Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

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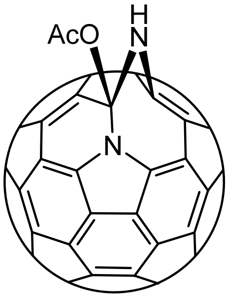
All reagents were used as received. All solvents were used as received. The reactions were carried out under atmosphere condition. The NMR spectra were obtained at 25 °C with 400, 500 and 600 MHz spectrometers (^{1}H and ^{13}C NMR spectra for the same compound were obtained with different spectrometers and different solvents in some cases). Chemical shifts are given in ppm relative to TMS or $CDCl_3$ (for ^{13}C NMR). ESI-FT-ICR-HRMS spectra were recorded in positive or negative mode. Chromatographic purifications were carried out with silica gel of mesh 200-300. Known compounds were characterized by comparison of their ^{1}H NMR data with literature data as cited.

Note: common impurities such as residue solvent toluene and grease from silica gel are sometimes impossible to be removed from the fullerene derivatives by routine flash column chromatography. Further purification by a diffusion-precipitation procedure was usually required to obtain pure samples. The diffusion-precipitation solvents are CS_2/CH_3CN or CS_2/hexane.

Preparation of Compound 4b:



A solution of compound **4a** (50 mg, 0.060 mmol) in 12.5 mL of PhCl was stirred in a sealed tube. Then HOAc (2.5 mL) and Ac_2O (2.5 mL) were added to the solution. The resulting solution was stirred at 150 ^{o}C for 18 hours. The solution was evaporated under vacuum. And then the residue was directly chromatographed on a silica gel column. Eluting with CS_2 gave the first brown band as unreacted compound **4a** (11.7 mg, 0.014 mmol, 23.4%), the second brown band as compound **4b** (20 mg, 0.025 mmol, 42%).

Characterization data for 4b:

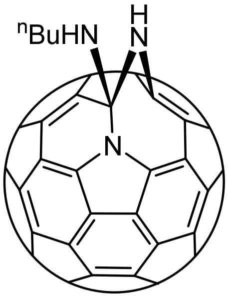
^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 8.92 (s, 1H), 2.63 (s, 3H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 170.13 (C=O), 148.65, 148.55, 148.18, 147.31, 147.25, 146.89, 146.85, 146.70, 146.12 (2C), 145.96, 145.89, 145.84, 145.78, 145.73, 145.58, 145.51, 145.40, 145.36, 144.99, 144.94, 144.92, 144.67, 144.56 (2C), 144.15, 143.70, 143.49, 143.41, 143.17, 142.90 (2C), 142.84, 142.20, 141.69, 141.57, 141.53, 141.47, 140.95, 140.33, 138.58, 137.95, 137.82, 137.80, 137.62, 136.71, 136.54, 135.95, 134.66, 134.44, 133.65, 133.59, 133.45, 133.30, 132.73, 131.78, 126.28, 125.91, 99.12 (1C, sp^3), 21.67 (CH_3).

FT-IR (microscope): 3320, 1754, 1542, 1512, 1456, 1425, 1364, 1225, 1212, 1005, 931, 921cm^{-1}.

ESI-FT-ICR-HRMS-Positive $C_{59}HN_2$ (M - OAc^-) calculated 737.0134, found 737.0149; $C_{60}H_5N_2O$ (M - OAc^- + CH_3OH) calculated 769.0396, found 769.0423.

Preparation of Compound 5a:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then *n*-butylamine (1 mL) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene:trimethylamine(TEA) (600:1) as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2:TEA:EA (600:1:6) to give compound **5a** as a brown solid (9.8 mg, 0.012 mmol, 14%).

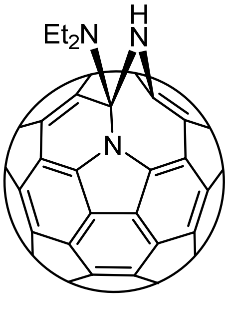
Characterization data for 5a:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 12.82 (s, 1H), 6.92 (s, 1H), 4.82-4.75 (m, 1H), 4.68-4.61 (m, 1H), 2.43-2.37 (m, 2H), 2.04-1.96 (m, 2H), 1.37-1.33 (m, 3H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (Because of poor solubility of compound **5a** in common organic solvent, the suitable ^{13}C NMR cannot be obtained. Selective signals, which correspond to the butyl groups, were shown below based on ^{13}C NMR we obtained.) δ 42.59 (1CH_2), 31.87 (1CH_2), 20.88 (1CH_2), 14.28 (1CH_3).

ESI-FT-ICR-HRMS-Positive $C_{63}H_{12}N_3$ (M + H^+) calculated 810.1026, found 810.1049.

Preparation of Compound 5b:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then diethylamine (1 mL) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2 to give compound **5b** as a brown solid (19.7 mg, 0.024 mmol, 27%).

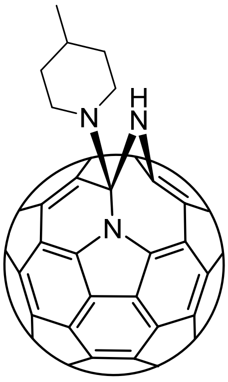
Characterization data for 5b:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 13.40 (s, 1H), 5.07-5.03 (m, 1H), 4.72-4.67 (m, 1H), 4.38-4.31 (m, 2H), 2.11-2.08 (t, J = 7.15 Hz, 3H), 1.87-1.84 (t, J = 7.17 Hz, 3H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 157.63, 151.13, 150.31, 149.24, 148.40, 148.09, 147.93, 147.78, 147.61, 147.25, 147.11, 146.94, 146.88, 146.81, 146.75 (2C), 146.55, 146.30, 146.17, 146.06, 146.05, 145.73, 145.52, 145.51, 145.49, 145.17, 145.12, 144.78, 143.69, 143.17, 143.14, 143.07, 142.84, 142.72, 142.71, 142.57, 141.59, 141.52, 141.35, 140.66, 140.36, 139.89, 139.21, 138.20, 137.90, 137.75, 137.13, 135.58, 135.49, 135.47, 135.25, 134.36, 133.27, 131.00, 128.45, 128.32, 127.77, 124.18, 113.38 (1C, sp^3), 46.47 (1CH_2), 43.11 (1CH_2), 15.29 (1CH_3), 13.36 (1CH_3).

ESI-FT-ICR-HRMS-Positive $C_{63}H_{12}N_3$ (M + H$^+$) calculated 810.1026, found 810.1040.

Preparation of Compound 5c:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then 4-methylpiperidine (1 mL) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2 to give compound **5c** as a brown solid (18 mg, 0.022 mmol, 24%).

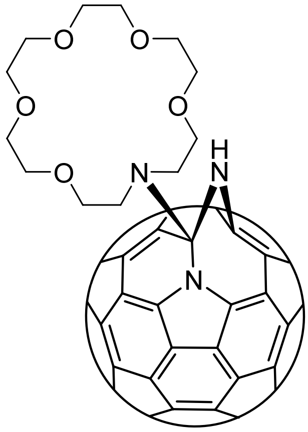
Characterization data for 5c:

Because of steric hindrance and restricted rotation in compound **5c**, signal broadening phenomenon has been observed. It was not possible to give accurate integrals for ^{1}H NMR. Selective signals of NMR data were shown below. ^{13}C NMR was not obtained.

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 13.49, 13.32, 7.26, 6.47, 6.45, 6.21, 6.18, 5.11, 5.08, 4.99, 4.96, 4.46, 4.44, 4.28, 4.27, 4.26, 4.25, 4.25, 4.23, 4.22, 4.21, 3.95, 3.93, 3.82, 3.79, 3.77, 3.77, 3.67, 3.67, 3.66, 3.65, 3.64, 3.62, 3.62, 3.54, 3.52, 3.49, 2.57, 2.54, 2.46, 2.45, 2.43, 2.41, 2.31, 2.29, 2.28, 2.27, 2.26, 2.24, 2.22, 2.19, 2.15, 2.12, 2.04, 2.01, 1.32, 1.30, 1.29, 1.27, 1.21, 1.20.

ESI-FT-ICR-HRMS-Positive $C_{65}H_{14}N_3$ (M + H$^+$) calculated 836.1182, found 836.1154.

Preparation of Compound 5d:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then pyridine (1 mL) and N-18-c-6 (100 mg) were added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene/dichloromethane/methanol (16:4:1) as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2/EA (ethylacetate) (3:1) to give compound **5d** as a brown solid (10 mg, 0.010 mmol, 11%).

Characterization data for 5d:

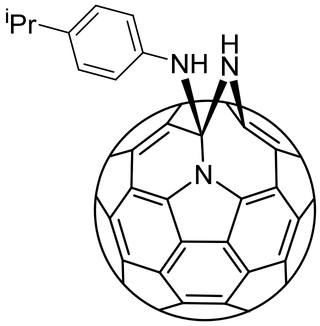
Because of poor solubility of compound **5d** in common organic solvent, the suitable CNMR cannot be obtained.

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 13.02 (s, 1H), 5.31-5.27 (m, 1H), 4.95-4.92 (m, 1H), 4.71-4.51 (m, 5H), 4.29-4.25 (m, 1H), 4.22-4.19 (m, 1H), 4.11-3.69 (m, 15H).

ESI-FT-ICR-HRMS-Positive $C_{71}H_{26}N_3O_5$ (M + H$^+$) calculated 1000.1867, found 1000.1851.

MS for (5d+KBPh$_4$): ESI-FT-ICR-HRMS-Positive $C_{71}H_{26}N_3O_5$ (M + H$^+$) calculated 1000.1867, found 1000.1847; $C_{71}H_{25}N_3O_5Na$ (M + Na$^+$) calculated 1022.1686, found 1022.1665; $C_{71}H_{25}N_3O_5K$ (M + K^+) calculated 1038.1426, found 1038.1409

Preparation of Compound 5e:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then 4-isopropylaniline (1 mL) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2:EA (100:1) to give compound **5e** as a brown solid (13.6 mg, 0.016 mmol, 18%).

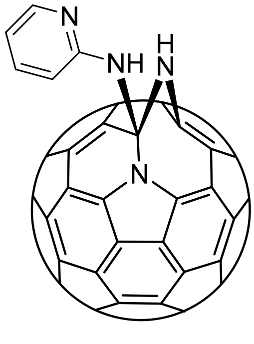
Characterization data for 5e:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 7.68 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.3 Hz, 2H), 7.29 (s, 1H), 5.89 (s, 1H), 3.04-2.98 (m, 1H), 1.40 (s, 3H), 1.38 (s, 3H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 148.76, 148.55, 148.10, 147.54, 147.43, 146.89, 146.83, 146.34, 146.19, 146.17, 146.07, 146.05, 145.87, 145.85, 145.69, 145.58, 145.49, 145.42, 145.15, 145.08, 145.02, 144.82, 144.56, 144.49, 144.28, 143.69, 143.58, 143.39, 143.29, 143.09, 142.99, 142.96, 142.91, 142.21, 141.78, 141.64, 141.52, 141.46, 140.93, 139.72, 138.89, 138.55, 137.92, 137.73, 137.66, 137.17, 136.88, 136.59, 135.95, 134.84, 134.81, 134.35, 134.03, 133.45, 133.29, 132.53, 127.64, 127.15, 125.75, 120.76, 89.99 (1C, sp^3), 33.68 (1C, **C**H(CH_3)$_2$), 24.10 (2C, CH(**C**H_3)$_2$).

ESI-FT-ICR-HRMS-Positive $C_{68}H_{14}N_3$ (M + H^+) calculated 872.1182, found 872.1201.

Preparation of Compound 5f:



To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then 2-aminopyridine (500 mg) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2:EA (50:1) to give compound **5f** as a brown solid (13.6 mg, 0.016 mmol, 18%).

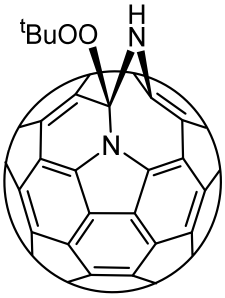
Characterization data for 5f:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 10.48 (s, 1H), 8.65 (d, J = 4.7 Hz, 1H), 7.81-7.78 (m, 1H), 7.08-7.06 (m, 2H), 7.00 (d, J = 8.2 Hz, 1H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 156.03, 151.48, 148.98, 148.86, 148.36, 147.89, 147.62, 147.44, 146.92, 146.67, 146.24, 146.16, 146.10, 145.97, 145.85, 145.64, 145.62, 145.47, 145.42, 145.18, 145.08, 144.68, 144.62, 144.05, 143.76, 143.02, 142.98, 142.74, 142.12, 141.97, 141.73, 141.28, 140.93, 138.75, 138.44, 138.13, 137.56, 137.45, 137.38, 136.99, 136.83, 135.87, 134.90, 134.31, 134.22, 133.73, 133.53, 132.96, 132.54, 126.71, 124.75, 116.10, 111.12, 89.33 (1C, sp^3).

ESI-FT-ICR-HRMS-Positive $C_{64}H_7N_4$ (M + H$^+$) calculated 831.0665, found 831.0679.

Preparation of Compound 6:



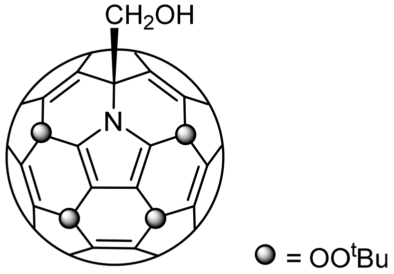
To a solution of the compound **3** (100 mg, 0.089 mmol) in 20 mL toluene was added BBr_3 (0.1 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 20 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then *t*-butyl hydroperoxide (70%, 3 ml) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene as a greenish band. The crude product was further purified by silica gel column chromatography eluting with CS_2 to give compound **6** as a brown solid (12 mg, 0.015 mmol, 16%).

Characterization data for 6:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 7.78 (s, 1H), 1.77 (s, 9H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 149.63, 148.73, 148.25, 147.95, 147.56, 147.46, 146.95, 146.91, 146.23, 146.15, 146.08 (2C), 145.95 (2C), 145.85, 145.73 (2C), 145.65, 145.47, 145.26, 145.16, 145.08, 144.72, 144.66, 144.56, 144.18, 143.78, 143.67, 143.39, 143.19, 143.05, 143.00, 142.99, 142.26, 141.96, 141.85, 141.70, 141.47, 141.14, 140.32, 138.84, 137.94, 137.89, 137.80, 137.73, 137.50, 137.25, 135.53 (2C), 135.09, 134.11, 134.07, 133.69, 133.68, 132.75, 130.57, 126.59, 125.50, 102.15 (1C, sp^3), 82.90 ($\mathbf{C}(CH_3)_3$), 26.97 (3C, $C(\mathbf{C}H_3)_3$).
ESI-FT-ICR-HRMS-Positive $C_{63}H_{11}N_2O_2$ (M + H$^+$) calculated 827.0815, found 827.0823.

Preparation of Compound 7a:

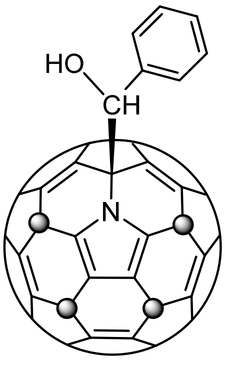


A solution of compound **1** (1 g, 0.927 mmol) in 167 mL of PhCl was stirred at 80 °C. Then paraformaldehyde (2.78 g, 92.7 mmol) and DABCO (1.04 g, 9.27 mmol) were added to the solution. The resulting solution was stirred at 80 °C for 2 hours. The solution was directly chromatographed on a silica gel column eluting with toluene to separate unreacted compound **1** and byproduct. Eluting with toluene/ethyl acetate (20:1) gave the red band as compound **7a** (513 mg, 0.463 mmol, 50%).

Characterization data for 7a:

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.90 (d, J = 6.9 Hz, 2H), 2.76 (t, J = 7.1 Hz, 1H), 1.46 (s, 18H), 1.44 (s, 18H).
^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 2C except as noted) δ 149.74, 149.39, 149.13 (1C), 149.10, 149.04, 148.65, 147.43, 147.40, 147.27, 147.22, 147.19, 147.12, 146.77, 146.66, 146.58, 146.50, 146.44 (1C), 146.26, 145.35, 145.24, 145.18, 143.56, 143.37, 143.30, 142.69, 141.28, 137.17, 129.79, 83.13 (2C, sp^3), 82.06 (2C, $\mathbf{C}(CH_3)_3$), 81.40 (2C, $\mathbf{C}(CH_3)_3$), 81.32 (2C, sp^3), 73.64 (1C, sp^3), 68.28 (1C), 26.82 (6C), 26.81 (6C).
ESI-FT-ICR-HRMS-Positive $C_{76}H_{40}NO_9$ (M + H$^+$) calculated 1110.2698, found 1110.2685.

Preparation of Compound 7b:



A solution of compound **1** (105 mg, 0.097 mmol) in 15 mL of toluene was stirred at 25 °C. Then benzaldehyde (6 mL) and 4-methylpiperidine (1.5 mL) were added to the solution. The resulting solution was stirred at 25 °C for 2.5 hours. Then the solution was washed by hydrochloric acid (2M) and water for 3 times respectively. The organic layer was first purified by silica gel column chromatography eluting with petroleum ether. The crude product was further washed by toluene and methanol to give the compound **7b** (34 mg, 0.029 mmol, 30%).

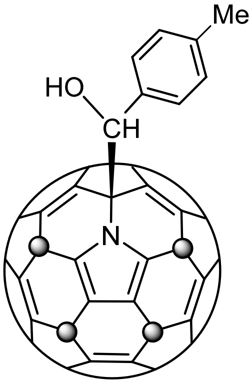
Characterization data for 7b:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.56 (m, 2H), 7.30-7.28 (m, 3H), 6.13 (d, J = 3.6 Hz, 1H), 3.62 (d, J = 4.1 Hz, 1H), 1.53 (s, 9H), 1.50 (s, 9H), 1.47 (s, 9H), 1.45 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 150.56, 149.69, 149.04, 149.02 (2C), 148.98, 148.95, 148.86, 148.72, 147.50, 147.42, 147.35, 147.30, 147.29, 147.22, 147.21, 147.20, 147.19, 146.94, 146.81, 146.79, 146.72, 146.69, 146.67, 146.61, 146.57, 146.56, 146.48, 146.43, 146.41, 146.37, 146.29, 146.21, 145.53, 145.41, 145.12, 145.10, 145.08, 144.76, 144.54, 143.38 (2C), 143.26, 143.23, 143.16, 142.65, 142.58, 141.65, 140.54, 137.62 (2C), 137.58, 136.72, 130.25, 129.66, 128.66, 128.05 (2C), 127.83 (2C), 83.20 (1C, sp^3), 83.09 (1C, sp^3), 82.21 ($\mathbf{C}(CH_3)_3$), 81.90 ($\mathbf{C}(CH_3)_3$), 81.51 ($\mathbf{C}(CH_3)_3$), 81.40 (1C, sp^3), 81.37 (1C, sp^3), 81.34 ($\mathbf{C}(CH_3)_3$), 78.81 (1C, sp^3), 77.63 (1C), 27.01 (3C), 26.84 (6C), 26.81 (3C).

ESI-FT-ICR-HRMS-Positive $C_{82}H_{44}NO_9$ (M + H$^+$) calculated 1186.3011 found 1186.2985.

Preparation of Compound 7c:



A solution of compound **1** (75 mg, 0.070 mmol) in 10 mL of toluene was stirred at

25 °C. Then *p*-tolualdehyde (4 mL) and 4-methylpiperidine (1 mL) were added to the solution. The resulting solution was stirred at 25 °C for 2 hours. Then the solution was washed by hydrochloric acid (2M) and water for 3 times respectively. And the organic layer was separated and evaporated. The residue was directly chromatographed on a silica gel column eluting with toluene to separate unreacted compound **1** and byproduct. Eluting with toluene/ethyl acetate (40:1) gave the red band as compound **7c** (26.3 mg, 0.022 mmol, 31%).

Characterization data for 7c:

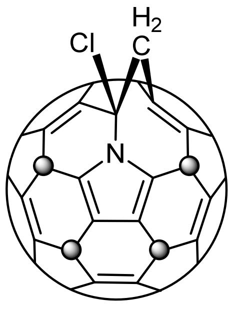
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, J = 8.1 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H), 6.09 (d, J = 3.7 Hz, 1H), 3.57 (d, J = 4.1 Hz, 1H), 2.28 (s, 3H), 1.53 (s, 9H), 1.50 (s, 9H), 1.47 (s, 9H), 1.45 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 150.62, 149.69, 149.04, 149.02, 149.00 (2C), 148.96, 148.94, 148.91, 148.86, 147.54, 147.49, 147.34, 147.29, 147.28, 147.23, 147.21, 147.19 (2C), 146.95, 146.94, 146.85, 146.78, 146.72, 146.68, 146.61, 146.60, 146.56, 146.48, 146.43 (2C), 146.37, 146.29, 146.21, 145.41, 145.14, 145.10 (2C), 144.77, 144.55, 143.36, 143.35 (2C), 143.25, 143.22, 143.13, 142.64, 142.57, 141.62, 140.49, 138.29, 137.55, 136.72, 134.63, 130.21, 129.60, 128.77 (2C), 127.69 (2C), 83.20 (1C, sp^3), 83.10 (1C, sp^3), 82.16 (**C**$(CH_3)_3$), 81.87 (**C**$(CH_3)_3$), 81.50 (**C**$(CH_3)_3$), 81.41 (1C, sp^3), 81.37 (1C, sp^3), 81.33 (**C**$(CH_3)_3$), 78.68 (1C, sp^3), 77.76 (1C), 26.99 (3C), 26.84 (6C), 26.81 (3C), 21.25 (1C).

ESI-FT-ICR-HRMS-Positive $C_{83}H_{46}NO_9$ $(M + H^+)$ calculated 1200.3167, found 1200.3162.

Preparation of Compounds 8 and 9:

A solution of compound **7a** (335 mg, 0.302 mmol) in 56 mL of freshly distilled CH_2Cl_2 was stirred at 25 °C. Then PCl_5 (1.675 g, 8.03 mmol) were added to the solution. The resulting solution was stirred at 25 °C for 5-10 minutes. Then the reaction was quenched with water. The solution was washed by saturated sodium bicarbonate solution and water for 3 times respectively. And the organic layer was separated and evaporated. The residue was directly chromatographed on a silica gel column. Eluting with toluene gave the first red band as an inseparable mixture of compound **8** and **9** (250 mg, 0.222 mmol, 74%, ratio= 20:1).

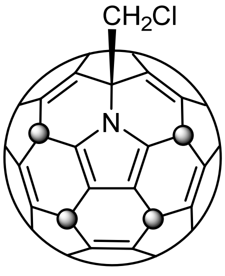


8

Characterization data for 8:

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.96 (d, J = 13.1 Hz, 1H), 4.29 (d, J = 13.1 Hz, 1H), 1.49 (s, 9H), 1.45 (s, 9H), 1.42 (s, 9H), 1.41 (s, 9H).

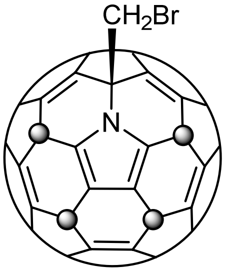
^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 151.13, 150.82, 149.67, 149.29, 149.08, 148.51, 148.12 (2C), 147.95, 147.90, 147.70, 147.51, 147.47, 147.43, 147.28, 147.25, 147.19, 147.15 (2C), 146.94, 146.92, 146.69, 146.54, 146.47, 146.44, 146.31, 146.29, 145.87, 145.75, 145.54, 145.35, 145.24, 145.09, 144.27, 144.24 (3C), 143.46, 143.43, 143.38, 143.36, 142.75, 142.61, 142.54, 141.34, 139.71, 138.73, 136.50, 136.33, 135.43, 134.14, 132.61, 130.67, 129.68, 88.93 (1C, sp^3), 85.12 (1C, sp^3), 82.78 (1C, sp^3), 82.16 (**C**$(CH_3)_3$), 81.76 (1C, sp^3), 81.52 (**C**$(CH_3)_3$), 81.48 (**C**$(CH_3)_3$), 81.37 (**C**$(CH_3)_3$), 77.06 (1C, sp^3), 38.79 (1C), 26.95 (3C), 26.88 (3C), 26.86 (3C), 26.81 (3C).
ESI-FT-ICR-HRMS-Positive $C_{76}H_{42}ClN_2O_8$ (M + NH_4^+) calculated 1145.2624, found 1145.2636.



9

Characterization data for 9:
^1H NMR (400 MHz, $CDCl_3$) δ 4.91 (s, 2H), 1.46 (s, 18H), 1.44 (s, 18H).

Preparation of Compound 10:



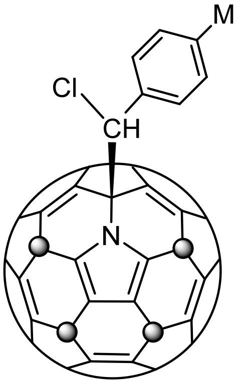
A solution of compound **7a** (50 mg, 0.045 mmol) in 10 mL of toluene was stirred at 35 °C. Then PPh_3 (200 mg, 0.763 mmol) and NBS (200 mg, 1.12 mmol) were added to the solution. The resulting solution was stirred at 35 °C for 50 minutes. The solution was directly chromatographed on a silica gel column. Eluting with toluene gave the first red band as compound **10** (18 mg, 0.015 mmol, 33%).

Characterization data for 10:
^1H NMR (500 MHz, $CDCl_3$) δ 4.79 (s, 2H), 1.46 (s, 18H), 1.44 (s, 18H).
^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 2C except as noted) δ 149.81, 149.11 (1C), 149.08, 149.07, 148.67, 148.48, 147.46, 147.31, 147.28, 147.26, 147.08, 146.88, 146.83, 146.55, 146.41 (3C), 146.35, 146.12, 145.29, 145.12, 144.64, 143.71, 143.41, 143.30, 142.76, 141.15, 137.47, 129.14, 83.14 (2C, sp^3), 82.18 (2C, **C**$(CH_3)_3$), 81.45 (2C, **C**$(CH_3)_3$), 80.96 (2C, sp^3), 70.42 (1C, sp^3), 36.18 (1C), 26.81 (6C), 26.74 (6C).

ESI-FT-ICR-HRMS-Positive $C_{76}H_{39}BrNO_8$ (M + H$^+$) calculated 1172.1854, found 1172.1875.

Crystal of compound **10** suitable for X-ray diffraction was obtained from slow evaporation in a mixture of carbon disulfide and ethanol. **Crystal Data** for $C_{76}H_{38}BrNO_8$ (M =1172.98 g/mol): monoclinic, space group P2$_1$/n (no. 14), a = 13.8397(9) Å, b = 24.2642(13) Å, c = 16.7166(13) Å, β = 109.444(8)°, V = 5293.4(7) Å^3, Z = 4, T = 180.00(10) K, μ(MoKα) = 0.845 mm^{-1}, $Dcalc$ = 1.472 g/cm^3, ? reflections measured (6.144° ≤ 2Θ ≤ 52.044°), 10314 unique (R_{int} = ?, R_{sigma} = 0.1648) which were used in all calculations. The final R_1 was 0.0699 (I > 2σ(I)) and wR_2 was 0.1714 (all data). CCDC 1522610 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

Preparation of Compound 11:



A solution of compound **7c** (16 mg, 0.013 mmol) in 4 mL of toluene was stirred at 25 °C. Then PCl_5 were added to the solution. The resulting solution was stirred at 25 °C for 10 minutes. Then the reaction was quenched with water. The solution was washed by saturated sodium bicarbonate solution and water for 3 times respectively. And the organic layer was directly chromatographed on a silica gel column eluting with toluene to give the compound **11** almost quantitatively (15 mg, 0.012 mmol, 92%).

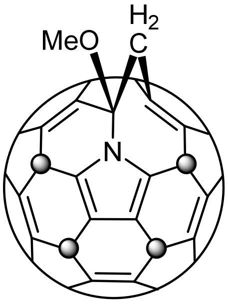
Characterization data for 11:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, J = 7.9 Hz, 2H), 7.10 (d, J = 7.9 Hz, 2H), 6.46 (s, 1H), 2.29 (s, 3H), 1.53 (s, 9H), 1.48 (s, 18H), 1.44 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 151.34, 149.80, 149.09, 149.08, 149.06, 149.00, 148.96, 148.71, 148.42, 148.07, 147.58, 147.50, 147.37 (2C), 147.31 (2C), 147.28, 147.21, 147.16, 147.03, 146.72 (3C), 146.69 (2C), 146.49, 146.48, 146.47, 146.42 (3C), 146.36, 146.27, 146.24, 145.65, 145.09, 145.06, 145.05, 144.62, 144.27, 143.45, 143.42, 143.34, 143.32, 143.28, 143.23, 142.73, 142.63, 142.52, 140.42, 139.09, 137.73, 137.62, 133.04, 130.07, 129.28 (2C), 129.03, 128.98 (2C), 83.21 (1C, sp^3), 83.17 (1C, sp^3), 82.60 (**C**(CH_3)$_3$), 81.59 (**C**(CH_3)$_3$), 81.48 (**C**(CH_3)$_3$), 81.41 (1C, sp^3), 81.31 (**C**(CH_3)$_3$), 81.21 (1C, sp^3), 75.84 (1C, sp^3), 66.41 (1C), 26.95 (3C), 26.94 (3C), 26.87 (3C), 26.81 (3C), 21.28 (1C).

ESI-FT-ICR-HRMS-Positive $C_{83}H_{45}ClNO_8$ (M + H^+) calculated 1218.2828, found 1218.2865.

Preparation of Compound 12a:



A solution of compound **8** and **9** (30 mg, ratio= 20:1, **8**: 0.025 mmol) in 5 mL of freshly distilled CH_2Cl_2 was stirred at 25 °C. Then $AgClO_4 \cdot H_2O$ (330 mg, 1.46 mmol) and MeOH (3 ml) were added to the solution. The resulting solution was stirred at 25 °C for 7 hours. Then the solution was washed by water for 3 times. And the organic layer was directly chromatographed on a silica gel column eluting with petroleum ether/toluene (1:1) to separate the first red band as unreacted compound **8** and **9**. Eluting with toluene gave the second red band as compound **12a** (8 mg, 0.007 mmol, 28%).

Characterization data for 12a:

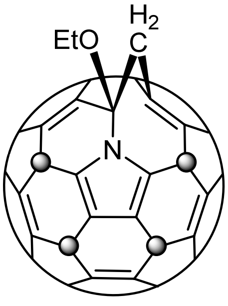
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.69 (d, J = 12.2 Hz, 1H), 3.80 (d, J = 12.2 Hz, 1H), 3.66 (s, 3H), 1.48 (s, 9H), 1.46 (s, 9H), 1.43 (s, 9H), 1.38 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 192.58, 151.83, 150.69, 149.66, 149.36, 148.96, 148.49, 148.18, 148.00, 147.86, 147.78 (2C), 147.50 (2C), 147.35 (2C), 147.30, 147.24 (2C), 147.12, 147.06, 147.04, 146.84, 146.54, 146.53, 146.45, 146.33, 145.94, 145.83, 145.65, 145.47, 144.78, 144.40 (2C), 144.11, 144.10, 144.04, 143.40, 143.38, 143.27, 143.24, 142.40, 142.33, 142.15, 141.15, 139.58, 138.79, 137.73, 137.09, 136.00, 133.41, 133.16, 129.74, 129.54, 90.55 (1C, sp^3), 88.71 (1C, sp^3), 85.60 (1C, sp^3), 82.84 (1C, sp^3), 81.86 (1C, sp^3), 81.77 (**C**$(CH_3)_3$), 81.37 (**C**$(CH_3)_3$), 81.26 (**C**$(CH_3)_3$), 81.17 (**C**$(CH_3)_3$), 51.01 (1C), 31.04 (1C), 26.88 (3C), 26.86 (3C), 26.82 (3C), 26.75 (3C).

ESI-FT-ICR-HRMS-Positive $C_{77}H_{42}NO_9$ (M + H^+) calculated 1124.2854, found 1124.2821.

