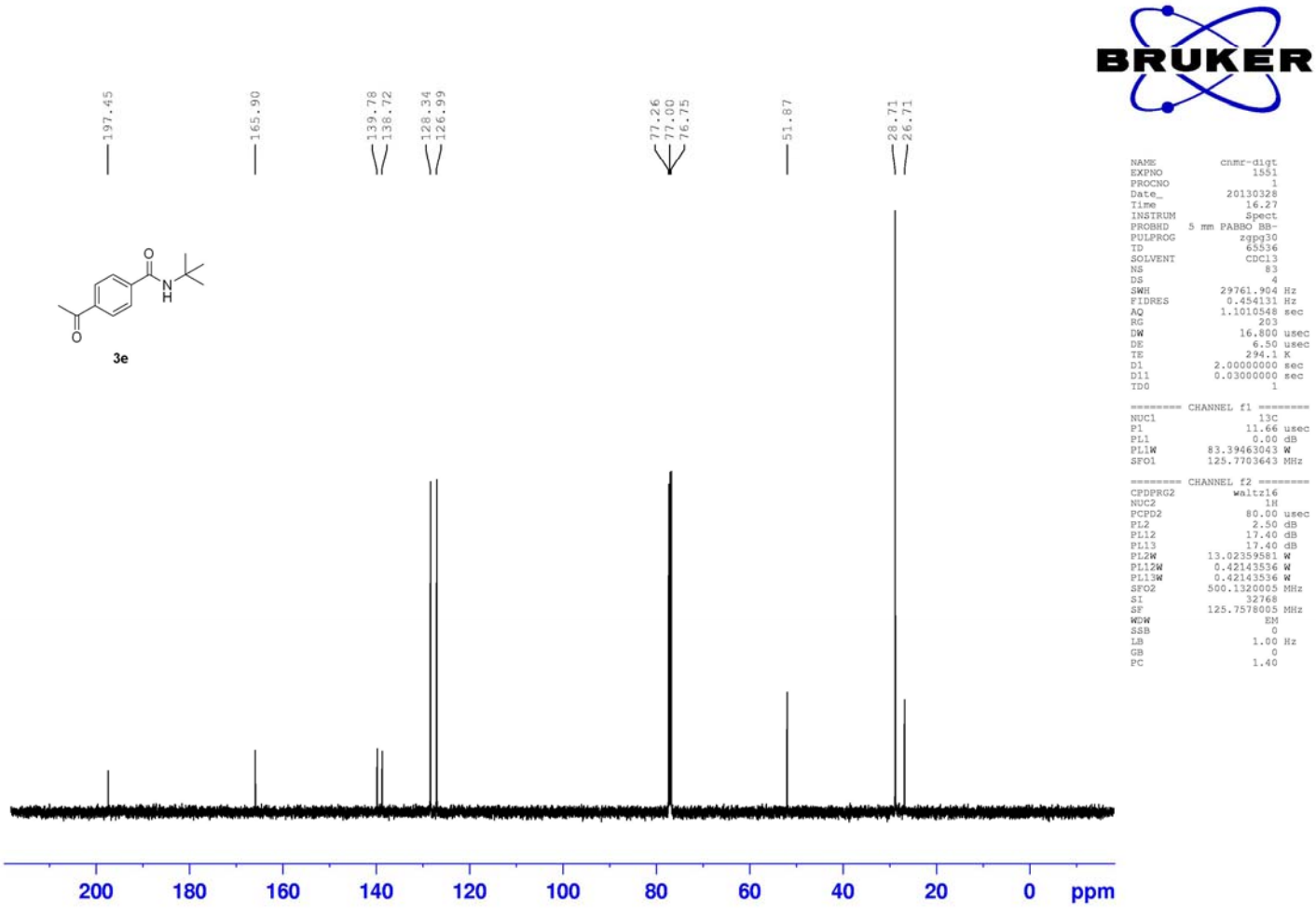
197.45
165.90
139.78
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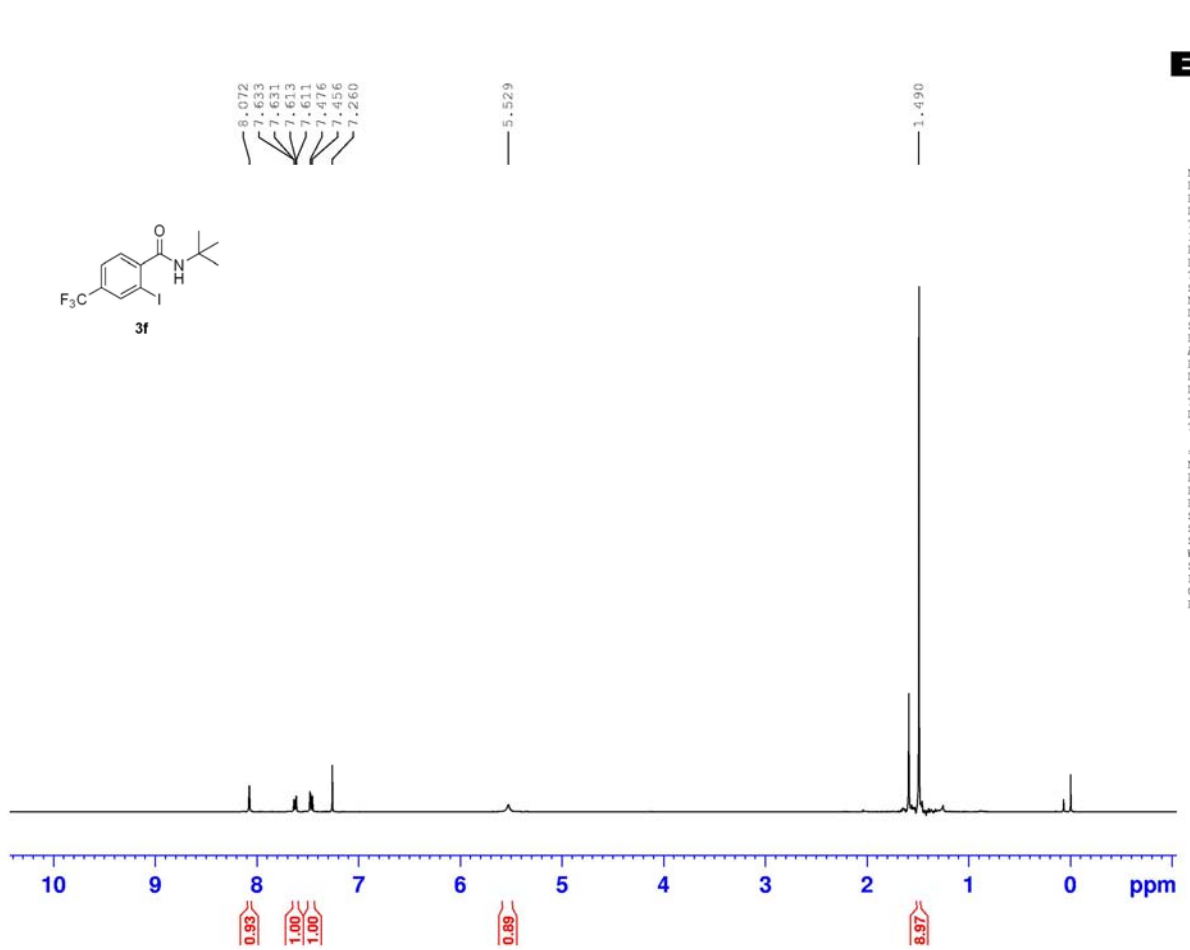
126.99
77.26
77.00
76.75
51.87
28.71
26.71
3e
200
180
160
140
120
100
80
60
40
20
0
ppm

NAME cnmr-diqt
EXPNO 1551
PROCNO 1
Date_ 20130328
Time 16.27
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 83
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.66 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578005 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

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PROCNO	1
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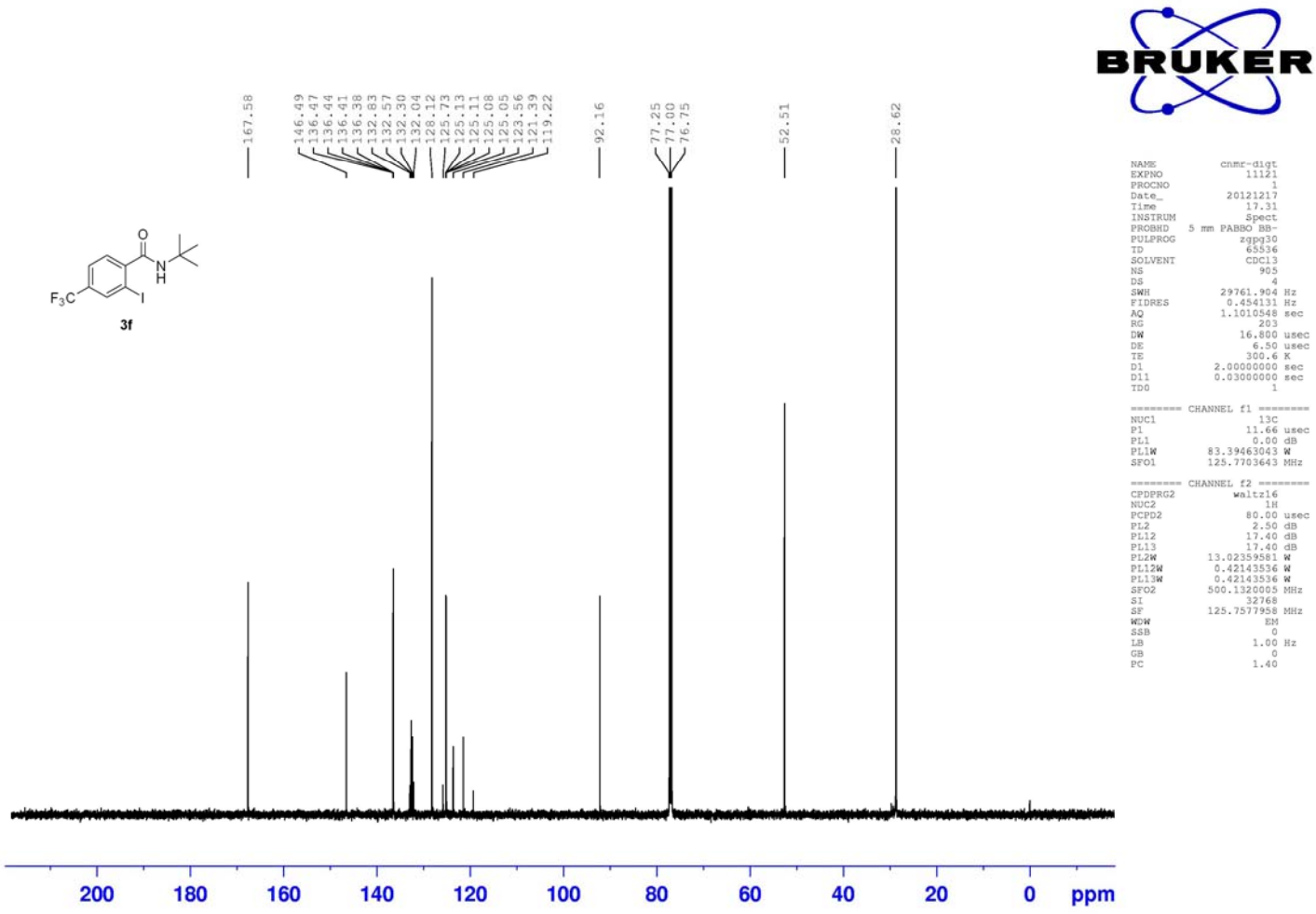
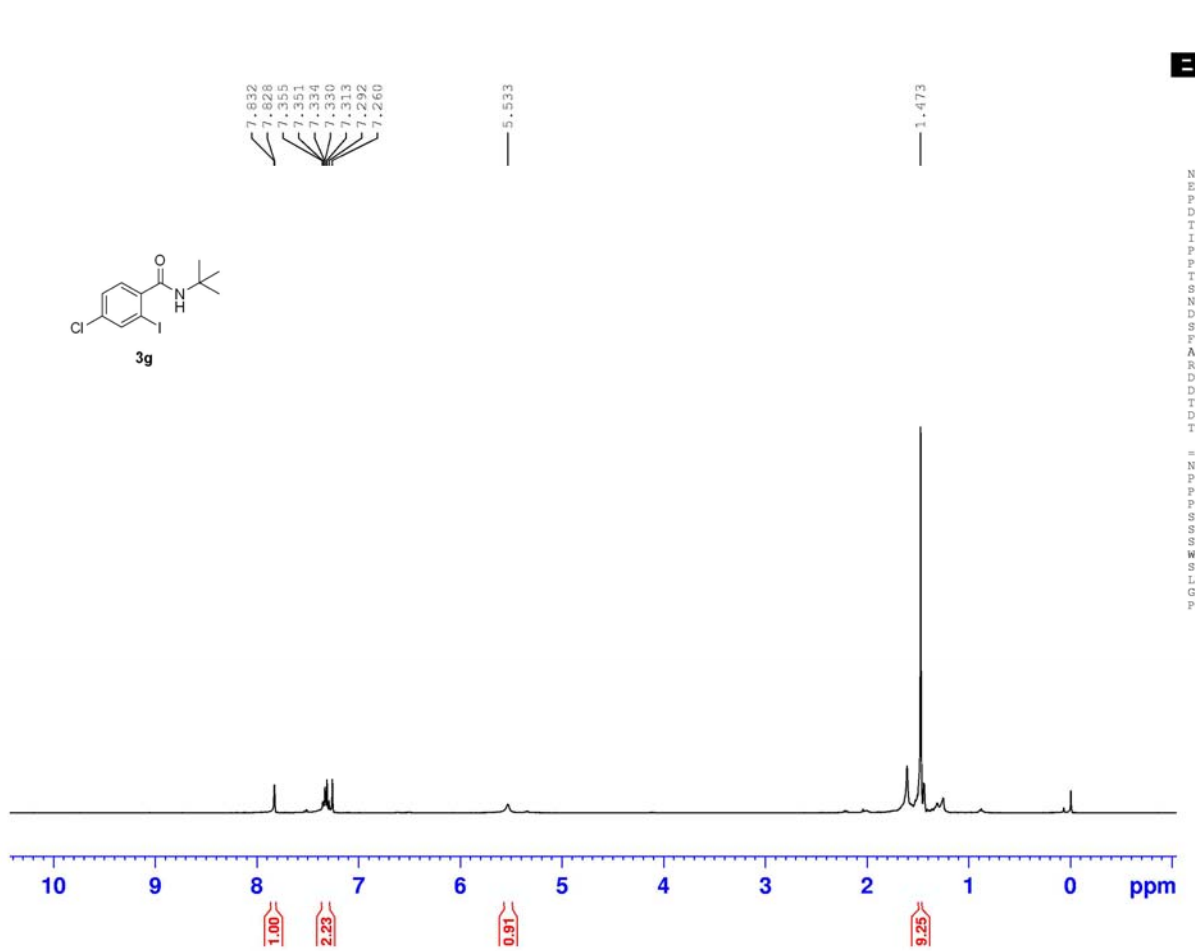
Time	19.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	299.7 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



