SUPPORTING INFORMATION

Thio FCMA Intermediates as Strong Acyl Donors: A General Solution to the Formation of Complex Amide Bonds

Yu Rao, Xuechen Li, Samuel J Danishefsky*

Laboratory for Bioorganic Chemistry, Sloan-Kettering Institute for Cancer Research, 1275 York Avenue, New York, NY 10065, USA

Department of Chemistry, Columbia University, 3000 Broadway, New York, NY 10027.

Materials and Methods

All commercial materials (Aldrich, Fluka) were used without further purification. All solvents were reagent grade or HPLC grade (Fisher). Anhydrous THF, diethyl ether, CH_2Cl_2, toluene, and benzene were obtained from a dry solvent system (passed through column of alumina) and used without further drying. All reactions were performed under an atmosphere of pre-purified dry Ar(g). ^{1}H NMR spectra and ^{13}C NMR spectra were recorded on a Bruker Advance DRX-500 MHz or DRX-600 MHz at ambient temperature unless otherwise stated. Chemical shifts are reported in parts per million relative to residual solvent $CDCl_3$ (^{1}H, δ 7.27 ppm; ^{13}C, 77.23 ppm). All ^{13}C NMR spectra were recorded with complete proton decoupling. Low-resolution mass spectral analyses were performed with a JOEL JMS-DX-303-HF mass spectrometer or Waters Micromass ZQ mass spectrometer. All reactions were carried out in oven-dried glassware under argon atmosphere unless otherwise noted. Analytical TLC was performed on E. Merck silica gel 60 F254 plates and flash column chromatography was performed on E. Merck silica gel 60 (40–63 μm).

General Procedure for the Preparation of Thio Acid:

To an oven dried round bottom flask was charged amino acid (2.00 mmol) and 5 mL anhydrous THF. The resulting solution was cooled to -10 °C and N-methyl morpholine (2.50 mmol) was added dropwise, followed by the addition of isobutyl chloroformate

(2.50 mmol). The reaction mixture was warmed to 0 °C and stirred for another 30 min, then a stream of H_2S gas (Cautions: H_2S is highly toxic and must be used in a well ventilated fume hood) was bubbled though the reaction mixture for 1 h at room temperature. THF was removed by a stream of N_2 in the fume hood and the residue was suspended in 40 mL EtOAc. The organic layer was washed by 1 N HCl and brine, dried over anhydrous sodium sulfate. Solvent was removed under vacuum and the residue was used as such without further purification.

General Procedure for Three-Component-Coupling Reactions:

To a CH_2Cl_2 (1.0 mL) solution of thio acid (0.20 mmol) was added t-butyl isonitrile (0.40 mmol) and amine (0.60 mmol). This reaction mixture was stirred at room temperature for 2 h before quenching with 1N HCl. After sequential washing with 1N HCl, $NaHCO_3$(sat.) and brine, the organic layer was dried over Na_2SO_4. Then, after filetration, CH_2Cl_2 was removed under vacuum. The residue was purified by flash column chromatography.

(S)-allyl 4-(((9H-fluoren-9-yl)methoxy)carbonylamino)-5-(N-cyclohexylmethanethioamido)-5-oxopentanoate (12)

To a CH_2Cl_2 (1.0 mL) solution of thio acid **10** (0.10 mmol) was added cyclohexyl isonitrile (0.15 mmol). This reaction mixture was stirred at room temperature for 4 h and CH_2Cl_2 was removed under vacuum. The residue was purified by flash column chromatography. Compound **12** was isolated in 55% yield (29.4 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 10.50 (s, 1H), 7.71 (m, 2H), 7.52 (m, 2H), 7.35 (m, 2H), 7.25 (m, 2H), 5.87 (m, 1H), 5.49 (d, J = 8.4 Hz, 1H), 5.27 (m, 3H), 5.04 (m, 1H), 4.56 (d, J = 5.4 Hz, 2H), 4.36 (d, J = 6.7 Hz, 2H), 4.15 (t, J = 6.7 Hz, 1H), 2.42 (m, 2H), 2.13 (m, 2H), 1.77 (m, 2H), 1.61 (m, 2H), 1.20 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 194.4, 174.1, 172.0, 156.1, 143.7, 143.5, 141.3, 131.8, 127.9, 127.2, 125.0, 120.1, 118.8, 67.3, 65.7, 60.4, 57.7, 52.5, 47.1, 31.6, 29.7, 29.5, 29.0, 28.3, 26.2, 25.3, 22.64, 22.61; IR (thin film) 2934, 2855, 1711, 1540, 1450, 1403, 1358, 1316, 1245, 1179, 1129, 1071, 988, 936, 894 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{30}H_{34}N_2O_5S$ 557.2086, found 557.2082.

(S)-allyl 4-(((9H-fluoren-9-yl)methoxy)carbonylamino)-5-oxo-5-(phenylamino)pentanoate (13)

To a CH_2Cl_2 (1.0 mL) solution of thio acid **10** (0.10 mmol) were added t-butyl isonitrile (0.20 mmol) and aniline (0.30 mmol). This reaction mixture was stirred at room temperature for 2 h before quenching with 1N HCl. After sequential washing with 1N HCl, $NaHCO_3$(sat.) and brine, the organic layer was dried over Na_2SO_4. Then, after filetration, CH_2Cl_2 was removed under vacuum. The residue was purified by flash column chromatography. Compound **13** was isolated in 77% yield (37.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 8.25 (brs, 1H), 7.70 (m, 2H), 7.52 (m, 2H), 7.46 (m, 2H), 7.32 (m, 2H), 7.22 (m, 4H), 7.07 (m, 1H), 5.87 (m, 1H), 5.67 (brs, 1H), 5.28 (m, 2H), 4.56 (d, J = 5.6 Hz, 2H), 4.37 (d, J = 6.7 Hz, 2H), 4.29 (s, 1H), 4.16 (t, J = 6.9 Hz, 1H), 2.60 (m, 1H), 2.44 (m, 1H), 2.17 (m, 1H), 1.98 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 173.5, 169.3, 156.6, 143.7, 143.6, 141.31, 141.30, 137.4, 131.8, 129.0, 127.8, 127.1, 125.0, 124.6, 120.0, 119.9, 118.7, 67.3, 65.6, 54.8, 47.1, 38.6, 30.5, 28.1; IR (thin film) 3293, 1729, 1689, 1659, 1599, 1536, 1445, 1389, 1276, 1257, 1187, 1168, 1085, 1046, 845 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{29}H_{28}N_2O_5$ 507.1896, found 507.1898.

N-phenylbenzamide (18)

Following the general procedure, compound **18** was isolated in 88% yield (34.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 7.91 (m, 2H), 7.81 (s, 1H), 7.68 (d, J = 7.7 Hz, 2H), 7.59 (m, 1H), 7.53 (m, 2H), 7.43 (m, 2H), 7.20 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 165.8, 137.9, 135.0, 131.8, 129.3, 129.1, 128.8, 127.0, 124.6, 120.2, 118.5, 115.1; IR (thin film) 3344, 1735, 1654, 1600, 1537, 1438, 1323, 1260, 1116, 928, 886 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{13}H_{11}NO$ 198.0919, found 198.0916.

(R)-benzyl 2-(tert-butoxycarbonylamino)-4-oxo-4-(phenylamino)butanoate (19)

Following the general procedure, compound **19** was isolated in 86% yield (68.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 7.49 (m, 2H), 7.44 (s, 1H), 7.36 (m, 7H), 7.16 (m, 1H), 5.81 (d, J = 6.9 Hz, 1H), 5.26 (d, J = 12.2 Hz, 1H), 5.21 (d, J = 12.2 Hz, 1H), 4.65 (m, 1H), 3.12 (dd, J = 3.6 Hz, J = 12.9 Hz, 1H), 2.96 (dd, J = 3.9 Hz, J = 15.9 Hz, 1H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 172.7, 169.7, 157.3, 139.0, 136.8, 130.4, 129.9,

129.8, 129.6, 125.9, 121.4, 81.6, 68.9, 61.9, 51.9, 40.5, 29.7, 22.5, 15.6; IR (thin film) 3318, 2978, 1736, 1686, 1600, 1544, 1497, 1443, 1367, 1250, 1213, 1161, 1054, 1026, 978 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{22}H_{26}N_2O_5$ 421.1753, found 421.1749.

N-phenylpivalamide (20)

Following the general procedure, compound **20** was isolated in 83% yield (29.4 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.45 (m, 2H), 7.25 (m, 3H), 7.03 (m, 1H), 1.29 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 176.6, 138.0, 129.0, 124.2, 120.0, 39.6, 27.6; IR (thin film) 3313, 2985, 2965, 2931, 1654, 1596, 1533, 1490, 1437, 1400, 1317, 1241, 1169, 928, 903 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{11}H_{15}NO$ 178.1232, found 178.1225.

N-Phenyl-1-adamantanecarboxamide (21)

Following the general procedure, compound **21** was isolated in 85% yield (43.4 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.35 (m, 2H), 7.13 (m, 3H), 6.91 (m, 1H), 1.91 (s, 3H), 1.78 (s, 6H), 1.58 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 176.1, 138.1, 128.9, 124.1, 120.0, 41.5, 39.3, 36.5, 28.2; IR (thin film) 3295, 2899, 2850, 1718, 1644, 1596, 1537, 1503, 1490, 1438, 1328, 1309, 1254, 1178, 925 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{17}H_{21}NO$ 256.1701, found 256.1704.

N-tert-butylpivalamide (22)

Following the general procedure, compound **22** was isolated in 61% yield (19.1 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 5.32 (s, 1H), 1,25 (s, 9H), 1.10 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 179.2, 52.1, 40.4, 30.1, 29.1; IR (thin film) 3375, 2966, 2930, 1637, 1525, 1449, 1361, 1297, 1238, 1223, 1198, 907 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_9H_{19}NO$ 158.1545, found 158.1543.

Adamantane-1-carboxylic acid tert-butylamide (23)

Following the general procedure, compound **23** was isolated in 72% yield (33.8 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 5.38 (s, 1H), 2.05 (m, 3H), 1.84 (m, 6H), 1.74 (m, 6H), 1.36 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 178.8, 51.9, 42.3, 40.8, 38.0, 30.2, 29.6; IR (thin

film) 3332, 2957, 2910, 2849, 1732, 1635, 1533, 1445, 1359, 1287, 1229, 1177, 1105, 912 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{15}H_{25}NO$ 236.2014, found 236.2025.

N-phenylcinnamamide (24)

Following the general procedure, compound **24** was isolated in 86% yield (38.3 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.81 (d, J = 15.5 Hz, 1H), 7.66 (m, 2H), 7.58 (m, 2H), 7.44-7.38 (m, 6H), 7.18 (m, 1H), 6.59 (d, J = 15.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 164.2, 142.4, 138.1, 134.6, 129.9, 129.1, 128.8, 128.0, 124.4, 120.9, 120.1; IR (thin film) 3273, 3060, 1661, 1622, 1596, 1542, 1498, 1441, 1343, 1295, 1246, 1184, 988, 976, 904, 862 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{15}H_{13}NO$ 224.1075, found 224.1070.

N-tert-butylbenzamide (25)

Following the general procedure, compound **25** was isolated in 83% yield (29.4 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.53 (m, 2H), 7.29 (m, 1H), 7.21 (m, 2H), 5.74 (s, 1H), 1.29 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 166.9, 135.9, 131.0, 128.4, 126.7, 51.6, 28.9; IR (thin film) 3320, 2976, 1634, 1533, 1490, 1451, 1362, 1312, 1217, 1027, 936, 876 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{11}H_{15}NO$ 178.1232, found 178.1228.

(R)-benzyl 2-(tert-butoxycarbonylamino)-4-(tert-butylamino)-4-oxobutanoate (26)

Following the general procedure, compound **26** was isolated in 77% yield (58.2 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.37 (m, 5H), 5.86 (d, J = 8.3 Hz, 1H), 5.35 (s, 1H), 5.26 (d, J = 12.4 Hz, 1H), 5.18 (d, J = 12.4 Hz, 1H), 4.54 (m, 1H), 2.85 (dd, J = 4.8 Hz, J = 16.1 Hz, 1H), 2.67 (dd, J = 3.9 Hz, J = 15.4 Hz, 1H), 1.45 (s, 9H), 1.32 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 171.5, 169.1, 155.8, 135.5, 128.5, 128.2, 128.0, 79.8, 67.2, 60.4, 51.5, 50.7, 38.6, 28.7, 28.3; IR (thin film) 3339, 2974, 2933, 1741, 1715, 1654, 1540, 1497, 1455, 1366, 1253, 1223, 1166, 1053, 1026 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{20}H_{30}N_2O_5$ 401.2052, found 401.2056.

N-tert-butylcinnamamide (27)

Following the general procedure, compound **27** was isolated in 81% yield (32.9 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.39 (d, J = 15.6 Hz, 1H), 7.29 (m, 2H), 7.16 (m, 3H), 6.15

(d, J = 15.6 Hz, 1H), 5.26 (s, 1H), 1.25 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 166.8, 141.5, 136.5, 130.8, 130.2, 129.1, 123.6, 52.9, 30.3; IR (thin film) 3289, 2966, 1655, 1617, 1545, 1448, 1342, 1246, 1224, 1213, 986, 977 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{13}H_{17}NO$ 204.1388, found 204.1392.

2,2-dimethyl-1-(pyrrolidin-1-yl)propan-1-one (28)

Following the general procedure, compound **28** was isolated in 85% yield (26.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 3.32 (brs, 4H), 1.63 (brs, 4H), 1.04 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 176.4, 47.8, 38.9, 27.5, 23.0; IR (thin film) 2968, 2874, 1616, 1479, 1407, 1363, 1339, 1213, 1134, 1167, 948, 918 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_9H_{17}NO$ 178.1208, found 178.1201.

2,2-dimethyl-1-morpholinopropan-1-one (29)

Following the general procedure, compound **29** was isolated in 79% yield (27.0 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 3.48 (m, 8H), 1.09 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 177.8, 68.3, 47.1, 40.0, 29.7; IR (thin film) 2965, 2859, 1613, 1416, 1271, 1260, 1186, 1104, 1018, 934, 841 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_9H_{17}NO_2$ 172.1338, found 172.1332.

1-(1-adamantylcarbonyl)-Pyrrolidine (30)

Following the general procedure, compound **30** was isolated in 82% yield (38.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 3.60 (s, 4H), 2.05 (m, 9H), 1.85 (m, 4H), 1.76 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 177.3, 49.5, 43.1, 39.6, 38.1, 29.8; IR (thin film) 2976, 2904, 2848, 1604, 1446, 1399, 1368, 1335, 1186, 1103, 976, 937 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{15}H_{23}NO$ 234.1858, found 234.1857.

4-(1-adamantylcarbonyl)-Morpholine (31)

Following the general procedure, compound **31** was isolated in 80% yield (39.8 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 3.72 (m, 8H), 2.07 (m, 3H), 2.02 (m, 6H), 1.78 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 177.2, 68.4, 47.3, 43.0, 40.4, 38.0, 29.8; IR (thin film) 2903,

2849, 1623, 1447, 1402, 1258, 1227, 1115, 1022, 973, 940 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{15}H_{23}NO_2$ 250.1807, found 250.1803.

(E)-3-phenyl-1-(pyrrolidin-1-yl)prop-2-en-1-one (32)

Following the general procedure, compound **32** was isolated in 83% yield (33.3 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.73 (d, J = 12.9 Hz, 1H), 7.56 (m, 2H), 7.39 (m, 3H), 6.76 (d, J = 12.9 Hz, 1H), 3.66 (t, J = 5.7 Hz, 2H), 3.62 (t, J = 5.8 Hz, 2H), 2.04 (m, 2H), 1.94 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 164.7, 141.7, 135.4, 129.5, 128.8, 127.8, 118.9, 53.4, 46.6, 46.0, 38.6, 26.1, 24.4; IR (thin film) 2971, 2873, 1650, 1603, 1451, 1427, 1339, 1227, 1044, 979, 853 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{13}H_{15}NO$ 202.1232, found 202.1228.

phenyl(pyrrolidin-1-yl)methanone (33)

Following the general procedure, compound **33** was isolated in 88% yield (30.8 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.54 (m, 2H), 7.43 (m, 3H), 3.69 (t, J = 7.0 Hz, 2H), 3.46 (t, J = 6.7 Hz, 2H), 1.98 (m, 2H), 1.89 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 169.7, 137.3, 129.7, 128.2, 127.1, 49.6, 46.1, 26.4, 24.4; IR (thin film) 2970, 2875, 1617, 1574, 1446, 1415, 1339, 1252, 1076, 1026, 926, 873, 841 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{11}H_{13}NO$ 176.1075, found 176.1073.

morpholino(phenyl)methanone (34)

Following the general procedure, compound **34** was isolated in 82% yield (31.4 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.44 (m, 5H), 3.79-3.47 (m, 8H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 170.4, 135.3, 129.9, 128.5, 127.1, 66.9, 43.1; IR (thin film) 2855, 1628, 1426, 1277, 1257, 1112, 1067, 1016, 933, 841 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{11}H_{13}NO_2$ 214.0841, found 214.0837.

(R)-benzyl 2-(tert-butoxycarbonylamino)-4-oxo-4-(pyrrolidin-1-yl)butanoate (35)

Following the general procedure, compound **35** was isolated in 85% yield (63.9 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.24 (m, 5H), 5.86 (d, J = 9.2 Hz, 1H), 5.13 (d, J = 12.5 Hz, 1H), 5.07 (d, J = 12.5 Hz, 1H), 4.52 (m, 1H), 3.34 (t, J = 6.9 Hz, 2H), 3.27 (t, J = 6.8 Hz,

2H), 3.01 (dd, J = 3.9 Hz, J = 16.7 Hz, 1H), 2.64 (dd, J = 4.1 Hz, J = 16.7 Hz, 1H), 1.86 (m, 2H), 1.77 (m, 2H) 1.45 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 171.7, 168.6, 155.9, 135.8, 128.4, 128.0, 127.9, 79.4, 67.1, 50.2, 46.5, 45.5, 36.8, 28.3, 25.9, 24.3; IR (thin film) 3427, 2974, 2876, 1712, 1635, 1495, 1449, 1366, 1215, 1161, 1054, 1025, 862 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{20}H_{28}N_2O_5$ 399.1909, found 399.1903.

(R)-benzyl 2-(tert-butoxycarbonylamino)-4-morpholino-4-oxobutanoate (36)

Following the general procedure, compound **36** was isolated in 81% yield (63.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 7.37 (m, 5H), 5.83 (d, J = 9.0 Hz, 1H), 5.26 (d, J = 12.4 Hz, 1H), 5.17 (d, J = 12.4 Hz, 1H), 4.65 (m, 1H), 3.68 (m, 6H), 3.43 (m, 2H), 3.17 (dd, J = 4.0 Hz, J = 16.6 Hz, 1H), 2.81 (dd, J = 4.0 Hz, J = 16.6 Hz, 1H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 171.4, 168.7, 155.8, 135.6, 128.4, 128.2, 128.0, 79.8, 67.1, 66.7, 66.4, 50.1, 45.7, 41.8, 35.4, 28.3; IR (thin film) 3433, 3332, 2975, 2929, 1741, 1712, 1640, 1495, 1455, 1366, 1273, 1236, 1163, 1115, 1056, 1028, 975 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{20}H_{28}N_2O_6$ 415.1845, found 415.1847.

(E)-1-morpholino-3-phenylprop-2-en-1-one (37)

Following the general procedure, compound **37** was isolated in 86% yield (37.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 7.74 (d, J = 15.4 Hz, 1H), 7.56 (m, 2H), 7.41 (m, 3H), 6.88 (d, J = 15.4 Hz, 1H), 3.76-3.69 (m, 8H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 165.6, 143.2, 135.1, 129.8, 128.8, 127.9, 116.6, 66.8, 60.4, 46.5, 43.1, 38.6; IR (thin film) 2964, 2898, 2855, 1646, 1599, 1495, 1455, 1428, 1263, 1227, 1201, 1113, 1043, 976, 881, 844 cm^{-1}; HRMS $[M+H]^+$ Calculated for $C_{13}H_{15}NO_2$ 218.1181, found 218.1176.

(S)-isopropyl 2-(((9H-fluoren-9-yl)methoxy)carbonylamino)propanoate (38)

Following the general procedure, compound **38** was isolated in 43% yield (30.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$), δ 7.70 (m, 2H), 7.54 (m, 2H), 7.34 (m, 2H), 7.26 (m, 2H), 5.30 (d, J = 7.2 Hz, 1H), 5.01 (m, 1H), 4.33 (m, 3H), 4.17 (t, J = 7.1 Hz, 1H), 1.36 (d, J = 7.1 Hz, 3H), 1.19 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 172.6, 155.6, 143.9, 143.8, 141.32, 141.30, 127.7, 127.1, 125.14, 125.10, 119.9, 69.1, 66.9, 49.8, 47.2, 21.7, 21.6, 18.8; IR (thin film) 3338, 2980, 2937, 1718, 1524, 1450, 1375, 1330, 1247, 1212, 1105,

1073, 1034, 940 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{21}H_{23}NO_4$ 376.1522, found 376.1525.

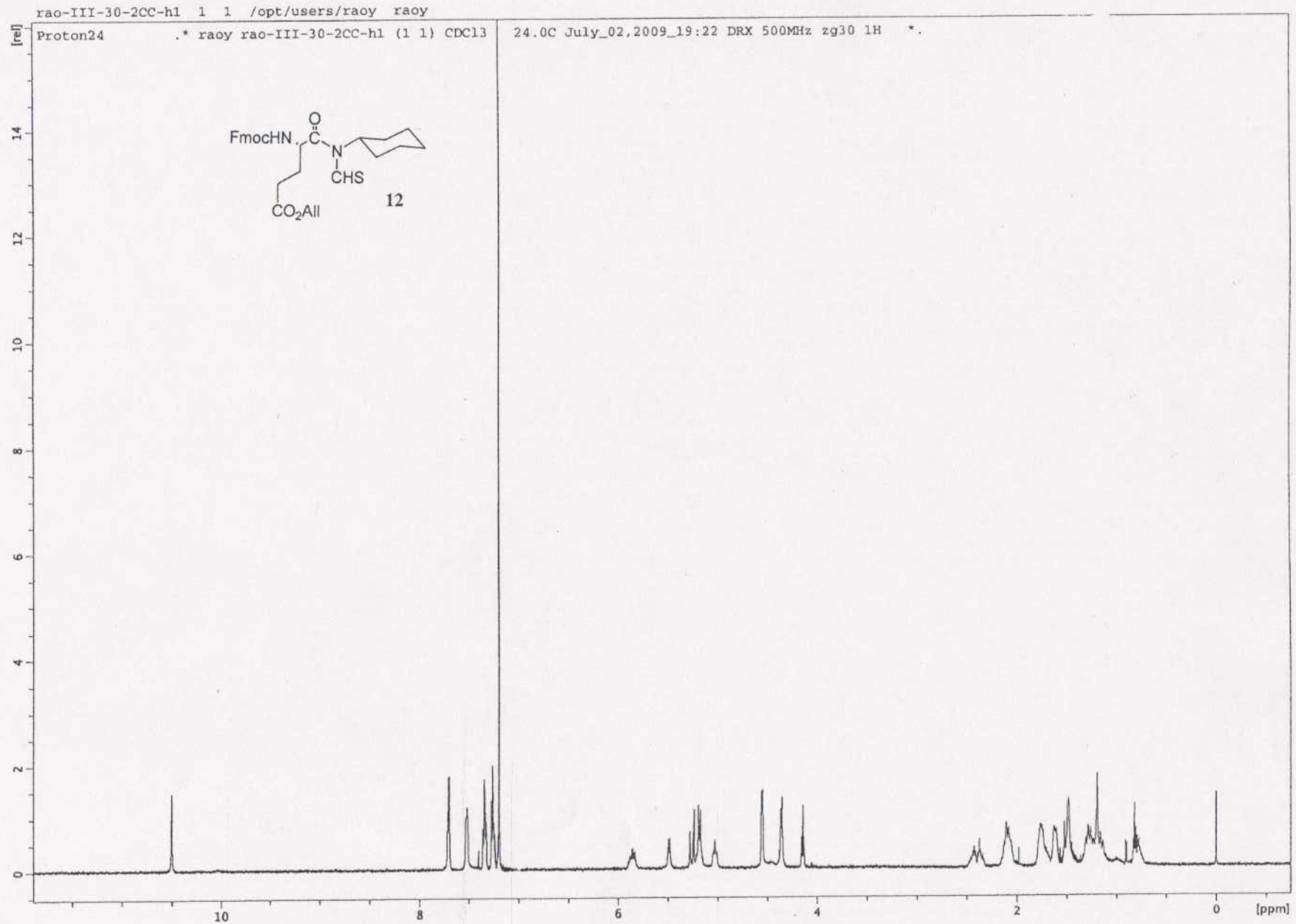
(S)-1-benzyl 4-isopropyl 2-(tert-butoxycarbonylamino)succinate (39)

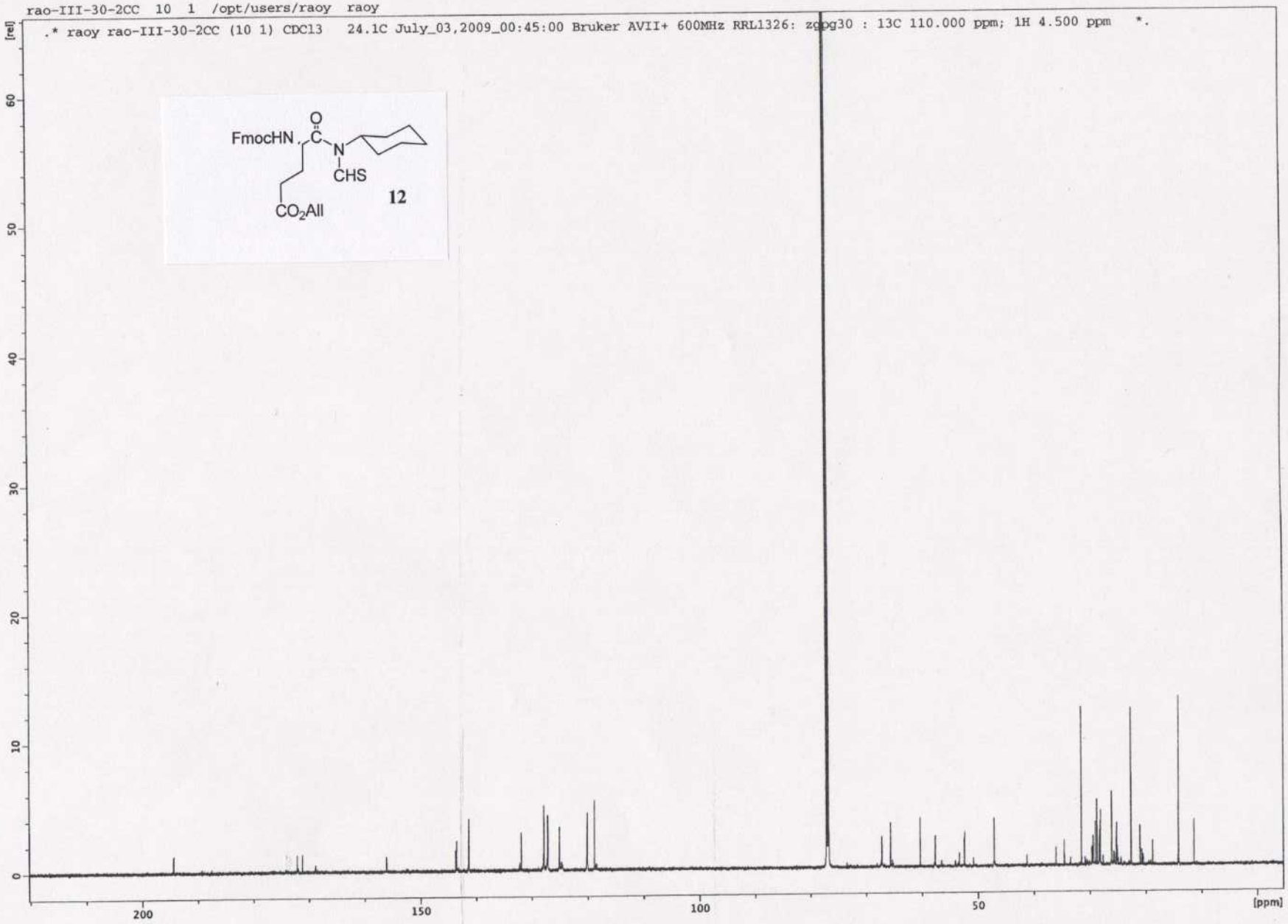
Following the general procedure, compound **39** was isolated in 64% yield (46.7 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.29 (m, 5H), 5.43 (d, J = 8.1 Hz, 1H), 5.15 (d, J = 12.3 Hz, 1H), 5.08 (d, J = 12.3 Hz, 1H), 4.92 (m, 1H), 4.55 (m, 1H), 2.92 (dd, J = 4.3 Hz, J = 16.8 Hz, 1H), 2.73 (m, 1H), 1.45 (s, 9H), 1.13 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 171.0, 170.3, 155.4, 135.3, 128.5, 128.3, 128.1, 80.0, 68.6, 67.3, 50.1, 37.0, 28.3, 21.7; IR (thin film) 3374, 2979, 2936, 1718, 1498, 1368, 1340, 1211, 1162, 1106, 1051, 1025, 976 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{19}H_{27}NO_6$ 388.1733, found 388.1732.