Preparation of Compounds 12b and 12d:

A solution of compound **8** and **9** (60 mg, ratio= 20:1, **8**: 0.051 mmol) in 10 mL of freshly distilled CH_2Cl_2 was stirred at 25 °C. Then $AgClO_4 \cdot H_2O$ (600 mg, 2.66 mmol) and EtOH (3 ml) were added to the solution. The resulting solution was stirred at 25 °C for 4 hours and then first purified by silica gel column chromatography eluting with CH_2Cl_2. The crude product was further purified by silica gel column chromatography eluting with petroleum ether/toluene (1:1) to separate the first red band as unreacted compound **8** and **9**, then give the second red band as compound **12b** (41mg, 0.036 mmol, 71%). Eluting with toluene gave the third red band as byproduct difficult to be clearly characterized and the fourth band as compound **12d** (11 mg, 0.010 mmol, 20%).



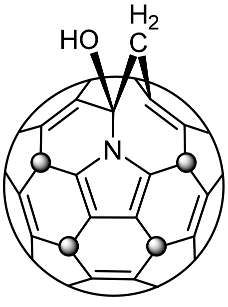
12b

Characterization data for 12b:

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.72 (d, J = 12.2 Hz, 1H), 4.03-3.97 (m, 1H), 3.85-3.80 (m, 2H), 1.54 (t, J = 7.0 Hz, 3H), 1.47 (s, 9H), 1.46 (s, 9H), 1.42 (s, 9H), 1.38 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 152.04, 150.65, 149.63, 149.34, 148.92, 148.45, 148.08, 147.96, 147.84, 147.78, 147.71, 147.58, 147.48, 147.37, 147.33, 147.25, 147.18, 147.07, 147.02 (2C), 146.79 (2C), 146.53, 146.49, 146.43, 146.31 (2C), 145.88, 145.77, 145.53, 145.43, 144.76, 144.38, 144.30, 144.05, 144.03, 144.00, 143.33 (2C), 143.28, 143.18, 142.35, 142.33, 142.32, 141.09, 139.56, 138.73, 137.80, 137.10, 136.10, 133.36, 133.00, 129.98, 129.18, 90.24 (1C, sp^3), 88.67 (1C, sp^3), 85.74 (1C, sp^3), 82.80 (1C, sp^3), 81.82 (1C, sp^3), 81.75 ($\mathbf{C}(CH_3)_3$), 81.38 ($\mathbf{C}(CH_3)_3$), 81.30 ($\mathbf{C}(CH_3)_3$), 81.20 ($\mathbf{C}(CH_3)_3$), 59.72 (1C), 31.58 (1C), 26.87 (3C), 26.83 (3C), 26.80 (3C), 26.77 (3C), 15.20 (1C).

ESI-FT-ICR-HRMS-Positive $C_{78}H_{44}NO_9$ (M + H$^+$) calculated 1138.3011, found 1138.2997.



12d

Characterization data for 12d:

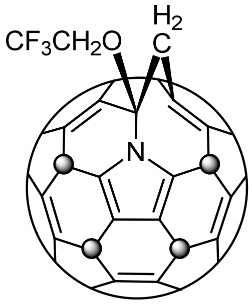
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.95 (s, 1H), 4.52 (d, J = 12.7 Hz, 1H), 3.97 (d, J = 12.7 Hz, 1H), 1.47 (s, 9H), 1.45 (s, 9H), 1.44 (s, 9H), 1.42 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 150.72, 149.66, 149.30, 149.13, 148.96, 148.67, 148.51, 148.24, 148.05, 147.97, 147.95, 147.78, 147.57, 147.54 (2C), 147.33, 147.17, 147.13, 147.07, 147.06, 146.95, 146.57, 146.44, 146.39, 146.34, 146.28, 146.20, 146.11, 145.69, 145.39, 145.36, 145.05, 144.36, 144.20, 144.18, 144.07, 143.82, 143.70, 143.54, 143.28, 143.20, 142.53 (2C), 142.36, 141.12, 139.98, 138.74, 138.00, 137.12, 134.75, 133.05, 131.44, 130.11, 129.12, 88.70 (1C, sp^3), 86.18 (1C, sp^3), 85.05 (1C, sp^3), 82.78 (**C**$(CH_3)_3$), 82.69 (1C, sp^3), 81.89 (**C**$(CH_3)_3$), 81.86 (1C, sp^3), 81.46 (**C**$(CH_3)_3$), 81.32 (**C**$(CH_3)_3$), 34.04 (1C), 26.86 (3C), 26.81 (3C), 26.79 (3C), 26.64 (3C).

ESI-FT-ICR-HRMS-Positive $C_{76}H_{40}NO_9$ ($M + H^+$) calculated 1110.2698, found 1110.2676; $C_{76}H_{43}N_2O_9$ ($M + NH_4^+$) calculated 1127.2963, found 1127.2970.

Crystal of compound **12d** suitable for X-ray diffraction was obtained from slow evaporation in a mixture of carbon disulfide and methanol. **Crystal Data** for $C_{76}H_{39}NO_9$ (M =1110.08 g/mol): N/A, space group Pna2_1 (no. 33), a = 21.489(4) Å, b = 10.179(2) Å, c = 22.215(4) Å, V = 4859.2(17) Å^3, Z = 4, T = 100 K, μ(MoKα) = 0.100 mm^{-1}, $Dcalc$ = 1.517 g/cm^3, 27095 reflections measured (6.62° ≤ 2Θ ≤ 62.52°), 14273 unique (R_{int} = 0.0348, R_{sigma} = N/A) which were used in all calculations. The final R_1 was 0.0656 (>2sigma(I)) and wR_2 was 0.1998 (all data). CCDC 1522611 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

Preparation of Compound 12c:



A solution of compound **8** and **9** (60 mg, ratio= 20:1, **8**: 0.051 mmol) in 6 mL of CH_2Cl_2 was stirred at 25 °C. Then $AgClO_4 \cdot H_2O$ (600 mg, 2.663 mmol) and CF_3CH_2OH (2 ml) were added to the solution. The resulting solution was stirred at 25 °C for 5 minutes. The reaction was terminated upon confirmation of complete conversion of compound **8** as indicated by TLC. Then the solution was washed by water for 3 times. And the organic layer was directly chromatographed on a silica gel column eluting with petroleum ether/toluene (1:1) to separate the first red band as byproduct difficult to be clearly characterized and give the second red band as compound **12c** (18 mg, 0.015 mmol, 29%).

Characterization data for 12c:

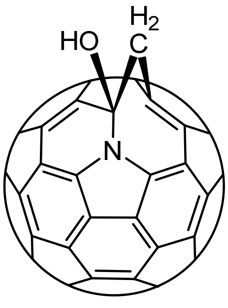
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.72 (d, J = 12.2 Hz, 1H), 4.19-4.30 (m, 2H), 3.92 (d, J = 12.2 Hz, 1H), 1.49 (s, 9H), 1.46 (s, 9H), 1.42 (s, 9H), 1.37 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) (all signals represent 1C except as noted) δ 151.46, 150.74, 149.70, 149.36, 149.05, 148.56, 148.09, 148.01, 147.87 (2C), 147.54 (2C), 147.51, 147.36, 147.23, 147.18, 147.10, 147.06, 147.01 (2C), 146.91, 146.60, 146.55, 146.46 (2C), 146.32, 146.16, 145.84, 145.52 (2C), 145.49, 145.16, 144.44, 144.43, 144.13 (2C), 144.11, 143.47, 143.38, 143.32, 143.30, 142.55, 142.27, 141.41, 140.58, 139.55, 138.86, 136.97, 136.92, 136.57, 133.30, 132.82, 130.43, 129.59, 123.53 (q, J = 277.7 Hz, CF_3), 90.58 (1C, sp^3), 88.60 (1C, sp^3), 85.51 (1C, sp^3), 82.73 (1C, sp^3), 82.12 (**C**(CH$_3$)$_3$), 81.83 (1C, sp^3), 81.56 (**C**(CH$_3$)$_3$), 81.52 (**C**(CH$_3$)$_3$), 81.37 (**C**(CH$_3$)$_3$), 62.00 (q, *J* = 36.7 Hz, CH$_2$), 31.59 (1C), 26.83 (3C), 26.81 (3C), 26.77 (3C), 26.63 (3C).

ESI-FT-ICR-HRMS-Positive $C_{78}H_{41}F_3NO_9$ (M + H$^+$) calculated 1192.2728, found 1192.2735; $C_{78}H_{44}F_3N_2O_9$ (M + NH$_4^+$) calculated 1209.2993, found 1209.2992.

Preparation of Compounds 13 and 14:

To a solution of the compound **8** and **9** (58 mg, ratio= 3:1, **8**: 0.039mmol, **9**: 0.013mmol) in 11 mL freshly distilled toluene was added BBr$_3$ (0.05 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 12 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh$_3$ (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene. The crude product was further purified by silica gel column chromatography eluting with CS$_2$ to give the first brown band as compound **14** (4.6 mg, 0.006 mmol, 46%), and with toluene to give the second brown band as compound **13** (6.2 mg, 0.008 mmol, 21%).



13

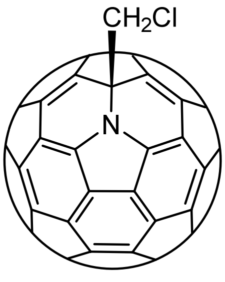
Characterization data for 13:

^{1}H NMR (500 MHz, CS$_2$/CDCl$_3$) δ 5.73 (d, *J* = 12.4 Hz, 1H), 5.30 (d, *J* = 12.4 Hz, 1H), 4.51 (s, 1H).

^{13}C NMR (126 MHz, CS$_2$/1,4-Dioxane-d$_8$) (all signals represent 1C except as noted) δ 149.34, 149.23, 149.15, 148.91 (2C), 147.44, 147.42, 147.41, 147.12, 146.78, 146.71, 146.61, 146.46, 146.33, 146.27, 146.20 (2C), 146.10, 146.07, 145.91, 145.84, 145.82, 145.58, 145.23, 145.17, 145.08, 144.32, 144.12, 143.95, 143.82, 143.51, 143.49, 142.97, 142.83, 142.69, 142.67, 142.51, 142.45, 142.39, 142.35, 141.95, 141.56, 140.15, 140.11, 140.06, 139.87, 139.22, 138.92, 138.06, 135.92, 135.63, 134.95, 134.64, 134.16, 134.10, 130.76, 128.75, 126.75, 88.65 (1C, sp^3), 40.47 (1C).

ESI-FT-ICR-HRMS-Positive $C_{60}H_4NO$ (M + H$^+$) calculated 754.0287, found 754.0299.

MALDI-TOF-MS $C_{60}H_3NO$ calculated 753.0, found 753.2.



14

Characterization data for 14:

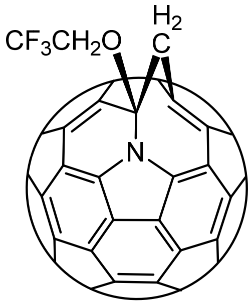
^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 5.64 (s, 2H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 2C except as noted) δ 154.81, 147.56 (1C), 147.33, 147.29, 147.27, 147.09, 146.44, 146.40, 146.10 (4C), 145.68 (3C), 145.64, 144.94, 144.74, 144.42, 144.18, 143.82, 143.09, 142.71, 142.02, 141.70, 141.38, 140.99, 140.86, 140.65, 139.65, 137.42, 134.52, 123.95, 81.71 (1C, sp^3), 53.01 (1C).

ESI-FT-ICR-HRMS-Positive $C_{60}H_2ClN$ (M – e$^-$) calculated 770.9870, found 770.9871.

MALDI-TOF-MS $C_{60}H_2N$ (M – Cl$^-$) calculated 736.0, found 736.1.

Preparation of Compound 15:



To a solution of the compound **12c** (34 mg, 0.029 mmol) in 7 mL freshly distilled toluene was added BBr_3 (0.05 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 7 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene. The crude product was further purified by silica gel column chromatography eluting with CS_2 to give the brown band as compound **15** (6 mg, 0.007 mmol, 24%)

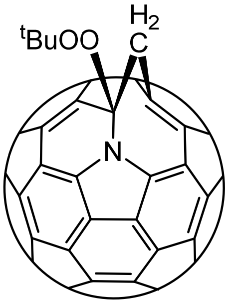
Characterization data for 15:

^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 5.65 (d, J = 12.1 Hz, 1H), 5.22 (d, J = 12.1 Hz, 1H), 4.81-4.96 (m, 2H).

^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 148.25, 148.07, 148.02, 147.86, 147.40, 146.54, 146.49, 146.43, 145.99, 145.97, 145.87, 145.60, 145.55, 145.50 (2C), 145.32, 145.29, 145.25, 145.10, 145.02, 144.88, 144.85, 144.49, 144.41, 144.28, 143.52, 143.37, 143.16, 142.99, 142.96, 142.74, 142.26, 142.02, 141.86, 141.73, 141.68, 141.56, 141.18, 140.98, 140.29, 140.14, 139.43,

139.32, 138.67, 138.28, 138.21, 137.57, 135.45, 135.29, 134.81, 133.96, 133.87, 133.66, 133.56, 130.92, 128.53, 128.39, 126.04, 123.30 (q, J = 278.2 Hz, CF_3), 91.82 (1C, sp^3), 62.36 (q, J = 35.8 Hz, CH_2), 35.17 (1C).
MALDI-TOF-MS $C_{62}H_5ONF_3$ ($M + H^+$) calculated 836.0, found 836.1.

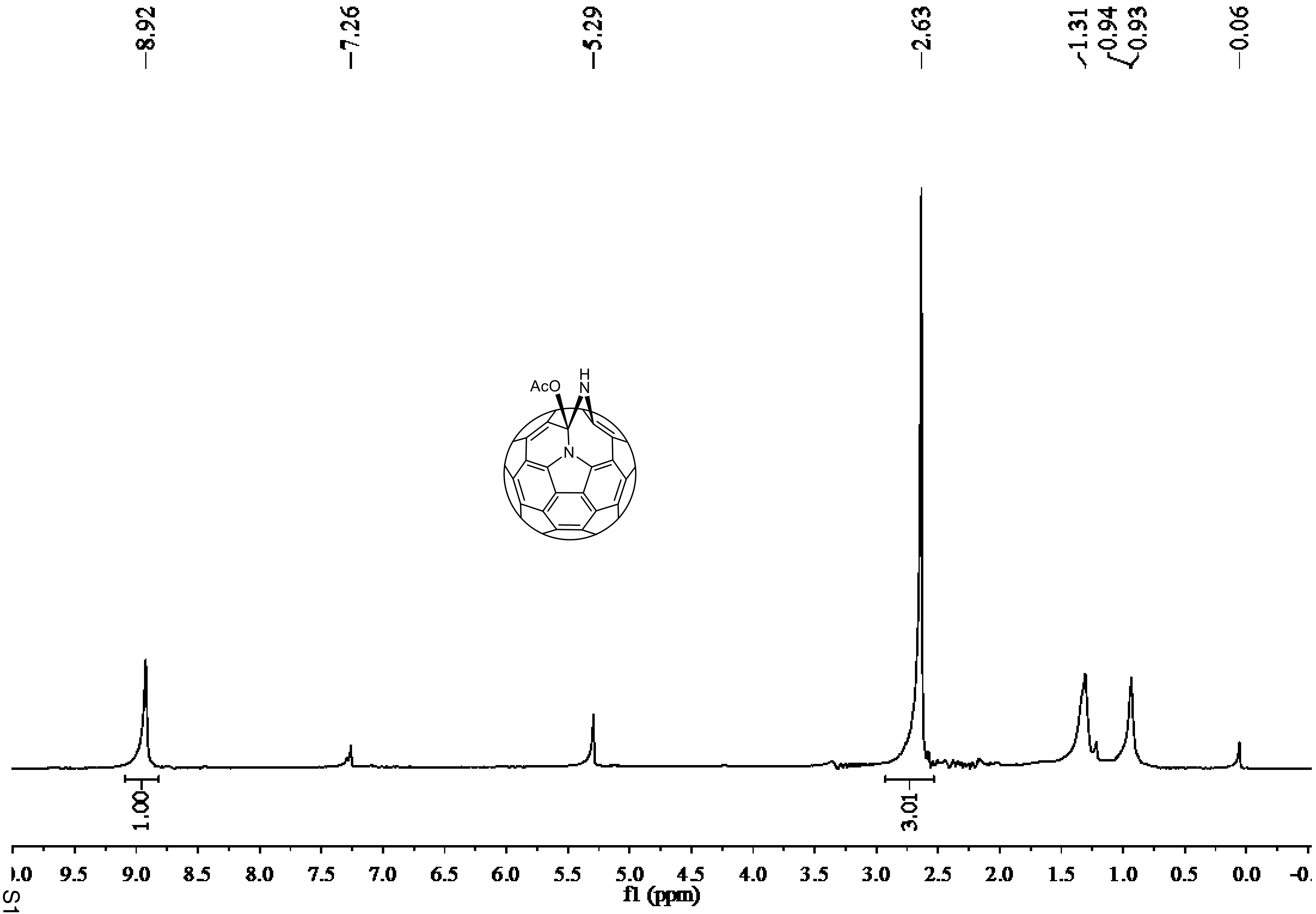
Preparation of Compound 16:

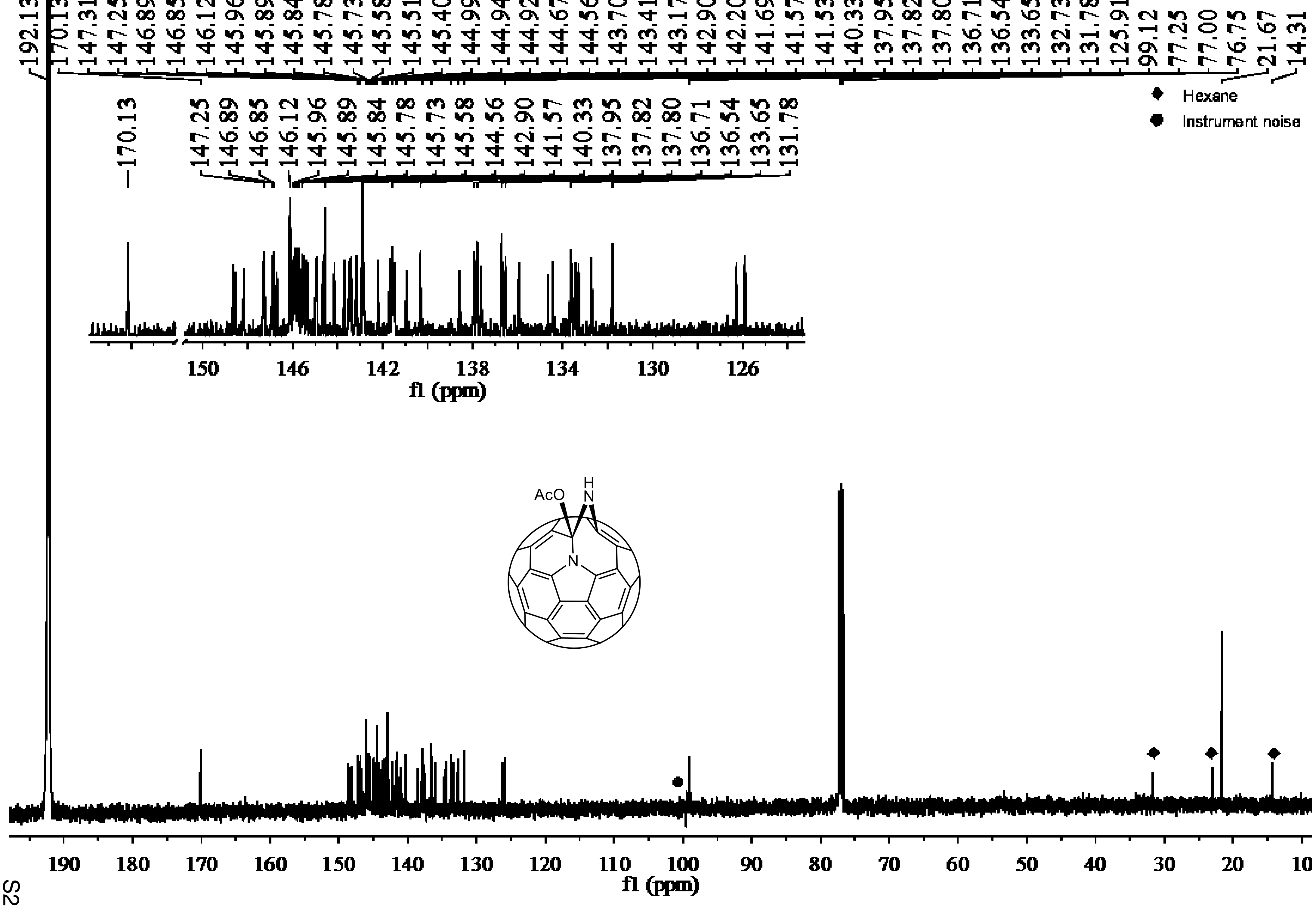


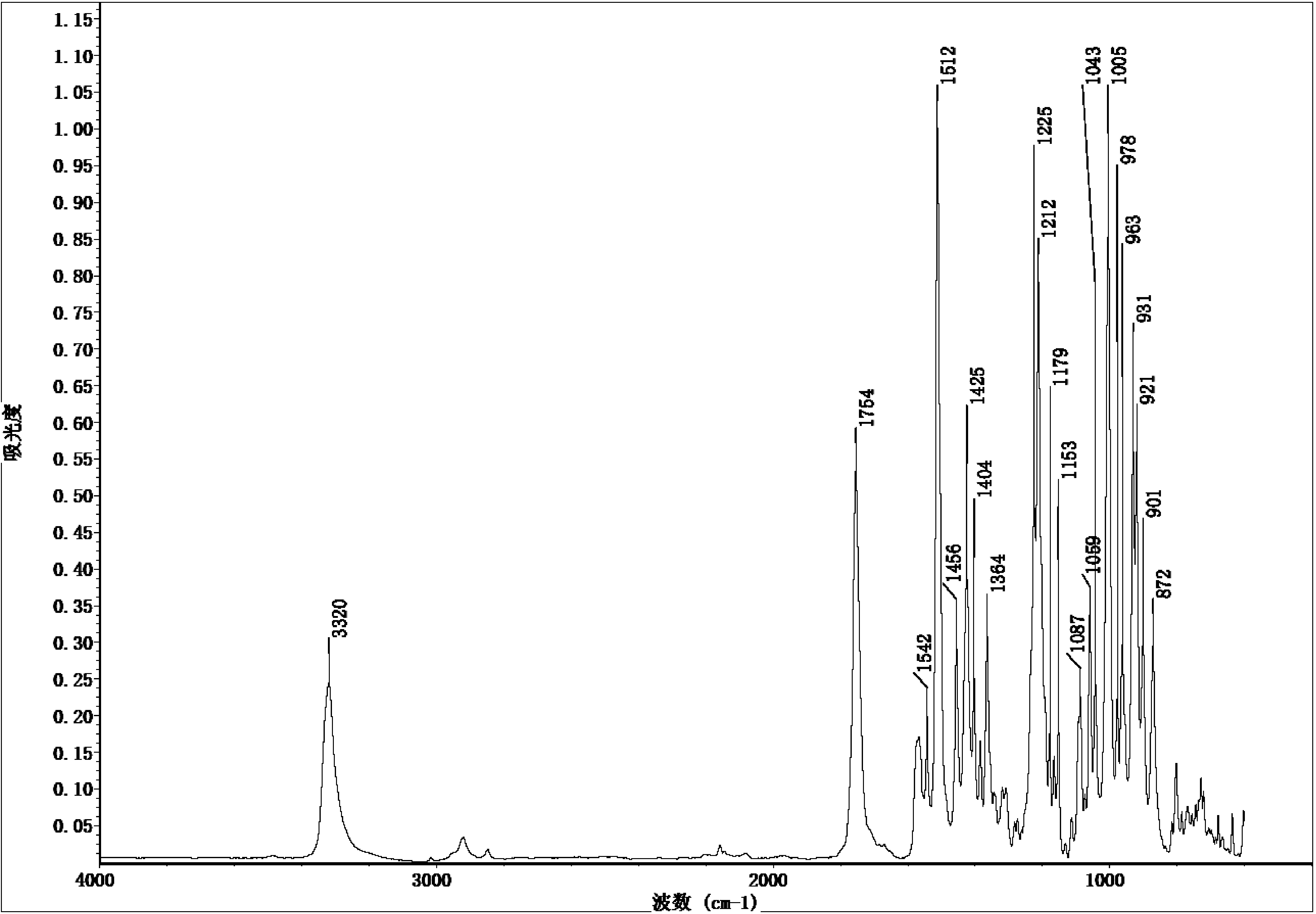
To a solution of the compound **8** and **9** (119 mg, ratio= 20:1, **8**: 0.101 mmol) in 24 mL freshly distilled toluene was added BBr_3 (0.14 mL). The mixture was stirred for about 10 minutes at room temperature followed by adding 24 mL toluene. Then the solution was quenched by water and the organic layer was mixed with PPh_3 (100 mg, 0.38 mmol). The solution was stirred at room temperature for 5 minutes and then *t*-butyl hydroperoxide (70%, 1.5 ml) was added. The resulting solution was stirred at room temperature for 5 minutes and then first purified by silica gel column chromatography eluting with toluene. The crude product was further purified by silica gel column chromatography eluting with CS_2 to give an inseparable mixture of compound **16** and **14** (9.8 mg, 0.012 mmol, 12%).

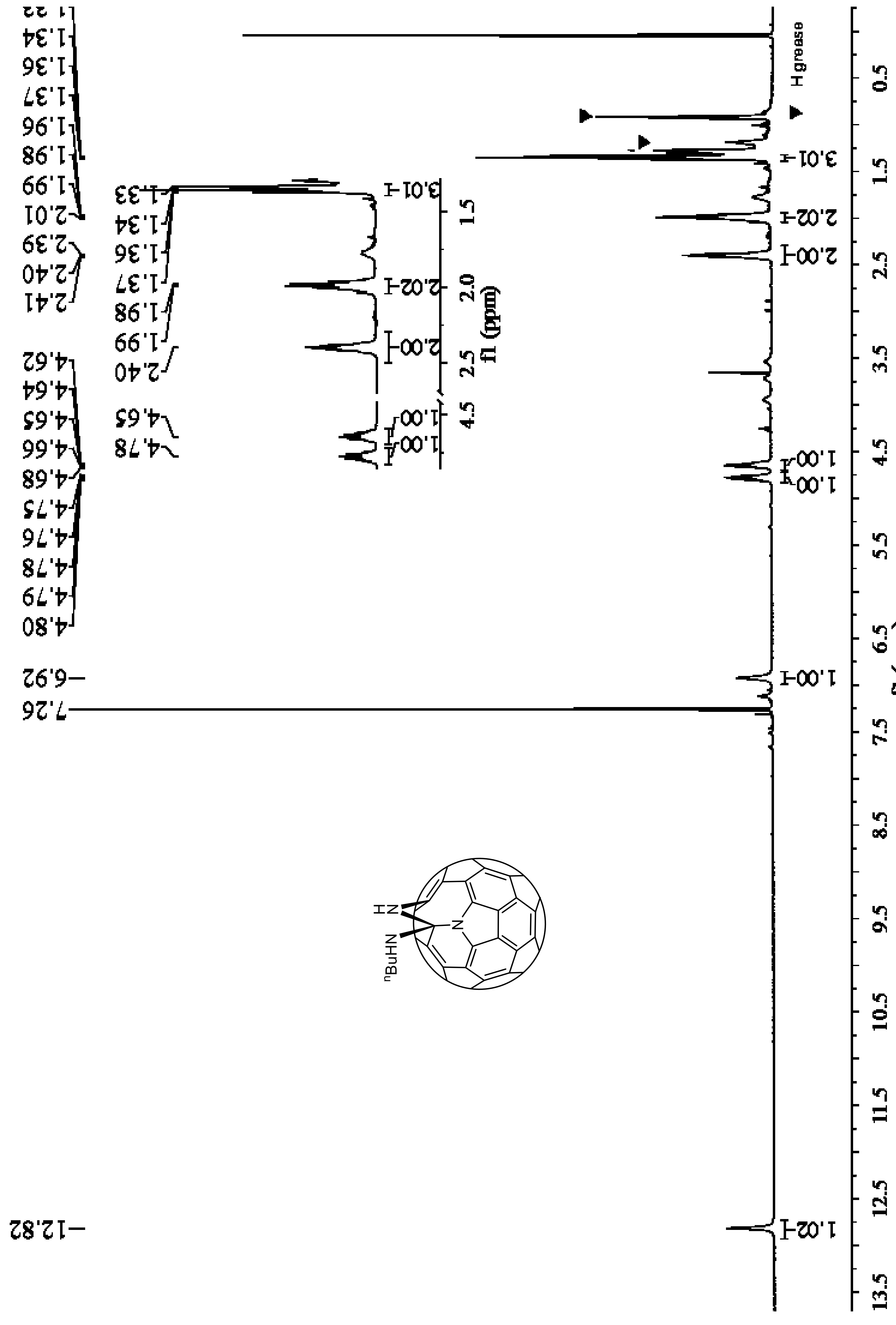
Characterization data for 16:

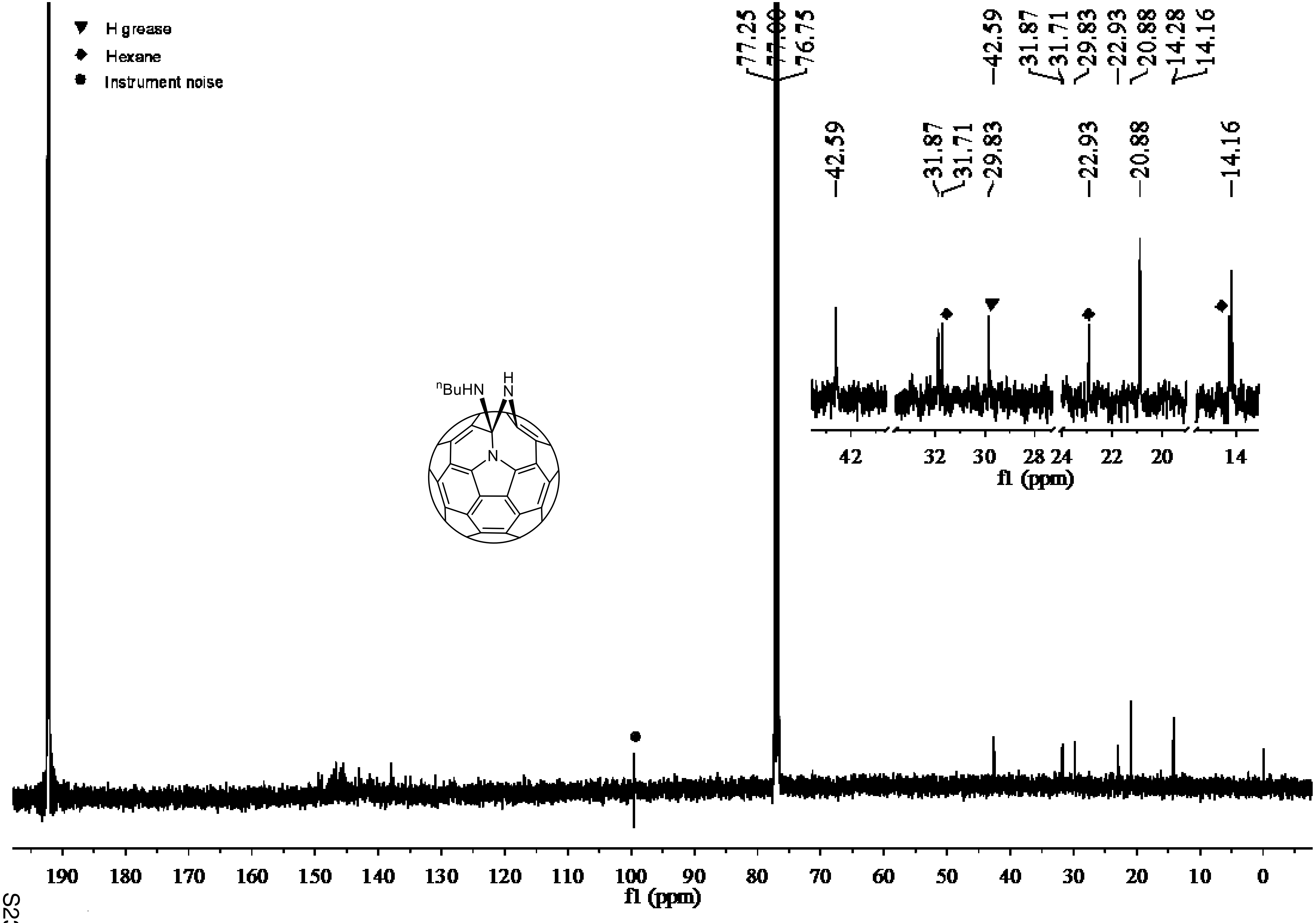
^{1}H NMR (500 MHz, $CS_2/CDCl_3$) δ 5.62 (q, J = 12.6 Hz, 1H), 1.67 (s, 9H).
^{13}C NMR (126 MHz, $CS_2/CDCl_3$) (all signals represent 1C except as noted) δ 148.48, 148.35, 148.01, 147.97, 147.92, 146.61 (2C), 146.50, 146.48, 145.82, 145.62 (2C), 145.56, 145.39, 145.33, 145.29, 145.28 (2C), 145.26, 145.02, 144.94, 144.90, 144.81, 144.35, 144.22, 144.16, 143.42, 143.18, 143.03, 142.84, 142.65, 142.59, 142.05, 142.01, 141.80 (2C), 141.54, 141.51, 141.48, 141.13, 140.74, 140.53, 139.32, 139.22, 139.09, 138.03, 137.92, 137.22, 135.79, 135.12, 134.26, 133.85, 133.48, 133.34, 133.29, 129.73, 127.60, 126.21, 94.50 (1C, sp^3), 81.46 ($\mathbf{C}(CH_3)_3$, 35.70 (1C), 26.86 (3C).
ESI-FT-ICR-HRMS-Positive $C_{64}H_{12}NO_2$ ($M + H^+$) calculated 826.0863, found 826.0855.
MALDI-TOF-MS $C_{60}H_2NO$ ($M - {}^tBuO^-$) calculated 752.0136, found 751.9977.

