Supplementary Information

Structure-Activity Relationship Studies on 2,5,6-trisubstituted benzimidazoles targeting *Mtb*-FtsZ as antitubercular agents

Krupanandan Haranahalli,[ab] Simon Tong,[b] Saerom Kim,[b] Monaf Awwa,[b] Lei Chen,[b] Susan E. Knudson,[c] Richard A. Slayden,[c] Eric Singleton,[d] Riccardo Russo[d], Nancy Connell,[d,e] and Iwao Ojima*[ab]

[a]*Institute of Chemical Biology & Drug Discovery, Stony Brook University, Stony Brook, NY, USA.*
[b]*Department of Chemistry, Stony Brook University, Stony Brook, NY, USA.*
[c]*Department of Microbiology, Immunology and Pathology, Colorado State University, Fort Collins, Colorado 80523-1682, USA.*
[d]*Department of Medicine, Center for Emerging and Re-emerging Pathogens, Rutgers University, Newark, New Jersey, 07103, USA.*
[e]*Department of Physiology, Rutgers University, Newark, New Jersey, 07103, USA.*

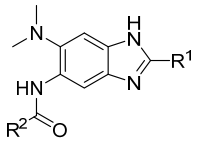
Table of contents

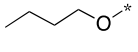
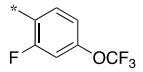
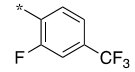
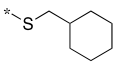
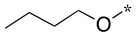
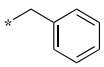
*To whom correspondences should be addressed. Phone 631-632-1339; fax 631-632-7942; e-mail, iwao.ojima@stonybrook.edu

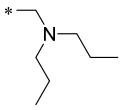
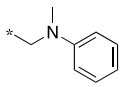
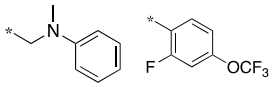
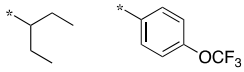
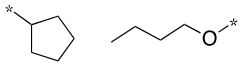
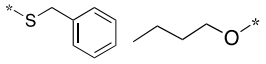
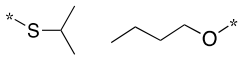
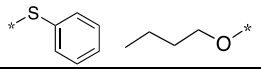
Computational analysis of preliminary pharmacokinetic toxicities and docking studies

i. pkCSM prediction of possible toxicities for top 13 compounds. Top 13 compounds based on the MIC values were analysed by the pkCSM program[28] for their predicted pharmacokinetic and toxicity profiles. Results are summarized in Table S1. For the virtual AMES test, all compounds bearing an aryl group-containing substituent at the 2-position are predicted to be positive, except for **10e-6** and **11e**. For the hERG I toxicity, two lead compounds, SB-P17G-A38 and SB-P-17G-A42, are predicted to be positive, which are 5-(substituted benzamido)benzimidazoles, although **5c-7** and **10e-6** were predicted to be non-toxic for hERG I. All top 14 compounds (entries 4-16) were predicted to be negative. However, all compounds in this table are predicted to be positive against hERG II, as well as hepatotoxicity. Oral toxicity (LD_{50}) of all compounds in this table is predicted to be very low, i.e., 2.3-2.7 mol/kg (320-420 g/kg). For Caco-2 permeability predictions (log P_{app}: >0.9 is considered high permeability), most of the compounds show good permeability, which corresponds to potential oral bioavailability. The log P_{app} value for the most potent compound **20g** is 0.69, which is considered fairly good. For the predictions on their properties with cytochrome P3A4 (CYP3A4), only aromatic group-containing compounds show interaction as substrate and/or inhibitor. Thus, all 6-dimethylaminobenzimidazoles with 5-(alkyl carbamate) and 2-alkylsulfanyl substituents do not have appreciable interactions with CYP3A4.

Table S1 pkCSM prediction of pharmacokinetic and toxicity profiles of top 13 compounds



Entry	Compound	R^1	R^2	MIC[a] (μg/mL)	AMES	hERG1	LD_{50} (mol/Kg)	Caco-2[d]	CYP3A4 inhibitor	CYP3A4 substrate
1	SB-P17G-C2			0.06	No	No	2.55	0.85	No	No
2	SB-P17G-A38			0.16	No	Yes	2.39	0.83	No	Yes
3	SB-P17G-A42			0.16	No	Yes	2.43	0.23	No	Yes
4	**20g**			0.015[c] (0.0039[b])	No	No	2.58	0.69	No	No
5	**20f**			<0.031[c] (<0.0081[b])	No	No	2.59	0.80	No	No
6	**6b**			<0.078	Yes	No	2.30	0.88	Yes	No
7	**6c**			0.078	No	No	2.65	0.89	No	No
8	**11c**			0.14[c] (0.09[b])	No	No	2.60	0.85	No	No

9	**11a**		0.16	No	No	2.74	0.89	No	No
10	**11e**		0.16	No	No	2.31	0.84	Yes	No
11	**10e-6**		0.29[c] (0.19[b])	No	No	2.40	0.83	Yes	Yes
12	**5c-7**		0.31	No	No	2.43	1.14	Yes	Yes
13	**6a**		0.31	No	No	2.57	0.87	No	No
14	**20b**		0.31	Yes	No	2.31	0.75	Yes	No
15	**20c**		0.31	No	No	2.68	0.88	No	No
16	**20e**		0.31	Yes	No	2.27	0.86	No	No

[a,b,c] See the caption in Table 1.
[d] Caco-2 prediction value is in log P_{app} 10^{-6} cm/s

ii. Docking analysis of 20g in the putative binding site on *Mtb*-FtsZ. Next, we performed docking analysis of the most potent compound **20g** by following up Li's *Mtb*-FtsZ homology model for the putative binding site of our 2,5,6-trisubstituted benzimidazoles,[29] using the Autodock 4.2 program. The comparison of docking poses of SB-P17G-C2, SB-P17G-A38 and **20g** based on the energy minimization protocol shows very clear overlap of cyclohexyl moieties in three compounds, and a recognizable deviation of the benzimidazole moiety of **20g** downward (Fig. S1A). However, the docking poses of these compounds based on simple docking are very different from the poses in Fig. S1A, especially **20g** takes a unique pose, illustrated in Fig. S1B and Fig. S1C. In this docking pose, **20g** holds 3 hydrogen bonding interactions with Thr298 and Arg296, as well as vander Waals interactions with Leu189, Val203, Met215 and IIle300. Fig. S1D illustrates a rather narrow and deep cleft, where in the 2-cyclohexymethylsufanyl and 6-dimethylamino groups fit very well and the n-butyl ester moiety is exposed. This docking analysis appears to explain the excellent MIC value (0.0039 μg/mL; normalized value 0.015 μg/mL) of **20g**. The PDB file of Fig. S1B-D is provided as a separate Supplemental Material.

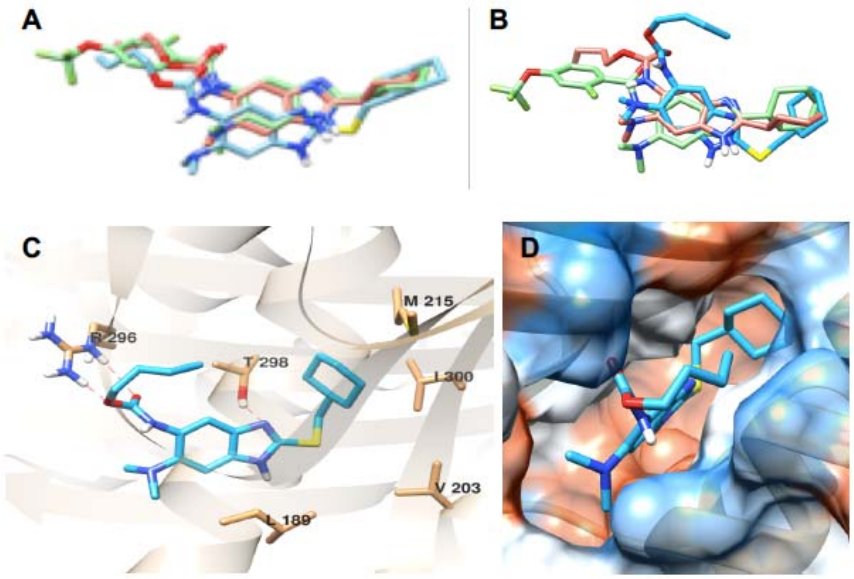


Fig. S1 Molecular docking analysis of **20g** as compared to SB-P17G-C2 and SB-P17G-A38. (A) Overlay of **20g**, SB-P17G-C2 and SB-P17G-A38 based on energy-minimization method; (B) Overlay of **20g**, SB-P17G-C2 and SB-P17G-A38 based on molecular docking method; (C) Polar and hydrophobic interactions of **20g** with Mtb-FtsZ; (D) Protein surface interaction with **20g** at the binding cleft.

Synthetic procedures and characterization data for intermediates

1-(Cyclopentanecarboxamido)-5-dimethylamino-2,4-dinitrobenzene (3a). A solution of **1.1** (1 g, 4.42 mmol) and cyclopentanecarbonyl chloride (5.30 mmol) in pyridine (4 mL) was refluxed overnight. After the starting material disappeared (TLC analysis), the reaction mixture was concentrated under reduced pressure to afford a brown residue. The brown residue was triturated in methanol to give **3a** (0.99 g, 70% yield) as yellow solid: mp 118-120 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.62-1.71 (m, 2 H), 1.74- 1.81 (m, 2 H), 1.82-1.93 (m, 2 H), 1.99-2.07 (m, 2 H), 2.81- 2.90 (quintet, *J* = 8.1 Hz, 1 H), 3.04 (s, 6 H), 8.57 (s, 1 H), 8.83 (s, 1 H), 10.96 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 30.4, 42.7, 48.2, 106.2, 125.2, 127.7, 131.6, 138.9, 150.4, 176.4; LRMS (ESI) *m/z* calculated for $C_{14}H_{18}N_4O_5$: 322.1, found: 323.1 $(M+1)^+$.

The same procedure as that for **3a** was used for the synthesis of compounds **3b**-**3g**.

5-Dimethylamino-2,4-dinitro-1-(phenylacetamido)benzene (3b). Yellow solid; 55% yield; mp 125-128 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.02 (s, 6 H), 3.82 (s, 2 H), 7.33-7.37 (m, 3 H), 7.37-7.43 (m, 2 H), 8.53 (s, 1 H), 8.75 (s, 1 H), 10.82 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.7, 46.1, 106.2, 125.1, 127.5, 128.2, 129.5, 129.7, 131.6, 133.0, 138.4, 150.2, 171.1; LRMS (ESI) *m/z* calculated for $C_{16}H_{16}N_4O_5$: 344.1, found: 345.1 $(M+1)^+$.

5-Dimethylamino-1-(2-ethylbutanamido)-2,4-dinitrobenzene (3c). Yellow solid; 92% yield; mp 98-100 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.97 (t, *J* = 7.4 Hz, 6 H), 1.58-1.79 (m, 4 H), 2.19-2.27 (m, 1 H), 3.06 (s, 6 H), 8.63 (s, 1 H), 8.85 (s, 1 H), 10.96 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.1, 25.8, 42.7, 53.6, 106.3, 125.2, 127.7, 131.7, 138.7, 150.4, 176.3; LRMS (ESI) *m/z* calculated for $C_{14}H_{20}N_4O_5$: 324.2, found: 325.2 $(M+1)^+$.

5-Dimethylamino1-(2-methylpropanamido)-2,4-dinitrobenzene (3d). Yellow solid; 74% yield; mp 105-107 ºC; 1H NMR (400 MHz, $CDCl_3$) δ 1.30 (d, *J* = 7.0 Hz, 6 H), 2.62-2.72 (m, 1 H), 3.05 (s, 6 H), 8.57 (s, 1 H), 8.78 (s, 1 H), 10.99 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.4, 38.0, 42.7, 106.2, 125.2, 127.6, 131.6, 138.8, 150.3, 177.0; LRMS (ESI) *m/z* calculated for $C_{12}H_{16}N_4O_5$: 296.1, found: 297.2 $(M+1)^+$.

1-Benzamido-5-dimethylamino-2,4-dinitrobenzene (3e). Yellow solid;96% yield; mp 190-193 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.1 (s, 6 H), 7.52-7.56 (m, 2 H), 7.60-7.64 (m, 1 H), 7.96-7.99 (m, 2 H), 8.75 (s, 1 H), 8.87 (s, 1 H), 11.88 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.8, 106.2, 125.4, 127.5, 127.8, 129.3, 131.8, 133.2, 133.7, 139.0, 150.4, 166.3; LRMS (ESI) *m/z* calculated for $C_{15}H_{14}N_4O_5$: 330.1, found: 331.1 $(M+1)^+$.

5-Dimethylamino-2,4-dinitro-1-(thiophene-2-carboxamido)benzene (3f). Yellow solid; 91% yield; mp 212-215 ºC; 1H NMR (400 MHz, $CDCl_3$) δ 3.08 (s, 6 H), 7.18-7.20 (m, 1 H), 7.67 (s, 1 H), 7.76 (s 1 H), 8.64 (s, 1 H), 8.87 (s, 1 H), 11.82 (s, 1 H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 42.8, 106.1, 125.2, 127.8, 128.6, 129.6, 131.8, 133.1, 138.8, 138.9, 150.3, 160.9; LRMS (ESI) *m/z* calculated for $C_{13}H_{12}N_4O_5S$: 336.1, found: 337.1 $(M+1)^+$.

1-(Furan-2-carboxamido)-5-dimethylamino-2,4-dinitrobenzene (3g). Yellow solid; 81% yield; mp 185-188 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.07 (s, 6 H), 6.61 (d, *J* = 5.3 Hz, 1 H), 7.30 (d, *J* = 5.3 Hz, 1 H), 7.63 (s, 1 H), 8.65 (s, 1 H), 8.85 (s, 1 H), 11.88 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.7, 106.3, 113.1, 117.1, 125.3, 127.7, 131.7, 138.4, 146.0, 147.1, 150.2, 157.0; LRMS (ESI) *m/z* calculated for $C_{13}H_{12}N_4O_6$: 320.1, found: 321.1 $(M+1)^+$.

5-Dimethylamino-2,4-dinitro-1-(tetrahydropyran-4-carboxamido)benzene (3h). To a solution of **2** (0.44 mmol) in acetonitrile was added tetrahydropyran-4-carboxylic acid (3 equiv.) and phosphorus trichloride (3 equiv.). The reaction mixture was refluxed overnight. After the completion of the reaction, solvent from the reaction mixture was evaporated under vacuum to give the crude reaction mixture, which was washed with sodium bicarbonate (30 mL x 3) and water (30 mL x 3). The product was extracted with ethyl acetate (30 mL x 3) and dichloromethane (DCM) (30 mL x 3). The organic layers were combined and dried over anhydrous $MgSO_4$, filtered and concentrated by a rotary evaporator to give the crude product. Purification of the crude product by flash chromatography on silica gel using DCM and hexanes (9:1), followed by 100 % ethyl acetate as eluent afforded **3h** as a yellow solid (71 mg, 47 % yield): ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.79 – 1.93 (m, 4 H), 2.61 – 2.67 (m, 1 H), 3.06 (s, 6 H), 3.46 – 3.51 (m, 2 H), 4.05 – 4.09 (m, 2 H), 8.56 (s, 1 H), 8.83 (s, 1 H), 11.06 (s, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 29.1, 42.8, 44.2, 67.2, 106.4, 127.7, 138.7, 150.4, 174.3; LRMS (ESI) *m/z* calculated for $C_{14}H_{18}N_4O_6$: 338.1, found: 339.1 $(M+1)^+$.

5-Amino-6-dimethylamino-2-cyclopentyl-1H-benzo[d]imidazole (4a). To a solution of **3a** (1.4 g, 4.5 mmol) in ethanol (25 mL), solid stannous chloride dihydrate ($SnCl_2.2H_2O$) (7.1 g, 31.6 mmol) was added. Concentrated hydrochloric acid (18 mL) was added to the reaction mixture such that the final concentration of HCl was 4 N in the reaction flask. The reaction mixture was refluxed for 4 h. Upon completion, the reaction mixture was basified with 30% sodium hydroxide solution. Excess stannous chloride formed a soluble salt in presence of excess base. It was then extracted with dichloromethane (30 mL x 3), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to give a dark brown oil. The brown oil was purified by flash chromatography on alumina using 100% ethyl acetate as eluent to afford **4a** as a pale brown solid: 87% yield; mp 100-103 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.54-1.64 (m, 2 H), 1.68-1.79 (m, 2 H), 1.86-1.95 (m, 2 H), 2.04- 2.12 (m, 2 H), 2.61 (s, 6 H), 3.19- 3.27 (m, 1 H), 6.78 (s, 1 H), 7.22 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.5, 32.4, 39.6, 44.6, 98.5, 106.5, 132.9, 134.2, 138.0, 138.2, 157.2; HRMS (ESI) m/z calcd for $C_{14}H_{20}N_4H^+$: 245.1687, Found: 245.1759 (Δ = 0.38 ppm).

The same procedure as that for **4a** was used for the synthesis and characterization of compounds **4b**-**4h** and **9a-9e.**

5-Amino-6-dimethylamino-2-benzyl-1H-benzo[d]imidazole (4b). Brown Solid; 59% yield; 80-83 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.61 (s, 6 H), 4.09 (s, 2 H), 6.68 (s, 1 H), 7.16-7.24 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 35.6, 44.5, 98.7, 106.6, 126.9, 128.7, 128.9, 133.1, 134.6, 137.0, 138.1, 138.4, 151.8; HRMS (ESI) m/z calcd for $C_{16}H_{18}N_4H^+$: 267.1527, Found: 267.1600 (Δ = 1.73 ppm).

5-Amino-6-dimethylamino-2-(pentan-3-yl)-1H-benzo[d]imidazole (4c). Brown solid; 87 % yield; mp 80-82 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.4 Hz, 6 H), 1.71-1.89 (m, 4 H), 2.62 (s, 6 H), 2.71-2.79 (m, 1 H), 6.81 (s, 1 H), 7.23 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.3, 27.7, 44.1, 44.6, 98.7, 106.5, 133.1, 134.4, 137.8, 138.0, 157.1; HRMS (ESI) m/z calcd for $C_{14}H_{22}N_4H^+$: 247.1845, Found: 247.1918 (Δ = -0.35 ppm).

5-Amino-6-dimethylamino-2-isopropyl-1H-benzo[d]imidazole (4d). Brown solid; 69% yield; mp 83-85 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.38 (d, *J* = 7.0 Hz, 6 H), 2.62 (s, 6 H), 3.11-3.22 (m, 1 H), 6.79 (s, 1 H), 7.23 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 29.1, 44.7, 98.7, 106.8, 133.1, 134.3, 138.1, 138.3, 158.8; HRMS (ESI) m/z calcd for $C_{12}H_{18}N_4H^+$: 219.1530, Found: 219.1603 (Δ = 0.68 ppm).

5-Amino-6-dimethylamino-2-phenyl-1H-benzo[d]imidazole (4e). Brown solid; 96% yield; mp 90-93 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.57 (s, 6 H), 6.77 (s, 1 H), 7.22 (s, 1 H), 7.29-7.33 (m, 3 H), 8.06-8.09 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 44.4, 98.9, 107.4, 126.4, 128.9, 129.3, 130.4, 134.5, 138.8, 139.1, 150.6, 171.3; HRMS (ESI) m/z calcd for $C_{15}H_{16}N_4H^+$: 253.1376, Found: 253.1449 (Δ = -0.47 ppm).

5-Amino-6-dimethylamino-2-(thiophen-2-yl)-1H-benzo[d]imidazole (4f). Brown solid; 87% yield; mp 100-102 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.58 (s, 6 H), 6.77 (s, 1 H), 6.94-6.96 (m, 1 H), 7.21 (s, 1 H), 7.23-7.25 (m, 1 H), 7.62-7.63 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 44.4, 98.3, 107.0, 125.8, 127.0, 127.9, 133.9, 134.9, 138.9, 139.1, 146.1; HRMS (ESI) m/z calcd for $C_{13}H_{14}N_4SH^+$: 259.0941, Found: 259.1013 (Δ = -0.89 ppm).

5-Amino-6-dimethylamino-2-(furan-2-yl)-1H-benzo[d]imidazole (4g). Yellow solid; 99% yield; mp 92-95 ºC; 1H NMR (400 MHz, $CDCl_3$) δ 2.62 (s, 6 H), 6.42-6.43 (m, 1 H), 6.80 (s, 1 H), 7.02 (d, , *J* = 4 Hz , 1 H), 7.25 (s, 1 H), 7.37-7.38 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 44.4, 98.4, 107.2, 109.2, 112.1, 134.3, 139.9, 139.2, 142.6, 143.0, 145.9; HRMS (ESI) m/z calcd for $C_{13}H_{14}N_4OH^+$: 243.1159, Found: 243.1232 (Δ = 3.44 ppm).

5-Amino-6-dimethylamino-2-tetrahydropyranyl-1H-benzo[d]imidazole (4h). Brown solid; 91% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.94 – 2.01 (m, 4 H), 2.66 (s, 6 H), 3.02 – 3.14 (m, 1 H), 3.48 – 3.56 (m, 2 H), 4.04 – 4.08 (m, 2 H), 6.82 (s, 1 H), 7.29 (s ,1 H); HRMS (ESI) m/z calcd for $C_{14}H_{20}N_4OH^+$: 261.171, Found: 261.1708 (Δ = 0.69 ppm).

Chloroacetamido-5-dimethylamino-2,4-dinitrobenzene (7). To a solution of **2** (1.00 g, 3.31 mmol.) in DCM (20 mL) was added chloroacetyl chloride (315 µL, 3.96 mmol.) and triethylamine (TEA) (553 µL, 3.96 mmol). The reaction mixture was refluxed overnight. Upon confirming the full consumption of the starting material **2** by TLC, the reaction mixture was diluted with DCM (30 mL), washed thrice with water (50 mL), dried over anhydrous magnesium sulfate, filtered and concentrated under reduced pressure to give a crude product. The crude product was purified by flash column chromatography on silica gel with 1:3 AcOEt: hexanes as the eluent to give **7** (1.24 g, 93% yield) as a yellow solid: mp 152 - 153 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.07 (s, 6 H), 4.26 (s, 2 H), 8.48 (s, 1 H), 8.81 (s, 1 H), 11.83 (bs, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 42.5, 43.2, 106.2, 125.1, 127.3, 131.7, 137.2, 149.8, 166.0; HRMS (ESI-TOF) *m/z* calcd for $C_{10}H_{11}ClN_4O_5H^+$: 303.0491, found 303.0490 (Δ 0.26 ppm).

5-Dimethylamino-2,4-dinitro-1-*N*,*N*-dipropylaminomethylcarboxamidobenzene (8a). To a solution of chloroacetamido-5-dipropylamino-2,4-dinitrobenzene in THF (10 mL) was added *N*,*N*-diisopropylethylamine (DIPEA) (0.35 mL, 2.0 mmol) and 2 M dimethylamine in THF (0.35 mL, 2.0 mmol). The mixture was stirred under reflux overnight. The reaction mixture was evaporated to get yellow residue. The residue was dissolved in DCM, washed three times with water (30 mL), dried over anhydrous magnesium sulfate and filtered. The filtrate was concentrated under reduced pressure to give a crude product. The crude product was purified using flash column chromatography on silica gel, using AcOEt:hexanes (1:1) as eluent to give **8a** (0.3 g, 71% yield) as a yellow solid: mp 94 - 97 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.86 (t, *J* = 7.3 Hz, 6 H), 1.46-1.57 (m, 4 H), 2.49-2.55 (, 4 H), 3.03 (s, 6 H), 3.25 (s, 2 H), 8.61 (s, 1 H), 8.82 (s, 1 H), 12.31 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 11.6, 20.3, 42.4, 57.7, 59.9, 106.1, 125.2, 127.3, 131.2, 137.9, 149.9, 173.1; HRMS (ESI-TOF) *m/z* calculated for $C_{16}H_{25}N_5O_5H^+$: 368.1928, found 368.1934 (Δ -1.41 ppm).

Compounds **8b-8e** were synthesized using the same procedure as that for **8a**.

5-Dimethylamino-2,4-dinitro-1-(morphorlin-4-y)lacetamidobenzene (8b). Yellow solid; 78% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.52 (br. s., 4 H), 2.90 (s, 6H), 3.09 (s, 2 H), 3.67 (bs, 4 H), 8.36 (s, 1 H), 8.51 (s, 1 H), 12.11 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 42.0, 53.3, 62.2, 66.2, 105.3, 124.4, 126.7, 130.7, 137.3, 149.3, 170.4; HRMS (ESI-TOF) m/z calculated for $C_{14}H_{19}N_5O_6H^+$: 354.1408, found 354.1412 (Δ -1.11 ppm).

5-Dimethylamino-2,4-dinitro-1-(piperadin-1-yl)methylcarboxamidobenzene (8c). Yellow solid; 93% Yield; mp 152-154 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.45 (bs, 2 H), 1.66-1.70 (m, 4 H), 2.52 (bs, 4 H), 3.01 (s, 6 H), 3.12 (s, 2 H), 8.57 (s, 1 H), 8.77 (s, 1 H), 12.34 (bs, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.4, 25.7, 42.4, 54.9, 63.2, 105.9, 125.1, 127.2, 131.1, 137.9, 149.8, 171.9; HRMS (ESI-TOF) m/z calculated for $C_{15}H_{21}N_5O_5H^+$: 352.1615, found 352.1622 (Δ -1.77 ppm).

5-Dimethylamino-2,4-dinitro-1-(pyrrolidin-1-yl)methylcarboxamidobenzene (8d). Yellow solid; 79% yield; mp 94-95 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.83 (bs, 4 H), 2.66 (bs, 4 H), 2.97 (s, 6 H), 3.30 (s, 2 H), 8.45 (s, 1 H), 8.64 (s, 1 H), 12.18 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.8, 42.1, 54.0, 59.1, 105.5, 124.7, 126.9, 130.8, 137.6, 149.5, 171.5; HRMS (ESI-TOF) m/z calculated for $C_{14}H_{19}N_5O_5H^+$: 338.1459, found 338.1466 (Δ -2.07 ppm).

5-Dimethylamino-1-(*N*-methyl-*N*-phenylamino)methylcarboxamido-2,4-dinitrobenzene (**8e**). Yellow solid; 65% yield; mp 142-144 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.06 (s, 6 H), 3.18 (s, 3 H), 4.05 (s, 2 H), 6.78 - 6.83 (m, 2 H), 6.86 (s, 1 H), 7.23 - 7.30 (m, 2 H), 8.61 (s, 1 H), 8.78 (s, 1 H), 11.94 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 40.4, 42.5, 59.9, 106.1, 113.5, 119.2, 125.1, 127.3, 129.4, 131.5, 137.7, 148.7, 150.0, 171.4; HRMS (ESI-TOF) m/z calculated for $C_{17}H_{19}N_5O_5H^+$: 374.1459, found 374.1463 (Δ -1.12 ppm).

5-Amino-6-dimethylamino-2-*N,N*-dipropylaminomethyl-1H-benzo[d]imidazole (9a). Brown solid; 45% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6 Hz, 6 H), 1.52-1.59 (m, 4 H), 2.54-2.58 (m, 4 H), 2.70 (s, 6 H), 3.92 (s, 2 H), 6.86 (s, 1 H), 7.28 (s, 1 H); LRMS (ESI) *m/z* calculated for $C_{16}H_{27}N_5$: 289.2, found: 290.2 $(M+1)^+$.

5-Amino-6-dimethylamino-2-morpholin-4-ylmethyl-1H-benzo[d]imidazole (9b). Brown solid; 28% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.48-2.55 (m, 4 H), 2.68 (s, 6 H), 3.65-3.72 (m, 4 H), 3.74 (s, 2 H), 6.82 (s, 1 H), 7.28 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 44.5, 53.7, 56.8, 66.8, 77.2, 98.3, 107.1, 132.9, 133.9, 138.5, 149.6; LRMS (ESI) *m/z* calculated for $C_{14}H_{21}N_5O$: 275.2, found: 276.2 $(M+1)^+$.

5-Amino-6-dimethylamino-2-piperadin-1-ylmethyl-1H-benzo[d]imidazole (9c). Brown solid; 91% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.44-1.46 (m, 2 H), 1.54-1.61(m, 4 H), 2.45 (s, 4 H), 2.68 (s, 6 H), 3.68 (s, 2 H), 6.75 (s, 1 H), 7.35 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.6, 25.5, 44.3, 54.3, 56.9, 60.1, 98.3, 106.7, 133.0, 134.2, 137.9, 138.0, 150.2; LRMS (ESI) *m/z* calculated for $C_{15}H_{23}N_5$: 273.2, found: 274.2 $(M+1)^+$.

5-Amino-6-dimethylamino-2-pyrrolidin-1-ylmethyl-1H-benzo[d]imidazole (9d). Brown solid; 45 % yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.77-1.80 (m, 4 H), 2.59 - 2.70 (m, 10 H), 3.88 (s, 2 H), 6.74 (s, 1H), 7.23 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.5, 44.5, 53.8, 54.2, 77.2, 98.2, 107.1, 133.4, 134.0, 138.3, 150.6; LRMS (ESI) *m/z* calculated for $C_{14}H_{21}N_5$: 259.2, found: 260.2 $(M+1)^+$.