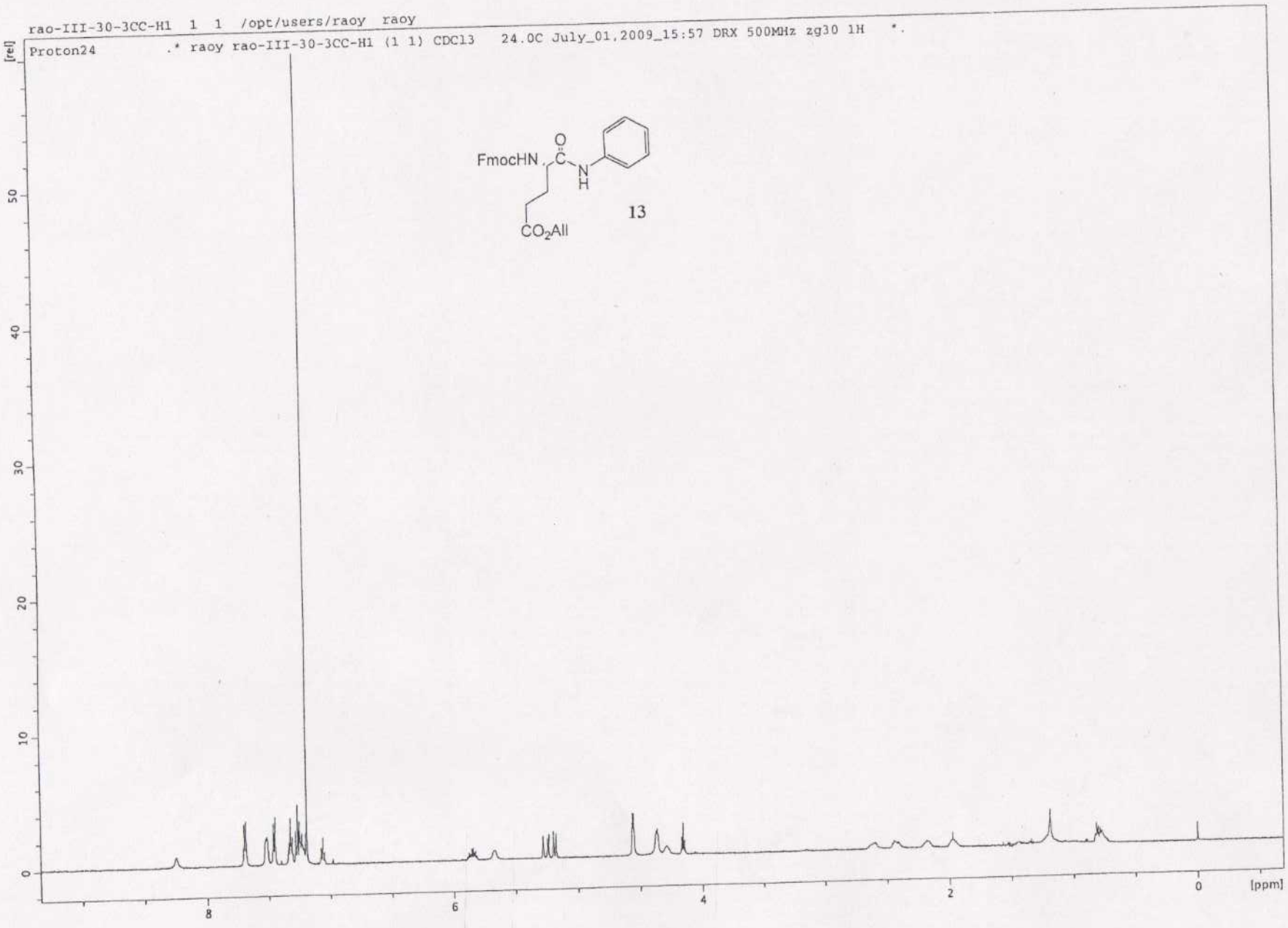
(S)-methyl 2-(2-(((9H-fluoren-9-yl)methoxy)carbonylamino)-N,2-dimethylpropanamido)-3-methylbutanoate (46)

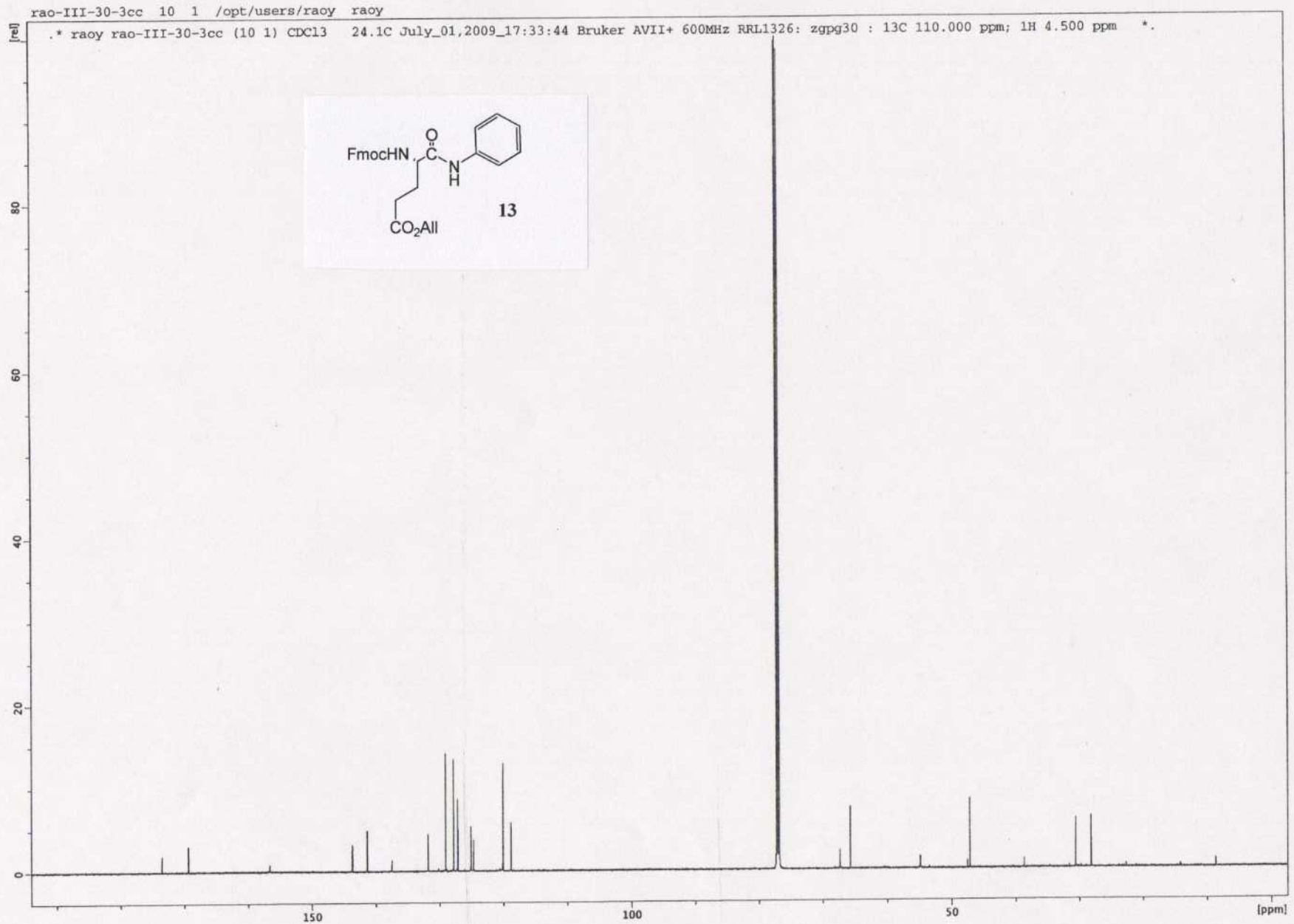
To a CH_2Cl_2 (1.0 mL) solution of thio acid **40** (0.10 mmol) were added t-butyl isonitrile (0.20 mmol) and N-methyl valine **41** (0.30 mmol). This reaction mixture was stirred at room temperature for 30 mins and CH_2Cl_2 was removed under vacuum. The residue was purified by flash column chromatography and acid **45** was isolated in 75% yield (32.9 mg). Then, to a THF/MeOH (1.0 mL, 3:1/v:v) solution of acid **45** (0.05 mmol) was added $TMSCH_2N_2$ (0.06 mmol). This reaction mixture was stirred at room temperature for 30 mins before quenching with acetic acid. After dilution with CH_2Cl_2 (5.0 mL) and sequential washing with $NaHCO_3$(sat.) and brine, the organic layer was dried over Na_2SO_4. Then, after filetration, CH_2Cl_2 was removed under vacuum. The residue was purified by flash column chromatography. Compound **46** was obtained in 90% yield (20.4 mg). ^1H NMR (500 MHz, $CDCl_3$), δ 7.79 (m, 2H), 7.62 (m, 2H), 7.44 (m, 2H), 7.34 (m, 2H), 5.59 (s, 1H), 4.83 (m, 1H), 4.46 (m, 2H), 4.23 (t, J = 6.5 Hz, 1H), 3.71 (s, 3H), 3.06 (s, 3H), 2.27 (m, 1H), 1.63 (s, 3H), 1.61 (s, 3H), 1.04 (d, J = 11.5 Hz, 3H), 0.90 (d, J = 12.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$), δ 173.5, 171.6, 154.1, 143.9, 143.8, 141.4, 141.3, 127.6, 127.1, 124.9, 119.9, 66.1, 60.4, 57.3, 53.4, 51.8, 47.3, 31.6, 29.7, 25.4, 22.6, 20.0, 18.9; IR (thin film) 3303, 2964, 2931, 1727, 1628, 1525, 1466, 1450, 1394, 1253,

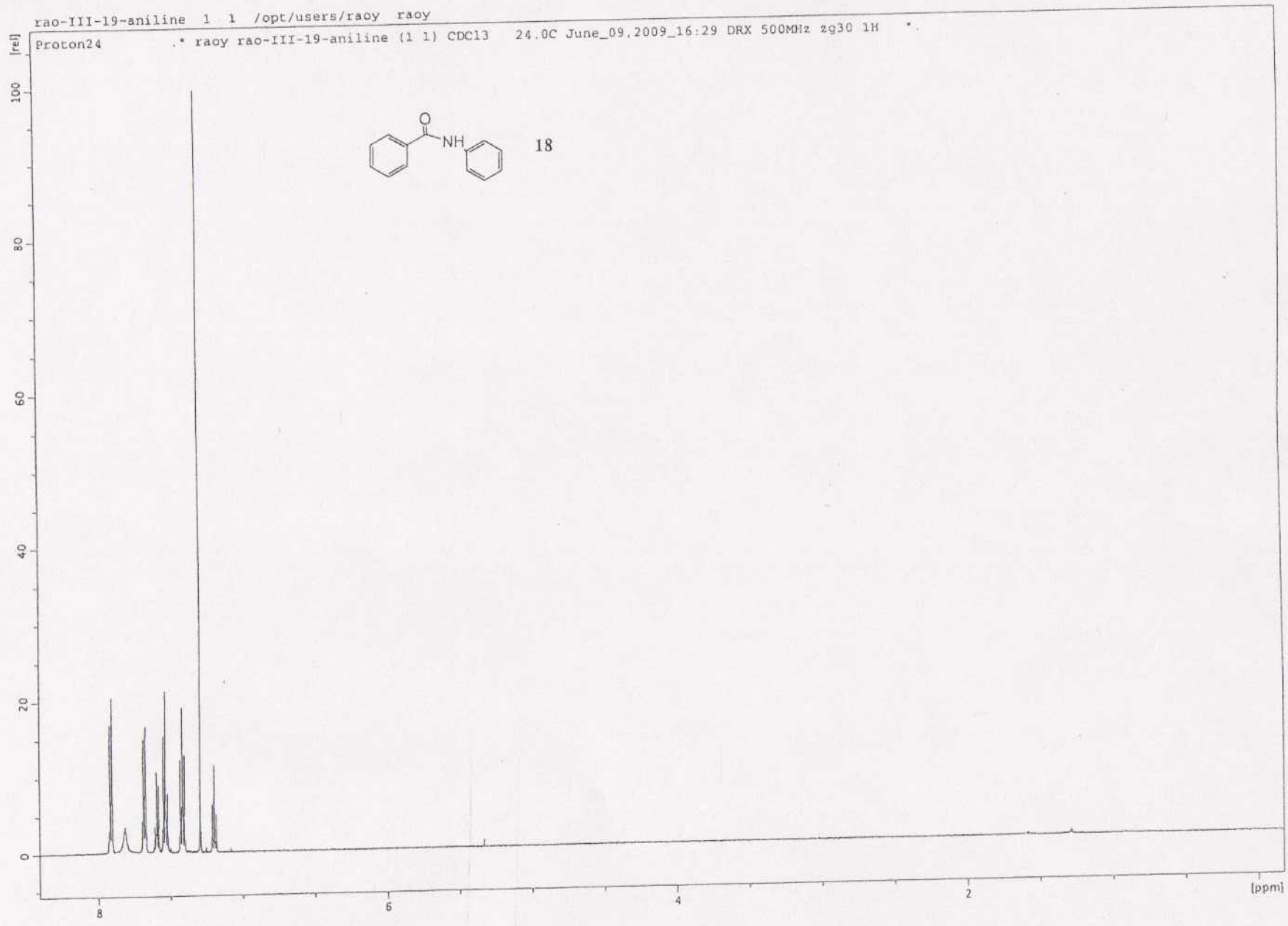
1210, 1105, 1081, 1015, 952, 910 cm^{-1}; HRMS $[M+Na]^+$ Calculated for $C_{26}H_{32}N_2O_5$ 475.2209, found 475.2204.


rao-III-30-2CC-h1 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-30-2CC-h1 (1 1) CDCl3
24.0C July_02,2009_19:22 DRX 500MHz zg30 1H *.
FmocHN
O
C
N
CHS
CO2All
12
[rel]
14
12
10
8
6
4
2
0
10
8
6
4
2
0
[ppm]

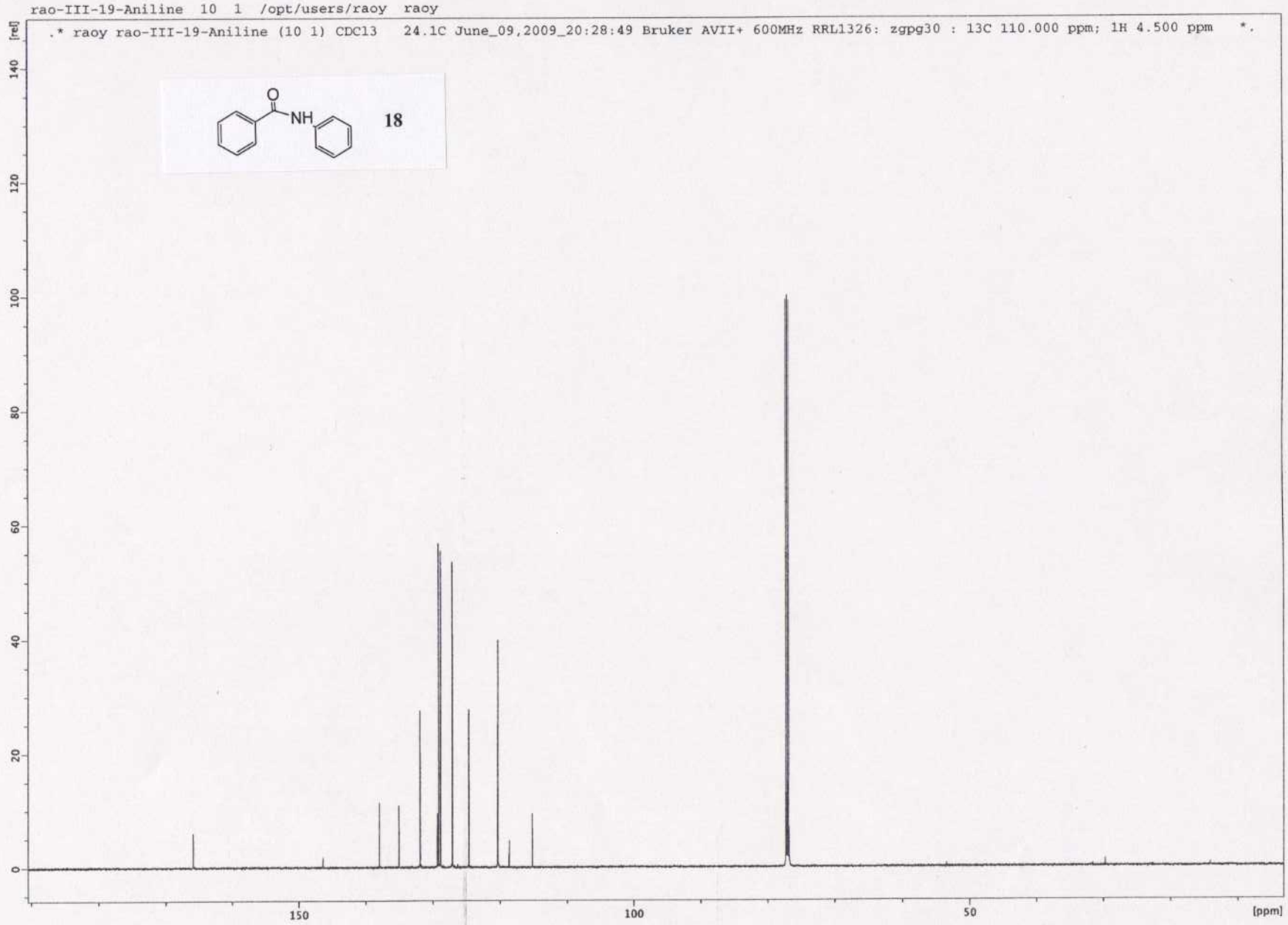

rao-III-30-2CC 10 1 /opt/users/raoy raoy
.* raoy rao-III-30-2CC (10 1) CDCl3 24.1C July_03,2009_00:45:00 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
FmocHN
O
C
N
CHS
CO2All
12
0
10
20
30
40
50
60
200
150
100
50
[ppm]

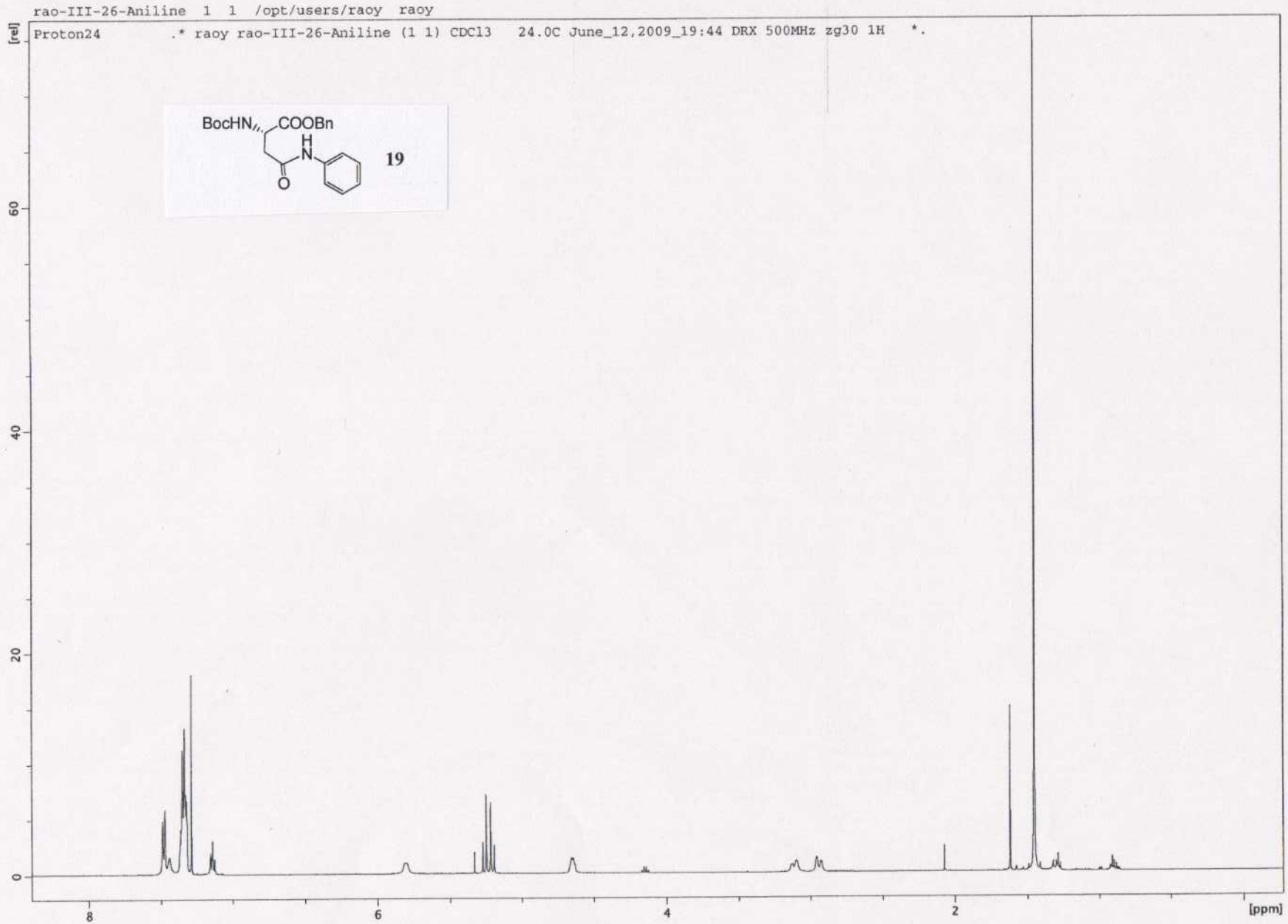

rao-III-30-3CC-H1 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-30-3CC-H1 (1 1) CDCl3 24.0C July_01,2009_15:57 DRX 500MHz zg30 1H *.
[rel]
FmocHN
O
C
N
H
CO2All
13
0
10
20
30
40
50
8
6
4
2
0
[ppm]

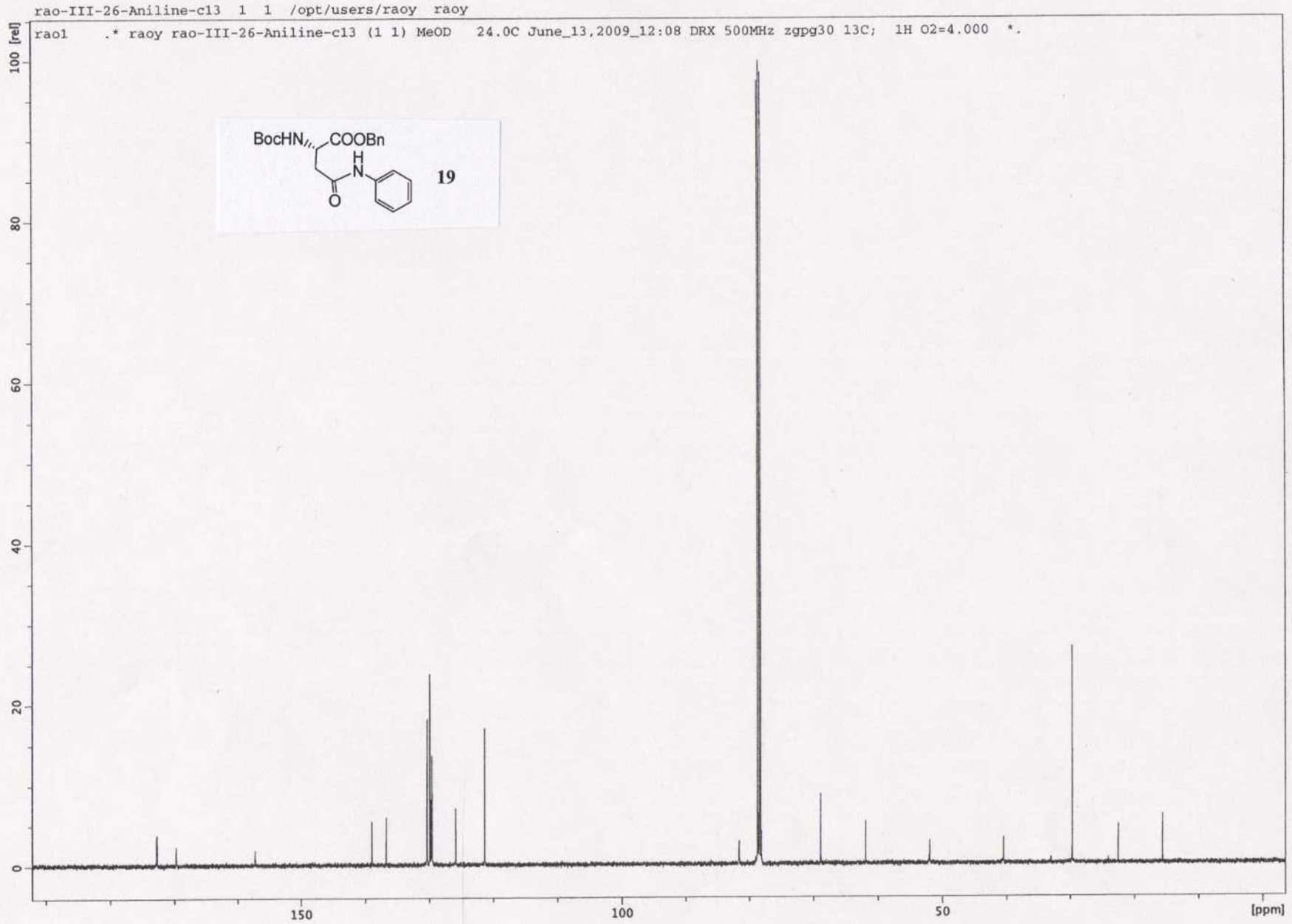

rao-III-30-3cc 10 1 /opt/users/raoy raoy
.* raoy rao-III-30-3cc (10 1) CDCl3 24.1C July_01,2009_17:33:44 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
FmocHN
O
C
N
H
CO2All
13
0
20
40
60
80
150
100
50
[ppm]