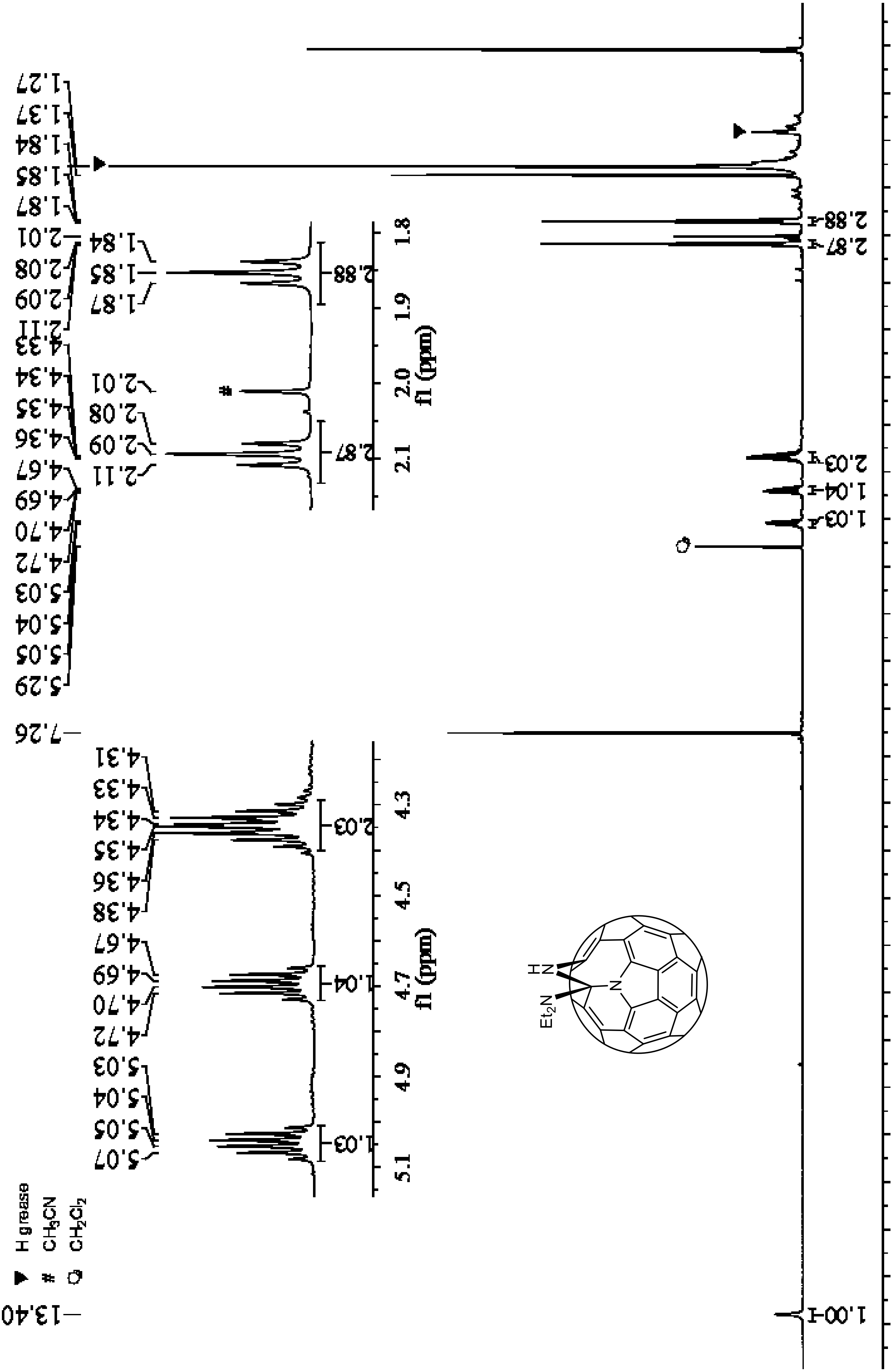

8.92
7.26
5.29
2.63
1.31
0.94
0.93
0.06
AcO
H
N
N
1.00
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

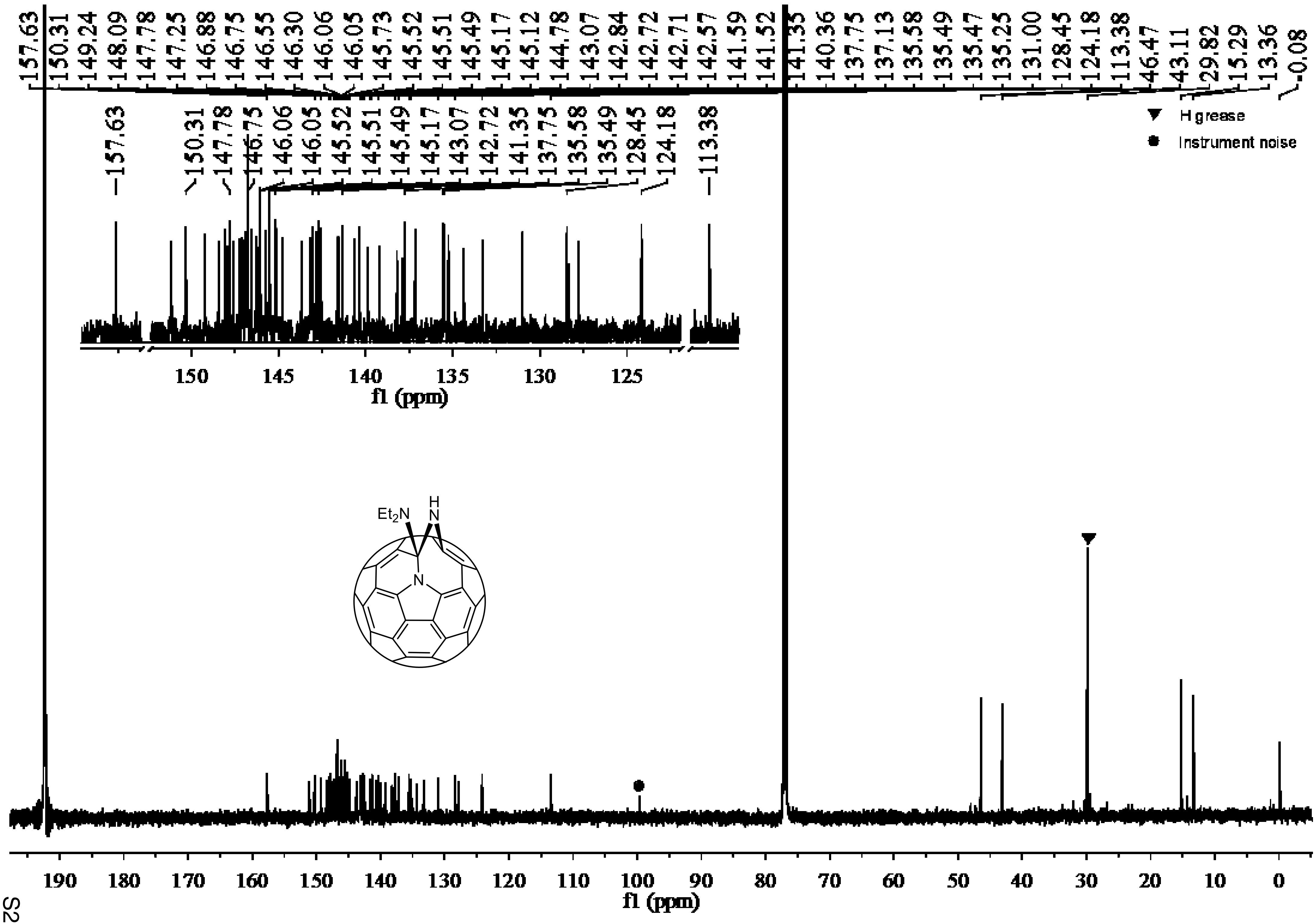

192.13
170.13
147.31
147.25
146.89
146.85
146.12
145.96
145.89
145.84
145.78
145.73
145.58
145.51
145.40
144.99
144.94
144.92
144.67
144.56
143.70
143.41
143.17
142.90
142.20
141.69
141.57
141.53
140.33
137.95
137.82
137.80
136.71
136.54
133.65
132.73
131.78
125.91
99.12
77.25
77.00
76.75
21.67
14.31
◆ Hexane
● Instrument noise
170.13
147.25
146.89
146.85
146.12
145.96
145.89
145.84
145.78
145.73
145.58
144.56
142.90
141.57
140.33
137.95
137.82
137.80
136.71
136.54
133.65
131.78
150
146
142
138
134
130
126
f1 (ppm)
AcO
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

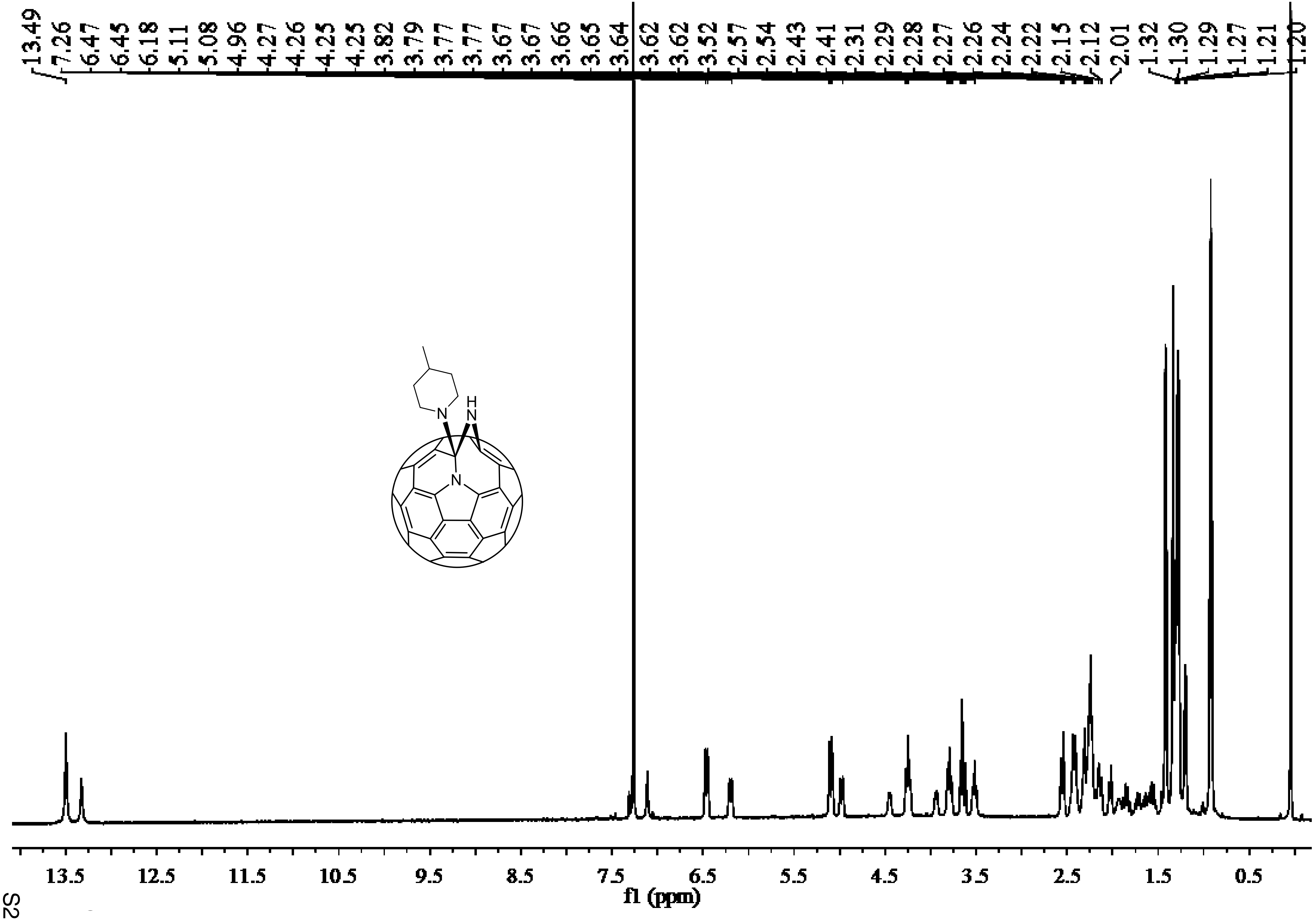

吸光度
1.15
1.10
1.05
1.00
0.95
0.90
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
4000
3000
2000
1000
波数 (cm-1)
3320
1754
1542
1512
1456
1425
1404
1364
1225
1212
1179
1153
1087
1059
1043
1005
978
963
931
921
901
872

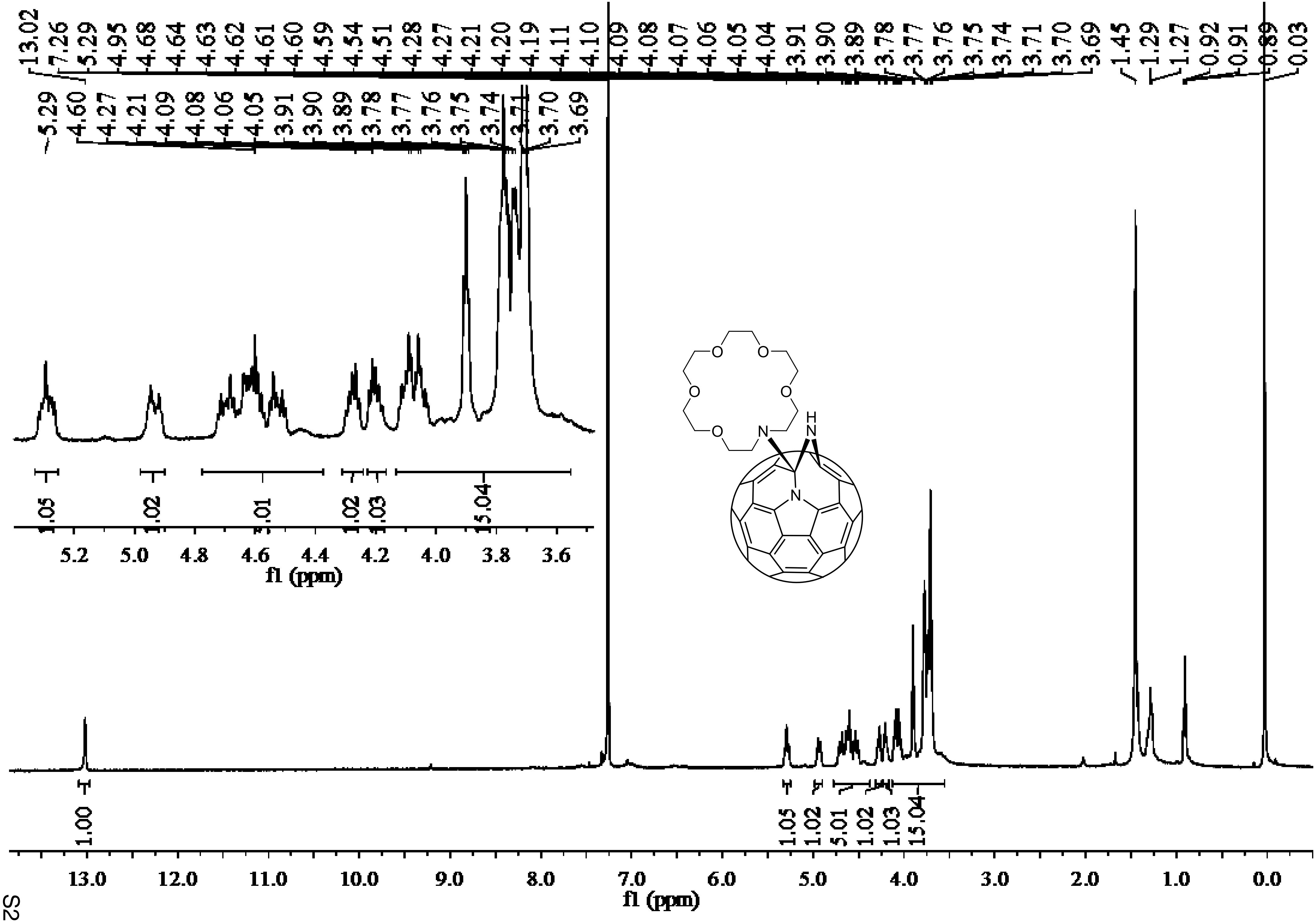
1.33
1.34
1.36
1.37
1.96
1.98
1.99
2.01
2.39
2.40
2.41
4.62
4.64
4.65
4.66
4.68
4.75
4.76
4.78
4.79
4.80
6.92
7.26
12.82
1.33
1.34
1.36
1.37
1.98
1.99
2.40
4.65
4.78
3.01
2.02
2.00
1.00
1.00
1.5
2.0
2.5
4.5
f1 (ppm)
Hgrease
3.01
2.02
2.00
1.00
1.00
1.00
1.02
0.5
1.5
2.5
3.5
4.5
5.5
6.5
7.5
8.5
9.5
10.5
11.5
12.5
13.5
f1 (ppm)
ⁿBuHN
NH
N


H grease
Hexane
Instrument noise
77.25
77.00
76.75
42.59
31.87
31.71
29.83
22.93
20.88
14.28
14.16
42.59
31.87
31.71
29.83
22.93
20.88
14.16
ⁿBuHN
H
N
N
42
32
30
28
24
22
20
14
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


13.40
▼ H grease
CH₃CN
○ CH₂Cl₂
7.26
5.29
5.05
5.04
5.03
4.72
4.70
4.69
4.67
4.36
4.35
4.34
4.33
2.11
2.09
2.08
2.01
1.87
1.85
1.84
1.37
1.27
5.07
5.05
5.04
5.03
4.72
4.70
4.69
4.67
4.38
4.36
4.35
4.34
4.33
4.31
1.03
1.04
2.03
5.1
4.9
4.7
4.5
4.3
f1 (ppm)
2.11
2.09
2.08
2.01
1.87
1.85
1.84
2.87
2.88
2.1
2.0
1.9
1.8
f1 (ppm)
Et₂N
N
H
N
1.00
1.03
1.04
2.03
2.87
2.88
13.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

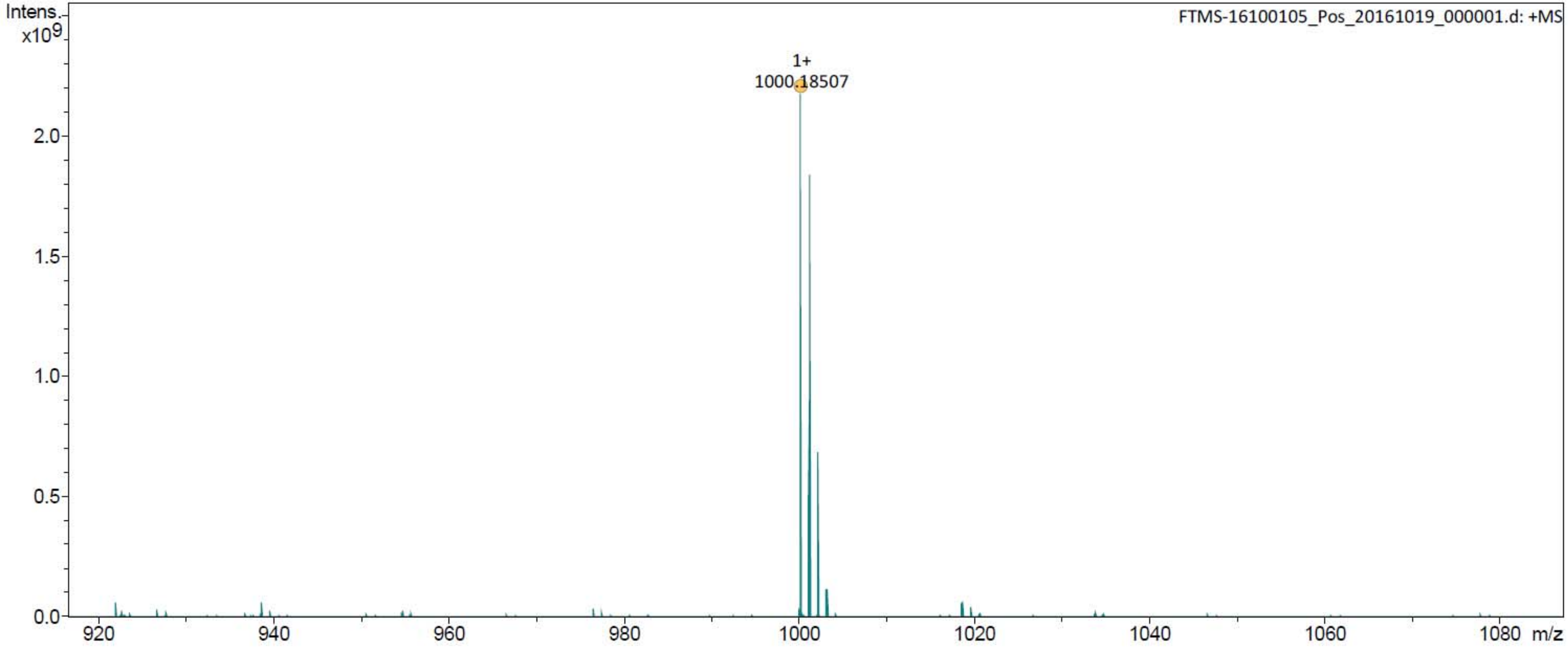

157.63
150.31
149.24
148.09
147.78
147.25
146.88
146.75
146.55
146.30
146.06
146.05
145.73
145.52
145.51
145.49
145.17
145.12
144.78
143.07
142.84
142.72
142.71
142.57
141.59
141.52
141.35
140.36
137.75
137.13
135.58
135.49
135.47
135.25
131.00
128.45
124.18
113.38
46.47
43.11
29.82
15.29
13.36
-0.08
H grease
Instrument noise
157.63
150.31
147.78
146.75
146.06
146.05
145.52
145.51
145.49
145.17
143.07
142.72
141.35
137.75
135.58
135.49
128.45
124.18
113.38
150
145
140
135
130
125
f1 (ppm)
Et$_2$N
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


13.49
7.26
6.47
6.45
6.18
5.11
5.08
4.96
4.27
4.26
4.25
4.25
3.82
3.79
3.77
3.77
3.67
3.67
3.66
3.65
3.64
3.62
3.62
3.52
2.57
2.54
2.43
2.41
2.31
2.29
2.28
2.27
2.26
2.24
2.22
2.15
2.12
2.01
1.32
1.30
1.29
1.27
1.21
1.20
N
H
N
N
13.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


13.02
7.26
5.29
4.95
4.68
4.64
4.63
4.62
4.61
4.60
4.59
4.54
4.51
4.28
4.27
4.21
4.20
4.19
4.11
4.10
4.09
4.08
4.07
4.06
4.05
4.04
3.91
3.90
3.89
3.78
3.77
3.76
3.75
3.74
3.71
3.70
3.69
1.45
1.29
1.27
0.92
0.91
0.89
0.03
5.29
4.60
4.27
4.21
4.09
4.08
4.06
4.05
3.91
3.90
3.89
3.78
3.77
3.76
3.75
3.74
3.71
3.70
3.69
1.05
1.02
5.01
1.02
1.03
15.04
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
f1 (ppm)
O
O
O
O
O
N
H
N
N
1.00
1.05
1.02
5.01
1.02
1.03
15.04
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

Analysis Info

Analysis Name	FTMS-16100105_Pos_20161019_000001.d	Acquisition Date	10/19/2016 2:25:40 PM
Sample	LYB-N-1	Instrument	Bruker Solarix XR FTMS
Comment		Operator	Peking University



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
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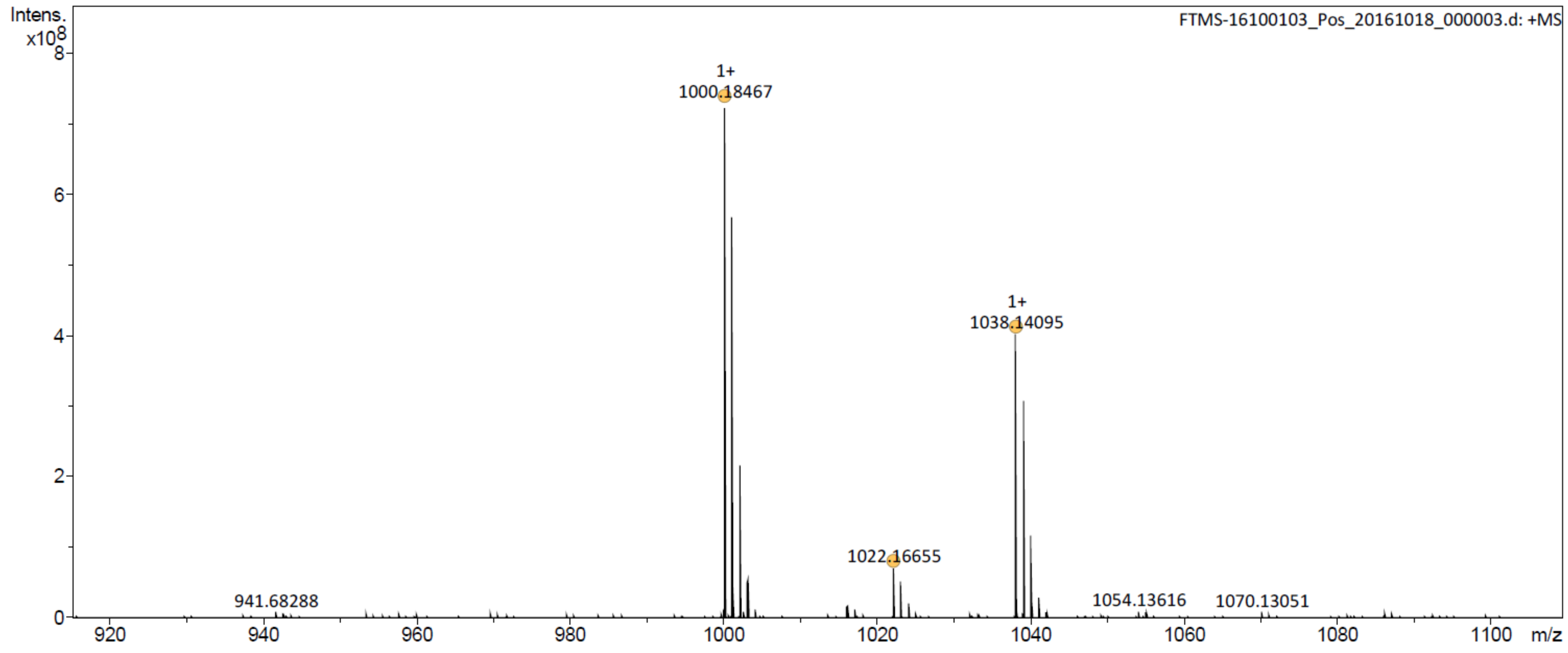
Analysis Info

Analysis Name	FTMS-16100103_Pos_20161018_000003.d	Acquisition Date	10/18/2016 4:55:44 PM
Sample	LYB-N-18-c-6	Instrument	Bruker Solarix XR FTMS
Comment		Operator	Peking University



