Eur. J. Org. Chem. **2013** · © WILEY-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2013 · ISSN 1434–193X

SUPPORTING INFORMATION

DOI: 10.1002/ejoc.201201631
Title: Synthesis, Electrochemistry, and Hierarchical Self-Organization of Fulleropyrrolidine–Phthalimide Dyads
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General experimental:

FTIR spectra were recorded on Perkin-Elmer-FT-IR 1725X spectrophotometer; values are given in cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Gemini 200 and Bruker Avance III 500 spectrometers at 200/50 and 500/125 MHz, respectively. The chemical shifts were measured to residual nondeuterated solvent resonances or TMS. Fullerenic carbons, presented as Cf, were numbered in a simplified way, according to the literature.[1] Mass spectra were obtained on Agilent technologies 6210 TOF LC/MS instrument. UV spectra were recorded on a GBC-Cintra 40 spectrophotometer. Reactions were monitored by TLC using plates precoated with silica gel 60 F254. Column chromatography was performed on Silica, 10-18, 60A, ICN Biomedicals. Standard techniques were used for the purification of reagent and solvents.[2] Reactions induced by microwave irradiation were performed in a Milestone MultiSynth microwave multimode oven, using a MedCHEM kit and MonoPREP kit.

Investigations of samples morphology were carried out with scanning electron microscopy, using a JEOL JSM-840A instrument, at an acceleration voltage of 30 kV. A several drops of a dilute solution (~1 mM in toluene, dioxane, methanol, toluene/dioxane (2:1), toluene/*iso*-propanol (1:1 and 2:1), and chloroform/methanol (2:1)) of fullero-phthalimide dyads were deposited on the surface of Si wafer and then slowly evaporated in a glass petri dish (diameter 10 cm) under toluene atmosphere at the room temperature. The investigated samples were gold sputtered in a JFC 1100 ion sputterer and then subjected to SEM observations.

Preparation of mono-protected diamines 2a-f

To a stirred solution of diamine (0.06 mol) in chloroform (150 mL), di-tert-butyl dicarbonate (0.01 mol) dissolved in chloroform (50 mL) was added via dropping funnel over 1 hour at room temperature. The reaction mixture was stirred overnight and the solvent was removed under reduced pressure. Pure amides **2a-f** were obtained as oils in 37-66% yields by SiO_2 column chromatography using EtOAc-toluene mixtures.

Tert-butyl 2-aminoethylcarbamate (2a): 1.9 g (66%); All obtained spectra were in accordance with reported procedure.[3]

Tert-butyl 4-aminobutylcarbamate (2b): 1.1 g (43%); IR (ATR): 3365, 2934, 2858, 1686, 1526, 1280, 1251, 1173, 780 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 5.09 (bs, 1H); 3.13 (m, 2H); 2.71 (t, 2H, *J*=6.2); 1.60-1.44 (m, 13H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 155.9 (C); 78.6 (C); 41.4 (CH_2); 40.1 (CH_2); 33.4 (CH_2); 28.1 ($3CH_3$); 27.2 (CH_2); HRMS: *m/z* calcd for $[C_9H_{20}N_2O_2+H]^+$ 189.1597, measured 189.1597; All obtained spectra were in accordance with reported procedure.[3]

Tert-butyl 6-aminohexylcarbamate (2c): 1.1g (42%); IR (ATR): 3344, 2934, 2868, 1690, 1525, 1276, 1254, 1172, 751 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 4.90 (s, 1H); 3.11-3.07 (m, 2H); 2.69 (t, 2H, *J*=7); 1.56-1.33 (m, 17H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 155.9 (C); 78.7

(C); 41.3 (CH_2); 40.2 (CH_2); 32.4 (CH_2); 29.8 (CH_2); 28.2 ($3CH_3$); 26.3 (CH_2); 26.2 (CH_2); HRMS: *m/z* calcd for $[C_{11}H_{24}N_2O_2+H]^+$ 217.1910, measured 217.1912; All obtained spectra were in accordance with reported procedure.[3]

Tert-butyl 8-aminooctylcarbamate (2d): 1.3 g (58%); IR (ATR): 3367, 2924, 2853, 1688, 1523, 1249, 1172, 807 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz): δ = 4.51 (bs, 1H); 3.09 (q, 2H, *J*=6.2); 2.68 (t, 2H, *J*=4.6); 1.51-1.36 (m, 6H); 1.44 (s, 9H); 1.36-1.20 (m, 6H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 156.0 (C); 79.1 (C); 42.1 (CH_2); 40.6 (CH_2); 33.6 (CH_2); 29.9 (CH_2); 29.3 (CH_2); 29.2 (CH_2); 28.4 ($3CH_3$); 26.7 (CH_2); 26.6 (CH_2); HRMS: *m/z* calcd for $[C_{13}H_{28}N_2O_2+H]^+$ 245.2223, measured 245.2224; All obtained spectra were in accordance with reported procedure.[3]

Tert-butyl 10-aminodecylcarbamate (2e): 1.2 g (37%); IR (ATR): 3367, 2924, 2854, 1690, 1524, 1283, 1249, 1175, 782 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz): δ = 4.65 (bs, 1H); 3.09 (q, 2H, *J*=6.2); 2.7 (t, 2H, *J*=6.8); 1.55-1.44 (m, 13H); 1.32-1.12 (m, 12H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 155.9 (C); 78.8 (C); 41.7 (CH_2); 40.5 (CH_2); 29.9 (CH_2); 29.4 ($2CH_2$); 29.3 (CH_2); 29.2 (CH_2); 29.1 (CH_2); 28.3 ($3CH_3$); 26.7 (CH_2); 26.6 (CH_2); HRMS: *m/z* calcd for $[C_{15}H_{32}N_2O_2+H]^+$ 273.2536, measured 273.2546; All obtained spectra were in accordance with reported procedure.[3]

Tert-butyl 12-aminododecylcarbamate (2f): 2.0 g (55%); IR (ATR): 3362, 2922, 2853, 1689, 1524, 1281, 1248, 1173.0, 722.3 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz): δ = 4.64 (bs, 1H); 3.10 (q, 2H, *J*=6.2); 2.68 (t, 2H, *J*=6.6); 1.55-1.38 (m, 12H); 1.33-1.23 (m, 17H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 155.9 (C); 78.8 (C); 42.1 (CH_2); 40.5 (CH_2); 33.6 (CH_2); 29.9 ($2CH_2$); 29.4 ($2CH_2$); 29.3 ($2CH_2$); 29.1 (CH_2); 28.3 ($3CH_3$); 26.8 (CH_2); 26.7 (CH_2); HRMS: *m/z* calcd for $[C_{17}H_{36}N_2O_2+H]^+$ 301.2849, measured 301.2863; All obtained spectra were in accordance with reported procedure.[3]

Preparation of benzyloxy-derivates (3a-f)

To a stirred, ice bath cooled solution of amine **2a-f** (2 mmol) and Et_3N (6 mmol, 0.44 mL) in dry CH_2Cl_2 (8 mL) a solution of benzyl bromoacetate (1.6 mmol, 1.1 mL) in dry CH_2Cl_2 (2 mL) was added dropwise over 1 hour. The reaction mixture was stirred at room temperature for an additional 30 h. The resulting mixture was diluted with water, organic phase washed with H_2O (2 x 10 mL), brine (2 x 10 mL) and dried over anh. Mg_2SO_4. The solvent was evaporated and the remaining crude product was chromatographed on a SiO_2 column. Elution with EtOAc-toluene (6/4) mixture gave the products **3a-f** as yellow oils in 39-62% yields.

Benzyl 2-(2-(tert-butoxycarbonylamino)ethylamino)acetate (3a): 271.0 mg (55%); The obtained spectra were in accordance with reported procedure.[4]

Benzyl 2-(4-(tert-butoxycarbonylamino)butylamino)acetate (3b): 229.9 mg (41%); IR (ATR): 3338, 2933, 1740, 1704, 1521, 1174, 748 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz): δ = 7.36 (s, 5H); 5.16 (s, 2H); 4.70 (bs, 1H); 3.44 (s, 2H); 3.12-3.09 (m, 2H); 2.64-2.58 (m, 2H); 1.56-1.47

(m, 13H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 172.4 (C); 155.9 (C); 135.5 (C); 128.6 (2CH); 128,3 (3CH); 78.6 (C); 66.5 (CH_2); 50.8 (CH_2); 49.0 (CH_2); 40.3 (CH_2); 28.3 ($3CH_3$); 27.6 (CH_2); 27.2 (CH_2); HRMS: *m/z* calcd for $[C_{18}H_{28}N_2O_4+H]^+$ 337.2122, measured 337.2131; The obtained spectra were in accordance with reported procedure.[5]

Benzyl 2-(6-(tert-butoxycarbonylamino)hexylamino)acetate (3c): 235.9 mg (39%); IR (ATR): 3343, 2931, 2859,1740, 1707, 1523, 1173, 755 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.34 (s, 5H); 5.15 (s, 2H), 4.85 (bs, 1H); 3.43 (s, 2H); 3.09-3.06 (m, 2H); 2.58 (t, 2H , *J*=6.8); 1.54-1.34 (m, 17H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 171.9 (C,); 155.5 (C); 135.1 (C); 128.1 (2CH); 127.9 (3CH); 78.3 (C); 65.9 (CH_2); 50.4 (CH_2); 48.9 (CH_2); 39.9 (CH_2); 29.4 (CH_2); 27.9 ($3CH_3$); 26.3 (CH_2); 26.1 (CH_2); HRMS: *m/z* calcd for $[C_{20}H_{32}N_2O_4+H]^+$ 365.2435, measured 365.2445; The obtained spectra were in accordance with reported procedure.[6]

Benzyl 2-(8-(tert-butoxycarbonylamino)octylamino)acetate (3e): 364.0 mg (58%); IR (ATR): 3357, 2930, 2859, 1741, 1689, 1172, 737 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.36 (s, 5H); 5.17 (s, 2H); 3.45 (s, 2H); 3.14-3.04 (m, 2H); 2.59 (t, 2H , *J*=7); 1.56-1.26 (m, 22H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 172.5 (C); 155.9 (C); 135.6 (C); 128.6 (2CH); 128.3 (3CH); 78.9 (C); 66.4 (CH_2); 50.9 (CH_2); 49.5 (CH_2); 40.5 (CH_2); 30.0 (CH_2); 29.3 (CH_2); 29.1 (CH_2); 28.3 ($3CH_3$); 27.0 (CH_2); 26.6 (CH_2); HRMS: *m/z* calcd for $[C_{22}H_{36}N_2O_4+H]^+$ 393.2748, measured 393.2753;

Benzyl 2-(10-(tert-butoxycarbonylamino)decylamino)acetate (3e): 286.2 mg (48%); IR (ATR): 3340, 2927, 2854, 1712, 1518, 1174, 735; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.36 (s, 5H); 5.17 (s, 2H); 4.52 (bs, 1H); 3.46 (s, 2H); 3.09 (q, 2H, *J*=6.2); 2.59 (t, 2H , *J*=6.6); 1.60-1.44 (m, 13H); 1.33-1.15 (m, 12H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 172.5 (C); 168.7(C); 135.6 (C); 128.6 (2CH); 128,4 (3CH); 78.3 (C); 66,5 (CH_2); 50.9 (CH_2); 49.6 (CH_2); 40.6 (CH_2); 30.0 (CH_2); 29.4 ($2CH_2$); 29.2 ($2CH_2$); 28.4 ($3CH_3$); 27.1 (CH_2); 26.7 (CH_2); HRMS: *m/z* calcd for $[C_{24}H_{40}N_2O_4+H]^+$ 421.3061, measured 421.3043;

Benzyl 2-(12-(tert-butoxycarbonylamino)dodecylamino)acetate (3f): 445.0 mg, (62%); IR (ATR): 3353, 2923, 2855, 1731, 1521, 1177, 745 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.36 (s, 5H); 5.17 (s, 2H); 4.53 (bs, 1H); 3.45 (s, 2H); 3.09 (q, 2H, *J*=6.2); 2.59 (t, 2H , *J*=7.2); 1.60-1.44 (m, 13H); 1.33-1.14 (m, 16H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 172.5 (C); 160.0(C) ; 135.6 (C); 128.6 (2CH); 128.3 (3CH); 78.9 (C); 66.4 (CH_2); 50.9 (CH_2); 49.6 (CH_2); 40.6 (CH_2); 30.0 (CH_2); 29.5 ($2CH_2$); 29.4 ($2CH_2$); 29.2 ($2CH_2$); 28.4 ($3CH_3$); 27.1 (CH_2); 26.7 (CH_2); HRMS: *m/z* calcd for $[C_{26}H_{44}N_2O_4+H]^+$ 449.3374, measured 449.3369;

Preparation of acids (4a-f)

Pd/C (20 mg, 1 mol %) was added to a solution of **3a-f** (0.8 mmol) in methanol (20 mL) and the obtained suspension was hydrogenated for 4 h with 50 psi H_2 at room temperature. The catalyst was removed by filtration and the solvent was evaporated to dryness, leaving acids as white solids in 77-100% yields, which were used in the next step without further purification.

2-(2-(tert-butoxycarbonylamino)ethylamino)acetic acid (4a): 174.5 mg (91%); The obtained spectra were in accordance with reported procedure.[4]

2-(4-(tert-butoxycarbonylamino)butylamino)acetic acid (4b): 191.1 mg (97%); m.p. 173.0-177.4 ^{o}C; IR: 3368, 2979, 2867, 1690, 1636, 1387, 1177 cm^{-1}; ^{1}H NMR (CD_3OD, 200 MHz): δ = 3.50 (s, 2H); 3.10-2.88 (m, 4H); 1.79-1.68 (m, 2H); 1.60-1.31 (m, 11H); ^{13}C NMR (CD_3OD, 50 MHz): δ = 171.2 (C); 158.5 (C); 79.9 (C); 50.6 (CH_2); 48.2 (CH_2); 40.5 (CH_2); 28.8 ($3CH_3$); 28.0 (CH_2); 24.5 (CH_2); HRMS: *m/z* calcd for $[C_{11}H_{22}N_2O_4+H]^+$ 247.1652, measured 247.1653;

2-(6-(tert-butoxycarbonylamino)hexylamino)acetic acid (4c): 220 mg (100%); m.p. 169.7-173.1 ^{o}C; IR (ATR): 3371, 2978, 2861, 1690, 1617, 1389, 1176 cm^{-1}; ^{1}H NMR (CD_3OD, 200 MHz): δ = 3.47 (s, 2H); 3.05-2.87 (m, 4H); 1.72-1.61 (m, 3H); 1.54-1.30 (m, 14H); ^{13}C NMR (D_2O, 50 MHz): δ = 170.9 (C); 158.5 (C); 79.8 (C); 50.6 (CH_2); (CH_2); 41.1 (CH_2); 30.7 (CH_2); 28.8 ($3CH_3$); 27.3 (CH_2); 27.1 (CH_2); HRMS: *m/z* calcd for $[C_{13}H_{26}N_2O_4+H]^+$ 275.1965, measured 275.1969;

2-(8-(tert-butoxycarbonylamino)octylamino)acetic acid (4d): 186.1 mg (77%); m.p. 119.5-121.7 ^{o}C; IR (ATR): 3362, 2978, 2856, 1689,1596, 1386, 1176 cm^{-1}; ^{1}H NMR (CD_3OD, 200 MHz): δ = 3.59 (s, 2H); 3.08-2.99 (m, 4H); 1.80-1.54 (m, 4H); 1.42 (s, 9H); 1.39-1.28 (m, 8H); ^{13}C NMR (D_2O, 50 MHz): δ = 174.2 (C); 161.3 (C); 79.0 (C); 56.8 (CH_2); 51.9 (CH_2); 50.2 (CH_2); 30.8 (CH_2); 30.4 (CH_2); 28.5 (CH_2); 28.3 ($3CH_3$); 28.2 (CH_2); 21.8 (CH_2); 17.7 (CH_2); HRMS: *m/z* calcd for $[C_{15}H_{30}N_2O_4+H]^+$ 303.2278, measured 303.2275;

2-(10-(tert-butoxycarbonylamino)decylamino)acetic acid (4e): 242.0 mg (100%); m.p. 120.3-123.5 ^{o}C; IR (ATR): 3380, 2924, 2854, 1691, 1564, 1370, 1176 cm^{-1}; ^{1}H NMR (D_2O, 500 MHz): δ = 3.59 (s, 2H); 3.01-2.95 (m, 4H); 1.73-1.64 (m, 2H); 1.41-1.31 (m, 23H); ^{13}C NMR (D_2O, 125 MHz): δ = 170.9 (C); 158.7 (C); 79.9 (C); 50.7 (CH_2); 42.5 (CH_2); 41.1 (CH_2); 31.1 (CH_2); 30.7 (CH_2); 30.6 (CH_2); 30.5 (CH_2); 30.3 (CH_2); 28.9 ($3CH_3$); 27.9 (CH_2); 27.7 (CH_2); 27.4 (CH_2); HRMS: *m/z* calcd for $[C_{17}H_{34}N_2O_4+H]^+$ 331.2591, measured 331.2606;

2-(12-(tert-butoxycarbonylamino)dodecylamino)acetic acid (4f): 275.2 mg (96%); mp 164.5-167.3 ^{o}C ; IR (ATR): 3380, 2920, 2852, 172, 1691, 1382, 1174 cm^{-1}; ^{1}H NMR (CD_3OD, 200 MHz): δ = 3.46 (s, 2H); 3.04-2.93 (m, 4H); 1.74-1.64 (m, 3H); 1.42-1.30 (m, 26H); ^{13}C NMR (CD_3OD, 50 MHz): δ = 170.7 (C); 168.9 (C); 79.7 (C); 50.6 (CH_2); 41.3 (CH_2); 30.9 (CH_2); 30.7 (CH_2); 30.5 (CH_2); 30.2 (CH_2); 28.9 ($3CH_3$); 27,8 (CH_2); 27.5 (CH_2); 27.2 (CH_2); HRMS: *m/z* calcd for $[C_{19}H_{38}N_2O_4+H]^+$ 359.2904, measured 359.2918;

Preparation of Boc-protected aminoalkyl fulleropyrrolidines (5a-f)

A suspension of C_{60} (0.1 mol), acid **5a-f** (0.1 mmol) and paraformaldehyde (0.5 mmol) in toluene (100 mL) was refluxed for 45 min. The reaction mixture was cooled down and the solvent was evaporated to dryness. Column chromatography on SiO_2 using toluene gave unreacted C_{60}. Further elution with EtOAc/toluene (1/9) and subsequent precipitation, from

CH_2Cl_2/CS_2 highly concentrated solution with MeOH, gave pure products **5a-f** as brown powders in 21-36% yields.

Boc-protected aminoethyl fulleropyrrolidine (**5a):** 24.5 mg (27%); The obtained spectra were in accordance with reported procedure.[7]

Boc-protected aminobutyl fulleropyrrolidine (5b): 19.5 mg (21%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 309, 320 and 431 (ε /$dm^3mol^{-1}cm^{-1}$ 120000, 49000, 56000 and 4600); IR (ATR): 3440, 2927, 2777, 1708, 1428, 1166, 526; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 4.88 (bs, 1H); 4.41 (s, 4H); 3.34-3.25 (m, 2H); 3.11 (t, 2H, *J*=7); 1.98 (quintet, 2H, *J=7*) ; 1.85 (quintet, 2H, *J*= 7); 1.45 (s, 9H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 155.8 (C); 154.8 (C_f(12)); 147.2 (C_f(17)); 146.2 (C_f(7)); 146.0 (C_f(11)); 145.9 (C_f(16)); 145.6 (C_f(5)); 145.3 (C_f(9)); 144.5 (C_f(15)); 143.0 (C_f(8)); 142.6 (C_f(6)); 142.2 (C_f(14)); 141.9 (C_f(4)); 141.8 (C_f(12,13)); 140.8 (C_f(10)); 136.2 (C_f(3)); 78.9 (C); 70.5 (2C); 67.8 ($2CH_2$); 54.3 (CH_2); 40.5 (CH_2); 28.4 ($3CH_3$); 28.0 (CH_2); 26.2 (CH_2): HRMS: *m/z* calcd for $[C_{71}H_{22}N_2O_2+H]^+$ 935.1754, measured 935.1785

Boc-protected aminohexyl fulleropyrrolidine (5c): 34.7 mg (36%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 252, 308, 322 and 432 (ε /$dm^3mol^{-1}cm^{-1}$ 130000, 48000, 55000 and 4400); IR (ATR): 3442, 2928, 2775, 1688, 1428, 1166, 526. cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 4.55 (bs, 1H); 4.39 (s, 4H); 3.19-3.18 (m, 2H); 3.07 (t, 2H, *J*=7.5); 1.95 (quintet, 2H); 1.68-1.58 (m, 4H); 1.54-1.49 (m, 11H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 155.7 (C); 154.9 (C_f(12)); 147.2 (C_f(17)); 146.1 (C_f(7)); 146.0 (C_f(11)); 145.9 (C_f(16)); 145.6 (C_f(5)); 145.3 (C_f(9)); 144.5 (C_f(15)); 142.9 (C_f(8)); 142.5 (C_f(6)); 142.1 (C_f(14)); 141.9 (C_f(4)); 141.8 (C_f(12,13)); 140.1 (C_f(10)); 136.1 (C_f(3)); 78.8 (C); 70.5 (2C); 67.9 ($2CH_2$); 54.9 (CH_2); 40.6 (CH_2); 30.2 (CH_2); 28.8 (CH_2); 28.4 ($3CH_3$); 27.4 (CH_2); 26.8 (CH_2); HRMS: *m/z* calcd for $[C_{73}H_{28}N_2O_2+H]^+$ 963.2067, measured 963.2067;

Boc-protected aminooctyl fulleropyrrolidine (5d): 22.8 mg (23%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 308, 320 and 430 (ε /$dm^3mol^{-1}cm^{-1}$ 130000, 51000, 54000 and 4800); IR (ATR): 3446, 2928, 2796, 1703, 1513, 1171, 737 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 4.44 (bs, 1H); 4.39 (s, 4H); 3.14-3.10 (m, 2H); 3.08-3.05 (m, 2H); 1.96-1.90 (m, 2H); 1.65-1.59 (m, 2H); 1.53-1.43 (m, 11H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 155.5 (C); 154.9 (C_f(12)); 147.1 (C_f(17)); 146.1 (C_f(7)); 146.0 (C_f(11)); 145.5 C_f(16)); 145.3 (C_f(5)); 145.2(C_f(9)); 144.4 (C_f(15)); 143.0 (C_f(8)); 142.5 (C_f(6)); 142.1 (C_f(14)); 141.9 (C_f(4)); 141.8 (C_f(12,13)); 140.0 (C_f(10)); 136.1 (C_f(3)); 78.9 (C); 70.5 (2C); 67.9 ($2CH_2$); 55.0 (CH_2); 40.6 (CH_2); 30.21 (CH_2); 29.7 (CH_2); 29.4 (CH_2); 28.9 (CH_2); 28.3 ($3CH_3$); 27.7 (CH_2); 26.9 (CH_2); HRMS: *m/z* calcd for $[C_{75}H_{32}N_2O_2+H]^+$ 991.2380, measured 991.2386;

Boc-protected aminodecyl fulleropyrrolidine (**5e):** 27.5 mg (27%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 309, 321 and 430 (ε /$dm^3mol^{-1}cm^{-1}$ 115000, 49000, 54000 and 4900); IR (ATR); 3446, 2928, 2855, 1703, 1513, 1171, 737; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 4.72 (bs, 1H);

4.41(s, 4H); 3.12-3.04 (m, 2H); 2.02-1.87 (m, 2H); 1.57-1.13 (m, 24H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 155.9 (C); 155.0 (C_f(12)); 147.3 (C_f(17)); 146.2 (C_f(7)); 146.0 (C_f(11)); 145.7 (C_f(16)); 145.4 (C_f(5)); 145.3 (C_f(9)); 144.6 (C_f(15)); 143.1 (C_f(8)); 142.6 (C_f(6)); 142.3 (C_f(14)); 142.1 (C_f(4)); 141.9 (C_f(12,13)); 140.1 (C_f(10)); 136.2 (C_f(3)); 78.9 (C); 70.5 (2C); 68.0 ($2CH_2$); 55.3 (CH_2); 40.6 (CH_2); 30.1 ($2CH_2$); 29.6 ($2CH_2$); 29.3 (CH_2); 28.9 (CH_2); 28.4 ($3CH_3$,); 27.7 (CH_2); 26.8 (CH_2); MALDI/TOF: *m/z* measured for $[C_{77}H_{36}N_2O_2+H]^+$

Boc-protected aminododecyl fulleropyrrolidine 5f: 24.1 mg (23%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 254, 310, 322 and 432 (ε /$dm^3mol^{-1}cm^{-1}$ 120000, 48000, 55000 and 4800); IR (ATR): 3448, 3366, 2927, 1741, 1464, 1174 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 4.39 (s, 4H); 3.09-3.05 (s, 2H); 1.96-1.90 (m, 2H); 1.64-1.90 (s, 2H); 1.49-1.30 (s, 25H); ^{13}C NMR ($CDCl_3$, 125 Mhz): δ = 155.1 (C_f(12)); 147.3 (C_f(17)); 146.3 (C_f(7)); 146.1 (C_f(11)); 146.0 (C_f(16)); 145.7 (C_f(5)); 145.5 (C_f(9)); 144.6 (C_f(15)); 143.1 (C_f(8)); 142.7 (C_f(6)); 142.3 (C_f(14)); 142.1 (C_f(4)); 141.9 (C_f(12,13)); 140.2 (C_f(10)); 136.3 (C_f(3)); 70.7 (2C); 68.1 ($2CH_2$,); 55.2 (CH_2); 40.7 (CH_2); 30.4 (CH_2); 29.9 ($4CH_2$); 29.6 ($2CH_2$); 29.1 (CH_2); 28.4 ($3CH_3$); 27.9 (CH_2); 27.0 (CH_2); HRMS: *m/z* calcd for $[C_{79}H_{40}N_2O_2+H]^+$ 947.2482, measured 947.2482;

Preparation of fulleropyrrolidine alkyl ammonium salts (6a-f)

A solution of *t*-butyl ester **5a-f** (0.02 mmol) in 0.45 mL CH_2Cl_2/TFA mixture (1/2) was stirred at room temperature for 2 h and than evaporated to dryness. Excess of TFA was removed by co-evaporation with toluene leaving amines **6a-f** as dark brown powders in almost quantitative yields.

6a: 17.0 mg (94%); The obtained spectra were in accordance with reported procedure.[8]

6b: 19.0 mg (100%); UV-VIS (MeOH): λ_{max} (nm): 254, 308, 320 and 431 (ε /$dm^3mol^{-1}cm^{-1}$ 130000, 47000, 58000 and 4900); IR (ATR): 2946, 1675, 1200, 798, 722, 525 cm^{-1}; HRMS: *m/z* calcd for $[C_{66}H_{14}N_2+H]^+$ 835.1230, measured 835.1228;

6c: 19.5 mg (100%); UV-VIS (MeOH): λ_{max} (nm): 254, 309, 322 and 430 (ε /$dm^3mol^{-1}cm^{-1}$ 119000, 47000, 58000 and 4600); IR (ATR): 2915, 1668, 1167, 794, 725, 523 cm^{-1}; HRMS: *m/z* calcd for $[C_{68}H_{18}N_2+H]^+$ 863.1543, measured 863.1527;

6d: 15.5 mg (97%); UV-VIS (MeOH): λ_{max} (nm): 252, 306, 321 and 431 (ε /$dm^3mol^{-1}cm^{-1}$ 110000, 48000, 54000 and 4900); IR (ATR): 2926, 1666, 1180, 798, 720, 522 cm^{-1}; HRMS: *m/z* calcd for $[C_{70}H_{22}N_2+H]^+$ 891.1856, measured 891.1851;

6e: 20.7 mg (100%); UV-VIS (MeOH): λ_{max} (nm): 254, 309, 320 and 432 (ε /$dm^3mol^{-1}cm^{-1}$ 120000, 51000, 55000 and 4600); IR (ATR): 2928, 1673, 1137, 796, 721, 523 cm^{-1}; HRMS: *m/z* calcd for $[C_{72}H_{26}N_2+H]^+$ 919.2169, measured 919.2129;

6f: 21.2 mg (100%); UV-VIS (MeOH): λ_{max} (nm): 253, 308, 322 and 432 (ε /$dm^3mol^{-1}cm^{-1}$ 125000, 48000, 54000 and 4700); IR (ATR): 2923, 1683, 1181, 557 cm^{-1} HRMS: *m/z* calcd for $[C_{74}H_{30}N_2+H]^+$ 947.2482 measured 947.2483;

Preparation of fulleropyrrolidine phthalimide dyads 1a-f

A suspension of **6a-f** (0.015 mmol) and phthalic anhydride (0.015 mmol eq.) in AcOH/Pyr, 3:2 mixture (1 mL) was irradiated in microwave reactor for 30 min., with inner temperature 130°C and applied pulse of 300W. Obtained reaction mixture was evaporated to dryness and the excess of acetic acid was removed by co-evaporation with toluene. The crude product was purified by column chromatography on SiO_2 with EtOAc-toluene (9/1) mixture as an eluent. Subsequent precipitation from CH_2Cl_2/CS_2 highly concentrated solution with MeOH gave pure products **1a-f** as a brown powder in 40-59% yields.

