

Electronic Supplementary Information (ESI)

A Metal-Free Synthesis of 2-Aminobenzothiazoles through Aminyl Radical Addition to Aryl Isothiocyanates

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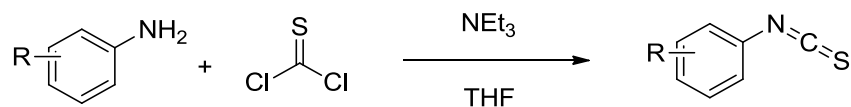
1. General Information

Reactions were monitored by using thin-layer chromatography (TLC) on commercial silica gel plates (GF 254). Visualization of the developed plates was performed under UV lights (GF 254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^1H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to the CDCl_3 residual peak (δ 7.26) or the DMSO-d_6 residual peak (δ 2.50) for ^1H NMR. Chemical shifts of ^{13}C NMR were reported relative to CDCl_3 (δ 77.0) or $\text{D}_6\text{-DMSO}$ (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 constant, J , was reported in Hertz unit (Hz). Melting points (mp) were taken on a MEL-TEMP® apparatus and were uncorrected. High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

Caution: Thiophosgene and thioisocyanates are toxic, and we must handle carefully in a fume hood.

2. Preparation of Aryl Isothiocyanates

All aryl isothiocyanates were prepared according to the reported method.¹ Analytical data of aryl isothiocyanates are in agreement with those reported in the literature.¹

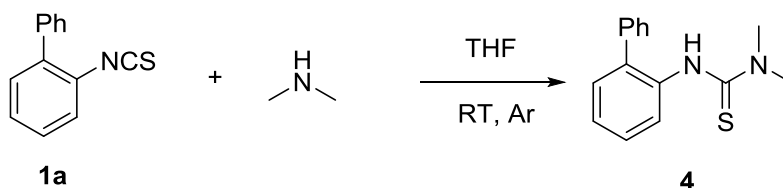


Thiophosgene (47 μL , 0.6 mmol) was added dropwise to a stirring suspension of the aromatic amine (0.5 mmol, 1.0 equiv) and triethylamine (170 μL , 1.2 mmol) in THF (3 mL) at 0 $^{\circ}\text{C}$ over a period of 30 minutes under argon. The resulting solution was allowed to warm to room temperature and stirred for 2 hours. The reaction mixture was concentrated to dryness and the residue was dissolved in EtOAc, washed with saturated aqueous solution of NaHCO_3 , dried over Na_2SO_4 , and concentrated under reduced pressure. The residue was separated by column chromatography

(petroleum ether) to get the pure product.

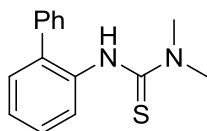
3. Preparation and Characterization of 4

3-([1,1'-biphenyl]-2-yl)-1,1-dimethylthiourea **4** was prepared according to the reported method.²



1a (106 mg, 0.5 mmol) was added dropwise to a stirred suspension of dimethylamine (68 μ L, 0.6 mmol, 40 wt% in water) in THF (5 mL) at 25 $^{\circ}$ C over a period of 30 minutes under argon. The resulting solution stirred for 1 hour. The reaction mixture was concentrated to dryness. The residue was dissolved in EtOAc, washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure. The residue was purified by column chromatography (petroleum ether/EtOAc = 2:1) to get the pure **4**.

3-([1,1'-biphenyl]-2-yl)-1,1-dimethylthiourea **4**

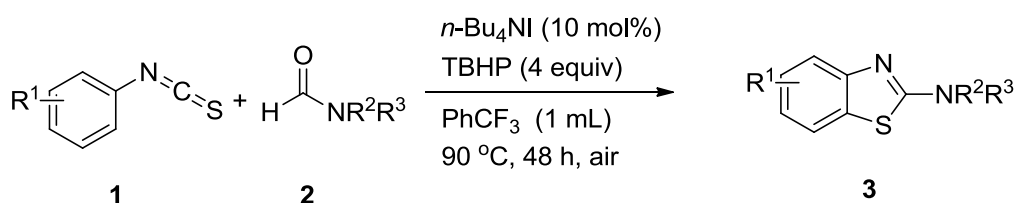


105 mg, 82% yield; mp 131 $^{\circ}$ C; white solid; ¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.28-7.45 (m, 8H), 6.80 (s, 1H), 3.19 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 182.3, 138.5, 137.0, 136.0, 130.1, 129.2, 128.7, 127.8, 127.1, 125.9, 41.1; IR (KBr/cm⁻¹): ν 3216, 3057, 2917, 1599, 1582, 1528, 1470, 1452, 1435, 1371, 1338, 864, 840, 767; HRMS (ESI): Exact mass calcd for C₁₅H₁₆N₂S [M+H]⁺, 257.1107; Found: 257.1108.

4. General Procedures and Product Characterization

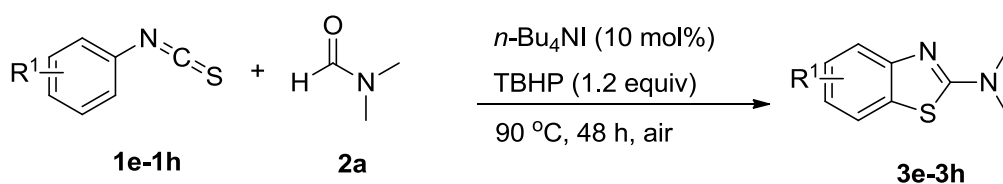
4.1 General Procedures

Procedure A:



To a solution of aryl isothiocyanates **1** (0.2 mmol), formamides **2** (2 mmol) and *n*-Bu₄NI (7mg, 10 mol%) in trifluoromethylbenzene (1.0 mL) was added 1.2 equiv of TBHP (44 μL, 5.5 M in decane). The reaction was stirred at 90 °C for 6 h in air, then another 2.8 equiv of TBHP (103 μL, 5.5 M in decane) was added in 7 portions during 42 h at the same temperature. The solution was then cooled to r.t., diluted with ethyl acetate (5 mL), and washed with brine. The aqueous layers were extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 16:1) to afford the desired product **3**.

Procedure B:

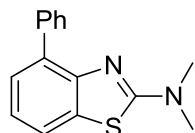


To a solution of arylisothiocyanates **1e-1h** (0.2 mmol), *n*-Bu₄NI (7 mg, 10 mol%) in formamide **2a** (1 mL) was added TBHP (44 μL, 5.5 M in decane). The reaction was stirred for 48 h at 90 °C in air. The reaction was monitored by TLC. The solution was then cooled to r.t., diluted with ethyl acetate (5 mL), washed with brine. The aqueous layers was extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over Na₂SO₄, filtered, and evaporated under vacuum. The residue was purified

bycolumn chromatography on silica gel (petroleum ether/ethyl acetate = 16:1) to afford the desired product **3e-3h**.

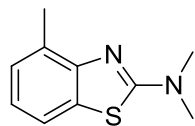
4.2 Product Characterization

N, N-dimethyl-4-phenylbenzo[*d*]thiazol-2-amine (**3a**)



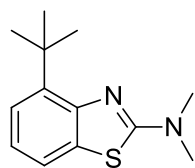
Following the general procedure A, 38 mg, 75% yield; yellow solid; mp 171-173 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.41-7.46 (m, 3H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 3.19 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 168.0, 150.7, 139.7, 132.5, 131.5, 129.4, 127.8, 126.8, 126.1, 121.0, 119.7, 39.9; IR (KBr/cm⁻¹): ν 3361, 3028, 2922, 2853, 1603, 1575, 1547, 1495, 1460, 1423, 1400, 787, 751, 709; HRMS (ESI): Exact mass calcd for C₁₅H₁₄N₂S [M+H]⁺, 255.0951; Found: 255.0948.

N, N, 4-trimethylbenzo[*d*]thiazol-2-amine (**3b**)



Following the general procedure A, 29 mg, 75% yield; white solid; mp 64-66 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.43 (d, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 7.2 Hz, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 3.20 (s, 6H), 2.58 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 167.8, 152.3, 131.0, 128.6, 126.5, 120.6, 117.9, 39.9, 18.2; IR (KBr/cm⁻¹): ν 3372, 3196, 2970, 2922, 1596, 1544, 1472, 1455, 1222, 743, 697; HRMS (ESI): Exact mass calcd for C₁₀H₁₂N₂S [M+H]⁺, 193.0794; Found: 193.0791.

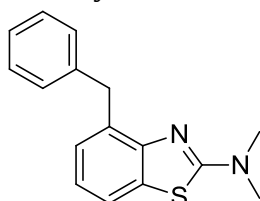
4-(*tert*-butyl)-*N, N*-dimethylbenzo[*d*]thiazol-2-amine (**3c**)



Following the general procedure A, 38 mg, 82% yield; white solid; mp 122-124 °C;

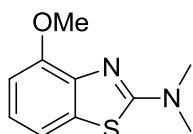
^1H NMR (400 MHz, CDCl_3): δ 7.49 (d, J = 8.0 Hz, 1H), 7.23 (d, J = 7.6 Hz, 1H), 6.99 (t, J = 7.6 Hz, 1H), 3.20 (s, 6H), 1.55 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.0, 150.9, 141.0, 132.6, 122.2, 120.5, 118.5, 39.9, 35.6, 29.7; IR (KBr/ cm^{-1}): ν 3404, 3052, 2950, 2932, 1597, 1517, 1470, 1455, 1401, 771, 745; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 235.1264; Found: 235.1259.

4-benzyl-*N,N*-dimethylbenzo[*d*]thiazol-2-amine (3d)



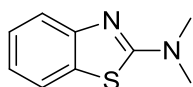
Following the general procedure A, 38 mg, 71% yield; white solid; mp 83-85 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.45 (d, J = 8.0 Hz, 1H), 7.36 (d, J = 7.6 Hz, 2H), 7.27 (d, J = 10.0 Hz, 1H), 7.25 (d, J = 4.8 Hz, 1H), 7.17 (t, J = 7.2 Hz, 1H), 7.04 (d, J = 7.2 Hz, 1H), 6.96 (t, J = 8.0 Hz, 1H), 4.34 (s, 2H), 3.21 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 167.8, 152.1, 141.6, 131.8, 131.3, 129.2, 128.1, 126.0, 125.7, 120.8, 118.5, 40.0, 38.3; IR (KBr/ cm^{-1}): ν 3396, 3057, 3022, 2919, 1595, 1579, 1554, 1492, 1468, 1451, 1434, 1416, 752, 741, 701; HRMS (ESI): Exact mass calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 269.1107; Found: 269.1108.

4-methoxy-*N,N*-dimethylbenzo[*d*]thiazol-2-amine (3e)



Following the general procedure B, 17 mg, 41% yield; yellow solid; mp 123-125 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.21 (d, J = 7.6 Hz, 1H), 7.02 (t, J = 8.0 Hz, 1H), 6.81 (d, J = 8.0 Hz, 1H), 3.98 (s, 3H), 3.22 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 168.1, 150.4, 142.7, 132.1, 121.4, 113.0, 107.0, 55.8, 40.3; IR (KBr/ cm^{-1}): ν 3415, 3060, 2920, 2837, 1597, 1564, 1476, 1441, 1418, 1403, 737, 706; HRMS (ESI): Exact mass calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{OS}$ $[\text{M}+\text{H}]^+$, 209.0743; Found: 209.0741.

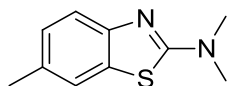
***N,N*-dimethylbenzo[*d*]thiazol-2-amine (3f) ³**



Following the general procedure B, 15 mg, 42% yield; white solid; mp 84-86 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 8.0 Hz, 1H), 7.28 (t,

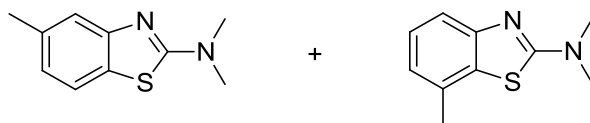
$J = 8.0$ Hz, 1H), 7.05 (t, $J = 7.6$ Hz, 1H), 3.21 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 153.6, 147.9, 128.9, 128.0, 125.0, 122.9, 118.4, 38.9; IR (KBr/ cm^{-1}): ν 3362, 3051, 2920, 2852, 1600, 1564, 1547, 1451, 1443, 1421, 753; HRMS (ESI): Exact mass calcd for $\text{C}_9\text{H}_{10}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 179.0565; Found: 179.0564.

***N, N*, 6-trimethylbenzo[d]thiazol-2-amine (3g)**



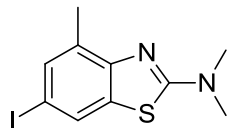
Following the general procedure B, 20 mg, 52% yield; yellow solid; mp 78-80 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.45 (d, $J = 8.4$ Hz, 1H), 7.40 (s, 1H), 7.09 (d, $J = 8.0$ Hz, 1H), 3.19 (s, 6H), 2.39 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 168.2, 151.1, 131.1, 130.5, 127.0, 120.6, 118.4, 40.1, 21.1; IR (KBr/ cm^{-1}): ν 3042, 3015, 2920, 2854, 1608, 1577, 1549, 1468, 821; HRMS (ESI): Exact mass calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 193.0752; Found: 193.0751.

***N, N*, 5-trimethylbenzo[d]thiazol-2-amine (3h) and *N, N*, 7-trimethylbenzo[d]thiazol-2-amine (3h')**



Following the general procedure B, 22 mg, 56% yield; **3h** and **3h'** were obtained as a inseperable mixture, **3h** : **3h'** = 1:1; gray solid; ^1H NMR (400 MHz, CDCl_3): δ 7.45 (d, $J = 8.0$ Hz, 1H), 7.40 (t, $J = 8.4$ Hz, 2H), 7.21 (t, $J = 7.6$ Hz, 1H), 6.87 (t, $J = 7.2$ Hz, 2H), 3.21 (s, 6H), 3.19 (s, 6H), 2.45 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 169.0, 168.4, 153.5, 153.1, 135.8, 131.4, 130.6, 127.9, 125.9, 122.1, 121.4, 120.1, 119.3, 116.2, 40.2, 40.1, 21.5, 21.3; IR (KBr/ cm^{-1}): ν 3386, 2963, 2920, 2855, 1599, 1559, 1448, 1403, 800, 784, 775.

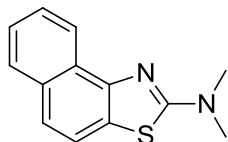
6-iodo-*N, N*, 4-trimethylbenzo[d]thiazol-2-amine (3i)



Following the general procedure A, 39 mg, 62% yield; white solid; mp 127-129 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.71 (s, 1H), 7.40 (s, 1H), 3.19 (s, 6H), 2.50 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 167.8, 152.0, 135.2, 132.8, 130.7, 126.2, 82.6, 40.0, 17.9; IR (KBr/ cm^{-1}): ν 3391, 3052, 2973, 2922, 1593, 1521, 1468, 1455, 1401, 766,

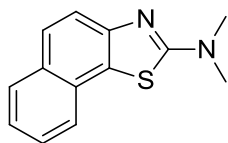
743; HRMS (ESI): Exact mass calcd for C₁₀H₁₁IN₂S [M+H]⁺, 318.9761; Found: 318.9761.

***N,N*-dimethylnaphtho[1,2-*d*]thiazol-2-amine (3j)**



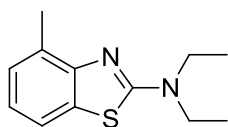
Following the general procedure A, 26 mg, 57% yield; gray solid; mp 115-117 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.61 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.52-7.56 (m, 2H), 7.47 (t, *J* = 7.2 Hz, 1H), 3.28 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 169.3, 149.0, 132.2, 127.8, 126.8, 125.6, 125.5, 125.2, 124.0, 120.9, 118.6, 40.1; IR (KBr/cm⁻¹): ν 3362, 3042, 2920, 2853, 1621, 1589, 1561, 1502, 1423, 1405, 781, 677; HRMS (ESI): Exact mass calcd for C₁₃H₁₂N₂S [M+H]⁺, 229.0743; Found: 229.0746.

***N,N*-dimethylnaphtho[2,1-*d*]thiazol-2-amine (3k)**



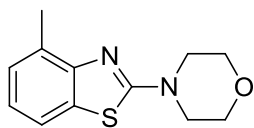
Following the general procedure A, 30 mg, 65% yield; gray solid; mp 127-129 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.87 (d, *J* = 8.4 Hz, 1H), 7.77-7.74 (m, 3H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 3.27 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 169.1, 151.1, 129.2, 128.9, 128.2, 126.6, 126.5, 125.3, 123.7, 123.6, 119.6, 40.3; IR (KBr/cm⁻¹): ν 3385, 3045, 2920, 1591, 1571, 1546, 1505, 1452, 1422, 1405, 813, 750; HRMS (ESI): Exact mass calcd for C₁₃H₁₂N₂S [M+H]⁺, 229.0794; Found: 229.0793.

***N,N*-diethyl-4-methylbenzo[*d*]thiazol-2-amine (3l)**



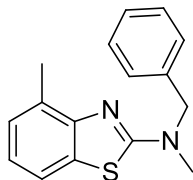
Following the general procedure A, 39 mg, 89% yield; yellow oil; ¹H NMR (400 MHz, CDCl₃): δ 7.42 (d, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 7.2 Hz, 1H), 6.93 (t, *J* = 7.6 Hz, 1H), 3.59 (t, *J* = 7.2 Hz, 4H), 2.56 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 166.2, 152.3, 130.4, 128.4, 126.4, 120.4, 117.9, 45.2, 18.2, 12.8; IR (KBr/cm⁻¹): ν 3422, 3067, 2972, 2930, 1593, 1513, 1468, 1455, 1404, 765, 741; HRMS (ESI): Exact mass calcd for C₁₂H₁₆N₂S [M+H]⁺, 221.1107; Found: 221.1105.

4-(4-methylbenzo[d]thiazol-2-yl)morpholine (3m)



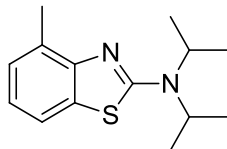
Following the general procedure A, 41 mg, 88% yield; white solid; mp 143-146 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.46 (d, J = 8.0 Hz, 1H), 7.12 (d, J = 7.6 Hz, 1H), 7.00 (t, J = 7.6 Hz, 1H), 3.84 (t, J = 4.8 Hz, 4H), 3.63 (t, J = 5.2 Hz, 4H), 2.56 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 168.0, 151.6, 130.4, 129.3, 126.7, 121.5, 118.1, 66.3, 48.5, 18.2; IR (KBr/ cm^{-1}): ν 2971, 2919, 2850, 1590, 1541, 1474, 1448, 1433, 1412, 700, 673; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{OS}$ $[\text{M}+\text{H}]^+$, 235.0900; Found: 235.0897.

N-benzyl-*N*, 4-dimethylbenzo[d]thiazol-2-amine (3n)



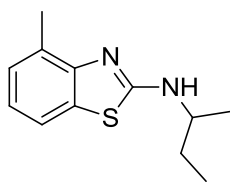
Following the general procedure A, 28 mg, 52% yield; yellow solid; mp 68-70 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.44 (d, J = 7.6 Hz, 1H), 7.29-7.35 (m, 5H), 7.12 (d, J = 7.6 Hz, 1H), 6.98 (t, J = 7.2 Hz, 1H), 4.81 (s, 2H), 3.14 (s, 3H), 2.59 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 167.8, 152.2, 136.8, 130.8, 128.7, 128.6, 127.8, 127.6, 126.6, 120.8, 118.0, 56.2, 37.7, 18.3; IR (KBr/ cm^{-1}): ν 3424, 3027, 3005, 2942, 2918, 1597, 1554, 1494, 1470, 1453, 1437, 1408, 1222, 753, 744, 703; HRMS (ESI): Exact mass calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 269.1107; Found: 269.1105.

N, *N*-diisopropyl-4-methylbenzo[d]thiazol-2-amine (3o)



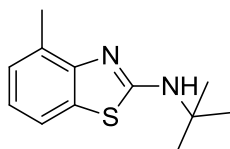
Following the general procedure A, 17 mg, 35% yield; gray solid; mp 44-46 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.42 (d, J = 7.6 Hz, 1H), 7.07 (d, J = 7.2 Hz, 1H), 6.91 (t, J = 7.6 Hz, 1H), 3.84-3.90 (m, 2H), 2.56 (s, 3H), 1.44 (d, J = 6.8 Hz, 12H); ^{13}C NMR (125 MHz, CDCl_3): δ 164.4, 152.5, 129.4, 128.3, 126.1, 120.2, 117.5, 51.1, 20.2, 18.2; IR (KBr/ cm^{-1}): ν 3418, 3066, 2970, 2930, 1590, 1515, 1470, 1453, 1404, 759, 743; HRMS (ESI): Exact mass calcd for $\text{C}_{14}\text{H}_{20}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 249.1420; Found: 249.1420.

***N*-(*sec*-butyl)-4-methylbenzo[*d*]thiazol-2-amine (3p)**



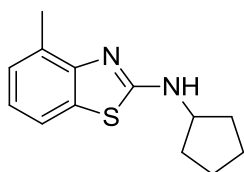
Following the general procedure A, 29 mg, 67% yield; yellow oil; ^1H NMR (400 MHz, CDCl_3): δ 7.43 (d, J = 8.0 Hz, 1H), 7.10 (d, J = 7.2 Hz, 1H), 6.97 (t, J = 8.0 Hz, 1H), 5.23 (s, 1H), 3.57 (s, 1H), 2.55 (s, 3H), 1.62 (t, J = 8.0 Hz, 2H), 1.29 (d, J = 6.4 Hz, 3H), 1.00 (t, J = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.4, 151.3, 130.0, 128.4, 126.7, 121.1, 118.2, 53.7, 30.0, 20.5, 18.3, 10.3; IR (KBr/ cm^{-1}): ν 3385, 3218, 2967, 2928, 1595, 1543, 1457, 1410, 1222, 760, 743; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 221.1107; Found: 221.1106.

***N*-(*tert*-butyl)-4-methylbenzo[*d*]thiazol-2-amine (3q)**



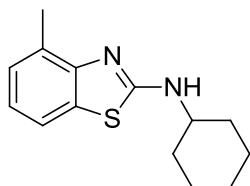
Following the general procedure A, 39 mg, 89% yield; yellow solid; mp 58-60 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.41 (d, J = 7.6 Hz, 1H), 7.09 (d, J = 7.6 Hz, 1H), 6.97 (t, J = 7.6 Hz, 1H), 5.16 (s, 1H), 2.56 (s, 3H), 1.50 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3): δ 163.6, 130.4, 128.7, 126.4, 121.3, 117.9, 53.2, 29.0, 18.3; IR (KBr/ cm^{-1}): ν 3372, 3196, 3016, 2970, 2921, 1596, 1544, 1472, 1455, 1429, 761, 743; HRMS (ESI): Exact mass calcd for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 221.1107; Found: 221.1105.

***N*-cyclopentyl-4-methylbenzo[*d*]thiazol-2-amine (3r)**



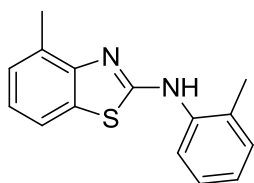
Following the general procedure A, 37 mg, 80% yield; yellow oil; ^1H NMR (400 MHz, CDCl_3): δ 7.45 (d, J = 8.0 Hz, 1H), 7.10 (d, J = 7.6 Hz, 1H), 6.98 (t, J = 7.6 Hz, 1H), 5.40 (s, 1H), 3.94 (s, 1H), 2.55 (s, 3H), 2.16-2.12 (m, 2H), 1.55-1.77 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.6, 151.3, 130.2, 128.5, 126.7, 121.1, 118.2, 57.4, 33.4, 23.7, 18.3; IR (KBr/ cm^{-1}): ν 3384, 3215, 2958, 2870, 1594, 1543, 1471, 1452, 1410, 760, 743; HRMS (ESI): Exact mass calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 233.1107; Found: 233.1105.

