Electronic Supplementary Material (ESI) for Chemical Communications

Stereo and regio-selective one-pot synthesis of triazole-based unnatural amino acids and β- amino triazoles

R. B. Nasir Baig and Rajender S. Varma*

Sustainable Technology Division, National Risk Management Research Laboratory, U. S. Environmental Protection Agency, MS 443, Cincinnati, Ohio 45268, USA. Fax: 513- 569-7677; Tel: 513-487-2701.

E-mail: varma.rajender@.epa.gov

Supporting Information

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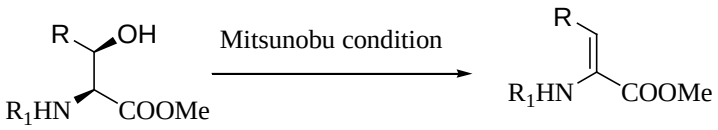


Scheme 1 dehydroamino acids

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}), and intensities of the peak are denoted as s (strong), w (weak), m (medium), broad (br). ^{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). CHN 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

(R)-tert-butyl-4-benzyl-1,2,3-oxathiazolidine-3-carboxylate 2,2-dioxide, (sulfamidate entry 9-13): Yield (87%) white solid; mp- 138^oC; $[\alpha]^{25}_D$ -41.2 (*c* = 0.7%, $CHCl_3$); I R (Neat), 2973(w), 2927(m), 1712(s), 1458(w), 1320(s), 1261(m), 1184(s), 1150(s), 1023(w), 838(m), 799(s), 784(m), 695(s), 657(m), 540(m), cm^{-1} ; ^1H 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.

(R)-tert-butyl-4-((S)-sec-butyl)-1,2,3-oxathiazolidine-3-carboxylate-2,2-dioxide, (sulfamidate entry 14-16)

Yield (91%) white solid; mp- 99^oC; $[\alpha]^{25}_D$ 2.16 (*c* = 0.5%, $CHCl_3$); I R (Neat), 2977(m), 2934(w), 1728(s), 1466(w), 1367(s), 1325(s), 1261(m), 1187(s), 1150(s), 1099(m), 968(m), 927(m), 826(s), 805(s), 653(s), 571(s), cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 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 procedure for the synthesis of triazole based unnatural amino acids.

1.0 mmol of sulfamidate, 1.2 mmol of NaN_3, 1.2 mmol of alkyne, 5 mol% of $CuSO_4$, 10 mol% of sodium ascorbate and 10 mg-Cu metal were placed in a crimp-sealed thick-walled glass tube equipped with a pressure sensor and a magnetic stirrer and 5 mL (*t*BuOH: H_2O) of solvent was added to the reaction mixture. The reaction tube was placed inside the cavity of a CEM Discover focused microwave synthesis system, operated at 120 ^oC (temperature monitored by a built-in infrared sensor), power 100 Watt and pressure 10–60 psi for 20-30 min min. After completion of the reaction 3-4 mL of saturated citric acid solution was added and stirs the reaction mixture for 5-10 min at room temperature. Dilute with water and extracted with ethyl acetate and purified by column chromatography. product were characterized using FT-IR, ^1H, and ^{13}C NMR and ele analysis.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-phenyl-1H-1,2,3-triazol-1-yl)butanoate, 1a : Yield (84%); white solid; mp- 149^oC; $[\alpha]^{25}_D$ 54.3 (*c* = 0.24% $CHCl_3$); I R (Neat), 3350 (m), 3154 (w), 1724(s), 1523(m), 1249(m), 1232(m), 1001(s), 971(s), 764(s), 692(s), cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 1.79 (3H, d, *J* = 6 Hz), 3.75 (3H, s), 4.81 (1H, dd, *J* = 4.5 Hz & 8.4 Hz), 5.11-5.17 (3H, m), 5.69 (1H, d, *J* = 6.6 Hz), 7.27-7.47 (8H, m), 7.74 (1H, s), 7.80-7.83 (2H, m); ^{13}C (75 MHz, $CDCl_3$) δ 16.61, 53.0, 58.2, 58.3, 67.4, 118.6, 125.7, 128.1, 128.2, 128.3, 128.5, 128.8, 130.1, 130.4; Analysis calculated for $C_{21}H_{22}N_4O_4$: C 63.95, H 5.62, N 14.20; Found C 63.97, H 5.61, N 14.18.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-(p-tolyl)-1H-1,2,3-triazol-1-yl)butanoate, 2a : Yield (85%); white solid; mp-138^oC; $[\alpha]^{25}_D$ 61.4 (*c* = 0.41%, $CHCl_3$); I R (Neat), 3349(w), 3155(w), 2858(m), 1719(s), 1478 (m), 1076(m), 998(s), 972(s), 820(s), 798(m), 723(w) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.78 (3H, d, *J* = 6.3Hz), 2.40 (3H, s), 3.73 (3H, s), 4.80 (1H, dd, *J* = 4.2 Hz, 8.4 Hz), 5.12-5.17 (3H, m), 5.70 (1H, d, *J* = 4.2), 7.23-7.37 (7H, m), 7.69-7.72 (3H, 8.1 Hz), 7.97 (1H, s); ^{13}C (75 MHz, $CDCl_3$) δ 16.6, 21.2, 52.9, 58.2, 58.7, 67.4, 118.2, 125.6, 128.1, 128.3, 128.5, 129.4, 130.6, 130.8, 138.0, 148.0, 155.7, 169.1; Analysis calculated for $C_{22}H_{24}N_4O_4$: C 64.69, H 5.92, N 13.72; Found C 64.67, H 5.89, N 13.76.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-(4-formylphenyl)-1H-1,2,3-triazol-1-yl)butanoate, 3a: ,Yield (81%); white solid; mp- 140^oC; $[\alpha]^{25}_D$ 60.2 (*c* = 0.22%, $CHCl_3$); I R (Neat), 3426(w), 3127(w), 2957(w), 2728(w), 1750(s), 1728(s), 1693(s), 1611(m), 1557(s), 1451(m), 1439(m), 1352(m), 1310(w), 1234(w), 1211(s), 1171(s), 1155(m), 1052(m), 985(m), 826(s), 751(s), 698(s), 673(m), cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 1.78 (3H, d, *J* = 6.9), 3.76 (3H, s), 4.84 (1H, dd, *J* = 4.5 Hz, 8.4 Hz) 5.04-5.37 (3H, m), 5.74 (1H, d, *J* = 6.9), 7.32 (5H, bs), 7.92 (1H, s), 7.95 (2H, d, *J* = 6.3 Hz), 7.98 (2H, *J* = 6.3 Hz), 10.03 (1H, s); ^{13}C (75 MHz, $CDCl_3$)

δ 16.4, 53.08, 58.2, 58.3, 67.4, 120.0, 126.0, 128.1, 128.3, 128.5, 130.3, 135.8, 136.2, 146.2, 155.6, 169.0, 191.6; Analysis calculated for $C_{22}H_{22}N_4O_5$: C 62.55, H 5.25, N 13.26; Found C 62.57, H 5.23, N 13.24.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-(4-nitrophenyl)-1H-1,2,3-triazol-1-yl)butanoate, 4a: Yield (76%); white solid; mp- 167^{o}C; $[\alpha]^{25}_D$ 45.1 (*c* = 0.16%, $CHCl_3$); I R (Neat), 3450 (w), 3126(w), 1752(m), 1728(s), 1609(w), 1515(s), 1352(s), 1336(m), 1210(m), 1173(m), 1053(m), 1210(m), 857(m), 751(s), 699(s), 680(m), cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.78 (3H, d, 7.2 Hz), 3.79 (3H, s), 4.84 (1H, dd, *J* = 4.5 Hz, 8.1 Hz), 5.05-5.25 (3H, m), 5.66 (1H, d, *J* = 5.7), 7.35 (5H, bs), 7.88 (1H, s), 7.97 (2H, d, *J* = 9 Hz), 8.30 (2H, d, *J* = 9 Hz); ^{13}C (75 MHz, $CDCl_3$) δ 16.5, 53.1, 58.2, 58.4, 67.5, 120.3, 124.2, 126.1, 128.1, 128.4, 128.6, 136.6, 145.4, 147.4, 154.0, 168.9; Analysis calculated for $C_{21}H_{21}N_5O_6$: C 57.40, H 4.82, N 15.94; Found C 57.38, H 4.84, N 15.91.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-(pyridin-2-yl)-1H-1,2,3-triazol-1-yl)butanoate, **5a: ,**Yield (79 %); white solid; mp- 155^{o}C; $[\alpha]^{25}_D$ 55.3 (*c* = 0.25%, $CHCl_3$); I R (Neat), 3235(m), 3131(m), 3059(w), 1745(s), 1750(s), 1725(s), 1559(m), 1555(s), 1472(m), 1355(s), 1314(s), 1254(s), 1215(s), 1191(m), 1083(s), 1066(s), 1000(s), 970(s), 971(m), 734(m), 725(s), 697(s), 697(s), 674(s), 619(m), cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.77 (3H, d, *J* = 6.9), 3.74 (3H, s), 4.83 (1H, dd, *J* = 4.2, 8.4), 5.09-5.22 (3H, m), 5.78 (1H, d, *J* = 6.3) 7.21-7.32 (6H, m), 7.75-7.80 (1H, m), 8.16-8.19 (2H, m), 8.57-8.59 (1H, m); ^{13}C (75 MHz, $CDCl_3$) δ 16.5, 52.9, 58.31, 58.38, 67.4, 120.2, 121.1, 122.8, 128.1, 128.2, 128.5, 136.8, 148.2, 149.3, 150.1, 155.7, 169.0; Analysis calculated for $C_{20}H_{21}N_5O_4$; C 60.75, H 5.35, N 15.46; Found C 60.73, H 5.33, N 15.44.

(2S,3S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-(2-hydroxyethyl)-1H-1,2,3-triazol-1-yl)butanoate, 6a: ,Yield (86%); white solid; mp- 98^{o}C; $[\alpha]^{25}_D$ 45.5 (*c* = 0.24%, $CHCl_3$); I R (Neat) 3450(w), 3316(s), 3040(w), 2901(w), 2827(s), 1742(s), 1696(s), 1654(m), 1335(s), 1491(s), 1436(s), 1535(s), 1289(s), 1223(s), 1063(s), 1223(s), 1063(s), 1009(m), 736(m), 594(s), cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.68 (3H, d, *J* = 7.2), 2.92 (2H, t, *J* = 6 Hz), 3.74 (3H, s), 3.89 (2H, t, *J* = 6 Hz), 4.75 (1H, dd, *J* = 4.2, 8.4), 5.06-5.15 (3H, m), 5.75 (1H, d, *J* = 8.4) 7.36 (5H, bs), 7.40 (1H, s); ^{13}C (75 MHz, $CDCl_3$) δ 15.7, 28.8, 53.0, 57.9, 58.3, 61.6, 67.4, 120.9, 128.1, 128.3, 128.5, 135.5, 135.7, 145.3, 155.8, 169.15; Analysis calculated for $C_{17}H_{22}N_4O_5$: C 56.34, H 6.12, N 15.46;Found C 56.32, H 6.10, N 15.43.

(S)-methyl-2-(((benzyloxy)carbonyl)amino)-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propanoate, 7a: ,Yield (85%); white solid; mp- 185^{o}C; $[\alpha]^{25}_D$ 50.1 (*c* = 0.14%, $CHCl_3$); I R (Neat), 3337(s), 3062(w), 2958(s), 1531(s), 1453(w), 1439(s), 1766(w), 1348(s), 1312(s), 1270(s), 1221(s), 1173(s), 1147(s), 1076(w), 1069(m), 1047(m), 997(m), 971(s), 930(m), 903(m), cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.81 (3H, s), 4.81-.498 (3H, m), 5.13 (1H, d, *J* = 12.3), 5.18 (1H, d, *J* = 12.3) 7.33-7.46 (8H, m), 7.66 (1H, m), 7.77-7.80 (2H, m); ^{13}C (75 MHz, $CDCl_3$) δ 50.7, 53.2,

54.1, 67.3, 120.7, 125.7, 128.1, 128.3, 128.5, 128.8, 130.2, 135.9, 147.8, 155.7, 169.1; Analysis calculated for $C_{20}H_{20}N_4O_4$: C 63.15, H 5.30, N 14.73;Found C 63.12, H 5.28, N 14.71.

(S)-methyl 2-(((benzyloxy)carbonyl)amino)-3-(4-(4-chlorophenyl)-1H-1,2,3-triazol-1-yl)propanoate, 8a: ,Yield (83%); white solid; mp- 202^oC; $[\alpha]^{25}_D$ 64.8 (*c* = 0.2%, $CHCl_3$); I R (Neat), 3331(s), 3144(w), 3047(w), 2958(w), 1733(s), 1682(s), 1532(m), 1484(m), 1453(s), 1439(s), 1367(s), 1146(m), 971(m), 938(m), 875(m), 838(m), 798(m), cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 3.82 (3H, s), 4.77-4.98 (3H, m), 5.13 (1H, d, *J* = 12.3 Hz), 5.18 (1H, d, *J* = 12.3 Hz), 7.36-7.41 (7H, m), 7.64 (1H, s), 7.71 (2H, d, *J* = 8.4); ^{13}C (75 MHz, $CDCl_3$) δ 50.7, 53.2, 54.1, 67.4, 120.8, 127.0, 128.1, 128.4, 128.6, 128.7, 129.0, 134.11, 135.8, 146.7, 155.6, 169.0; Analysis calculated for $C_{20}H_{19}ClN_4O_4$: C 57.90, H 4.62, N 13.51; Found C 57.92, H 4.59, N 13.53.

(S)-tert-butyl (1-phenyl-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-yl)carbamate, 9a: ,Yield (84%); white solid; mp- 169^oC; $[\alpha]^{25}_D$ -11.9 (*c* = 0.21%, $CHCl_3$); I R (Neat), 3386(s), 3144(w), 3027(w), 2978(w), 2931(s), 1698(s), 1515(s), 1455(m), 1444(m), 1364(m), 1247(s), 1169(s), 1015(m), 980(w), 882(w), 914(w), 885(s), 823(m), cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 1.39 (9H, s), 2.79-2.97 (2H, m), 4.23-4.34 (1H, m), 4.40-4.59 (2H, m), 4.94 (1H, d, *J* = 6.9), 7.19-7.37 (8H, m), 7.76 (1H, s), 7.82-7.85 (2H, m); ^{13}C (75 MHz, $CDCl_3$) δ 28.2, 29.7, 38.0, 52.0, 79.9, 120.8, 125.6, 126.9, 128.1, 128.7, 128.8, 129.3, 130.5, 136.7, 147.6, 155.2; Analysis calculated for $C_{25}H_{24}N_4O_2$: C 72.80, H 5.86, N 13.58; Found C 72.78, H 5.83, N 13.55.

(S)-tert-butyl (1-(4-(4-methoxyphenyl)-1H-1,2,3-triazol-1-yl)-3-phenylpropan-2-yl)carbamate , 10a: ,Yield (85%); white solid; mp- 189^oC; $[\alpha]^{25}_D$ 8.3 (*c* = 0.34%, $CHCl_3$); I R (Neat), 3365(s), 2981(m), 1686(s), 1522(s), 1499(s), 1455(m), 1444(m), 1367(m), 1357(m), 1276(m), 1247(s), 1171(s), 1058(m0, 1029(s), 976(s), 856(m), 834(s), 797(s), 755(s), cm^{-1} ;^1H NMR (300 MHz, $CDCl_3$) δ 1.39 (9H, s), 2.78-2.96 (2H, m), 3.85 (3H, s), 4.22-4.35 (1H, m), 4.43-4.58 (2H, m), 4.90 (1H, bs); 6.97(2H, d, *J* = 8.7 Hz), 7.23-7.37 (5H, m), 7.67 (1H, s), 7.76 (2H, d, *J* = 8.7 Hz); ^{13}C (75 MHz, $CDCl_3$) δ 27.7, 28.2, 37.9, 52.0, 55.3, 79.9, 114.3, 120.0, 123.2, 126.9, 128.7, 129.3, 136.7, 147.5, 155.2, 159.6; Analysis calculated for $C_{23}H_{28}N_4O_3$: C 67.63, H 6.91, N 13.72; Found C 67.61, H 6.88, N 13.69.

(S)-tert-butyl (1-(4-(4-nitrophenyl)-1H-1,2,3-triazol-1-yl)-3-phenylpropan-2-yl)carbamate , 11a: ,Yield (71%); white solid; mp- 186^oC; $[\alpha]^{25}_D$ 23.1 (*c* = 0.2%, $CHCl_3$); I R (Neat), 3367(m), 3137(w), 3060(w), 2982(w), 2933(w), 1686(s), 1607(s), 1516(s), 1339(s), 1274(m), 1248(s), 1167(s), 1110(m), 1060(m), 1045(m), 853(s), 754(s), 700(s), 629(s), cm^{-1} ;^1H NMR (300 MHz, $CDCl_3$) δ 1.39 (9H, s), 2.84-.3.00 (2H, m), 4.25-4.36 (1H, m), 4.50-4.65 (2H, m), 4.74 (1H, bs), 7.25-7.39 (5H, m), 7.92 (1H, s) 8.00 (2H, d, *J* = 9 Hz), 8.31 (2H, d, *J* = 9 Hz); ^{13}C (75 MHz, $CDCl_3$) δ 28.2, 29.6, 38.1, 52.0, 60.3, 80.2, 122.2, 124.3, 126.0, 127.1, 128.8, 129.2, 136.3, 136.3, 136.7, 145.5, 147.3, 155.2; Analysis calculated for $C_{22}H_{25}N_5O_4$: C 62.40, H 5.95, N 16.54; Found C 62.38, H 5.92, N 16.57.

(S)-tert-butyl (1-phenyl-3-(4-(p-tolyl)-1H-1,2,3-triazol-1-yl)propan-2-yl)carbamate, 12a: ,Yield (85%); white solid; mp- 182^{o}C; $[\alpha]^{25}_{D}$ 2.1 (*c* = 0.25%, $CHCl_3$); I R (Neat), 3372(s), 3128(w), 3066(w), 2981(m), 2936(m), 1720(S), 1686(s), 1545(m), 1518(s), 1495(m), 1457(m), 1446(m), 1390(m), 1366(m), 1218(s), 1168(s), 1059(m), 1025(m), 1042(m), 910(m), 835(m), 843(m), 776(s), 730(s), 700(s), cm^{-1} ;^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40 (9H, s), 2.40 (3H, s), 2.79-2.96 (2H, m), 4.22-4.34 (1H, m), 4.44-4.59 (2H, m), 4.87 (1H, bs), 7.24-7.38 (7H, m), 7.71 (1H, s), 7.73 (2H, d, *J* = 5.4 Hz) ; ^{13}C (75 MHz, $CDCl_3$) δ 21.2, 28.2, 31.2, 37.9, 52.0, 80.0, 120.4, 125.6, 126.9, 127.6, 128.7, 129.3, 129.5, 136.7, 138.0, 147.7, 155.2; Analysis calculated for $C_{23}H_{28}N_4O_2$: C 70.38, H 7.19, N 14.27; Found C 70.341, H 7.21, N 14.25.