Compound 1a: 8.3 mg (59%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 254, 308, 320, 431 and 704 (ε /$dm^3mol^{-1}cm^{-1}$ 120000, 48000, 55000, 4600 and 650); IR(ATR): 3464, 2926, 1711, 1391, 717 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.89-7.87 (m, 2H); 7.73-7.71 (m, 2H); 4.48 (s, 4H); 4.27 (t, 2H, *J*=6); 3.47 (t, 2H, *J*=6); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 168.5 (2C); 154.7 (C_f(12)); 147.2 (C_f(17)); 146.2 (C_f(7)); 146.1 (C_f(11)); 146.0 (C_f(16)); 145.3 (C_f(5)); 145.2 (C_f(9)); 144.5 (C_f(15)); 143.0 (C_f(8)); 142.5 (C_f(6)); 142.1 (C_f(14)); 142.0 (C_f(4)); 141.8 (C_f(12,13)); 140.0 (C_f(10)); 136.1 (C_f(3)); 133.9 (2C); 132.3 (2CH); 123.4 (2CH); 70.6 (2C); 67.8 ($2CH_2$); 50.9 (CH_2); 36.6 (CH_2); HRMS: *m/z* calcd for $[C_{72}H_{12}N_2O_2+H]^+$ 937.0971, measured 937. 0993;

Compound 1b: 5.8 mg (40%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 309, 321, 430 and 703 (ε /$dm^3mol^{-1}cm^{-1}$ 130000, 49000, 56000, 4800 and 700); IR (ATR): 3453, 2927, 2854, 1710, 1395, 710 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 7.88-7.82 (m, 2H); 7.75-7.69 (m, 2H); 4.38 (s, 4H); 3.88 (t, 2H, *J*=6); 3.13 (t, 2H, *J*=6); 2.08-1.94 (m, 4H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ 163.5 (2C); 154.8 (C_f(12)); 146.1 (C_f(11)); 145.9 (C_f(16)); 145.3 (C_f(5)); 145.1 (C_f(9)); 144.4 (C_f(15)); 143.0 (C_f(8)); 142.5 (C_f(6)); 142.1 (C_f(14)); 141.9 (C_f(4)); 141.7 (C_f(12,13)); 140.0 (C_f(10)); 136.1 (C_f(3)); 133.7 (2C); 132.1 (2CH); 123.0 (2CH); 70.4 (2C); 67.7 ($2CH_2$); 54.1 (CH_2); 37.6 (CH_2); 26.6 (CH_2); 26.0 (CH_2); HRMS: *m/z* calcd for $[C_{74}H_{16}N_2O_2+H]^+$ 965.1284, measured 965.1285; HRMS: $[M+H]^+$ calcd 965.1284, measured 965.1285;

Compound 1c: 7.5 mg (51%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 308, 320, 430 and 703 (ε /$dm^3mol^{-1}cm^{-1}$ 118000, 47000, 56000, 4900 and 500); IR (ATR): 3442, 2928, 2852, 1688, 1521, 718 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.87-7.81 (m, 2H); 7.76-7.69 (m, 2H); 4.39 (s, 2H);3.77 (t, 2H, *J*=7.2); 3.08 (t, 2H, *J*=7.2); 1.95-1.46 (m, 12H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 167.8 (2C); 155.1 (C_f(12)); 147.3 (C_f(17)); 146.2 (C_f(7)); 146.0 (C_f(11)); 145.4 (C_f(16)); 145.2 (C_f(5)); 145.1 (C_f(9)); 144.5 (C_f(15)); 143.0 (C_f(8)); 142.5 (C_f(6)); 142.2 (C_f(14)); 142.0 (C_f(4)); 141.8 (C_f(12,13)); 140.1 (C_f(10)); 136.2 (C_f(3)); 133.8 (2C); 132.1 (2CH; 123.2 (2CH); 70.6 (2C); 67.9 ($2CH_2$); 54.9 (CH_2); 37.9 (CH_2); 28.6 ($2CH_2$); 27.2 (CH_2); 26.8 (CH_2); HRMS: *m/z* calcd for $[C_{76}H_{20}N_2O_2+H]^+$ 993.1597, measured 993.1593;

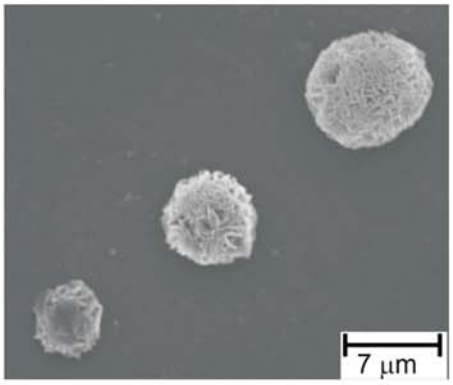
Compound 1d: 6.9 mg (45%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 254, 310, 322, 432 and 704 (ε /$dm^3mol^{-1}cm^{-1}$ 130000, 50000, 56000, 5100 and 700); IR (ATR): 3457, 2925, 2851, 1711, 1393, 717; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.86-7.79 (m, 2H); 7.74-7.66 (m, 2H); 4.39 (s, 4H); 3.71 (t, 2H, *J*=7.6); 3.06 (t, 2H, *J*=7.6); 2.03-1.86 (2H); 1.80-1.39 (m, 4H); ^{13}C NMR ($CDCl_3$, 50

MHz): δ = 168.3 (2C); 155.1 (C_f(12)); 147.2 (C_f(17)); 146.2 (C_f(7)); 146.1 (C_f(11)); 145.9 (C_f(16)); 145.6 (C_f(5)); 145.3 (C_f(9)); 145.2 (C_f(15)); 144.5 (C_f(8)); 142.5 (C_f(6)); 142.2 (C_f(14)); 142.0 (C_f(4)); 141.8 (C_f(12,13)); 140.1 (C_f(10)); 136.2 (C_f(3)); 133.8 (2C); 132.2 (2CH); 123.1 (2CH); 70.6 (2C); 67.9 ($2CH_2$); 55.1 (CH_2); 37.9 (CH_2); 29.5 ($2CH_2$); 29.2 ($2CH_2$); 28.8 (CH_2); 28.6 (CH_2); 27.6 (CH_2); 26.8 (CH_2); HRMS: *m/z* calcd for $[C_{78}H_{24}N_2O_2+H]^+$ 1021.1910, measured 1021.1912;

Compound 1e: 9.1 mg (58%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 252, 308, 322, 432 and 704 (ε /$dm^3mol^{-1}cm^{-1}$ 125000, 47000, 54000, 4800 and 600); IR (ATR): 3464, 2926, 2851, 1711, 1394, 718; ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 7.86-7.75 (m, 2H); 7.72-7.68 (m, 2H); 4.40 (s, 4H); 3.69 (t, 2H, *J*=7.5); 3.07 (t, 2H, *J*=7.5); 2.01-1.86 (m, 2H); 1.72-1.24 (m, 14H); ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 168.3 (2C); 155.1 (C_f(12)); 147.2 (C_f(17)); 146.2 (C_f(7)); 146.1 (C_f(11)); 146.0 (C_f(16)); 145.4 (C_f(5)); 145.3 (C_f(9)); 145.2 (C_f(15)); 144.5 (C_f(8)); 142.5 (C_f(6)); 142.2 (C_f(14)); 142.0 (C_f(4)); 141.8 (C_f(12,13)); 140.1 (C_f(10)); 136.2 (C_f(3)); 133.8 (2C); 132.2 (2CH); 123.1 (2CH); 70.6 (2C); 67.9 ($2CH_2$); 55.1 (CH_2); 38.0 (CH_2); 29.6 (CH_2); 29.5 (CH_2) 29.2 ($2CH_2$); 28.9 (CH_2); 28.6 (CH_2); 27.0 (CH_2); 26.9 (CH_2); HRMS: *m/z* calcd for $[C_{80}H_{28}N_2O_2+H]^+$ 1049.2223, measured 1049.2234;

Compound 1f: 9.2 mg (57%); UV-VIS (CH_2Cl_2): λ_{max} (nm): 253, 309, 320, 431 and 703 (ε /$dm^3mol^{-1}cm^{-1}$ 118000, 49000, 54000, 4800 and 540); IR(ATR): 3662, 2926, 2855, 1688, 1386, 720; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.85-7.83 (m, 2H); 7.71-7.69 (m, 2H); 4.41 (s, 4H); 3.68 (t, 2H, *J*=7.4); 3.08 (t, 2H, *J*=7.4); 1.98-1.92 (2H); 1.69-1.59 (m, 4H); 1.51-1.45 (2H); 1.45-1.32 (12H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 168.5 (2C); 155.2 (C_f(12)); 147.3 (C_f(17)); 146.2 (C_f(7)); 146.1 (C_f(11)); 146.0 (C_f(16)); 145.7 (C_f(5)); 145.4 (C_f(9)); 145.3 (C_f(15)); 144.6 (C_f(8)); 142.6 (C_f(6)); 142.3 (C_f(14)); 142.1 (C_f(4)); 141.9 (C_f(12,13)); 140.1 (C_f(10)); 136.2 (C_f(3)); 133.8 (2C); 132.2 (2CH); 123.1 (2CH); 70.7 (2C); 68.0 ($2CH_2$); 55.2 (CH_2); 38.1 (CH_2); 29.6 ($3CH_2$); 29.5 (CH_2); 29.2 ($2CH_2$); 28.8 (CH_2); 28.6 (CH_2); 27.7 (CH_2); 26.9 (CH_2); HRMS: *m/z* calcd for $[C_{82}H_{32}N_2O_2+H]^+$ 1077.2536, measured 1077.2550;

a)



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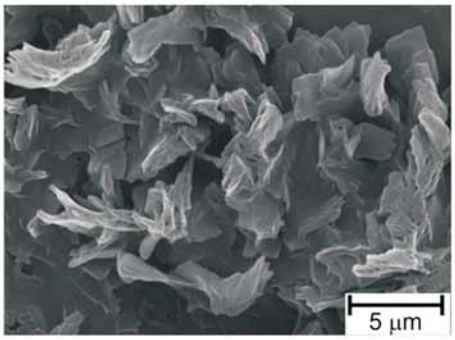

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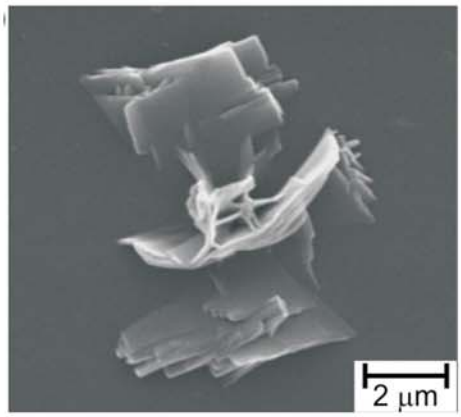
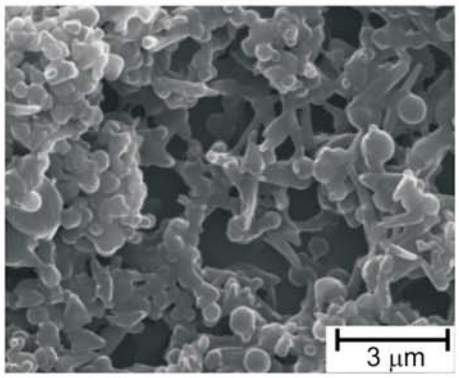
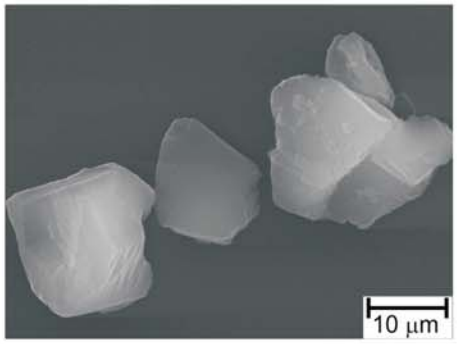


SEM images of **1b** prepared from:
a) PhMe, -20°C, 12h
b) PhMe, r.t.
c) PhMe/iPrOH (2/1), r.t.

d)



e)



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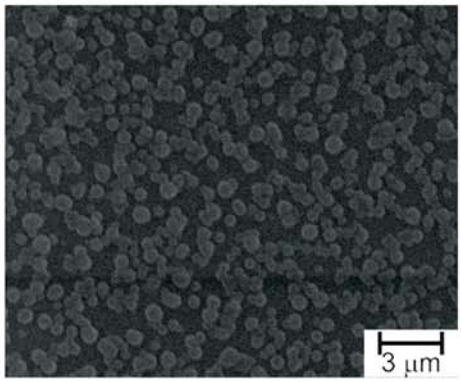
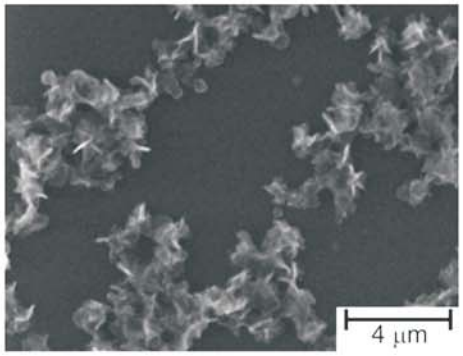
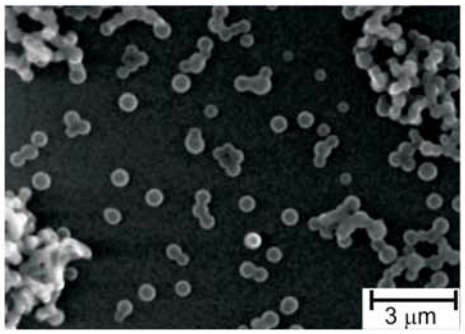


SEM images of **1c** prepared from:
d) MeOH, r.t.
e) PhMe, r.t.
f) PhMe/dioxane (2/1), r.t.

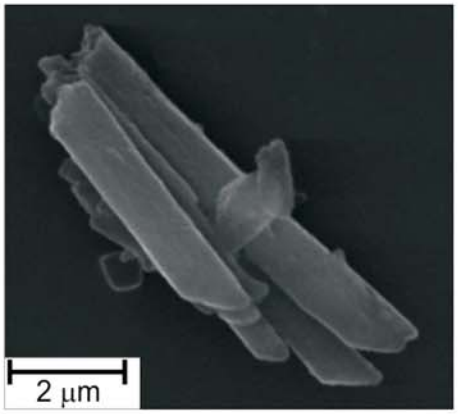


SEM image of **1d** prepared from $CHCl_3$/MeOH (2/1), r.t.

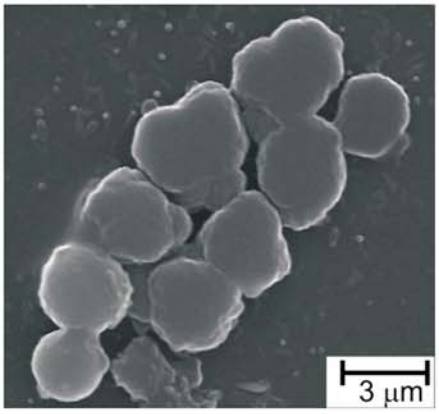


SEM image of **1e** prepared from $CHCl_3$/MeOH (2/1), r.t.

a)



b)



SEM images of **1f** prepared from:
a) dioxane, r.t.
b) PhMe/dioxane (2/1), r.t.

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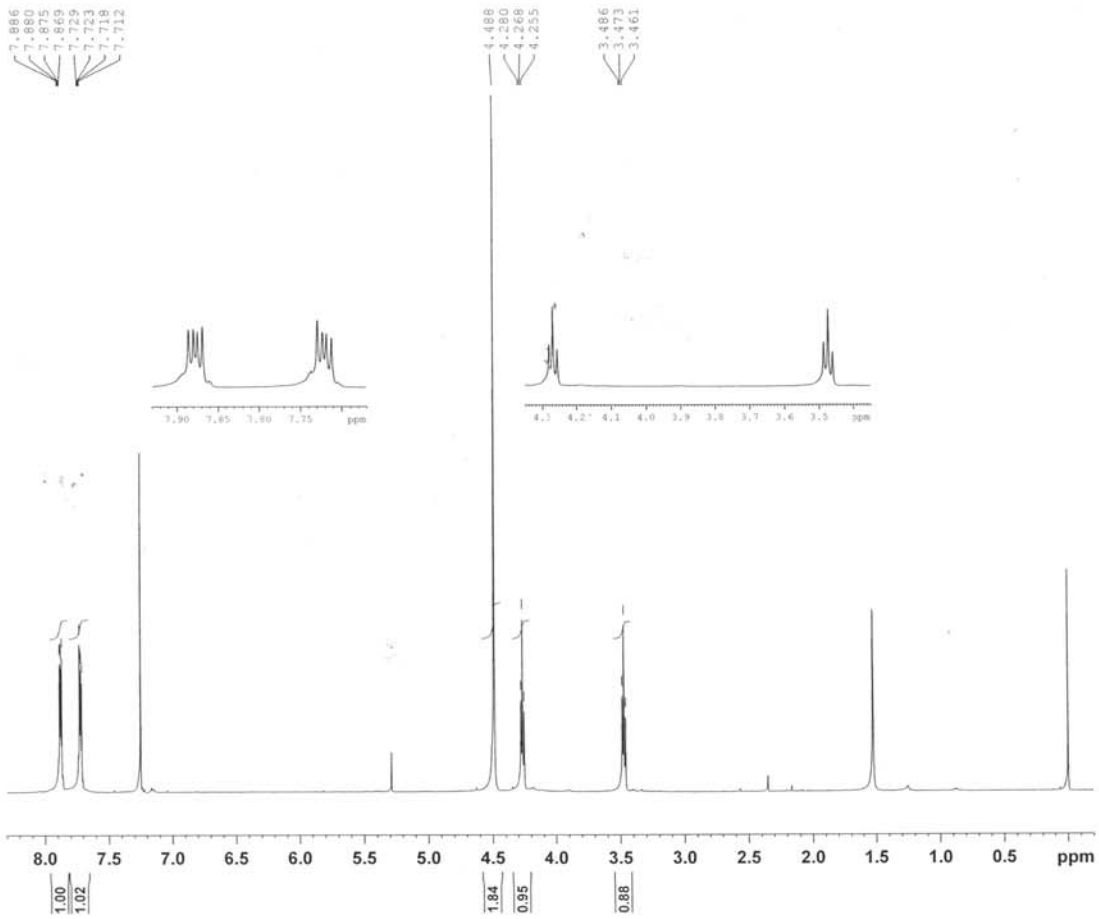


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FIDRES        0.140937 Hz
AQ            3.5477321 sec
RG                  362
DW             108.267 usec
DE                6.50 usec
TE              298.0 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
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PL1                0.00 dB
PL1W       27.37956238 W
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LB                 0.30 Hz
GB                    0
PC                 1.00
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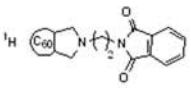

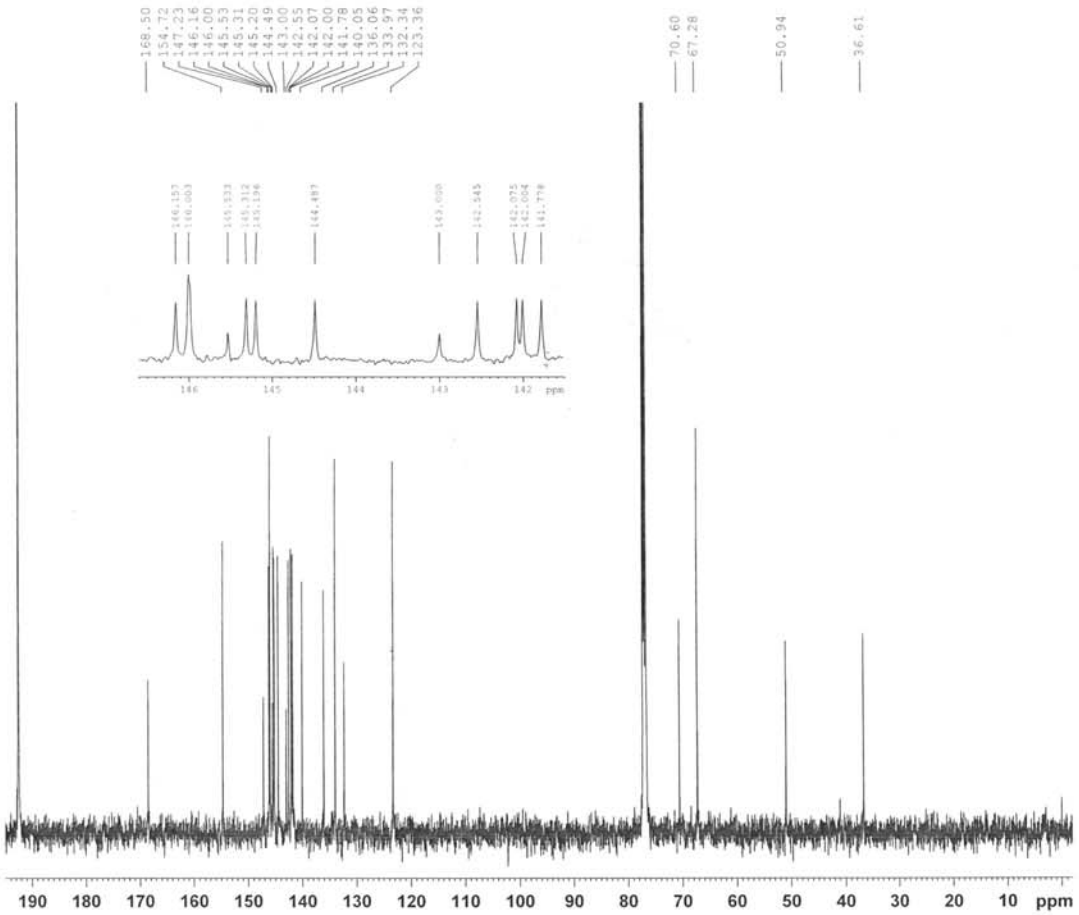


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TD                32768
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TE              298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.50 usec
PL1                3.00 dB
PL1W       32.22848892 W
SFO1       125.8043140 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.20 dB
PL12              18.40 dB
PL13              18.40 dB
PL2W       20.76952171 W
PL12W       0.39575511 W
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SF         125.7904872 MHz
WDW                  EM
SSB                   0
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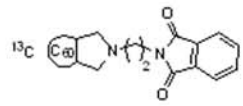
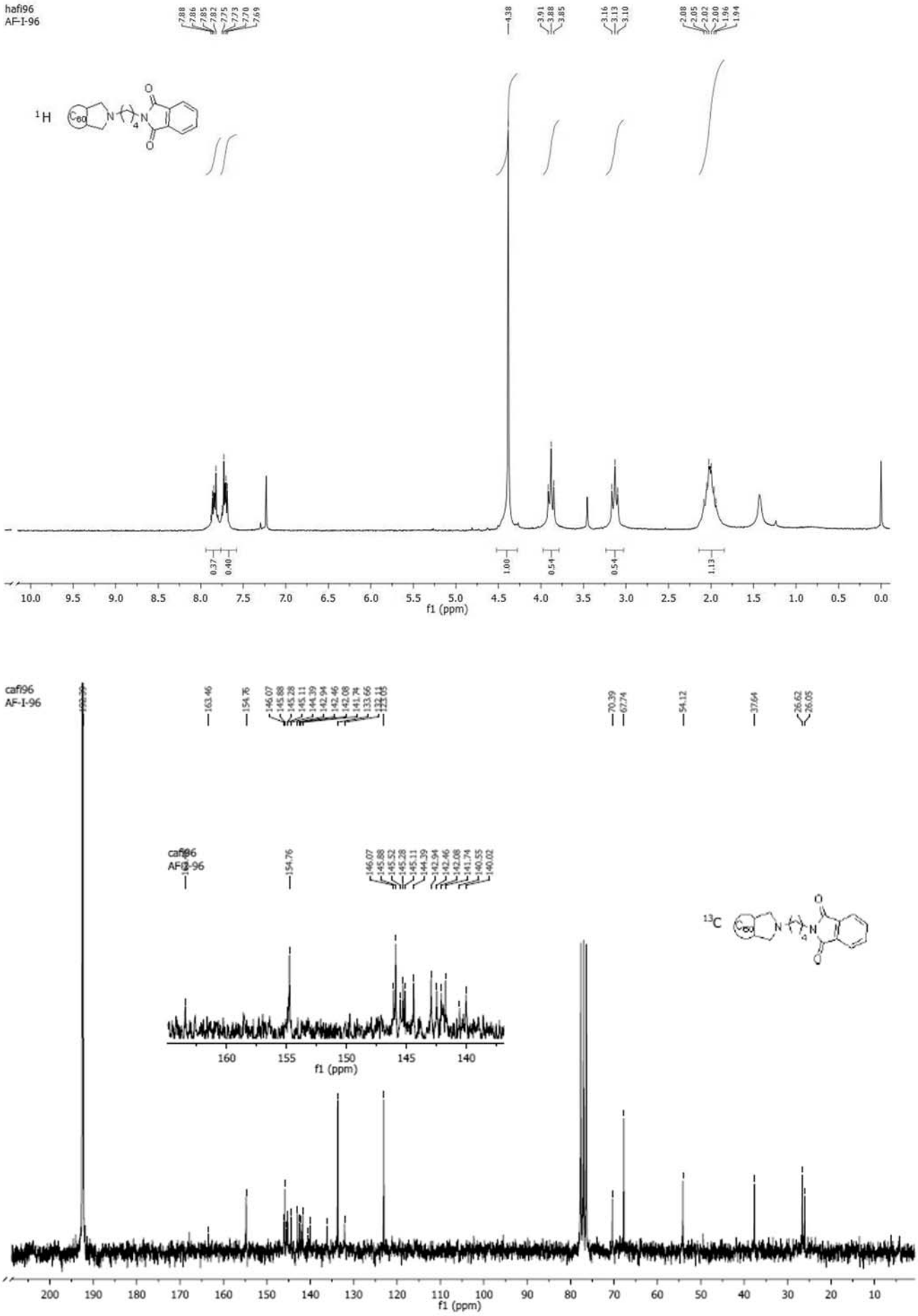
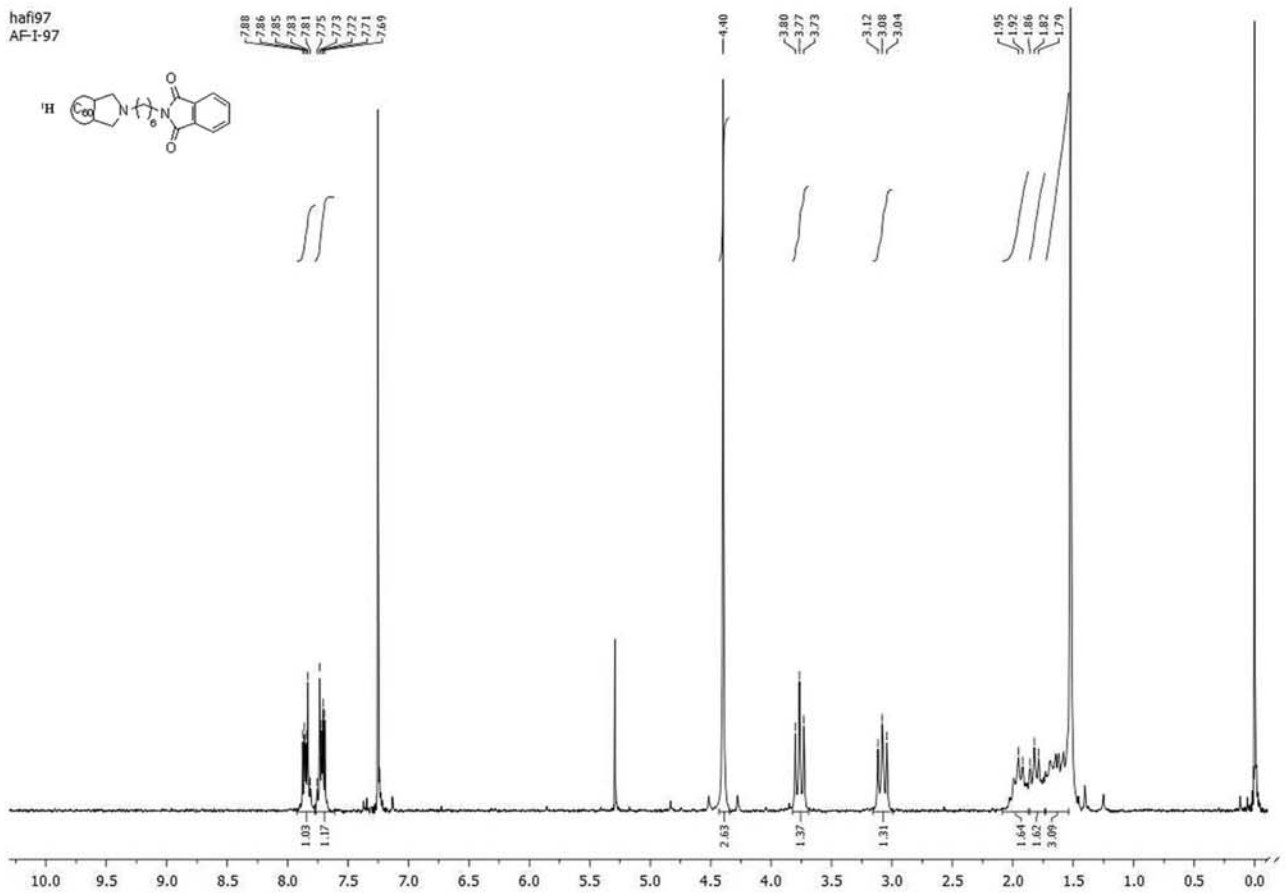
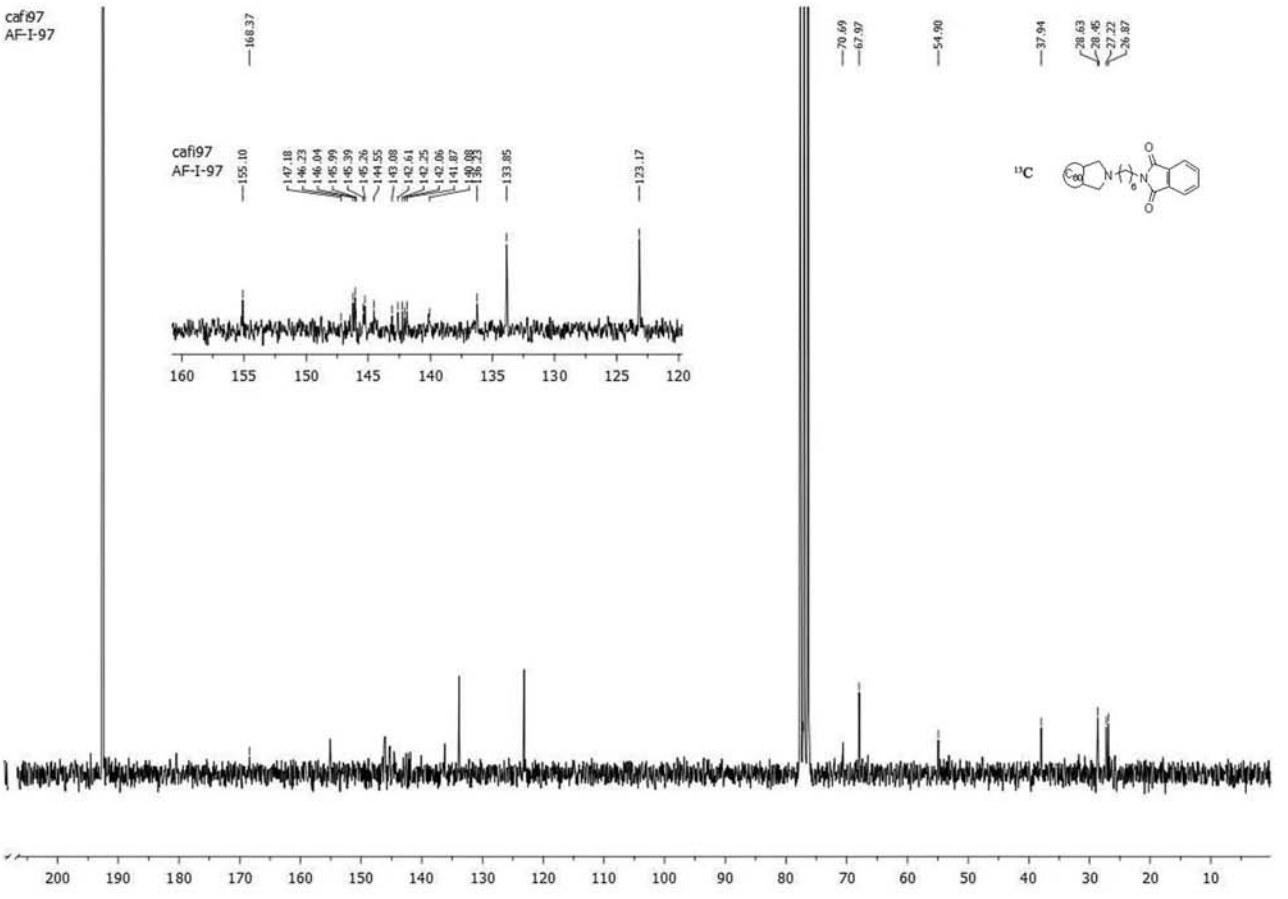