***N*-cyclohexyl-4-methylbenzo[*d*]thiazol-2-amine (3s)**



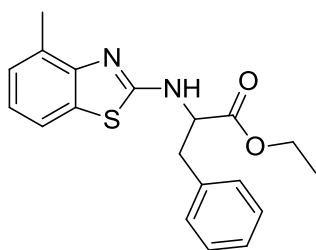
Following the general procedure A, 25 mg, 50% yield; yellow oil; ^1H NMR (400 MHz, CDCl_3): δ 7.43 (d, J = 7.6 Hz, 1H), 7.10 (d, J = 7.2 Hz, 1H), 6.97 (t, J = 8.0 Hz, 1H), 5.30 (d, J = 7.2 Hz, 1H), 3.41-3.44 (m, 1H), 2.55 (s, 3H), 2.12 (d, J = 10.4 Hz, 2H), 1.76-1.80 (m, 2H), 1.26-1.44 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.0, 130.1, 128.4, 126.6, 121.1, 118.2, 54.8, 33.2, 25.4, 24.6, 18.3; IR (KBr/ cm^{-1}): ν 3379, 3218, 2930, 2854, 1593, 1543, 1451, 1409, 759, 743; HRMS (ESI): Exact mass calcd for $\text{C}_{14}\text{H}_{18}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 247.1264; Found: 247.1261.

4-methyl-*N*-(*o*-tolyl)benzo[*d*]thiazol-2-amine (3t)



Following the general procedure A, 25 mg, 50% yield; gray solid; mp 130-132 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 7.71 (d, J = 8.0 Hz, 1H), 7.44 (d, J = 7.6 Hz, 1H), 7.28-7.30 (m, 2H), 7.16 (t, J = 7.6 Hz, 2H), 7.05 (t, J = 7.6 Hz, 1H), 2.60 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 164.9, 150.8, 138.2, 131.1, 131.0, 130.1, 129.1, 127.2, 126.8, 125.7, 122.8, 122.2, 118.3, 18.3, 17.8; IR (KBr/ cm^{-1}): ν 3168, 3118, 3054, 3010, 2922, 2852, 1611, 1594, 1556, 1493, 1454, 762, 755; HRMS (ESI): Exact mass calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{S}$ $[\text{M}+\text{H}]^+$, 255.0951; Found: 255.0949.

ethyl 2-((4-methylbenzo[*d*]thiazol-2-yl)amino)-3-phenylpropanoate (3u)



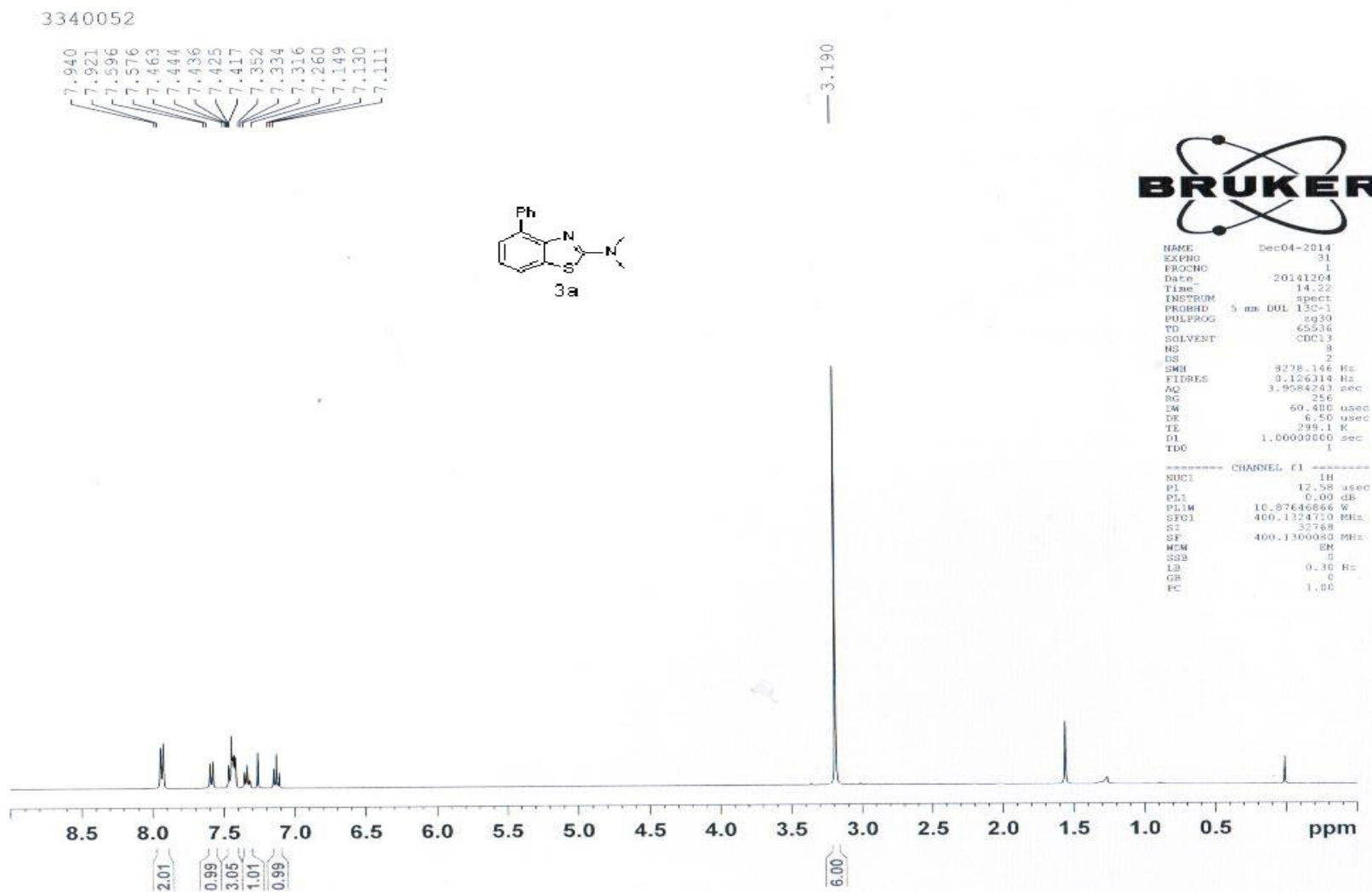
Following the general procedure A, 41 mg, 60% yield; gray solid; mp 83-86 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 7.43 (d, J = 8.0 Hz, 1H), 7.22-7.31 (m, 3H), 7.17 (d, J = 7.2 Hz, 2H), 7.11 (d, J = 7.2 Hz, 1H), 7.01 (t, J = 7.6 Hz, 1H), 5.60 (s, 1H), 4.90 (s, 1H), 4.15-4.23 (m, 2H), 3.53-3.40 (m, 1H), 3.23-3.29 (m, 1H), 2.57 (s, 3H), 1.25 (t, J

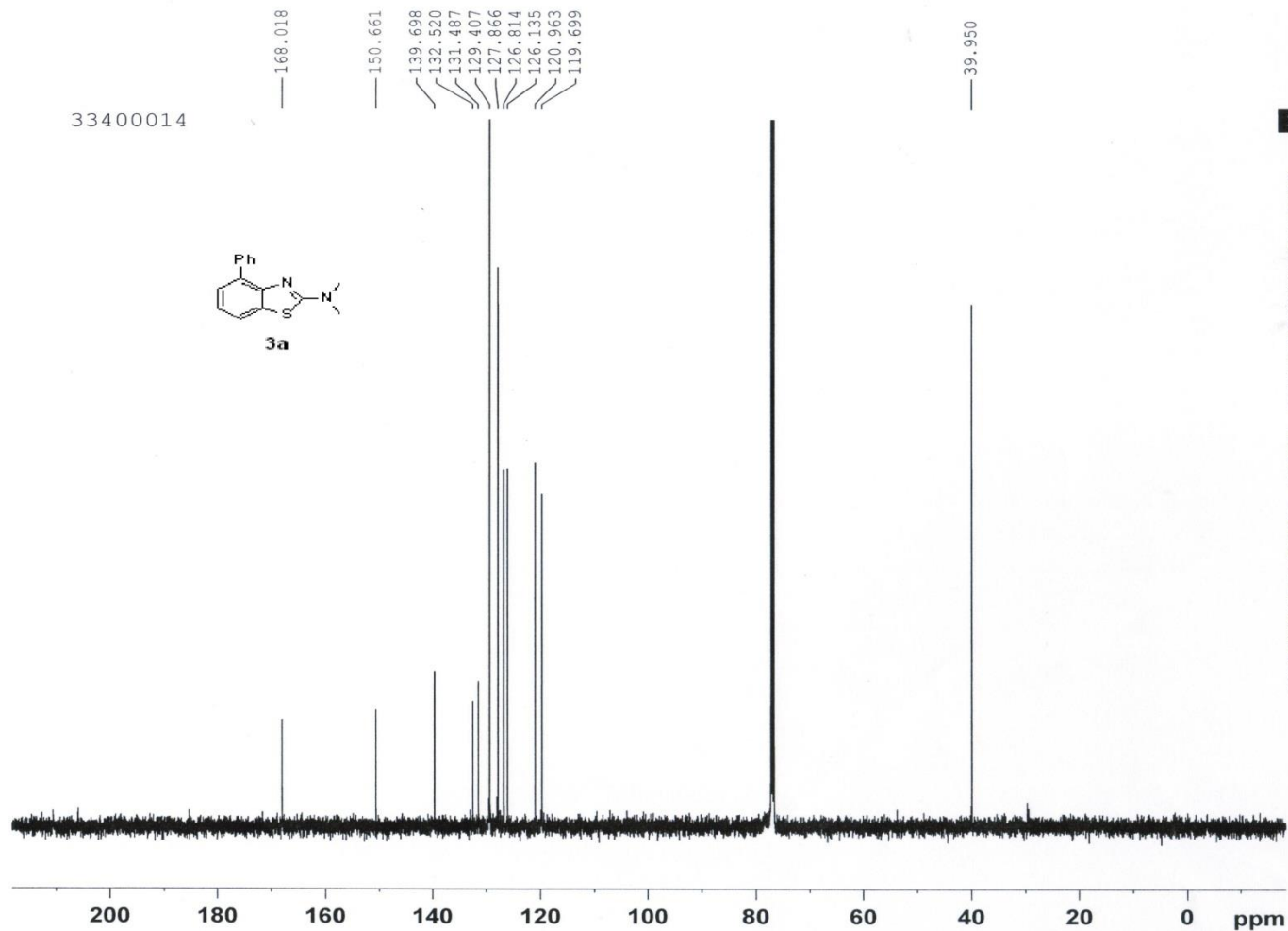
= 6.8 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 171.6, 163.9, 151.3, 135.9, 130.6, 129.4, 129.3, 128.5, 127.1, 126.6, 121.9, 118.2, 61.5, 58.3, 37.8, 18.3, 14.1; IR ($\text{KBr}/\text{cm}^{-1}$): ν 3336, 3023, 2980, 2923, 1734, 1719, 1591, 1542, 1495, 1472, 1454, 1407, 1207, 758, 744, 701; HRMS (ESI): Exact mass calcd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$, 341.1318; Found: 341.1319.

5. References

- 1 (a) S. Degorce, H. F. Jung, C. S. Harris, P. Koza, J. Lecoq and A. Stevenin, *Tetrahedron Lett.*, 2011, **52**, 6719; (b) A. K. Yadav and D. S. Yadav, *Org. Biomol. Chem.*, 2015, **13**, 2606; (c) W. Guo, S. Li, L. Tang, M. Li, L. Wen and C. Chen, *Org. Lett.*, 2015, **17**, 1232.
- 2 K. Biswas and M. F. Greaney, *Org. Lett.*, 2011, **13**, 4946.
- 3 (a) B. K. Lee, M. R. Biscoe and S. L. Buchwald, *Tetrahedron Lett.*, 2009, **50**, 3672; (b) T. P. Petersen, A. F. Larsen, A. Ritzen and T. Ulven, *J. Org. Chem.*, 2013, **78**, 4190; (c) D. Kumar, B. Mishra and V. K. Tiwari, *J. Org. Chem.*, 2014, **79**, 251.

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





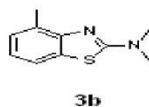
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 PROCNO 1
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 Time 11.02
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 298.6 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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

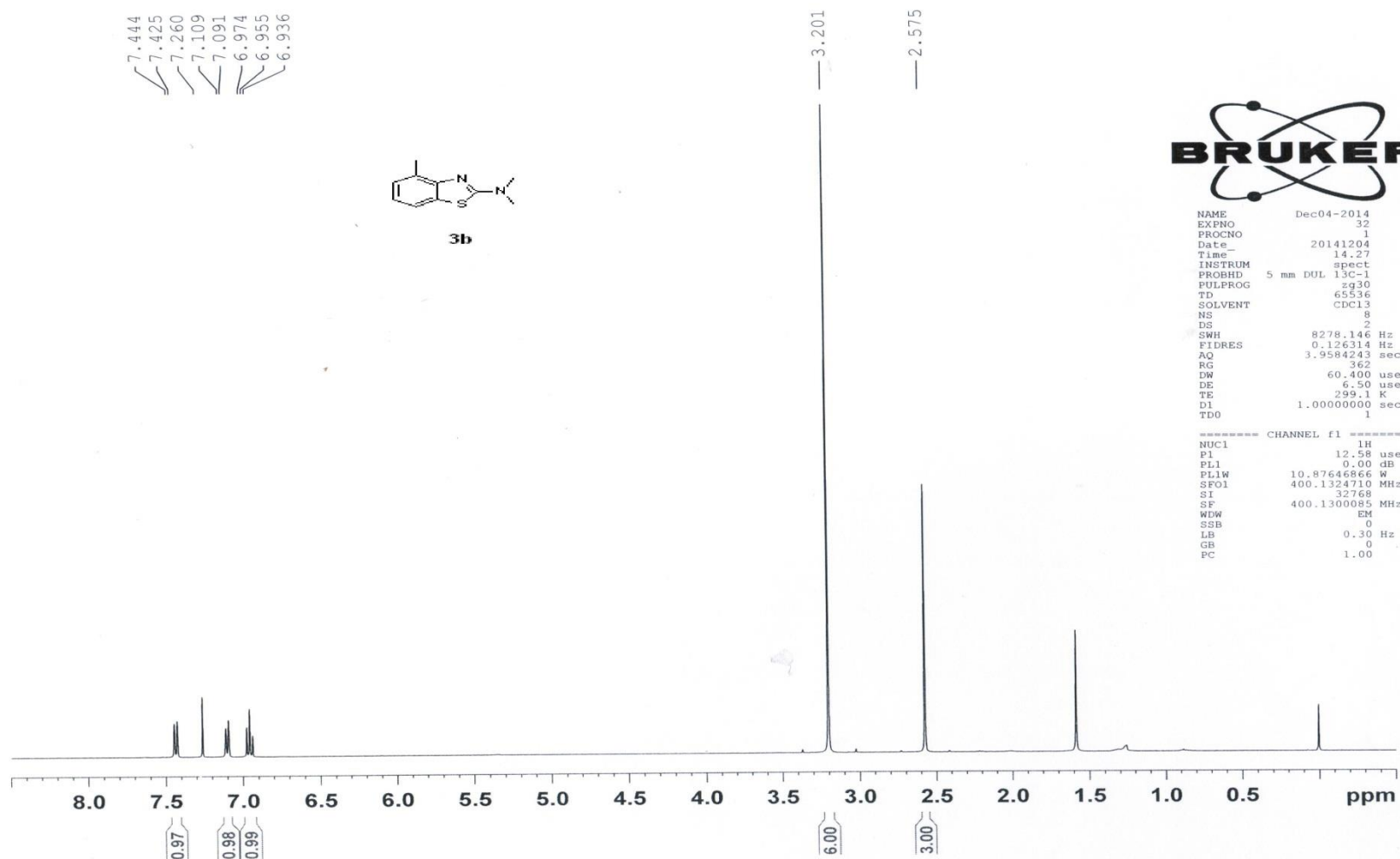
3340060

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7.091
6.974
6.955
6.936

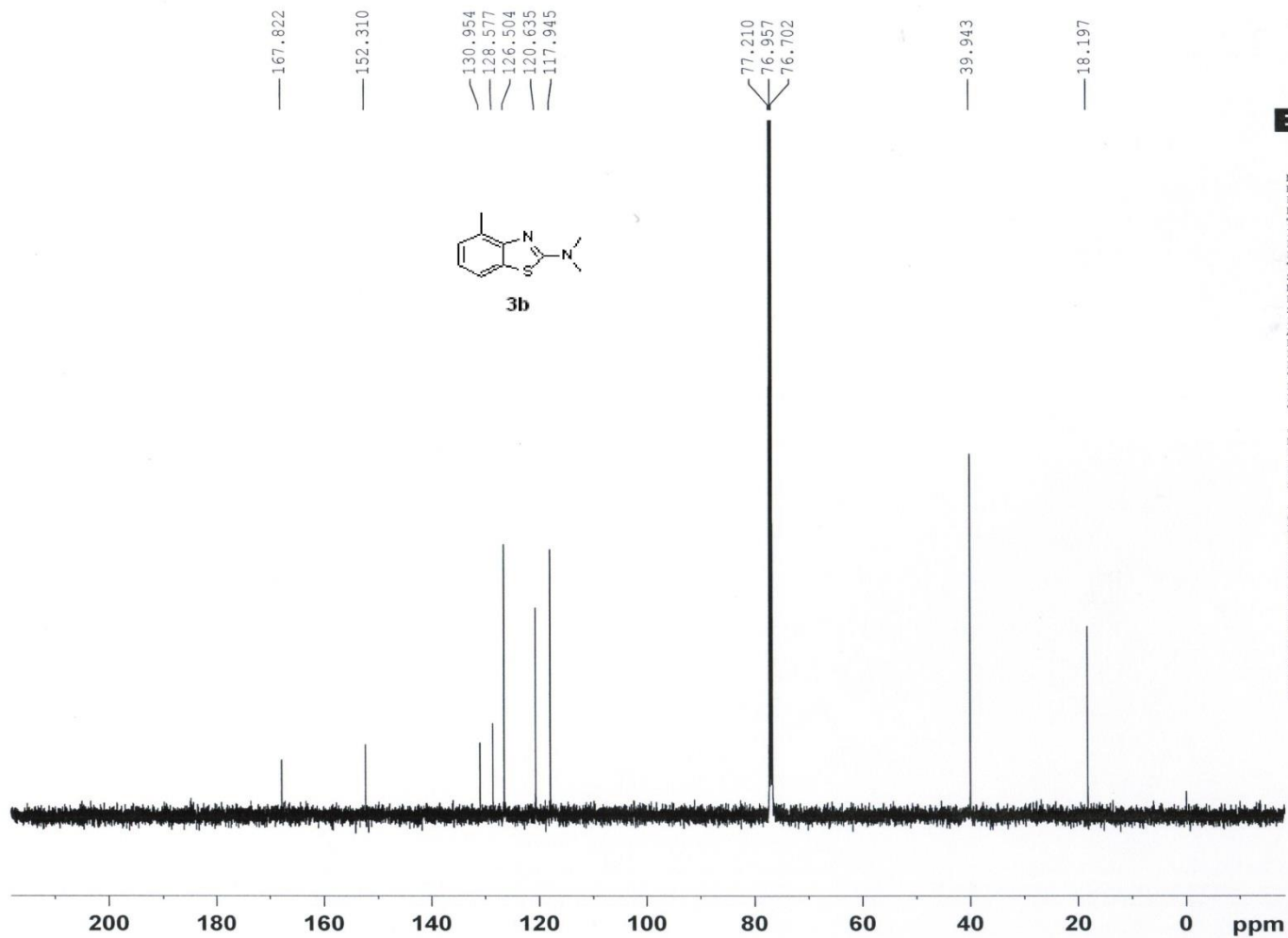


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PROCNO 1
Date 20141204
Time 14.27
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.0000000 sec
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 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3340060

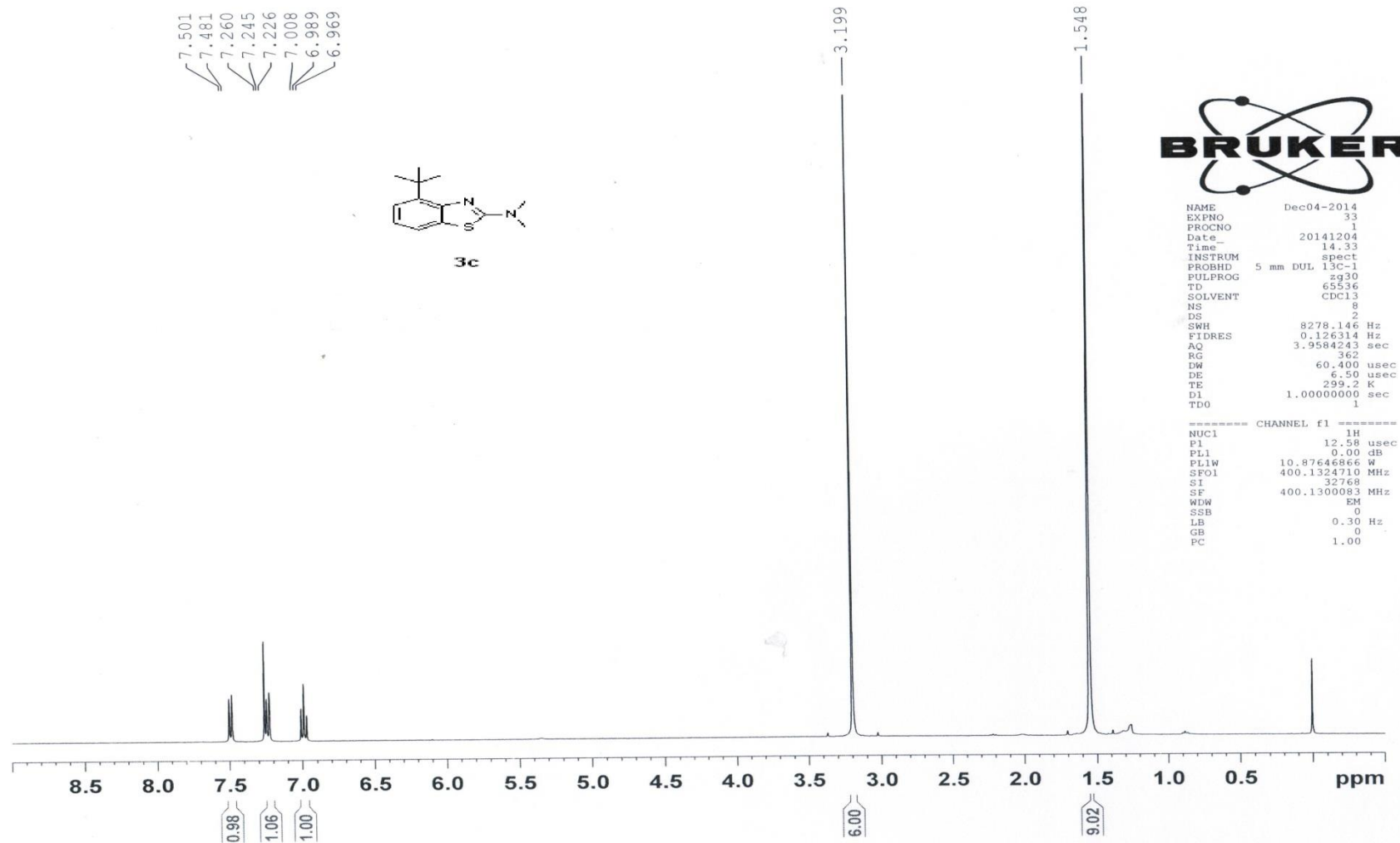


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PROCNO 1
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Time 13.06
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 892
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 301.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