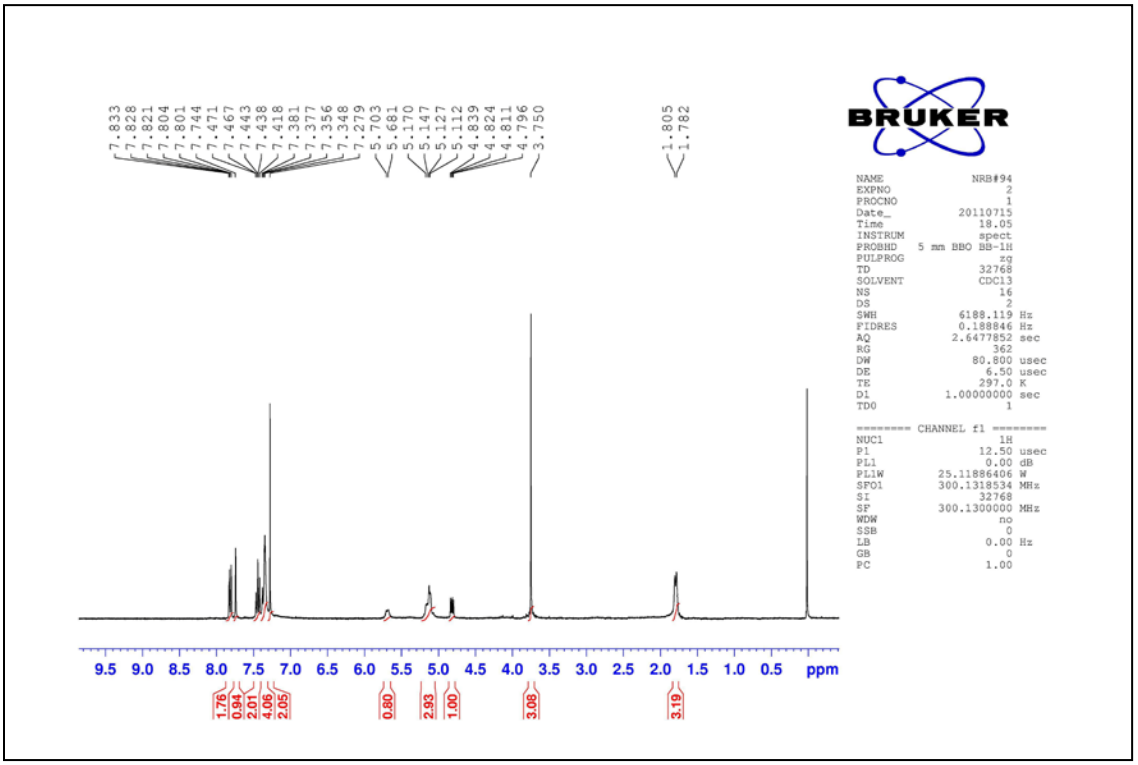
(S)-ethyl 1-(2-((tert-butoxycarbonyl)amino)-3-phenylpropyl)-1H-1,2,3-triazole-4-carboxylate, 13a: ,Yield (79%); white solid; mp- 145^{o}C; $[\alpha]^{25}_{D}$ -7.29 (*c* = 0.38%, $CHCl_3$); I R (Neat)- 3369(m), 3945(w), 2980(m), 2928(m), 1687(s), 1521(s), 1448(s), 1359(s), 1250(s), 1168(s), 1045(s), 856(m), 796(s), 756(s), 702(s), 626(s), cm^{-1} ;^{1}H NMR (300 MHz, $CDCl_3$) δ 1.37 (9H, s), 1.40 (3H, t, *J* = 7.2 Hz), 2.79-2.94 (2H, m), 4.16-4.27 (1H, m), 4.41 (2H, q, *J* = 7.2 Hz), 4.53-4.67 (2H, m), 7.75(1H, d, *J* = 7.2 Hz), 7.19-7.35 (5H, m); ^{13}C (75 MHz, $CDCl_3$) δ 14.2, 28.2, 31.2, 37.9, 52.1, 61,2, 80.1, 127.0, 128.5, 128.8, 129.2, 136.3, 140.1,155.1, 160.6; Analysis calculated for $C_{19}H_{26}N_4O_4$: C 60.95, H 7.00, N 14.96; Found C 60.92, H 7.02, N 14.98.

tert-butyl ((2R,3S)-3-methyl-1-(4-phenyl-1H-1,2,3-triazol-1-yl)pentan-2-yl)carbamate, 14a: ,Yield (85%); white solid; mp- 156^{o}C; $[\alpha]^{25}_{D}$ 16.9 (*c* = 0.23%, $CHCl_3$); I R (Neat), 3364(m), 3129(w), 2967(m), 2940(m), 2877(w), 1686(s), 1518(s), 1464(m), 1449(m), 1366(m), 1277(m), 1252(m), 1231(m), 1177(m), 1252(m), 1231(m), 1172(s), 1157(s), 1082(w), 1045(w), 977(m), 855(m), 778(m), 764(s), 739(m), 710(m), 694(s), cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.94 (3H, t, *J* = 7.5 Hz), 1.03 (3H, d, *J* = 6.6) 1.12-1.27 (1H, m), 1.36 (9H, s), 1.49-1.69 (2H, m), 3.89-3.98 (1H, m), 4.54 (2H, bd, *J* = 5.1), 4.75 (1H, d, *J* = 8.7) ,7.28-7.44 (3H, m), 7.81-7.84 (3H, m); ^{13}C (75 MHz, $CDCl_3$) δ 11.2, 15.6, 24.9, 28.2, 36.3, 51.4, 54.9, 79.7, 120.3, 125.6, 128.0, 128.7, 130.6, 147.7, 155.5; Analysis calculated for $C_{19}H_{28}N_4O_2$: C 66.25, H 8.19, N 16.27; Found C 66.22, H 8.17, N 16.25.

tert-butyl ((2R,3S)-1-(4-(4-methoxyphenyl)-1H-1,2,3-triazol-1-yl)-3-methylpentan-2-yl)carbamate , 15a: ,Yield (86%); white solid; mp- 160^{o}C; $[\alpha]^{25}_{D}$ 14.9 (*c* = 0.32%, $CHCl_3$); I R (Neat), 3351(m), 3147(w), 2967(m), 2936(m), 2876(w), 1682(s), 1619(w), 1523(s), 1457(s), 1170(s), 1026(s), 976(w), 835(m), 804(m), 743(m), 648(s), 612(m), cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.93 (3H, J = 7.2 Hz), 1.03 (3H, d, J = 6.6), 1.21-1.28 (1H, m), 1.37 (9H, s), 1.48-1.65 (2H, m), 3.84 (3H, s), 3.87-3.95 (1H, m), 5.52 (2H, d, *J* = 5.4), 4.74 (1H, d, *J* = 8.7 Hz), 6.96 (2H, d, *J* = 9 Hz), 7.35 (1H, s), 7.75 (2H, d, *J* = 9 Hz); ^{13}C (75 MHz, $CDCl_3$) δ 11.2, 15.6, 24.9, 28.2, 36.3, 51.3, 54.9, 55.3, 79.7, 114.2, 119.5, 123.3, 126.9, 147.6, 155.5, 159.5; Analysis calculated for $C_{20}H_{30}N_4O_3$: C 64.15, H 8.07, N 14.96; Found C 64.13, H 8.04, N 14.93.

tert-butyl-((2R,3S)-3-methyl-1-(4-(4-nitrophenyl)-1H-1,2,3-triazol-1-yl)pentan-2-yl)carbamate, 16a: Yield (72%); white solid; mp- 183^{o}C; $[\alpha]^{25}_{D}$ 37.5 (*c* = 0.18%, $CHCl_3$); I R

(Neat), 3344(m), 2960(m), 2931(m), 2878(w), 1676(s), 1608(s), 1512(s), 1459(m), 1443(m), 1353(s), 1341(s), 1276(m), 1231(m), 1156(s), 1109(m), 1058(w), 754(s), 690(m), 657(s).cm^{-1} ; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.96 (3H, t, J = 7.2 Hz), 1.05 (3H, d, J = 6.6 Hz), 1.18-1.28 (1H, m), 1.34 (9H, s), 1.55-1.71 (2H, m), 3.93-4.02 (1H, m), 4.49-4.69 (3H, m), 7.99 (2H, d, J = 9 Hz), 8.01 (1H, s), 8.29 (2H, d, J = 9 Hz); ^{13}C (75 MHz, $CDCl_3$) δ 11.2, 15.6, 25.0, 28.1, 36.6, 51.0, 54.9, 79.9, 121.9, 124.2, 126.0, 136.9, 145.5, 147.3, 155.5; Analysis calculated for $C_{19}H_{27}N_5O_4$: C 58.60, H 6.99, N 17.98; Found. C 58.63, H 6.96, N 17.95.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5.170
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EXPNO 2
PROCNO 1
Date_ 20110715
Time 18.05
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SOLVENT CDCl3
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GB 0
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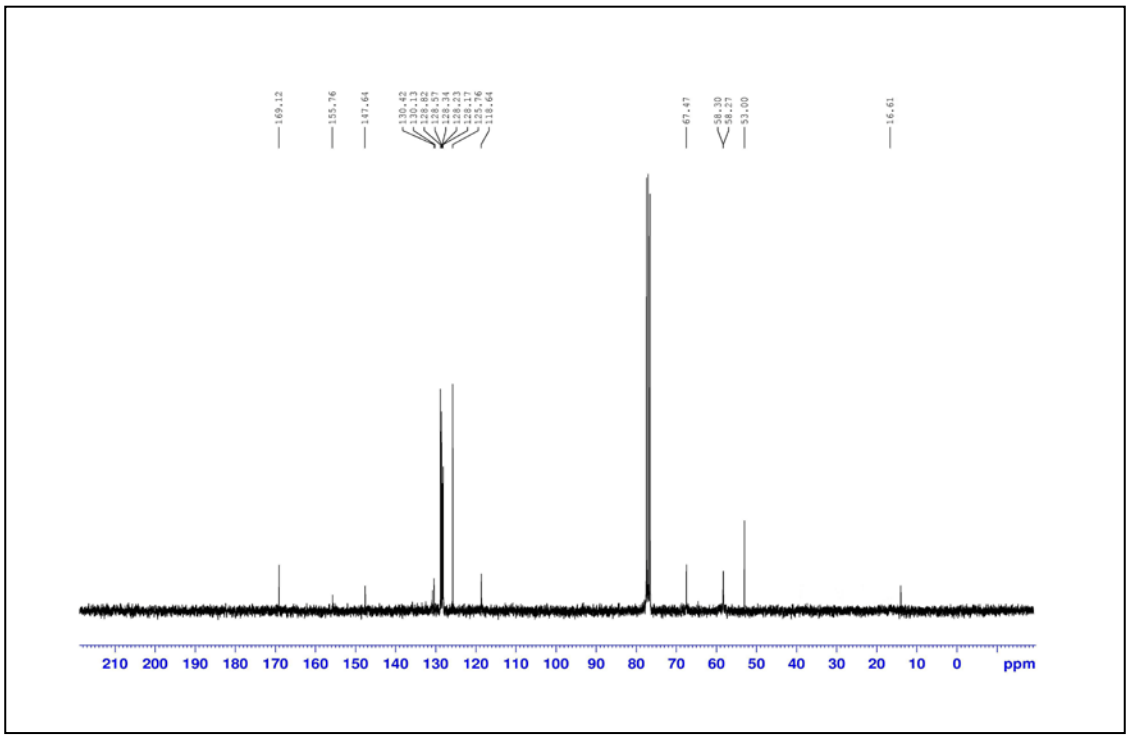
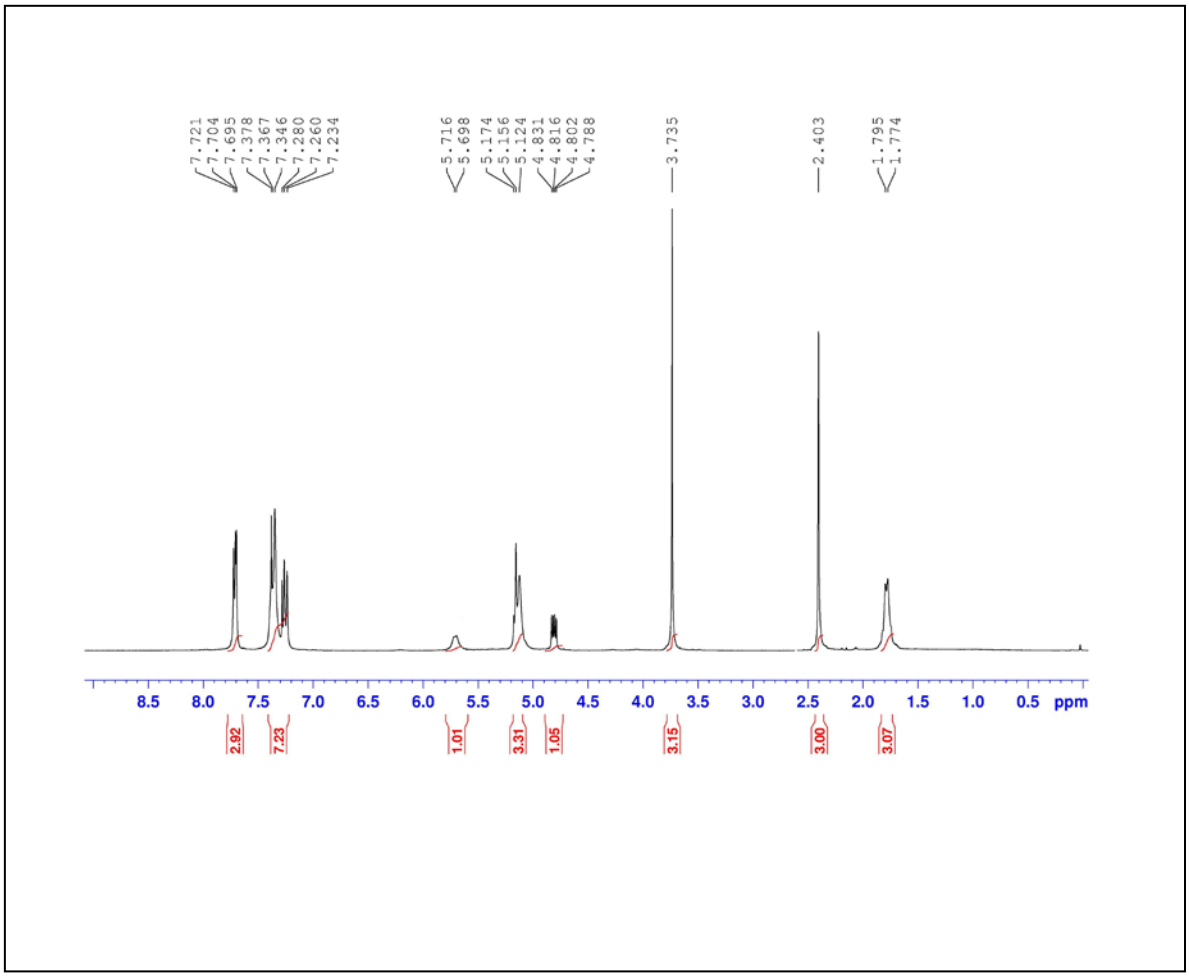
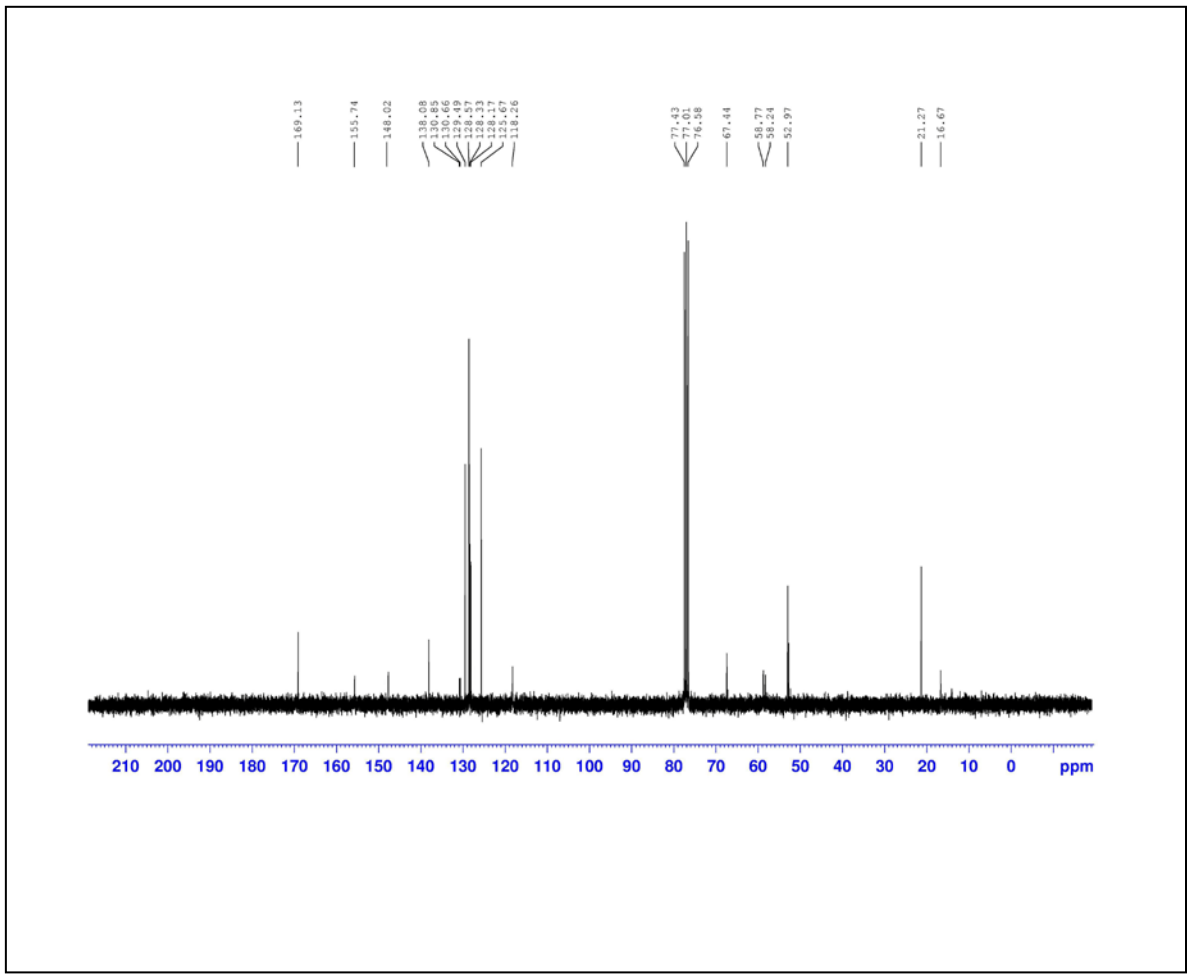
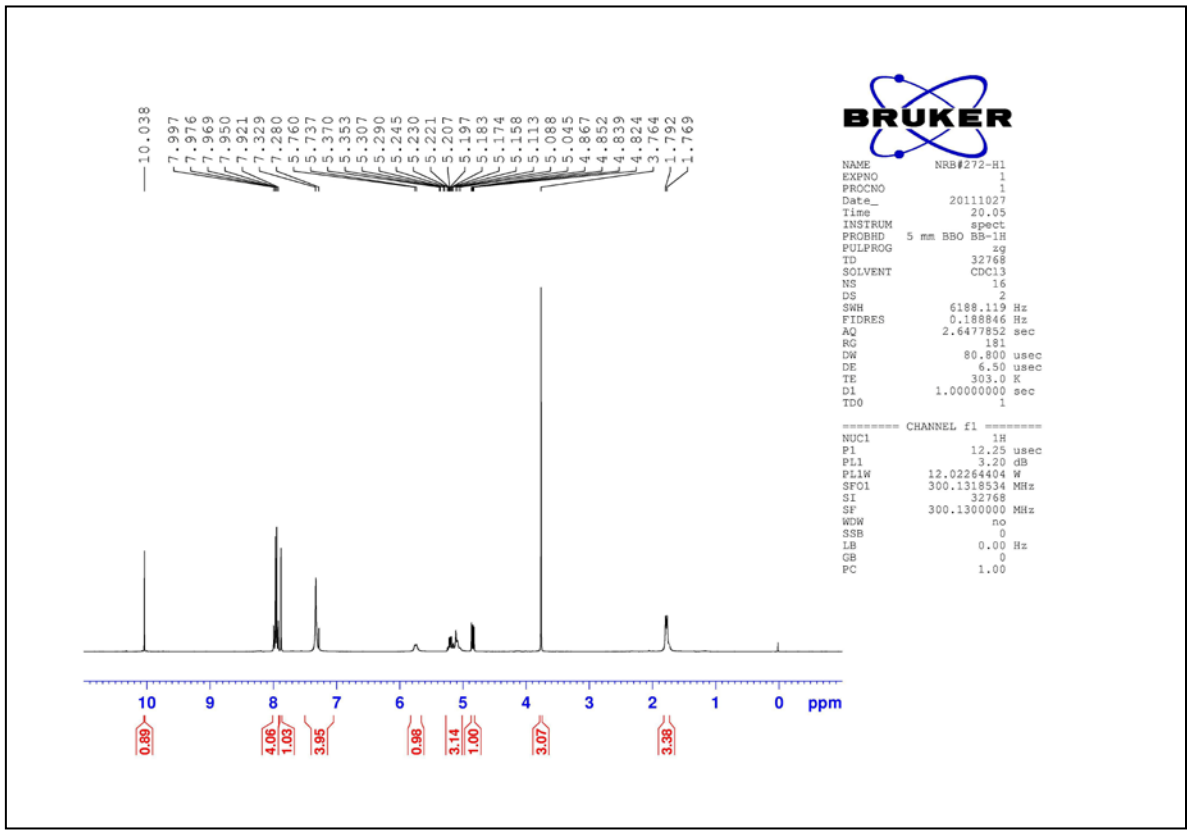
1.00
3.08
3.19