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
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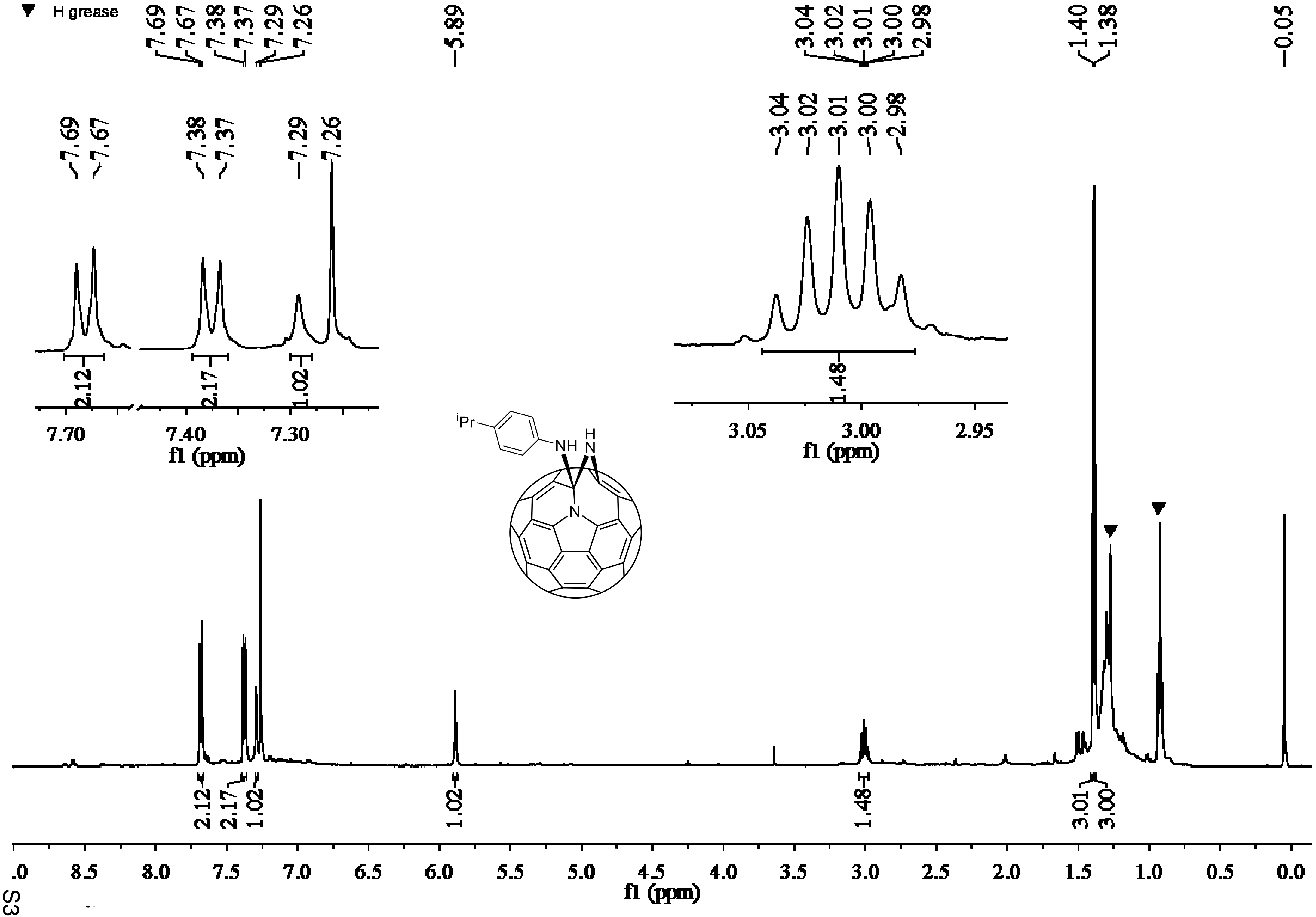
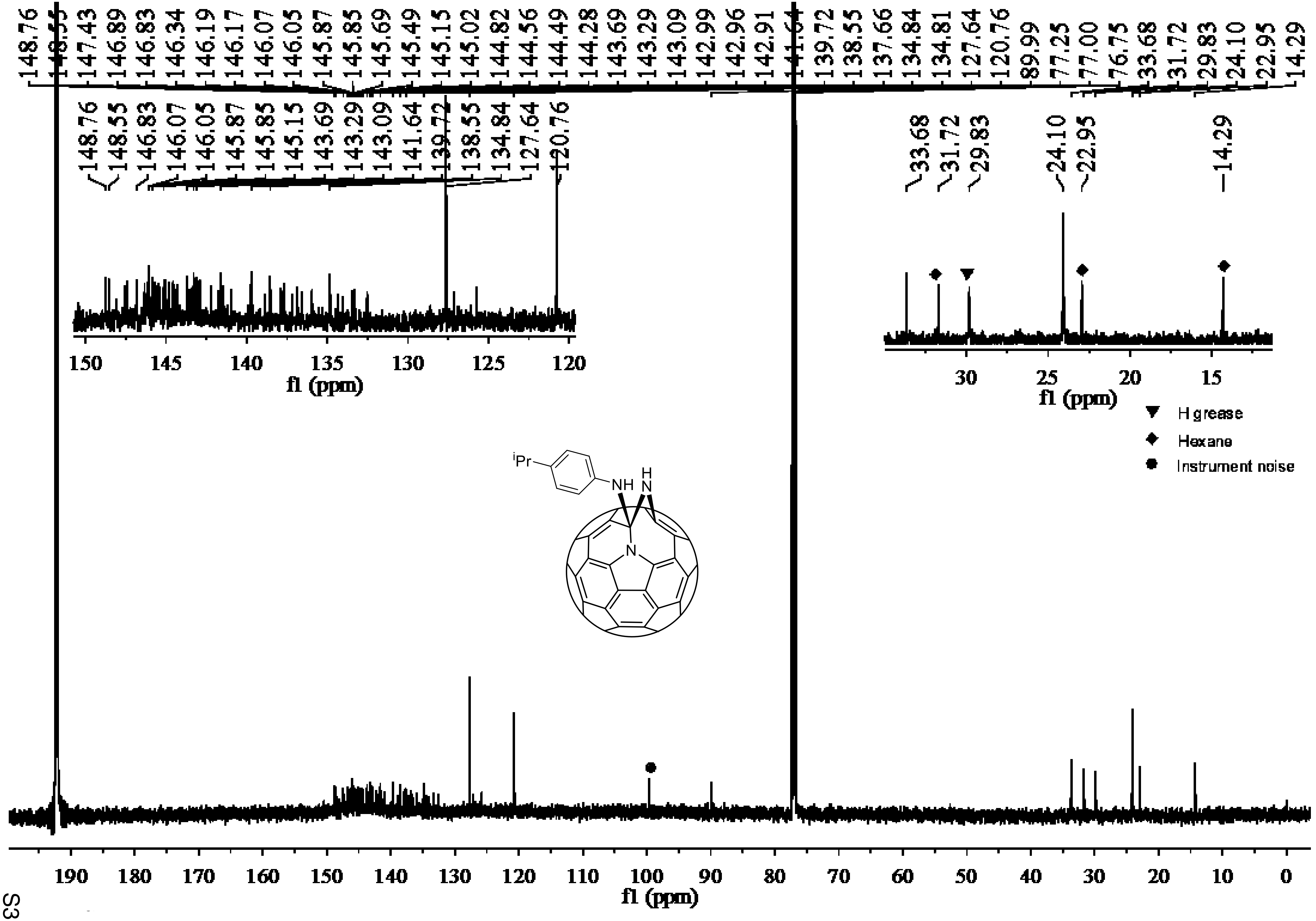
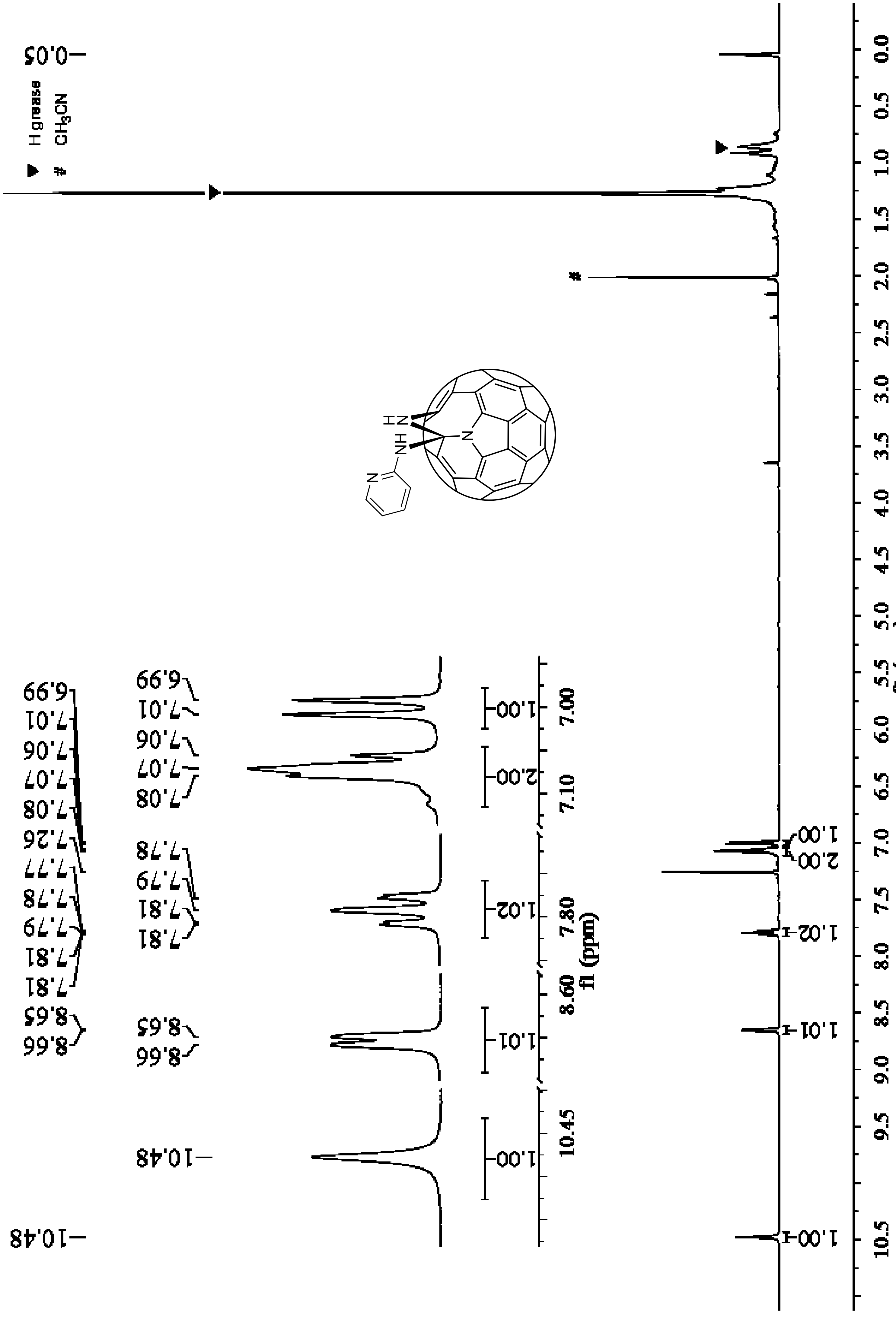
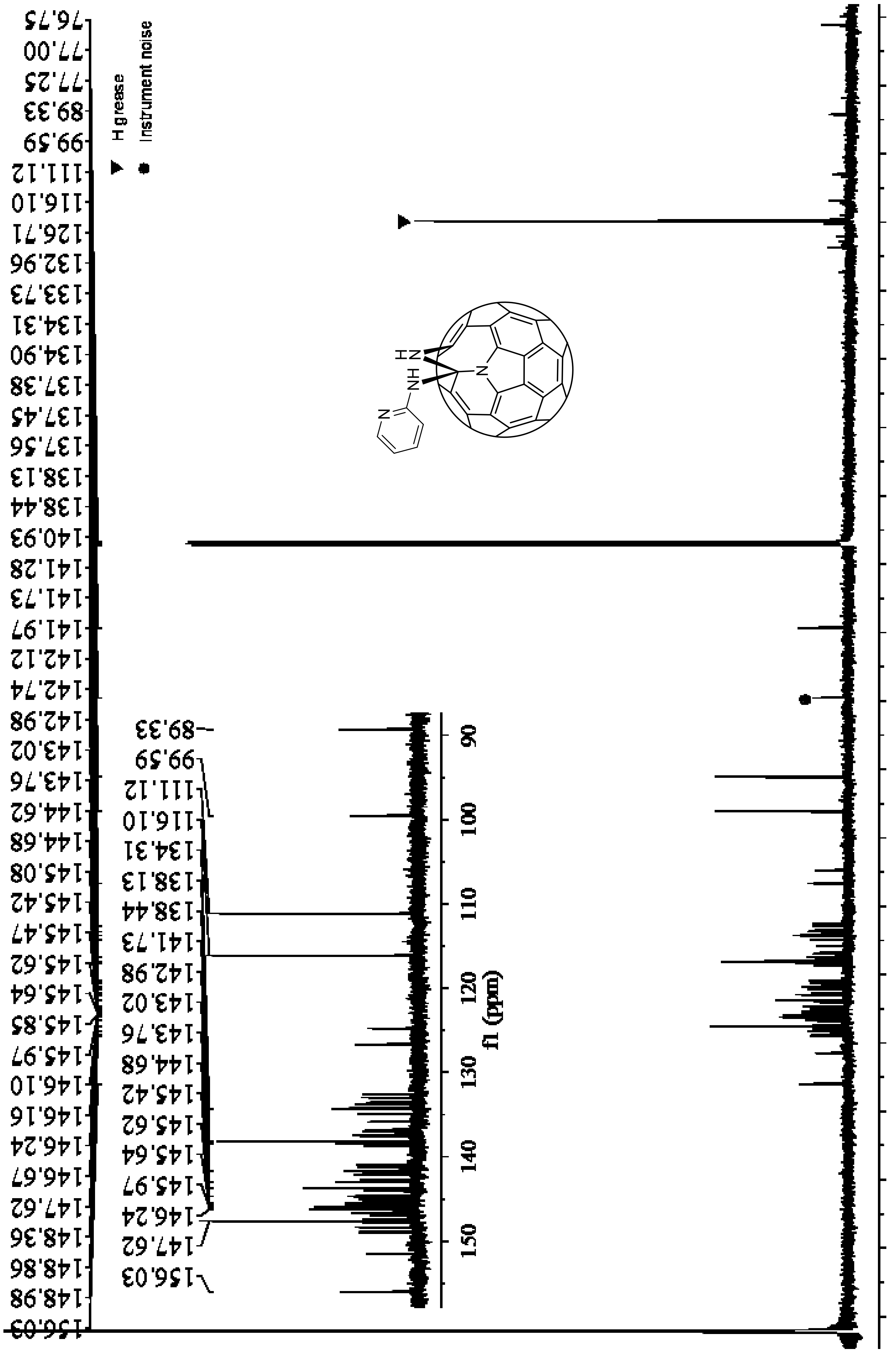
1022.166548	1	$C_{71}H_{25}N_3NaO_5$	100.00	1022.168642	2.0	1.3	21.8	61.0	even	ok
1038.140946	1	$C_{71}H_{25}KN_3O_5$	100.00	1038.142579	1.6	0.9	53.3	61.0	even	ok

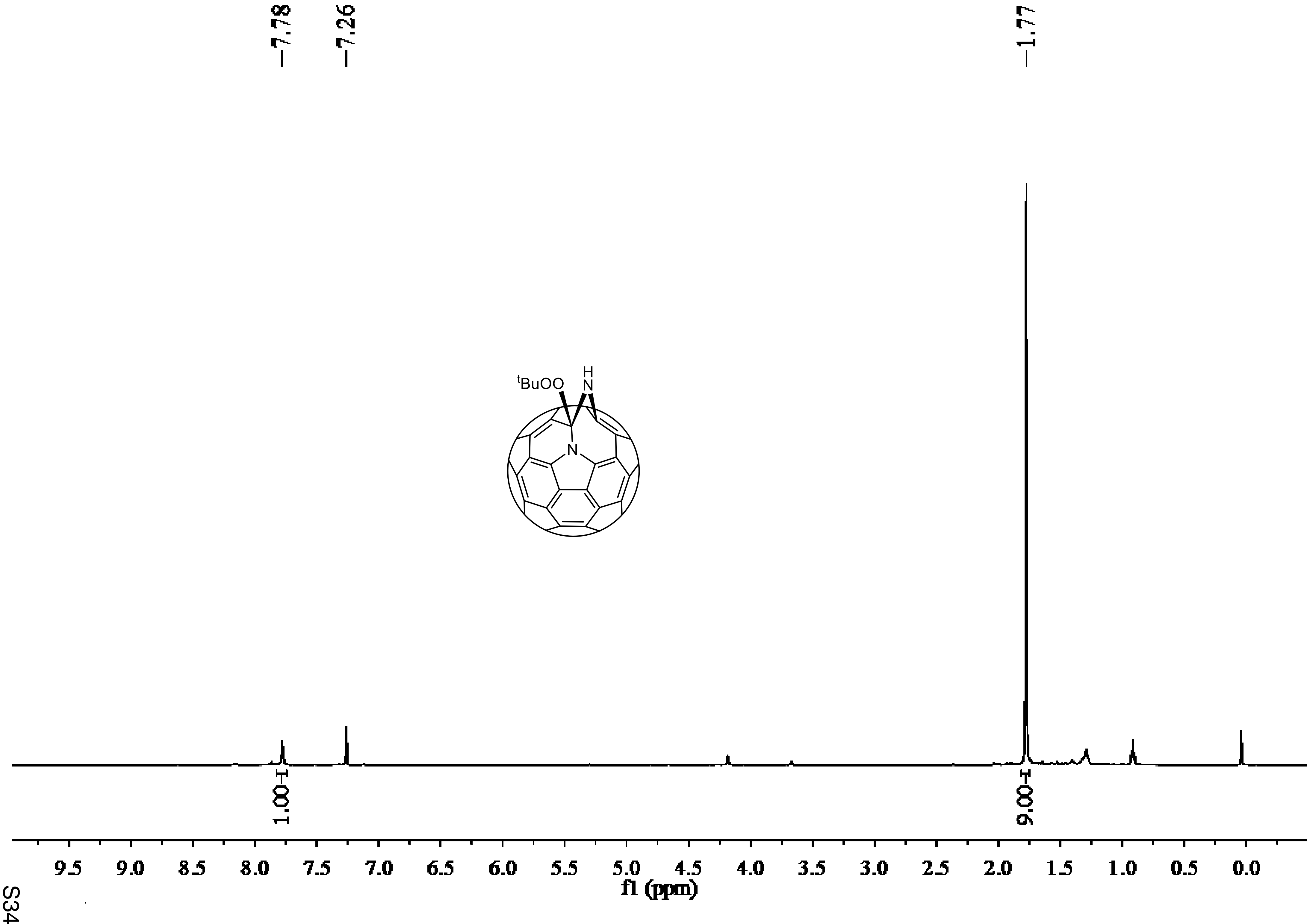

▼ H grease
7.69
7.67
7.38
7.37
7.29
7.26
5.89
3.04
3.02
3.01
3.00
2.98
1.40
1.38
0.05
2.12
2.17
1.02
1.48
3.01
3.00
7.70
7.40
7.30
3.05
3.00
2.95
fl (ppm)
iPr
NH
H
N
N
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

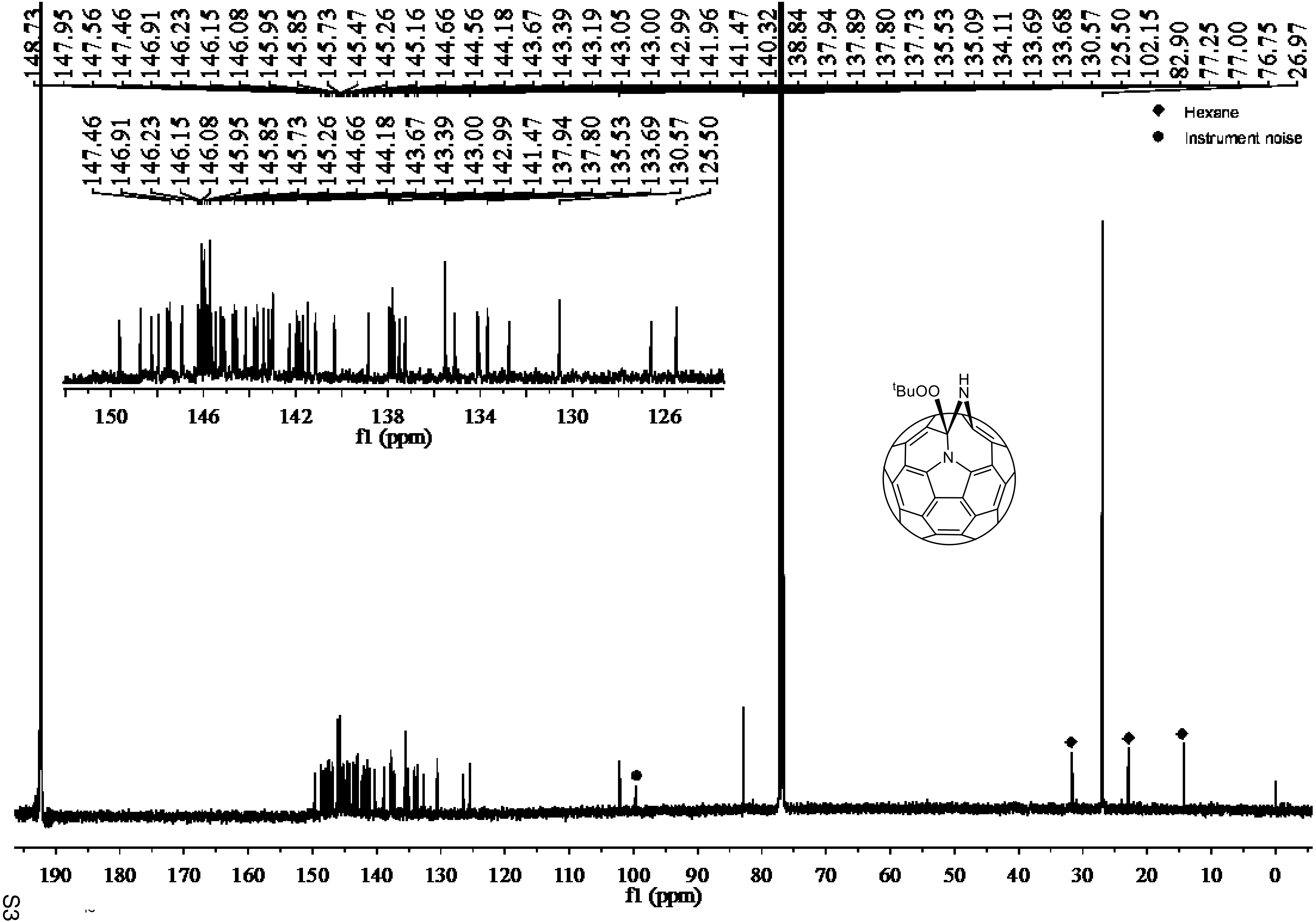

148.76
148.55
147.43
146.89
146.83
146.34
146.19
146.17
146.07
146.05
145.87
145.85
145.69
145.49
145.15
145.02
144.82
144.56
144.49
144.28
143.69
143.29
143.09
142.99
142.96
142.91
141.64
139.72
138.55
137.66
134.84
134.81
127.64
120.76
89.99
77.25
77.00
76.75
33.68
31.72
29.83
24.10
22.95
14.29
148.76
148.55
146.83
146.07
146.05
145.87
145.85
145.15
143.69
143.29
143.09
141.64
139.72
138.55
134.84
127.64
120.76
150
145
140
135
130
125
120
f1 (ppm)
33.68
31.72
29.83
24.10
22.95
14.29
30
25
20
15
f1 (ppm)
▼ H grease
◆ Hexane
● Instrument noise
ⁱPr
NH
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



▼ H grease
CH$_3$CN
0.05
6.99
7.01
7.06
7.07
7.08
7.26
7.77
7.78
7.79
7.81
7.81
8.65
8.66
10.48
NH
HN
N
N
1.00
2.00
1.02
1.01
1.00
7.00
7.10
7.80
8.60
10.45
f1 (ppm)
0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 8.0 8.5 9.0 9.5 10.5


76.75
77.00
77.25
89.33
99.59
111.12
116.10
126.71
132.96
133.73
134.31
134.90
137.38
137.45
137.56
138.13
138.44
140.93
141.28
141.73
141.97
142.12
142.74
142.98
143.02
143.76
144.62
144.68
145.08
145.42
145.47
145.62
145.64
145.85
145.97
146.10
146.16
146.24
146.67
147.62
148.36
148.86
148.98
156.03
H grease
Instrument noise
NH
HN
N
N
89.33
99.59
111.12
116.10
134.31
138.13
138.44
141.73
142.98
143.02
143.76
144.68
145.42
145.62
145.64
145.97
146.24
147.62
156.03
90
100
110
120
130
140
150
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)


7.78
7.26
1.77
tBuOO
N
H
N
1.00
9.00
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)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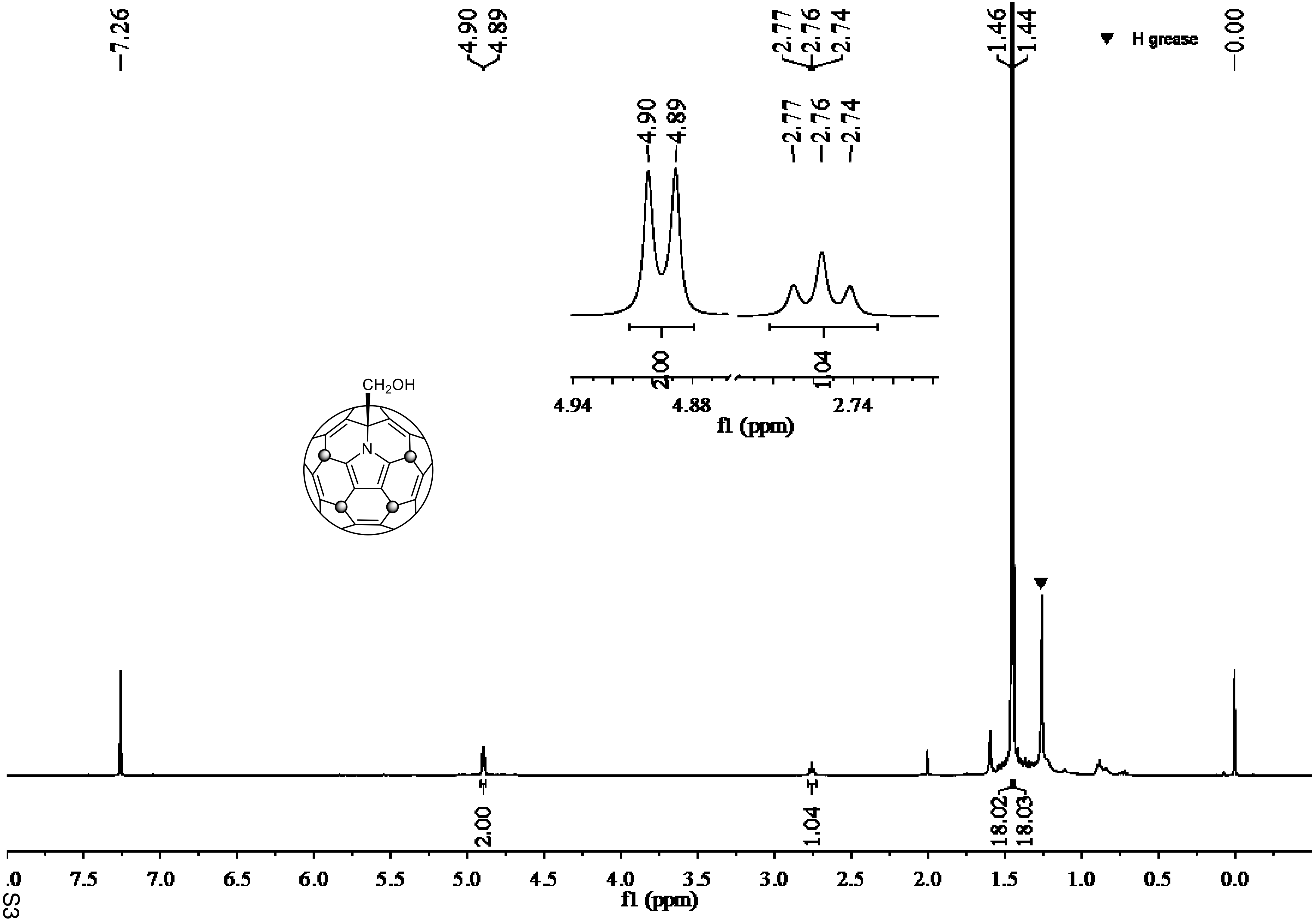
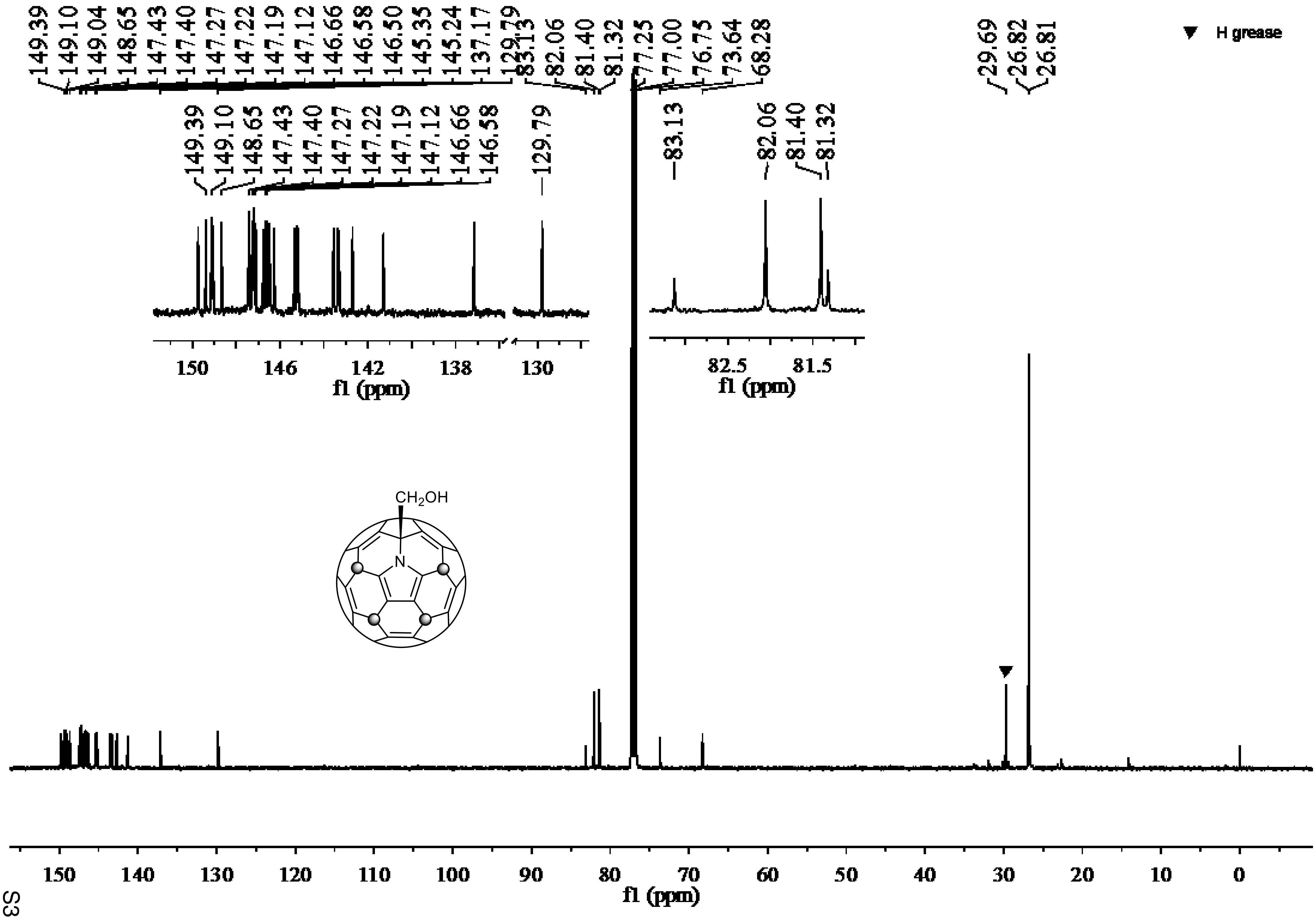
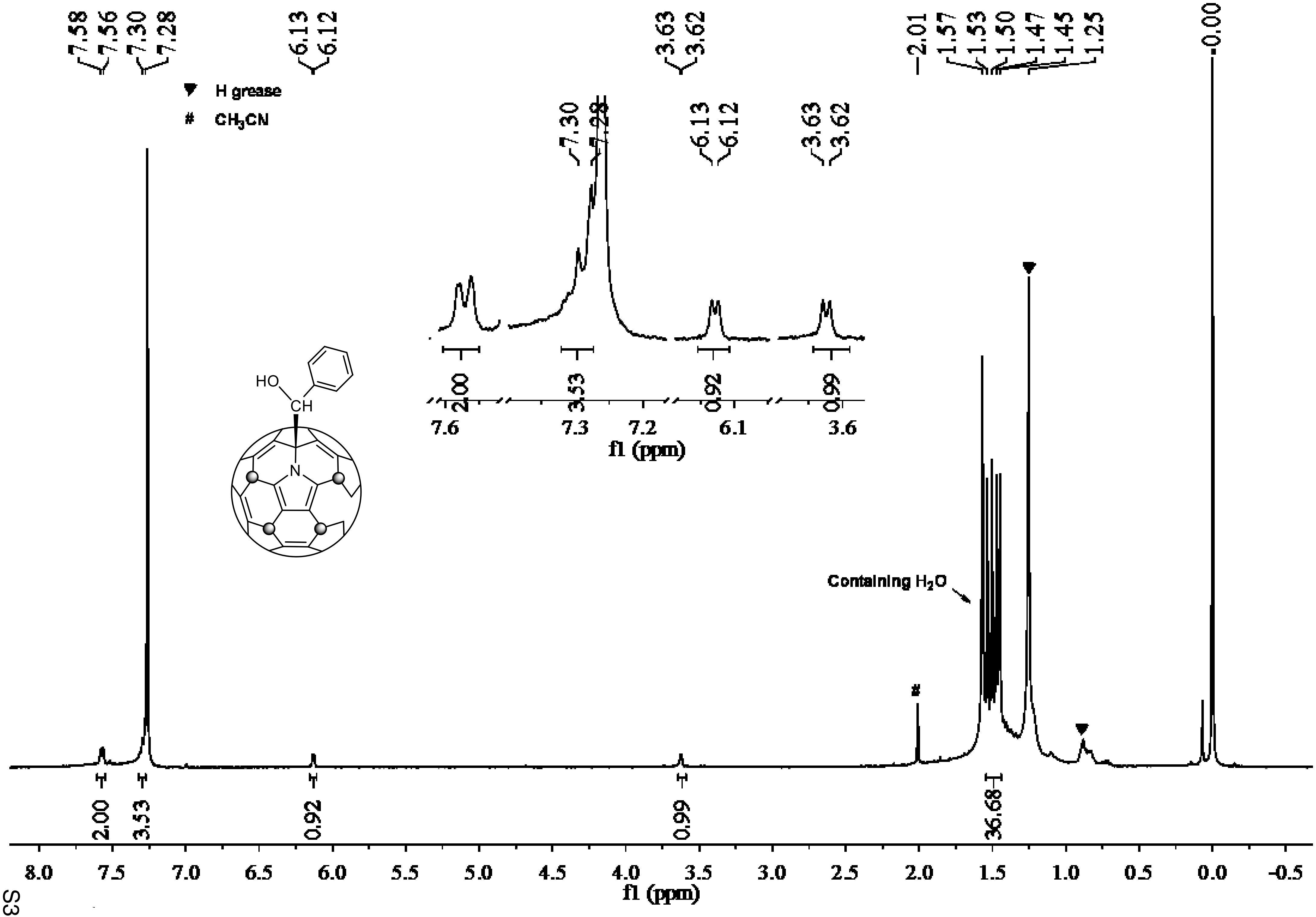
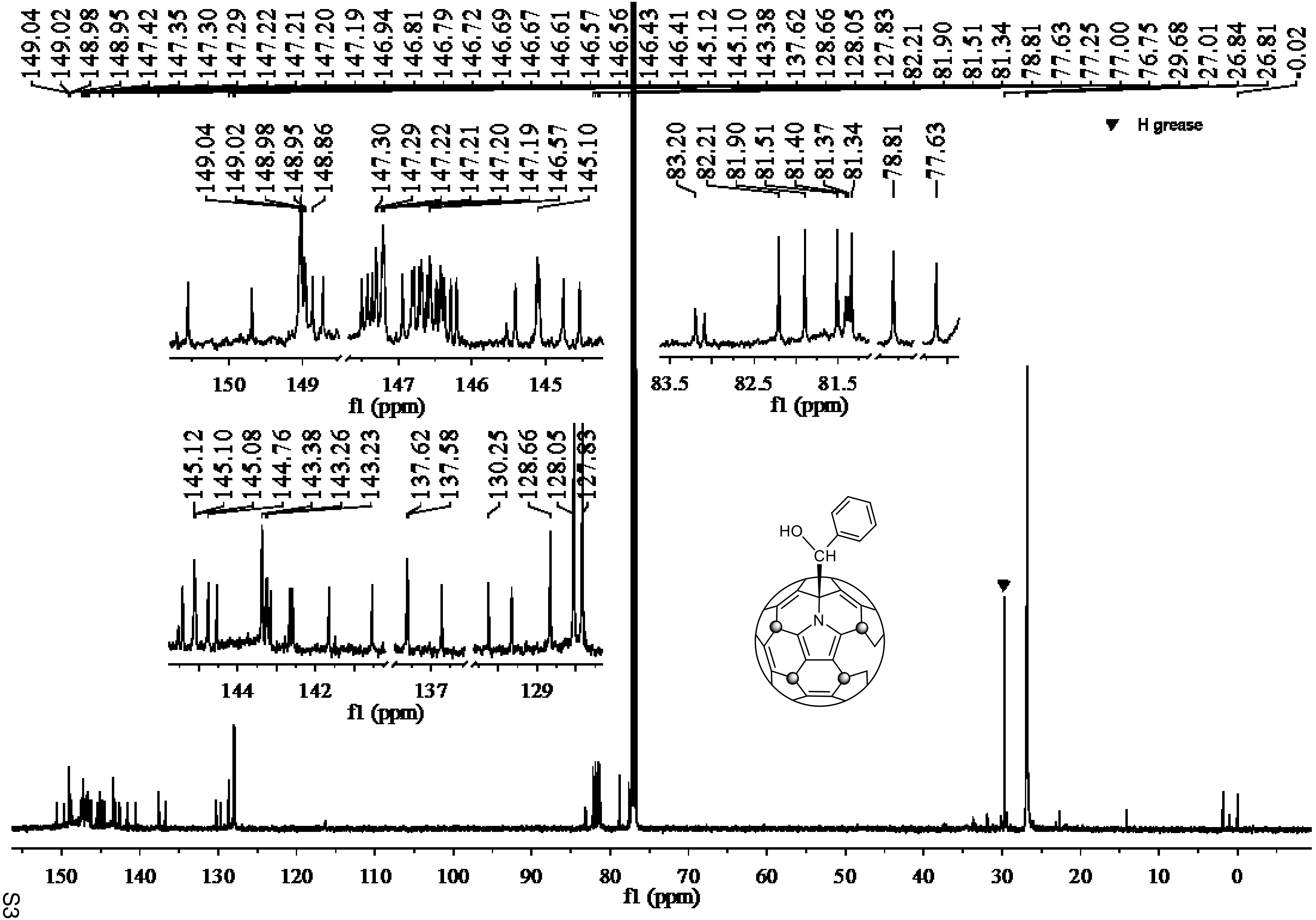
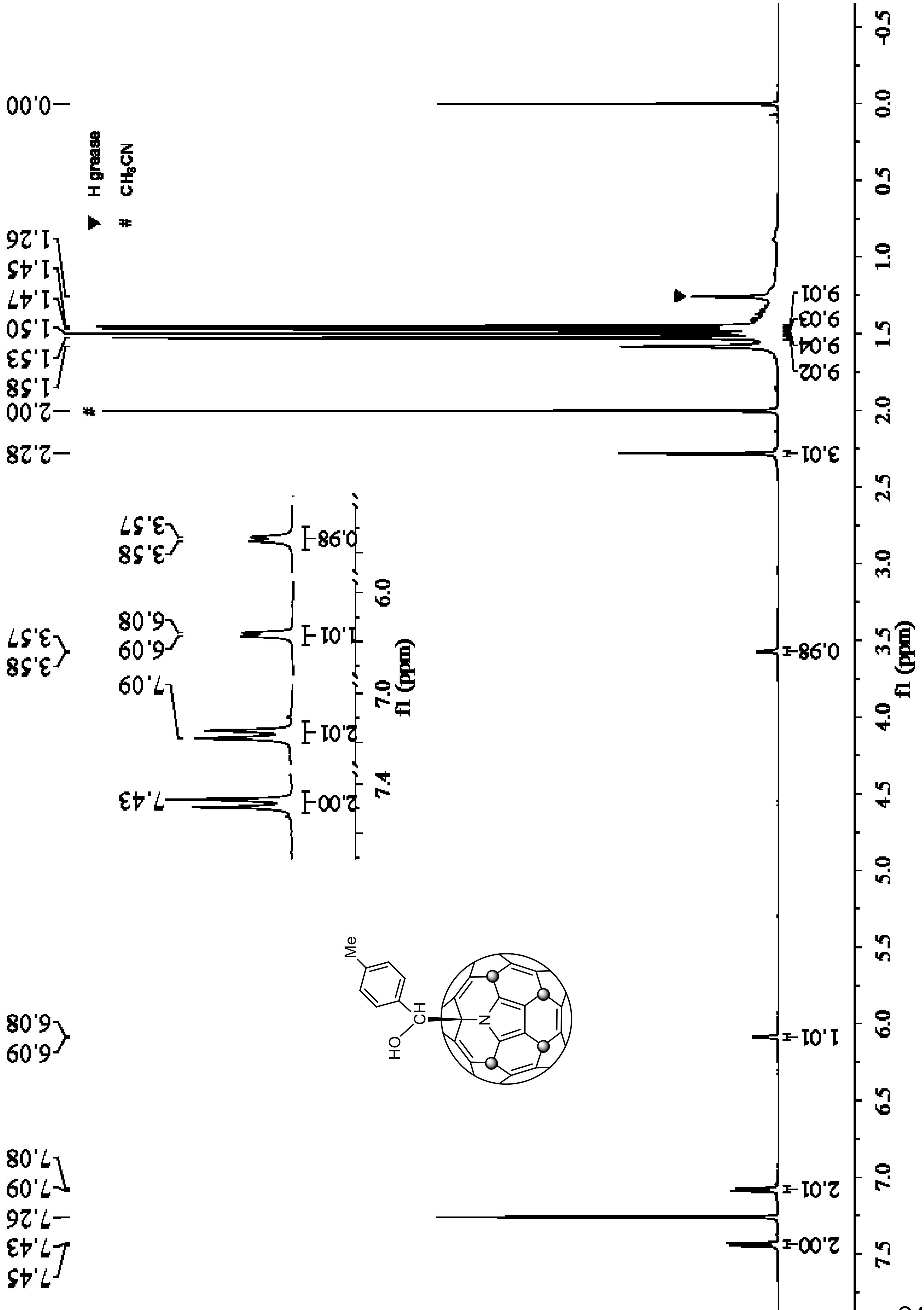
147.95
147.56
147.46
146.91
146.23
146.15
146.08
145.95
145.85
145.73
145.47
145.26
145.16
144.66
144.56
144.18
143.67
143.39
143.19
143.05
143.00
142.99
141.96
141.47
140.32
138.84
137.94
137.89
137.80
137.73
135.53
135.09
134.11
133.69
133.68
130.57
125.50
102.15
82.90
77.25
77.00
76.75
26.97
147.46
146.91
146.23
146.15
146.08
145.95
145.85
145.73
145.26
144.66
144.18
143.67
143.39
143.00
142.99
141.47
137.94
137.80
135.53
133.69
130.57
125.50
◆ Hexane
● Instrument noise
150
146
142
138
134
130
126
f1 (ppm)
ᵗBuOO
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.26
4.90
4.89
2.77
2.76
2.74
1.46
1.44
▼ H grease
0.00
CH$_2$OH
N
2.00
1.04
18.02
18.03
4.94
4.88
2.74
fl (ppm)
.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