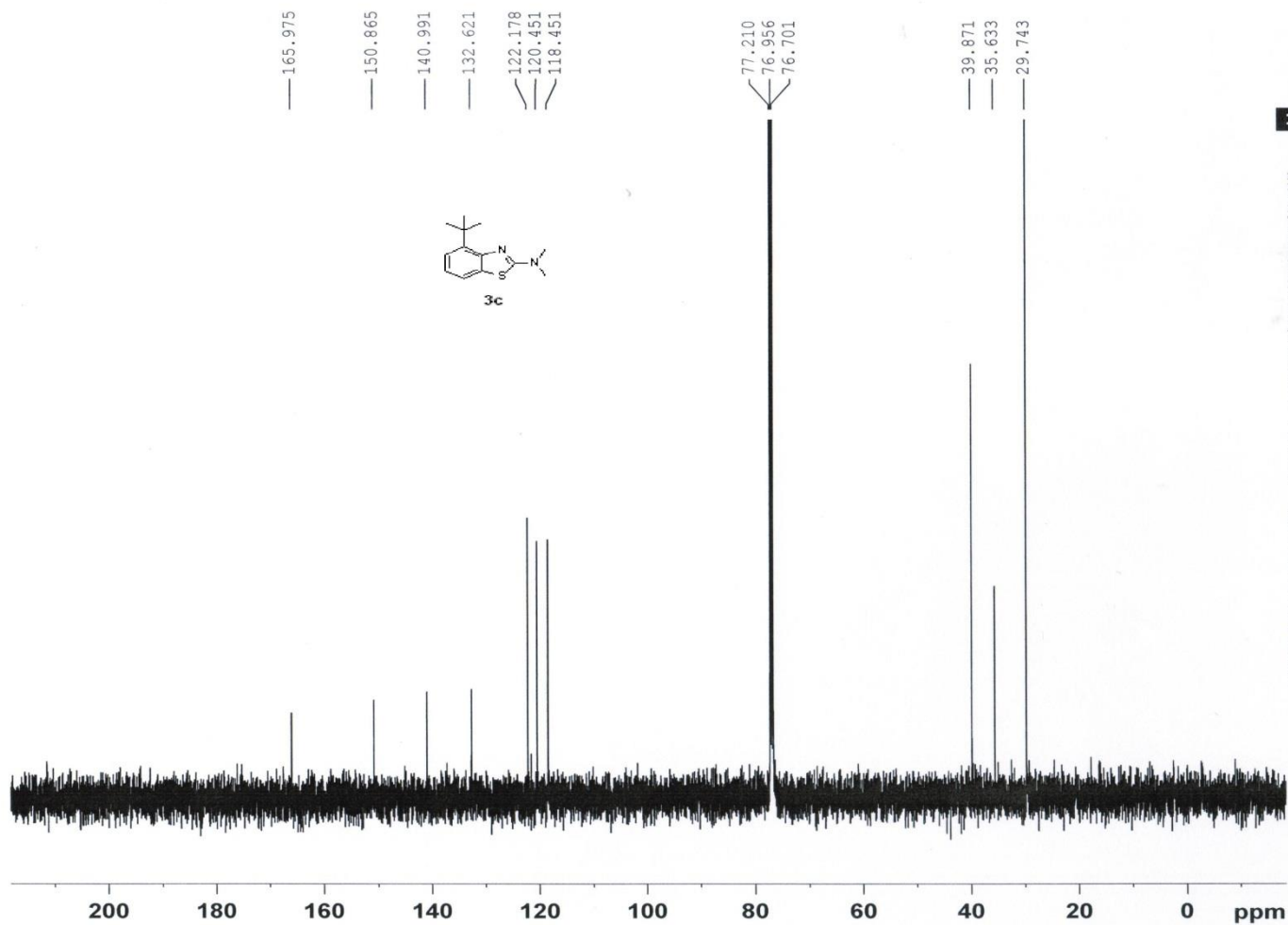
===== CHANNEL f1 =====
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
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

4760067



3340067



NAME 3340067
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 PROCNO 1
 Date 20141218
 Time 10.42
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 693
 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 299.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

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

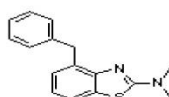
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.50 dB
 PL12 17.40 dB
 PL13 17.40 dB
 PL2W 13.02359581 W
 PL12W 0.42143536 W
 PL13W 0.42143536 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577966 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

3340129

7.460
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7.350
7.285
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7.172
7.154
7.050
7.032
6.978
6.958
6.939

— 4.336

— 3.205



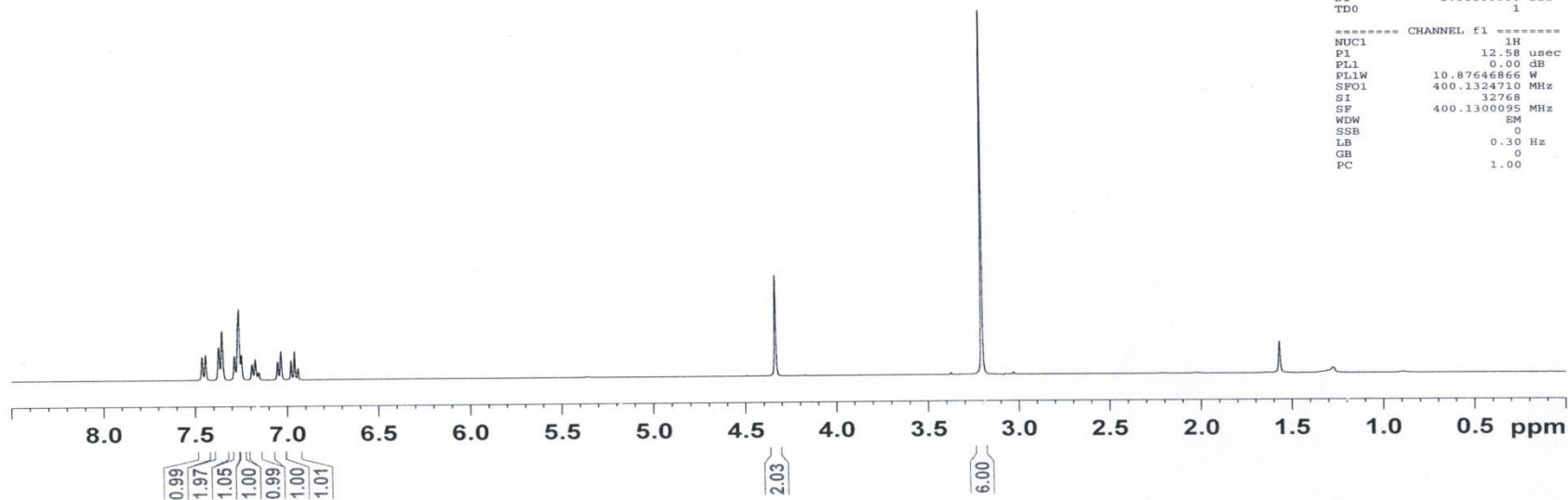
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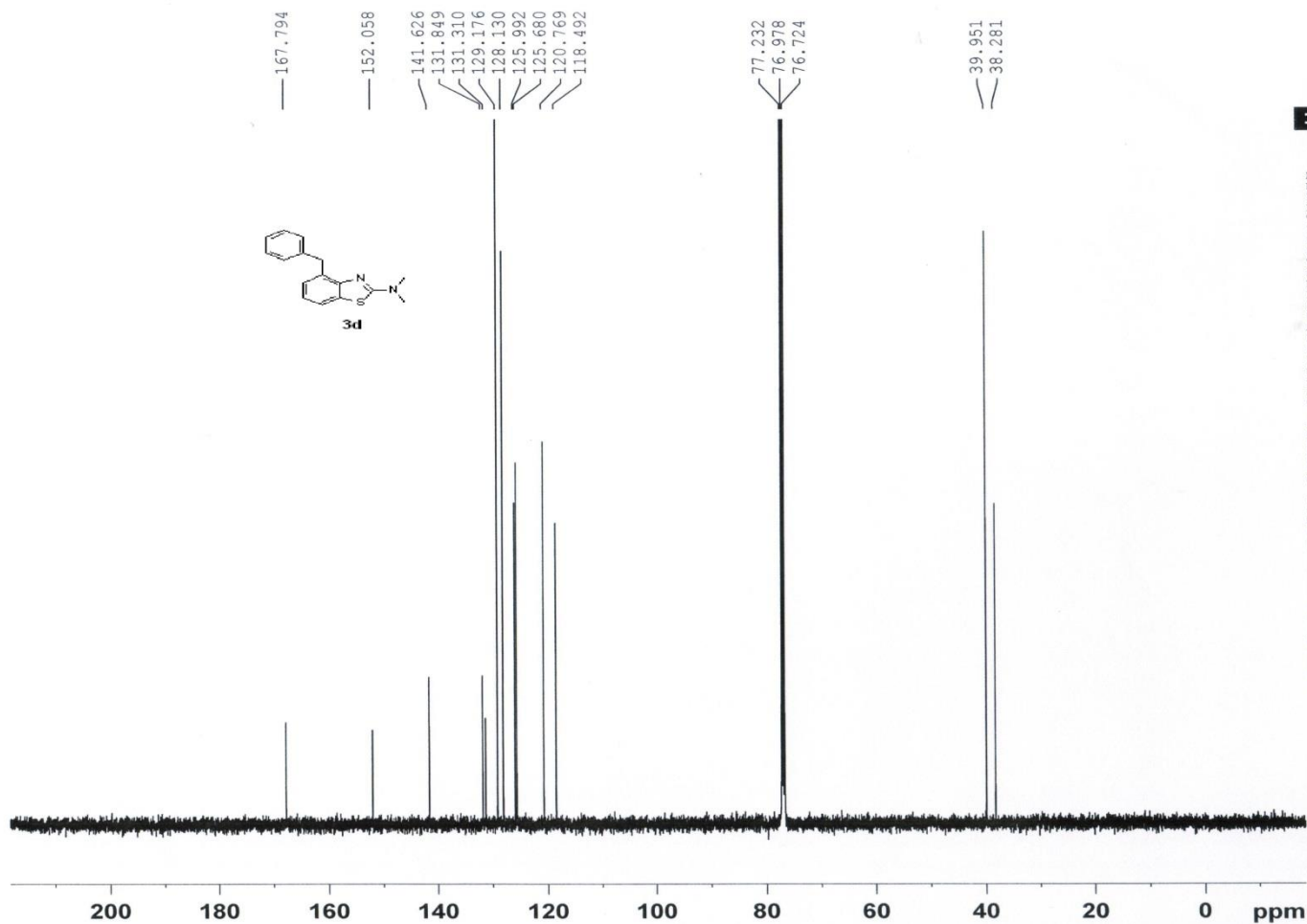
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Time          16.58
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PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            256
DW            60.400 usec
DE            6.50 usec
TE            297.1 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            400.1300095 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
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3340129



```

NAME      3340129
EXPNO     1
PROCNO    1
Date_     20150120
Time      11.52
INSTRUM   Spect
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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        299.3 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

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

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

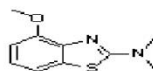
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3340061

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—3.979

—3.219



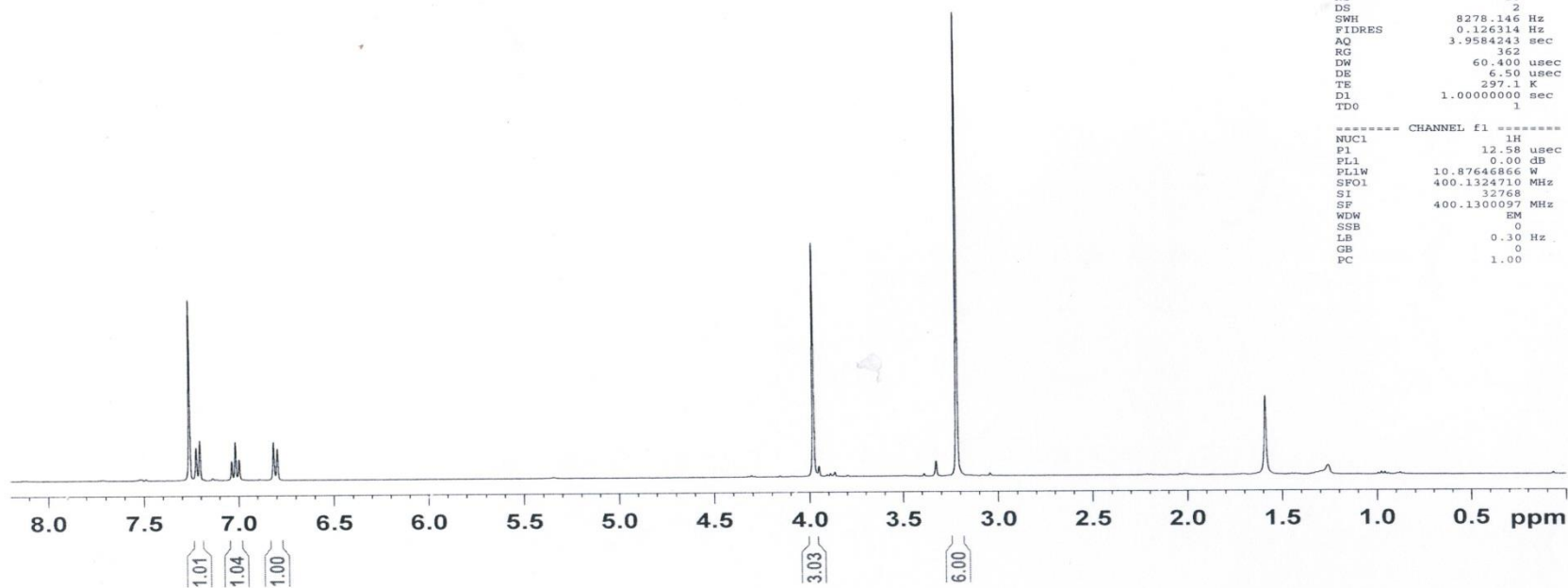
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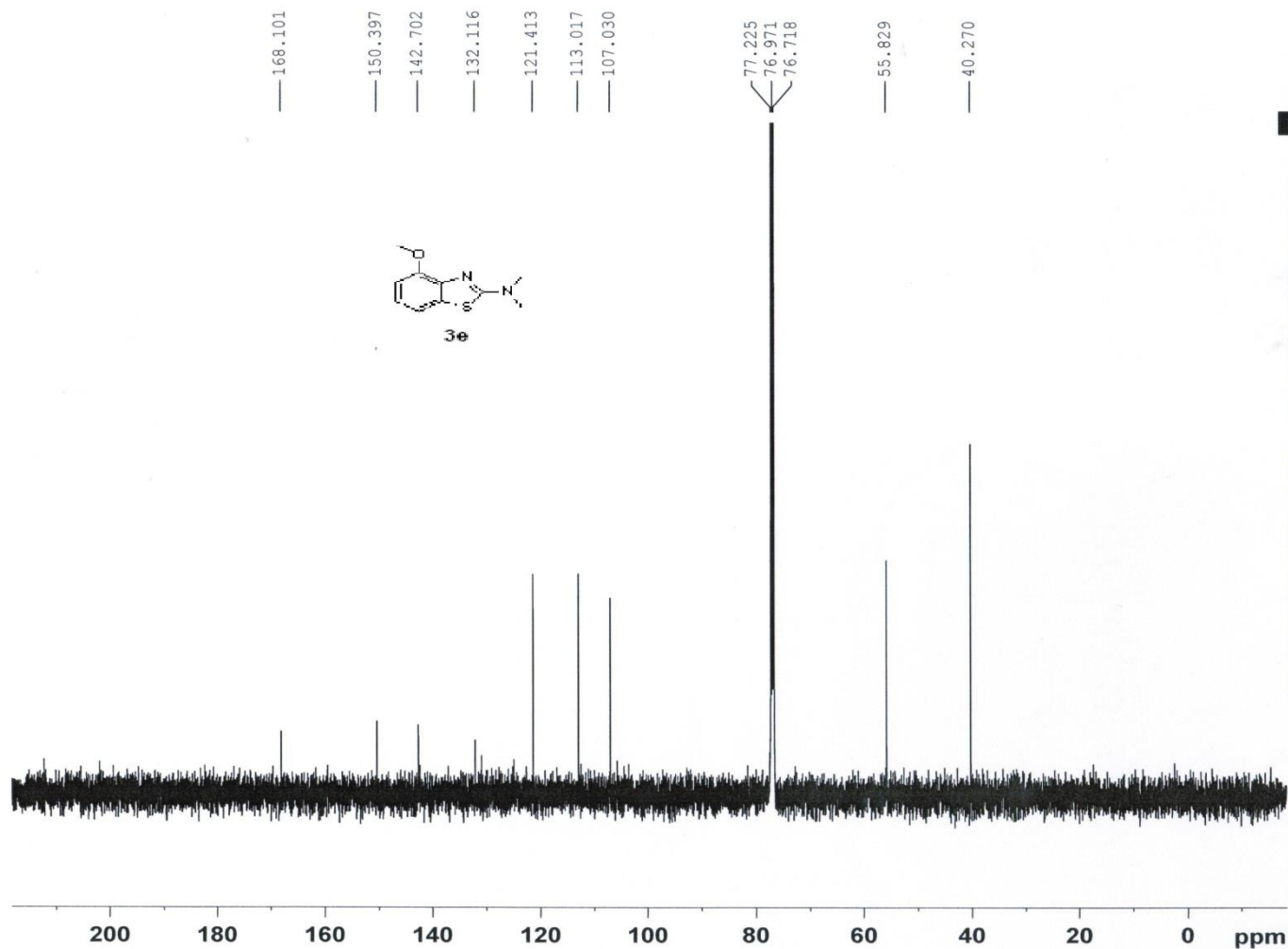
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Date_      20150122
Time       10.32
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PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         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         400.1300097 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
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3340061



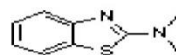
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PROCNO 1
Date 20150122
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PULPROG zgpg30
TD 65536
SOLVENT CDC13
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SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

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

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

33400221

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7.032



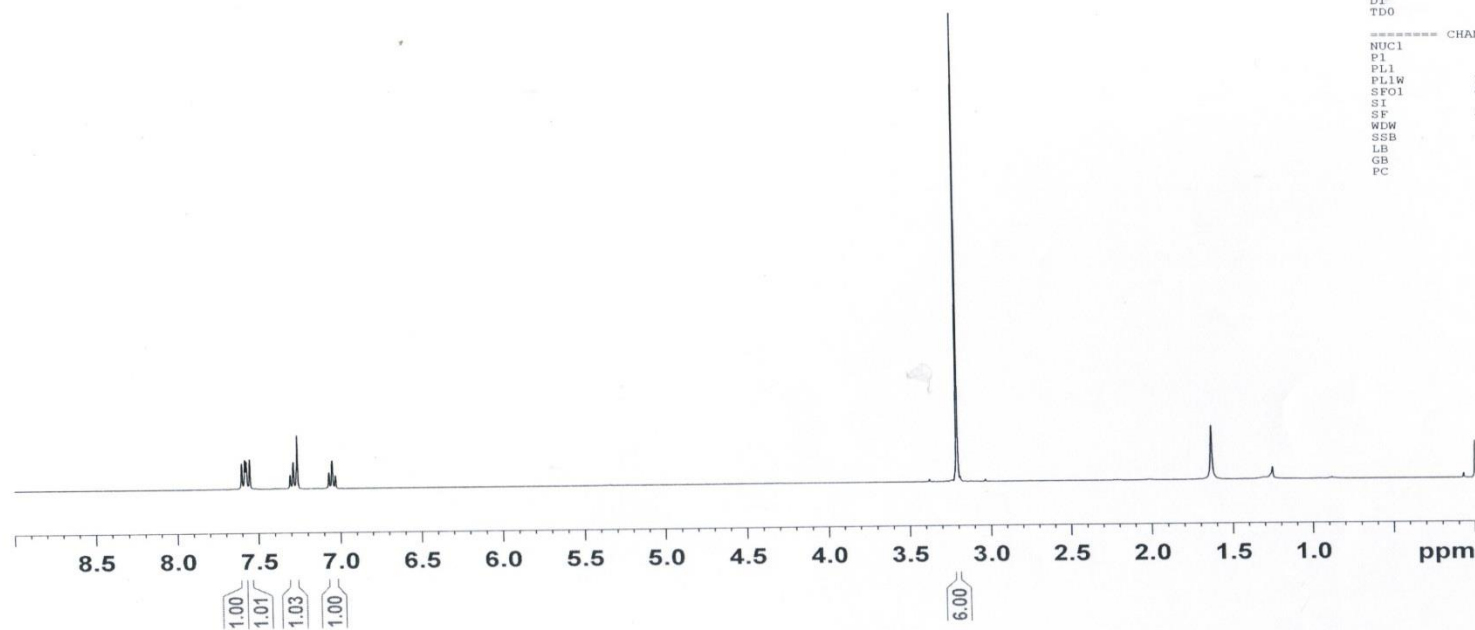
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3.207

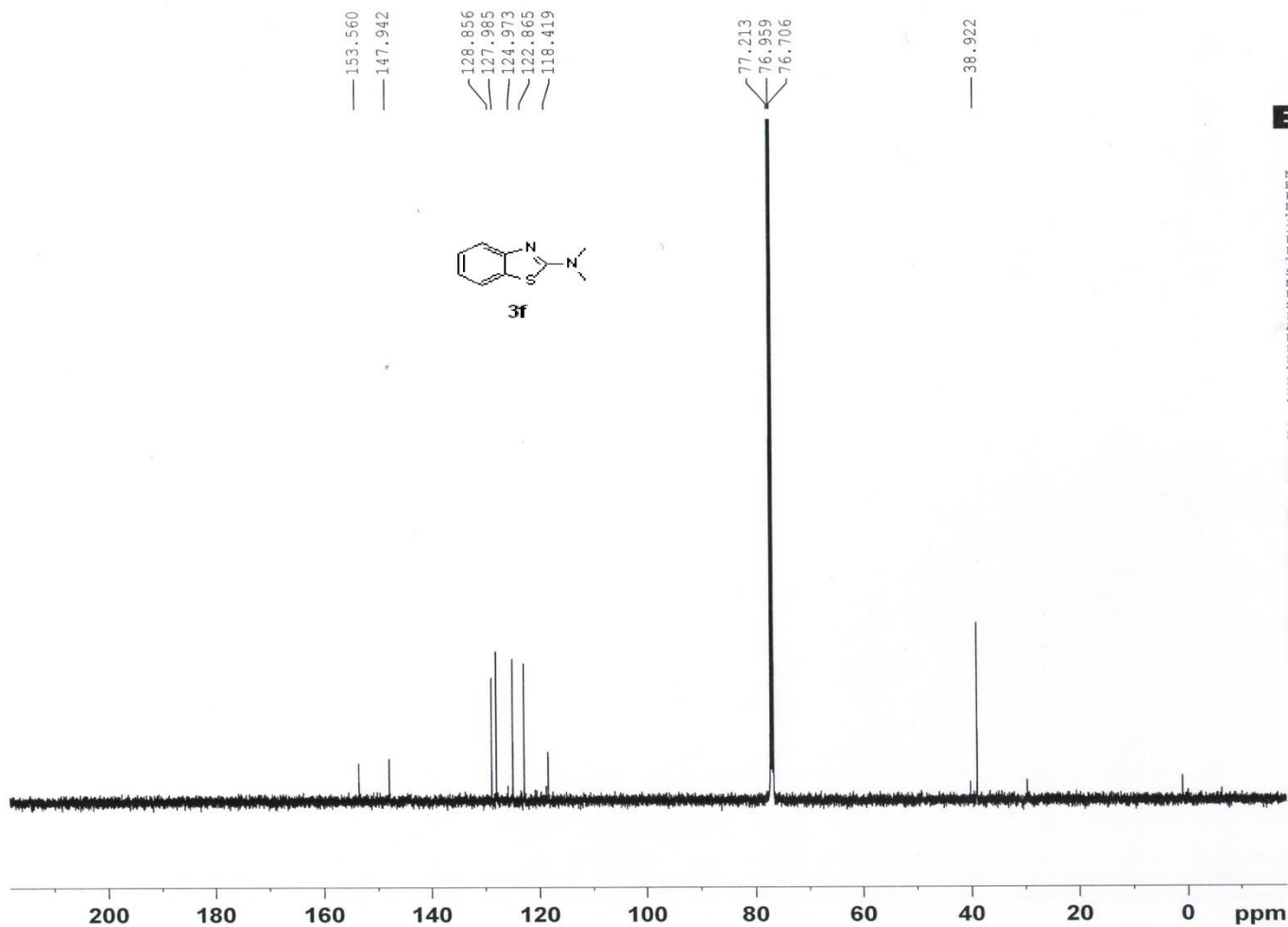


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PROCNO 1
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PULPROG zg30
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SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
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SI 32768
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SSB 0
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GB 0
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33400221



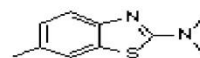
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 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 302.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCPD2 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

3340072

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3g

— 3.186

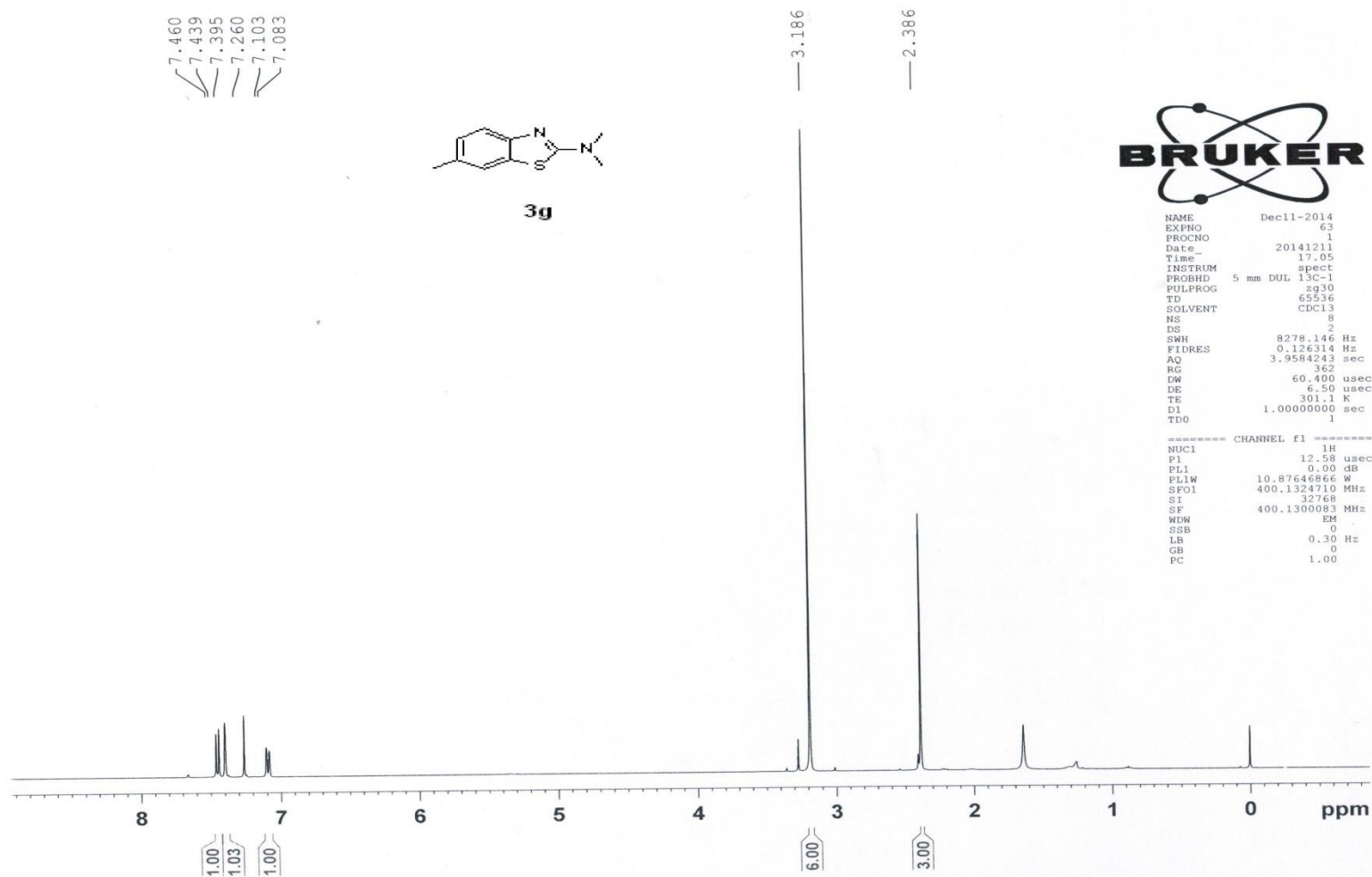
— 2.386



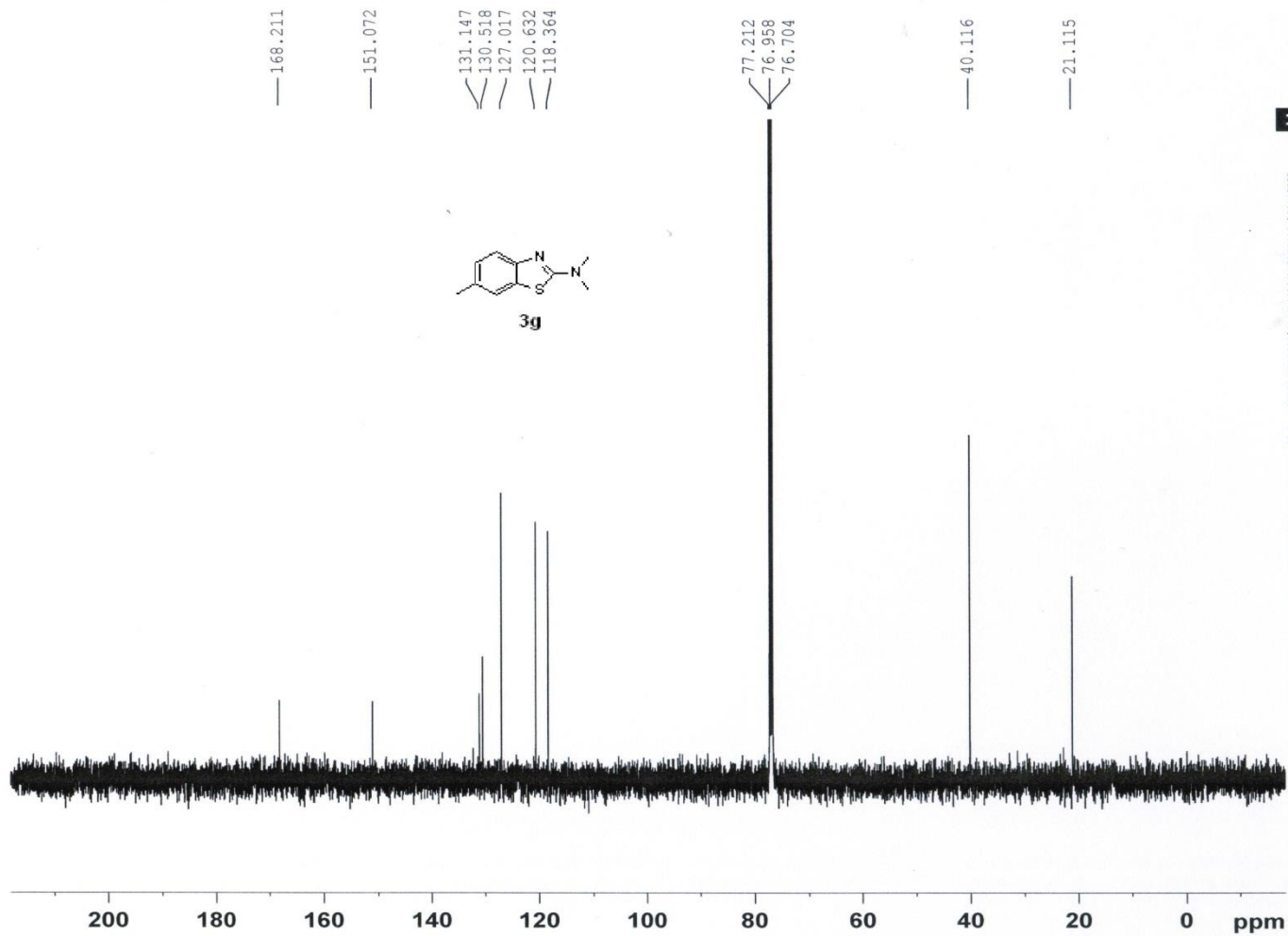
```

