Supporting Information

Synthesis of Biphenyl-2-carbonitrile Derivatives via a Palladium-Catalyzed sp^2 C-H Bond Activation Using Cyano as Directing Group

Wu Li, Zhipeng Xu, Peipei Sun*, Xiaoqing Jiang and Min Fang

Jiangsu Key Laboratory of Biofunctional Materials, College of Chemistry and Materials Science, Nanjing Normal University, Nanjing, 210097, China.

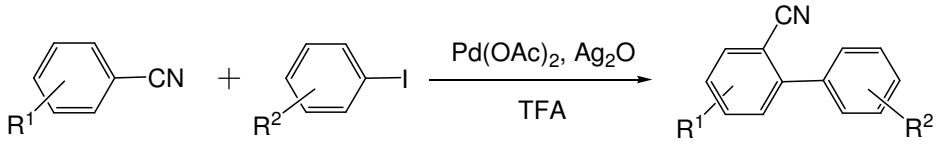
E-mail: sunpeipei@njnu.edu.cn

1. General

^{1}H NMR (400MHz) spectra were recorded on a Bruker Avance 400 spectrometers in $CDCl_3$ [using $(CH_3)_4Si$ (for ^{1}H, δ = 0.00) as internal standard]. ^{13}C NMR (100 MHz) spectra on a Bruker Avance 400 spectrometers in $CDCl_3$ [using $CDCl_3$ (for ^{13}C, δ = 77.00) as internal standard]. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, m = multiplet, br = broad. IR spectra were recorded on a Shimazu IR Prestige-21 FT-IR Spectrometer. Mass and high-resolution mass spectra were obtained with a Mariner ABI mass spectrometer. Melting points were uncorrected and were recorded on a Buchi B-54 melting point apparatus.

Flash column chromatography was performed using Qingdao Haiyang silica gel (300-400) with distilled solvents. All chemical reagents were purchased from Alfa Aesar or Sigma-Aldrich Co., Inc.

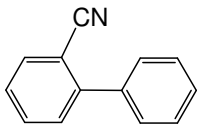
2. Synthesis of Biphenyl-2-carbonitrile Derivatives



A seal tube (15 mL) initially fitted with a septum containing $Pd(OAc)_2$ (22 mg, 0.10 mmol) and silver(I) oxide (231 mg, 1.00 mmol) was evacuated and purged with nitrogen gas three times. Trifluoroacetic acid (2.0 mL), aryl nitrile (1.00 mmol) and aryl iodide (2.00 mmol) were added to the system and the reaction mixture was stirred at 110 °C for 9 h. The mixture was filtered through a short Celite pad and washed with dichloromethane several times. The filtrate was concentrated by vacuum and separated on a silica gel column using hexane/EtOAc as eluent to give the corresponding pure biphenyl-2-carbonitrile derivatives.

The spectral data and a copy of ^{1}H and ^{13}C NMR spectra of all compounds are listed below.

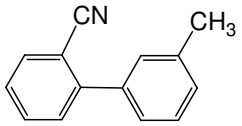
biphenyl-2-carbonitrile (**3aa**)



Pale yellow oil;[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ7.46-7.54 (m, 5H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.66

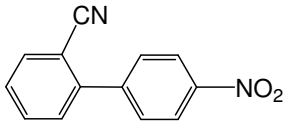
(m, 1 H), 7.78 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.3, 118.7, 127.5, 128.3, 128.6, 128.7, 130.1, 132.4, 132.8, 133.7, 138.1, 145.5; MS (ESI) m/z 179.2 (M^+); IR (KBr): 2227, 1610, 1495 cm^{-1}.

3'-methylbiphenyl-2-carbonitrile (**3ab**)



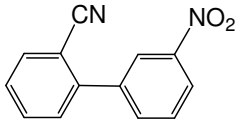
Pale yellow oil;[2] ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.44 (s, 3H), 7.29 (s, 1H), 7.36-7.47 (m, 4H), 7.50 (d, J = 8.0 Hz, 1H), 7.63 (t, J = 7.2 Hz, 1H), 7.74 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4, 111.3, 118.7, 125.8, 127.4, 128.6, 128.9, 129.4, 130.3, 132.7, 133.7, 138.1, 138.4, 145.6; MS (ESI) m/z 193.2 (M^+); IR (KBr): 2963, 2228, 1615, 1489 cm^{-1}.

4'-nitrobiphenyl-2-carbonitrile (**3ac**)



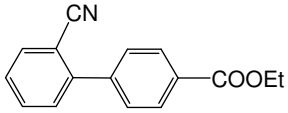
Pale yellow solid, mp 128-129 °C (lit.[3] 127-128 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56-7.59 (m, 2H), 7.73-7.77 (m, 3H), 7.83 (d, J = 8.0 Hz, 1H), 8.34 (d, J = 8.8 Hz, 1H), 8.35 (d, J = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.2, 118.1, 124.0, 129.0, 129.9, 130.0, 133.3, 134.0, 142.8, 144.4, 147.9; MS (ESI) m/z 224.1 (M^+); IR (KBr): 2227, 1617, 1525, 1489, 1342 cm^{-1}.

3'-nitrobiphenyl-2-carbonitrile (**3ad**)



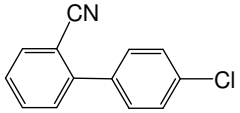
Pale yellow solid,[4] mp 155-156 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, J = 8.0 Hz, 2H), 7.72-7.75 (m, 2H), 7.85 (d, J = 8.0 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 8.34 (d, J = 8.0 Hz, 1H), 8.43 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.4, 118.0, 123.6, 123.8, 128.8, 129.9, 130.1, 133.3, 133.9, 134.8, 139.7, 142.8, 148.4; MS (ESI) m/z 224.1 (M^+); IR (KBr): 2228, 1615, 1523, 1490, 1341 cm^{-1}.

2'-cyanobiphenyl-4-carboxylic acid ethyl ester (**3ae**)



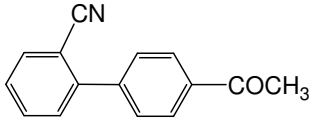
Pale yellow solid, mp 107-108 °C (lit.[5] 112 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43 (t, J = 7.2 Hz, 3H), 4.43 (q, J = 7.2 Hz, 2H), 7.49-7.56 (m, 2H), 7.65 (d, J = 8.0 Hz, 2H), 7.67-7.71 (m, 1H), 7.80 (d, J = 8.0 Hz, 1H), 8.19 (d, J =8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.3, 61.2, 111.3, 118.4, 128.2, 128.8, 130.0, 130.7, 133.0, 133.9, 142.4, 144.3, 166.1; MS (ESI) m/z 251.1 (M^+); IR (KBr): 2226, 1728, 1617, 1487, 1280, 1182 cm^{-1}.

4'-chlorobiphenyl-2-carbonitrile (**3af**)



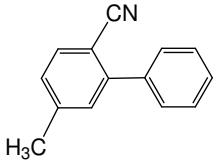
Pale yellow solid, mp 112-113 ^{o}C (lit.[6] 115-116 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.55 (m, 6H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.2, 118.5, 127.9, 129.0, 129.9, 130.1, 133.0, 133.8, 135.1, 136.5, 144.2; MS (ESI) m/z 213.1 (M^+); IR (KBr): 2230, 1613, 1495, 755 cm^{-1}.

4'-acetylbiphenyl-2-carbonitrile (**3ag**)



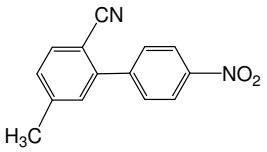
Pale yellow solid,[1] mp 70 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.65 (s, 3H), 7.48-7.54 (m, 2H), 7.65-7.70 (m, 3H), 7.79 (d, *J* = 8.0 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.7, 111.3, 118.3, 127.4, 128.3, 128.7, 129.0, 129.1, 130.0, 133.0, 133.9, 137.0, 142.6, 144.2, 197.5; MS (ESI) m/z 221.1 (M^+); IR (KBr): 2985, 2232, 1689, 1610, 1495 cm^{-1}.

5-methylbiphenyl-2-carbonitrile (**3ba**)



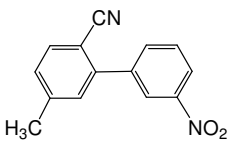
Pale yellow oil;[7] ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.48 (s 3H), 7.26-7.33 (m, 2H), 7.46-7.51 (m, 3H), 7.55-7.58 (m, 2H), 7.65 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.5, 108.0, 118.7, 128.1, 128.3, 128.4, 128.7, 129.4, 130.5, 133.4, 138.0, 143.4, 145.1, 146.5; MS (ESI) m/z 193.1 (M^+); IR (KBr): 2983, 2230, 1612, 1488 cm^{-1}.

5-methyl-4'-nitrobiphenyl-2-carbonitrile (**3bc**)



Pale yellow solid, mp 132-133 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.52 (s, 3H), 7.36-7.38 (m, 2H), 7.71-7.75 (m, 3H), 8.35 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.9, 108.2, 118.3, 123.9, 129.8, 130.7, 133.9, 142.8, 144.4, 144.6, 147.9; HRMS (ESI) calcd for $C_{14}H_{10}N_2O_2$[Na] 261.0634, found 261.0640; IR (KBr): 2993, 2228, 1607, 1523, 1489, 1345 cm^{-1}.

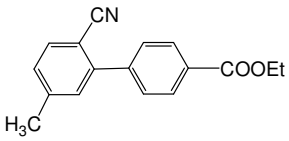
5-methyl-3'-nitrobiphenyl-2-carbonitrile (**3bd**)



Pale yellow solid, mp 146-147 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.50 (s, 3H), 7.34-7.36 (m, 2H),

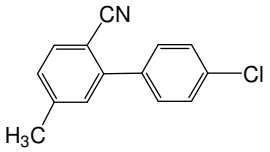
7.66-7.71 (m, 2H), 7.93 (d, *J* = 8.0 Hz, 1H), 8.30 (d, *J* = 8.0 Hz, 1H), 8.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.9, 108.3, 118.3, 123.4, 123.7, 129.6, 129.8, 130.8, 133.8, 134.8, 139.8, 142.7, 144.4, 148.4; HRMS (ESI) calcd for $C_{14}H_{10}N_2O_2$[Na] 261.0634, found 261.0622; IR (KBr): 2989, 2227, 1605, 1530, 1490, 1342 cm^{-1}.

2'-cyano-5'-methylbiphenyl-4-carboxylic acid ethyl ester (**3be**)



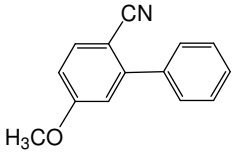
Pale yellow solid, mp 106-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.41 (t, *J* = 7.2 Hz, 3H), 2.47 (s, 3H), 4.40 (q, *J* = 7.2 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 1H), 7.33 (s, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 1H), 8.15 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.3, 21.8, 61.2, 108.2, 118.7, 128.8, 129.0, 129.9, 130.5, 130.7, 133.7, 142.5, 144.0, 144.2, 166.2; HRMS (ESI) calcd for $C_{17}H_{15}NO_2$[Na] 288.0995, found 288.1001; IR (KBr): 2953, 2228, 1725, 1612, 1489, 1280, 1132 cm^{-1}.

5-methyl-4'-chlorobiphenyl-2-carbonitrile (**3bf**)



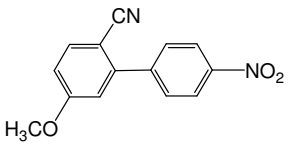
Pale yellow solid, mp 88-89 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.49 (s, 3H), 7.29-7.31 (m, 2H), 7.46-7.52 (m, 4H), 7.67 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.9, 108.2, 118.8, 128.7, 128.9, 130.1, 130.7, 133.7, 134.9, 136.7, 143.9, 144.1; HRMS (ESI) calcd for $C_{14}H_{10}NCl$[H] 228.0575, found 228.0568; IR (KBr): 2955, 2226, 1617, 1489, 758 cm^{-1}.

5-methoxylbiphenyl-2-carbonitrile (**3ca**)



Pale yellow solid, mp 82-83 °C (lit.[8] 85-86 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.92 (s, 3H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.01, (s, 1H), 7.47-7.51 (m, 3H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.2, 102.8, 113.2, 114.5, 115.2, 118.8, 128.3, 128.4, 128.5, 128.6, 135.1, 137.9, 147.3, 162.4; MS (ESI) m/z 209.1 (M^+); IR (KBr): 2963, 2228, 1615, 1487 cm^{-1}.

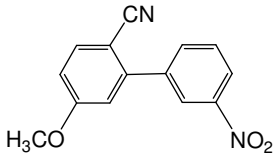
5-methoxyl-4'-nitrobiphenyl-2-carbonitrile (3**cc**)



Pale yellow solid, mp 165-166 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.95 (s, 3H), 7.02-7.07 (m, 2H),

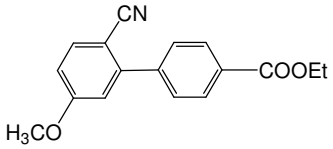
7.74-7.78 (m, 3H), 8.37 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.8, 103.0, 114.3, 115.9, 118.4, 124.0, 129.8, 135.8, 144.4, 145.0, 148.0, 162.9; HRMS (ESI) calcd for $C_{14}H_{10}N_2O_3$[Na] 277.0584, found 277.0573; IR (KBr): 2962, 2227, 1605, 1528, 1490, 1345 cm^{-1}.

5-methoxyl-3'-nitrobiphenyl-2-carbonitrile (3**cd**)



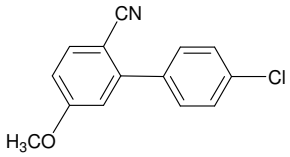
Pale yellow solid, mp 153-154 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.96 (s, 3H), 7.04-7.07 (m, 2H), 7.70-7.78 (m, 2H), 7.95 (d, *J* = 6.8 Hz, 1H), 8.34 (d, *J* = 6.8 Hz, 1H), 8.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.8, 103.1, 114.3, 115.8, 118.3, 123.6, 123.7, 129.8, 134.7, 135.7, 139.7, 144.8, 148.4, 163.0; HRMS (ESI) calcd for $C_{14}H_{10}N_2O_3$[Na] 277.0584, found 277.0577; IR (KBr): 2968, 2225, 1608, 1526, 1488, 1346 cm^{-1}.

2'-cyano-5'-methoxylbiphenyl-4-carboxylic acid ethyl ester (3**ce**)



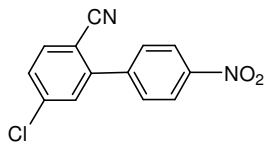
Pale yellow solid, mp 85-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.43 (t, *J* = 7.2 Hz, 3H), 3.91 (s, 3H), 4.42 (q, *J* = 7.2 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 1H), 7.00 (s, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 1H), 8.17 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.3, 55.7, 61.2, 103.0, 113.9, 115.6, 118.7, 128.7, 129.9, 130.7, 135.6, 142.4, 146.4, 162.8, 166.1; HRMS (ESI) calcd for $C_{17}H_{15}NO_3$[Na] 304.0944, found 304.0955; IR (KBr): 2976, 2224, 1723, 1612, 1490, 1283, 1128 cm^{-1}.

5-methoxyl-4'-chlorobiphenyl-2-carbonitrile (3**cf**)



Pale yellow solid, mp 107-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.92 (s, 3H), 6.97-9.99 (m, 2H), 7.47-7.53 (m, 4H), 7.72 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.7, 103.0, 113.6, 115.5, 118.9, 129.0, 130.0, 135.1, 135.5, 136.6, 146.3, 162.8; HRMS (ESI) calcd for $C_{14}H_{10}ClNO$[H] 244.0524, found 244.0521; IR (KBr): 2975, 2226, 1615, 1493, 765 cm^{-1}.

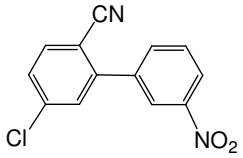
5-chloro-4'-nitrobiphenyl-2-carbonitrile (3**dc**)



Pale yellow solid, mp 187-188 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 7.2 Hz, 1H), 7.58 (s, 1H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.79 (d, *J* = 7.2 Hz, 1H), 8.40 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100

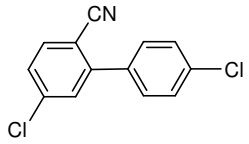
MHz, $CDCl_3$): δ 109.7, 117.2, 124.2, 129.3, 129.8, 130.3, 135.1, 139.9, 143.2, 144.5; HRMS (ESI) calcd for $C_{13}H_7ClN_2O_2$[Na] 281.0088, found 280.9993; IR (KBr): 2223, 1624, 1522, 1491, 1349, 760 cm^{-1}.

5-chloro-3'-nitrobiphenyl-2-carbonitrile (3**dd**)



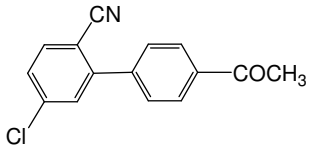
Pale yellow solid, mp 210-211 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, J = 8.0 Hz, 1H), 7.59 (s, 1H), 7.74-7.80 (m, 2H), 7.94 (d, J = 8.0 Hz, 1H), 8.38 (d, J = 8.4 Hz, 1H), 8.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 109.8, 117.2, 123.7, 124.1, 129.2, 130.1, 130.3, 134.6, 135.0, 138.3, 139.9, 144.3, 148.5; HRMS (ESI) calcd for $C_{13}H_7ClN_2O_2$[Na] 281.0088, found 281.0094; IR (KBr): 2226, 1620, 1526, 1492, 1347, 762 cm^{-1}.

5-chloro-4'-chlorobiphenyl-2-carbonitrile (3**df**)



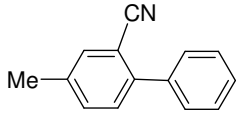
Pale yellow solid, mp 148-149 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45-7.49 (m, 6H), 7.71 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 109.6, 117.7, 128.3, 129.2, 130.0, 130.2, 134.9, 135.2, 135.7, 139.5, 145.8; HRMS (ESI) calcd for $C_{13}H_7Cl_2N$[Na] 269.9948, found 269.9954; IR (KBr): 2225, 1623, 1482, 768 cm^{-1}.

5-chloro-4'-acetylbiphenyl-2-carbonitrile (3**dg**)



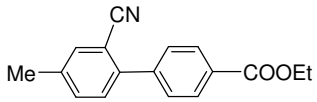
Pale yellow solid, mp 108-109 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.69 (s, 3H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.56 (s, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.8, 109.7, 117.6, 128.3, 128.7, 129.0, 129.9, 130.2, 131.5, 134.9, 137.4, 139.6, 141.2, 145.8, 197.4; HRMS (ESI) calcd for $C_{15}H_{10}ClNO$[Na] 278.0343, found 278.0340; IR (KBr): 2953, 2226, 1685, 1623, 1489, 765 cm^{-1}.

4-methylbiphenyl-2-carbonitrile (**3ea**)



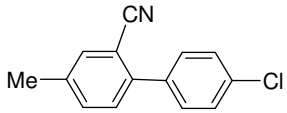
Pale yellow solid, mp 76-77 °C (lit.[9] 79-80 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.45 (s 3H), 7.45-7.52 (m, 5H), 7.56-7.59 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.5, 110.7, 118.7, 128.2, 128.4, 129.7, 133.5, 133.7, 137.4, 137.8, 138.9, 142.4; MS (ESI) m/z 193.1 (M^+); IR (KBr): 2982, 2232, 1615, 1488 cm^{-1}.

2'-cyano-4'-methylbiphenyl-4-carboxylic acid ethyl ester (**3ee**)



Pale yellow solid, mp 127-128 ^{o}C (lit.[10] 133-134 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.44 (t, *J* = 7.2 Hz, 3H), 2.47 (s, 3H), 4.43 (q, *J* = 7.2 Hz, 2H), 7.43-7.51 (m, 2H), 7.62-7.65 (m, 3H), 8.17 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 20.5, 60.9, 110.7, 118.3, 128.5, 129.6, 130.1, 133.6, 133.9, 138.2, 141.2, 142.1, 165.9; MS (ESI) m/z 265.1 (M^+); IR (KBr): 2950, 2228, 1722, 1615, 1491, 1280, 1128 cm^{-1}.

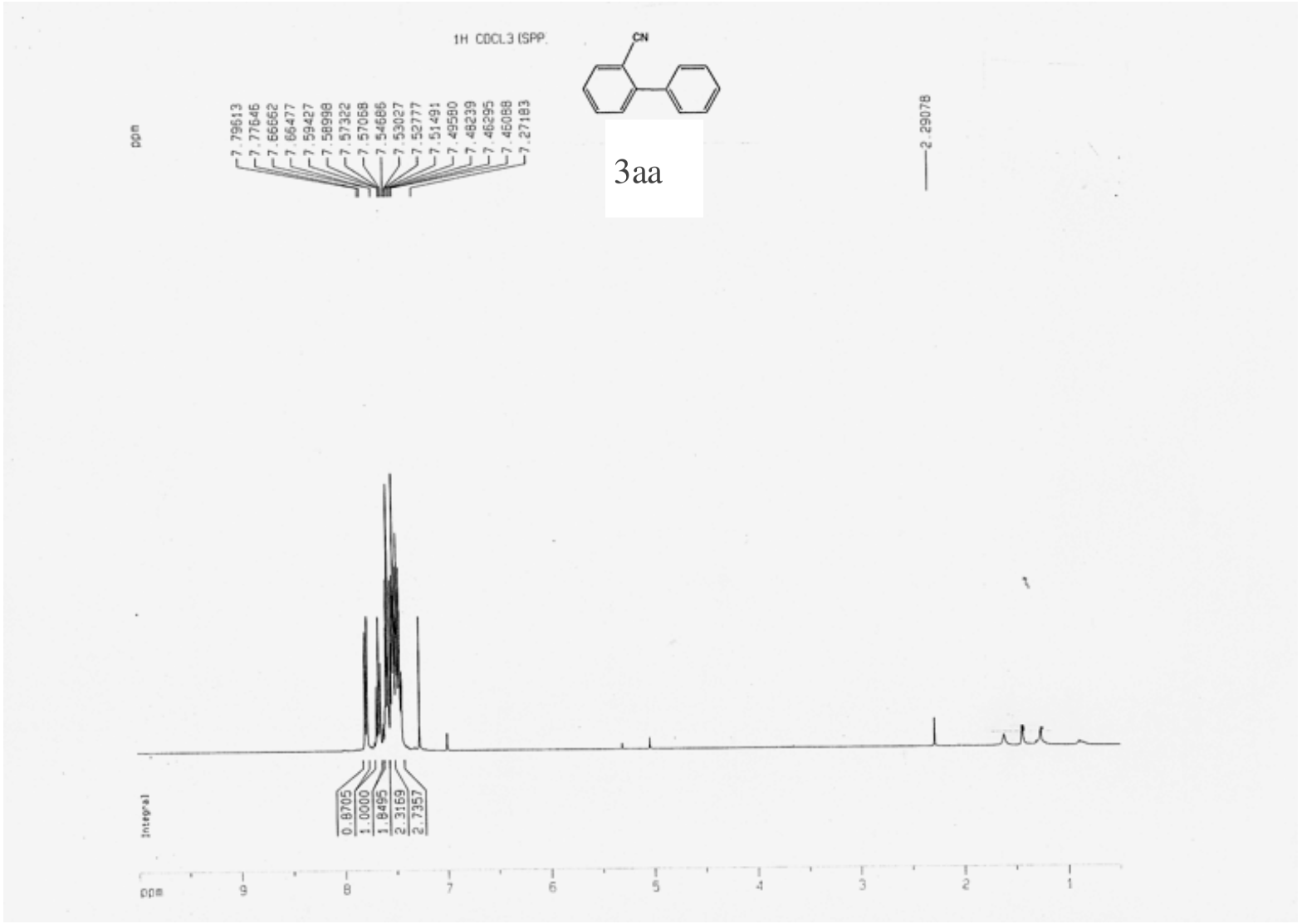
4-methyl-4'-chlorobiphenyl-2-carbonitrile (**3ef**)