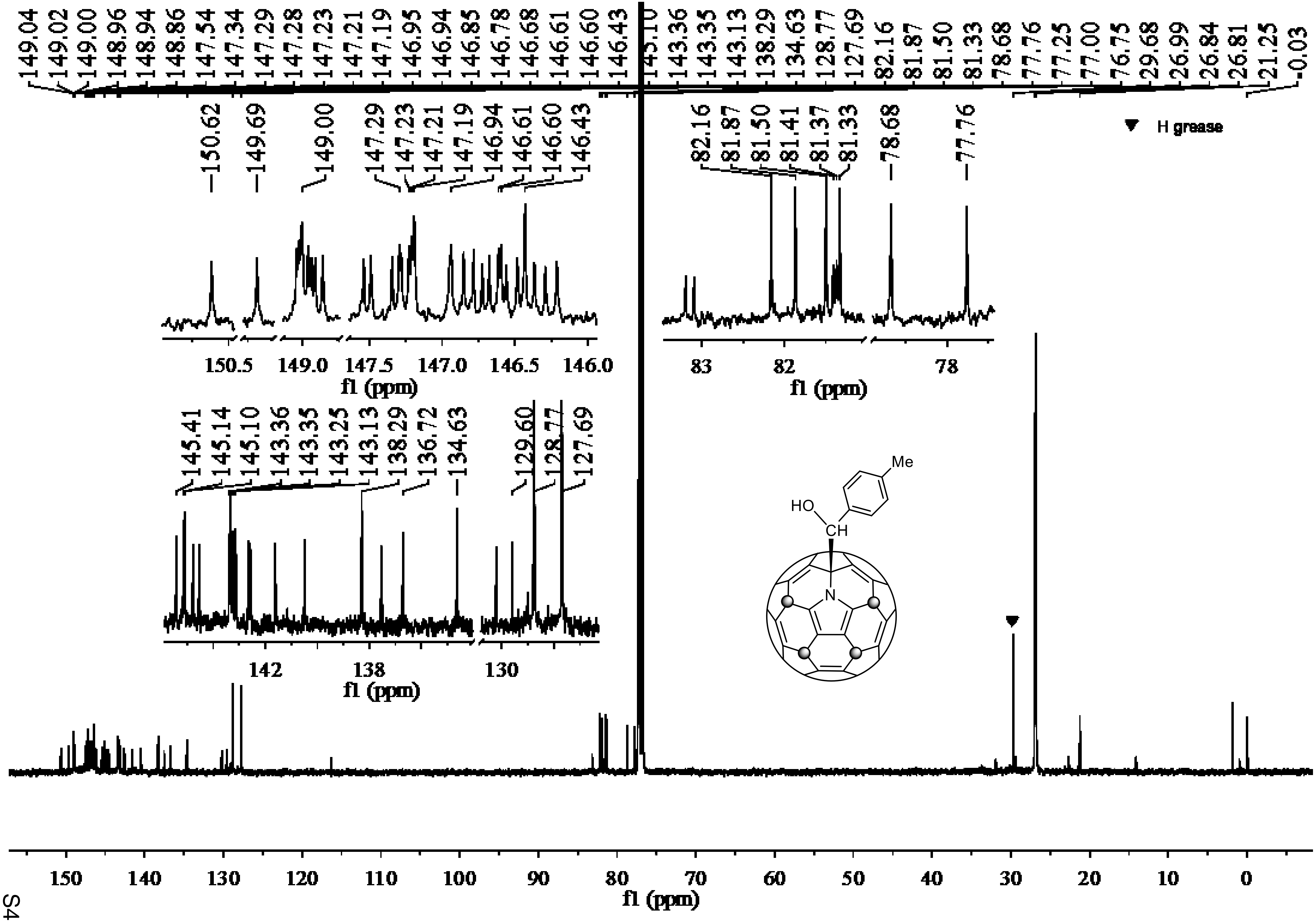

149.39
149.10
149.04
148.65
147.43
147.40
147.27
147.22
147.19
147.12
146.66
146.58
146.50
145.35
145.24
137.17
129.79
83.13
82.06
81.40
81.32
77.25
77.00
76.75
73.64
68.28
29.69
26.82
26.81
H grease
149.39
149.10
148.65
147.43
147.40
147.27
147.22
147.19
147.12
146.66
146.58
129.79
150
146
142
138
130
f1 (ppm)
83.13
82.06
81.40
81.32
82.5
81.5
f1 (ppm)
CH_2OH
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

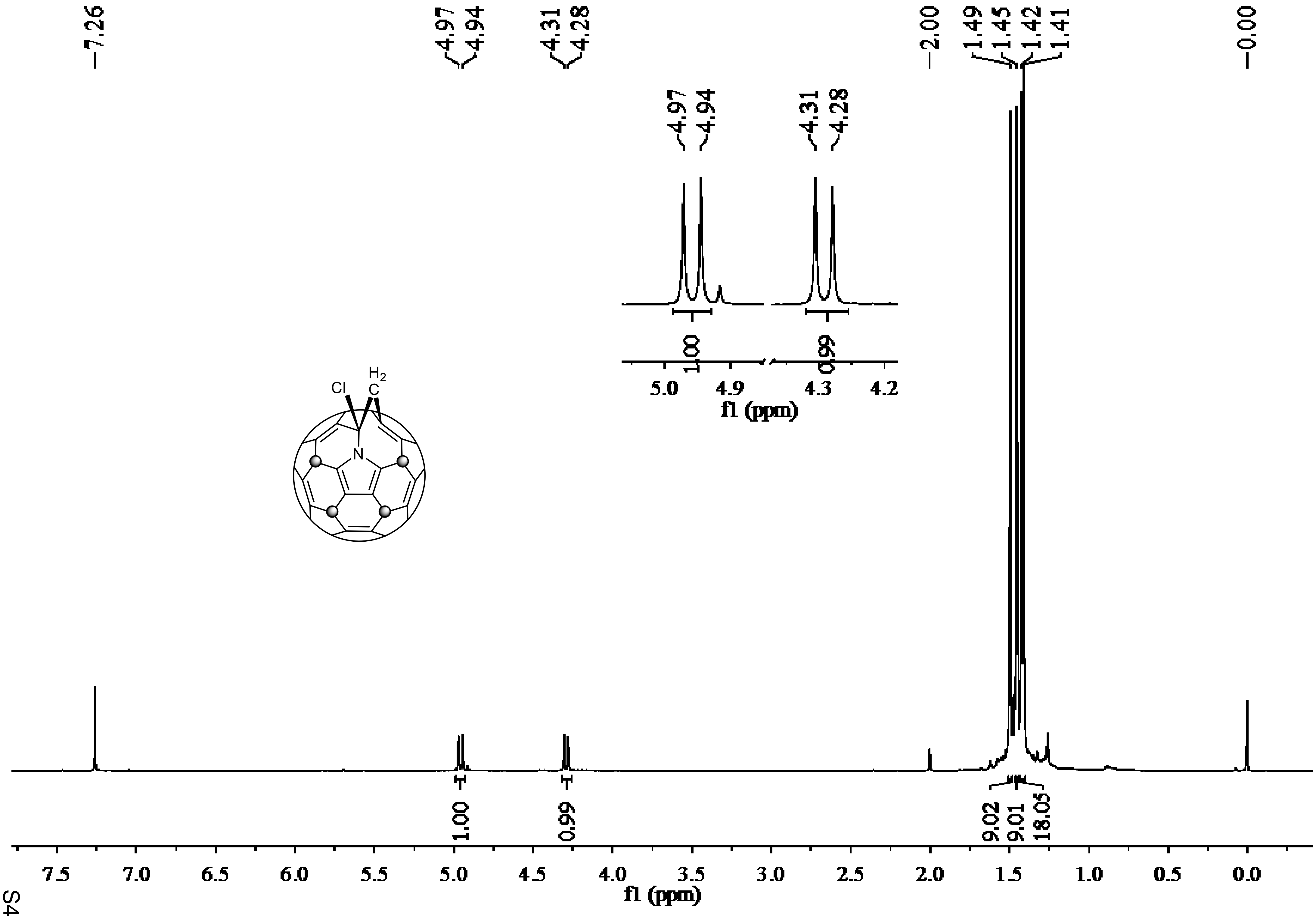

7.58
7.56
7.30
7.28
6.13
6.12
3.63
3.62
2.01
1.57
1.53
1.50
1.47
1.45
1.25
-0.00
▼ H grease
CH$_3$CN
HO
CH
N
7.30
7.28
6.13
6.12
3.63
3.62
2.00
3.53
0.92
0.99
7.6
7.3
7.2
6.1
3.6
f1 (ppm)
Containing H$_2$O
#
▼
2.00
3.53
0.92
0.99
36.68
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


149.04
149.02
148.98
148.95
147.42
147.35
147.30
147.29
147.22
147.21
147.20
147.19
146.94
146.81
146.79
146.72
146.69
146.67
146.61
146.57
146.56
146.43
146.41
145.12
145.10
143.38
137.62
128.66
128.05
127.83
82.21
81.90
81.51
81.34
78.81
77.63
77.25
77.00
76.75
29.68
27.01
26.84
26.81
-0.02
▼ H grease
149.04
149.02
148.98
148.95
148.86
147.30
147.29
147.22
147.21
147.20
147.19
146.57
145.10
150
149
147
146
145
f1 (ppm)
83.20
82.21
81.90
81.51
81.40
81.37
81.34
78.81
77.63
83.5
82.5
81.5
f1 (ppm)
145.12
145.10
145.08
144.76
143.38
143.26
143.23
137.62
137.58
130.25
128.66
128.05
127.83
144
142
137
129
f1 (ppm)
HO
CH
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

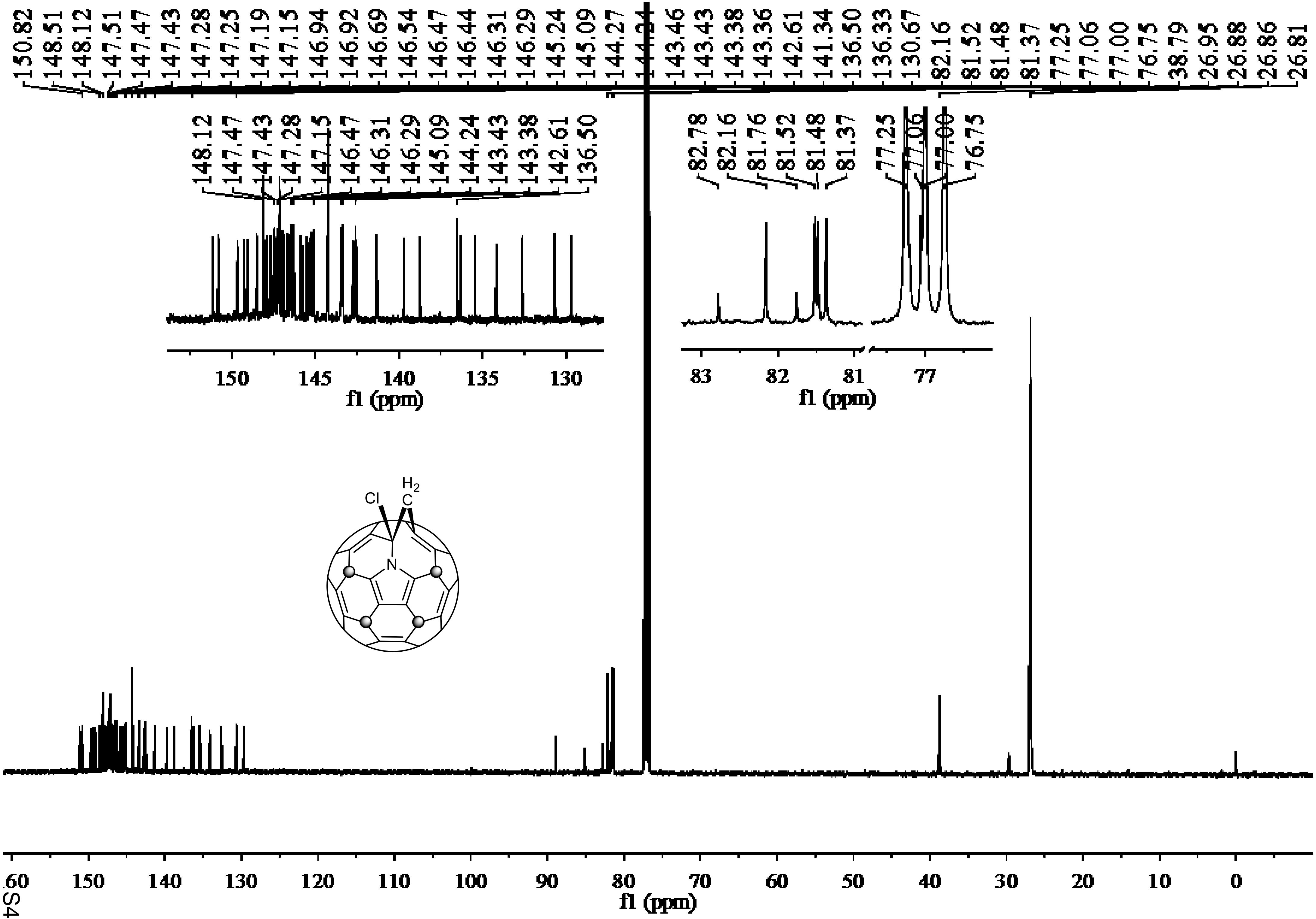


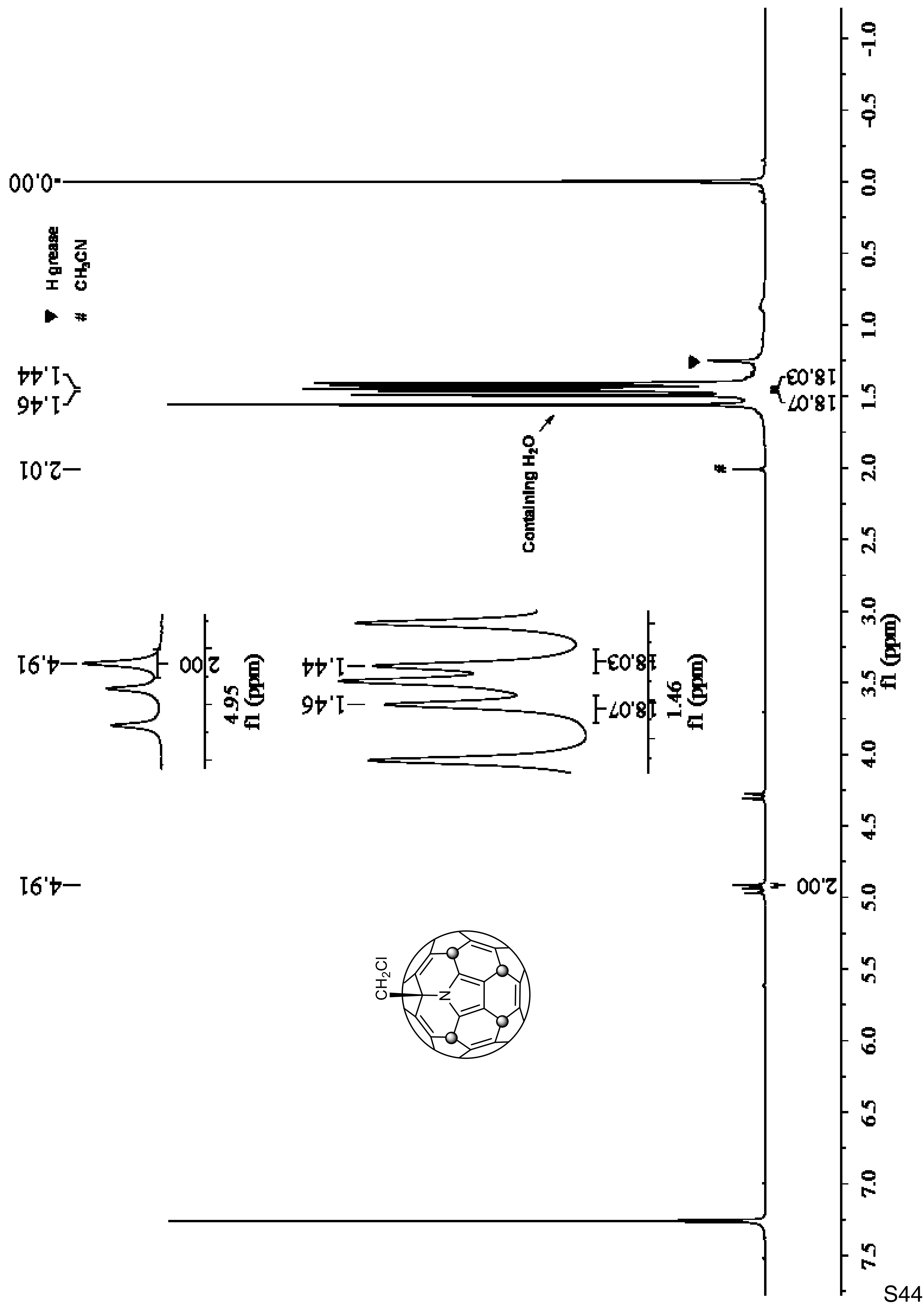
0.00
1.26
1.45
1.47
1.50
1.53
1.58
2.00
2.28
3.57
3.58
6.08
6.09
7.08
7.09
7.26
7.43
7.45
H grease
CH$_3$CN
9.01
9.03
9.04
9.02
3.01
0.98
1.01
2.01
2.00
7.4
7.0
6.0
f1 (ppm)
Me
HO
CH
N
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5

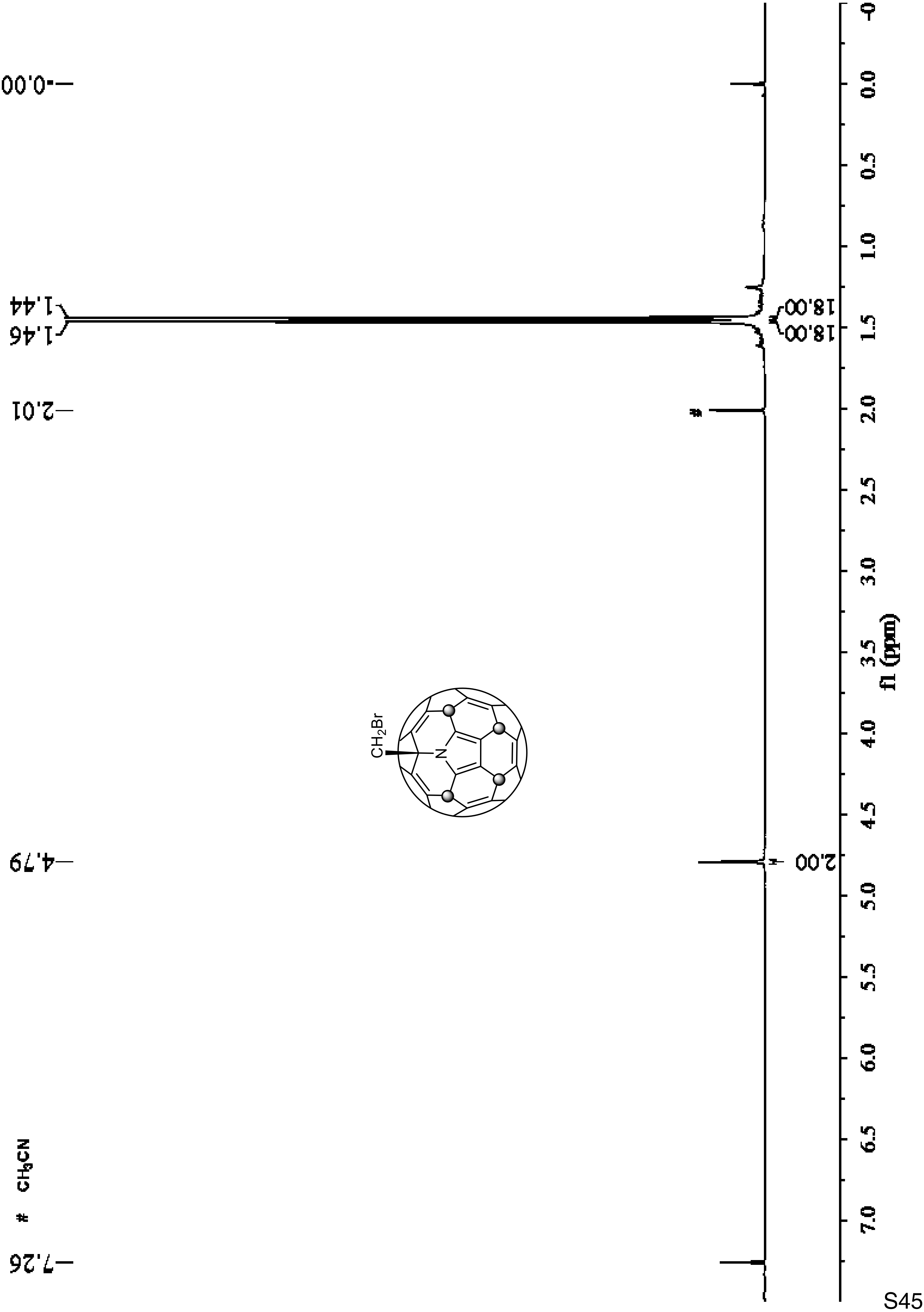

149.04
149.02
149.00
148.96
148.94
148.86
147.54
147.34
147.29
147.28
147.23
147.21
147.19
146.95
146.94
146.85
146.78
146.68
146.61
146.60
146.43
145.10
143.36
143.35
143.13
138.29
134.63
128.77
127.69
82.16
81.87
81.50
81.33
78.68
77.76
77.25
77.00
76.75
29.68
26.99
26.84
26.81
21.25
-0.03
150.62
149.69
149.00
147.29
147.23
147.21
147.19
146.94
146.61
146.60
146.43
150.5
149.0
147.5
147.0
146.5
146.0
f1 (ppm)
82.16
81.87
81.50
81.41
81.37
81.33
78.68
77.76
83
82
78
f1 (ppm)
▼ H grease
145.41
145.14
145.10
143.36
143.35
143.25
143.13
138.29
136.72
134.63
129.60
128.77
127.69
142
138
130
f1 (ppm)
Me
HO
CH
N
▼
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



