Electronic Supplementary Material (ESI) for Chemical Communications

A modular synthesis of dithiocarbamate pendant unnatural α- amino acids

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Supporting Information

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General Methods.

All the reactions were performed in oven dried apparatus and were stirred magnetically. Melting points and optical rotation values reported are uncorrected. Infrared spectra were recorded using an FTIR instrument, the frequencies are reported in wave numbers (cm^{-1}). ^{1}H and ^{13}C spectra were recorded at 300 MHz and 75 MHz NMR instruments, respectively. Chemical shifts are reported in parts per million downfield from the internal reference, tetramethylsilane (TMS). Multiplicity is indicated using the following abbreviations: s (singlet), d (doublet), dd (double doublet), t (triplet), m (multiplet), bs (broad singlet). elemental analysis has been performed using Perkin Elmer instrument. References for the compound reported previously are indicated against each of them along with the characterization data.

General Procedure for the Synthesis of *N*-Boc- and *N*-Cbz-, Sulfamidates.

Step I. A solution of $SOCl_2$ (1.2 equiv) in dry CH_3CN under nitrogen was cooled to -40 ^oC, and then Boc-Threo-OMe (1.0 equiv) in dry CH_3CN was added dropwise over 10 min and stirring continued for a further 45 min at the same temperature. Dry pyridine (4.0 equiv) was then added. The reaction mixture was further stirred for 1 h and then allowed to warm to room temperature. The reaction mixture was quenched with water and extracted with ethyl acetate. The combined organic extract was washed with water, dried over anhydrous sodium sulfate (Na_2SO_4), and concentrated in vacuum to afford the crude sulfamidite. This was used without further purification in the next step.

Step II. To a cooled (ice bath) solution of crude (step I) sulfamidite in MeCN was added ruthenium- (III) chloride (5 mol %) followed by $NaIO_4$ (1.2 equiv) and then water (CH_3CN:H_2O, 1:1). The mixture was stirred at ^oC for 1-3h and diluted with ether, and the phases were separated. The aqueous phase was extracted with ether. The combined organic portions were washed with $NaHCO_3$ solution and then brine. The solution was dried over anhydrous (Na_2SO_4) and concentrated. The crude product was purified by silica gel (100-200 mesh) column chromatography.

Spectral Data.

(4S,5R)-3-benzyl 4-methyl 5-methyl-1,2,3-oxathiazolidine-3,4-dicarboxylate 2,2-dioxide: Yield 87 % Gummy solid; $[\alpha]^{25}D$ -30.51 (*c* = 1, $CHCl_3$); FT-IR (cm^{-1}), 2983, 1739, 1440, 1384, 1326, 1192, 1074, 1018, 751; ^1H NMR (300 MHz, $CDCl_3$), δ 7.61 (5H, m), 5.37 (1H, d, *J* = 12 Hz), 5.35-5.25 (1H, m), 4.95-.4.87 (1H, m), 4.55 (1H, d, *J* = 6 Hz), 3.75 (3H, s), 1.70 (3H, d, *J* = 6Hz); ^{13}C NMR (75 MHz, $CDCl_3$), δ 166.5, 149.3, 134.1, 128.6, 128.5, 127.9, 77.8, 69.6, 63.7, 53.7, 18.8; Analysis calculated for $C_{13}H_{15}NO_7S$: C, 47.41; H, 4.59; N, 4.25; O, 34.01; S, 9.74; Found C, 47.39; H, 4.57; N, 4.27; O, 34.04; S, 9.72.

(S)-3-benzyl 4-methyl 1,2,3-oxathiazolidine-3,4-dicarboxylate 2,2-dioxide: Yield 84%; Gummy solid; FT-IR (cm^{-1}), 2981, 1736, 1442, 1377, 1259, 1187, 1150, 789, 784, 691, 657; ^1H NMR (400 MHz, $CDCl_3$), δ 7.42-7.27(5H, m), 5.40 (1H, d, *J* = 9 Hz), 5.32 (1H, d, *J* = 9 Hz), 4.89-4.73(3H, m), 3.82 (3H, s) ^{13}C NMR (100 MHz, $CDCl_3$), δ 167.1, 149.3, 134.2, 128.78, 128.71, 128.0, 69.8, 67.9, 57.7, 53.7; Analysis calculated for $C_{12}H_{13}NO_7S$: C, 45.71; H, 4.16; N, 4.44; O, 35.52; S, 10.17; Found C, 45.73; H, 4.15; N, 4.41; O, 35.54; S, 10.15.

(S)-tert-butyl 4-benzyl-1,2,3-oxathiazolidine-3-carboxylate 2,2-dioxide: Yield (87%) white solid; mp- 138^{o}C; $[\alpha]^{25}_{D}$ -41.2 (*c* = 0.7%, $CHCl_3$); FT-IR (cm^{-1}), 2973, 2927, 1712, 1458, 1320, 1261, 1184, 1150, 1023, 838, 799, 784, 695, 657, 540 ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.55 (9H, s), 2.92(1H, dd, *J* =9.9, 13.5), 3.36 (1H, dd, *J* = 3.3, 13.5), 4.28-4.34(1H, m), 4.40-4.48(2H, m), 7.21-7.37(5H, m); (75 MHz, $CDCl_3$) δ 27.9, 37.8, 58.5, 68.7, 85.5, 127.4, 129.0, 129.4, 135.1, 148.4. Analysis calculated for $C_{14}H_{19}NO_5S$: C 53.66, H 6.11, N 4.47; Found C 53.63, H 6.13, N 4.45.

(S)-tert-butyl 4-((S)-sec-butyl)-1,2,3-oxathiazolidine-3-carboxylate 2,2-dioxide:

Yield (91%) white solid; mp- 99^{o}C; $[\alpha]^{25}_{D}$ 2.16 (*c* = 0.5%, $CHCl_3$); FT-IR (cm^{-1}), 2977, 2934, 1728, 1466, 1367, 1325, 1261, 1187, 1150, 1099, 968, 927, 826, 805, 653, 571, cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 0.96-1.04 (6H, m), 1.15-1.44(2H, m), 1.57 (9H, s), 2.03-2.12(1H, m), 4.28-4.33(1H, m), 4.39 (1H, dd, *J* = 2.4, 9.6), 4.57 (1H, dd, *J* = 9.3, 6.6); (75 MHz, $CDCl_3$) δ 11.7, 13.0, 25.3, 27.9, 36.1, 60.8, 66.3, 85.3, 149.0. Analysis calculated for $C_{11}H_{21}NO_5S$: C 47.29, H 7.58, N 5.01; Found C 47.27, H 7.55, N 5.05.

General Experimental Procedure for the Synthesis fo Dithiocarbamate Side Chain Bearing Unnatural α-Amino Acids.

Carbon disulfide (1.5 mmol) was added drop wise to a DMF (1 mL) solution of amine (1 mmol) at 0-5 ^{o}C. The mixture was stirred for 2-3 min. Sulfamidate (1 mmol) solution in DMF (1 mL) was added to the solution of dithiocarbamate at room temperature and was stirred for the required time period (TLC). The crude reaction mixture was extracted with ethyl acetate and was washed with water (three times). The organic layer was concentrated and was purified by column chromatography to obtain desired dithiocarbamate α-amino acid. The product was characterized by FT-IR, ^{1}H and ^{13}C NMR spectroscopy, elemental analysis and polarimetry.

Spectral Data.

(2R,3S)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((piperidine-1-carbonothioyl)thio)butanoate, Table 2, Entry 1: Colorless viscous liquid, $[\alpha]^{23}_{D}$= -18.2 (*c* = 0.28, $CHCl_3$). FT-IR (cm^{-1}): 603, 667, 701, 755, 892, 975, 1005, 1031, 1077, 1205, 1227, 1241, 1270, 1307, 1357, 1386, 1434, 1468, 1508, 1718,

1744, 2944, 3362, 3431. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.38 (d, J = 7.2 Hz, 3H), 1.67 (bs, 6H), 3.78-3.84 (m, 5H), 4.25 (bs, 2H), 4.66-4.74 (m, 1H), 4.92 (dd, J_1 = 9Hz, J_2 = 3.6 Hz, 1H), 5.06-5.16 (m, 2H), 5.62 (d, J = 8.1 Hz, 1H), 7.28-7.32 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.62, 24.26, 25.76 (2C), 48.84, 51.60, 52.58, 52.99, 57.48, 67.05, 128.01, 128.08 (2C), 128.46 (2C), 136.30, 155.99, 170.89, 193.44. Analysis calculated for $C_{19}H_{26}N_2O_4S_2$: C, 55.58; H, 6.38; N, 6.82. Found: C, 55.55; H, 6.37; N, 6.80.

(2R,3S)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((pyrrolidine-1-carbonothioyl)thio)butanoate, Table 2, Entry 2: Colorless viscous liquid, $[\alpha]^{23}_D$ = -18.9 (c = 0.42, $CHCl_3$). FT-IR (cm^{-1}): 579, 608, 670, 699, 749, 775, 869, 915, 955, 1004, 1034, 1078, 1090, 1159, 1207, 1269, 1304, 1349, 1384, 1438, 1509, 1718, 1744, 2953, 3363, 3425. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.39 (d, J =7.5 Hz, 3H), 1.93-2.06 (m, 4H), 3.58 (t, J = 6.3 Hz, 2H), 3.78 (s, 3H), 3.90 (t, J = 6.6 Hz, 2H), 4.66-4.74 (m, 1H), 4.88 (dd, J_1 = 9 Hz, J_2 = 3.6 Hz, 1H), 5.07-5.16 (m, 2H), 5.65 (d, J = 7.8 Hz, 1H), 7.28-7.32 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.92, 24.16, 25.95, 48.16, 50.72, 52.59, 55.07, 57.63, 67.08, 128.05, 128.09 (2C), 128.46 (2C), 136.27, 155.99, 170.81, 190.72. Analysis calculated for $C_{18}H_{24}N_2O_4S_2$: C, 54.52; H, 6.10; N, 7.06. Found: C, 54.53; H, 6.08; N, 7.05.

(2R,3S)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((diisopropylcarbamothioyl)thio)butanoate, Table 2, Entry 3: Pale yellow viscous liquid, $[\alpha]^{23}_D$ = -11.8 (c = 2.00, $CHCl_3$). FT-IR (cm^{-1}): 583, 697, 734, 772, 851, 913, 960, 1026, 1085, 1142, 1193, 1212, 1313, 1370, 1439, 1476, 1508, 1720, 2968, 3348. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.37 (d, J = 7.2 Hz, 17H), 3.78 (s, 3H), 4.79 (bs, 1H), 4.94 (dd, J_1 = 9Hz, J_2 = 3.6 Hz, 1H), 5.06-5.17 (m, 2H), 5.60 (d, J = 8.7 Hz, 1H), 7.30-7.35 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.18, 19.97 (4C), 47.44, 47.71, 52.54, 54.13, 57.36, 66.99, 128.00, 128.08 (2C), 128.47 (2C), 136.33, 156.00, 171.06, 198.06. Analysis calculated for $C_{20}H_{30}N_2O_4S_2$: C, 56.31; H, 7.09; N, 6.57. Found: C, 56.28; H, 7.08; N, 6.57.

(2R,3S)-methyl 3-((benzylcarbamothioyl)thio)-2-(((benzyloxy)carbonyl)amino)butanoate, Table 2, Entry 4: Colorless viscous liquid, $[\alpha]^{23}_{D}$ = -10.3 (*c* = 2.80, $CHCl_3$). FT-IR (cm^{-1}): 632, 666, 696, 751, 795, 932, 1028, 1086, 1215, 1339, 1379, 1437, 1454, 1494, 1509, 1707, 3033, 3276. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.37 (d, *J* = 7.2 Hz, 3H), 3.76 (s, 3H), 4.59-4.67 (m, 2H), 4.85-4.98 (m, 2H), 5.09 (s, 2H), 5.66 (bs, 1H), 7.24-7.38 (m, 10H), 7.64 (bs, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.3, 47.76, 51.16, 52.72, 57.63, 67.28, 127.58 (2C), 127.99, 128.20, 128.29 (2C), 128.52 (2C), 128.82 (2C), 136.11, 137.24, 156.15, 170.70, 196.01. Analysis calculated for $C_{21}H_{24}N_2O_4S_2$: C, 58.31; H, 5.59; N, 6.48. Found: C, 58.30; H, 5.60; N, 6.45.

(2R,3S)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((((S)-1-phenylethyl)carbamothioyl)thio)butanoate, Table 2, Entry 5: Pale yellow viscous liquid, $[\alpha]^{23}_{D}$ = -55.8 (*c* = 2.42, $CHCl_3$). FT-IR (cm^{-1}): 542, 600, 631, 697, 754, 845, 885, 913, 973, 1028, 1086, 1137, 1212, 1342, 1453, 1497, 1509, 1702, 2975, 3257. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.36 (d, *J* = 7.2 Hz, 3H), 1.60 (d, *J* = 6.6 Hz, 3H), 3.78 (s, 3H), 4.60-4.66 (m, 1H), 4.89 (dd, J_1 = 8.7 Hz, J_2 = 3 Hz, 1H), 5.13 (s, 2H), 5.67-5.78 (m, 2H), 7.29-7.38 (m, 10 H), 7.57 (bs, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.69, 20.73, 47.64, 52.70, 56.07, 57.74, 67.27, 126.53 (2C), 127.72, 128.09, 128.19 (2C), 128.52 (2C), 128.75 (2C), 136.17, 141.61, 156.12, 170.73, 194.72. Analysis calculated for $C_{22}H_{26}N_2O_4S_2$: C, 59.17; H, 5.87; N, 6.27. Found: C, 59.15; H, 5.85; N, 6.28.

(R)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((piperidine-1-carbonothioyl)thio)propanoate, Table 2, Entry 6: Colorless viscous liquid, $[\alpha]^{23}_{D}$ = +11.7 (*c* = 0.48, $CHCl_3$). FT-IR (cm^{-1}): 614, 641, 718, 850, 895, 922, 973, 1019, 1052, 1130, 1151, 1175, 1221, 1274, 1432, 1524, 1682, 1735, 2947, 3336. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.69 (bs, 6H), 3.77 (s, 3H), 3.87-3.94 (m, 4H), 4.26 (bs, 2H), 4.63-4.70 (m, 1H), 5.12-5.13 (m, 2H), 5.87 (d, *J* = 7.2 Hz, 1H), 7.28-7.36 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 24.22, 25.46, 25.92, 38.30, 51.64, 52.67, 53.67, 54.00, 66.97, 128.02, 128.08 (2C), 128.46 (2C), 136.33, 155.93, 170.97, 194.25. Analysis calculated for $C_{18}H_{24}N_2O_4S_2$: C, 54.52; H, 6.10; N, 7.06. Found: C, 54.51; H, 6.07; N, 7.03.

(R)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((pyrrolidine-1-carbonothioyl)thio)propanoate, Table 2, Entry 7: Colorless viscous liquid, $[\alpha]^{23}_{D}$= +18.7 (*c* = 0.50, $CHCl_3$). FT-IR (cm^{-1}): 545, 580, 623, 644, 699, 758, 742, 781, 799, 826, 865, 895, 919, 951, 975, 1022, 1055, 1157, 1178, 1227, 1242, 1277, 1319, 1343, 1388, 1434, 1456, 1528, 1681, 1737, 2946, 3329. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.93-2.13 (m, 4H), 3.65 (t, *J* = 6.9 Hz, 2H), 3.78 (s, 3H), 3.90-3.94 (m, 4H), 4.63-4.70 (m, 1H), 5.08-5.17 (m, 2H), 5.91 (d, *J* = 7.2 Hz, 1H), 7.31-7.38 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 24.21, 26.00, 37.58, 50.77, 52.67, 54.15, 55.49, 66.96, 128.02, 128.06 (2C), 128.45 (2C), 136.36, 155.97, 170.93, 191.48. Analysis calculated for $C_{17}H_{22}N_2O_4S_2$: C, 53.38; H, 5.80; N, 7.32. Found: C, 53.40; H, 5.79; N, 7.33.

(R)-methyl 2-(((benzyloxy)carbonyl)amino)-3-((diethylcarbamothioyl)thio)propanoate, Table 2, Entry 8: Colorless viscous liquid, $[\alpha]^{23}_{D}$ = +24.4 (*c* = 0.52, $CHCl_3$). FT-IR (cm^{-1}): 557, 577, 666, 697, 752, 832, 916, 982, 1006, 1028, 1055, 1144, 1205, 1269, 1301, 1343, 1380, 1420, 1438, 1455, 1490, 1514, 1719, 2934, 3340. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.24-1.30 (m, 6H), 3.70-3.78 (m, 5H), 3.92 (d, J = 6.3 Hz, 2H), 4.02 (q, J = 6.9 Hz, 2H), 4.63-4.70 (m, 1H), 5.12-5.13 (m, 2H), 5.85 (d, J = 7.2 Hz, 1H), 7.35-7.37 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 11.48, 12.54, 38.25, 46.99, 50.19, 52.66, 54.05, 66.94, 127.99, 128.06 (2C), 128.45 (2C), 136.35, 155.93, 170.95, 194.35. Analysis calculated for C17H24N2O4S2: C, 53.10; H, 6.29; N, 7.29. Found: C, 53.07; H, 6.29; N, 7.30.

(S)-2-((tert-butoxycarbonyl)amino)-3-phenylpropyl pyrrolidine-1-carbodithioate, Table 2, Entry 9: Pale yellow solid, Mp. 157-159 ^{o}C, $[\alpha]^{23}_{D}$= +2.8 (*c* = 0.34, $CHCl_3$). FT-IR (cm^{-1}): 657, 701, 745, 833, 857, 955, 1008, 1028, 1044, 1080, 1167, 1249, 1270, 1331, 1367, 1384, 1438, 1523, 1686, 2979, 3356. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.41 (s, 9H), 1.93-2.13 (m, 4H), 2.84-2.91 (m, 1H), 3.02 (bs, 1H), 3.55 (d, *J* = 6H, 2H), 3.68 (t, *J* = 6.6 Hz, 2H), 3.94 (t, *J* = 6.9 Hz, 2H), 4.01-4.11 (m, 1H), 5.04 (bs, 1H), 7.22-7.32 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 24.27, 26.02, 28.38 (3C), 39.89, 40.89, 50.73, 52.42, 55.31, 79.11, 126.47, 128.44 (2C), 129.47 (2C), 137.75, 155.45, 192.78. Analysis calculated for $C_{19}H_{28}N_2O_2S_2$: C, 59.96; H, 7.42; N, 7.36. Found: C, 59.97; H, 7.40; N, 7.35.

tert-butyl ((2S,3S)-1-((diethylcarbamothioyl)thio)-3-methylpentan-2-yl)carbamate, Table 2, Entry 10: Pale yellow solid, Mp. 104-105 ^{o}C, $[\alpha]^{23}_{D}$= -56.8 (*c* = 0.48, $CHCl_3$). FT-IR (cm^{-1}): 648, 744, 775, 835, 869, 918, 982, 1008, 1043, 1071, 1143, 1172, 1208, 1249, 1267, 1301, 1352, 1366, 1388, 1458, 1489, 1523, 1686, 2932, 2965, 3361. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 0.92-1.00 (m, 6H), 1.06-1.24 (m, 1H), 1.30 (t, *J* =7.2 Hz, 6H), 1.42 (s, 9H), 1.51-1.68 (m, 2H), 3.41-3.45 (m, 1H), 3.64-3.82 (m, 4H), 3.93-3.97 (m, 1H), 4.13-4.14 (m, 1H), 4.98 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 11.58, 11.70, 12.48, 15.13, 25.37, 28.37 (3C), 38.98, 39.44, 46.82, 49.91, 55.10, 78.67, 155.92, 196.38. Analysis calculated for $C_{16}H_{32}N_2O_2S_2$: C, 55.13; H, 9.25; N, 8.04. Found: C, 55.10; H, 9.24; N, 8.05.