5-Amino-6-dimethylamino-2-(*N*-methyl-*N*-phenylamino)methyl-1H-benzo[d]imidazole (9e). Brown solid; 65% yield; mp 120-122 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.68 (s, 6 H), 3.03 (s, 3 H), 4.03 (bs, 2 H), 4.65 (s, 2 H), 6.79 - 6.83 (m, 4 H), 7.23 - 7.29 (m, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 39.4, 44.6, 52.2, 76.9, 98.6, 107.1, 113.1, 117.9, 129.4, 133.8, 138.3, 138.6, 149.5, 151.1; HRMS (ESI-TOF) *m/z* calculated for $C_{17}H_{21}N_5H^+$: 296.187, found 296.1868 (Δ 0.54 ppm).

4-Dimethylamino-1,2-di(4-methylbenzenesulfonylamino)-5-nitrobenzene (15). To a solution of 4-fluoro-5-nitro-1,2-di(4-methylbenzenesulfonylamino)benzene (100 mg, 0.21 mmol) and diisopropylethylamine (DIPEA) (0.23 mmol) in THF (1.0 mL) was added a solution of 2 M dimethylamine in THF (115 μL). The mixture was stirred at room temperature overnight. The reaction mixture was diluted with ethyl acetate, washed with water, dried over anhydrous magnesium sulfate and filtered. The filtrate was concentrated under reduced pressure to give a crude product. The crude product was purified on column chromatography on silica gel using 1:1 AcOEt: hexanes as eluent to give **15** (735 mg, 70% yield) as an amber solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.40 (s, 6 H), 2.78 (s, 6 H), 7.01 (s, 1 H), 7.07 (s, 1 H), 7.36 (d, *J* = 10.0 Hz, 2 H), 7.41 (d, *J* = 10.0 Hz, 2 H), 7.56 (d, *J* = 10.0 Hz, 2 H), 7.85 (d, *J* = 10.0 Hz, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.6, 42.4, 106.9, 113.8, 127.7, 127.7, 128.1, 129.8, 129.9, 132.4, 133.9, 135.6, 140.2, 144.7, 145.0, 146.9; LRMS (ESI) *m/z* calculated for $C_{22}H_{24}N_4O_6S_2$: 504.1, found: 505.1 $(M+1)^+$.

1,2-Diamino-4-dimethylamino-5-nitrobenzene (16). To a solution of 1,2-diamino-4-fluoro-5-nitrobenzene (50 mg, 0.29 mmol.), dissolved in anhydrous *N,N*-dimethylformamide (DMF) (10 mL), was added dimethylamine solution (2 M in THF) (1.5 mL, 3.0 mmol.). The mixture was heated to 120 ºC for 1 h in a sealed tube. Upon completion, the reaction mixture was diluted with ethyl acetate and the washed with 5 % aqueous lithium chloride solution three times. The organic layer was collected, dried over magnesium sulfate, and evaporated on a rotary evaporator. The resulting crude product was purified by column chromatography on alumina, using ethyl acetate as the eluent to give **16** as a red solid (19 mg, 98% yield) (This reaction was also performed in the microwave, and the reaction went to completion in 30 min.): ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.79 (s, 6 H), 3.16 (bs, 2 H), 4.26 (bs, 2 H), 6.26 (s, 1 H), 7.52 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 43.6, 103.1, 116.8, 125.5, 130.7, 144.3, 145.5. LRMS (ESI) *m/z* calculated for $C_8H_{12}N_4O_2$: 196.1, found: 197.1 $(M+1)^+$.

6-Dimethylamino-5-nitro-2-sulfhydryl-1H-benzo[d]imidazole (17). To a solution of **16** (1.0 g, 5.09 mmol.) in THF (10 mL) and methanol (10 mL) was added carbon disulfide (776 mg, 10.19 mmol.) and TEA (1.03g, 10.19 mmol). The reaction mixture was heated to 50 ºC for 4 h. The reaction was complete when a red solid crashed out of the solution and the reaction mixture was clear and colorless. The reaction mixture was monitored by FIA Mass to ensure all the starting material was consumed. The solvent was evaporated by a rotary evaporator to give a crude product as a red solid. The crude product was washed with water (20 mL), filtered, and washed further with water (20 mL x3). After the water washes, the solid was rinsed with methanol (10 mL) and allowed to dry in air overnight, and under vacuum for 4 h to give **17** (0.92 g, 76% yield) as a red solid: ^{1}H NMR (500 MHz, DMSO-*d*$_6$) δ 6.55 (s, 1 H), 7.31 (s, 1 H), 12.40 (bs, 1H), 12.48 (bs, 1H); ^{13}C NMR (126 MHz, DMSO-*d*$_6$) δ 43.3, 98.7, 106.7, 125.4, 136.1, 136.8, 144.2, 171.2; LRMS (ESI) *m/z* calculated for $C_9H_{10}N_4O_2S$: 238.1, found: 239.1 $(M+1)^+$.

6-Dimethylamino-2-(methylsulfanyl)-5-nitro-1H-benzo[d]imidazole (18a). To compound **17** (500 mg, 2.1 mmol.) was added water (5 mL), ethanol (5 mL), and potassium hydroxide (318 mg, 5.67 mmol.) to form a reddish-black solution. Methyl iodide (301 mg, 2.12

mmol.) was added to this solution, and the reaction mixture was allowed to stir at room temperature overnight. The reaction was monitored by FIA Mass for the consumption of the starting material. The reaction mixture was diluted with water (30 mL) and extracted with DCM (30 mL x 3), dried over anhydrous magnesium sulfate and filtered. The filtrate was concentrated under reduced pressure to give a crude product. The crude product was purified by flash column chromatography on silica gel, using 1:1 AcOEt:hexanes as eluent to give **18a** (445 mg, 84% yield) as a red solid: ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.60 (s, 6 H), 2.66 (s, 3 H), 6.79 (s, 1 H), 7.22 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 15.2, 44.4, 98.4, 105.8, 134.0, 135.6, 137.7, 138.1, 149.2; HRMS (ESI-TOF) *m/z* calculated for $C_{10}H_{12}N_4O_2SH^+$: 253.0754, found 253.0756 (Δ -1.01 ppm).

In a similar manner, compound **18b-18d** and **18f** were synthesized and characterized.

2-(Benzylsulfanyl)-6-*N,N*-dimethylamino-5-nitro-1H-benzo[d]imidazole (18b). Red solid; 83% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.81 (s, 6 H), 4.54 (s, 2 H), 7.12 (s, 1 H), 7.23-7.37 (m, 5 H), 8.05 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 37.11, 44.01, 102.6, 113.4, 127.9, 128.8, 128.9, 136.1, 138.4, 144.2, 154.3, 155.9; HRMS (ESI-TOF) *m/z* calculated for $C_{16}H_{16}N_4O_2SH^+$: 329.0994, found 329.1067 (Δ -1.46 ppm).

6-Dimethylamino-2-(isopropylsulfanyl)-5-nitro-1H-benzo[d]imidazole (18c). Red solid; 58% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.41 (d, J = 6.8 Hz, 6 H), 2.81 (s, 6 H), 4.01 (septet, J = 6.8 Hz, 1 H), 7.17 (s, 1 H), 8.11 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.65, 39.12, 44.25, 103.2, 113.4, 134.0, 138.6, 143.0, 144.3, 154.4; LRMS (ESI) *m/z* calculated for $C_{12}H_{16}N_4O_2S$: 280.1, found: 281.1 $(M+1)^+$.

6-Dimethylamino-2-(ethylsulfanyl)-5-nitro-1H-benzo[d]imidazole (18d). Red solid; 69% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.42 (t, J = 7.4 Hz, 3 H), 2.80 (s, 6 H), 3.31 (q, J = 7.4 Hz, 2 H), 7.12 (s, 1 H), 8.04 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.92, 26.96, 44.10, 102.9, 112.9, 133.5, 138.2, 142.8, 144.2, 155.2; HRMS (ESI-TOF) *m/z* calculated for $C_{11}H_{14}N_4O_2SH^+$: 267.091, found 267.0916 (Δ -1.99 ppm).

6-Dimethylamino-5-nitro-2-(phenylsulfanyl)-1H-benzo[d]imidazole (18e). To a solution of **17** (100 mg, 0.4 mmol) in DMF (2 mL) was added iodobenzene (81.6 mg, 0.4 mmol.), cuprous iodide (7.6 mg, 0.04 mmol.), 1,10-phenanthroline (7.2 mg, 0.04 mmol.), and potassium carbonate (110 mg, 0.8 mmol.). The reaction mixture was heated in a pressure vessel for 22 h at 140 °C. The reaction was monitored by FIA Mass. Upon completion of the reaction, the reaction mixture was diluted with ethyl acetate (40 mL) and washed thrice with brine (80 mL) and thrice with water (80 mL), dried over anhydrous magnesium sulfate, and filtered. The filtrate was concentrated under reduced pressure to give a crude product. The crude product was purified by flash column chromatography on silica gel using 1:3 AcOEt: hexanes as eluent to give **18e** (41 mg, 31% yield) as a red solid: ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.79 (s, 6 H), 7.05 (s, 1 H), 7.27-7.29 (m, 3 H), 7.45-7.54 (m, 2 H), 7.89 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 44.12, 100.4, 116.8, 128.7, 129.7, 129.8, 130.1, 133.8, 134.9, 139.0, 144.3; LRMS (ESI) *m/z* calculated for $C_{15}H_{14}N_4O_2S$: 314.1, found: 315.1 $(M+1)^+$.

2-(Cyclohexylsulfanyl)-6-dimethylamino-5-nitro-1H-benzo[d]imidazole (18f). Red solid; 58% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.21 - 1.44 (m, 3 H), 1.46 - 1.65 (m, 3 H), 1.73-1.75 (m, 2 H), 2.04 - 2.18 (m, 2 H), 2.83 (s, 6 H), 3.73 - 3.96 (m, 1 H), 7.16 (bs, 1 H), 8.07 (bs, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.3, 25.6, 33.4, 44.0, 46.6, 102.6, 113.2, 133.7, 138.3, 142.4, 144.1, 154.3; HRMS (ESI-TOF) *m/z* calculated for $C_{15}H_{20}N_4O_2SH^+$: 321.138, found 321.1385 (Δ -1.57 ppm).

5-Amino-6-dimethylamino-2-(methylsulfanyl)-1H-benzo[d]imidazole (19a). To a solution of **18a** (444 mg 1.8 mmol.), dissolved in ethanol (10 mL), was added solid stannous chloride dihydrate (1.42 g, 6.3 mmol.). The reaction mixture was refluxed for 3 h. The reaction was monitored by TLC for the consumption of the starting material. After completion of the reaction, the reaction mixture was diluted with water (100 mL), and solid sodium carbonate was added until the solution became basic. The reaction mixture was extracted thrice with DCM (50 mL), dried over anhydrous magnesium sulfate, and filtered. The filtrate was concentrated under reduced pressure to give a crude product. The crude product was purified by flash chromatography on alumina using 1.5% methanol/DCM as eluent to give **19a** (316 mg, 81% yield) as a beige solid: ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.60 (s, 6 H), 2.66 (s, 3 H), 6.79 (s, 1 H), 7.22 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 15.2, 44.4, 98.4, 105.8, 134.0, 135.6, 137.7, 138.1, 149.2; LRMS (ESI) *m/z* calculated for $C_{10}H_{14}N_4S$: 222.1, found: 223.1 $(M+1)^+$.

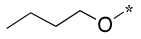
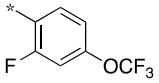
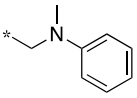
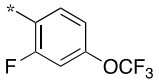
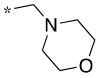
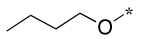
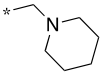
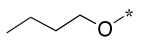
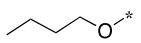
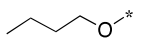
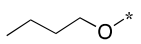
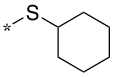
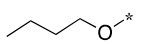
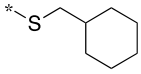
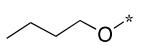
In a similar manner, **19b, 19c** and **19d** were synthesized and characterized.

5-Amino-2-(benzylsulfanyl)-6-dimethylamino-1H-benzo[d]imidazole (19b). Beige solid; 87% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.59 (s, 6 H), 4.41 (s, 2 H), 6.78 (s, 1 H), 7.15-7.23 (m, 5 H), 7.28 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 37.9, 44.3, 98.3, 106.1, 127.2, 128.4, 128.7, 133.8, 135.2, 136.7, 138.0, 138.4, 146.7; LRMS (ESI) *m/z* calculated for $C_{16}H_{18}N_4S$: 298.1, found: 299.1$(M+1)^+$.

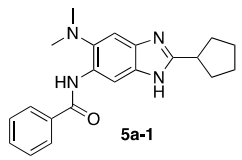
5-Amino-6-dimethylamino-2-(isopropylsulfanyl)-1H-benzo[d]imidazole (19c). Beige solid; 87% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.32 (d, J = 5.2 Hz, 6 H), 2.63 (s, 6 H), 3.87-3.89 (m, 1 H), 6.86 (s, 1 H), 7.33 (s, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.4, 38.9, 44.5, 98.4, 106.3, 134.1, 135.5, 138.0, 138.4, 146.4; LRMS (ESI) *m/z* calculated for $C_{12}H_{18}N_4S$: 250.1, found: 251.1 $(M+1)^+$.

5-Amino-6-dimethylamino-2-(ethylsulfanyl)-1H-benzo[d]imidazole (19d). Beige solid; 89% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.35 (t, J = 7.4 Hz, 3 H), 2.63 (s, 6 H), 3.22 (q, J = 7.4 Hz, 2 H), 6.84 (s, 1 H), 7.29 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ14.9, 27.4, 44.3, 98.3, 106.0, 134.0, 135.5, 137.7, 138.1, 147.4; LRMS (ESI) *m/z* calculated for $C_{11}H_{16}N_4S$: 236.1, found: 237.1 $(M+1)^+$.

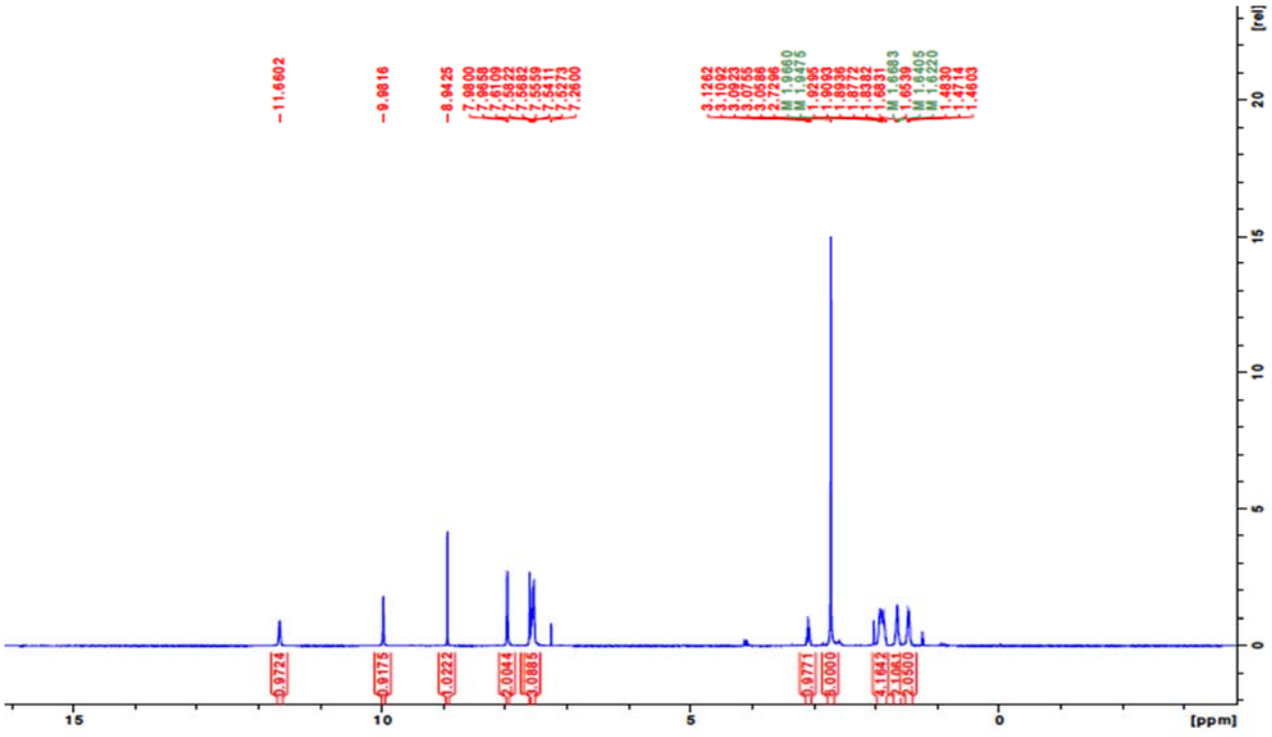
Table S2 Accurate MIC (μg/mL) of compounds determined by *Procedure B*.

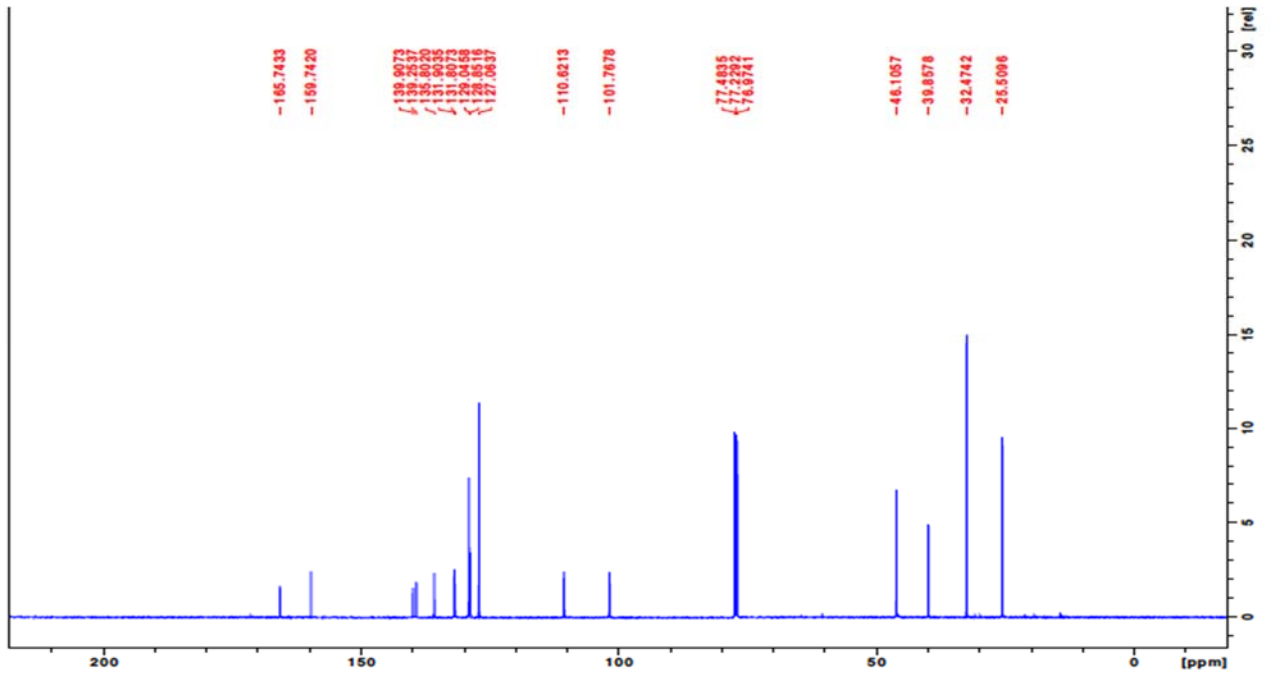
Compound	R^1	R^2	MIC
SB-P17G-C2			0.008
SB-P17G-A38			0.019
10e-6			0.19
11b			1.6
11c			0.09
11d			1.6
20a			6.25
20d			0.78
20f			<0.02
20g			0.0039

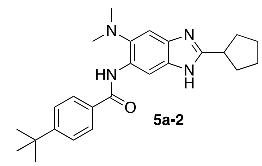
MIC: Minimum concentration of the compound required to inhibit growth of 99% of *Mtb* H37Rv cells.



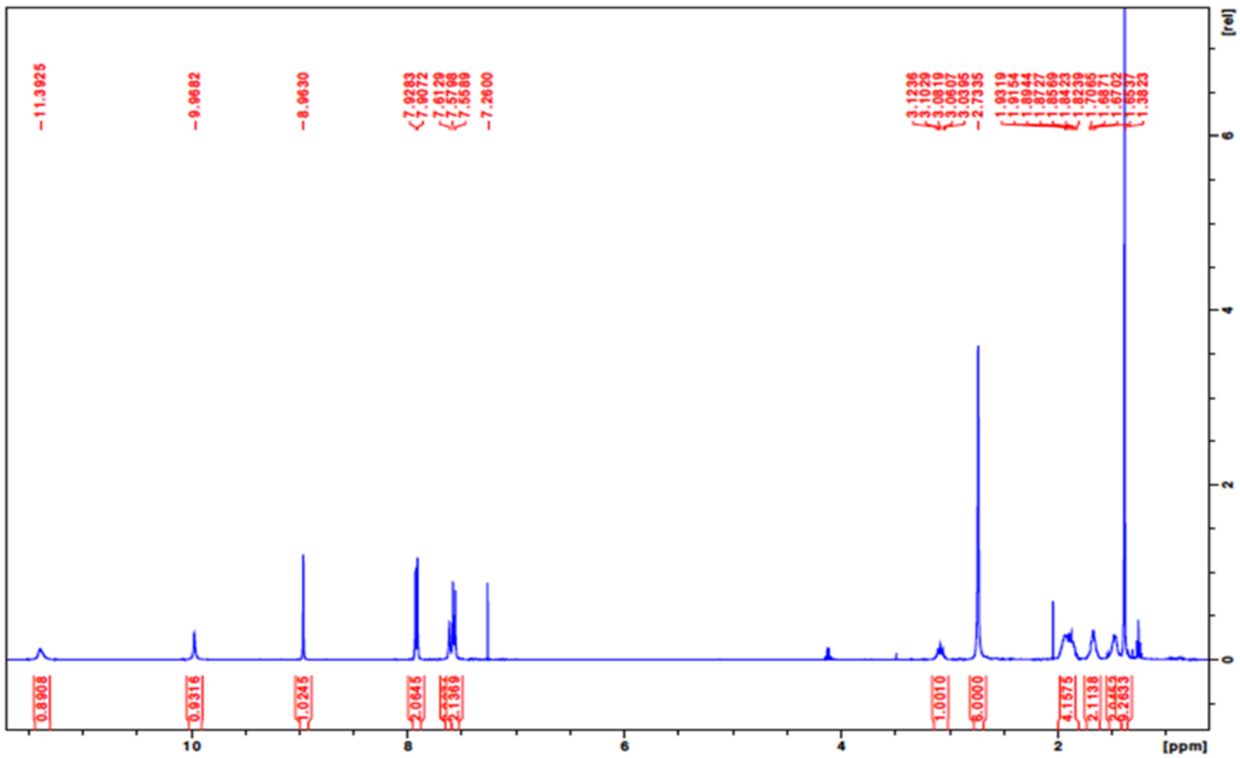
5a-1

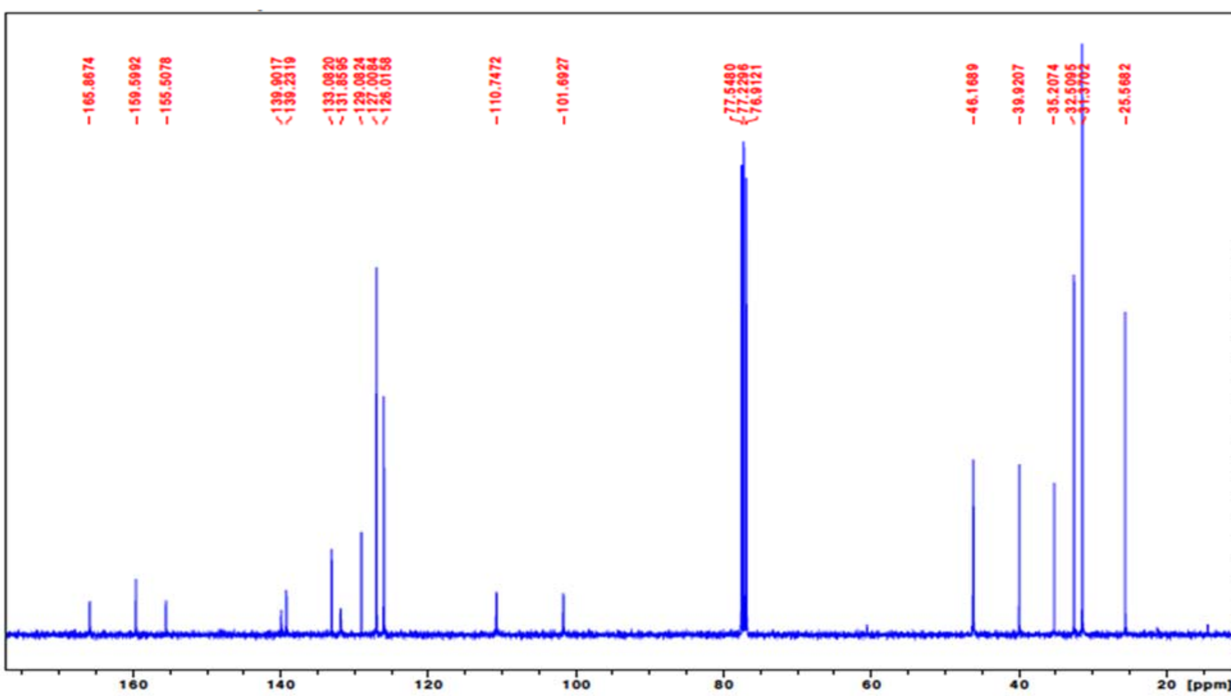

11.6602
9.9816
8.9425
7.9800
7.9658
7.6109
7.5922
7.5682
7.5559
7.5411
7.5273
7.2600
3.1262
3.1092
3.0922
3.0755
3.0586
2.7296
M 1.9660
M 1.9475
1.9295
1.9093
1.8936
1.8772
1.8582
1.6831
M 1.6683
1.6530
M 1.6205
M 1.6220
1.4830
1.4714
1.4603
0.9724
0.9175
0.9222
2.0044
3.0883
0.9771
6.0000
4.1842
2.1061
2.0500
[ppm]
[rel]

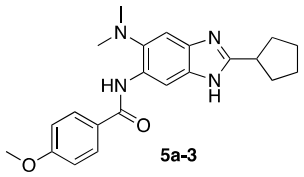

165.7433
159.7420
139.9073
139.2537
135.8020
131.9035
131.8073
129.0458
128.8516
127.0637
110.6213
101.7678
77.4835
77.2292
76.9741
46.1057
39.8578
32.4742
25.5086
[ppm]
[rel]