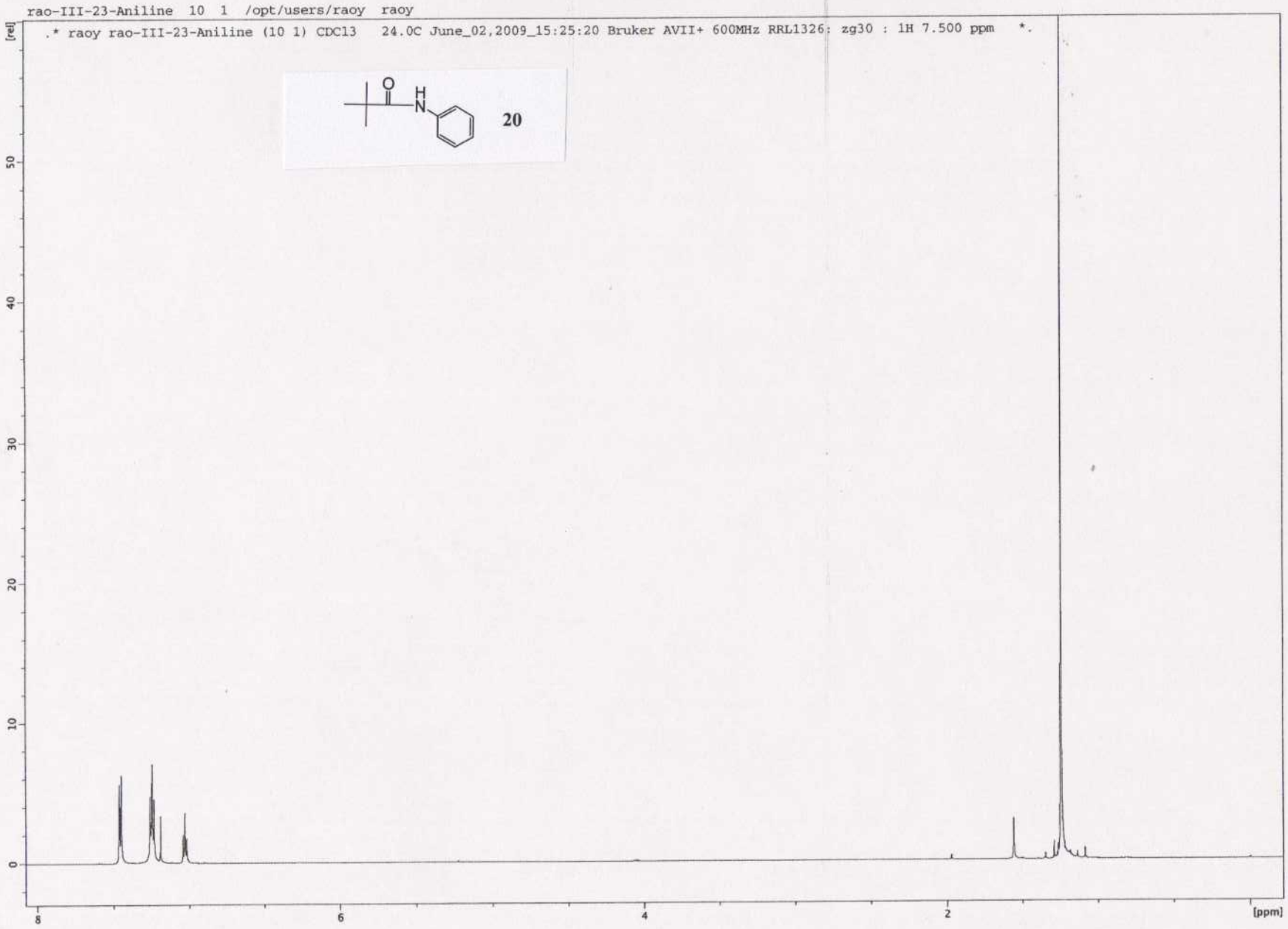

rao-III-19-aniline 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-19-aniline (1 1) CDCl3 24.0C June_09,2009_16:29 DRX 500MHz zg30 1H
[rel]
100
80
60
40
20
0
8
6
4
2
[ppm]
O
NH

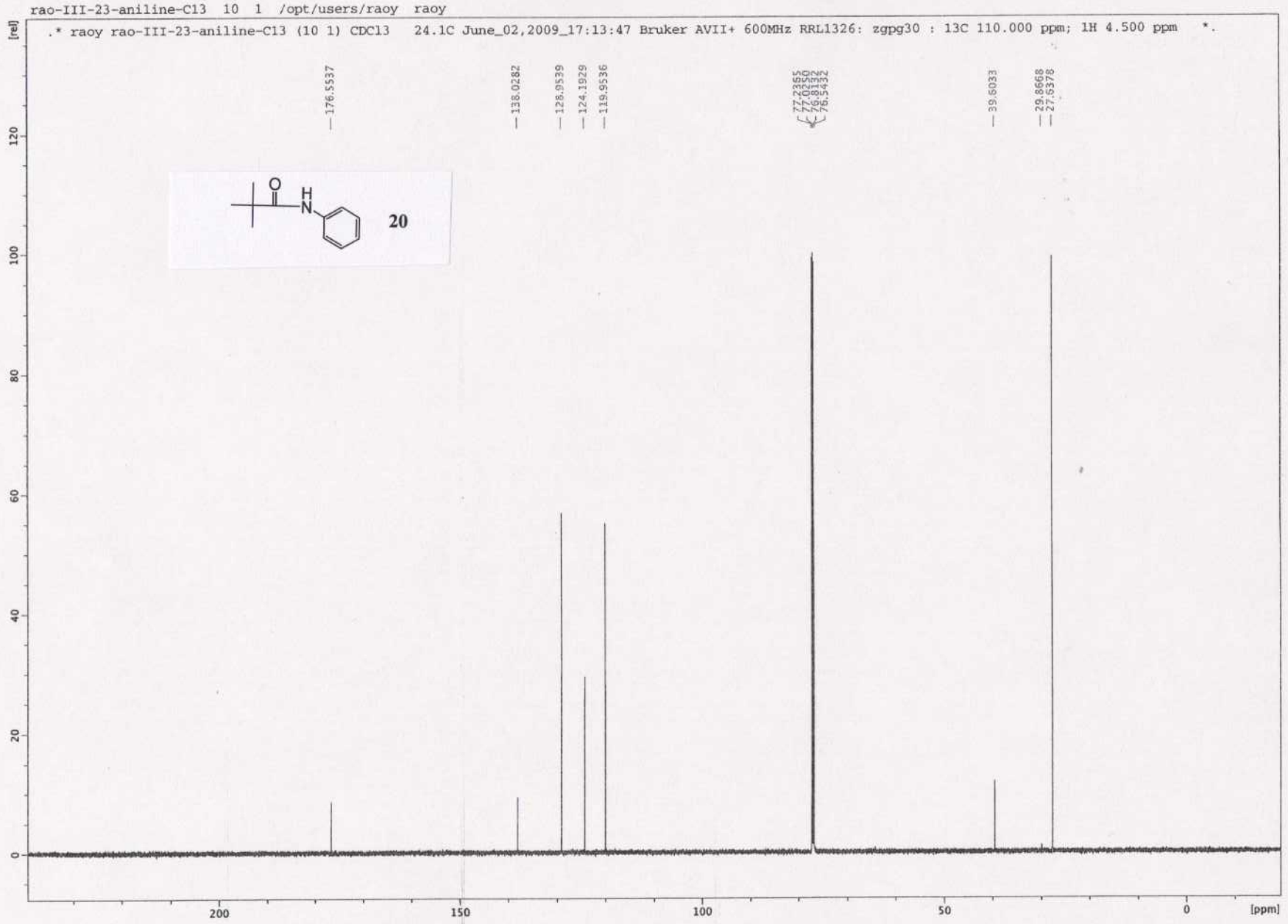
18

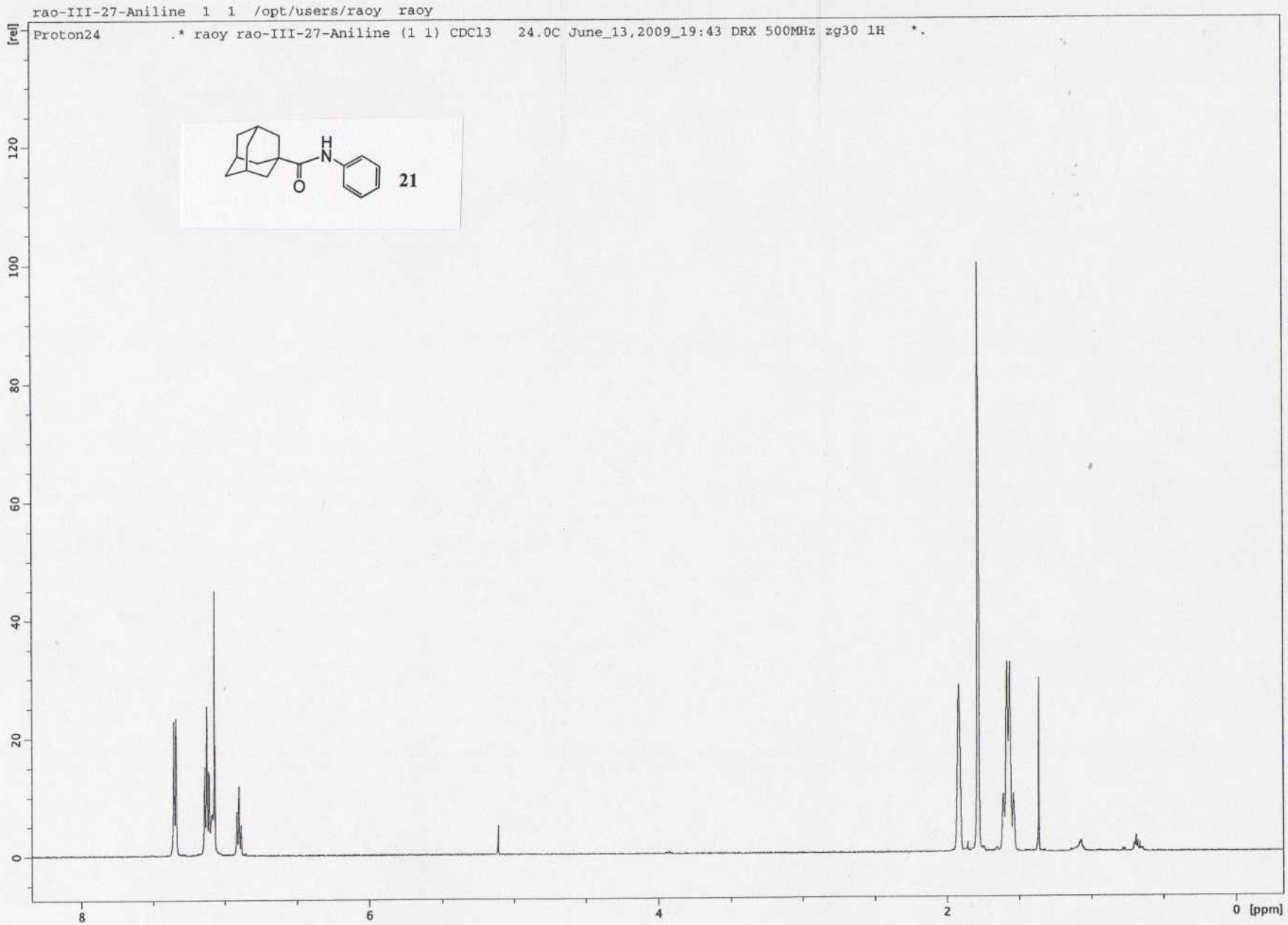

rao-III-19-Aniline 10 1 /opt/users/raoy raoy
.* raoy rao-III-19-Aniline (10 1) CDCl3 24.1C June_09,2009_20:28:49 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
140
120
100
80
60
40
20
0
O
NH
18
150
100
50
[ppm]

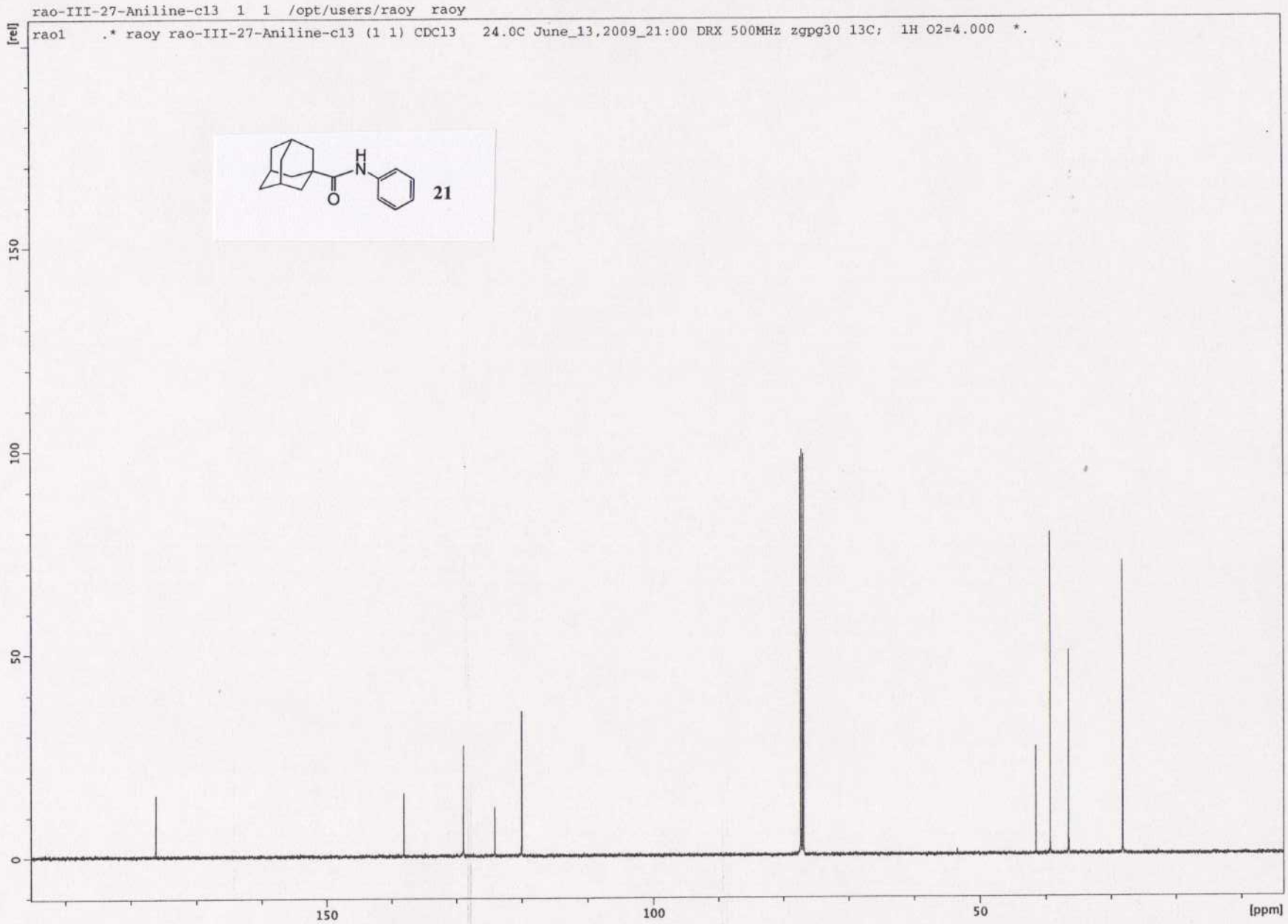

rao-III-26-Aniline 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-26-Aniline (1 1) CDCl3 24.0C June_12,2009_19:44 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
8
6
4
2
[ppm]
BocHN
COOBn
H
N
O
19

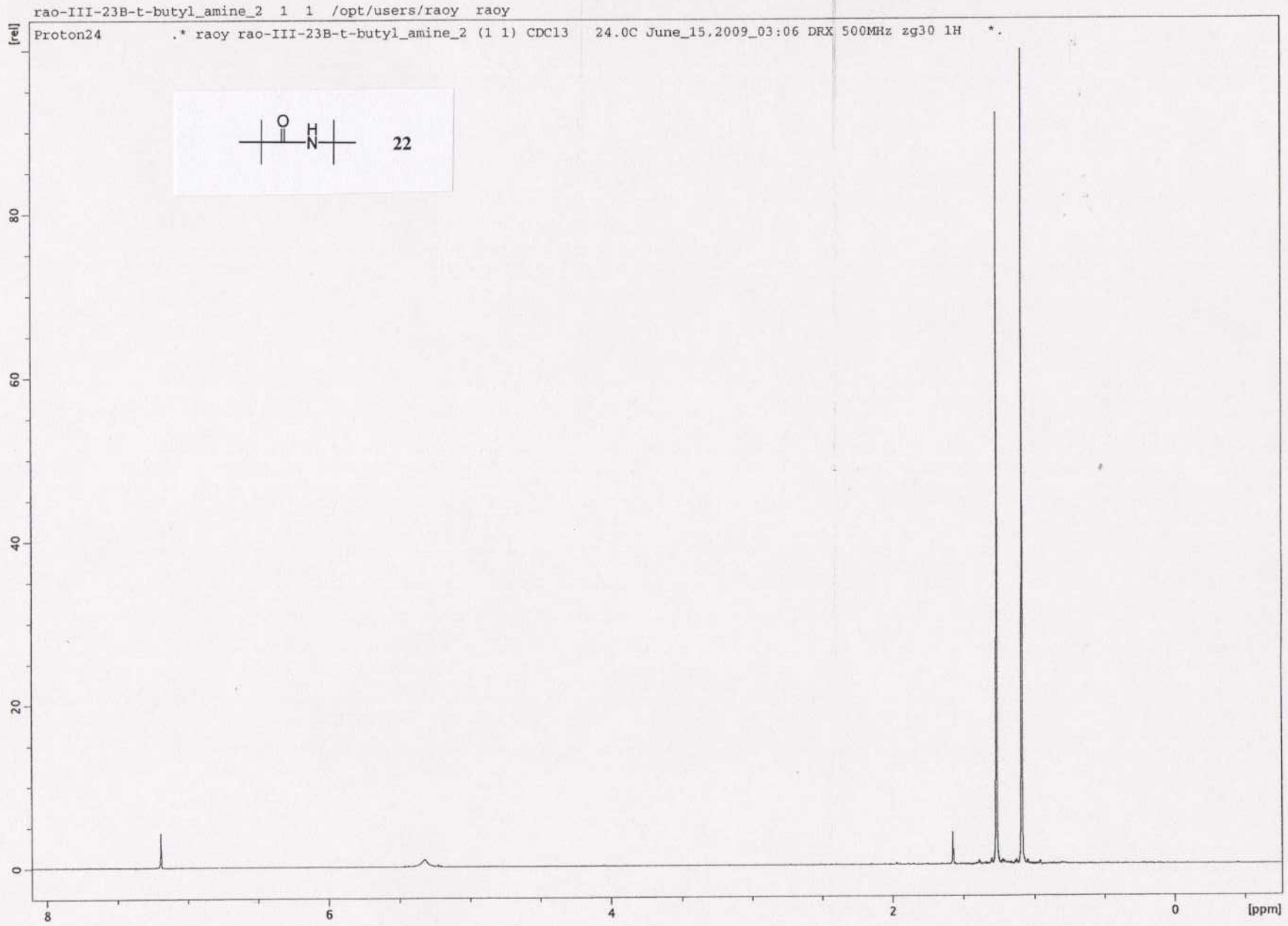

rao-III-26-Aniline-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-26-Aniline-c13 (1 1) MeOD 24.0C June_13,2009_12:08 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
100 [rel]
80
60
40
20
0
BocHN
COOBn
H
N
O
19
150
100
50
[ppm]

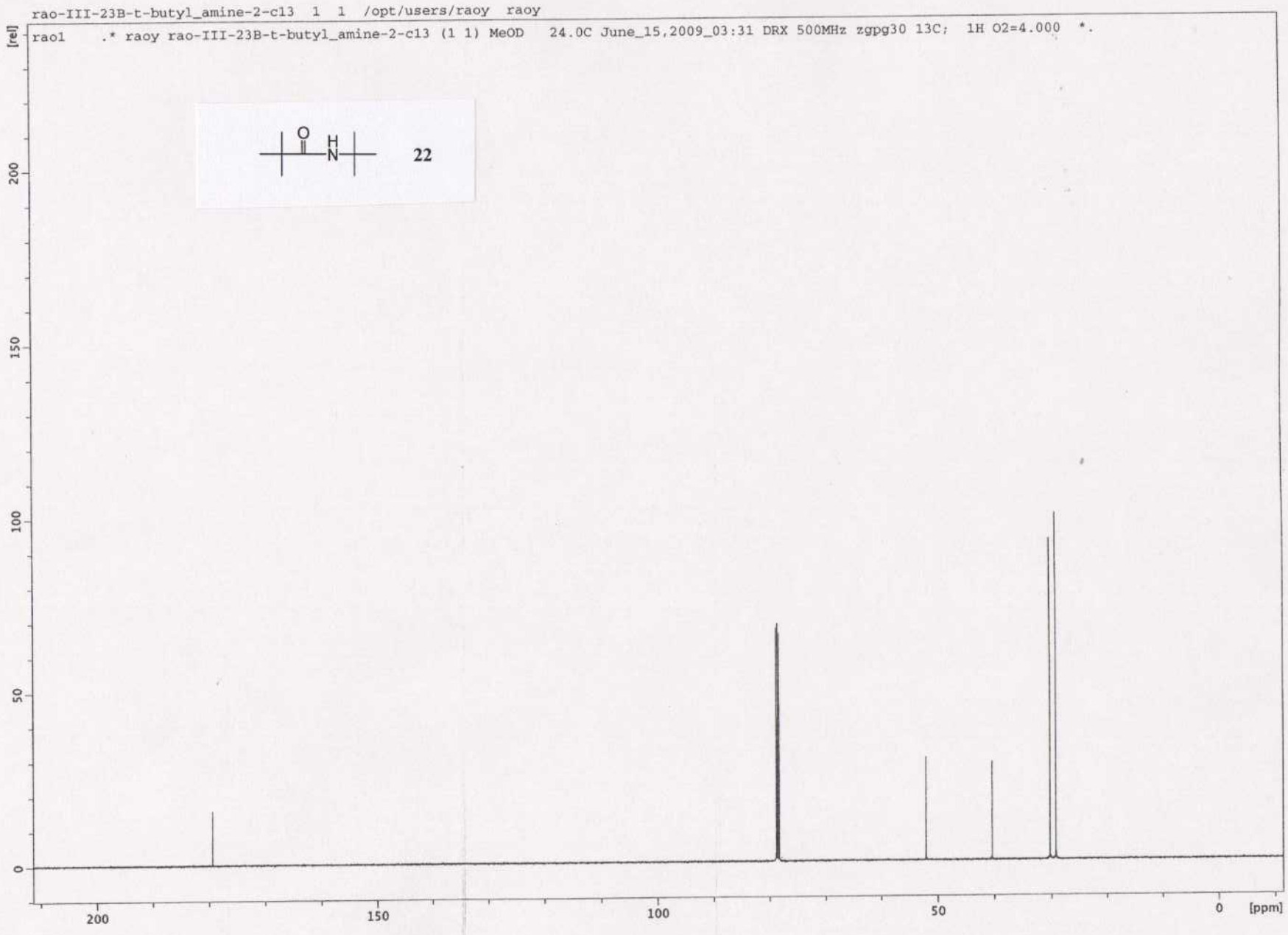

rao-III-23-Aniline 10 1 /opt/users/raoy raoy
.* raoy rao-III-23-Aniline (10 1) CDCl3 24.0C June_02,2009_15:25:20 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm *.
[rel]
0
10
20
30
40
50
8
6
4
2
[ppm]
O
H
N
20

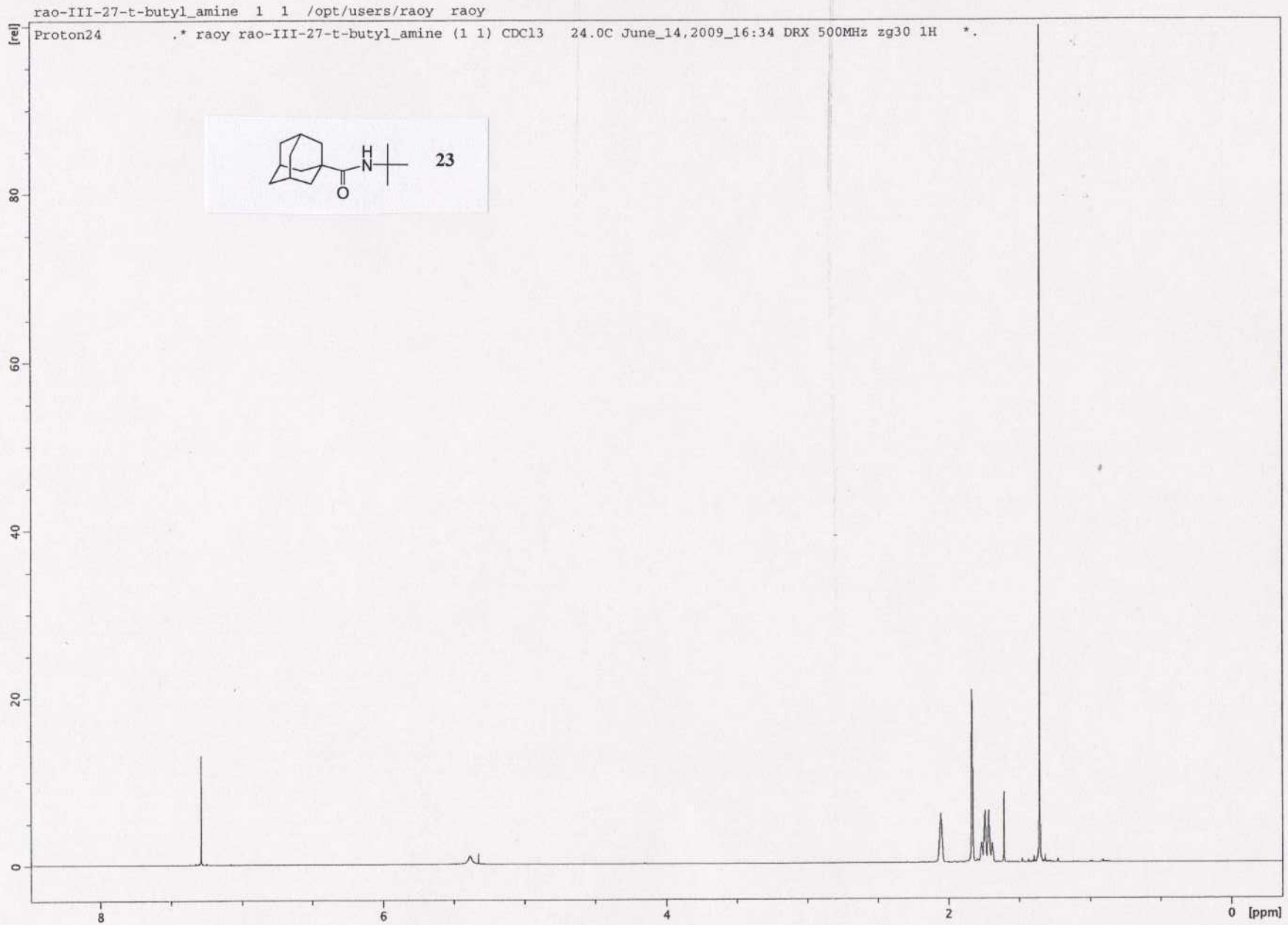

rao-III-23-aniline-C13 10 1 /opt/users/raoy raoy
.* raoy rao-III-23-aniline-C13 (10 1) CDCl3 24.1C June_02,2009_17:13:47 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
176.5537
138.0282
128.9539
124.1929
119.9536
77.2355
77.0250
76.8132
76.5432
39.6033
29.8668
27.6378
120
100
80
60
40
20
0
200
150
100
50
0
[ppm]
O
H
N
20