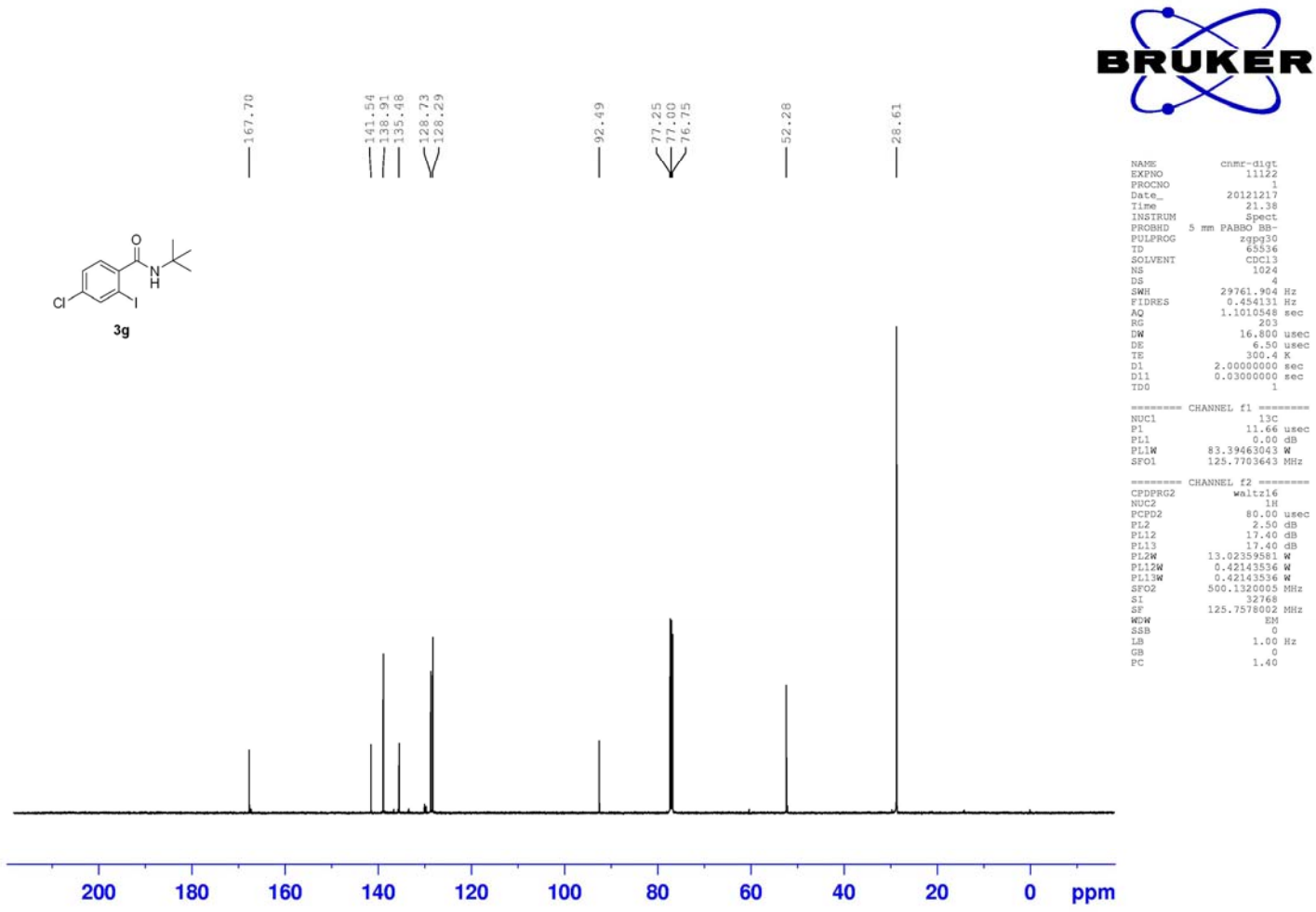
BRUKER

NAME	cnmr-dlgt
EXPNO	11121
PROCNO	1
Date_	20121217
Time	17.31
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	905
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577958 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



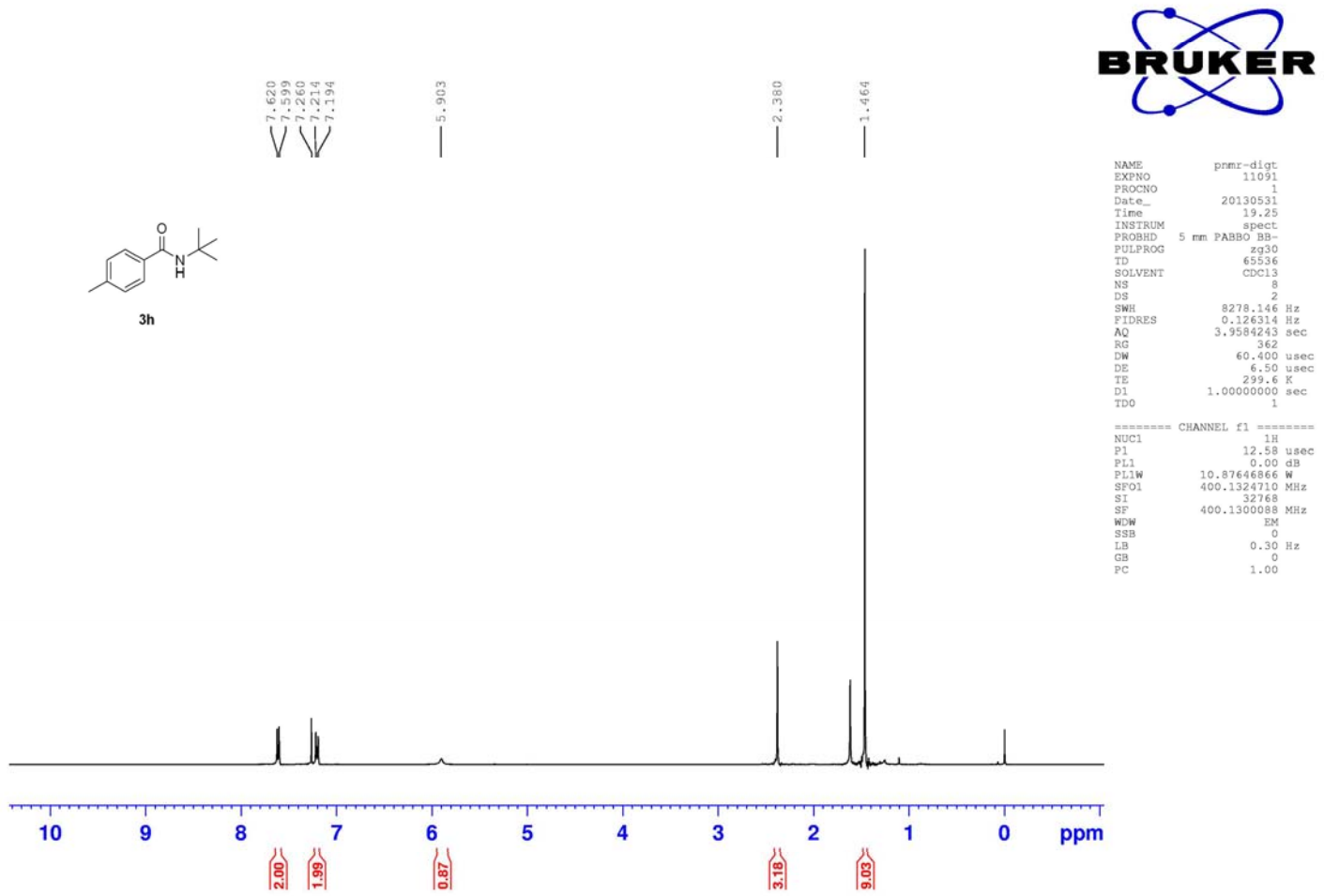
BRUKER

NAME	pnmr-digt
EXPNO	11122
PROCNO	1
Date_	20130326
Time	17.53
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	297.5 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

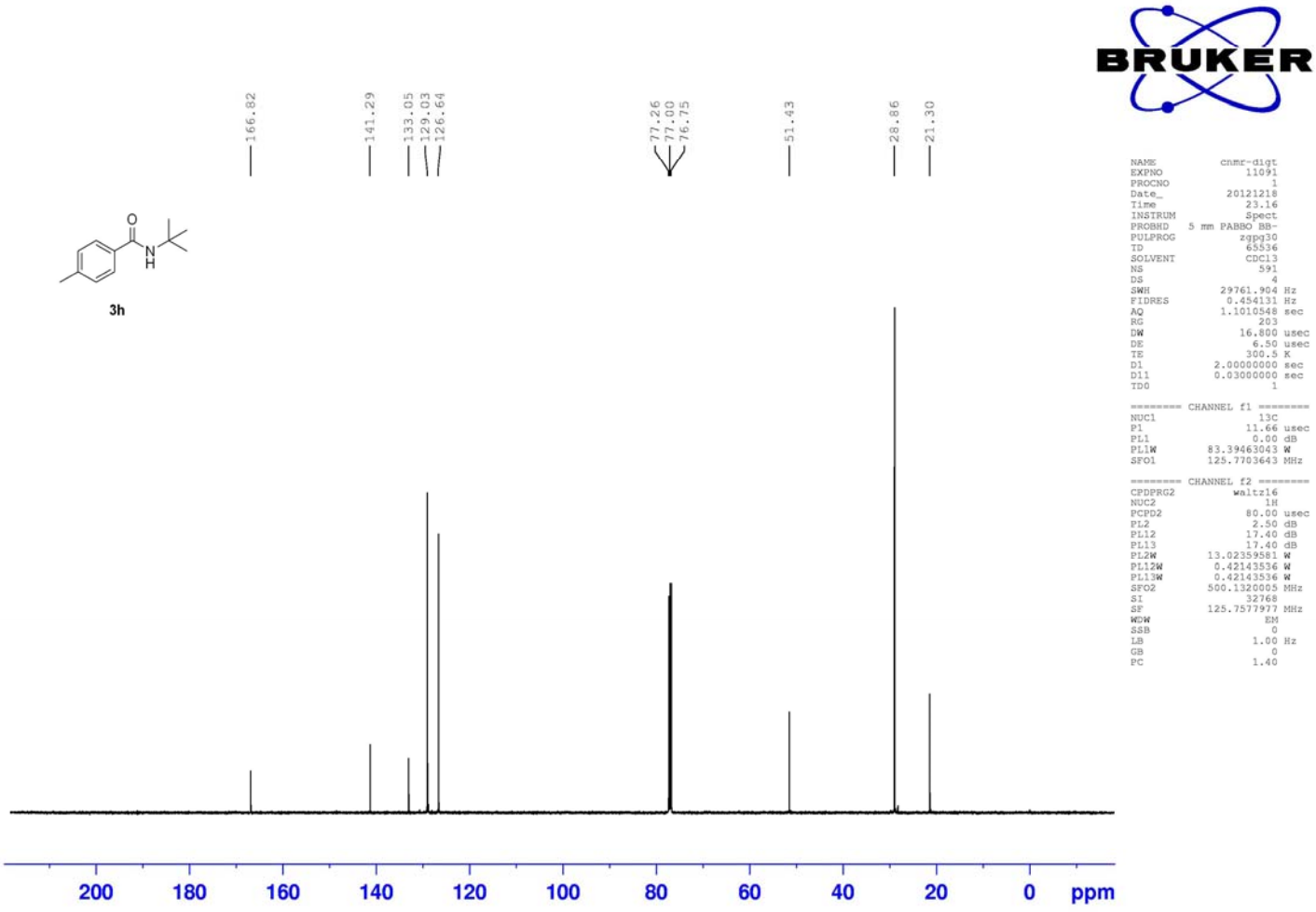


BRUKER

NAME	cnmr-dlgt
EXPNO	11122
PROCNO	1
Date_	20121217
Time	21.38
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578002 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

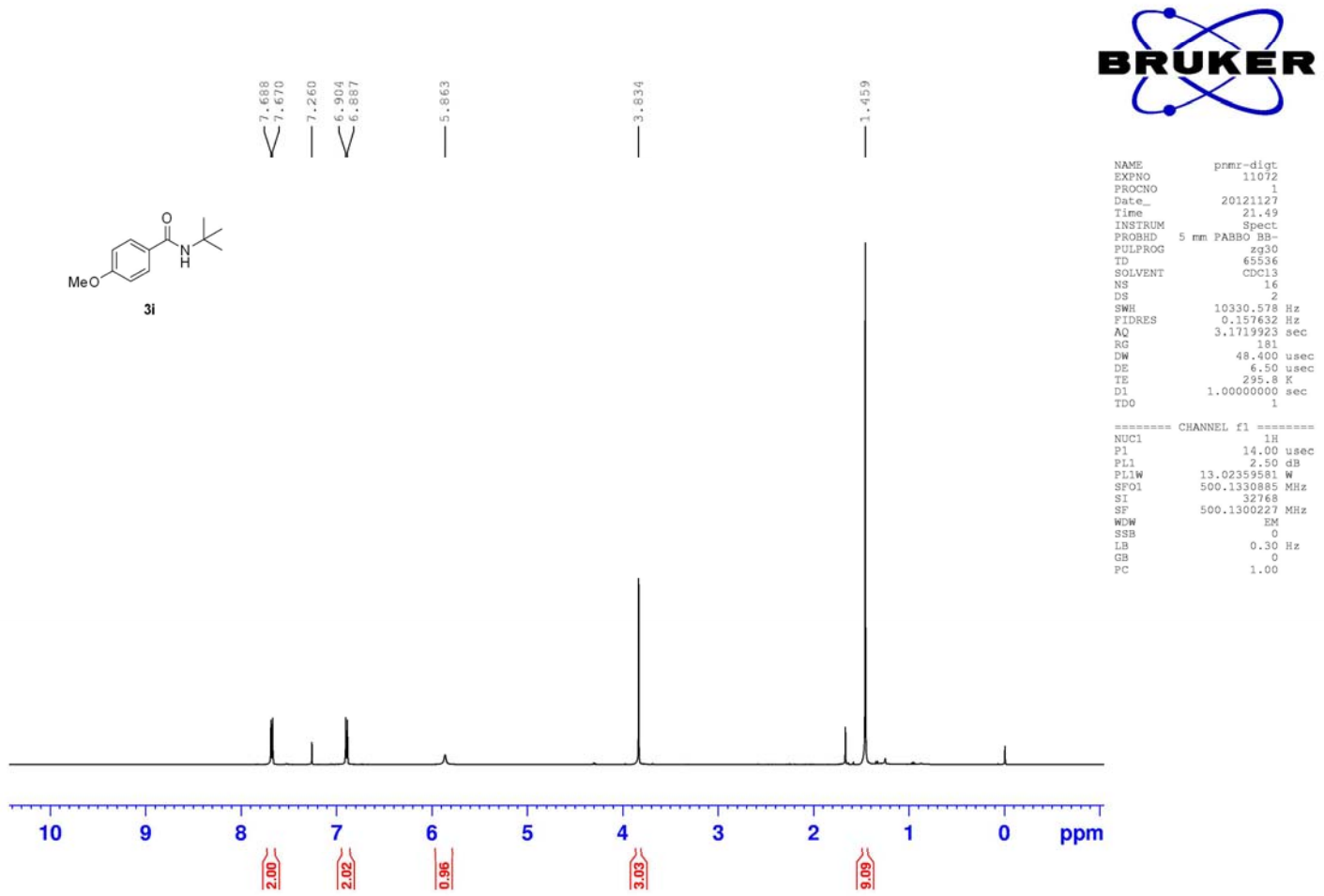



```
NAME            pnmr-digt
EXPNO               11091
PROCNO                  1
Date_            20130531
Time                19.25
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    362
DW                60.400 usec
DE                 6.50 usec
TE                299.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.58 usec
PL1                 0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300088 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

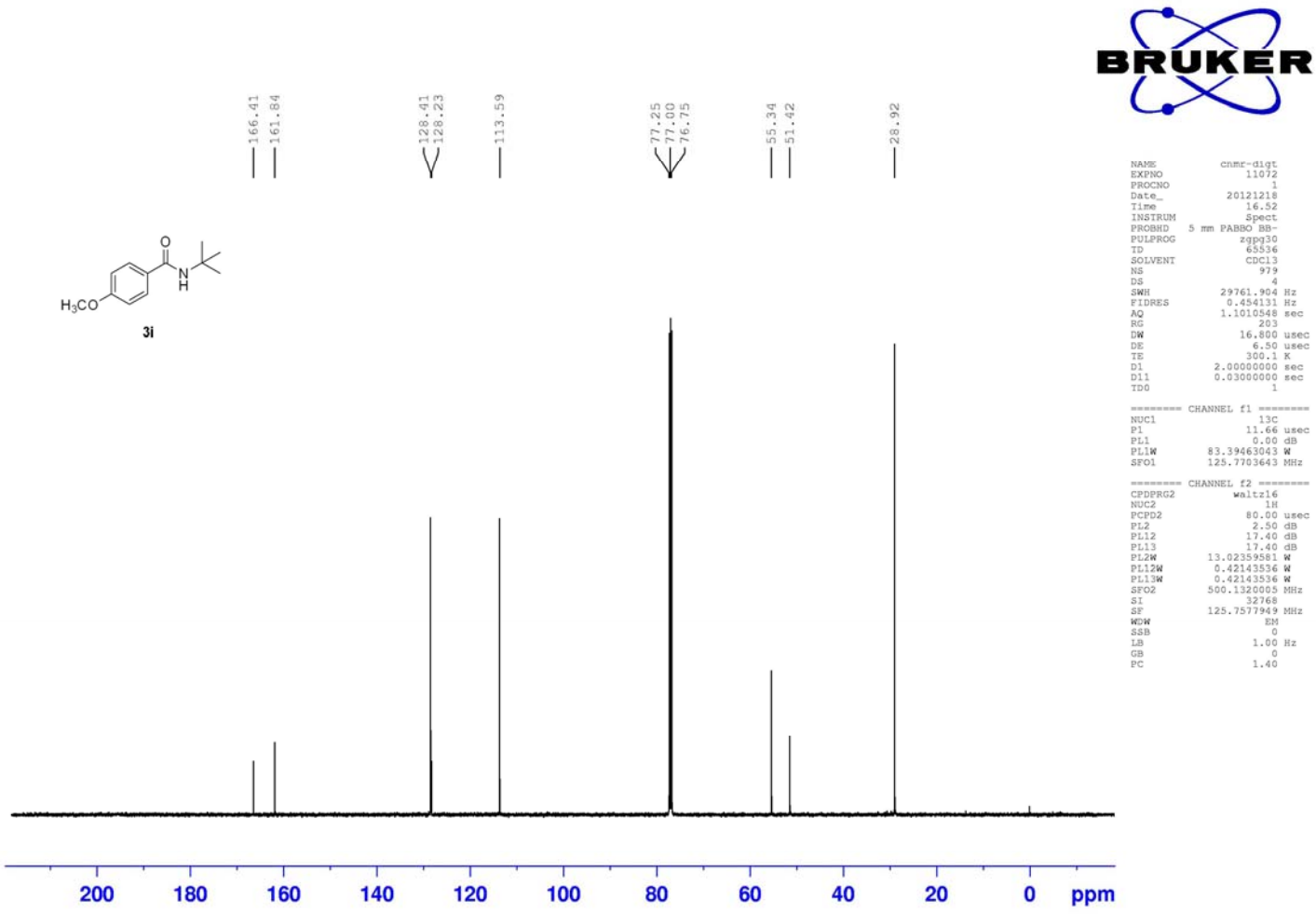


BRUKER

NAME	cnmr-dlgt
EXPNO	11091
PROCNO	1
Date_	20121218
Time	23.16
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	591
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577977 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

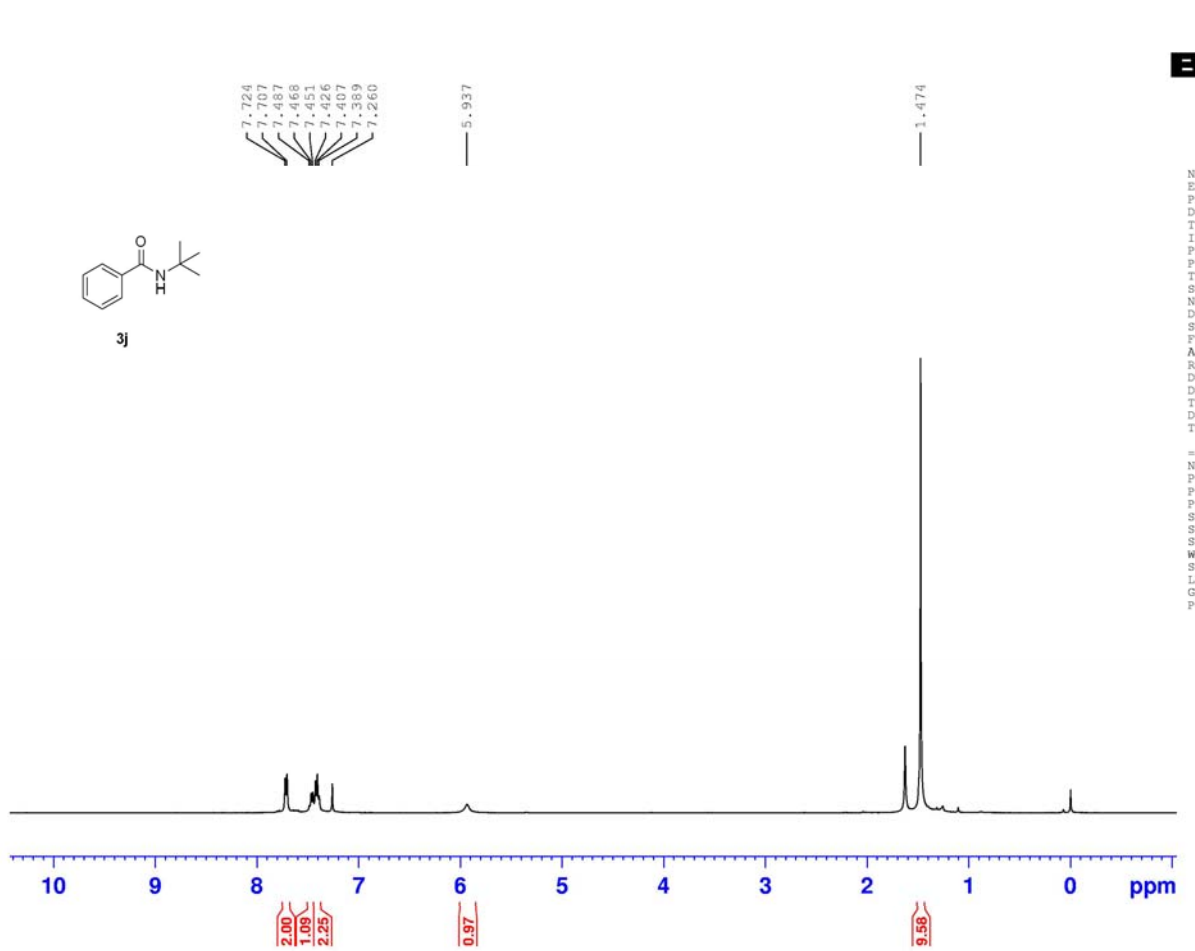