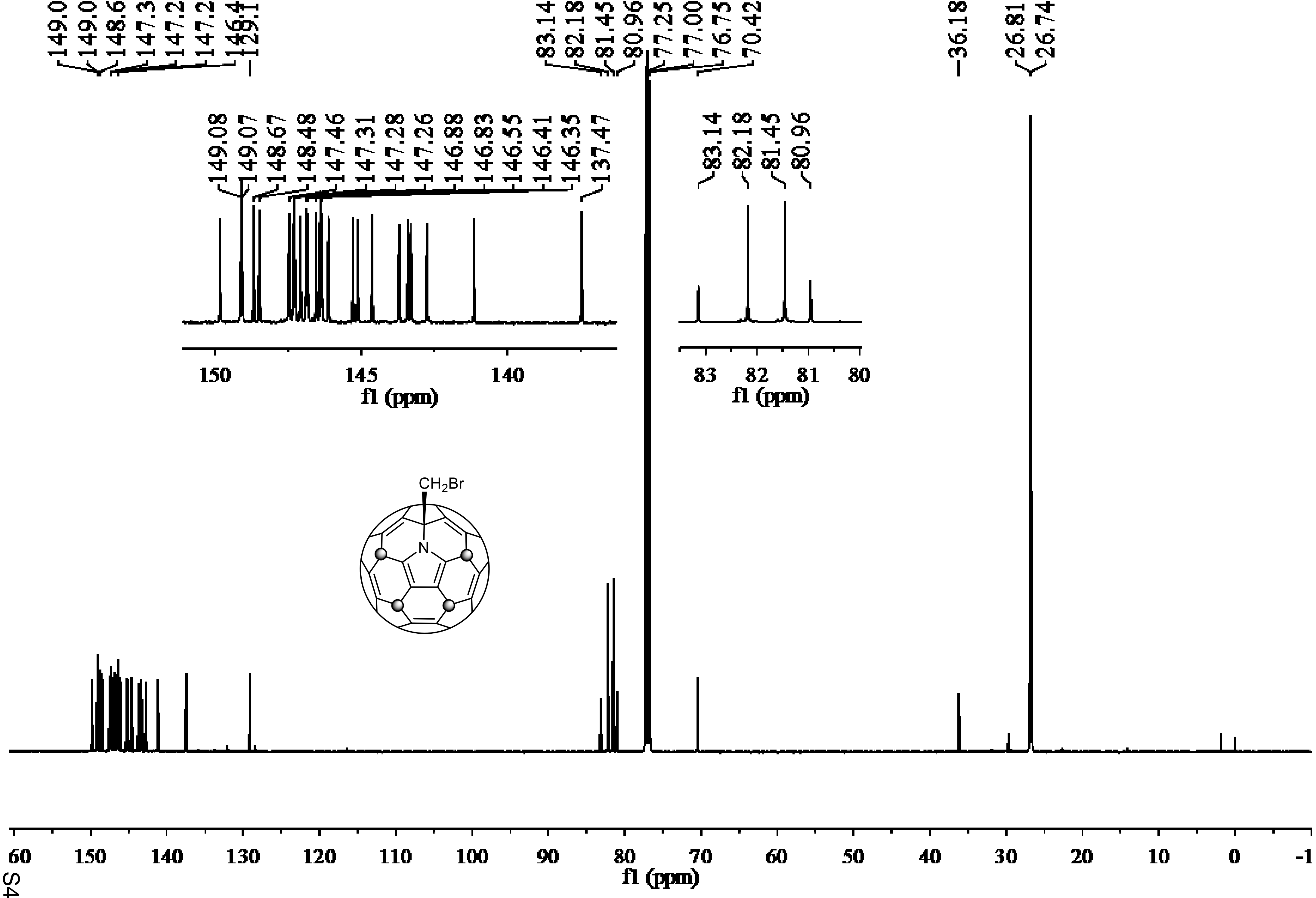
7.26
4.97
4.94
4.31
4.28
2.00
1.49
1.45
1.42
1.41
0.00
4.97
4.94
4.31
4.28
1.00
0.99
5.0
4.9
4.3
4.2
fl (ppm)
Cl
H_2
C
N
1.00
0.99
9.02
9.01
18.05
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
fl (ppm)

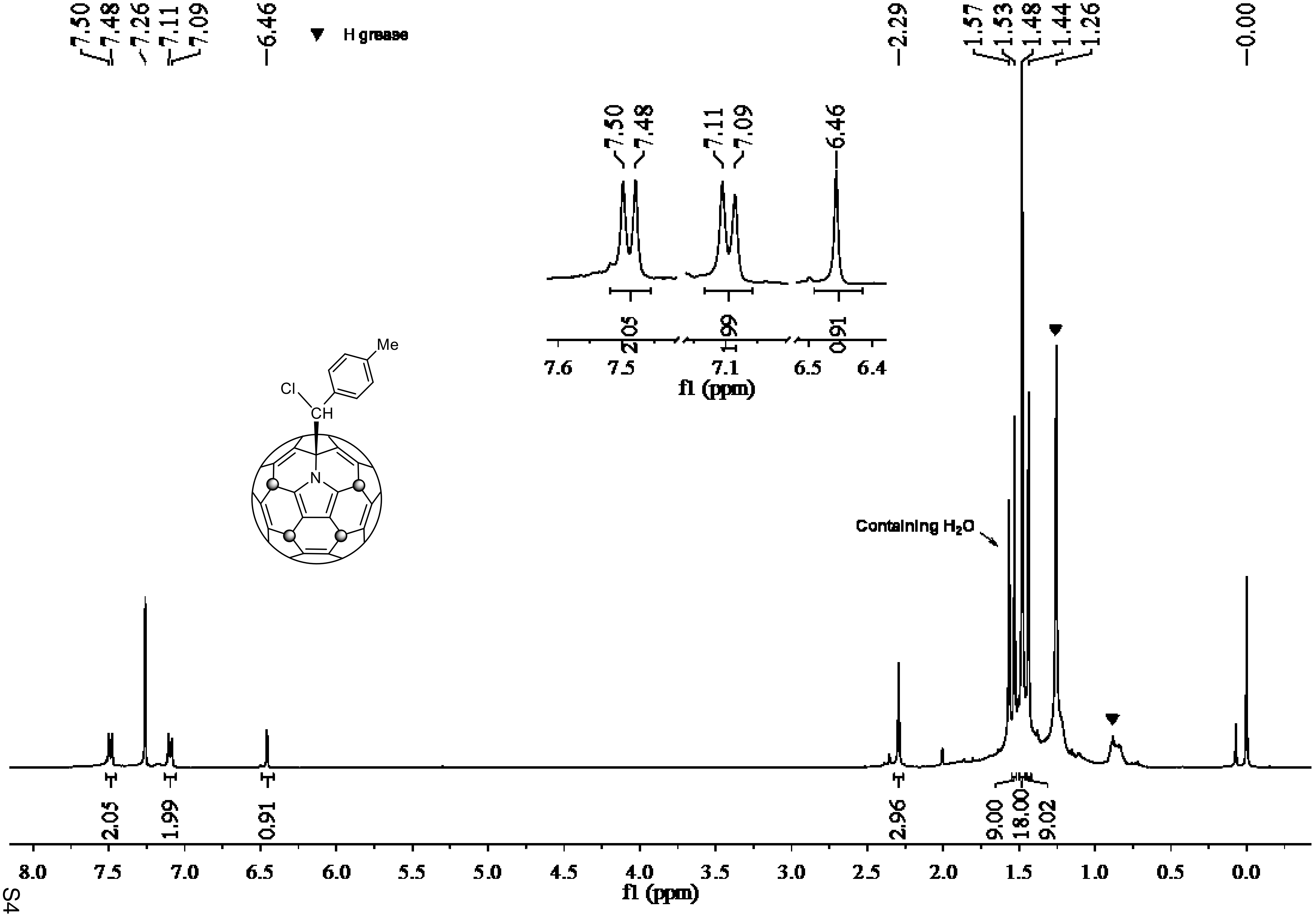

150.82
148.51
148.12
147.51
147.47
147.43
147.28
147.25
147.19
147.15
146.94
146.92
146.69
146.54
146.47
146.44
146.31
146.29
145.24
145.09
144.27
144.24
143.46
143.43
143.38
143.36
142.61
141.34
136.50
136.33
130.67
82.16
81.52
81.48
81.37
77.25
77.06
77.00
76.75
38.79
26.95
26.88
26.86
26.81
148.12
147.47
147.43
147.28
147.15
146.47
146.31
146.29
145.09
144.24
143.43
143.38
142.61
136.50
150
145
140
135
130
f1 (ppm)
82.78
82.16
81.76
81.52
81.48
81.37
77.25
77.06
77.00
76.75
83
82
81
77
f1 (ppm)
Cl
H_2
C
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

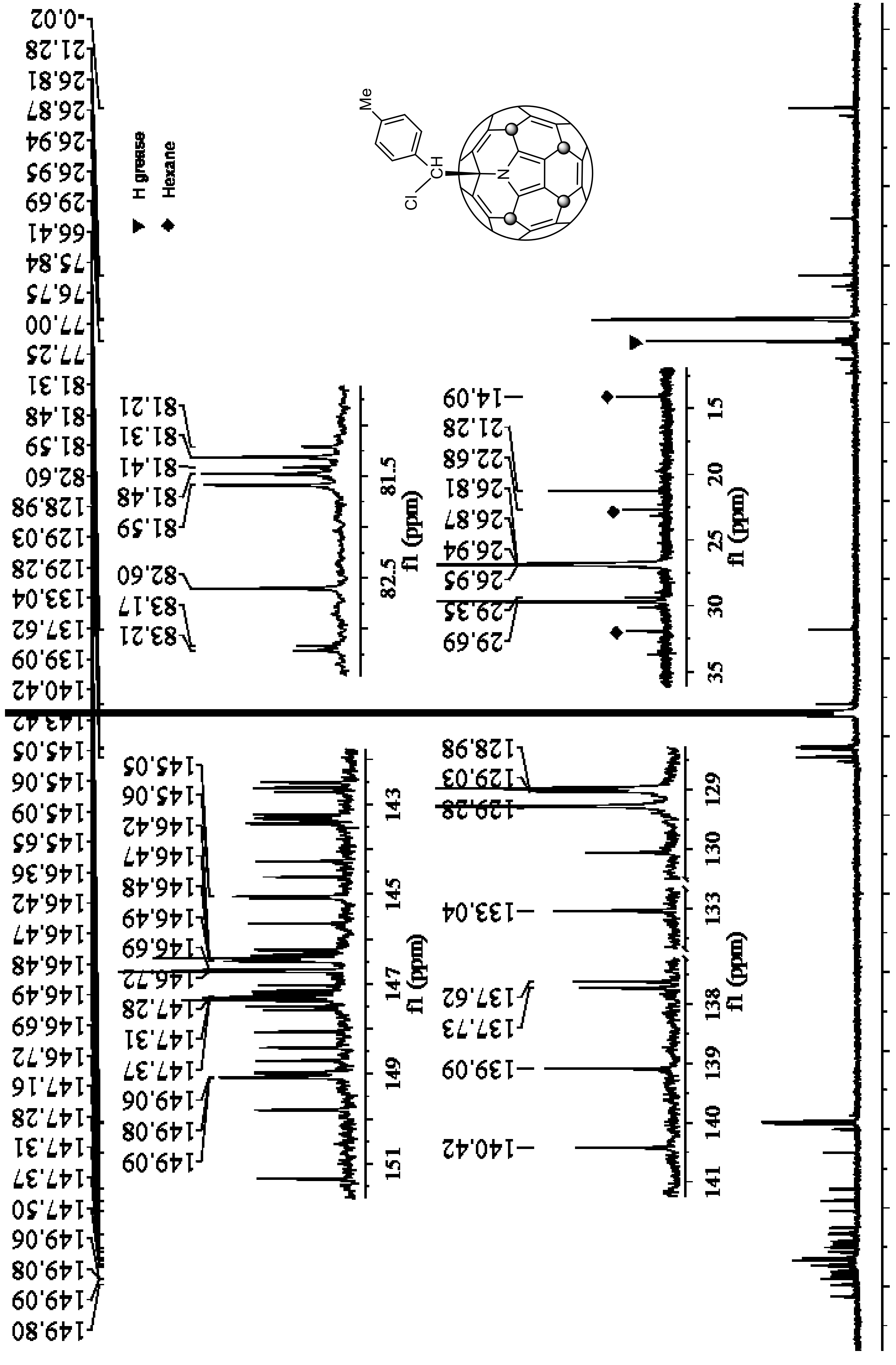

0.00
1.44
1.46
2.01
4.91
▼ H grease
CH$_3$CN
Containing H$_2$O
18.03
18.07
2.00
4.91
2.00
4.95
f1 (ppm)
1.44
1.46
18.03
18.07
1.46
f1 (ppm)
CH$_2$Cl
N
-1.0
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
f1 (ppm)

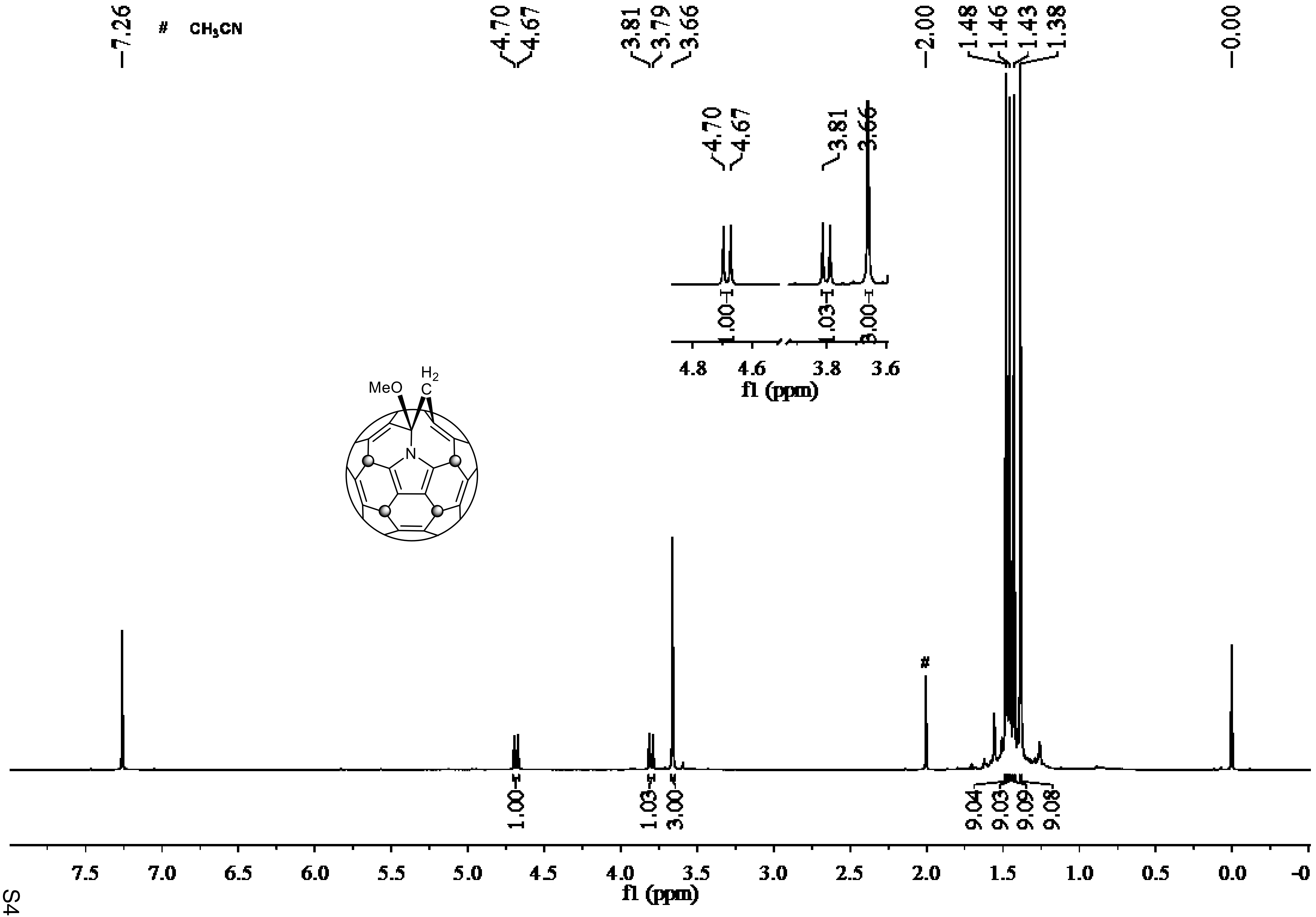


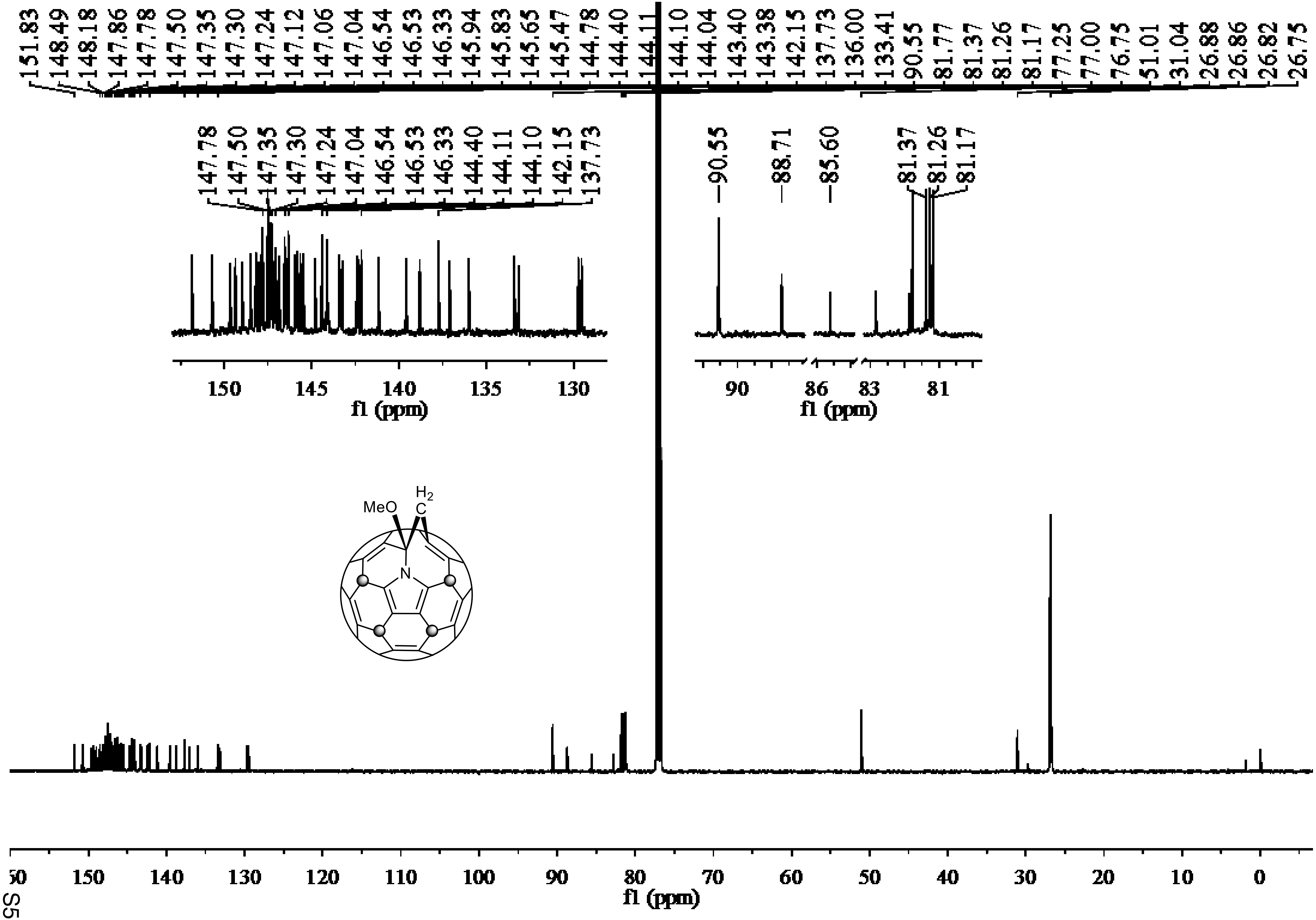
0.00
1.44
1.46
2.01
4.79
7.26
CH$_3$CN
#
CH$_2$Br
N
18.00
18.00
2.00
-0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
f1 (ppm)

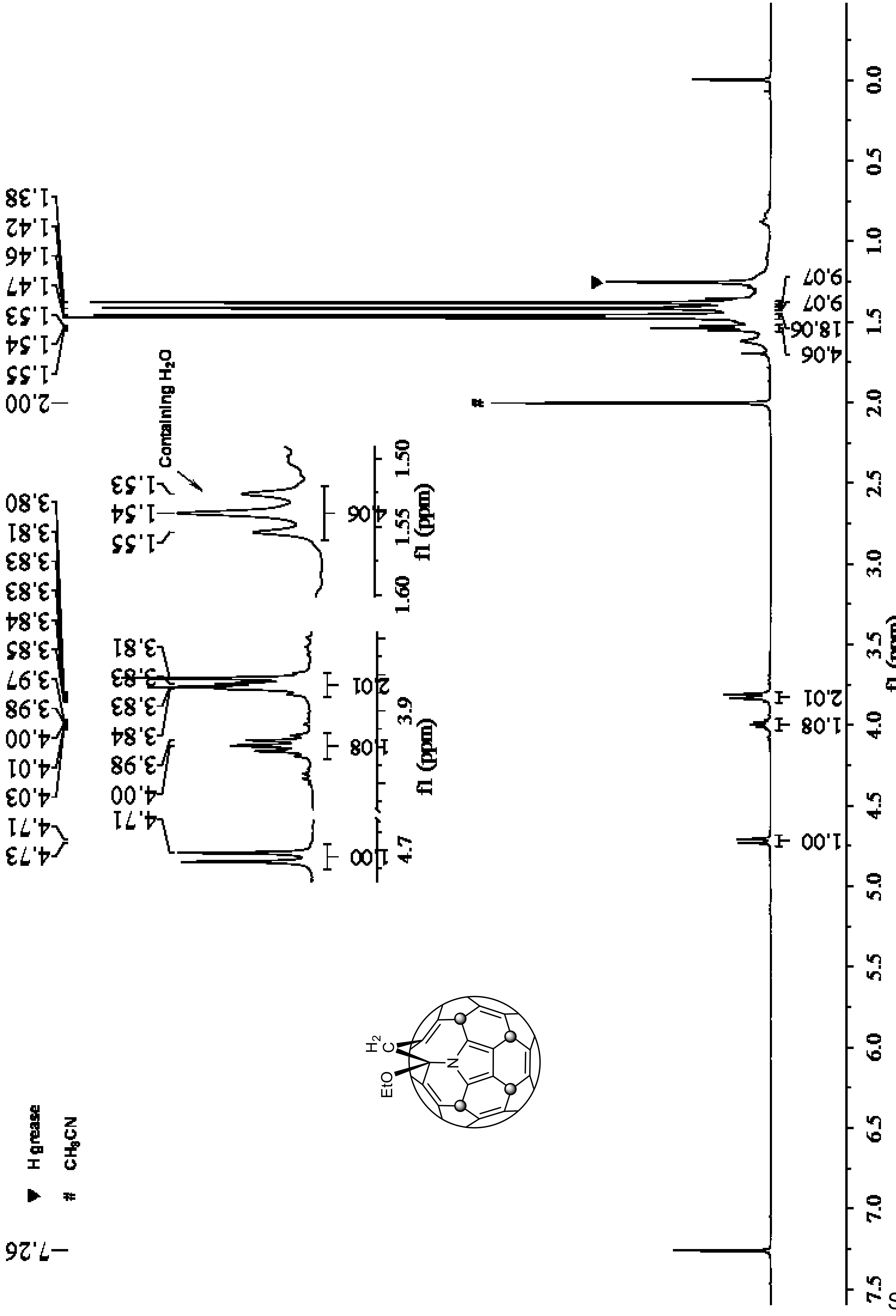

149.08
149.07
148.67
147.31
147.28
147.26
146.41
129.14
83.14
82.18
81.45
80.96
77.25
77.00
76.75
70.42
36.18
26.81
26.74
149.08
149.07
148.67
148.48
147.46
147.31
147.28
147.26
146.88
146.83
146.55
146.41
146.35
137.47
150
145
140
f1 (ppm)
83.14
82.18
81.45
80.96
83
82
81
80
f1 (ppm)
CH$_2$Br
N
60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

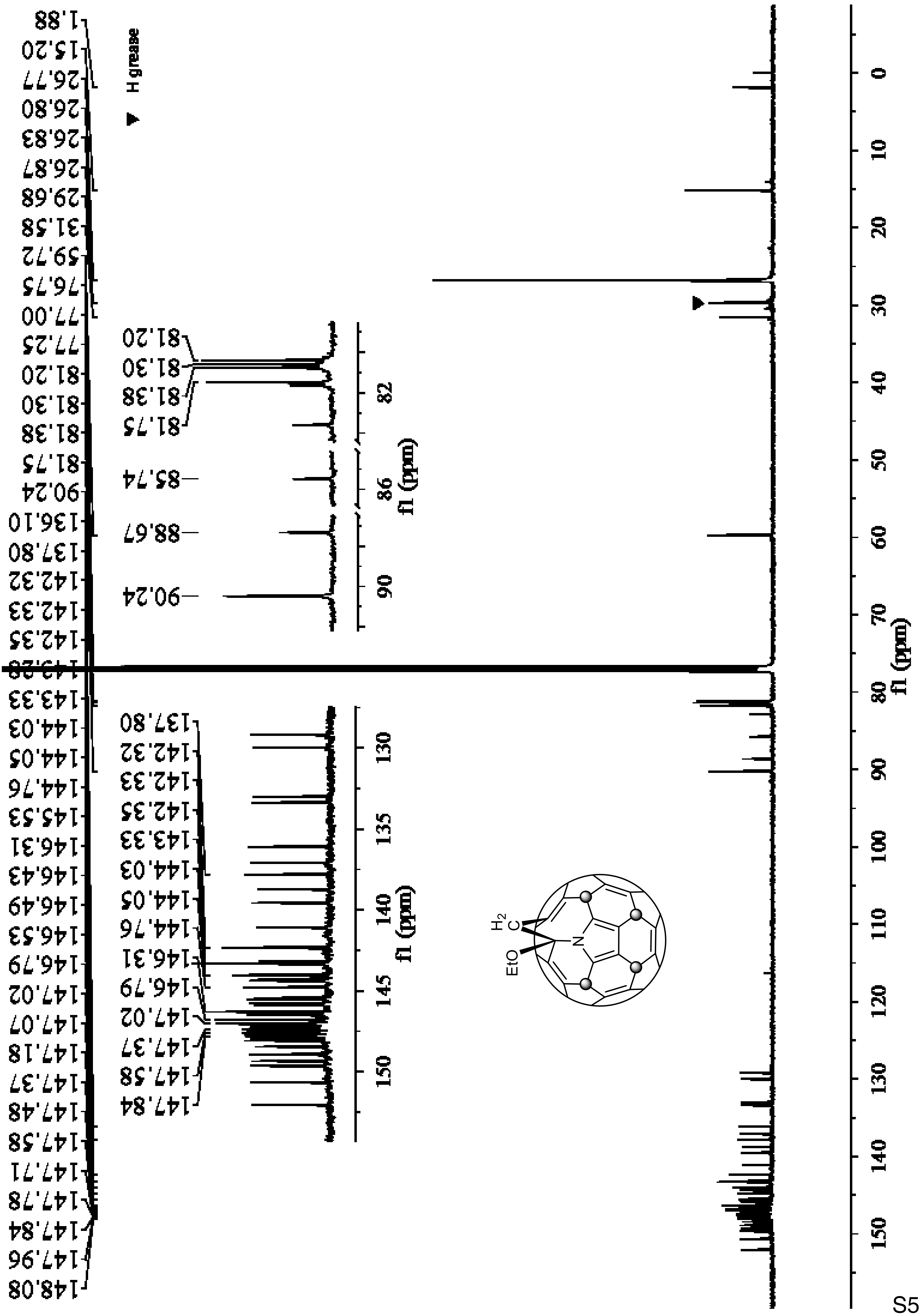

7.50
7.48
7.26
7.11
7.09
6.46
▼ H grease
2.29
1.57
1.53
1.48
1.44
1.26
0.00
7.50
7.48
7.11
7.09
6.46
2.05
1.99
0.91
7.6
7.5
7.1
6.5
6.4
fl (ppm)
Cl
CH
Me
N
Containing H_2O
2.05
1.99
0.91
2.96
9.00
18.00
9.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
fl (ppm)

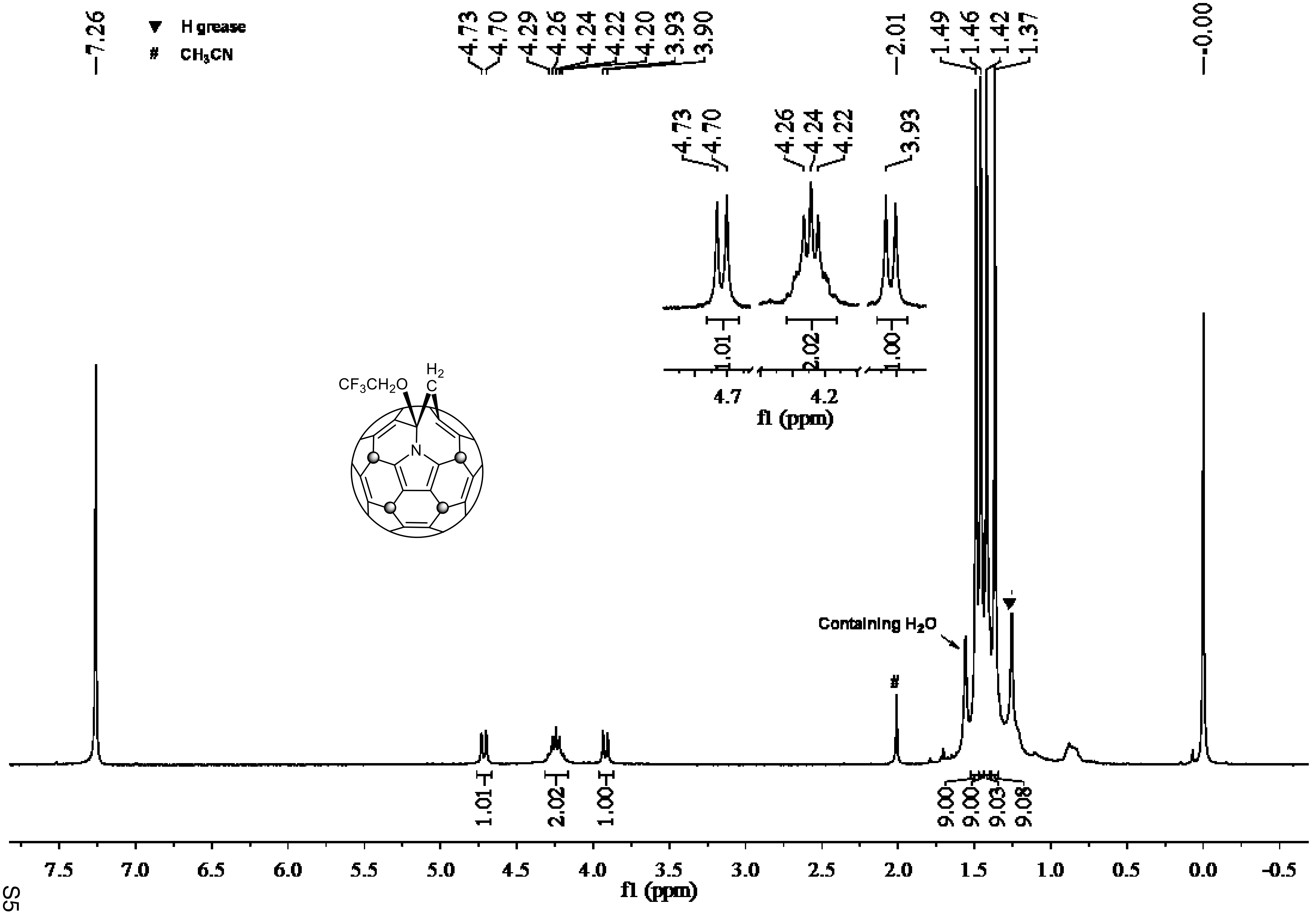

-0.02
21.28
26.81
26.87
26.94
26.95
29.69
66.41
75.84
76.75
77.00
77.25
81.31
81.48
81.59
82.60
128.98
129.03
129.28
133.04
137.62
139.09
140.42
143.42
145.05
145.06
145.09
145.65
146.36
146.42
146.47
146.48
146.49
146.69
146.72
147.16
147.28
147.31
147.37
147.50
149.06
149.08
149.09
149.80
H grease
Hexane
Me
CH
Cl
N
81.21
81.31
81.41
81.48
81.59
82.60
83.17
83.21
82.5
81.5
f1 (ppm)
14.09
21.28
22.68
26.81
26.87
26.94
26.95
29.35
29.69
35
30
25
20
15
f1 (ppm)
145.05
145.06
146.42
146.47
146.48
146.49
146.69
146.72
147.28
147.31
147.37
149.06
149.08
149.09
151
149
147
145
143
f1 (ppm)
128.98
129.03
129.28
133.04
137.62
137.73
139.09
140.42
141
140
139
138
133
130
129
f1 (ppm)
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
f1 (ppm)