(S)-methyl 1-((((2S,3R)-3-(((benzyloxy)carbonyl)amino)-4-methoxy-4-oxobutan-2-yl)thio)carbonothioyl)pyrrolidine-2-carboxylate, Table 3, Entry 1: Colorless viscous liquid, $[\alpha]^{23}_{D}$= -77.4 (*c* = 2.18, $CHCl_3$). FT-IR (cm^{-1}): 561, 698, 751, 843, 888, 913, 957, 1013, 1042, 1086, 1170, 1205, 1341, 1413, 1516, 1735, 2954, 3360. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.38 (d, *J* = 7.5 Hz, 3H), 2.03-2.28 (m, 4H), 3.71-3.77 (m, 8H), 4.59-4.63 (m, 1H), 4.91 (dd, J_1 = 8.7 Hz, J_2 = 3.3 Hz, 1H), 5.04-5.06 (m, 1H), 5.10-5.11 (m, 2H), 5.62 (d, *J* = 6 Hz, 1H), 7.30-7.36 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.38, 24.46, 29.03, 48.49, 50.95, 52.34, 52.62, 57.42, 66.20, 67.09, 127.98, 128.11 (2C), 128.47 (2C), 136.26, 156.03, 170.75, 171.02, 193.35. Analysis calculated for $C_{20}H_{26}N_2O_6S_2$: C, 52.85; H, 5.77; N, 6.16. Found: C, 52.82; H, 5.78; N, 6.18.

(2S,4R)-methyl 1-((((2S,3R)-3-(((benzyloxy)carbonyl)amino)-4-methoxy-4-oxobutan-2-yl)thio)carbonothioyl)-4-hydroxypyrrolidine-2-carboxylate, Table 3, Entry 2: Colorless viscous liquid, $[\alpha]^{23}_{D}$ = -32.2 (*c* =1.68, $CHCl_3$). FT-IR (cm^{-1}): 577, 667, 699, 752, 848, 897, 918, 975, 1014, 1040, 1081, 1176, 1210, 1336, 1414, 1522, 1735, 2954, 3349. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.31 (d, *J* = 6 Hz, 3H), 2.04-2.14 (m, 1H), 2.39-2.45 (m, 1H), 3.36 (bs, 1H), 3.74 (s, 3H), 3.79 (s, 3H), 3.91-3.95 (m, 1H), 4.46 (bs, 1H), 4.52-4.58 (m, 1H), 4.99 (dd, J_1 = 9 Hz, J_2 = 3 Hz, 1H), 5.08-5.13 (m, 3H), 5.71 (d, *J* =

6 Hz, 1H), 7.31-7.37 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 14.54, 30.37, 48.40, 52.44, 52.81, 56.52, 59.18, 64.46, 67.28, 69.70, 127.83 (2C), 128.19, 128.55 (2C), 136.10, 156.55, 170.71, 171.20, 194.15. Analysis calculated for $C_{20}H_{26}N_2O_7S_2$: C, 51.05; H, 5.57; N, 5.95. Found: C, 51.03; H, 5.58; N, 5.93.

(S)-1-((((2S,3R)-3-(((benzyloxy)carbonyl)amino)-4-methoxy-4-oxobutan-2-yl)thio)carbonothioyl)pyrrolidine-2-carboxylic acid, Table 3, Entry 3: Colorless viscous liquid, $[\alpha]^{23}_D$ = -69.7 (*c* =1.68, $CHCl_3$). FT-IR (cm^{-1}): 667, 697, 751, 795, 829, 920, 959, 1013, 1039, 1086, 1183, 1215, 1342, 1412, 1522, 1717, 2954, 3329. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.38 (d, J = 7.2 Hz, 3H), 2.02-2.33 (m, 4H), 3.65-3.79 (m, 5H), 4.59-4.63 (m, 1H), 4.94 (dd, J_1 = 8.7 Hz, J_2 = 3 Hz, 1H), 5.07-5.16 (m, 3H), 5.66 (d, J = 8.7 Hz, 1H), 7.31-7.35 (m, 5H), 8.84 (bs, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 16.28, 24.46, 28.94, 48.54, 50.96, 52.72, 57.36, 66.05, 67.22, 128.05, 128.15 (2C), 128.49 (2C), 136.20, 156.18, 170.84, 174.94, 193.73. Analysis calculated for $C_{19}H_{24}N_2O_6S_2$: C, 51.80; H, 5.49; N, 6.36. Found: C, 51.78; H, 5.48; N, 6.37.

(5R,6S,10R)-methyl 9-benzyl-10-((S)-1-hydroxyethyl)-5-(methoxycarbonyl)-6-methyl-3-oxo-1-phenyl-8-thioxo-2-oxa-7-thia-4,9-diazaundecan-11-oate, Table 3, Entry 4: Pale yellow viscous liquid, $[\alpha]^{23}_D$ = +11.4 (*c* =1.62, $CHCl_3$). FT-IR (cm^{-1}): 567, 698, 753, 771, 915, 1002, 1085, 1168, 1214, 1380, 1437, 1455, 1498, 1516, 1719, 2956, 3353. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.32-1.41 (m, 6H), 3.54 (bs, 2H), 3.80 (s, 6H), 4.67-4.71 (m, 1H), 5.07-5.18 (m, 3H), 5.27-5.45 (m, 2H), 5.61 (bs, 1H), 7.28-7.42 (m, 10H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 15.87, 23.11, 49.46, 52.62, 52.74, 54.15, 56.99, 57.40, 67.32, 70.34, 128.05 (2C), 128.16 (2C), 128.29 (2C), 128.49, 128.55 (2C), 128.59, 130.16, 136.00, 156.65, 169.51, 170.94, 198.10. Analysis calculated for $C_{26}H_{32}N_2O_7S_2$: C, 56.92; H, 5.88; N, 5.11. Found: C, 56. 90; H, 5.85; N, 5.12.

(S)-methyl 1-((((R)-2-(((benzyloxy)carbonyl)amino)-3-methoxy-3-oxopropyl)thio)carbonothioyl)pyrrolidine-2-carboxylate, Table 3, Entry 5: Colorless viscous liquid, $[\alpha]^{23}_{D}$ = -56.9 (*c* = 0.58, $CHCl_3$). FT-IR (cm^{-1}): 577, 667, 699, 752, 841, 890, 914, 958, 1013, 1046, 1087, 1170, 1209, 1266, 1341, 1415, 1515, 1720, 1736, 2957, 3346. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 2.05-2.28 (m, 4H), 3.70-3.77 (m, 7H), 3.82-4.00 (m, 3H), 4.66-4.72 (m, 1H), 5.03-5.05 (m, 1H), 5.13 (s, 2H), 5.77 (d, J = 7.6 Hz, 1H), 7.33-7.37 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 24.52, 29.12, 38.06, 51.03, 52.35, 52.72, 53.80, 66.68, 67.07, 128.05, 128.10 (2C), 128.46 (2C), 136.29, 156.32, 170.77, 170.79, 194.00. Analysis calculated for $C_{19}H_{24}N_2O_6S_2$: C, 51.80; H, 5.49; N, 6.36. Found: C, 51.82; H, 5.48; N, 6.34.

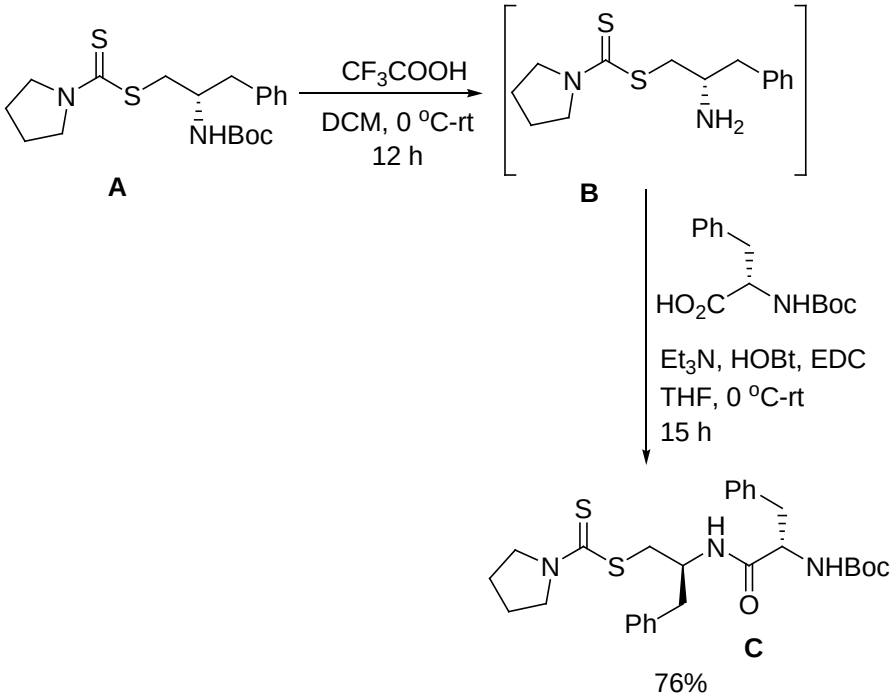
Boc-deprotection of the compound, A (entry 9, Table 2):

Compound, A (30 mg, 0.08 mmol) was dissolved in DCM (1.5 mL). Trifluoroacetic acid (TFA) (45 mg, 0.4 mmol) was added to the solution at 0 ^oC followed by stirring at room temperature for 12 h. After completion of the reaction (monitored by TLC), DCM and excess of TFA were removed under vacuum.

The crude product, B obtained was directly used in the next step of peptide coupling reaction without any purification.

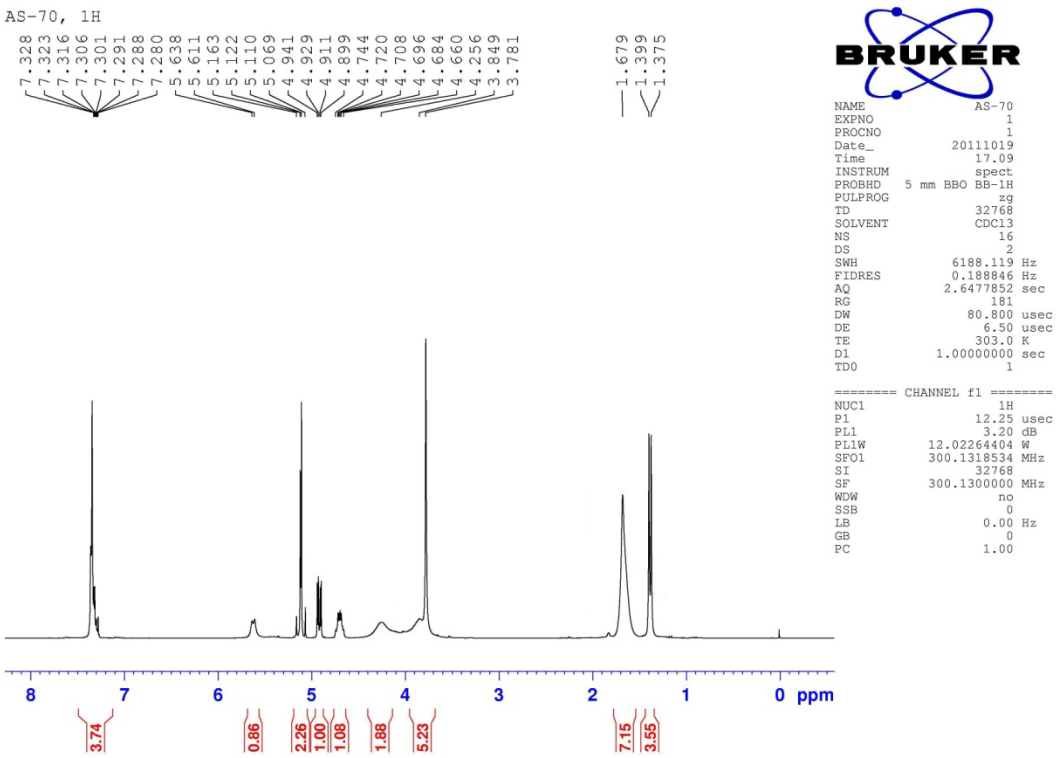
Peptide coupling of B with Boc-protected (*S*)-phenylalanine(*N*-Boc(*S*)-Phe):

Compound, B obtained from the previous step and *N*-Boc(*S*)-Phe (24 mg, 0.09 mmol) were dissolved in THF (2 mL). Triethylamine (32 mg, 0.32 mmol) was added to the solution. The reaction mixture was cooled to 0 ^{o}C. *N*-Hydroxybenzotriazole (HOBt) (16 mg, 0.12 mmol) and 3-(ethyliminomethyleneamino)-*N*,*N*-dimethyl-propan-1-amine (EDC) (18 mg, 0.12 mmol) were added drop wise to the solution followed by stirring for 1 h at 0 ^{o}C. The reaction mixture was continued to stir for 14 h at room temperature. After completion of the reaction (monitored by TLC), THF was removed under vacuum. The product was extracted using ethyl acetate and was washed with water. The organic layer was dried over anhydrous Na_2SO_4. The crude product was purified using column chromatography.