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5.207
5.197
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PROCNO 1
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Time 20.05
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PULPROG zg
TD 32768
SOLVENT CDCl3
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FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 181
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DE 6.50 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
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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
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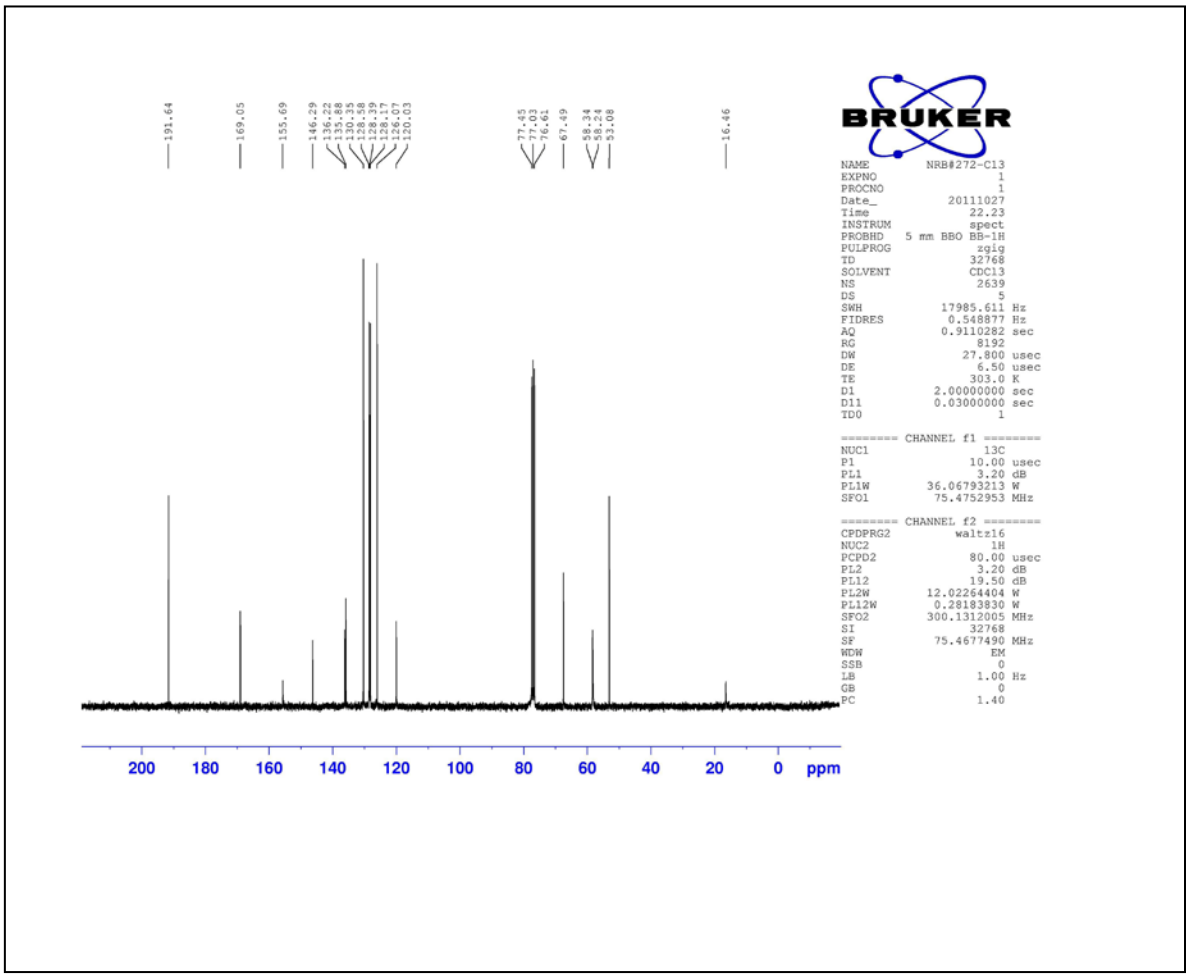
3.38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126.07
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77.03
76.61
67.49
58.34
58.24
53.08
16.46
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EXPNO 1
PROCNO 1
Date_ 20111027
Time 22.23
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PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 2639
DS 5
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
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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
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GB 0
PC 1.40
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120
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80
60
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20
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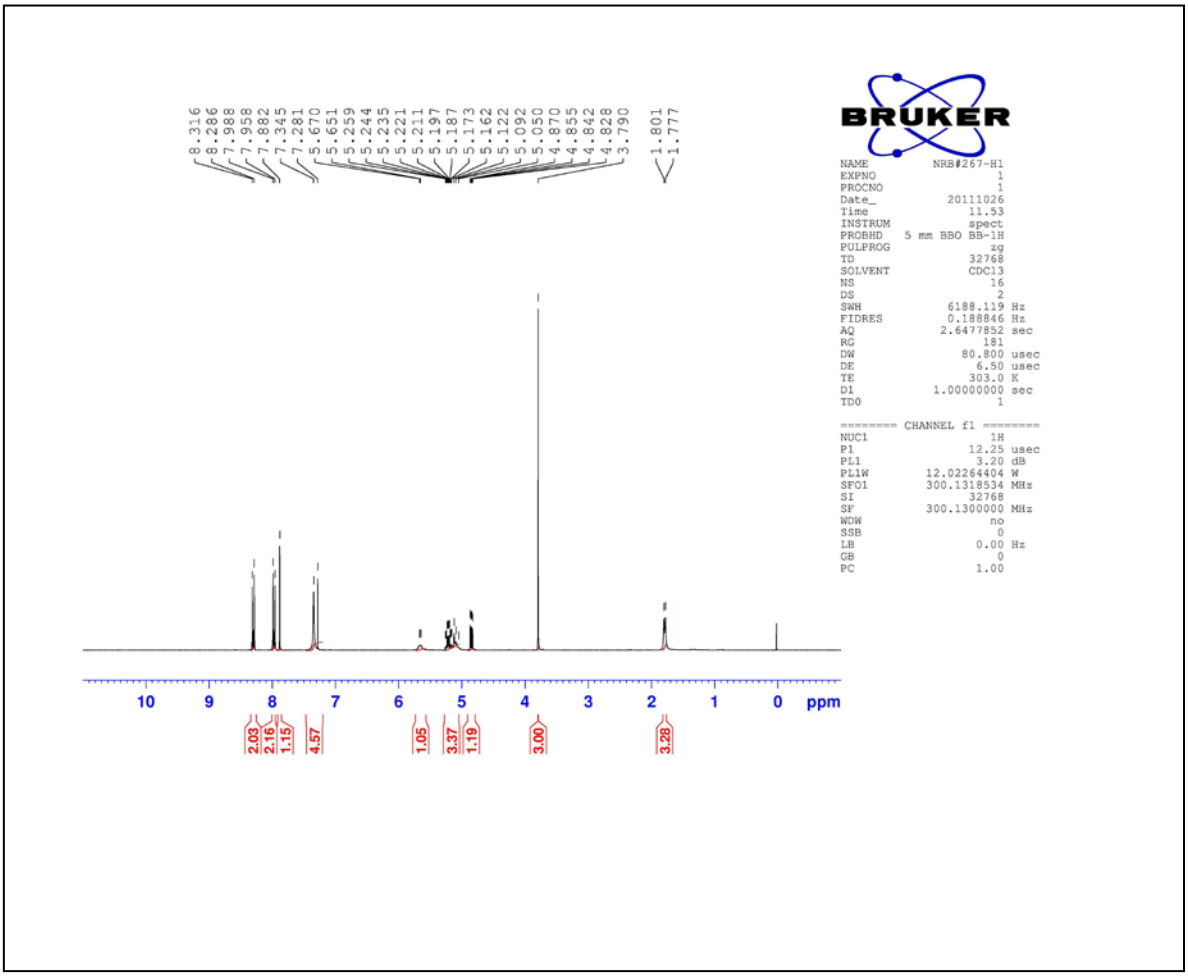
ppm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1.777
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PROCNO 1
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PULPROG zg
TD 32768
SOLVENT CDCl3
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FIDRES 0.188846 Hz
AQ 2.6477852 sec
RG 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
10
9
8
7
6
5
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3
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ppm
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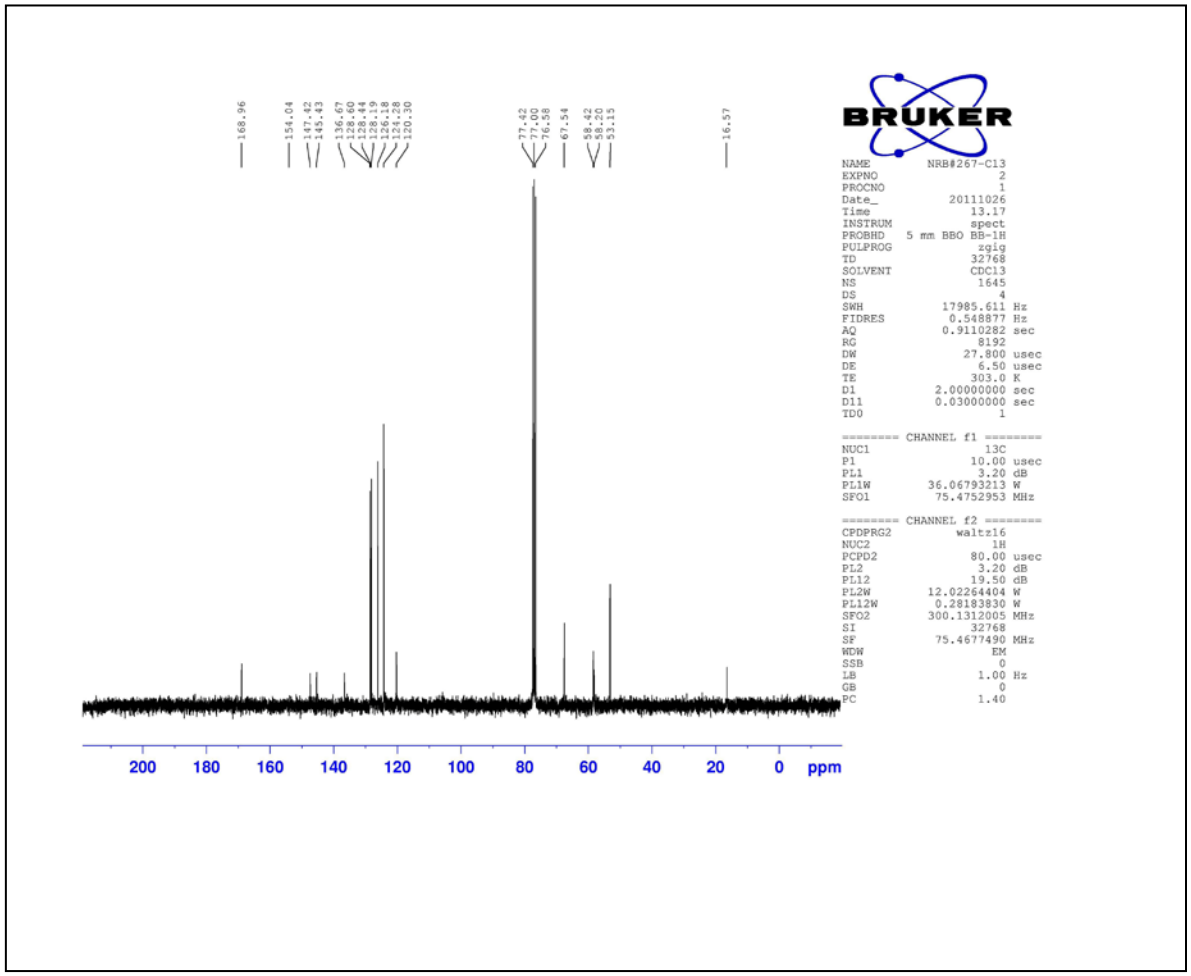
3.28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76.58
67.54
58.42
58.20
53.15
16.57
200
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160
140
120
100
80
60
40
20
0
ppm
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PROCNO 1
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PULPROG zgig
TD 32768
SOLVENT CDCl3