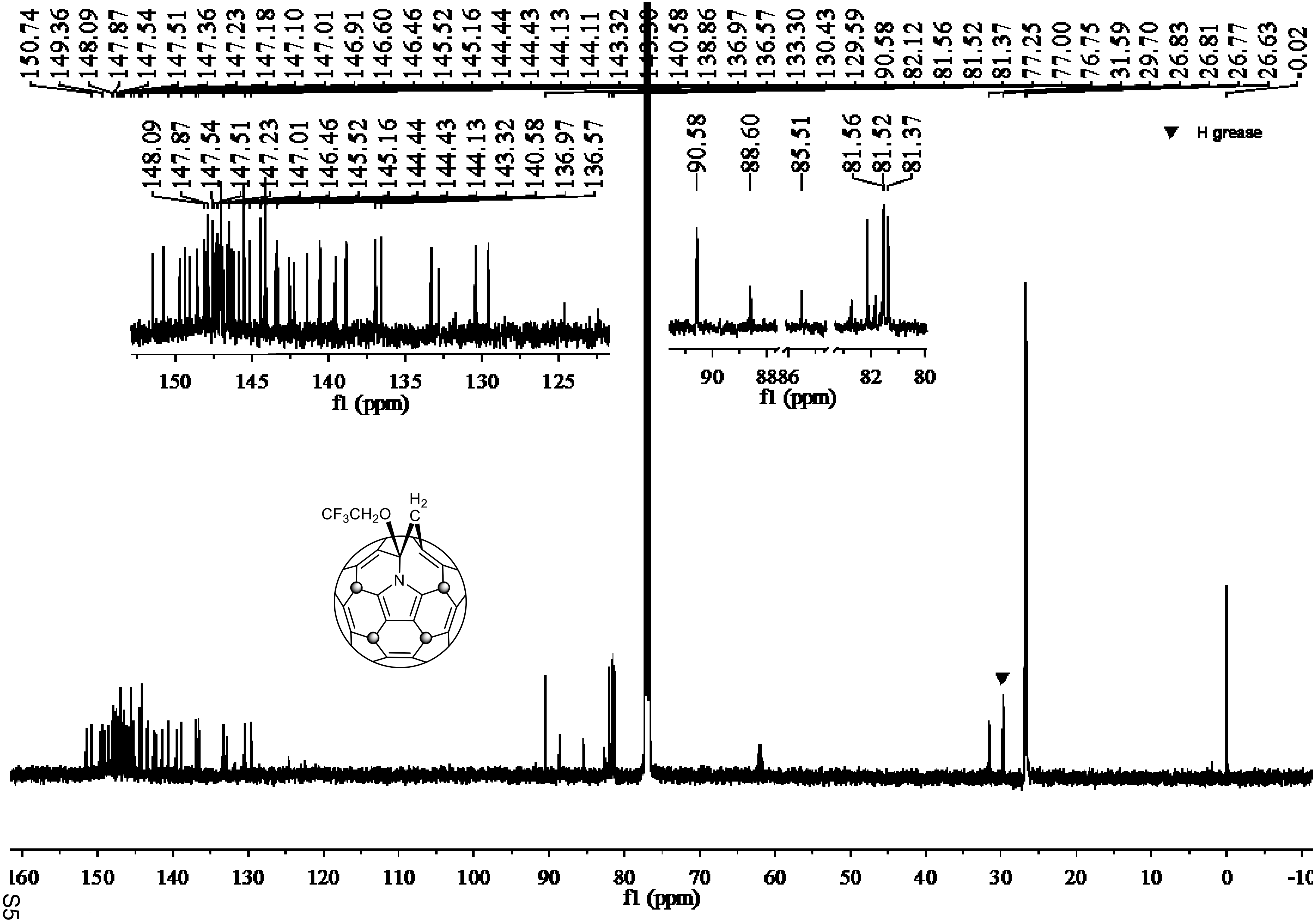

-7.26
CH$_3$CN
4.70
4.67
3.81
3.79
3.66
-2.00
1.48
1.46
1.43
1.38
-0.00
4.70
4.67
3.81
3.66
1.00
1.03
3.00
4.8
4.6
3.8
3.6
fl (ppm)
MeO
H$_2$
C
N
#
1.00
1.03
3.00
9.04
9.03
9.09
9.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
fl (ppm)

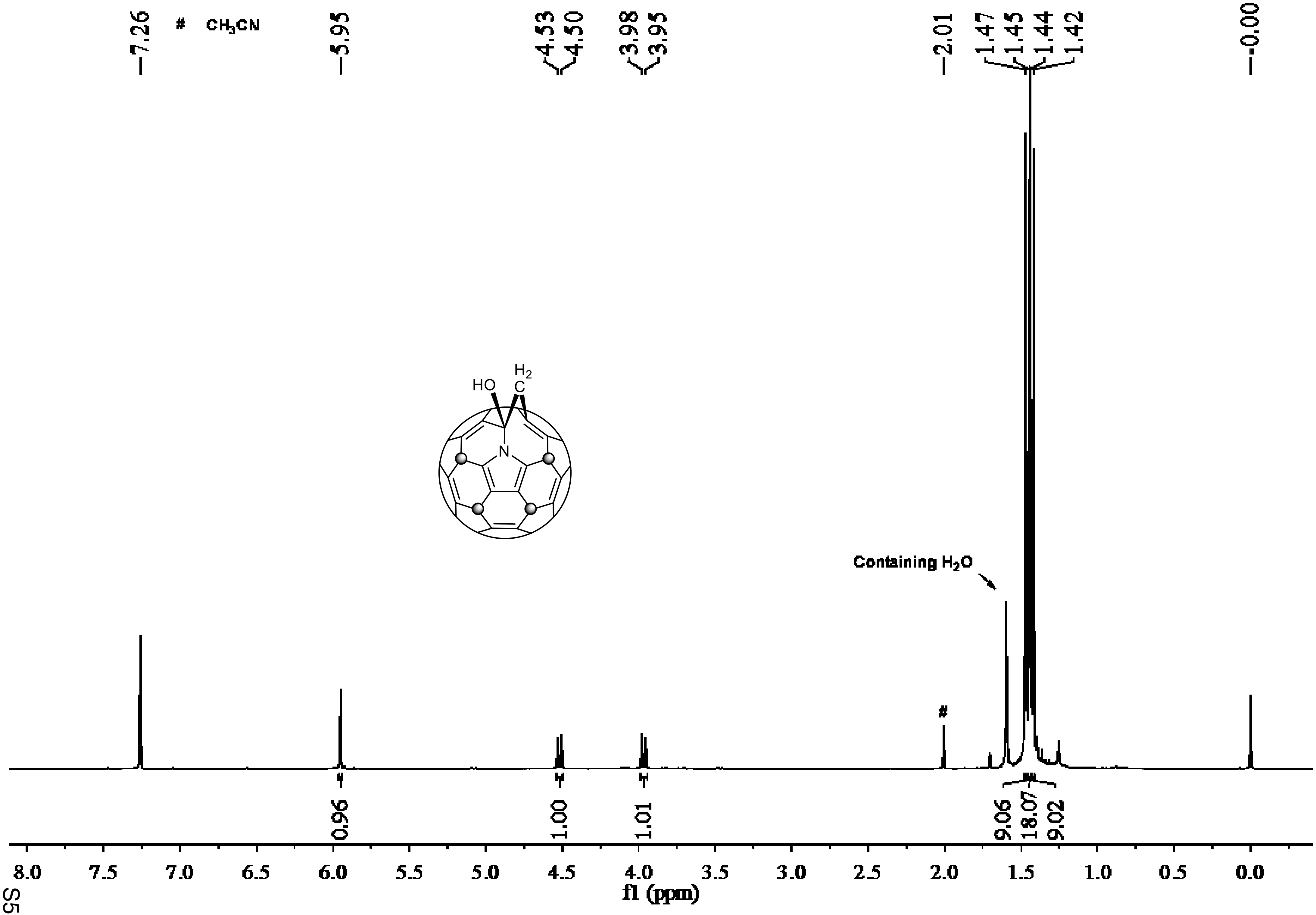

151.83
148.49
148.18
147.86
147.78
147.50
147.35
147.30
147.24
147.12
147.06
147.04
146.54
146.53
146.33
145.94
145.83
145.65
145.47
144.78
144.40
144.11
144.10
144.04
143.40
143.38
142.15
137.73
136.00
133.41
90.55
81.77
81.37
81.26
81.17
77.25
77.00
76.75
51.01
31.04
26.88
26.86
26.82
26.75
147.78
147.50
147.35
147.30
147.24
147.04
146.54
146.53
146.33
144.40
144.11
144.10
142.15
137.73
150
145
140
135
130
f1 (ppm)
90.55
88.71
85.60
81.37
81.26
81.17
90
86
83
81
f1 (ppm)
MeO
H_2
C
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


▼ H grease
CH_3CN
7.26
4.73
4.71
4.03
4.01
4.00
3.98
3.97
3.85
3.84
3.83
3.83
3.81
3.80
2.00
1.55
1.54
1.53
1.47
1.46
1.42
1.38
Containing H_2O
1.53
1.54
1.55
4.06
1.50
1.55
1.60
f1 (ppm)
3.81
3.83
3.83
3.84
3.98
4.00
4.71
2.01
1.08
1.00
3.9
4.7
f1 (ppm)
EtO
H_2
C
N
1.00
1.08
2.01
4.06
18.06
9.07
9.07
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
f1 (ppm)

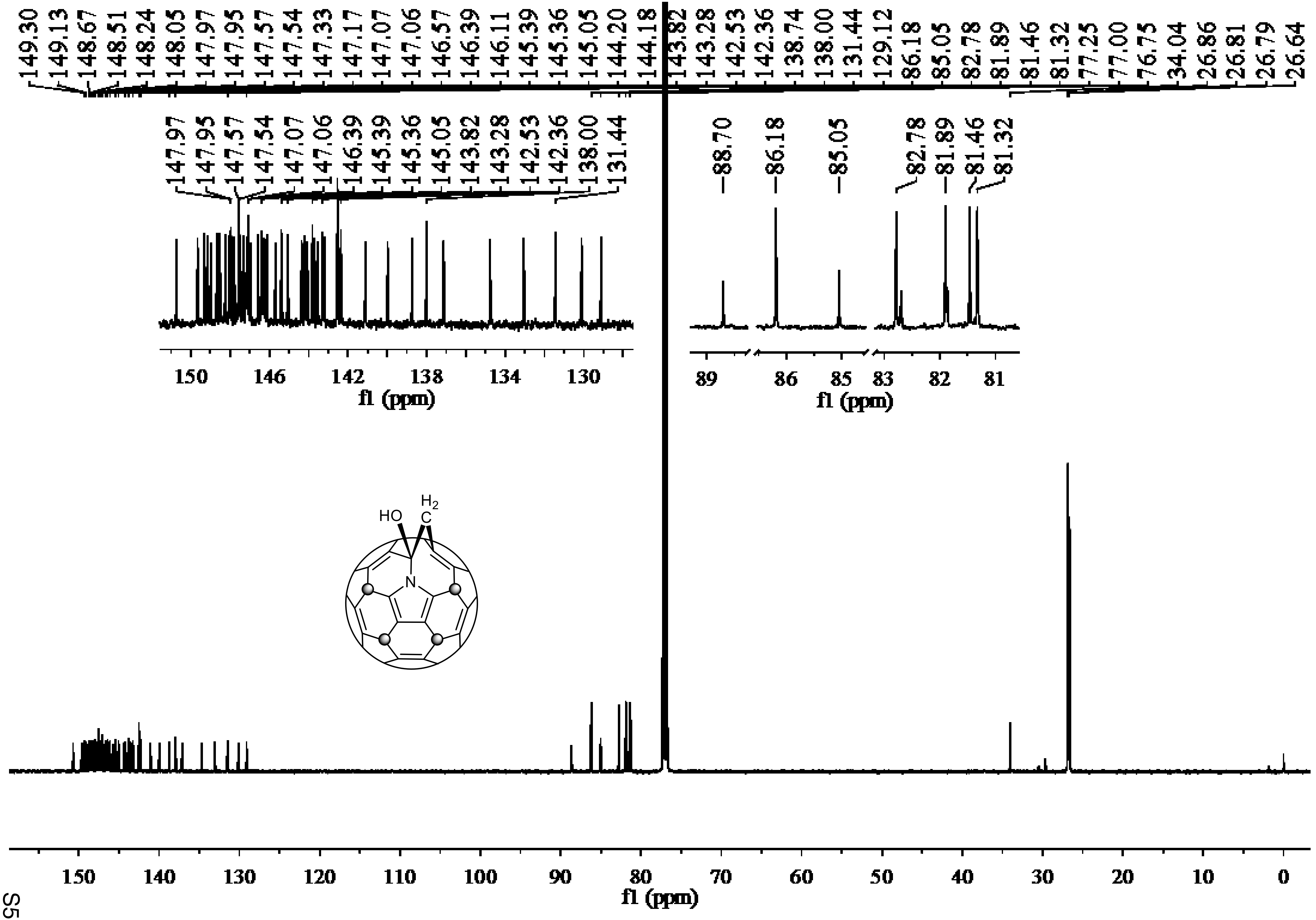

1.88
15.20
26.77
26.80
26.83
26.87
29.68
31.58
59.72
76.75
77.00
77.25
81.20
81.30
81.38
81.75
90.24
136.10
137.80
142.32
142.33
142.35
143.28
143.33
144.03
144.05
144.76
145.53
146.31
146.43
146.49
146.53
146.79
147.02
147.07
147.18
147.37
147.48
147.58
147.71
147.78
147.84
147.96
148.08
H grease
81.20
81.30
81.38
81.75
85.74
88.67
90.24
82
86
90
f1 (ppm)
137.80
142.32
142.33
142.35
143.33
144.03
144.05
144.76
146.31
146.79
147.02
147.37
147.58
147.84
130
135
140
145
150
f1 (ppm)
EtO
H_2
C
N
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
f1 (ppm)

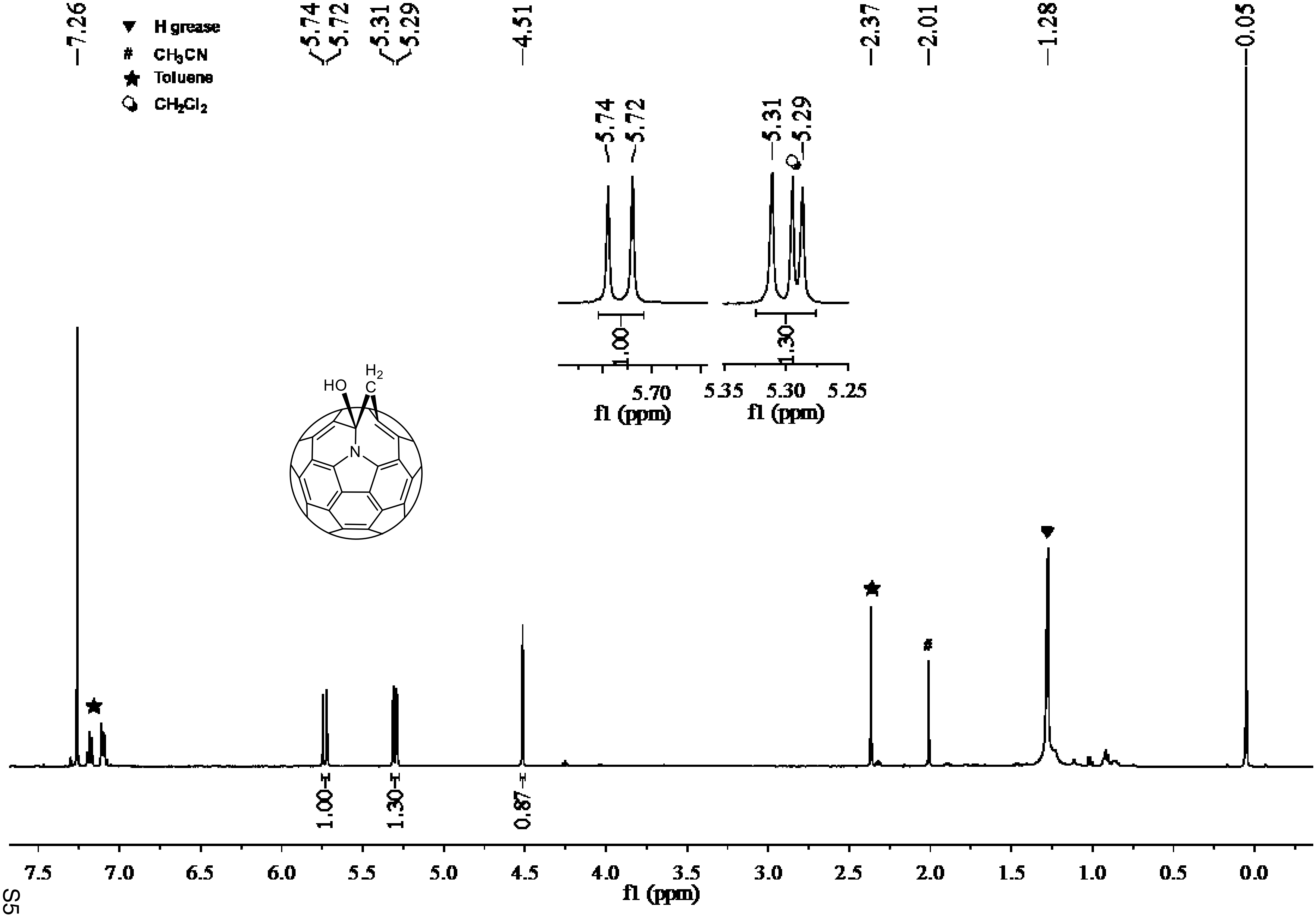

7.26
H grease
CH$_3$CN
4.73
4.70
4.29
4.26
4.24
4.22
4.20
3.93
3.90
2.01
1.49
1.46
1.42
1.37
-0.00
4.73
4.70
4.26
4.24
4.22
3.93
1.01
2.02
1.00
4.7
4.2
f1 (ppm)
CF$_3$CH$_2$O
H$_2$
C
N
Containing H$_2$O
#
1.01
2.02
1.00
9.00
9.00
9.03
9.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

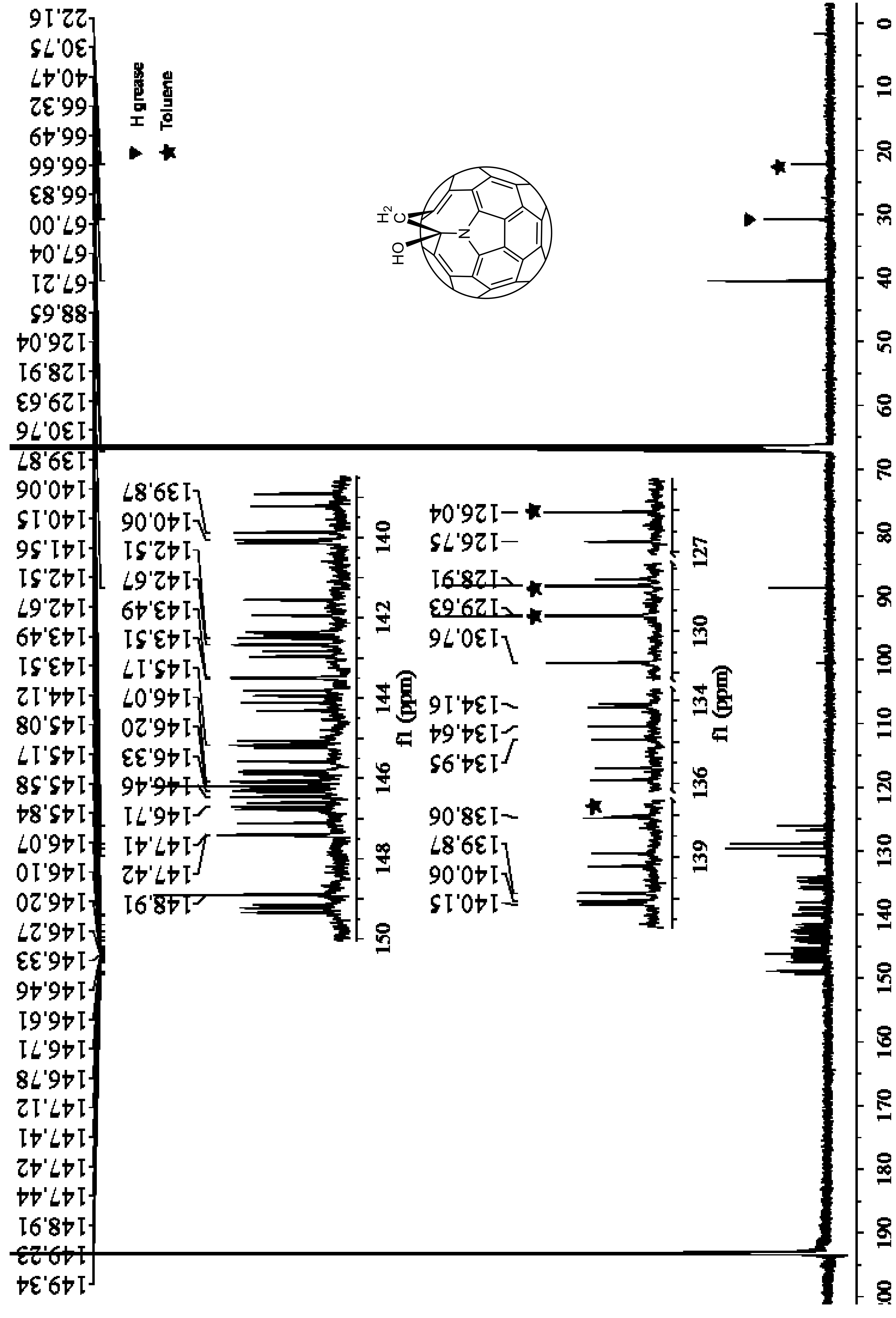

150.74
149.36
148.09
147.87
147.54
147.51
147.36
147.23
147.18
147.10
147.01
146.91
146.60
146.46
145.52
145.16
144.44
144.43
144.13
144.11
143.32
140.58
138.86
136.97
136.57
133.30
130.43
129.59
90.58
82.12
81.56
81.52
81.37
77.25
77.00
76.75
31.59
29.70
26.83
26.81
26.77
26.63
-0.02
148.09
147.87
147.54
147.51
147.23
147.01
146.46
145.52
145.16
144.44
144.43
144.13
143.32
140.58
136.97
136.57
150
145
140
135
130
125
f1 (ppm)
90.58
88.60
85.51
81.56
81.52
81.37
90
88
86
82
80
f1 (ppm)
▼ H grease
CF_3CH_2O
H_2
C
N
▼
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

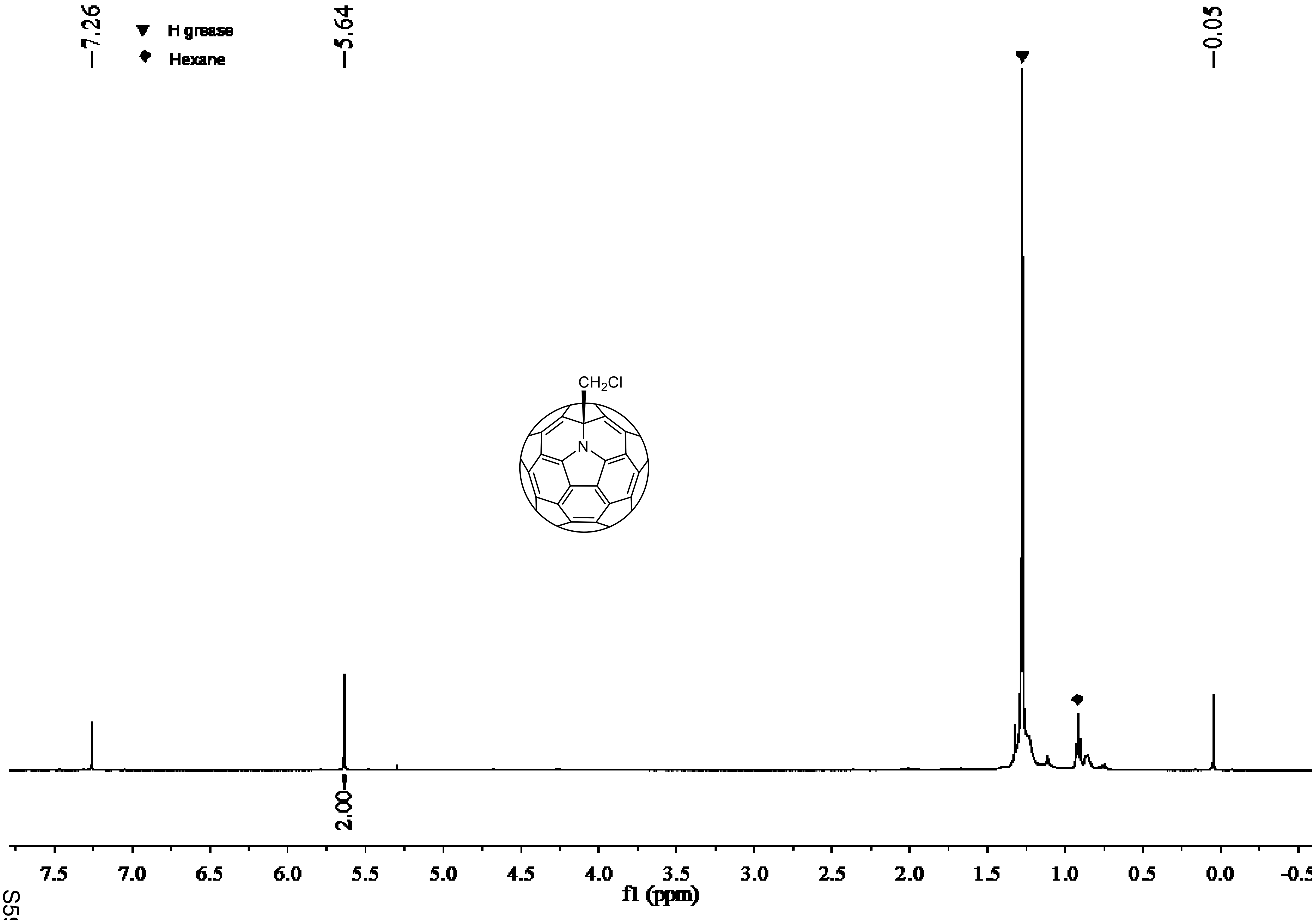


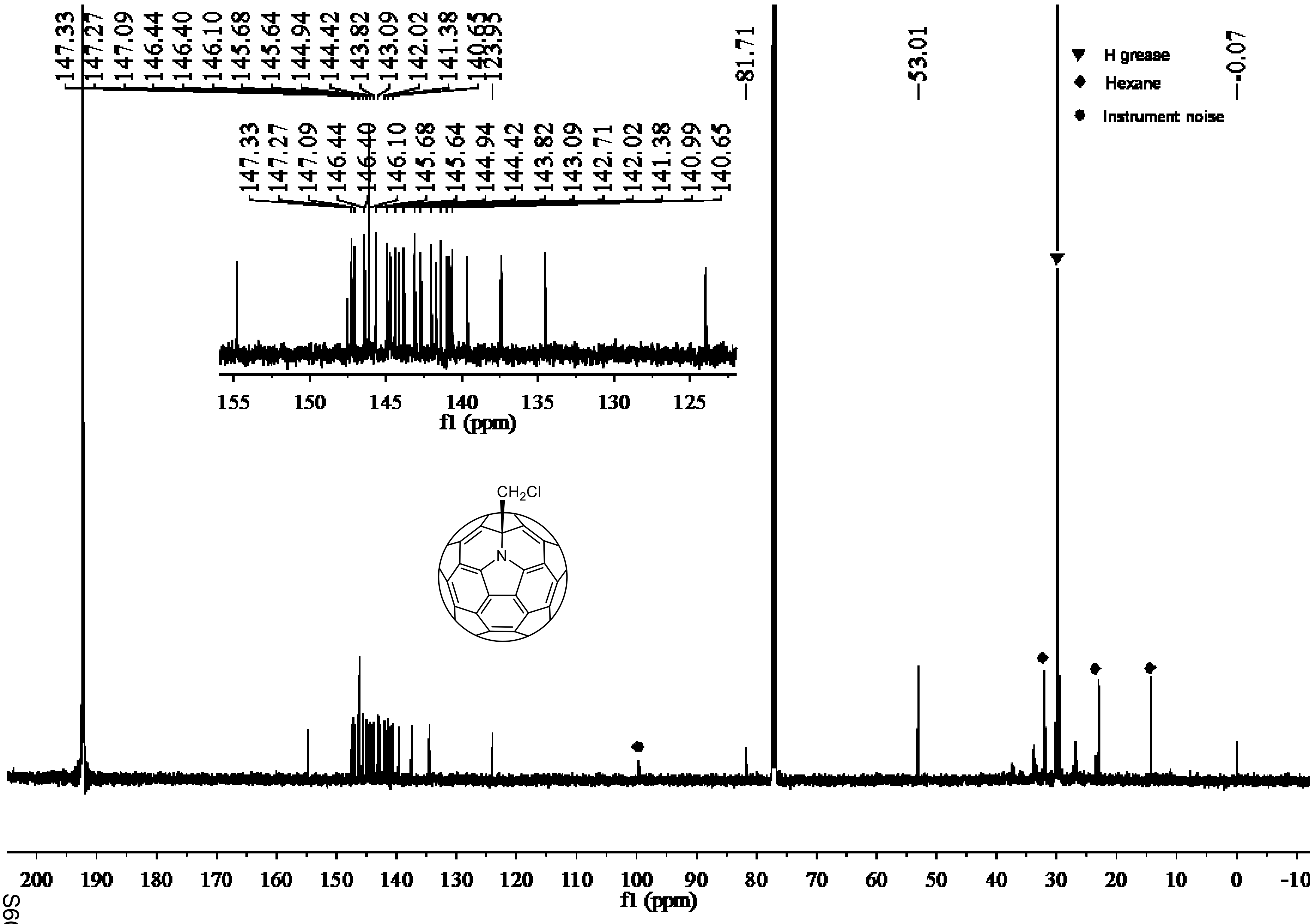
7.26
CH$_3$CN
5.95
4.53
4.50
3.98
3.95
2.01
1.47
1.45
1.44
1.42
-0.00
HO
H$_2$
C
N
Containing H$_2$O
#
0.96
1.00
1.01
9.06
18.07
9.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