HN
O
N
N
H

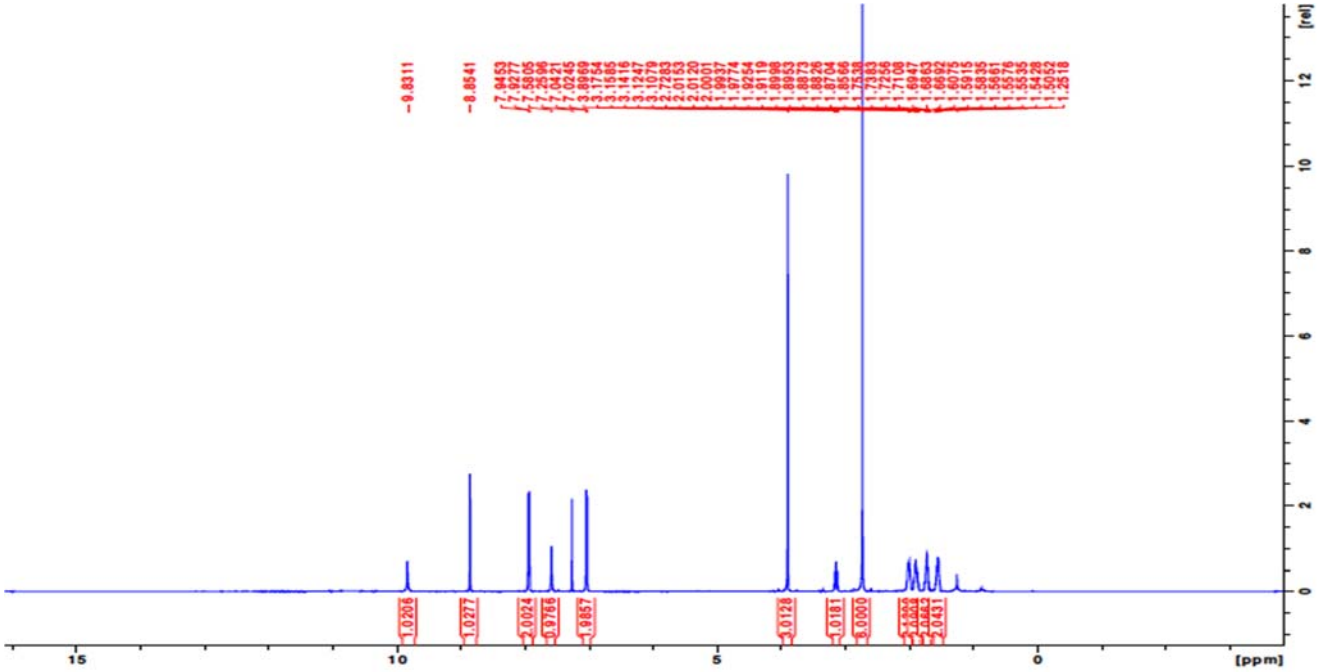
5a-2

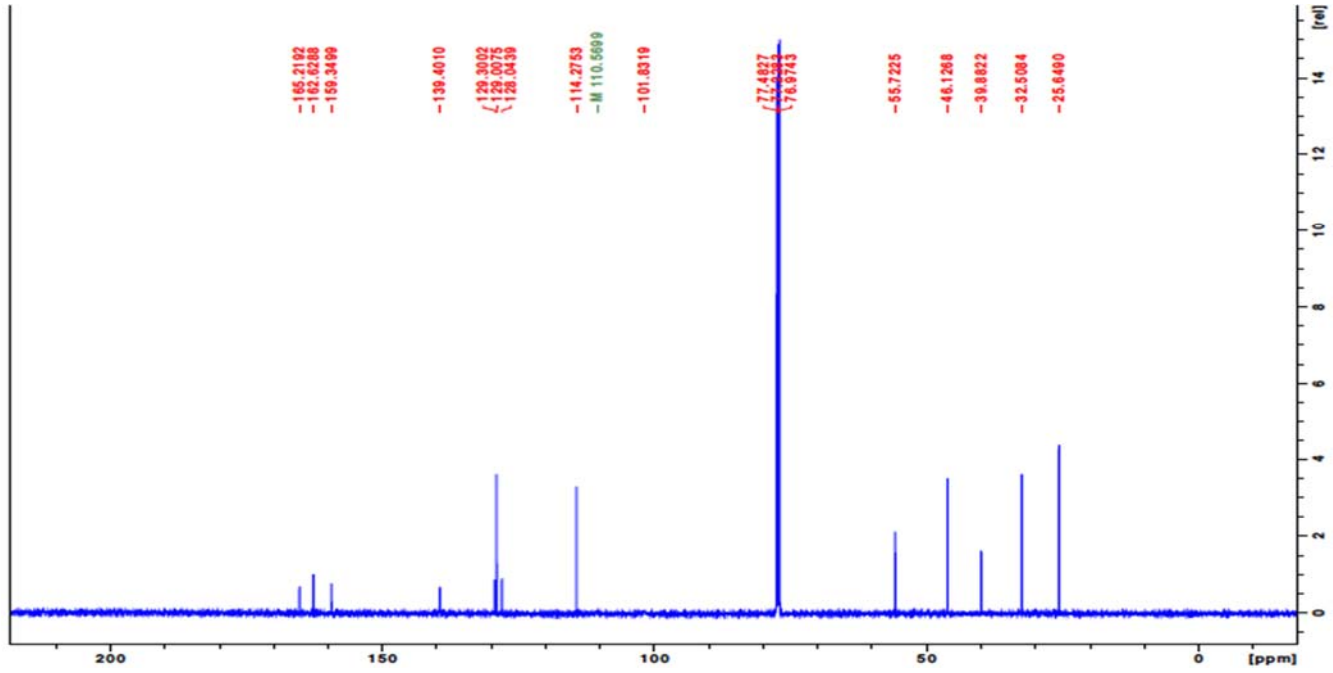

11.3925
9.9682
8.9630
7.9283
7.9072
7.6129
7.5798
7.5589
7.2600
3.1236
3.1029
3.0819
3.0607
3.0395
2.7335
1.9319
1.9154
1.8944
1.8727
1.8569
1.8423
1.8239
1.7065
1.6871
1.6702
1.6537
1.3823
0.8908
0.9316
1.0245
2.0645
2.1369
1.0010
6.0000
4.1575
2.1138
[rel]
[ppm]

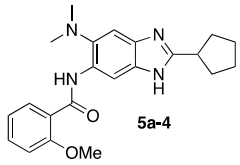

165.8674
159.5992
155.5078
139.9017
139.2319
133.0820
131.8595
129.0824
127.0084
126.0158
110.7472
101.6927
77.5480
77.2296
76.9121
46.1689
39.9207
35.2074
32.5095
31.3702
25.5682
[rel]
[ppm]


N
N
HN
N
H
O
O

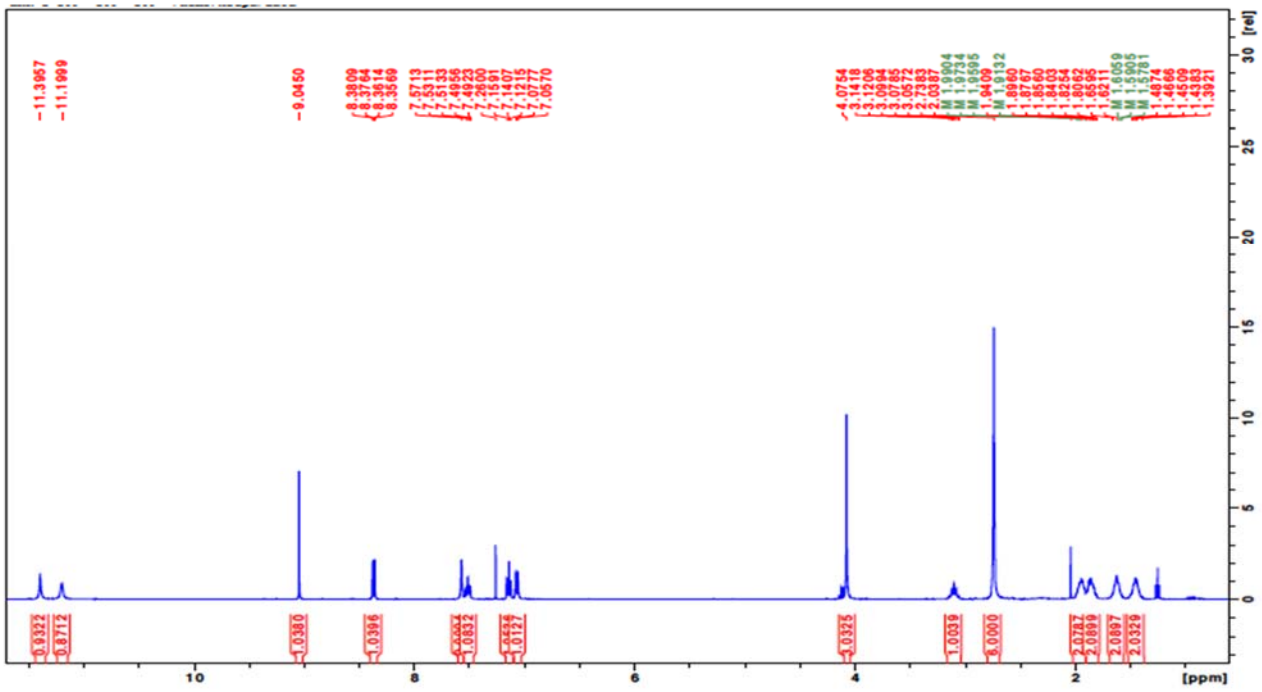
5a-3

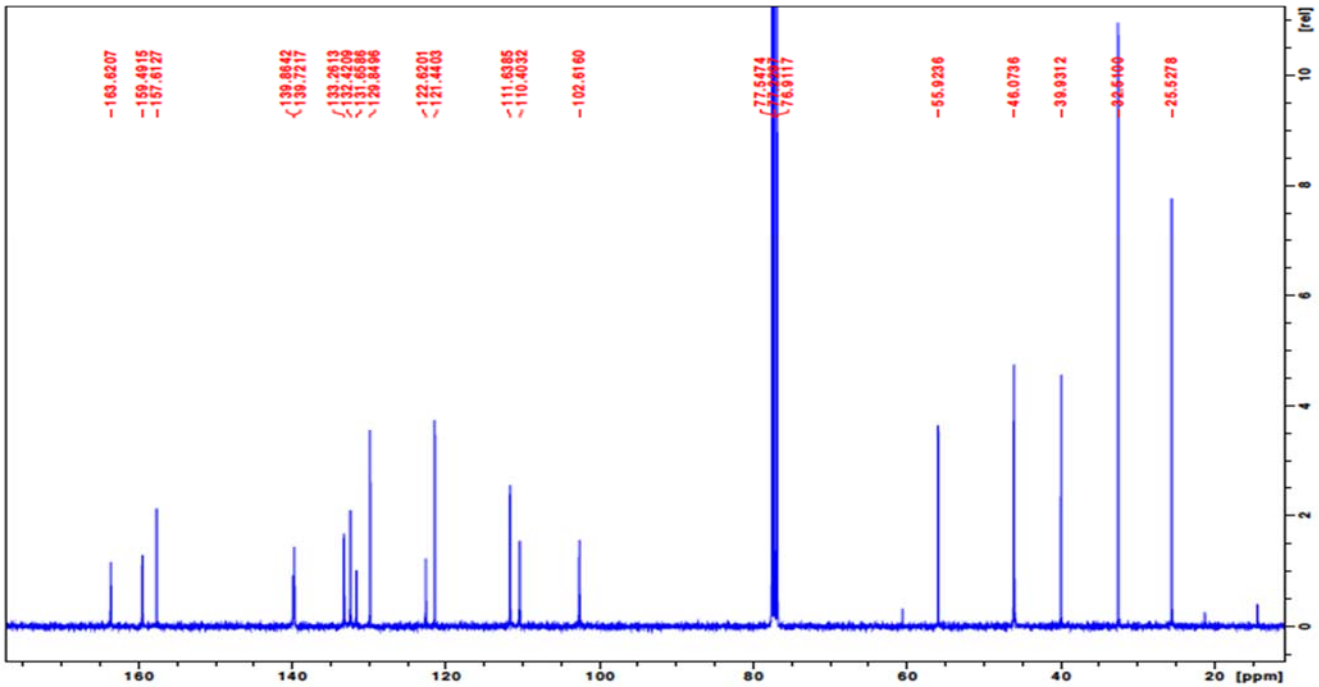

9.8311
8.8541
1.0206
1.0277
2.0024
0.9766
0.9857
3.0128
1.0181
3.0000
2.0431
15
10
5
0
[ppm]
[rel]

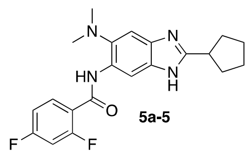

165.2182
162.6288
159.3499
139.4010
129.3002
129.0075
128.0439
114.2753
M 110.5699
101.8319
77.4827
77.1651
76.8743
55.7225
46.1268
39.8822
32.5084
25.6490
200
150
100
50
0
[ppm]
[rel]



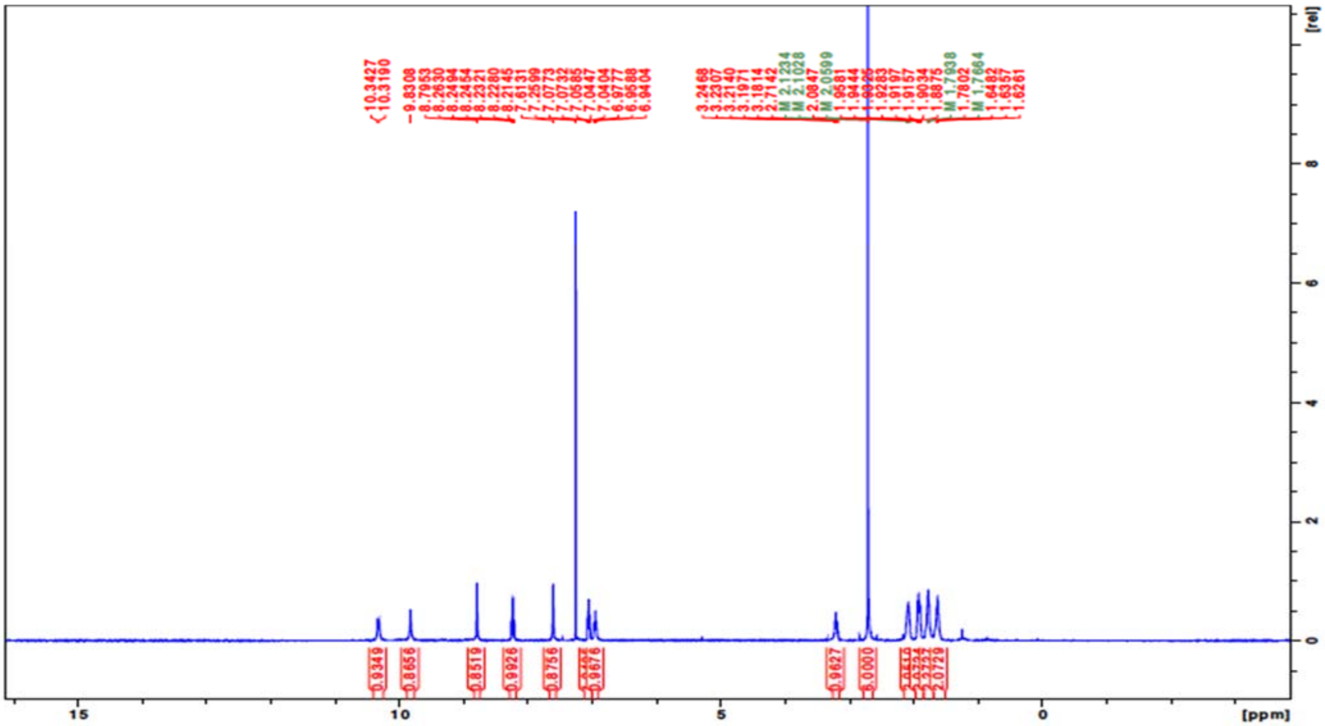
5a-4

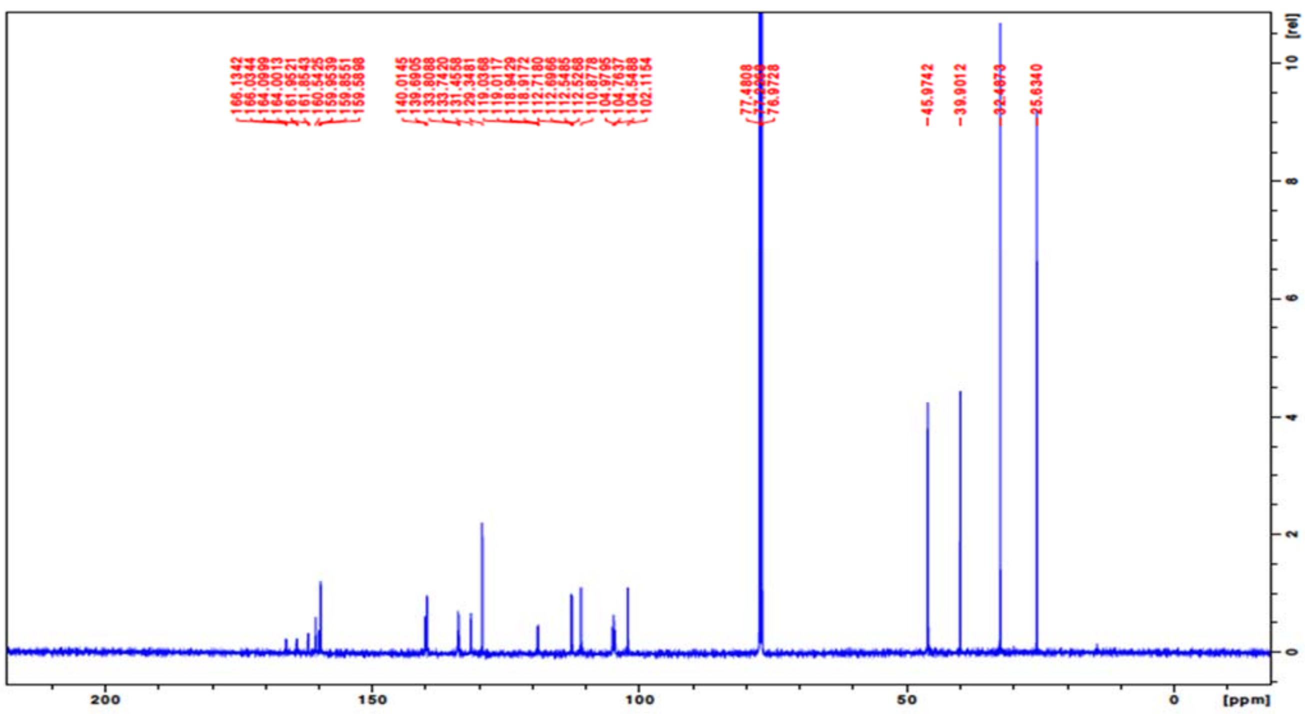


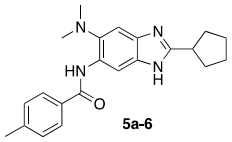




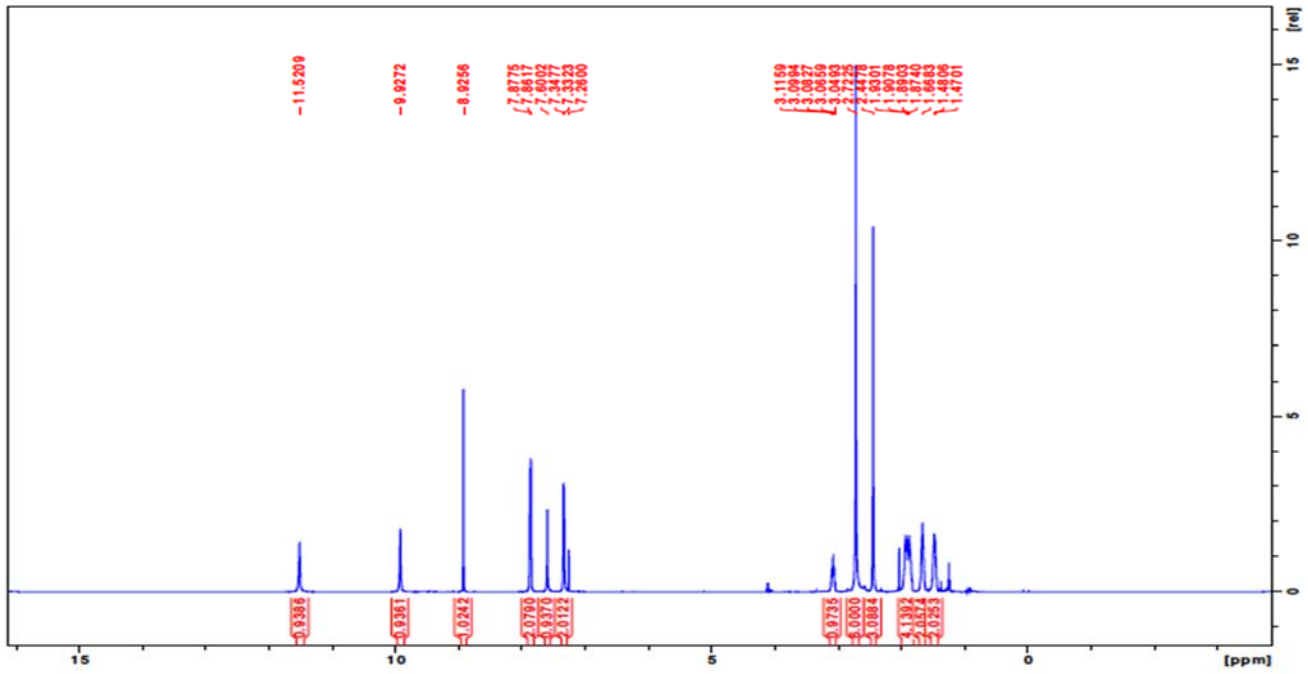
5a-5

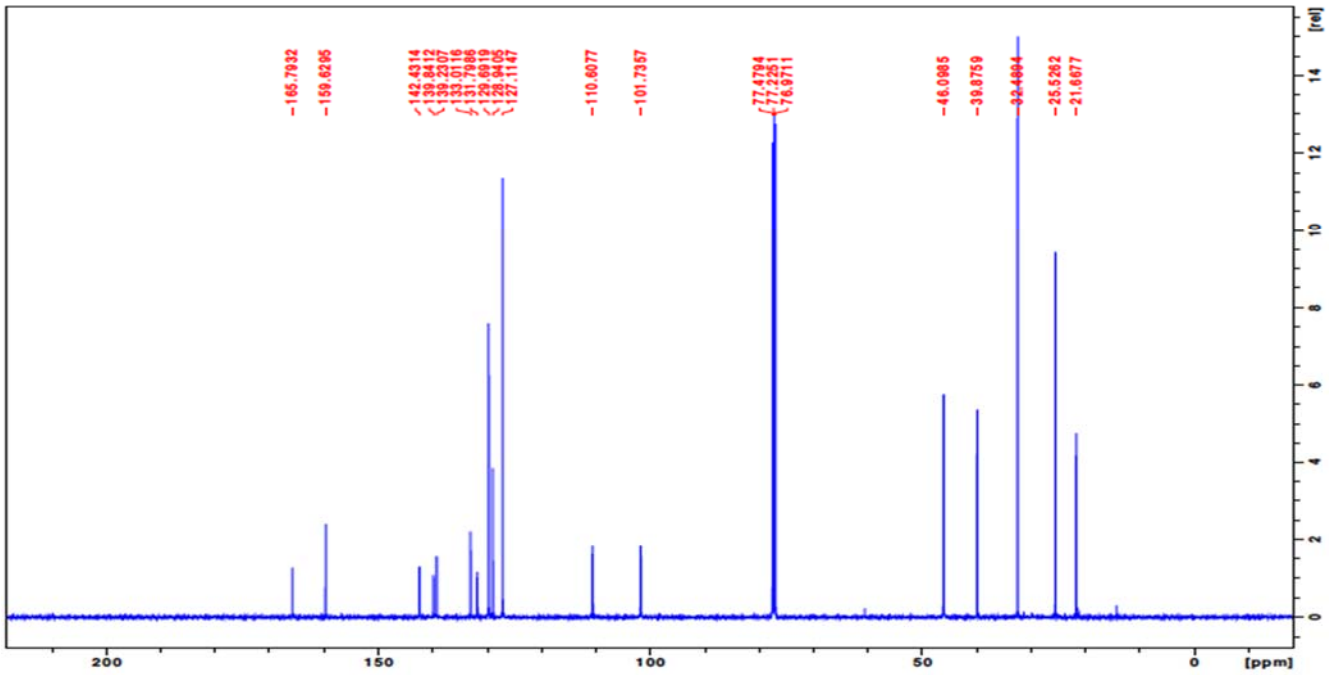

[ppm]
[rel]
15
10
5
0

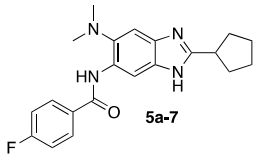

166.1342
166.0344
164.0999
164.0013
161.9521
161.8543
160.5425
159.9539
159.8551
159.5898
140.0145
139.6905
133.8088
132.7420
131.4558
129.3481
119.0368
119.0117
118.9429
118.9172
112.7180
112.6966
112.5485
112.5268
110.8778
104.9795
104.7637
104.5488
102.1154
77.4808
76.9728
45.9742
39.9012
32.4673
25.6340
[ppm]
[rel]
200
150
100
50
0


N
N
HN
N
H
O

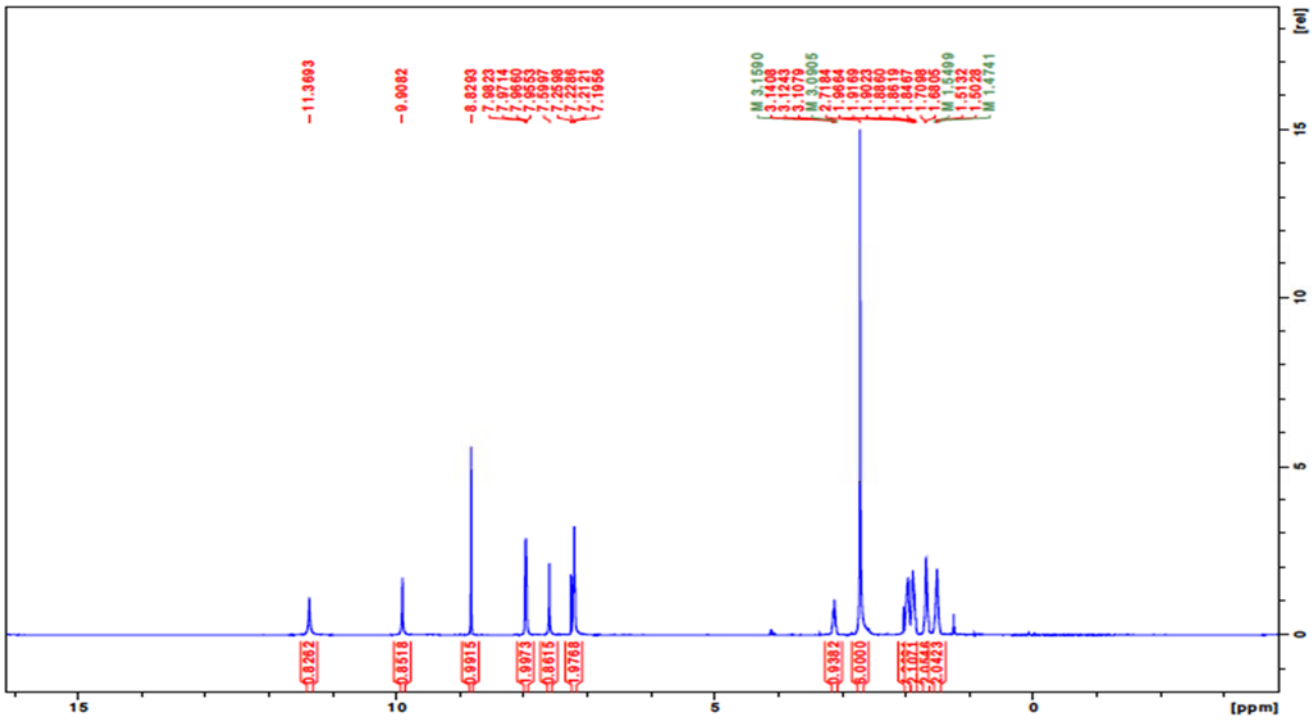
5a-6

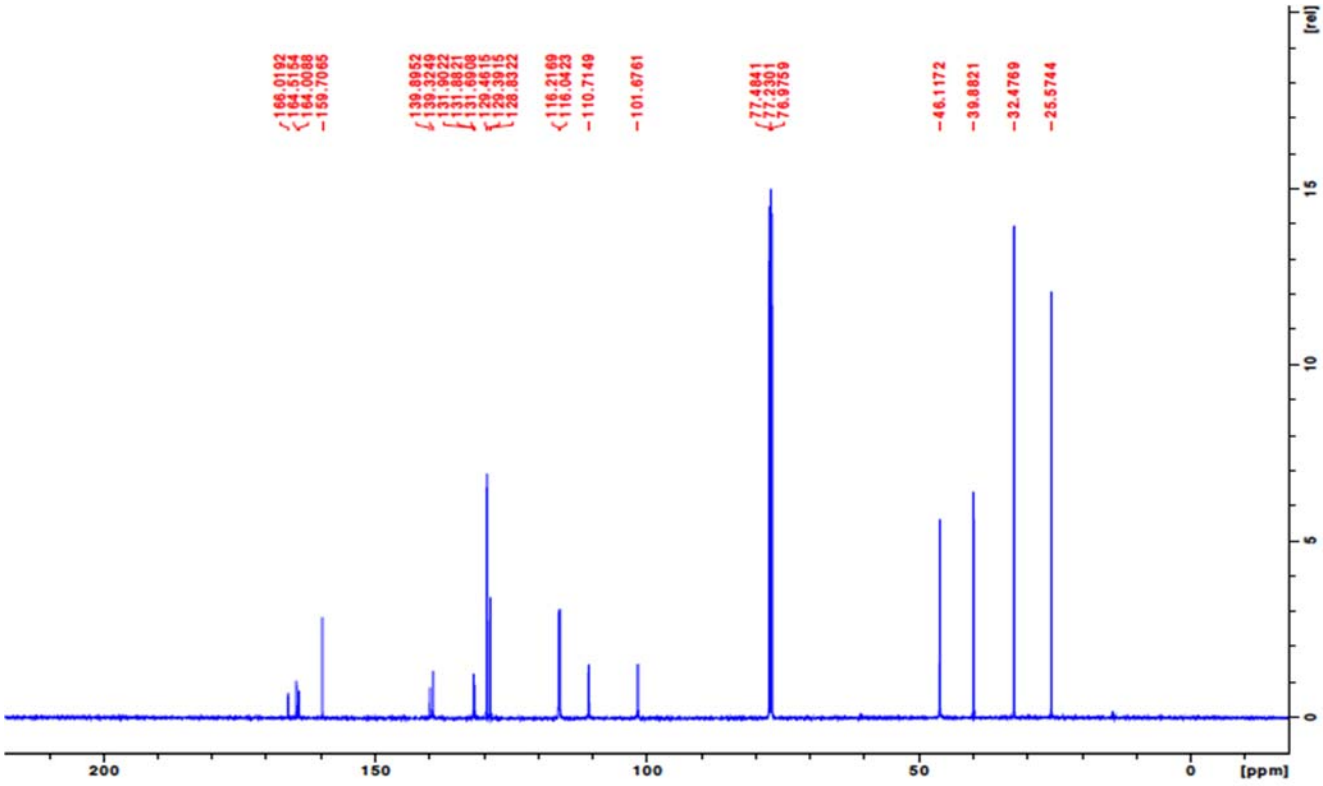

11.5209
9.9272
8.9256
7.8775
7.8617
7.6002
7.3477
7.3323
7.2600
3.1159
3.0994
3.0827
3.0659
3.0493
2.7225
2.4476
1.9301
1.9078
1.8903
1.8740
1.6683
1.4806
1.4701
0.9386
0.9361
1.0242
0.0790
0.9370
1.0122
0.9735
5.0000
2.0884
4.1382
2.0674
2.0253
15
10
5
0
[ppm]
[rel]