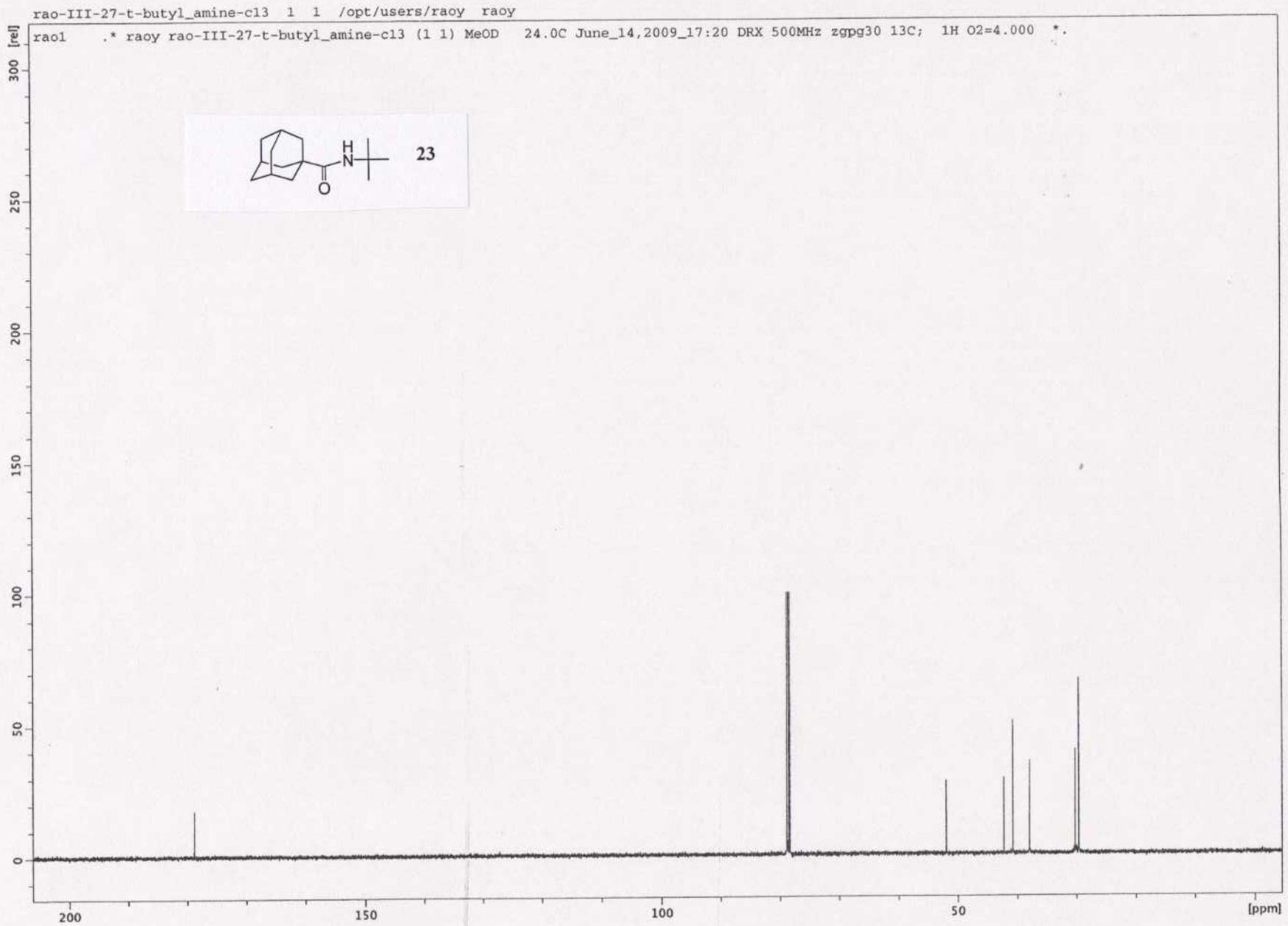

rao-III-27-Aniline 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-27-Aniline (1 1) CDCl3 24.0C June_13,2009_19:43 DRX 500MHz zg30 1H *.
[rel]
120
100
80
60
40
20
0
8
6
4
2
0 [ppm]
H
N
O
21

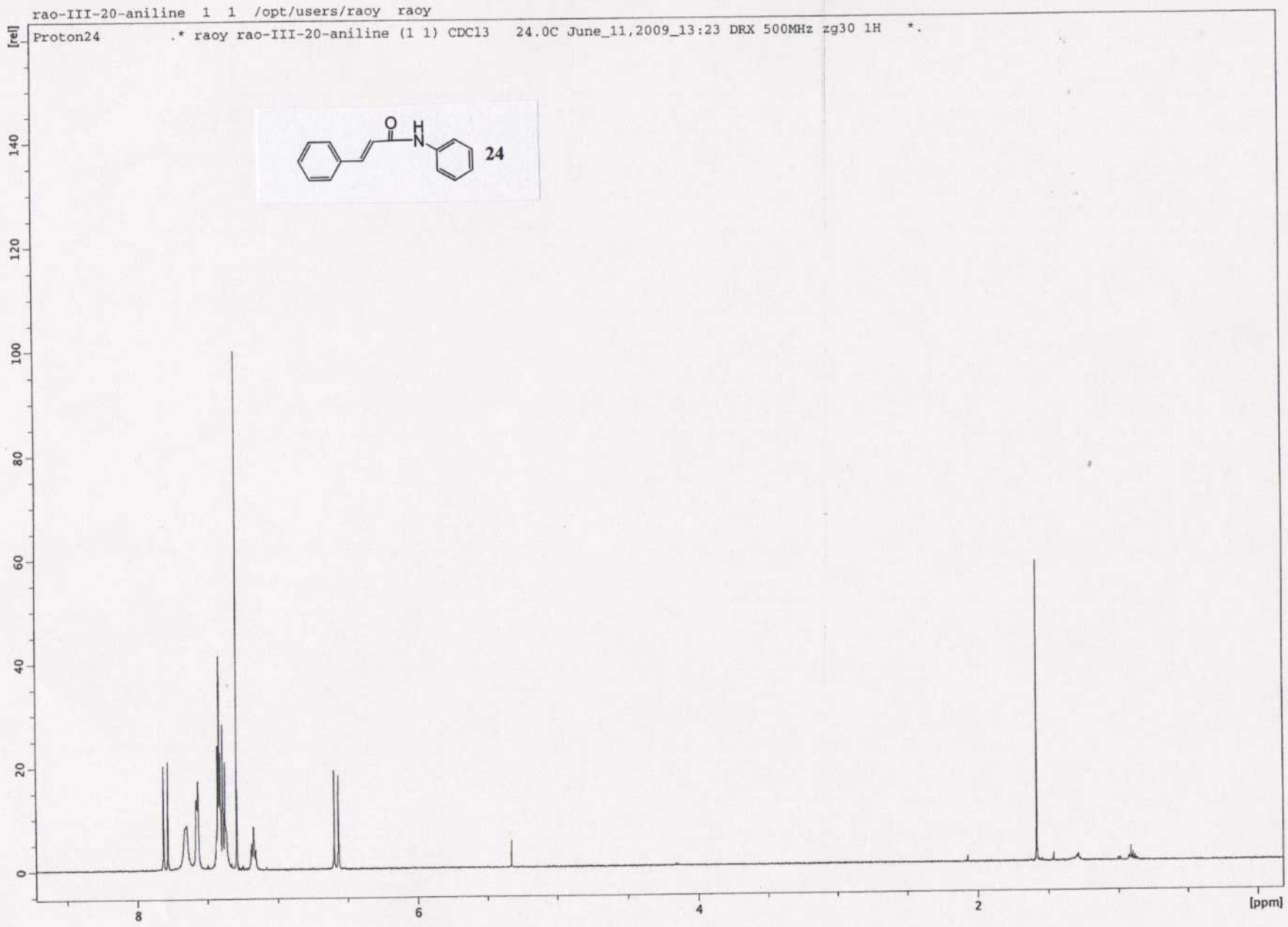

rao-III-27-Aniline-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-27-Aniline-c13 (1 1) CDCl3 24.0C June_13,2009_21:00 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
150
100
50
0
150
100
50
[ppm]
H
N
O
21

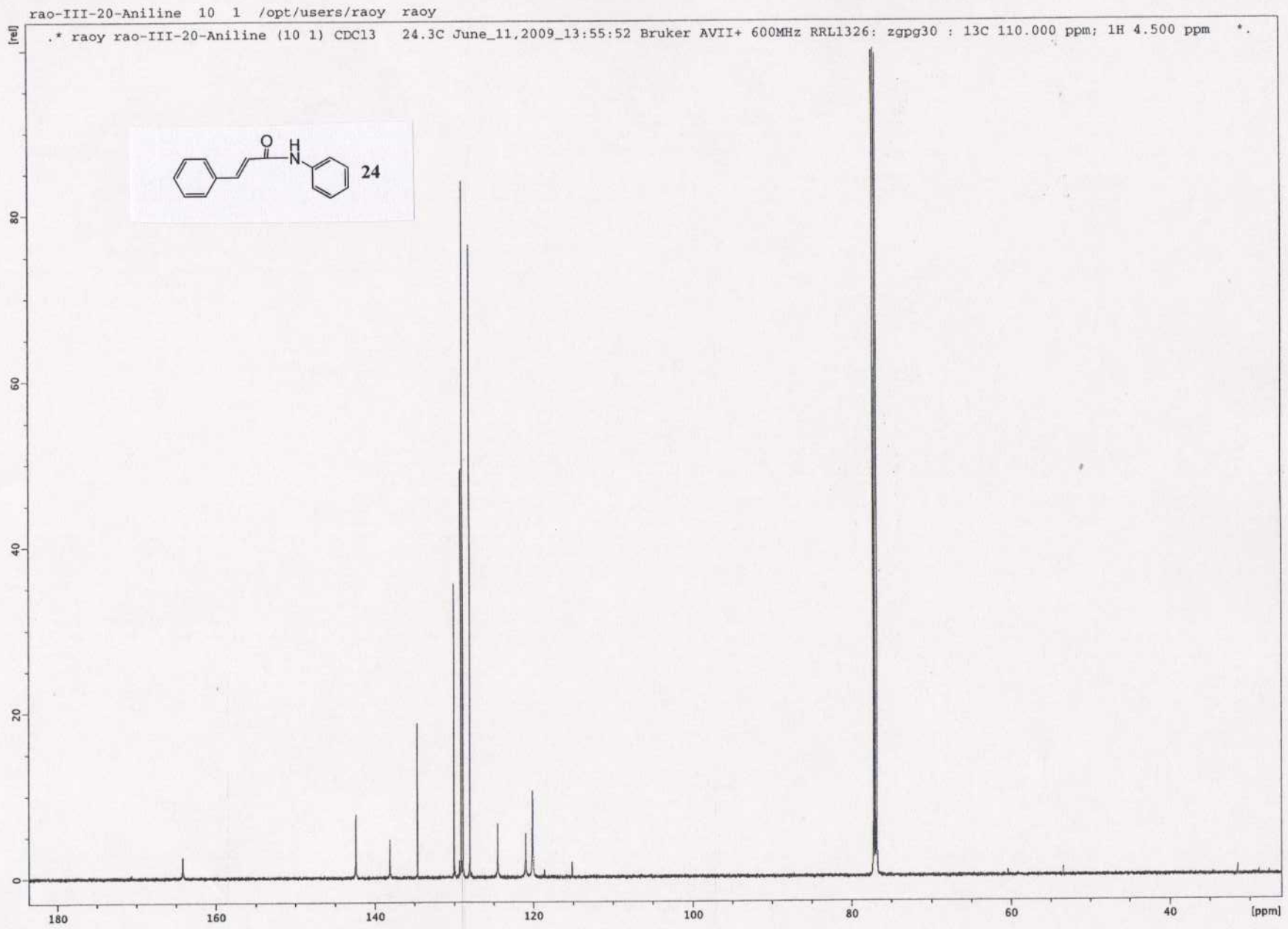

rao-III-23B-t-butyl_amine_2 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-23B-t-butyl_amine_2 (1 1) CDCl3 24.0C June_15,2009_03:06 DRX 500MHz zg30 1H *.
[rel]
22
0
20
40
60
80
8
6
4
2
0
[ppm]

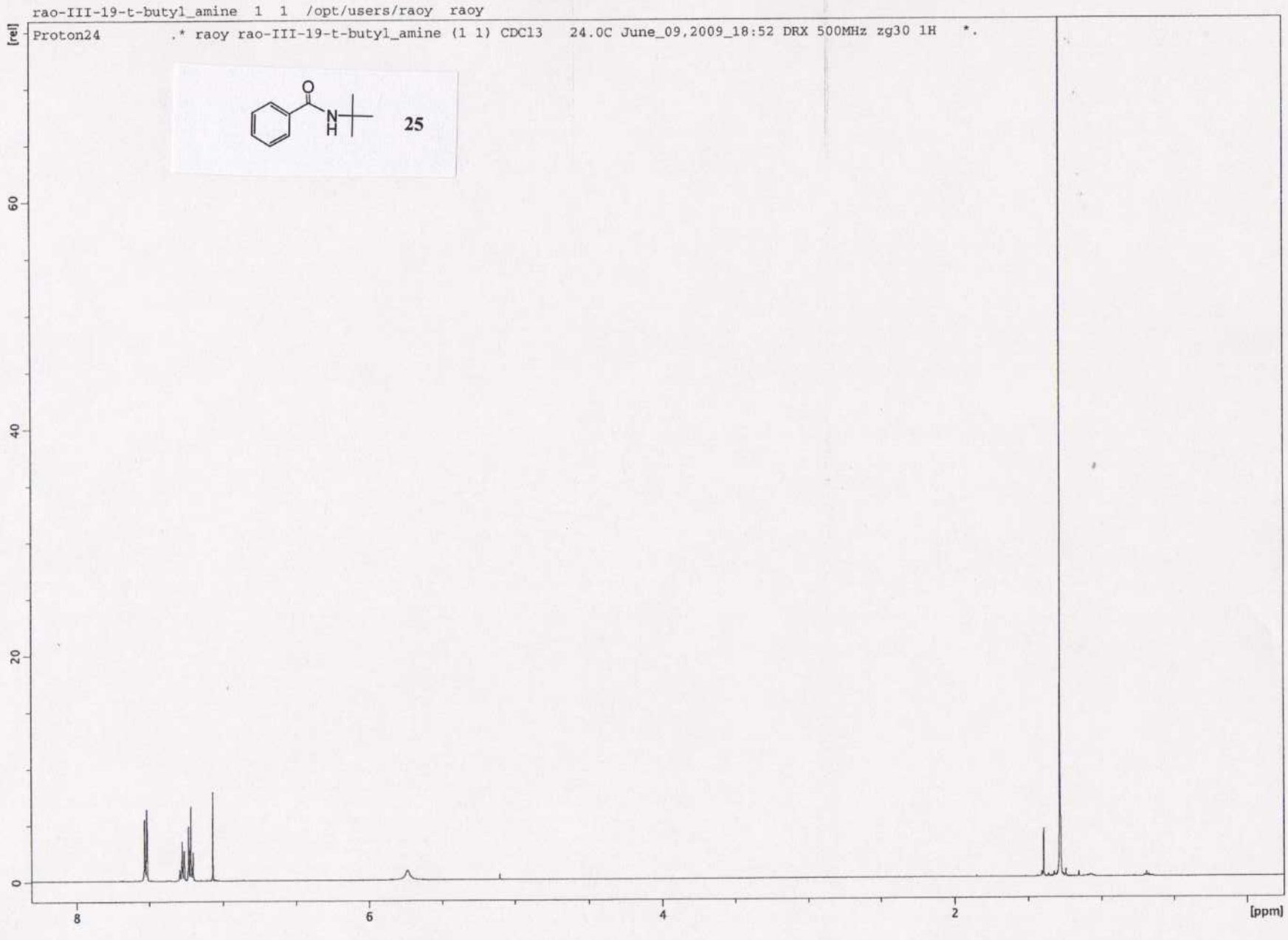

rao-III-23B-t-butyl_amine-2-c13 1 1 /opt/users/raoy raoy
raol .* raoy rao-III-23B-t-butyl_amine-2-c13 (1 1) MeOD 24.0C June_15,2009_03:31 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
200
150
100
50
0
O
H
N
22
200
150
100
50
0
[ppm]

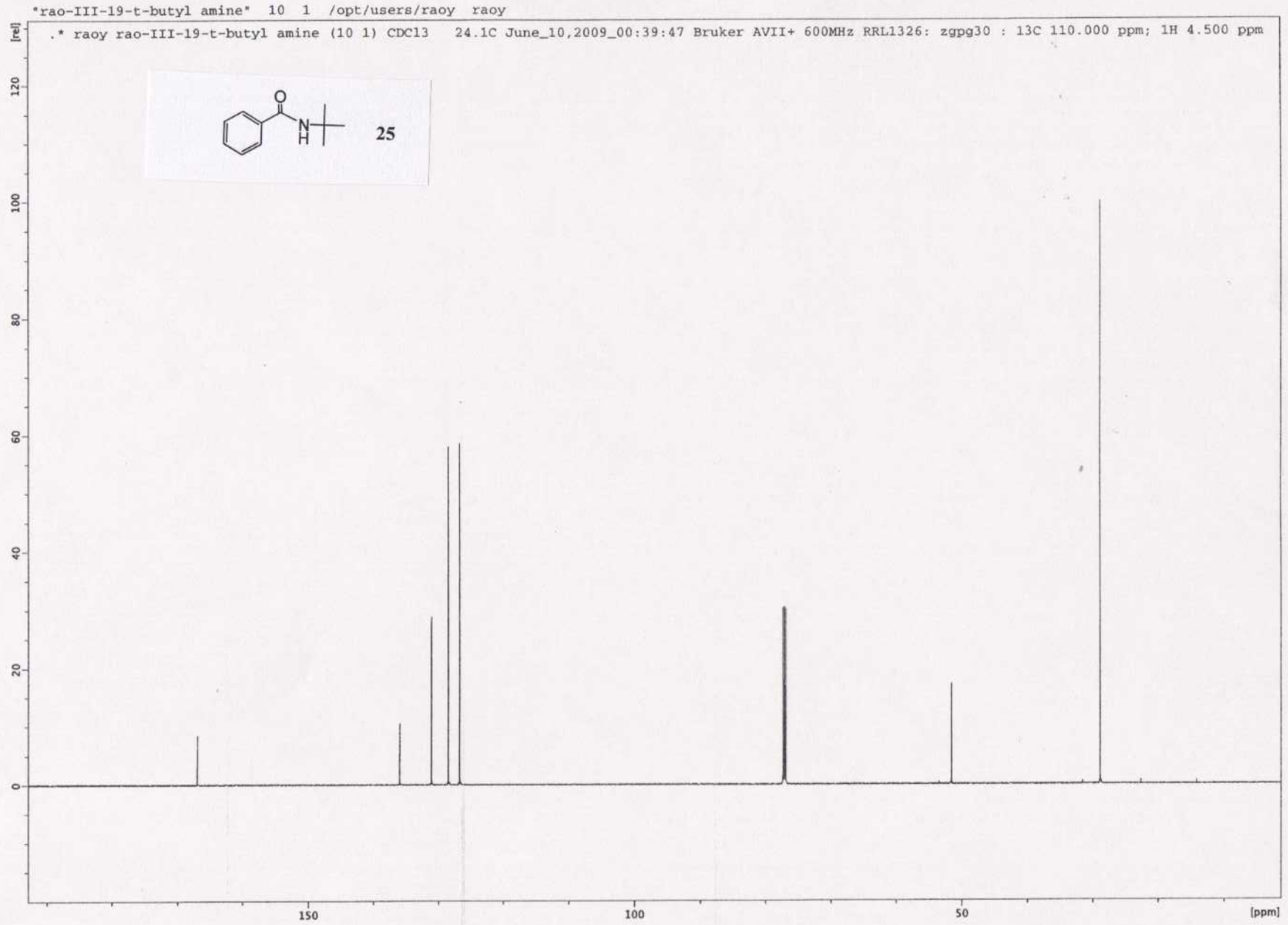

rao-III-27-t-butyl_amine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-27-t-butyl_amine (1 1) CDCl3 24.0C June_14,2009_16:34 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
80
8
6
4
2
0 [ppm]
H
N
O
23

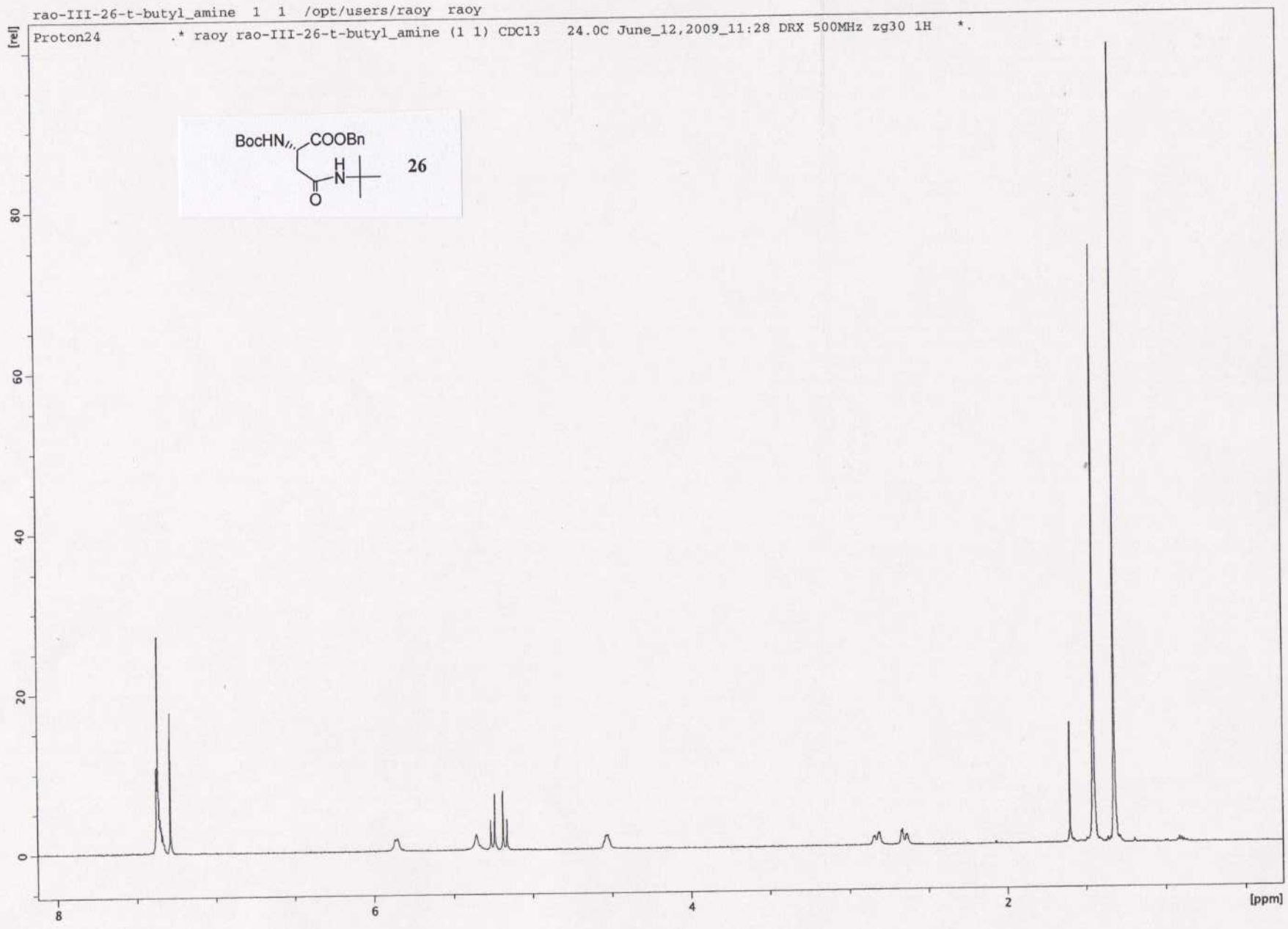

rao-III-27-t-butyl_amine-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-27-t-butyl_amine-c13 (1 1) MeOD 24.0C June_14,2009_17:20 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
23
300 [rel]
250
200
150
100
50
0
200
150
100
50
[ppm]

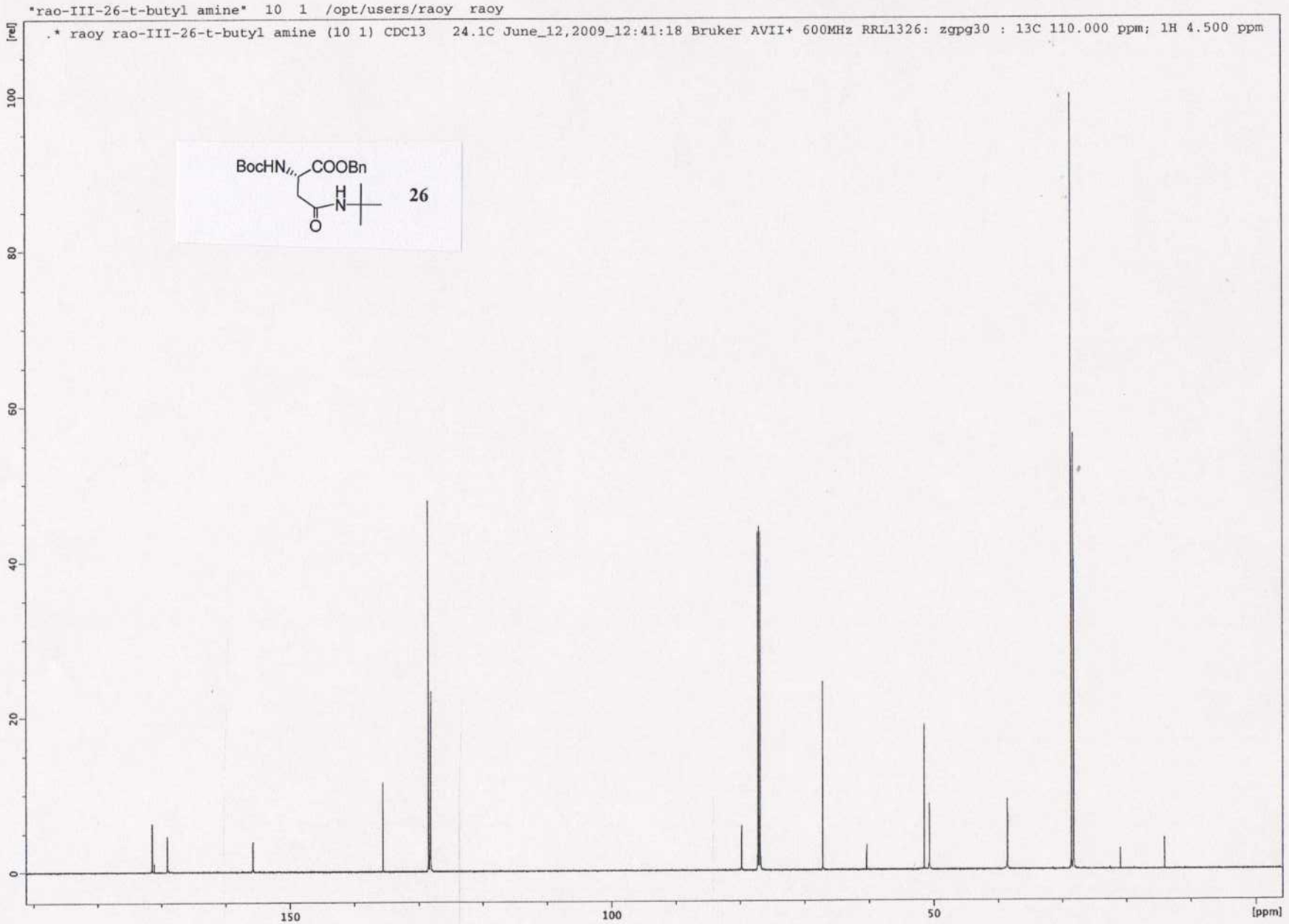

rao-III-20-aniline 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-20-aniline (1 1) CDCl3 24.0C June_11,2009_13:23 DRX 500MHz zg30 1H *.
[rel]
140
120
100
80
60
40
20
0
8
6
4
2
[ppm]
O
H
N
24