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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
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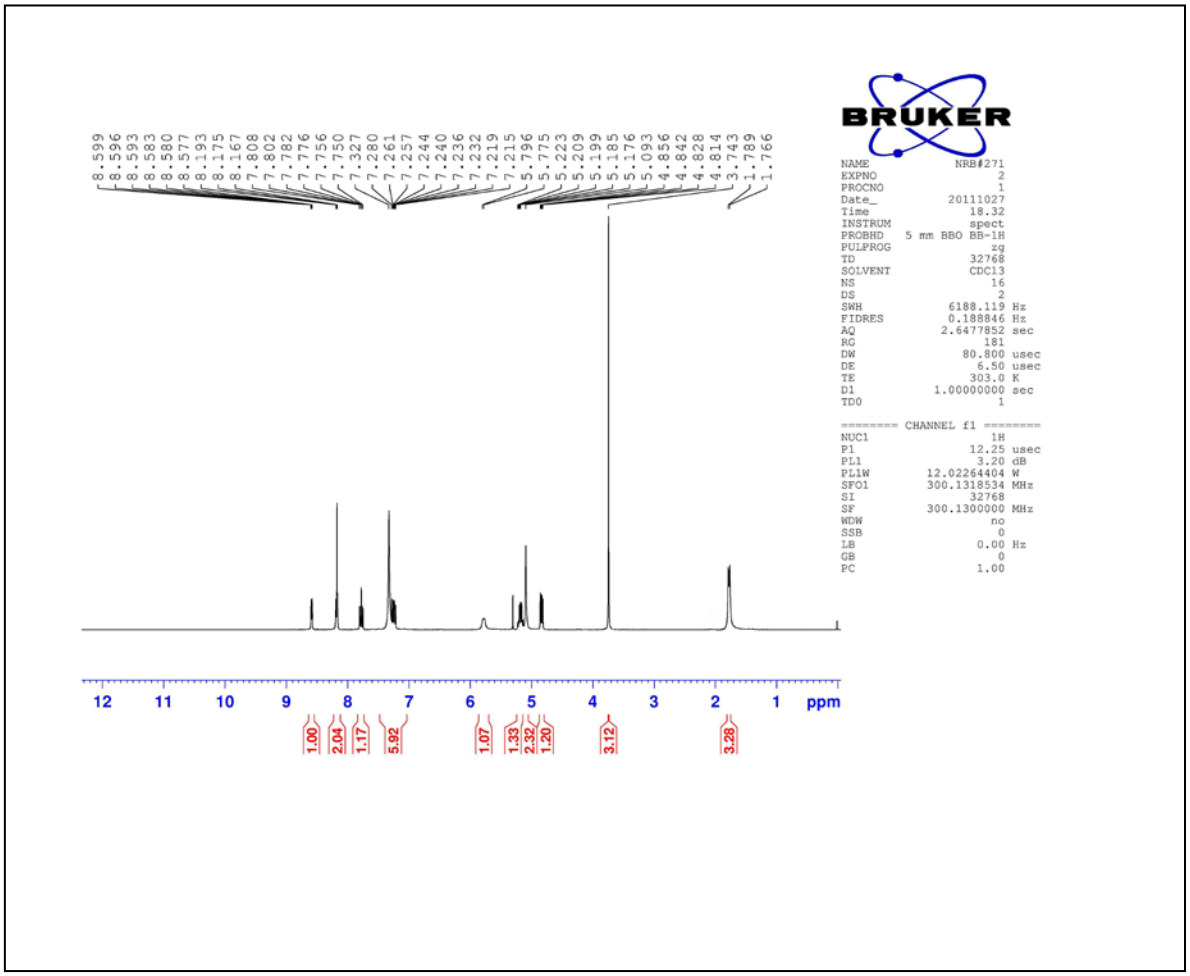
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5.223
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PROCNO 1
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Time 18.32
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PULPROG zg
TD 32768
SOLVENT CDC13
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FIDRES 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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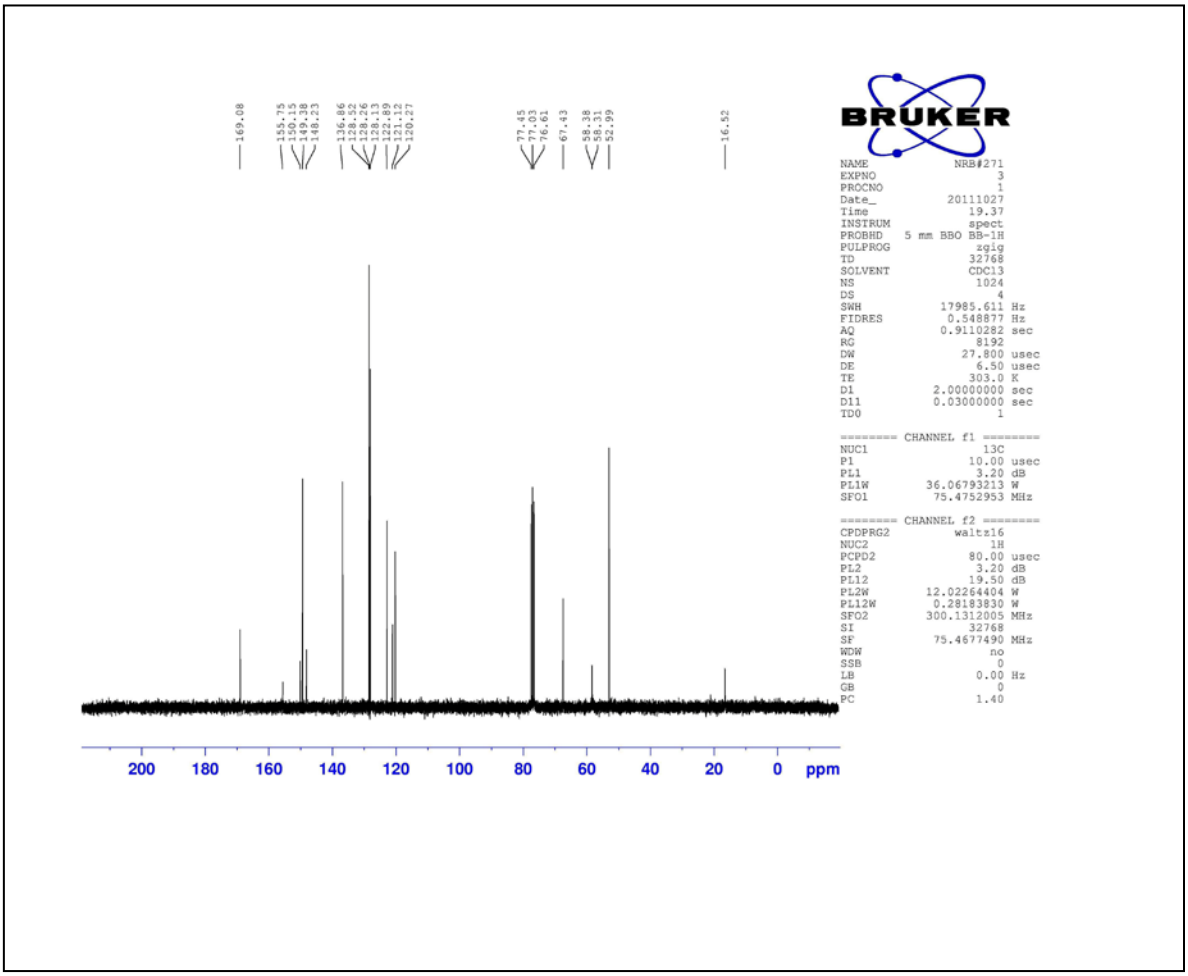
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76.61
67.43
58.38
58.31
52.99
16.52
200
180
160
140
120
100
80
60
40
20
0
ppm
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NAME NRB#271
EXPNO 3
PROCNO 1
Date_ 20111027
Time 19.37
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PULPROG 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
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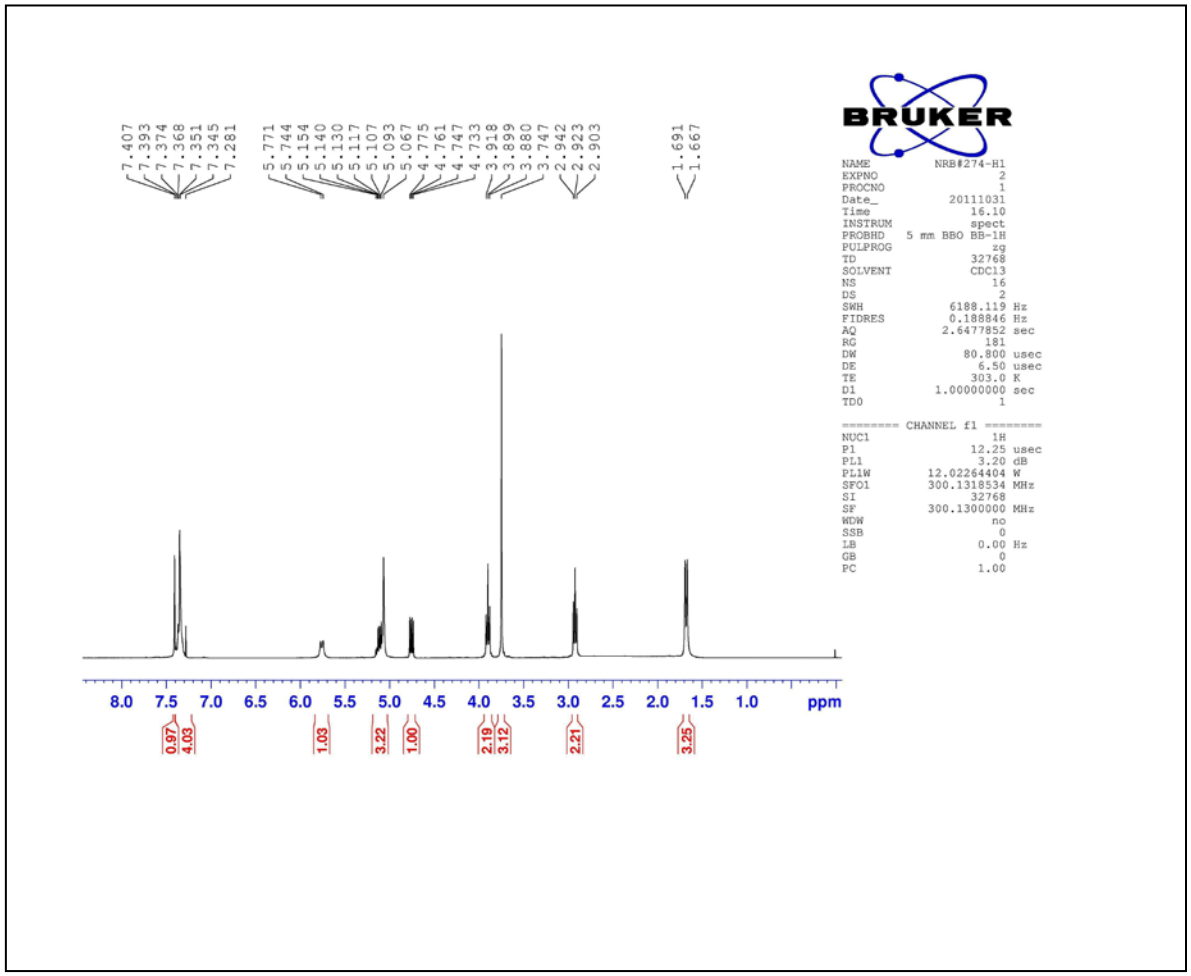
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3.747
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EXPNO 2
PROCNO 1
Date_ 20111031
Time 16.10
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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477852 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
8.0 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
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1.00
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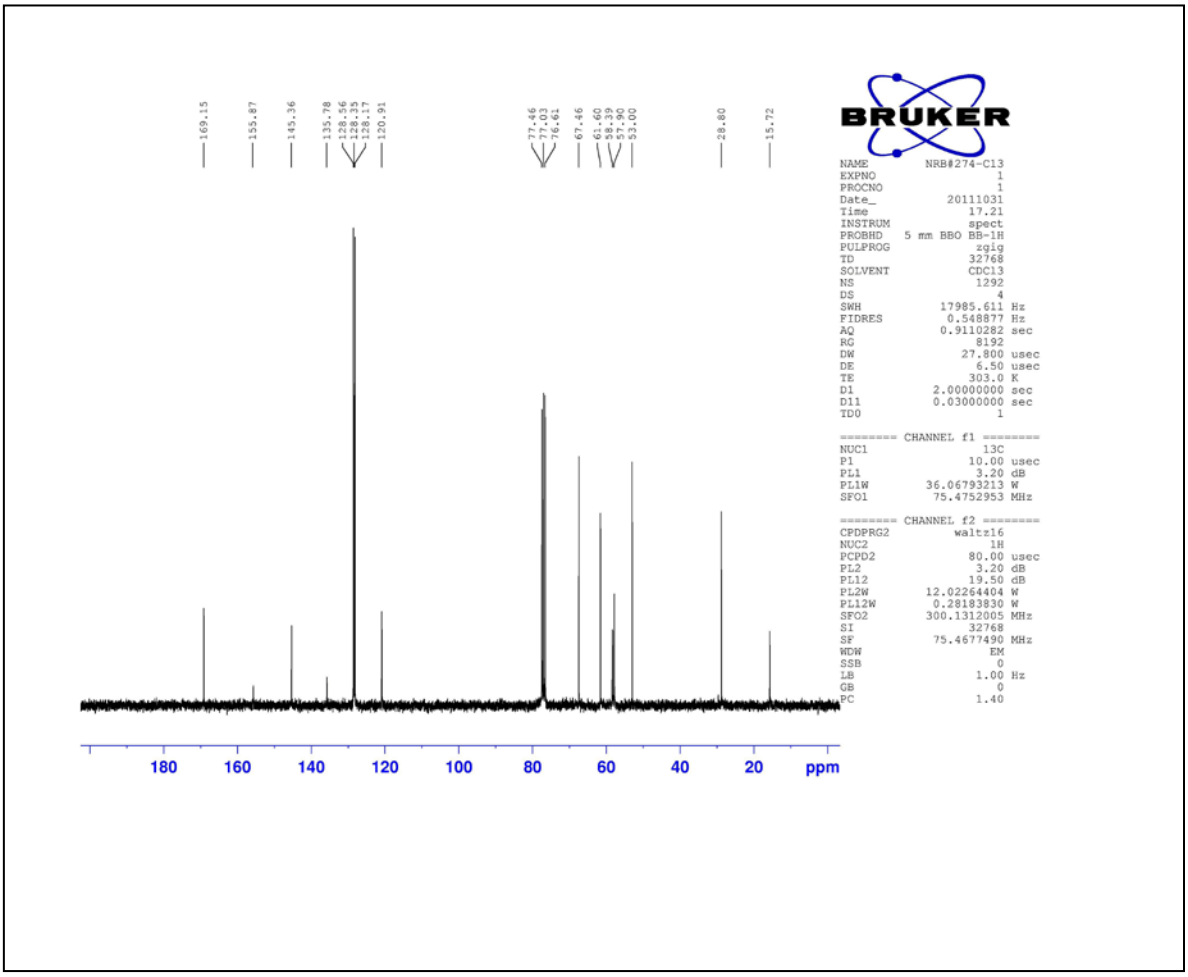
2.21
3.25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67.46
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59.39
57.80
53.00
28.80
15.72
180
160
140
120
100
80
60
40
20
ppm
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EXPNO 1
PROCNO 1
Date_ 20111031
Time 17.21
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PULPROG zgig
TD 32768
SOLVENT CDCl3
NS 1292
DS 4
SWH 17985.611 Hz
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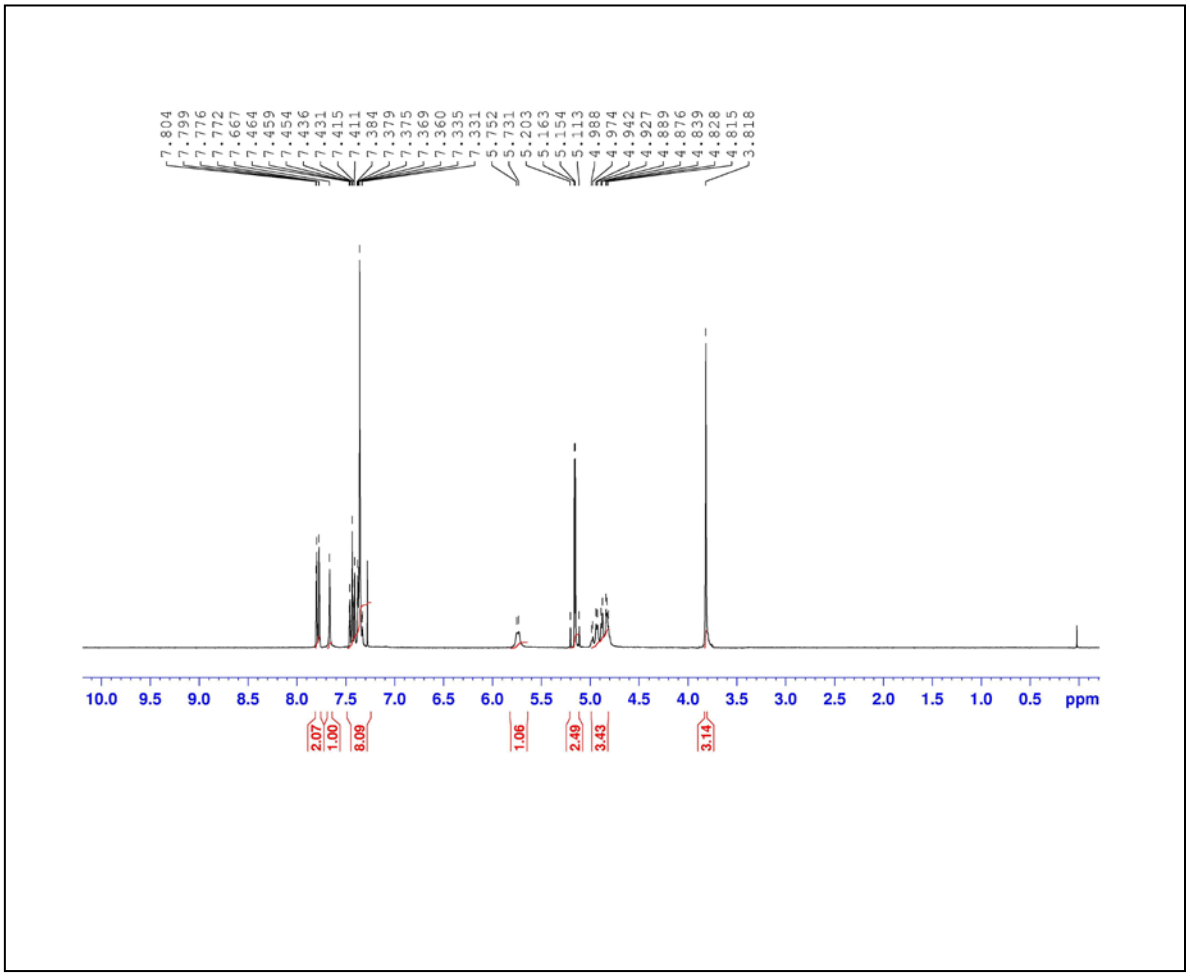
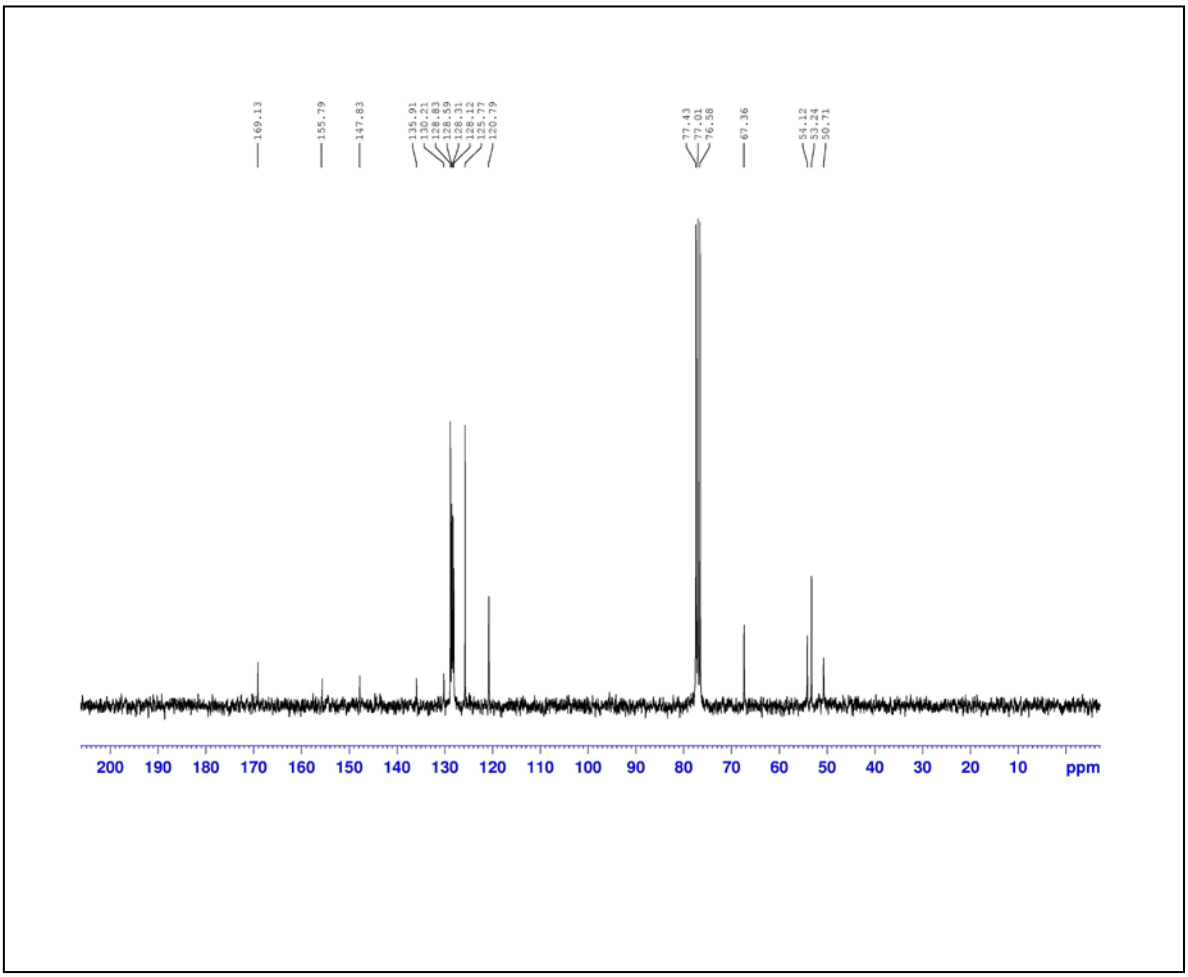
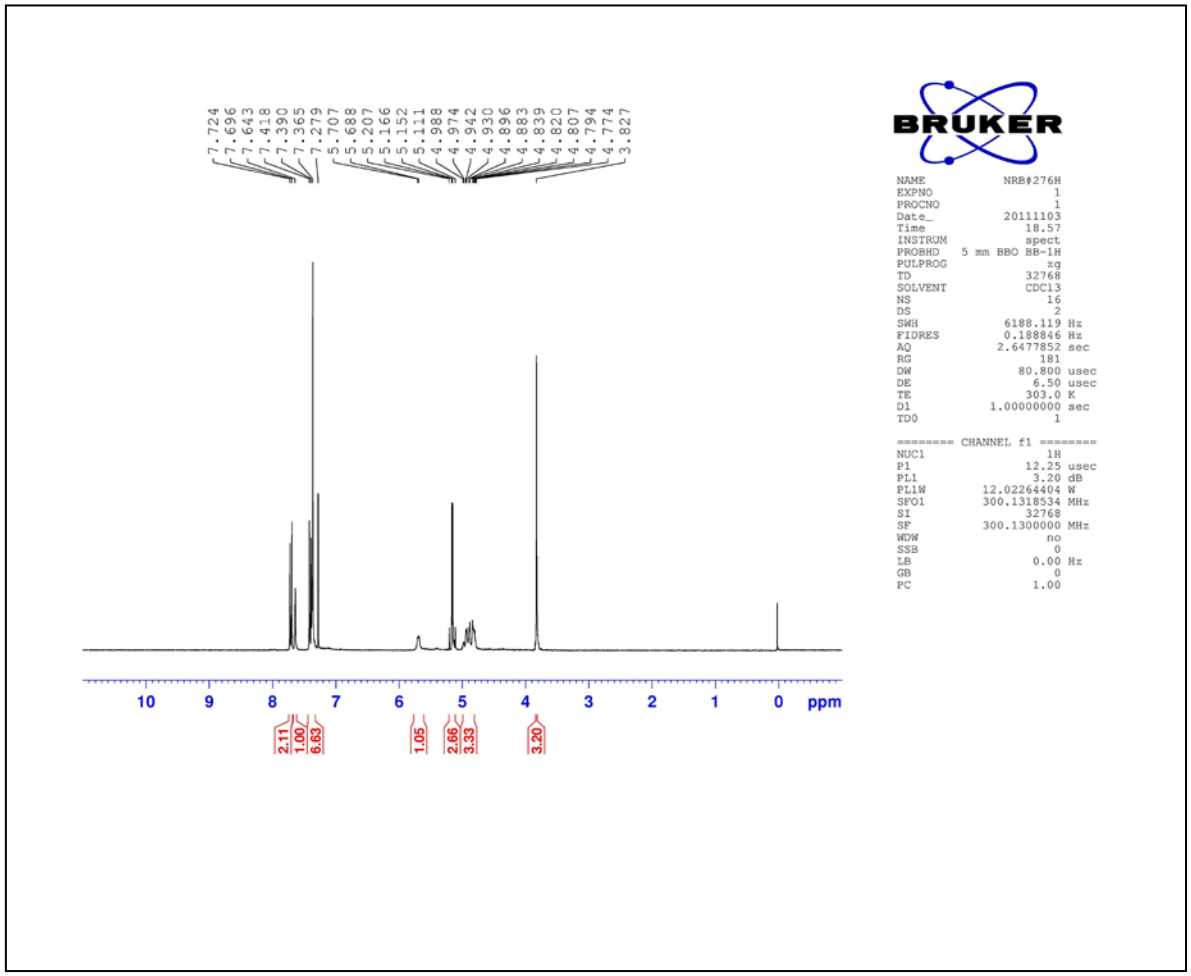
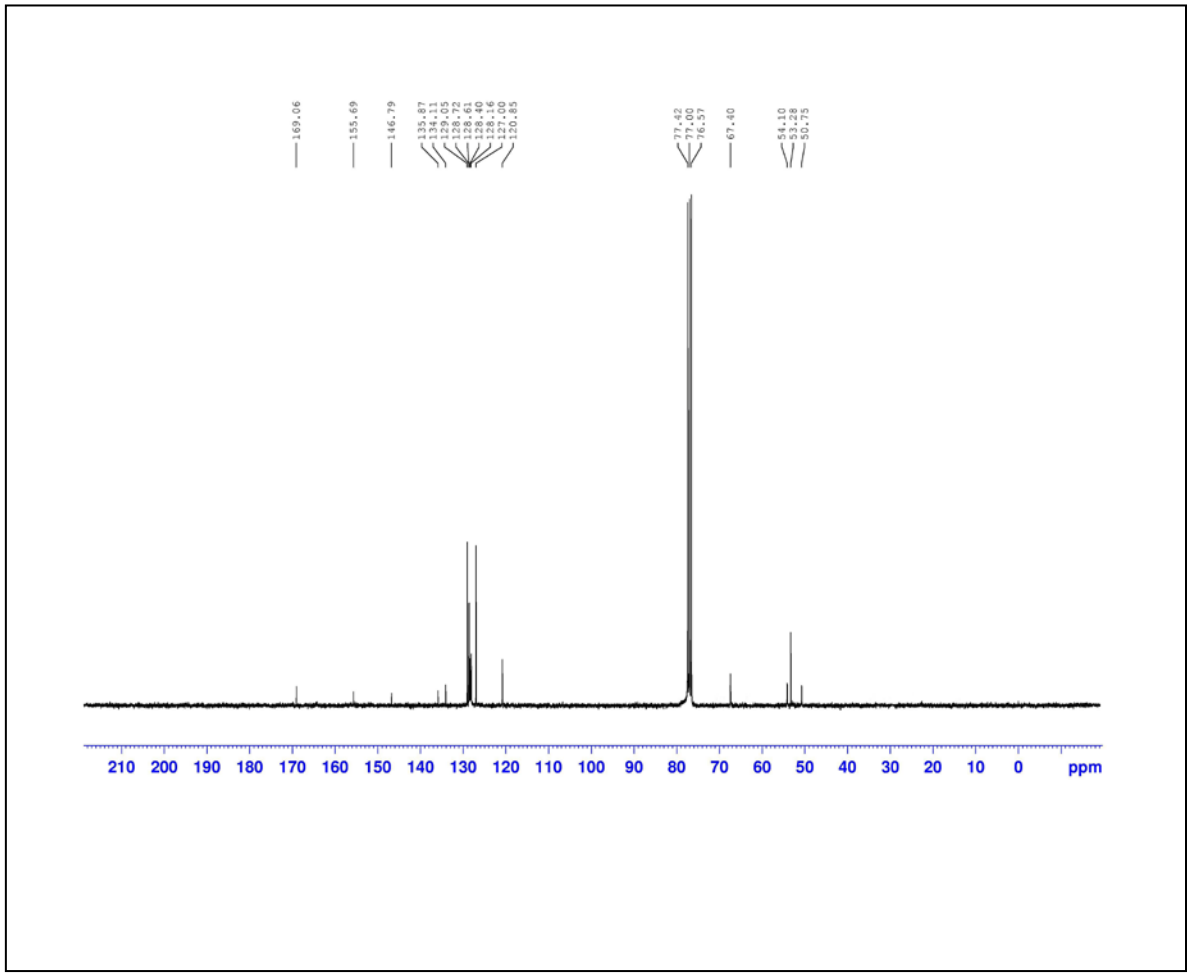
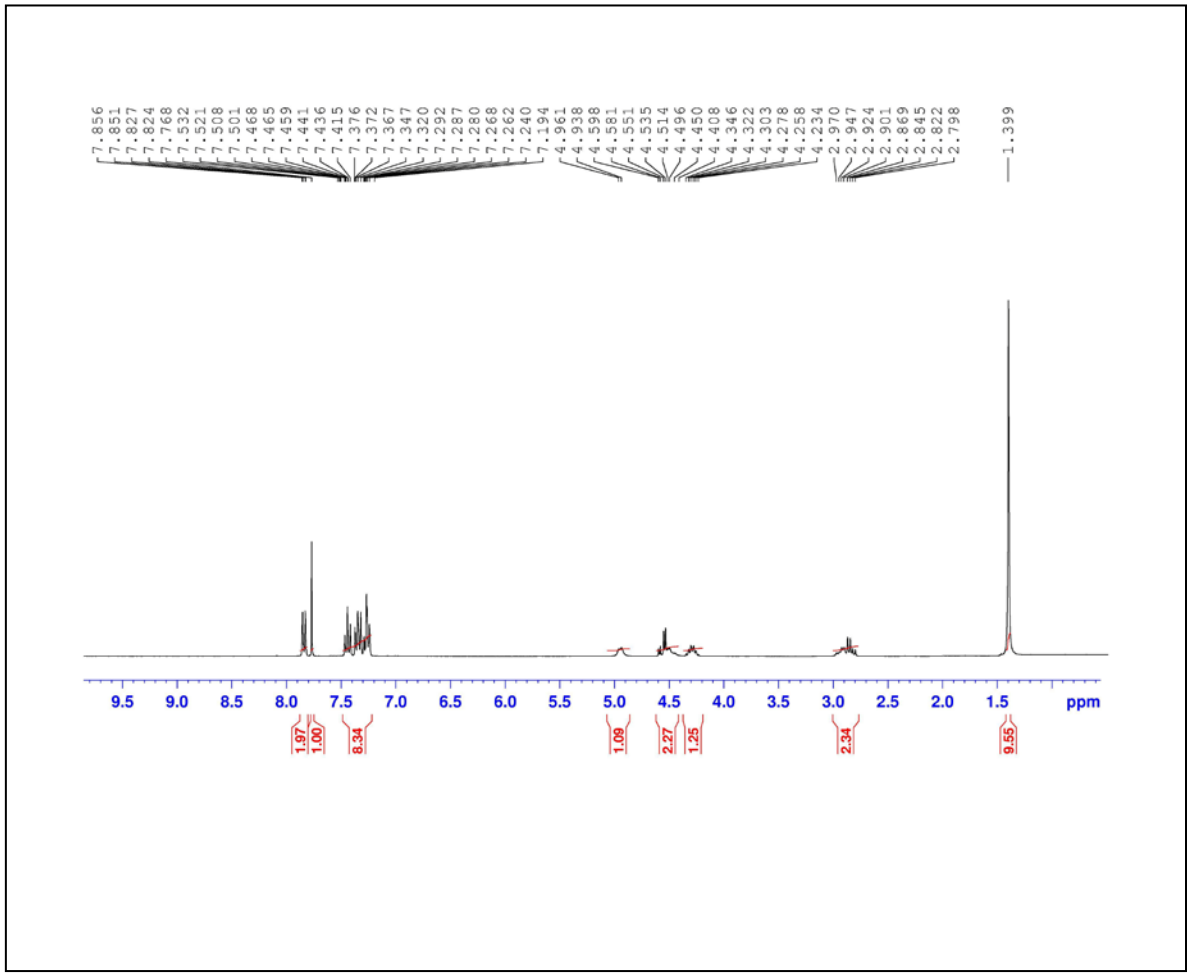
AQ 0.9110282 sec
RG 8192
DW 27.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 3.20 dB
PL1W 36.06793213 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.20 dB
PL12 19.50 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