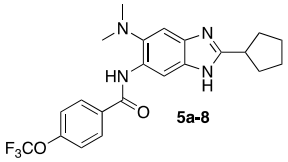

165.7932
159.6265
142.4314
139.8412
139.2307
133.0116
131.7986
129.6919
128.9405
127.1147
110.6077
101.7357
77.4794
77.2251
76.9711
46.0985
39.8759
32.4884
25.5282
21.6677
200
150
100
50
0
[ppm]
[rel]


N
N
HN
N
H
O
F

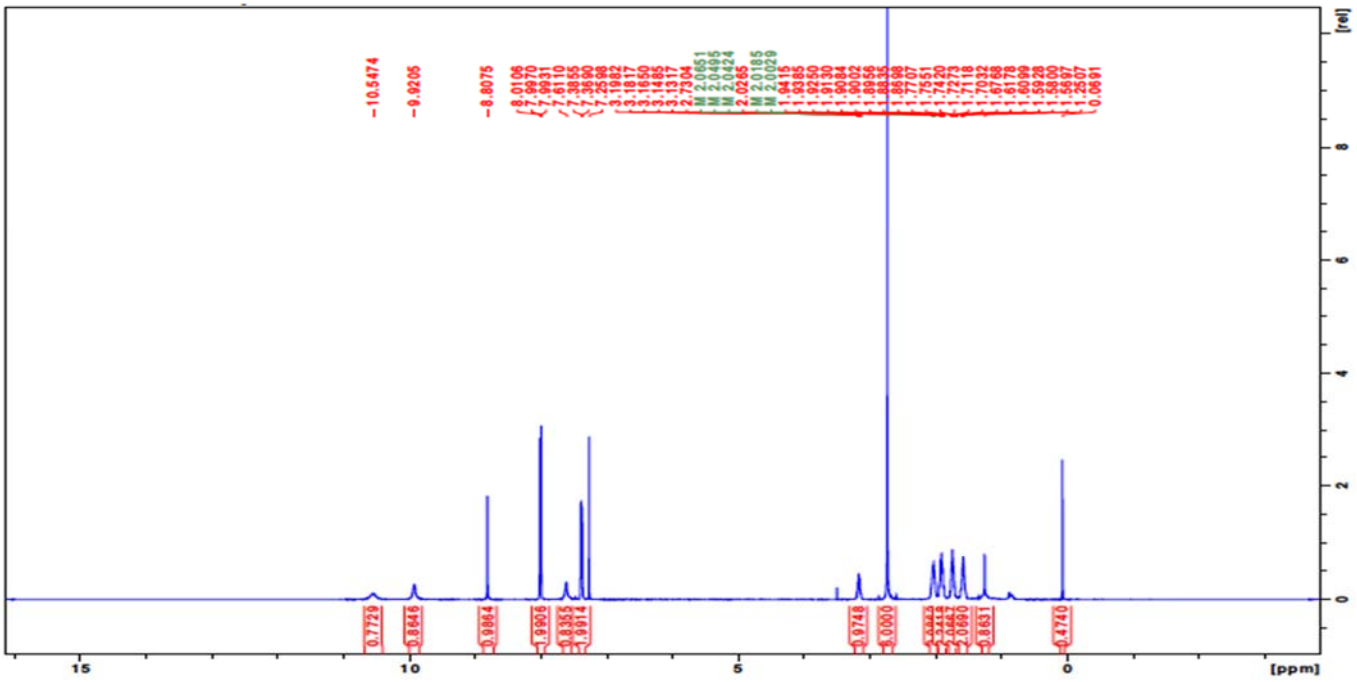
5a-7

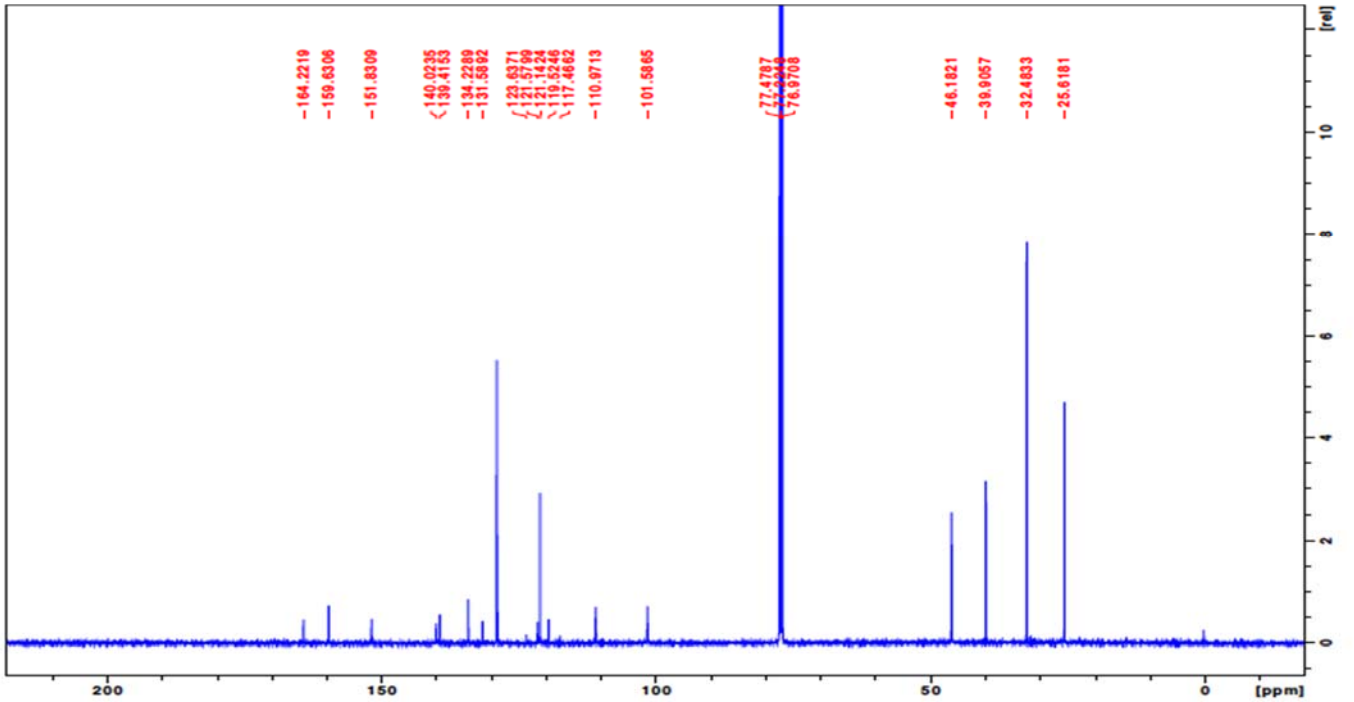

11.3693
9.9082
8.8293
7.9823
7.9714
7.9660
7.9553
7.5997
7.2598
7.2286
7.2121
7.1956
3.1590
3.1408
3.1243
3.1079
3.0905
2.7184
1.9964
1.9169
1.9023
1.8860
1.8619
1.8467
1.7098
1.6805
1.5499
1.5132
1.5028
1.4741
0.8282
0.8518
0.9915
0.9973
0.8615
0.9768
0.9382
5.0000
2.1021
2.0844
2.0423
[rel]
[ppm]

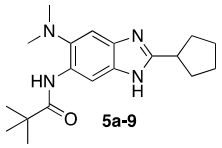

166.0192
164.5154
164.0088
159.7065
139.8952
139.3249
131.8922
131.8821
129.4815
129.3822
116.2169
116.0423
110.7149
101.6761
77.4841
77.2301
76.9759
46.1172
39.8821
32.4769
25.5744
[rel]
[ppm]


N
N
HN
N
H
O
F3CO

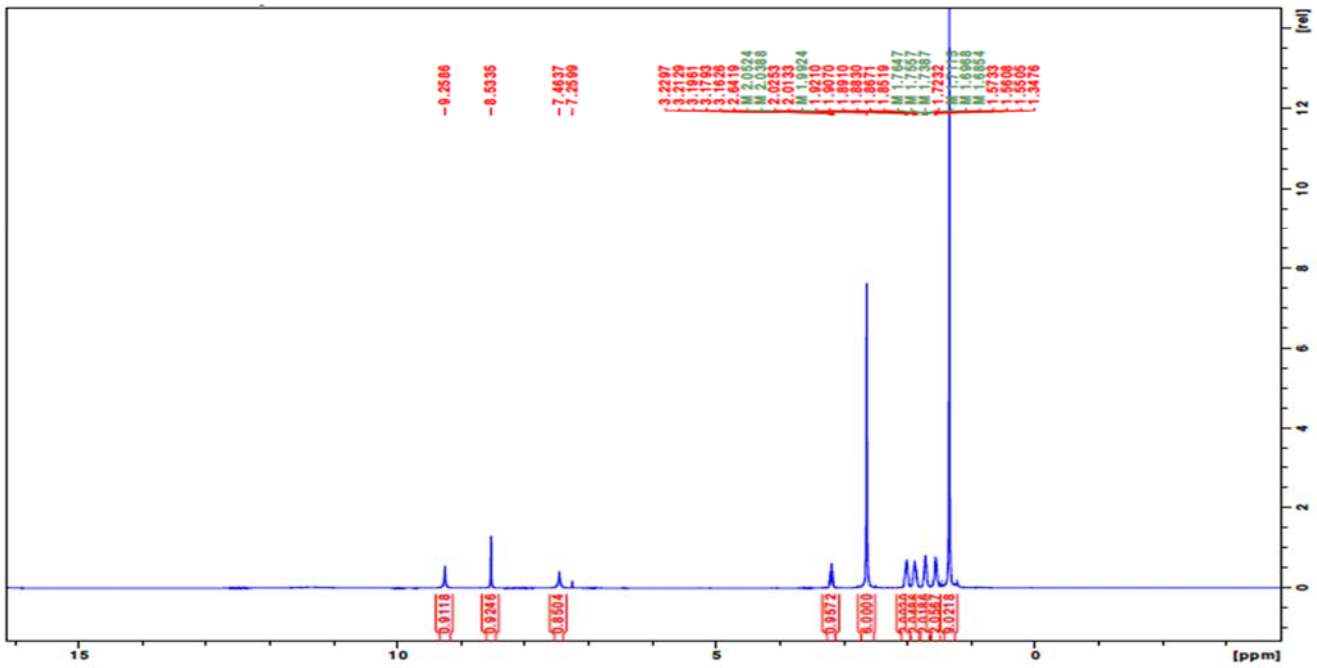
5a-8

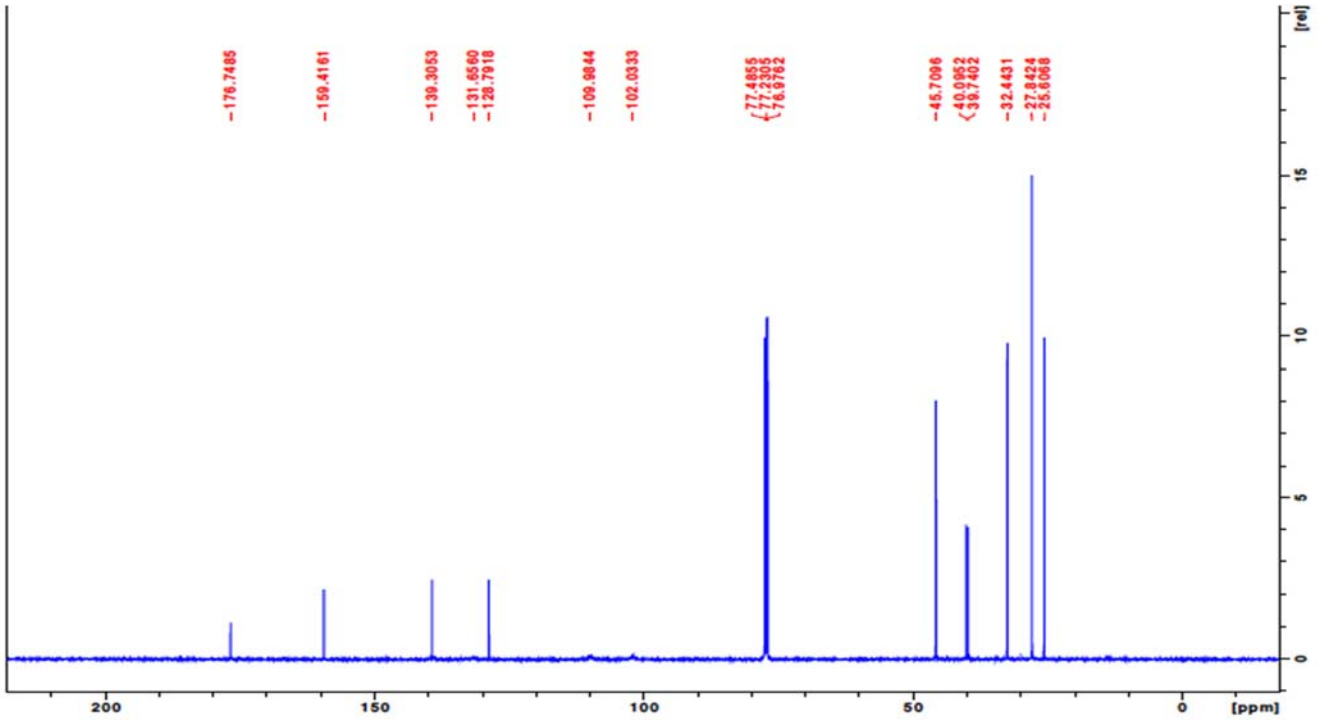

10.5474
9.9205
8.8075
8.0106
7.9970
7.9931
7.6110
7.3855
7.3690
7.2598
3.1982
3.1817
3.1650
3.1485
3.1317
2.7304
2.0651
2.0495
2.0424
2.0265
2.0185
2.0029
1.9415
1.9385
1.9250
1.9130
1.9084
1.9002
1.8956
1.8835
1.8698
1.7707
1.7551
1.7420
1.7273
1.7118
1.7032
1.6768
1.6178
1.6099
1.5928
1.5800
1.5697
1.2507
0.0691
0.7729
0.8646
0.9864
0.9906
0.9748
1.0000
0.8631
0.4740
15
10
5
0
[ppm]
[rel]

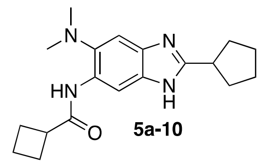

164.2219
159.6306
151.8309
140.0235
139.4153
134.2289
131.5892
123.6371
121.5799
121.1424
119.5246
117.4662
110.9713
101.5865
77.4787
77.3348
76.9708
46.1821
30.9057
32.4833
25.6181
200
150
100
50
0
[ppm]
[rel]


N
N
HN
N
H
O

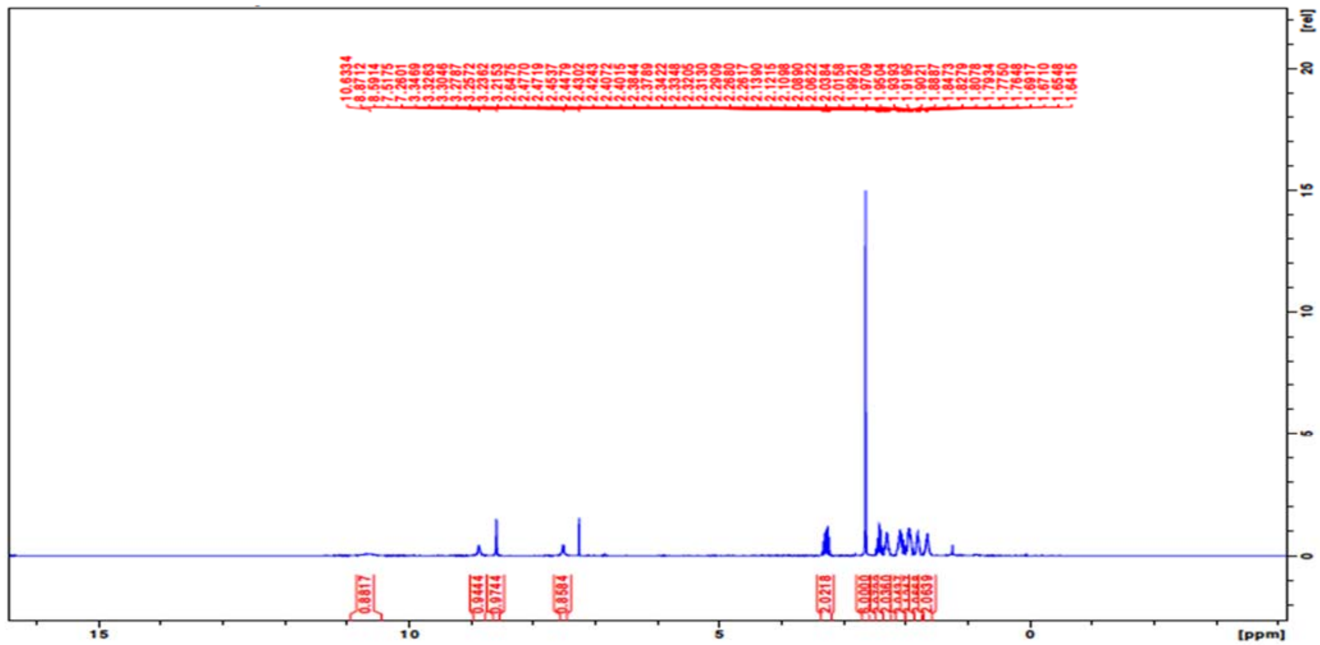
5a-9


9.2586
8.5335
7.4637
7.2599
3.2297
3.2129
3.1961
3.1793
3.1626
2.6419
M 2.0524
M 2.0388
2.0253
2.0133
M 1.9924
1.9210
1.9070
1.8910
1.8830
1.8671
1.8519
M 1.7647
M 1.7557
M 1.7387
M 1.7232
1.5783
1.5608
1.3476
0.9118
0.9246
0.8504
0.9572
6.0000
2.0211
15
10
5
0
[ppm]
[rel]

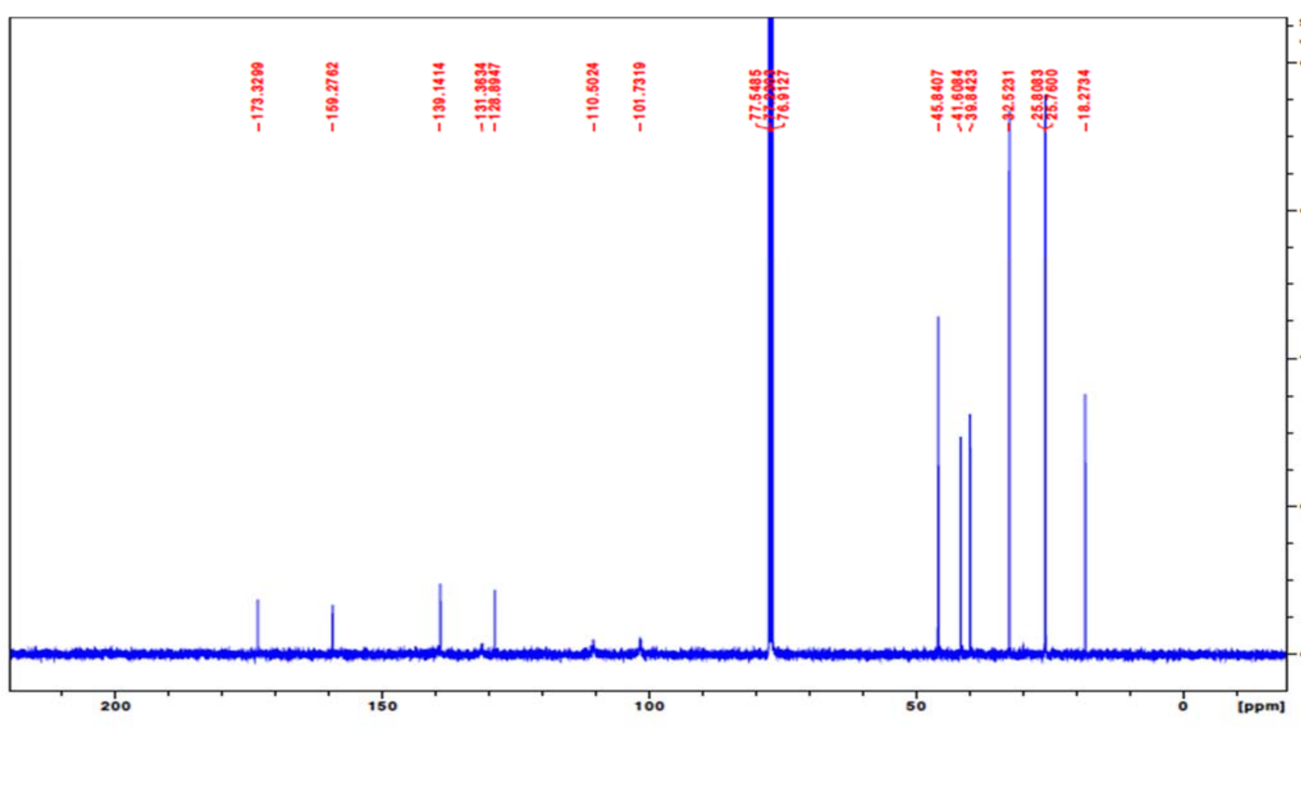

176.7485
159.4161
139.3053
131.6560
128.7918
109.8844
102.0333
77.4855
77.2305
76.9762
45.7096
40.0852
39.7402
32.4431
27.8424
25.6068
200
150
100
50
0
[ppm]
[rel]



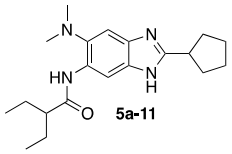
5a-10



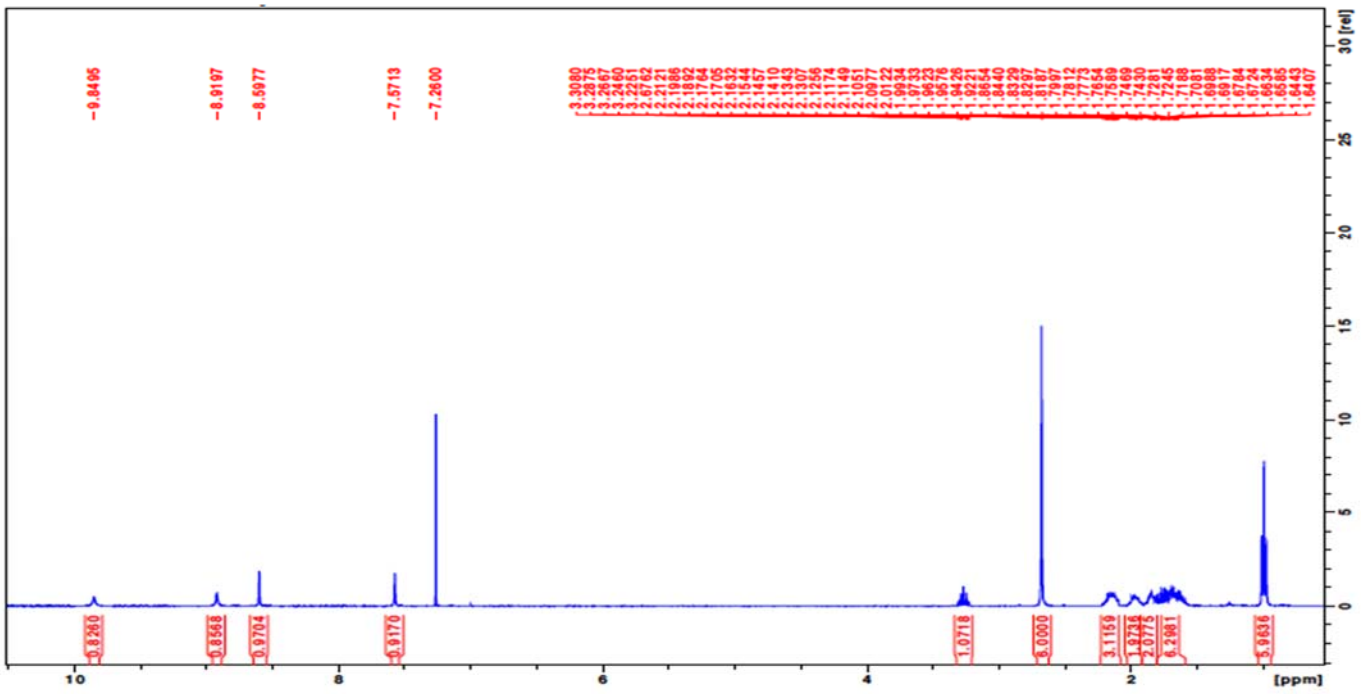
[rel]
0.8817
0.9444
0.9744
0.8584
2.0218
[ppm]

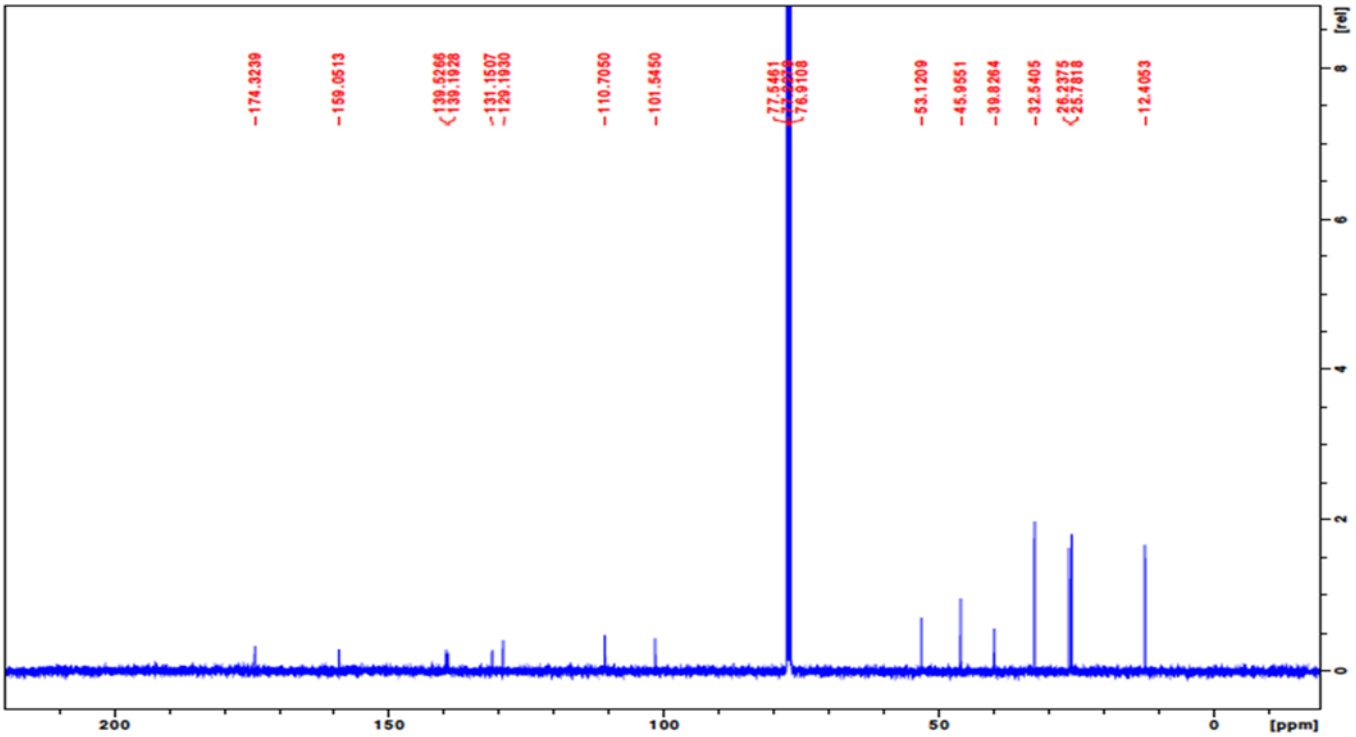


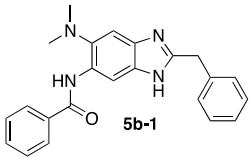
[rel]
173.3299
159.2762
139.1414
131.3634
128.8947
110.5024
101.7319
77.5485
76.9127
45.8407
41.6084
39.8423
32.5231
25.8083
25.7600
18.2734
[ppm]