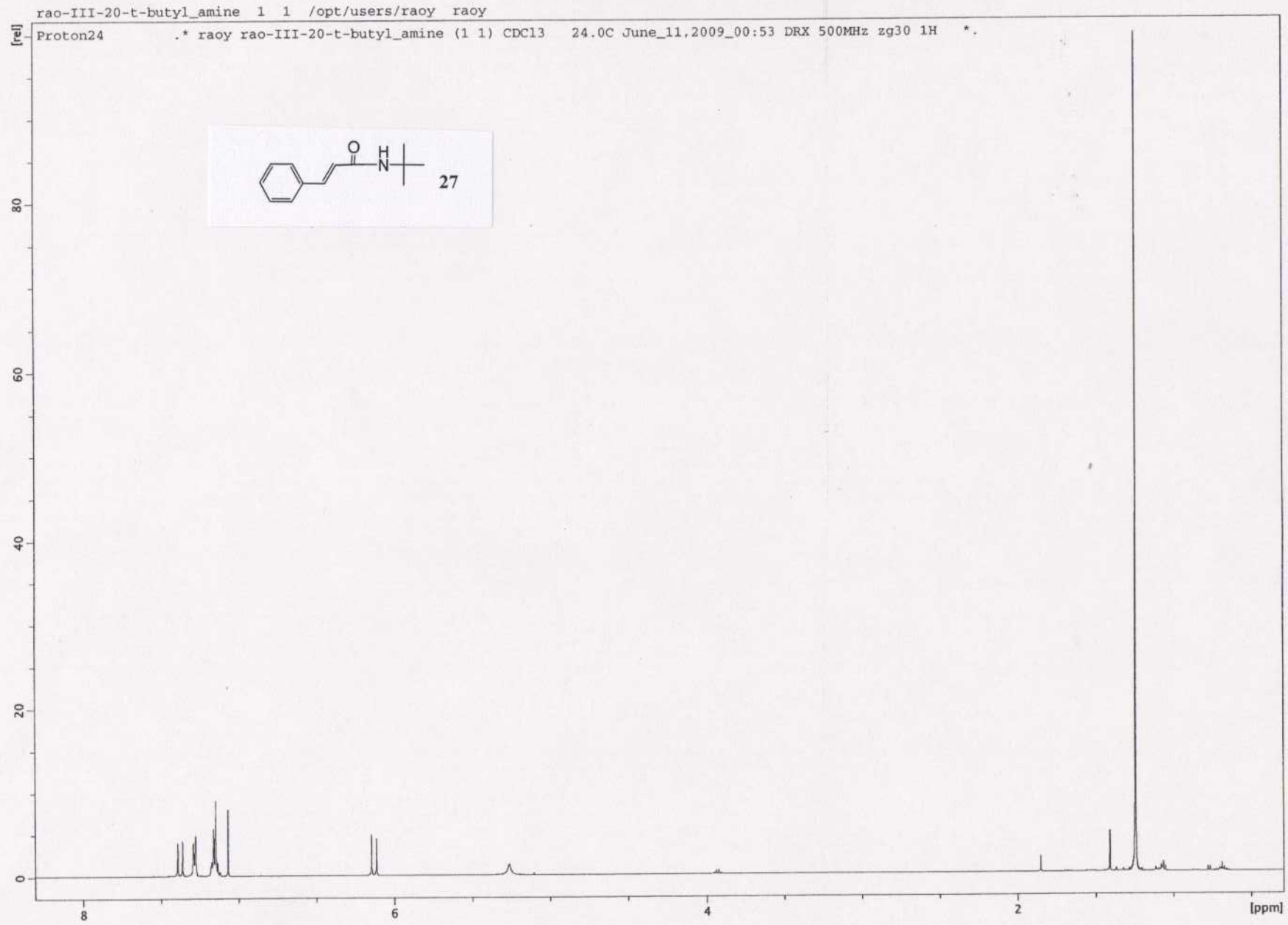

rao-III-20-Aniline 10 1 /opt/users/raoy raoy
.* raoy rao-III-20-Aniline (10 1) CDCl3 24.3C June_11,2009_13:55:52 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
0
20
40
60
80
180
160
140
120
100
80
60
40
[ppm]
O
H
N
24

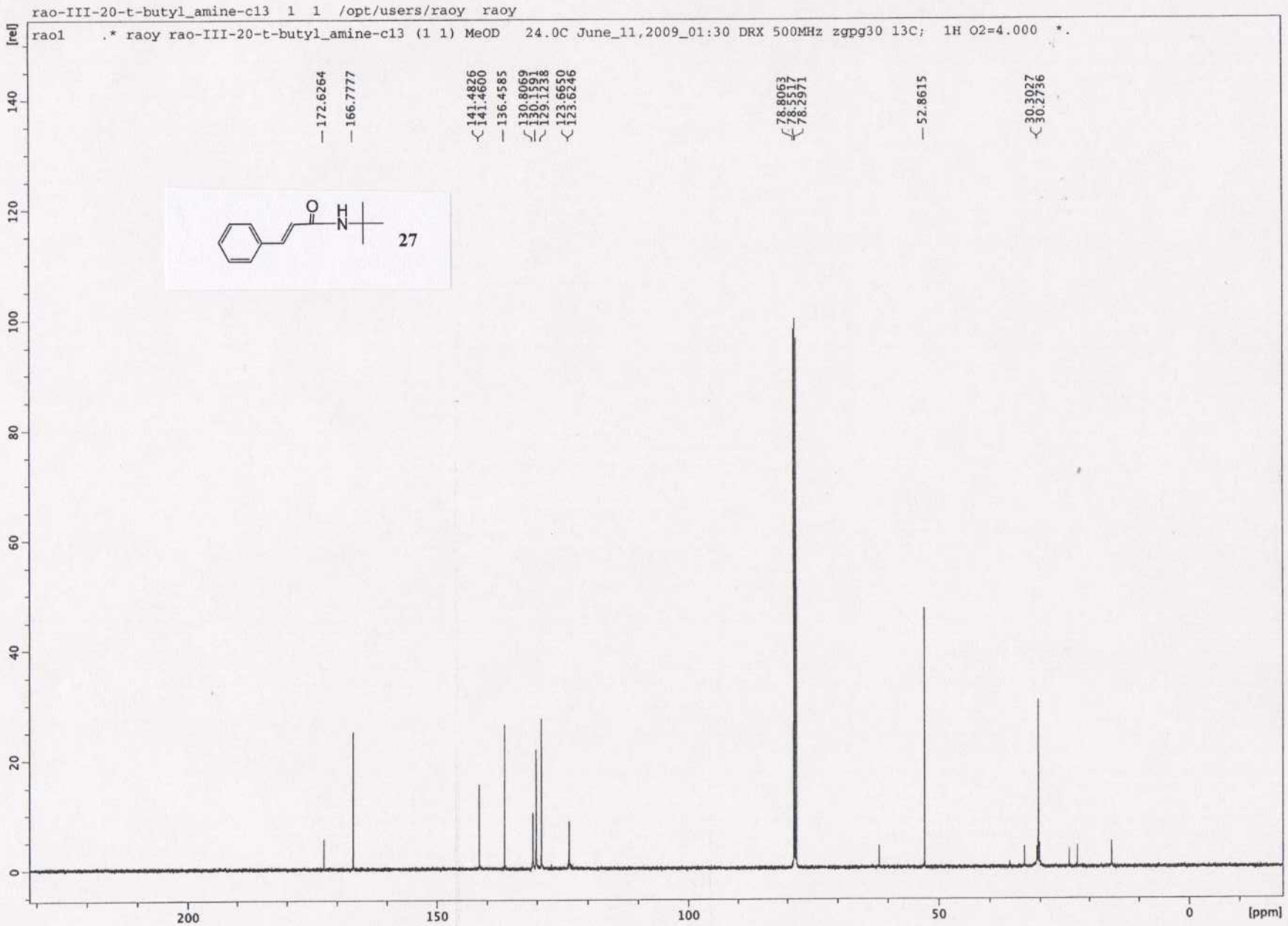

rao-III-19-t-butyl_amine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-19-t-butyl_amine (1 1) CDCl3 24.0C June_09,2009_18:52 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
8
6
4
2
[ppm]
25

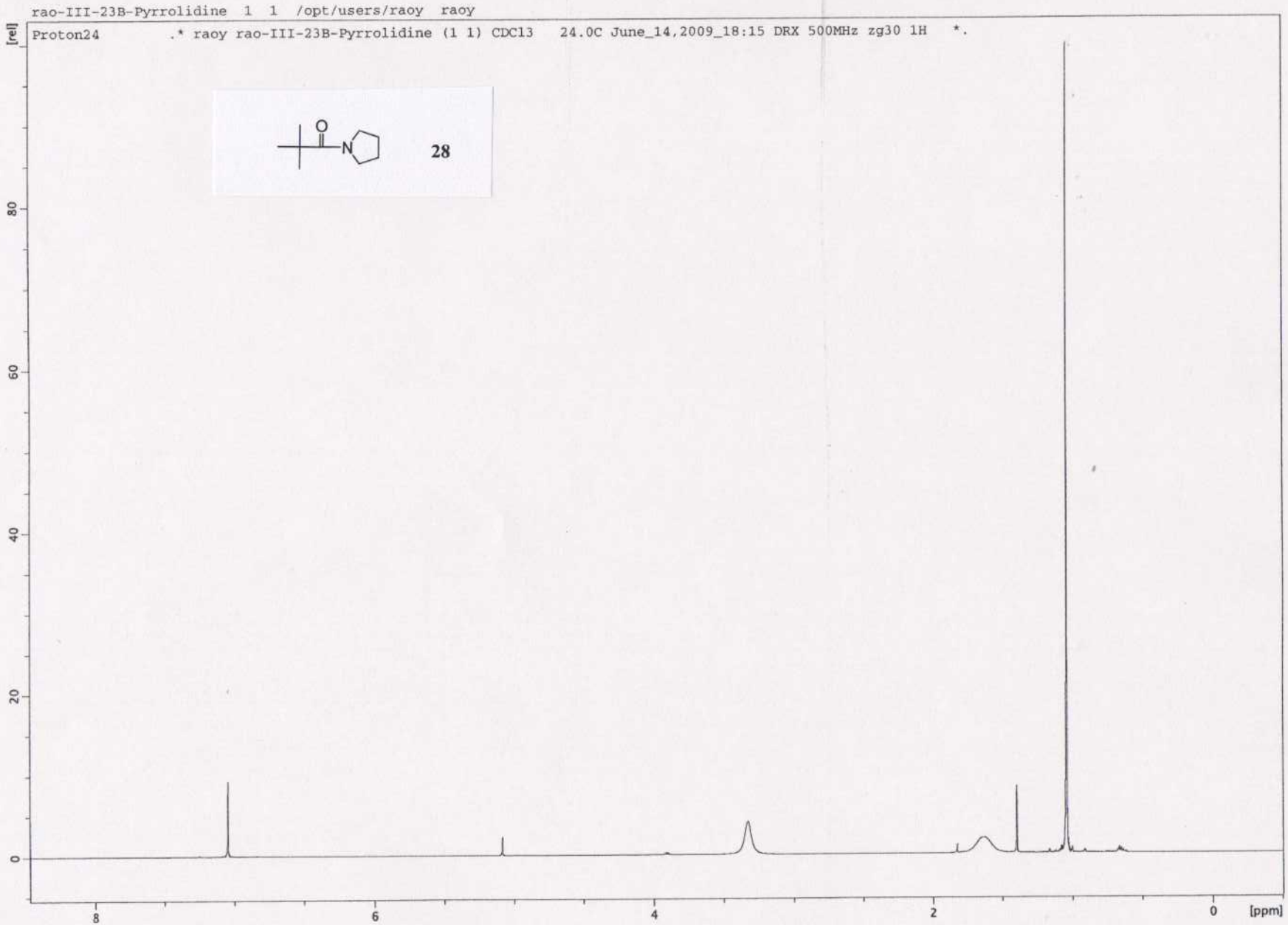

"rao-III-19-t-butyl amine" 10 1 /opt/users/raoy raoy
.* raoy rao-III-19-t-butyl amine (10 1) CDCl3 24.1C June_10,2009_00:39:47 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm
25
[rel]
120
100
80
60
40
20
0
150
100
50
[ppm]

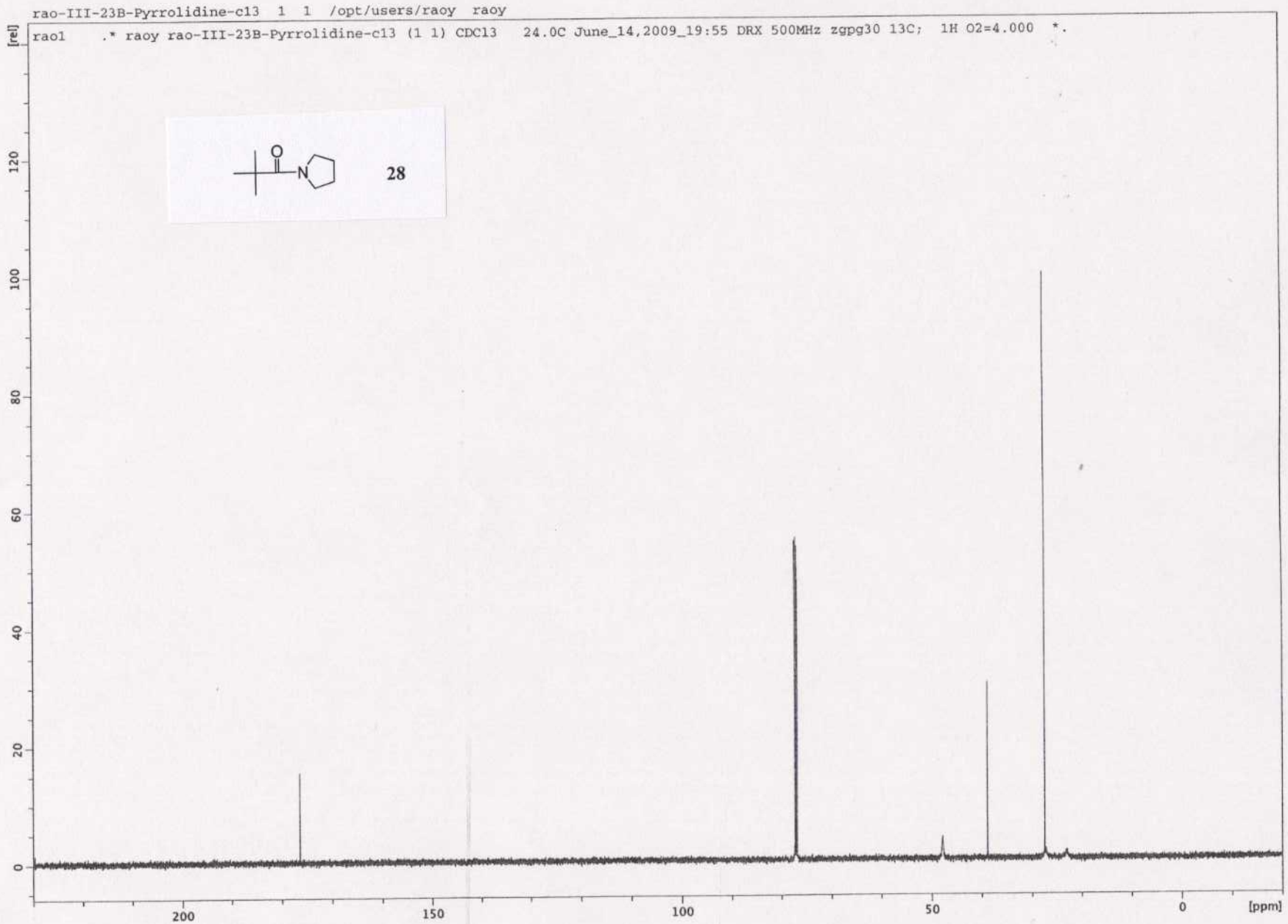

rao-III-26-t-butyl_amine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-26-t-butyl_amine (1 1) CDCl3 24.0C June_12,2009_11:28 DRX 500MHz zg30 1H *.
[rel]
BocHN
COOBn
H
N
O
26
0
20
40
60
80
8
6
4
2
[ppm]

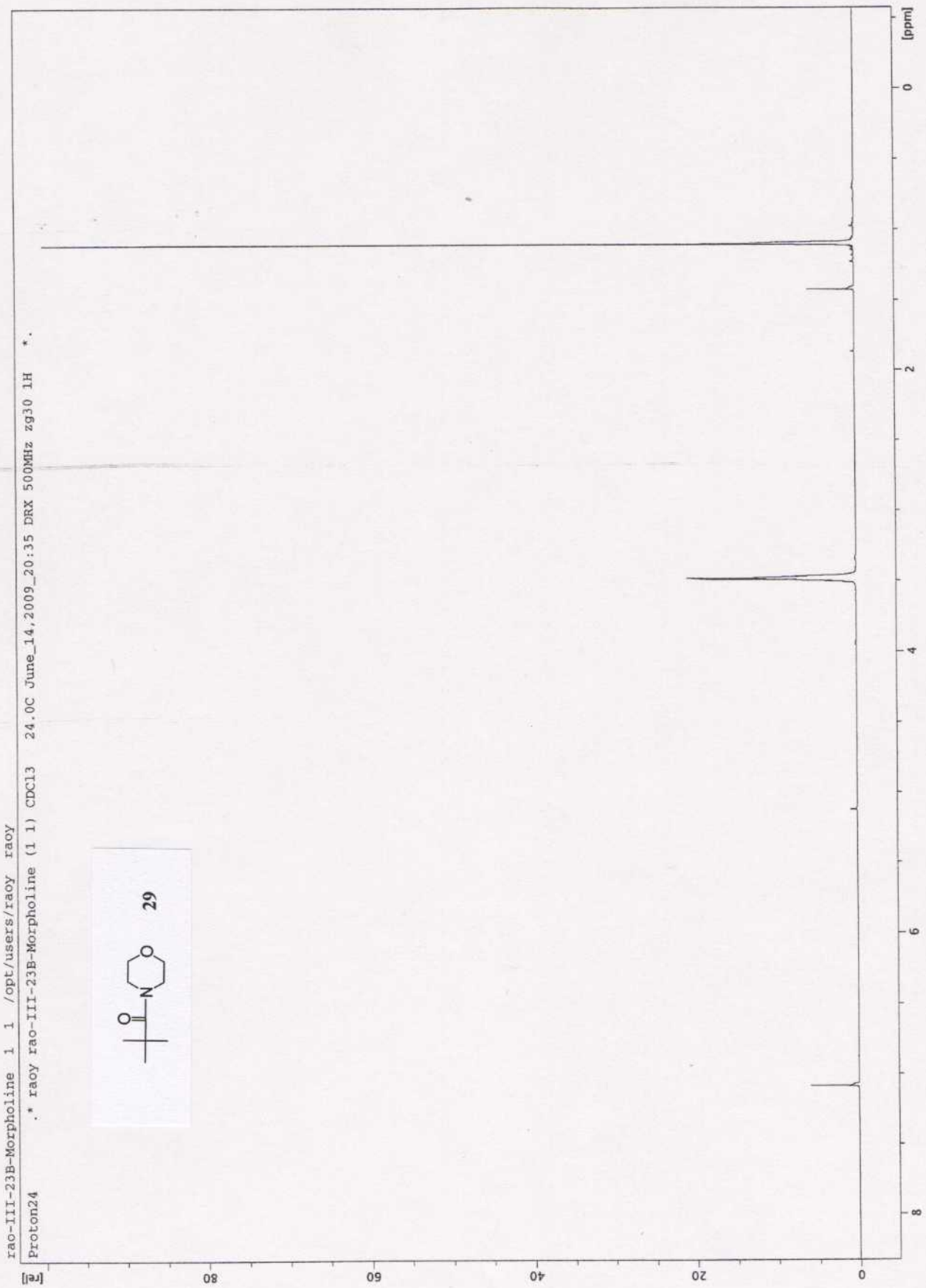

rao-III-26-t-butyl amine 10 1 /opt/users/raoy raoy
.* raoy rao-III-26-t-butyl amine (10 1) CDCl3 24.1C June_12,2009_12:41:18 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm
[rel]
100
80
60
40
20
0
150
100
50
[ppm]
BocHN
COOBn
H
N
O
26

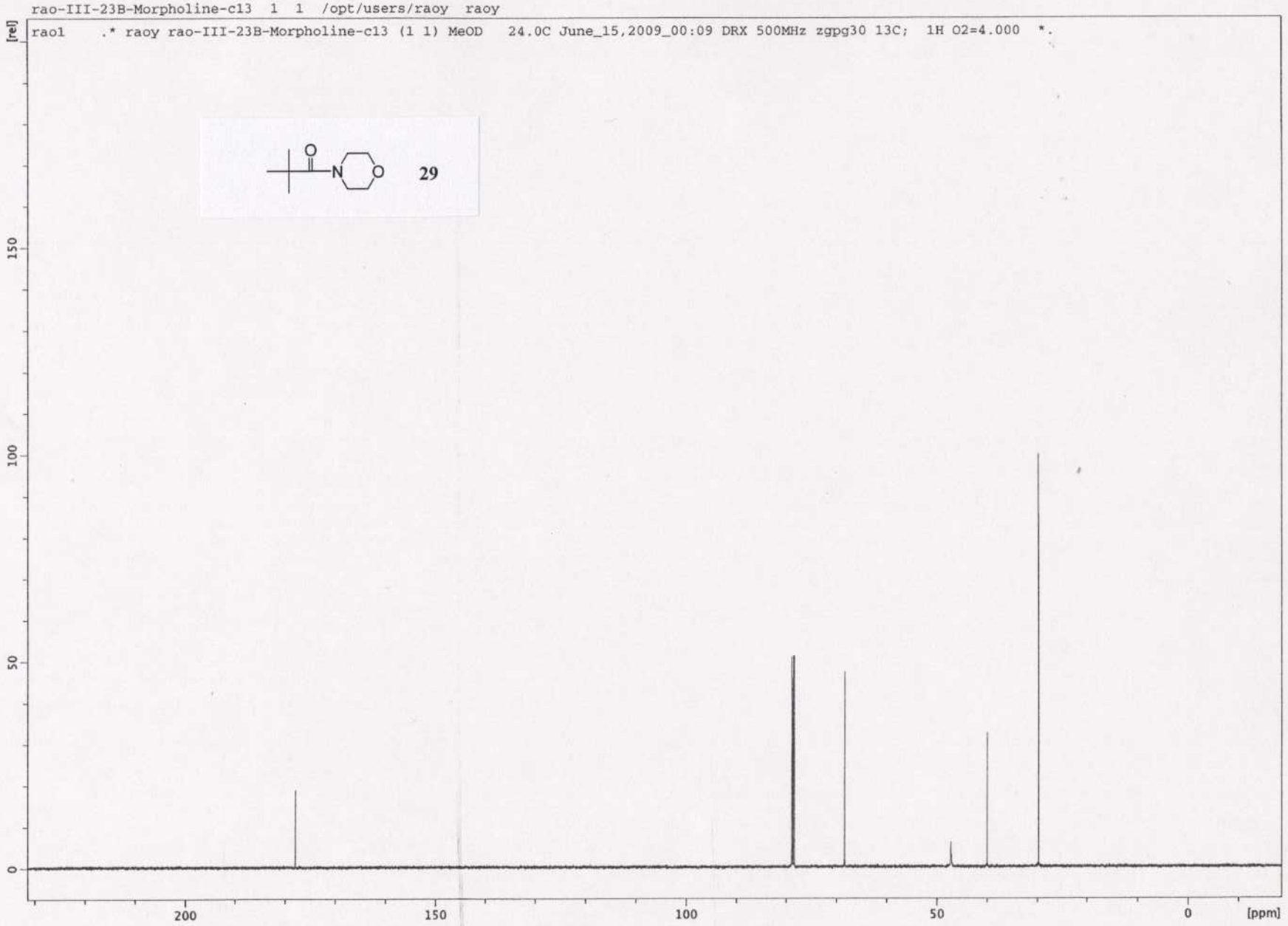

rao-III-20-t-butyl_amine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-20-t-butyl_amine (1 1) CDCl3 24.0C June_11,2009_00:53 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
80
8
6
4
2
[ppm]
O
H
N
27

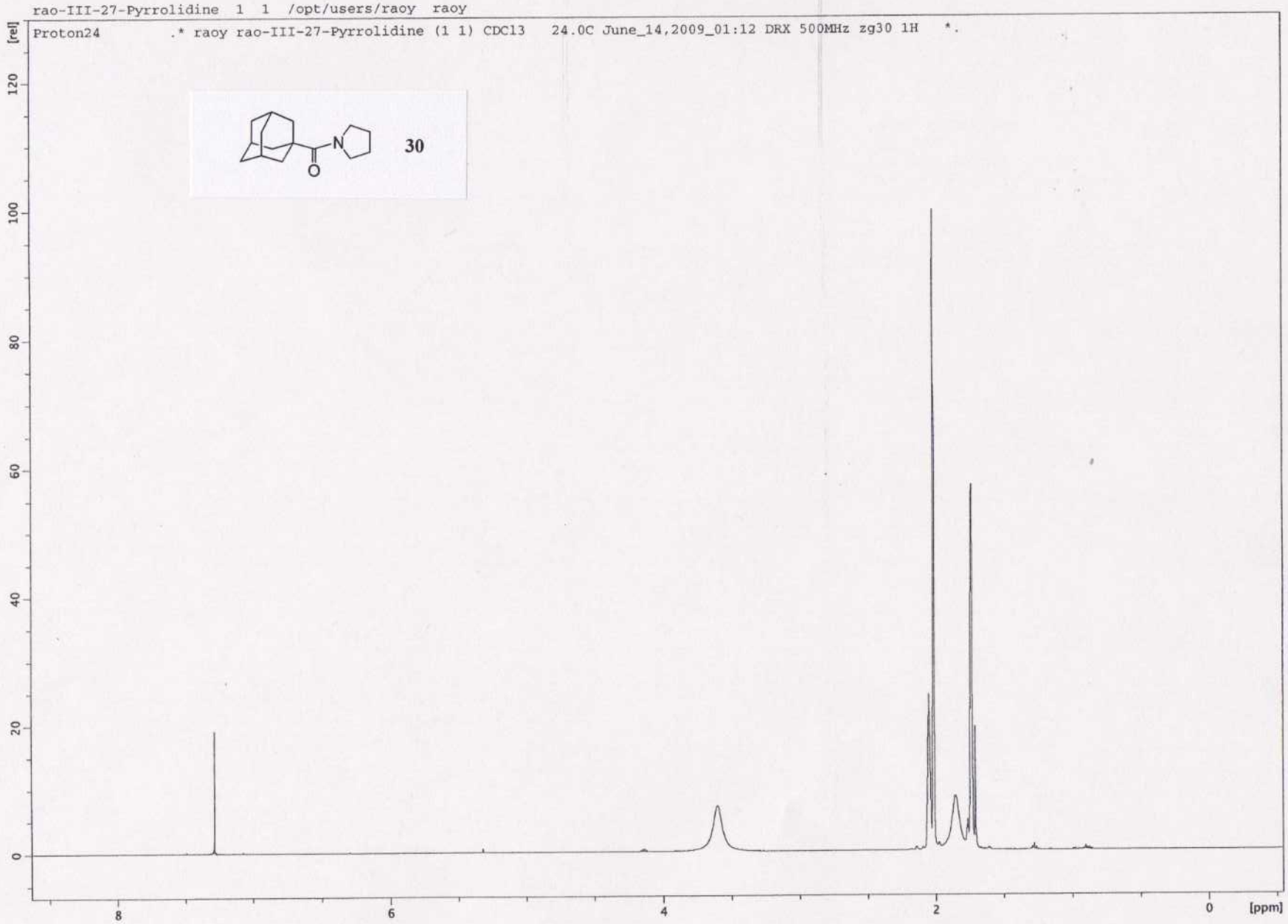

rao-III-20-t-butyl_amine-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-20-t-butyl_amine-c13 (1 1) MeOD 24.0C June_11,2009_01:30 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
172.6264
166.7777
141.4826
141.4600
136.4585
130.8069
130.1591
129.1238
123.6650
123.6246
78.8063
78.5517
78.2971
52.8615
30.3027
30.2736
27
[ppm]