NMR spectra of **1a**

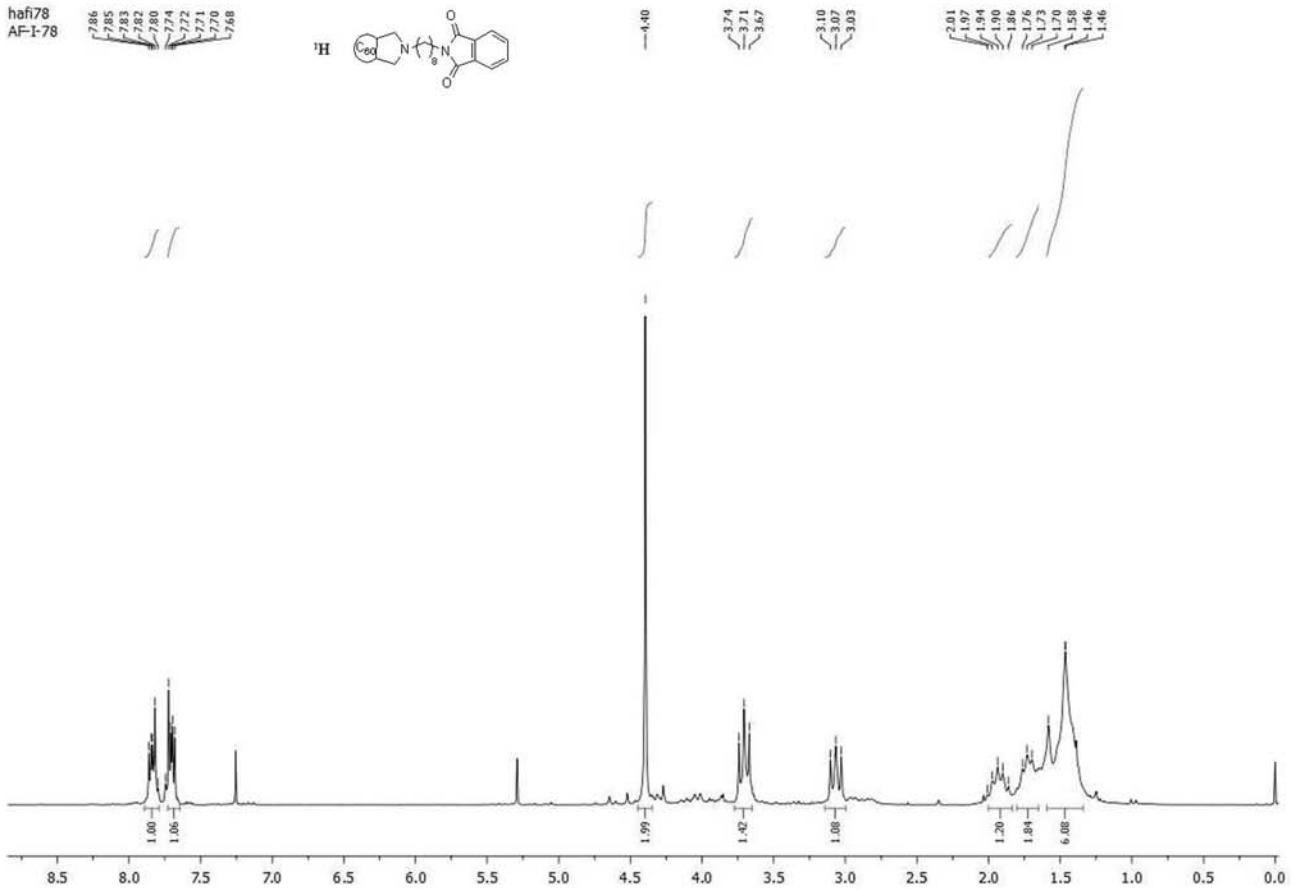


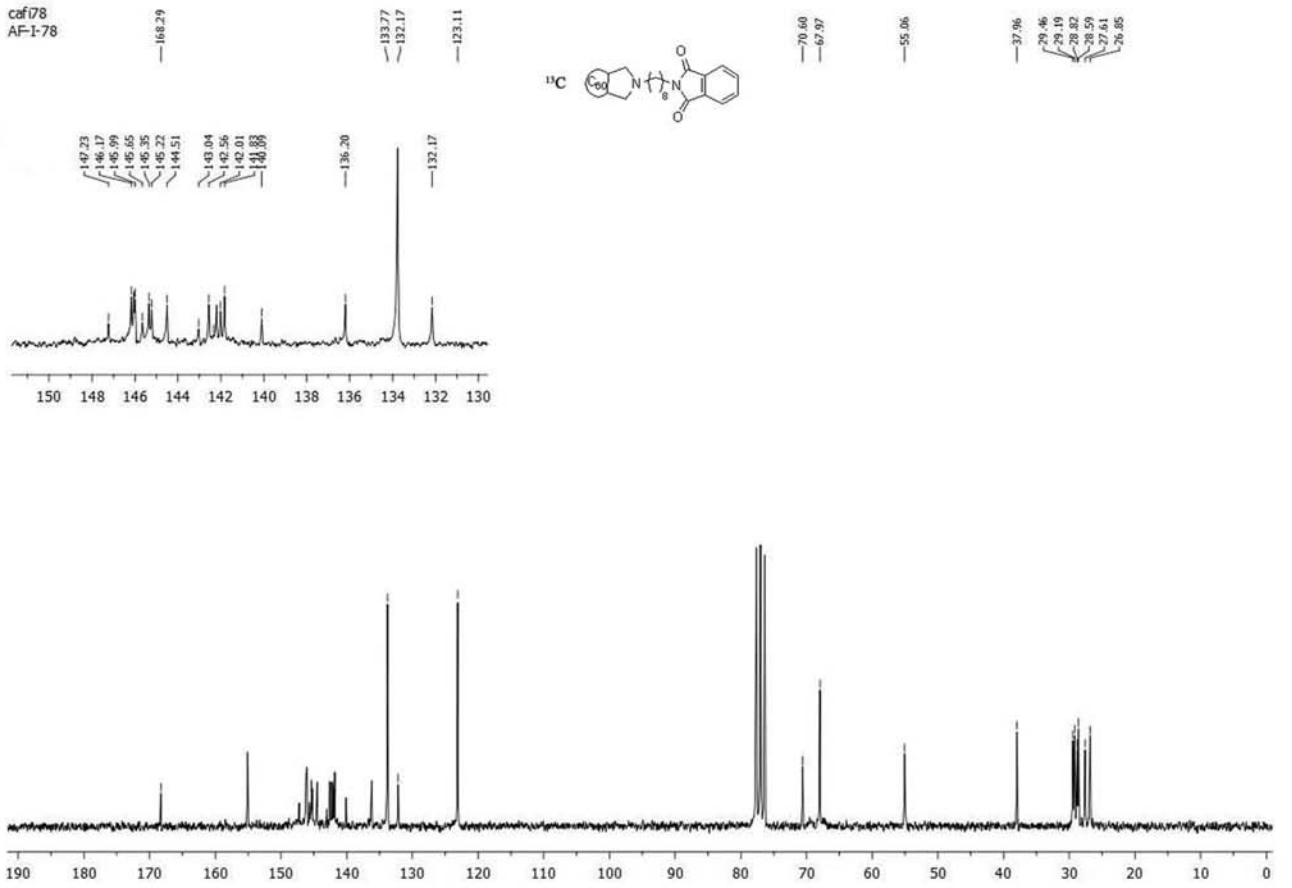
NMR spectra of **1b**



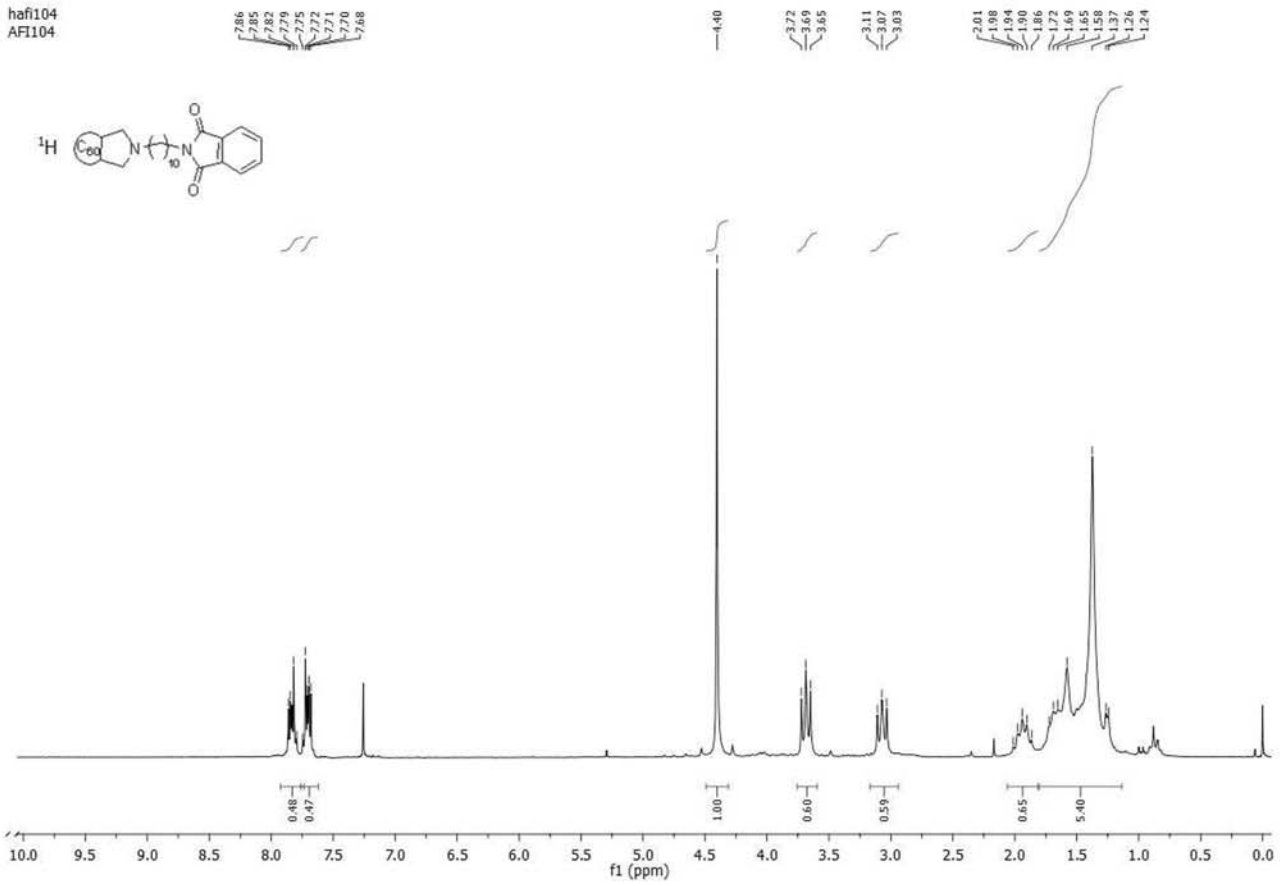


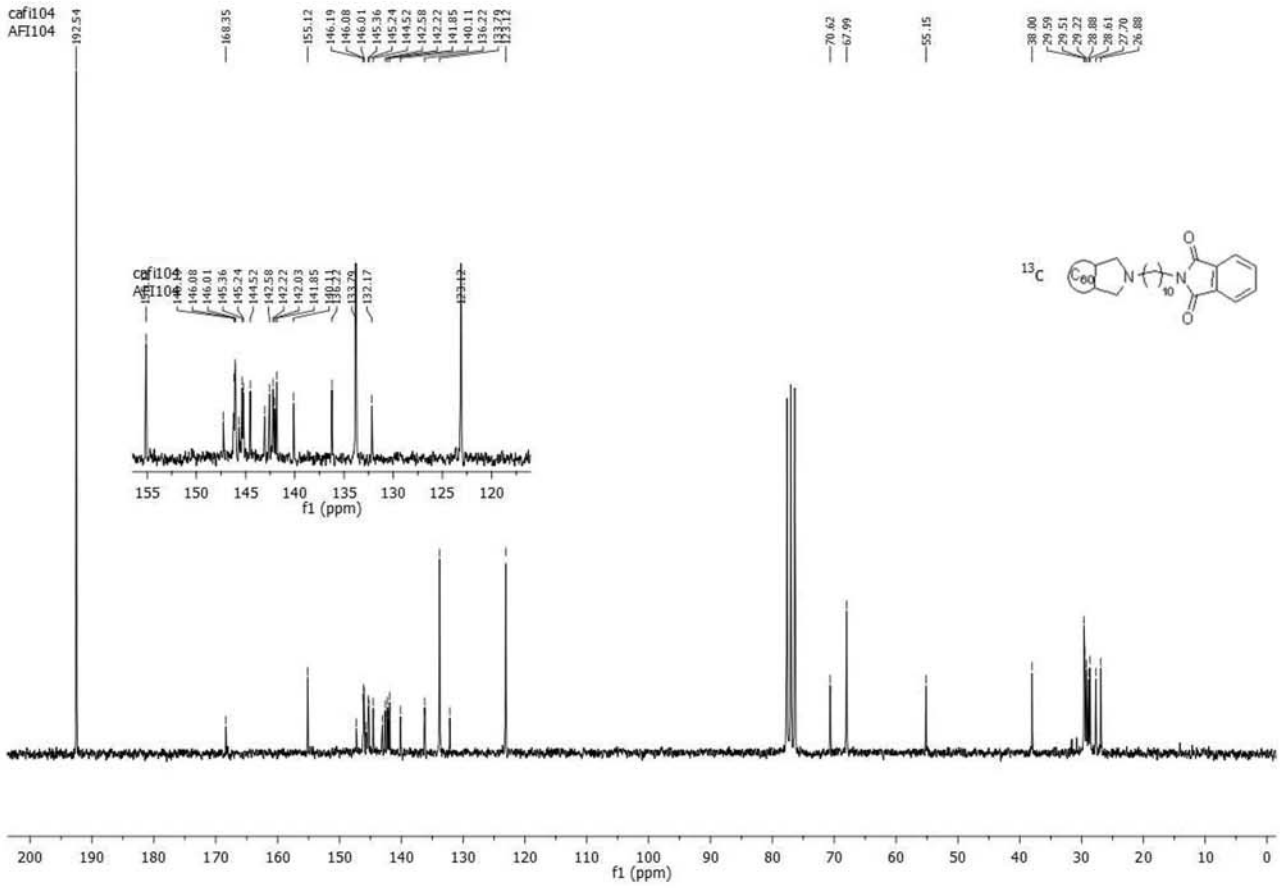
NMR spectra of **1c**



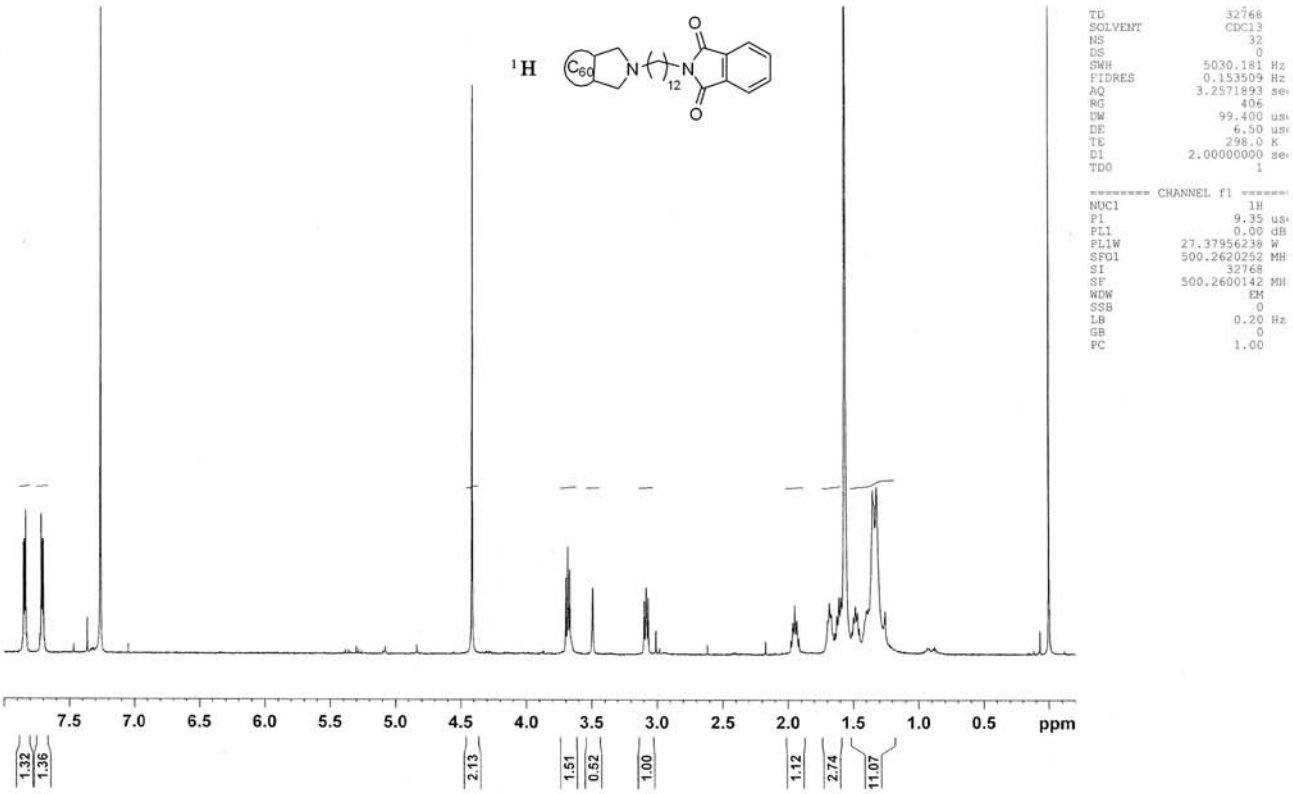


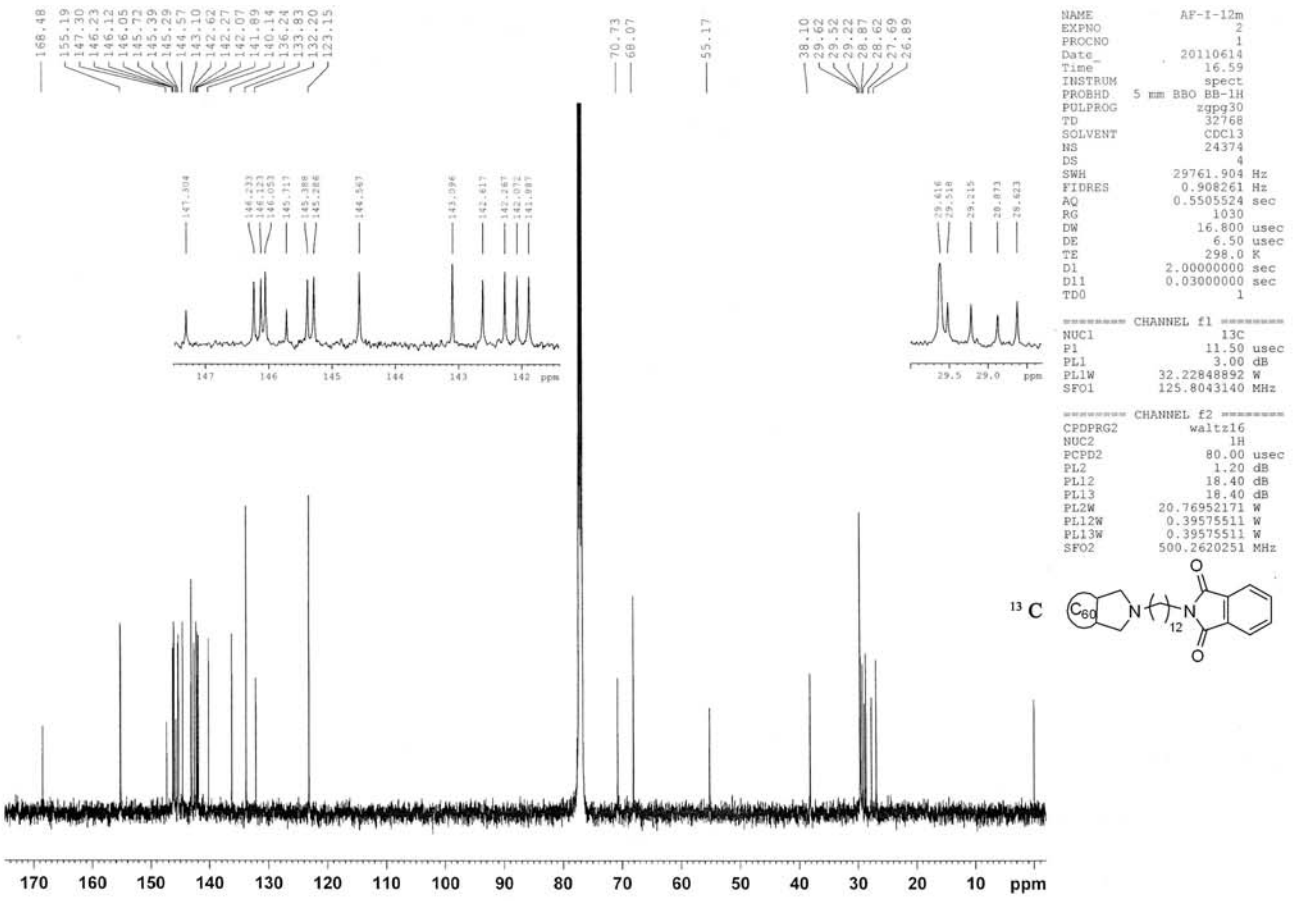
NMR spectra of **1d**



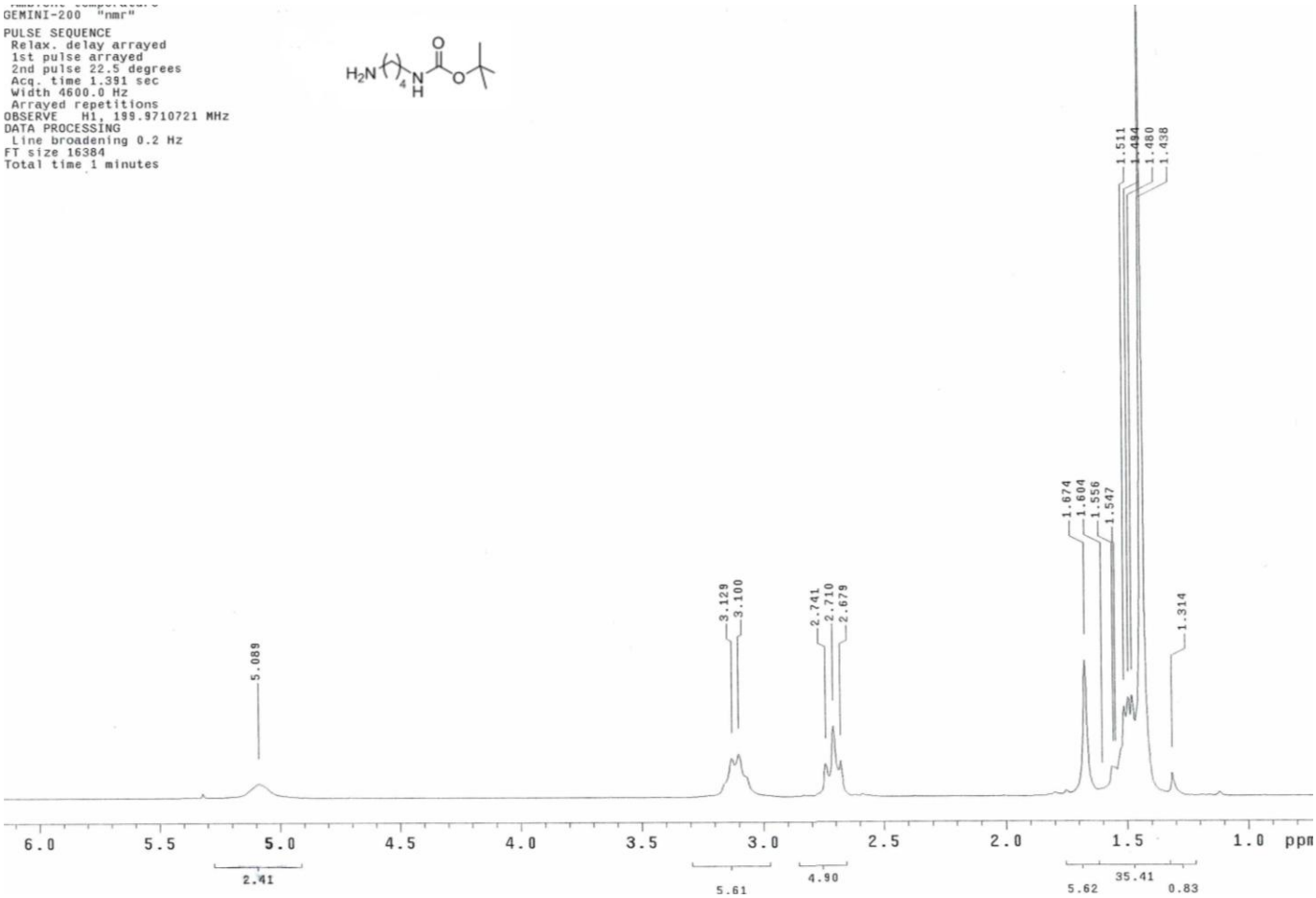


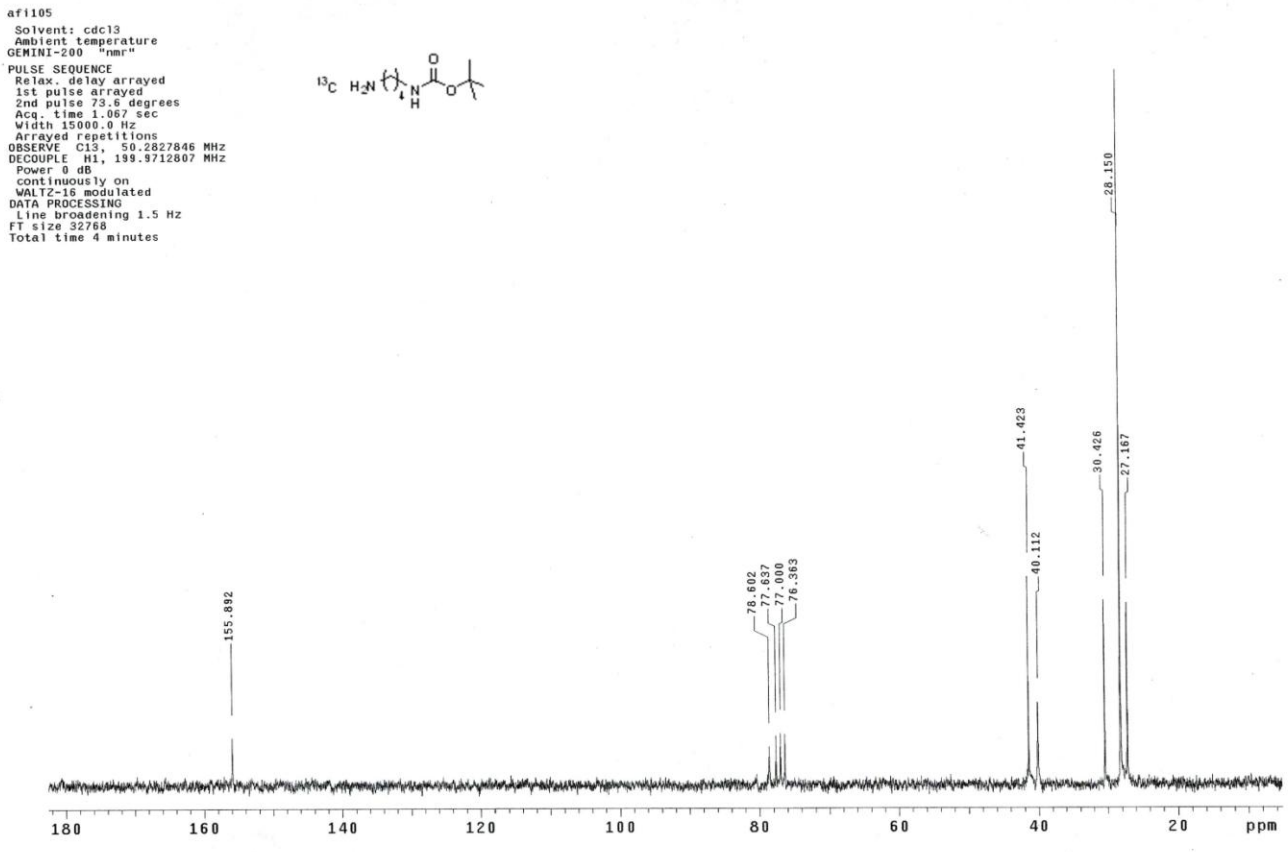
NMR spectra of **1e**



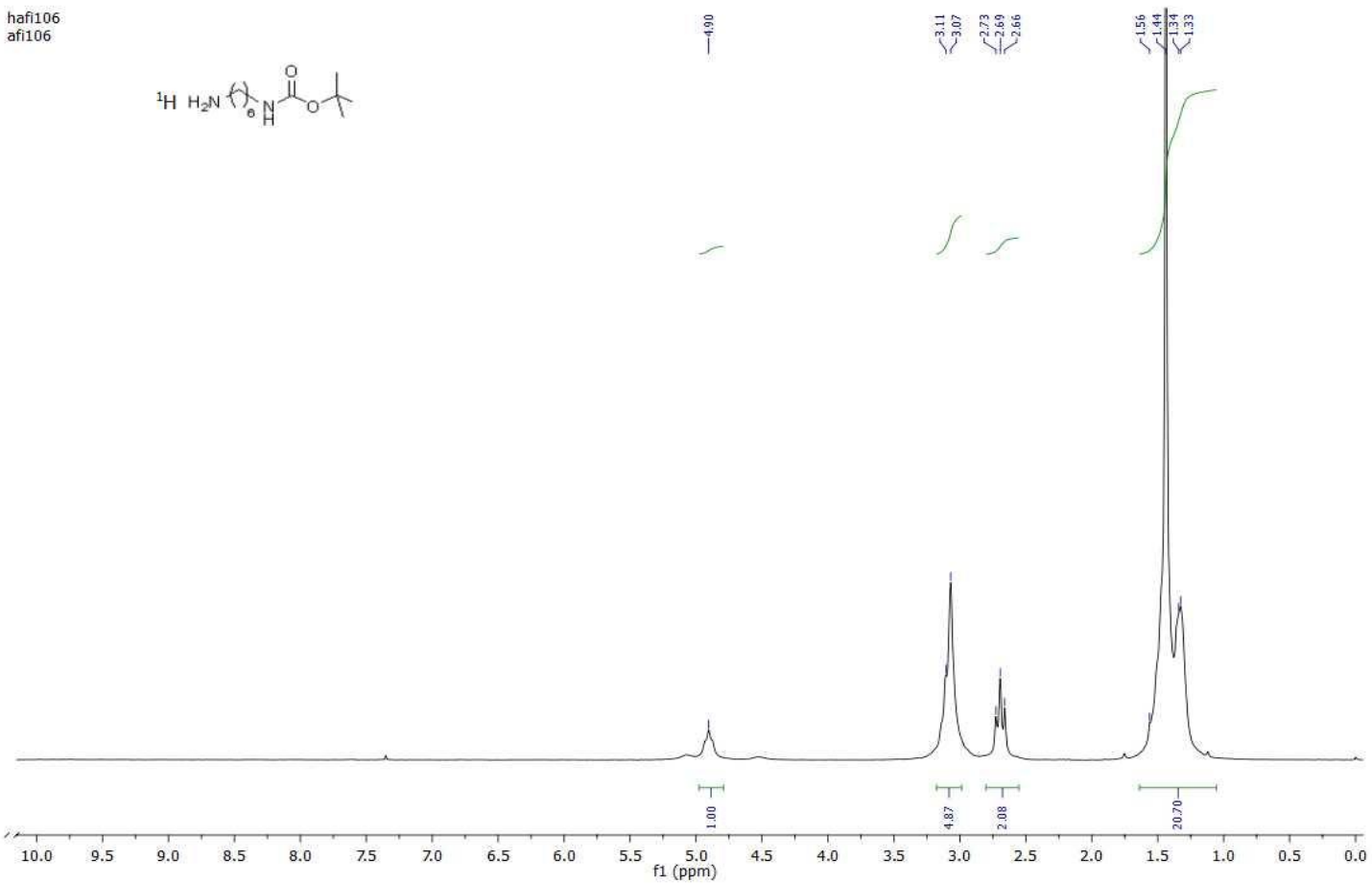


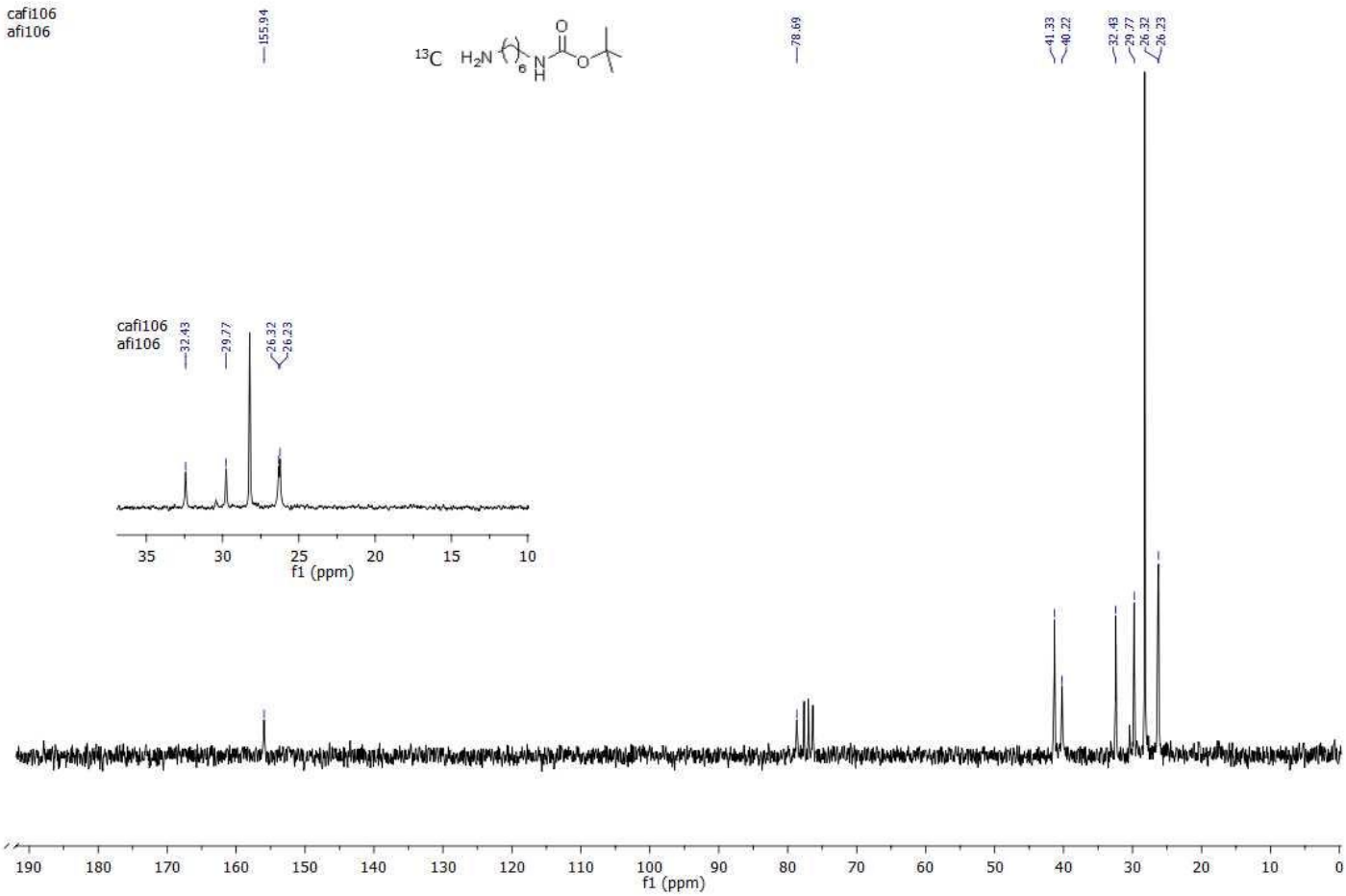
NMR spectra of **1f**



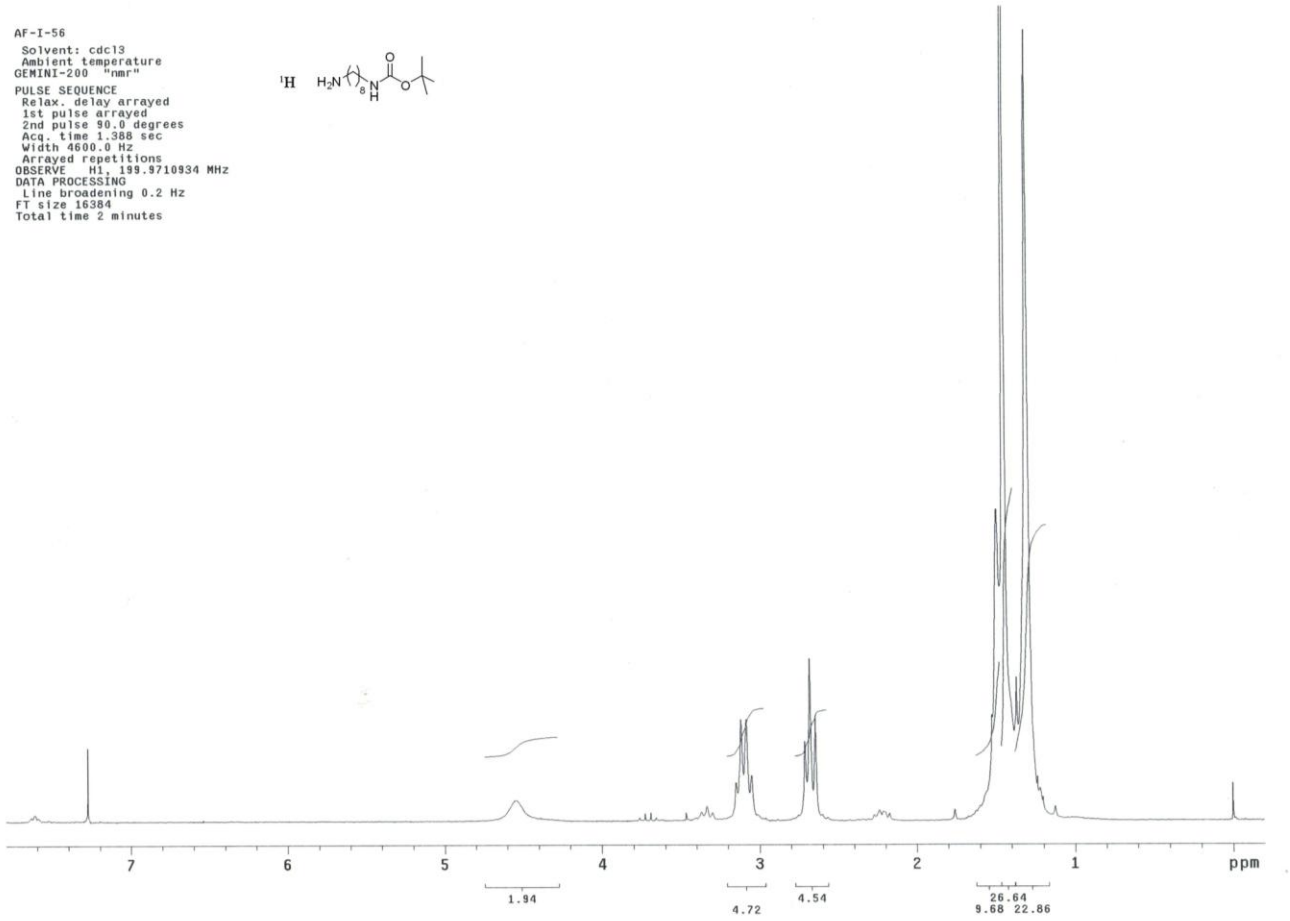


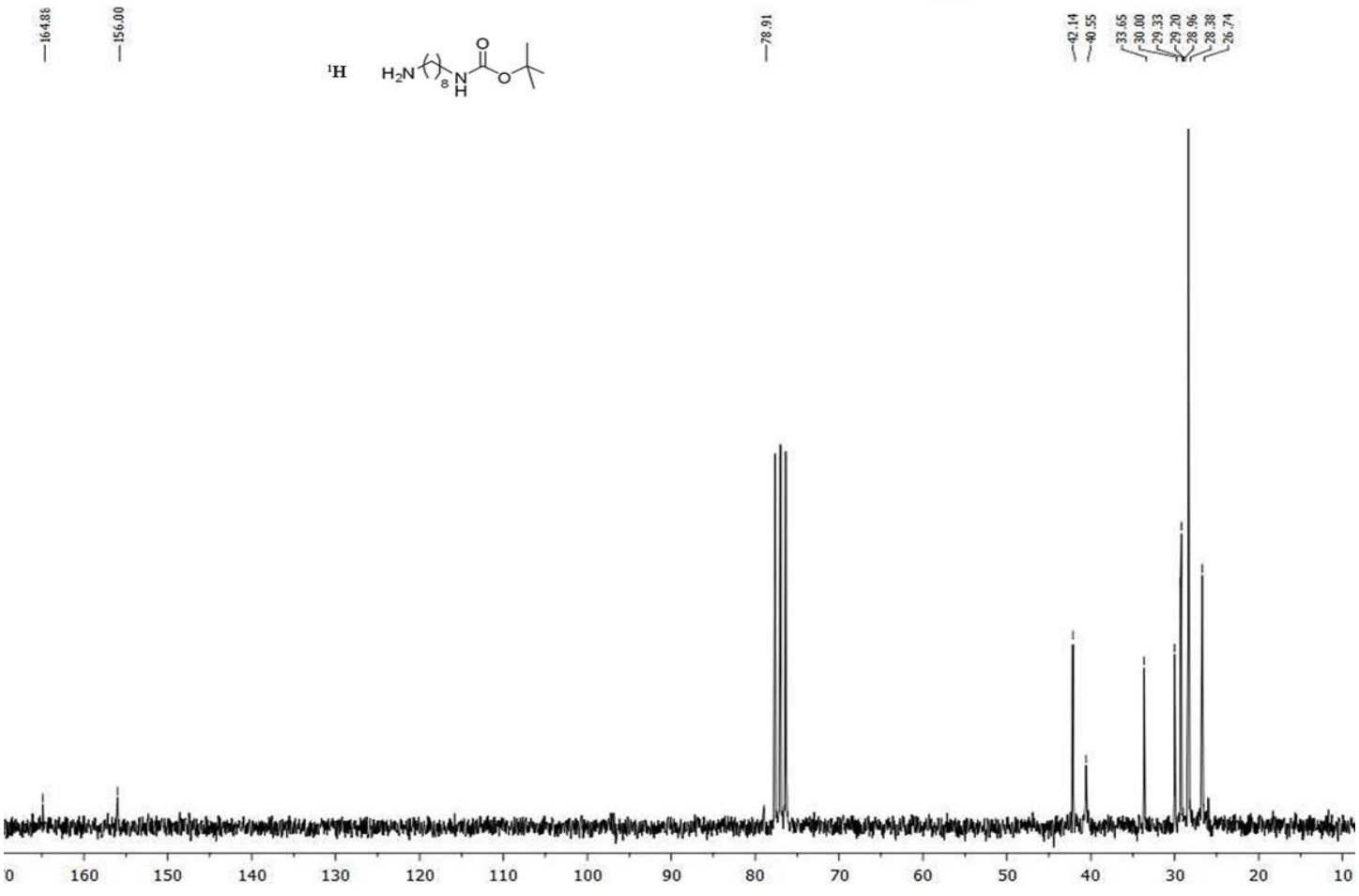
NMR spectra of 2b



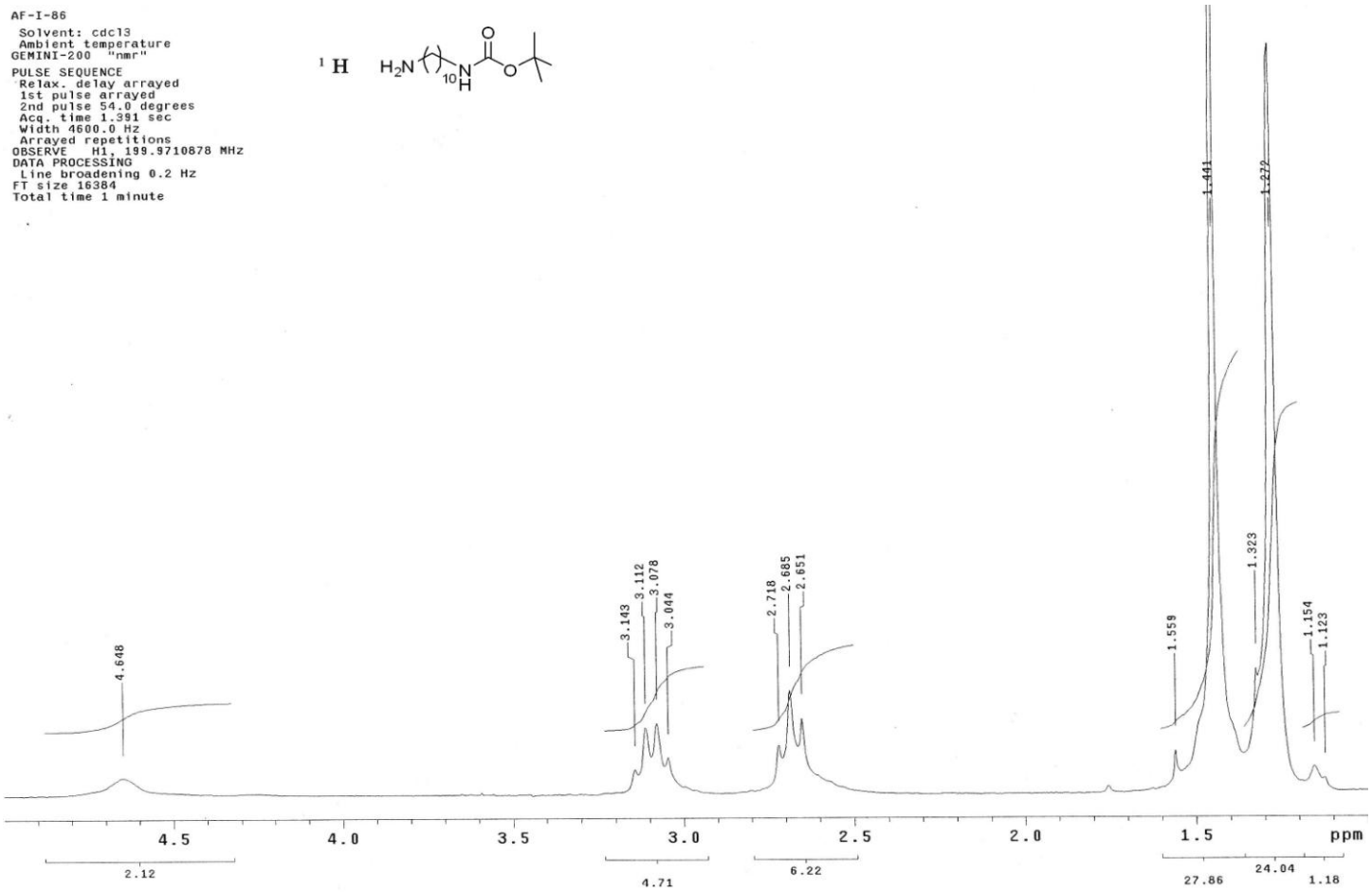


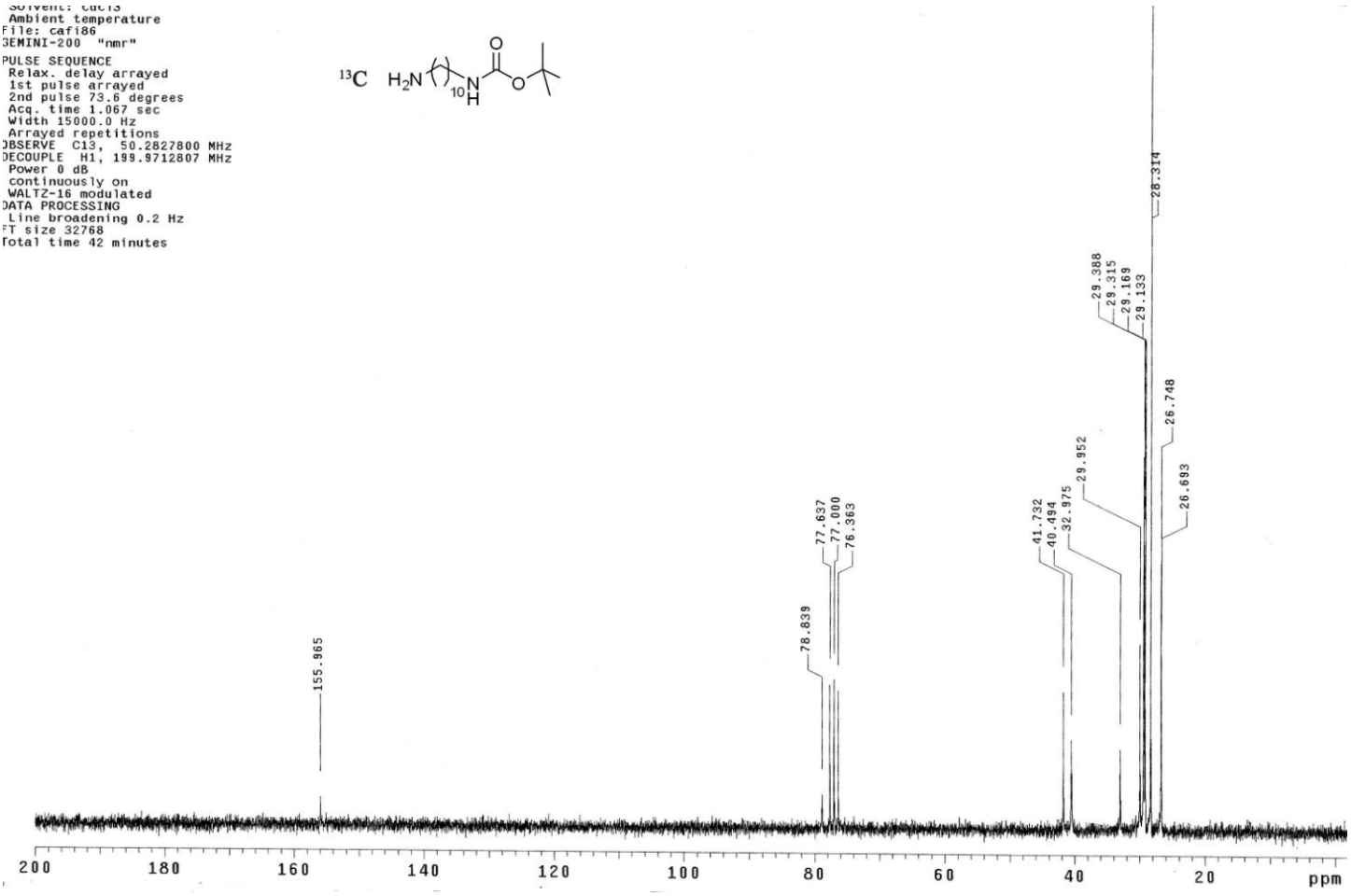
NMR spectra of 2c

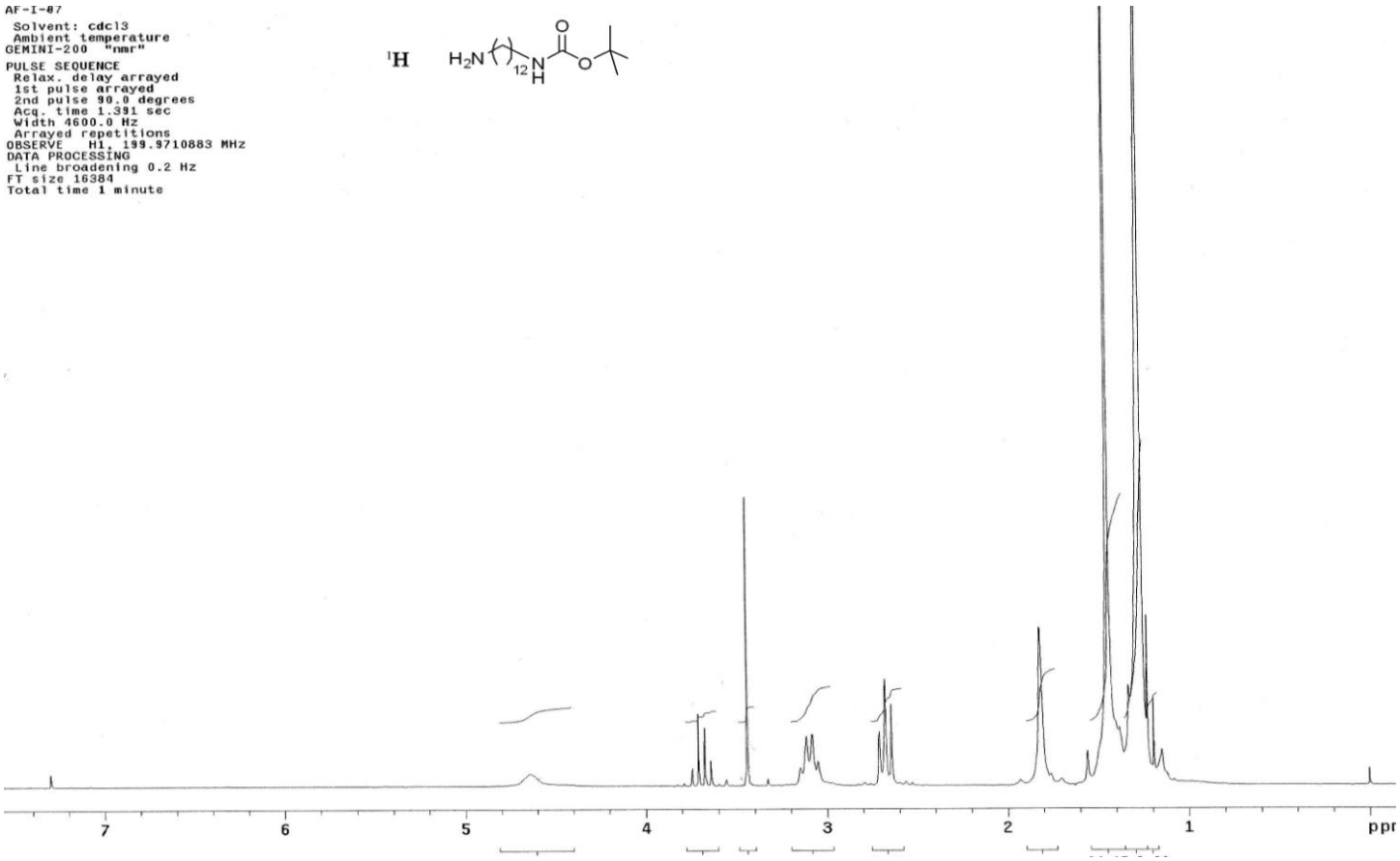