N
N
HN
N
H
O

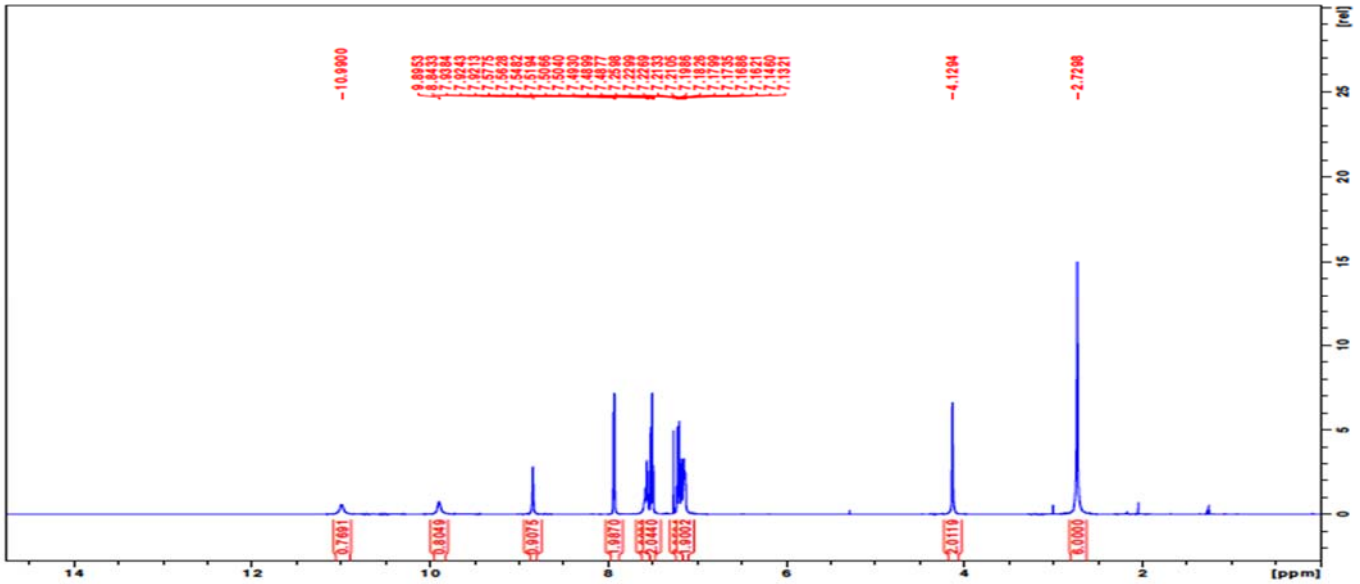
5a-11

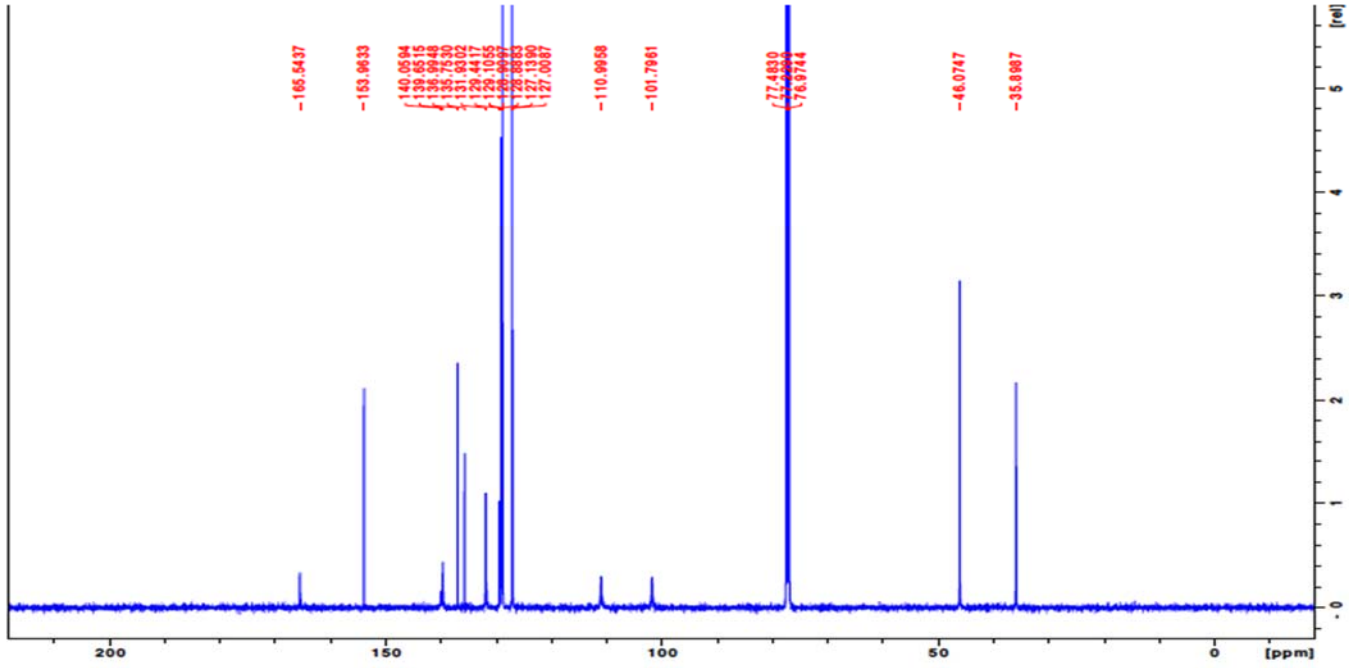

9.8495
8.9197
8.5977
7.5713
7.2600
0.8260
0.8568
0.9704
0.9170
1.0718
6.0000
3.1159
1.8734
2.0775
0.2981
5.9636
10
8
6
4
2
[ppm]
30 [rel]

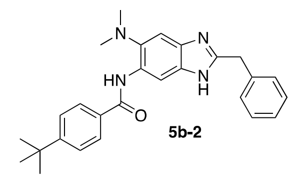

174.3239
159.0513
139.5266
139.1928
131.1507
129.1930
110.7050
101.5450
77.5461
77.2299
76.9108
53.1209
45.0551
39.8264
32.5405
26.2375
25.7818
12.4053
200
150
100
50
0
[ppm]
[rel]


N
HN
N
H
O

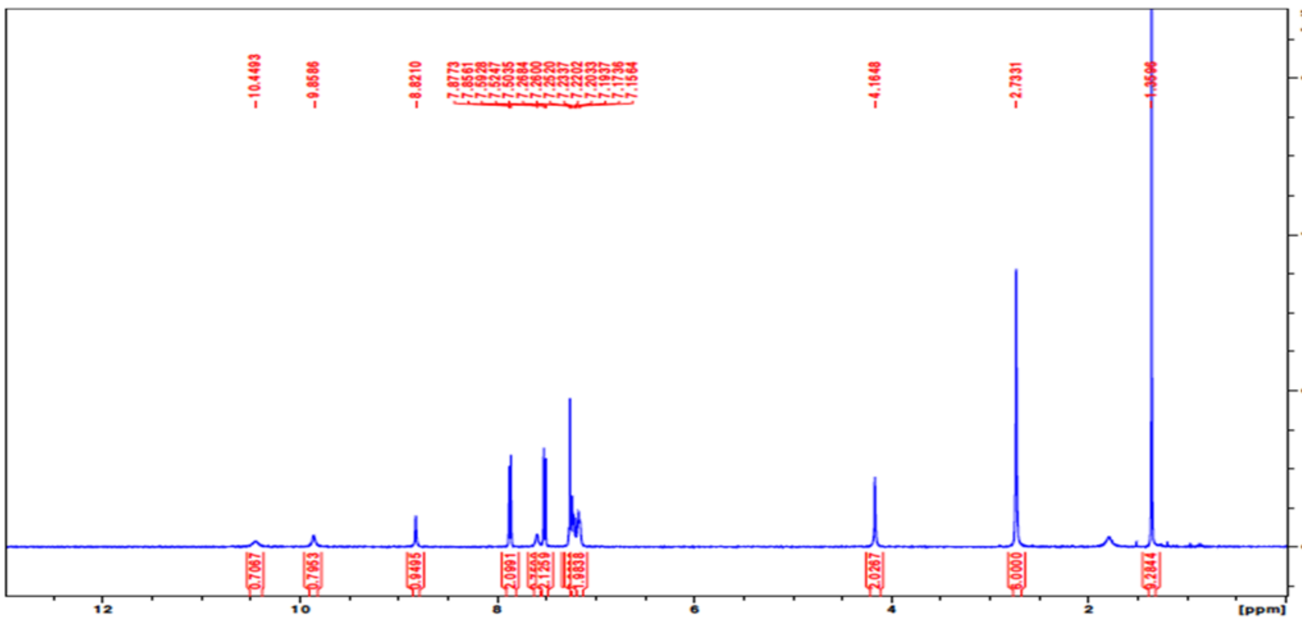
5b-1

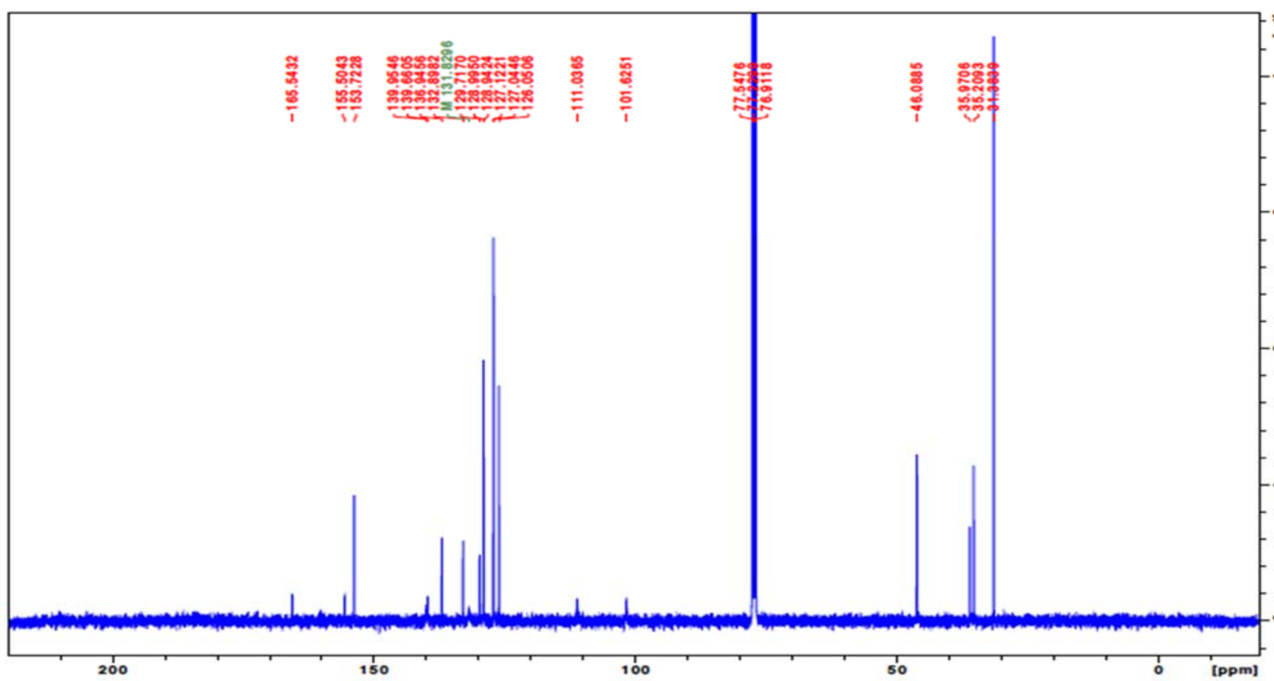

10.9900
9.8953
8.8433
8.3084
7.9243
7.9213
7.5775
7.5628
7.5482
7.5194
7.5046
7.5040
7.4930
7.4899
7.4877
7.2598
7.2299
7.2269
7.2133
7.2105
7.1986
7.1826
7.1799
7.1735
7.1688
7.1621
7.1460
7.1321
4.1294
2.7198
0.7691
0.8049
0.8075
0.9876
2.0440
1.0002
2.0119
3.0000
14
12
10
8
6
4
2
[ppm]
[rel]

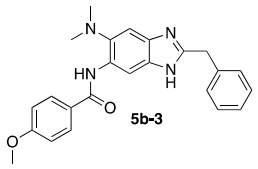

165.5437
153.9633
140.0594
139.6515
136.9948
135.7530
131.9302
129.4417
129.1055
128.8883
127.1390
127.0087
110.9958
101.7961
77.4830
76.9744
46.0747
35.8987
200
150
100
50
0
[ppm]
[rel]

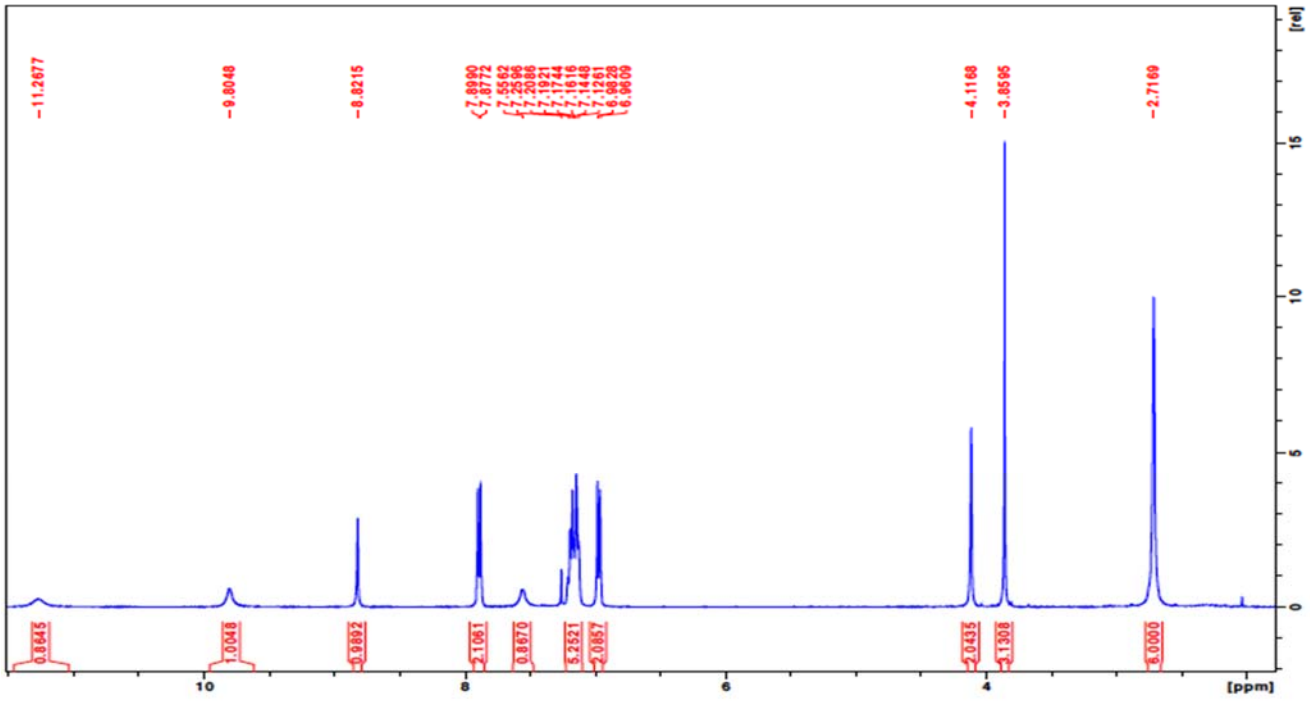


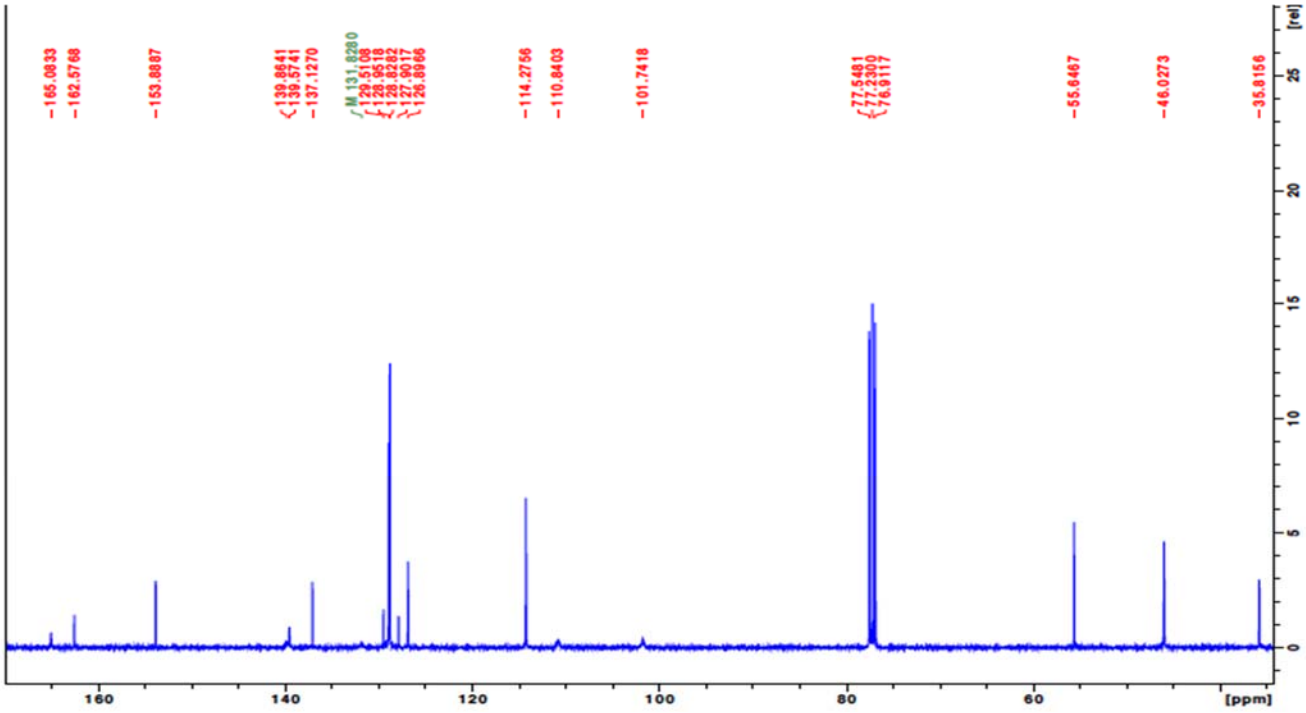
5b-2

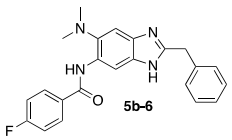


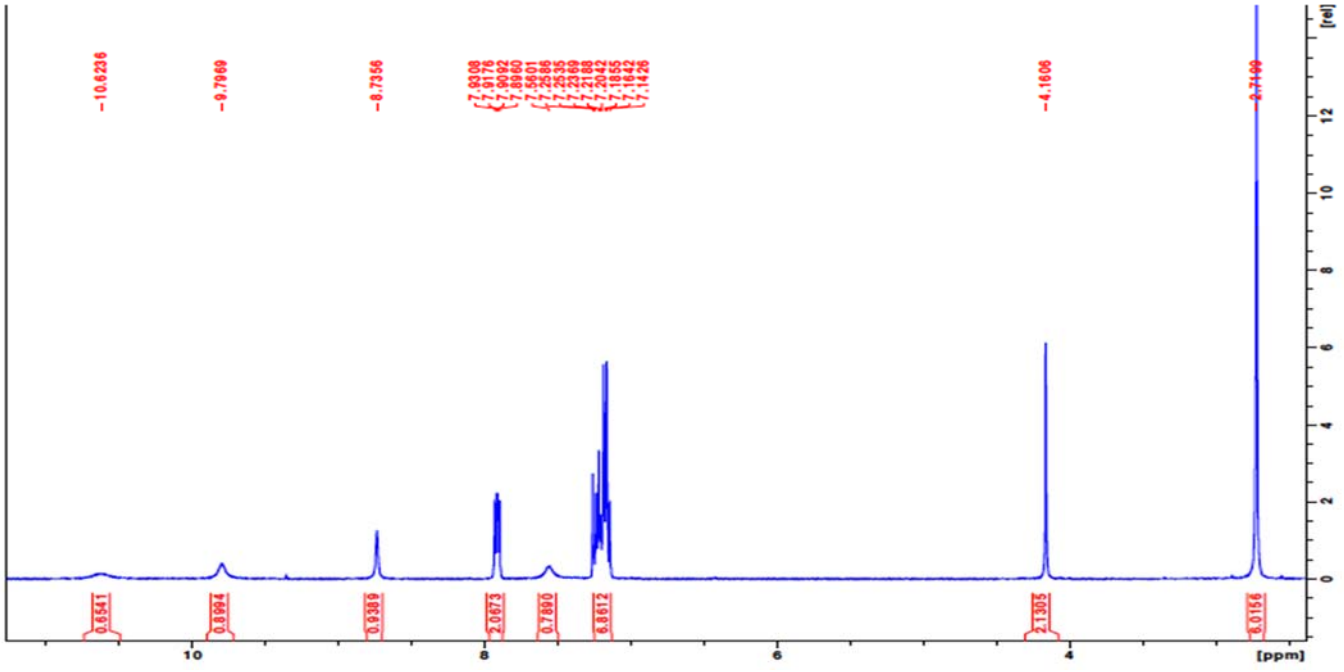


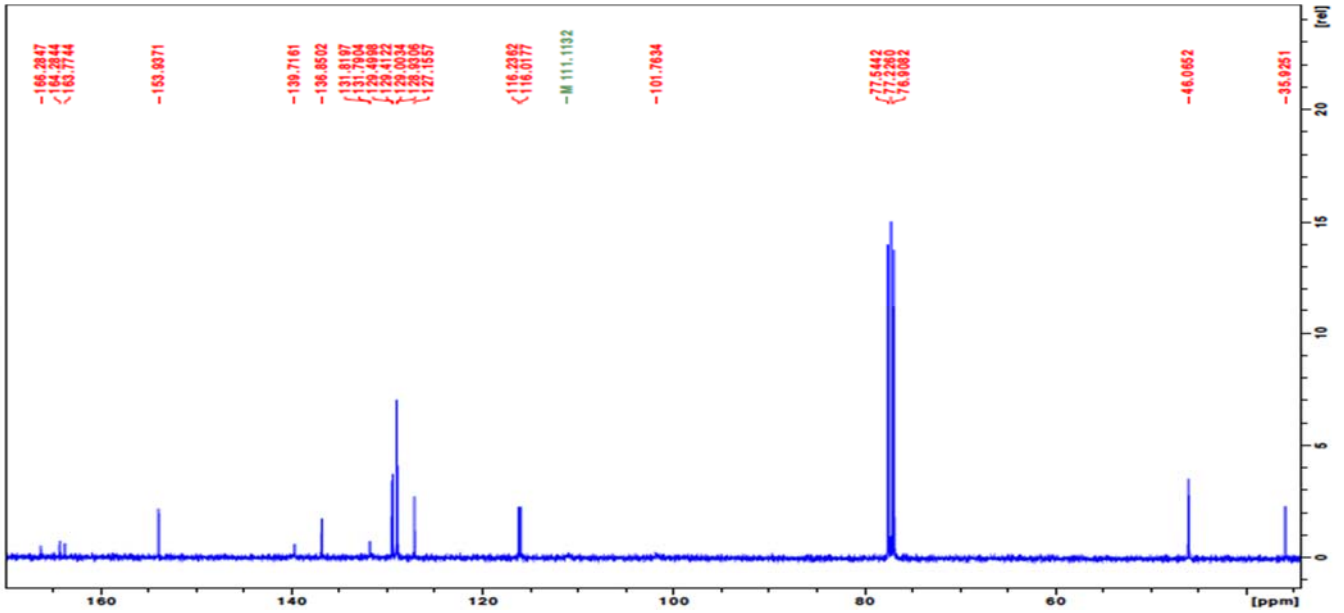

5b-3

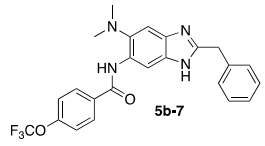

11.2677
9.8048
8.8215
7.8990
7.8772
7.5562
7.2596
7.2086
7.1921
7.1744
7.1616
7.1448
7.1261
6.9828
6.9609
4.1168
3.8595
2.7169
0.8645
1.0048
0.9892
2.1061
0.8670
5.2521
2.0857
2.0435
3.1308
6.0000
[rel]
[ppm]


165.0833
162.5768
153.8887
139.8641
139.5741
137.1270
131.8280
129.5108
128.9618
128.8282
127.9017
126.8966
114.2756
110.8403
101.7418
77.6481
77.2300
76.8117
55.6467
46.0273
35.8156
[rel]
[ppm]

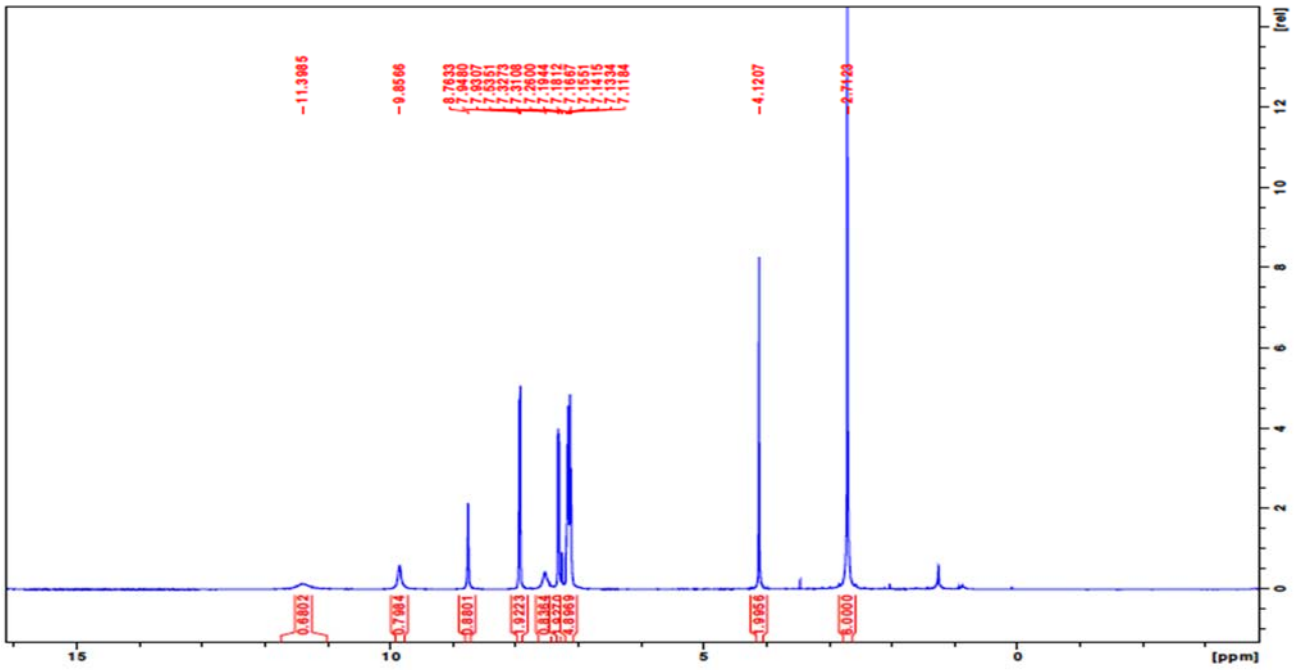

5b-6

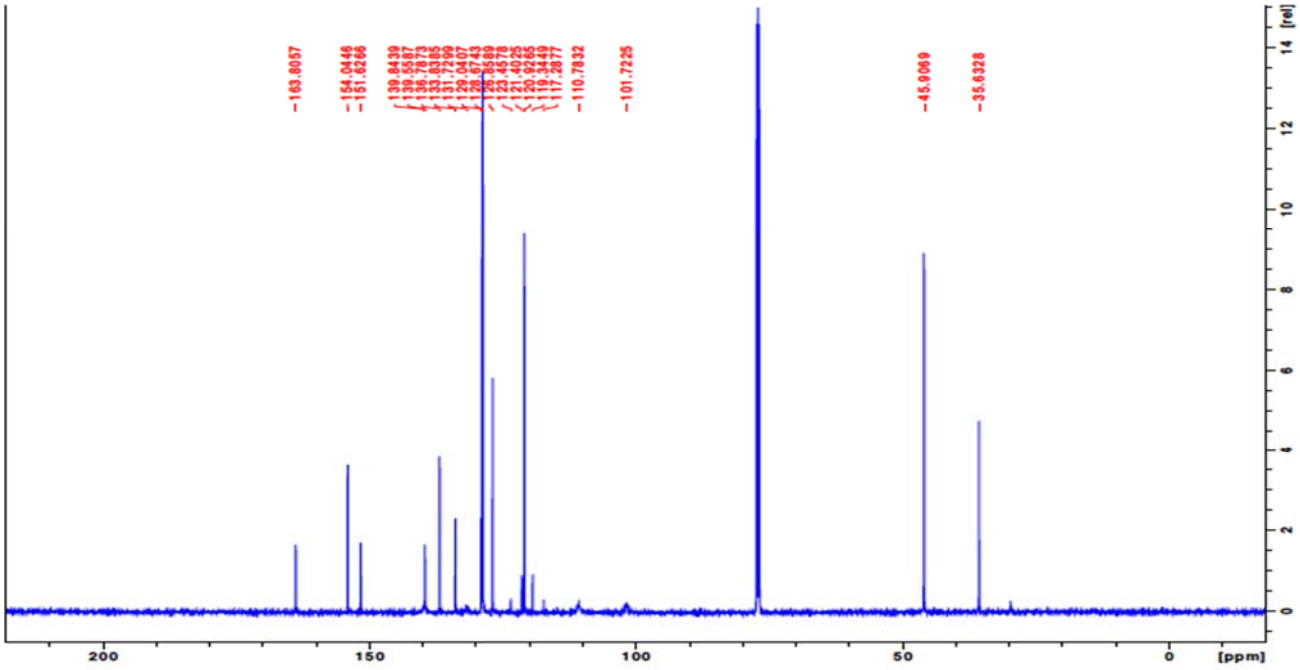

10.6236
9.7969
8.7356
7.9308
7.9176
7.9092
7.8960
7.5601
7.2586
7.2535
7.2369
7.2188
7.2042
7.1855
7.1642
7.1426
4.1606
2.7100
0.6541
0.8994
0.9389
2.0673
0.7890
6.8612
2.1305
8.0156
[rel]
[ppm]