Scheme 1 Synthesis of dithiocarbamate bearing peptide

(S)-2-((S)-2-((tert-butoxycarbonyl)amino)-3-phenylpropanamido)-3-phenylpropyl pyrrolidine-1-carbodithioate: White solid, Mp. 164-165 ^{o}C, $[\alpha]^{23}_{D}$ = -37.7 (*c* = 0.38, $CHCl_3$). FT-IR (cm^{-1}): 657, 701, 857, 833, 870, 955, 1008, 1044, 1154, 1167, 1249, 1270, 1438, 1523, 1687, 2930, 2979, 3349, 3356. ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 1.40 (s, 9H), 1.82-2.13 (m, 4H), 2.75-2.83 (m, 1H), 2.92-2.94 (m, 1H), 3.00-3.16 (m, 2H), 3.35 (dd, J1 = 3.9 Hz, J2 = 14.4 Hz, 1H), 3.60-3.74 (m, 3H), 3.90 (t, J = 6.3 Hz, 2H), 4.22-4.35 (m, 2H), 5.01 (bs, 1H), 7.01 (d, J = 6.6 Hz, 1H), 7.16-7.34 (m, 10H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 24.18, 25.95, 28.29, 38.16, 38.76, 40.80, 50.87, 52.65, 55.58, 79.70, 126.65, 126.67, 128.40, 128.55, 129.46, 129.48, 136.71, 137.38, 155.16, 171.07, 193.04. Analysis calculated for $C_{28}H_{37}N_3O_3S_2$: C, 63.72; H, 7.07; N, 7.96. Found: C, 63.74; H, 7.05; N, 7.93.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7.316
7.306
7.301
7.291
7.288
7.280
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5.611
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5.122
5.110
5.069
4.941
4.929
4.911
4.899
4.744
4.720
4.708
4.696
4.684
4.660
4.256
3.849
3.781
1.679
1.399
1.375
8
7
6
5
4
3
2
1
0 ppm
3.74
0.86
2.26
1.00
1.08
1.88
5.23
7.15
3.55
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EXPNO 1
PROCNO 1
Date_ 20111019
Time 17.09
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PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
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DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
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PL1 3.20 dB
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WDW no
SSB 0
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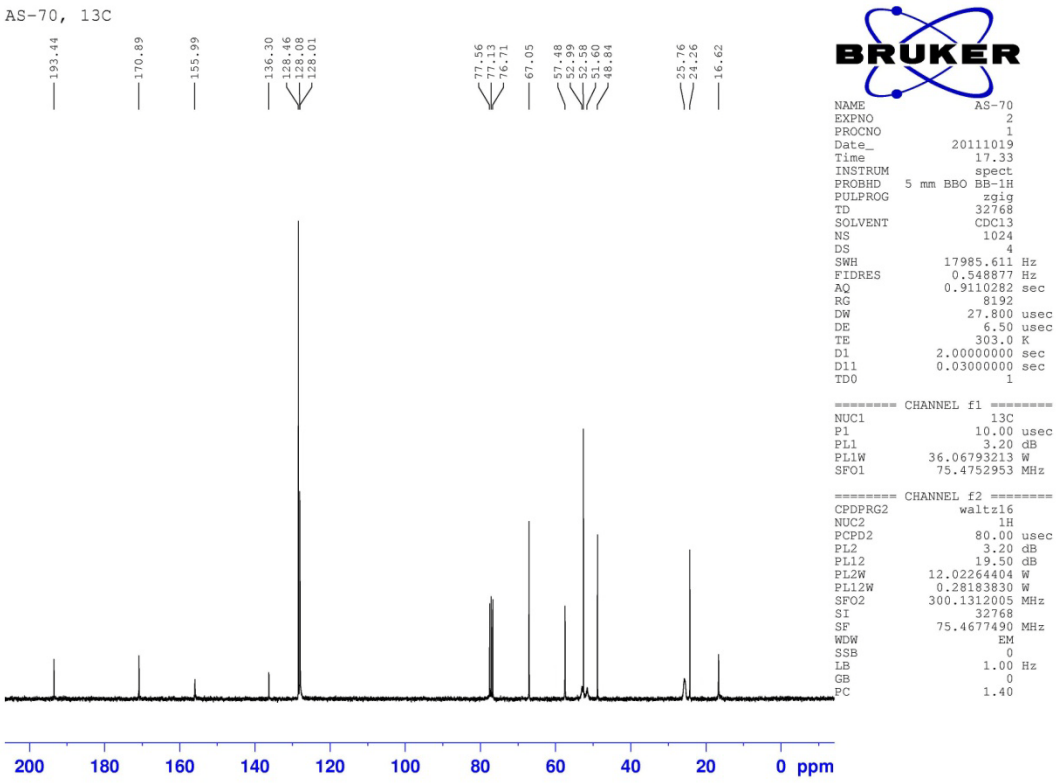
GB 0
PC 1.00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155.99
136.30
128.46
128.08
128.01
77.56
77.13
76.71
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57.48
52.99
52.58
51.60
48.84
25.76
24.26
16.62
200
180
160
140
120
100
80
60
40
20
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BRUKER
NAME AS-70
EXPNO 2
PROCNO 1
Date_ 20111019
Time 17.33
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PULPROG zgig
TD 32768
SOLVENT CDCl3
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FIDRES 0.548877 Hz
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RG 8192
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DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
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PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
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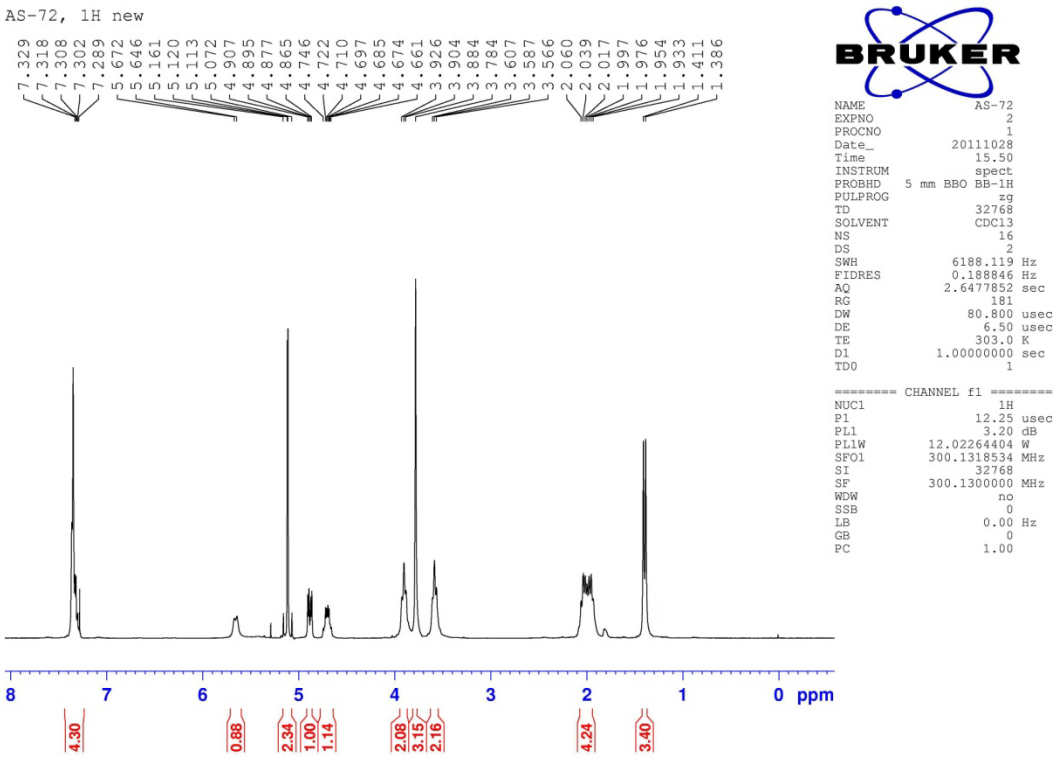
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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7.308
7.302
7.289
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5.646
5.161
5.120
5.113
5.072
4.907
4.895
4.877
4.865
4.746
4.722
4.710
4.697
4.685
4.674
4.661
3.926
3.904
3.884
3.784
3.607
3.587
3.566
2.060
2.039
2.017
1.997
1.976
1.954
1.933
1.411
1.386
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NAME AS-72
EXPNO 2
PROCNO 1
Date_ 20111028
Time 15.50
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PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
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SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
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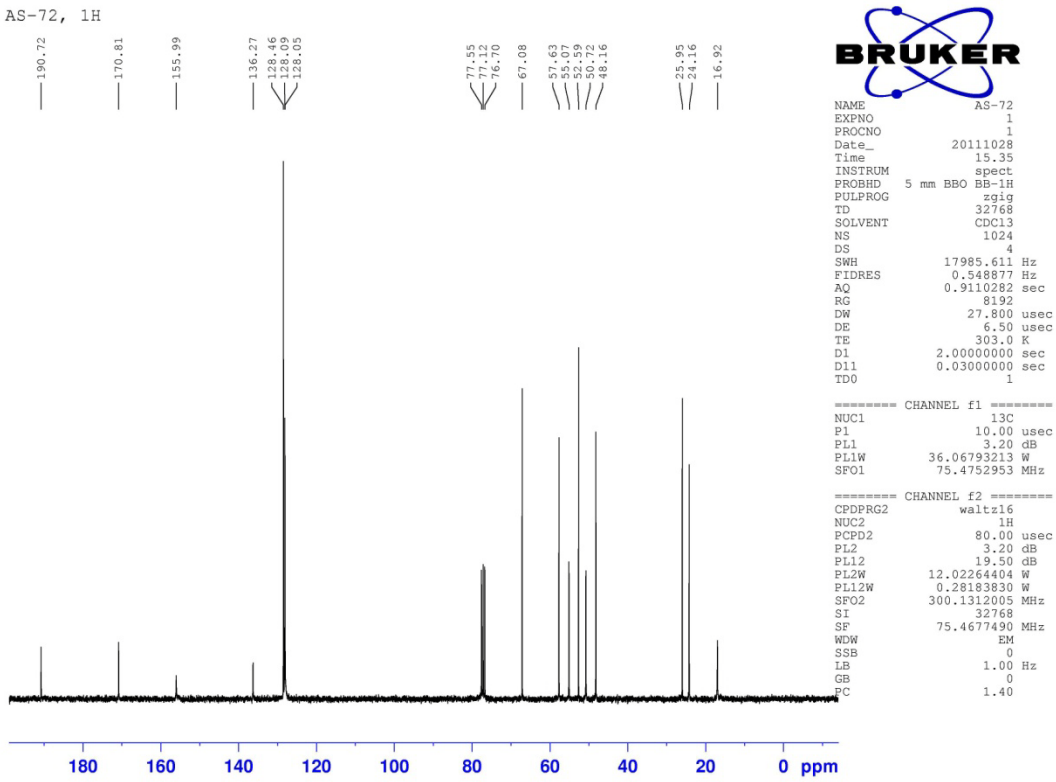
4.30
0.88
2.34
1.00
1.14
2.08
3.15
2.16
4.24
3.40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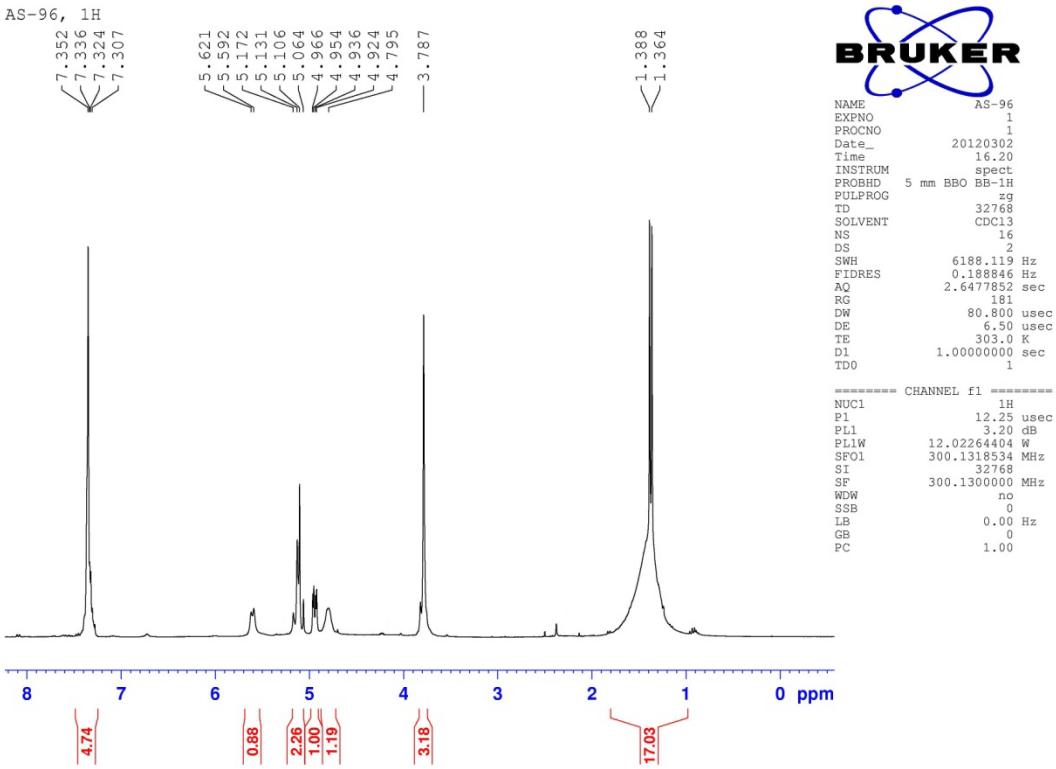
155.99
136.27
128.46
128.09
128.05
77.55
77.12
76.70
67.08
57.63
55.07
52.59
50.72
48.16
25.95
24.16
16.92
180
160
140
120
100
80
60
40
20
0 ppm
NAME AS-72
EXPNO 1
PROCNO 1
Date_ 20111028
Time 15.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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7.336
7.324
7.307
5.621
5.592
5.172
5.131
5.106
5.064
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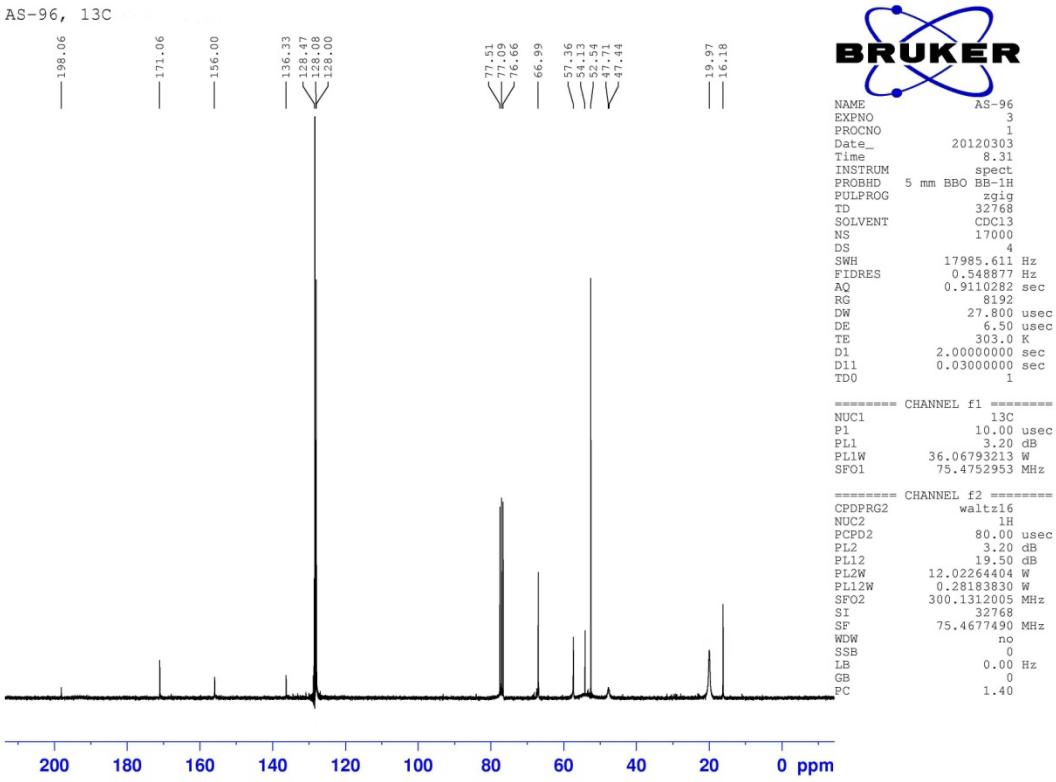
4.954
4.936
4.924
4.795
3.787
1.388
1.364
8
7
6
5
4
3
2
1
0 ppm
4.74
0.88
2.26
1.00
1.19
3.18
17.03
NAME AS-96
EXPNO 1
PROCNO 1
Date_ 20120302
Time 16.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

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PULPROG	zgig
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SOLVENT	CDCl3
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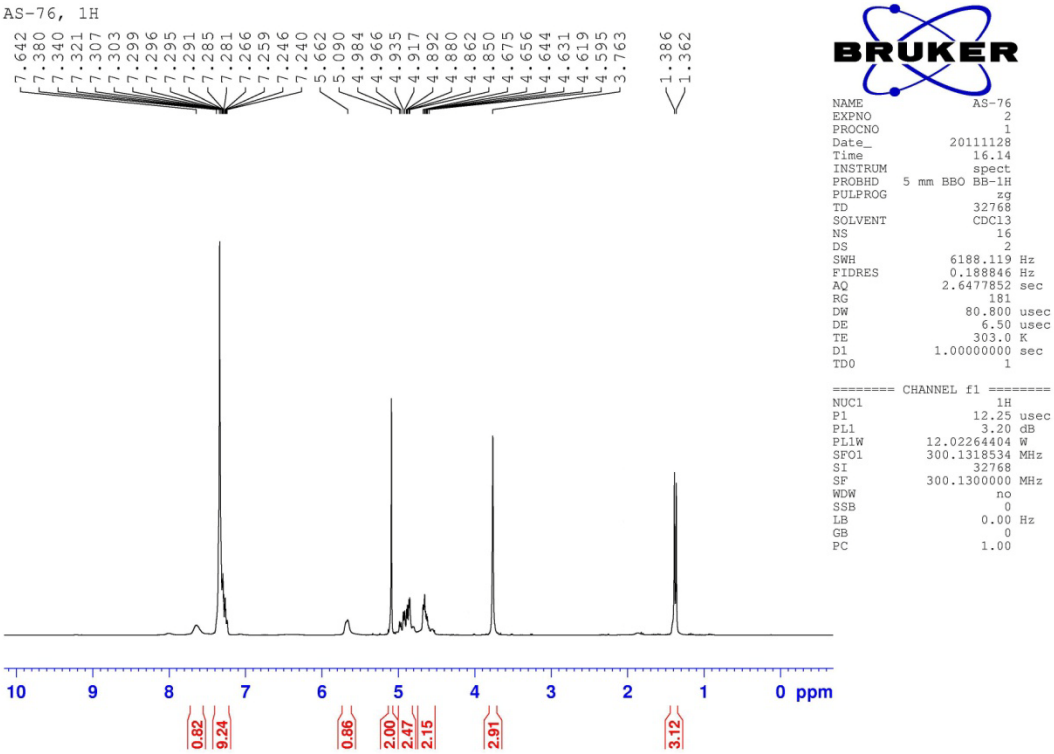
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40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7.340
7.321
7.307
7.303
7.299
7.296
7.295
7.291
7.285
7.281
7.266
7.259
7.246
7.240
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5.090
4.984
4.966
4.935
4.917
4.892
4.880
4.862
4.850
4.675
4.656
4.644
4.631
4.619
4.595
3.763
1.386
1.362
BRUKER
NAME AS-76
EXPNO 2
PROCNO 1
Date_ 20111128
Time 16.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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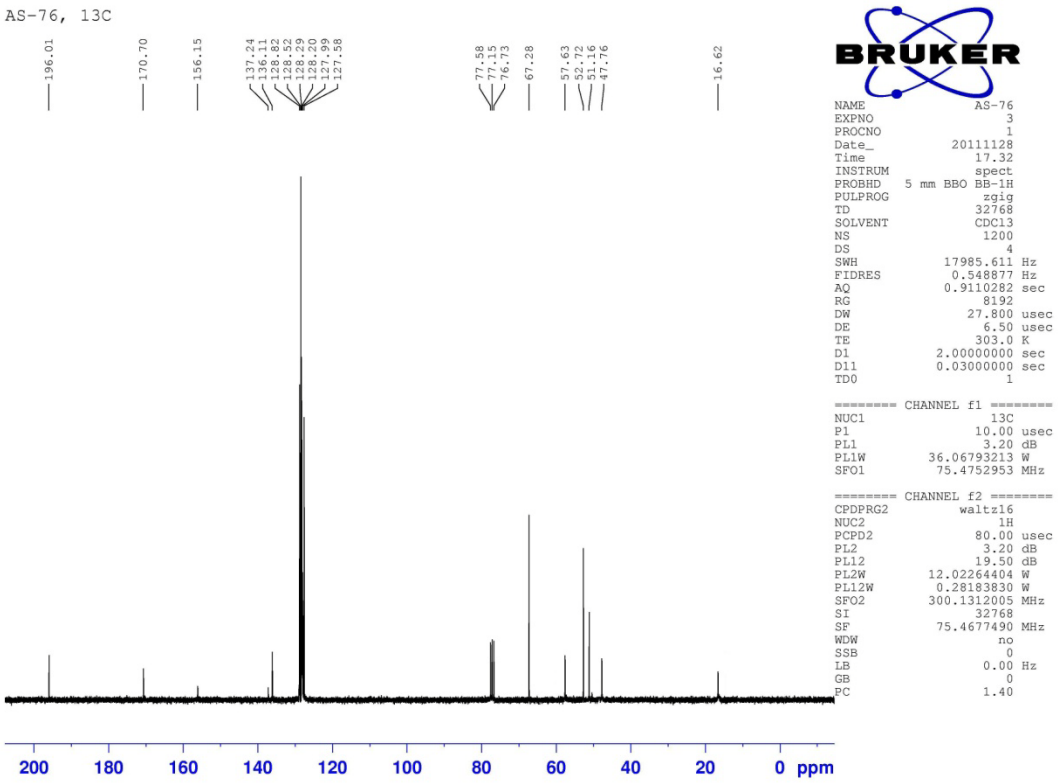
9.24
0.86
2.00
2.47
2.15
2.91
3.12