```
NAME            pnmr-digt
EXPNO               11072
PROCNO                  1
Date_            20121127
Time                21.49
INSTRUM             Spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719923 sec
RG                    181
DW                48.400 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                 2.50 dB
PL1W         13.02359581 W
SFO1         500.1330885 MHz
SI                  32768
SF           500.1300227 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

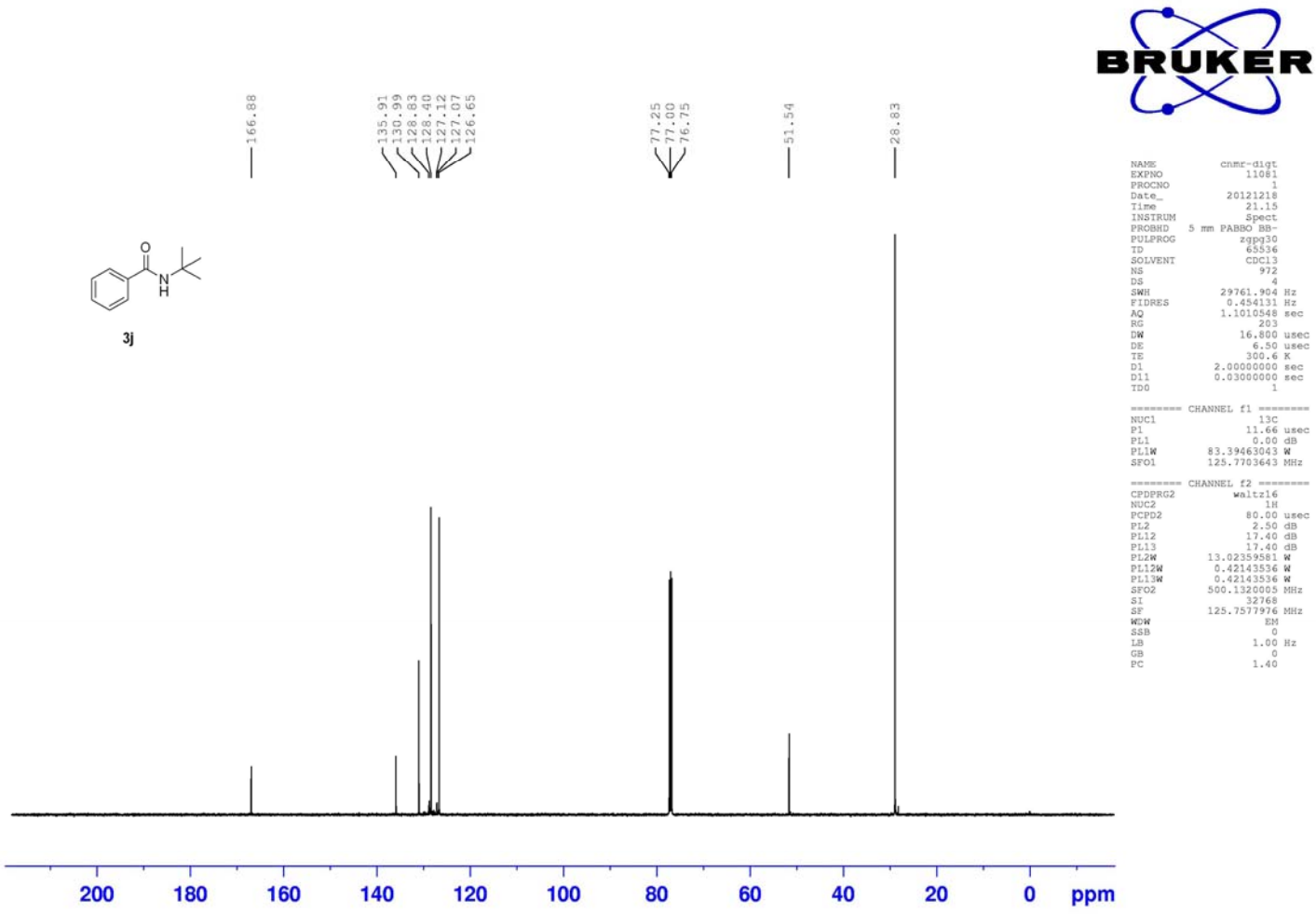


BRUKER

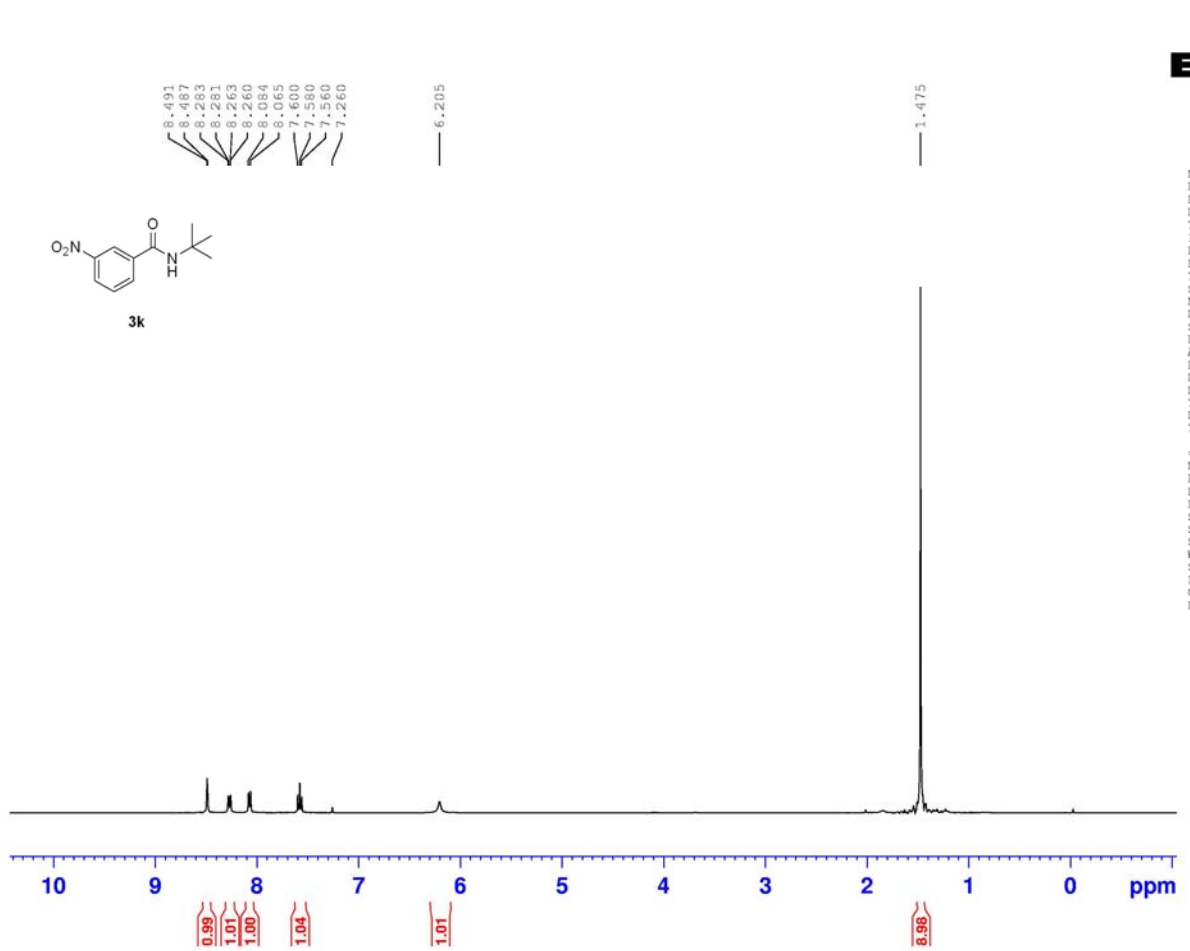
NAME	cnmr-dlgt
EXPNO	11072
PROCNO	1
Date_	20121218
Time	16.52
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	979
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577949 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40




```
NAME            pnmr-digt
EXPNO               11081
PROCNO                  1
Date_            20130531
Time                16.51
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    362
DW                 60.400 usec
DE                  6.50 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.58 usec
PL1                  0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300109 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

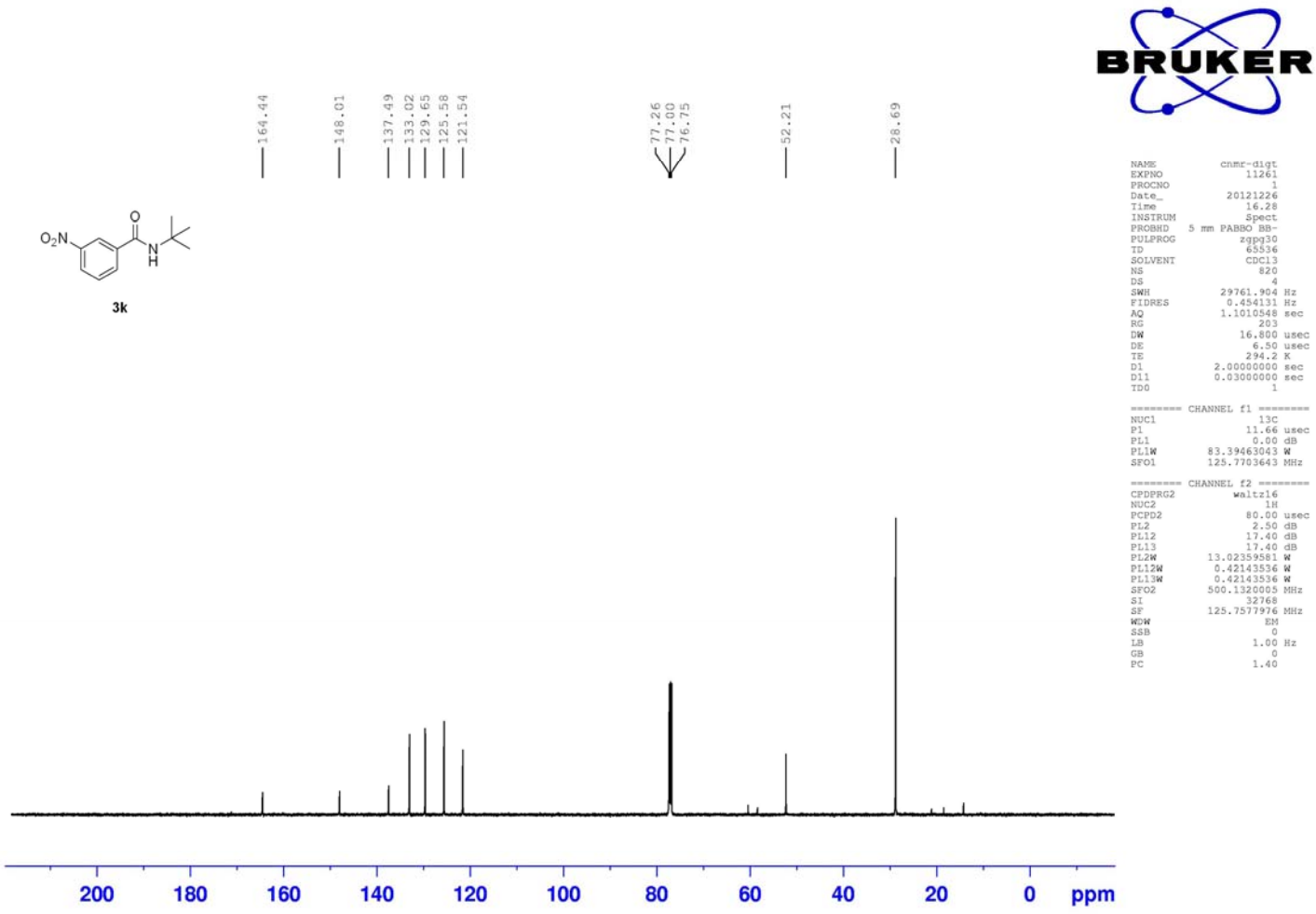