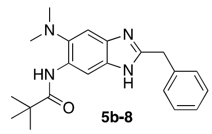

166.2847
164.2844
163.7744
153.8371
139.7161
136.8502
131.8197
131.7904
129.4998
129.4122
129.0034
128.9936
127.1557
118.2362
116.0177
M 111.1132
101.7634
77.5442
77.2260
76.9082
46.0652
35.9251
[rel]
[ppm]


N
N
HN
O
N
H
F3CO

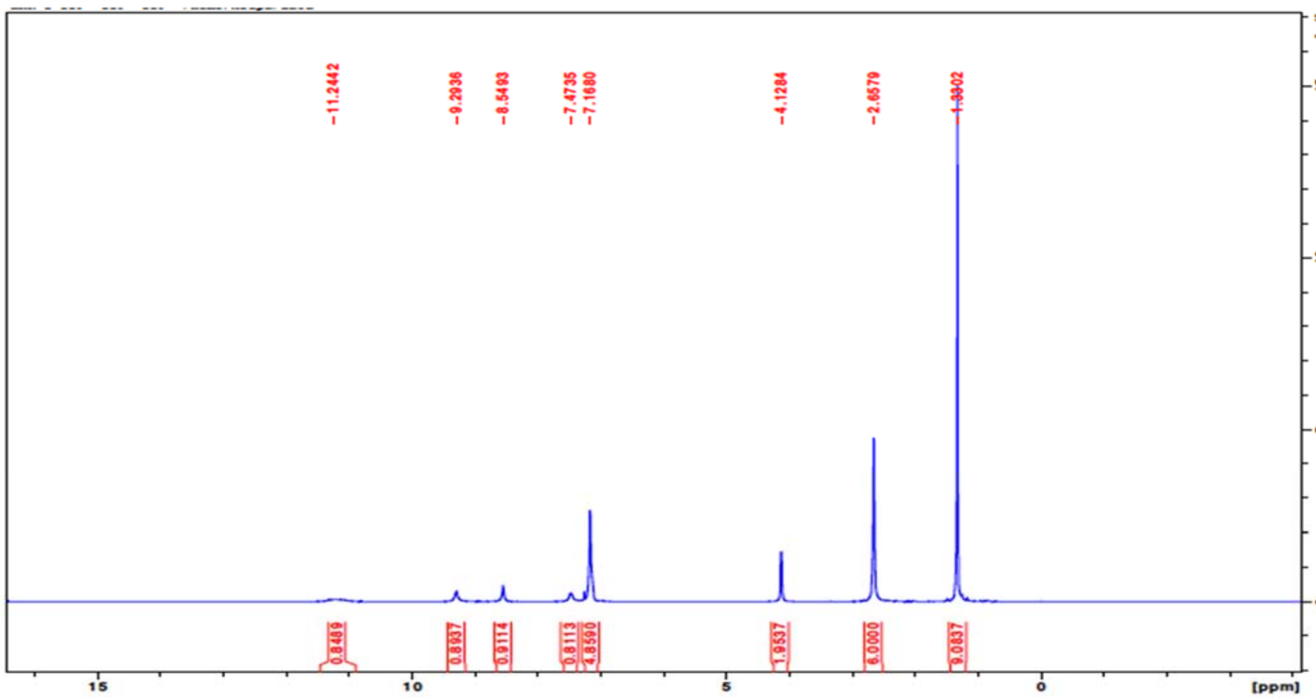
5b-7

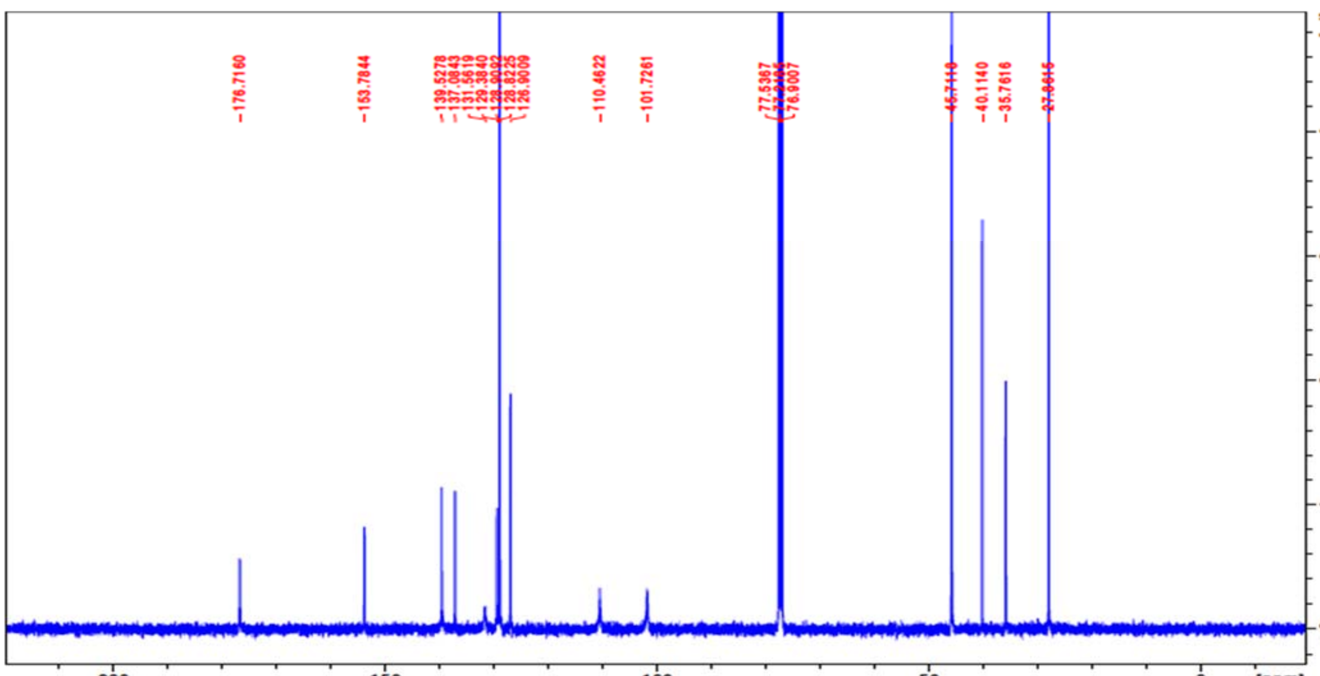

11.3985
9.8566
8.7633
8.4480
8.3307
7.5351
7.3273
7.3108
7.2600
7.1944
7.1812
7.1667
7.1551
7.1415
7.1334
7.1184
4.1207
2.7128
0.6802
0.7984
0.8801
1.9223
0.9384
1.0220
4.0969
1.9956
6.0000
15
10
5
0
[ppm]
[rel]

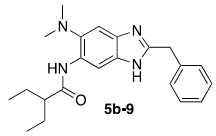

163.8057
154.0446
151.6286
139.8439
139.5587
136.7873
133.8385
131.7299
129.0407
128.6743
128.3589
123.4578
121.4025
120.9265
119.3449
117.2877
110.7832
101.7225
45.9069
35.6328
200
150
100
50
0
[ppm]
[rel]



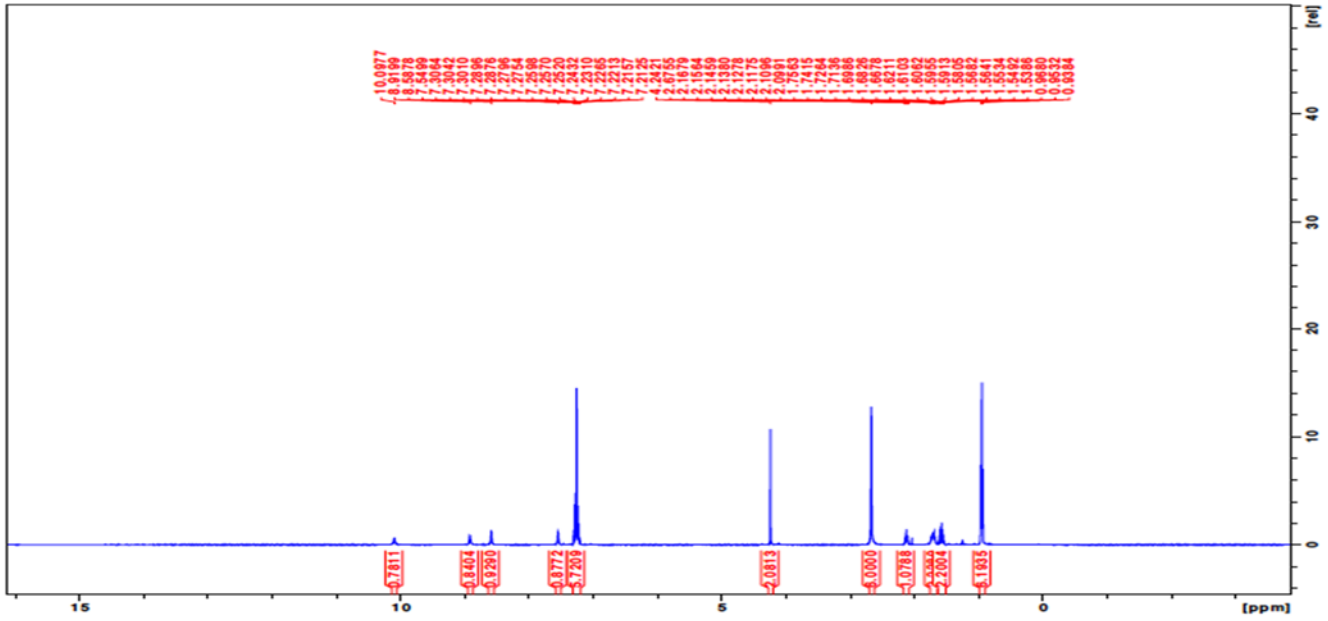
5b-8

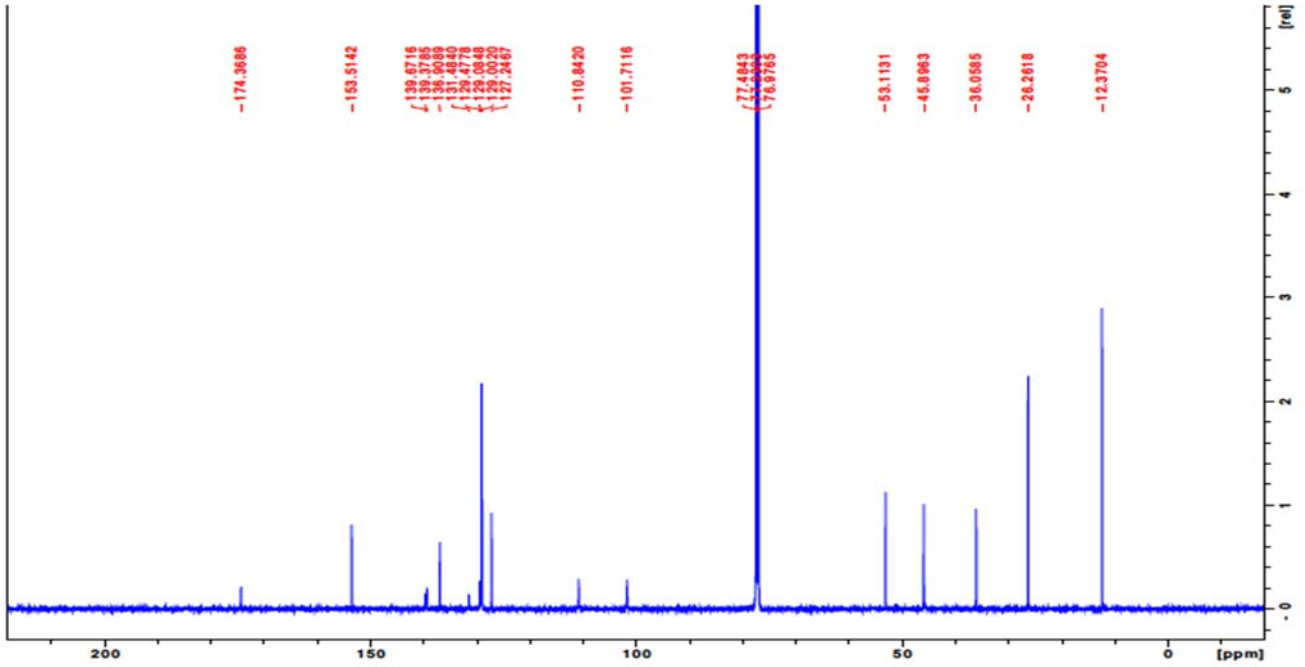


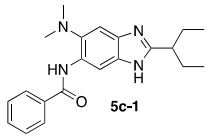




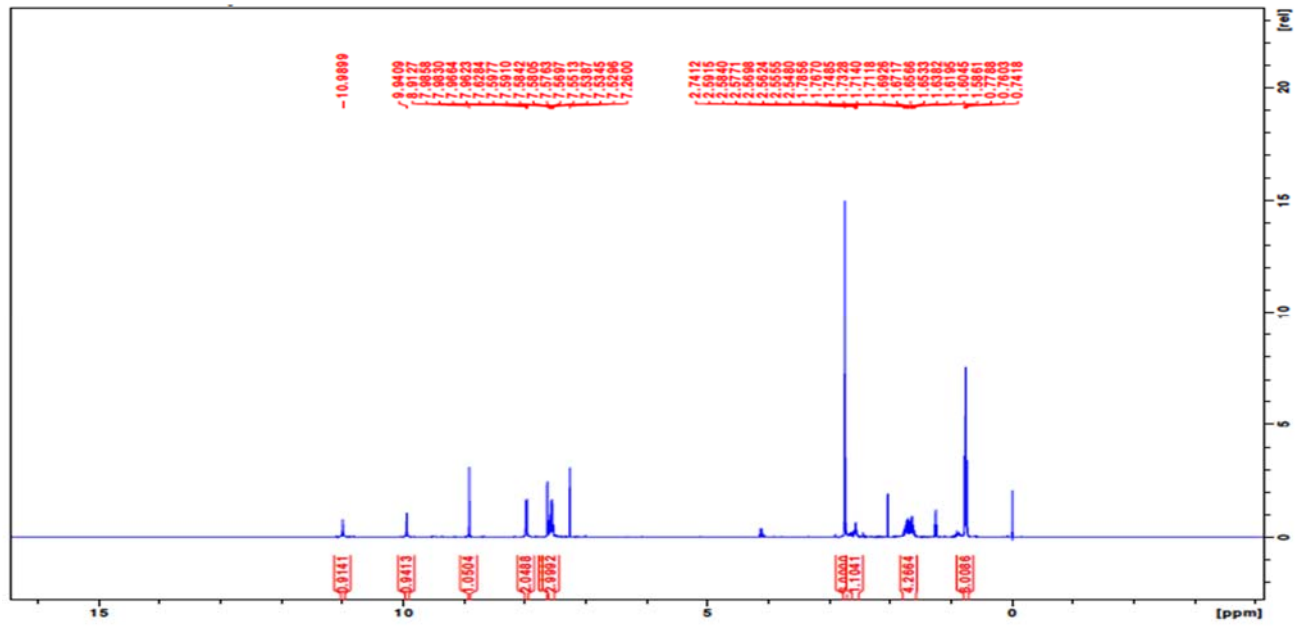
5b-9

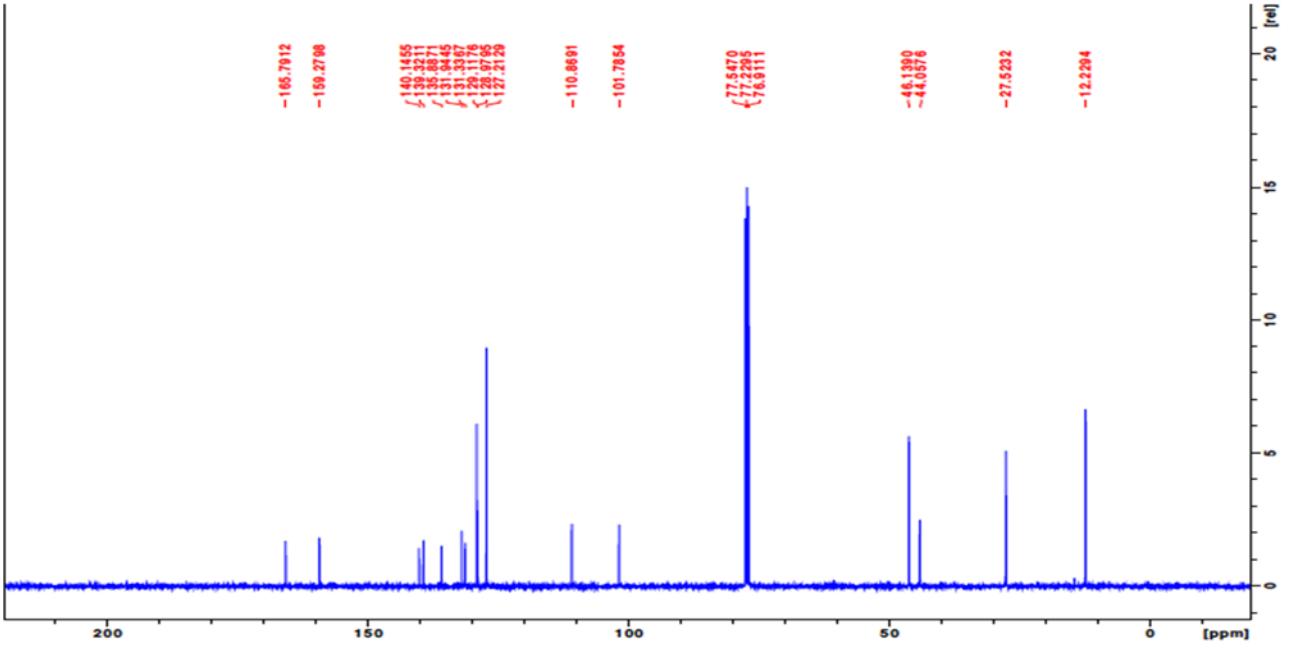


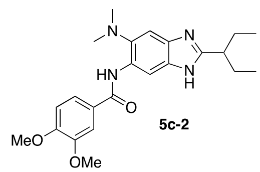



N
N
HN
O
N
H

5c-1

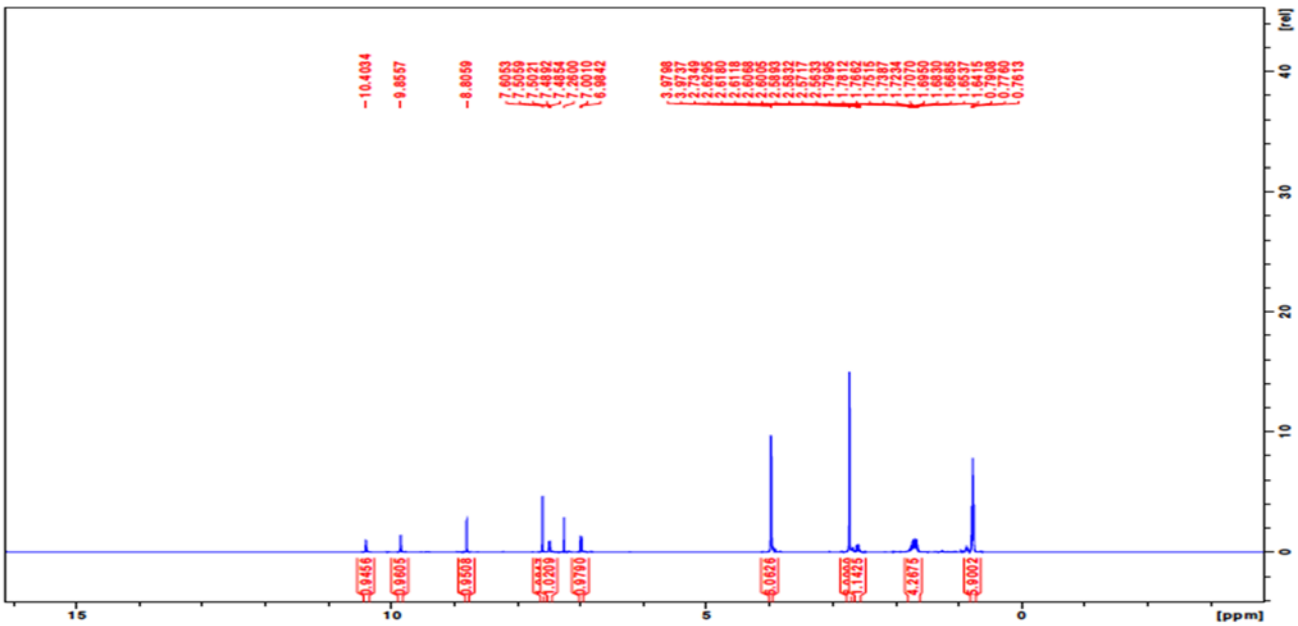

10.9899
9.9409
8.0127
7.9857
7.9830
7.9664
7.9623
7.6284
7.5977
7.5910
7.5842
7.5805
7.5763
7.5697
7.5513
7.5387
7.5345
7.5296
7.2600
2.7412
2.5915
2.5840
2.5771
2.5688
2.5654
2.5565
2.5480
1.7856
1.7670
1.7485
1.7328
1.7140
1.7118
1.6926
1.6717
1.6566
1.6533
1.6382
1.6196
1.6045
1.5861
0.7788
0.7603
0.7418
0.9141
0.9413
0.5504
2.0408
2.9992
4.0000
3.1041
4.2664
8.0086
15
10
5
0
[ppm]
[rel]
20
15
10
5
0


165.7912
159.2798
140.1455
139.3211
135.8871
131.9445
131.3367
129.1176
128.9795
127.2129
110.8691
101.7854
77.5470
77.2295
76.9111
46.1390
44.0576
27.5232
12.2284
200
150
100
50
0
[ppm]
[rel]
20
15
10
5
0

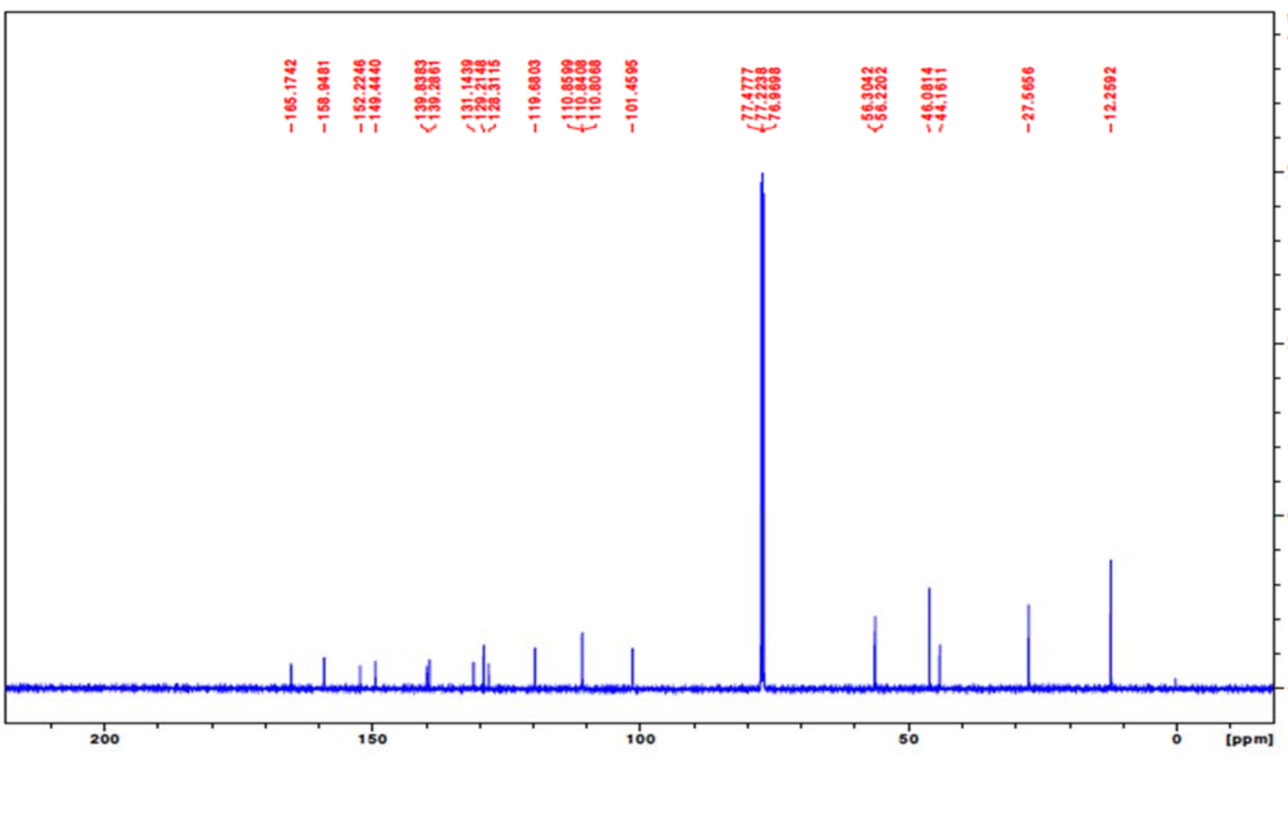


HN
N
H
O
MeO
OMe

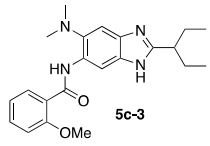
5c-2



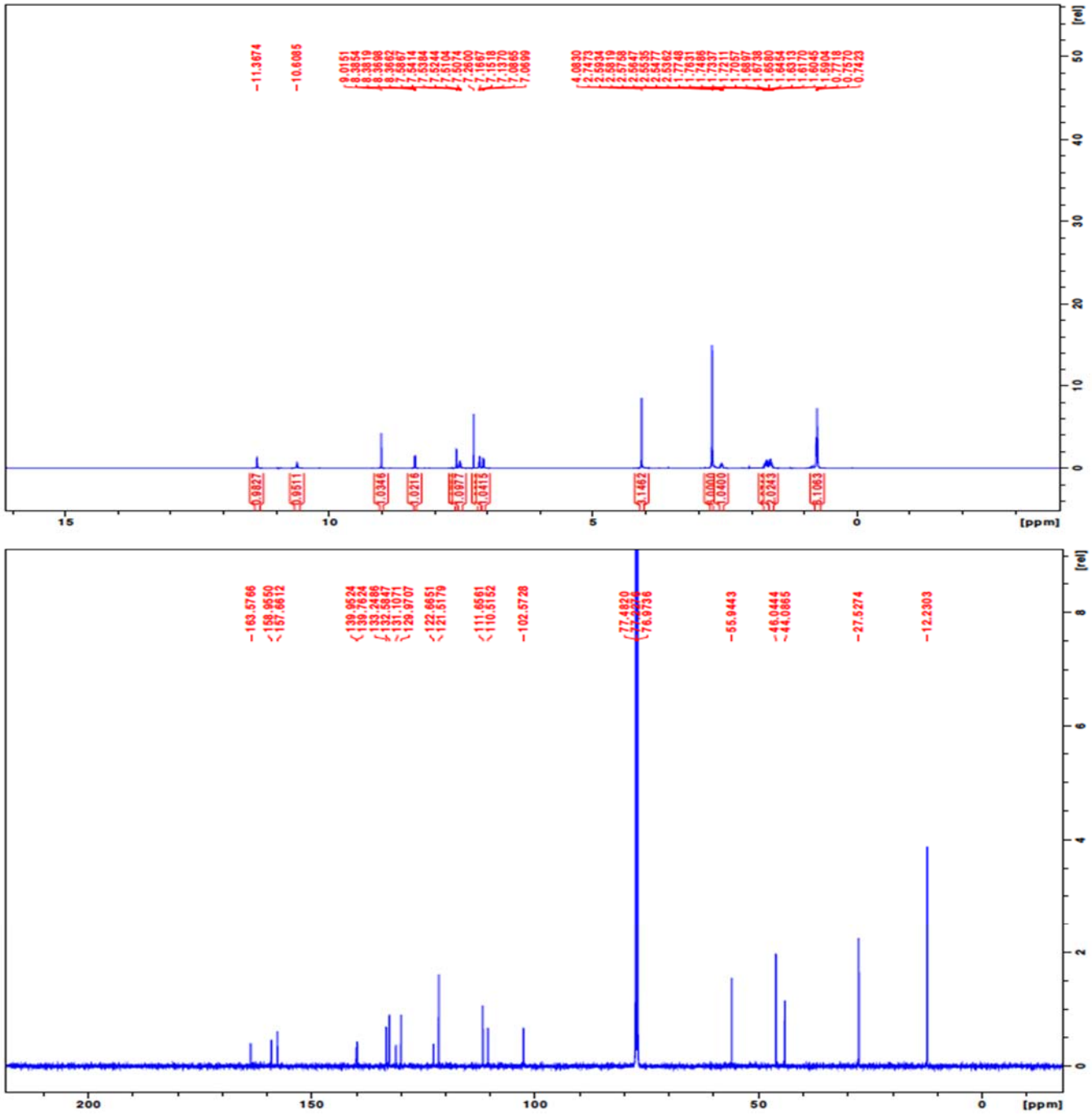
10.4034
9.8557
8.8059
7.6053 7.5059 7.5021 7.4892 7.4854 7.2600 7.0010 6.9842
3.9798 3.9737 2.7349 2.6295 2.6180 2.6118 2.6068 2.6005 2.5893 2.5832 2.5717 2.5533 1.7995 1.7812 1.7762 1.7515 1.7387 1.7234 1.7070 1.6950 1.6830 1.6685 1.6537 1.6415 0.7908 0.7760 0.7613
0.9456 0.9605 0.9508 1.0209 0.9790 6.0626 1.1425 4.2875 5.9002
15 10 5 0 [ppm]
[rel]