AS-76, 13C
196.01
170.70
156.15
137.24
136.11
128.82
128.52
128.29
128.20
127.99
127.58
77.58
77.15
76.73
67.28
57.63
52.72
51.16
47.76
16.62
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME AS-76
EXPNO 3
PROCNO 1
Date_ 20111128
Time 17.32
INSTRUM spect
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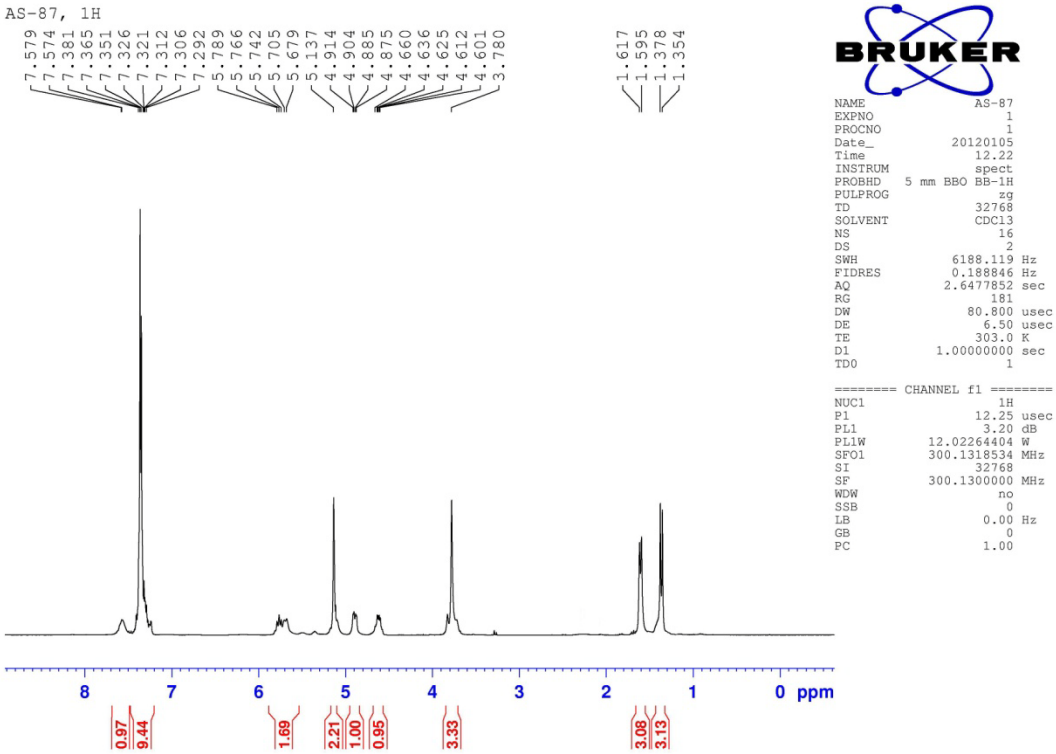
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 1200
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40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7.574
7.381
7.365
7.351
7.326
7.321
7.312
7.306
7.292
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5.766
5.742
5.705
5.679
5.137
4.914
4.904
4.885
4.875
4.660
4.636
4.625
4.612
4.601
3.780
1.617
1.595
1.378
1.354
BRUKER
NAME AS-87
EXPNO 1
PROCNO 1
Date_ 20120105
Time 12.22
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
0.97
9.44
1.69
2.21
1.00
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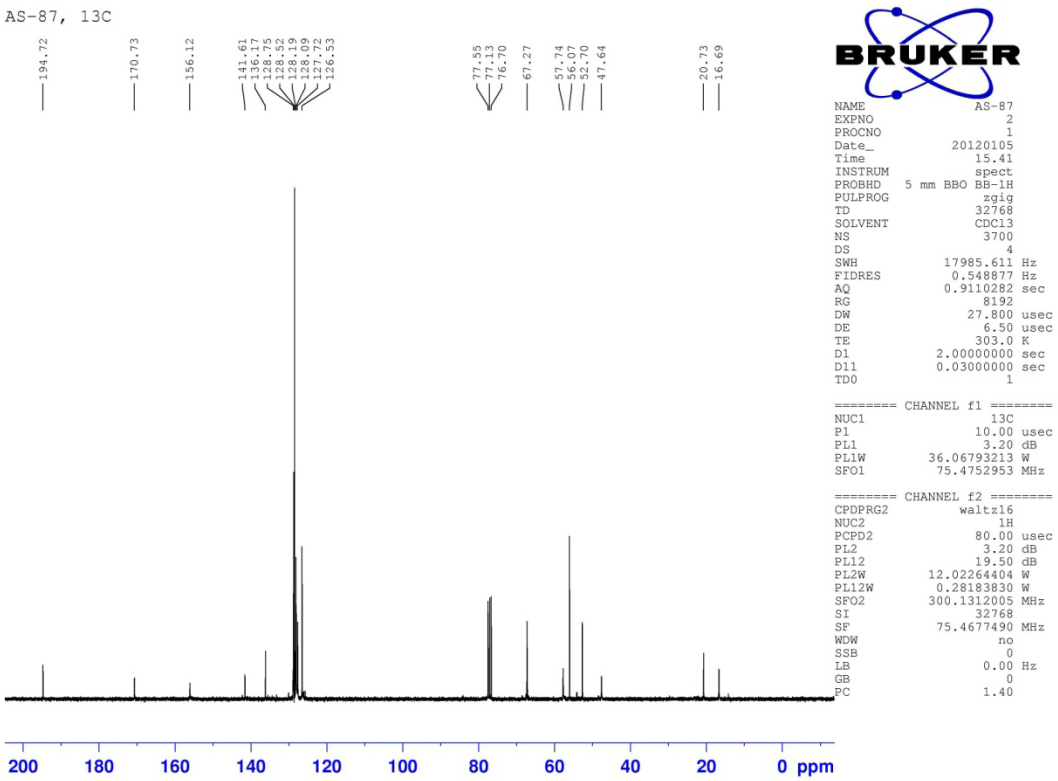
3.33
3.08
3.13


AS-87, 13C
194.72
170.73
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136.17
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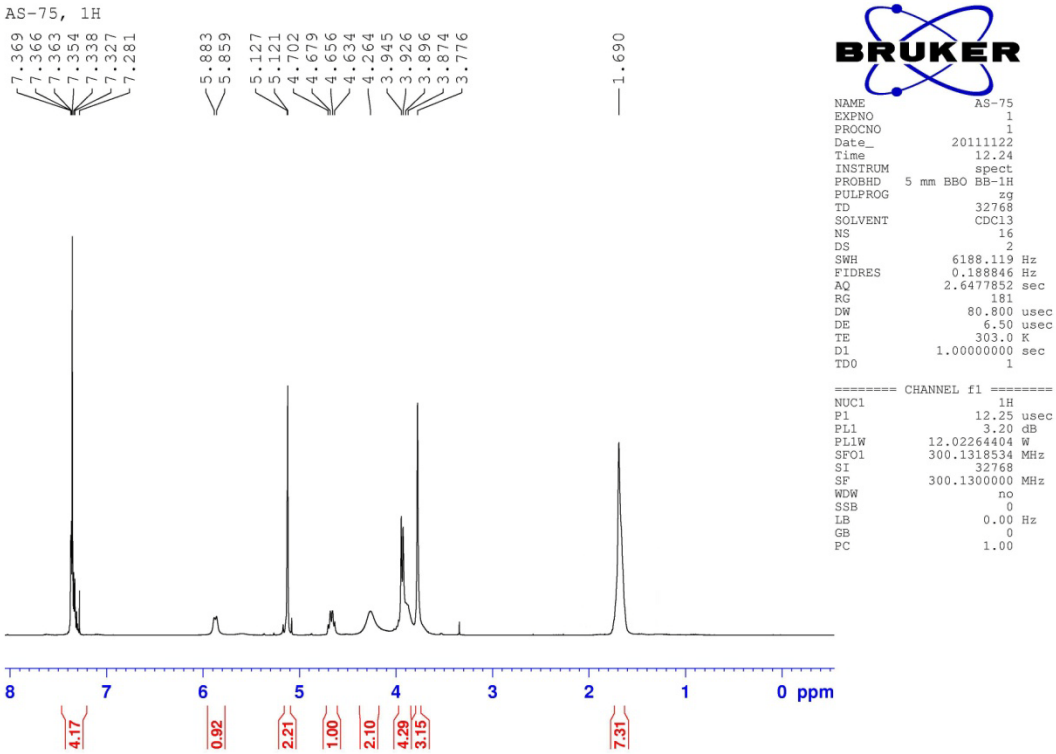
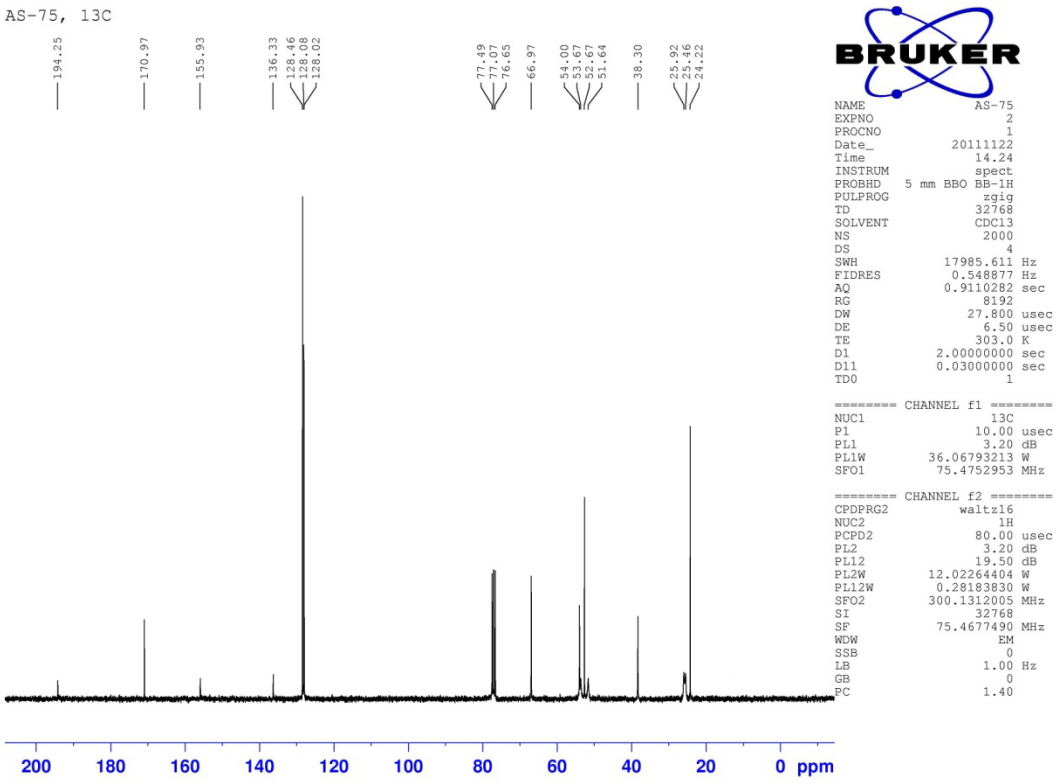
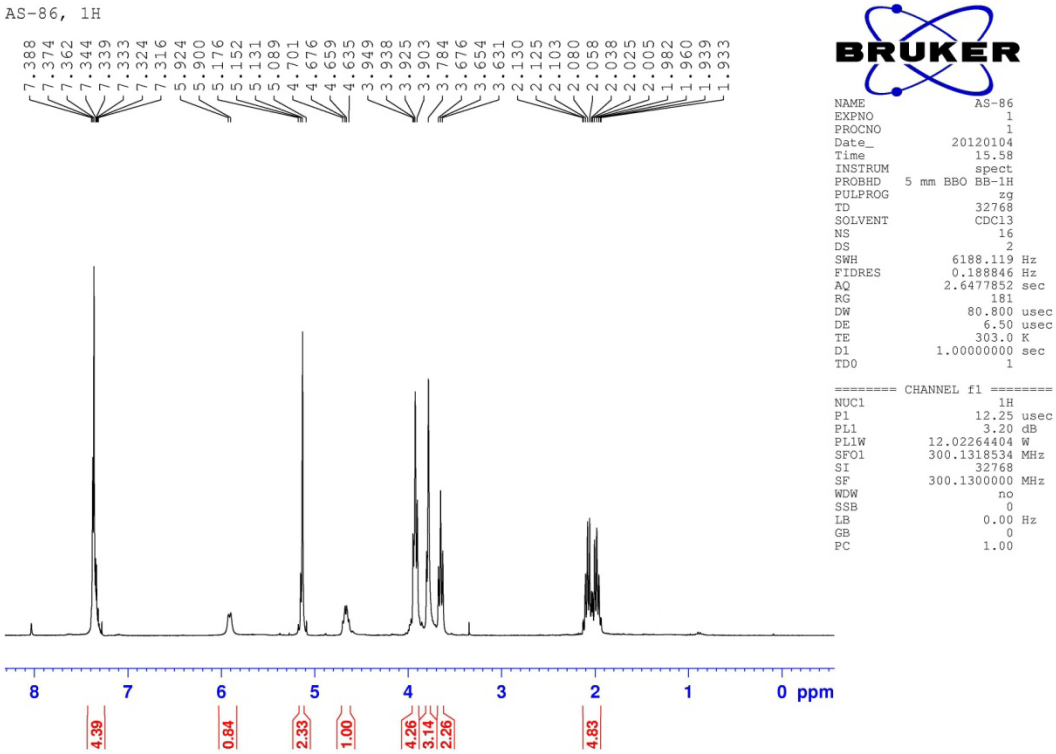
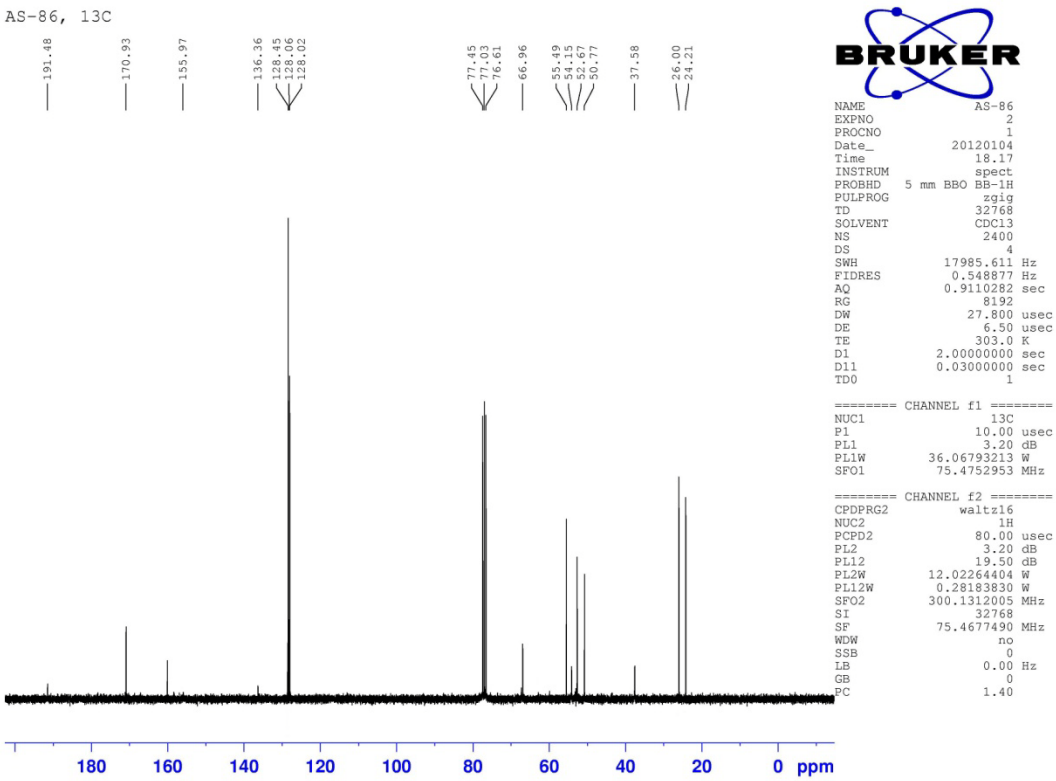
128.52
128.19
128.09
127.72
126.53
77.55
77.13
76.70
67.27
57.74
56.07
52.70
47.64
20.73
16.69
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME AS-87
EXPNO 2
PROCNO 1
Date_ 20120105
Time 15.41
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 3700
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40


AS-75, 1H
7.369
7.366
7.363
7.354
7.338
7.327
7.281
5.883
5.859
5.127
5.121
4.702
4.679
4.656
4.634
4.264
3.945
3.926
3.896
3.874
3.776
1.690
BRUKER
NAME AS-75
EXPNO 1
PROCNO 1
Date_ 20111122
Time 12.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
4.17
0.92
2.21
1.00
2.10
4.29
3.15
7.31

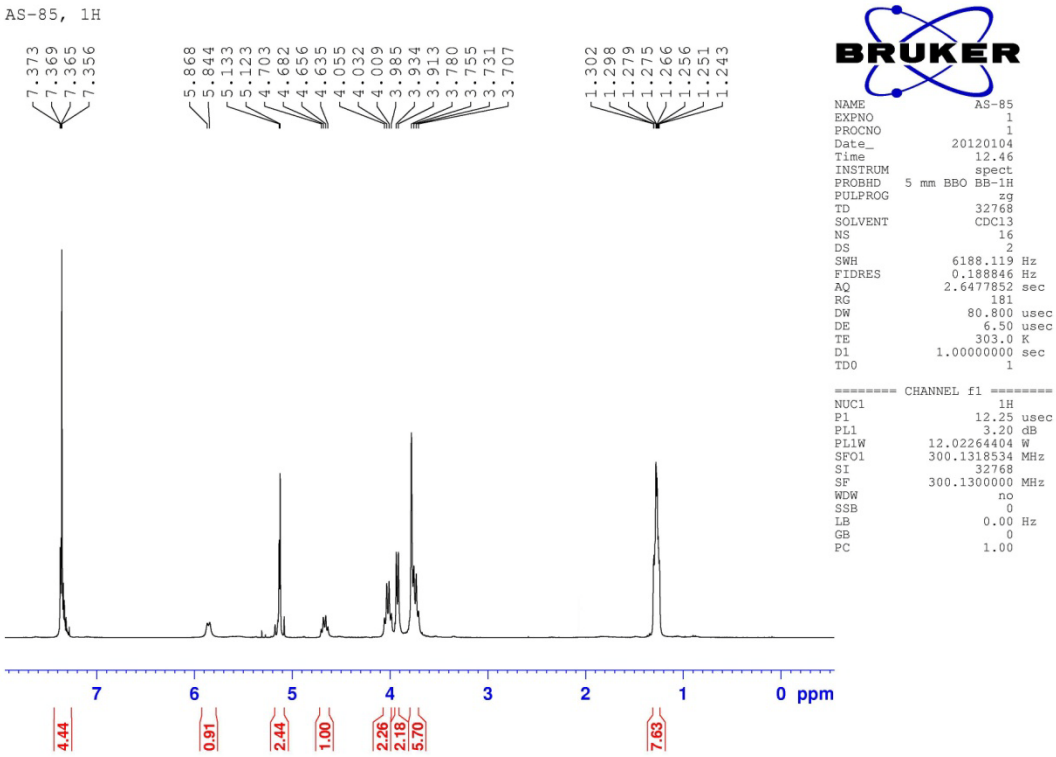

AS-75, 13C
194.25
170.97
155.93
136.33
128.46
128.08
128.02
77.49
77.07
76.65
66.97
54.00
53.67
52.67
51.64
38.30
25.92
25.46
24.22
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME AS-75
EXPNO 2
PROCNO 1
Date_ 20111122
Time 14.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 2000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