```
NAME                cnmr-digt
EXPNO                   11081
PROCNO                      1
Date_                20121218
Time                    21.15
INSTRUM                 Spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        972
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        203
DW                     16.800 usec
DE                      6.50 usec
TE                      300.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.66 usec
PL1                     0.00 dB
PL1W               83.39463043 W
SFO1               125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     2.50 dB
PL12                   17.40 dB
PL13                   17.40 dB
PL2W               13.02359581 W
PL12W               0.42143536 W
PL13W               0.42143536 W
SFO2               500.1320005 MHz
SI                       32768
SF                 125.7577976 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```



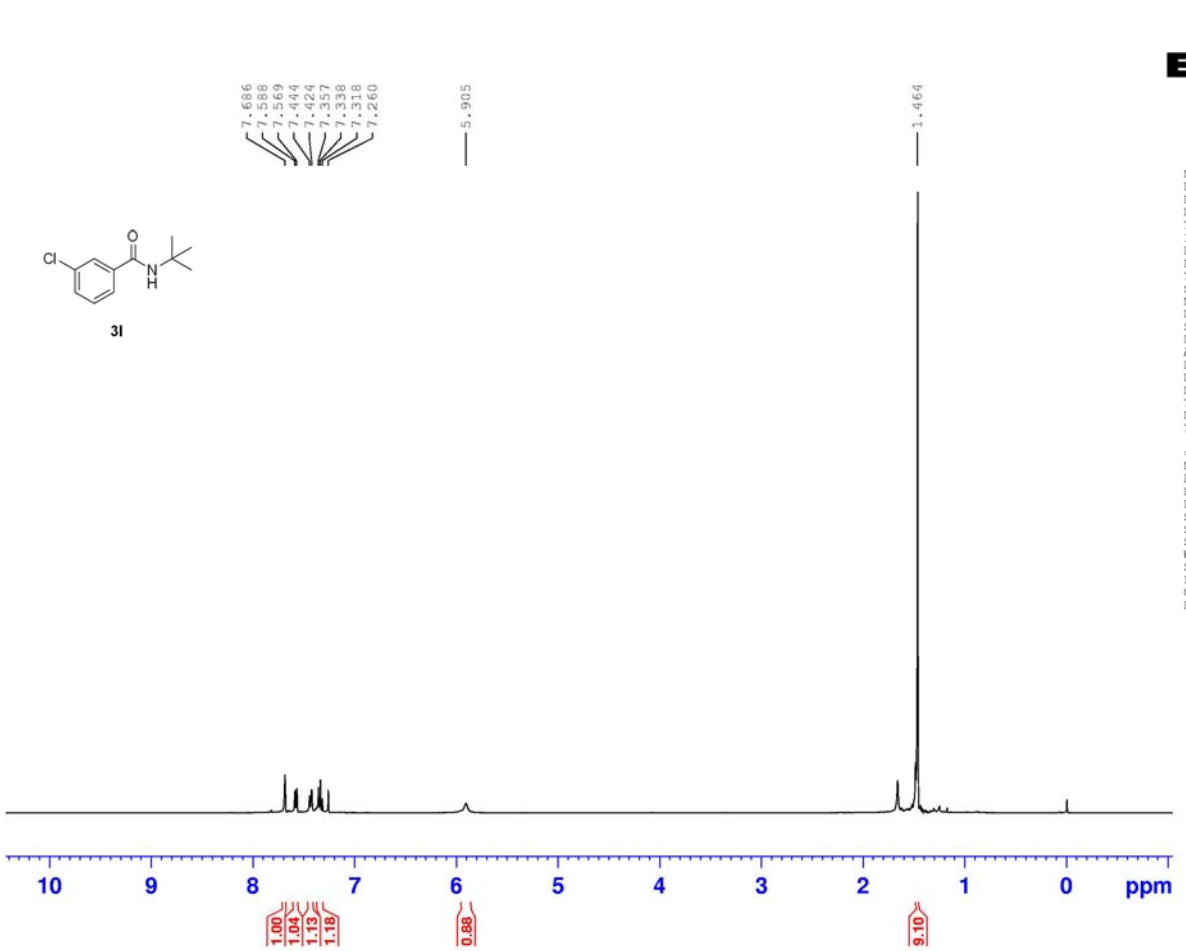
BRUKER

NAME	pnmr-digt
EXPNO	11261
PROCNO	1
Date_	20121218
Time	11.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	64
DW	60.400 usec
DE	6.50 usec
TE	296.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

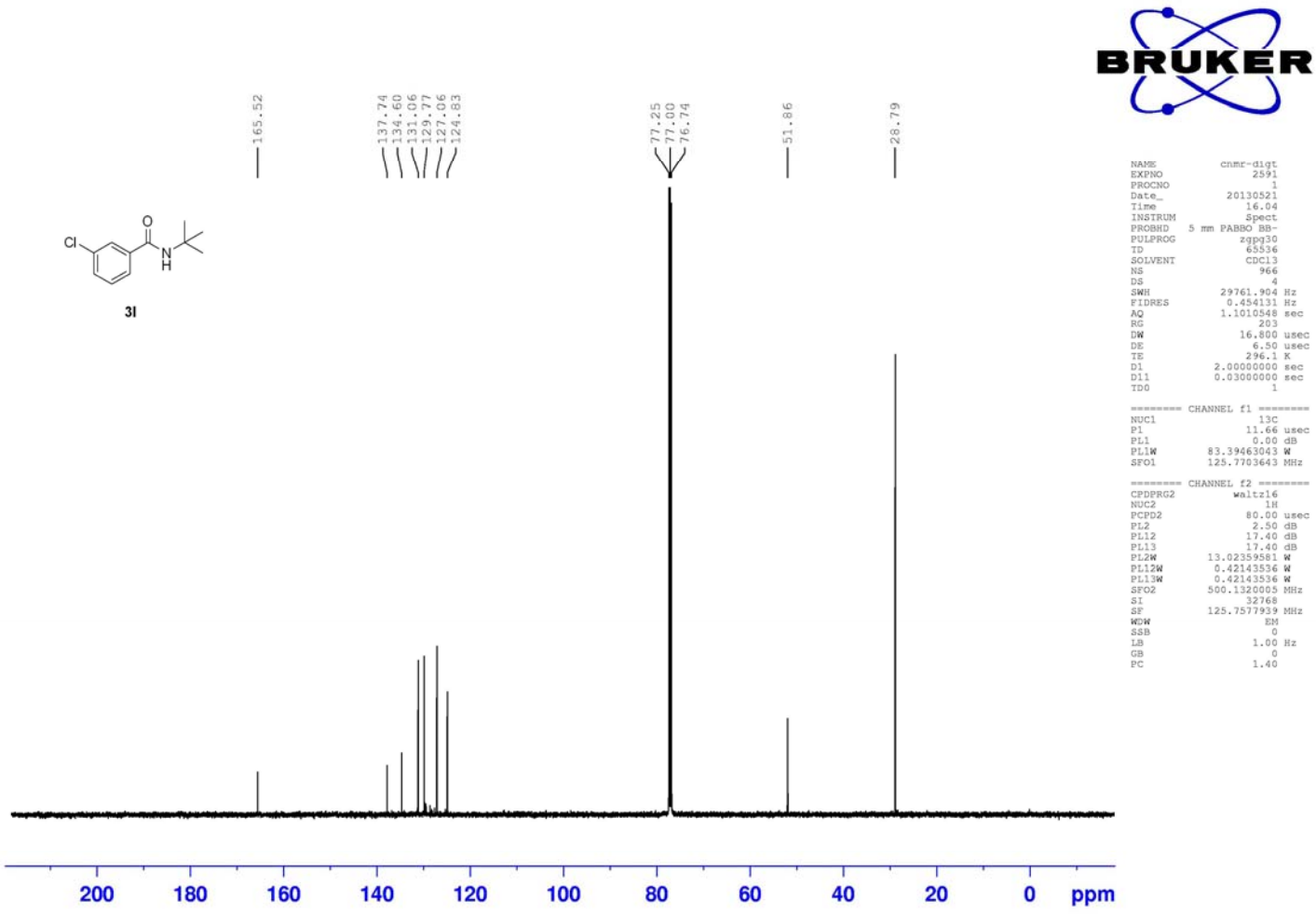


BRUKER

NAME	cnmr-dlgt
EXPNO	11261
PROCNO	1
Date_	20121226
Time	16.28
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	820
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	294.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577976 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