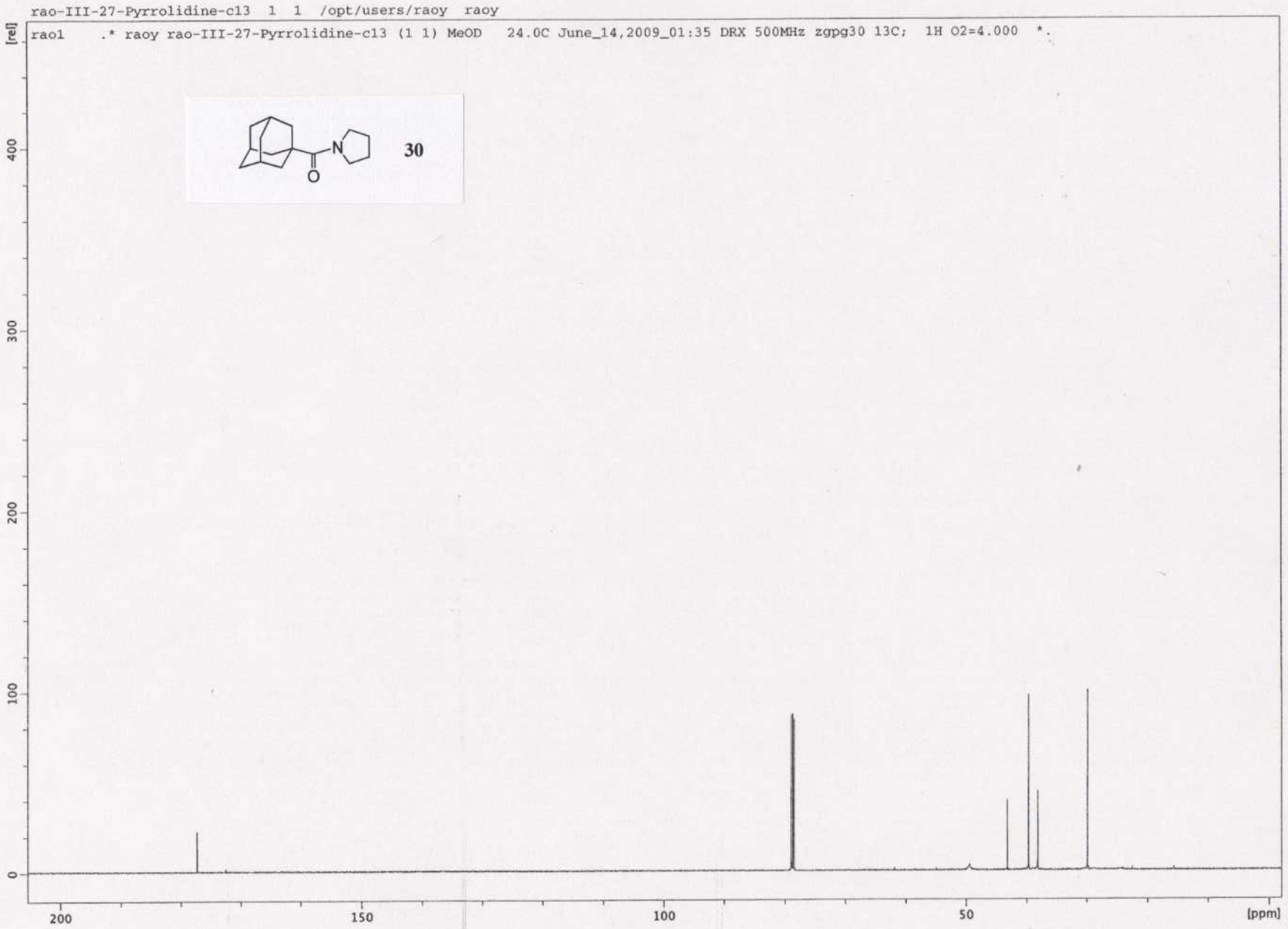

rao-III-23B-Pyrrolidine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-23B-Pyrrolidine (1 1) CDCl3 24.0C June_14,2009_18:15 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
80
8
6
4
2
0
[ppm]
O
N
28

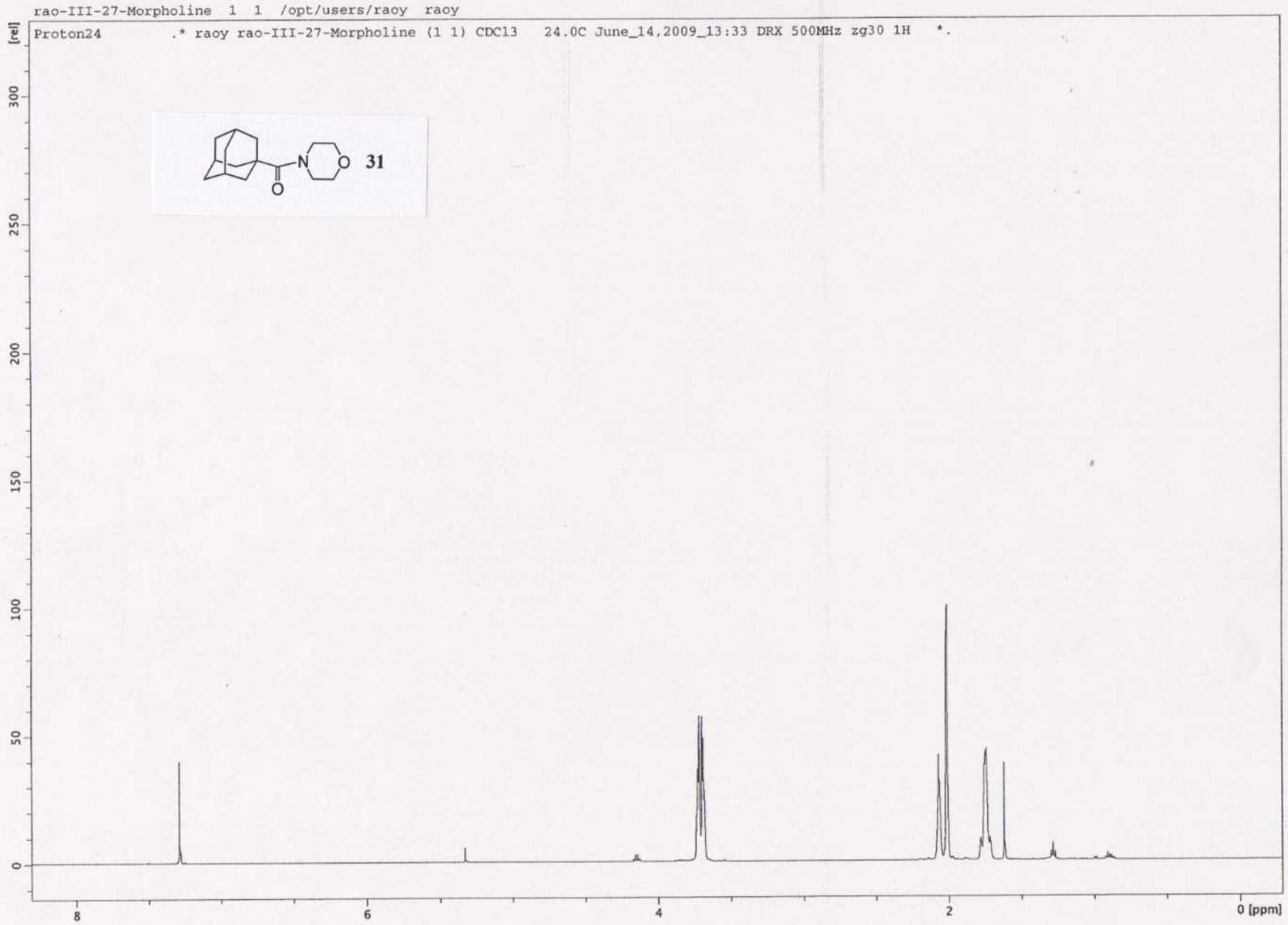

rao-III-23B-Pyrrolidine-c13 1 1 /opt/users/raoy raoy
raoy .* raoy rao-III-23B-Pyrrolidine-c13 (1 1) CDCl3 24.0C June_14,2009_19:55 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
120
100
80
60
40
20
0
28
200
150
100
50
0
[ppm]

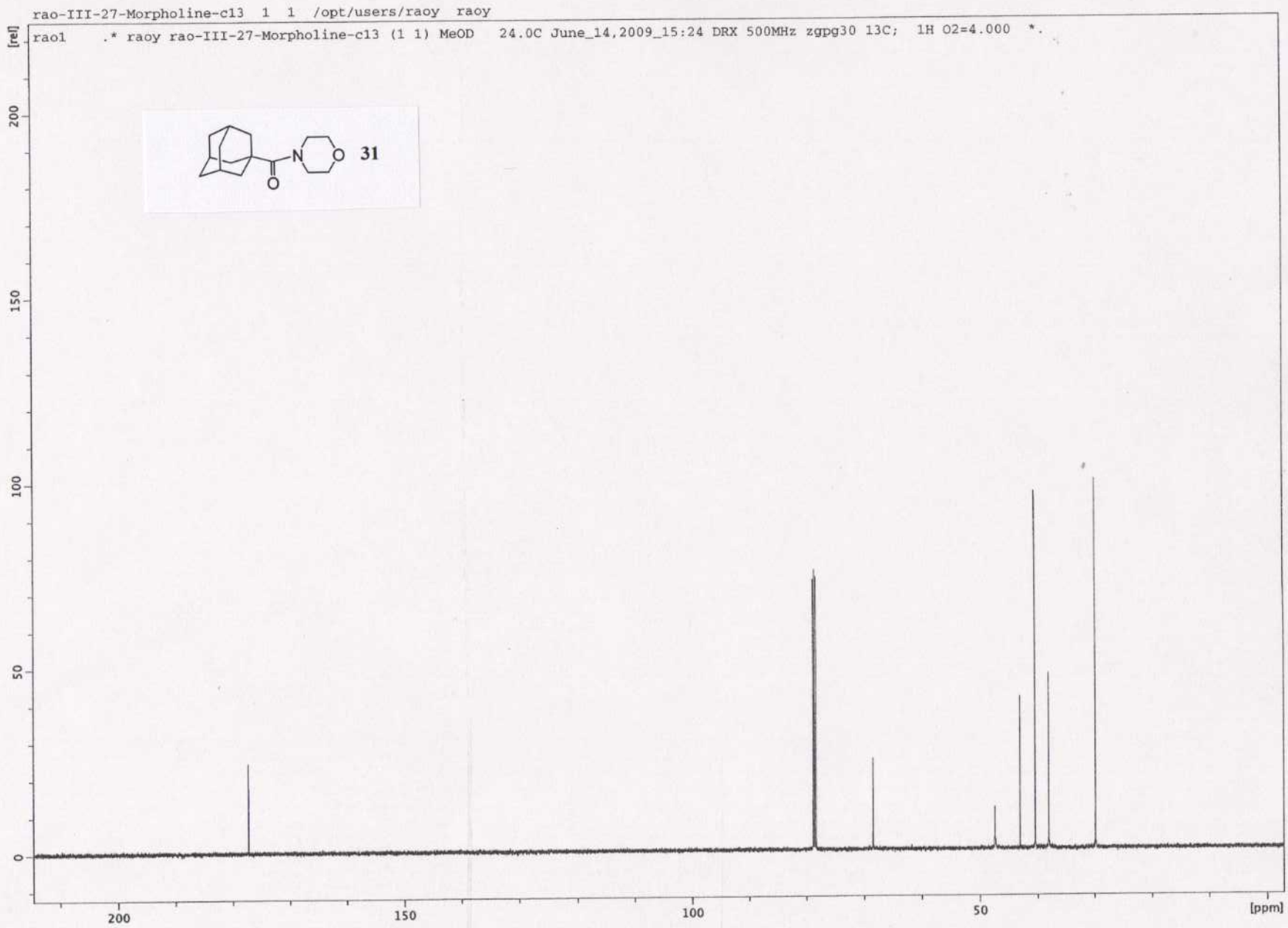

rao-III-23B-Morpholine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-23B-Morpholine (1 1) CDCl3 24.0C June_14,2009_20:35 DRX 500MHz zg30 1H *.
[rel]
0
20
40
60
80
8
6
4
2
0
[ppm]
O
N
O
29

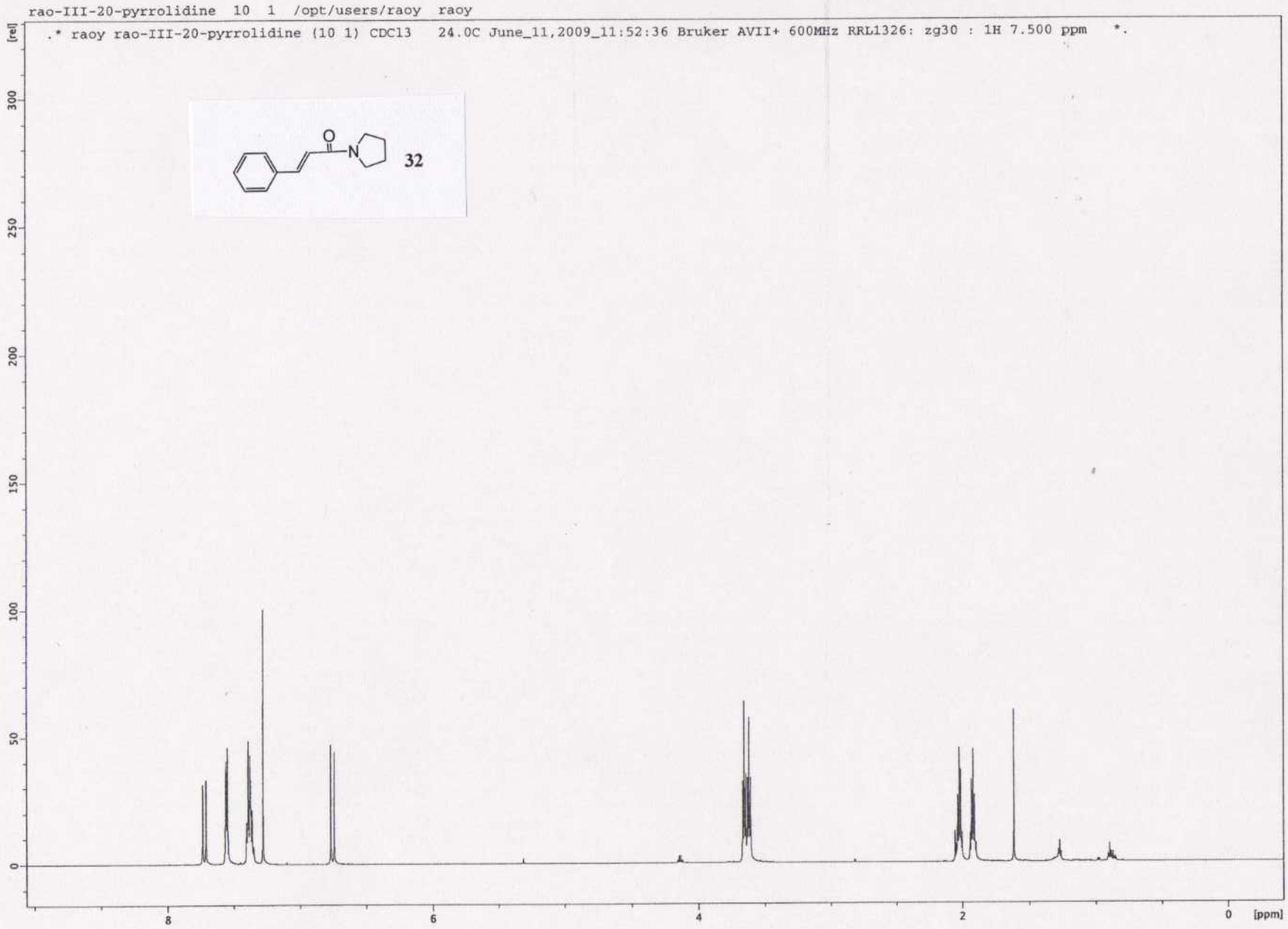

rao-III-23B-Morpholine-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-23B-Morpholine-c13 (1 1) MeOD 24.0C June_15,2009_00:09 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
29
[rel]
150
100
50
0
200
150
100
50
0
[ppm]

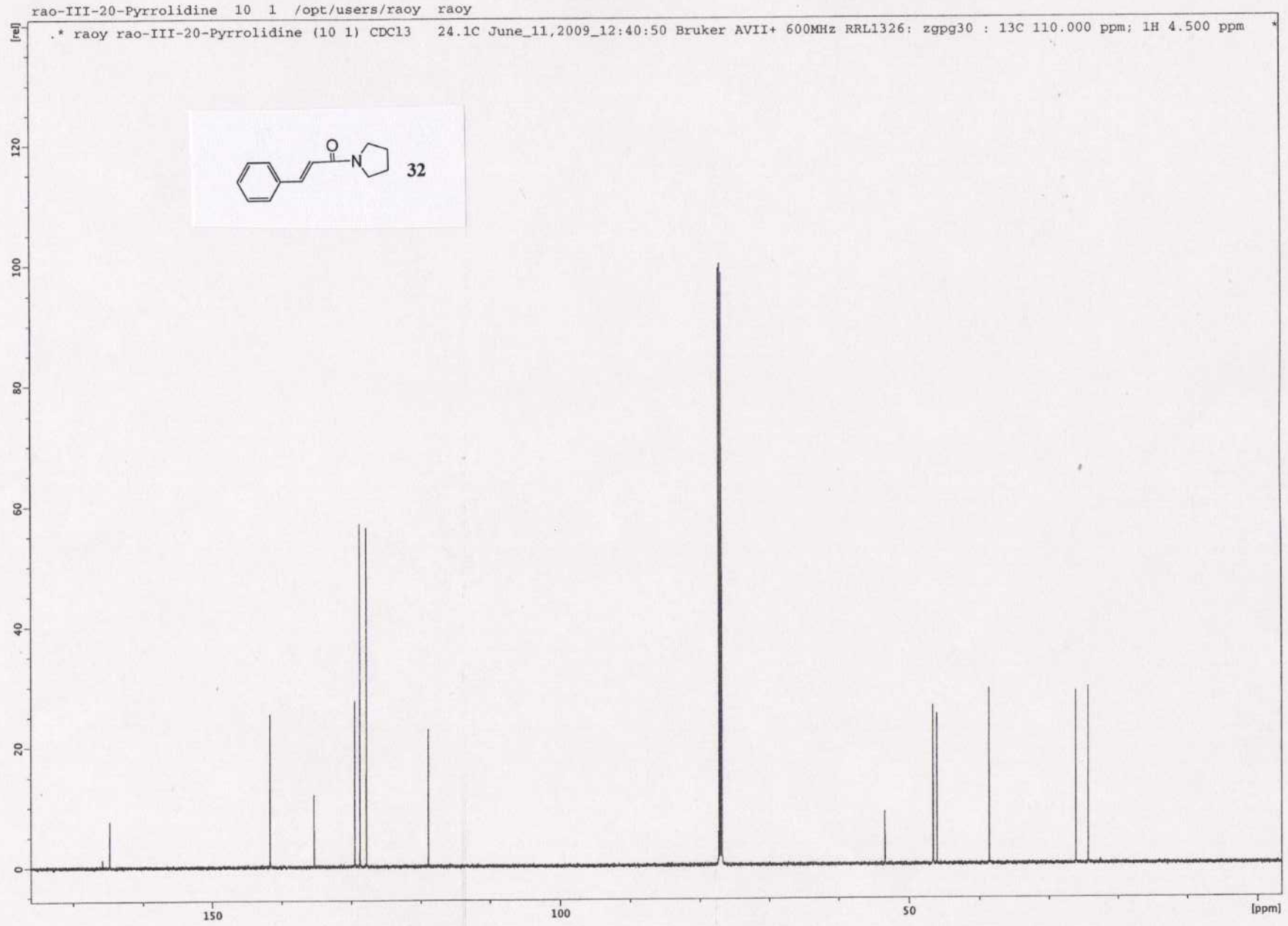

rao-III-27-Pyrrolidine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-27-Pyrrolidine (1 1) CDCl3 24.0C June_14,2009_01:12 DRX 500MHz zg30 1H *.
[rel]
120
100
80
60
40
20
0
8
6
4
2
0
[ppm]
N
O
30

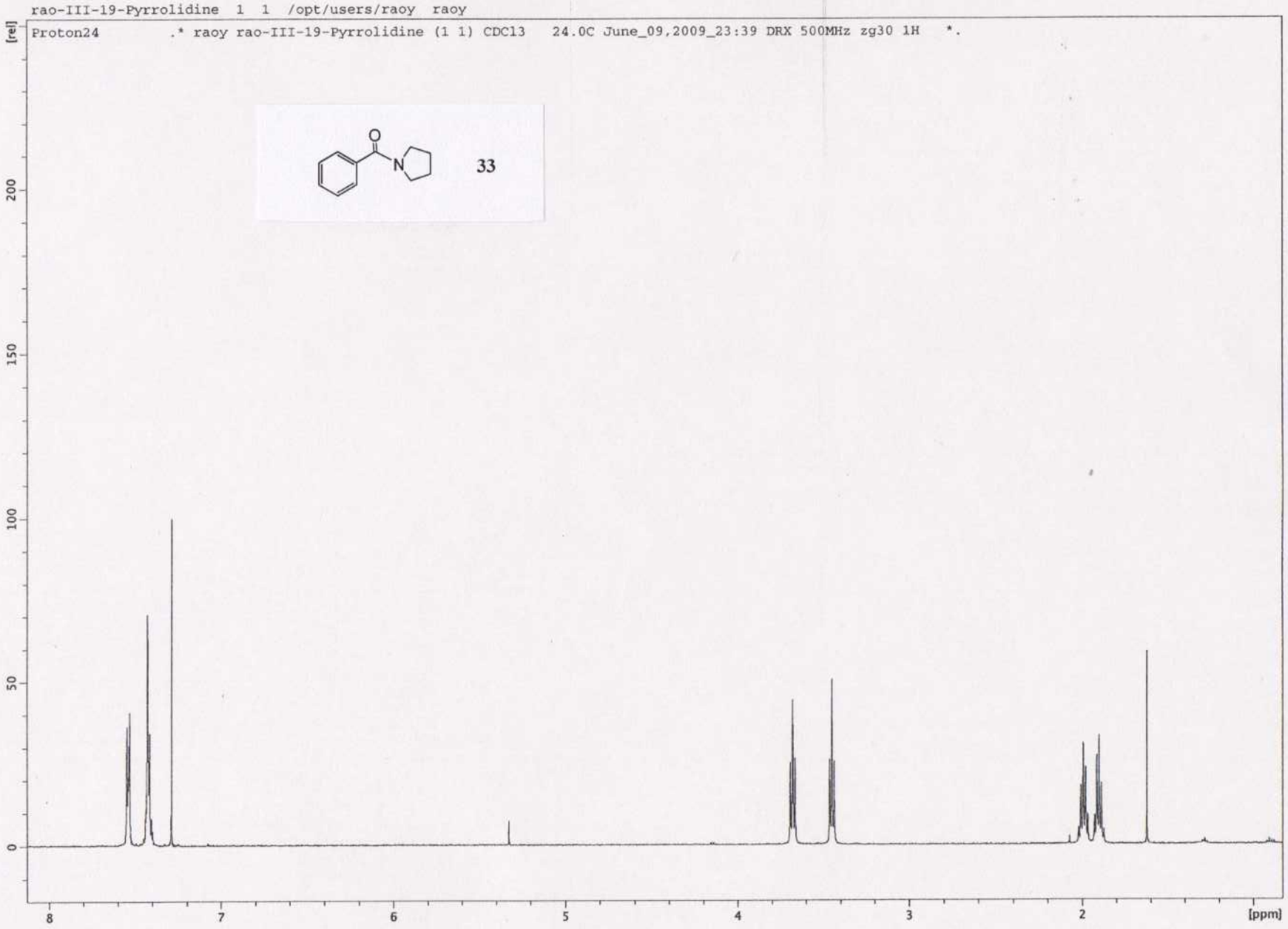

rao-III-27-Pyrrolidine-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-27-Pyrrolidine-c13 (1 1) MeOD 24.0C June_14,2009_01:35 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
400
300
200
100
0
N
O
30
200
150
100
50
[ppm]

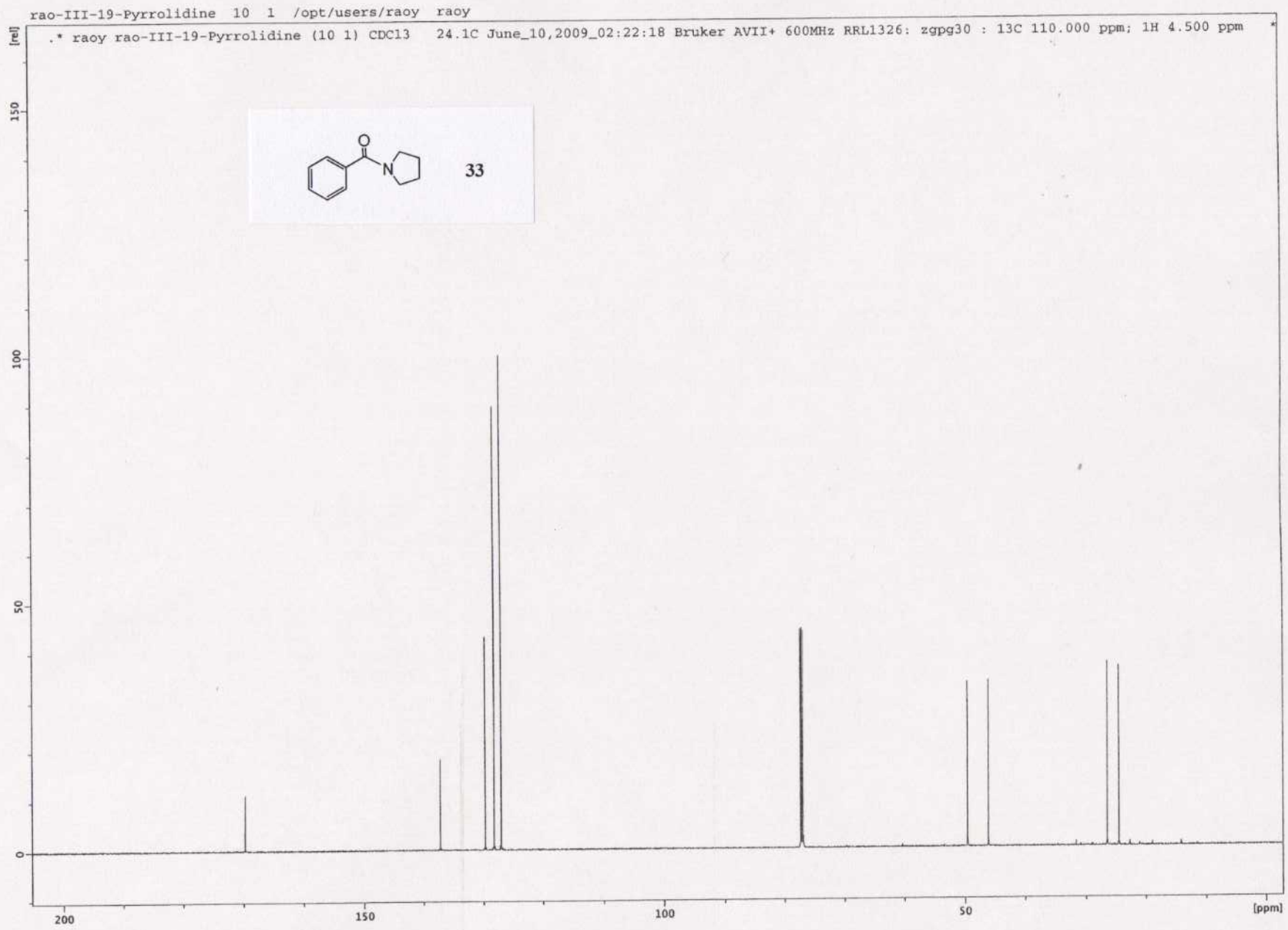

rao-III-27-Morpholine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-27-Morpholine (1 1) CDCl3 24.0C June_14,2009_13:33 DRX 500MHz zg30 1H *.
[rel]
300
250
200
150
100
50
0
8
6
4
2
0 [ppm]
N
O
O
31