NAME      Dec11-2014
EXPNO      63
PROCNO     1
Date_      20141211
Time       17.05
INSTRUM    spect
PROBHD     5 mm DUL 13C-1
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS          8
DS          2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9564243 sec
RG          362
DW         60.400 usec
DE         6.50 usec
TE         301.1 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.56 usec
PL1        0.00 dB
PL1W       10.87646866 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300083 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



3340072



NAME 3340072
EXPNO 1
PROCNO 1
Date_ 20141218
Time 14.52
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 753
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 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

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

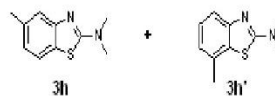
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3340073

7.466
7.446
7.425
7.404
7.390
7.260
7.227
7.208
7.188
6.891
6.873
6.855

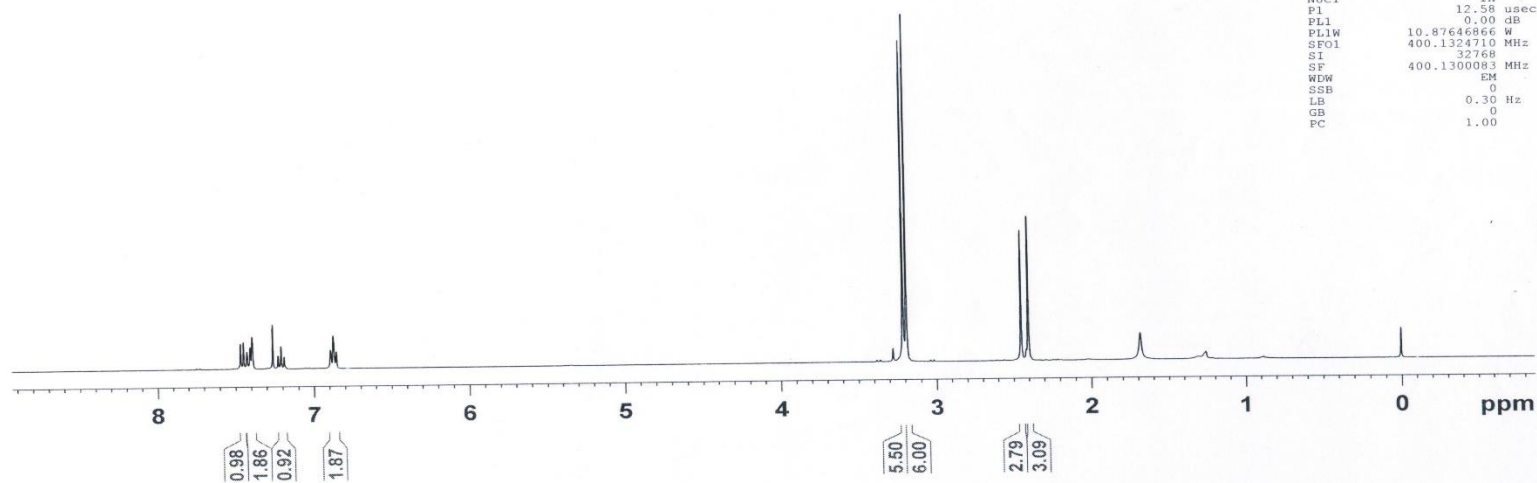
3.212
3.190

2.449
2.402

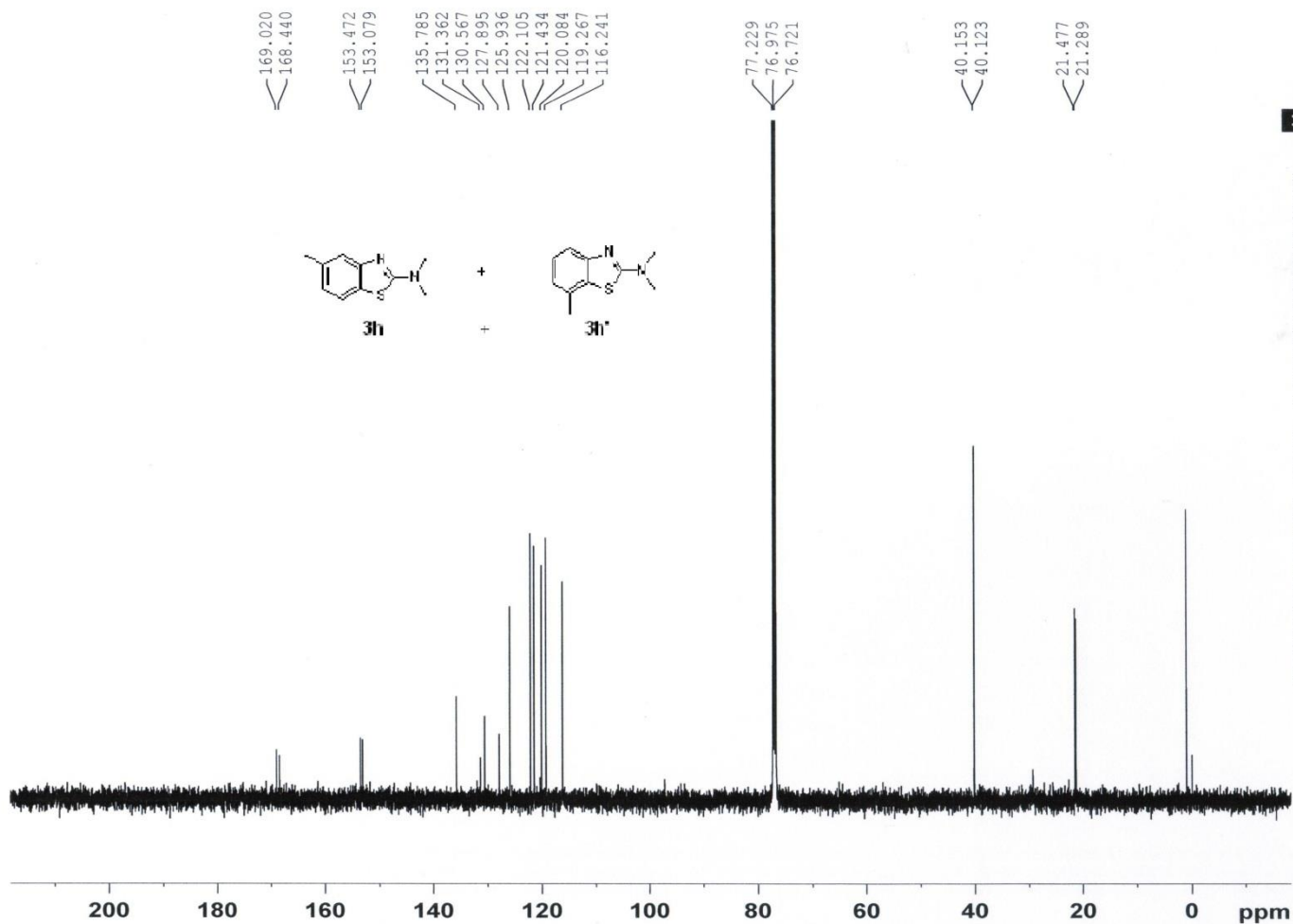


NAME Dec11-2014
 EXPNO 64
 PROCNO 1
 Date_ 20141211
 Time 17.11
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DW 60.400 usec
 DE 6.50 usec
 TE 301.1 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.58 usec
 PL1 0.00 dB
 PL1W 10.87646866 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300083 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3340073



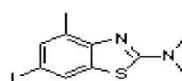
NAME 3340073
 EXPNO 1
 PROCNO 1
 Date 20150121
 Time 9.26
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 986
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 298.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

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

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

3340083

— 7.709
— 7.397
— 7.260



3i

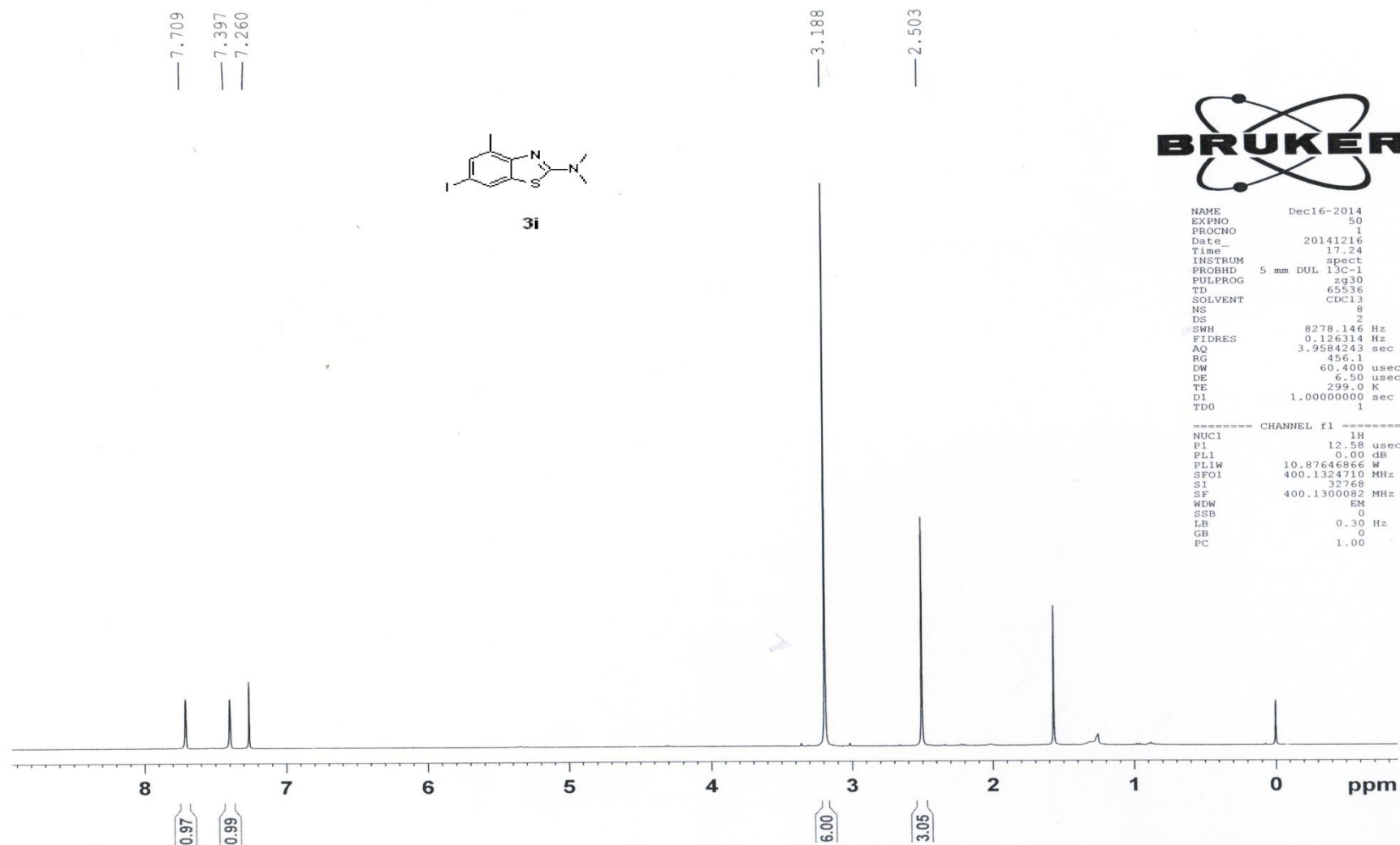
— 3.188

— 2.503

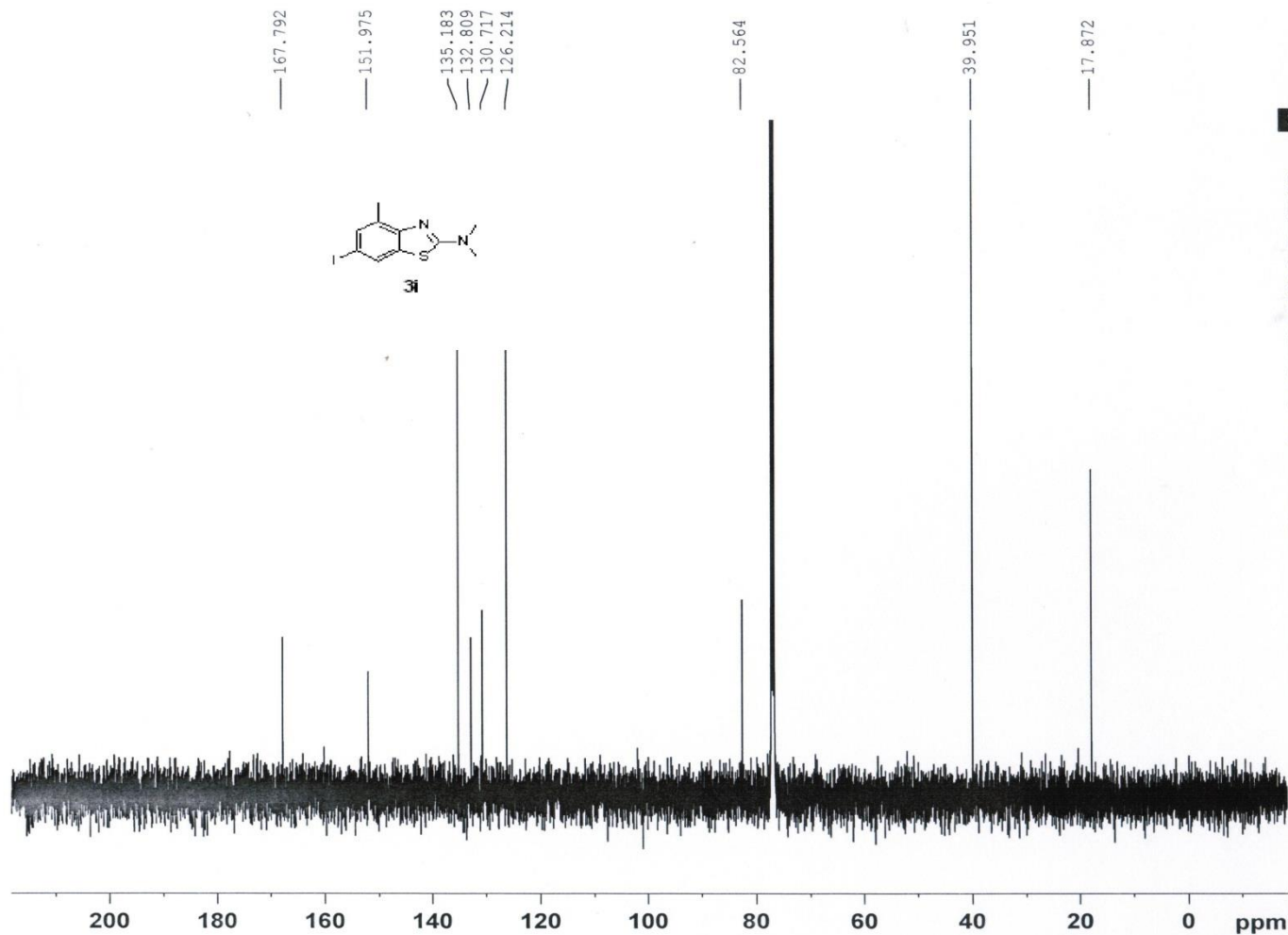


NAME Dec16-2014
EXPNO 50
PROCNO 1
Date_ 20141216
Time 17.24
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3340083