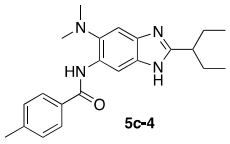


165.1742
158.9481
152.2246
149.4440
139.8383
139.2861
131.1439
129.2148
128.3115
119.6803
110.8599
110.8408
110.8068
101.4595
77.4777
77.2238
76.9698
56.3042
56.2202
46.0814
44.1611
27.5656
12.2592
200 150 100 50 0 [ppm]
[rel]

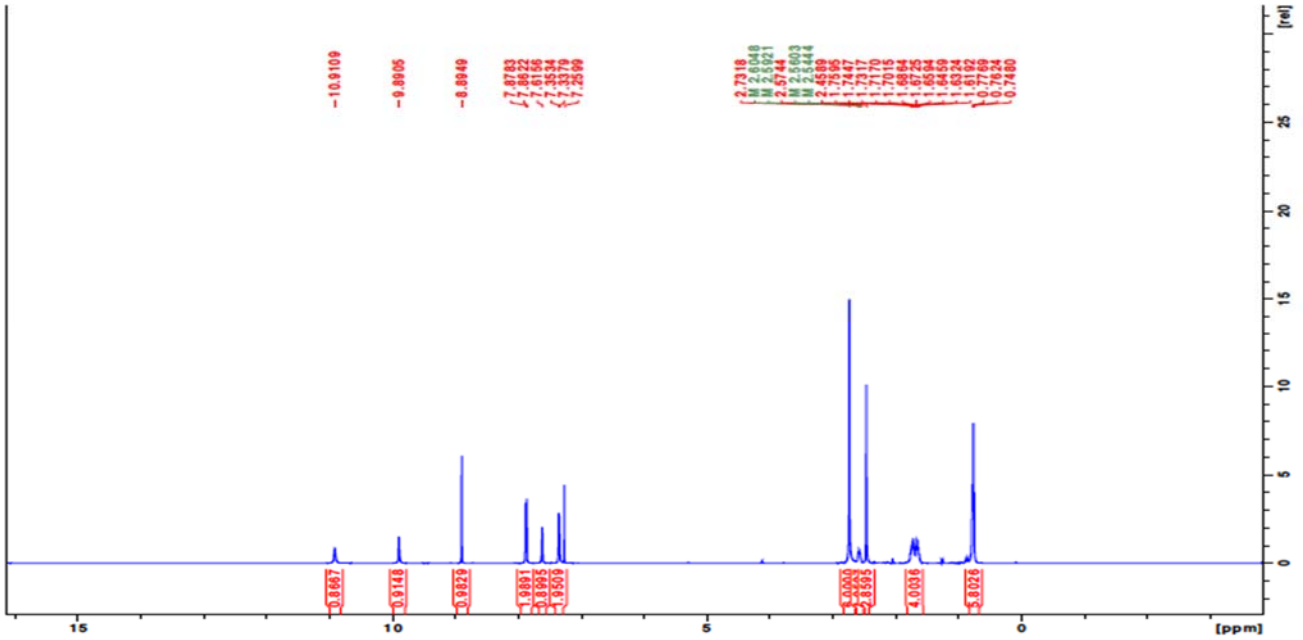

N
N
HN
N
H
O
OMe

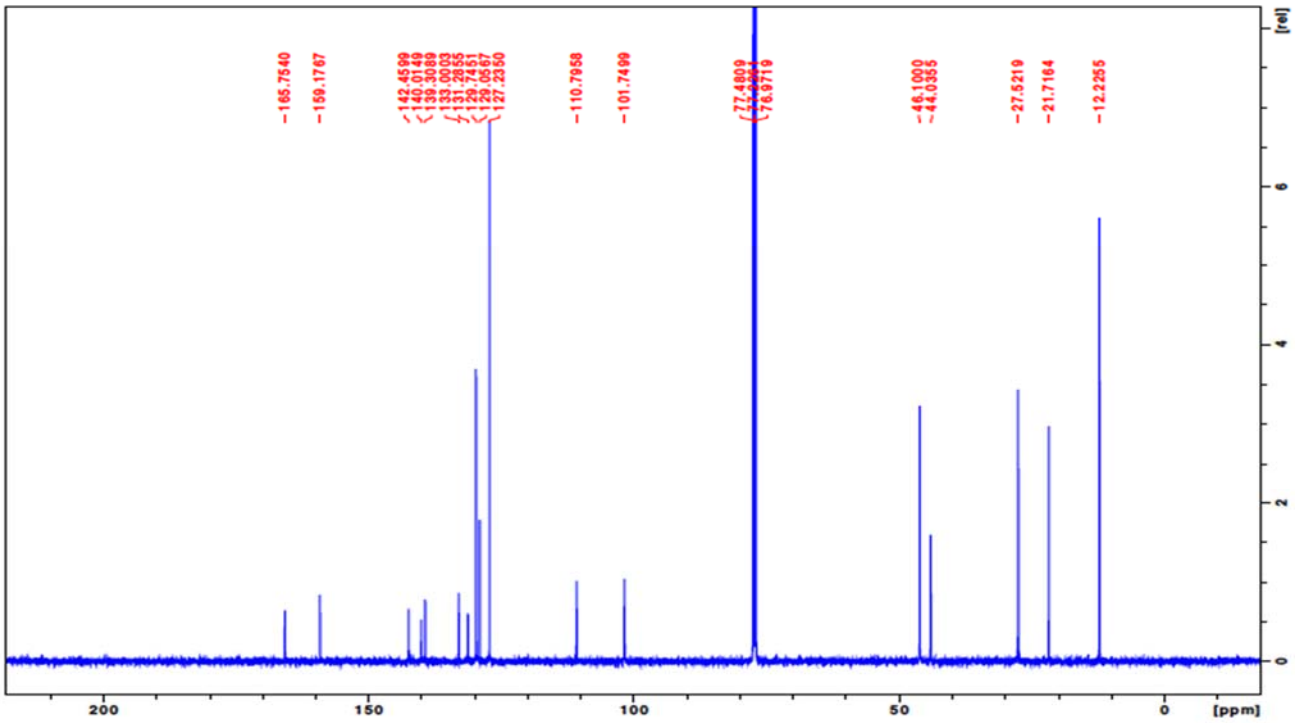
5c-3

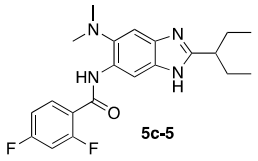

-11.3674
-10.6085
9.0151
8.3854
8.3819
8.3698
8.3662
7.5867
7.5414
7.5384
7.5244
7.5174
7.5074
7.2600
7.1697
7.1570
7.1370
7.0865
7.0699
4.0830
2.7473
2.5934
2.5819
2.5758
2.5647
2.5535
2.5477
2.5362
1.7748
1.7631
1.7486
1.7337
1.7211
1.7057
1.6897
1.6738
1.6580
1.6454
1.6313
1.6170
1.6045
1.5904
0.7718
0.7570
0.7423
0.9827
0.9511
1.0346
1.0216
1.0977
1.0415
5.1462
4.0000
1.0400
2.0243
5.1063
15
10
5
0
[ppm]
[rel]
-163.5766
-158.9550
-157.8612
139.9624
139.7624
133.2486
132.5847
131.1071
129.9707
122.6651
121.5179
111.6581
110.5152
-102.5728
77.4820
77.2274
76.9736
-55.9443
-46.0444
-44.0865
-27.5274
-12.2303
200
150
100
50
0
[ppm]
[rel]


N
N
HN
N
H
O

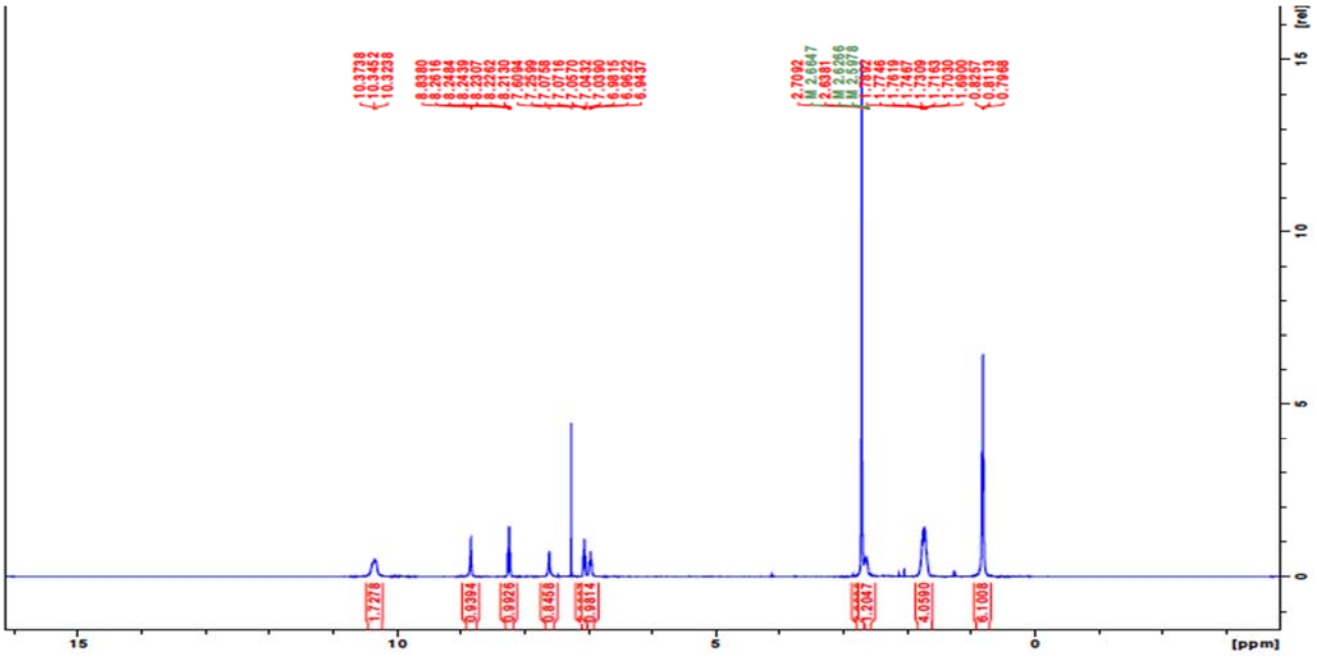
5c-4

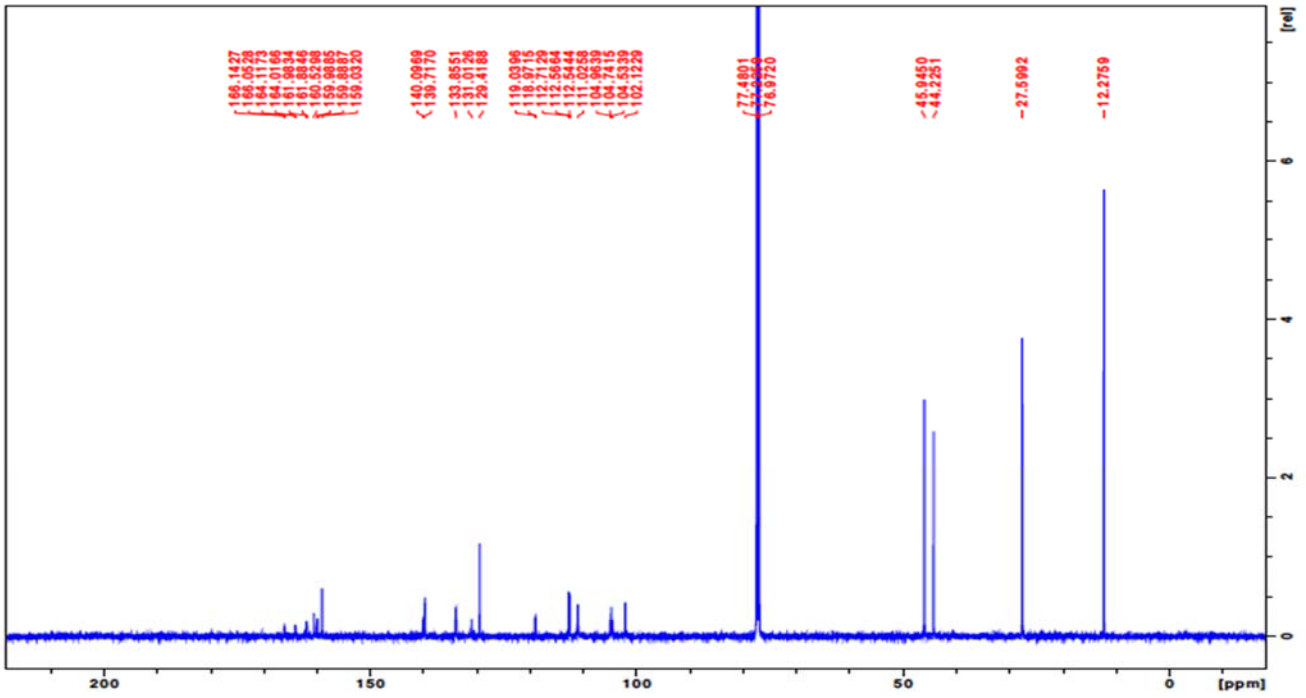

-10.9109
-9.8905
-8.8949
7.8783
7.8622
7.6156
7.3534
7.3379
7.2590
2.7318
2.6048
2.5921
2.5744
2.5603
2.5444
2.4589
1.7596
1.7447
1.7317
1.7170
1.7015
1.6864
1.6725
1.6594
1.6459
1.6324
1.6192
0.7769
0.7624
0.7480
0.8667
0.9148
0.8829
1.9891
0.8995
1.3509
4.0036
5.0026
15
10
5
0
[ppm]
[rel]

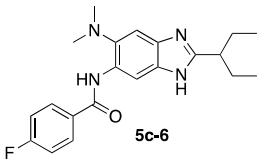

165.7540
159.1767
142.4599
140.0149
139.3089
133.0003
131.2855
129.7451
129.0567
127.2350
110.7958
101.7499
77.4809
77.0001
76.9719
46.1000
44.0355
27.5219
21.7164
12.2255
200
150
100
50
0
[ppm]
[rel]


N
N
HN
N
H
O
F
F

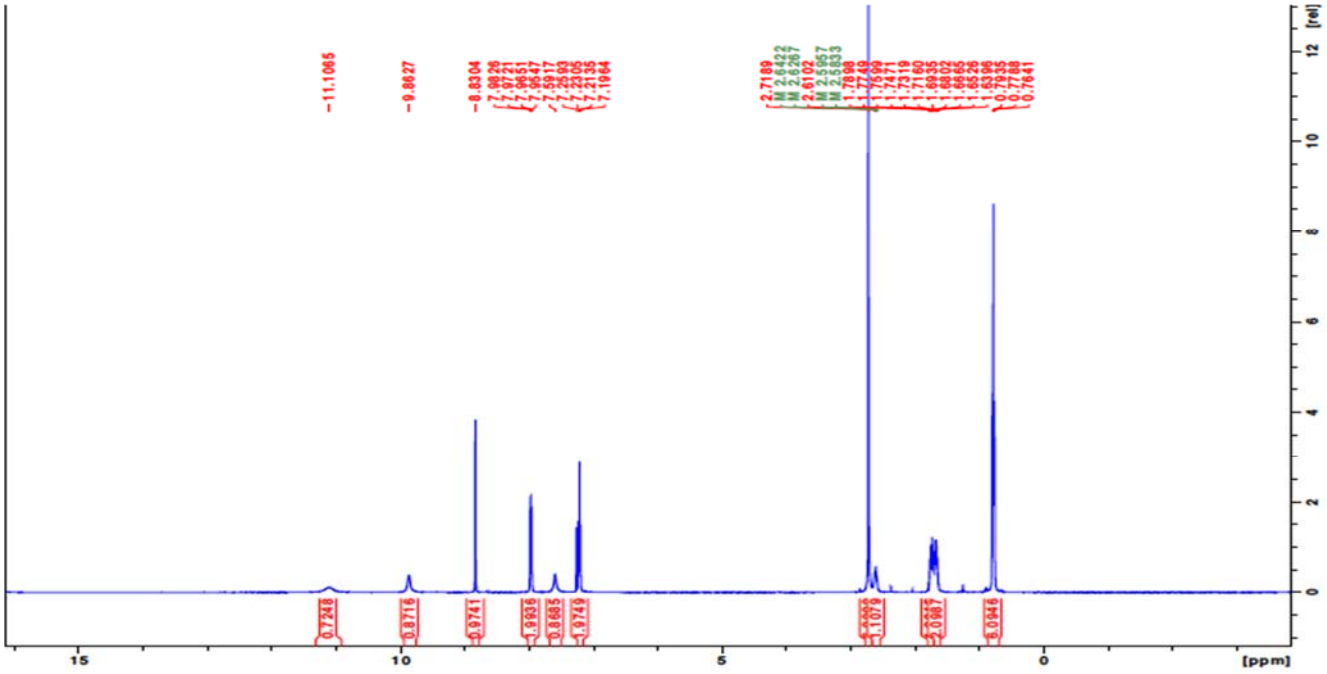
5c-5

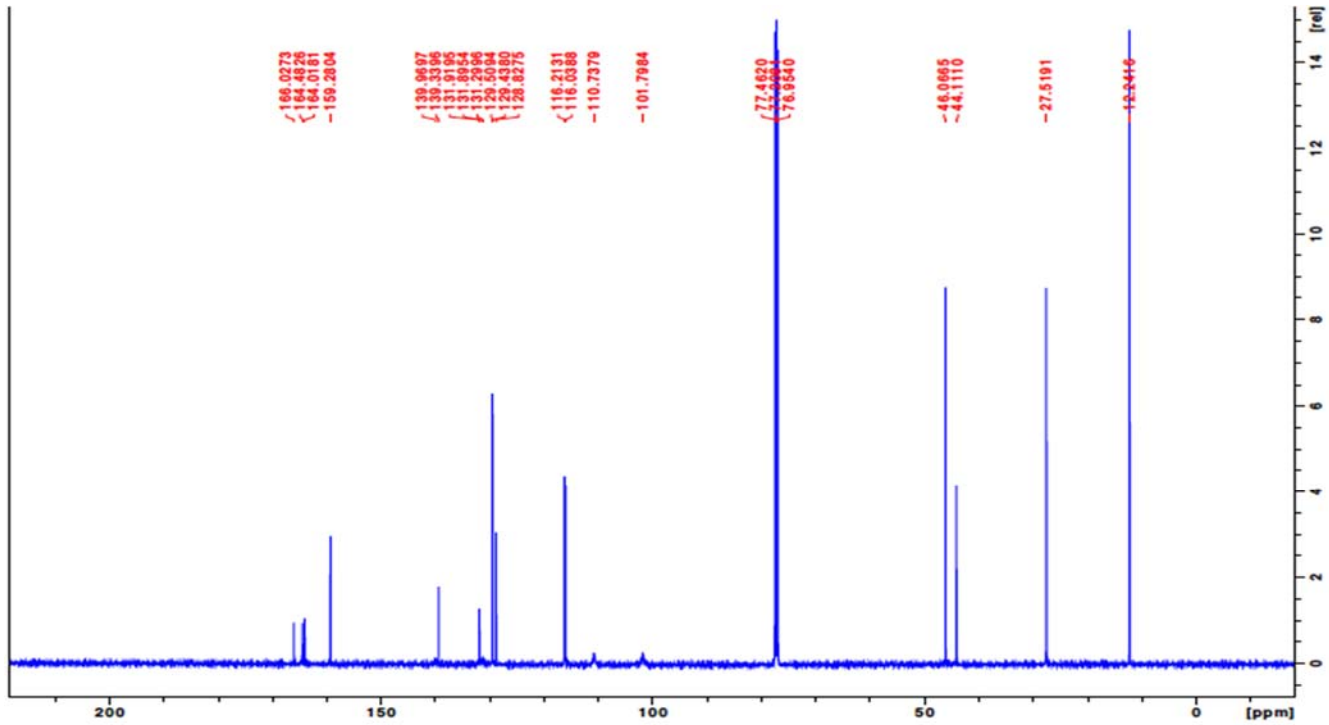

10.3738
10.3452
10.3238
8.8380
8.2616
8.2484
8.2439
8.2307
8.2262
8.2130
7.8094
7.7599
7.7058
7.7016
7.6570
7.6432
7.6390
6.9815
6.9622
6.9437
2.7092
M 2.6647
2.6381
M 2.6286
M 2.5978
1.7692
1.7746
1.7619
1.7467
1.7309
1.7163
1.7030
1.6900
0.8257
0.8113
0.7988
1.7278
0.9394
0.9926
0.8458
0.9814
1.2047
4.0590
6.1008
15
10
5
0
[ppm]
[rel]

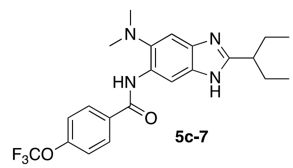

166.1427
166.0528
164.1173
164.0166
161.9834
161.8846
160.5298
159.9885
159.8887
159.0320
140.0969
139.7170
133.8551
131.0126
129.4188
119.0396
118.9715
112.7129
112.5664
112.5444
111.0258
104.9639
104.7415
104.5339
102.1229
77.4801
77.1679
76.8720
45.9450
44.2251
27.5992
12.2759
200
150
100
50
0
[ppm]
[rel]


N
HN
N
H
O
F

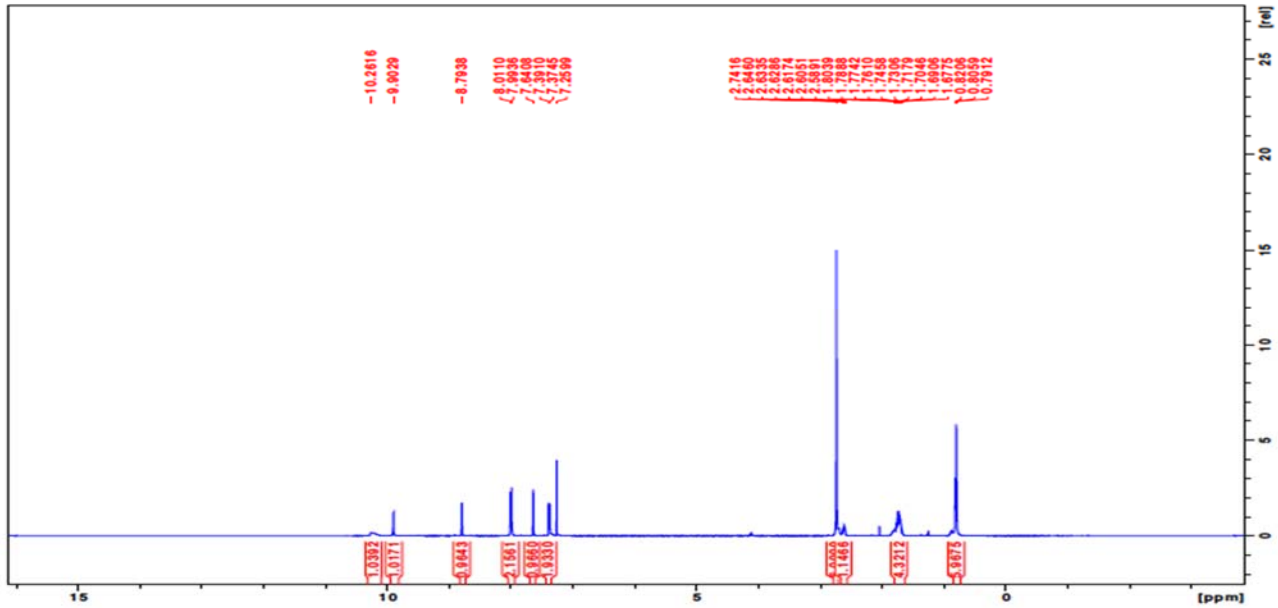
5c-6

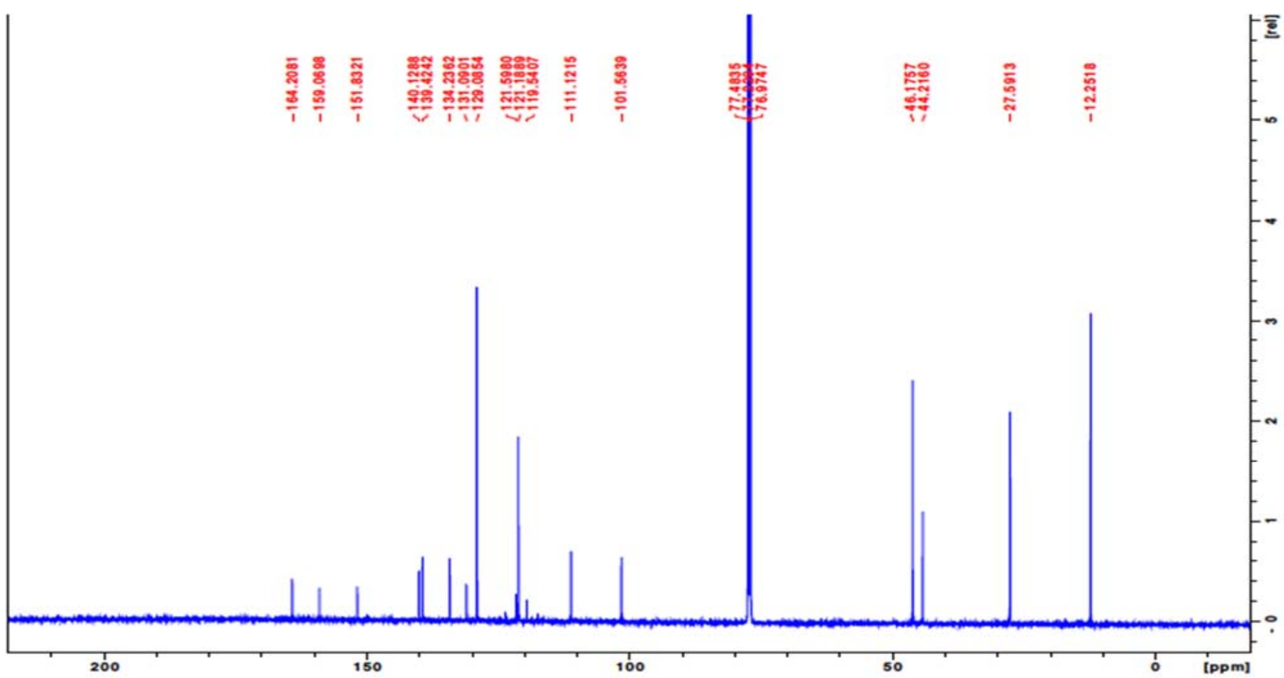

-11.1065
-9.8627
-8.8304
7.9826
7.9721
7.9651
7.9647
7.5917
7.2593
7.2305
7.2135
7.1964
2.7189
2.6422
2.6267
2.6102
2.5957
2.5833
1.7749
1.7591
1.7491
1.7310
1.7160
1.6932
1.6802
1.6662
1.6526
1.6396
0.7935
0.7788
0.7641
0.7248
0.8716
0.9741
0.9936
0.8685
1.9749
1.1079
1.1019
2.0987
8.0946
15
10
5
0
[ppm]
12
[rel]