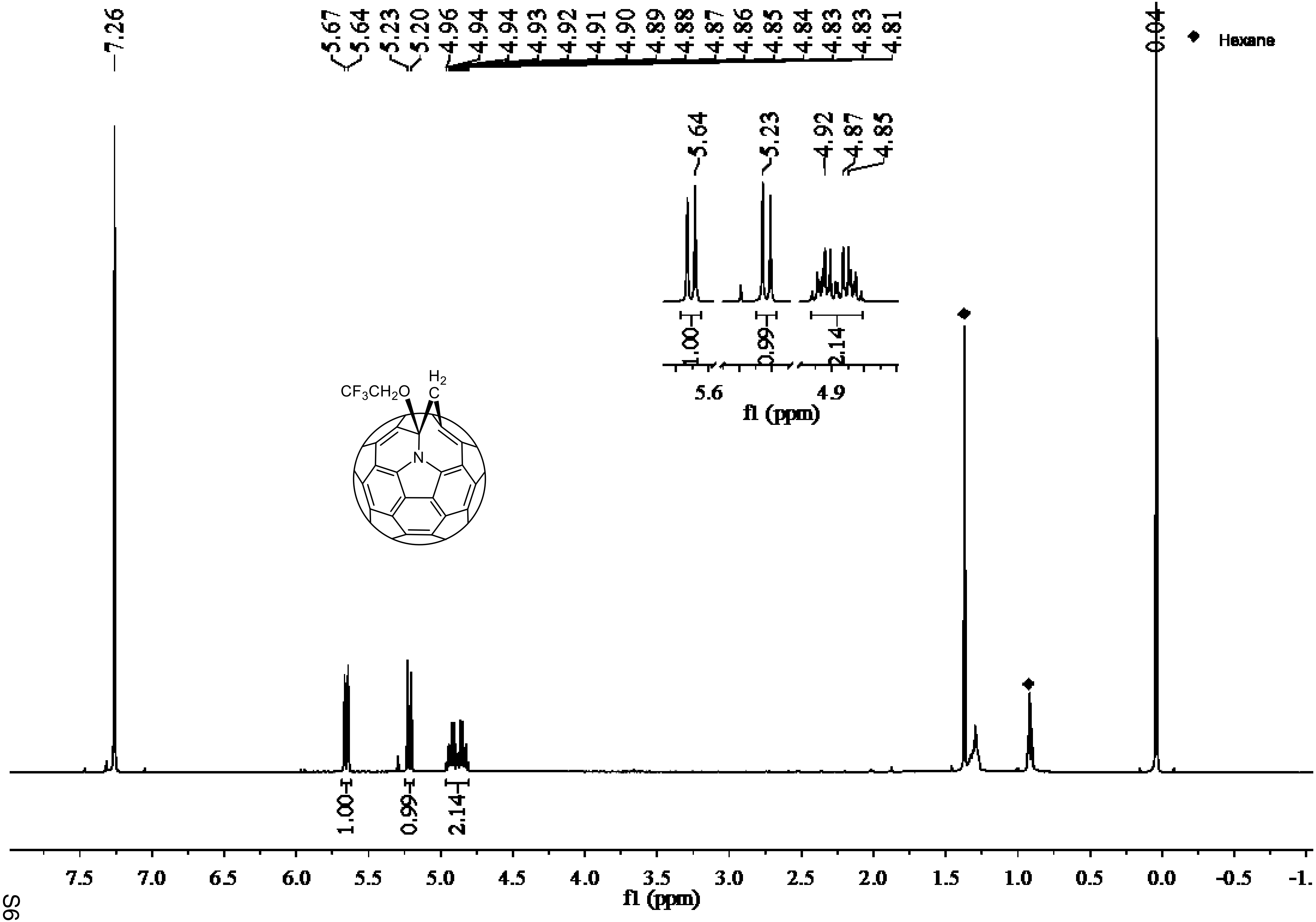

149.30
149.13
148.67
148.51
148.24
148.05
147.97
147.95
147.57
147.54
147.33
147.17
147.07
147.06
146.57
146.39
146.11
145.39
145.36
145.05
144.20
144.18
143.82
143.28
142.53
142.36
138.74
138.00
131.44
129.12
86.18
85.05
82.78
81.89
81.46
81.32
77.25
77.00
76.75
34.04
26.86
26.81
26.79
26.64
147.97
147.95
147.57
147.54
147.07
147.06
146.39
145.39
145.36
145.05
143.82
143.28
142.53
142.36
138.00
131.44
88.70
86.18
85.05
82.78
81.89
81.46
81.32
150
146
142
138
134
130
f1 (ppm)
89
86
85
83
82
81
f1 (ppm)
HO
H_2
C
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

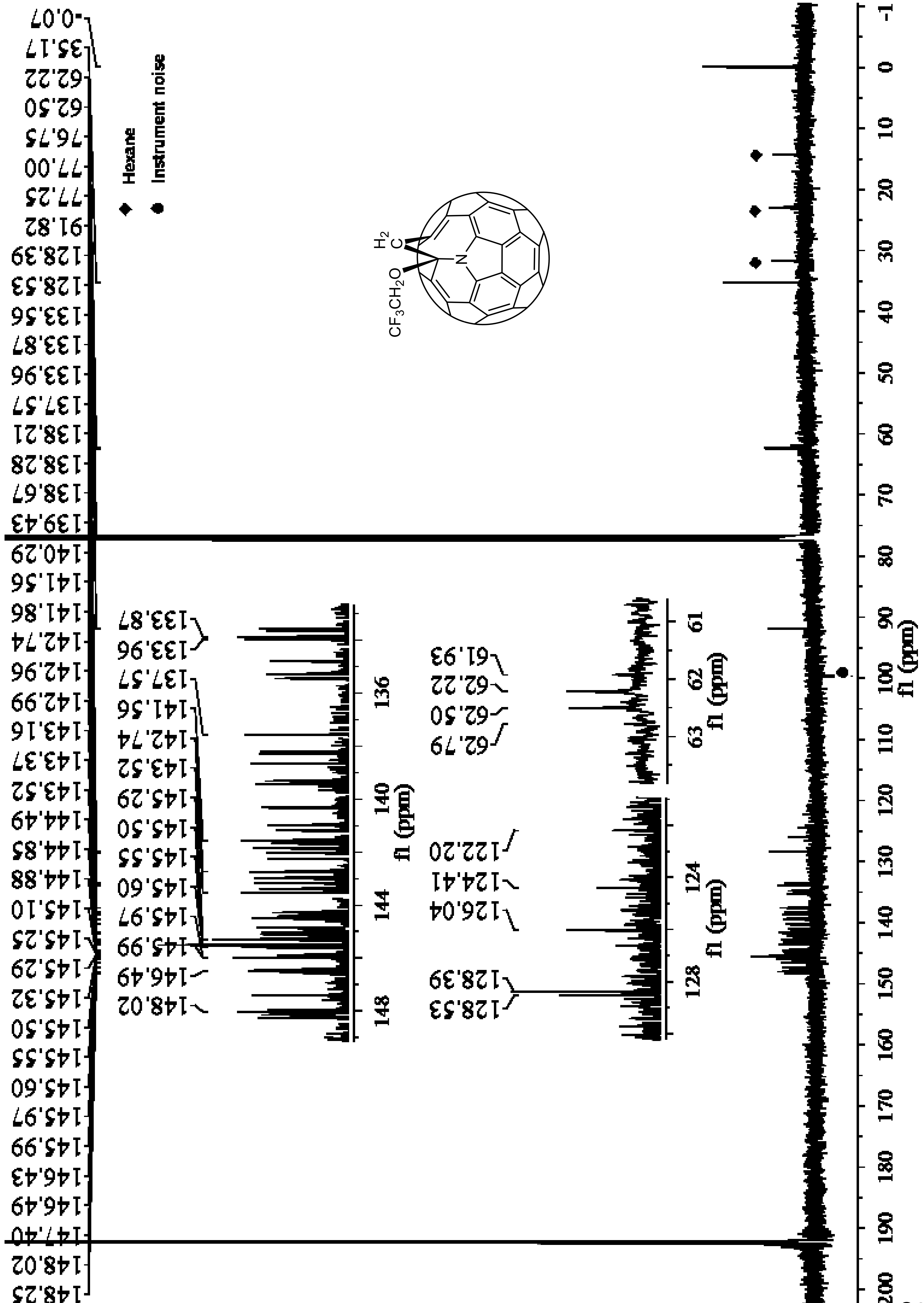

7.26
5.74
5.72
5.31
5.29
4.51
2.37
2.01
1.28
0.05
▼ H grease
CH_3CN
★ Toluene
◔ CH_2Cl_2
HO
H_2
C
N
5.74
5.72
1.00
5.70
f1 (ppm)
5.31
5.29
1.30
5.35
5.30
5.25
f1 (ppm)
1.00
1.30
0.87
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

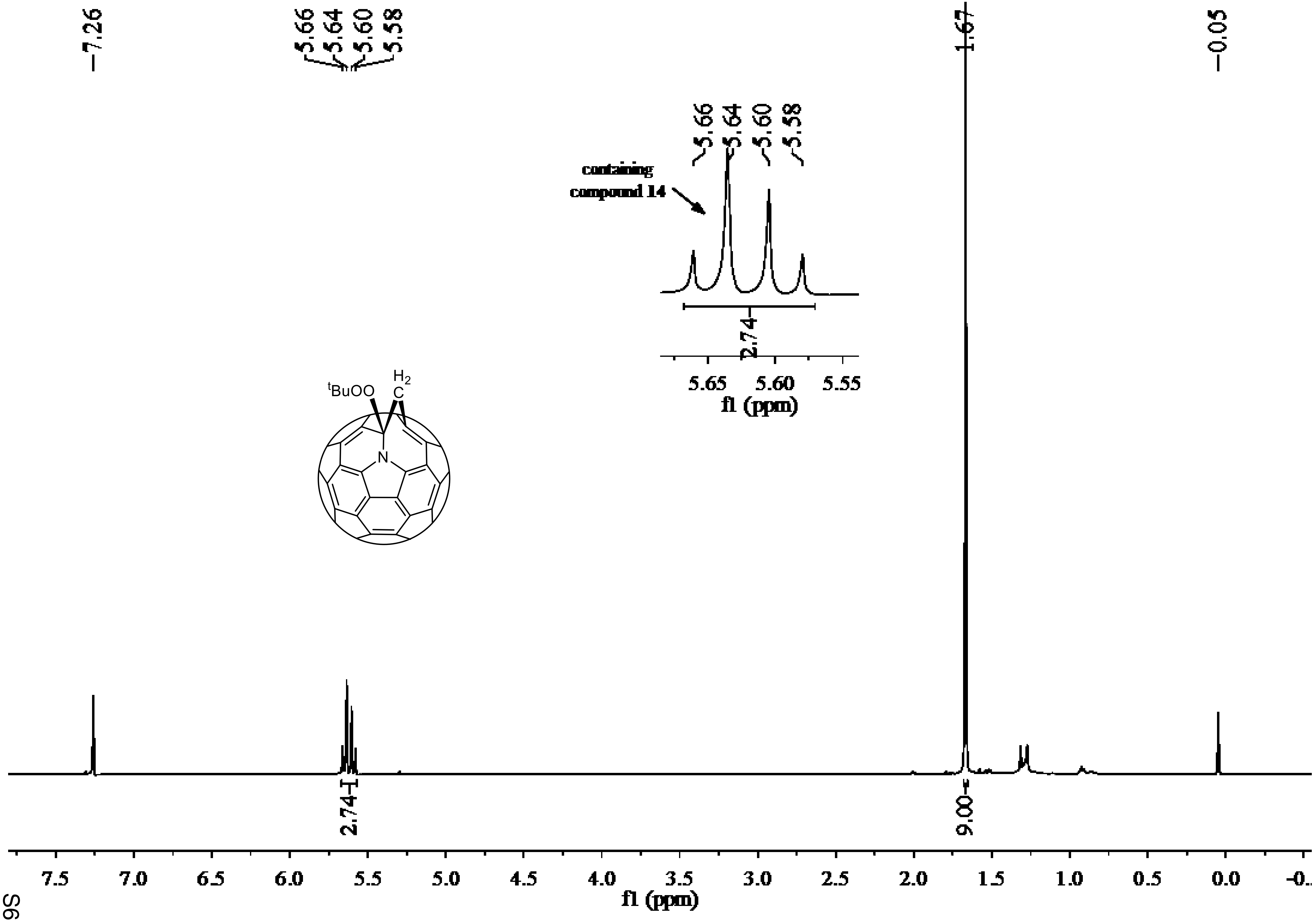

149.34
149.23
148.91
147.44
147.42
147.41
147.12
146.78
146.71
146.61
146.46
146.33
146.27
146.20
146.10
146.07
145.84
145.58
145.17
145.08
144.12
143.51
143.49
142.67
142.51
141.56
140.15
140.06
139.87
130.76
129.63
128.91
126.04
88.65
67.21
67.04
67.00
66.83
66.66
66.49
66.32
40.47
30.75
22.16
H grease
Toluene
HO
C
H2
N
148.91
147.42
147.41
146.71
146.46
146.33
146.20
146.07
145.17
143.51
143.49
142.67
142.51
140.06
139.87
150
148
146
144
142
140
f1 (ppm)
140.15
140.06
139.87
138.06
134.95
134.64
134.16
130.76
129.63
128.91
126.75
126.04
139
136
134
130
127
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.26
5.64
0.05
▼ H grease
◆ Hexane
CH$_2$Cl
N
2.00
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
-0.5
f1 (ppm)

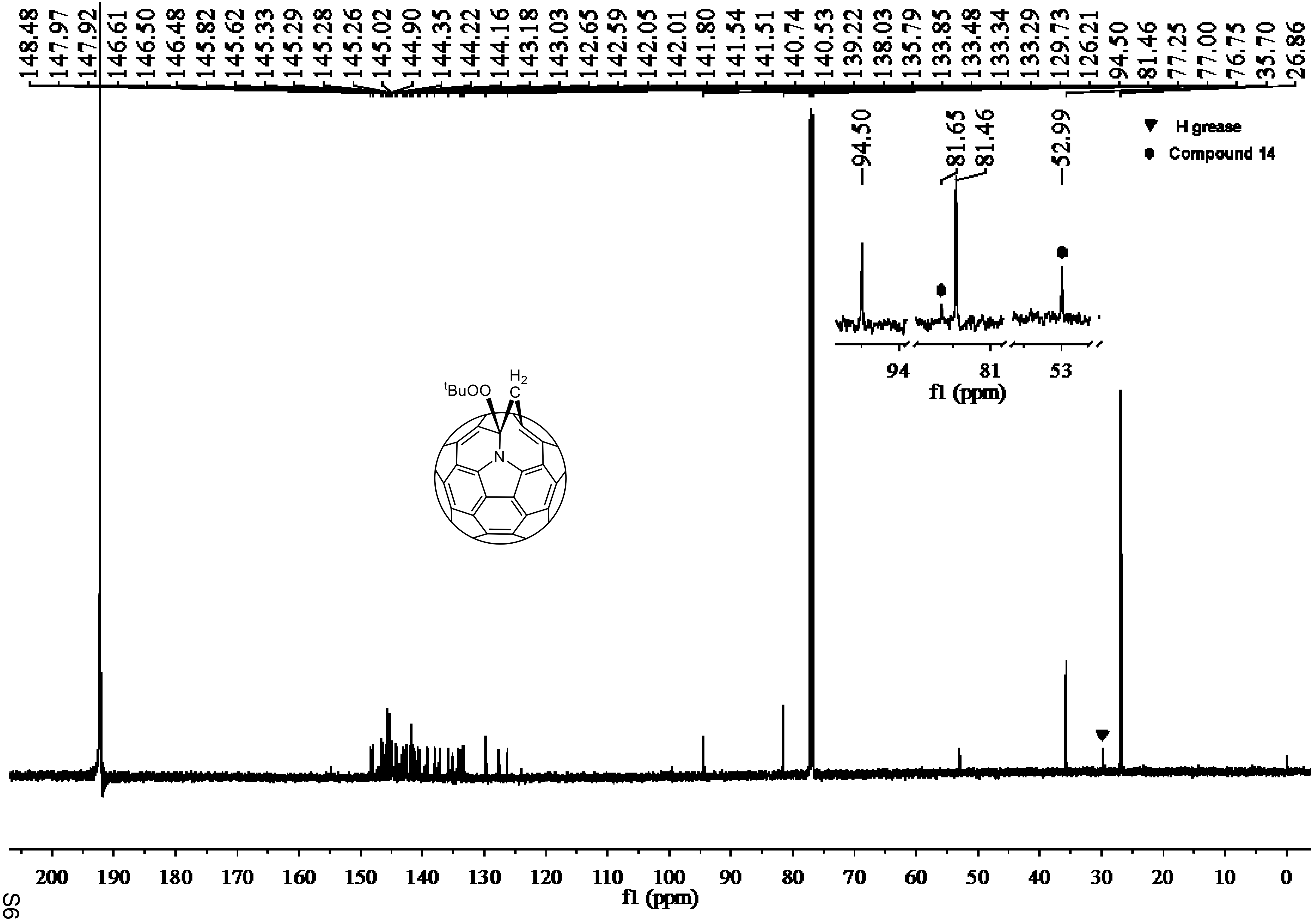

147.33
147.27
147.09
146.44
146.40
146.10
145.68
145.64
144.94
144.42
143.82
143.09
142.02
141.38
140.65
123.95
81.71
53.01
-0.07
147.33
147.27
147.09
146.44
146.40
146.10
145.68
145.64
144.94
144.42
143.82
143.09
142.71
142.02
141.38
140.99
140.65
155
150
145
140
135
130
125
f1 (ppm)
▼ H grease
◆ Hexane
● Instrument noise
CH_2Cl
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

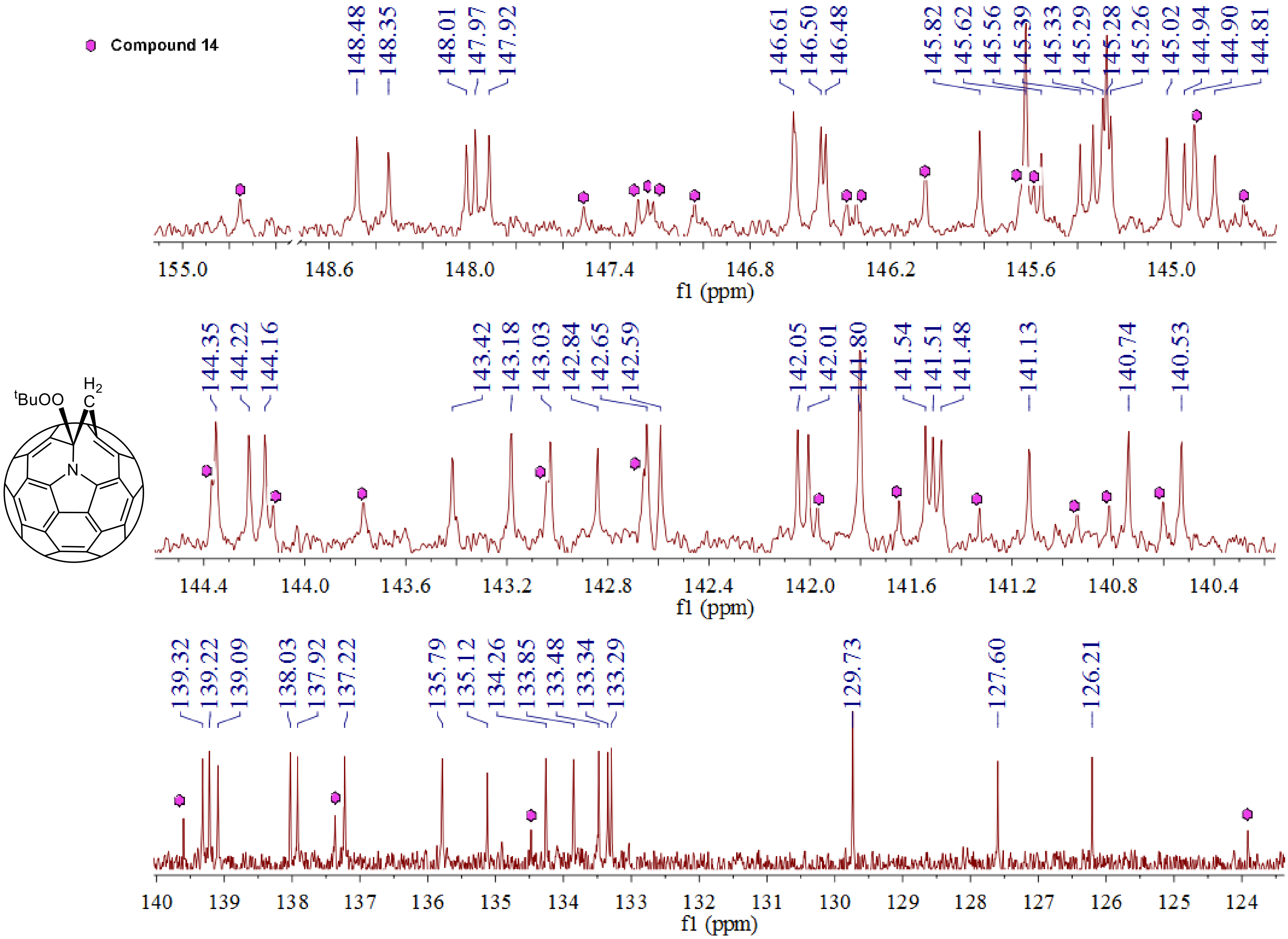

7.26
5.67
5.64
5.23
5.20
4.96
4.94
4.94
4.93
4.92
4.91
4.90
4.89
4.88
4.87
4.86
4.85
4.84
4.83
4.83
4.81
0.04
Hexane
5.64
5.23
4.92
4.87
4.85
1.00
0.99
2.14
5.6
4.9
fl (ppm)
CF_3CH_2O
H_2
C
N
1.00
0.99
2.14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.
fl (ppm)


148.25
148.02
147.40
146.49
146.43
145.99
145.97
145.60
145.55
145.50
145.32
145.29
145.25
145.10
144.88
144.85
144.49
143.52
143.37
143.16
142.99
142.96
142.74
141.86
141.56
140.29
139.43
138.67
138.28
138.21
137.57
133.96
133.87
133.56
128.53
128.39
91.82
77.25
77.00
76.75
62.50
62.22
35.17
-0.07
Hexane
Instrument noise
CF_3CH_2O
CH_2
N
148.02
146.49
145.99
145.97
145.60
145.55
145.50
145.29
143.52
142.74
141.56
137.57
133.96
133.87
148
144
140
136
f1 (ppm)
128.53
128.39
126.04
124.41
122.20
128
124
f1 (ppm)
62.79
62.50
62.22
61.93
63
62
61
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)



7.26
5.66
5.64
5.60
5.58
1.67
0.05
containing compound 14
5.65
5.60
5.55
fl (ppm)
2.74
9.00
^{t}BuOO
H_2
C
N
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
-0.
fl (ppm)


148.48
147.97
147.92
146.61
146.50
146.48
145.82
145.62
145.33
145.29
145.28
145.26
145.02
144.90
144.35
144.22
144.16
143.18
143.03
142.65
142.59
142.05
142.01
141.80
141.54
141.51
140.74
140.53
139.22
138.03
135.79
133.85
133.48
133.34
133.29
129.73
126.21
94.50
81.46
77.25
77.00
76.75
35.70
26.86
94.50
81.65
81.46
52.99
▼ H grease
● Compound 14
94
81
53
f1 (ppm)
^{t}BuOO
H_2
C
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Compound 14
148.48
148.35
148.01
147.97
147.92
146.61
146.50
146.48
145.82
145.62
145.56
145.39
145.33
145.29
145.28
145.26
145.02
144.94
144.90
144.81
155.0
148.6
148.0
147.4
146.8
146.2
145.6
145.0
f1 (ppm)
tBuOO
H2
C
N
144.35
144.22
144.16
143.42
143.18
143.03
142.84
142.65
142.59
142.05
142.01
141.80
141.54
141.51
141.48
141.13
140.74
140.53
144.4
144.0
143.6
143.2
142.8
142.4
142.0
141.6
141.2
140.8
140.4
f1 (ppm)
139.32
139.22
139.09
138.03
137.92
137.22
135.79
135.12
134.26
133.85
133.48
133.34
133.29
129.73
127.60
126.21
140
139
138
137
136
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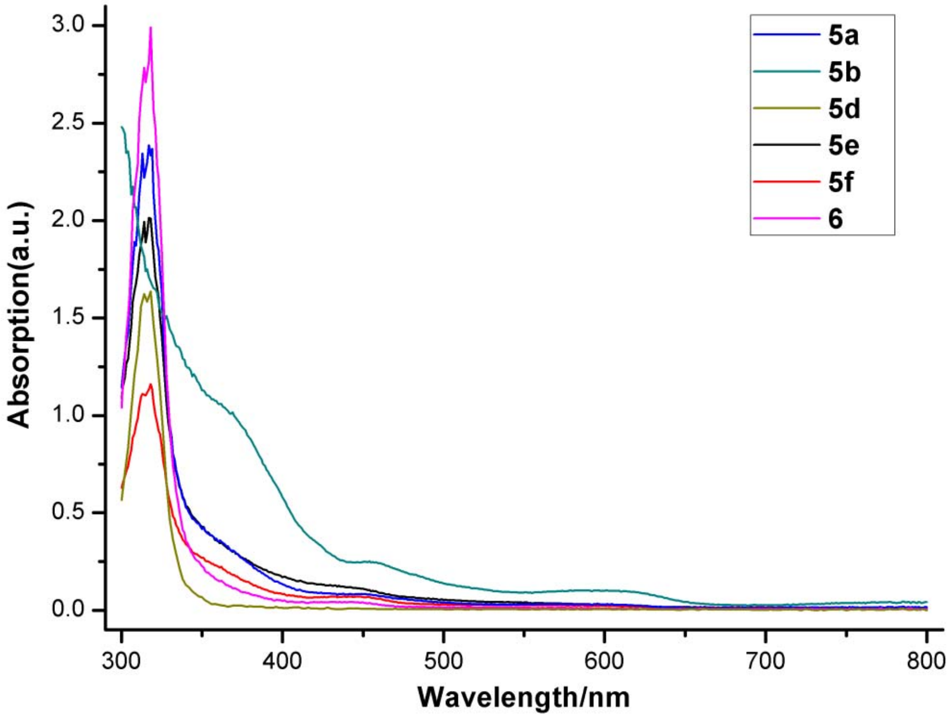
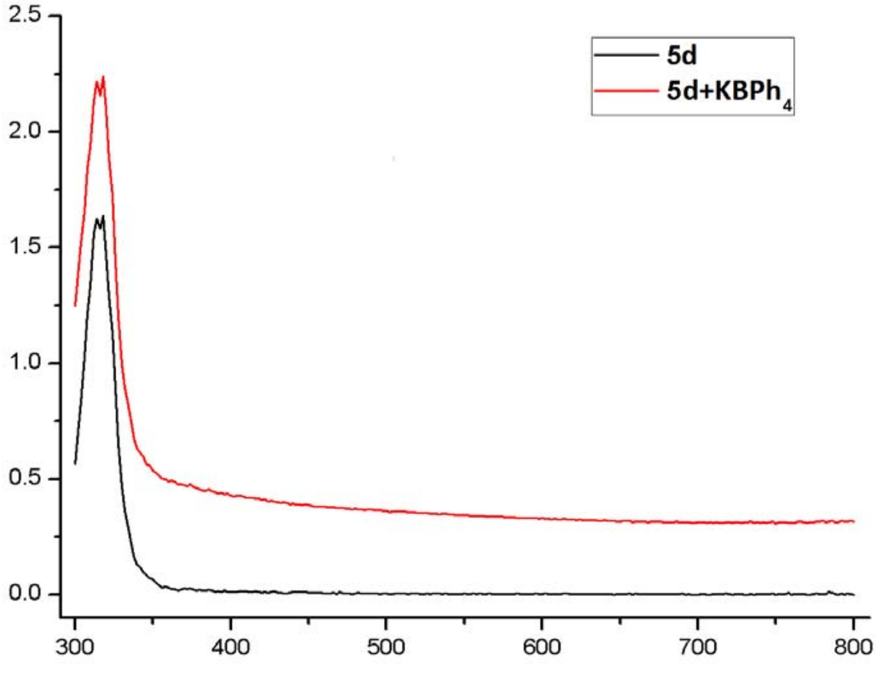
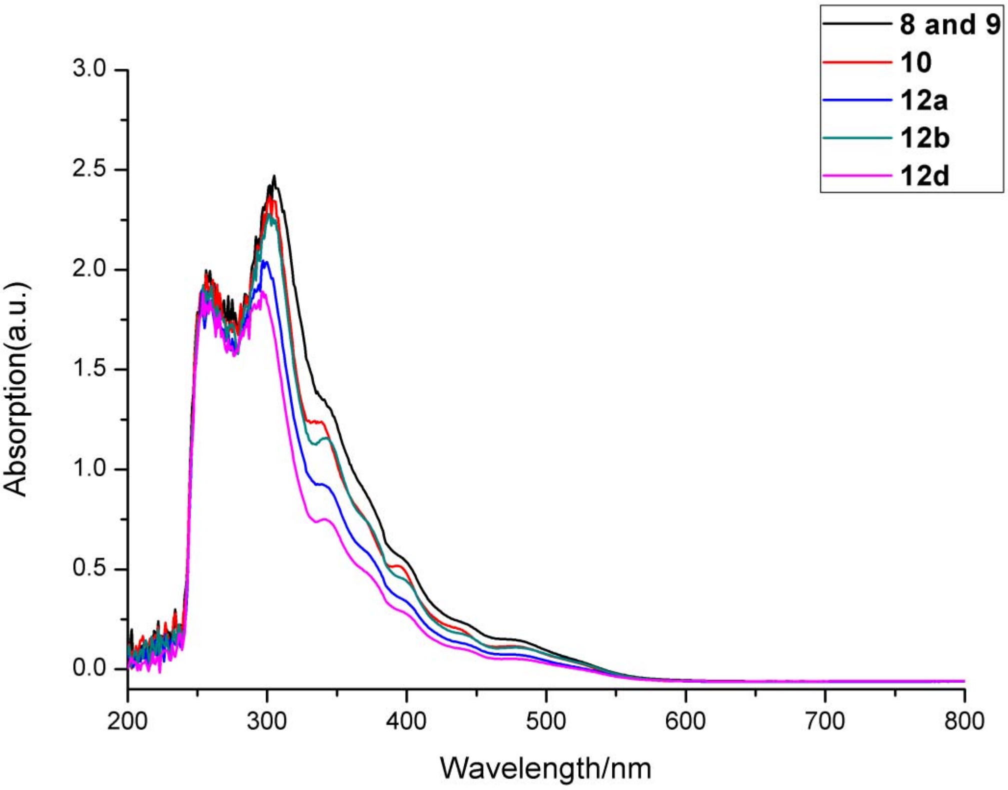
128
127
126
125
124
f1 (ppm)



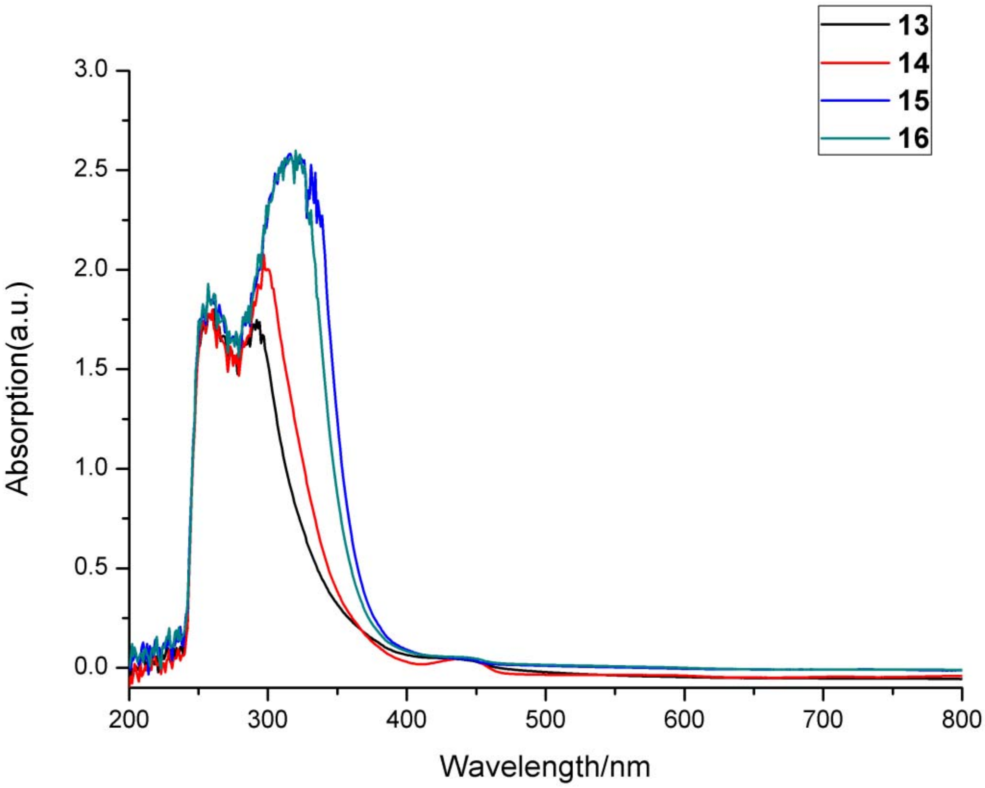
UV-Vis spectra for Compounds **5a**, **5b**, **5d**, **5e**, **5f**, **6** in $CHCl_3$



UV-Vis spectra for Compound **5d** & **5d**+$KBPh_4$ in $CHCl_3$



UV-Vis spectra for Compounds **8** and **9**, **10**, **12a**, **12b**, **12d** in $CHCl_3$



UV-Vis spectra for Compounds **13**, **14**, **15**, **16** in $CHCl_3$