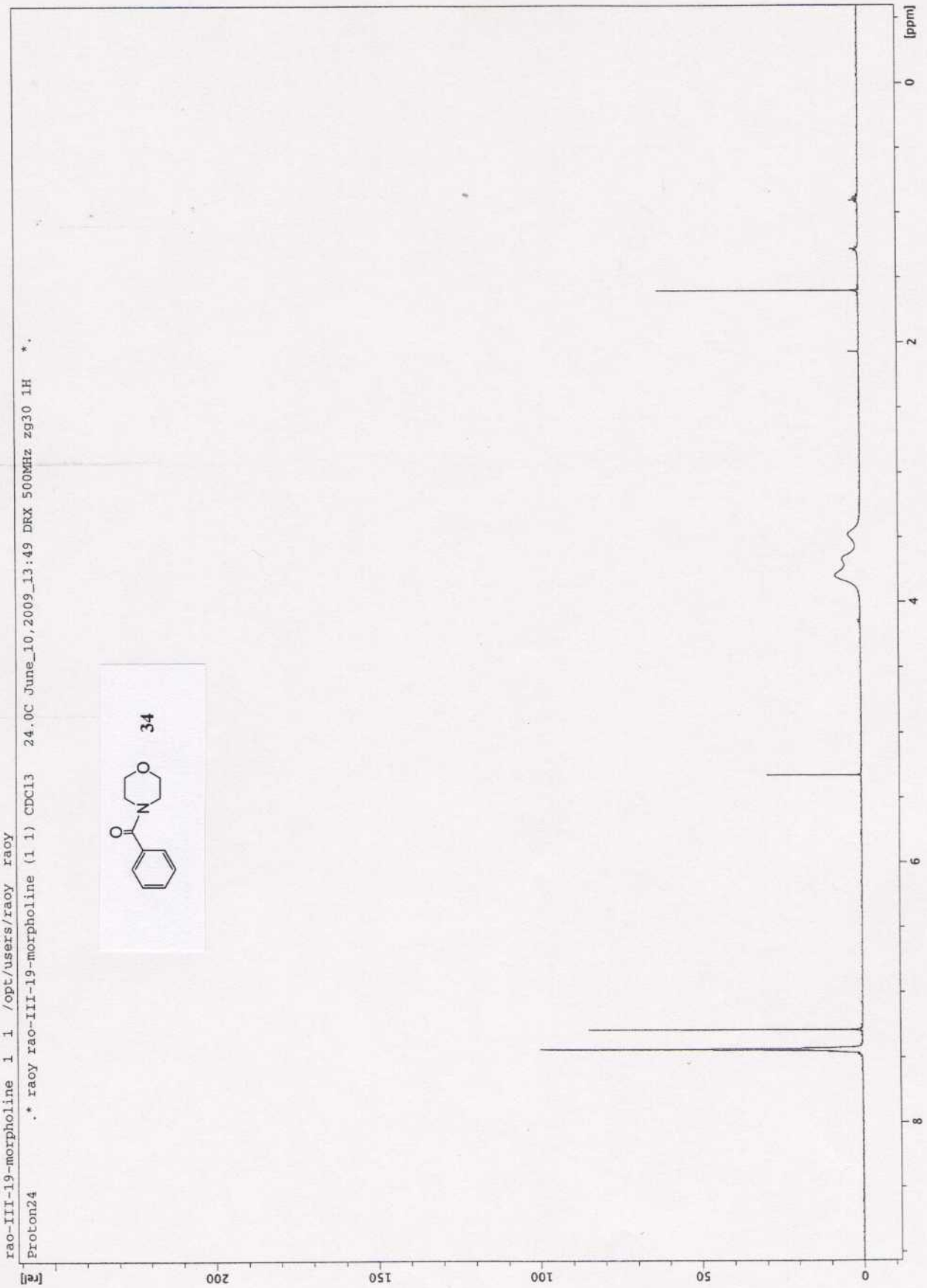

rao-III-27-Morpholine-c13 1 1 /opt/users/raoy raoy
raol .* raoy rao-III-27-Morpholine-c13 (1 1) MeOD 24.0C June_14,2009_15:24 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
200
150
100
50
0
N
O
O
31
200
150
100
50
[ppm]

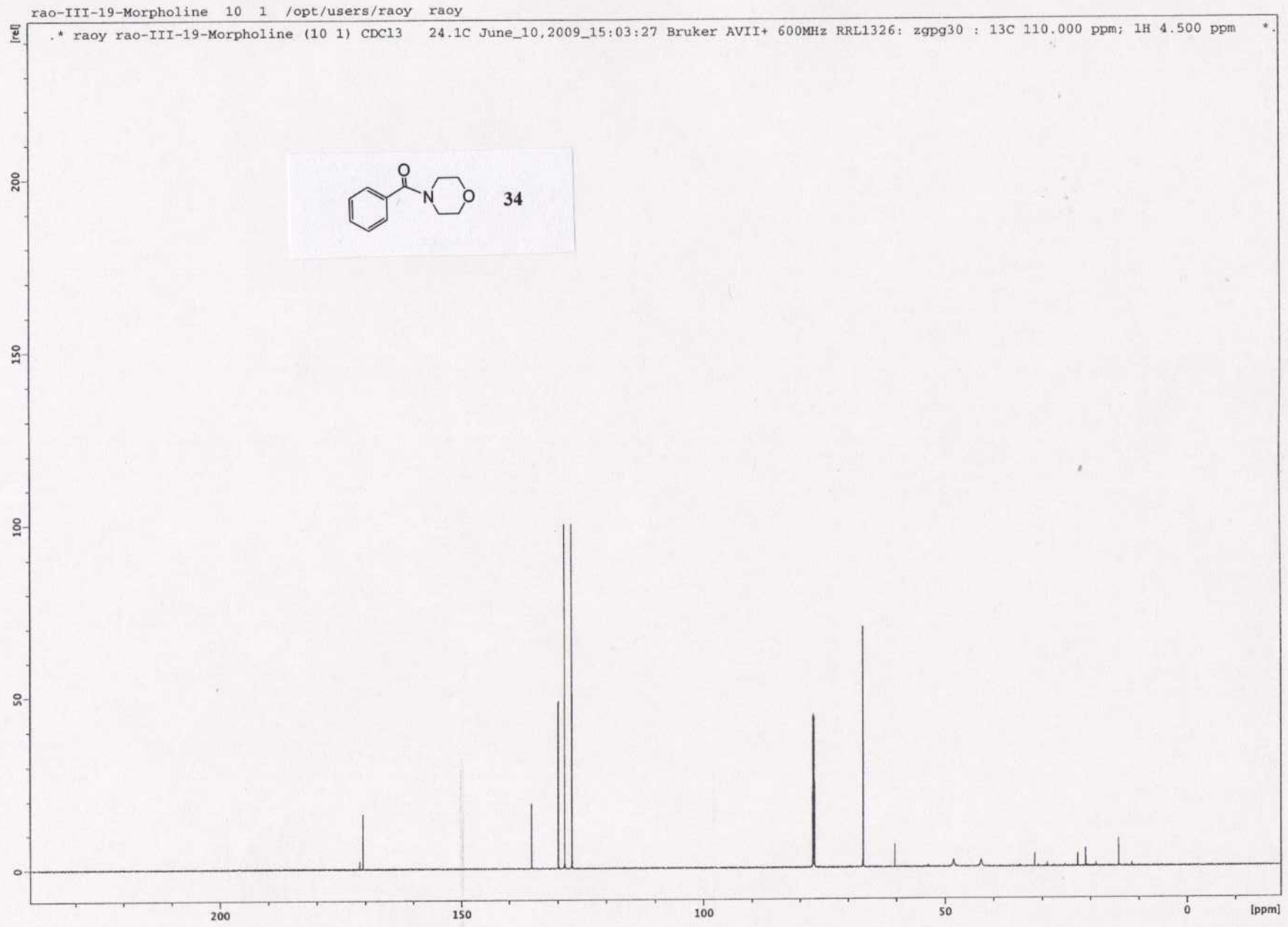

rao-III-20-pyrrolidine 10 1 /opt/users/raoy raoy
.* raoy rao-III-20-pyrrolidine (10 1) CDCl3 24.0C June_11,2009_11:52:36 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm *.
[rel]
300
250
200
150
100
50
0
8
6
4
2
0
[ppm]
O
N
32

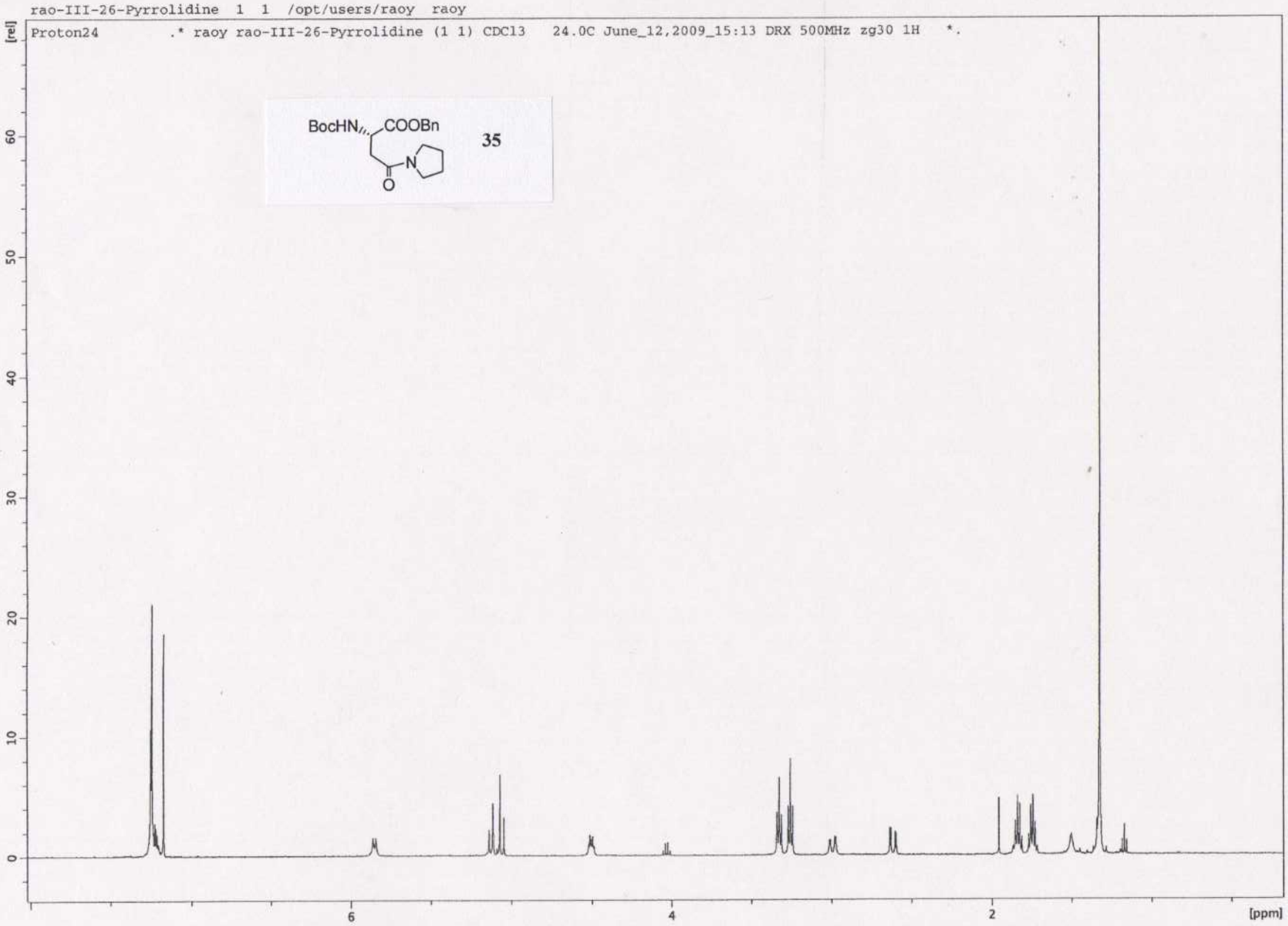

rao-III-20-Pyrrolidine 10 1 /opt/users/raoy raoy
.* raoy rao-III-20-Pyrrolidine (10 1) CDCl3 24.1C June_11,2009_12:40:50 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm
[rel]
120
100
80
60
40
20
0
150
100
50
[ppm]
O
N
32

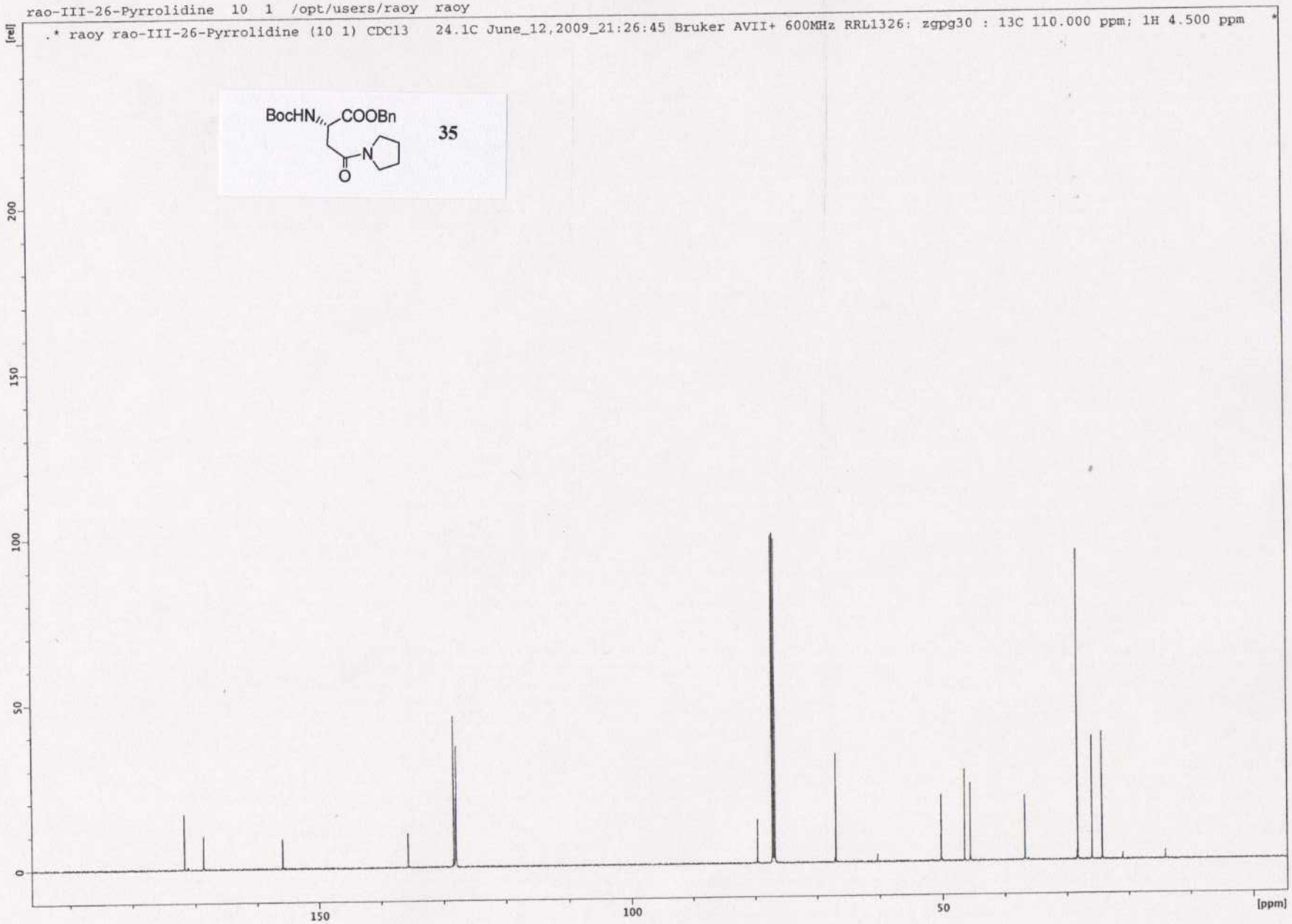

rao-III-19-Pyrrolidine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-19-Pyrrolidine (1 1) CDCl3 24.0C June_09,2009_23:39 DRX 500MHz zg30 1H *.
[rel]
200
150
100
50
0
O
N
33
8
7
6
5
4
3
2
[ppm]

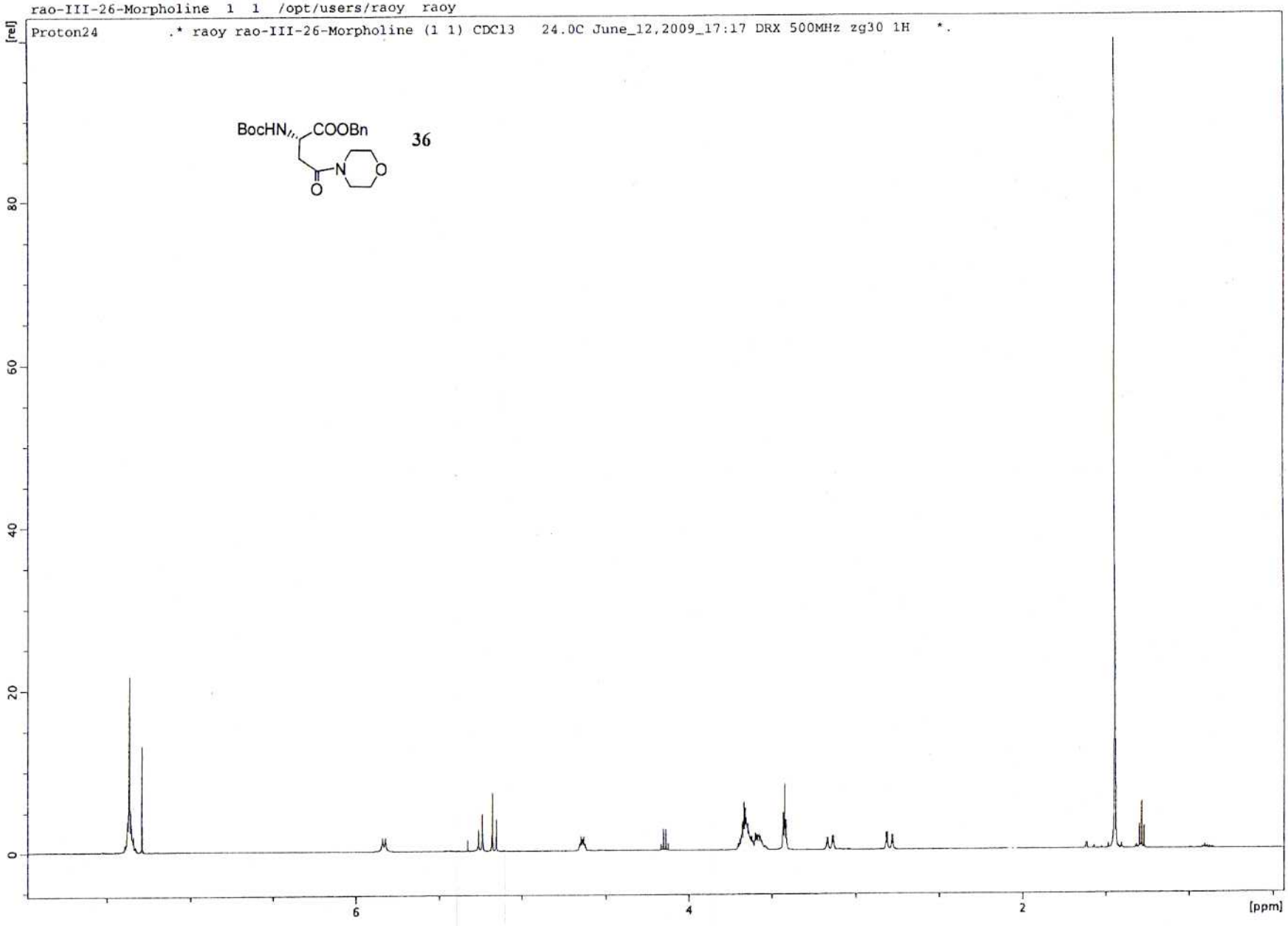

rao-III-19-Pyrrolidine 10 1 /opt/users/raoy raoy
.* raoy rao-III-19-Pyrrolidine (10 1) CDCl3 24.1C June_10,2009_02:22:18 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm
[rel]
150
100
50
0
200
150
100
50
[ppm]
33

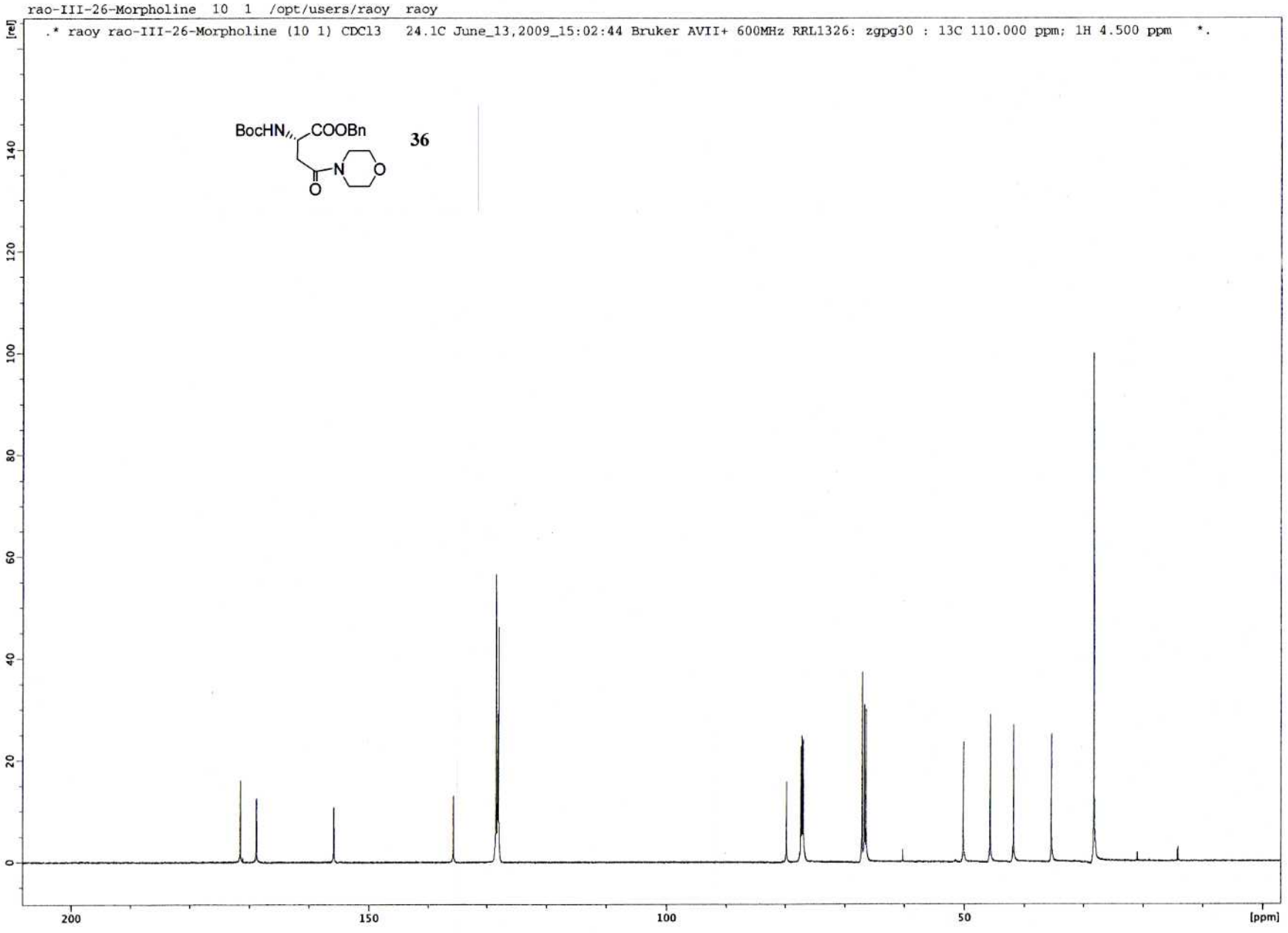

rao-III-19-morpholine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-19-morpholine (1 1) CDCl3 24.0C June_10,2009_13:49 DRX 500MHz zg30 1H *.
34
[rel]
200
150
100
50
0
8
6
4
2
0
[ppm]

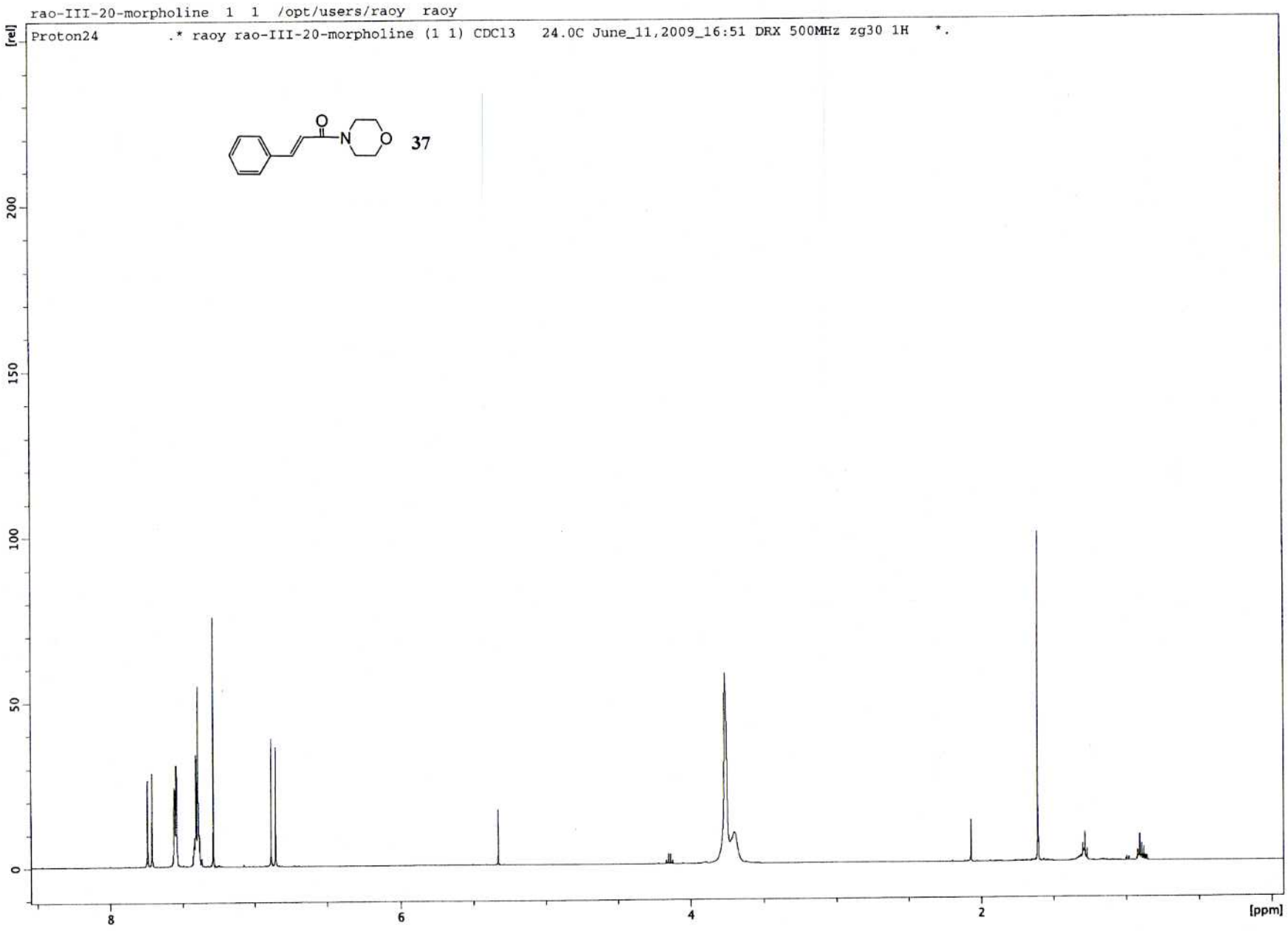

rao-III-19-Morpholine 10 1 /opt/users/raoy raoy
.* raoy rao-III-19-Morpholine (10 1) CDCl3 24.1C June_10,2009_15:03:27 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
200
150
100
50
0
O
N
O
34
200
150
100
50
0
[ppm]