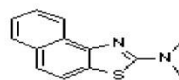
NAME 3340083
 EXPNO 2
 PROCNO 1
 Date_ 20141217
 Time 11.49
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 301.8 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 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

3340071

8.615
8.595
7.864
7.844
7.693
7.672
7.561
7.539
7.523
7.517
7.489
7.469
7.451
7.260



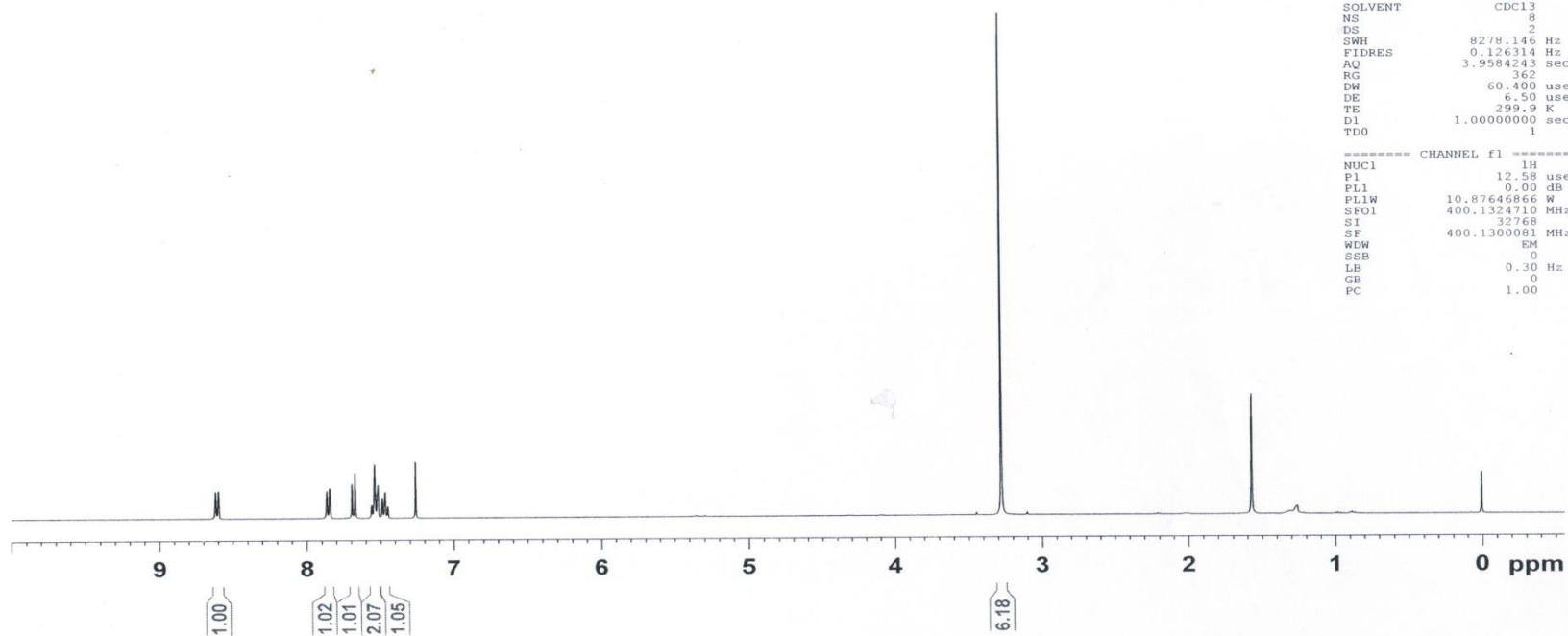
3j

3.277

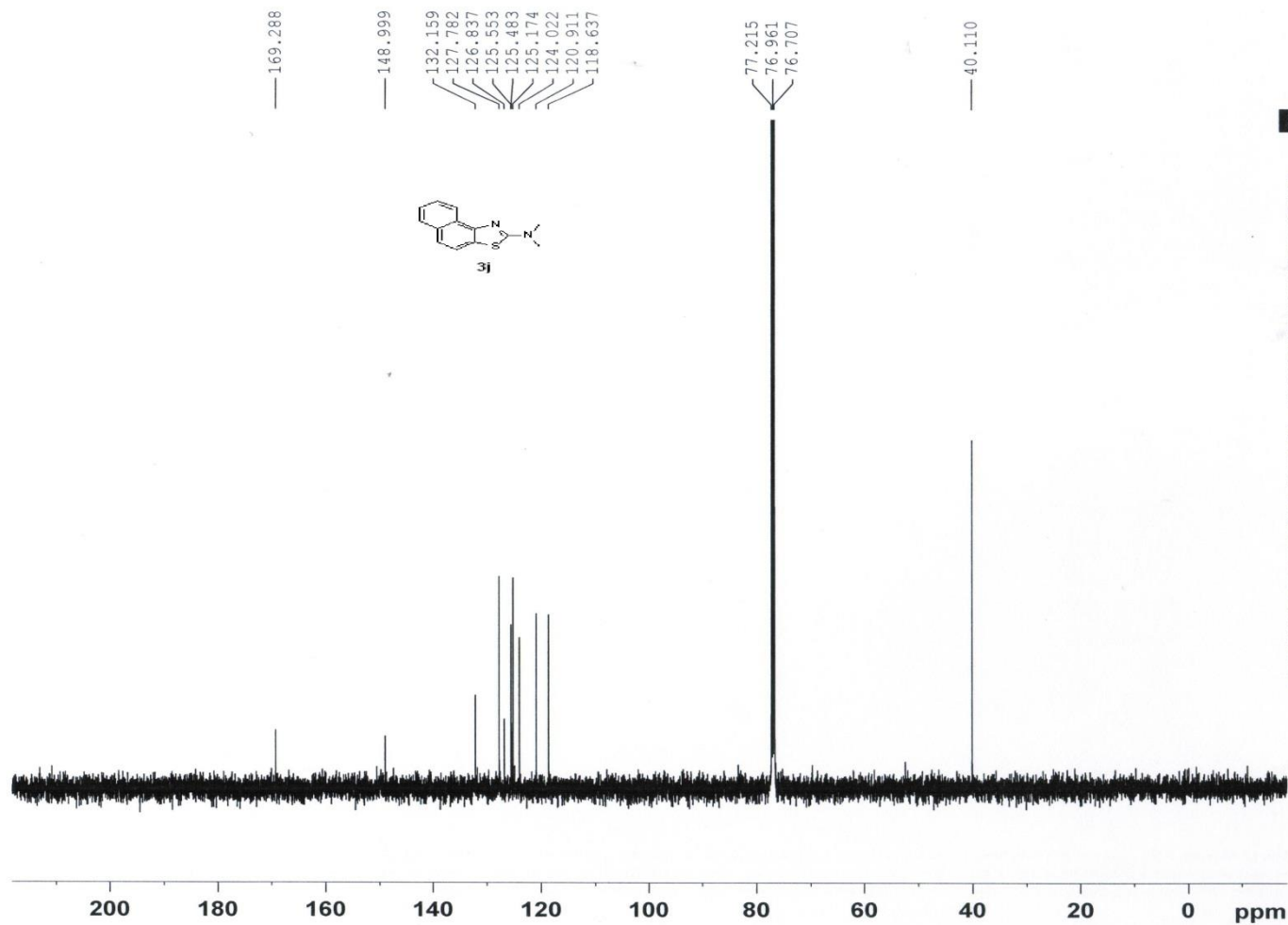


NAME Dec09-2014
EXPNO 9
PROCNO 1
Date 20141209
Time 9.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



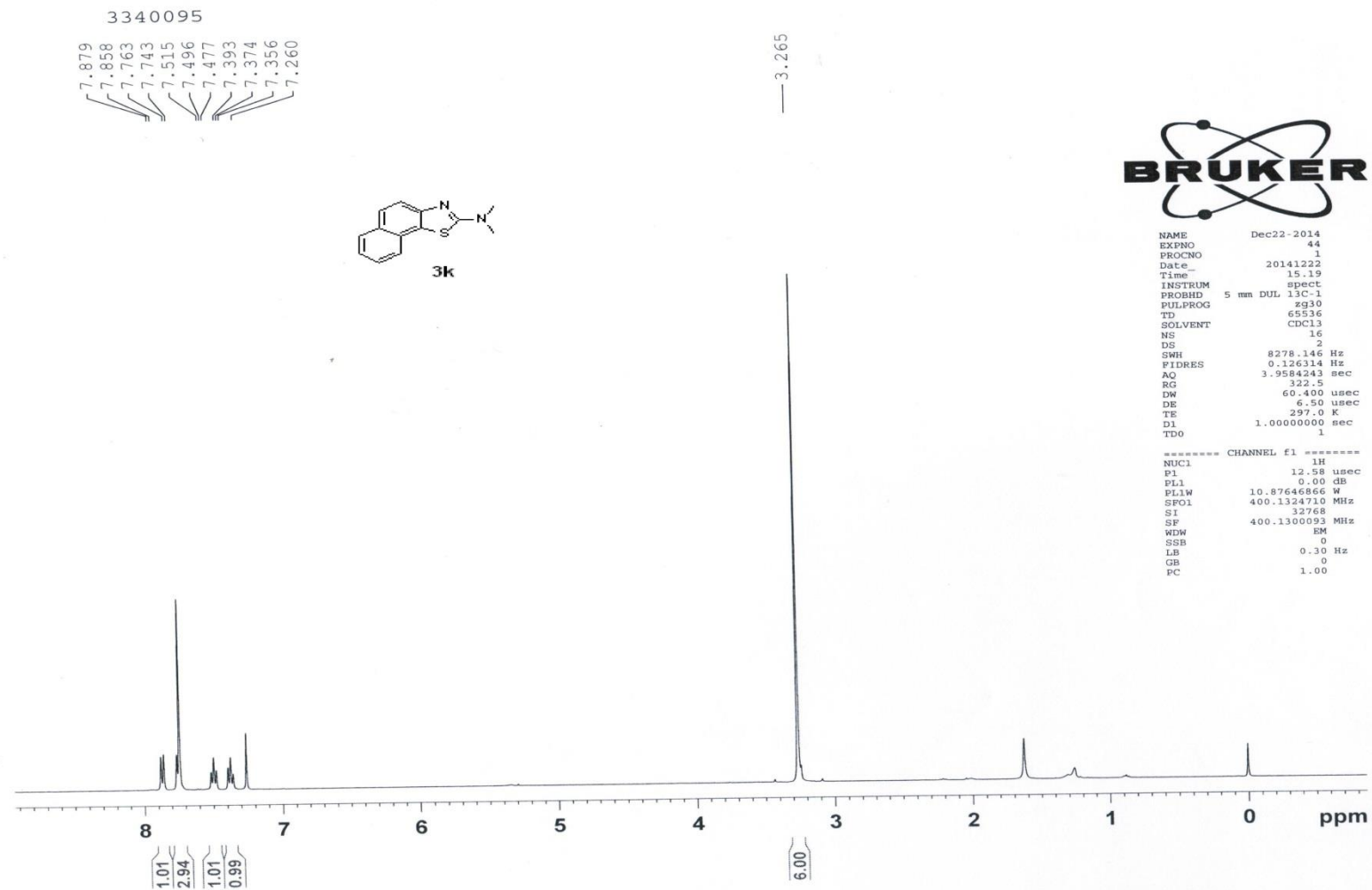
3340071



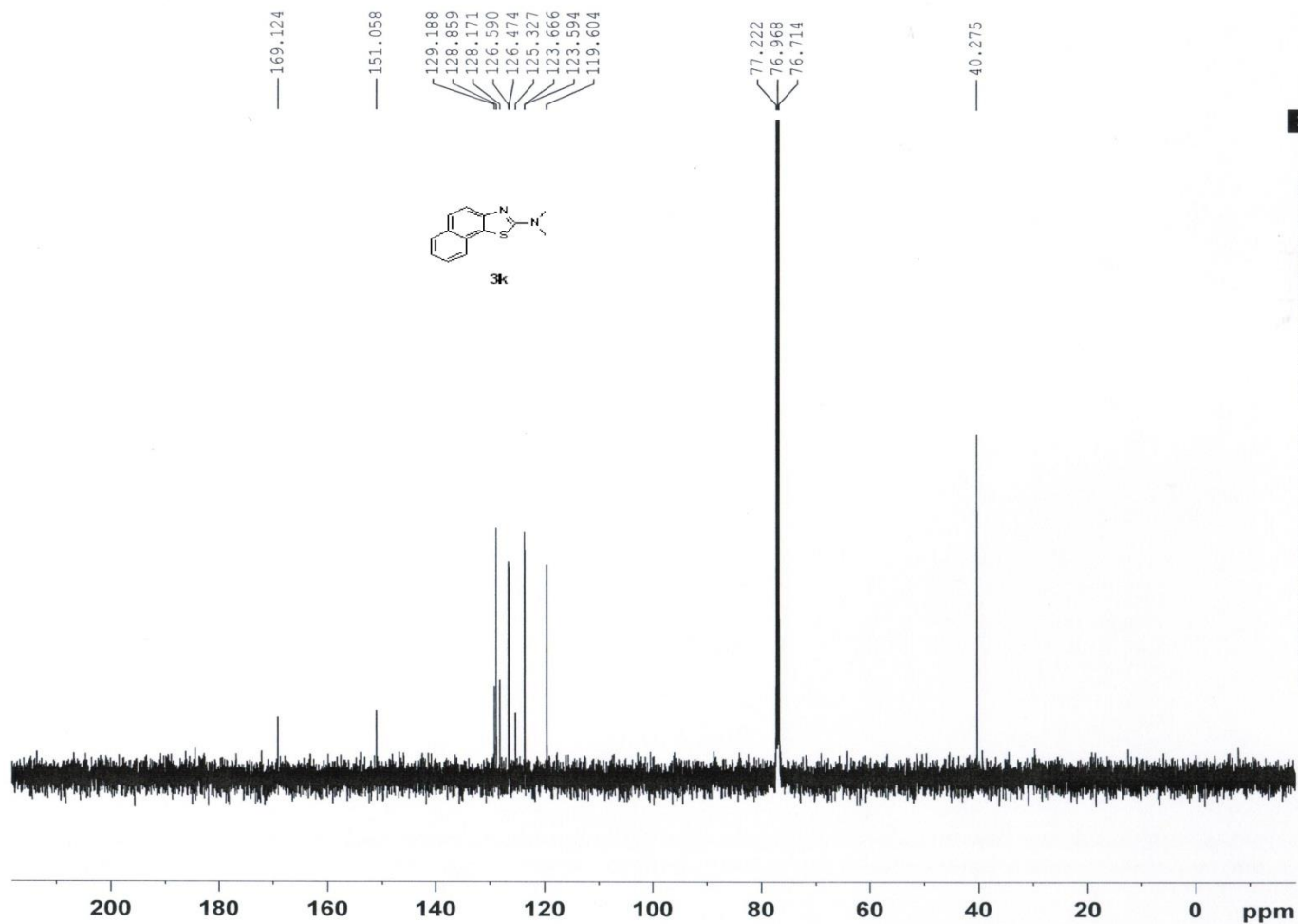
NAME 3340071
 EXPNO 1
 PROCNO 1
 Date 20141218
 Time 11.40
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 299.6 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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



3340095



```

NAME          3340095
EXPNO         1
PROCNO        1
Date_         20141225
Time          9.21
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            222
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            300.7 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

```

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

```

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

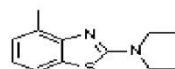
3340092

7.430
7.411
7.260
7.091
7.073
6.950
6.931
6.912

3.611
3.593
3.576
3.558

— 2.559

1.300
1.282
1.264

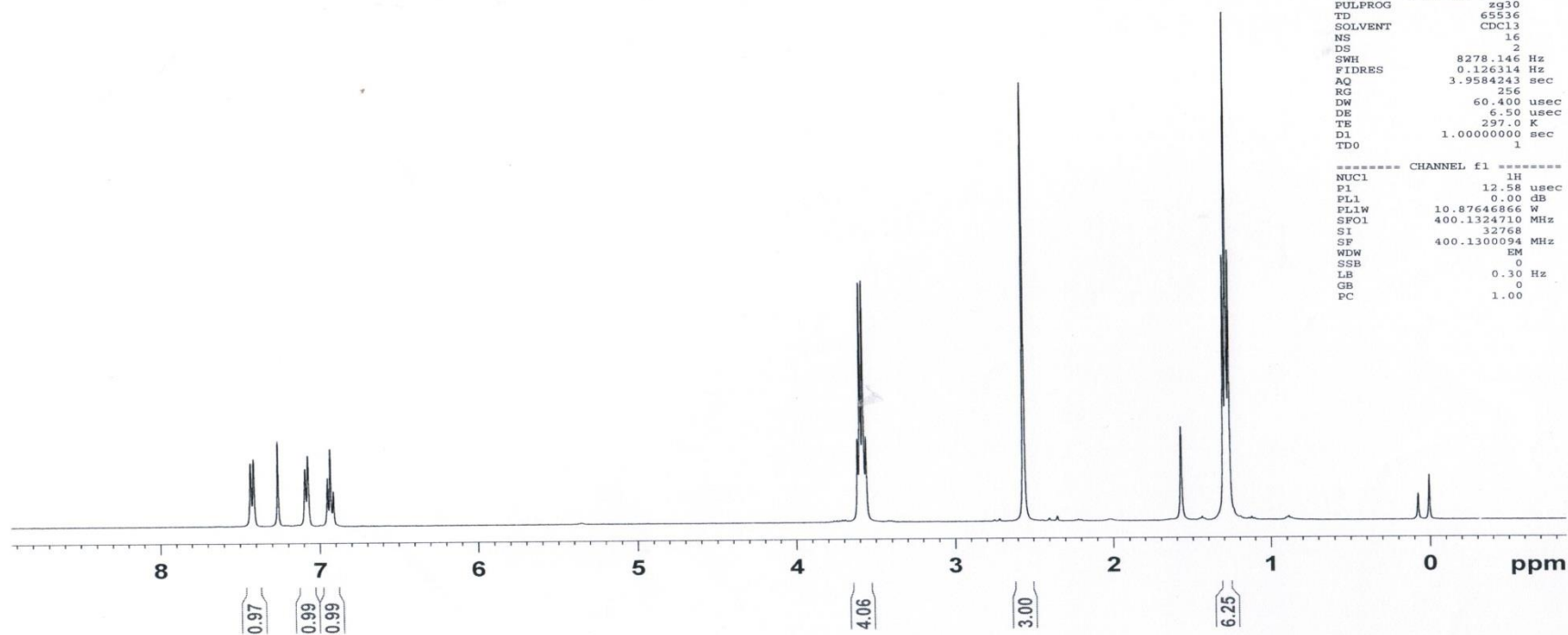


31

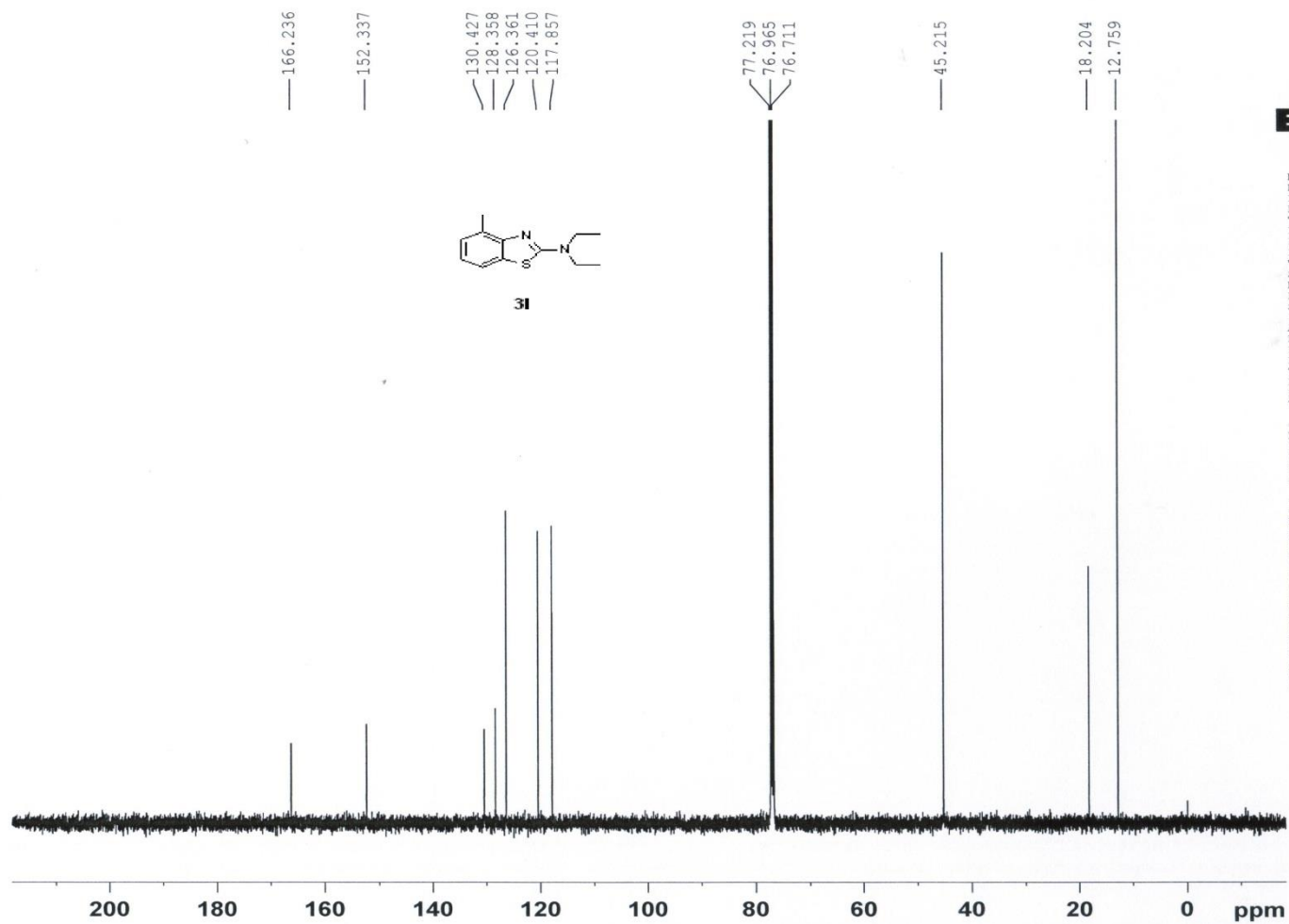


NAME Dec22-2014
EXPNO 42
PROCNO 1
Date_ 20141222
Time 15.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

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



3340092



```

NAME      3340092
EXPNO     1
PROCNO    1
Date_     20141224
Time      16.54
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         300.2 K
D1         2.0000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