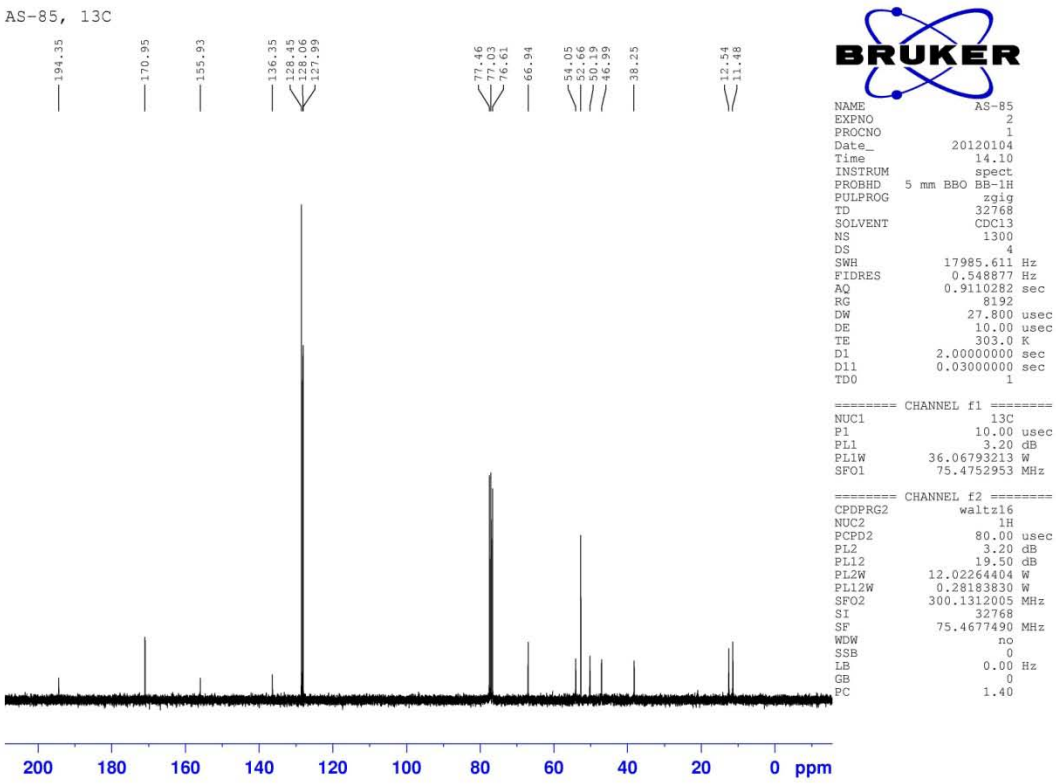

AS-86, 1H
7.388
7.374
7.362
7.344
7.339
7.333
7.324
7.316
5.924
5.900
5.176
5.152
5.131
5.089
4.701
4.676
4.659
4.635
3.949
3.938
3.925
3.903
3.784
3.676
3.654
3.631
2.130
2.125
2.103
2.080
2.058
2.038
2.025
2.005
1.982
1.960
1.939
1.933
8
7
6
5
4
3
2
1
0 ppm
4.39
0.84
2.33
1.00
4.26
3.14
2.26
4.83
BRUKER
NAME AS-86
EXPNO 1
PROCNO 1
Date_ 20120104
Time 15.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

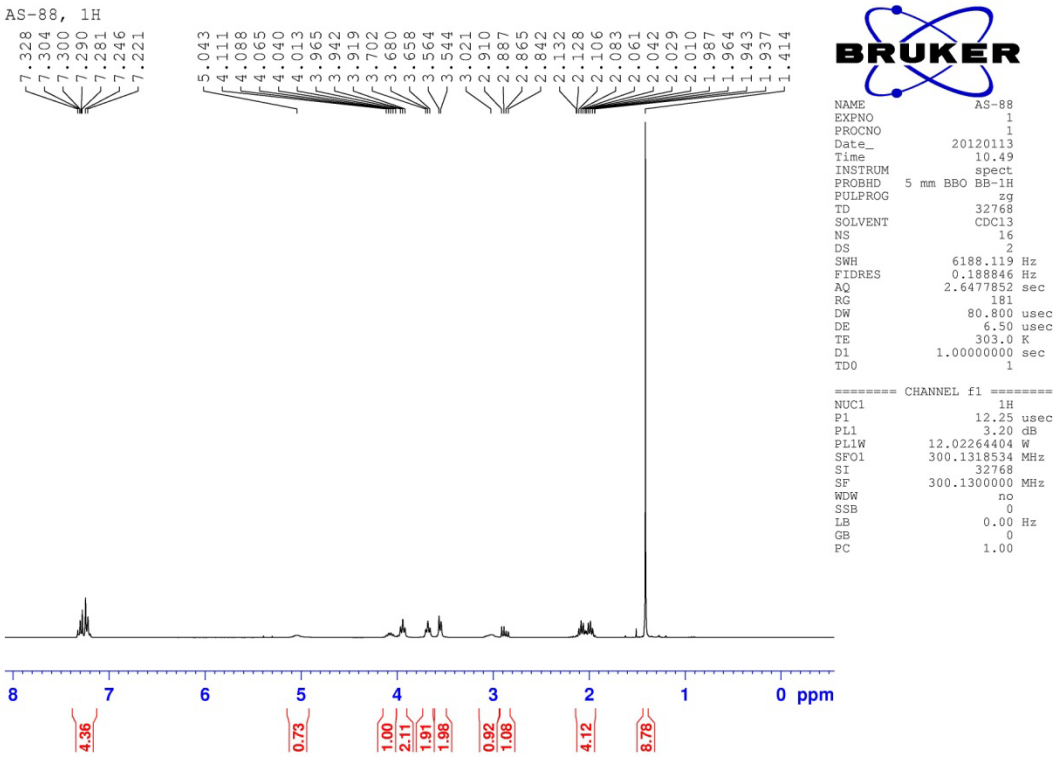


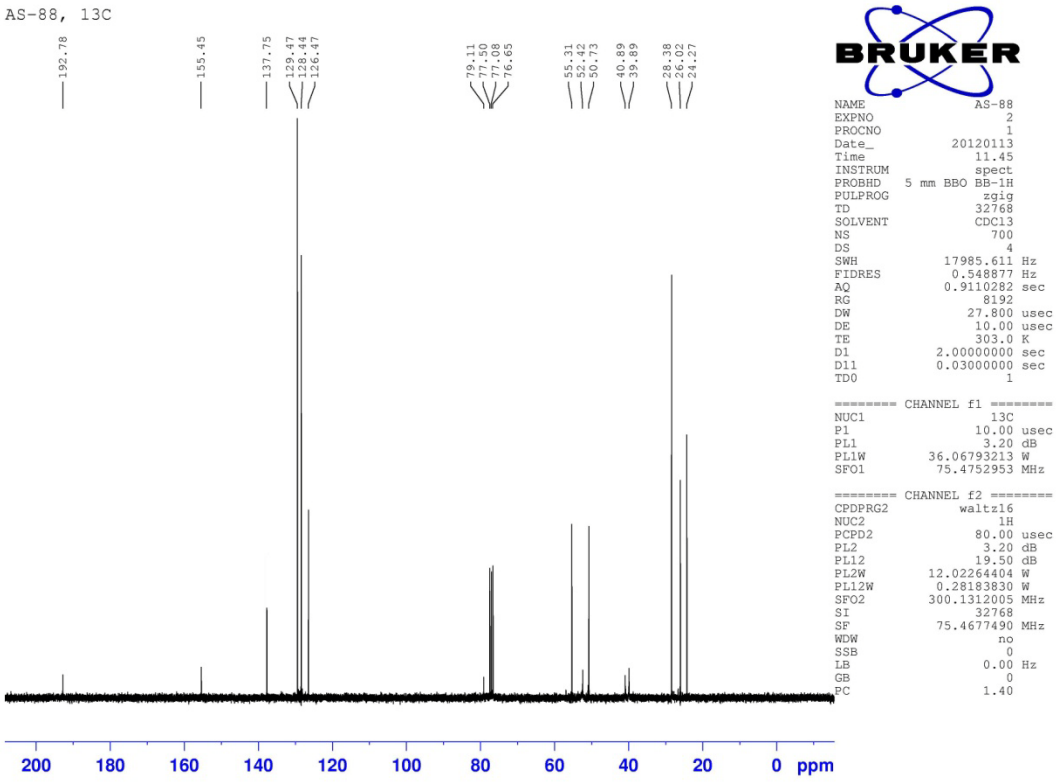
BRUKER

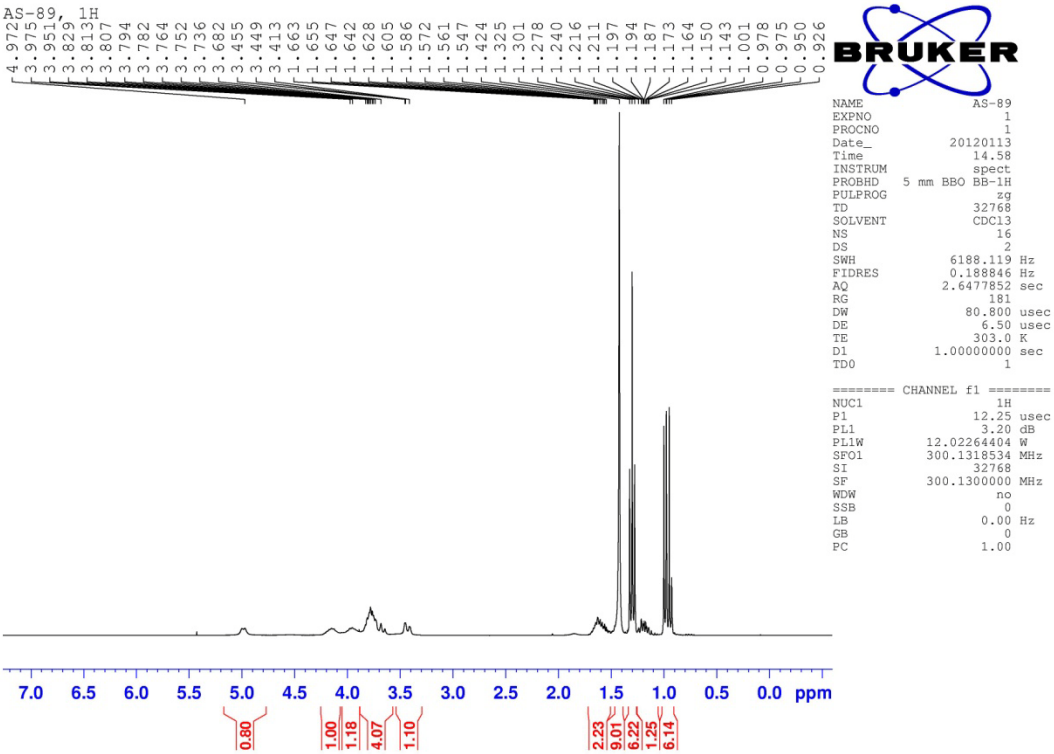
NAME	AS-86
EXPNO	2
PROCNO	1
Date_	20120104
Time	18.17
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	2400
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

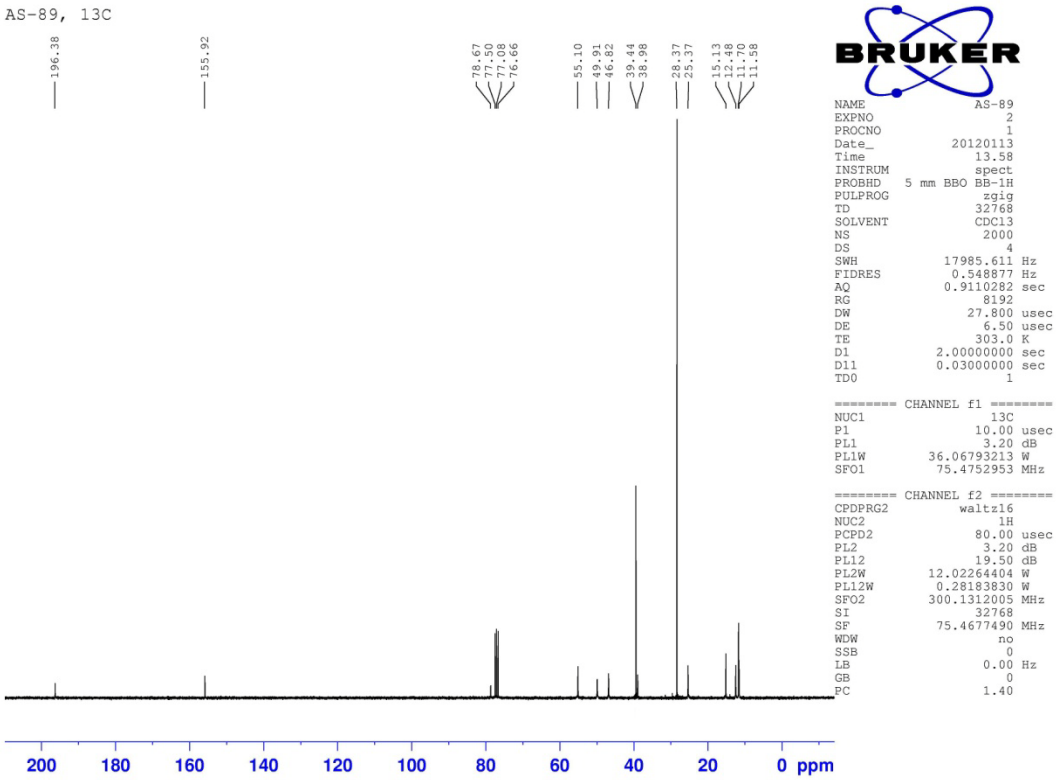

AS-85, 1H
7.373
7.369
7.365
7.356
5.868
5.844
5.133
5.123
4.703
4.682
4.656
4.635
4.055
4.032
4.009
3.985
3.934
3.913
3.780
3.755
3.731
3.707
1.302
1.298
1.279
1.275
1.266
1.256
1.251
1.243
BRUKER
NAME AS-85
EXPNO 1
PROCNO 1
Date_ 20120104
Time 12.46
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7
6
5
4
3
2
1
0 ppm
4.44
0.91
2.44
1.00
2.26
2.18
5.70
7.63


AS-85, 13C
194.35
170.95
155.93
136.35
128.45
128.06
127.99
77.46
77.03
76.61
66.94
54.05
52.66
50.19
46.99
38.25
12.54
11.48
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME AS-85
EXPNO 2
PROCNO 1
Date_ 20120104
Time 14.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 1300
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 10.00 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40


AS-88, 1H
7.328
7.304
7.300
7.290
7.281
7.246
7.221
5.043
4.111
4.088
4.065
4.040
4.013
3.965
3.942
3.919
3.702
3.680
3.658
3.564
3.544
3.021
2.910
2.887
2.865
2.842
2.132
2.128
2.106
2.083
2.061
2.042
2.029
2.010
1.987
1.964
1.943
1.937
1.414
8
7
6
5
4
3
2
1
0 ppm
4.36
0.73
1.00
2.11
1.91
1.98
0.92
1.08
4.12
8.78
BRUKER
NAME AS-88
EXPNO 1
PROCNO 1
Date_ 20120113
Time 10.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

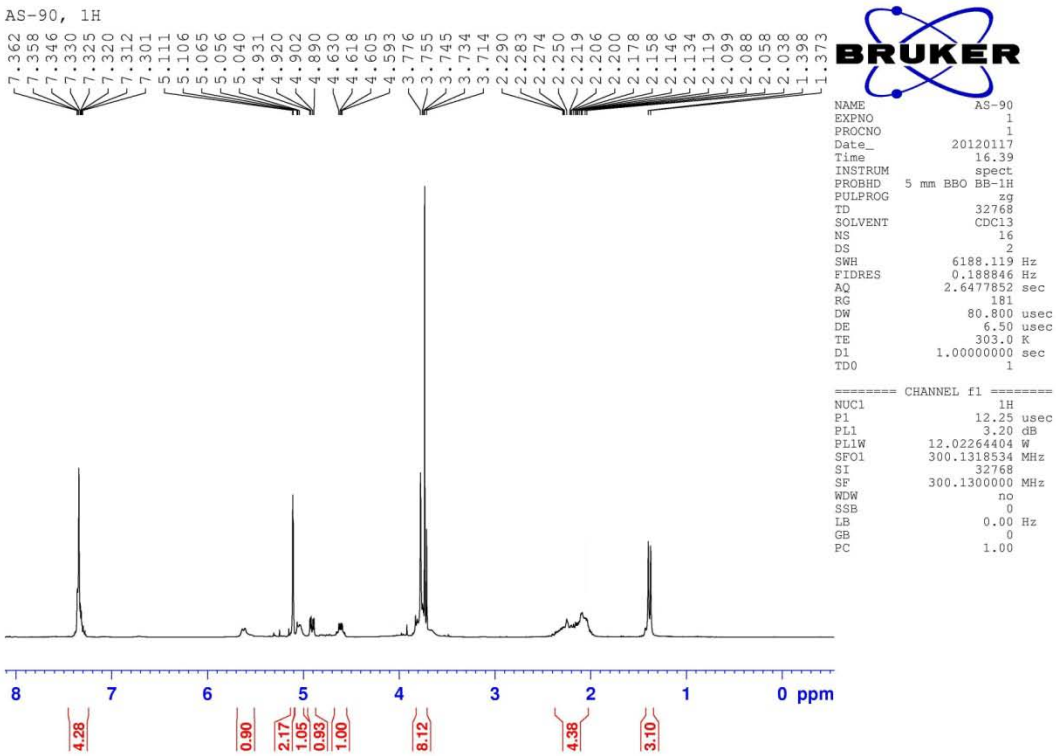

AS-88, 13C
192.78
155.45
137.75
129.47
128.44
126.47
79.11
77.50
77.08
76.65
55.31
52.42
50.73
40.89
39.89
28.38
26.02
24.27
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME AS-88
EXPNO 2
PROCNO 1
Date_ 20120113
Time 11.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 700
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 10.00 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 dB
PL2W 12.02264404 W
PL12W 0.28183830 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

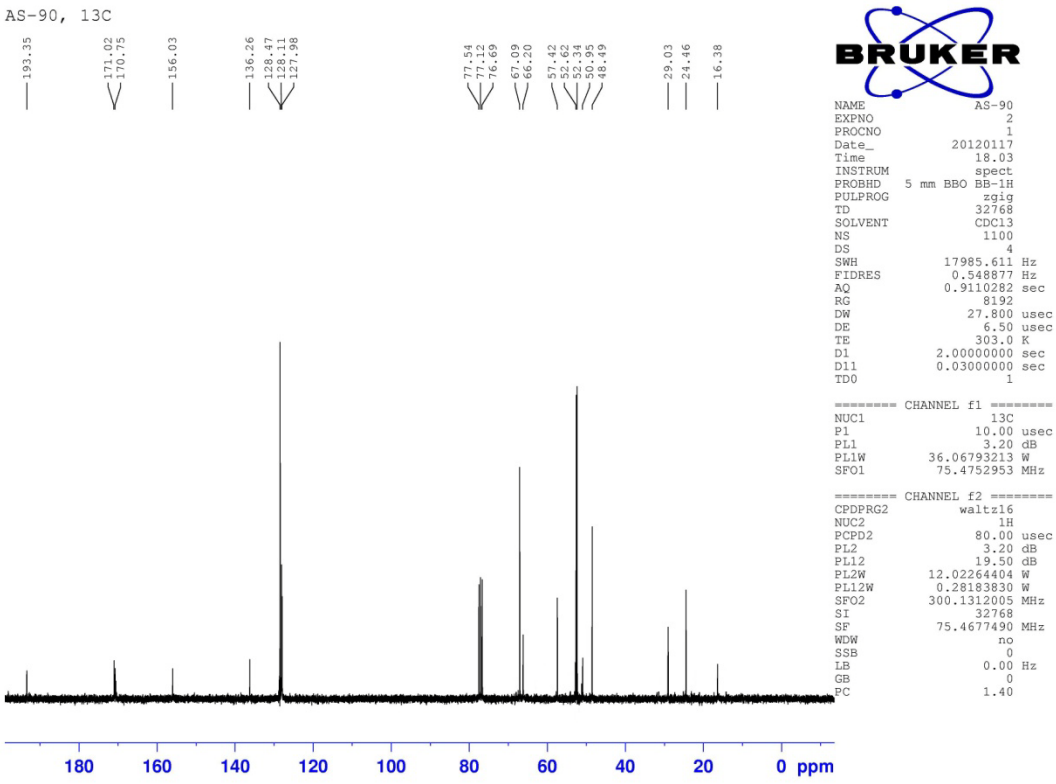

AS-89, 1H
4.972
3.975
3.951
3.829
3.813
3.807
3.794
3.782
3.764
3.752
3.736
3.682
3.455
3.449
3.413
1.663
1.655
1.647
1.642
1.628
1.605
1.586
1.572
1.561
1.547
1.424
1.325
1.301
1.278
1.240
1.216
1.211
1.197
1.194
1.187
1.173
1.164
1.150
1.143
1.001
0.978
0.975
0.950
0.926
BRUKER
NAME AS-89
EXPNO 1
PROCNO 1
Date_ 20120113
Time 14.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.80
1.00
1.18
4.07
1.10
2.23
9.01
6.22
1.25
6.14