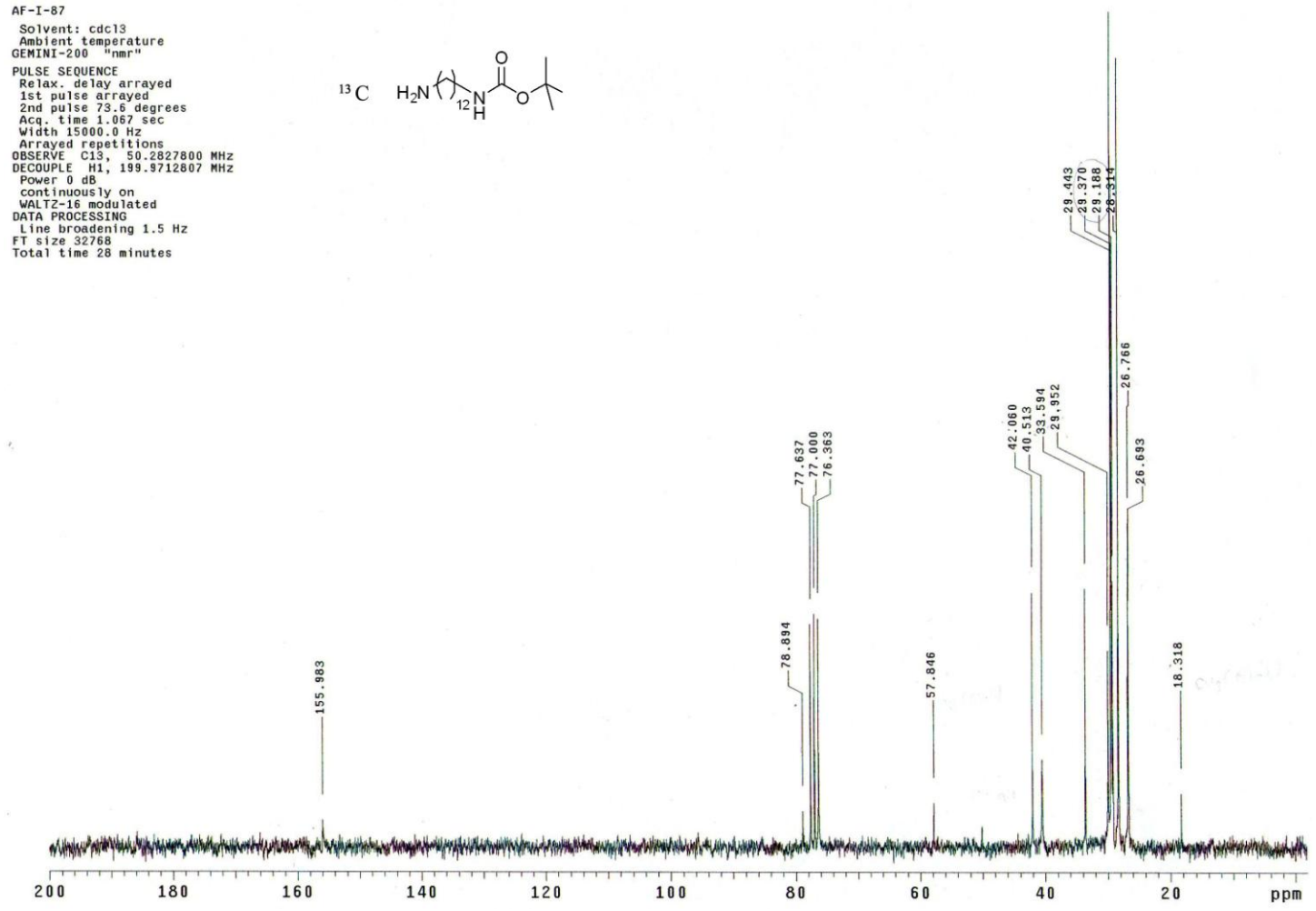


NMR spectra of 2d

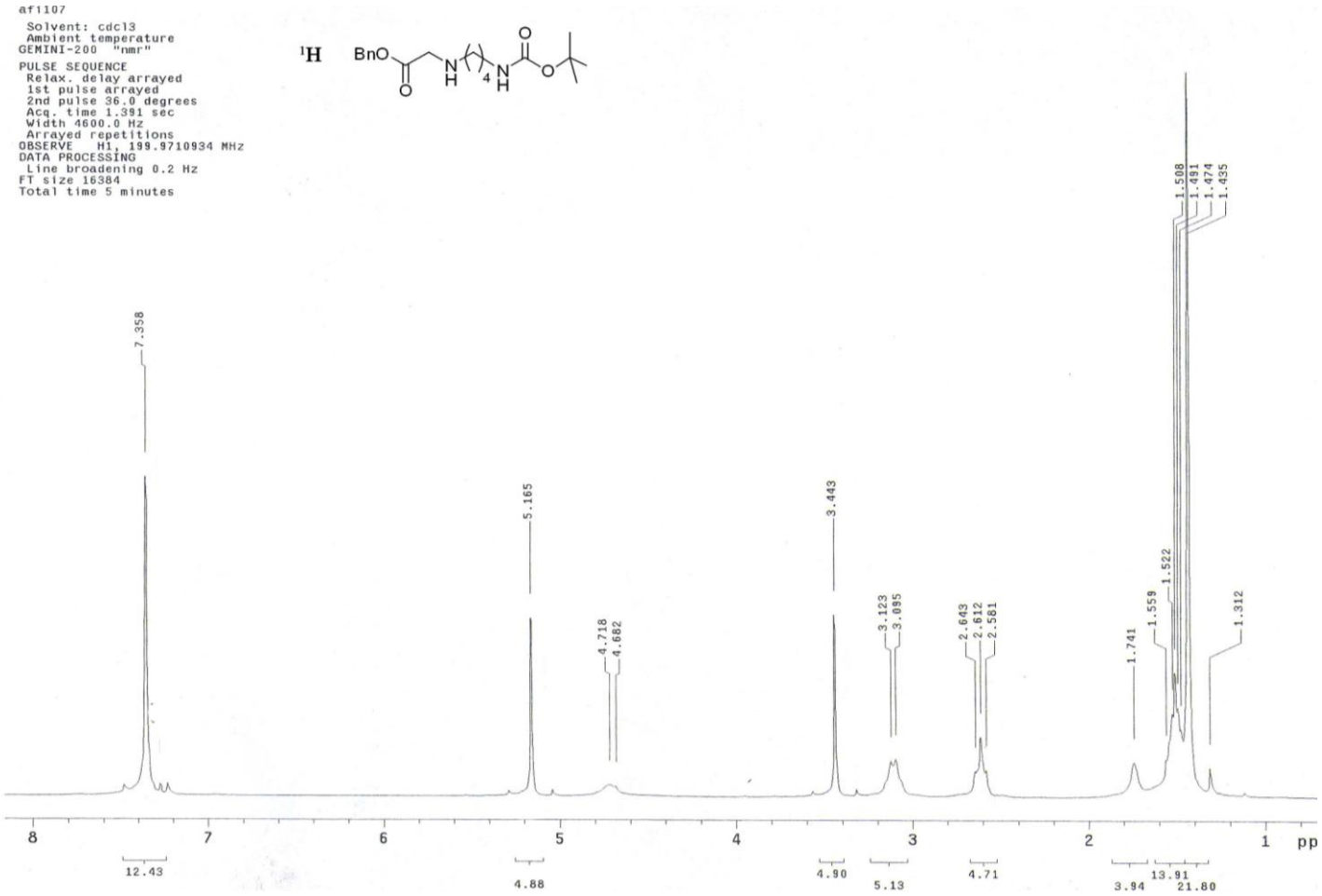

AF-I-86
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 54.0 degrees
Acq. time 1.391 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710878 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minute
1H
4.648
3.143
3.112
3.078
3.044
2.718
2.685
2.651
1.559
1.441
1.323
1.272
1.154
1.123
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
2.12
4.71
6.22
27.86
24.04
1.18

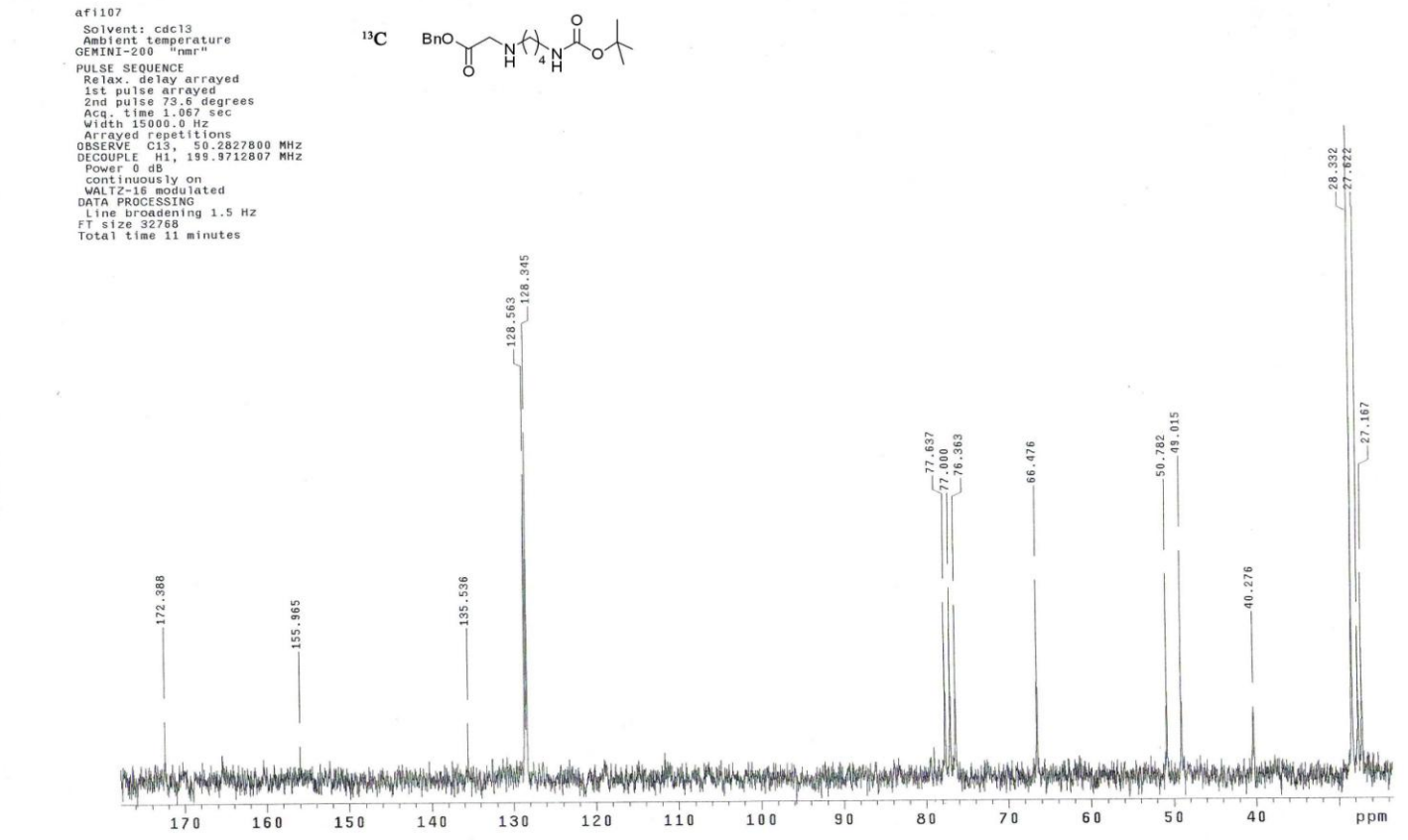

Ambient temperature
File: cafi86
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827800 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 42 minutes
13C
155.965
78.839
77.637
77.000
76.363
41.732
40.494
32.975
29.952
29.388
29.315
29.169
29.133
28.314
26.748
26.693
200
180
160
140
120
100
80
60
40
20
ppm