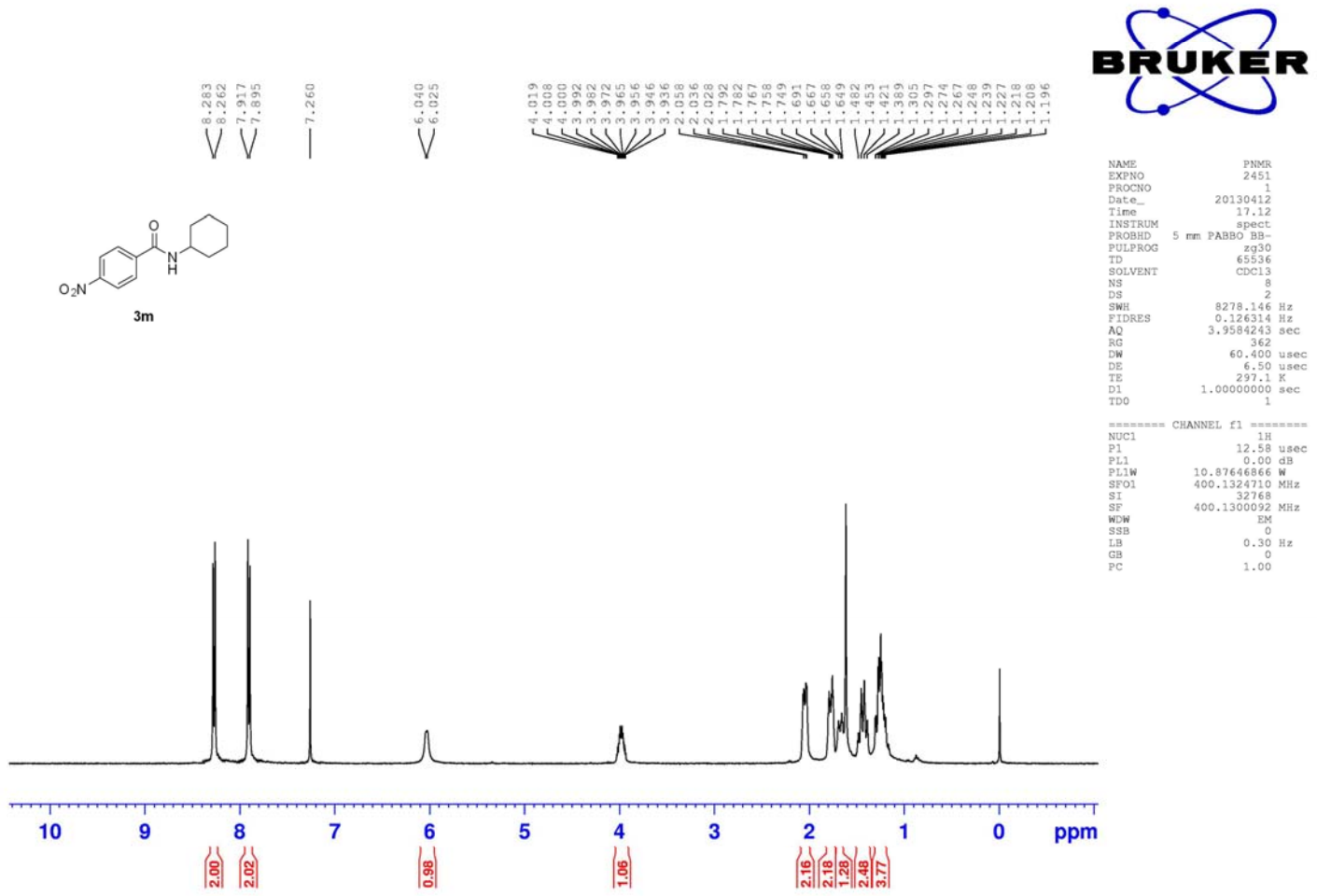



```
NAME            pnmr-digt
EXPNO                2591
PROCNO                  1
Date_            20130515
Time                19.45
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                  228.1
DW                 60.400 usec
DE                  6.50 usec
TE                 298.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.58 usec
PL1                  0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300087 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



BRUKER

NAME	cnmr-dlgt
EXPNO	2591
PROCNO	1
Date_	20130521
Time	16.04
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	966
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577939 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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6.040
6.025
4.019
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4.000
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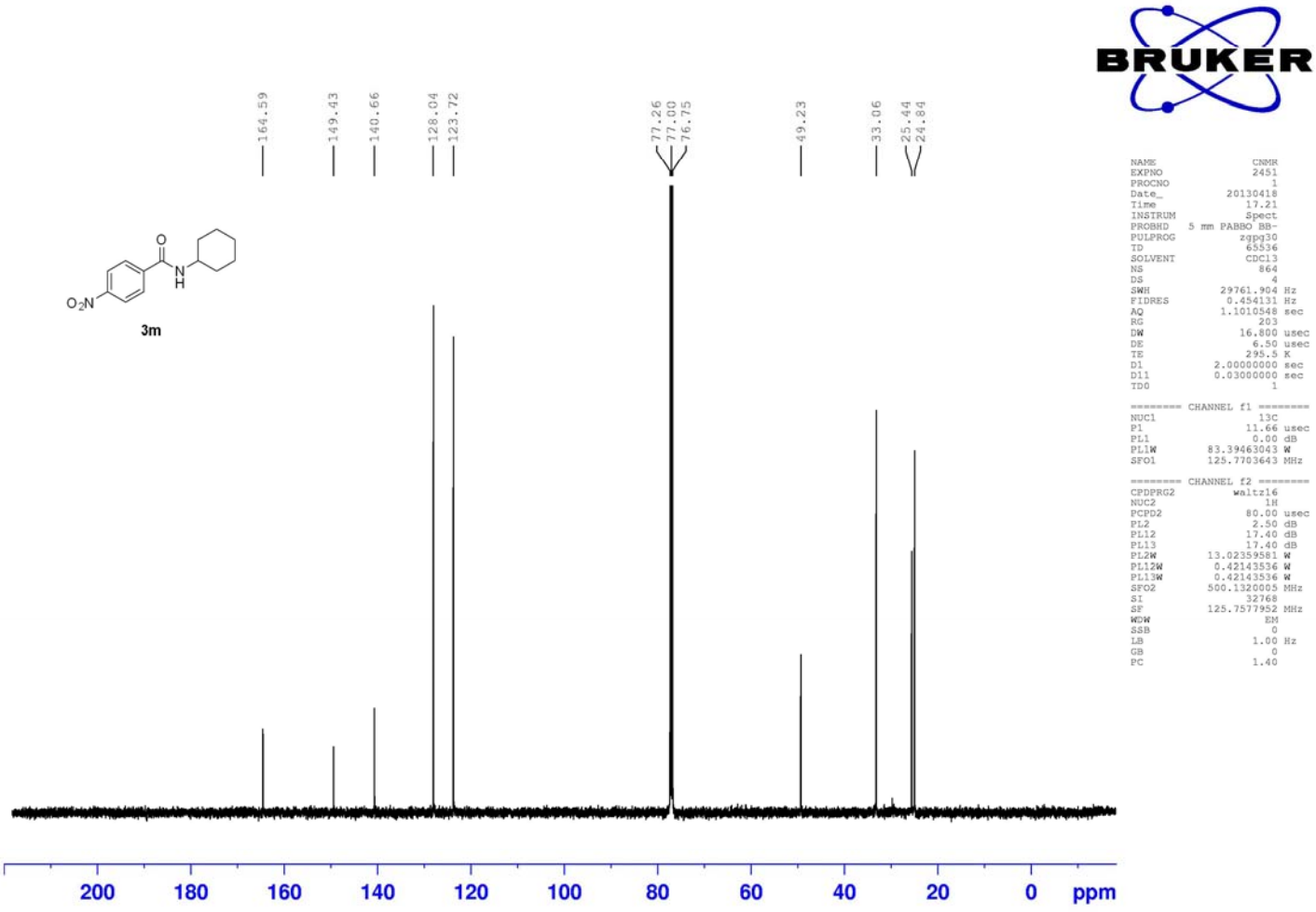
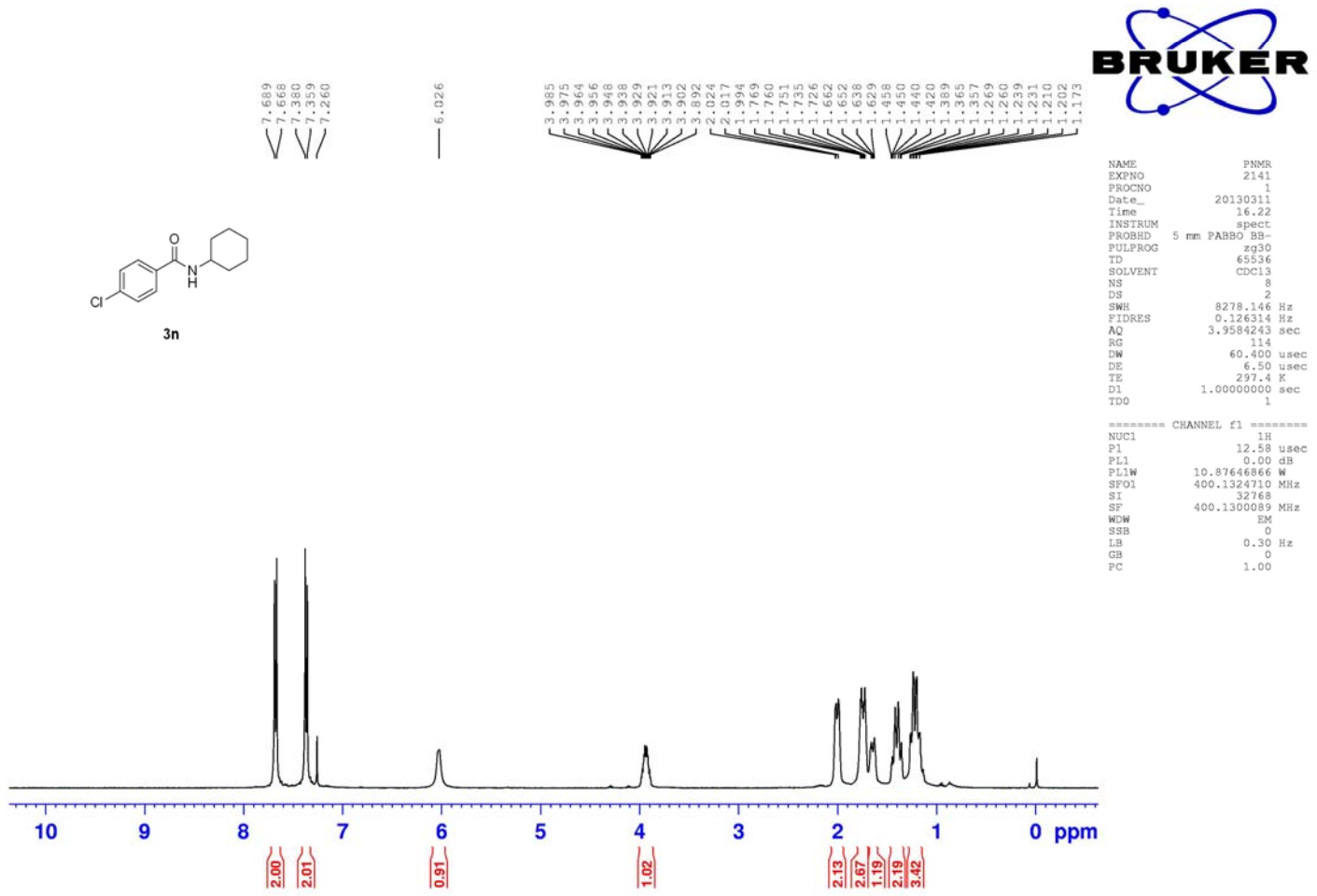
3.982
3.972
3.965
3.956
3.946
3.936
2.058
2.036
2.028
1.792
1.782
1.767
1.758
1.749
1.691
1.667
1.658
1.649
1.482
1.453
1.421
1.389
1.305
1.297
1.274
1.267
1.248
1.239
1.227
1.218
1.208
1.196
O
N
H
O2N
3m
BRUKER
NAME PNMR
EXPNO 2451
PROCNO 1
Date_ 20130412
Time 17.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.02
0.98
1.06
2.16
2.18
1.28
2.48
3.77



BRUKER

NAME	CNMR
EXPNO	2451
PROCNO	1
Date_	20130418
Time	17.21
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	864
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	295.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577952 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


7.689
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7.260
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3.938
3.929
3.921
3.913
3.902
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2.017
1.994
1.769
1.760
1.751
1.735
1.726
1.662
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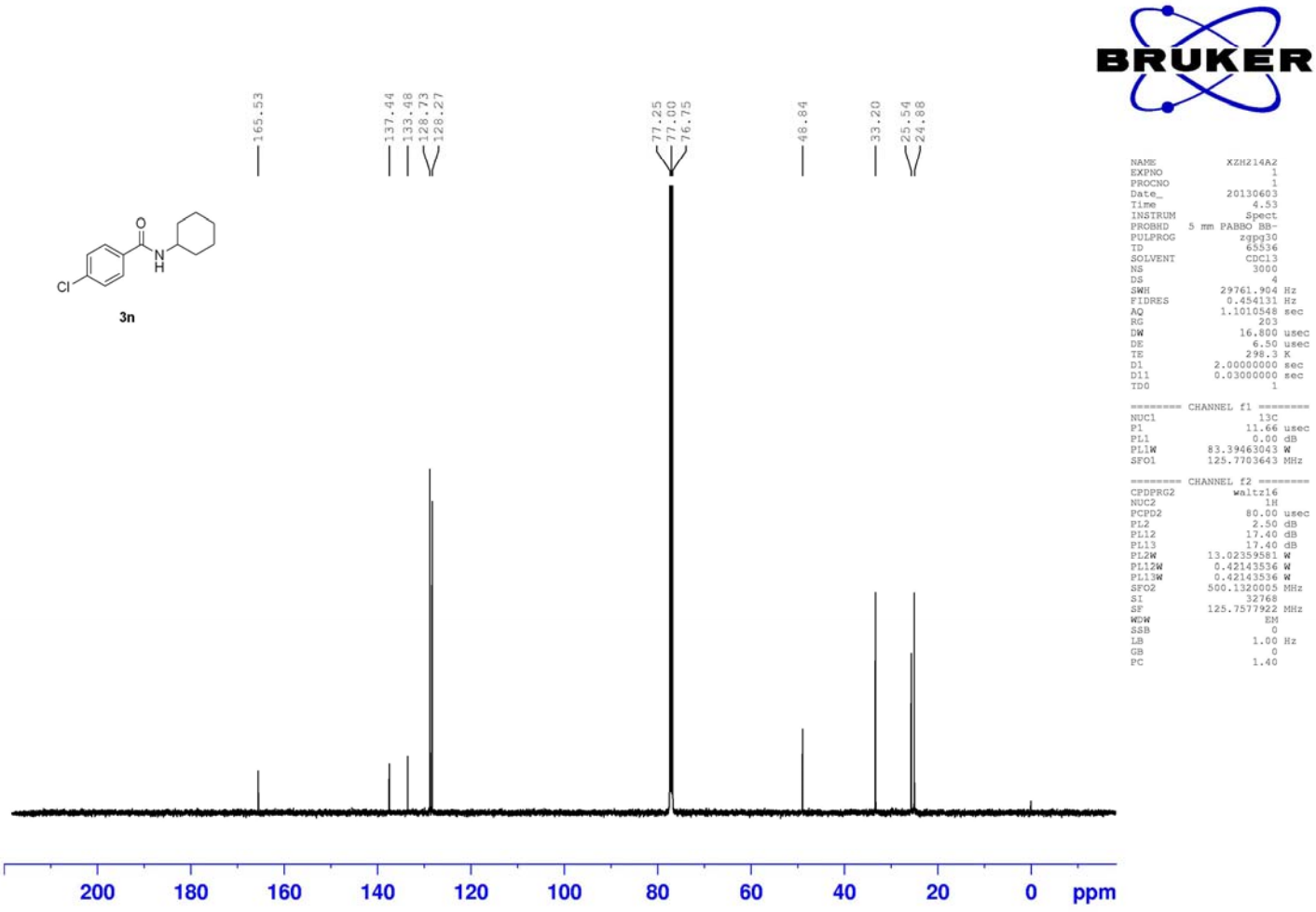
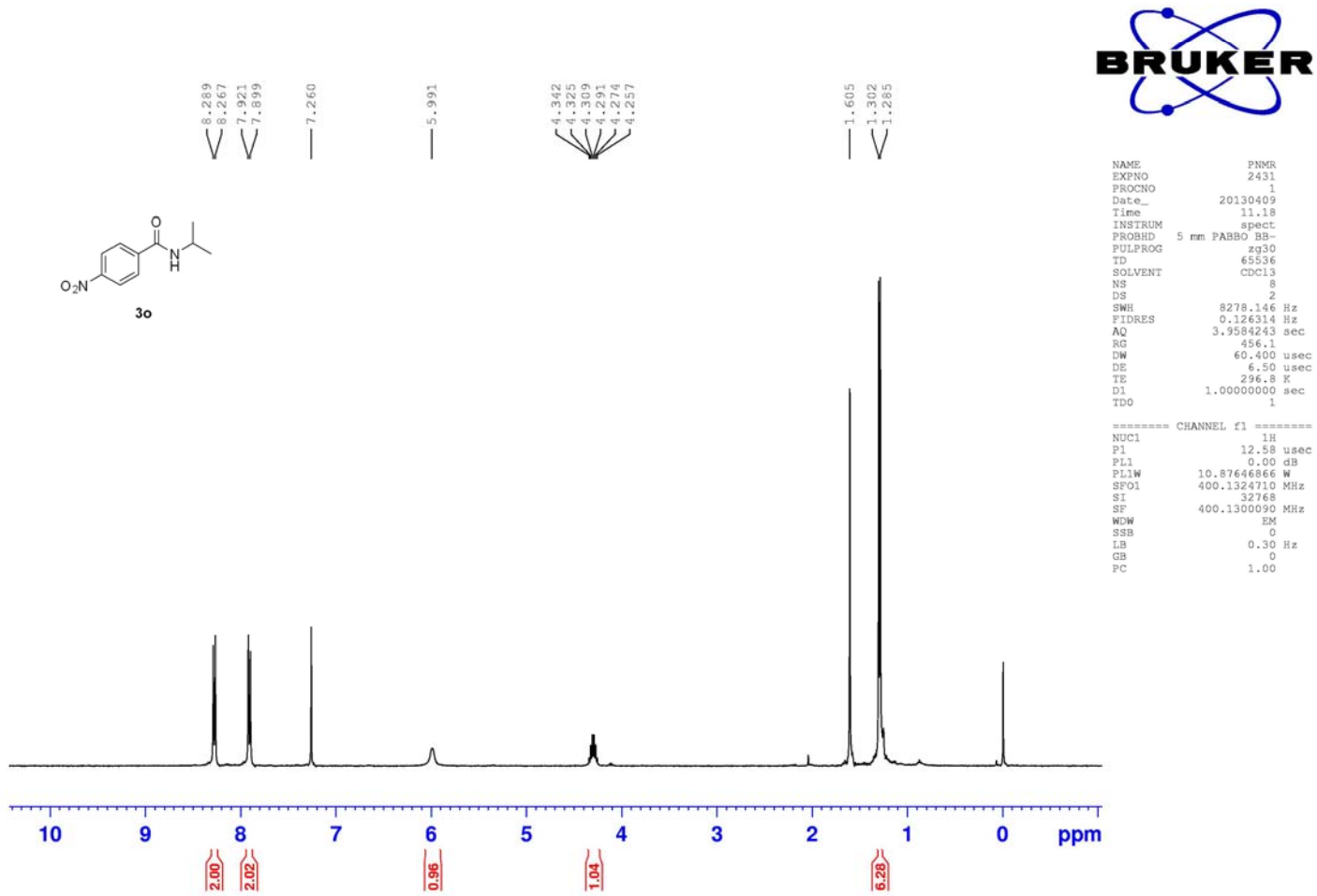
1.202
1.173
BRUKER
NAME PNMR
EXPNO 2141
PROCNO 1
Date_ 20130311
Time 16.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
H
Cl
3n
10
9
8
7
6
5
4
3
2
1
0 ppm
2.00
2.01
0.91
1.02
2.13
2.67
1.19
2.19
3.42

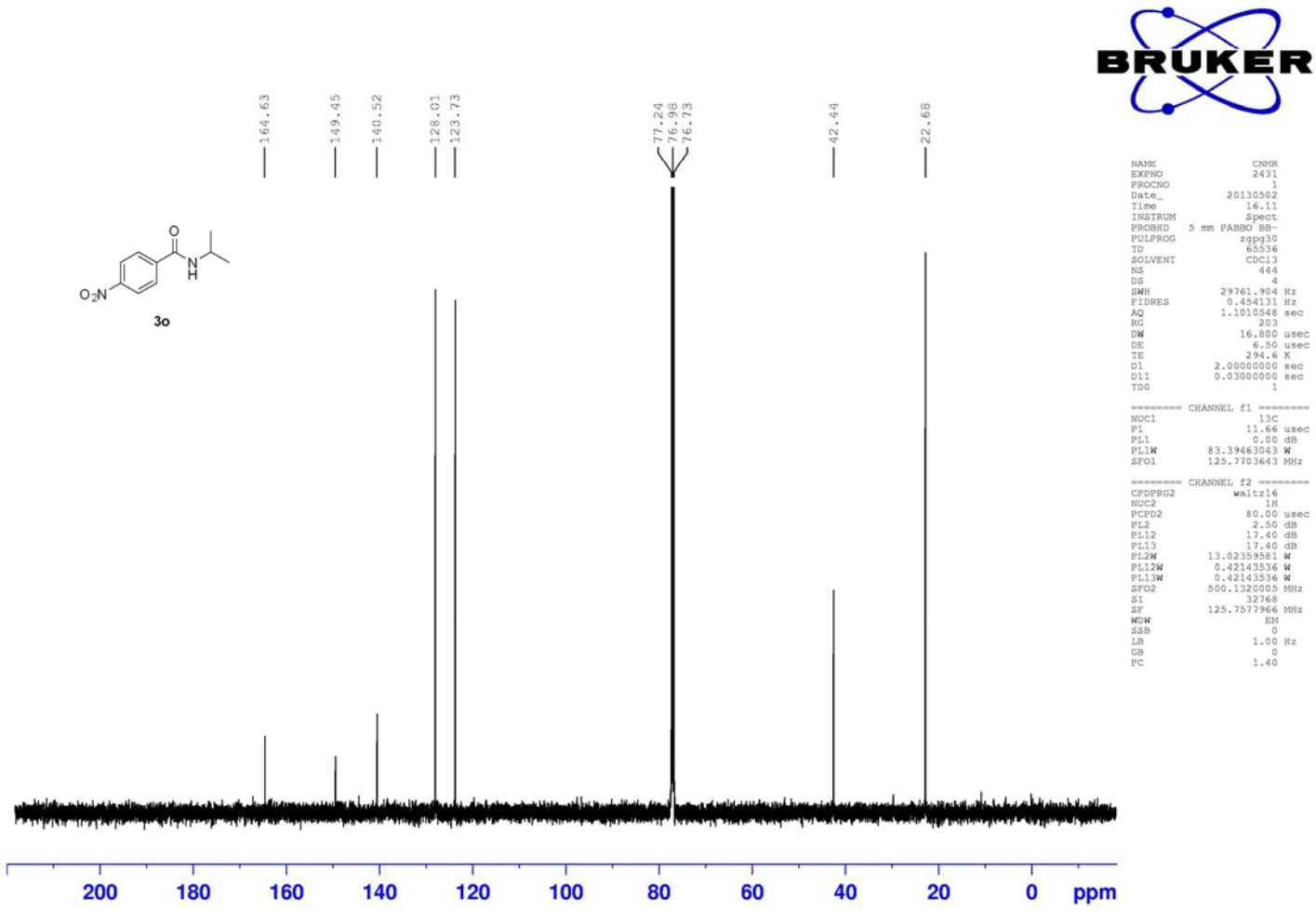


BRUKER

NAME	XZH214A2
EXPNO	1
PROCNO	1
Date_	20130603
Time	4.53
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3000
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577922 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



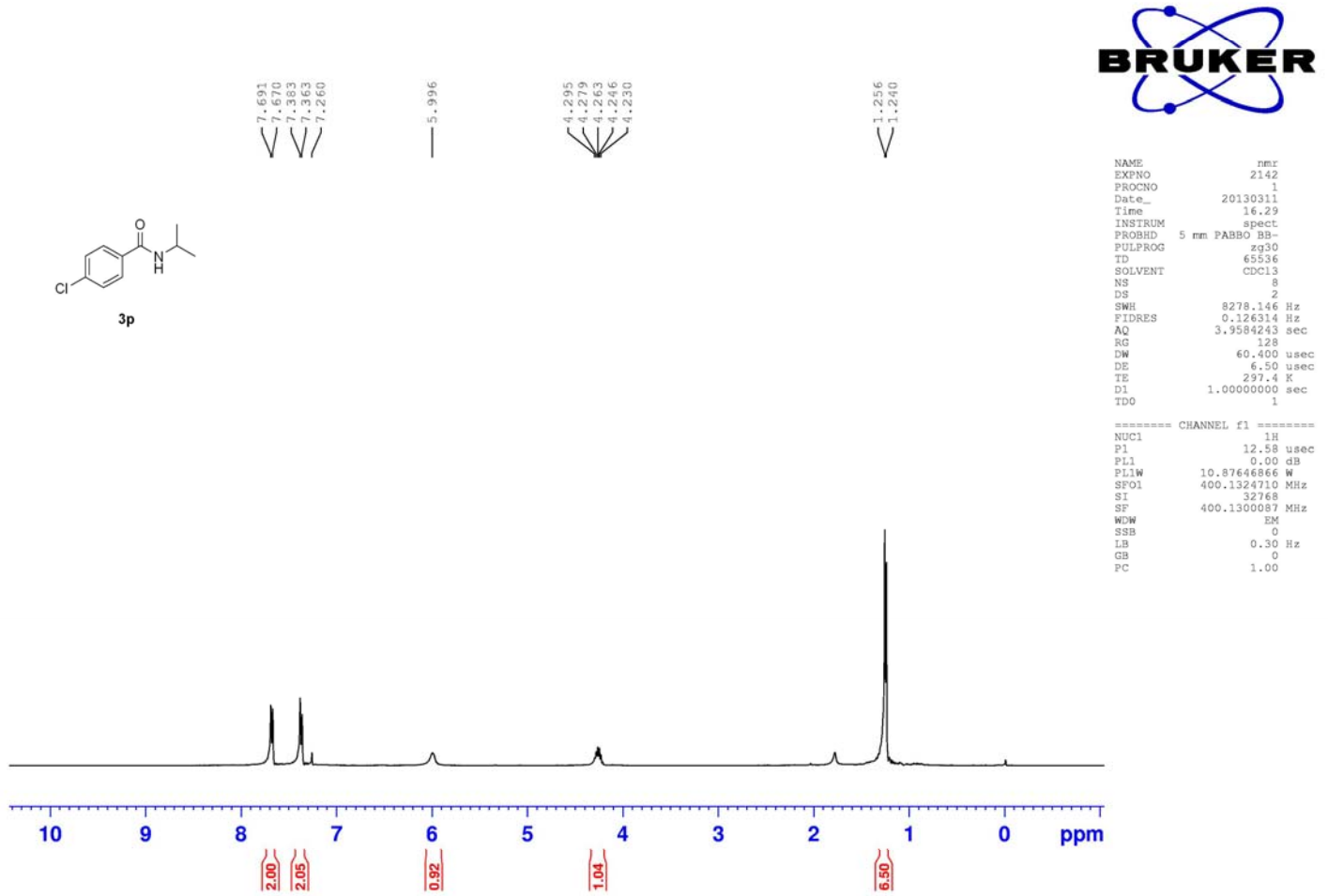