BRUKER

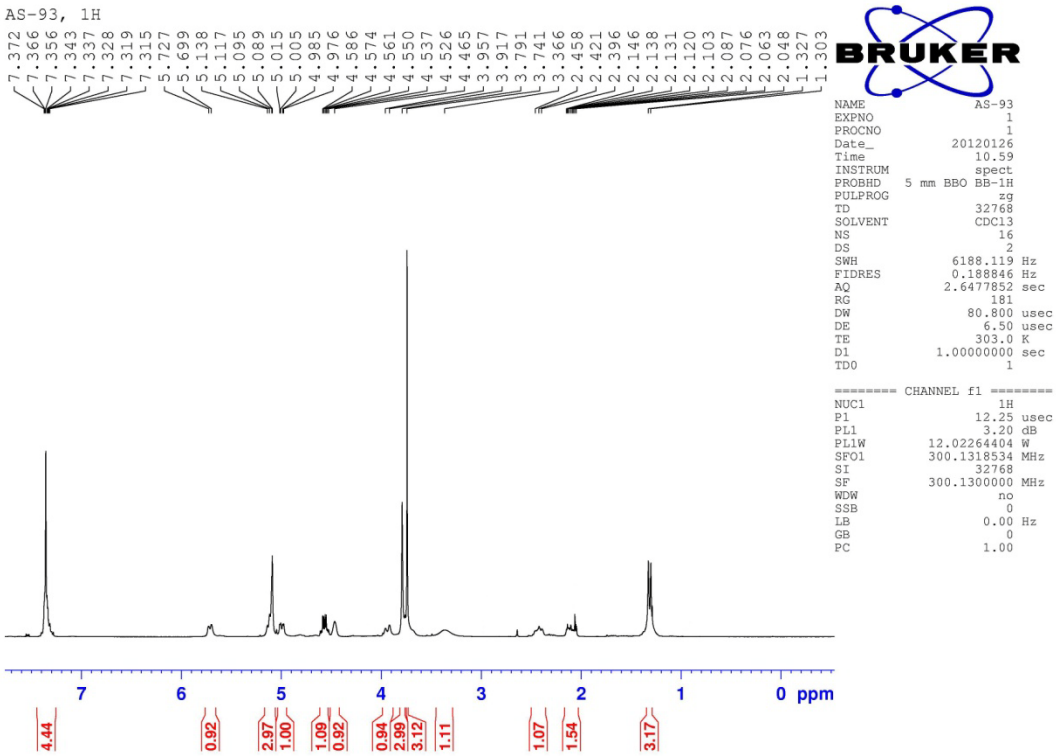
NAME	AS-89
EXPNO	2
PROCNO	1
Date_	20120113
Time	13.58
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	2000
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

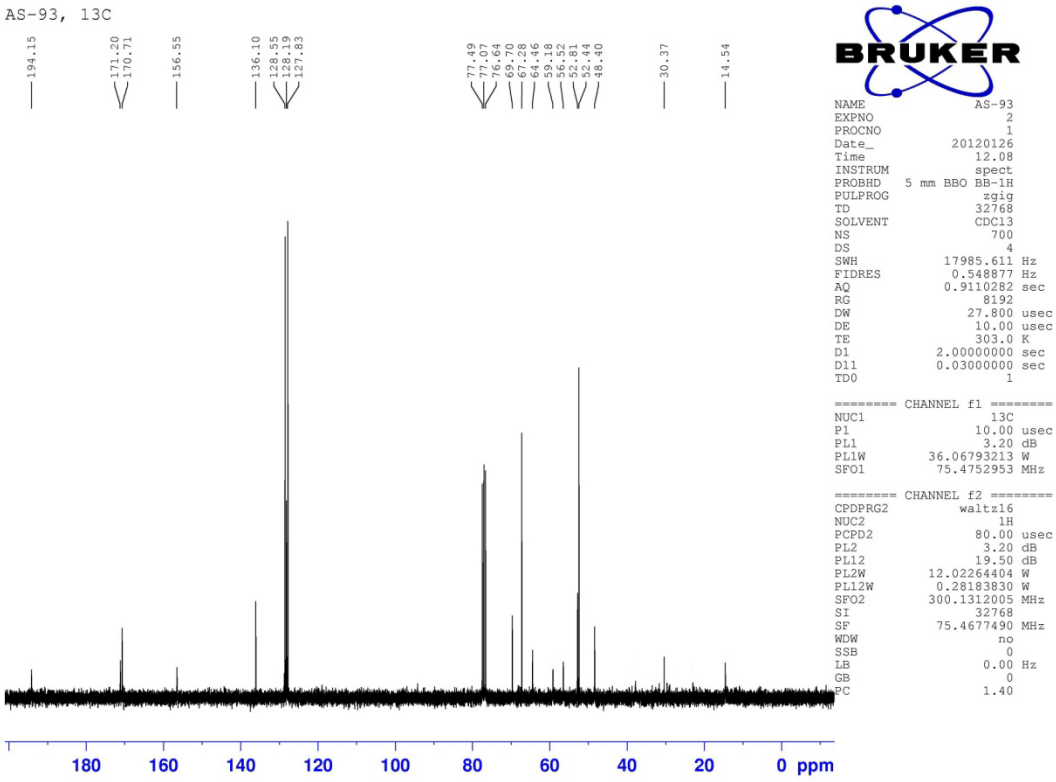

AS-90, 1H
7.362
7.358
7.346
7.330
7.325
7.320
7.312
7.301
5.111
5.106
5.065
5.056
5.040
4.931
4.920
4.902
4.890
4.630
4.618
4.605
4.593
3.776
3.755
3.745
3.734
3.714
2.290
2.283
2.274
2.250
2.219
2.206
2.200
2.178
2.158
2.146
2.134
2.119
2.099
2.088
2.058
2.038
1.398
1.373
BRUKER
NAME AS-90
EXPNO 1
PROCNO 1
Date_ 20120117
Time 16.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
4.28
0.90
2.17
1.05
0.93
1.00
8.12
4.38
3.10



BRUKER

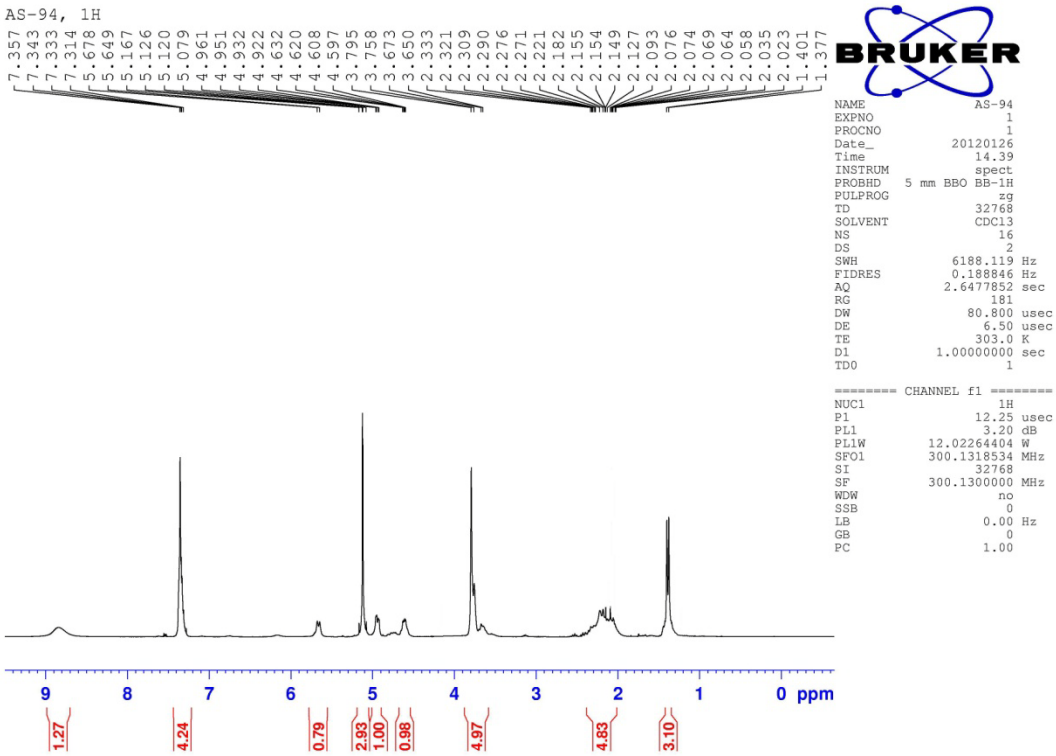
NAME	AS-90
EXPNO	2
PROCNO	1
Date_	20120117
Time	18.03
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	1100
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

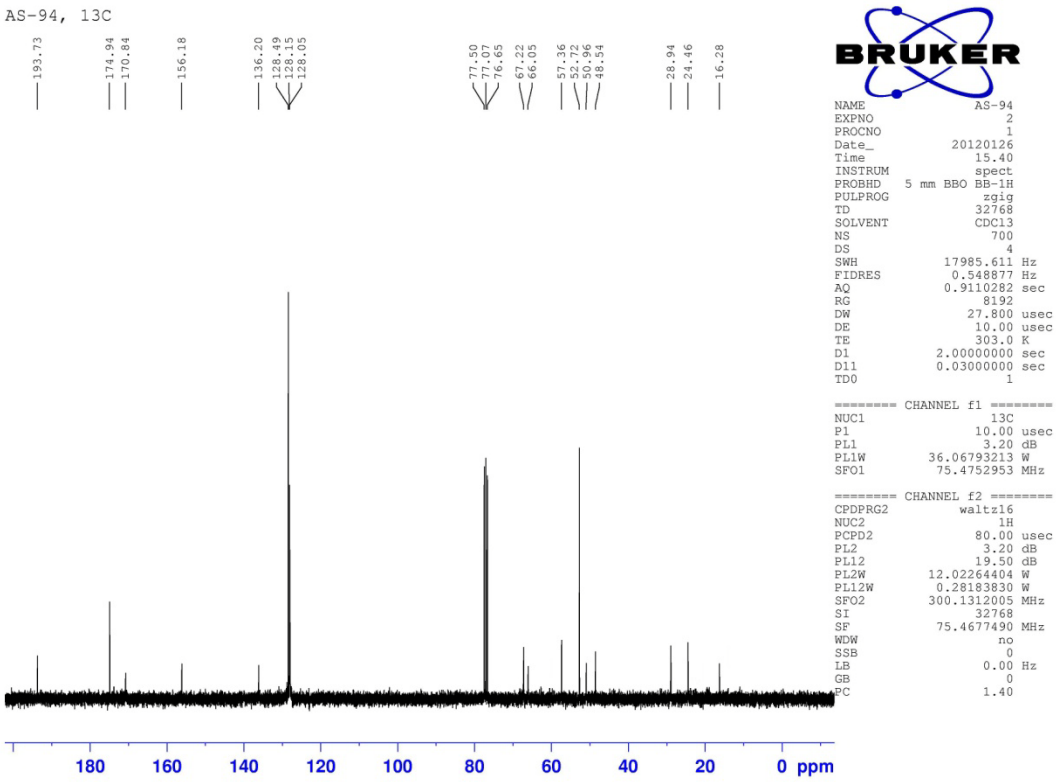

AS-93, 1H
7.372
7.366
7.356
7.343
7.337
7.328
7.319
7.315
5.727
5.699
5.138
5.117
5.095
5.089
5.015
5.005
4.985
4.976
4.586
4.574
4.561
4.550
4.537
4.526
4.465
3.957
3.917
3.791
3.741
3.366
2.458
2.421
2.396
2.146
2.138
2.131
2.120
2.103
2.087
2.076
2.063
2.048
1.327
1.303
BRUKER
NAME AS-93
EXPNO 1
PROCNO 1
Date_ 20120126
Time 10.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7
6
5
4
3
2
1
0 ppm
4.44
0.92
2.97
1.00
1.09
0.92
0.94
2.99
3.12
1.11
1.07
1.54
3.17



BRUKER

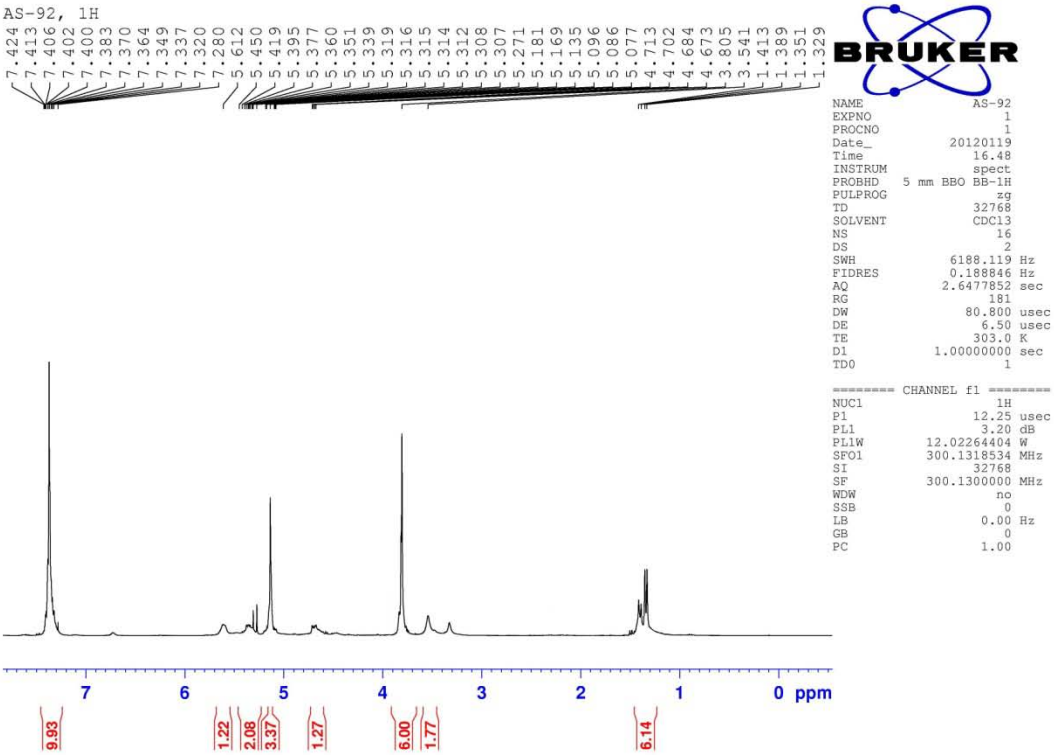
NAME	AS-93
EXPNO	2
PROCNO	1
Date_	20120126
Time	12.08
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	700
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	10.00 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

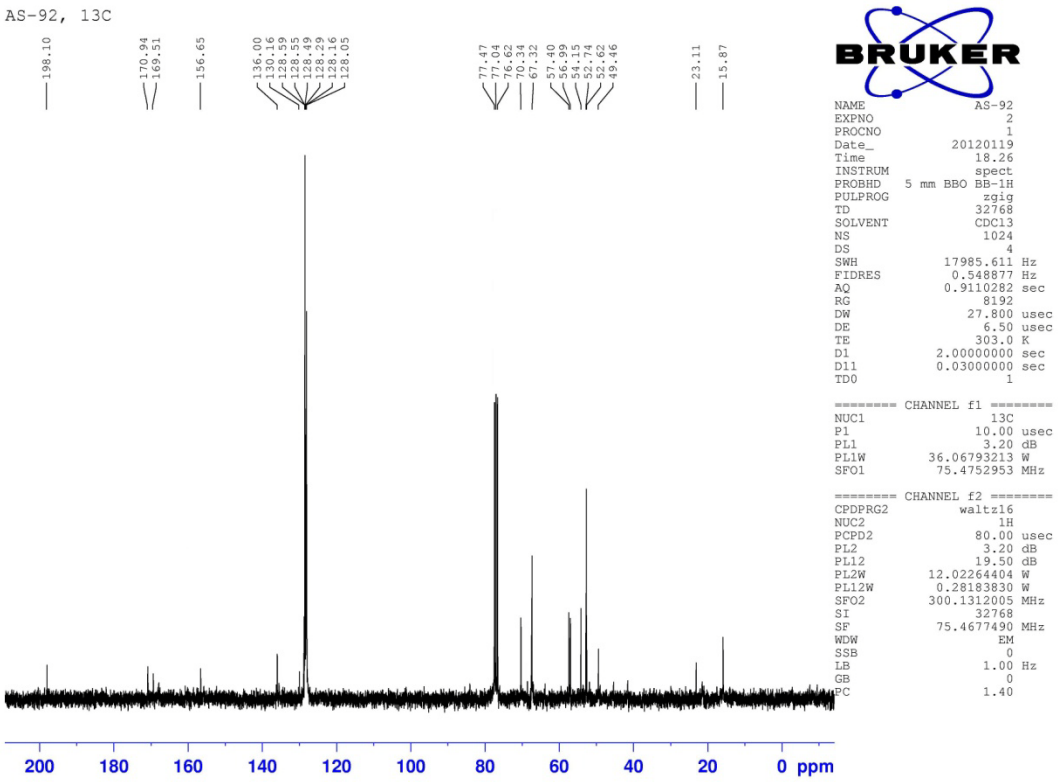

AS-94, 1H
7.357
7.343
7.333
7.314
5.678
5.649
5.167
5.126
5.120
5.079
4.961
4.951
4.932
4.922
4.632
4.620
4.608
4.597
3.795
3.758
3.673
3.650
2.333
2.321
2.309
2.290
2.276
2.271
2.221
2.182
2.155
2.154
2.149
2.127
2.093
2.076
2.074
2.069
2.064
2.058
2.035
2.023
1.401
1.377
BRUKER
NAME AS-94
EXPNO 1
PROCNO 1
Date_ 20120126
Time 14.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
1.27
4.24
0.79
2.93
1.00
0.98
4.97
4.83
3.10