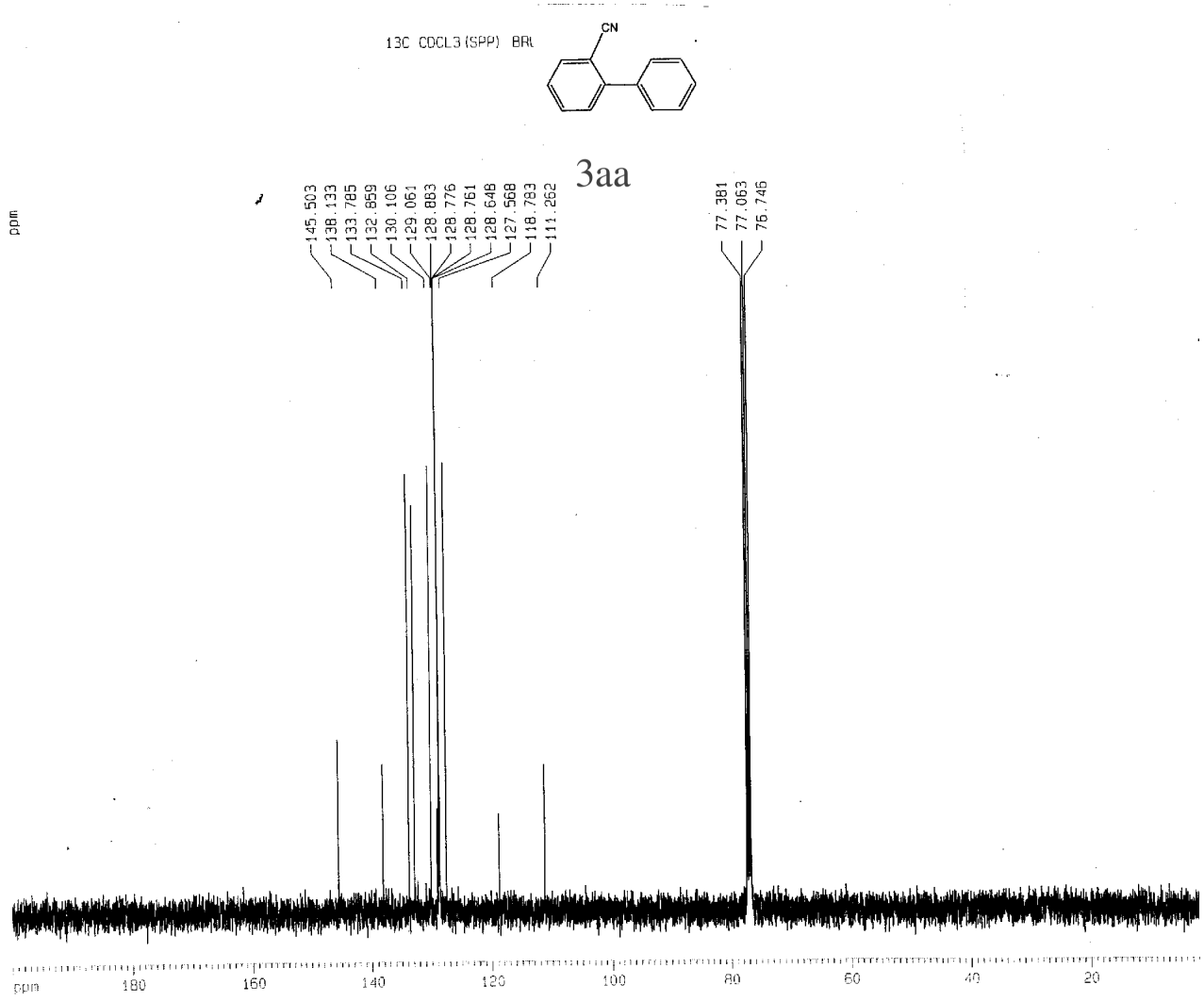


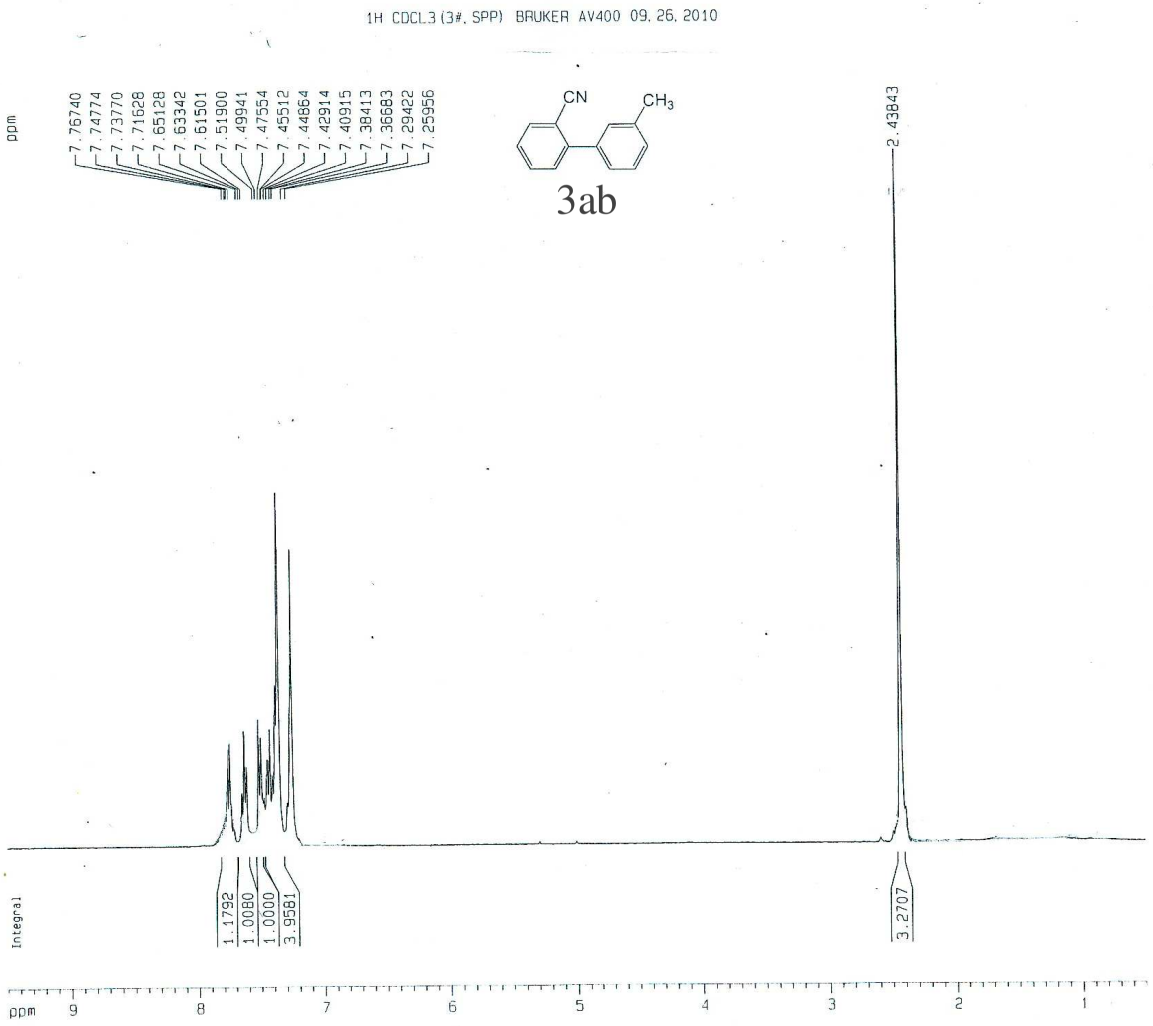
Pale yellow solid, mp 107-108 ^{o}C (lit.[10] 112-113 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.46 (s, 3H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.46-7.51 (m, 5H), 7.59 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.5, 110.6, 118.4, 128.7, 129.5, 129.7, 133.6, 133.8, 134.4, 136.2, 137.8, 141.1; MS (ESI) m/z 227.1 (M^+); IR (KBr): 2955, 2225, 1618, 1489, 756 cm^{-1}.

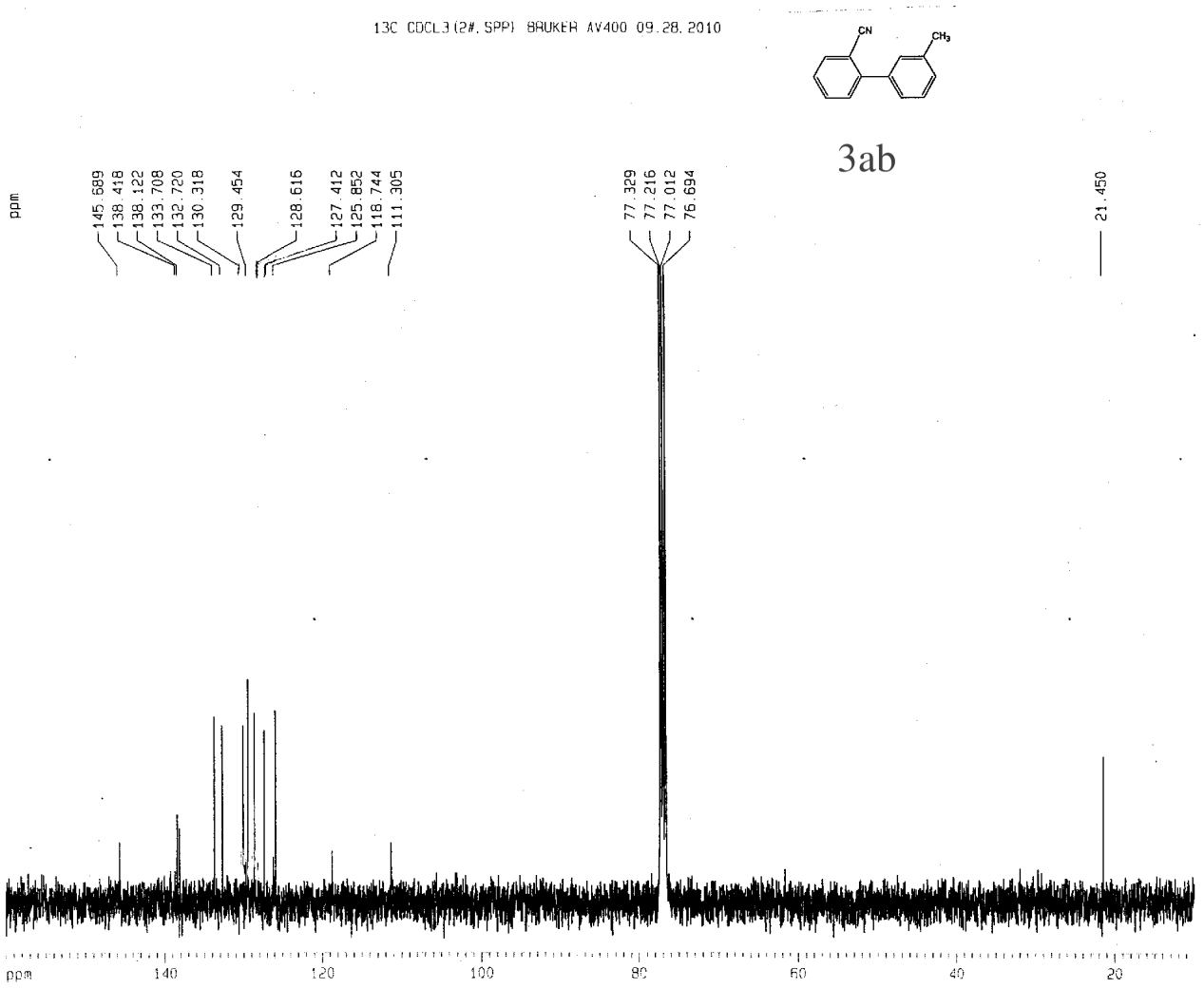
References:

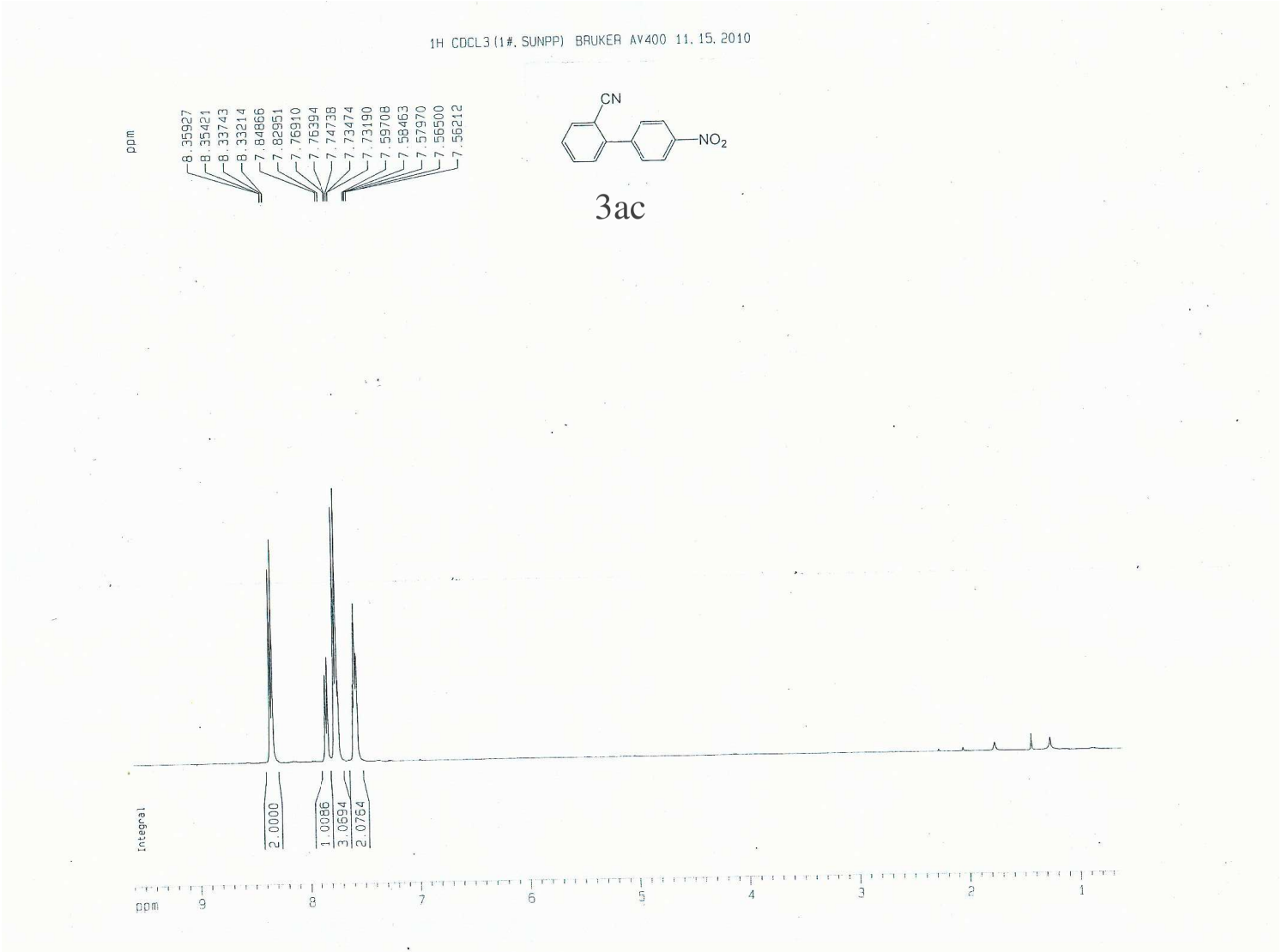
1. Bolliger, J. L.; Frech, C. M. *Adv. Synth. Catal.* **2010**, *352*, 1075.
2. Lipshutz, B. H.; Blomgren, P. A. *J. Am. Chem. Soc.* **1999**, *121*, 5819.
3. Kristensen, J.; Lysen, M.; Vedso, P.; Begtrup, M. *Org. Lett*. **2001**, *3*, 1435.
4. Stevens, M. F. G. *J. Chem. Soc., C* **1968**, 348.
5. Amatore, M.; Gosmini, C. *Angew. Chem., Int. Ed.* **2008**, *47*, 2089.
6. Sain, B.; Sandhu, J. S. *J. Org. Chem*. **1990**, *55*, 2545.
7. Fringuelli, F.; Girotti, R.; Piermatti, O.; Pizzo, F.; Vaccaro, L. *Org. Lett*. **2006**, *8*, 5741.
8. Bradsher, C. K.; Brown, F. C.; Porter, H. K. *J. Am. Chem. Soc.* **1954**, *76*, 2357.
9. Fisher, T. H.; Meierhoefer, A. W. *J. Org. Chem*. **1978**, *43*, 220.
10. Shabashov, D.; Maldonado, J. R. M.; Daugulis, O. *J. Org. Chem.* **2008**, *73*, 7818.

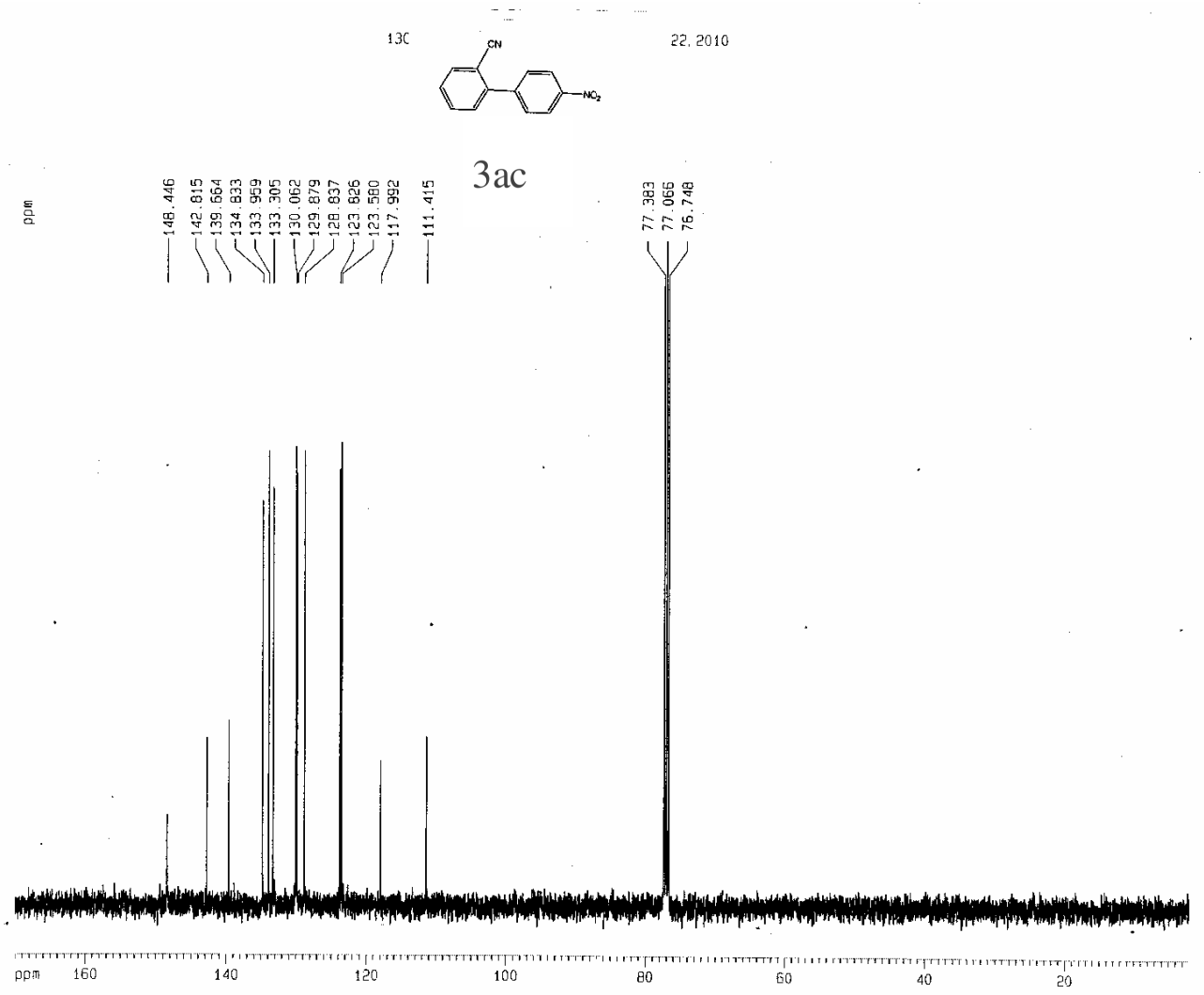

1H CDCL3 (SPP.
CN
3aa
ppm
7.79613
7.77646
7.66662
7.66477
7.59427
7.58998
7.57322
7.57068
7.54686
7.53027
7.52777
7.51491
7.49580
7.48239
7.46295
7.46088
7.27183
2.29078
Integral
0.8705
1.0000
1.8495
2.3169
2.7357
ppm
9
8
7
6
5
4
3
2
1

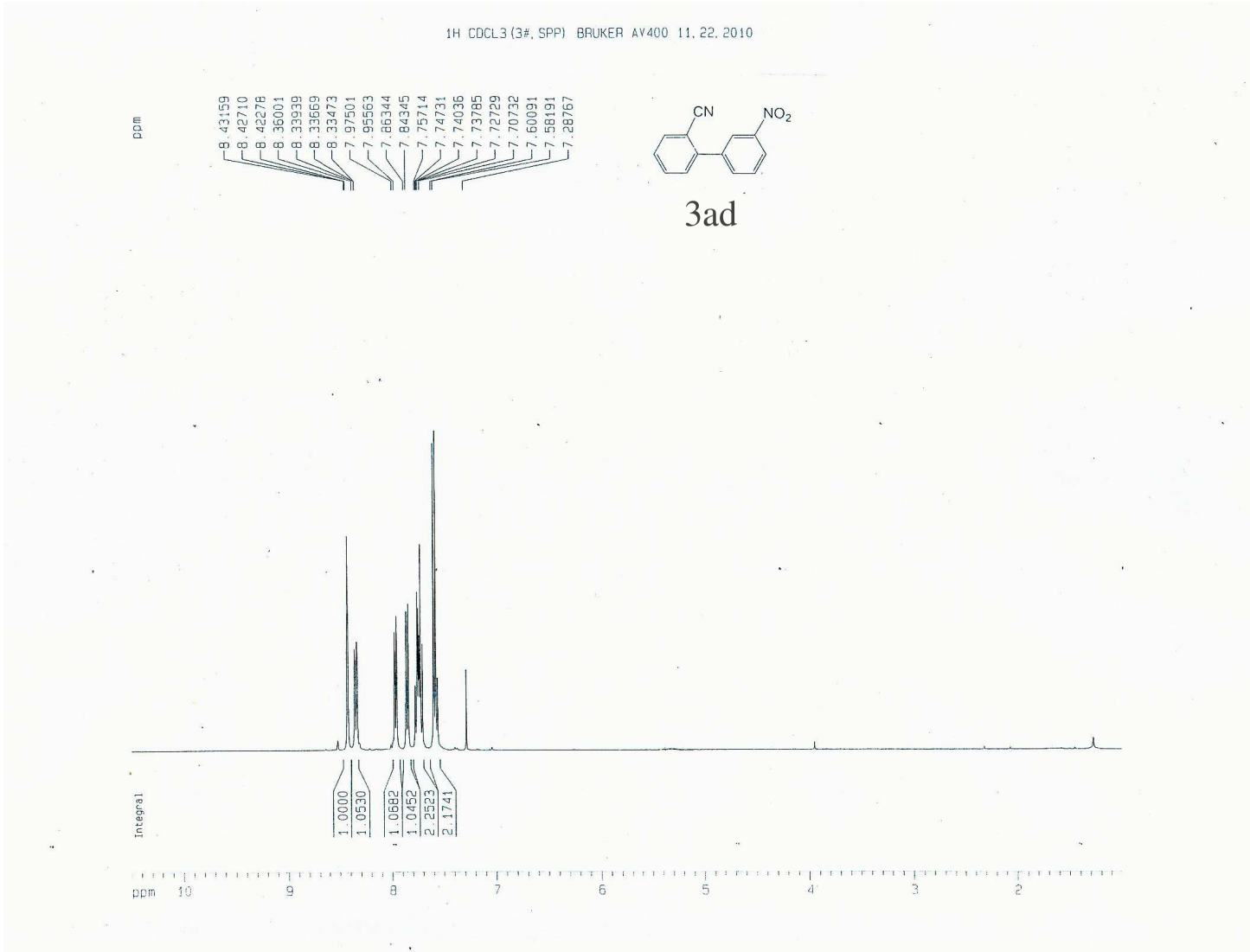

13C CDCL3 (SPP) BRU
CN
3aa
ppm
145.503
138.133
133.785
132.859
130.106
129.061
128.883
128.776
128.761
128.648
127.568
118.783
111.262
77.381
77.063
76.746
ppm
180
160
140
120
100
80
60
40
20

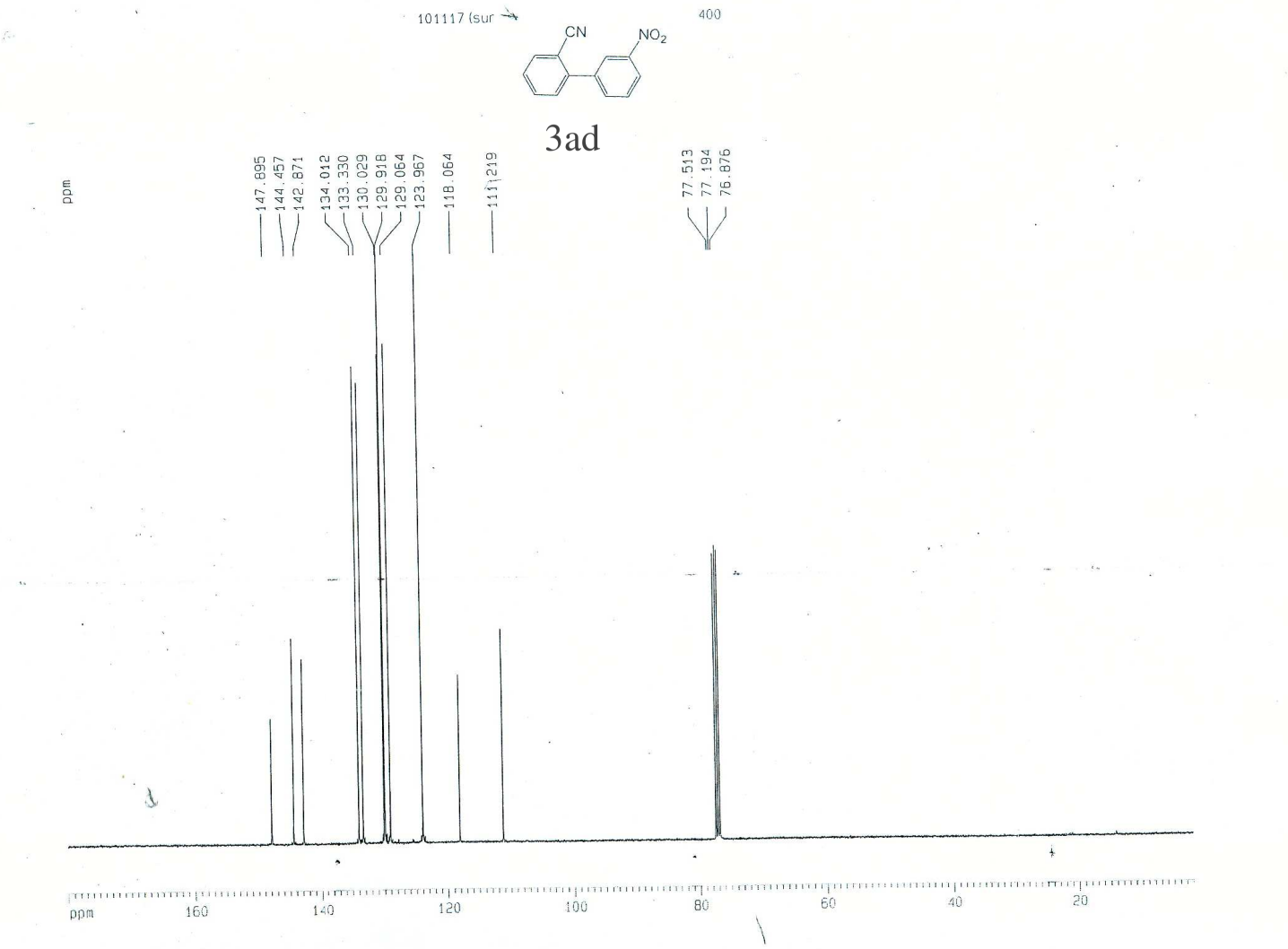

1H CDCL3 (3#, SPP) BRUKER AV400 09.26.2010
ppm
7.76740
7.74774
7.73770
7.71628
7.65128
7.63342
7.61501
7.51900
7.49941
7.47554
7.45512
7.44864
7.42914
7.40915
7.38413
7.36683
7.29422
7.25956
CN
CH3
3ab
2.43843
Integral
1.1792
1.0080
1.0000
3.9581
3.2707
ppm
9
8
7
6
5
4
3
2
1

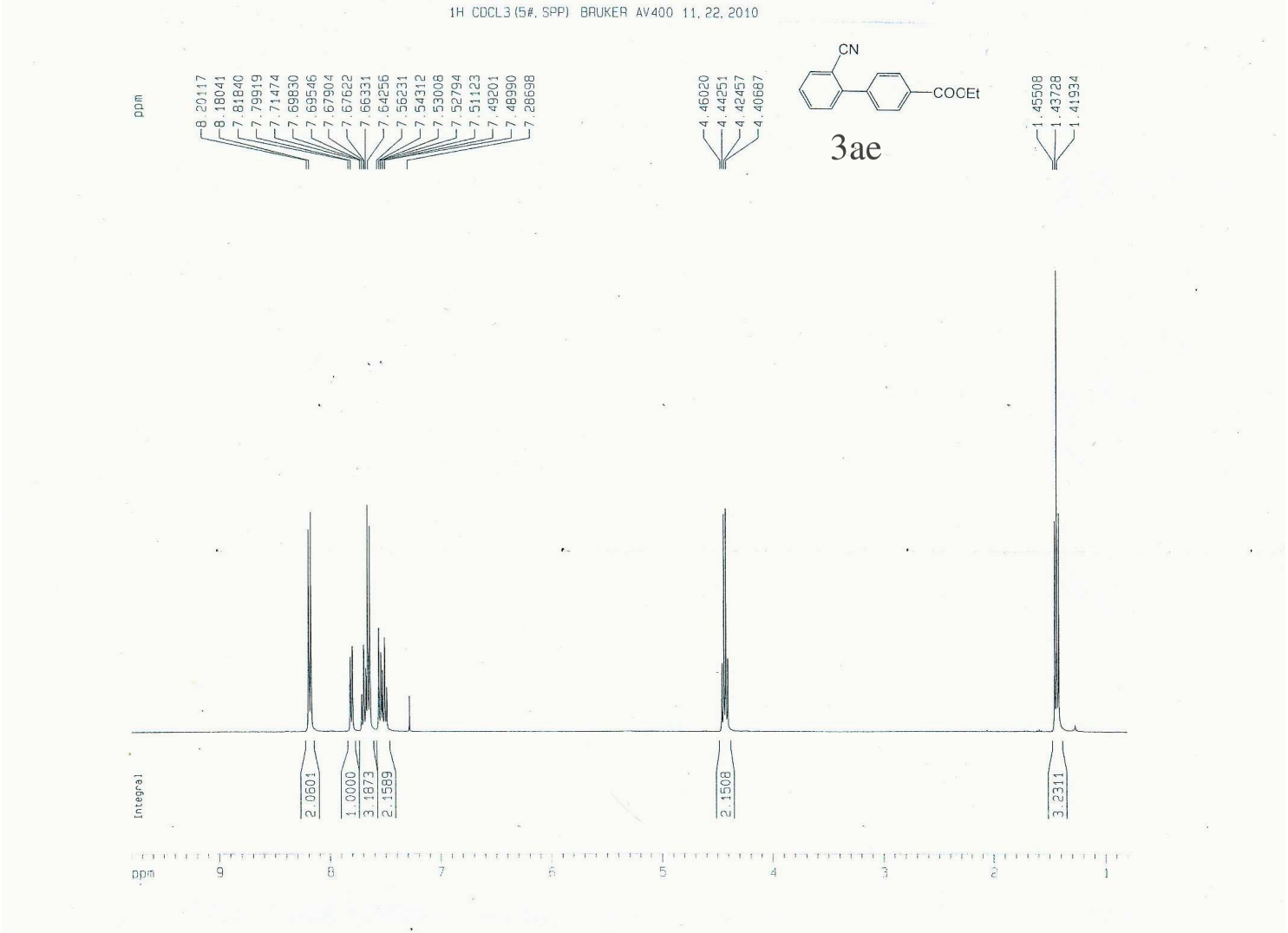

13C CDCL3 (2#.SPP) BRUKER AV400 09.28.2010
CN
CH3
3ab
ppm
145.689
138.418
138.122
133.708
132.720
130.318
129.454
128.616
127.412
125.852
118.744
111.305
77.329
77.216
77.012
76.694
21.450
ppm
140
120
100
80
60
40
20