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

```

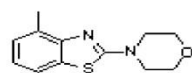
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        2.50 dB
PL12       17.40 dB
PL13       17.40 dB
PL2W       13.02359581 W
PL12W      0.42143536 W
PL13W      0.42143536 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7577966 MHz
WDW        EM
SSB  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

33400892

7.467
7.447
7.260
7.132
7.113
7.022
7.003
6.984

3.853
3.841
3.829
3.641
3.629
3.617

— 2.564

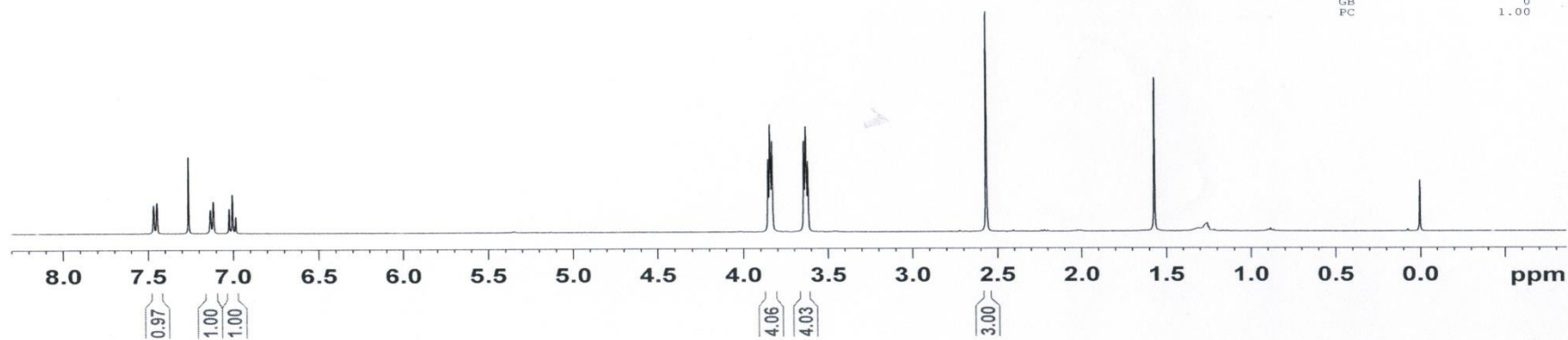


3m

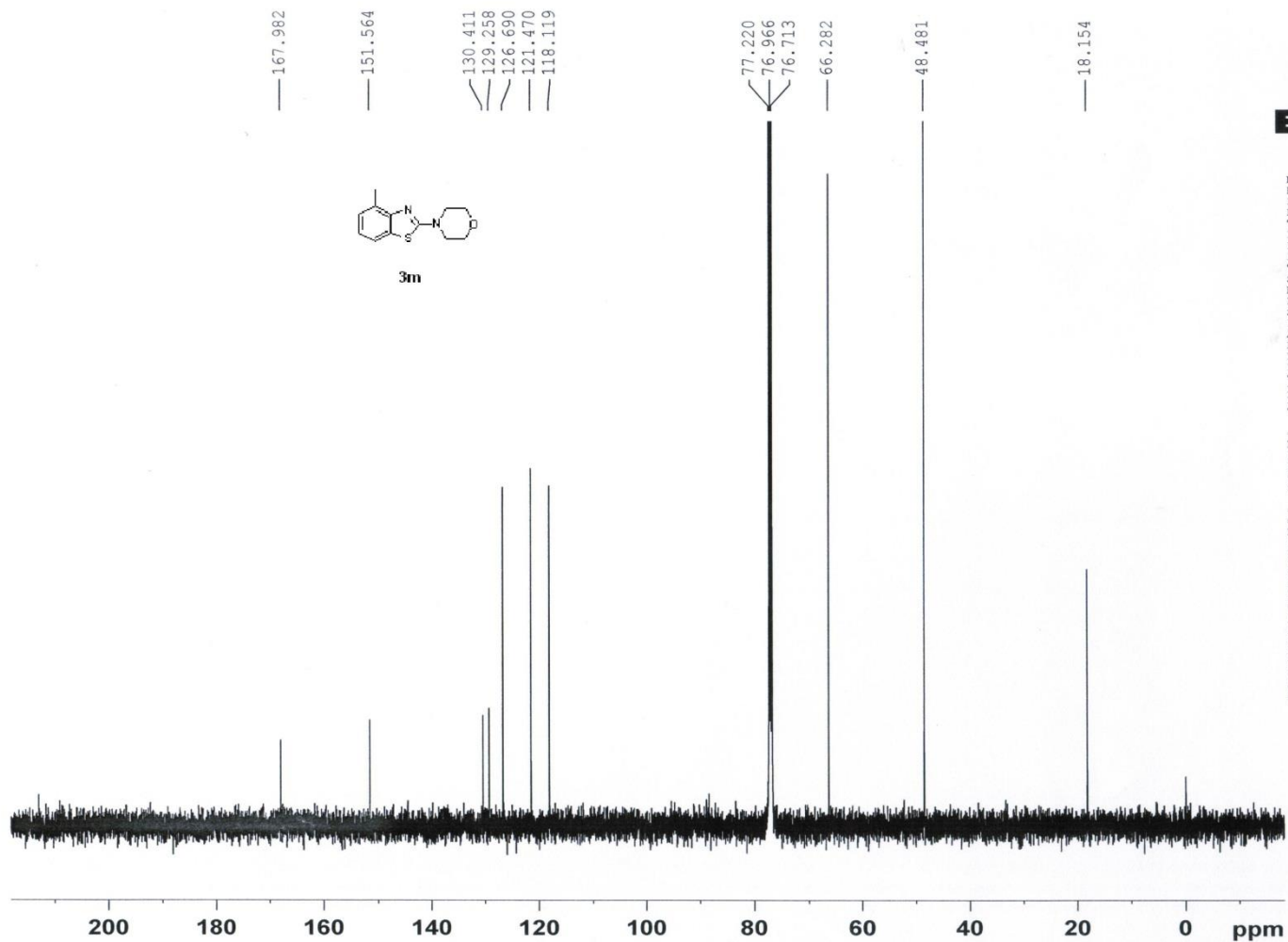


NAME Dec16-2014
EXPNO 51
PROCNO 1
Date_ 20141216
Time 17.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



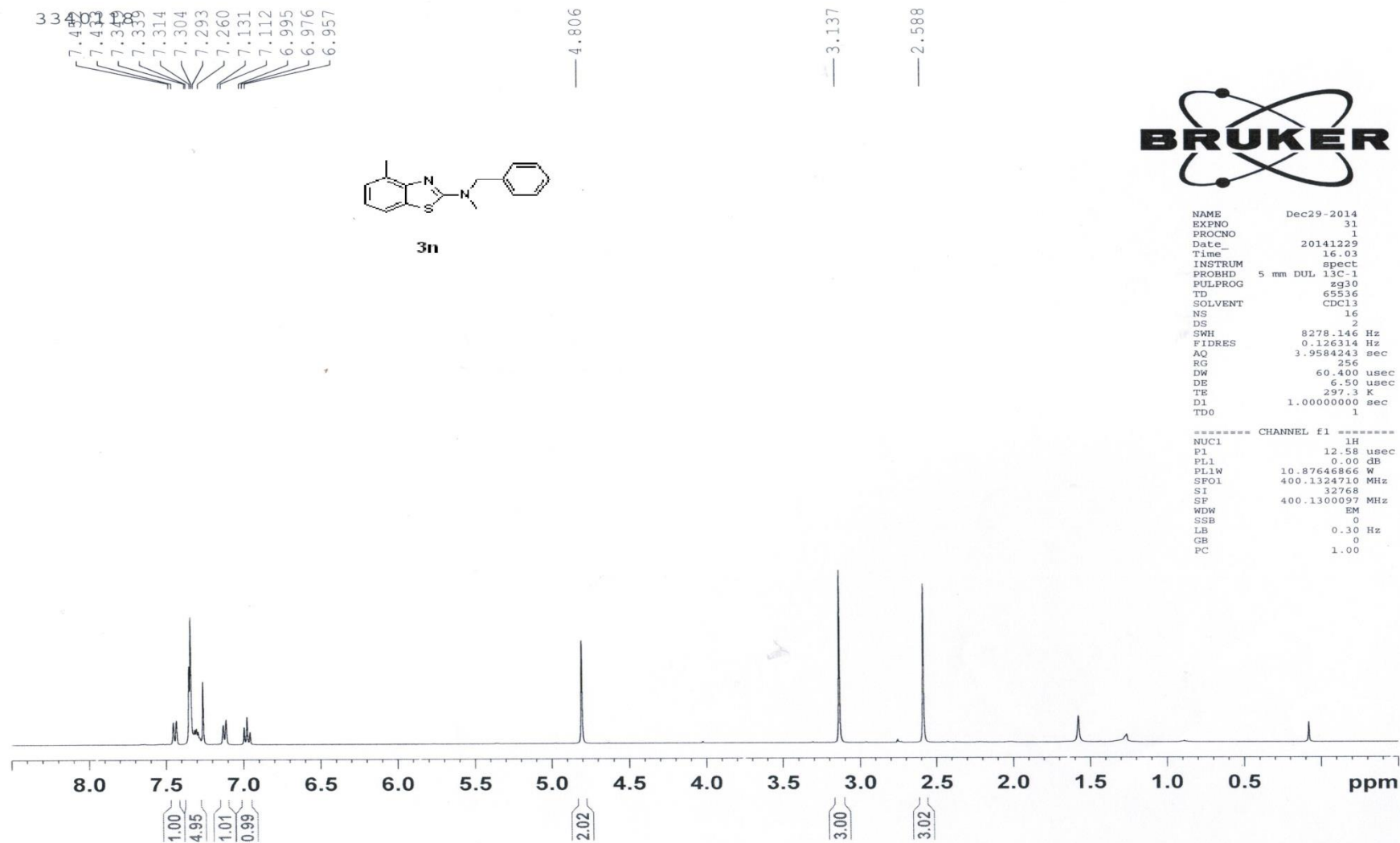
33400892



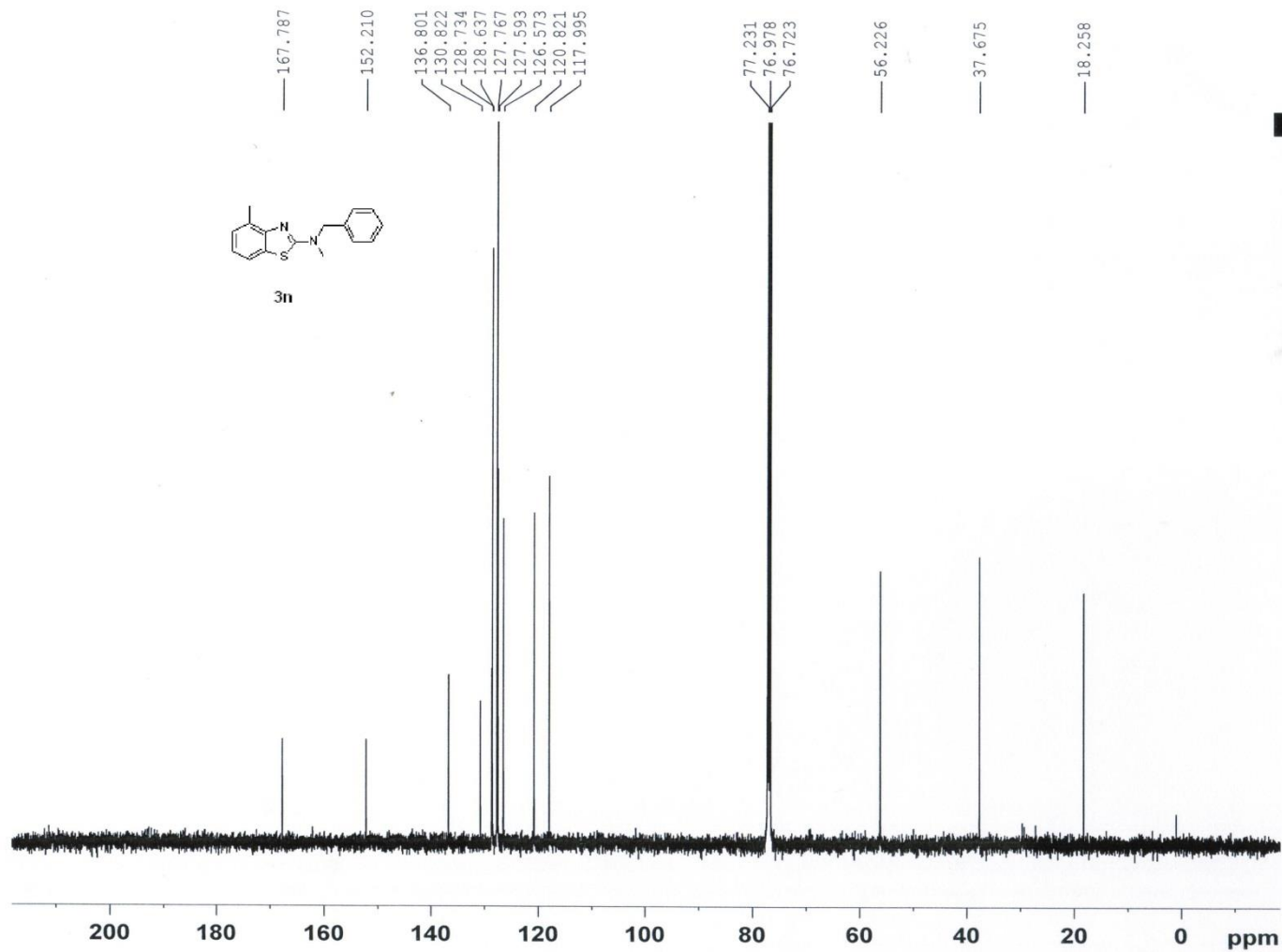
NAME 33400892
 EXPNO 1
 PROCNO 1
 Date_ 20141219
 Time 13.11
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 298.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

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

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



3340118



NAME 3340118
EXPNO 1
PROCNO 1
Date_ 20150114
Time_ 15.51
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 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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

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

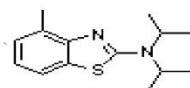
3340094

7.426
7.407
7.260
7.076
7.058
6.926
6.907
6.889

3.904
3.887
3.870
3.854
3.837

— 2.555

1.454
1.437

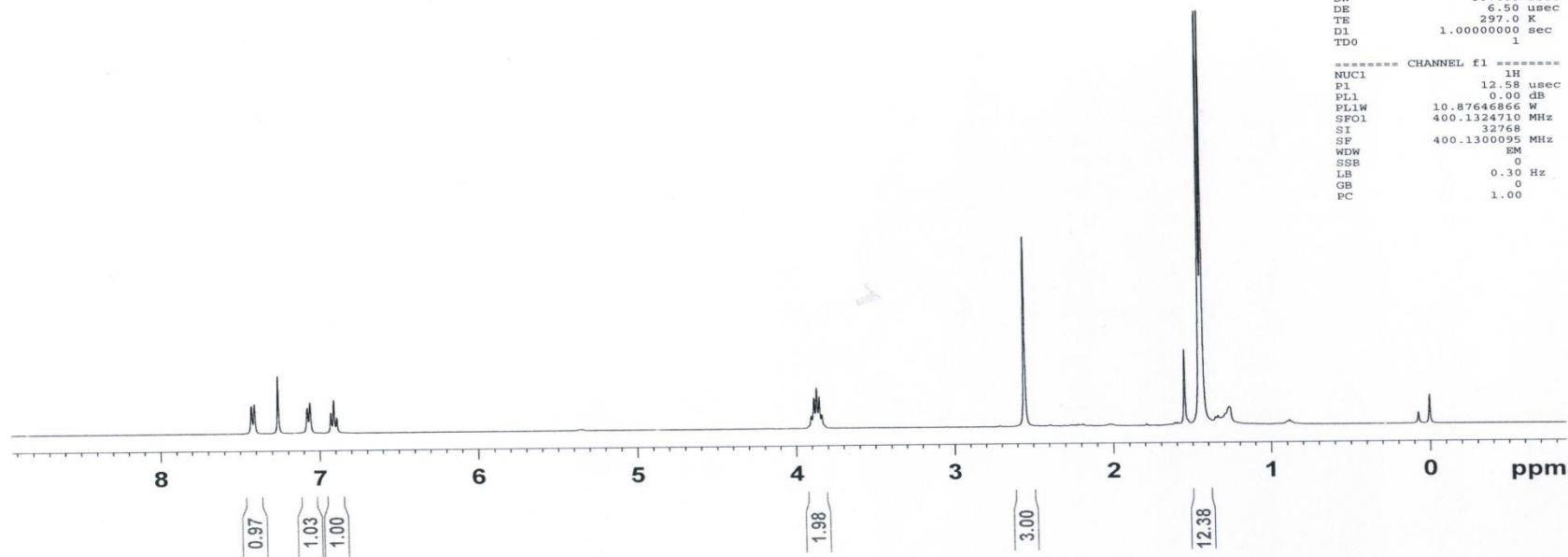


30

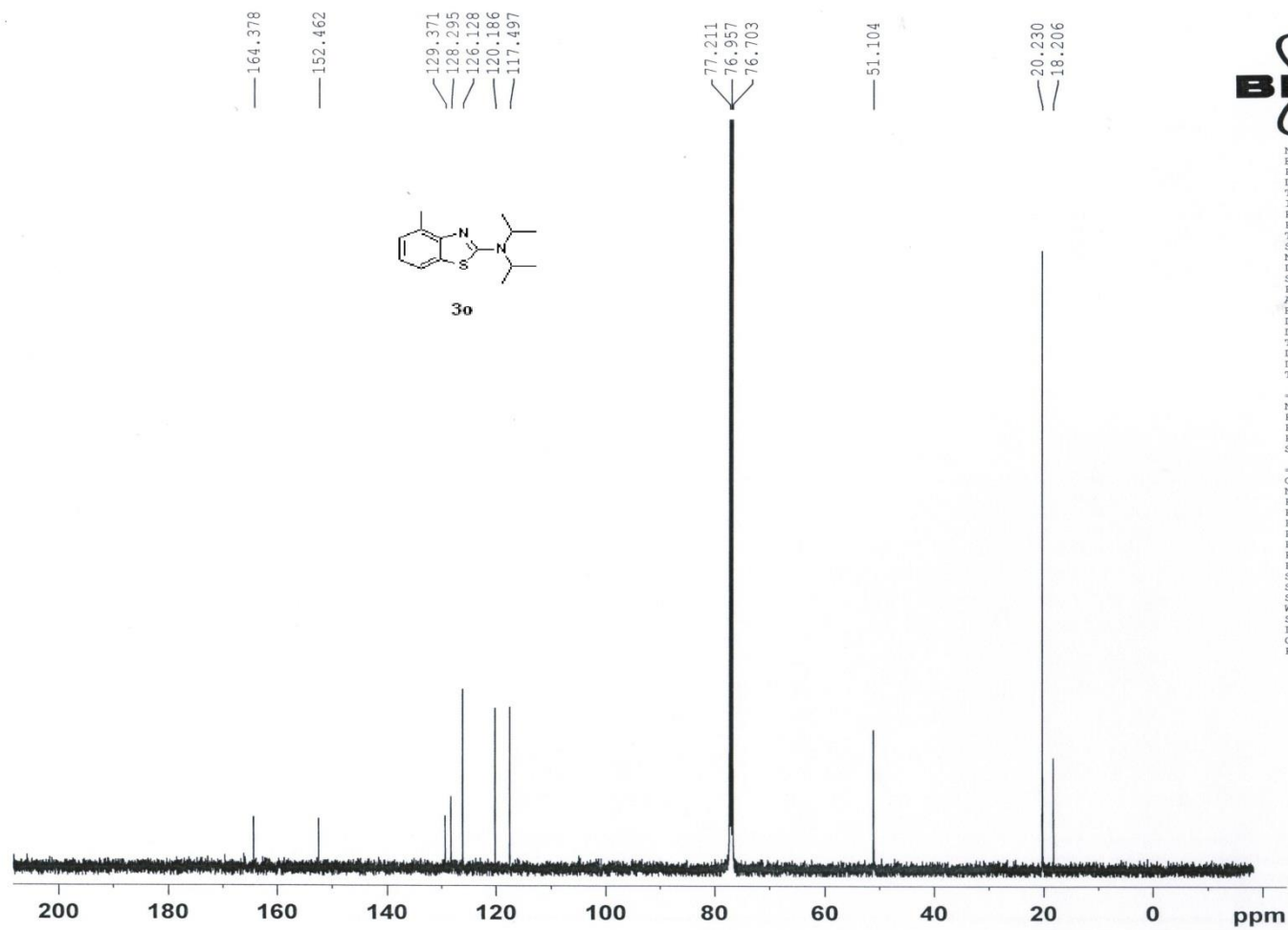


NAME Dec22-2014
EXPNO 43
PROCNO 1
Date_ 20141222
Time 15.13
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

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



3340049



```

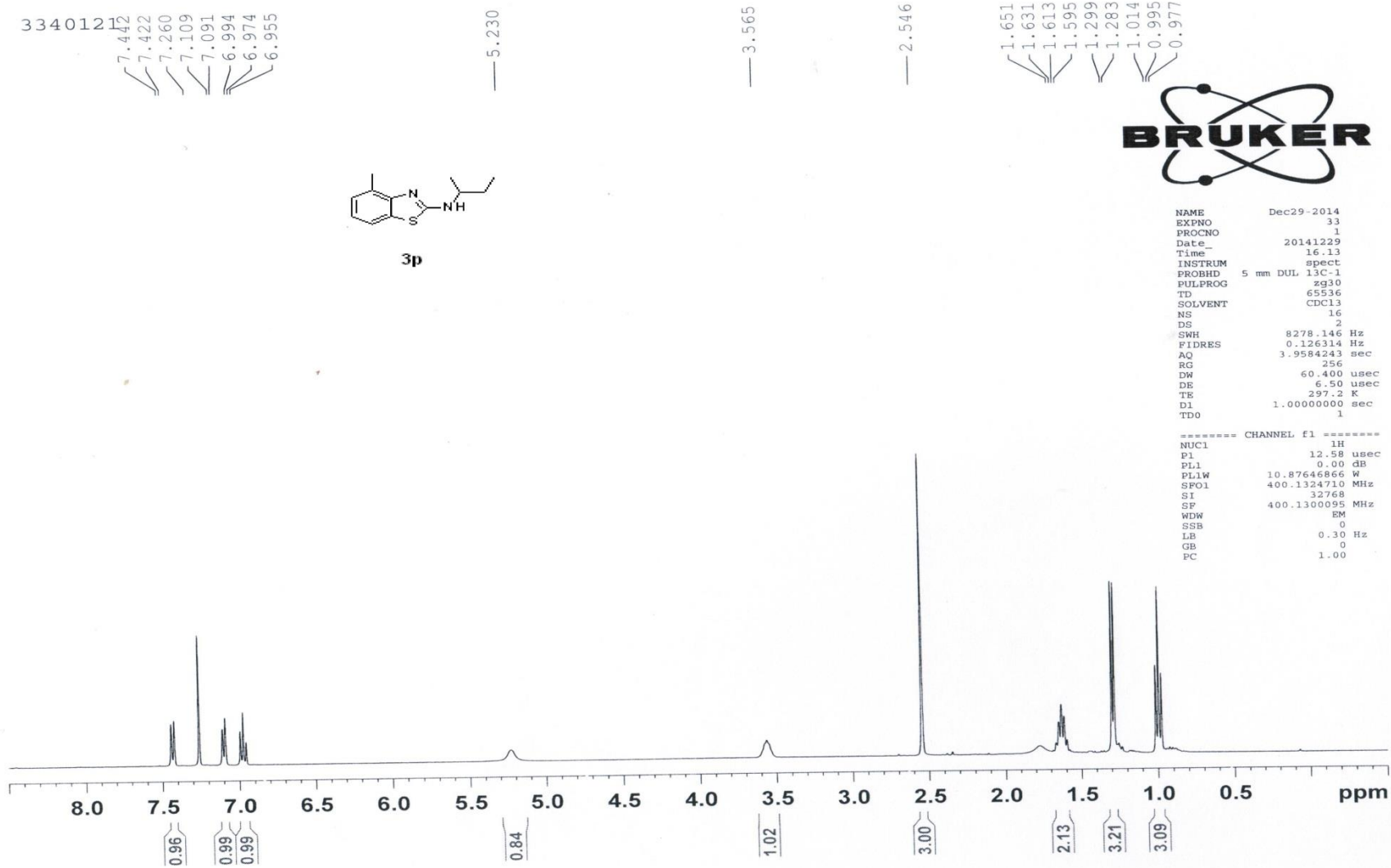
NAME          3340049
EXPNO         1
PROCNO        1
Date_         20141226
Time_         12.18
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            301.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