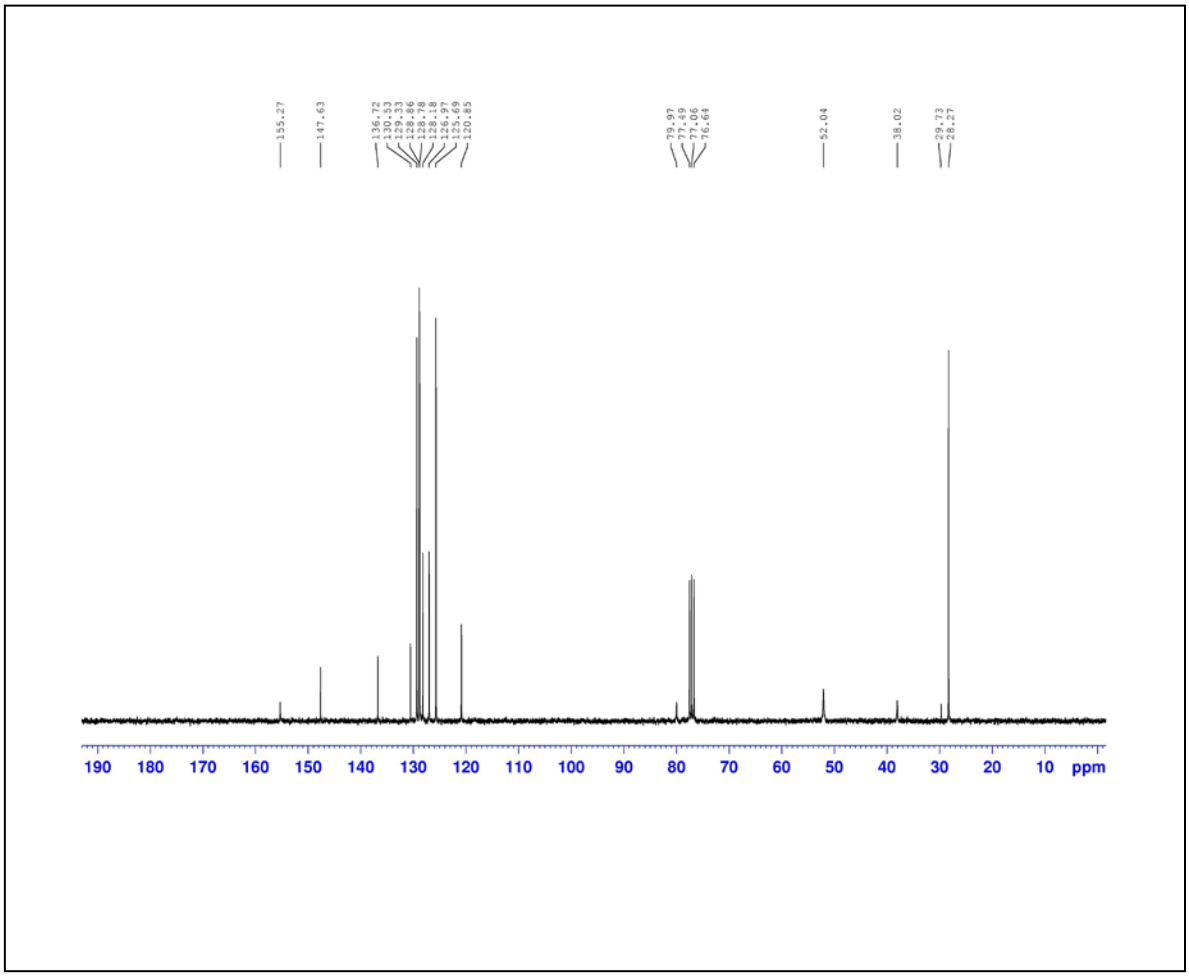


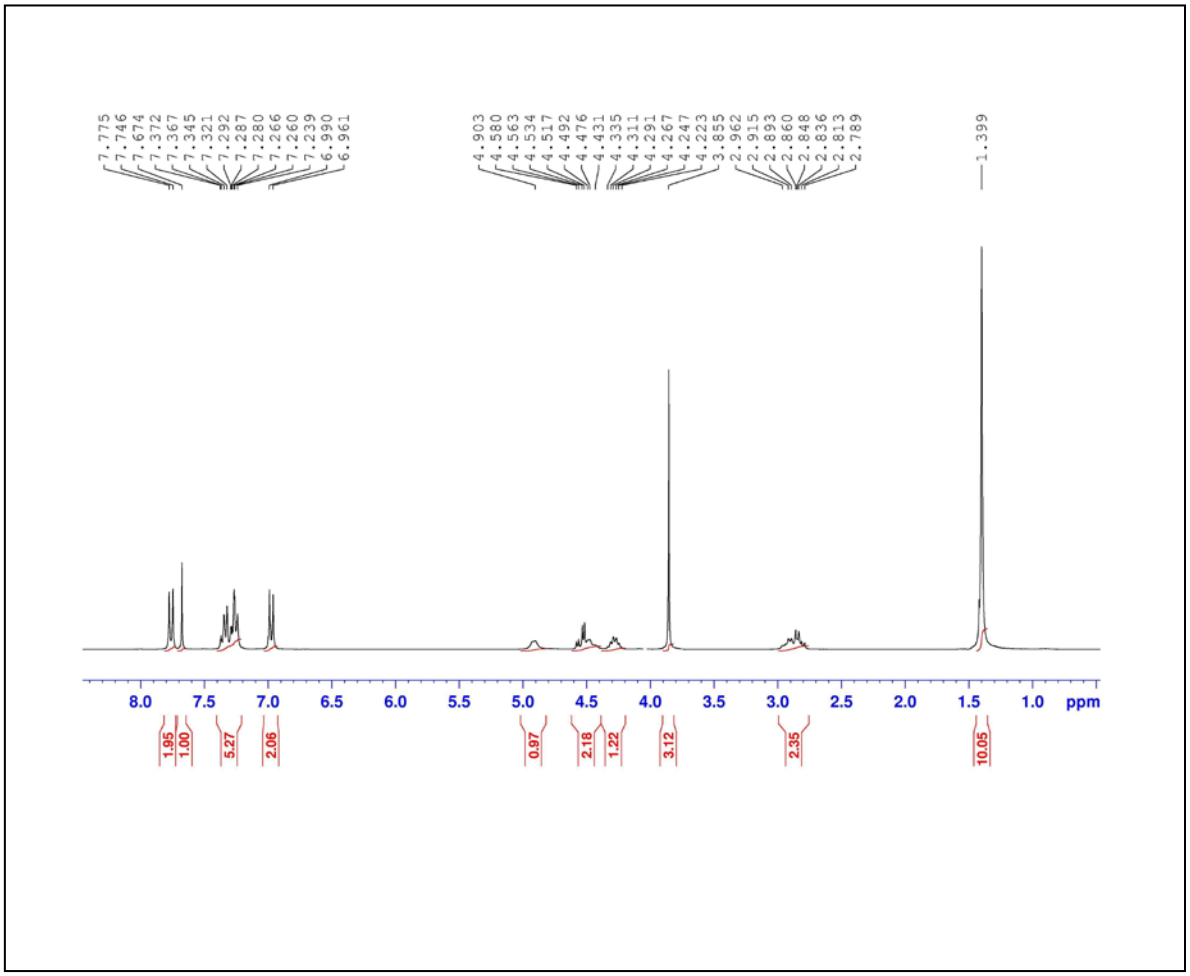

169.13
155.79
147.83
135.91
130.21
129.83
128.59
128.31
128.12
125.77
120.79
77.43
77.01
76.58
67.36
54.12
53.24
50.71
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

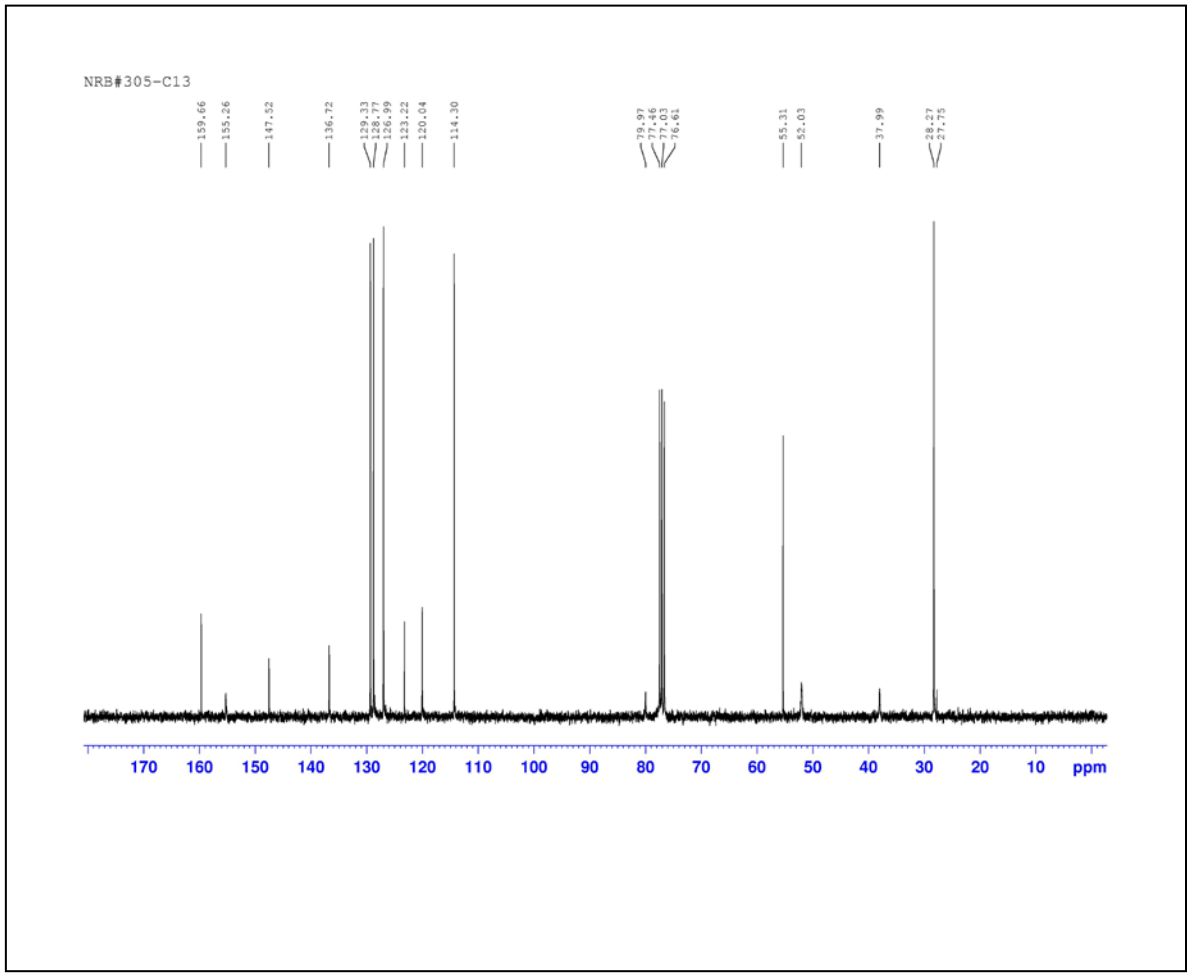

7.724
7.696
7.643
7.418
7.390
7.365
7.279
5.707
5.688
5.207
5.166
5.152
5.111
4.988
4.974
4.942
4.930
4.896
4.883
4.839
4.820
4.807
4.794
4.774
3.827
10
9
8
7
6
5
4
3
2
1
0
ppm
2.11
1.00
6.63
1.05
2.66
3.33
3.20
BRUKER
NAME NRB#276H
EXPNO 1
PROCNO 1
Date_ 20111103
Time 18.57
INSTRUM spect
PROBHD 5 mm 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