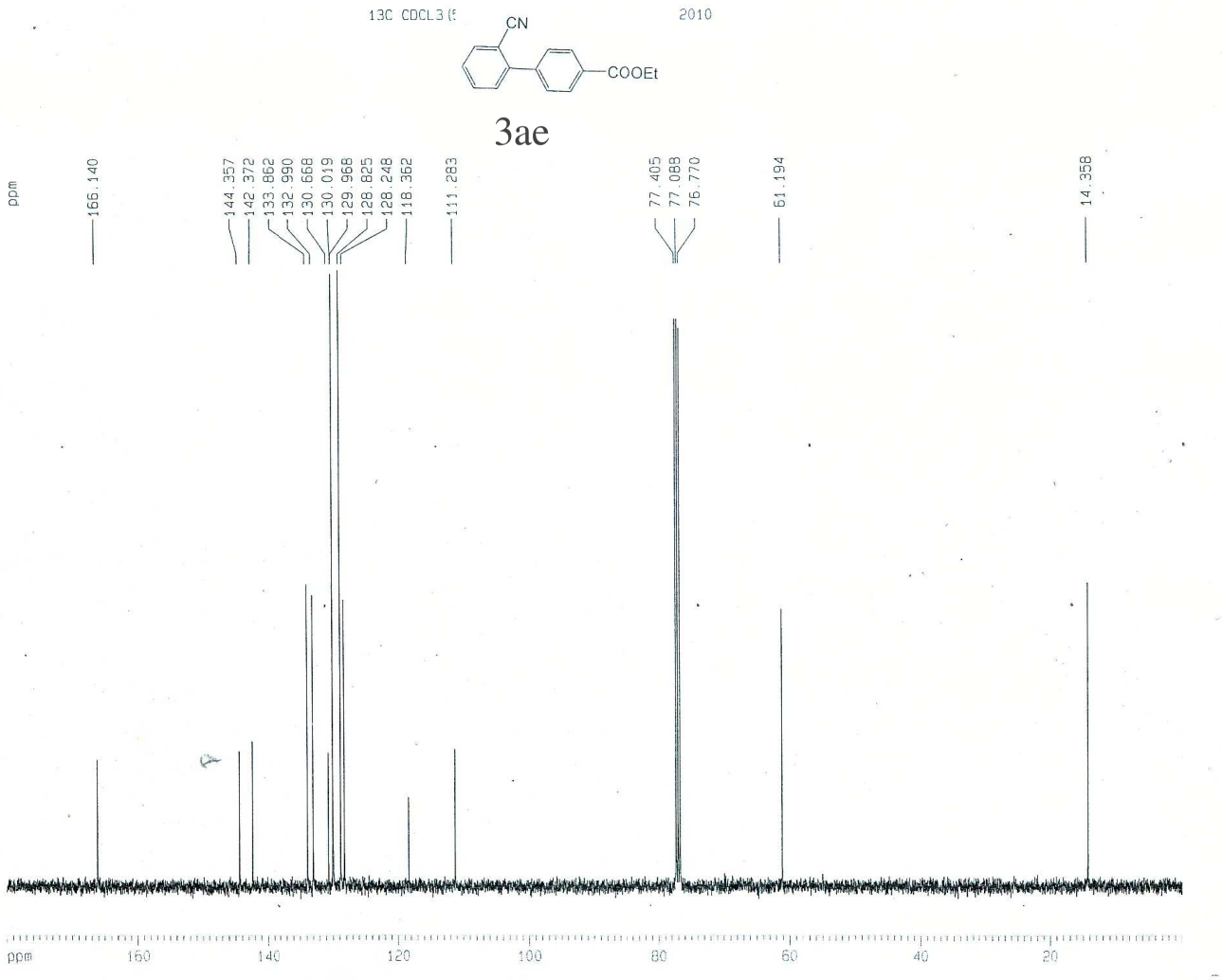

1H CDCL3 (1#. SUNPP) BRUKER AV400 11. 15. 2010
ppm
8.35927
8.35421
8.33743
8.33214
7.84866
7.82951
7.76910
7.76394
7.74738
7.73474
7.73190
7.59708
7.58463
7.57970
7.56500
7.56212
CN
NO2
3ac
Integral
2.0000
1.0086
3.0694
2.0764
ppm
9
8
7
6
5
4
3
2
1

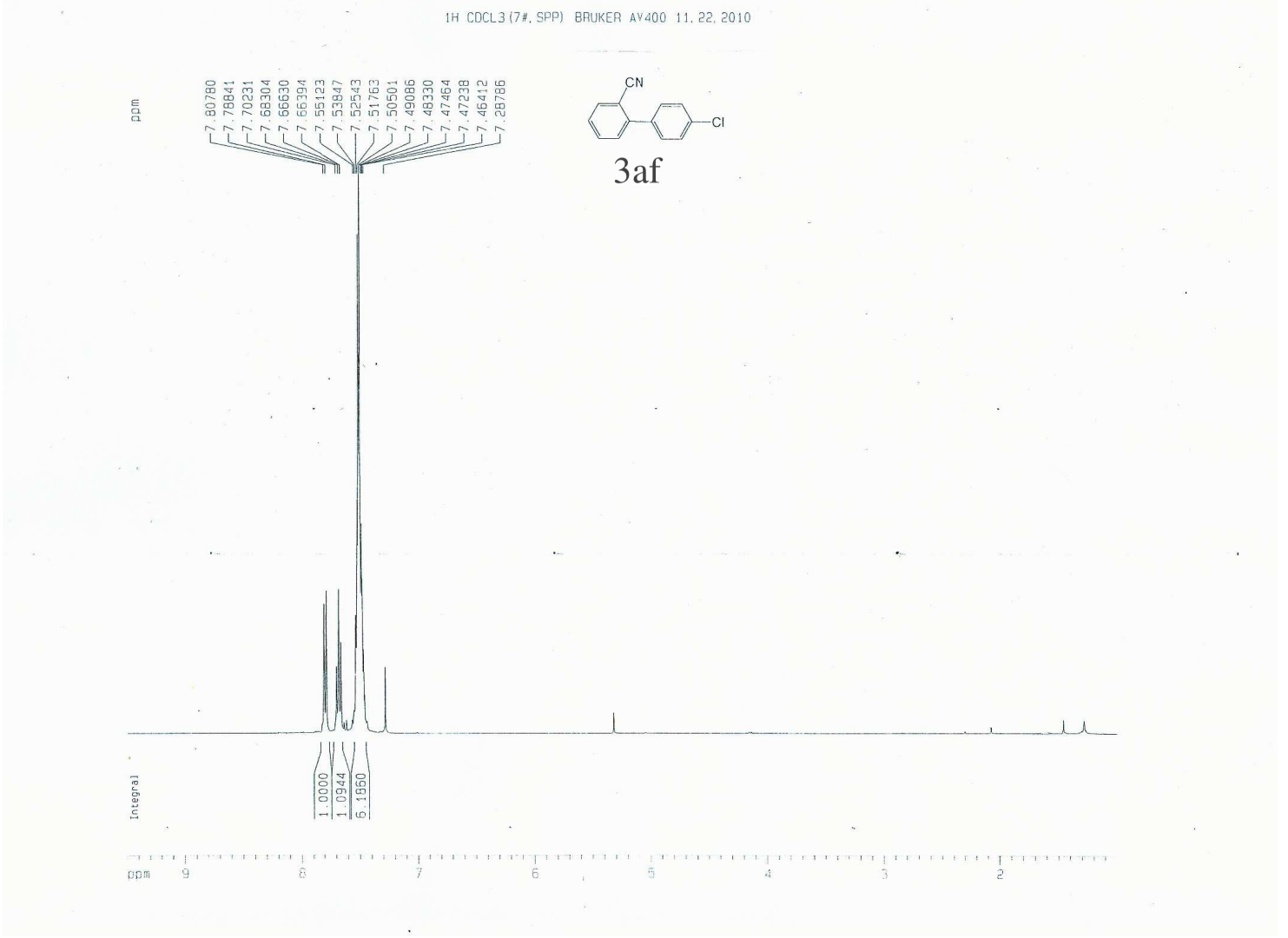

13C
22. 2010
CN
NO2
3ac
ppm
148.446
142.815
139.664
134.833
133.959
133.305
130.062
129.879
128.837
123.826
123.580
117.992
111.415
77.383
77.066
76.748
ppm
160
140
120
100
80
60
40
20

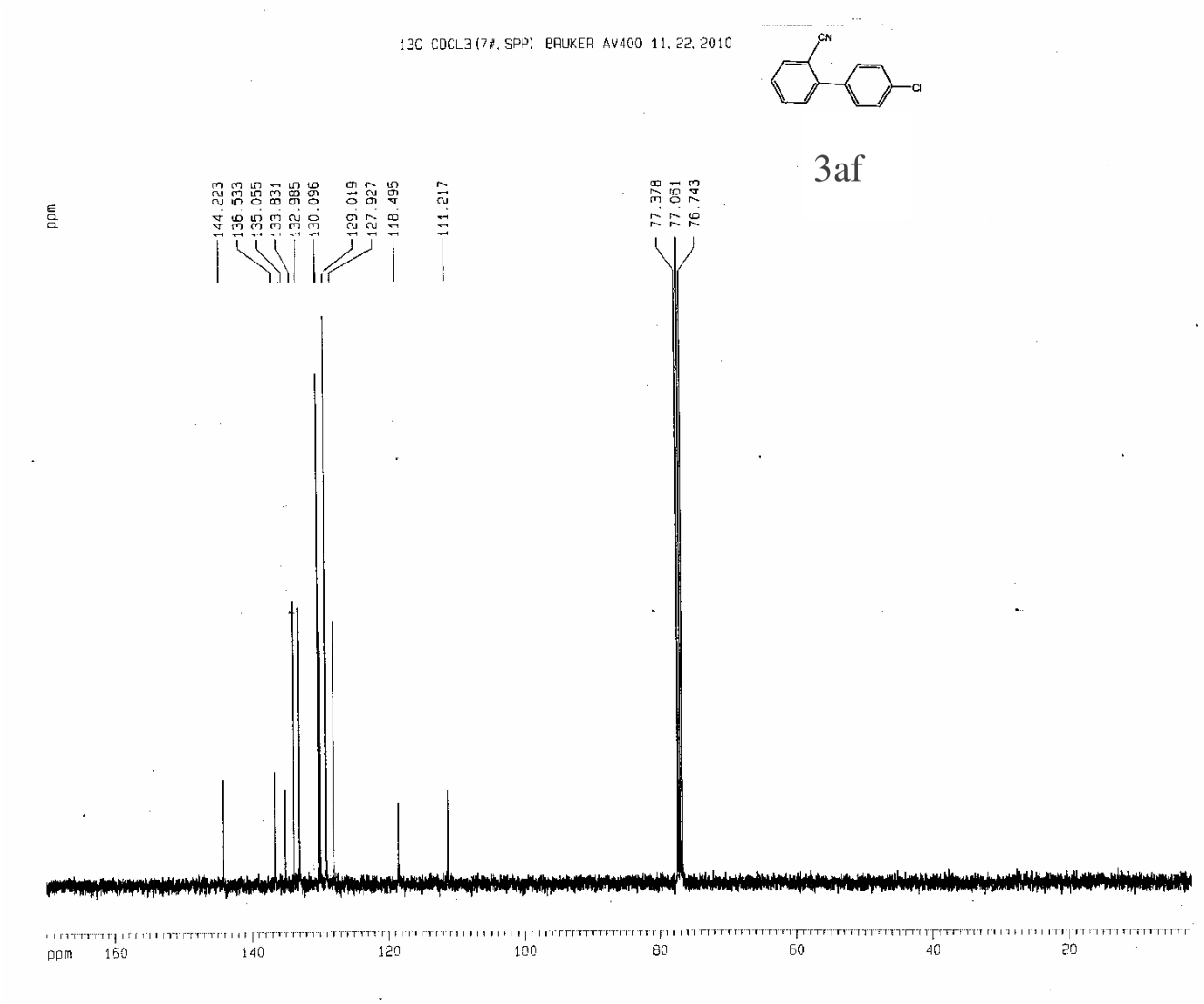

1H CDCL3 (3#, SPP) BRUKER AV400 11. 22. 2010
ppm
8.43159
8.42710
8.42278
8.36001
8.33939
8.33669
8.33473
7.97501
7.95563
7.86344
7.84345
7.75714
7.74731
7.74036
7.73785
7.72729
7.70732
7.60091
7.58191
7.28767
CN
NO2
3ad
Integral
1.0000
1.0530
1.0682
1.0452
2.2523
2.1741
ppm 10 9 8 7 6 5 4 3 2

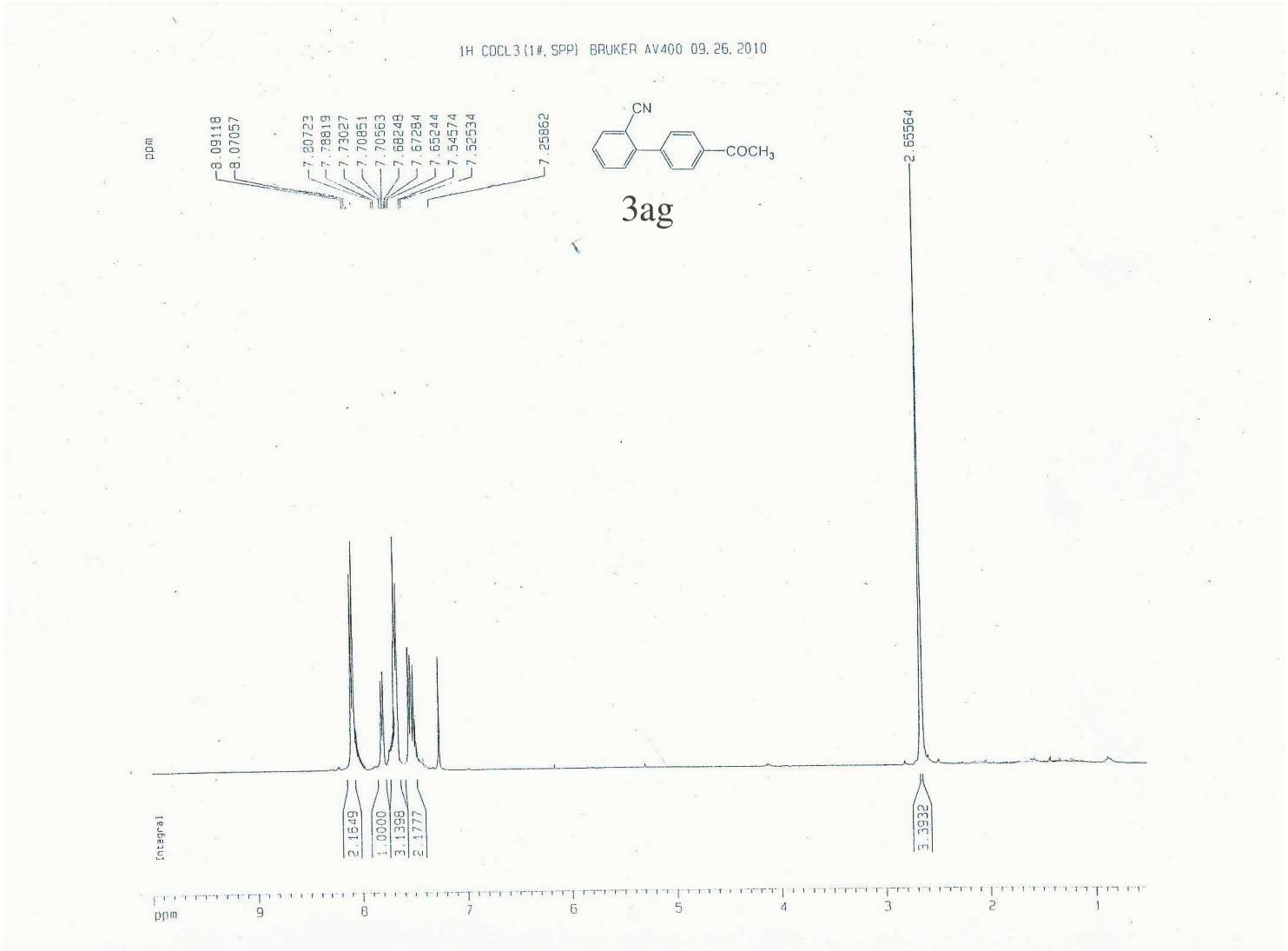

101117 (sur
400
CN
NO2
3ad
ppm
147.895
144.457
142.871
134.012
133.330
130.029
129.918
129.064
123.967
118.064
111.219
77.513
77.194
76.876
ppm
160
140
120
100
80
60
40
20


1H CDCL3 (5#, SPP) BRUKER AV400 11, 22, 2010
ppm
8.20117
8.18041
7.81840
7.79919
7.71474
7.69830
7.69546
7.67904
7.67622
7.66331
7.64256
7.56231
7.54312
7.53008
7.52794
7.51123
7.49201
7.48990
7.28698
4.46020
4.44251
4.42457
4.40687
1.45508
1.43728
1.41934
CN
COCEt
3ae
Integral
2.0601
1.0000
3.1873
2.1589
2.1508
3.2311
ppm
9
8
7
6
5
4
3
2
1

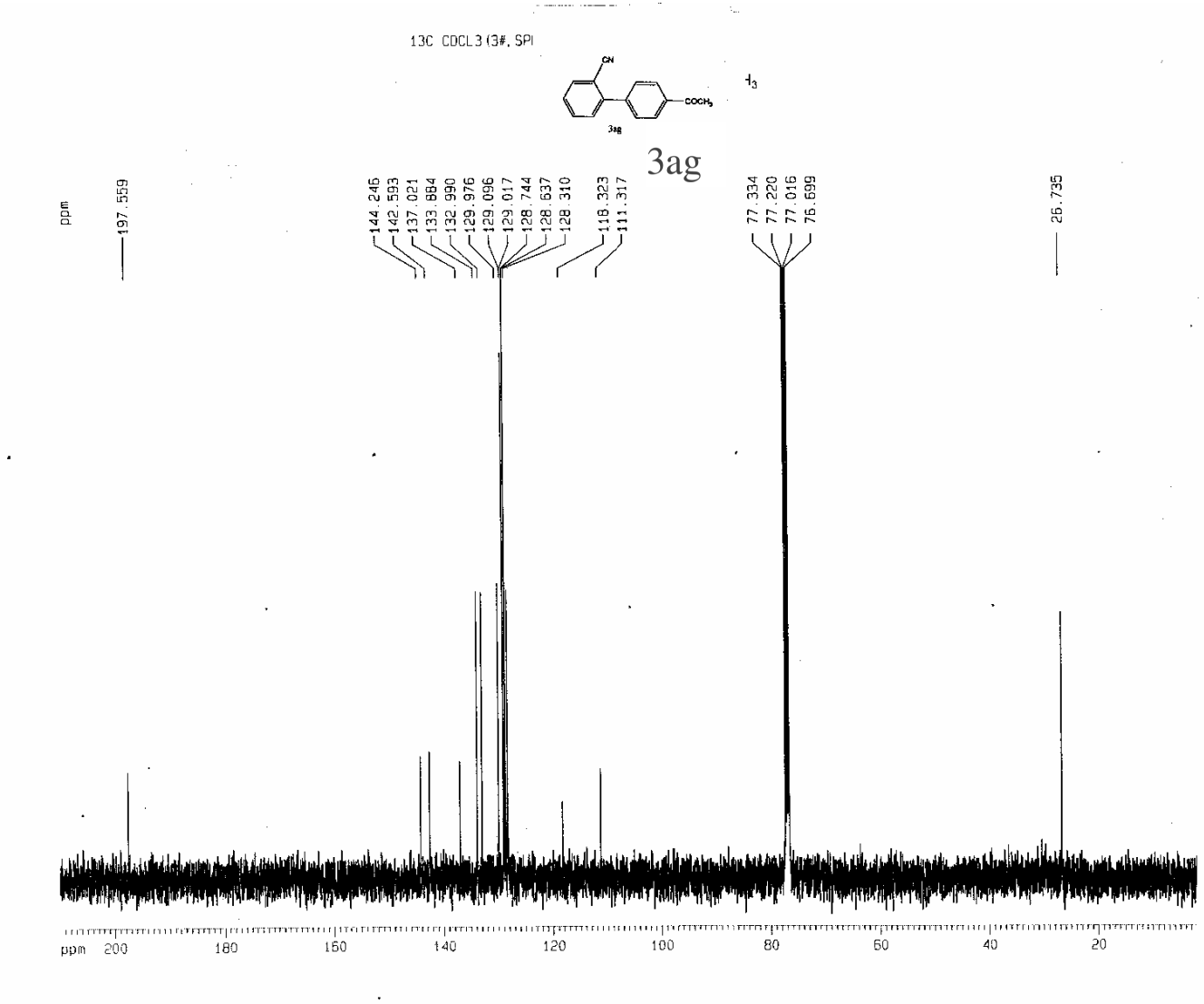

13C CDCL3 (5
2010
CN
COOEt
3ae
ppm
166.140
144.357
142.372
133.862
132.990
130.668
130.019
129.968
128.825
128.248
118.362
111.283
77.405
77.088
76.770
61.194
14.358
ppm
160
140
120
100
80
60
40
20

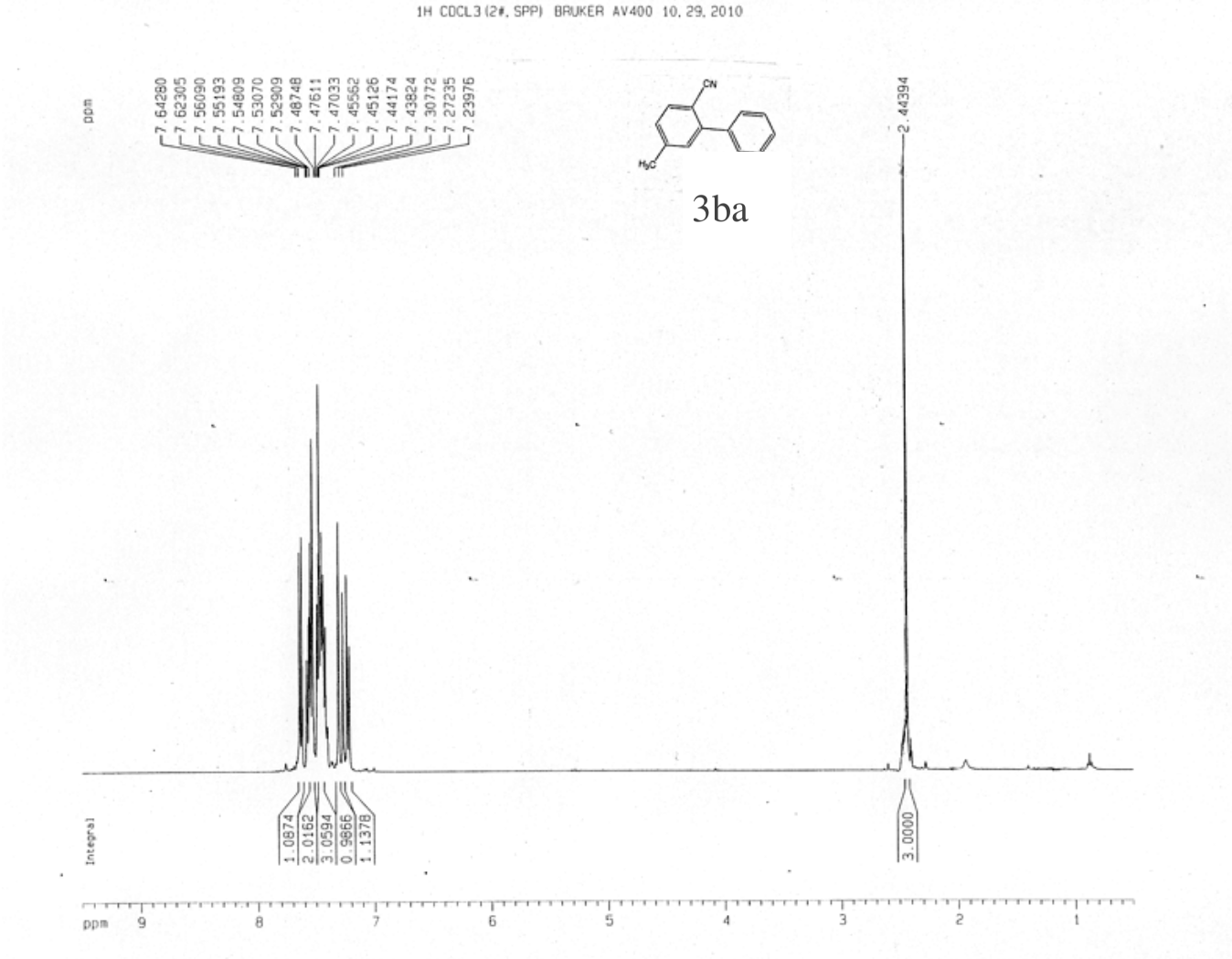

1H CDCL3 (7#, SPP) BRUKER AV400 11. 22. 2010
ppm
7.80780
7.78841
7.70231
7.68304
7.66630
7.66394
7.55123
7.53847
7.52543
7.51763
7.50501
7.49086
7.48330
7.47464
7.47238
7.46412
7.28786
CN
Cl
3af
Integral
1.0000
1.0944
6.1660
ppm
9
8
7
6
5
4
3
2