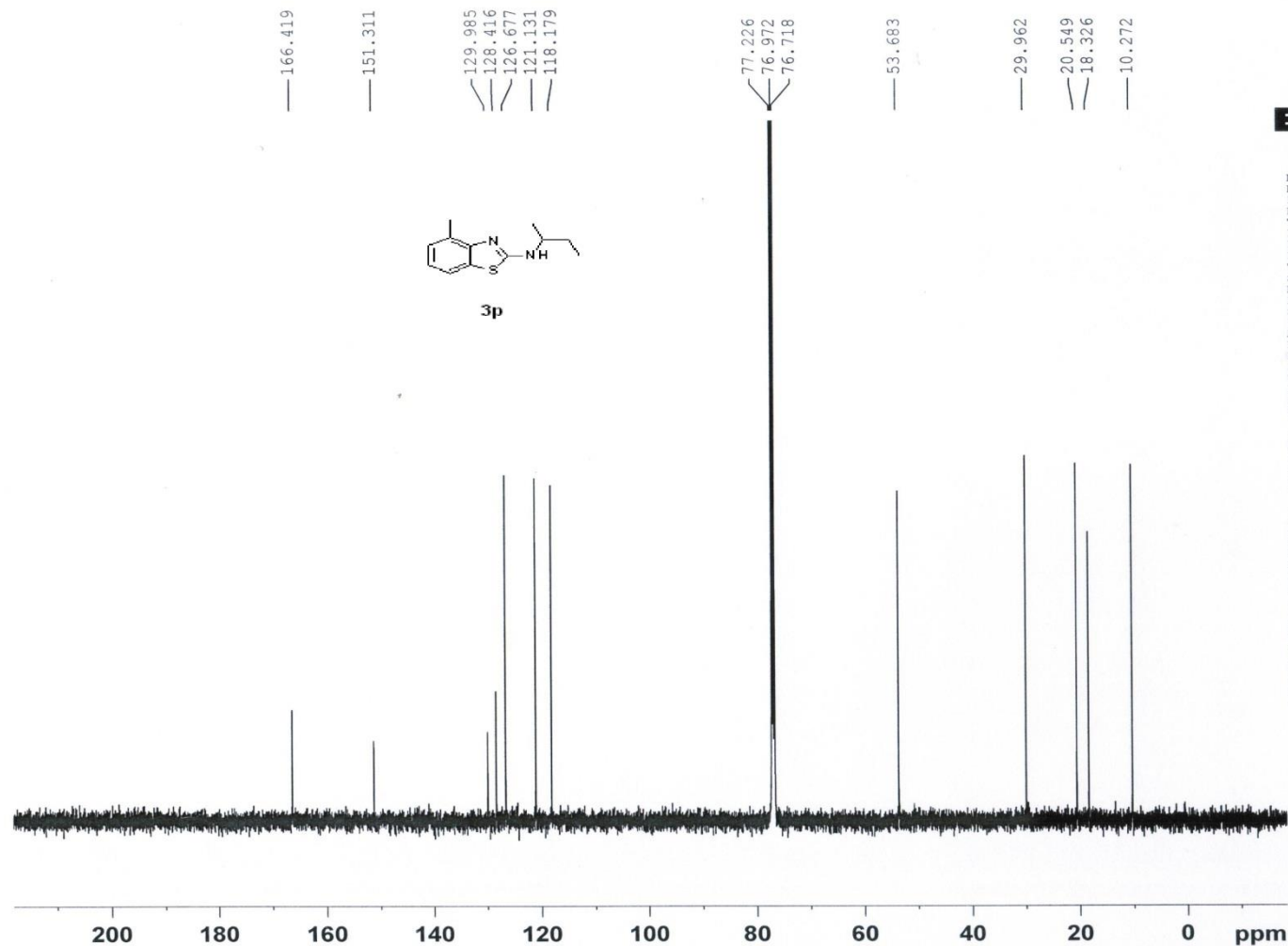
===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz
  
```

```

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



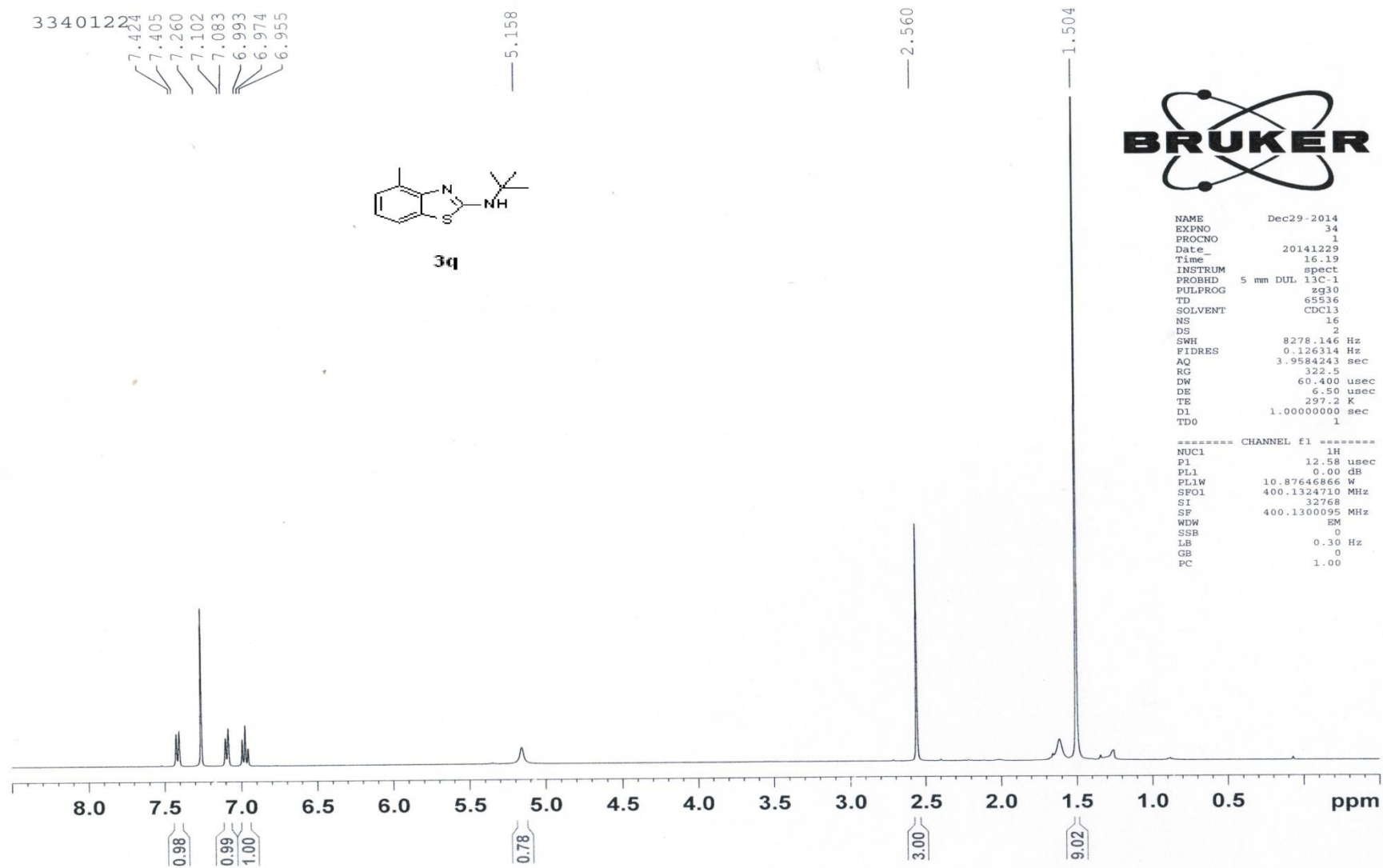
3340121



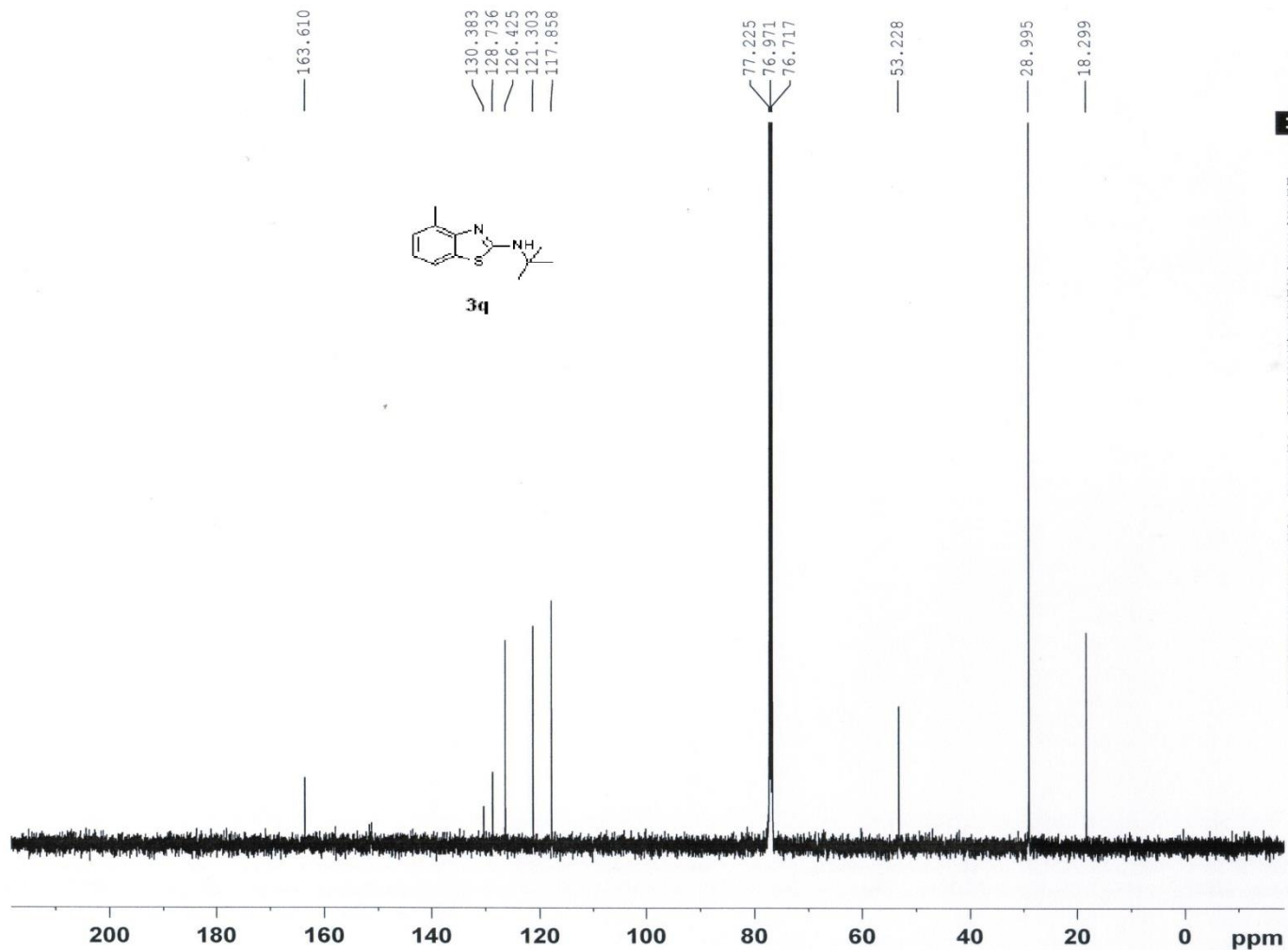
NAME 3340121
EXPNO 1
PROCNO 1
Date 20150112
Time 10.29
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1879
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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

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



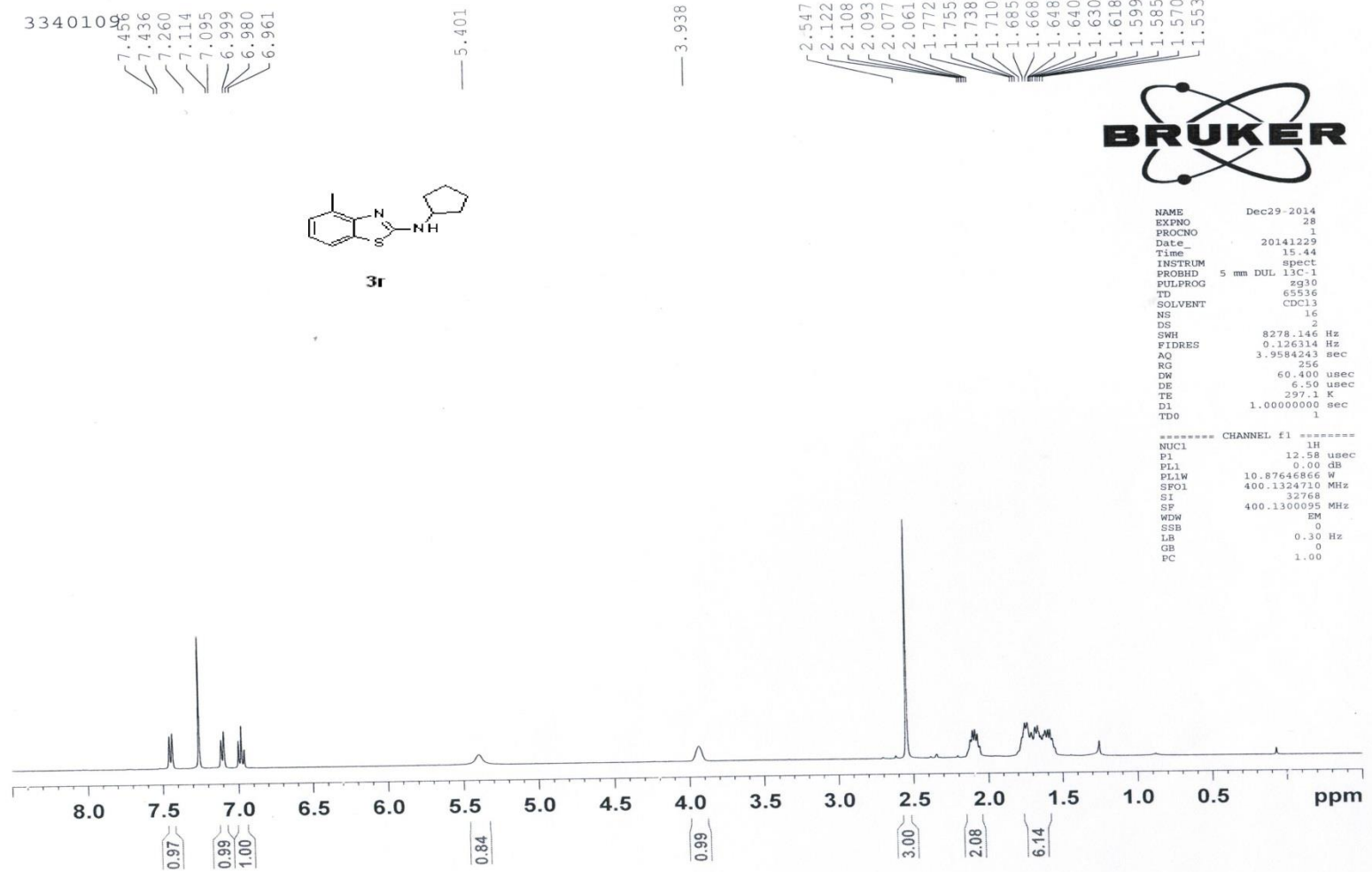
3340122



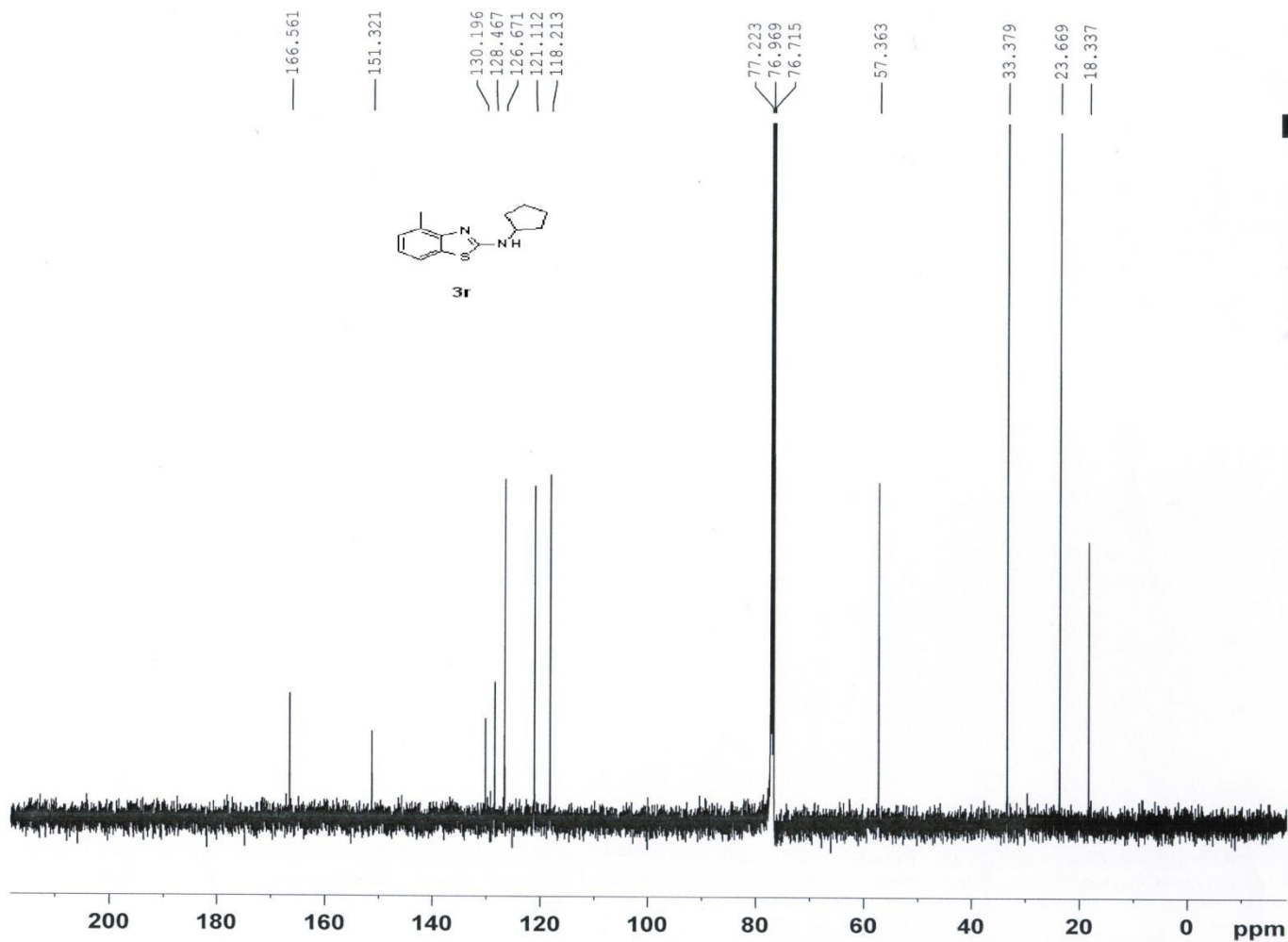
NAME 3340122
EXPNO 1
PROCNO 1
Date_ 20150108
Time_ 14.58
INSTRUM Spect
PROBHD 5 mm PABBO BB-
FULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

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

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
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