BRUKER

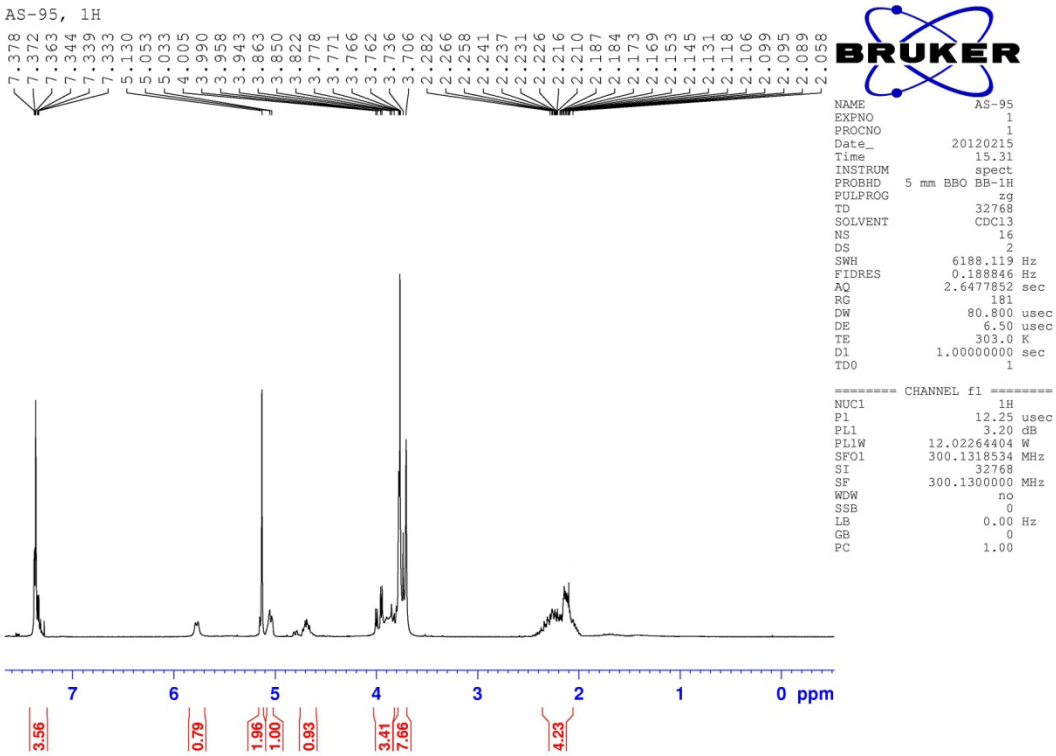
NAME	AS-94
EXPNO	2
PROCNO	1
Date_	20120126
Time	15.40
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	700
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	10.00 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

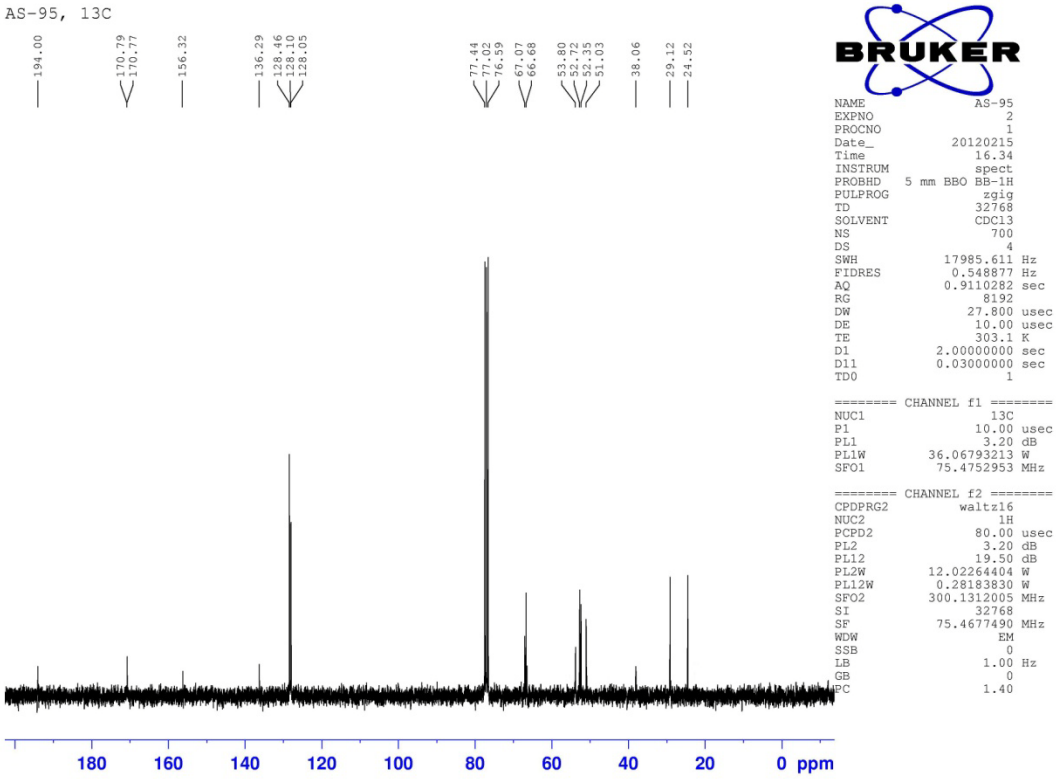

AS-92, 1H
7.424
7.413
7.406
7.402
7.400
7.383
7.370
7.364
7.349
7.337
7.320
7.280
5.612
5.450
5.419
5.395
5.377
5.360
5.351
5.339
5.319
5.316
5.315
5.314
5.312
5.308
5.307
5.271
5.181
5.169
5.135
5.096
5.086
5.077
4.713
4.702
4.684
4.673
3.805
3.541
1.413
1.389
1.351
1.329
BRUKER
NAME AS-92
EXPNO 1
PROCNO 1
Date_ 20120119
Time 16.48
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7
6
5
4
3
2
1
0 ppm
9.93
1.22
2.08
3.37
1.27
6.00
1.77
6.14



BRUKER

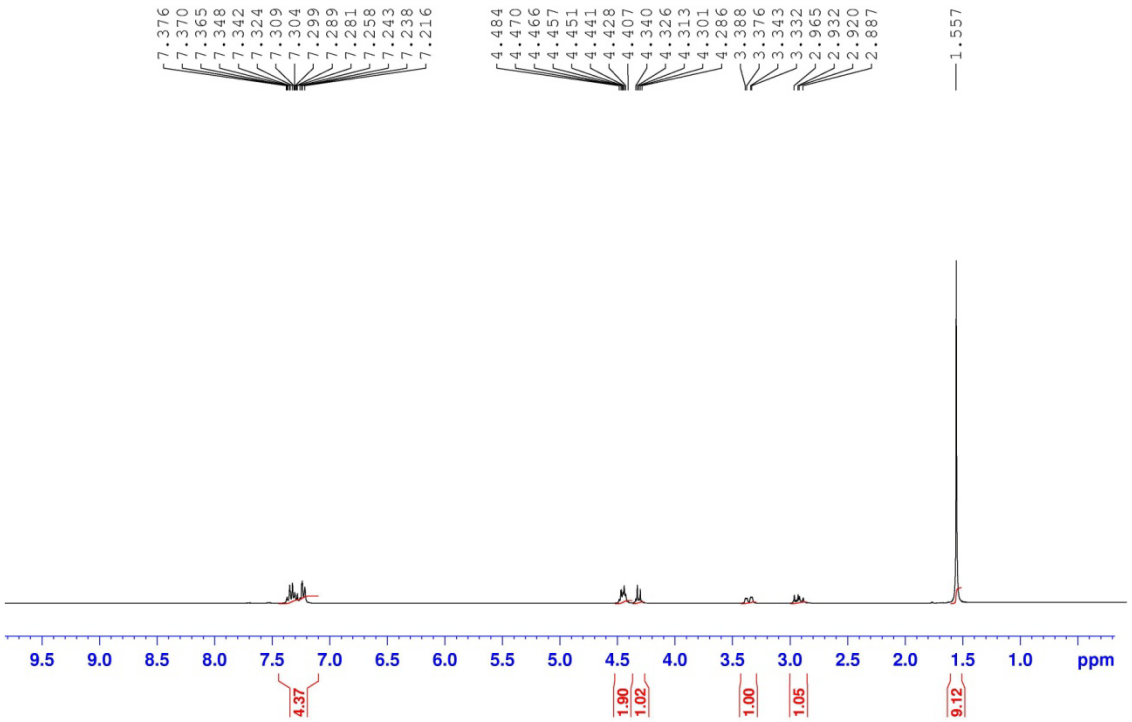
NAME	AS-92
EXPNO	2
PROCNO	1
Date_	20120119
Time	18.26
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	1024
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

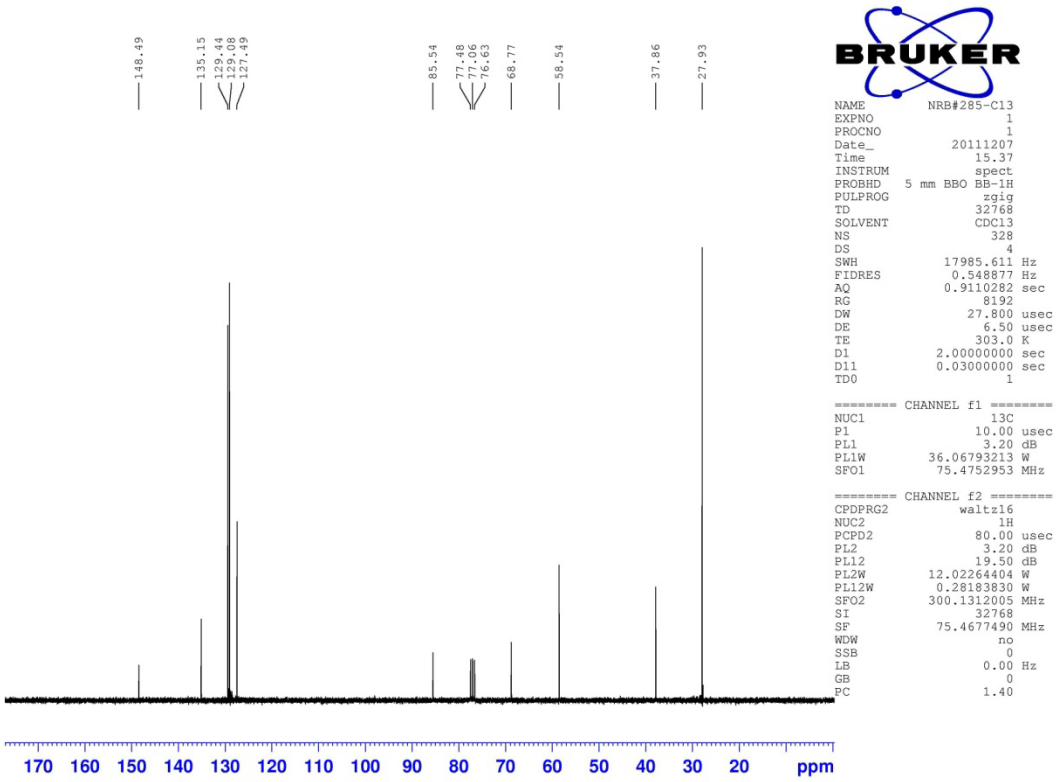

AS-95, 1H
7.378
7.372
7.363
7.344
7.339
7.333
5.130
5.053
5.033
4.005
3.990
3.958
3.943
3.863
3.850
3.822
3.778
3.771
3.766
3.762
3.736
3.706
2.282
2.266
2.258
2.241
2.237
2.231
2.226
2.216
2.210
2.187
2.184
2.173
2.169
2.153
2.145
2.131
2.118
2.106
2.099
2.095
2.089
2.058
BRUKER
NAME AS-95
EXPNO 1
PROCNO 1
Date_ 20120215
Time 15.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7
6
5
4
3
2
1
0 ppm
3.56
0.79
1.96
1.00
0.93
3.41
7.66
4.23



BRUKER

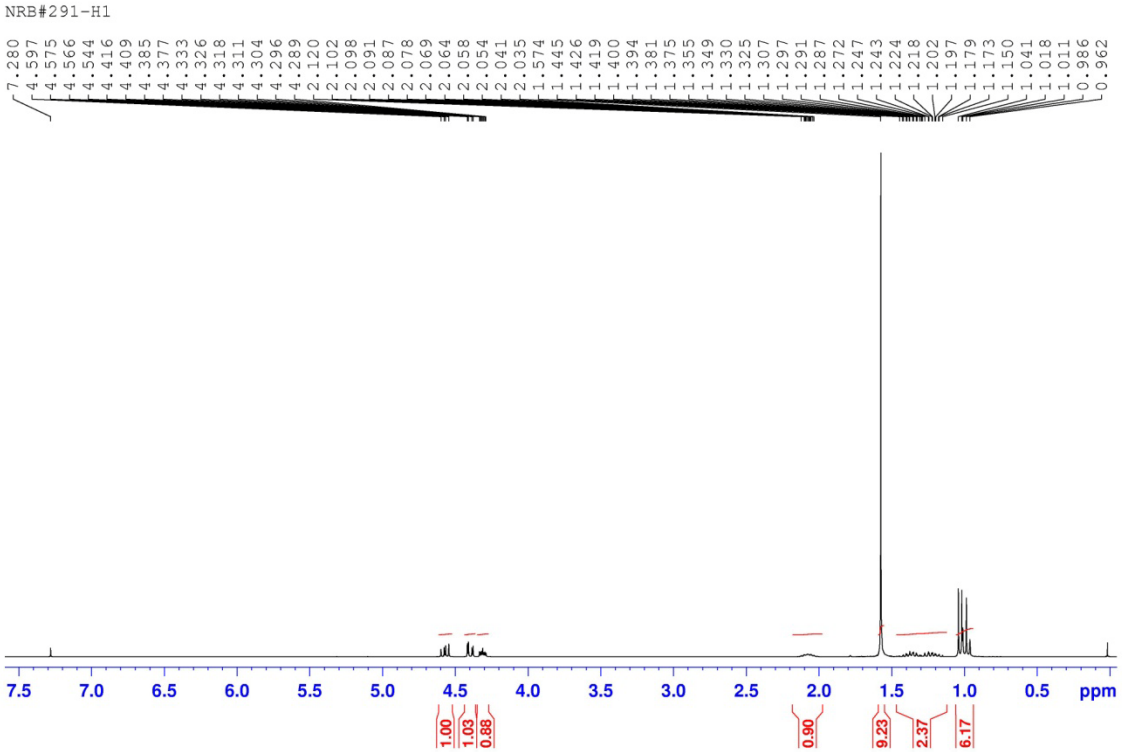
NAME	AS-95
EXPNO	2
PROCNO	1
Date_	20120215
Time	16.34
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	700
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	10.00 usec
TE	303.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

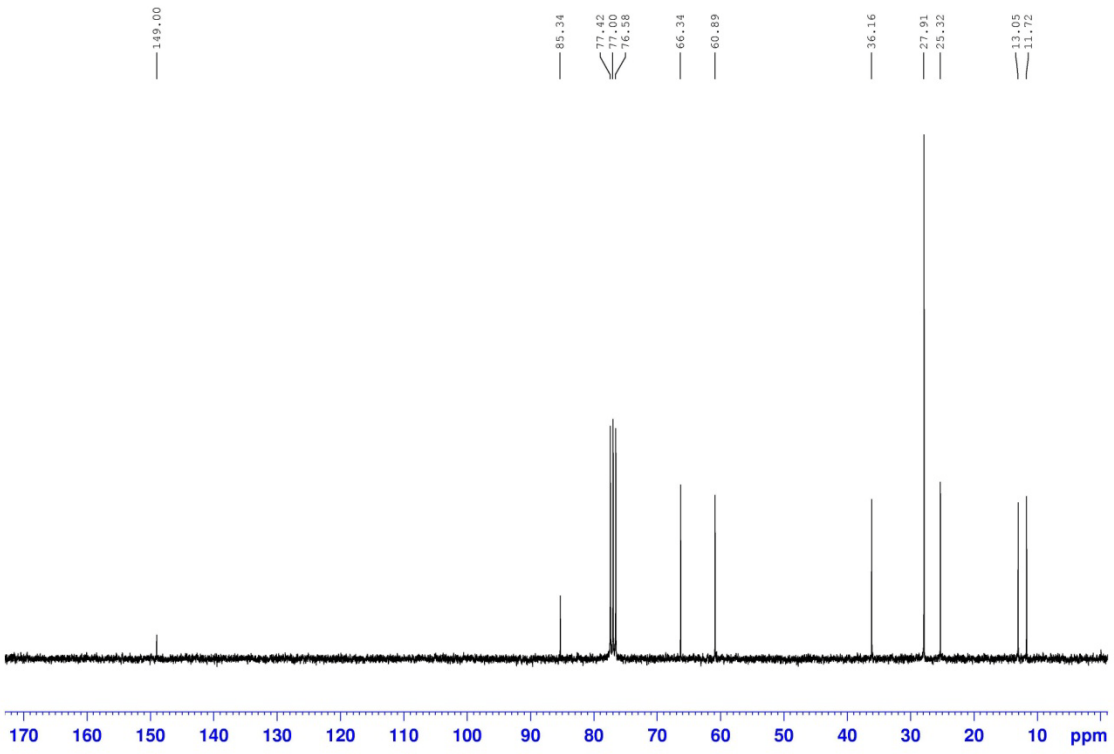

7.376
7.370
7.365
7.348
7.342
7.324
7.309
7.304
7.299
7.289
7.281
7.258
7.243
7.238
7.216
4.484
4.470
4.466
4.457
4.451
4.441
4.428
4.407
4.340
4.326
4.313
4.301
4.286
3.388
3.376
3.343
3.332
2.965
2.932
2.920
2.887
1.557
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
4.37
1.90
1.02
1.00
1.05
9.12