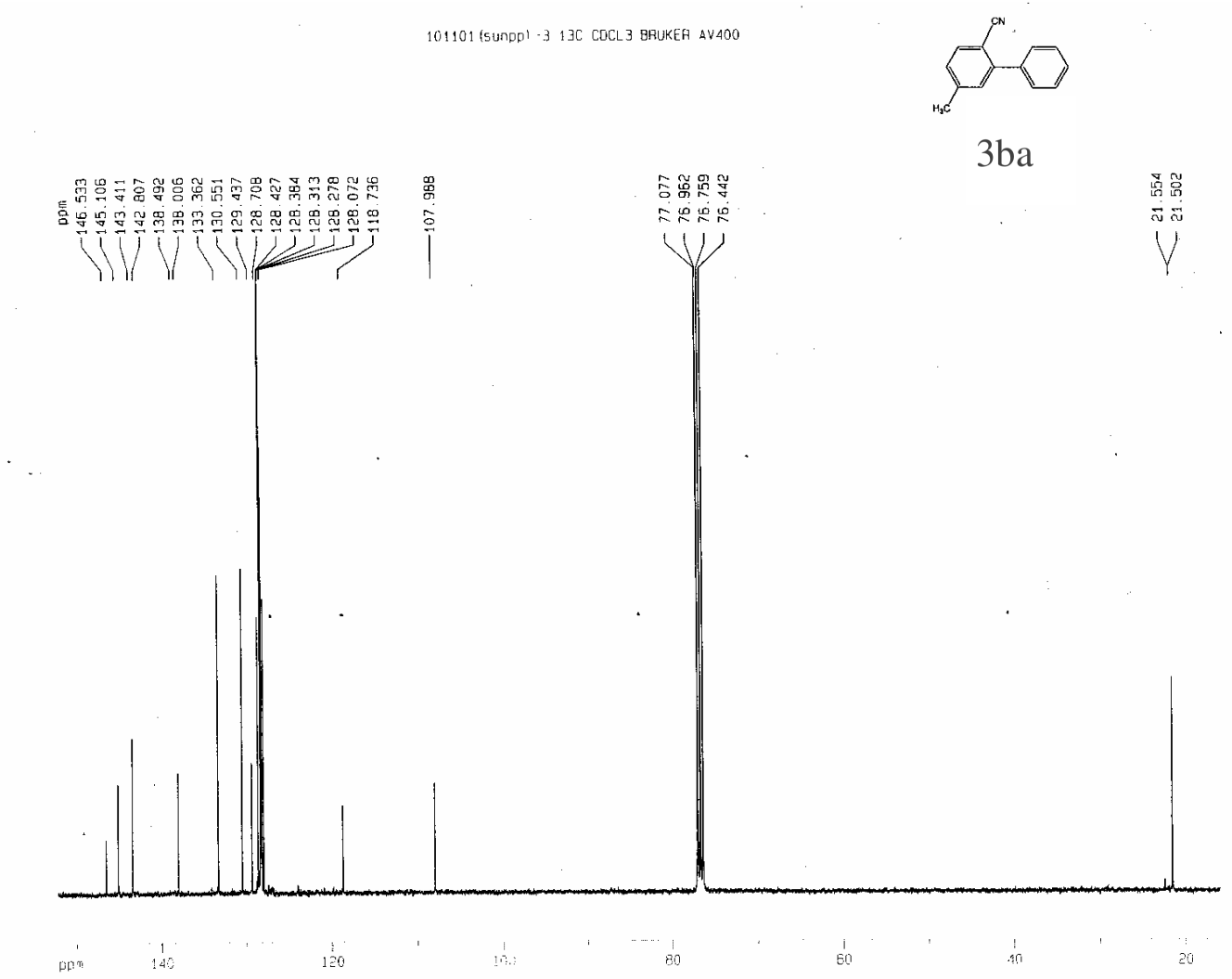

13C CDCL3 (7#.SPP) BRUKER AV400 11.22.2010
CN
Cl
3af
ppm
144.223
136.533
135.055
133.831
132.985
130.096
129.019
127.927
118.495
111.217
77.378
77.061
76.743
ppm 160
140
120
100
80
60
40
20

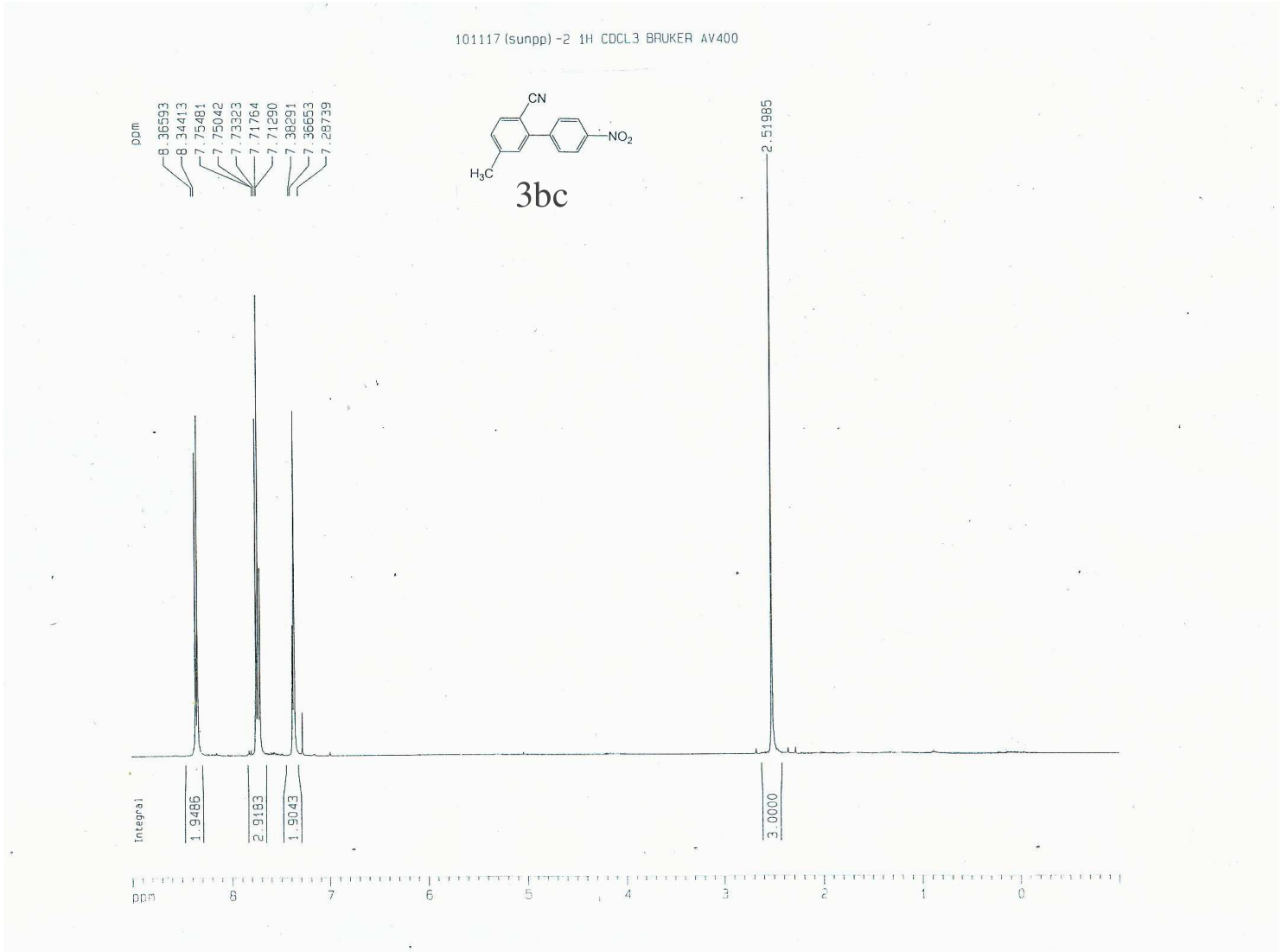

1H CDCL3 (1#, SPP) BRUKER AV400 09. 26. 2010
ppm
8.09118
8.07057
7.80723
7.78819
7.73027
7.70851
7.70563
7.68248
7.67284
7.65244
7.54574
7.52534
7.25862
CN
COCH3
2.65564
Integral
2.1649
1.0000
3.1398
2.1777
3.3932
ppm
9
8
7
6
5
4
3
2
1

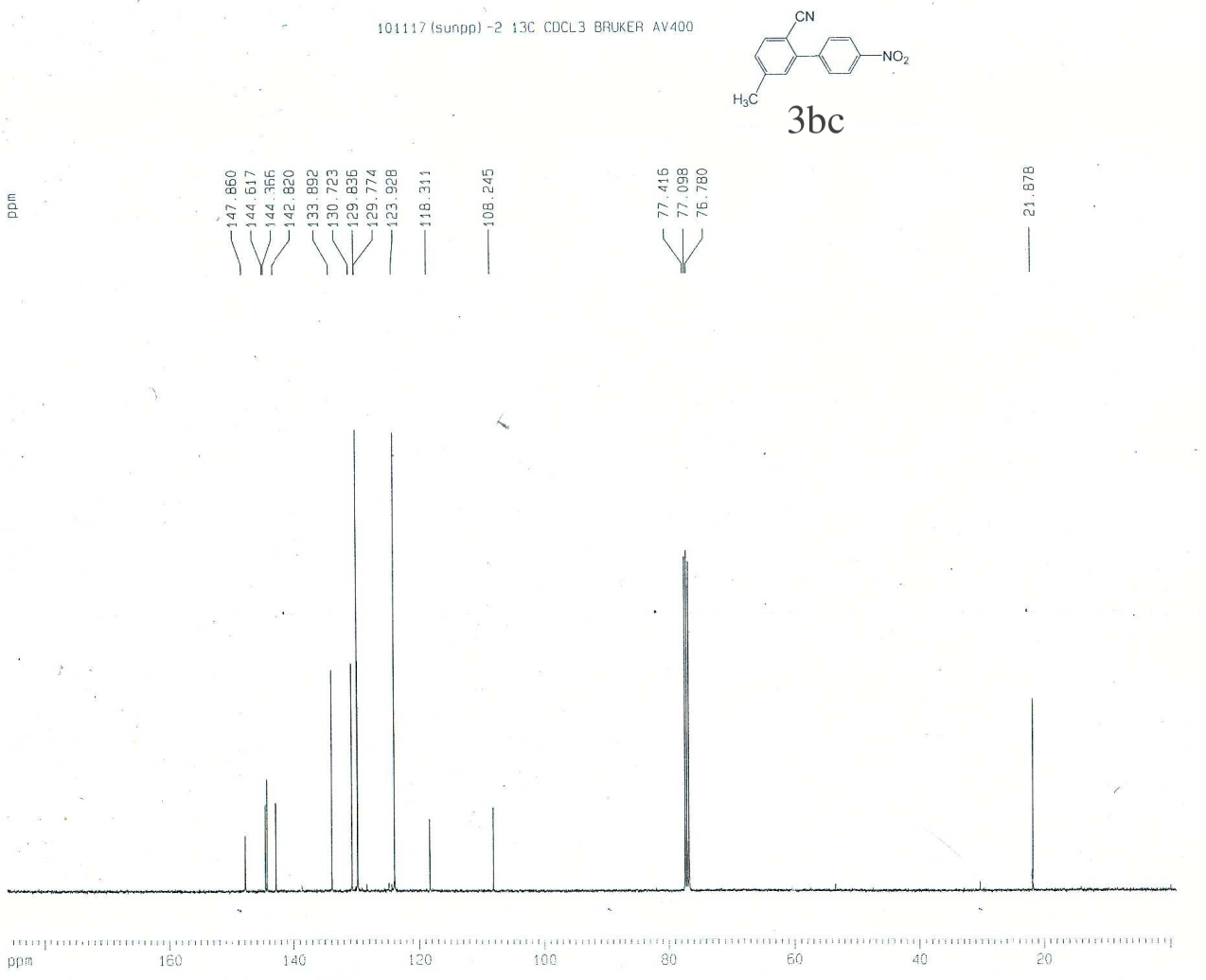
3ag

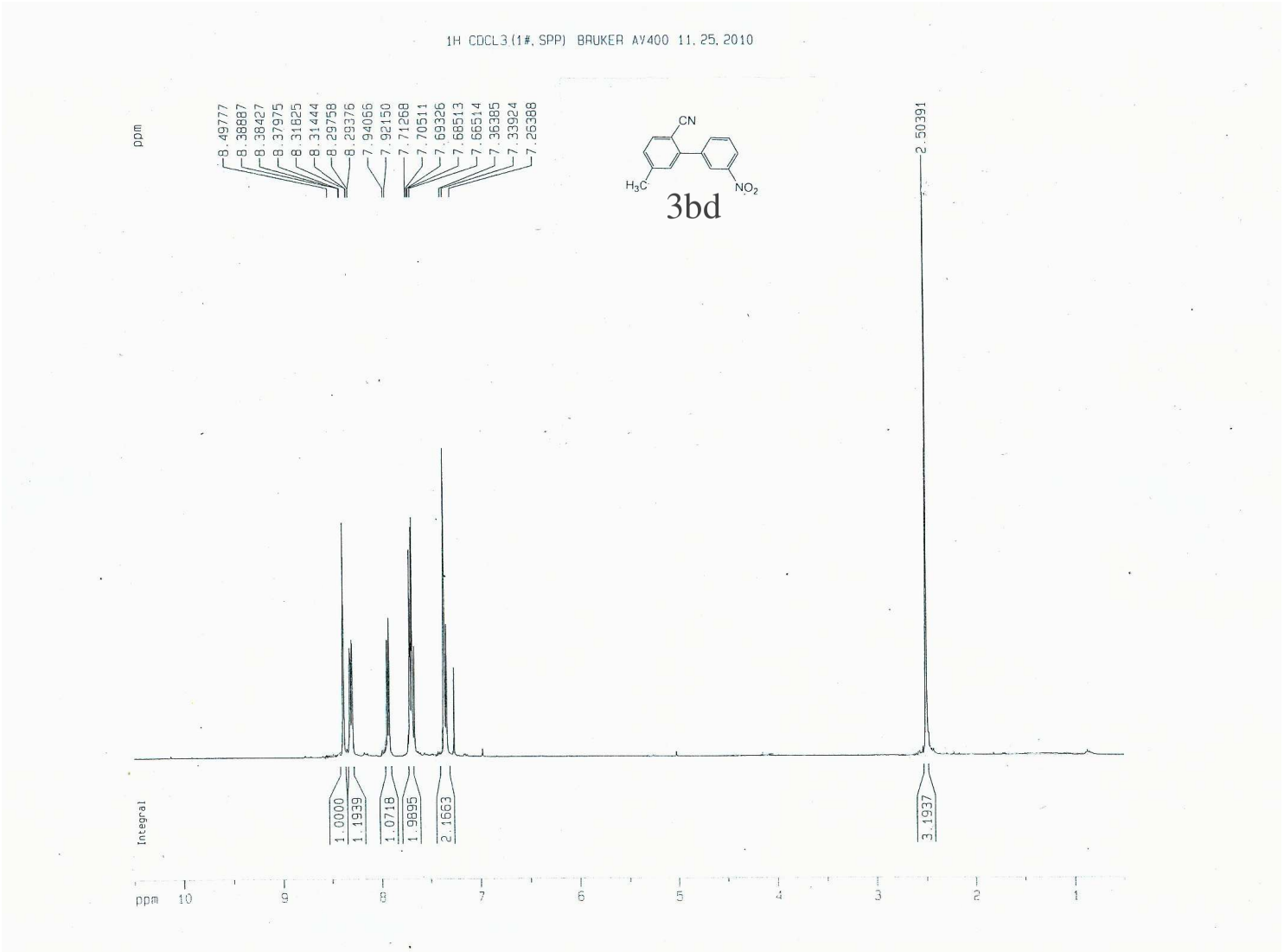

13C CDCL3 (3#. SPI
CN
COCH3
3ag
3ag
ppm
197.559
144.246
142.593
137.021
133.884
132.990
129.976
129.096
129.017
128.744
128.637
128.310
118.323
111.317
77.334
77.220
77.016
76.699
26.735
ppm 200
180
160
140
120
100
80
60
40
20

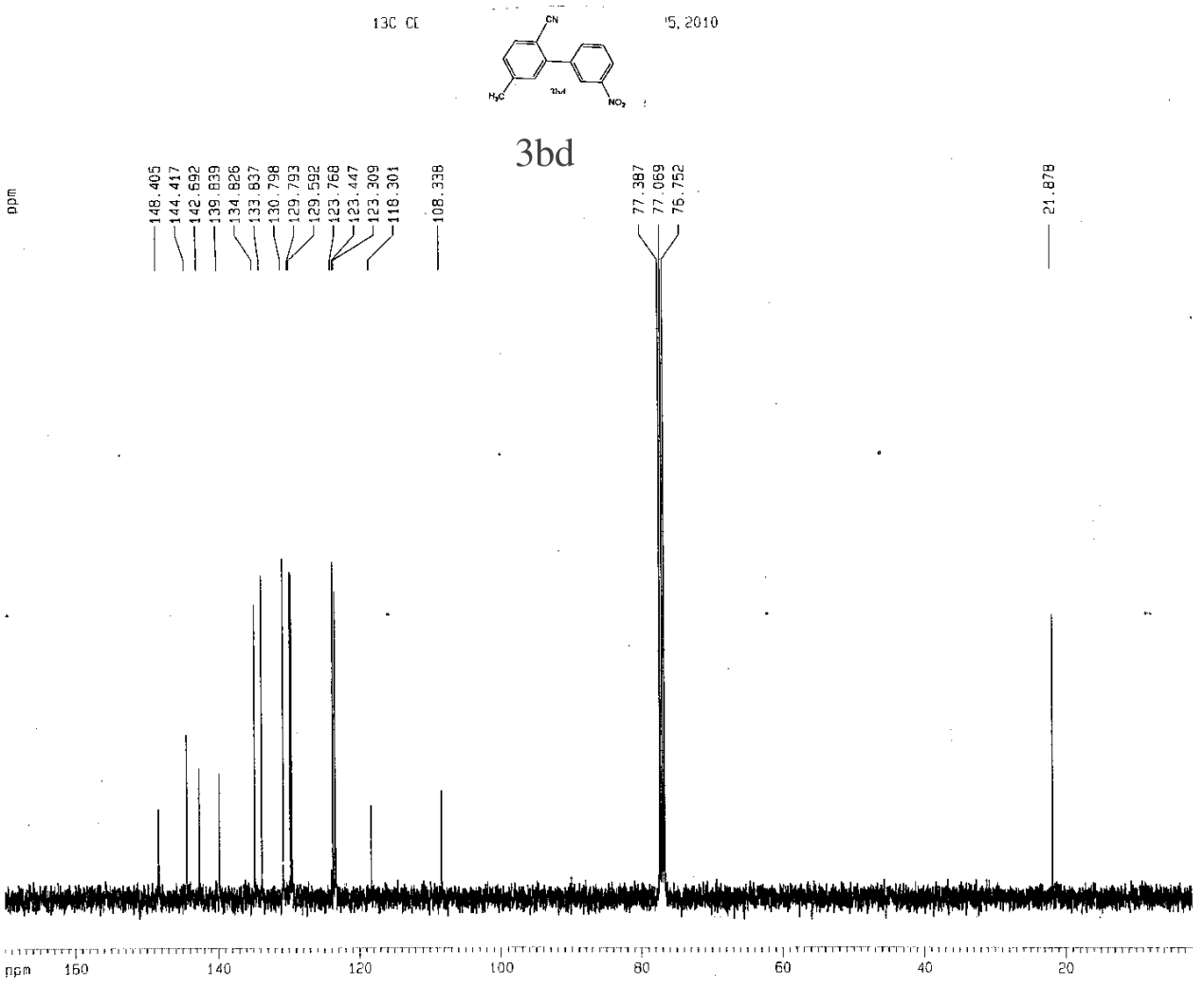

1H CDCL3 (2#, SPP) BRUKER AV400 10. 29. 2010
ppm
7.64280
7.62305
7.56090
7.55193
7.54809
7.53070
7.52909
7.48748
7.47611
7.47033
7.45562
7.45126
7.44174
7.43824
7.30772
7.27235
7.23976
2.44394
CN
H3C
3ba
Integral
1.0874
2.0162
3.0594
0.9866
1.1378
3.0000
ppm
9
8
7
6
5
4
3
2
1

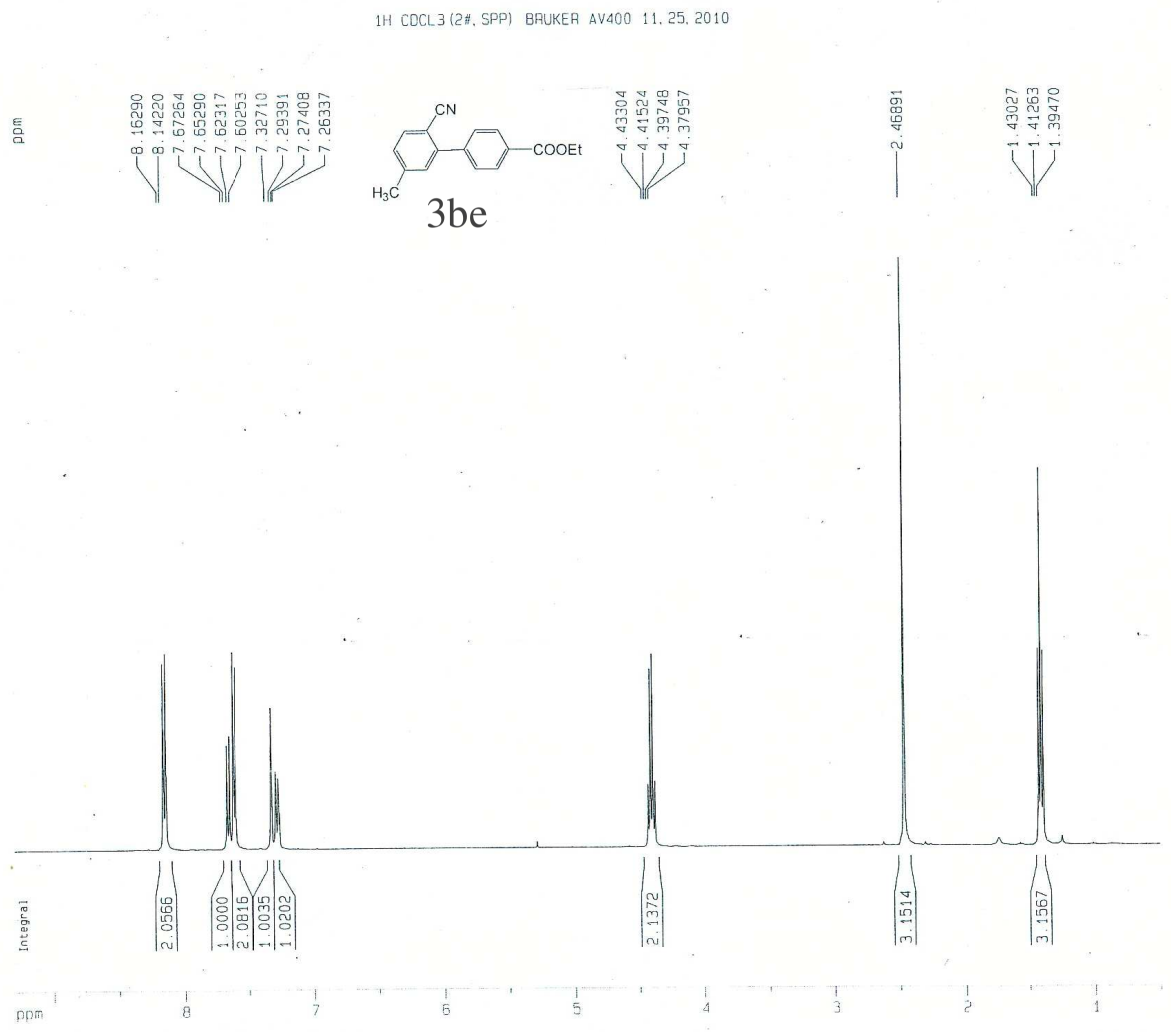

101101 (sunpp) -3 13C CDCL3 BRUKER AV400
CN
H3C
3ba
ppm
146.533
145.106
143.411
142.807
138.492
138.006
133.362
130.551
129.437
128.708
128.427
128.384
128.313
128.278
128.072
118.736
107.988
77.077
76.962
76.759
76.442
21.554
21.502
ppm
140
120
80
60
40
20


101117 (sunpp) -2 1H CDCL3 BRUKER AV400
ppm
8.36593
8.34413
7.75481
7.75042
7.73323
7.71764
7.71290
7.38291
7.36653
7.28739
2.51995
CN
NO2
H3C
3bc
Integral
1.9486
2.9183
1.9043
3.0000
ppm
8
7
6
5
4
3
2
1
0

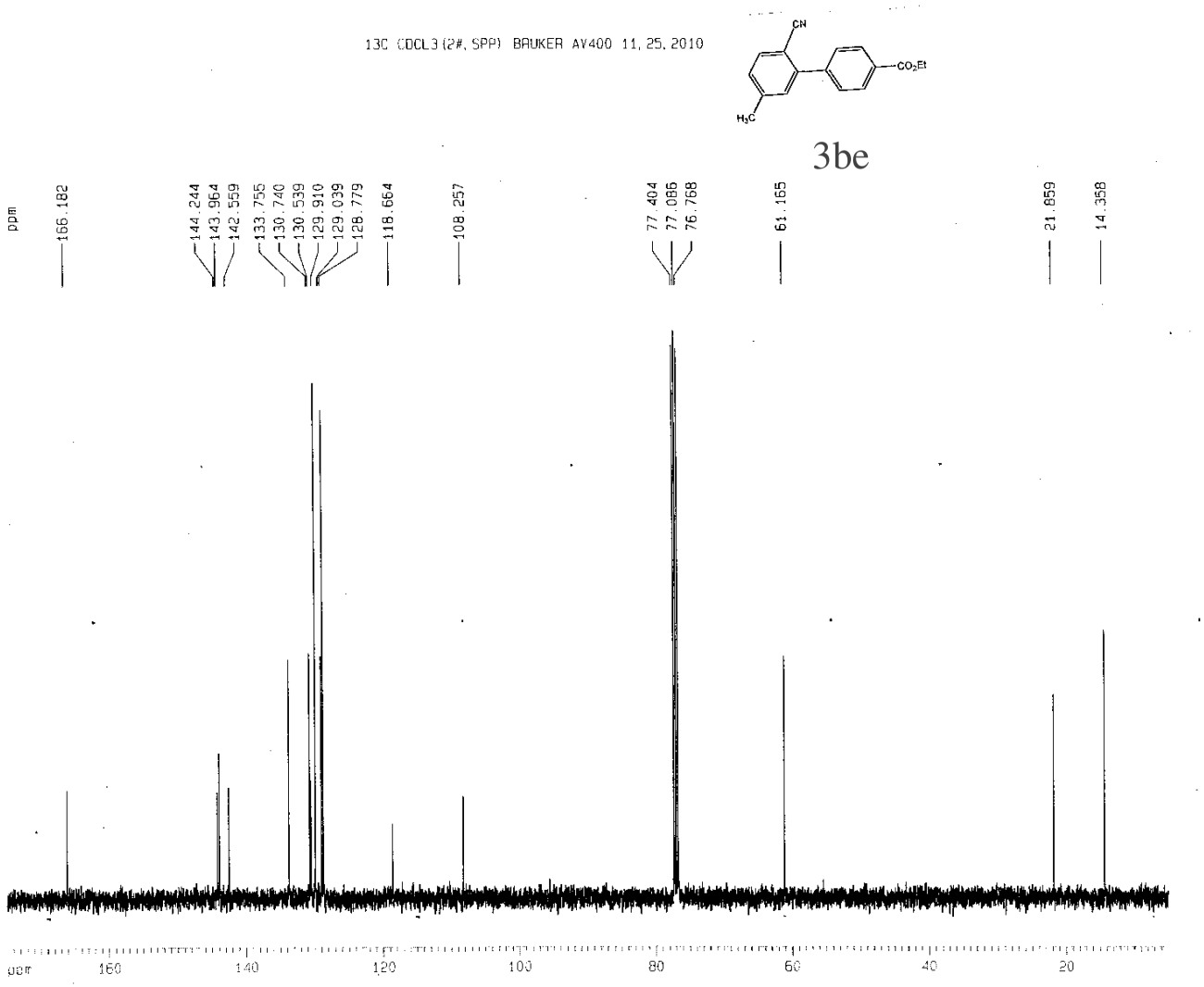

101117 (sunpp) -2 13C CDCL3 BRUKER AV400
CN
NO2
H3C
3bc
ppm
147.860
144.617
144.366
142.820
133.892
130.723
129.836
129.774
123.928
118.311
108.245
77.416
77.098
76.780
21.878
ppm
160
140
120
100
80
60
40
20