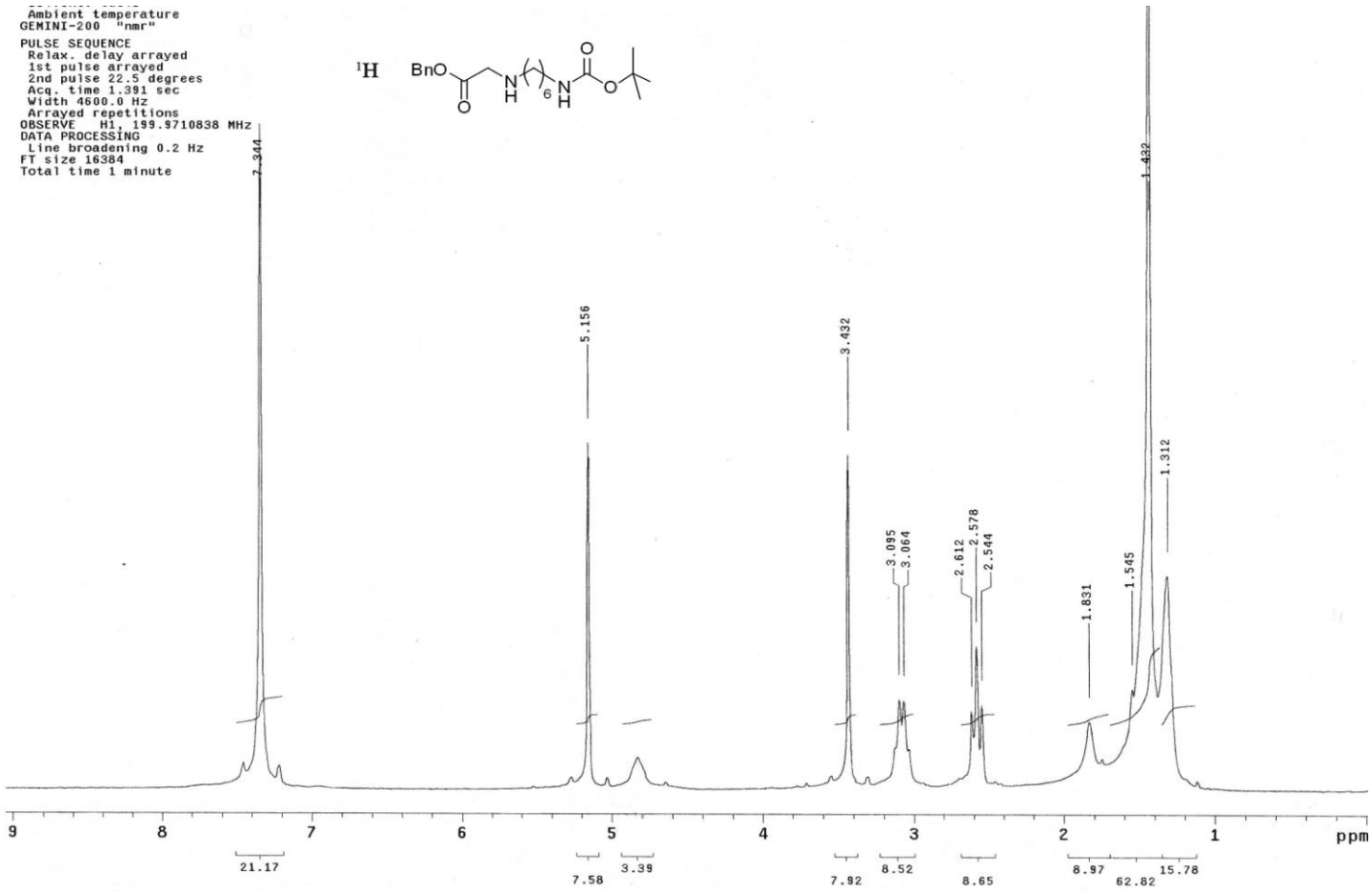


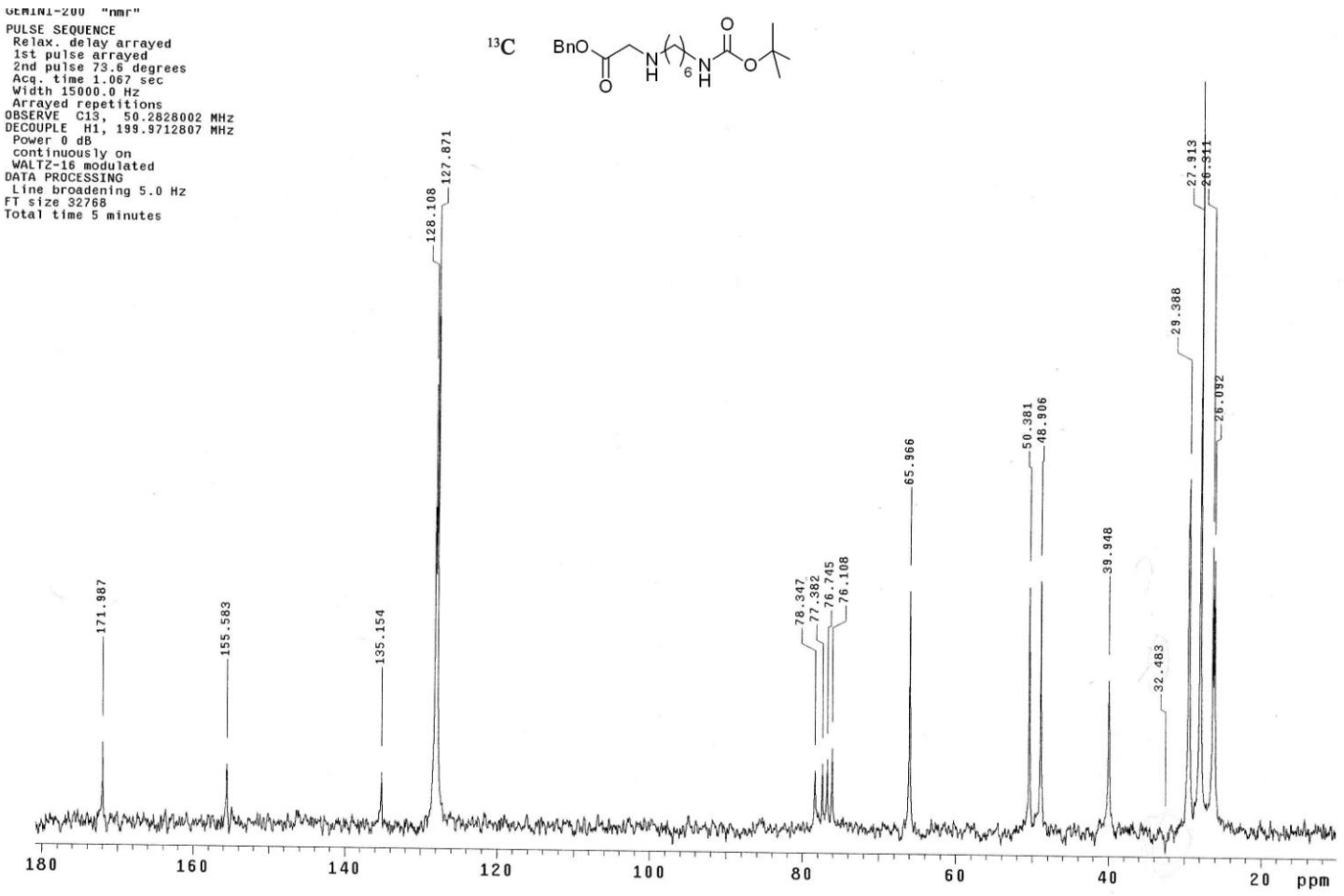
NMR spectra of 2f



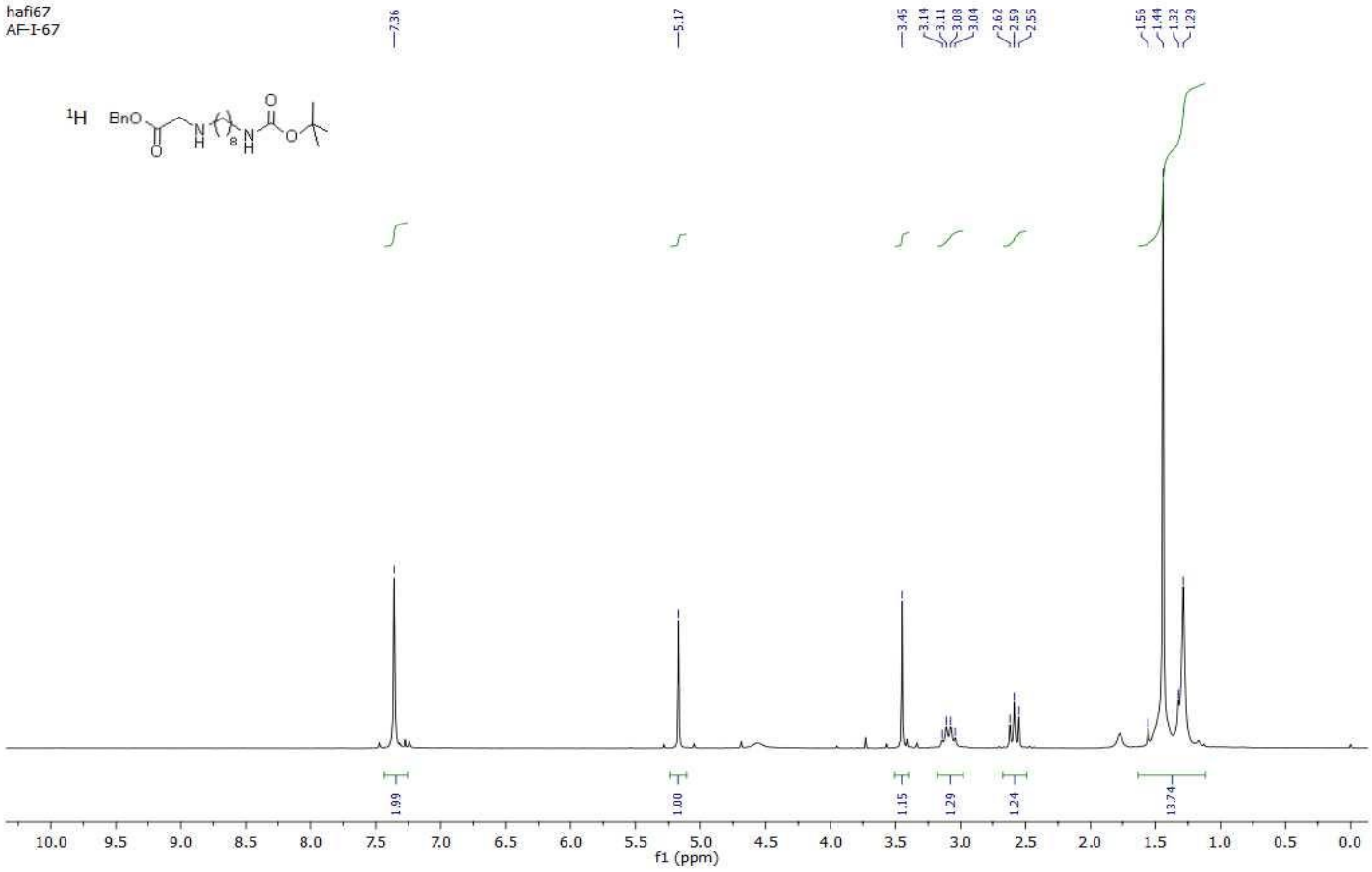


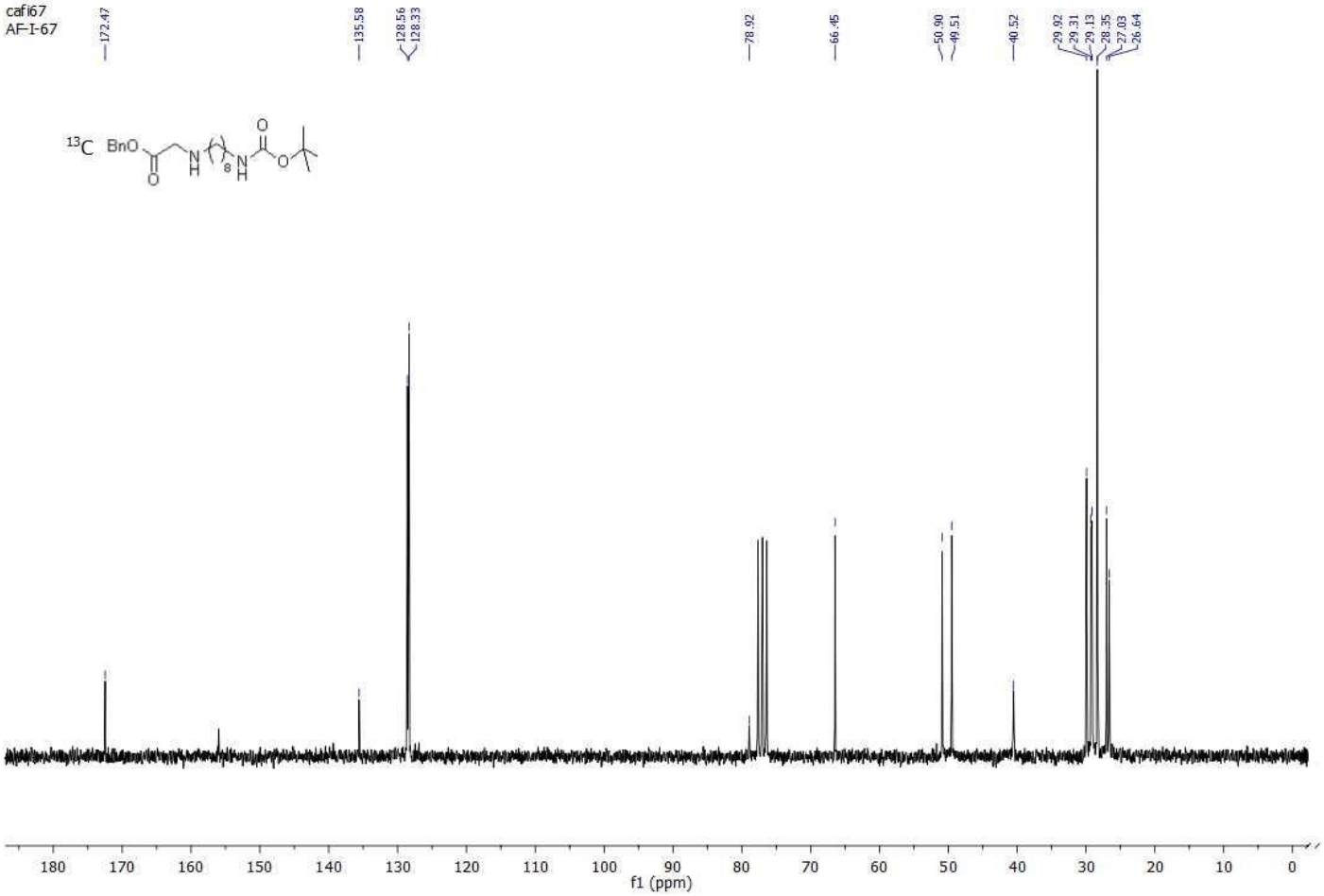
NMR spectra of 3b

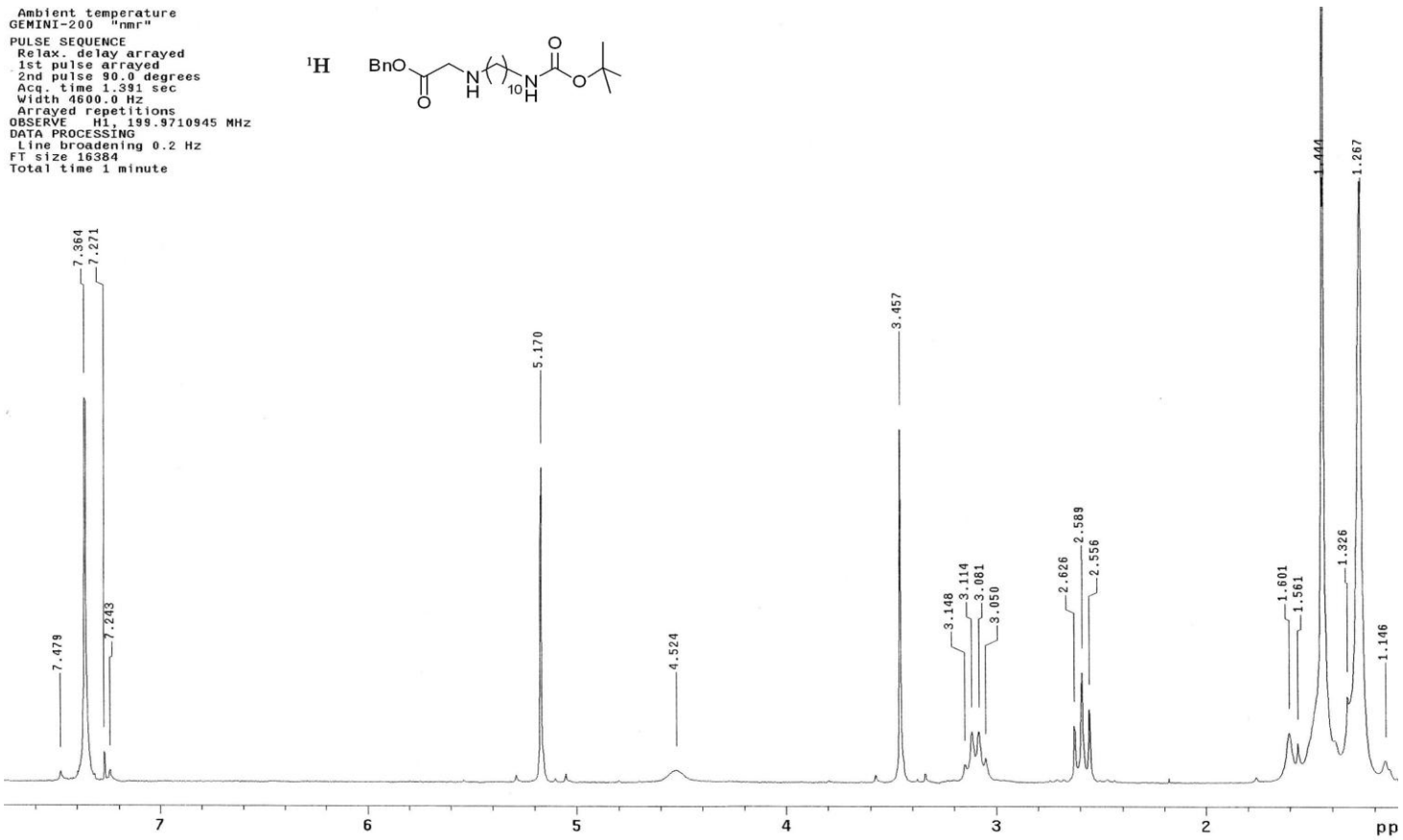


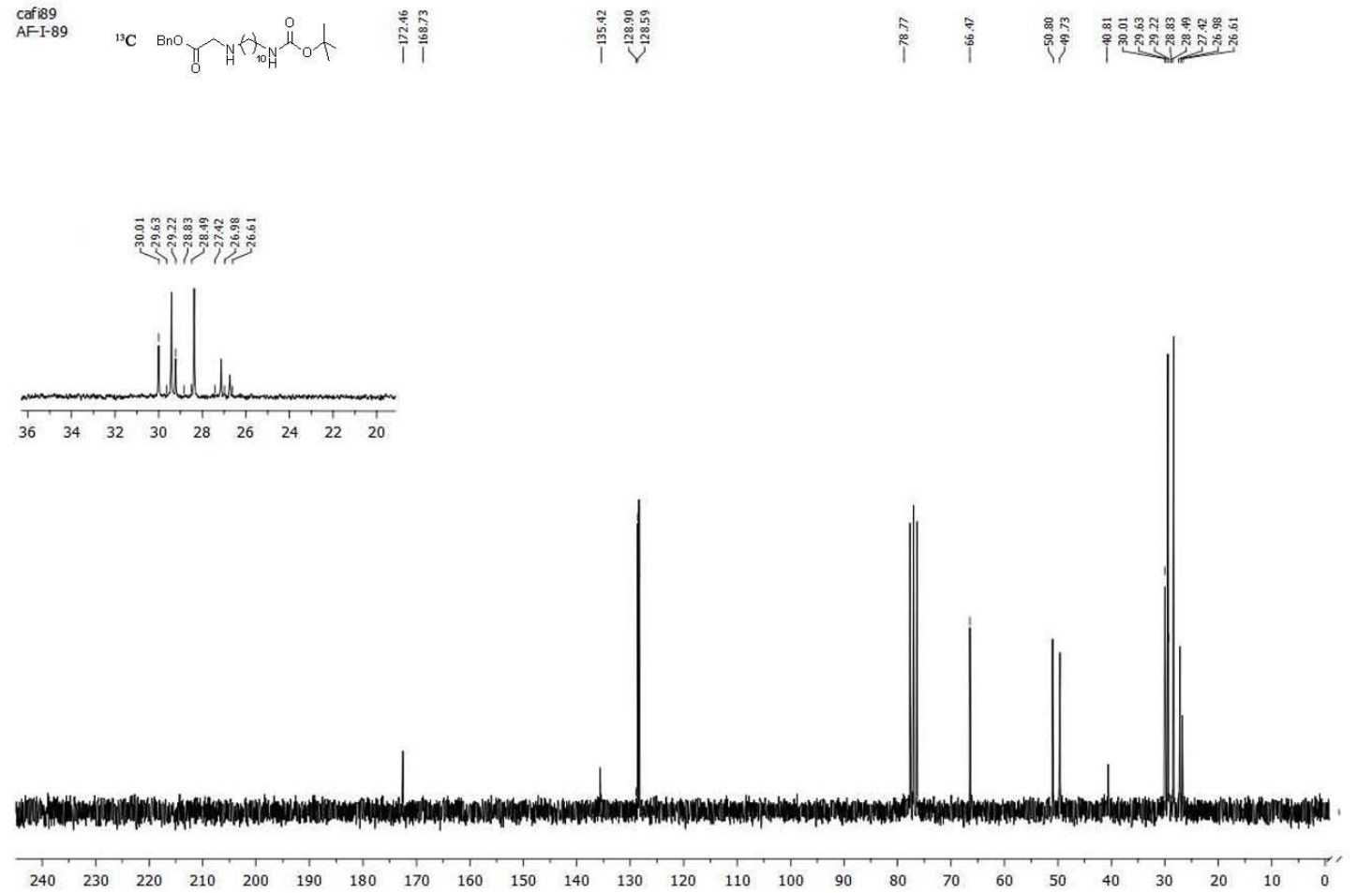


NMR spectra of 3c

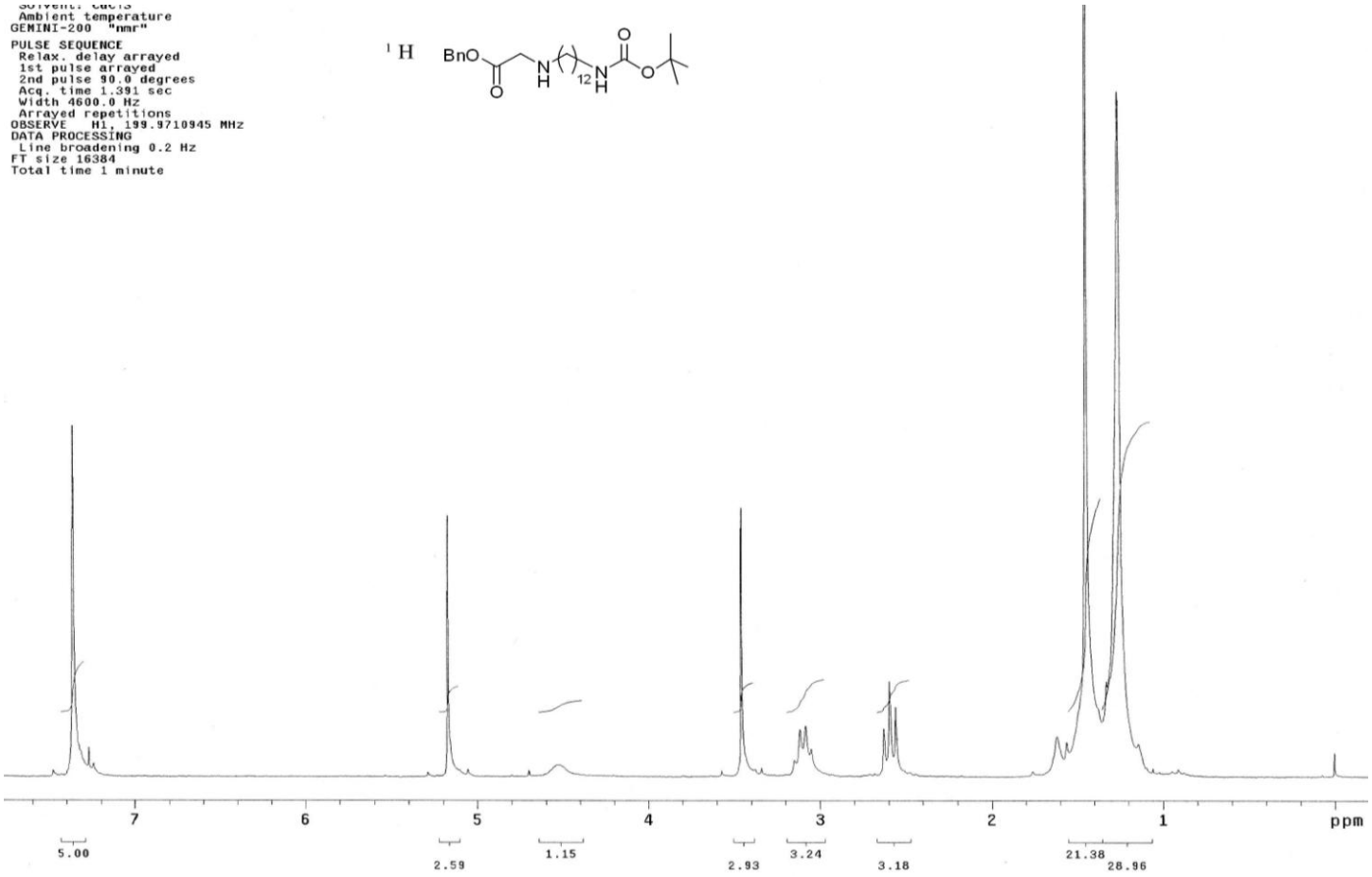

hafi67
AF-I-67
7.36
5.17
3.45
3.14
3.11
3.08
3.04
2.62
2.59
2.55
1.56
1.44
1.32
1.29
1H
BnO
1.99
1.00
1.15
1.29
1.24
13.74
f1 (ppm)

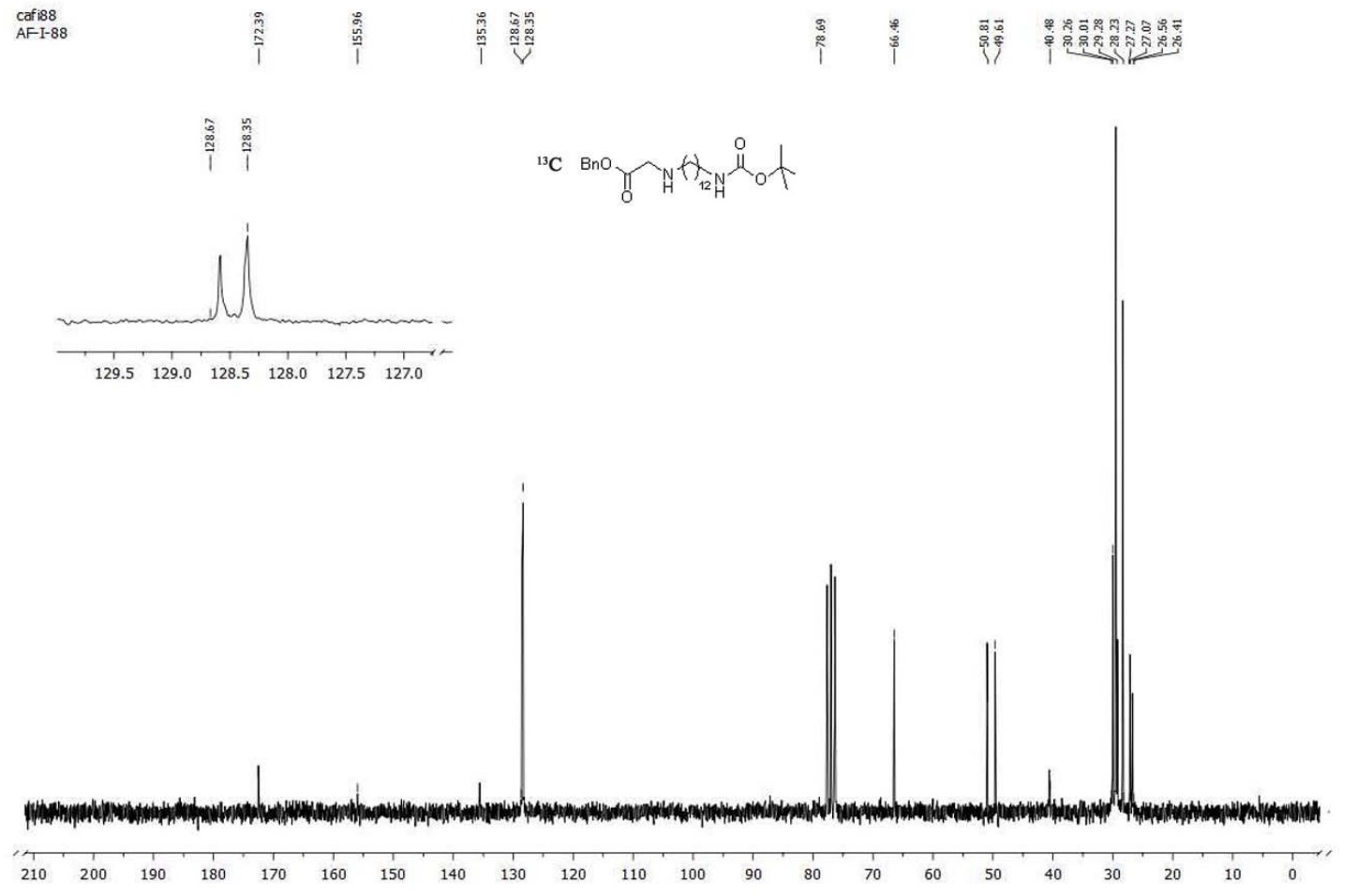

cafi67
AF-I-67
172.47
135.58
128.56
128.33
78.92
66.45
50.90
49.51
40.52
29.92
29.31
29.13
28.35
27.03
26.64
13C
BnO
f1 (ppm)



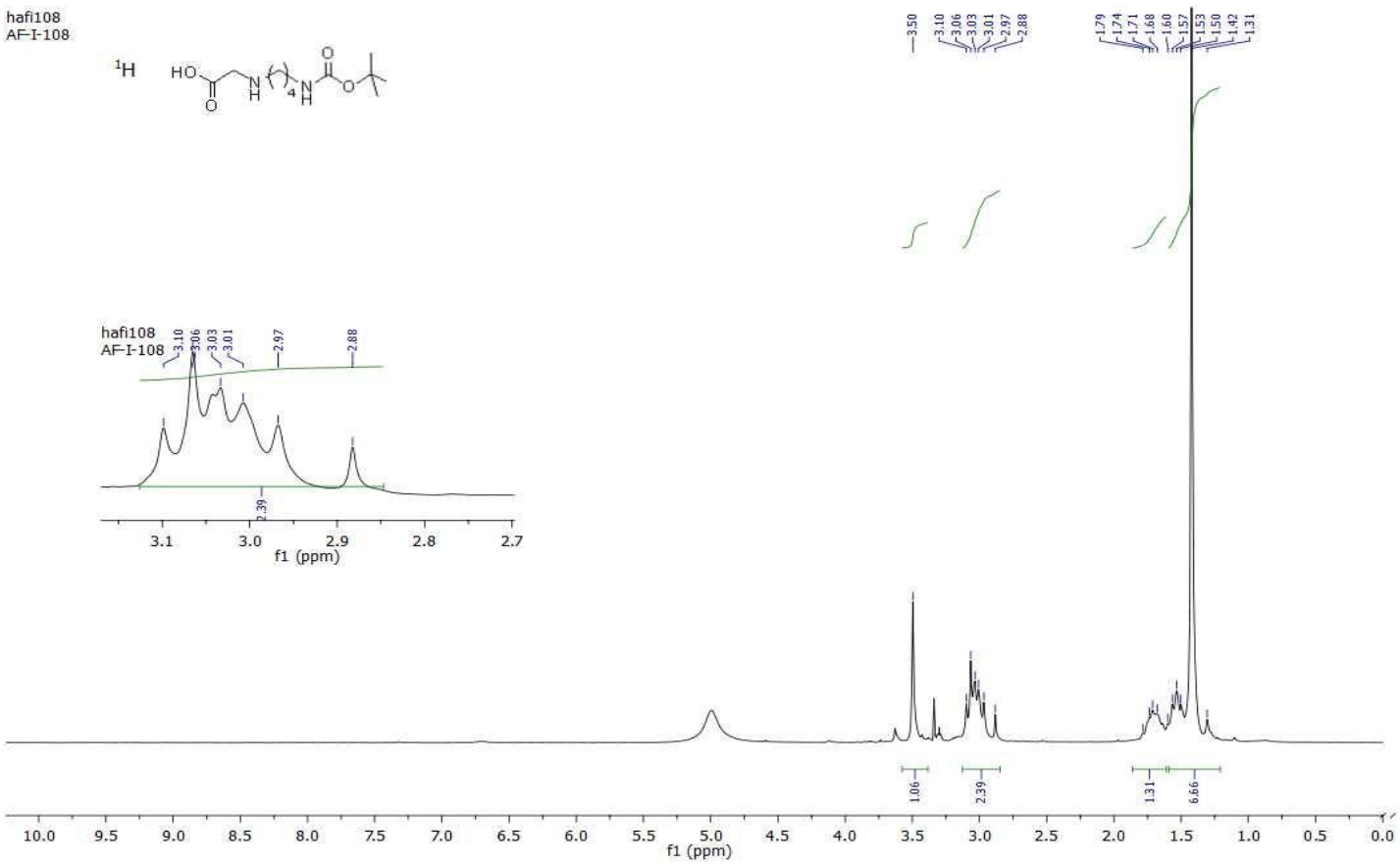