```
NAME                PNMR
EXPNO               2431
PROCNO                 1
Date_           20130409
Time               11.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 456.1
DW                60.400 usec
DE                 6.50 usec
TE                296.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300090 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



NAME CNMR
EXPNO 2431
PROCNO 1
Date_ 20130502
Time 16.11
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 444
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

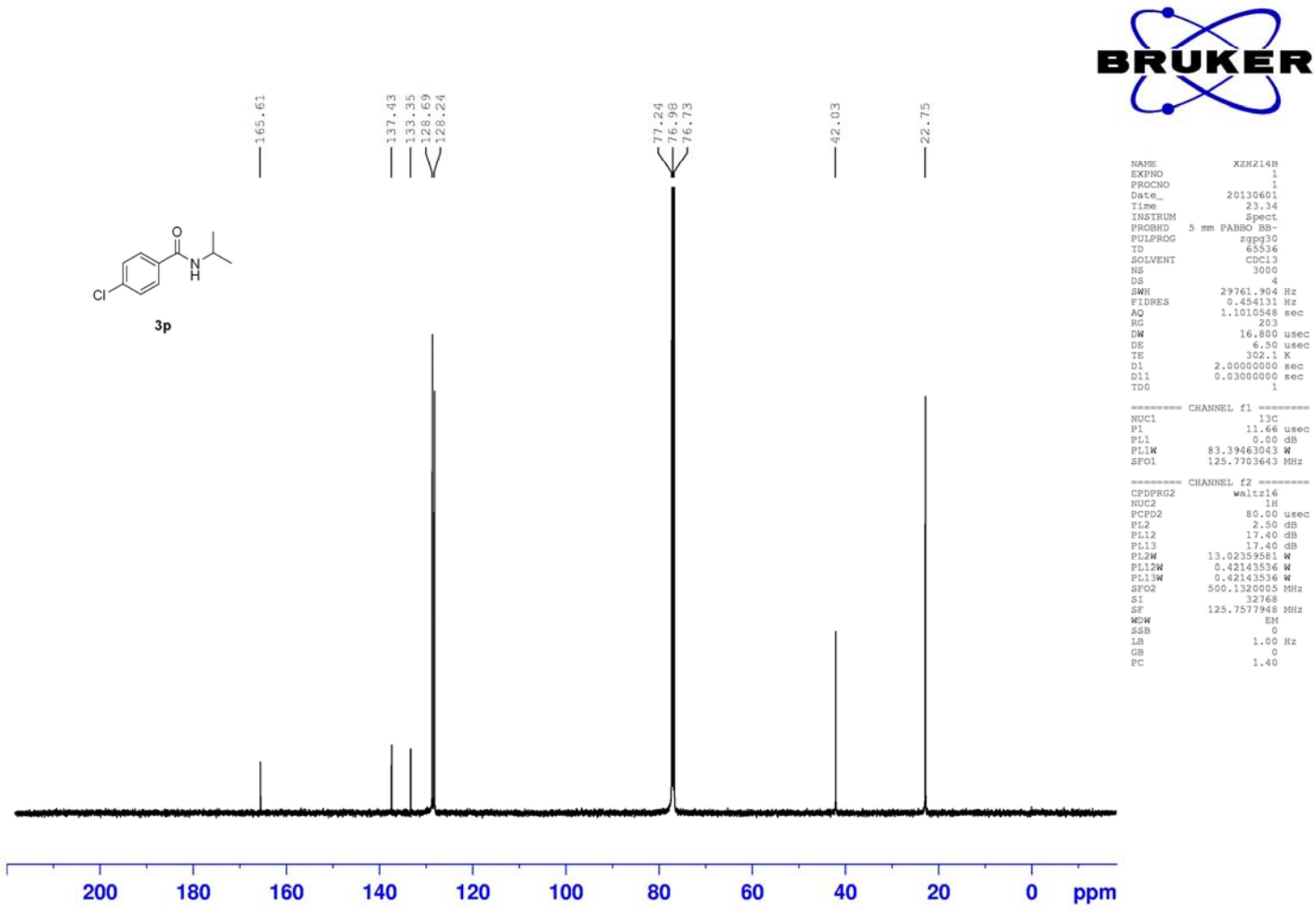
======== CHANNEL f1 ========
NUC1 13C
P1 11.66 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz