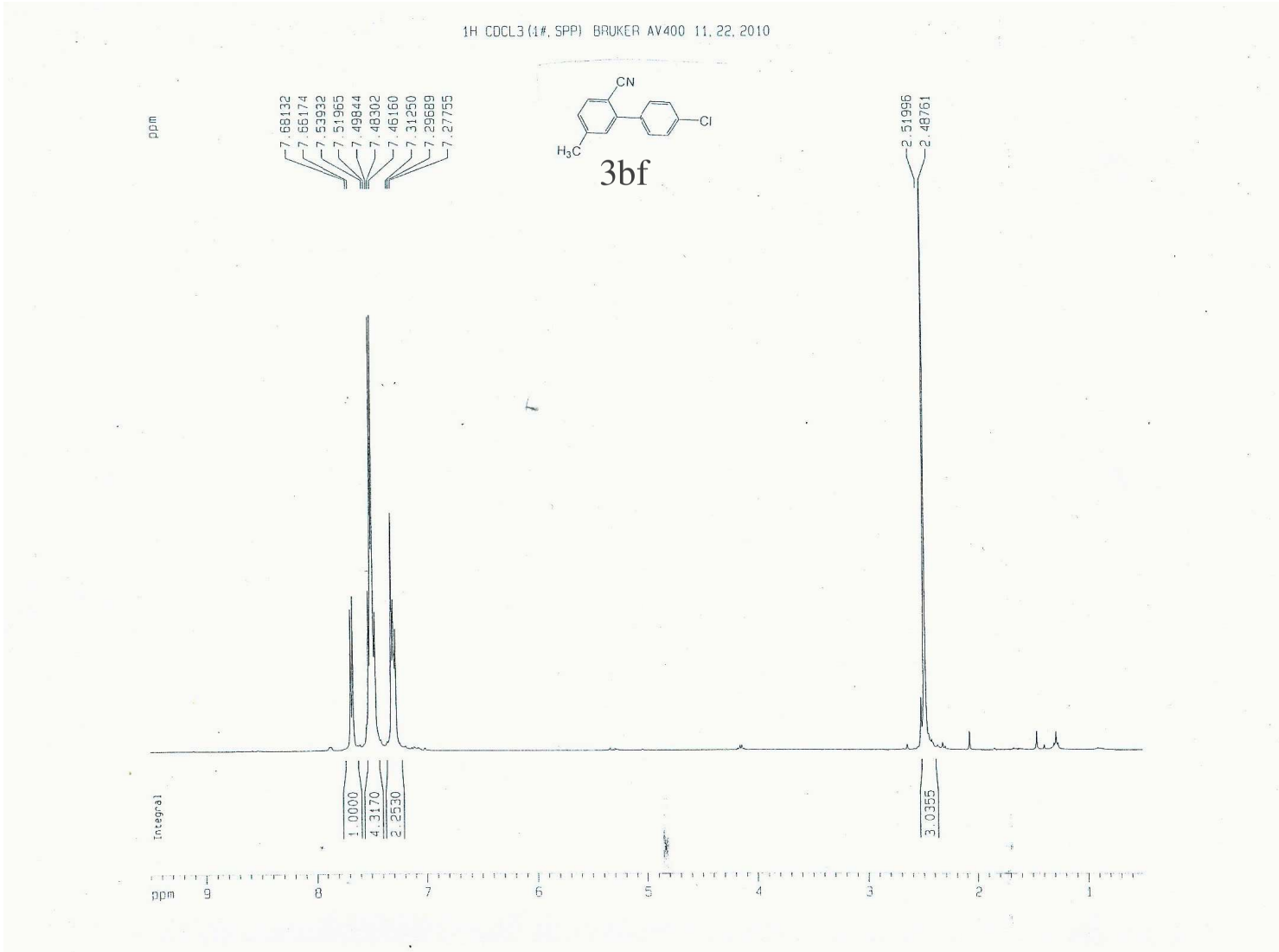

1H CDCL3 (1#, SPP) BRUKER AV400 11. 25. 2010
ppm
8.49777
8.38887
8.38427
8.37975
8.31825
8.31444
8.29758
8.29376
7.94066
7.92150
7.71268
7.70511
7.69326
7.66513
7.66514
7.36385
7.33924
7.26388
CN
H3C
NO2
3bd
2.50391
Integral
1.0000
1.1939
1.0718
1.9895
2.1663
3.1937
ppm
10
9
8
7
6
5
4
3
2
1

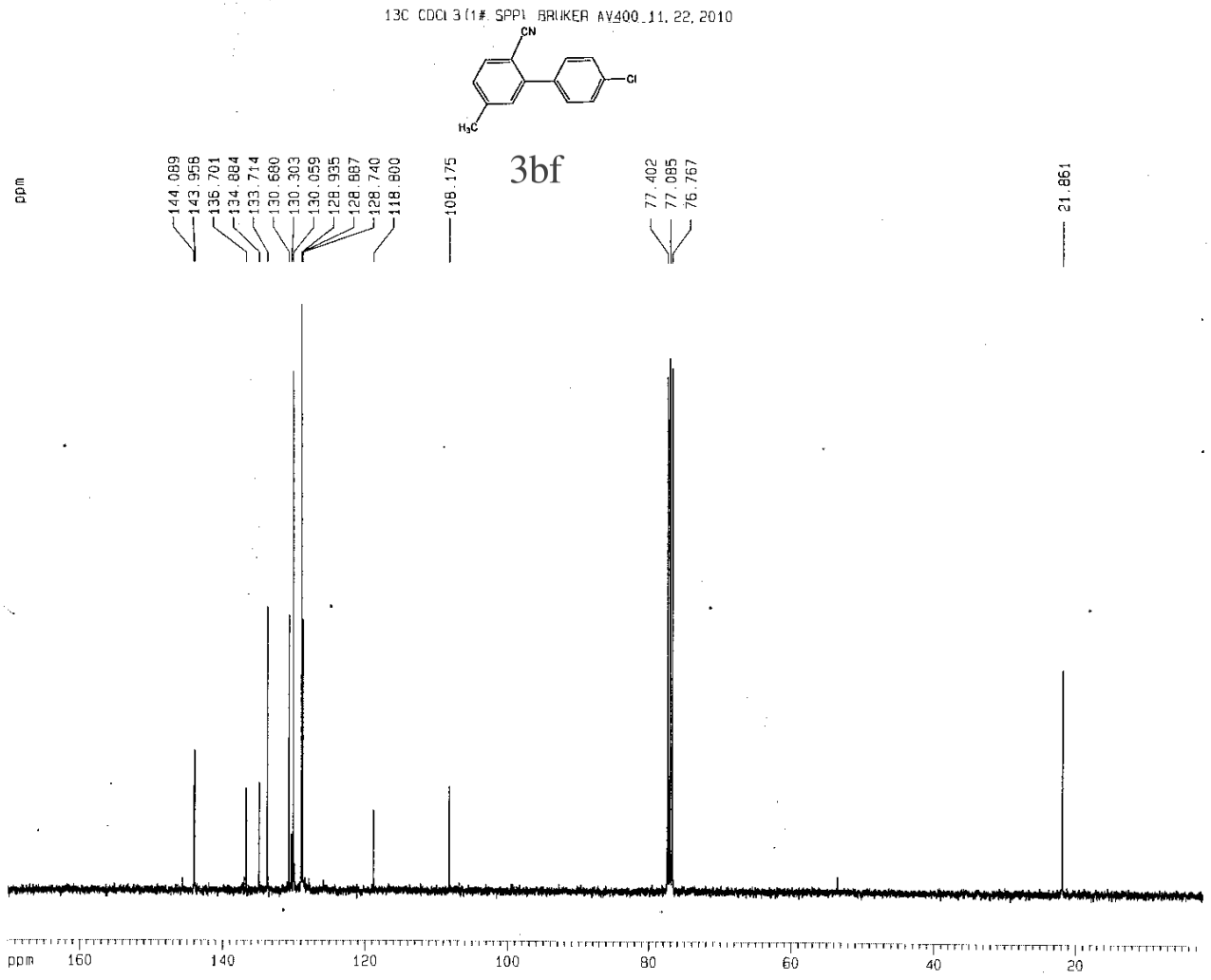

13C CI
5, 2010
CN
H_3C
NO_2
3bd
ppm
148.405
144.417
142.692
139.839
134.826
133.837
130.798
129.793
129.592
123.768
123.447
123.309
118.301
108.338
77.387
77.069
76.752
21.878
ppm 160
140
120
100
80
60
40
20

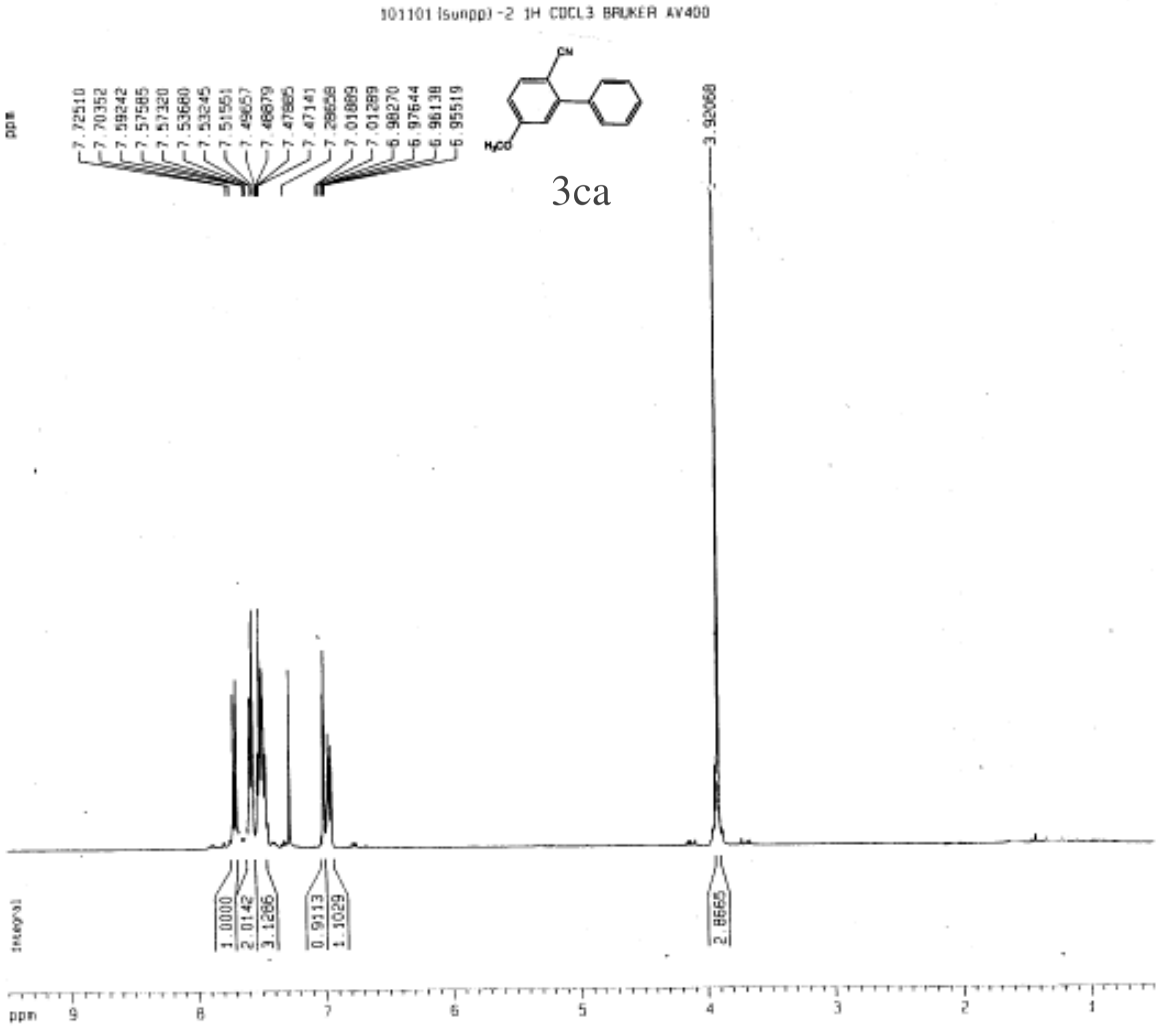

1H CDCL3 (2#, SPP) BRUKER AV400 11.25.2010
ppm
8.16290
8.14220
7.67264
7.65290
7.62317
7.60253
7.32710
7.29391
7.27408
7.26337
CN
COOEt
H3C
4.43304
4.41524
4.39748
4.37957
2.46891
1.43027
1.41263
1.39470
Integral
2.0566
1.0000
2.0816
1.0035
1.0202
2.1372
3.1514
3.1567
ppm
8
7
6
5
4
3
2
1

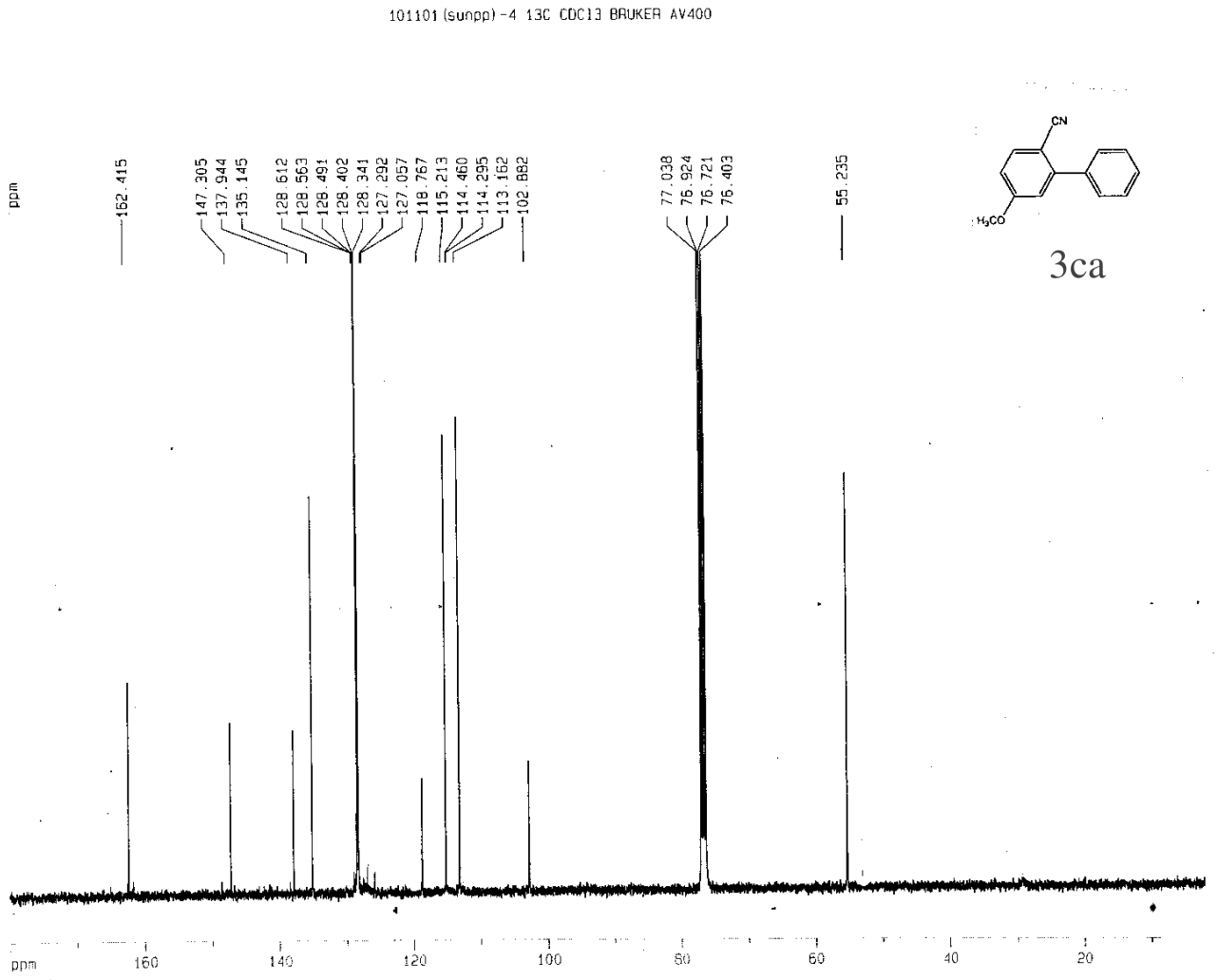
3be

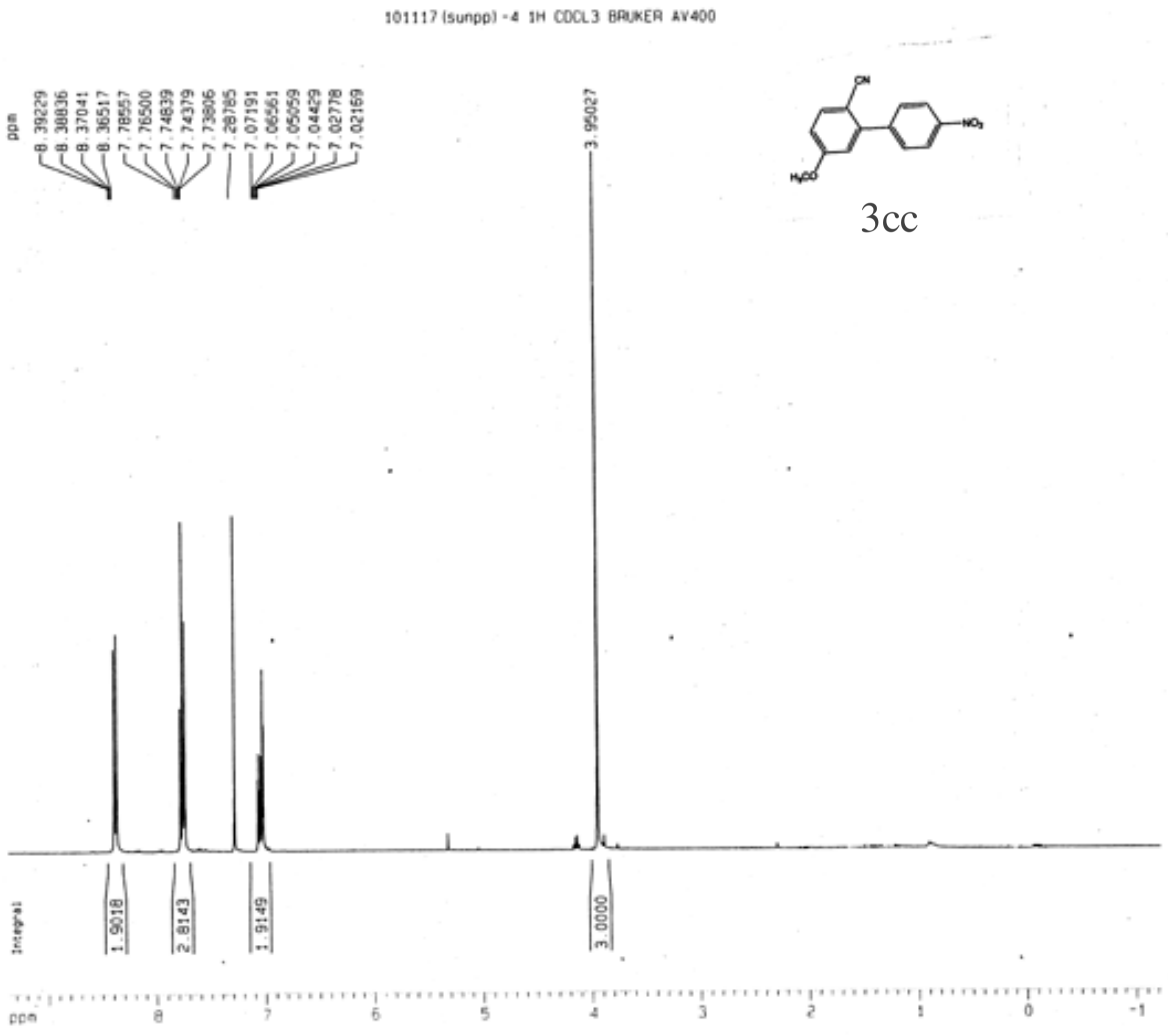

13C (CDCL3 (2#, SPP) BRUKER AV400 11, 25, 2010
CN
CO2Et
H3C
3be
ppm
166.182
144.244
143.964
142.559
133.755
130.740
130.539
129.910
129.039
128.779
118.664
108.257
77.404
77.086
76.768
61.165
21.859
14.358
ppm
160
140
120
100
80
60
40
20


1H CDCL3 (1#, SPP) BRUKER AV400 11.22.2010
ppm
7.68132
7.66174
7.53932
7.51965
7.49844
7.48302
7.46160
7.31250
7.29689
7.27755
CN
Cl
H3C
3bf
2.51996
2.48761
Integral
1.0000
4.3170
2.2530
3.0355
ppm 9 8 7 6 5 4 3 2 1

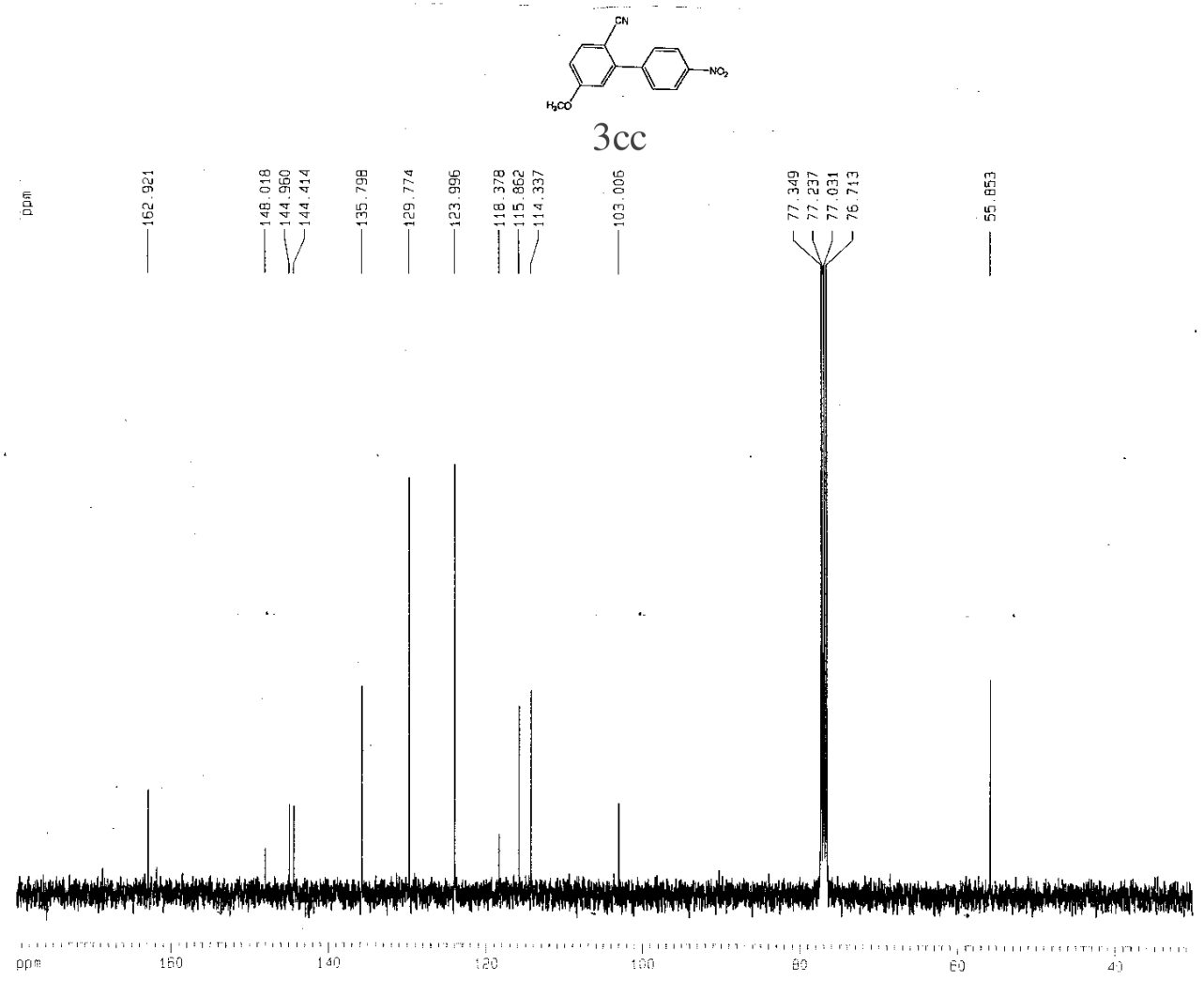

13C CDCl3 (1#.SPP) BRUKER AV400_11.22.2010
CN
Cl
H3C
3bf
ppm
144.089
143.958
136.701
134.884
133.714
130.680
130.303
130.059
128.935
128.887
128.740
118.800
108.175
77.402
77.085
76.767
21.861
ppm 160 140 120 100 80 60 40 20