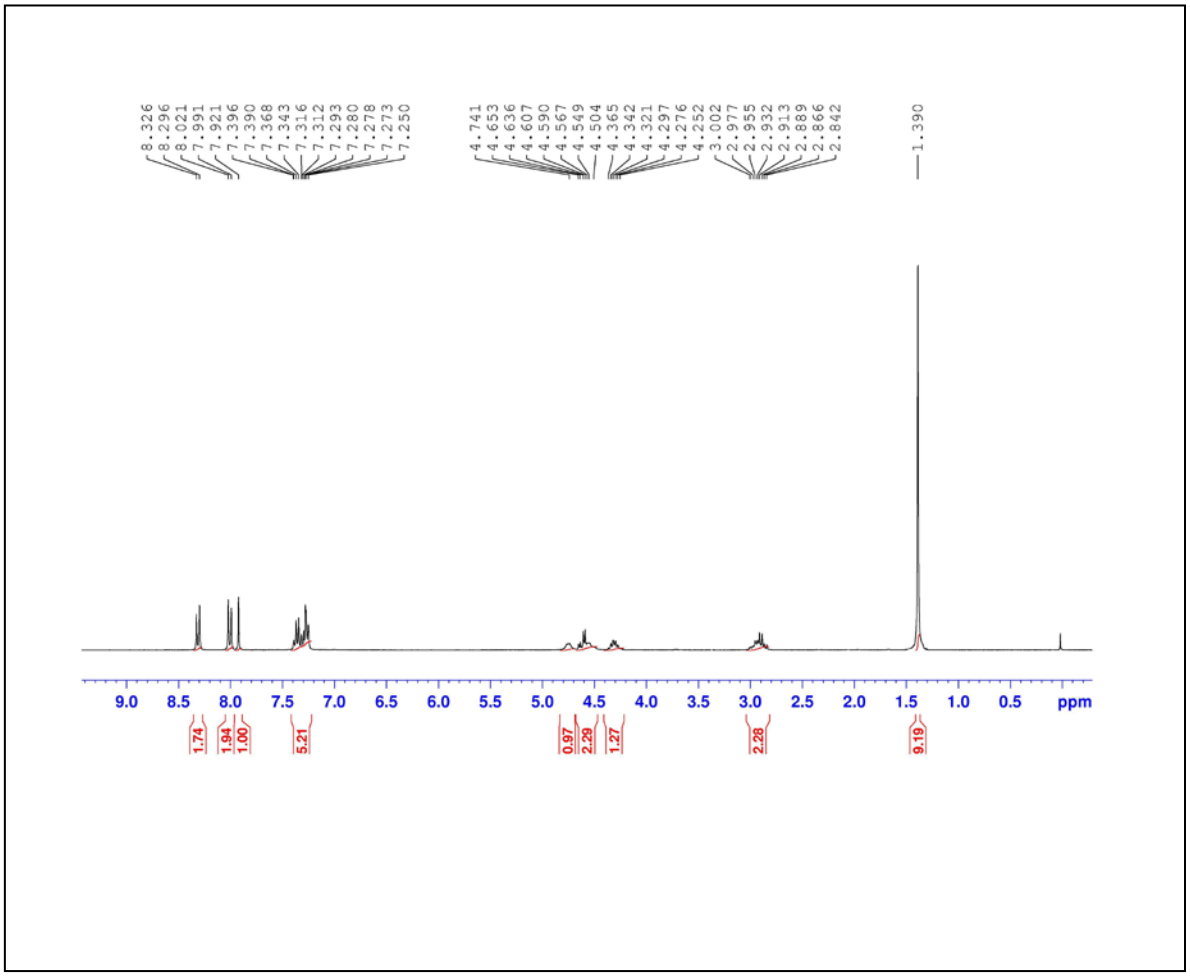


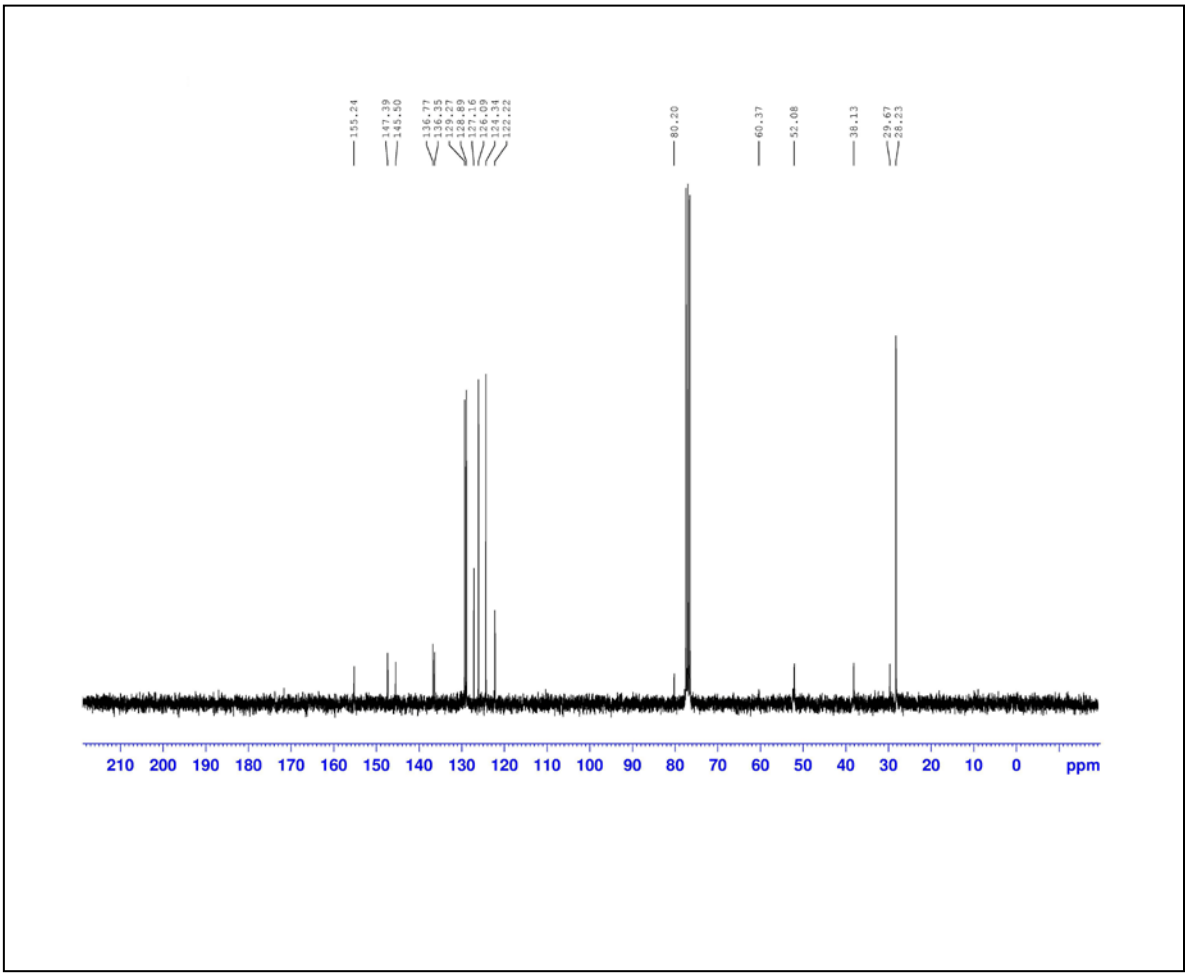


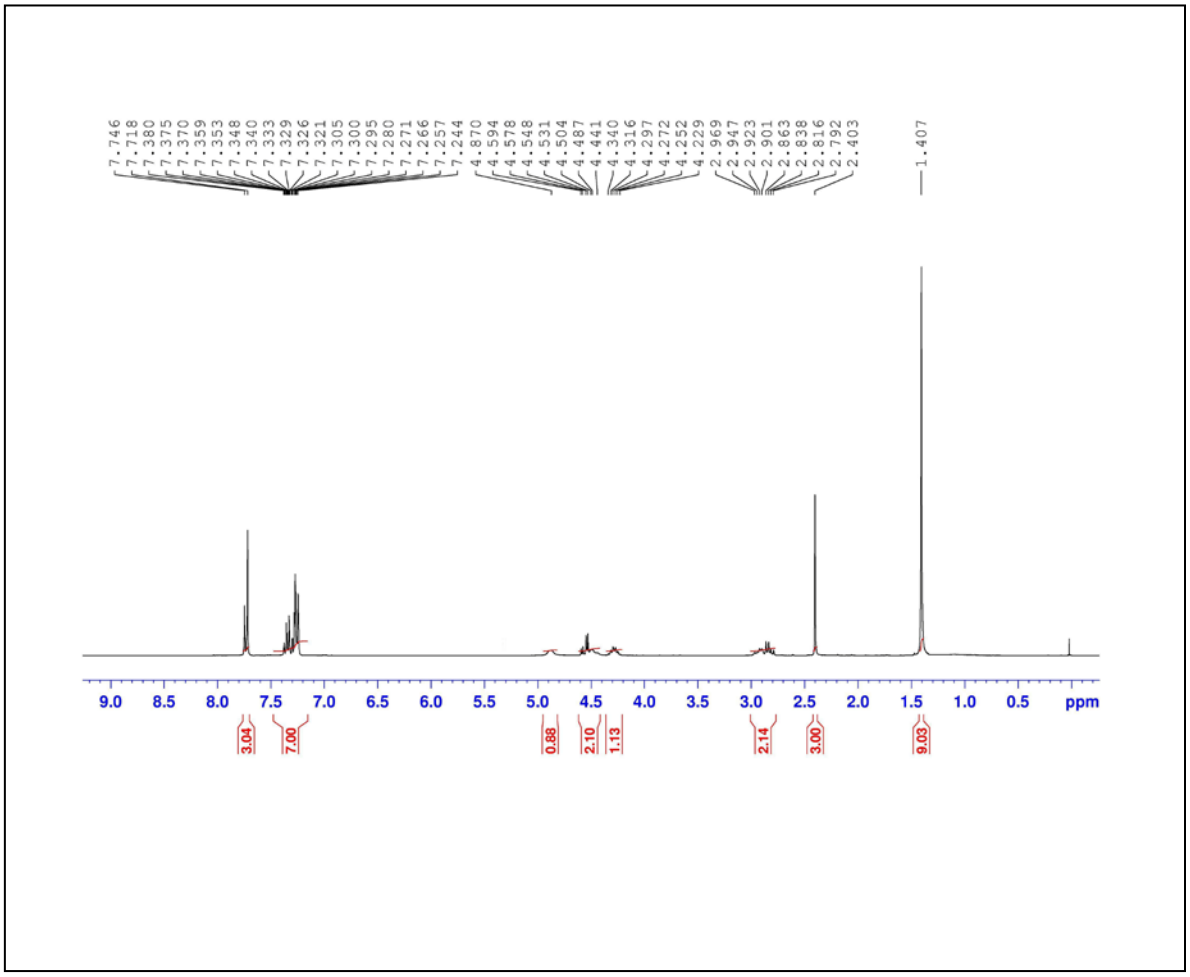


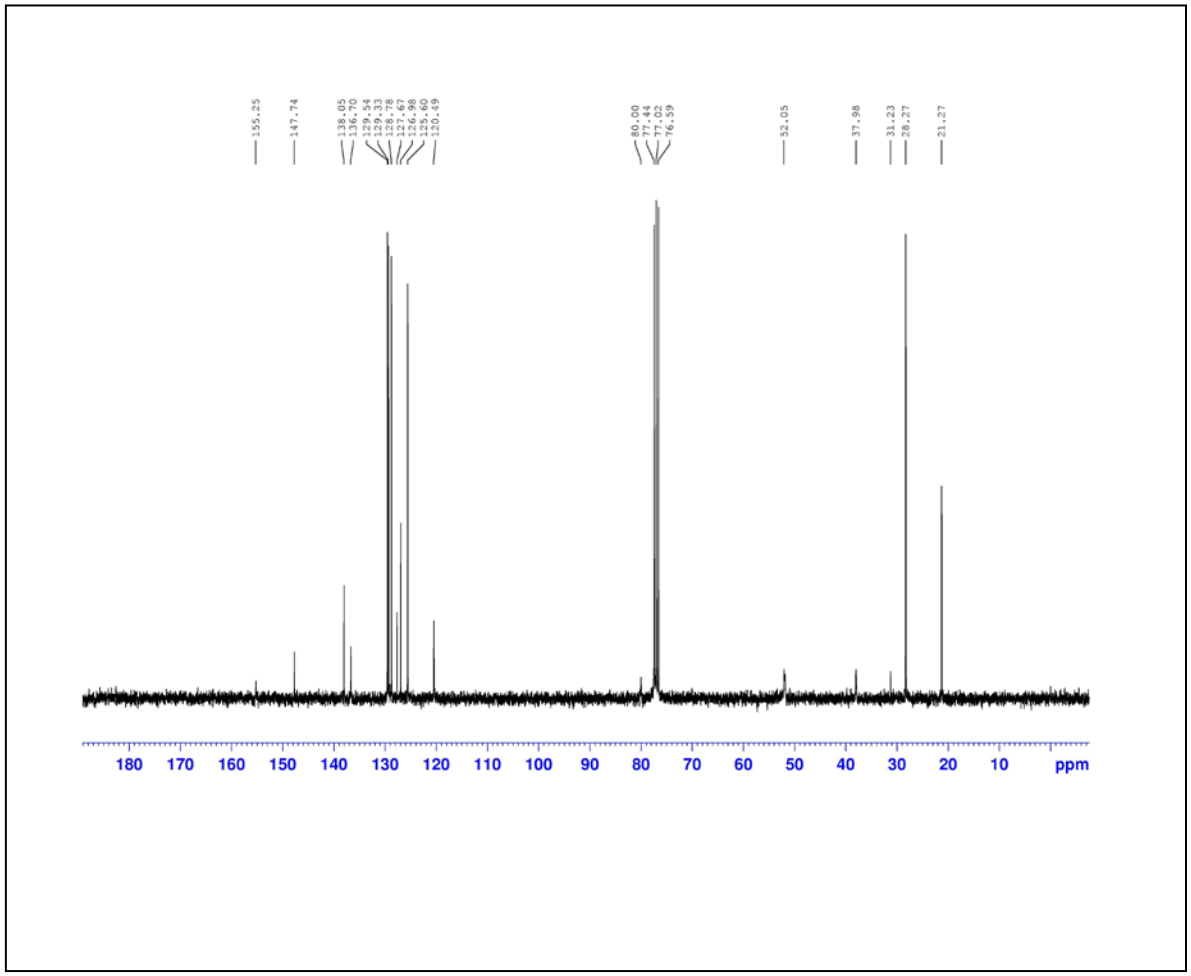


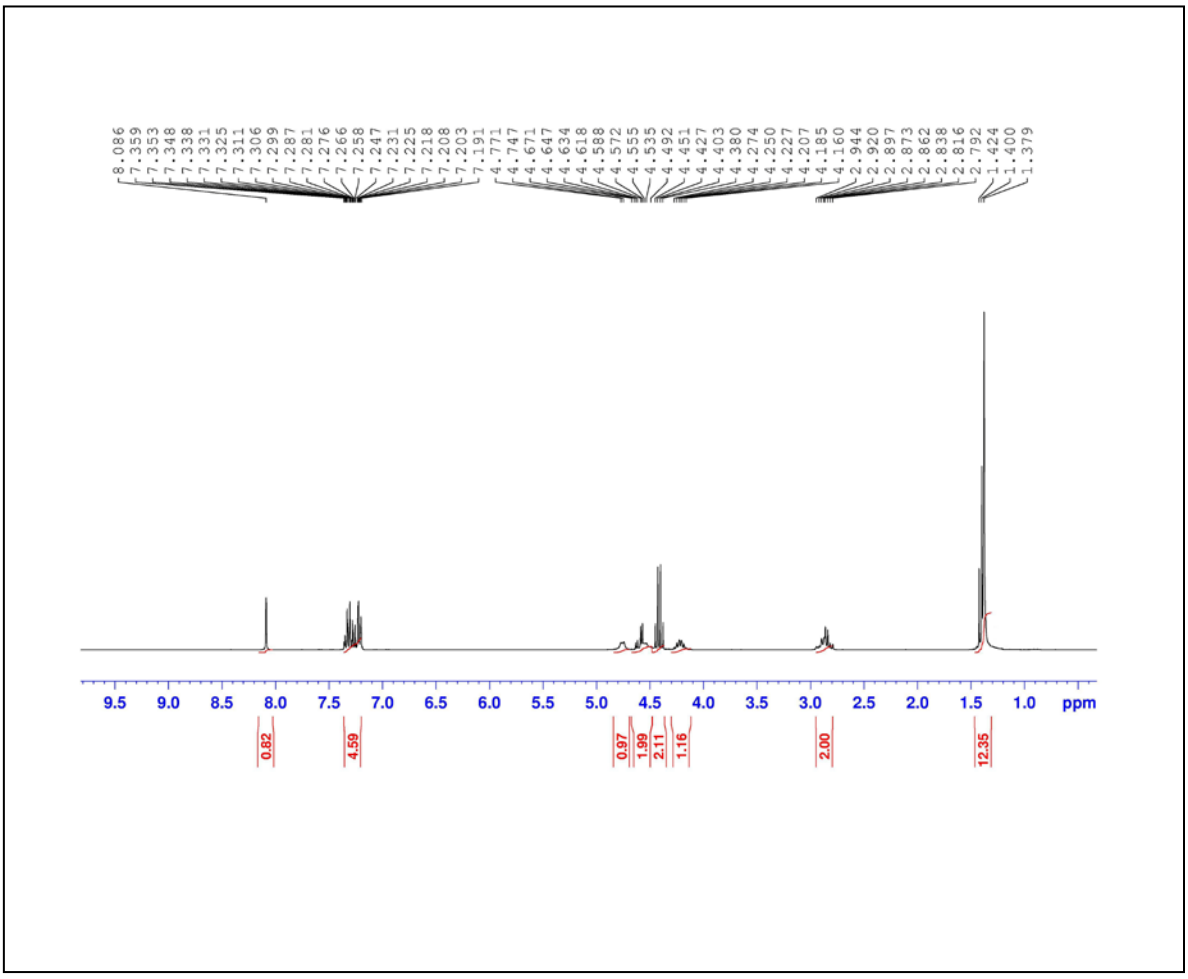

NRB#305-C13
159.66
155.26
147.52
136.72
129.33
128.77
126.99
123.22
120.04
114.30
79.97
77.66
77.03
76.61
55.31
52.03
37.99
28.27
27.75
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

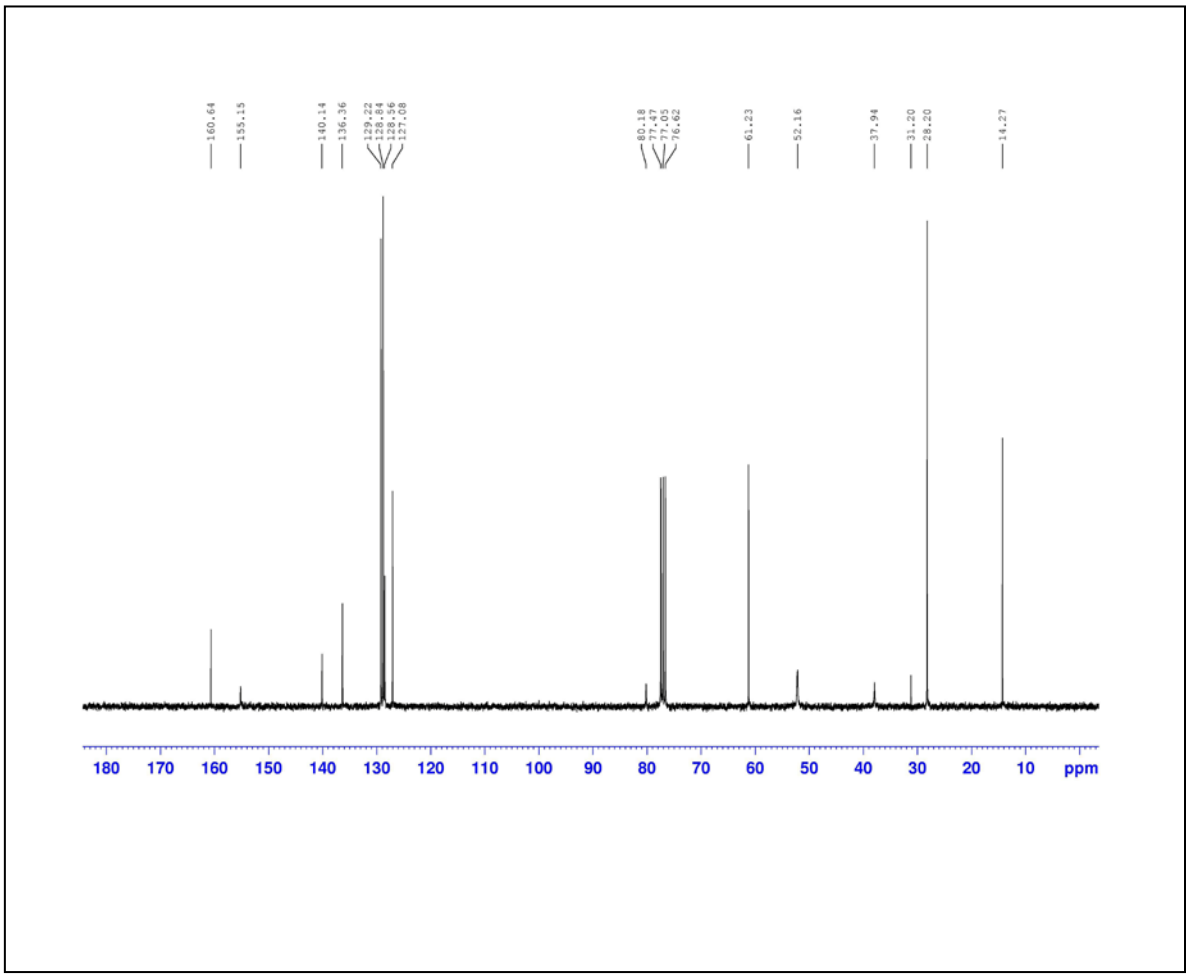

8.326
8.296
8.021
7.991
7.921
7.396
7.390
7.368
7.343
7.316
7.312
7.293
7.280
7.278
7.273
7.250
4.741
4.653
4.636
4.607
4.590
4.567
4.549
4.504
4.365
4.342
4.321
4.297
4.276
4.252
3.002
2.977
2.955
2.932
2.913
2.889
2.866
2.842
1.390
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.74
1.94
1.00
5.21
0.97
2.29
1.27
2.28
9.19