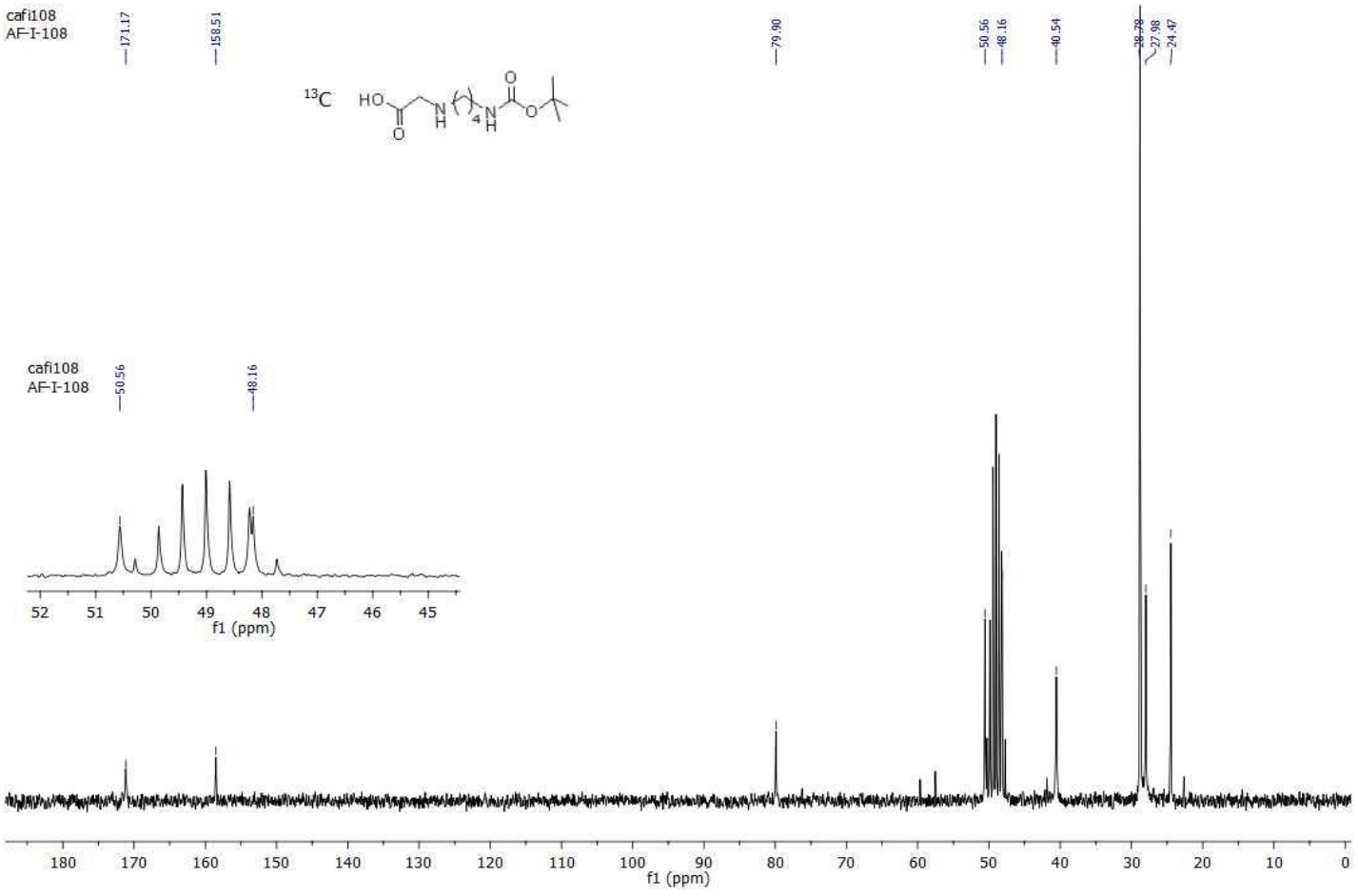
NMR spectra of 3e

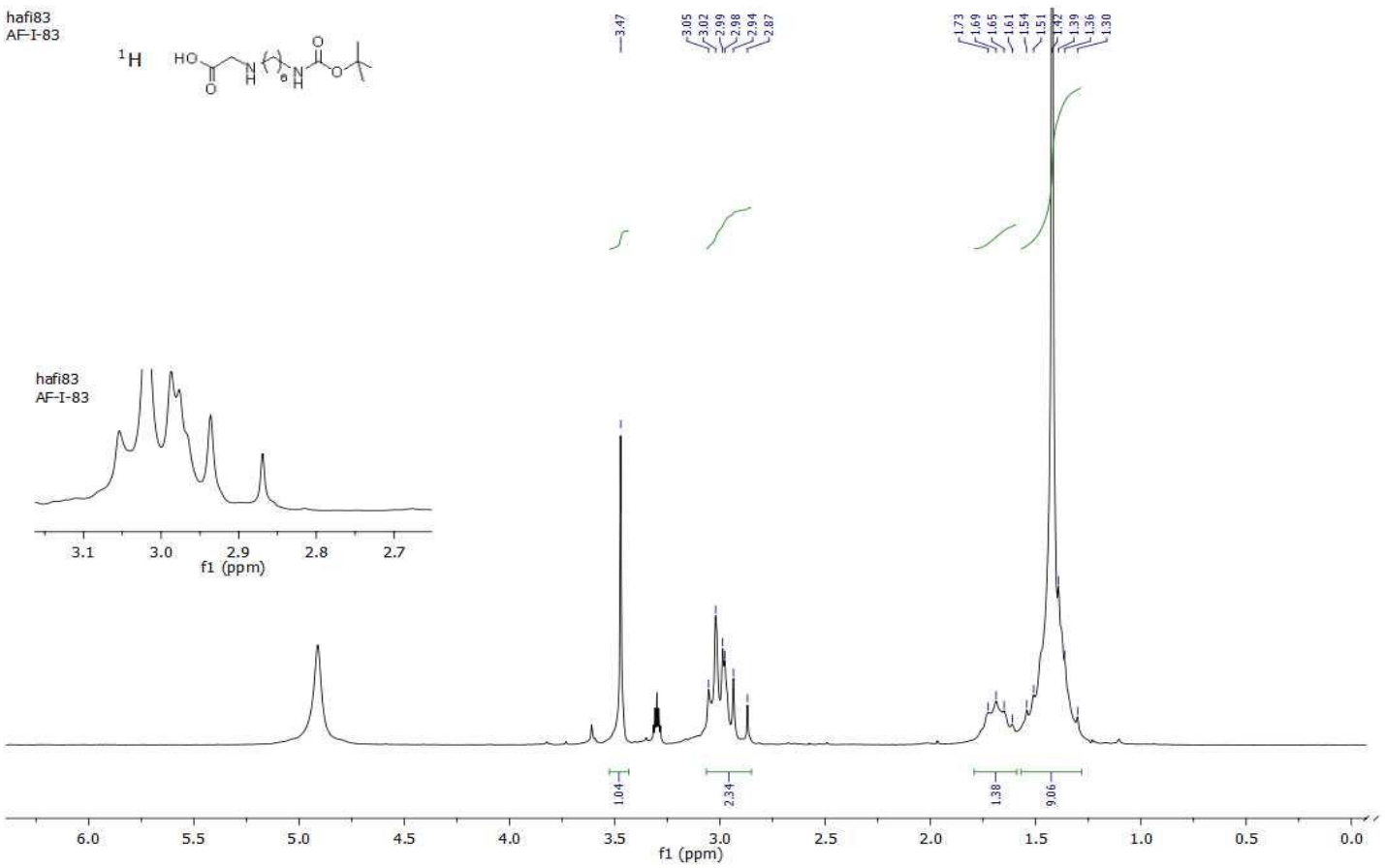


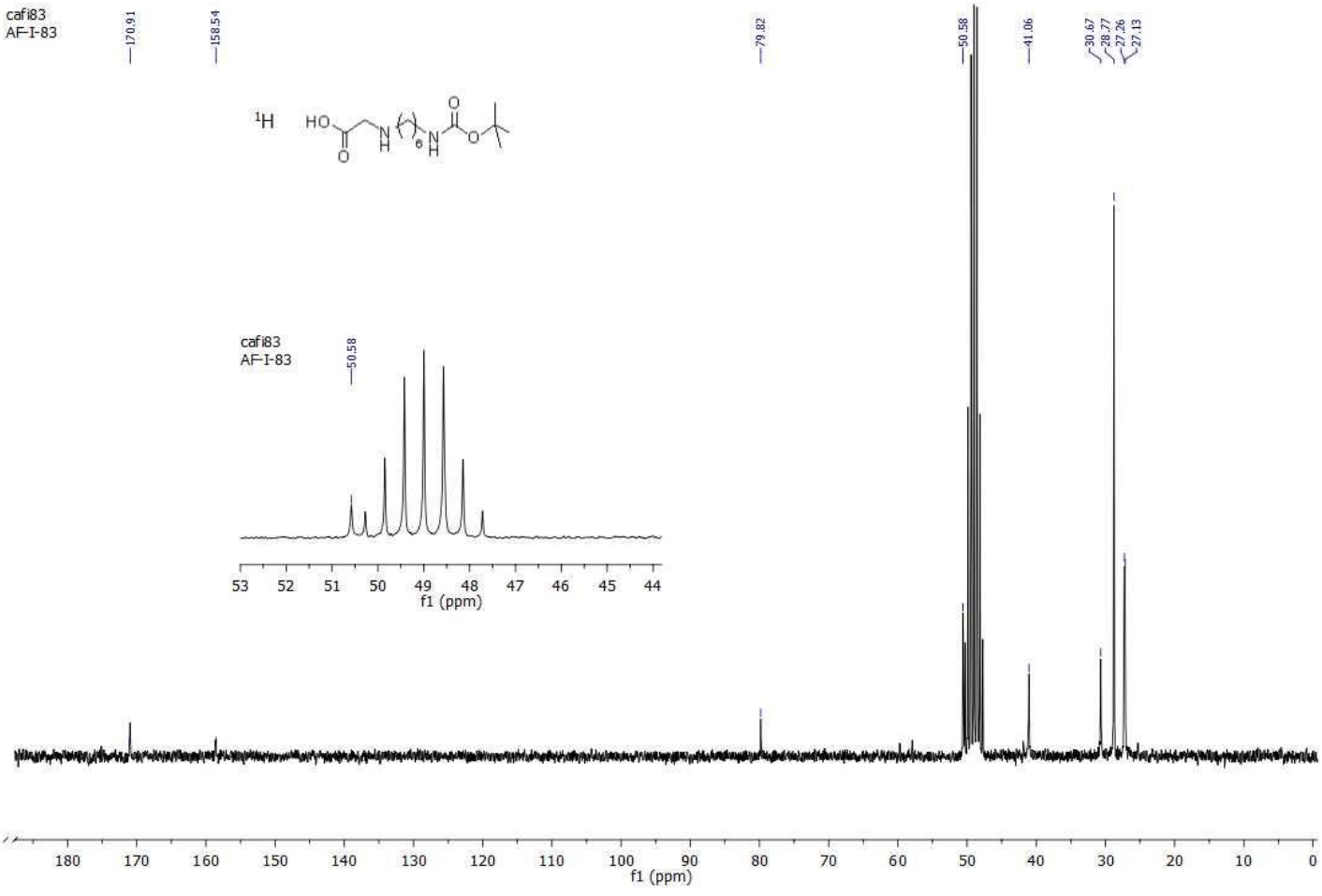


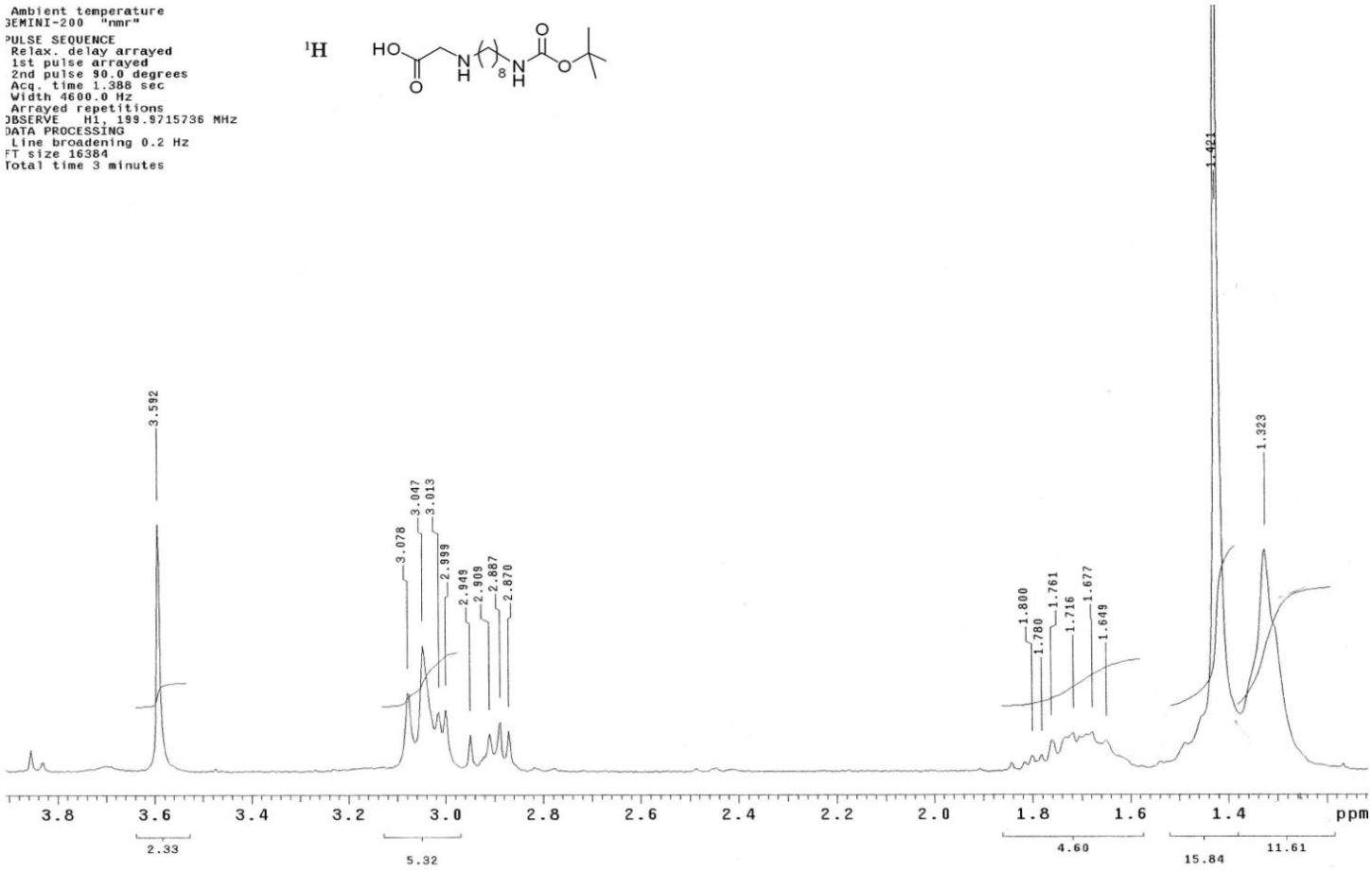
NMR spectra of 3f

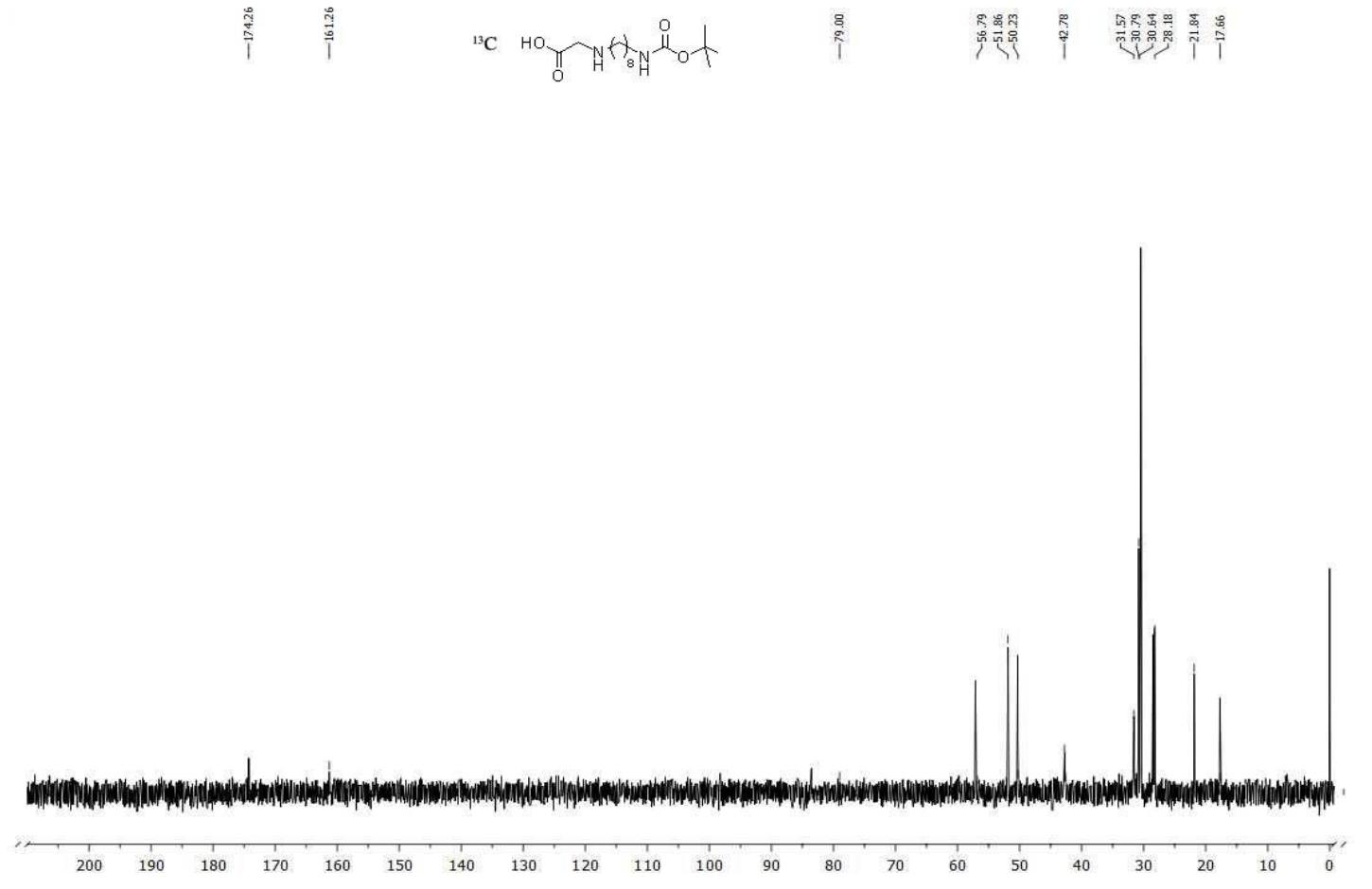

hafi108
AF-I-108
^{1}H
HO
O
N
H
4
N
H
O
O
3.50
3.10
3.06
3.03
3.01
2.97
2.88
1.79
1.74
1.71
1.68
1.60
1.57
1.53
1.50
1.42
1.31
hafi108
AF-I-108
2.39
3.1
3.0
2.9
2.8
2.7
f1 (ppm)
1.06
2.39
1.31
6.66
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


cafi108
AF-I-108
171.17
158.51
79.90
50.56
48.16
40.54
28.28
27.98
24.47
^{13}C
HO
O
N
H
4
N
H
O
O
cafi108
AF-I-108
50.56
48.16
52
51
50
49
48
47
46
45
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