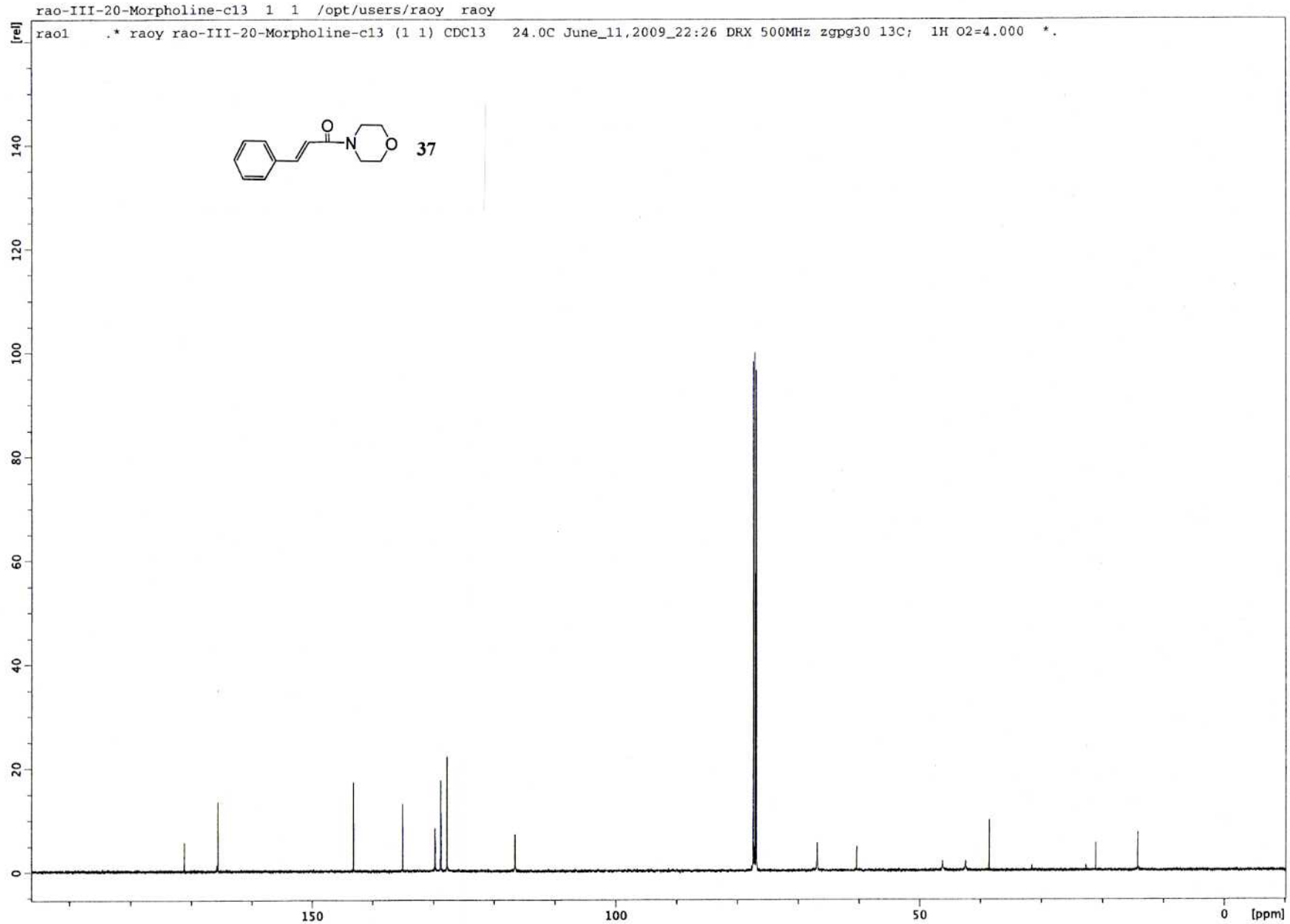

rao-III-26-Pyrrolidine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-26-Pyrrolidine (1 1) CDCl3 24.0C June_12,2009_15:13 DRX 500MHz zg30 1H *.
BocHN
COOBn
N
O
35
[rel]
60
50
40
30
20
10
0
6
4
2
[ppm]

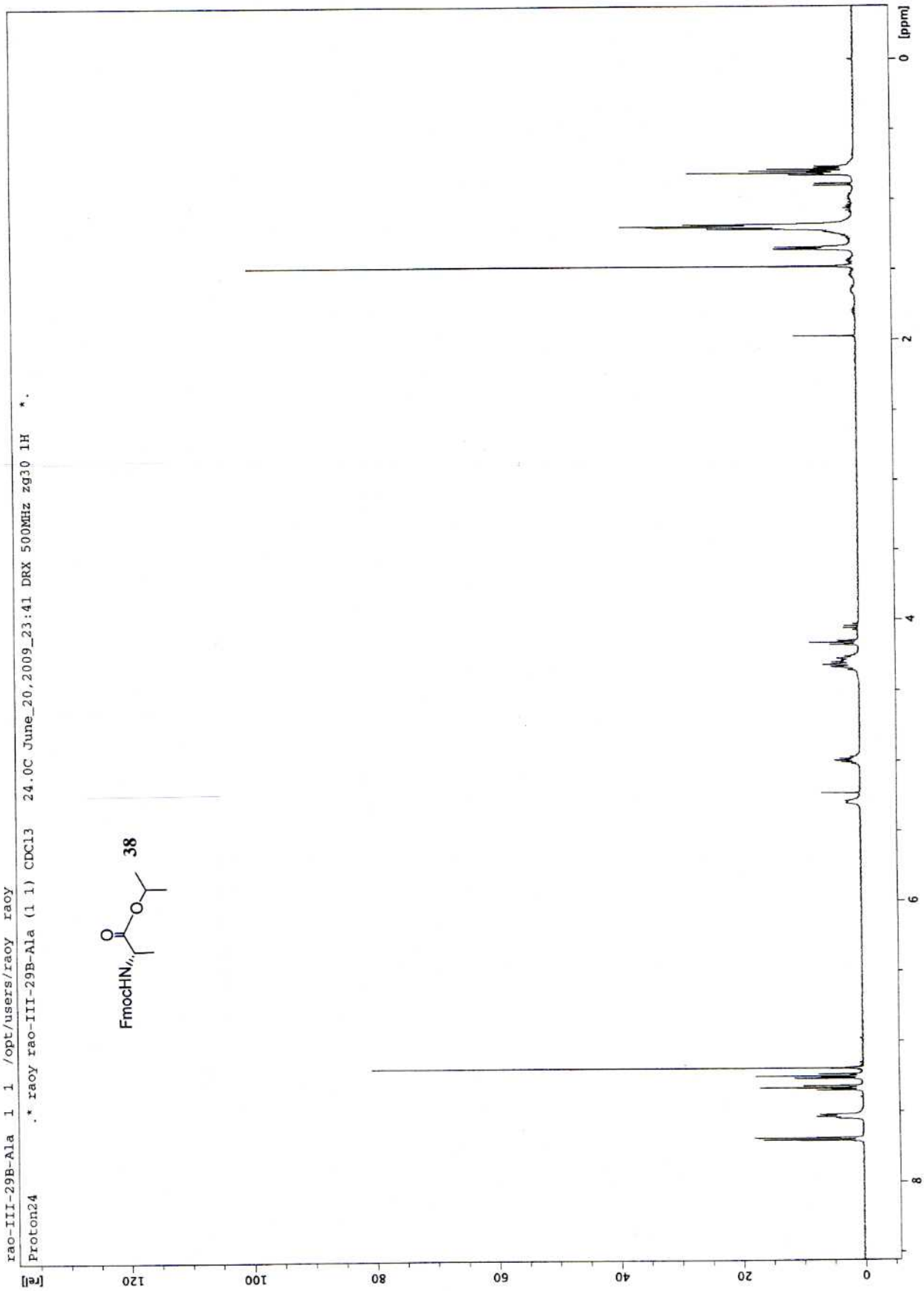

rao-III-26-Pyrrolidine 10 1 /opt/users/raoy raoy
.* raoy rao-III-26-Pyrrolidine (10 1) CDCl3 24.1C June_12,2009_21:26:45 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm
[rel]
BocHN
COOBn
35
N
O
200
150
100
50
0
150
100
50
[ppm]

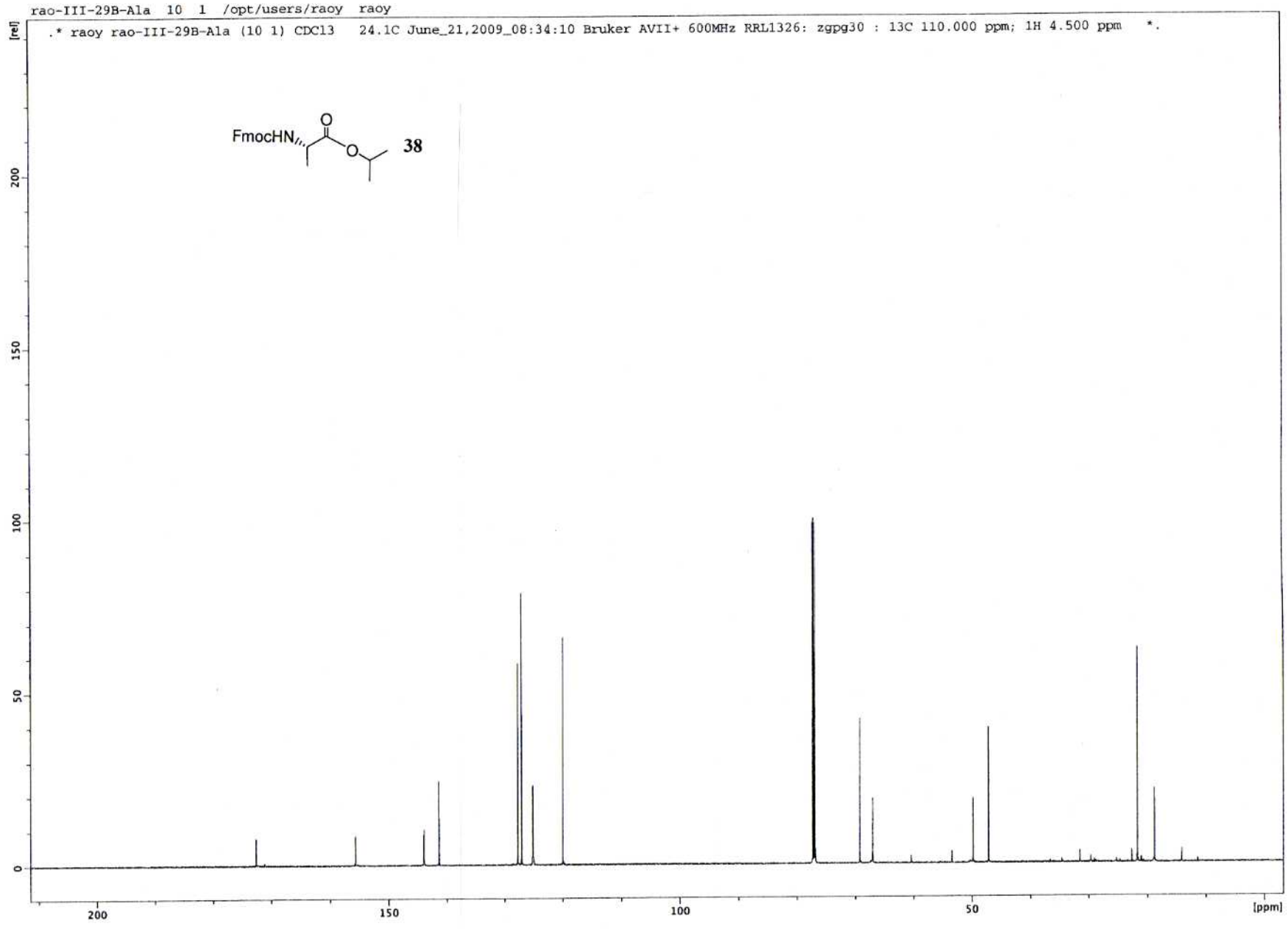

rao-III-26-Morpholine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-26-Morpholine (1 1) CDCl3 24.0C June_12,2009_17:17 DRX 500MHz zg30 1H *.
[rel]
BocHN
COOBn
O
N
O
36
0
20
40
60
80
6
4
2
[ppm]

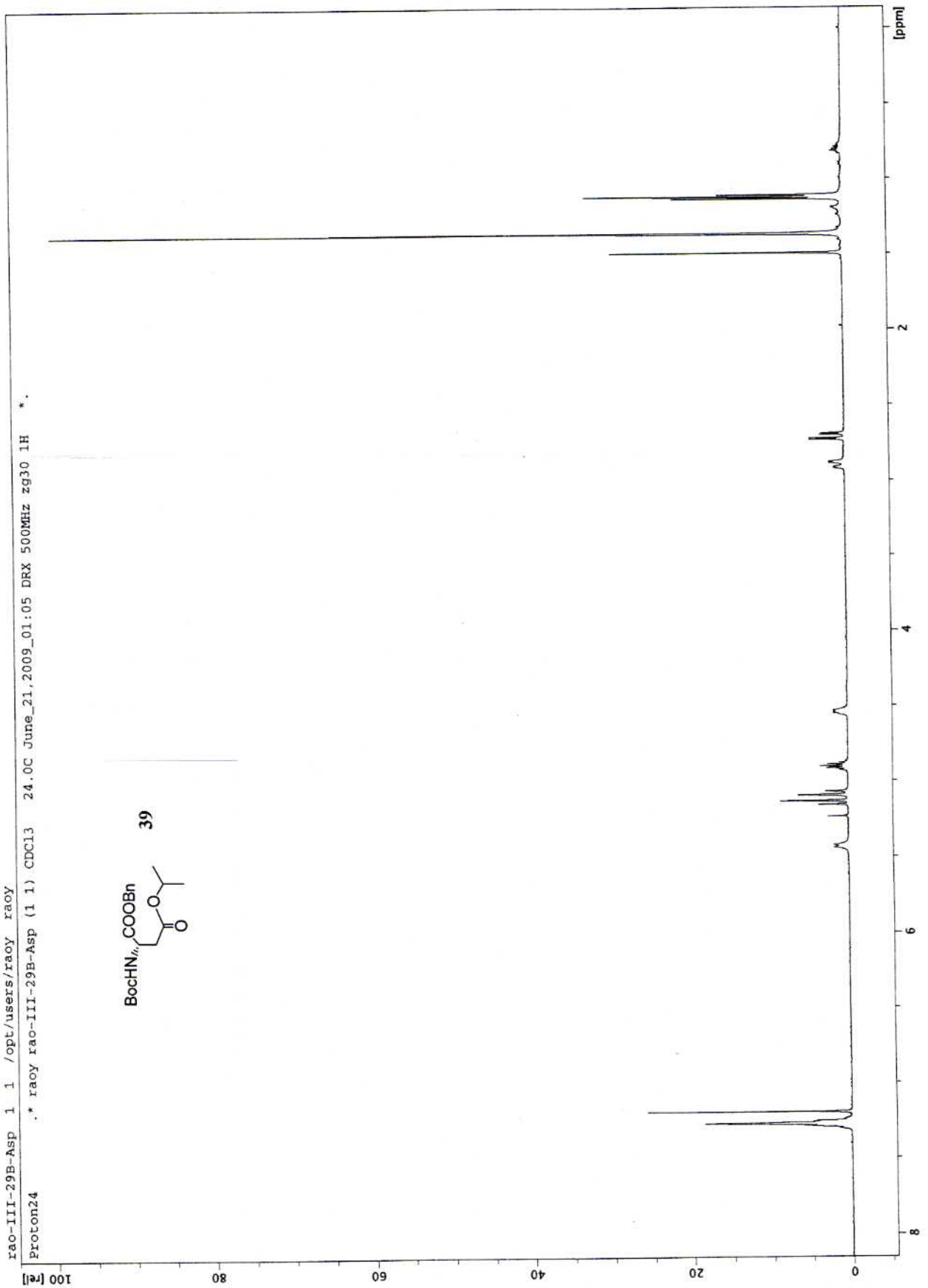

rao-III-26-Morpholine 10 1 /opt/users/raoy raoy
.* raoy rao-III-26-Morpholine (10 1) CDCl3 24.1C June_13,2009_15:02:44 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
BocHN
COOBn
N
O
O
36
0
20
40
60
80
100
120
140
200
150
100
50
[ppm]

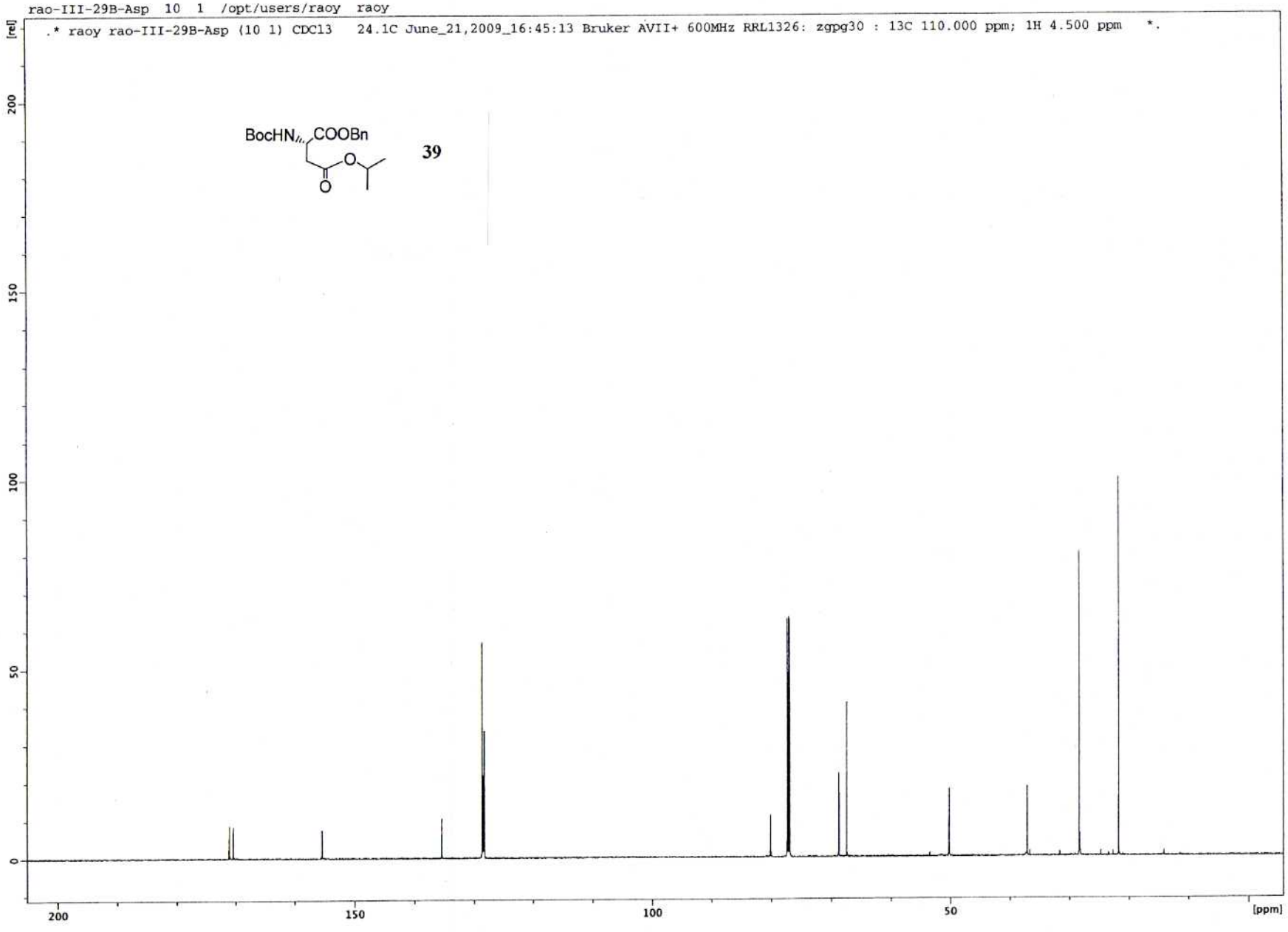

rao-III-20-morpholine 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-20-morpholine (1 1) CDCl3 24.0C June_11,2009_16:51 DRX 500MHz zg30 1H *.
[rel]
200
150
100
50
0
8
6
4
2
[ppm]
O
N
O
37

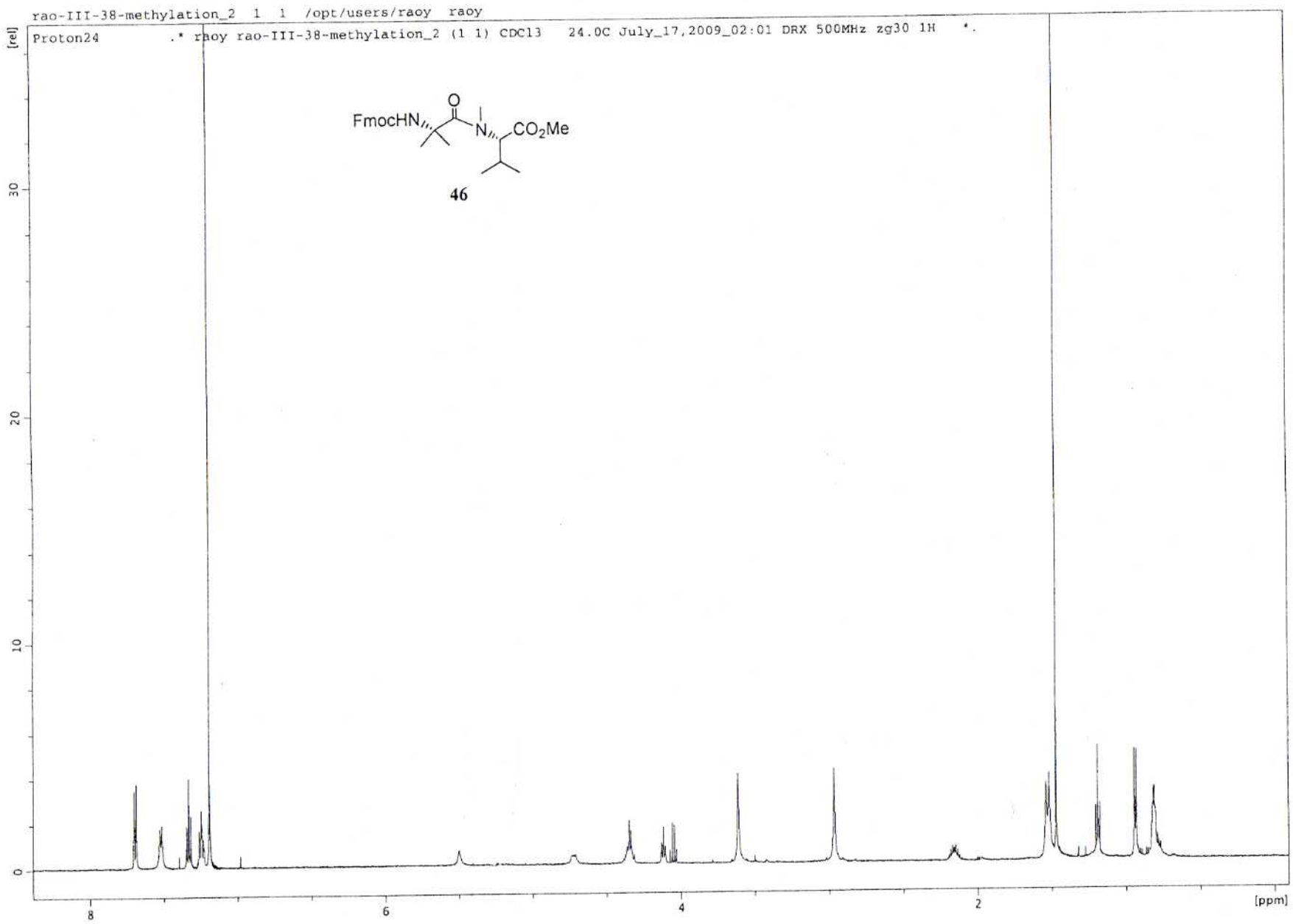

rao-III-20-Morpholine-c13 1 1 /opt/users/raoy raoy
rao1 .* raoy rao-III-20-Morpholine-c13 (1 1) CDCl3 24.0C June_11,2009_22:26 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.
[rel]
140
120
100
80
60
40
20
0
O
N
O
37
150
100
50
0
[ppm]


rao-III-29B-Ala 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-29B-Ala (1 1) CDCl3 24.0C June_20,2009_23:41 DRX 500MHz zg30 1H *.
38
FmocHN
[rel]
120
100
80
60
40
20
0
8
6
4
2
0 [ppm]

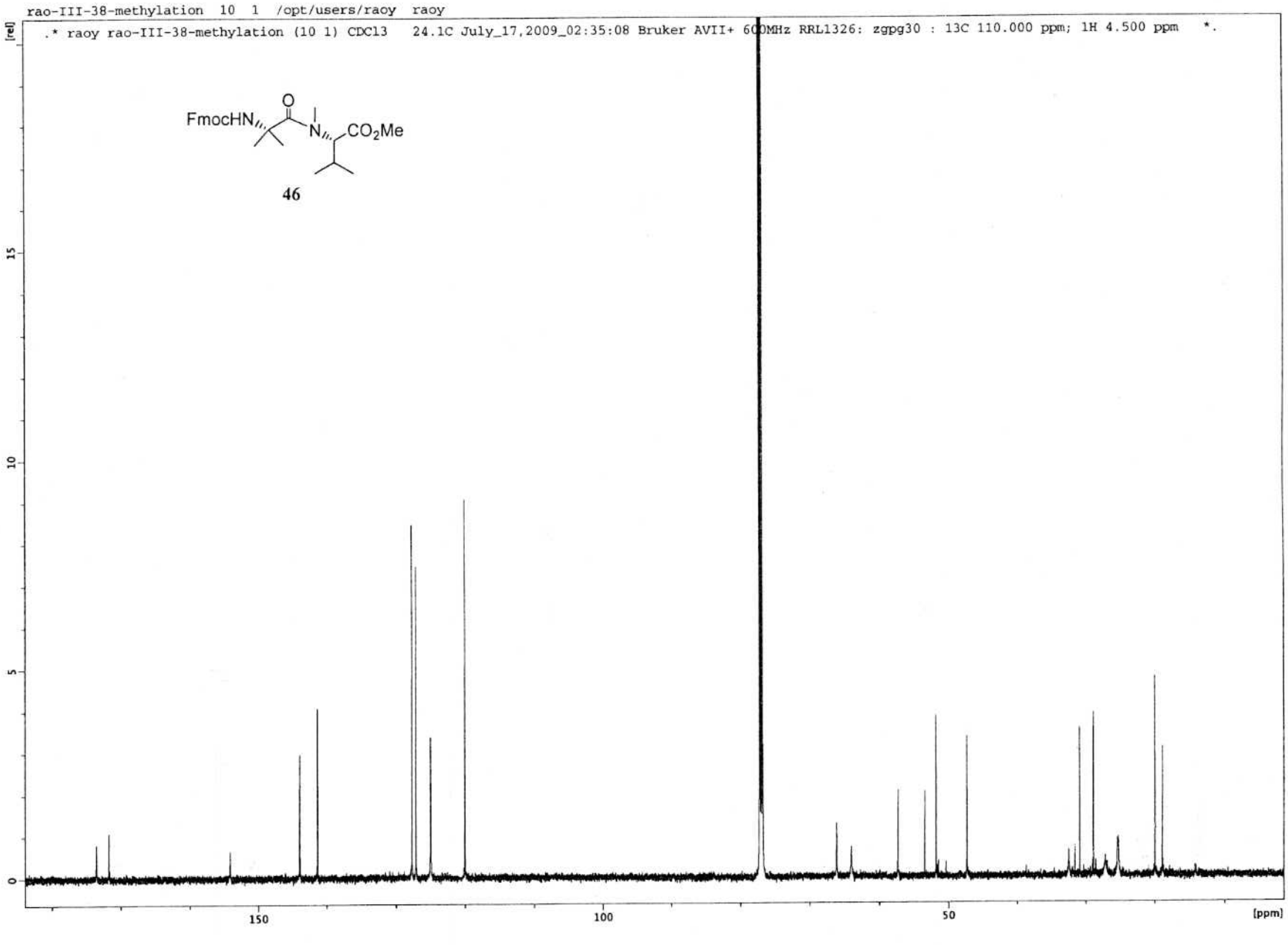

rao-III-29B-Ala 10 1 /opt/users/raoy raoy
.* raoy rao-III-29B-Ala (10 1) CDCl3 24.1C June_21,2009_08:34:10 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
FmocHN
O
O
38
200
150
100
50
0
200
150
100
50
[ppm]


rao-III-29B-Asp 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-29B-Asp (1 1) CDCl3 24.0C June_21,2009_01:05 DRX 500MHz zg30 1H *.
BocHN
COOBn
39
100 [rel]
80
60
40
20
0
8
6
4
2
[ppm]


rao-III-29B-Asp 10 1 /opt/users/raoy raoy
.* raoy rao-III-29B-Asp (10 1) CDCl3 24.1C June_21,2009_16:45:13 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
200
150
100
50
0
BocHN
COOBn
O
O
39
200
150
100
50
[ppm]


rao-III-38-methylation_2 1 1 /opt/users/raoy raoy
Proton24 .* raoy rao-III-38-methylation_2 (1 1) CDCl3 24.0C July_17,2009_02:01 DRX 500MHz zg30 1H *.
[rel]
30
20
10
0
8
6
4
2
[ppm]
FmocHN
O
N
CO2Me

46


rao-III-38-methylation 10 1 /opt/users/raoy raoy
.* raoy rao-III-38-methylation (10 1) CDCl3 24.1C July_17,2009_02:35:08 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.
[rel]
FmocHN
O
N
CO2Me
46
15
10
5
0
150
100
50
[ppm]