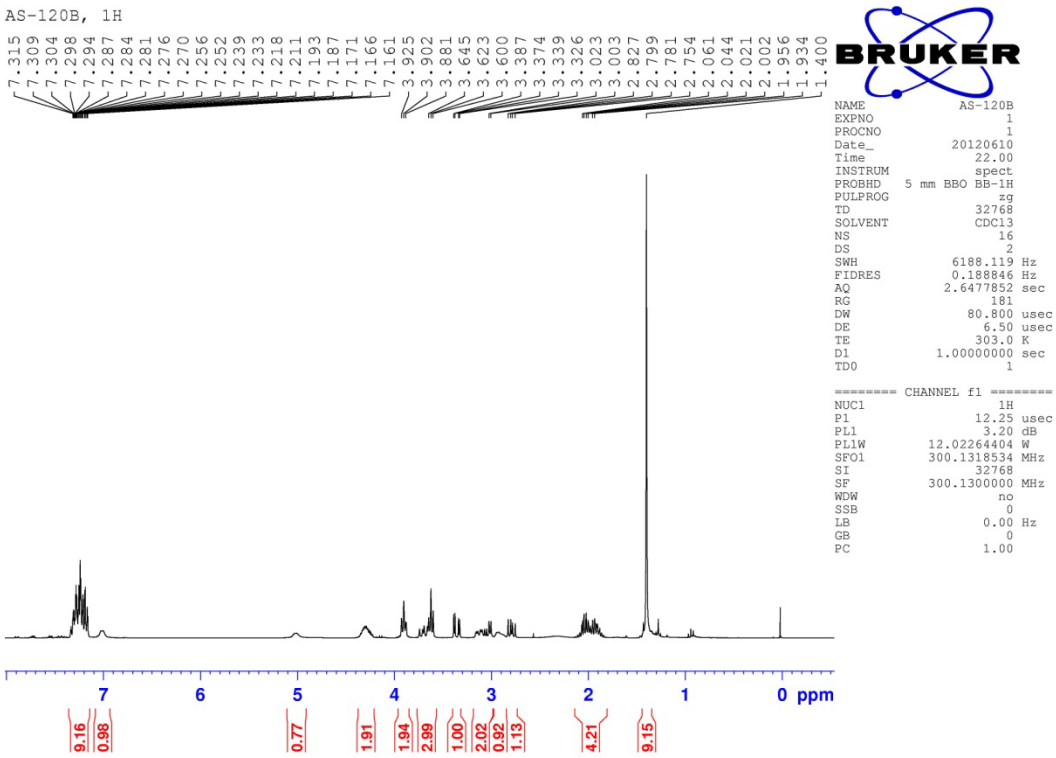


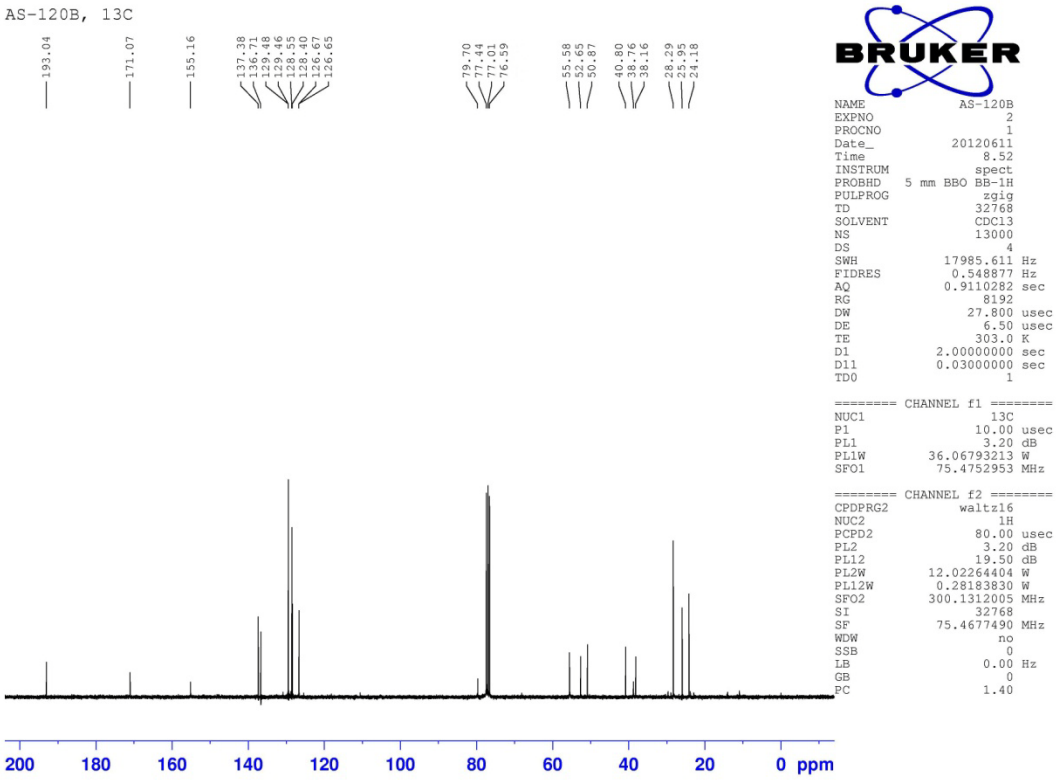
BRUKER

NAME	NRB#285-C13
EXPNO	1
PROCNO	1
Date_	20111207
Time	15.37
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	328
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40


NRB#291-H1
7.280
4.597
4.575
4.566
4.544
4.416
4.409
4.385
4.377
4.333
4.326
4.318
4.311
4.304
4.296
4.289
2.120
2.102
2.098
2.091
2.087
2.078
2.069
2.064
2.058
2.054
2.041
2.035
1.574
1.445
1.426
1.419
1.400
1.394
1.381
1.375
1.355
1.349
1.330
1.325
1.307
1.297
1.291
1.287
1.272
1.247
1.243
1.224
1.218
1.202
1.197
1.179
1.173
1.150
1.041
1.018
1.011
0.986
0.962
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.03
0.88
0.90
9.23
2.37
6.17

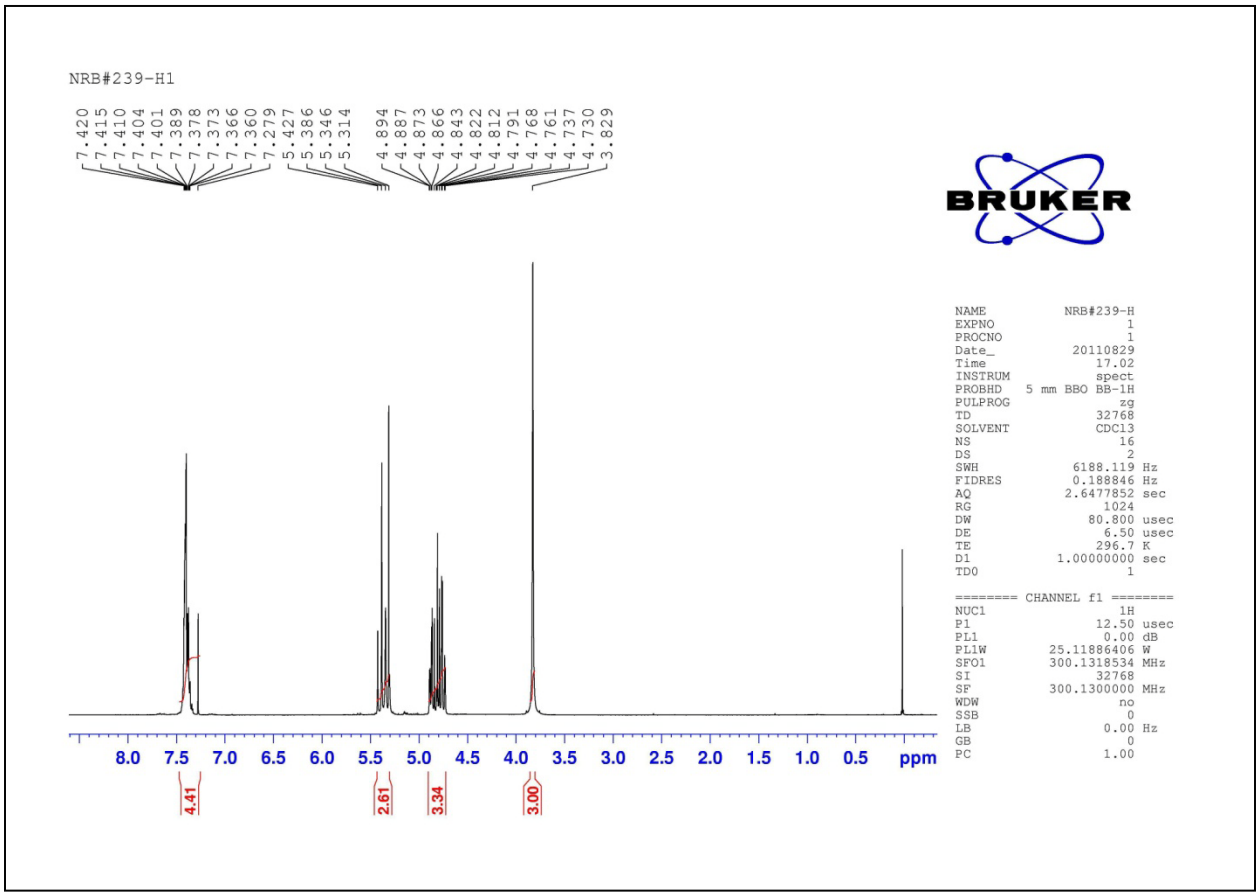

149.00
85.34
77.42
77.00
76.58
66.34
60.89
36.16
27.91
25.32
13.05
11.72
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

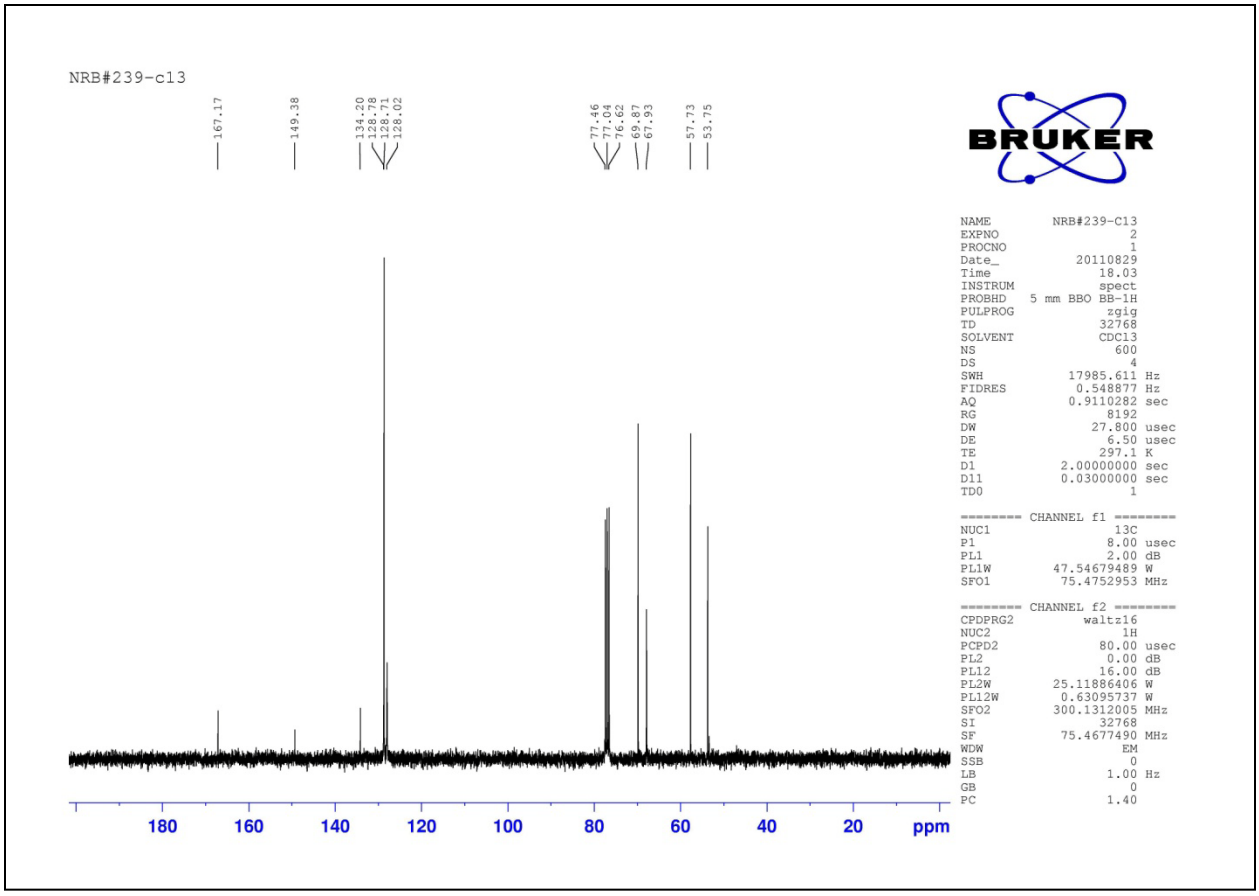

AS-120B, 1H
7.315 7.309 7.304 7.298 7.294 7.287 7.284 7.281 7.276 7.270 7.256 7.252 7.239 7.233 7.218 7.211 7.193 7.187 7.171 7.166 7.161 3.925 3.902 3.881 3.645 3.623 3.600 3.387 3.374 3.339 3.326 3.023 3.003 2.827 2.799 2.781 2.754 2.061 2.044 2.021 2.002 1.956 1.934 1.400
BRUKER
NAME AS-120B
EXPNO 1
PROCNO 1
Date_ 20120610
Time 22.00
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
DW 80.800 usec
DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 3.20 dB
PL1W 12.02264404 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7 6 5 4 3 2 1 0 ppm
9.16 0.98 0.77 1.91 1.94 2.99 1.00 2.02 0.92 1.13 4.21 9.15



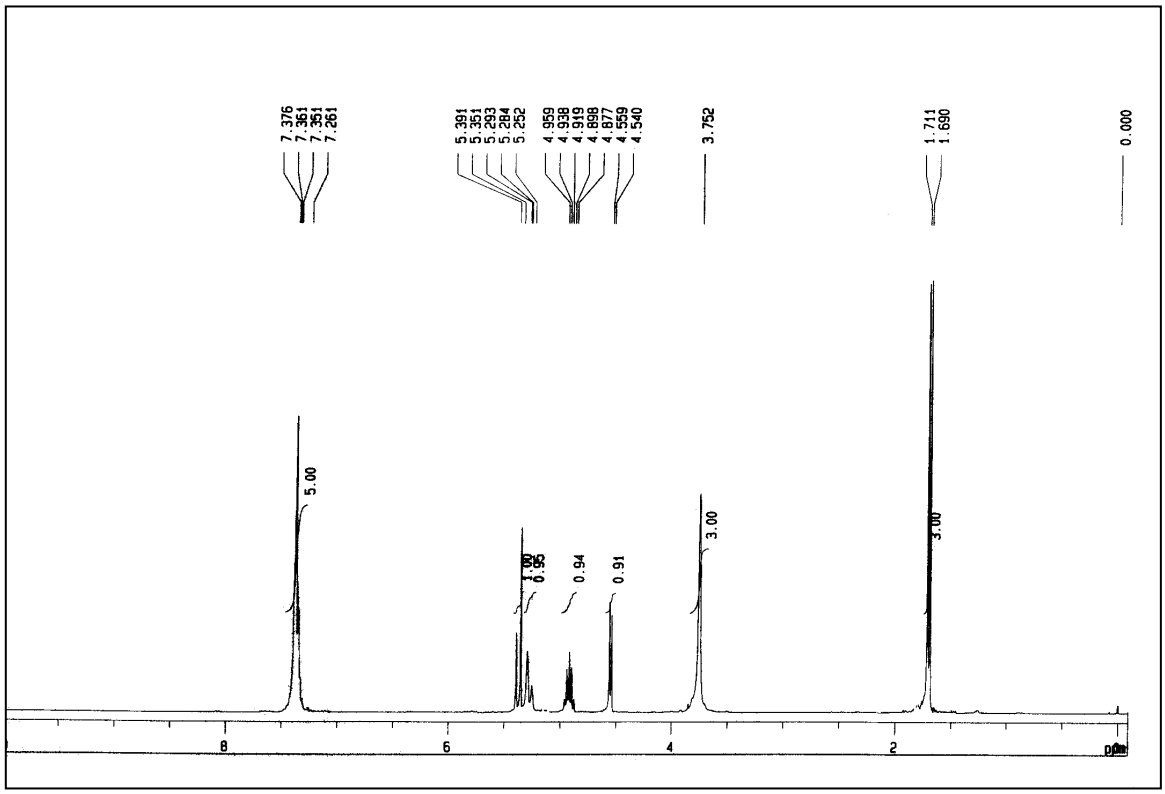
BRUKER

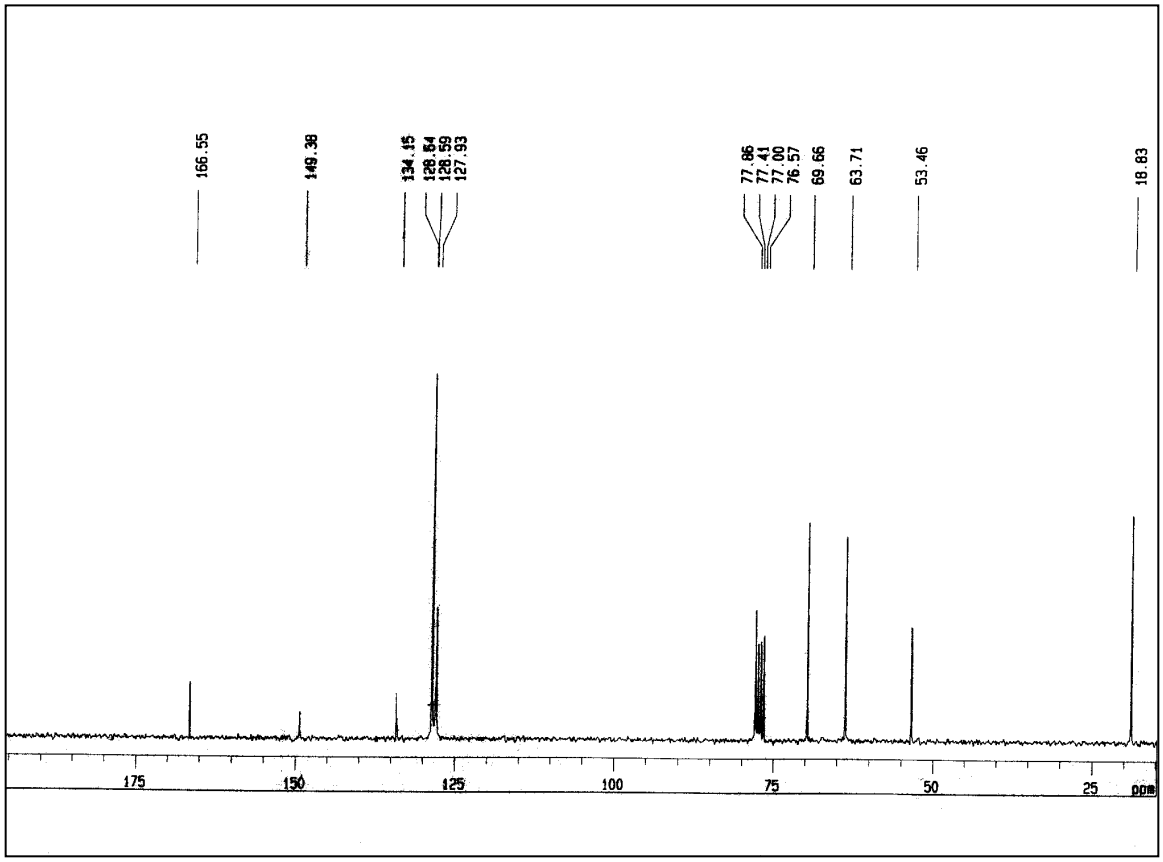
NAME	AS-120B
EXPNO	2
PROCNO	1
Date_	20120611
Time	8.52
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	13000
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	303.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	3.20 dB
PL1W	36.06793213 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	3.20 dB
PL12	19.50 dB
PL2W	12.02264404 W
PL12W	0.28183830 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40


NRB#239-H1
7.420
7.415
7.410
7.404
7.401
7.389
7.378
7.373
7.366
7.360
7.279
5.427
5.386
5.346
5.314
4.894
4.887
4.873
4.866
4.843
4.822
4.812
4.791
4.768
4.761
4.737
4.730
3.829
BRUKER
NAME NRB#239-H
EXPNO 1
PROCNO 1
Date_ 20110829
Time 17.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 1024
DW 80.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 0.00 dB
PL1W 25.11886406 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.41
2.61
3.34
3.00



NAME	NRB#239-C13
EXPNO	2
PROCNO	1
Date_	20110829
Time	18.03
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgig
TD	32768
SOLVENT	CDCl3
NS	600
DS	4
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110282 sec
RG	8192
DW	27.800 usec
DE	6.50 usec
TE	297.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	2.00 dB
PL1W	47.54679489 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	16.00 dB
PL2W	25.11886406 W
PL12W	0.63095737 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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