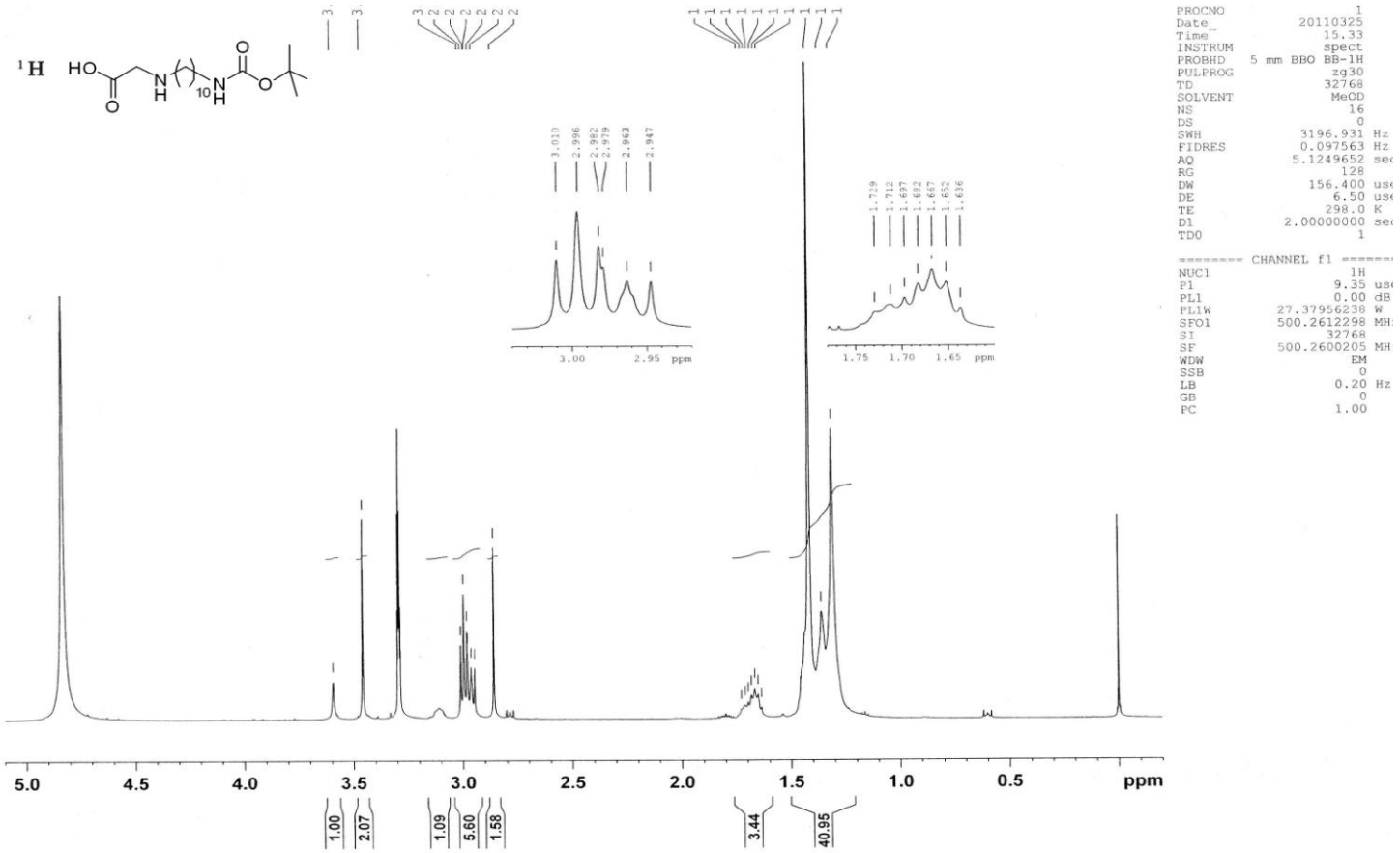

hafi83
AF-I-83
1H
HO
O
N
H
6
N
H
O
O
3.47
3.05
3.02
2.99
2.98
2.94
2.87
1.73
1.69
1.65
1.61
1.54
1.51
1.42
1.39
1.36
1.30
hafi83
AF-I-83
3.1
3.0
2.9
2.8
2.7
f1 (ppm)
1.04
2.34
1.38
9.06
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

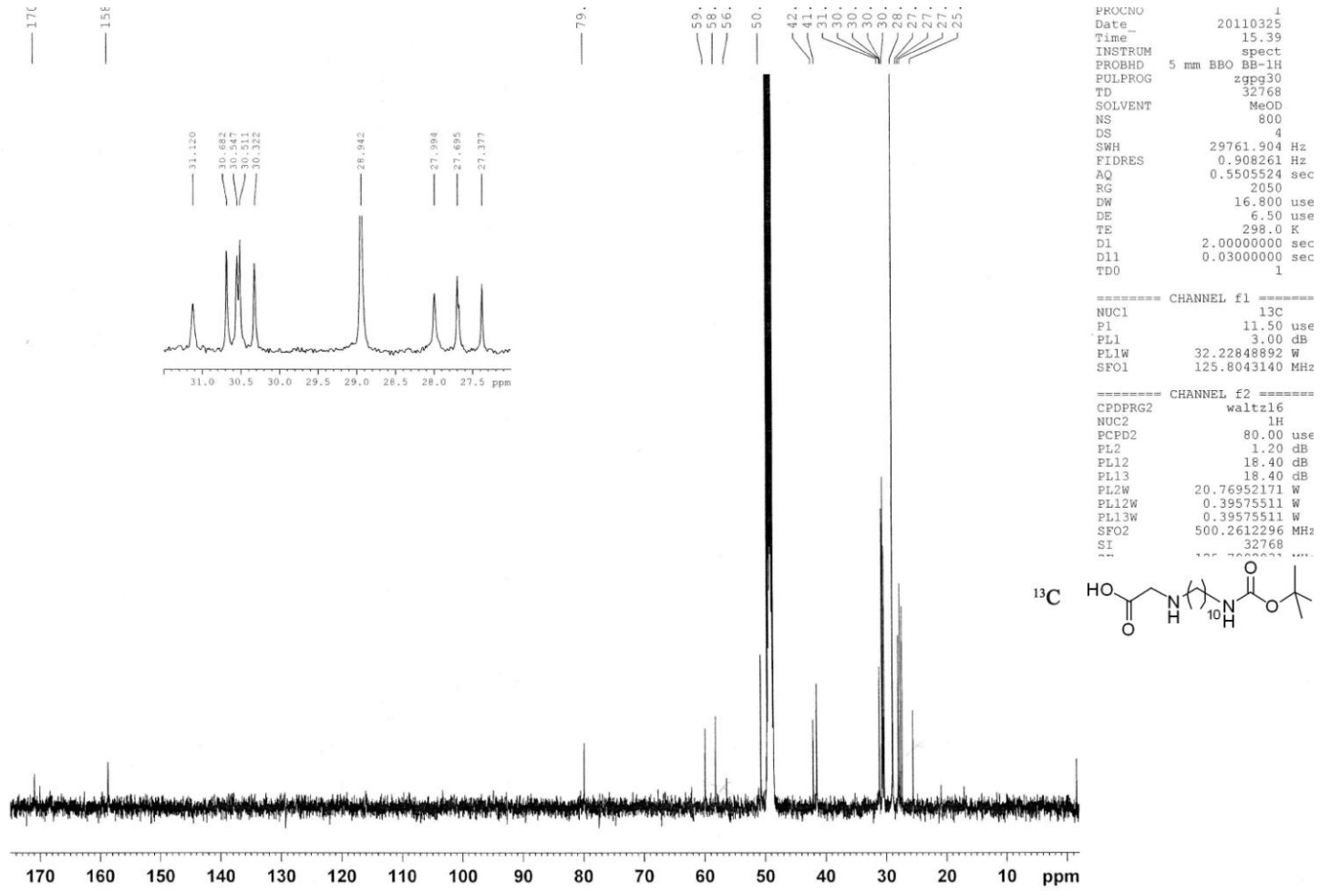

cafi83
AF-I-83
170.91
158.54
79.82
50.58
41.06
30.67
28.77
27.26
27.13
1H
HO
O
N
H
6
N
H
O
O
cafi83
AF-I-83
50.58
53
52
51
50
49
48
47
46
45
44
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



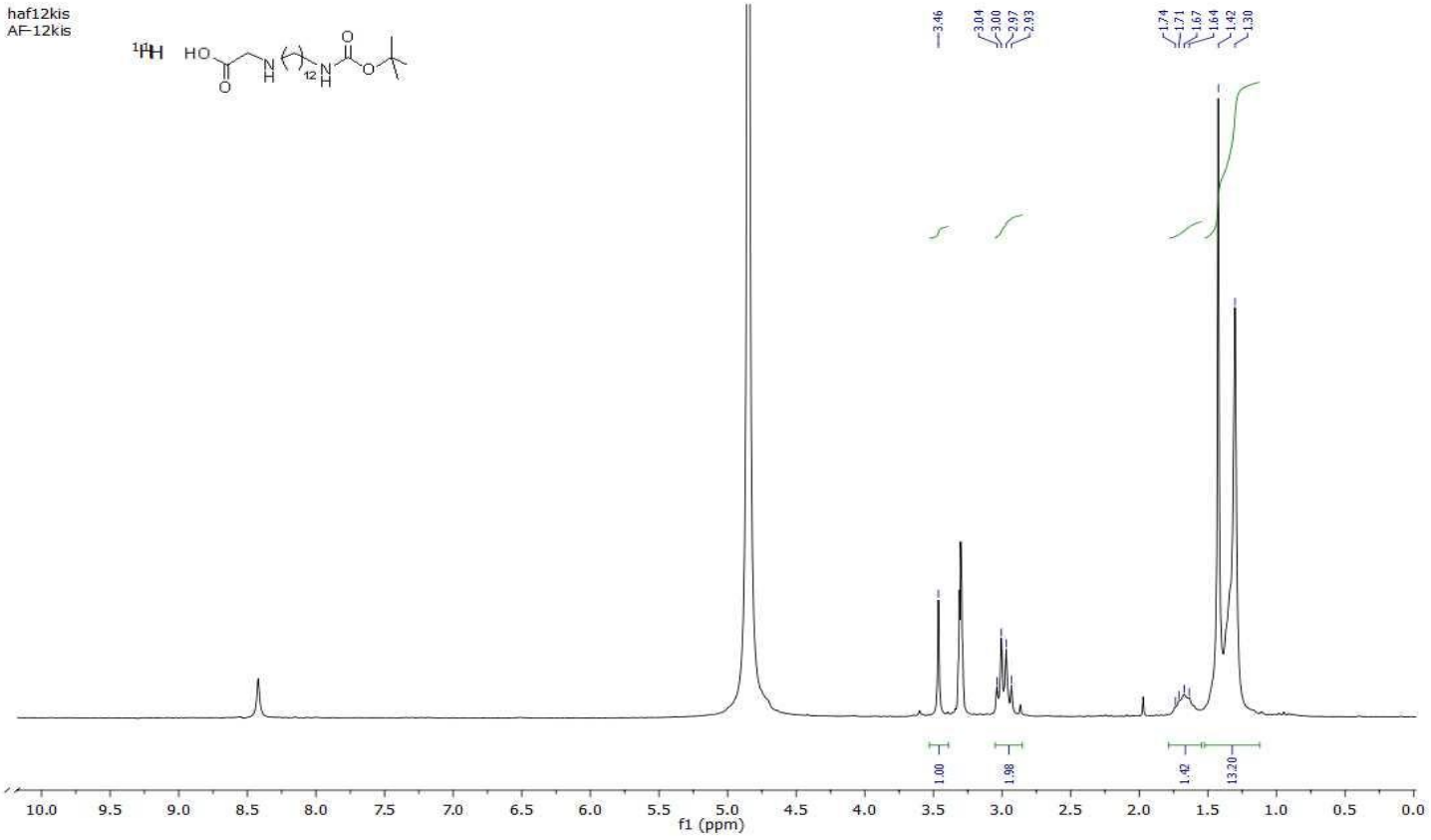


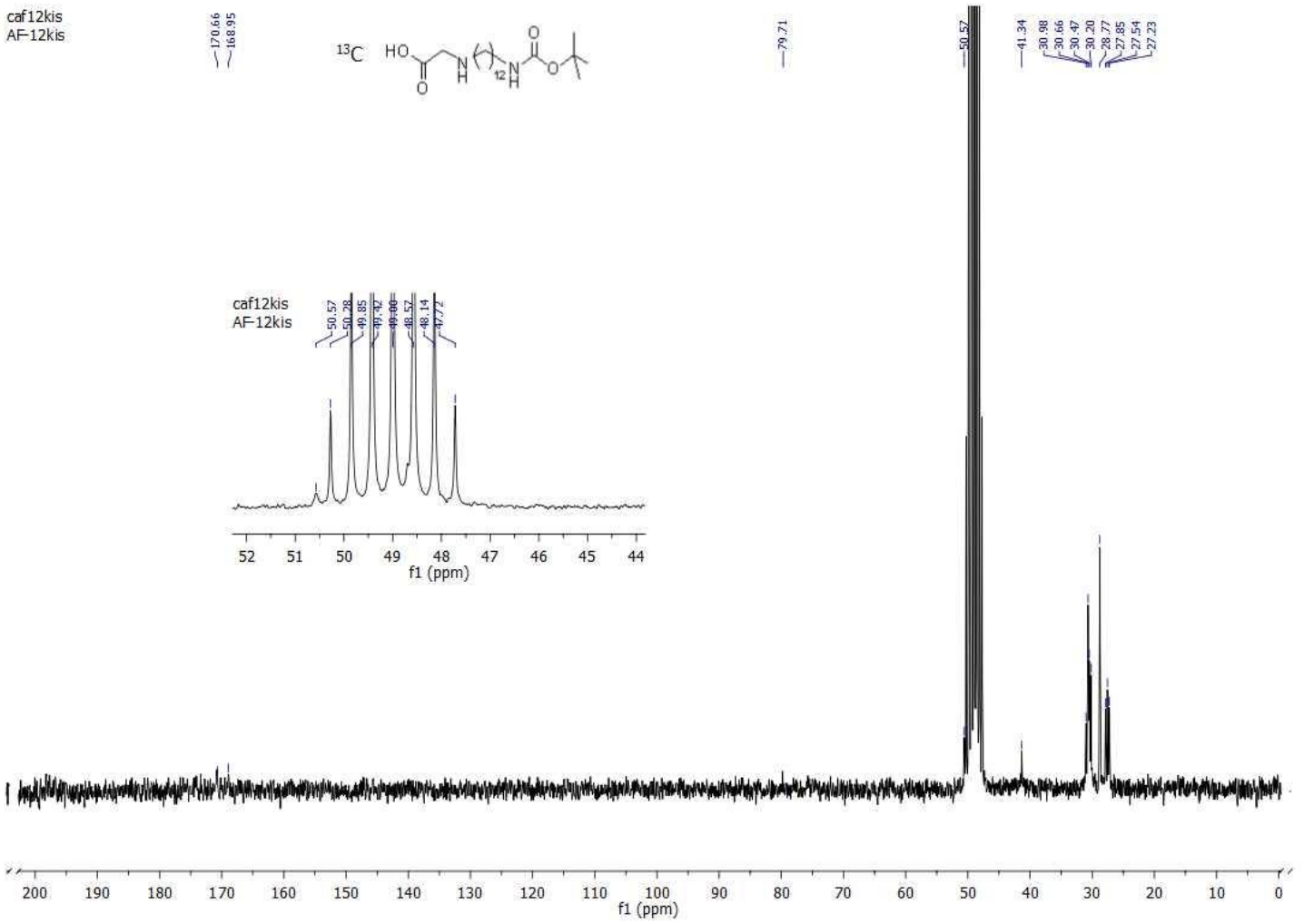
NMR spectra of 4d



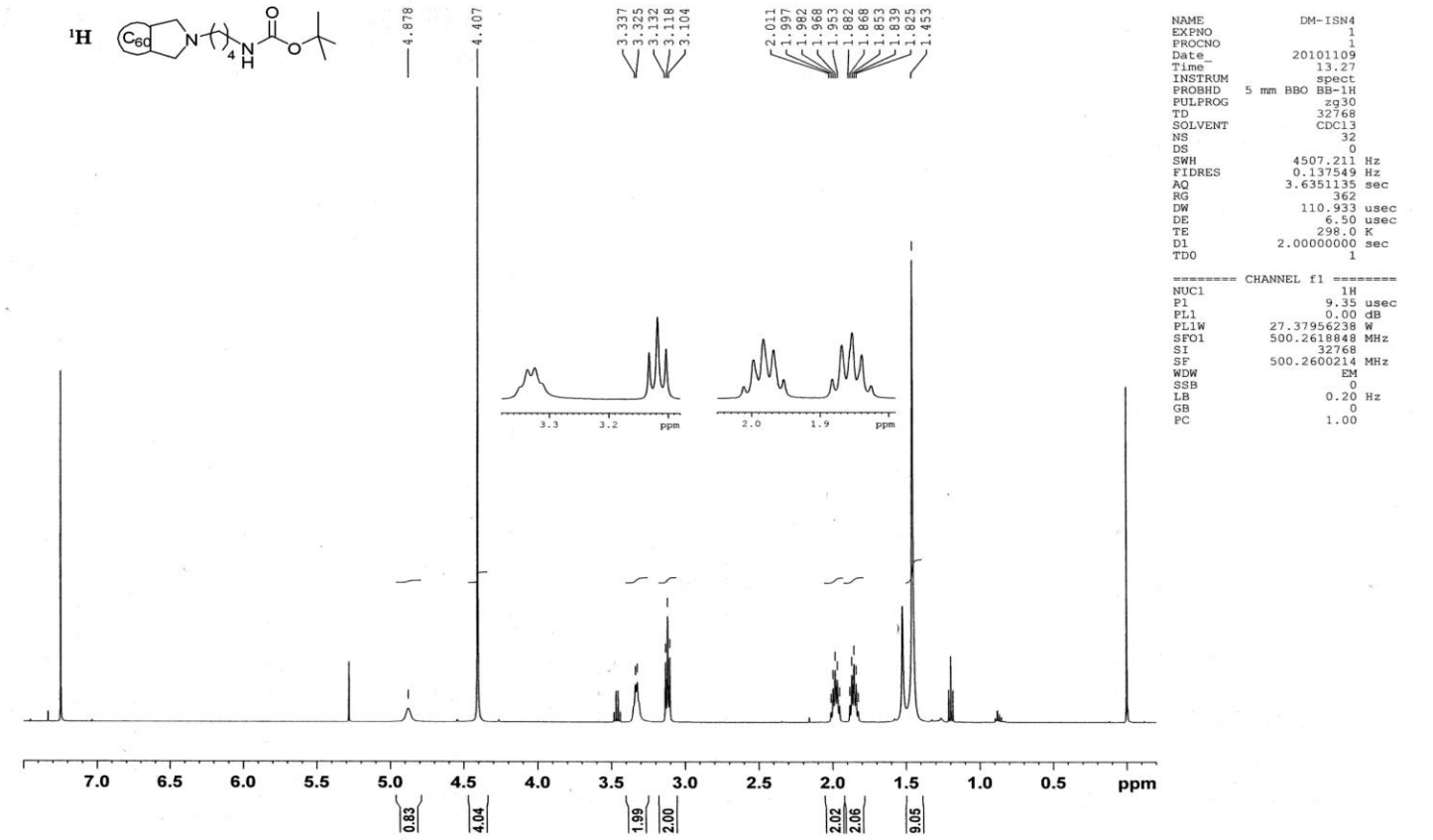


NMR spectra of 4e





NMR spectra of 4f




```
NAME                DM-ISN4
EXPNO                     1
PROCNO                    1
Date_              20101109
Time                  13.27
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        0
SWH                4507.211 Hz
FIDRES            0.137549 Hz
AQ                3.6351135 sec
RG                      362
DW                  110.933 usec
DE                     6.50 usec
TE                   298.0 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.35 usec
PL1                   0.00 dB
PL1W           27.37956238 W
SFO1           500.2618848 MHz
SI                    32768
SF             500.2600214 MHz
WDW                     EM
SSB                      0
LB                    0.20 Hz
GB                       0
PC                    1.00
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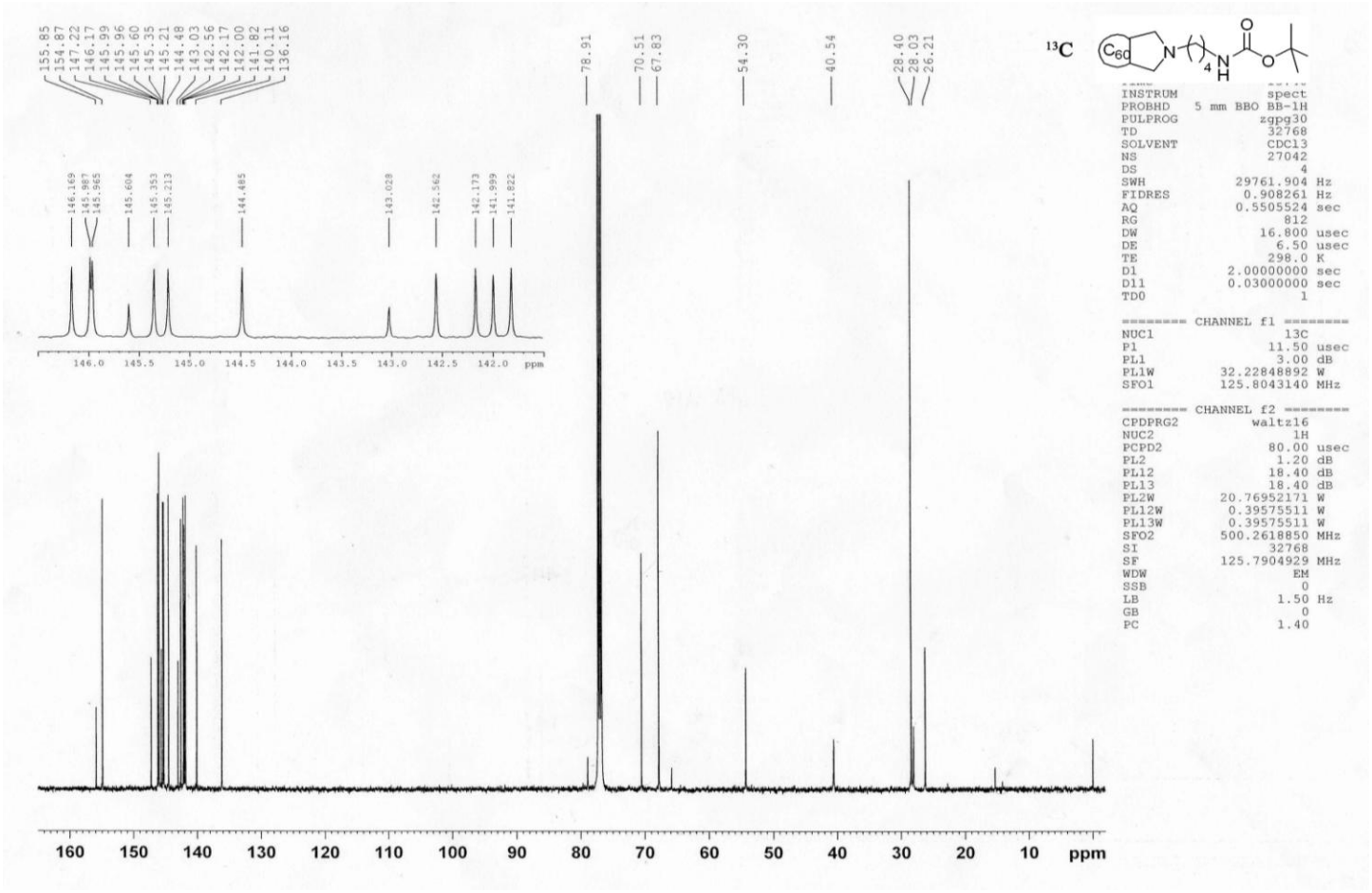
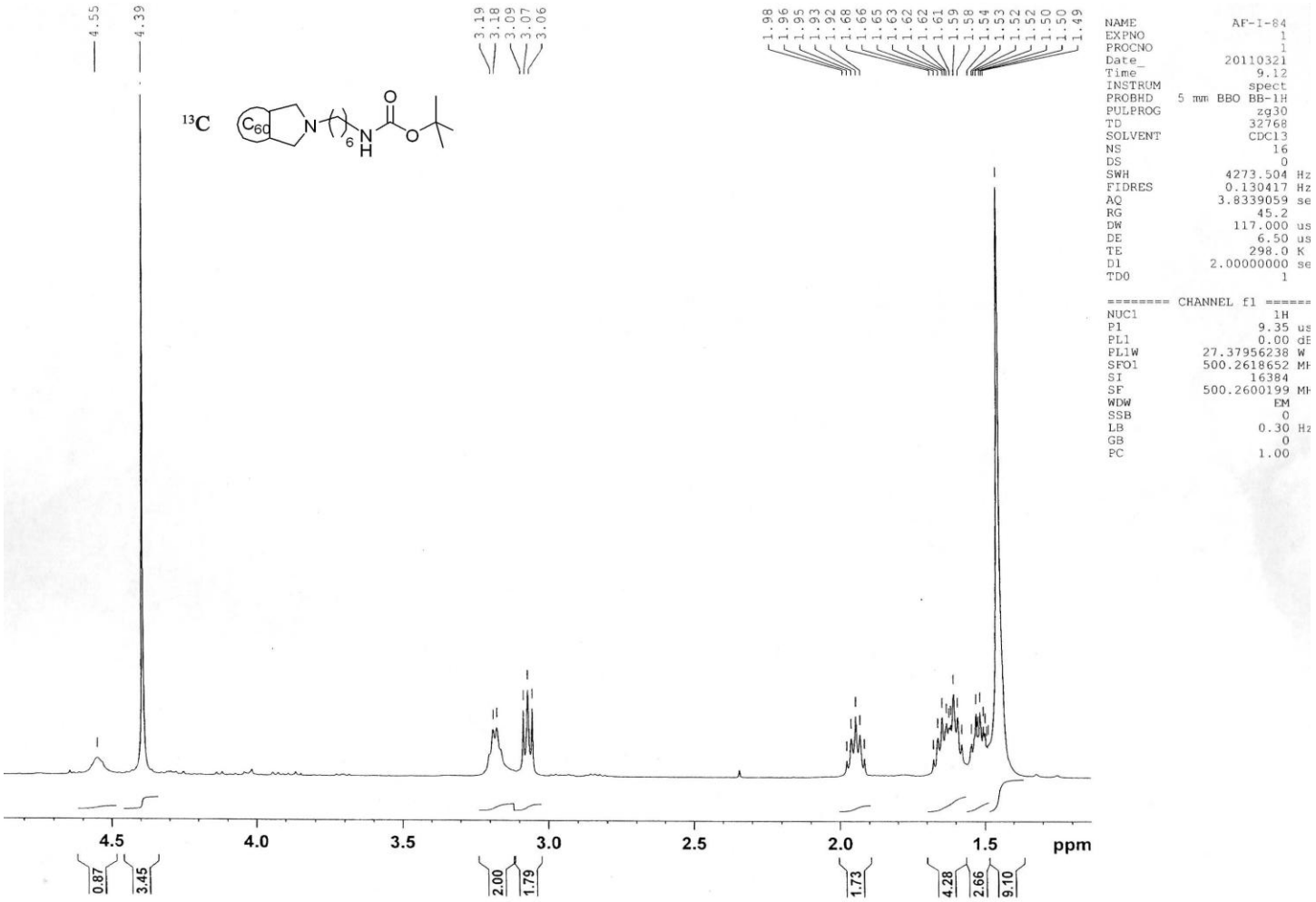