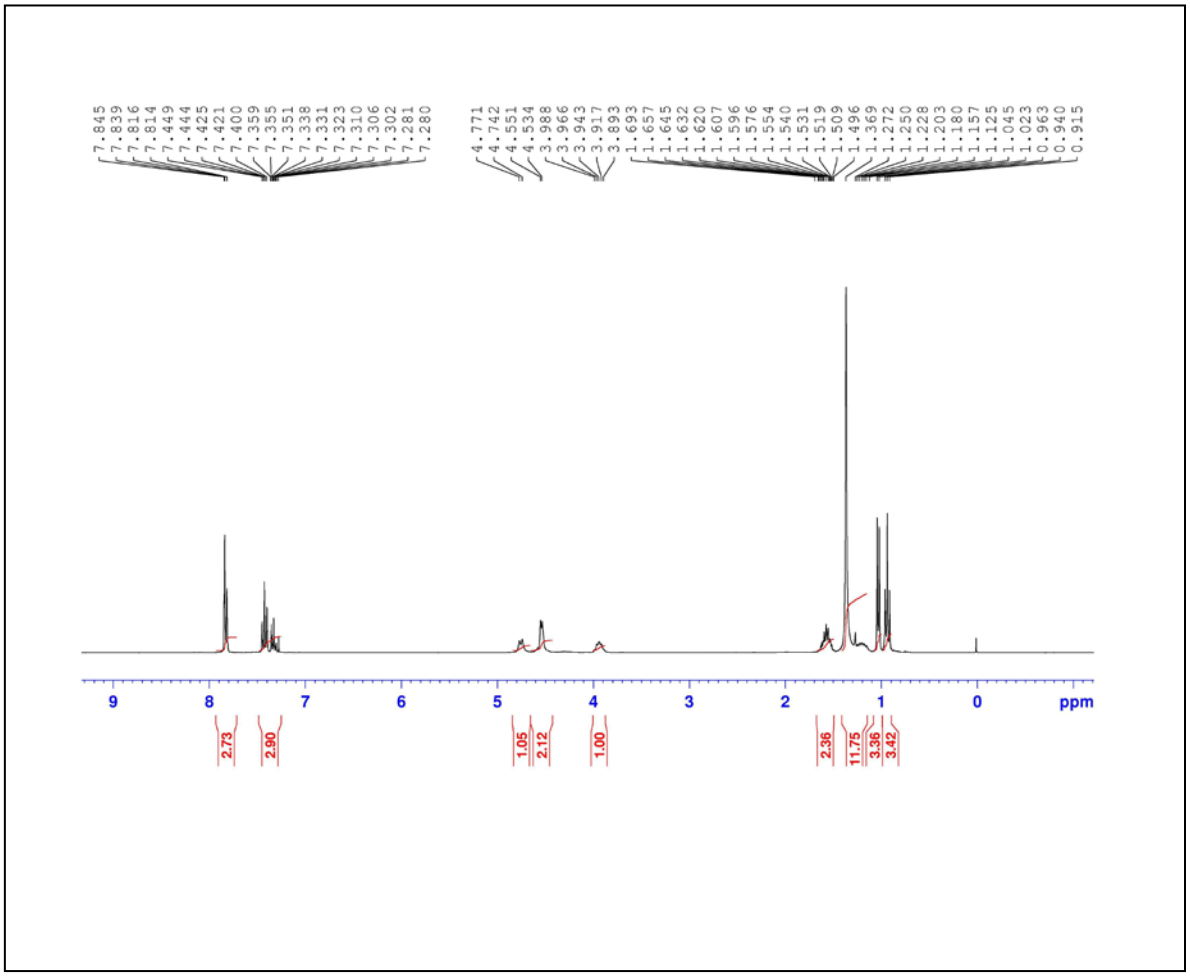


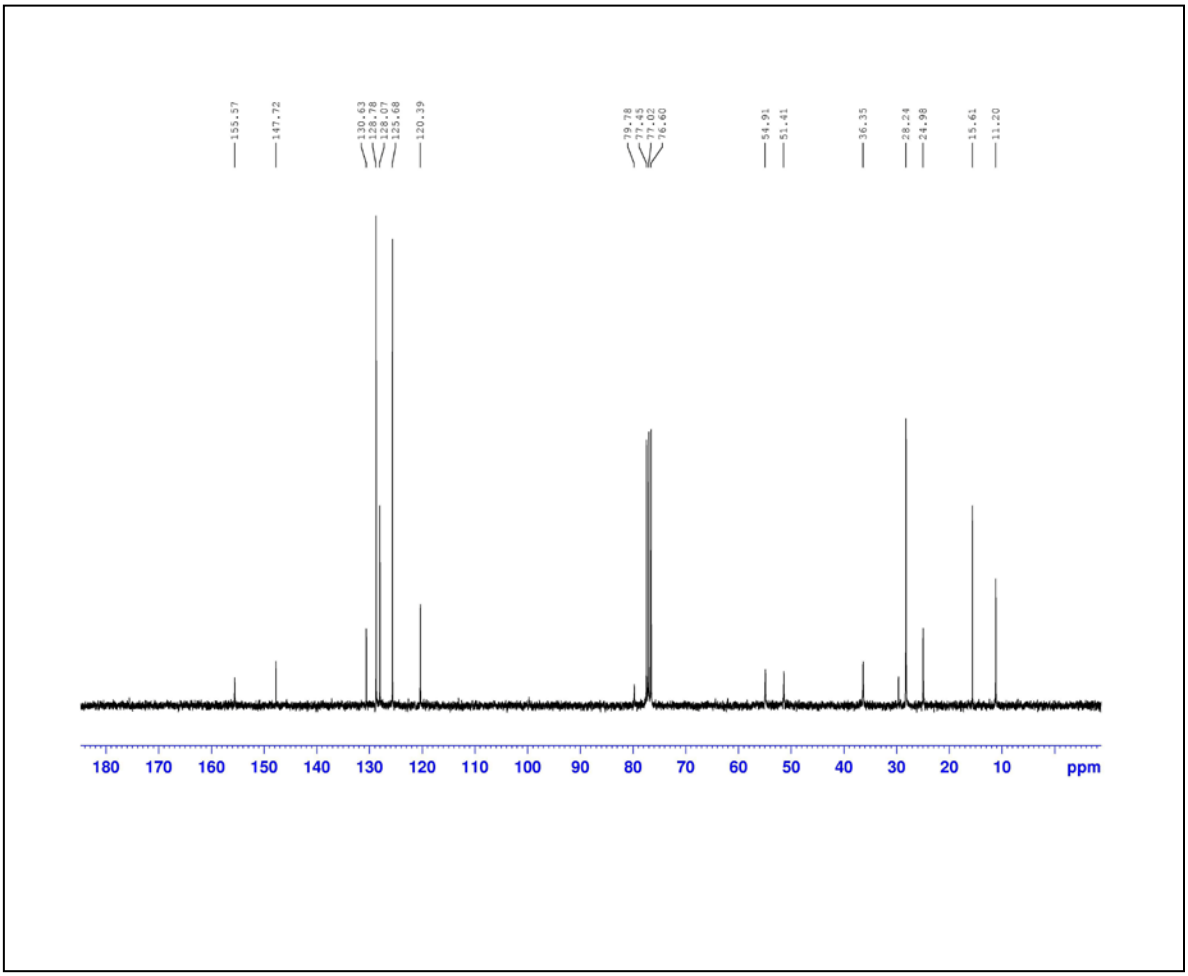

7.746
7.718
7.380
7.375
7.370
7.359
7.353
7.348
7.340
7.333
7.329
7.326
7.321
7.305
7.300
7.295
7.280
7.271
7.266
7.257
7.244
4.870
4.594
4.578
4.548
4.531
4.504
4.487
4.441
4.340
4.316
4.297
4.272
4.252
4.229
2.969
2.947
2.923
2.901
2.863
2.838
2.816
2.792
2.403
1.407
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.04
7.00
0.88
2.10
1.13
2.14
3.00
9.03

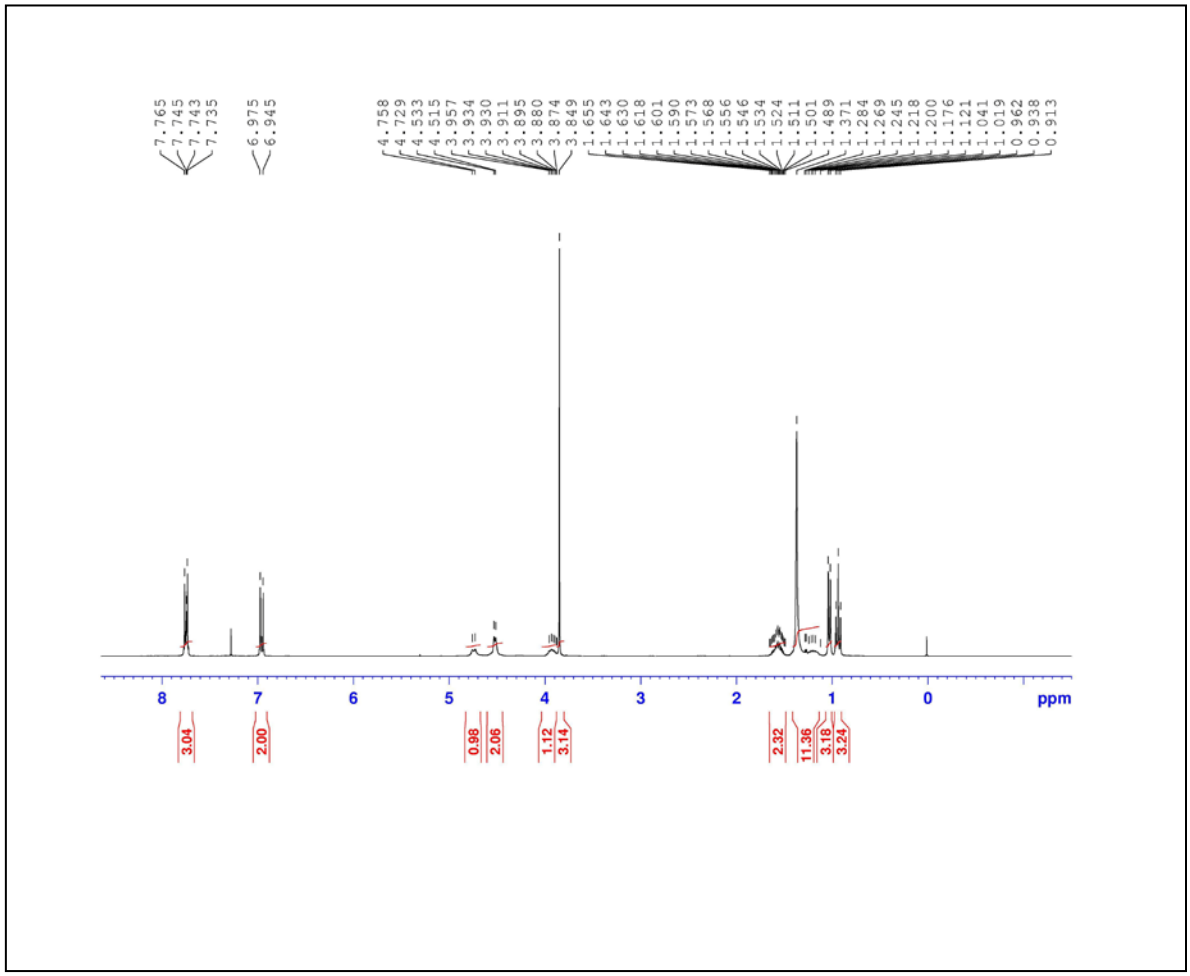

155.25
147.74
138.05
136.70
129.54
129.33
128.78
127.67
126.98
125.60
120.49
80.00
77.44
77.02
76.59
52.05
37.98
31.23
28.27
21.27
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

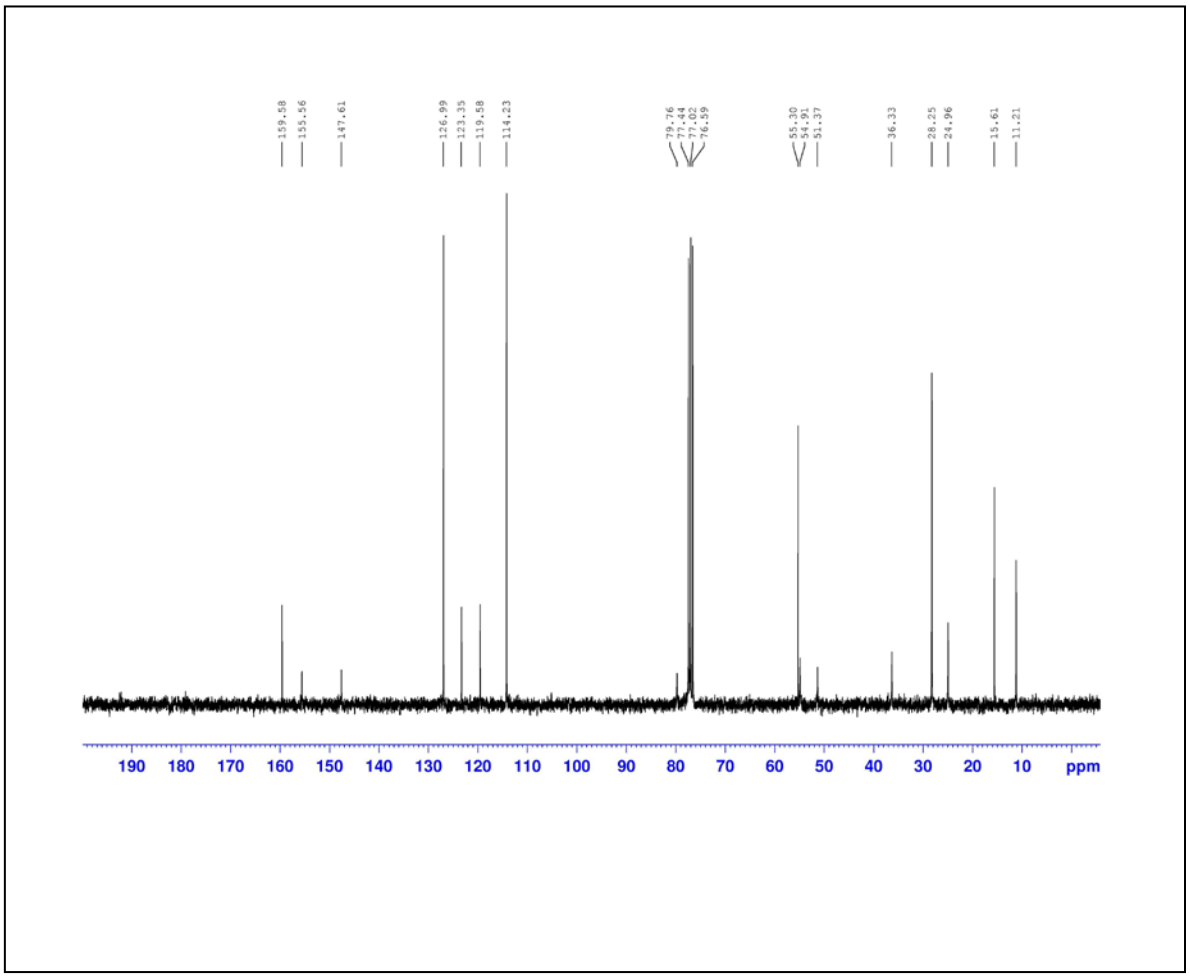


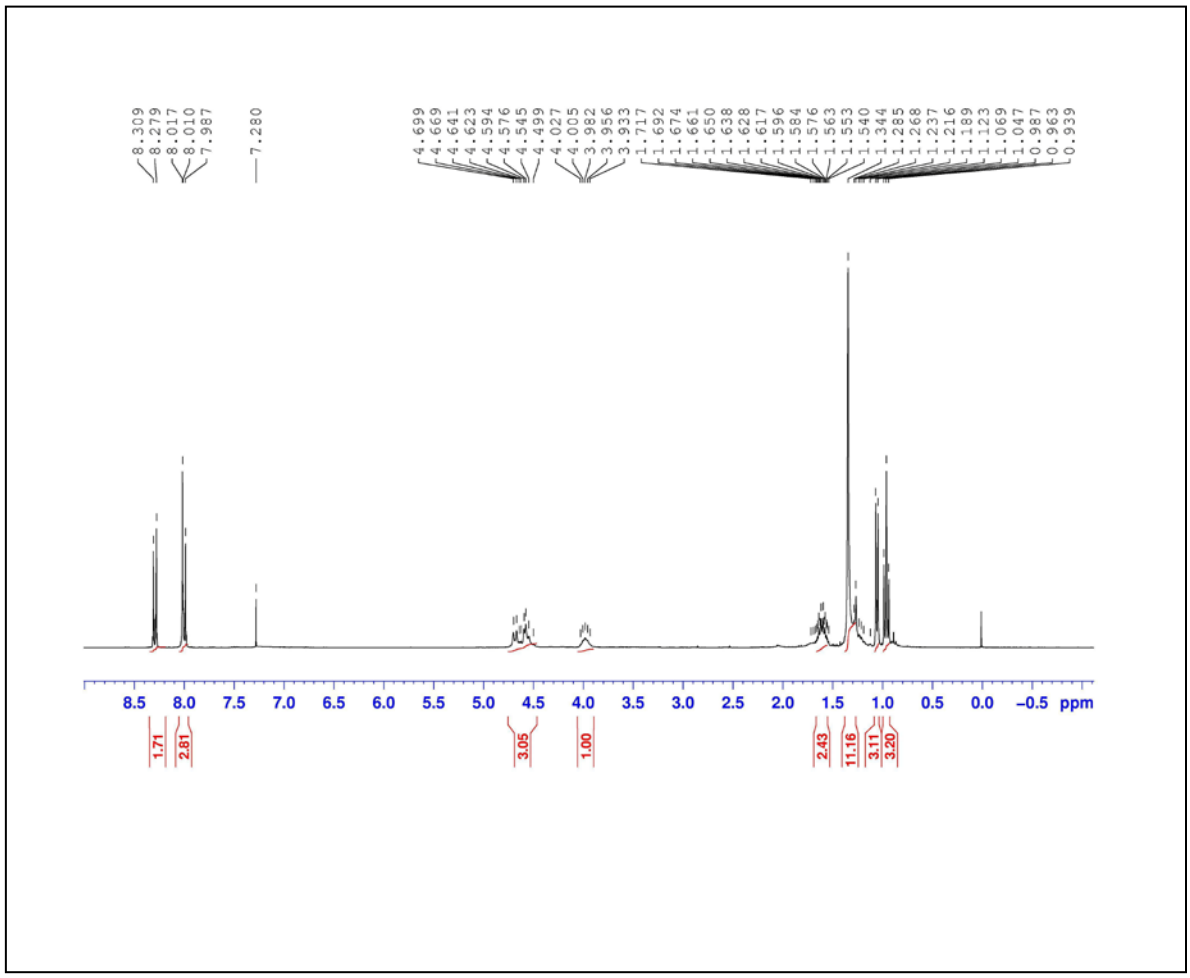

160.64
155.15
140.14
136.36
139.22
129.64
128.56
127.08
80.18
77.47
77.03
76.62
61.23
52.16
37.94
31.20
28.20
14.27
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