3340109



```

NAME      3340109
EXPNO     1
PROCNO    1
Date_     20150112
Time      14.18
INSTRUM    Spect
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         1519
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         298.7 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

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

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

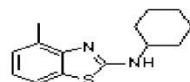
3340099

7.441
7.422
7.260
7.107
7.089
6.992
6.972
6.954

5.309
5.291

3.435
3.424
3.414

2.545
2.134
2.108
1.797
1.787
1.773
1.764
1.433
1.403
1.371
1.350
1.321
1.292
1.263

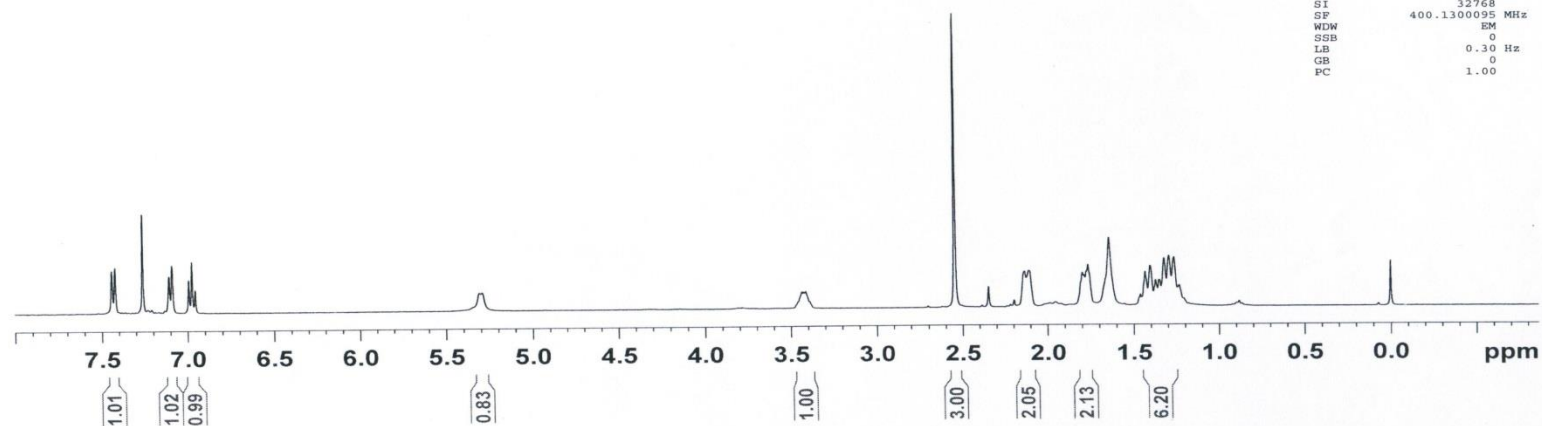


3s

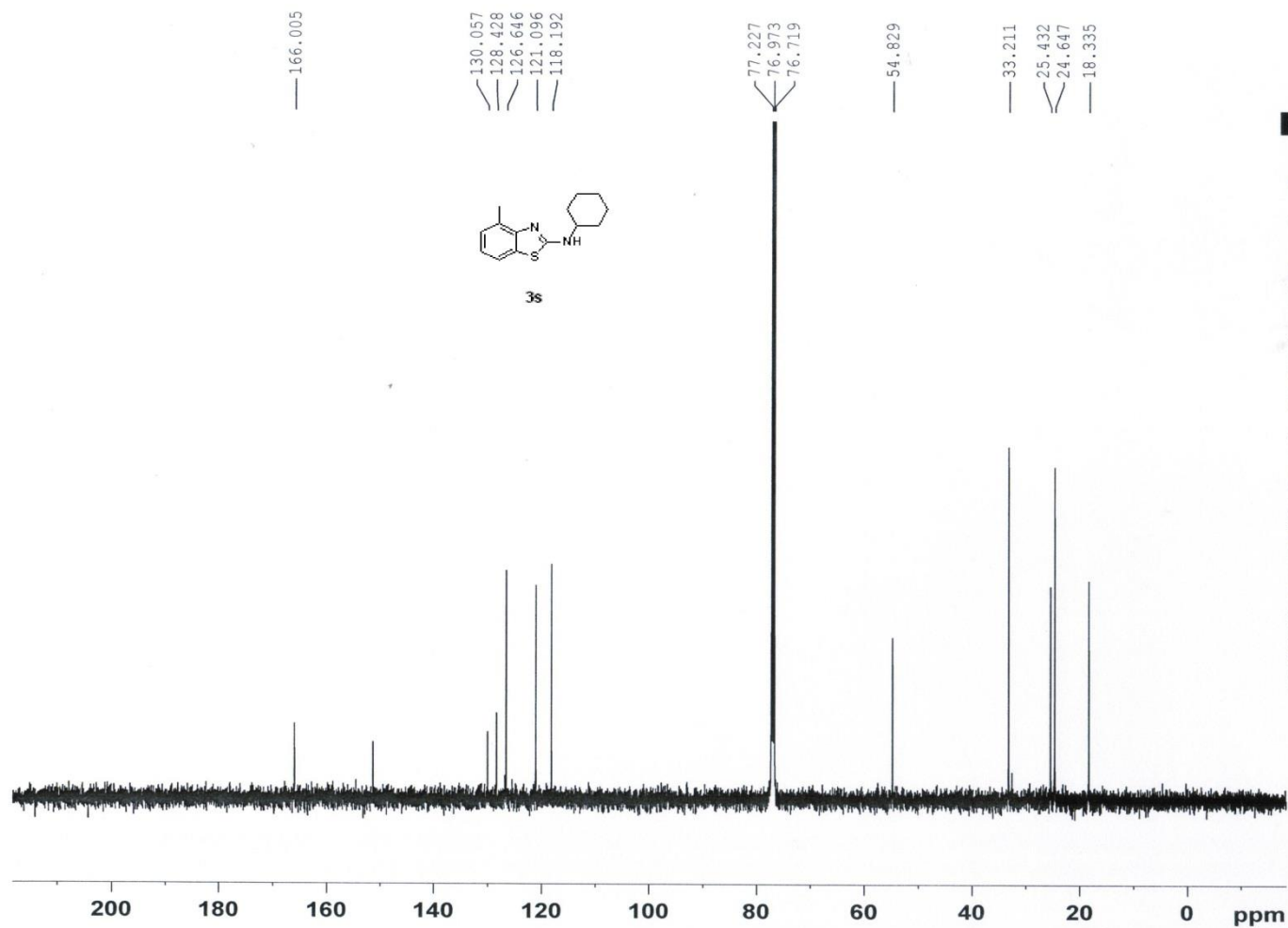


NAME Dec23-2014
EXPNO 41
PROCNO 1
Date_ 20141223
Time 14.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DM 60.400 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

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



3340099



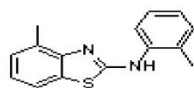
NAME 3340099
 EXPNO 1
 PROCNO 1
 Date_ 20141223
 Time 11.20
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 FULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 299.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

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

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 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

33401171

7.721
7.705
7.447
7.428
7.301
7.284
7.260
7.180
7.161
7.143
7.066
7.047
7.028



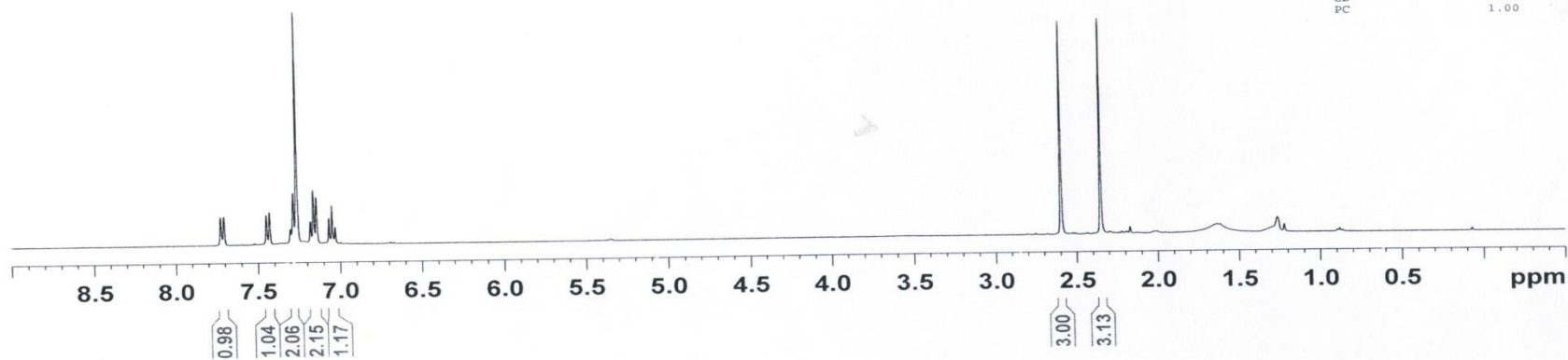
31

— 2.596
— 2.352

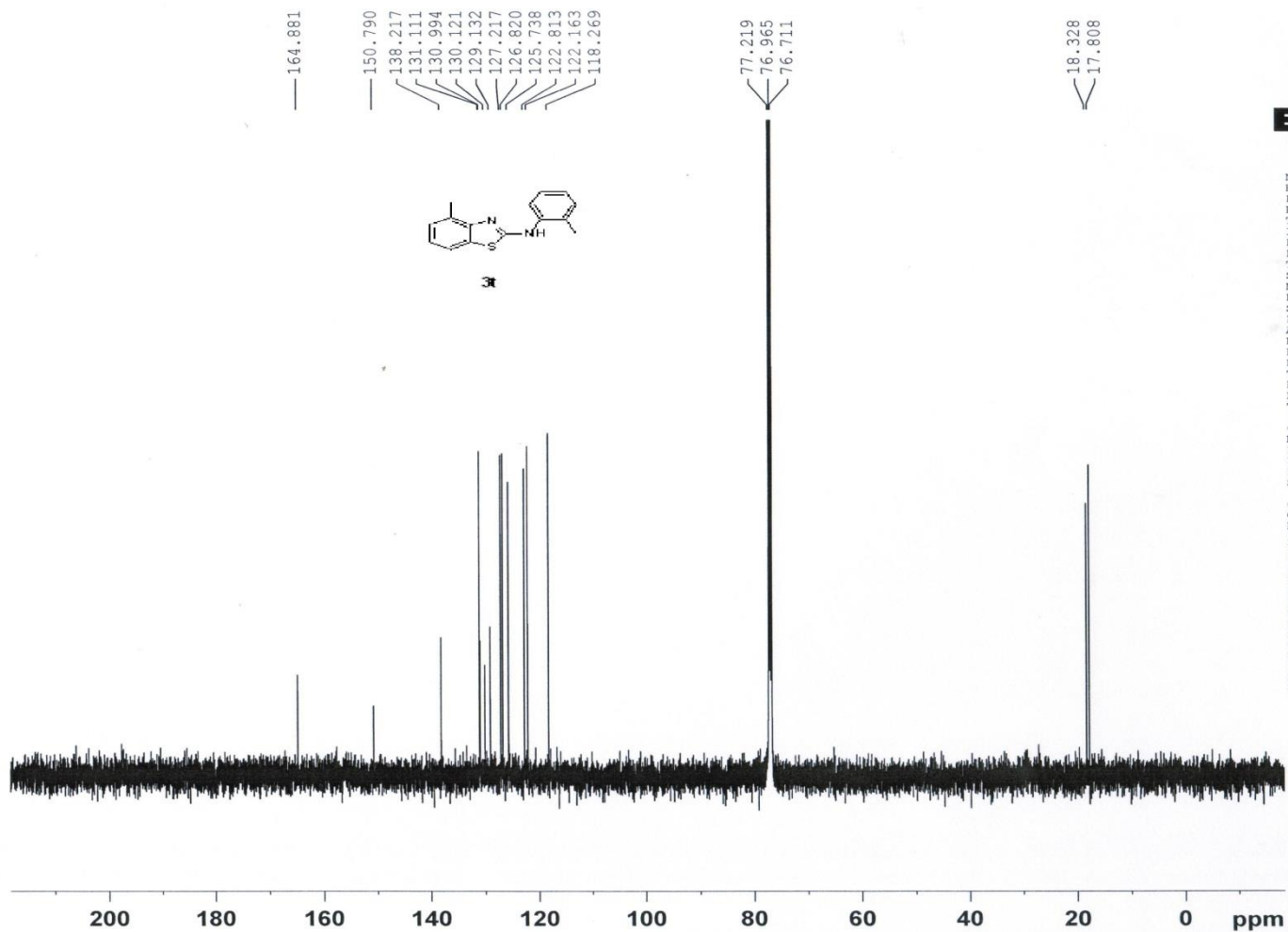


NAME Dec29-2014
EXPNO 29
PROCNO 1
Date_ 20141229
Time 15.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

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



33401171

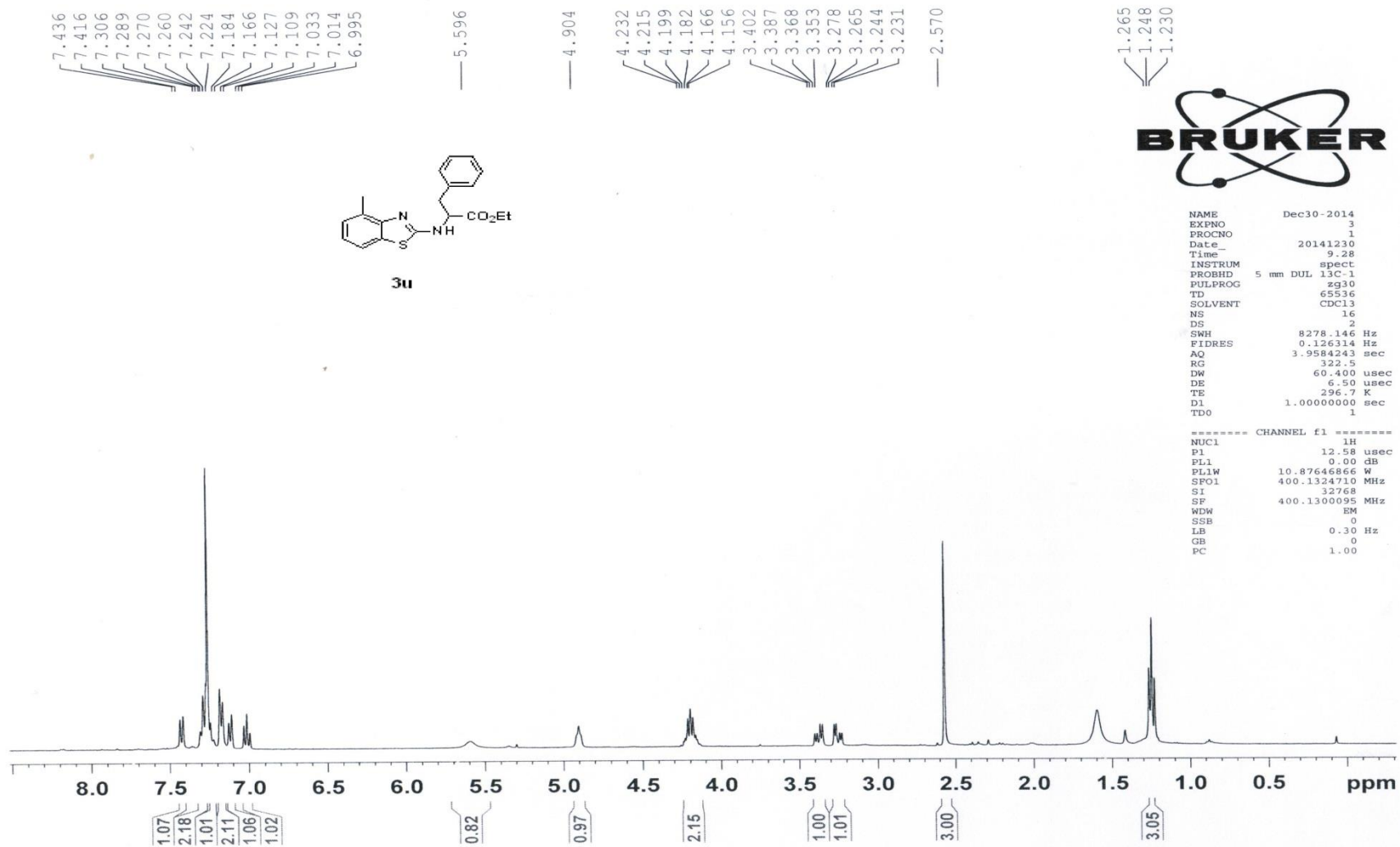


NAME 33401171
 EXPNO 1
 PROCNO 1
 Date 20150113
 Time 13.25
 INSTRUM Spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1289
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

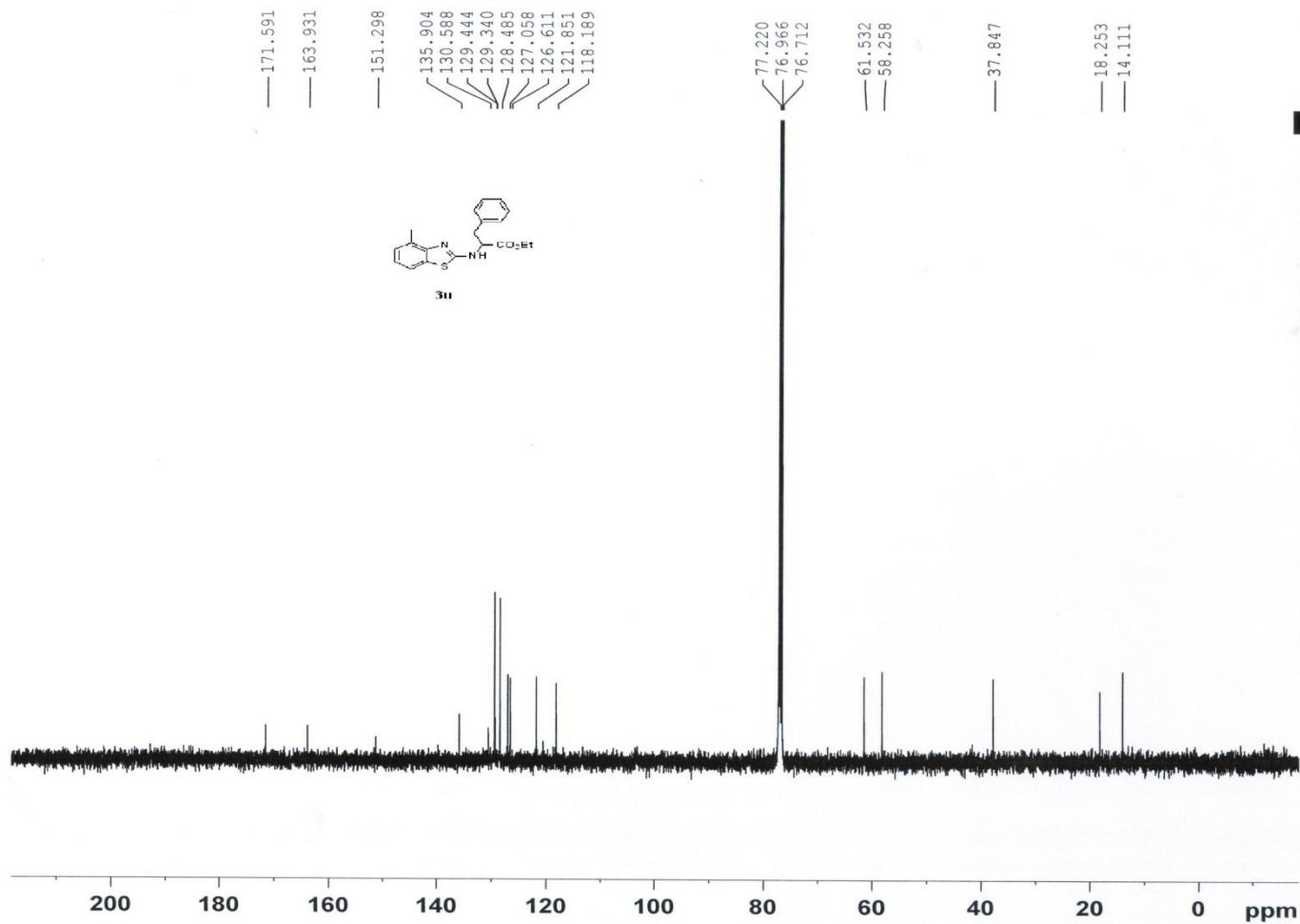
===== CHANNEL f1 =====
 NUC1 13C
 P1 13.84 usec
 PL1 2.50 dB
 PL1W 46.89624786 W
 SFO1 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 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

3340120



3340120



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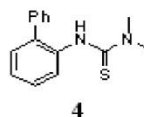
NAME 3340120
EXPNO 1
PROCNO 1
Date_ 20150113
Time 9.52
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1231
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
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3340187

7.764
7.744
7.428
7.412
7.379
7.362
7.333
7.314
7.280
7.260
6.804

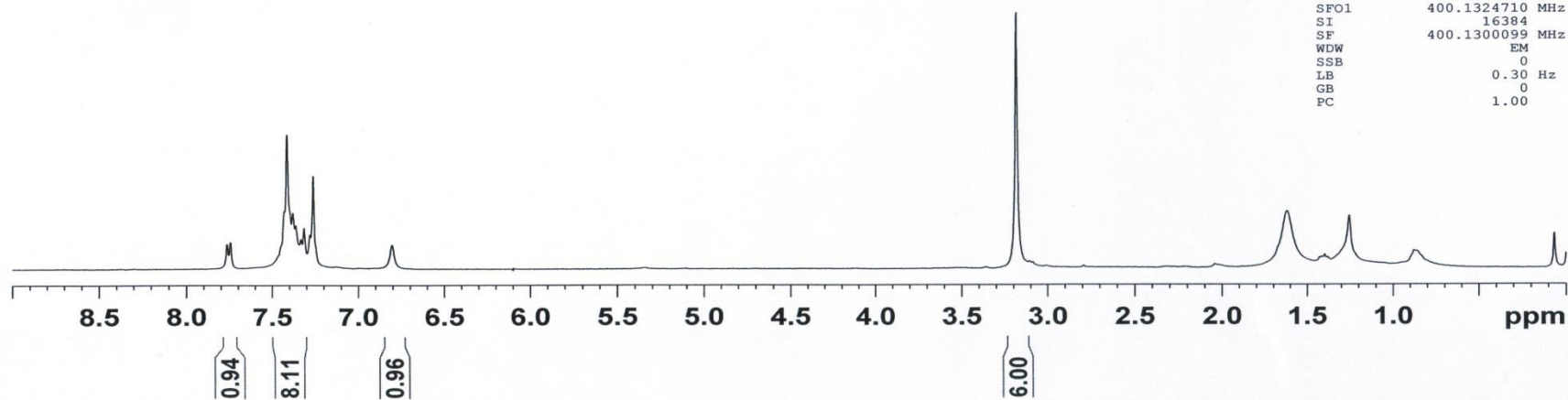


— 3.183

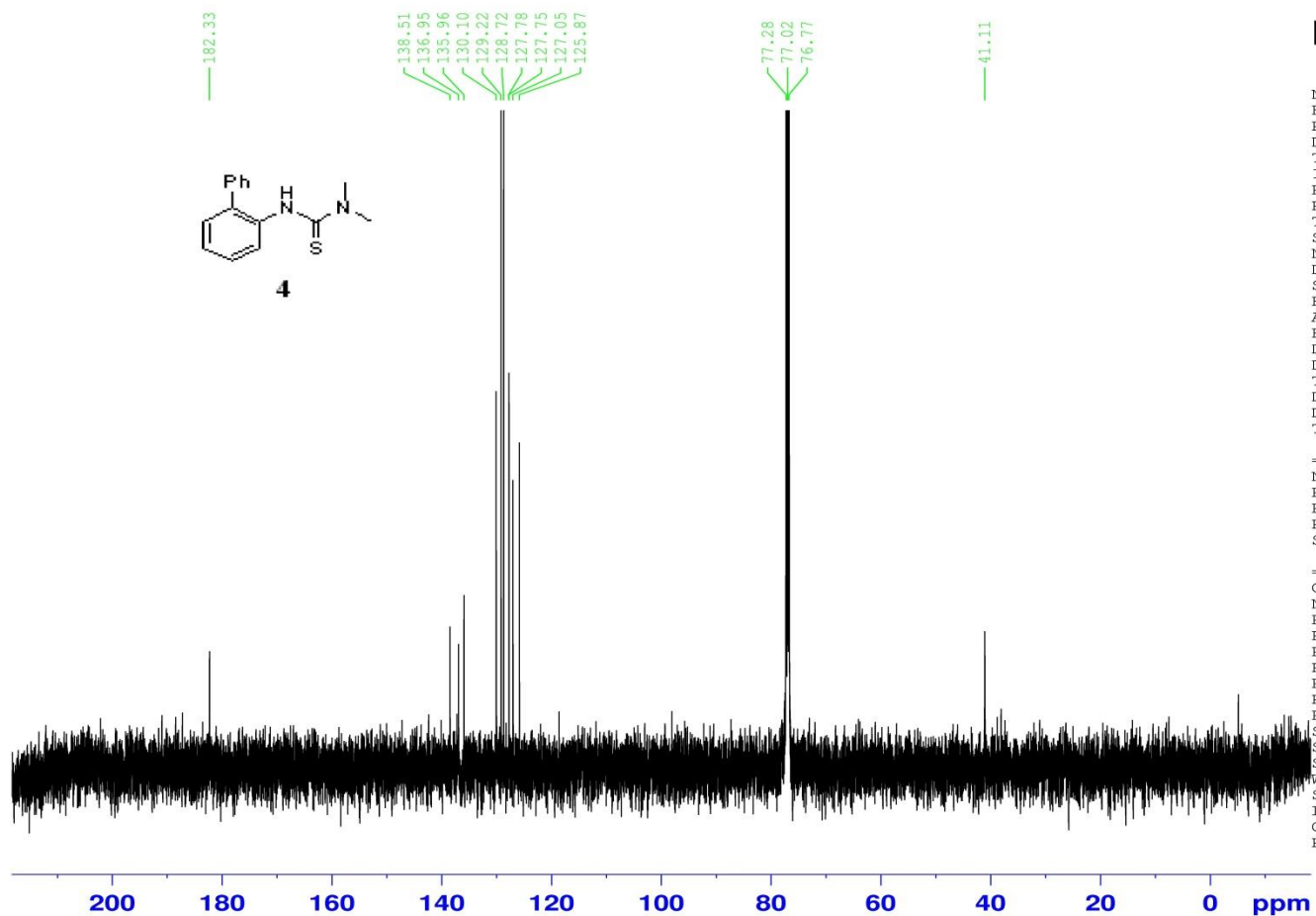


NAME Apr27-2016
EXPNO 60
PROCNO 1
Date 20160427
Time 17.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3340187



NAME Nov05-2015
 EXPNO 2
 PROCNO 1
 Date_ 20151105
 Time_ 10.35
 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 13.84 usec
 PL1 2.50 dB
 PL1W 46.89624786 W
 SFO1 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 2.50 dB
 PL12 17.40 dB
 PL13 17.40 dB
 PL2W 13.02359581 W
 PL12W 0.42143536 W
 PL13W 0.42143536 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577890 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40