```
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                    27042
DS                        4
SWH               29761.904 Hz
FIDRES            0.908261 Hz
AQ                0.5505524 sec
RG                      812
DW                  16.800 usec
DE                     6.50 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                   11.50 usec
PL1                   3.00 dB
PL1W           32.22848892 W
SFO1           125.8043140 MHz

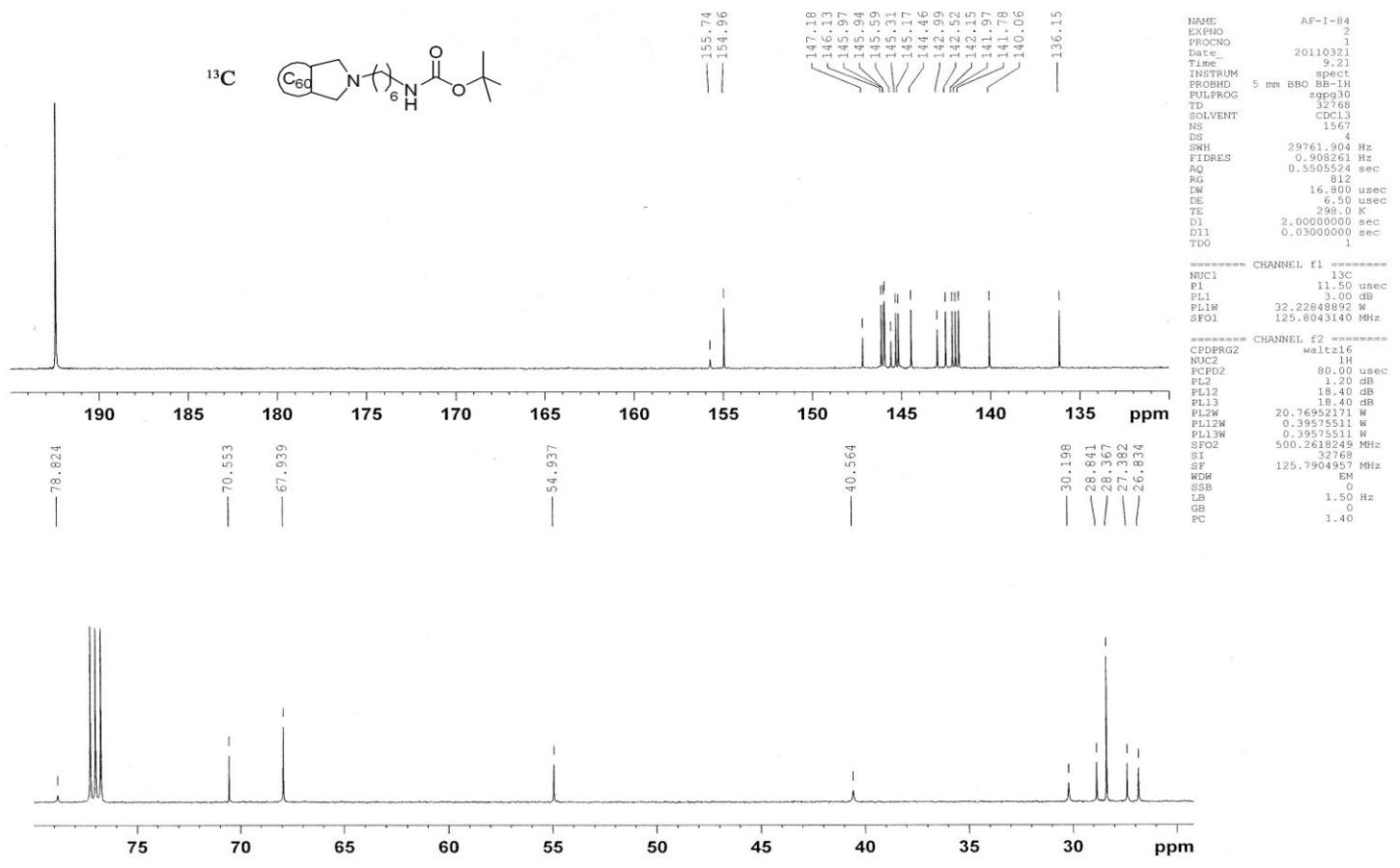
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.20 dB
PL12                 18.40 dB
PL13                 18.40 dB
PL2W           20.76952171 W
PL12W           0.39575511 W
PL13W           0.39575511 W
SFO2           500.2618850 MHz
SI                    32768
SF             125.7904929 MHz
WDW                     EM
SSB                      0
LB                    1.50 Hz
GB                       0
PC                    1.40
```



NAME	AF-I-84
EXPNO	1
PROCNO	1
Date_	20110321
Time	9.12
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4273.504 Hz
FIDRES	0.130417 Hz
AQ	3.8339059 se
RG	45.2
DW	117.000 us
DE	6.50 us
TE	298.0 K
D1	2.00000000 se
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	9.35 us
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2618652 MH
SI	16384
SF	500.2600199 MH
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



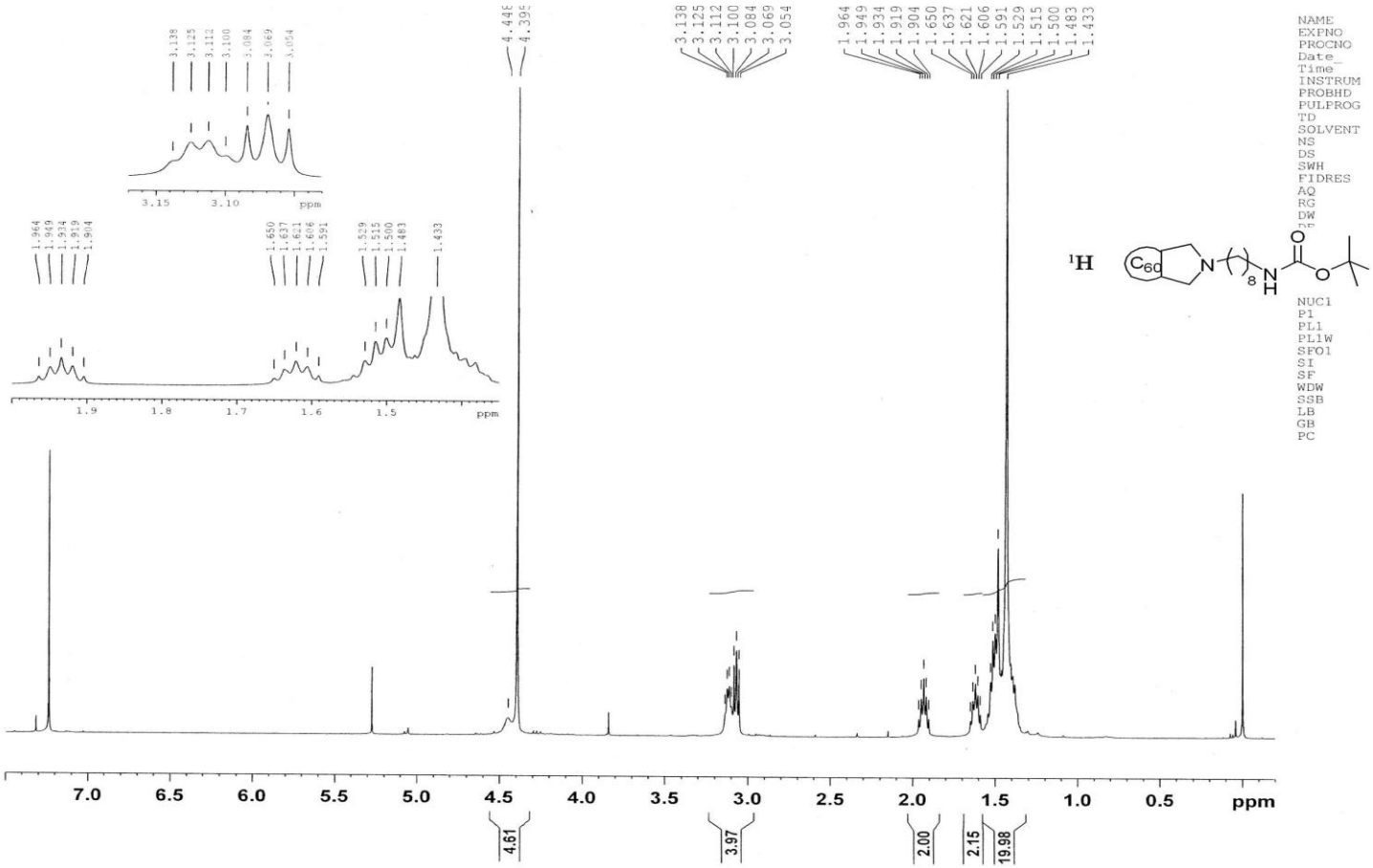
NAME	AF-I-84
EXPNO	2
PROCNO	1
Date_	20110321
Time	9.21
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1567
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	812
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

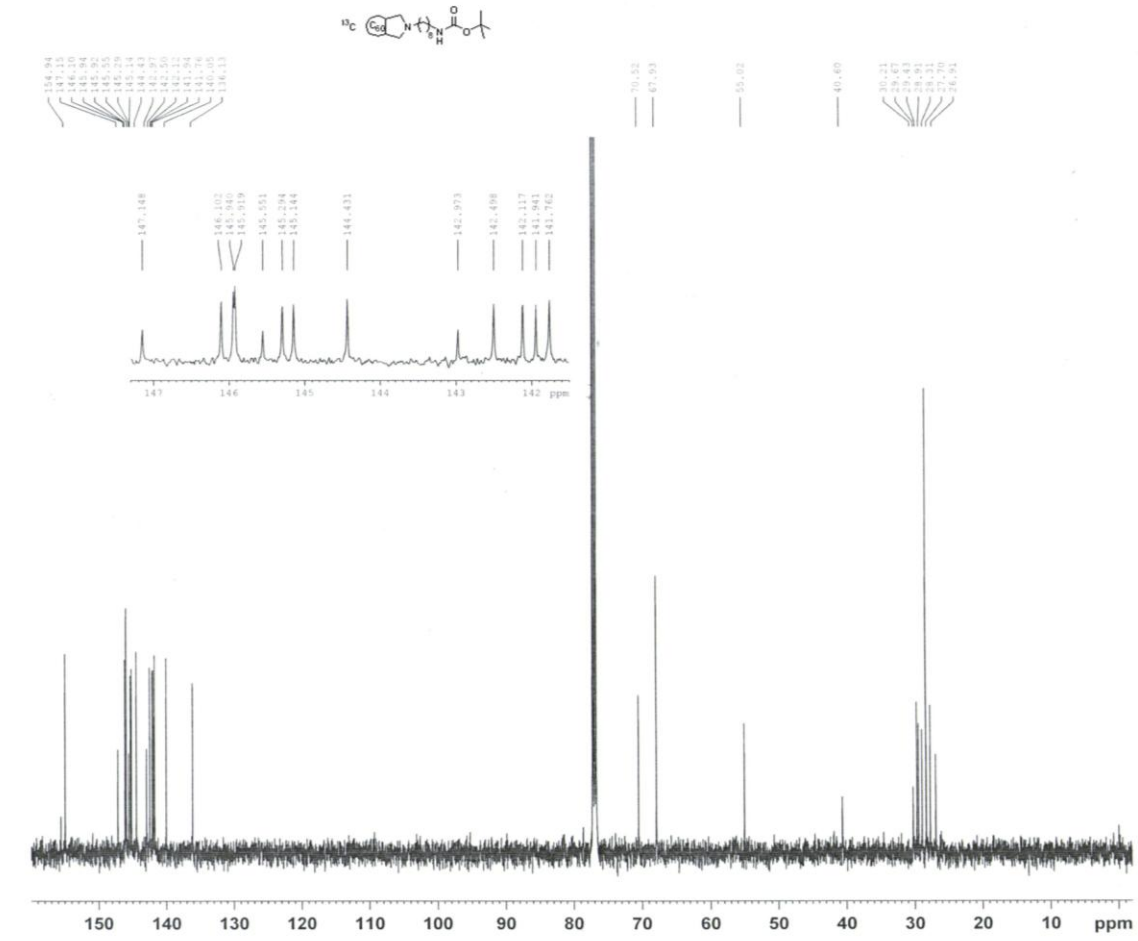
======== CHANNEL f1 ========

NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz

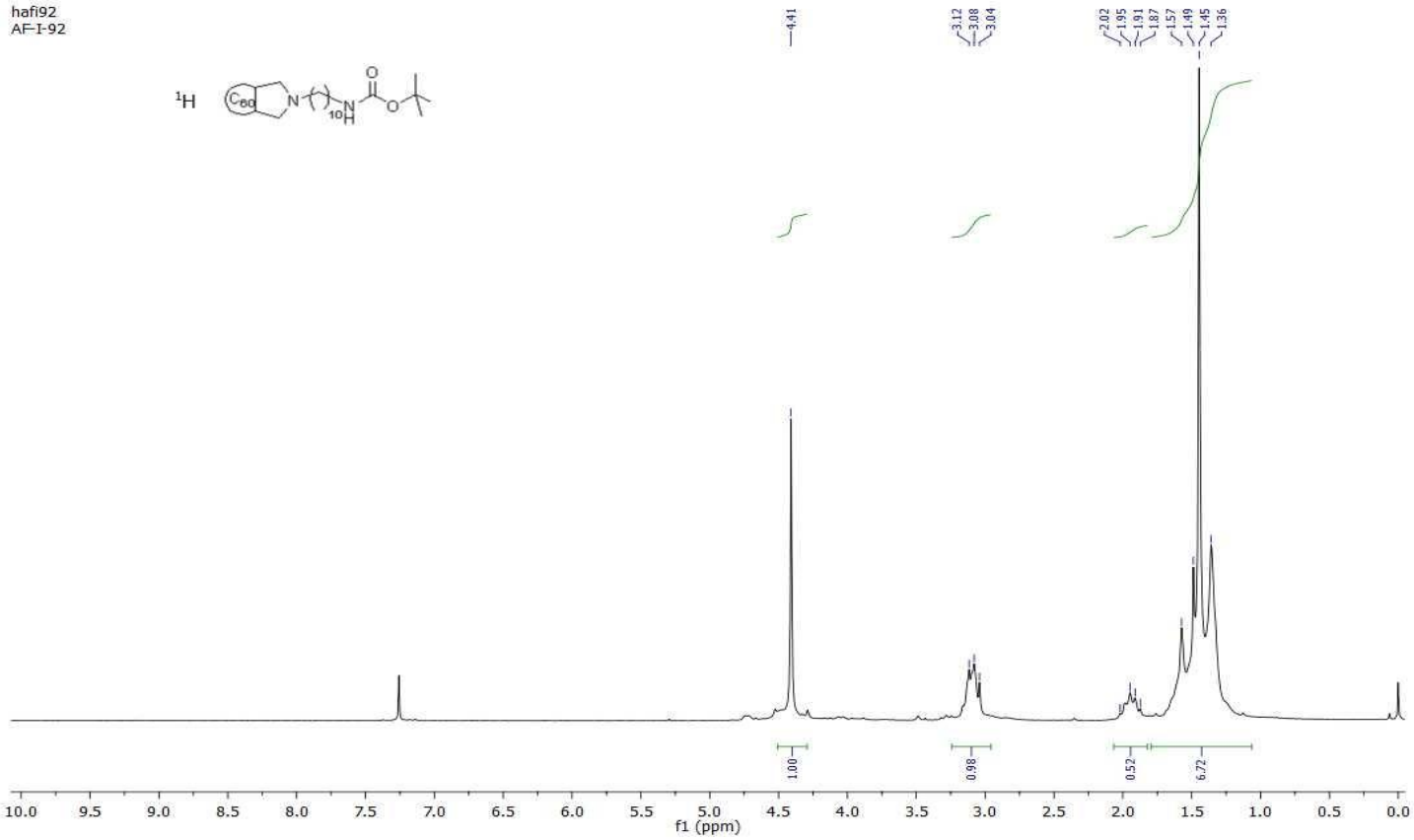
======== CHANNEL f2 ========

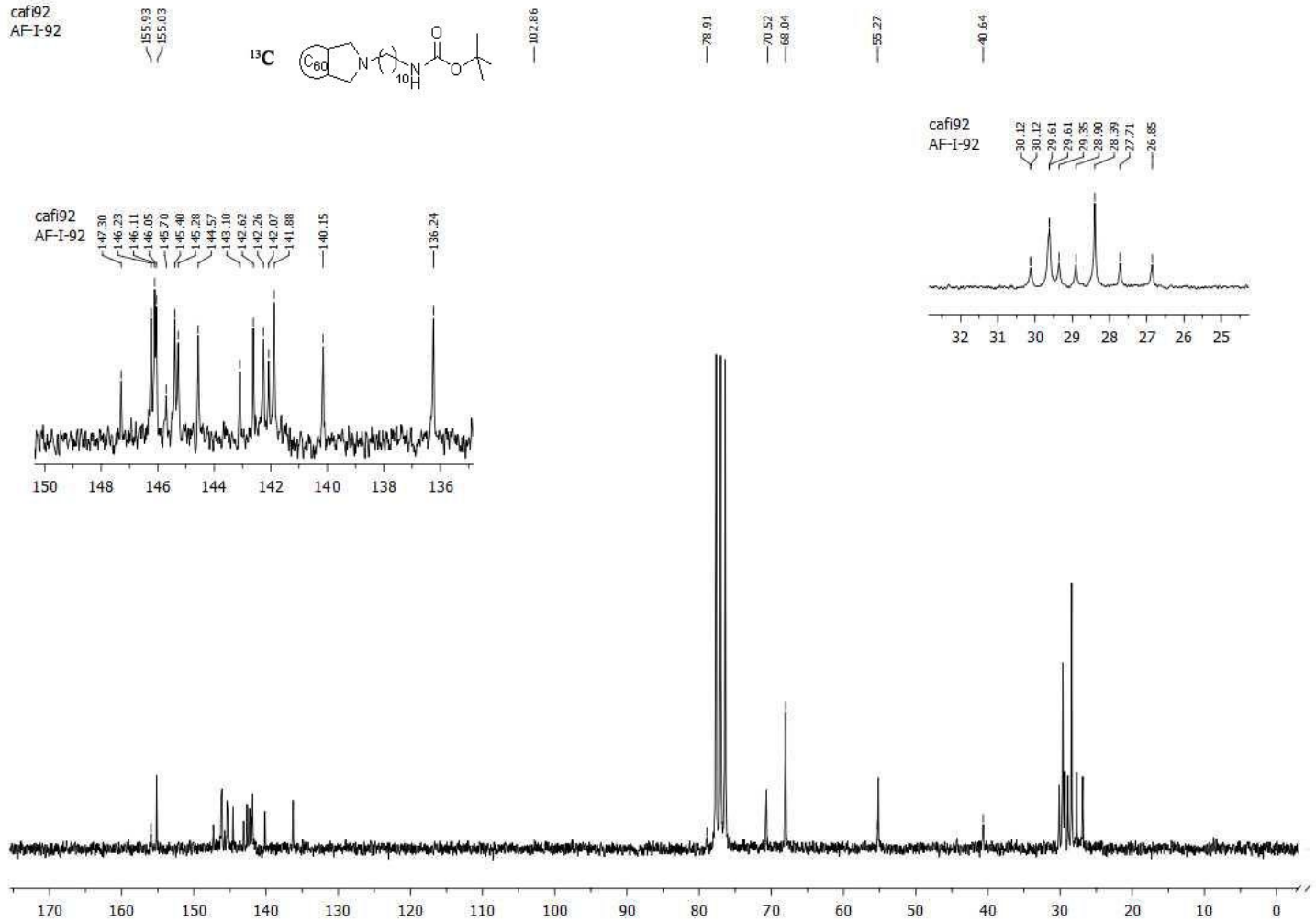
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618249 MHz
SI	32768
SF	125.7904957 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

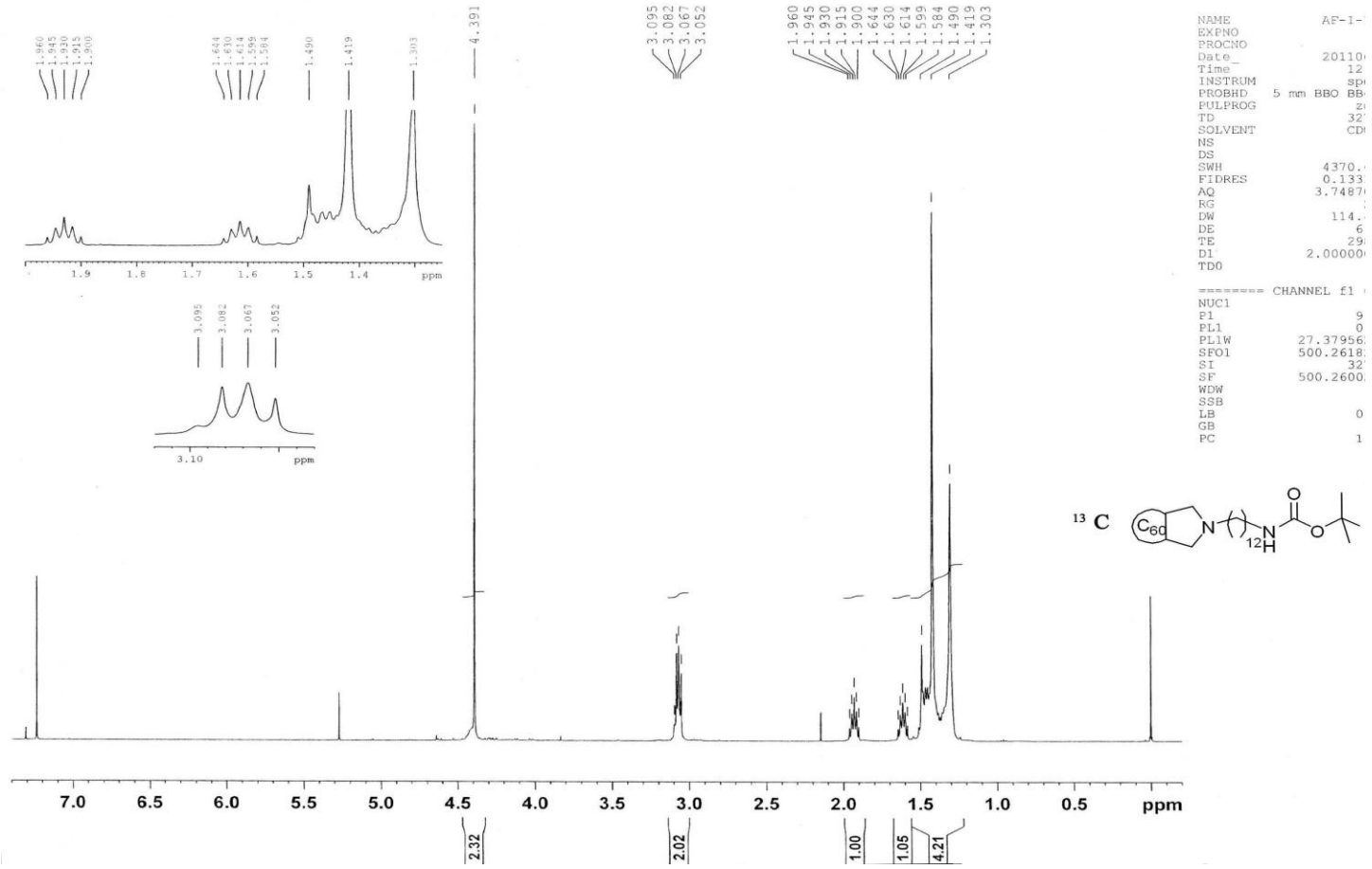


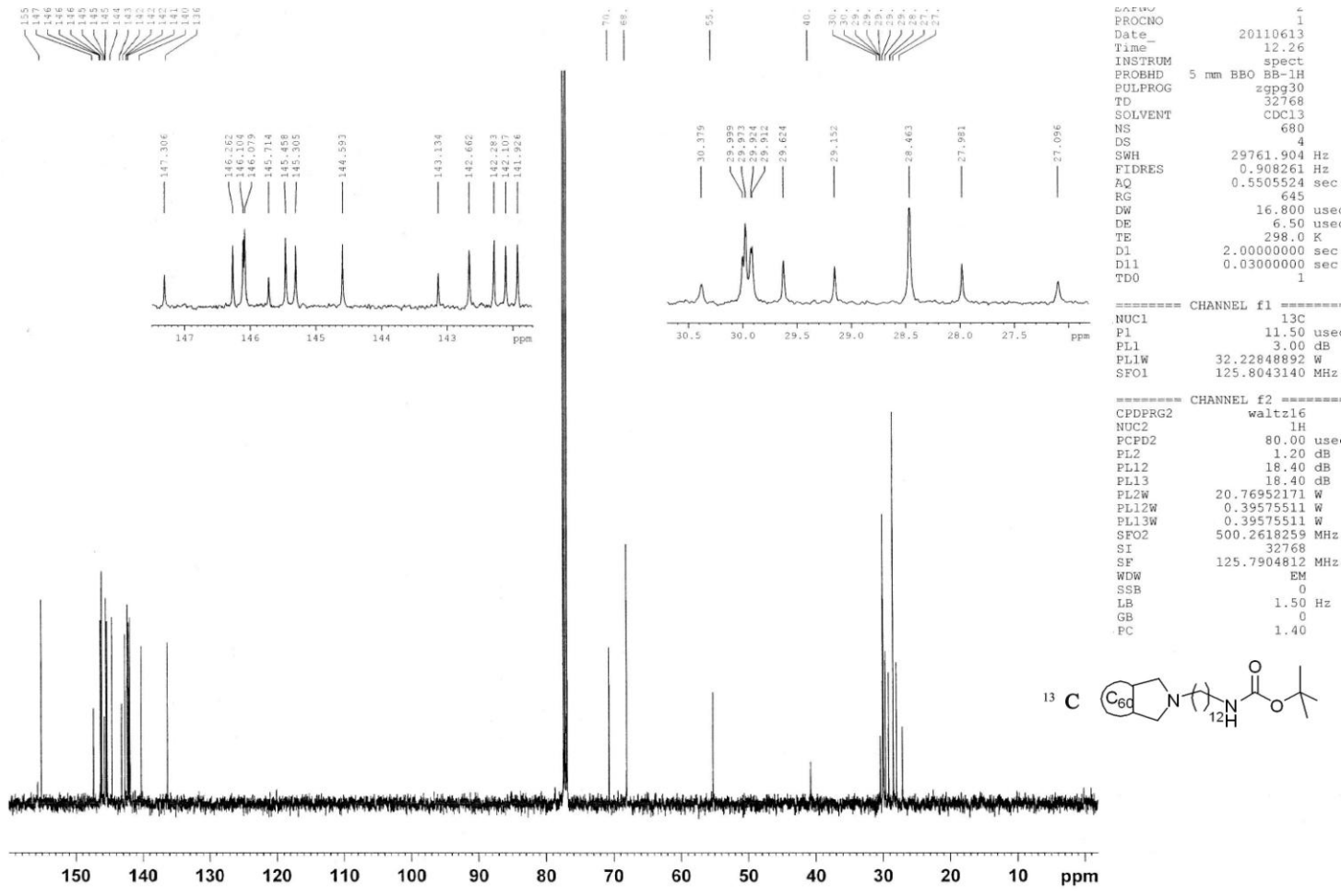


NAME	AF-I-70
EXPNO	2
PROCNO	1
Date_	20110624
Time_	11.32
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDC13
NS	1226
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2618950 MHz
SI	32768
SF	125.7905014 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40


hafi92
AF-I-92
^{1}H
4.41
3.12
3.08
3.04
2.02
1.95
1.91
1.87
1.57
1.49
1.45
1.36
1.00
0.98
0.52
6.72
f1 (ppm)


cafi92
AF-I-92
^{13}C
155.93
155.03
102.86
78.91
70.52
68.04
55.27
40.64
cafi92
AF-I-92
30.12
30.12
29.61
29.61
29.35
28.90
28.39
27.71
26.85
cafi92
AF-I-92
147.30
146.23
146.11
146.05
145.70
145.40
145.28
144.57
143.10
142.62
142.26
142.07
141.88
140.15
136.24





NMR spectra of 5f