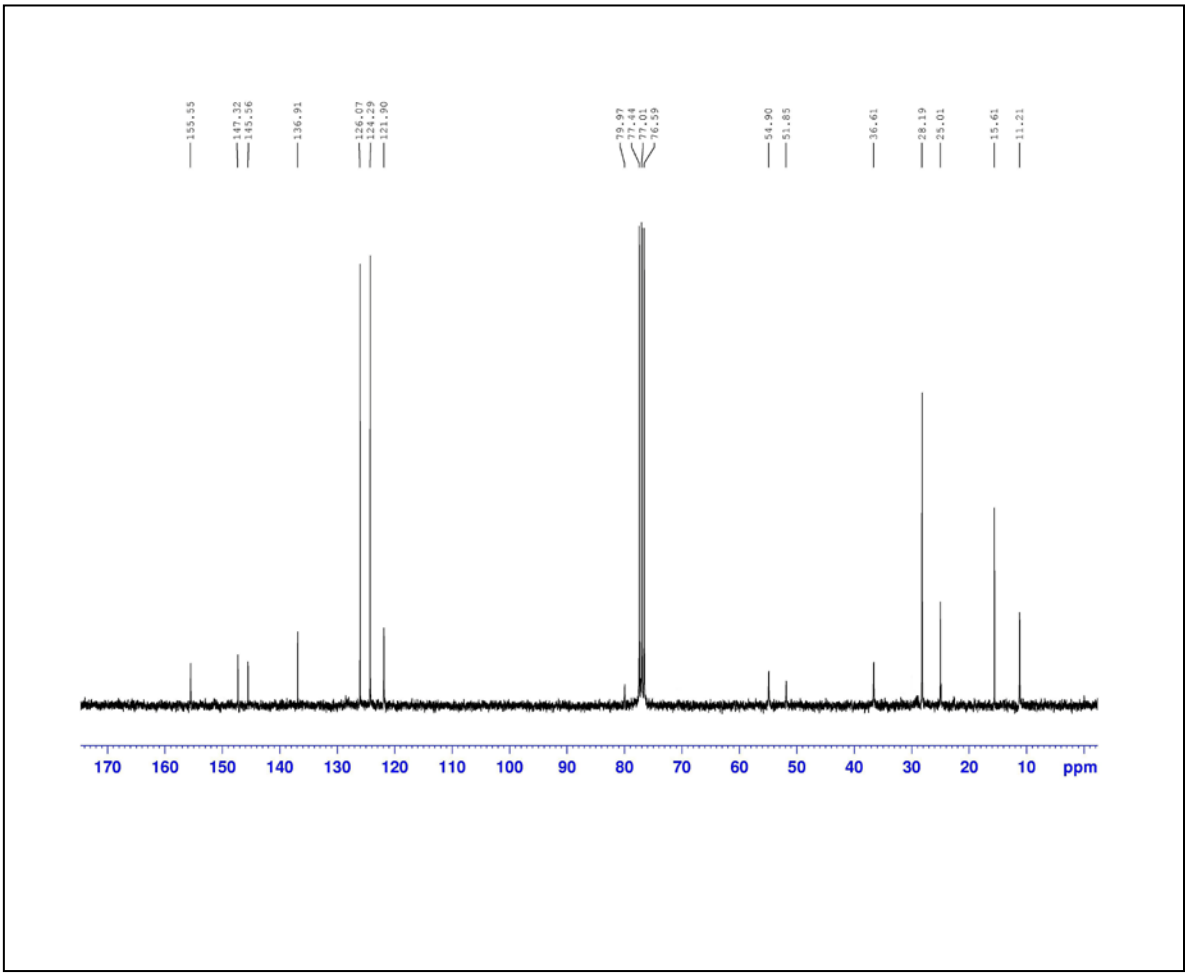

7.845
7.839
7.816
7.814
7.449
7.444
7.425
7.421
7.400
7.359
7.355
7.351
7.338
7.331
7.323
7.310
7.306
7.302
7.281
7.280
4.771
4.742
4.551
4.534
3.988
3.966
3.943
3.917
3.893
1.693
1.657
1.645
1.632
1.620
1.607
1.596
1.576
1.554
1.540
1.531
1.519
1.509
1.496
1.369
1.272
1.250
1.228
1.203
1.180
1.157
1.125
1.045
1.023
0.963
0.940
0.915
9
8
7
6
5
4
3
2
1
0
ppm
2.73
2.90
1.05
2.12
1.00
2.36
11.75
3.36
3.42

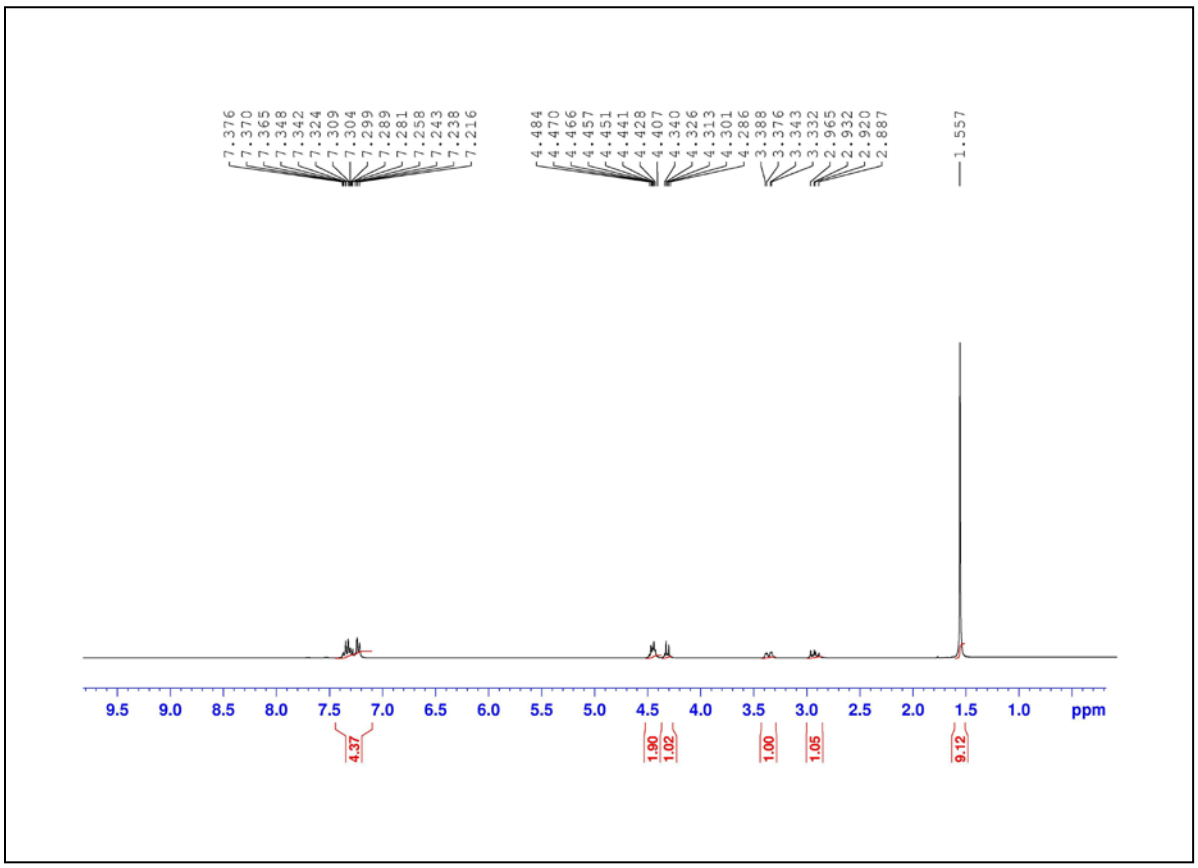

155.57
147.72
130.63
129.78
128.07
125.68
120.39
79.78
77.45
77.02
76.60
54.91
51.41
36.35
28.24
24.98
15.61
11.20
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

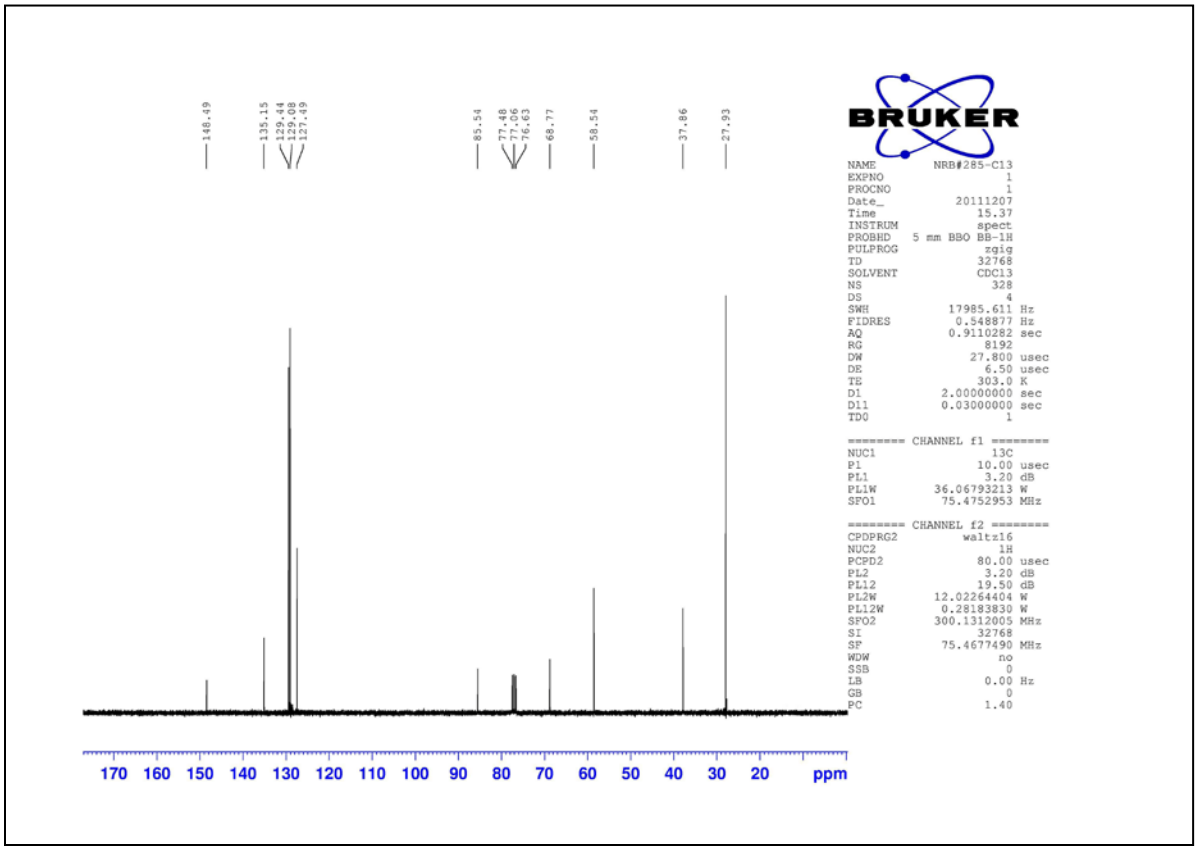

7.765
7.745
7.743
7.735
6.975
6.945
4.758
4.729
4.533
4.515
3.957
3.934
3.930
3.911
3.895
3.880
3.874
3.849
1.655
1.643
1.630
1.618
1.601
1.590
1.573
1.568
1.556
1.546
1.534
1.524
1.511
1.501
1.489
1.371
1.284
1.269
1.245
1.218
1.200
1.176
1.121
1.041
1.019
0.962
0.938
0.913
8
7
6
5
4
3
2
1
0
ppm
3.04
2.00
0.98
2.06
1.12
3.14
2.32
11.36
3.18
3.24

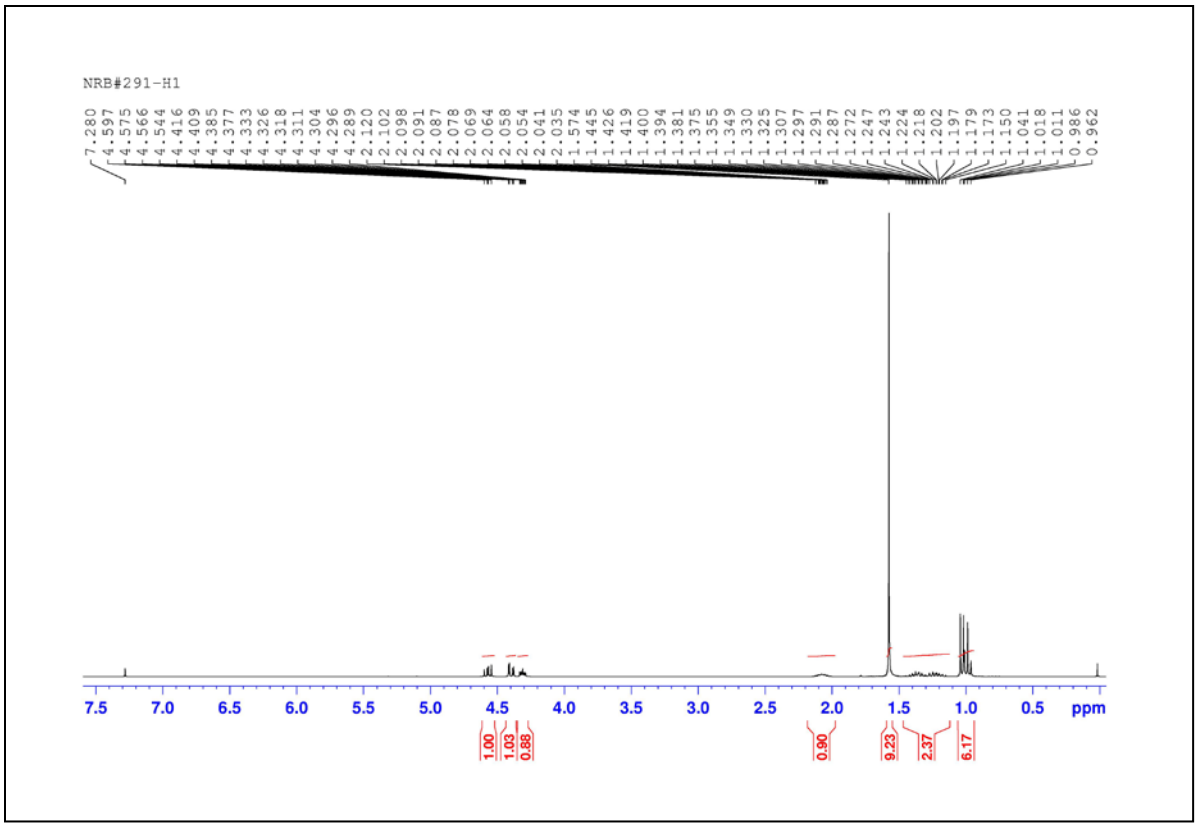

159.58
155.56
147.61
126.99
123.35
119.58
114.23
79.76
77.44
77.02
76.59
55.30
54.91
51.37
36.33
28.25
24.96
15.61
11.21
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm




155.55
147.32
145.56
136.91
126.07
124.29
121.90
79.97
77.64
77.01
76.59
54.90
51.85
36.61
28.19
25.01
15.61
11.21
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


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


148.49
135.15
129.44
129.08
127.49
85.54
77.48
77.06
76.63
68.77
58.54
37.86
27.93
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
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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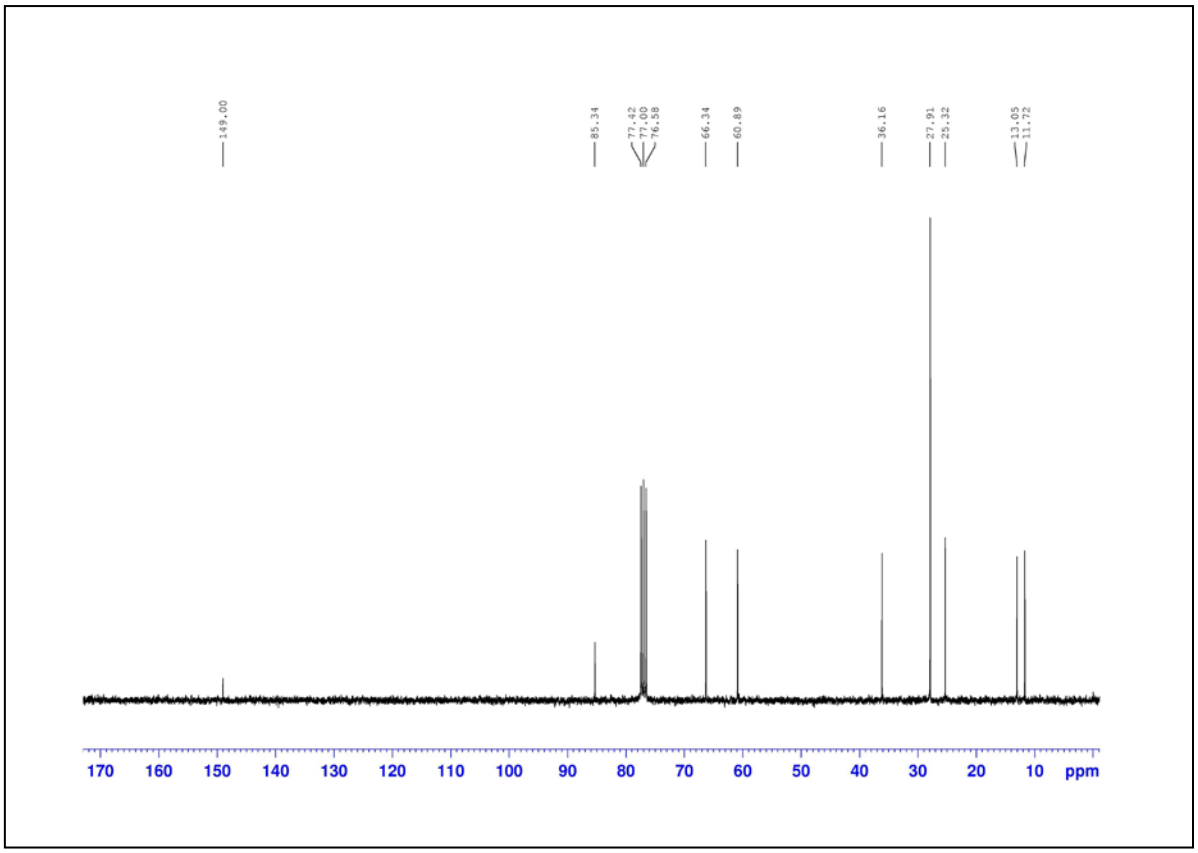
4.575
4.566
4.544
4.416
4.409
4.385
4.377
4.333
4.326
4.318
4.311
4.304
4.296
4.289
4.120
2.102
2.098
2.091
2.087
2.078
2.069
2.064
2.058
2.054
2.041
2.035
1.574
1.445
1.426
1.419
1.400
1.394
1.381
1.375
1.355
1.349
1.330
1.325
1.307
1.297
1.291
1.287
1.272
1.247
1.243
1.224
1.218
1.202
1.197
1.179
1.173
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