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

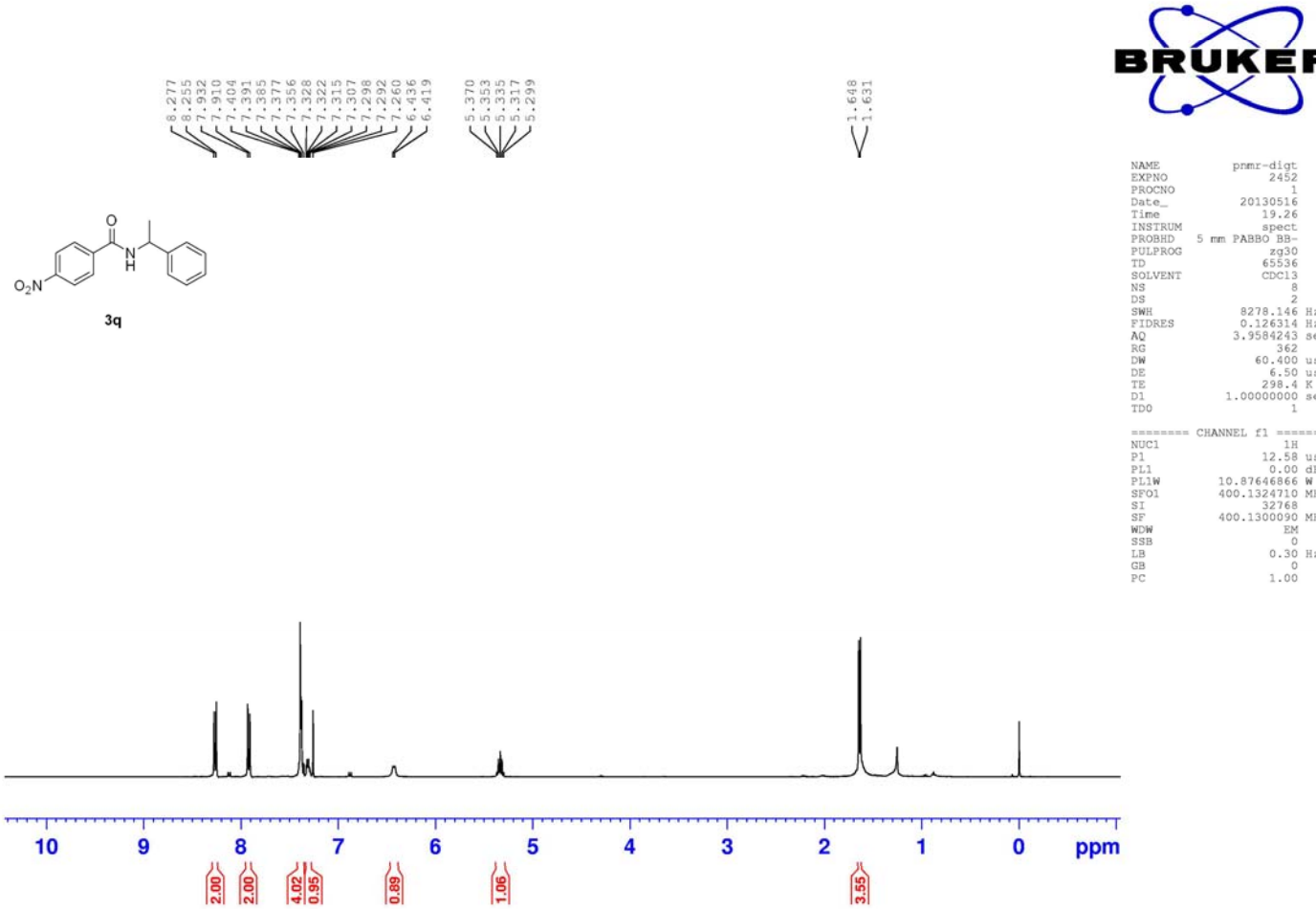


BRUKER

NAME	nmr
EXPNO	2142
PROCNO	1
Date_	20130311
Time	16.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DW	60.400 usec
DE	6.50 usec
TE	297.4 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	XZH214B
EXPNO	1
PROCNO	1
Date_	20130601
Time	23.34
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3000
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	302.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577948 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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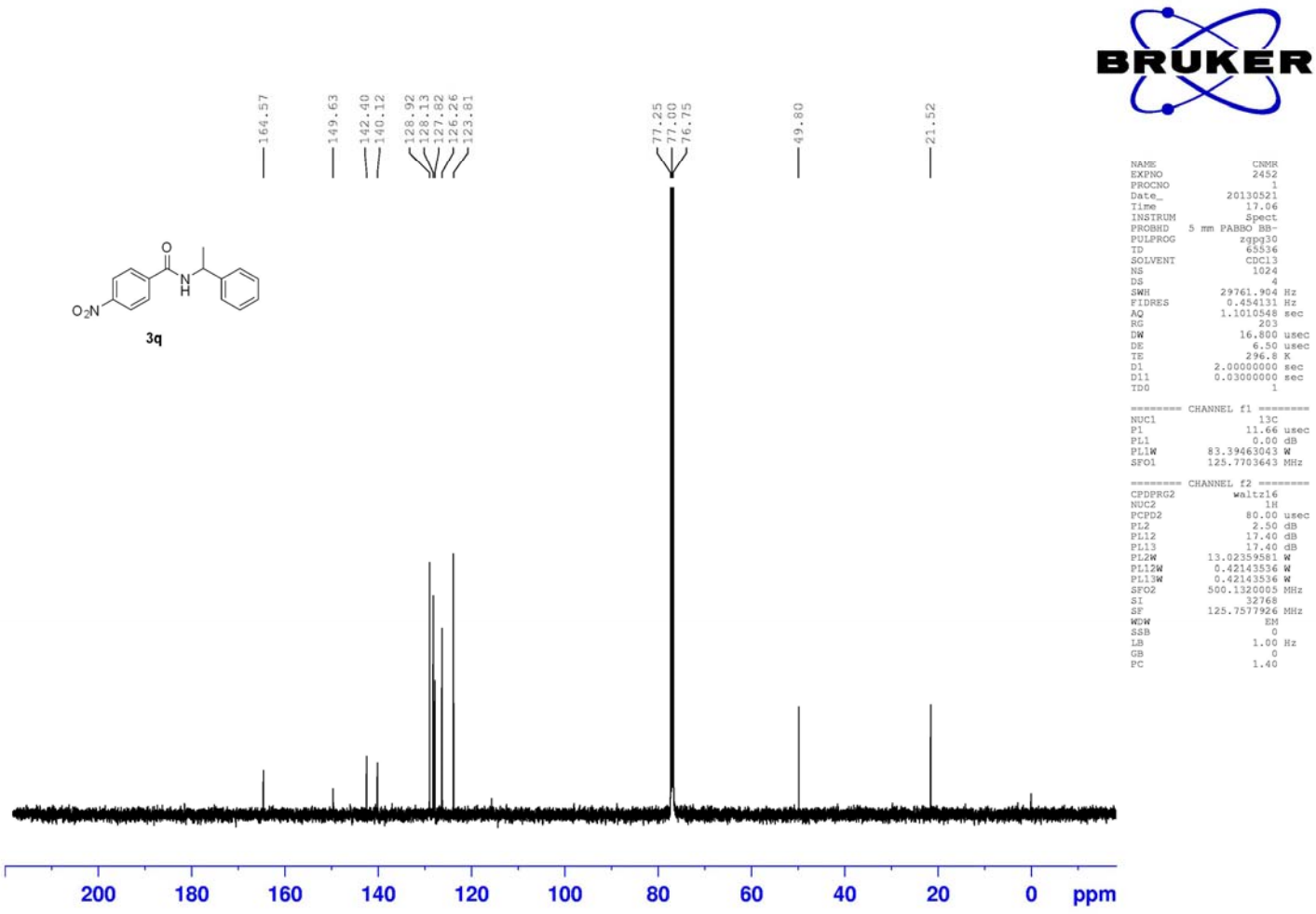
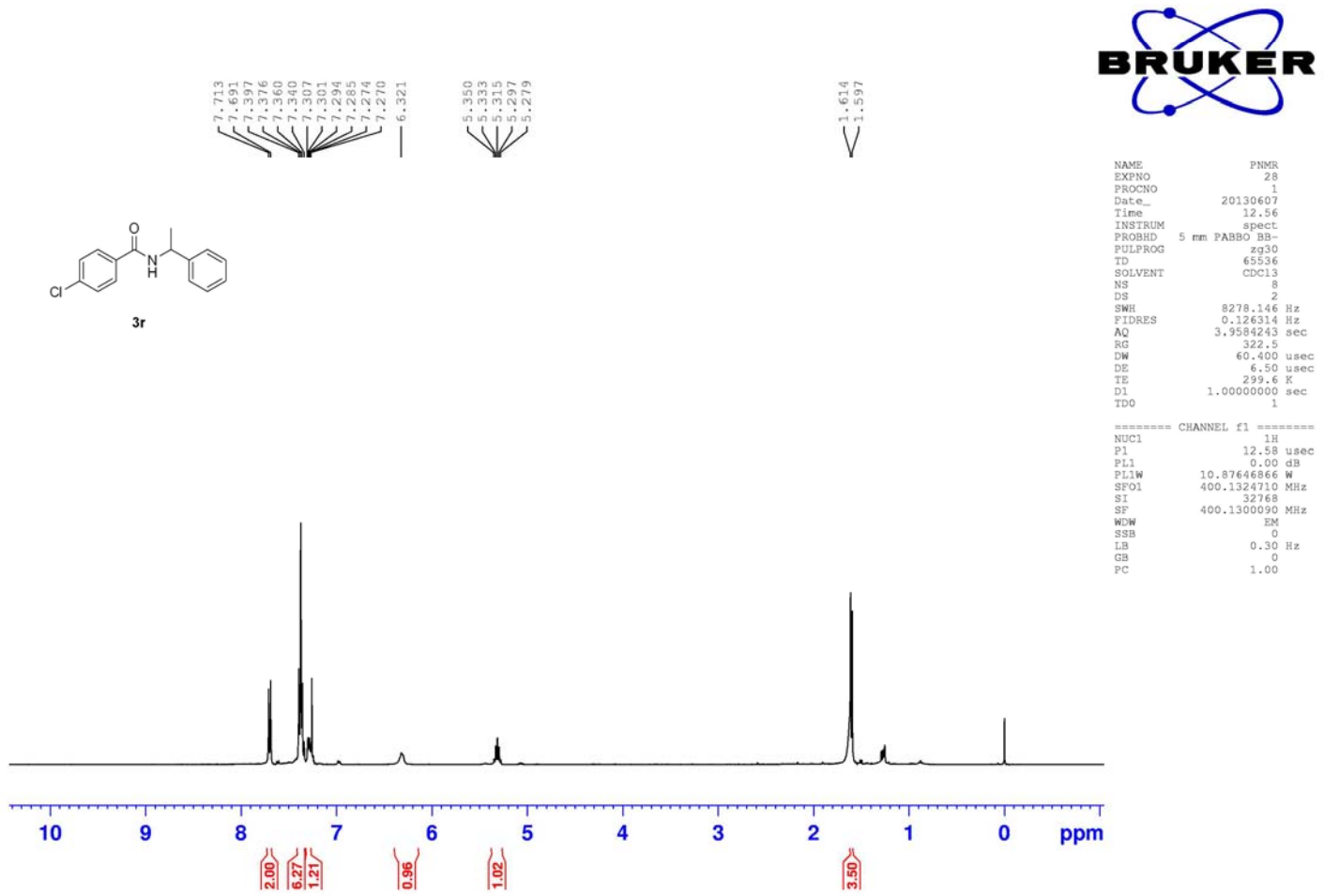
8.255
7.932
7.910
7.404
7.391
7.385
7.377
7.356
7.328
7.322
7.315
7.307
7.298
7.292
7.260
6.436
6.419
5.370
5.353
5.335
5.317
5.299
1.648
1.631
O
N
H
O_2N
3q
BRUKER
NAME pnmr-digt
EXPNO 2452
PROCNO 1
Date_ 20130516
Time 19.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.00
4.02
0.95
0.89
1.06
3.55

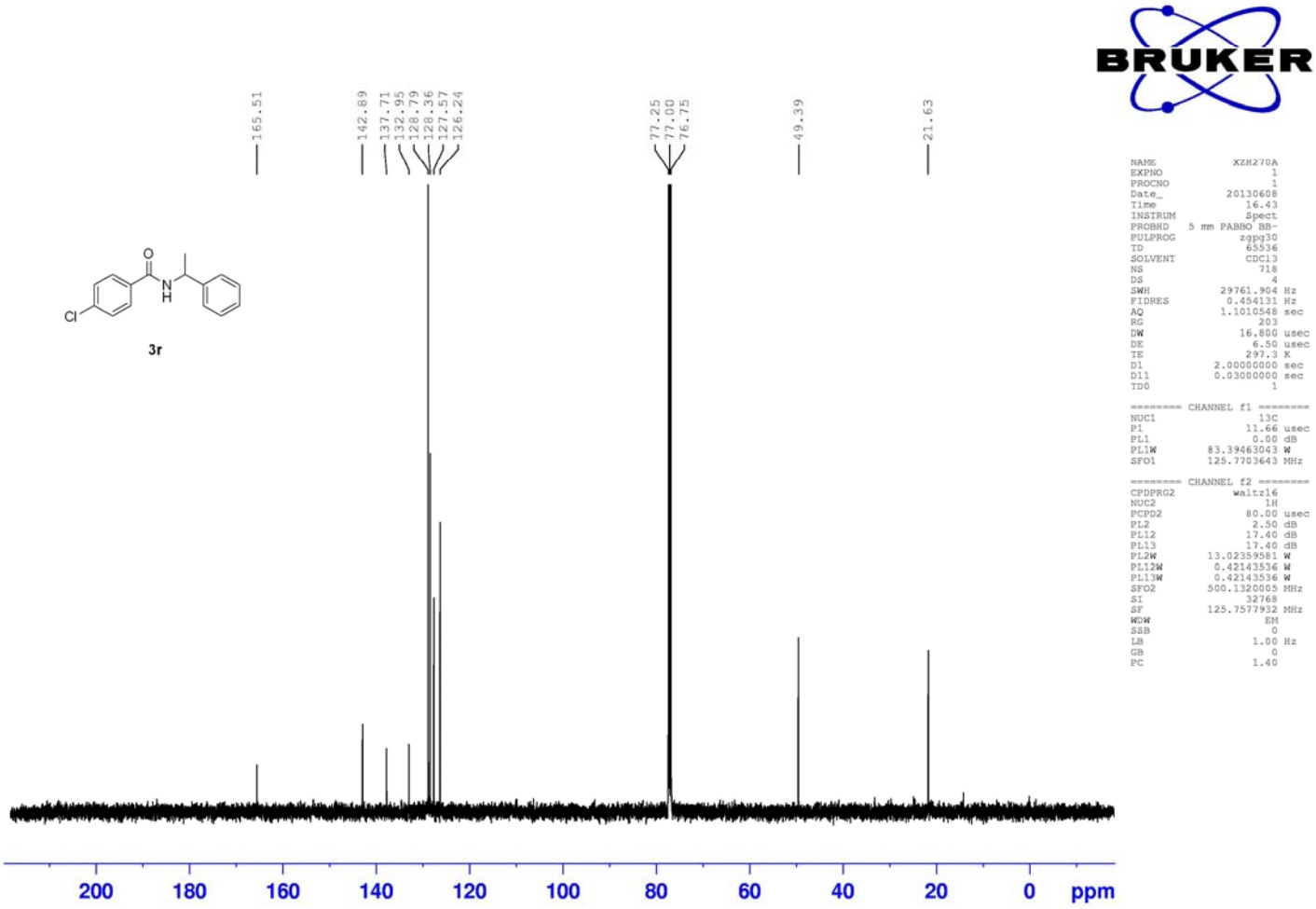


BRUKER

NAME	CNMR
EXPNO	2452
PROCNO	1
Date_	20130521
Time	17.06
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577926 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

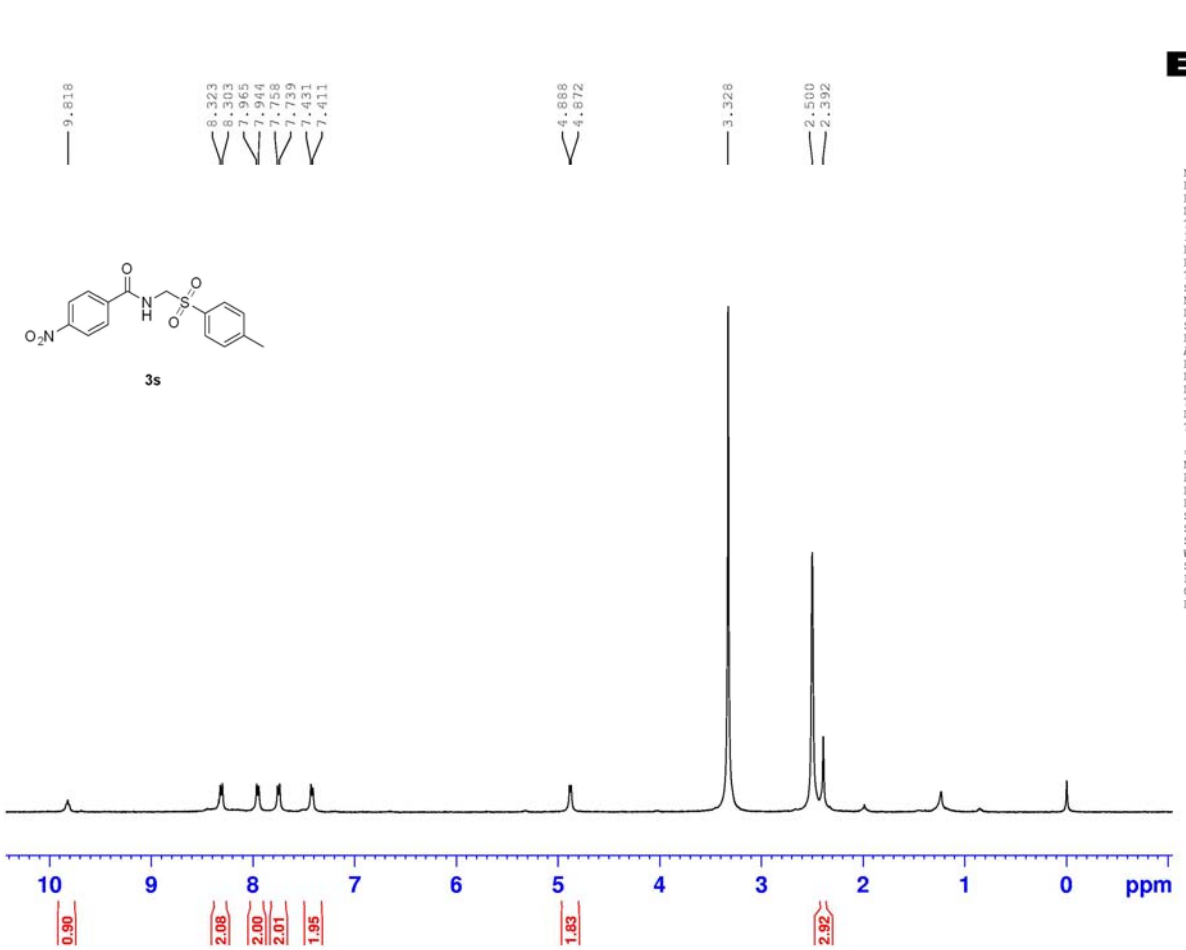


NAME	PNMR
EXPNO	28
PROCNO	1
Date_	20130607
Time	12.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	299.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



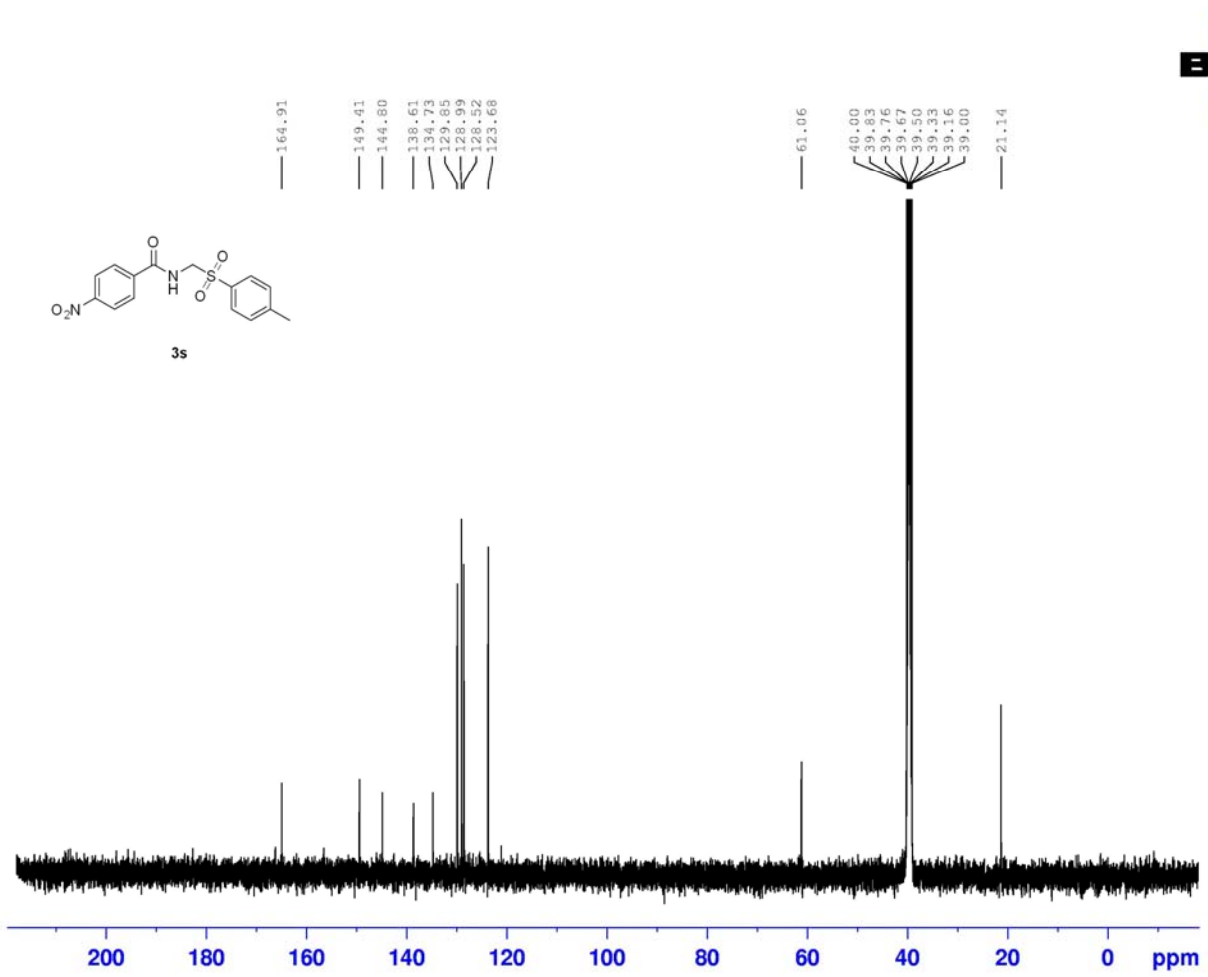
BRUKER

NAME	XZH270A
EXPNO	1
PROCNO	1
Date_	20130608
Time	16.43
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	718
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	297.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577932 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