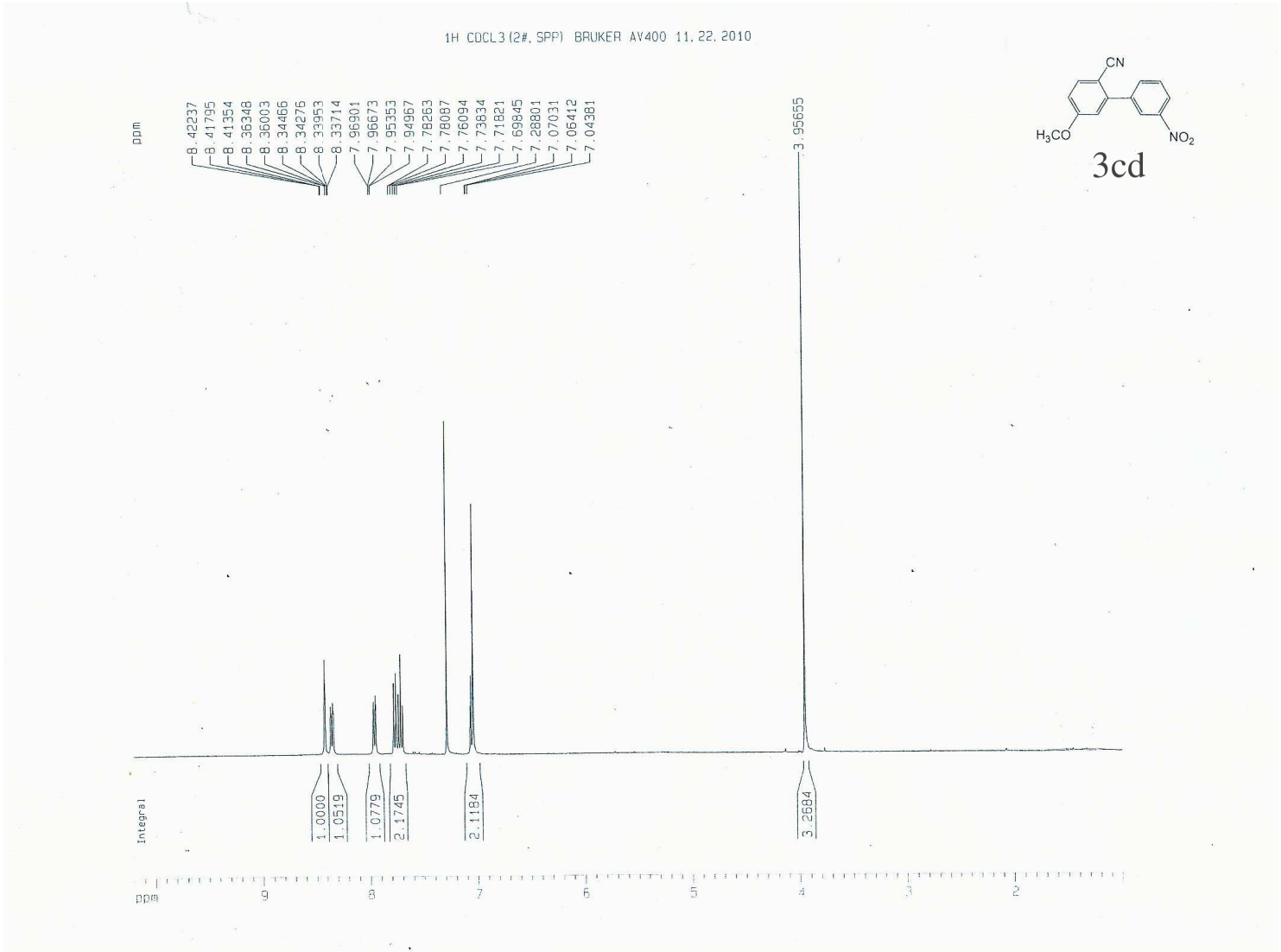

101101 (sunpp) -2 1H CDCL3 BRUKER AV400
ppm
7.72510
7.70352
7.59242
7.57585
7.57320
7.53660
7.53245
7.51551
7.49657
7.48879
7.47885
7.47141
7.28658
7.01889
7.01289
6.98270
6.97644
6.96138
6.95519
3.92068
3ca
integral
1.0000
2.0142
3.1286
0.9113
1.1029
2.8665
ppm 9 8 7 6 5 4 3 2 1

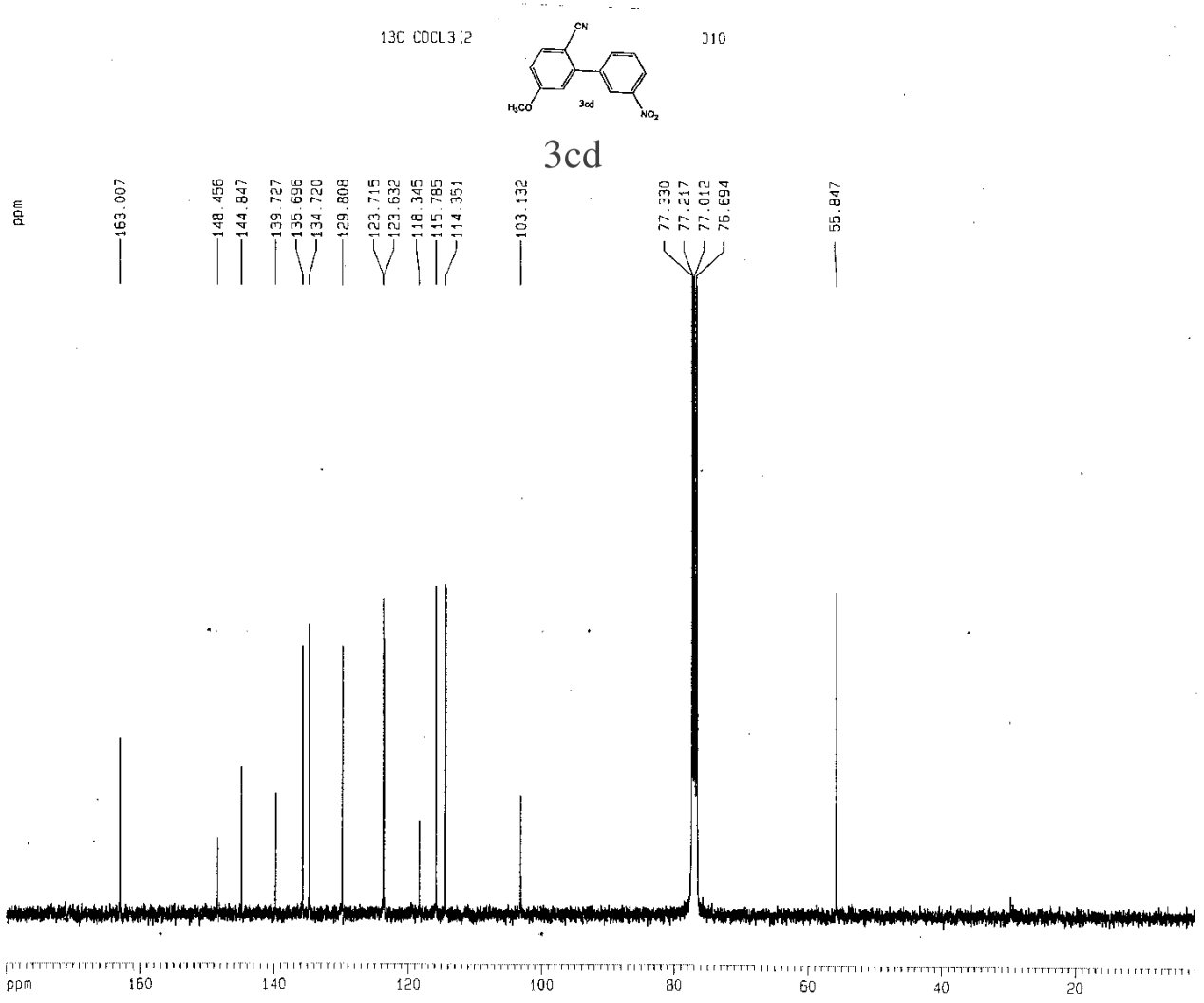

101101 (sunpp) -4 13C CDCl3 BRUKER AV400
ppm
162.415
147.305
137.944
135.145
128.612
128.563
128.491
128.402
128.341
127.292
127.057
118.767
115.213
114.460
114.295
113.162
102.882
77.038
76.924
76.721
76.403
55.235
CN
H3CO
3ca
ppm
160
140
120
100
80
60
40
20

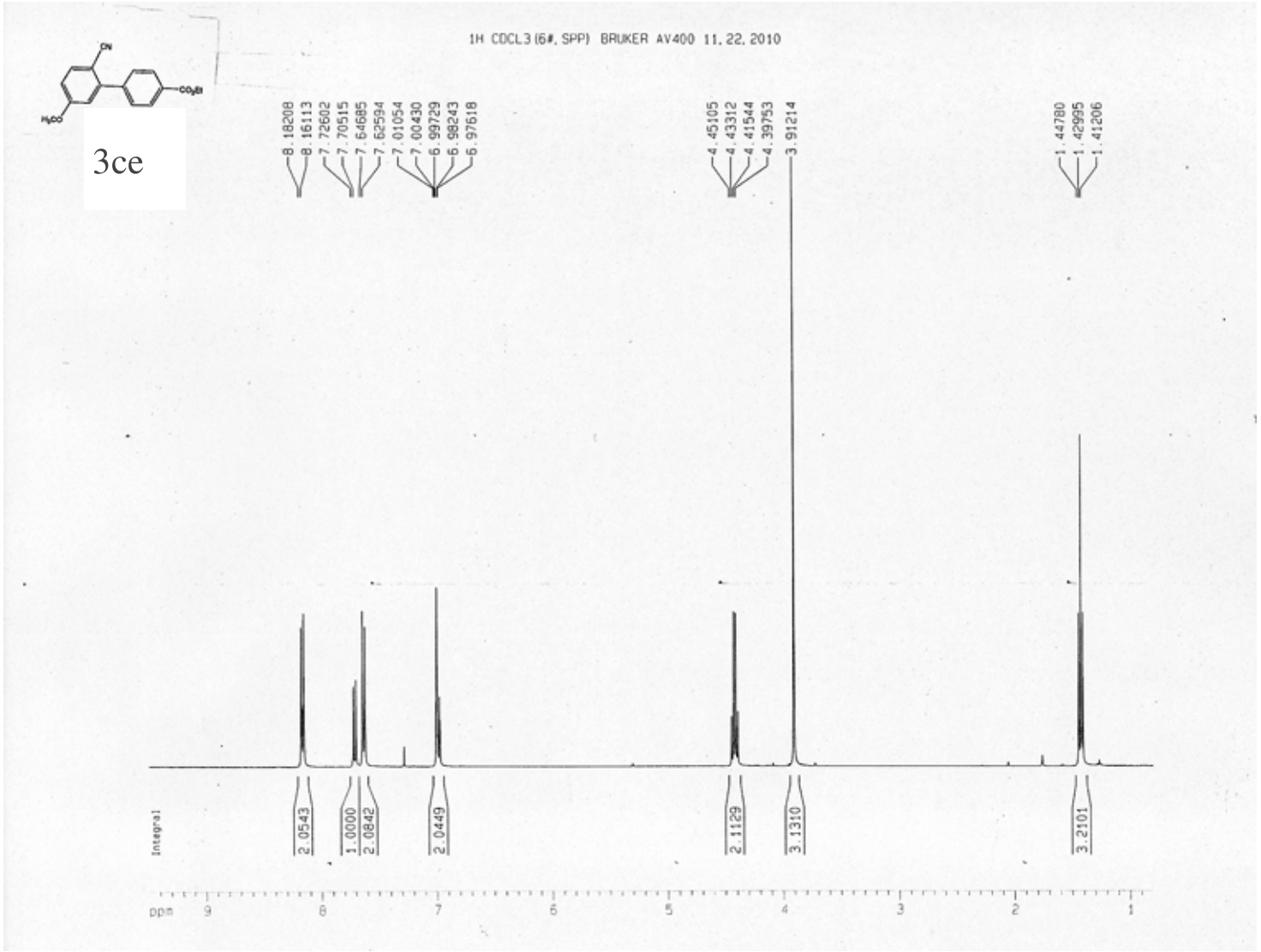

101117 (sunpp) -4 1H CDCL3 BRUKER AV400
ppm
8.39229
8.38836
8.37041
8.36517
7.78557
7.76500
7.74839
7.74379
7.73806
7.28785
7.07191
7.06561
7.05059
7.04429
7.02778
7.02169
3.95027
Integral
1.9018
2.8143
1.9149
3.0000
ppm
8
7
6
5
4
3
2
1
0
-1

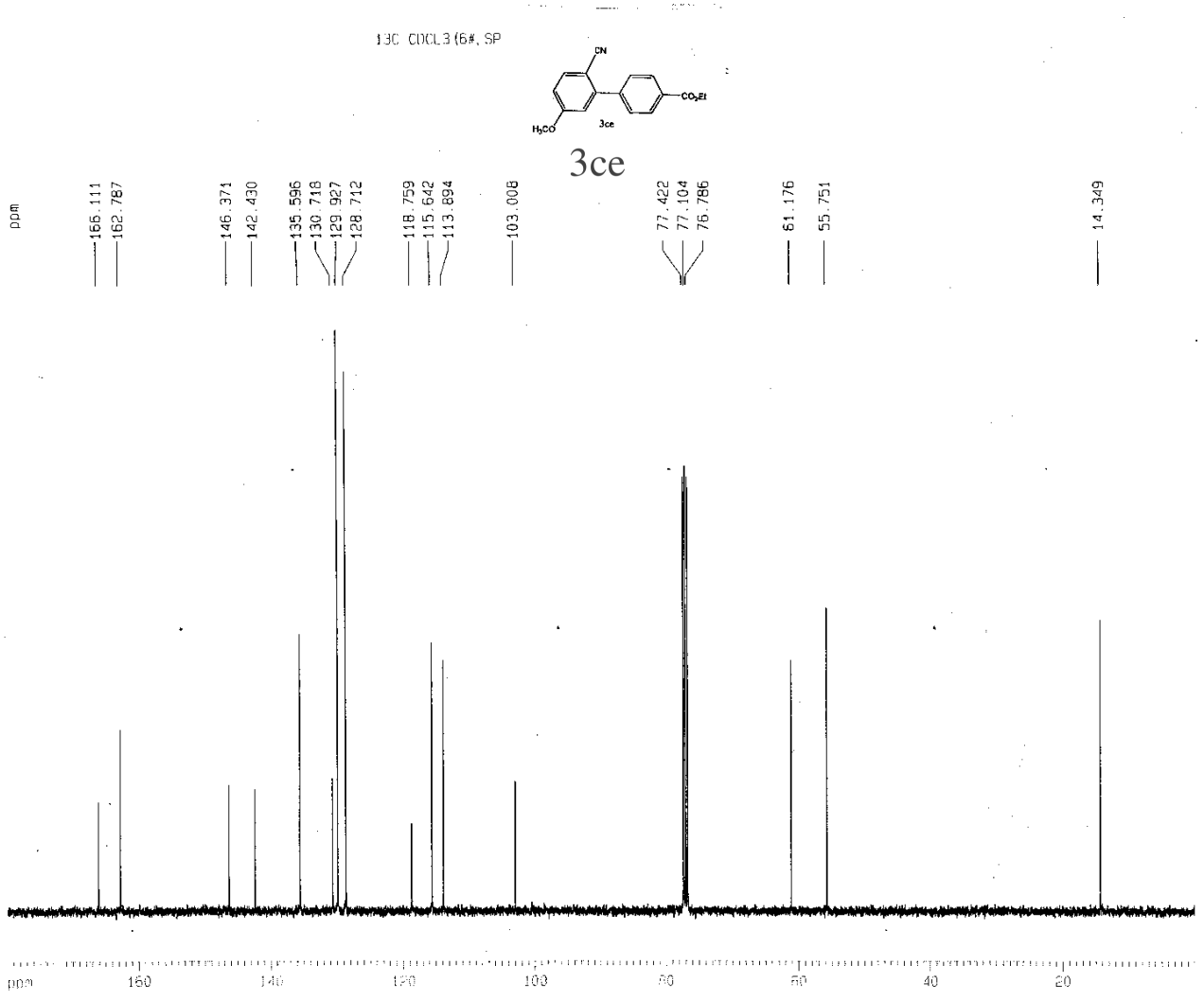
3cc

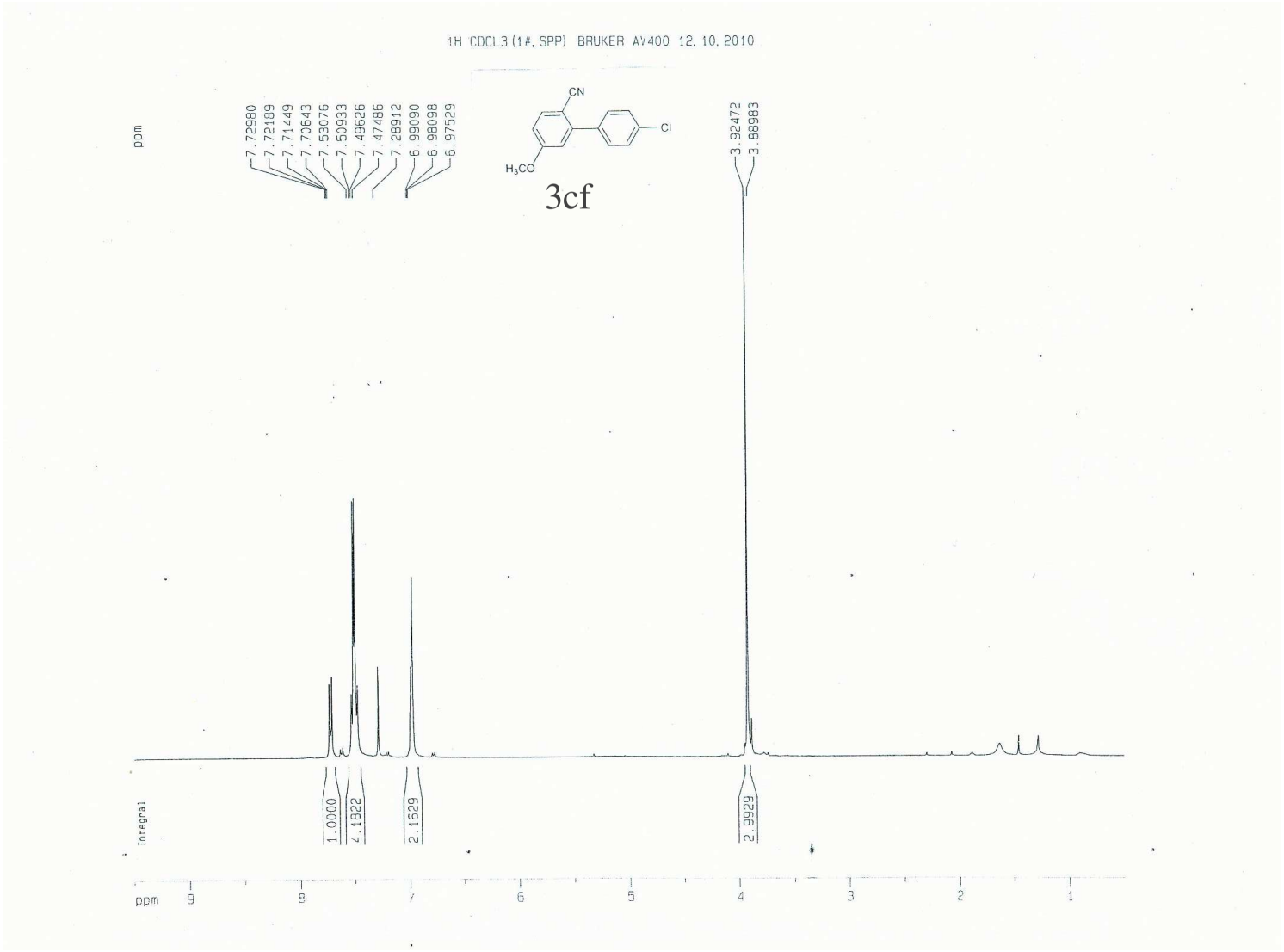

CN
NO2
H3CO
3cc
ppm
162.921
148.018
144.960
144.414
135.798
129.774
123.996
118.378
115.862
114.337
103.006
77.349
77.237
77.031
76.713
55.853
ppm
160
140
120
100
80
60
40

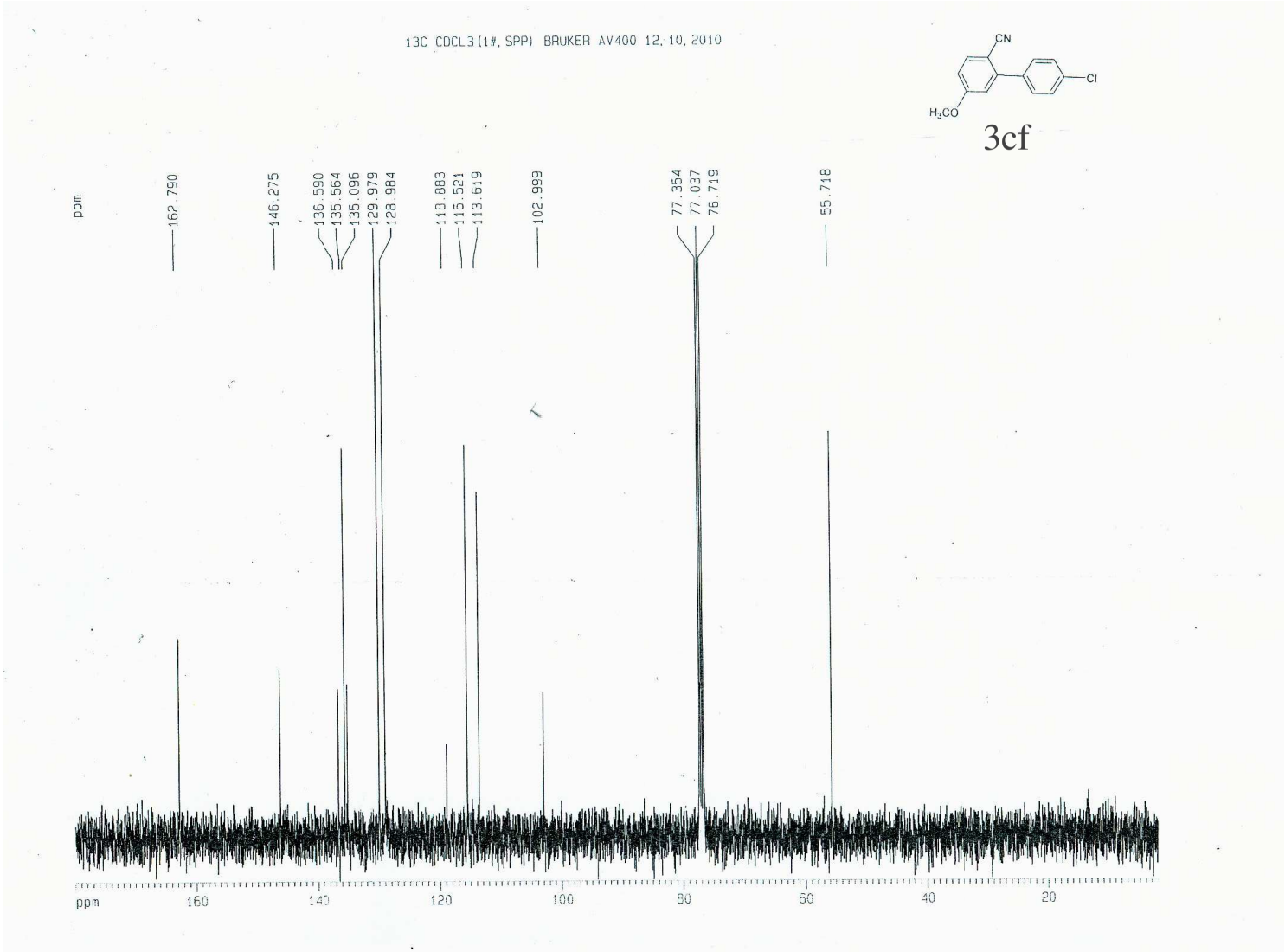

1H CDCL3 (2#, SPP) BRUKER AV400 11. 22. 2010
ppm
8.42237
8.41795
8.41354
8.36348
8.36003
8.34466
8.34276
8.33953
8.33714
7.96901
7.96673
7.95353
7.94967
7.78263
7.78087
7.76094
7.73834
7.71821
7.69845
7.28801
7.07031
7.06412
7.04381
3.95655
CN
H3CO
NO2
3cd
Integral
1.0000
1.0519
1.0779
2.1745
2.1184
3.2684
ppm
9
8
7
6
5
4
3
2

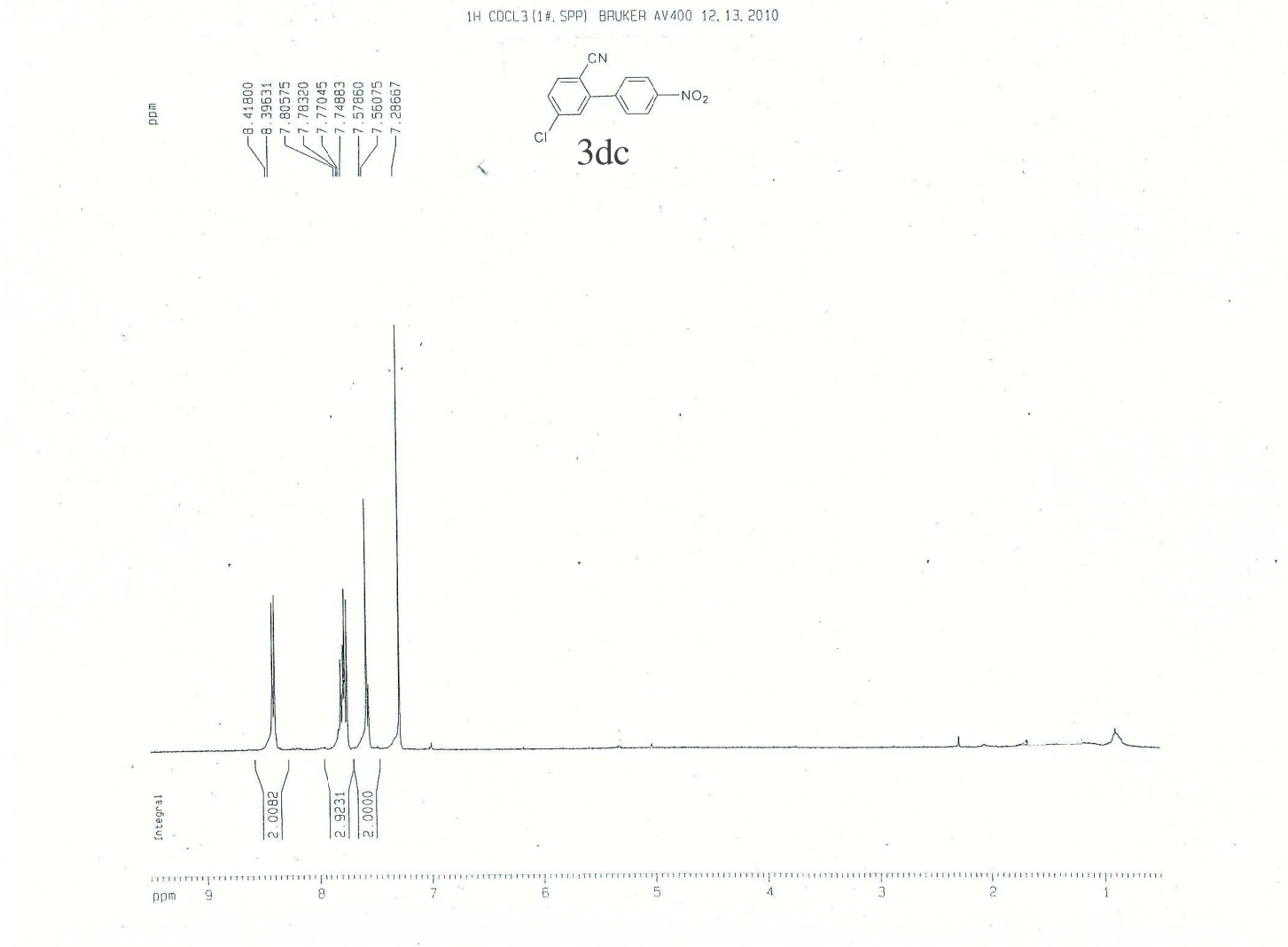

13C CDCL3 (2
CN
H3CO
3cd
NO2
010
3cd
ppm
163.007
148.456
144.847
139.727
135.696
134.720
129.808
123.715
123.632
118.345
115.785
114.351
103.132
77.330
77.217
77.012
76.694
55.847
ppm
160
140
120
100
80
60
40
20