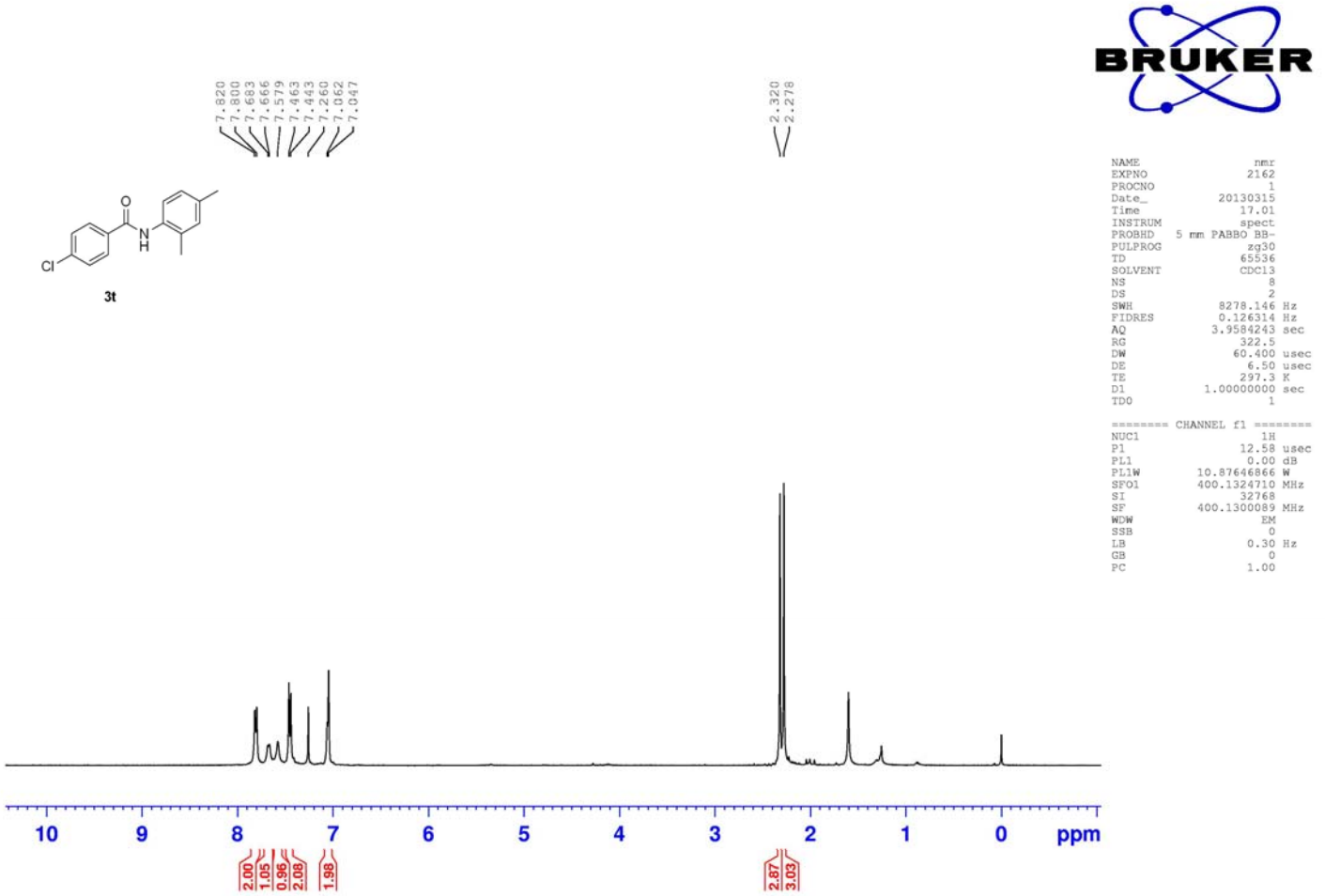
NAME	pnmr-digt
EXPNO	2192
PROCNO	1
Date_	20130318
Time	11.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	297.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300021 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00




```
NAME            cnmr-dlgt
EXPNO                2192
PROCNO                  1
Date_            20130329
Time                22.29
INSTRUM             Spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                  65536
SOLVENT              DMSO
NS                   1024
DS                      4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                    203
DW               16.800 usec
DE                6.50 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.66 usec
PL1                0.00 dB
PL1W         83.39463043 W
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W         13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SFO2        500.1320005 MHz
SI                 32768
SF          125.7578459 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```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7.666
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7.260
7.062
7.047
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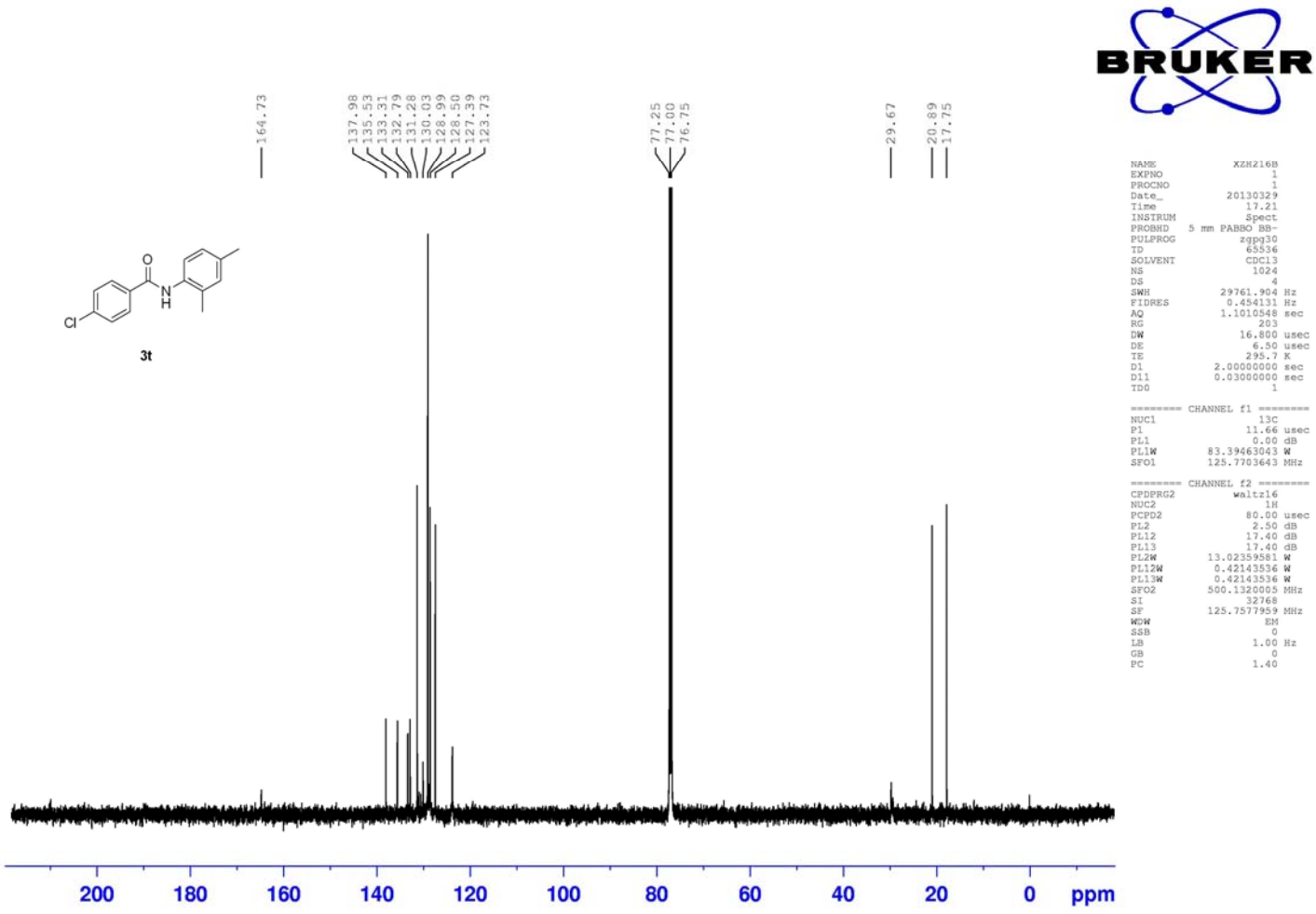
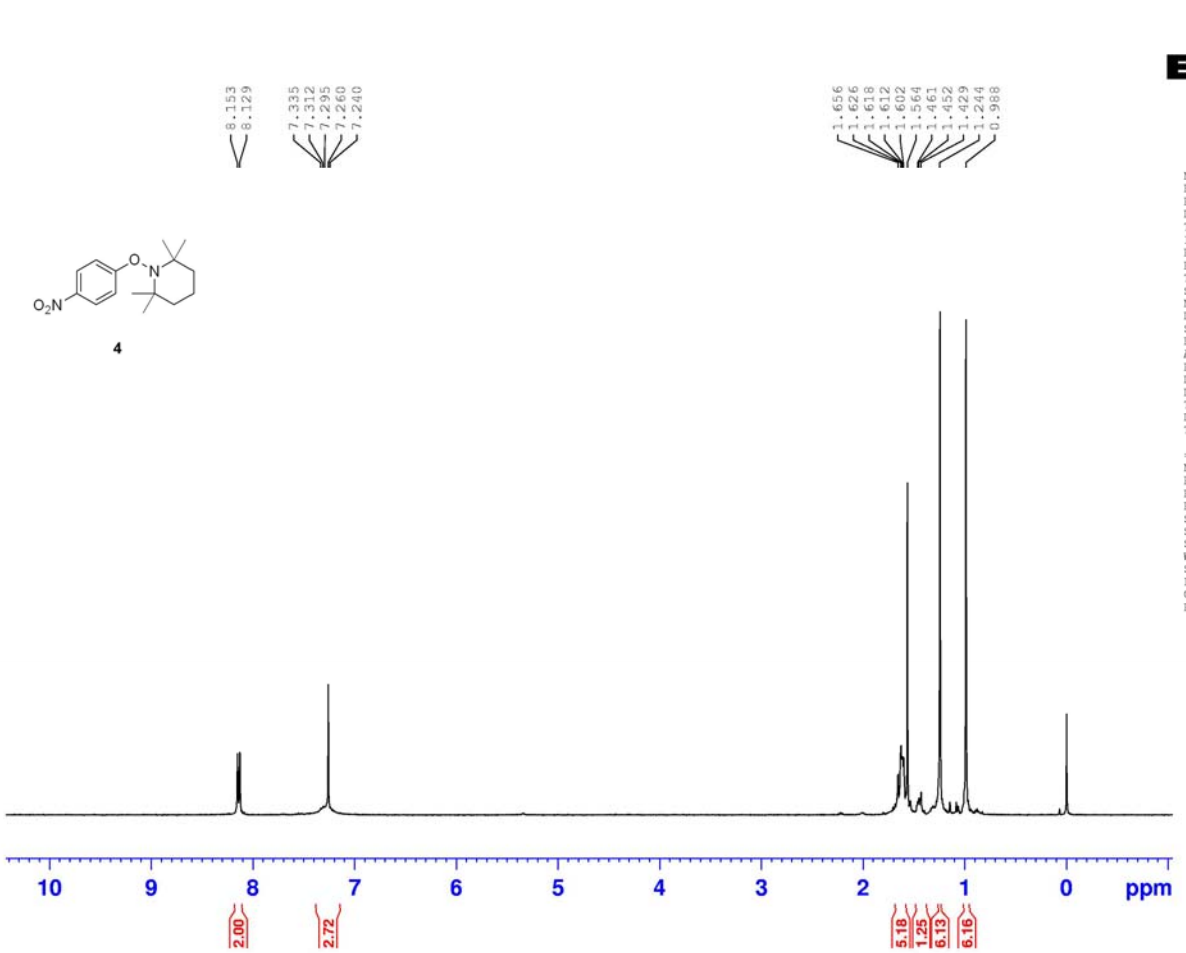
2.278
O
N
H
Cl
3t
BRUKER
NAME nmr
EXPNO 2162
PROCNO 1
Date_ 20130315
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.05
0.96
2.08
1.98
2.87
3.03



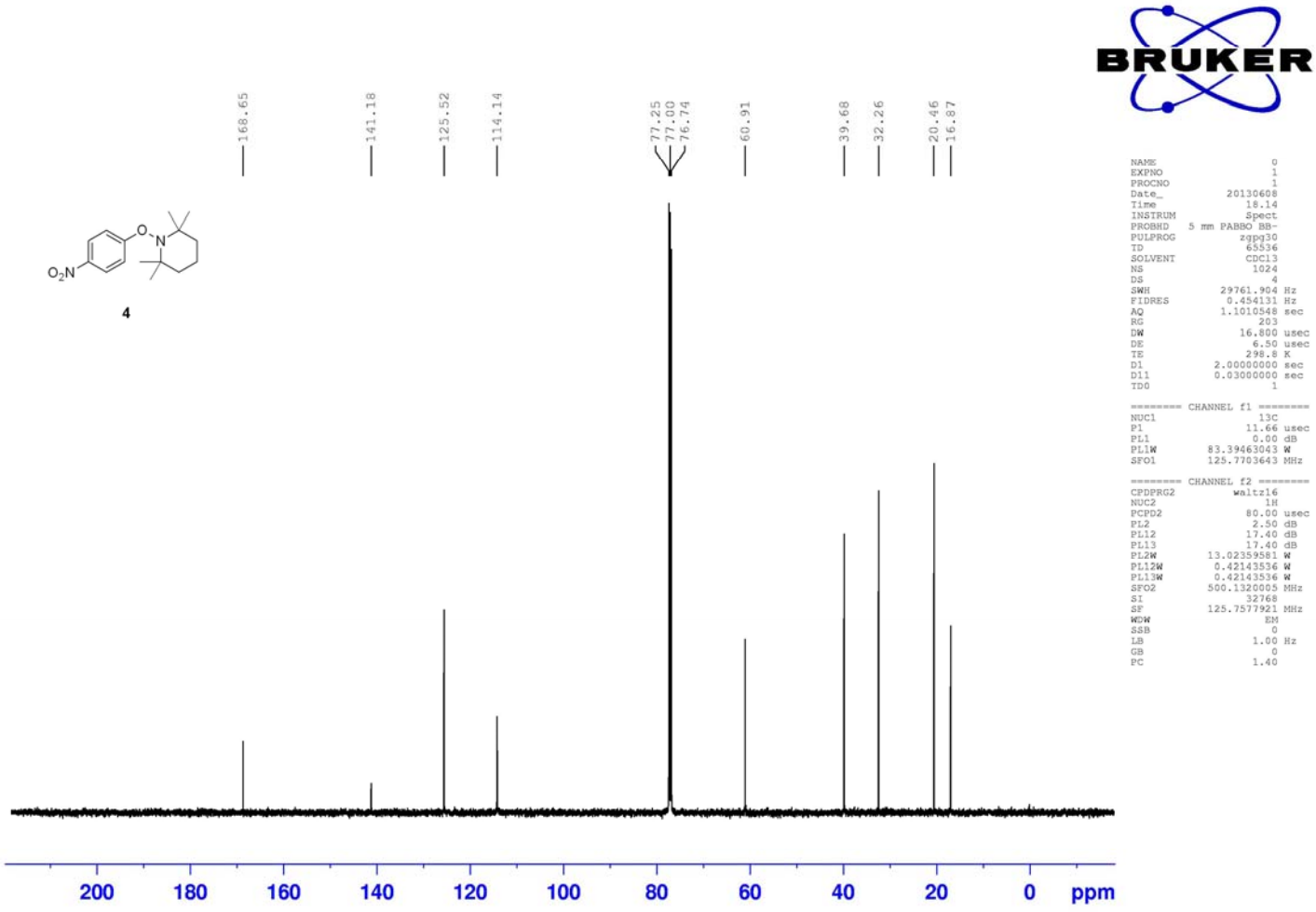
BRUKER

NAME	XZH216B
EXPNO	1
PROCNO	1
Date_	20130329
Time	17.21
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	295.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577959 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	pnmr-digt
EXPNO	2231
PROCNO	1
Date_	20130319
Time	19.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	297.6 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	0
EXPNO	1
PROCNO	1
Date_	20130608
Time	18.14
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577921 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40