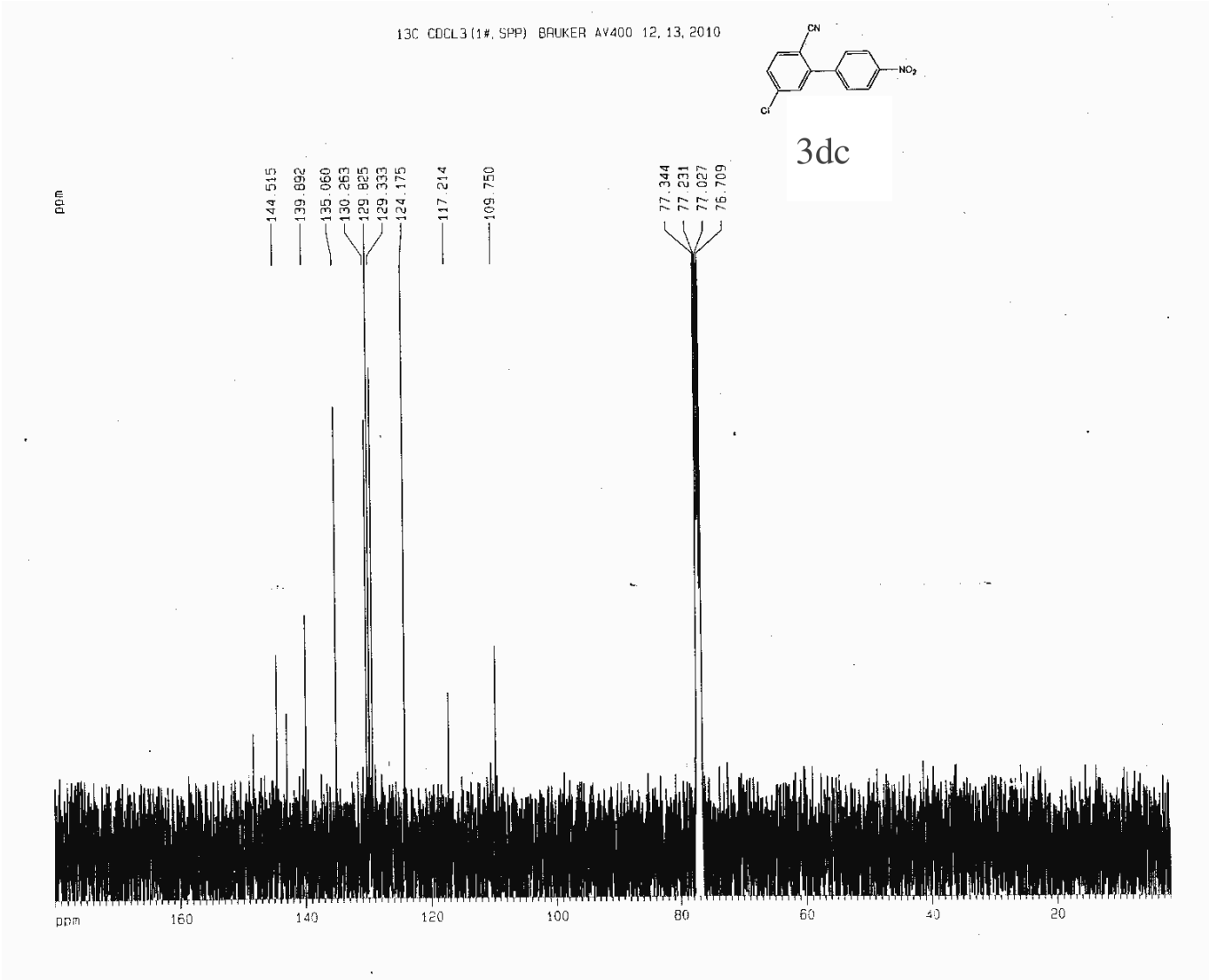

1H COCL3 (6#, SPP) BRUKER AV400 11.22.2010
3ce
8.18208
8.16113
7.72602
7.70515
7.64685
7.62594
7.01054
7.00430
6.99729
6.98243
6.97618
4.45105
4.43312
4.41544
4.39753
3.91214
1.44780
1.42995
1.41206
Integral
2.0543
1.0000
2.0842
2.0449
2.1129
3.1310
3.2101
ppm
9
8
7
6
5
4
3
2
1

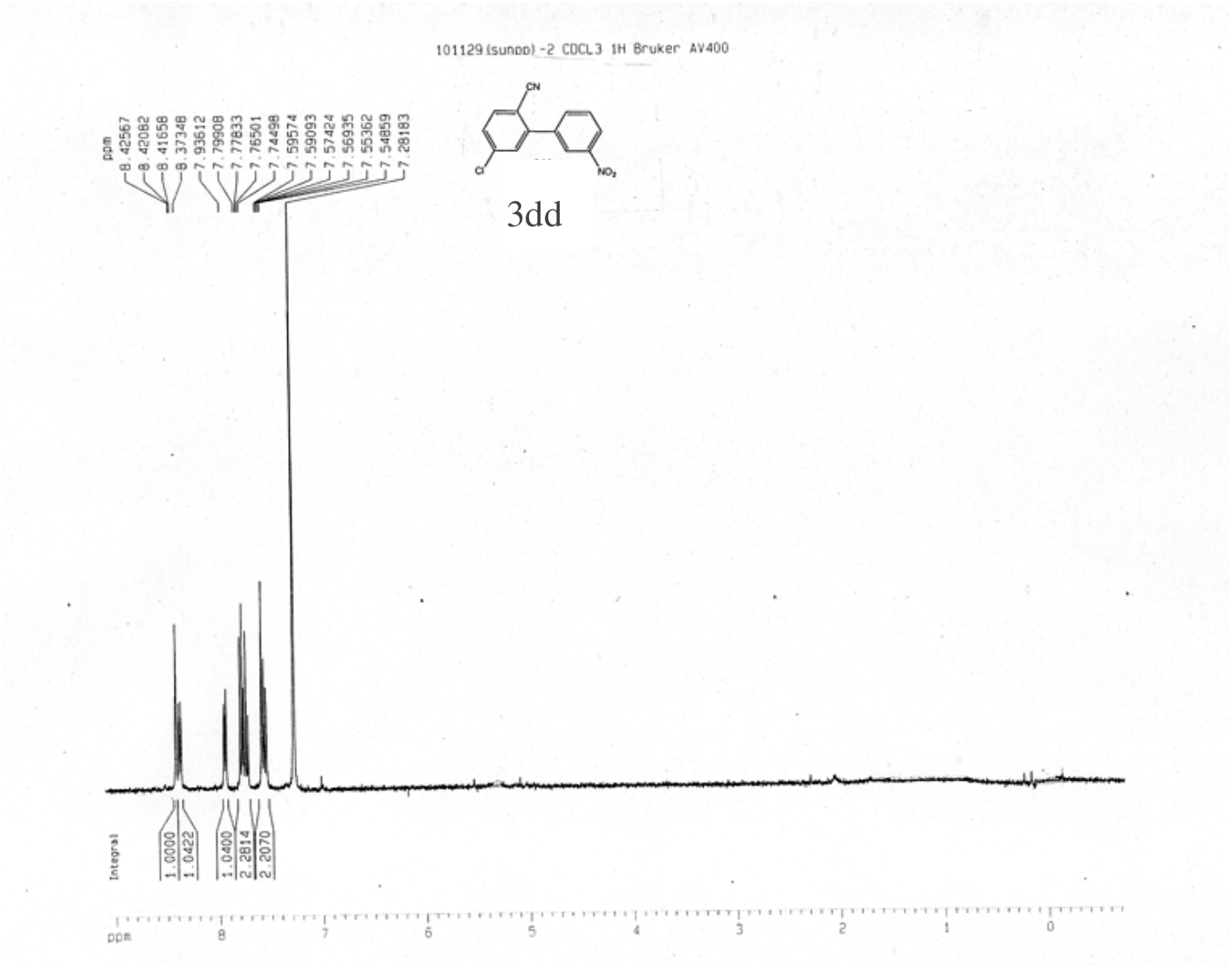

13C CDCL3 (6#, SP
CN
CO2Et
H3CO
3ce
3ce
ppm
166.111
162.787
146.371
142.430
135.596
130.718
129.927
128.712
118.759
115.642
113.894
103.008
77.422
77.104
76.786
61.176
55.751
14.349
ppm
160
140
120
100
80
60
40
20

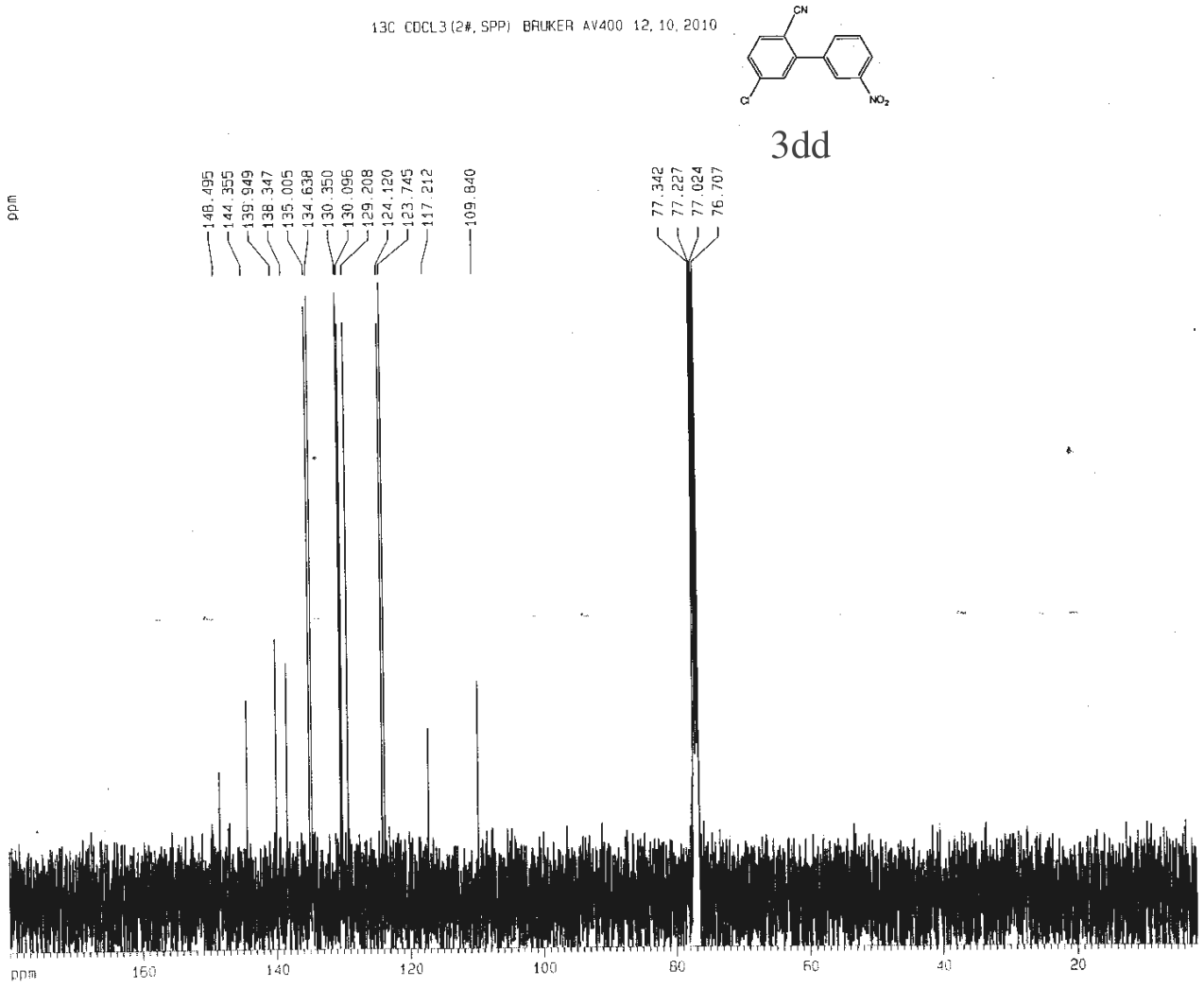

1H CDCL3 (1#, SPP) BRUKER AV400 12. 10. 2010
ppm
7. 72980
7. 72189
7. 71449
7. 70643
7. 53076
7. 50933
7. 49626
7. 47486
7. 28912
6. 99090
6. 98098
6. 97529
CN
Cl
H3CO
3cf
3. 92472
3. 88983
Integral
1. 0000
4. 1822
2. 1629
2. 9929
ppm
9
8
7
6
5
4
3
2
1

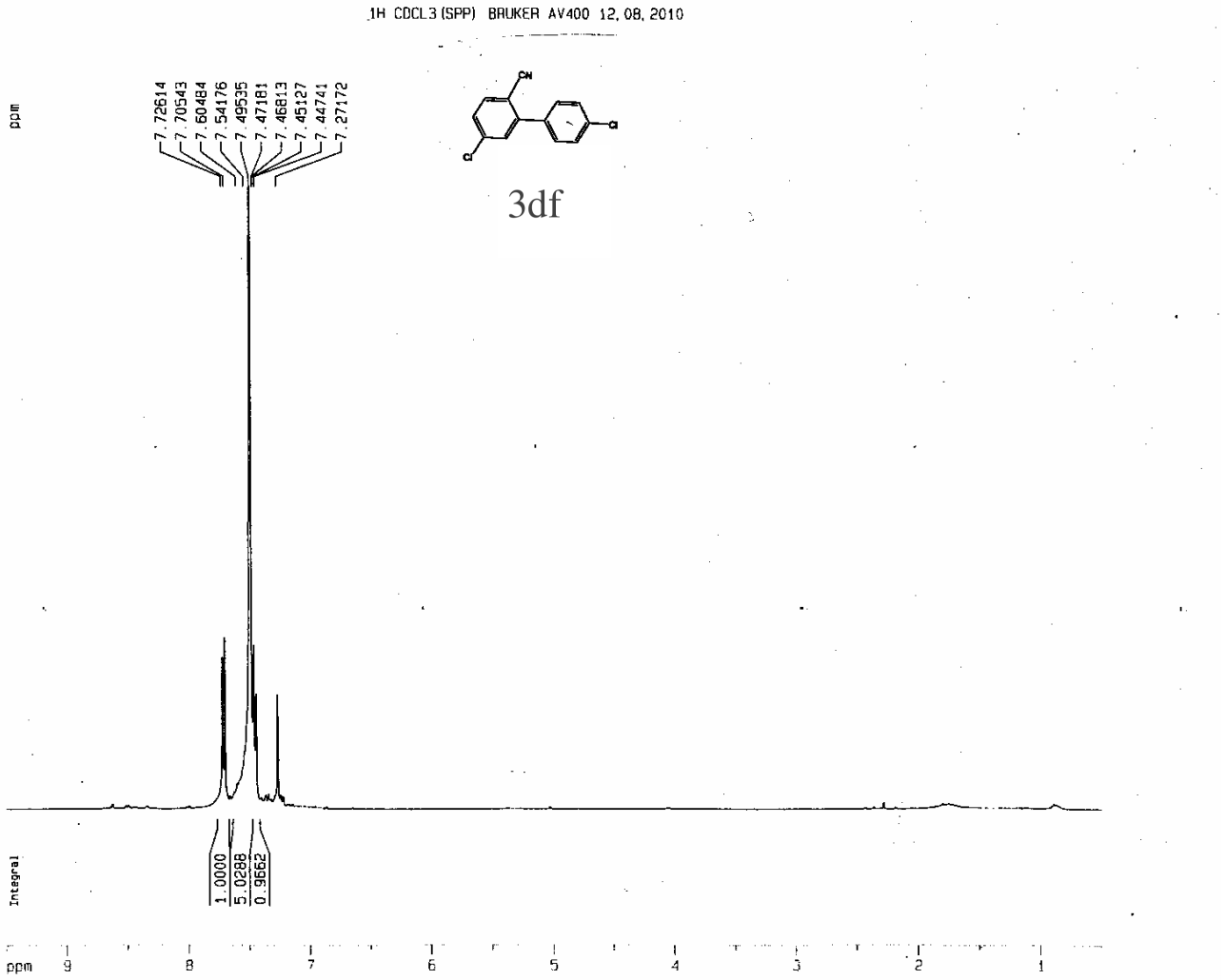

13C CDCL3 (1#, SPP) BRUKER AV400 12. 10. 2010
CN
Cl
H3CO
3cf
ppm
162.790
146.275
136.590
135.564
135.096
129.979
128.984
118.883
115.521
113.619
102.999
77.354
77.037
76.719
55.718
ppm
160
140
120
100
80
60
40
20

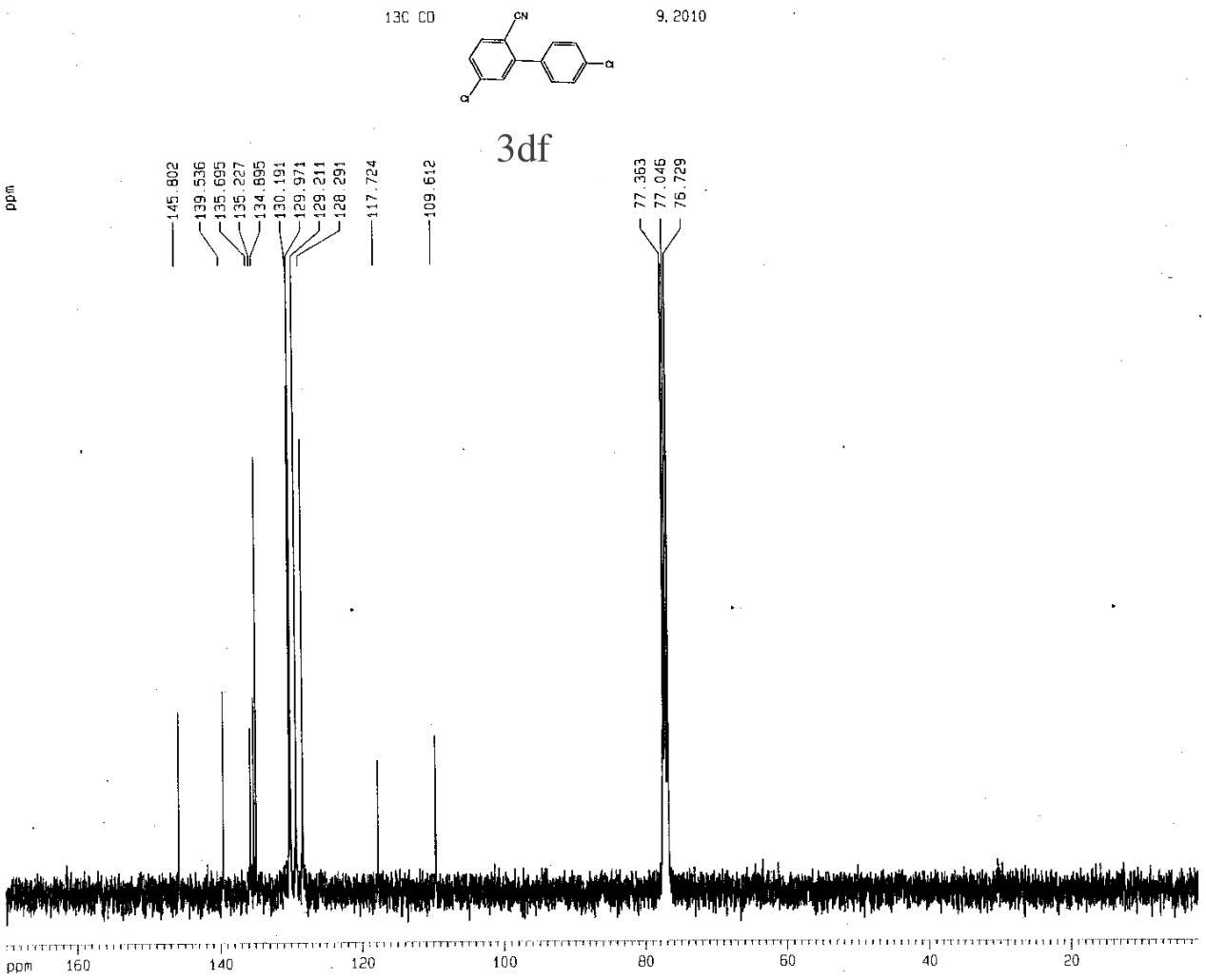

1H CDCL3 (1#, SPP) BRUKER AV400 12. 13. 2010
ppm
8.41800
8.39631
7.80575
7.78320
7.77045
7.74883
7.57860
7.56075
7.28667
CN
NO2
Cl
3dc
Integral
2.0082
2.9231
2.0000
ppm 9 8 7 6 5 4 3 2 1

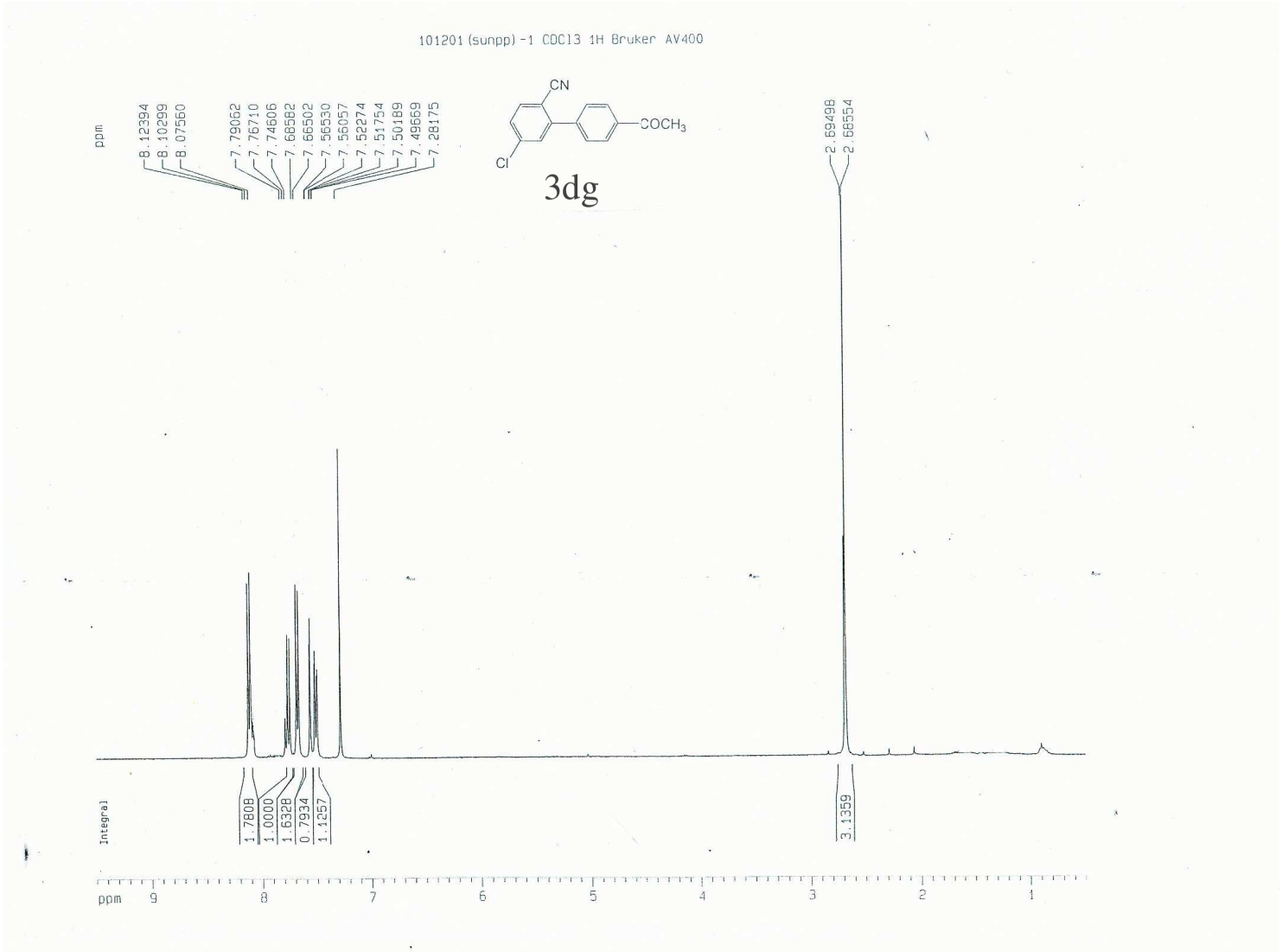

13C CDCL3 (1#, SPP) BRUKER AV400 12, 13, 2010
CN
NO2
Cl
3dc
ppm
144.515
139.892
135.060
130.263
129.825
129.333
124.175
117.214
109.750
77.344
77.231
77.027
76.709
ppm
160
140
120
100
80
60
40
20

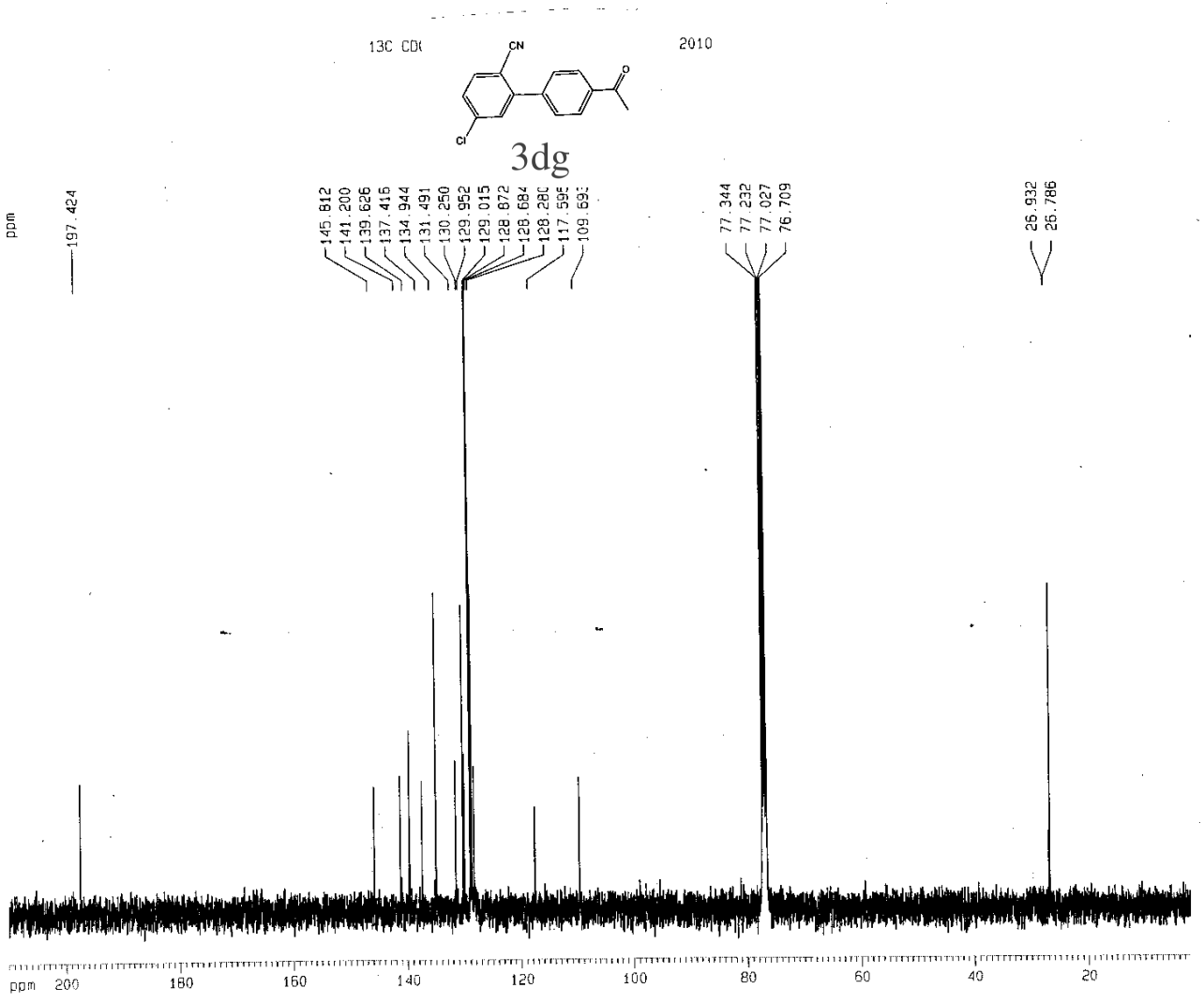

101129(sunpp)-2 CDCL3 1H Bruker AV400
ppm
8.42567
8.42062
8.41658
8.37348
7.93612
7.79908
7.77833
7.76501
7.74498
7.59574
7.59093
7.57424
7.56935
7.55362
7.54859
7.28183
CN
Cl
NO2
3dd
Integral
1.0000
1.0422
1.0400
2.2814
2.2070
ppm
8
7
6
5
4
3
2
1
0

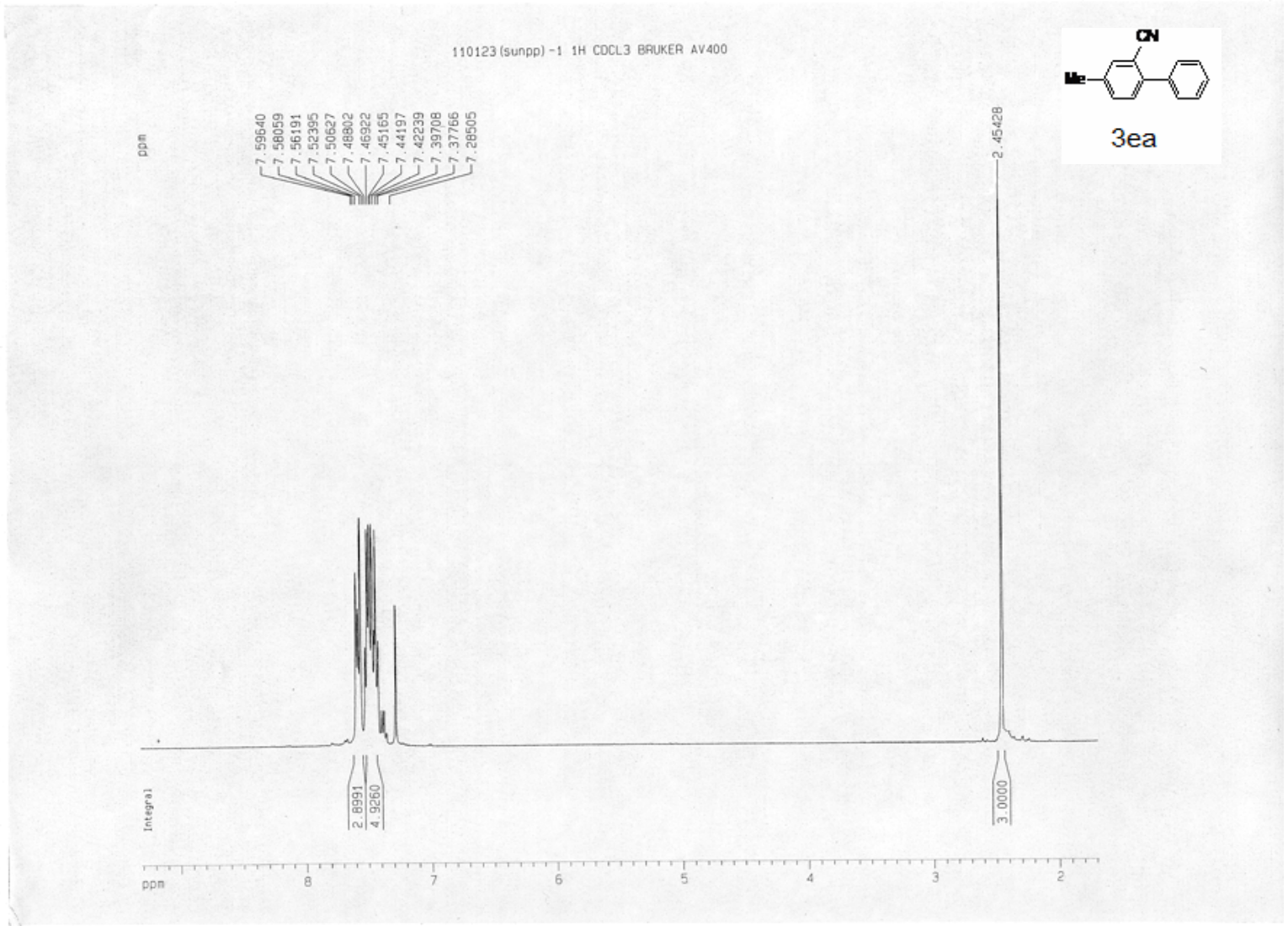

13C CDCL3 (2#, SPP) BRUKER AV400 12, 10, 2010
CN
Cl
NO2
3dd
ppm
148.495
144.355
139.949
138.347
135.005
134.638
130.350
130.096
129.208
124.120
123.745
117.212
109.840
77.342
77.227
77.024
76.707
ppm
160
140
120
100
80
60
40
20

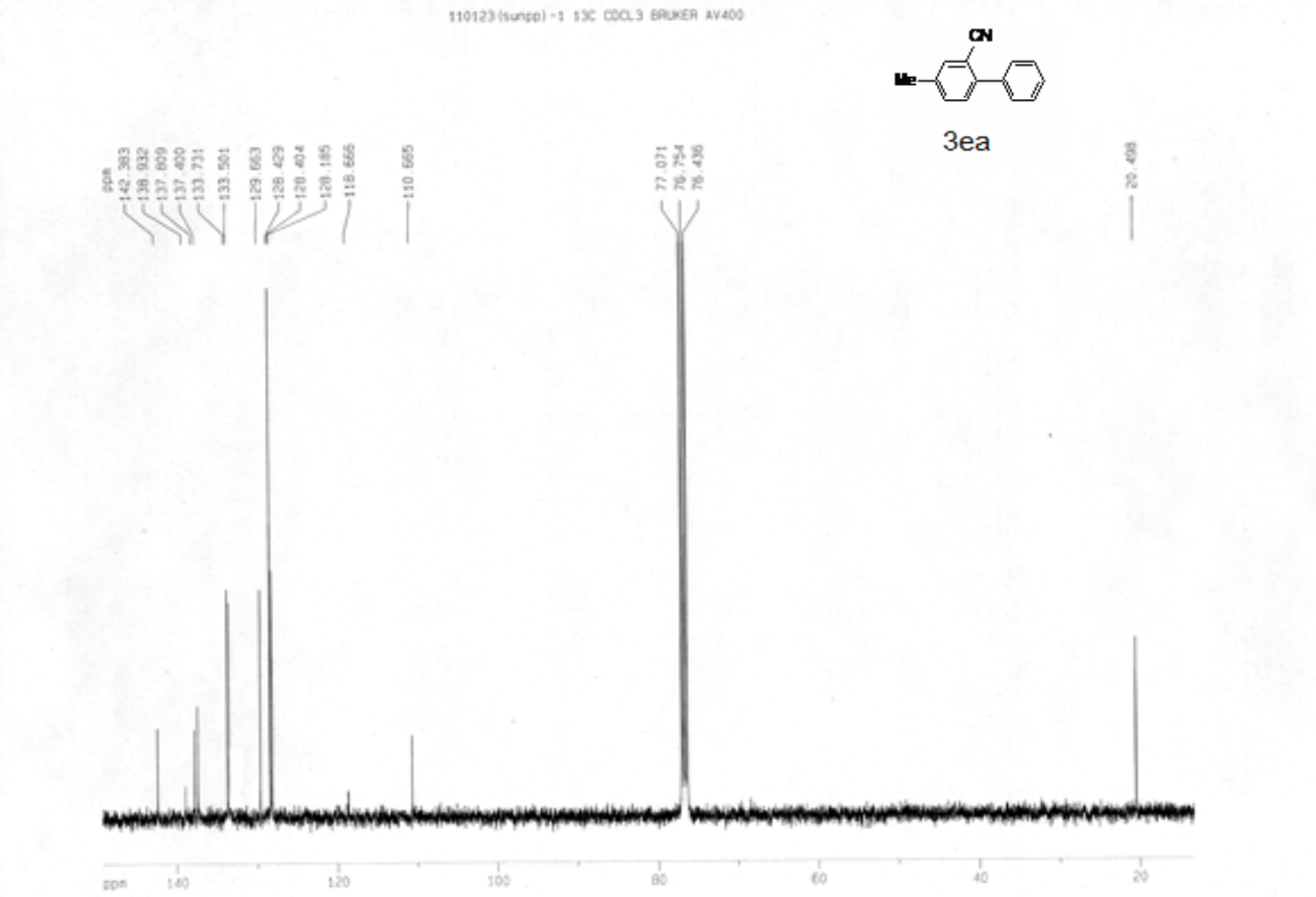

1H CDCL3 (SPP) BRUKER AV400 12, 08, 2010
ppm
7.72614
7.70543
7.60484
7.54176
7.49535
7.47181
7.46813
7.45127
7.44741
7.27172
CN
Cl
Cl
3df
Integral
1.0000
5.0288
0.9662
ppm
9
8
7
6
5
4
3
2
1

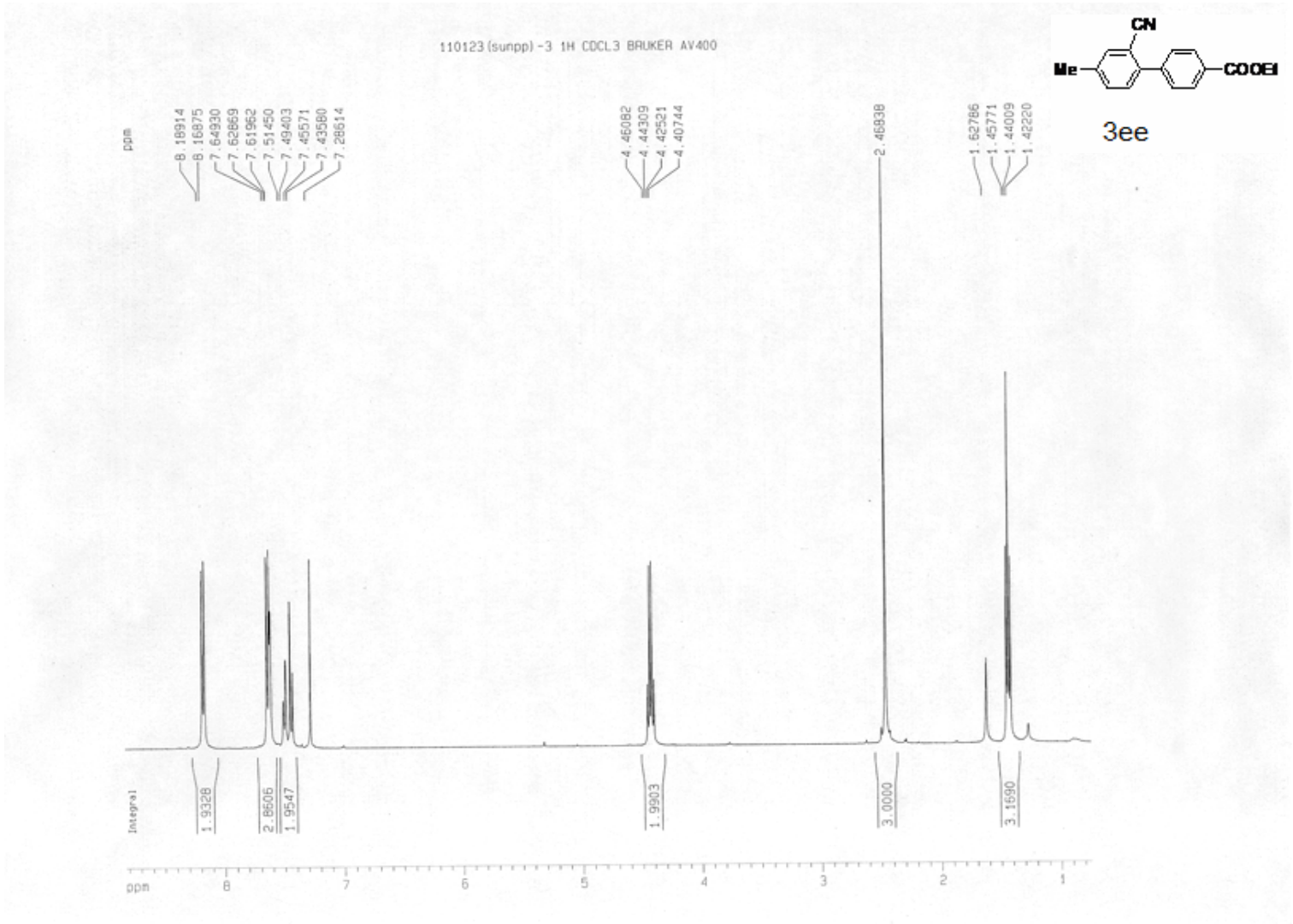

13C CD
9. 2010
CN
Cl
Cl
3df
ppm
145. 802
139. 536
135. 695
135. 227
134. 895
130. 191
129. 971
129. 211
128 291
117. 724
109. 612
77. 363
77. 046
76. 729
ppm 160
140
120
100
80
60
40
20

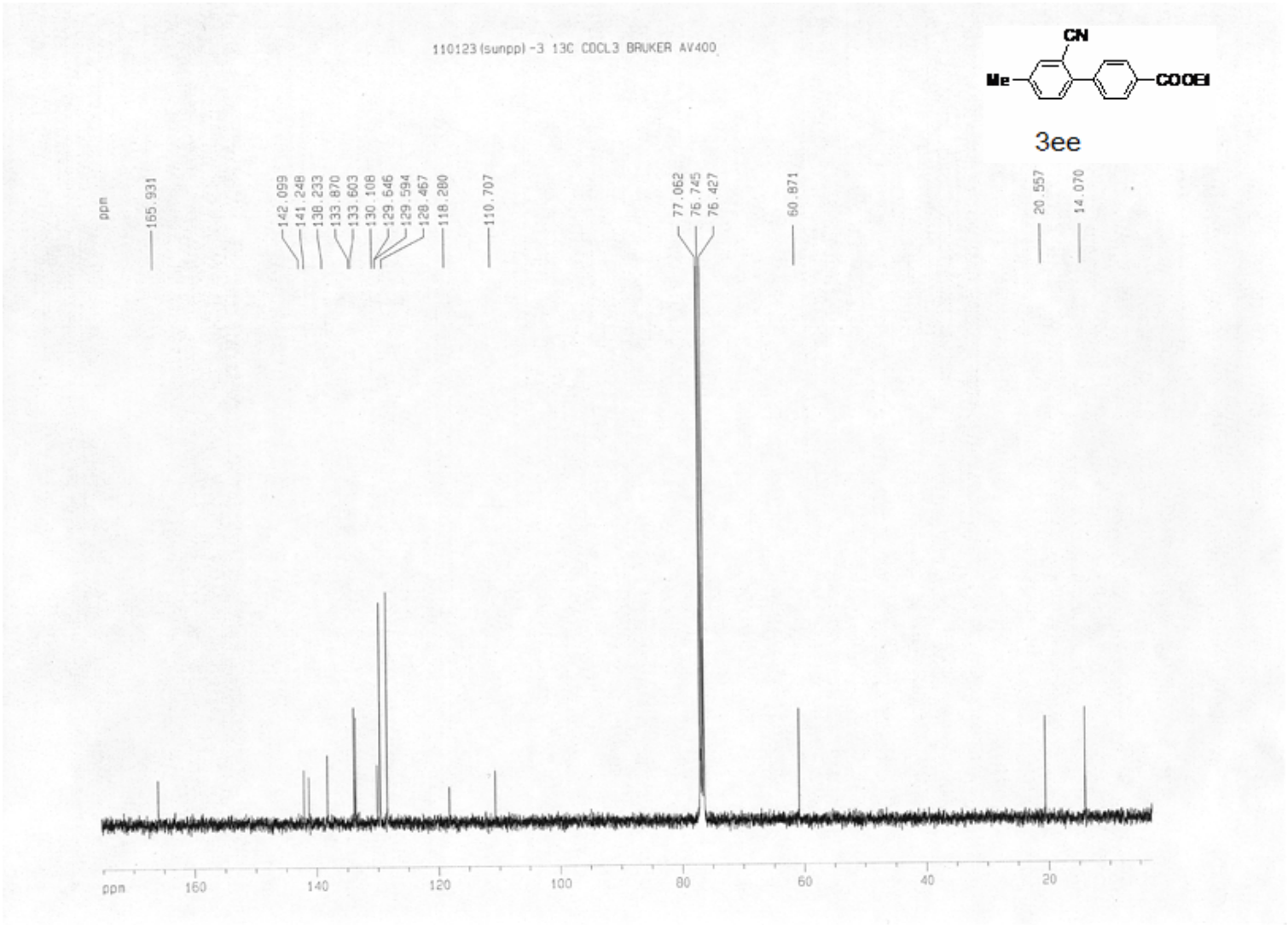

101201 (sunpp) -1 CDCl3 1H Bruker AV400
ppm
8.12394
8.10299
8.07560
7.79062
7.76710
7.74606
7.66582
7.66502
7.56530
7.56057
7.52274
7.51754
7.50189
7.49669
7.28175
CN
COCH3
Cl
3dg
2.69498
2.66554
Integral
1.7808
1.0000
1.6328
0.7934
1.1257
3.1359
ppm
9
8
7
6
5
4
3
2
1

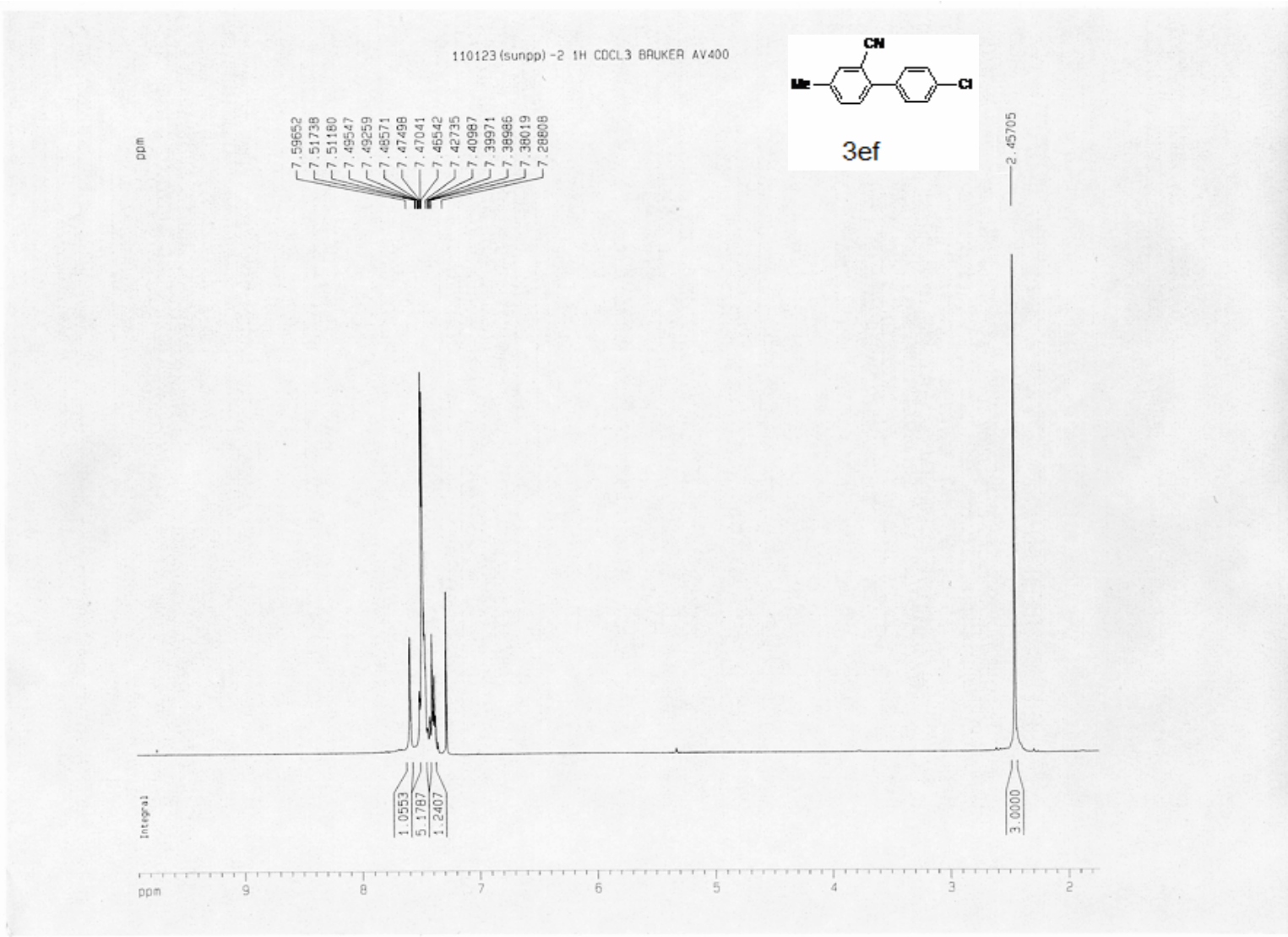

13C CDC
2010
CN
O
Cl
3dg
ppm
197.424
145.812
141.200
139.626
137.416
134.944
131.491
130.250
129.952
129.015
128.872
128.684
128.280
117.595
109.693
77.344
77.232
77.027
76.709
26.932
26.786
ppm 200
180
160
140
120
100
80
60
40
20

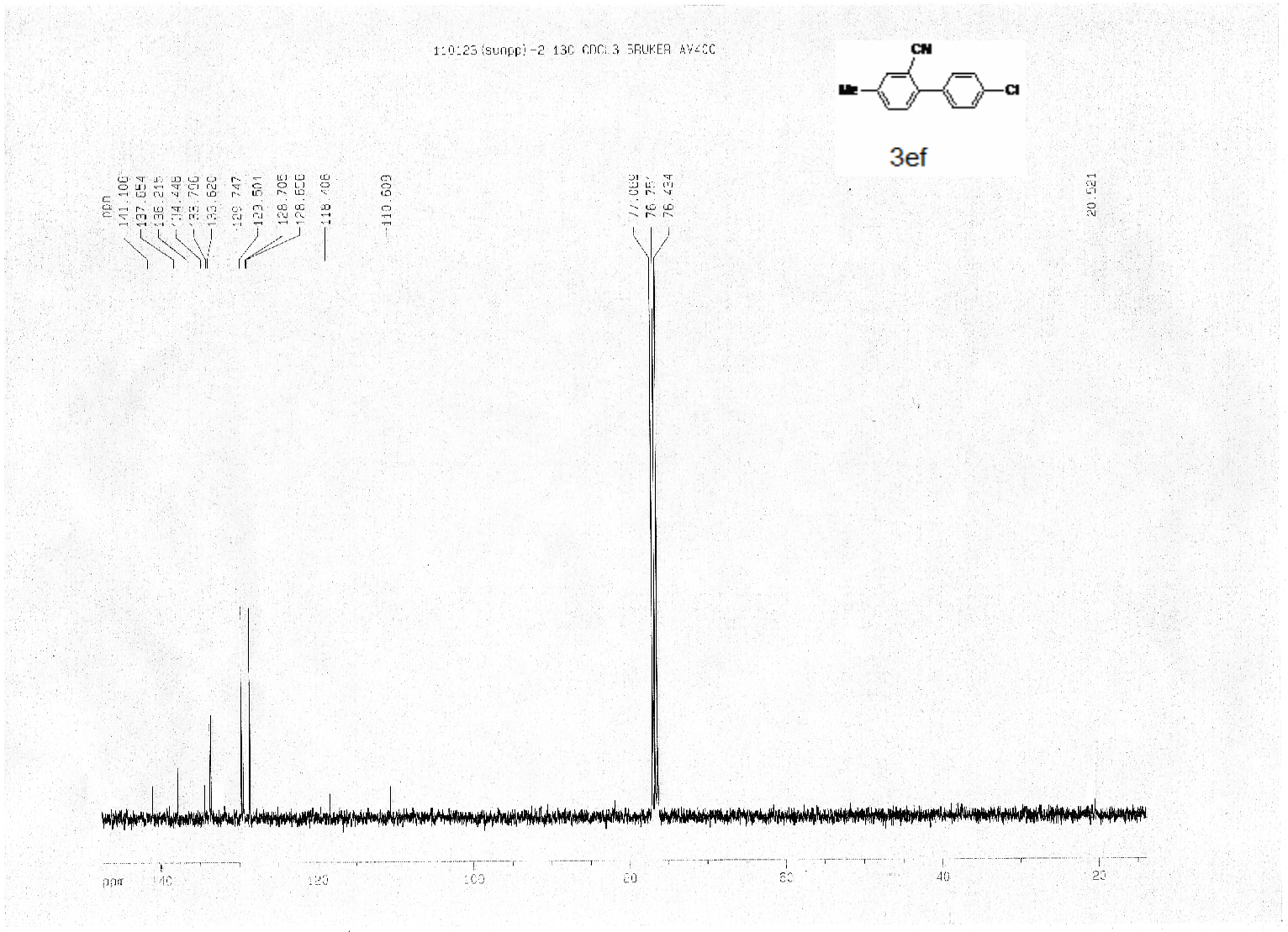

110123 (sunpp) -1 1H CDCL3 BRUKER AV400
CN
Me
3ea
ppm
7.59640
7.58059
7.56191
7.52395
7.50627
7.48802
7.46922
7.45165
7.44197
7.42239
7.39708
7.37766
7.28505
2.45428
Integral
2.8991
4.9260
3.0000
ppm
8
7
6
5
4
3
2


110123 (sunpp) -1 13C CDCL3 BRUKER AV400
CN
Me
3ea
ppm
142.383
138.932
137.609
137.400
133.731
133.501
129.663
128.429
128.404
128.185
118.666
110.685
77.071
76.754
76.436
20.498
ppm
140
120
100
80
60
40
20


110123(sunpp)-3 1H CDCL3 BRUKER AV400
CN
Me
COOEt
3ee
ppm
8.18914
8.16875
7.64930
7.62869
7.61962
7.51450
7.49403
7.45571
7.43580
7.28614
4.46082
4.44309
4.42521
4.40744
2.46838
1.62786
1.45771
1.44009
1.42220
Integral
1.9328
2.8606
1.9547
1.9903
3.0000
3.1690
ppm
8
7
6
5
4
3
2
1


110123(sunpp)-3 13C CDCL3 BRUKER AV400
CN
Me
COOEt
3ee
ppm
165.931
142.099
141.248
138.233
133.870
133.603
130.108
129.646
129.594
128.467
118.280
110.707
77.062
76.745
76.427
60.871
20.557
14.070
ppm
160
140
120
100
80
60
40
20


110123 (sunpp) -2 1H CDCL3 BRUKER AV400
CN
Me
Cl
3ef
ppm
7.59652
7.51738
7.51180
7.49547
7.49259
7.48571
7.47498
7.47041
7.46542
7.42735
7.40987
7.39971
7.38986
7.38019
7.28808
2.45705
Integral
1.0553
5.1787
1.2407
3.0000
ppm
9
8
7
6
5
4
3
2


110123(sunpp)-2 13C CDCl3 BRUKER AV400
CN
Me
Cl
3ef
ppm
141.100
137.654
136.215
114.446
133.620
129.747
123.501
128.705
128.656
118.406
110.609
77.089
76.757
76.434
20.581
ppm
140
120
100
80
60
40
20