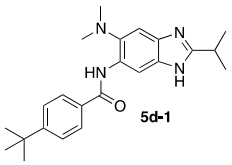

166.0273
164.4826
164.0181
159.2804
139.9697
139.3396
131.9195
131.8954
131.2996
129.5094
129.4380
128.8275
116.2131
116.0088
110.7379
101.7984
77.4620
77.0584
76.9540
46.0665
44.1110
27.5191
12.3416
200
150
100
50
0
[ppm]
14
[rel]



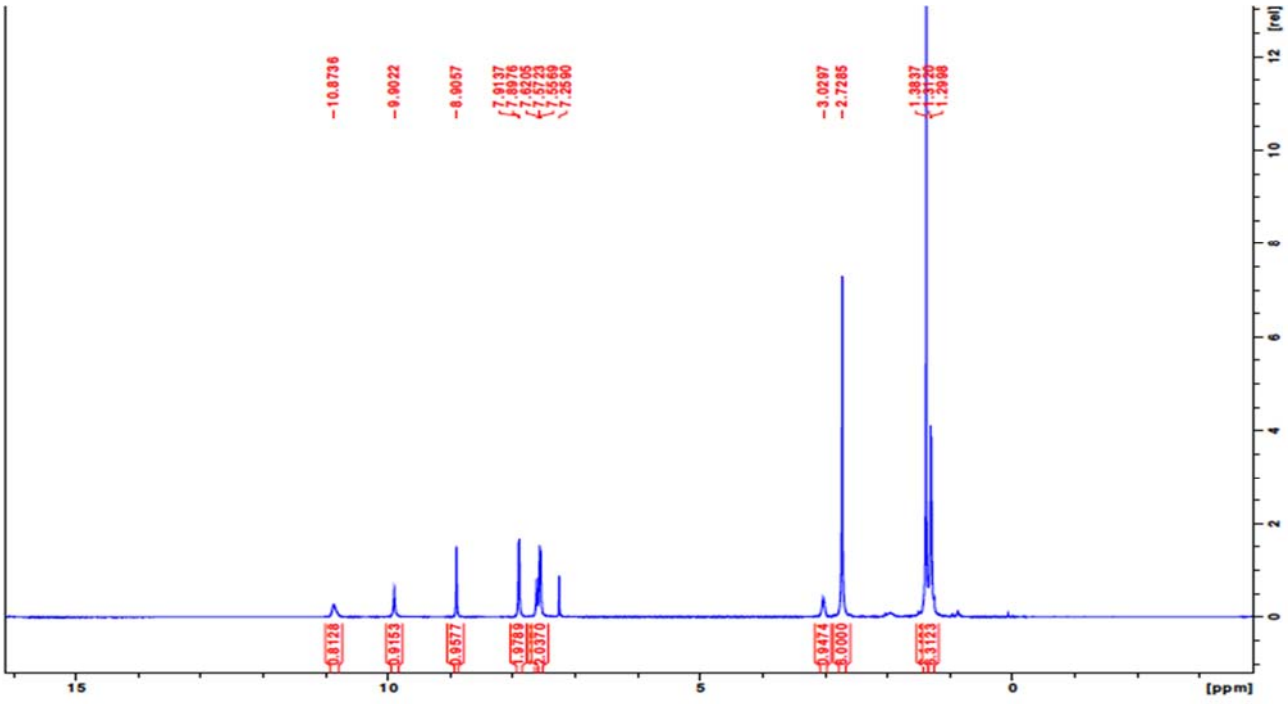
5c-7

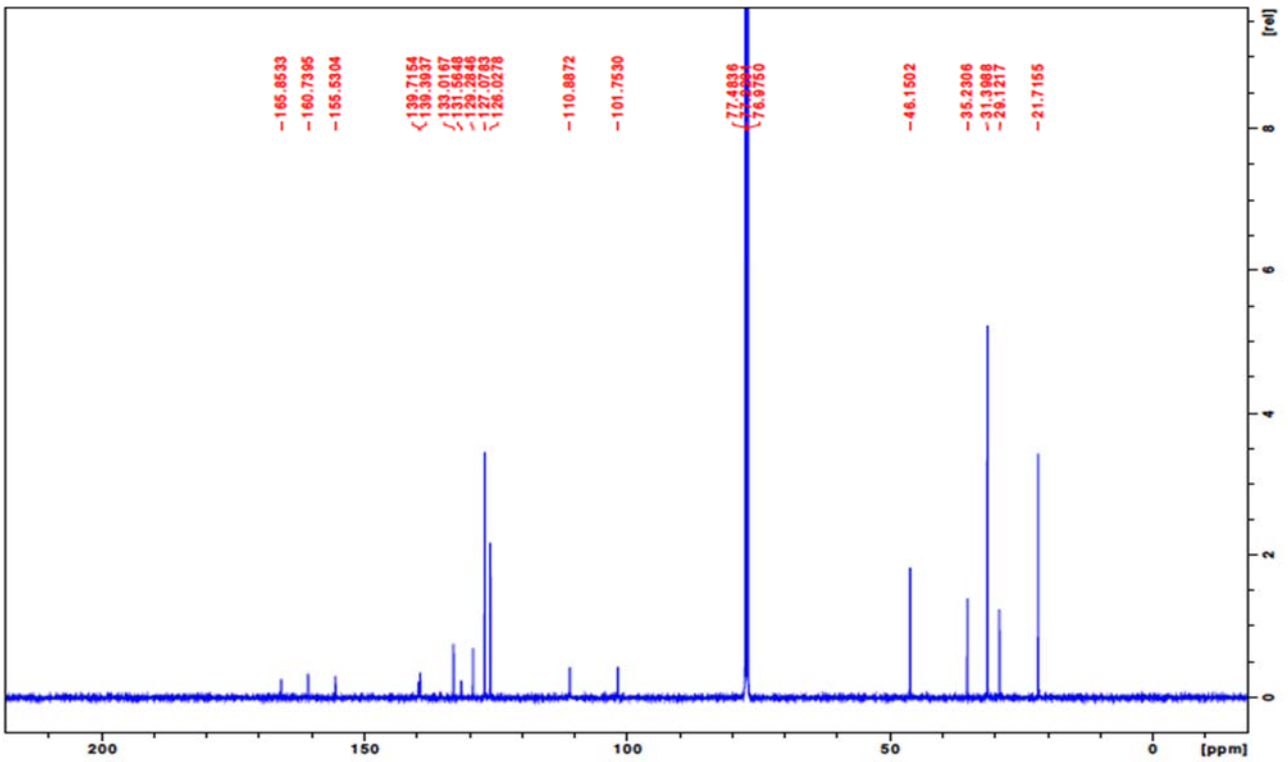


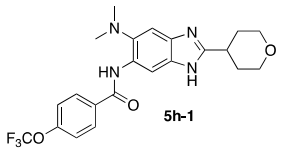



N
N
HN
O
N
H

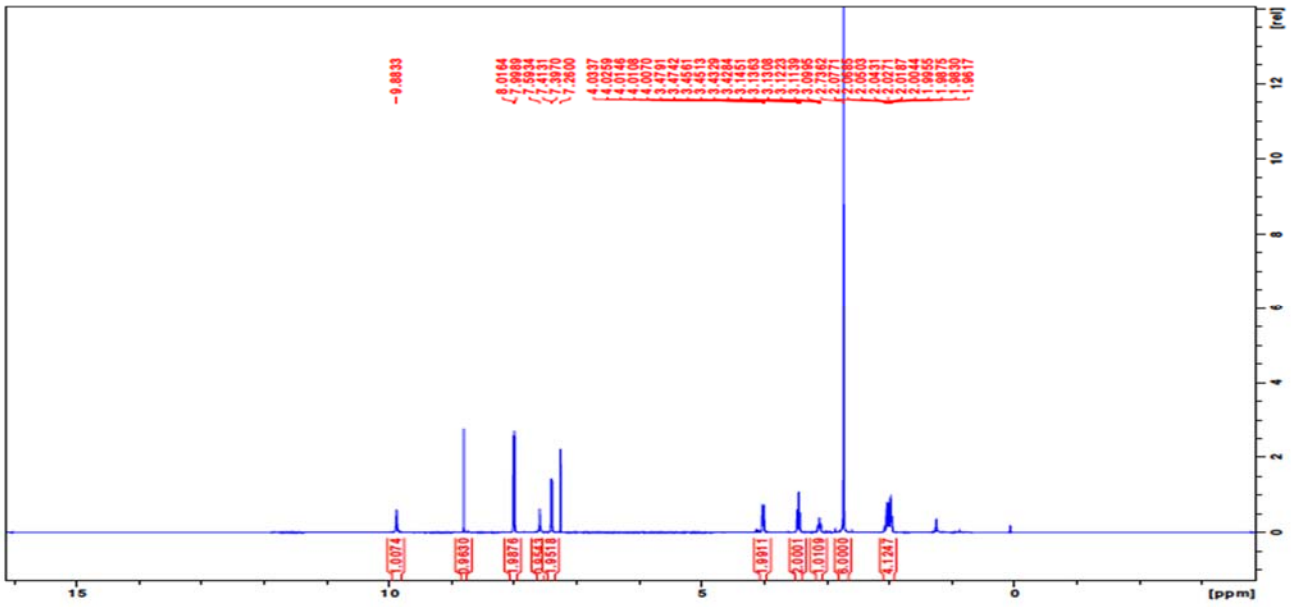
5d-1

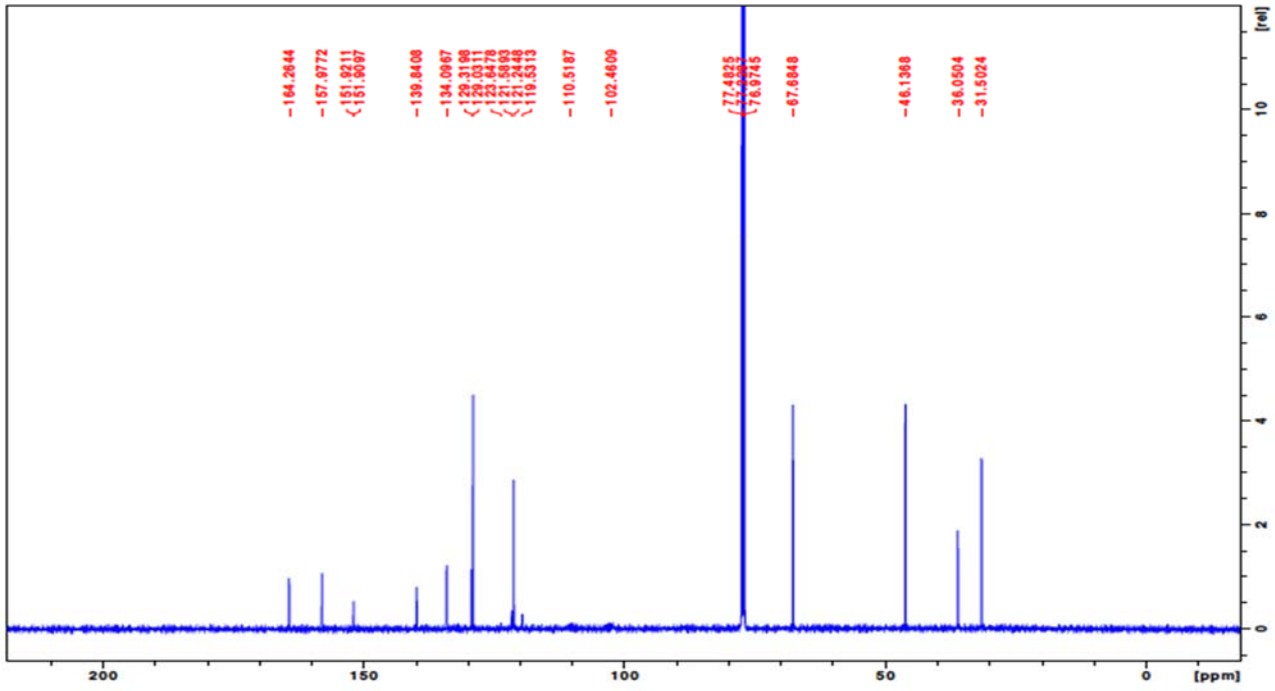

10.8736
9.9022
8.9057
7.9137
7.8876
7.6205
7.5723
7.5569
7.2590
3.0297
2.7285
1.3837
1.3120
1.2968
0.8128
0.9153
0.9577
1.9789
3.3370
2.0370
0.9474
3.0000
3.1123
0.3123
15
10
5
0
[ppm]
[rel]

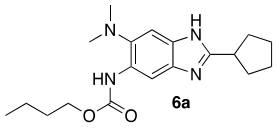

165.8533
160.7396
155.5304
139.7154
139.3937
133.0167
131.5648
129.2846
127.0763
126.0278
110.8872
101.7530
77.4836
77.0672
76.9750
46.1502
35.2306
31.3088
29.1217
21.7155
200
150
100
50
0
[ppm]
[rel]


N
N
HN
O
N
H
O
F3CO

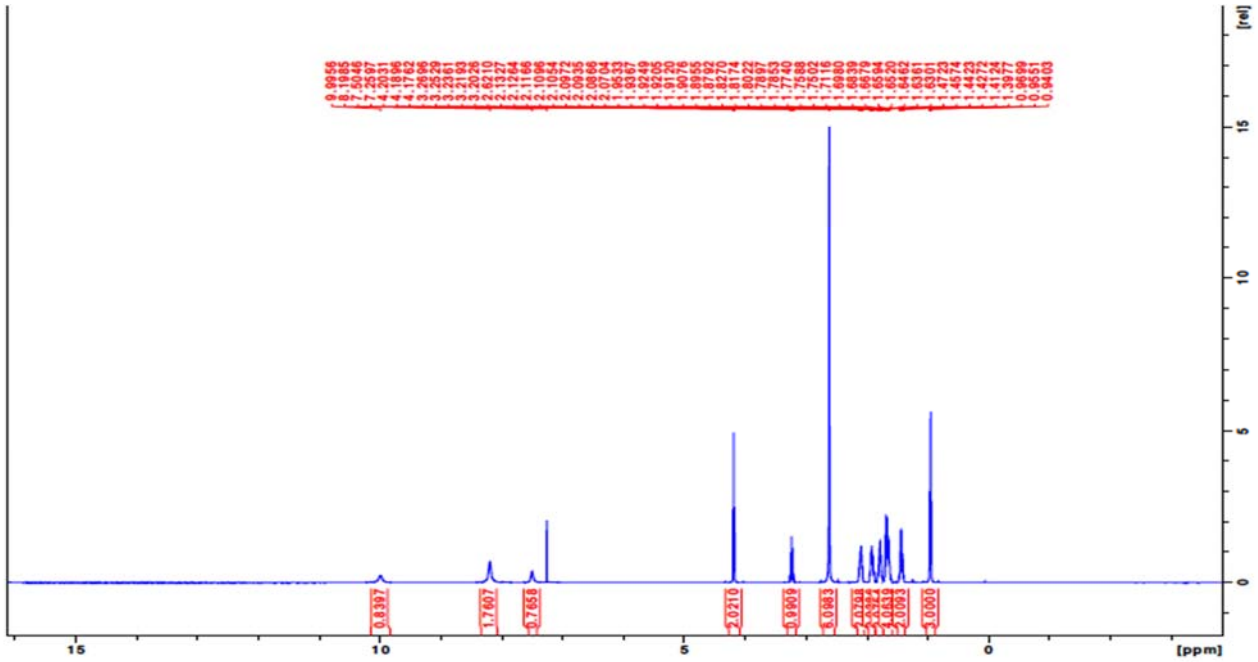
5h-1

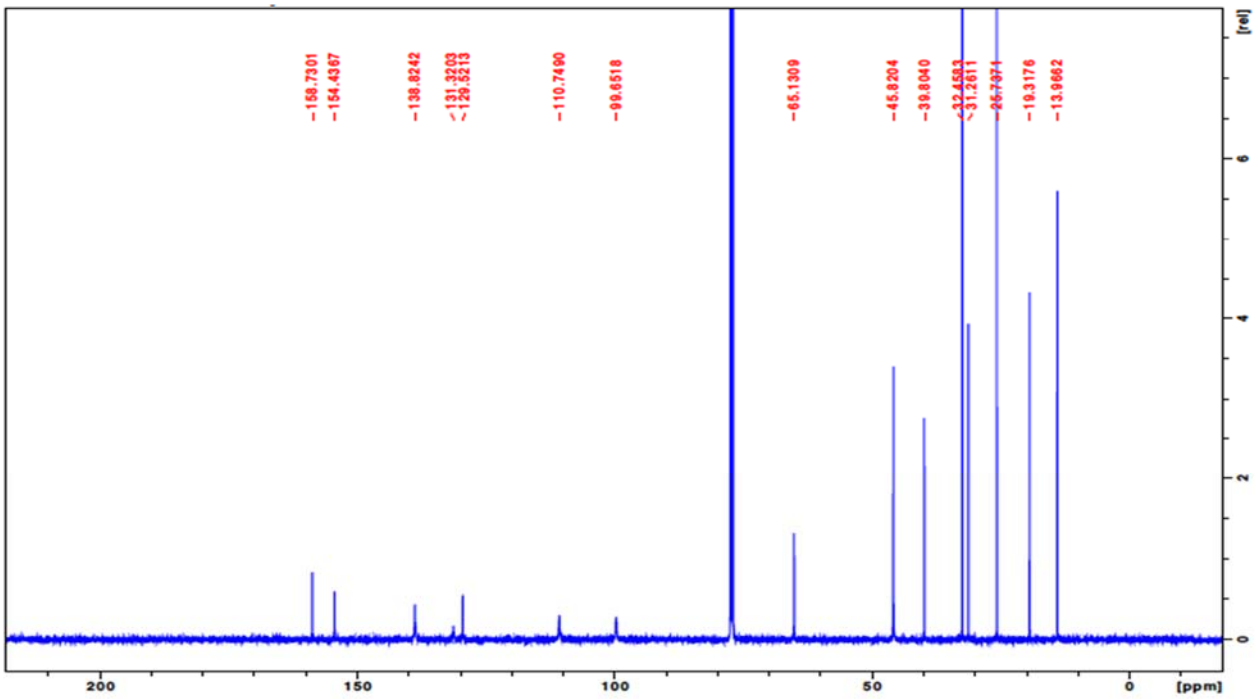

9.8833
8.0184
7.9989
7.5934
7.4131
7.3970
7.2600
4.0337
4.0259
4.0148
4.0070
3.4711
3.4742
3.4561
3.4513
3.4329
3.4284
3.1451
3.1308
3.1223
3.1139
3.0996
2.7342
2.0771
2.0593
2.0503
2.0431
2.0271
2.0167
2.0044
1.9845
1.9810
1.9617
1.0074
0.9830
1.9876
1.9543
1.9518
1.9911
2.0001
1.0109
0.0002
4.1247
15
10
5
0
[ppm]
[rel]

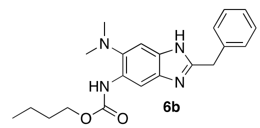

164.2644
157.9772
151.9211
151.9097
139.8408
134.0967
129.3190
129.0311
123.6478
121.5893
121.2448
119.5313
110.5187
102.4609
77.4825
77.2303
76.9745
67.6848
46.1388
36.0504
31.5024
200
150
100
50
0
[ppm]
[rel]



6a

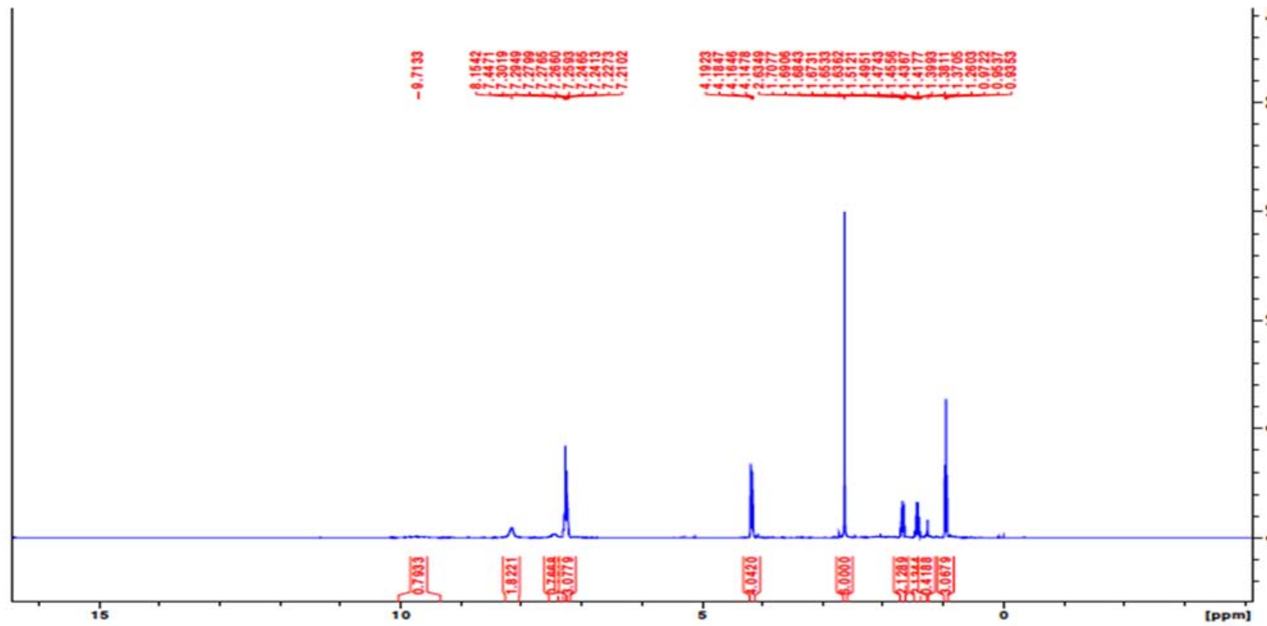




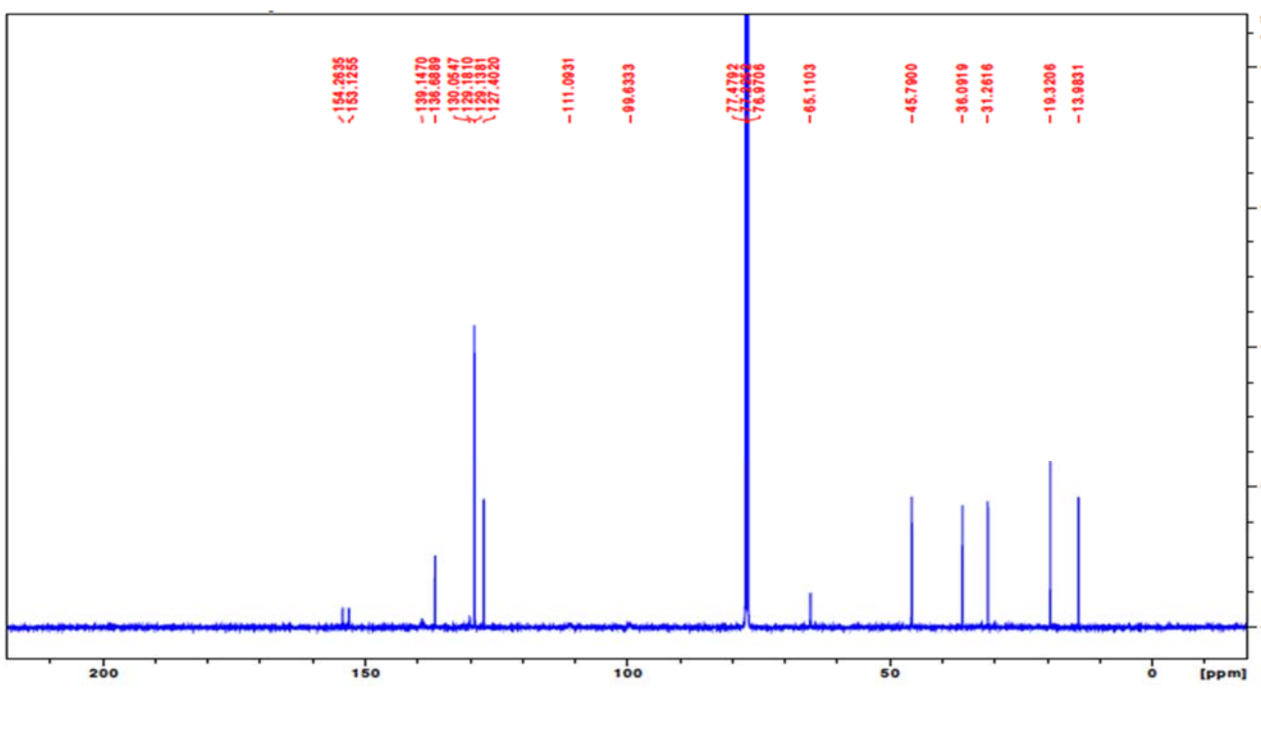


HN
N
H
N
O
O

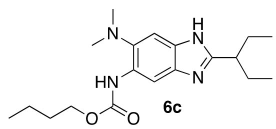
6b



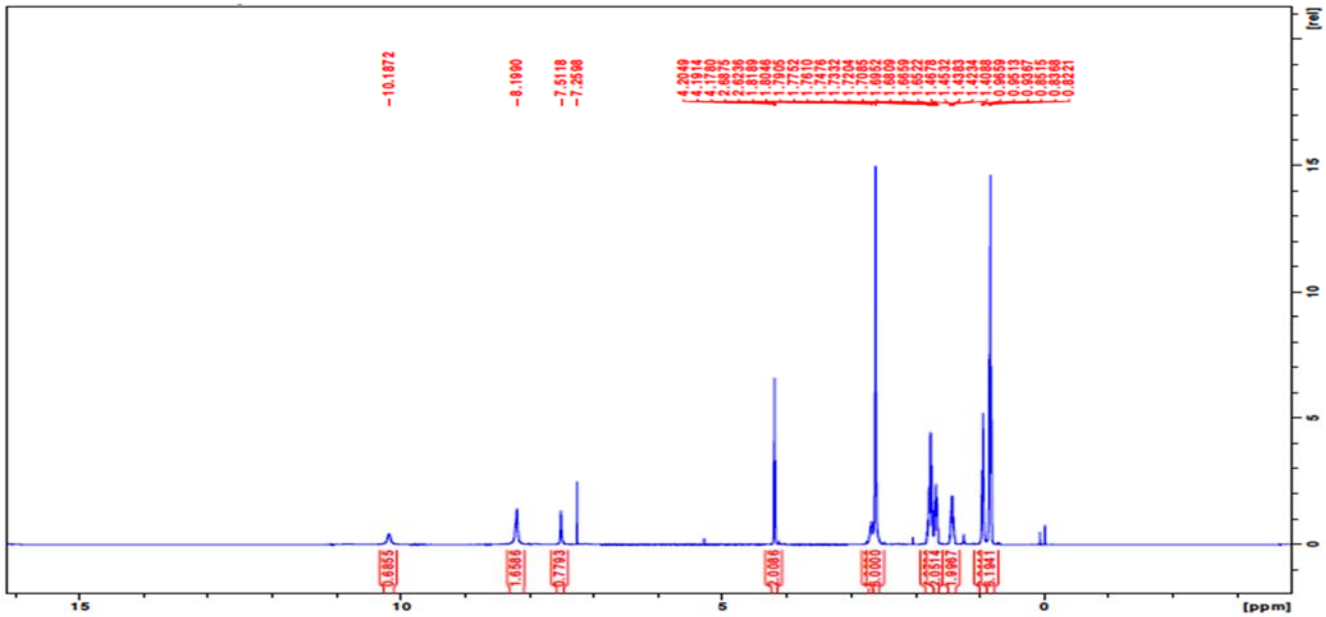
9.7133
8.1542
7.4471
7.3019
7.2949
7.2799
7.2765
7.2660
7.2593
7.2465
7.2413
7.2273
7.2102
4.1923
4.1847
4.1646
4.1478
2.6349
1.7077
1.6906
1.6843
1.6731
1.6533
1.6362
1.5121
1.4951
1.4743
1.4556
1.4367
1.4177
1.3993
1.3811
1.3705
1.2603
0.9722
0.9537
0.9353
0.7933
1.8221
3.0779
4.0420
6.0000
3.0679
15
10
5
0
[ppm]

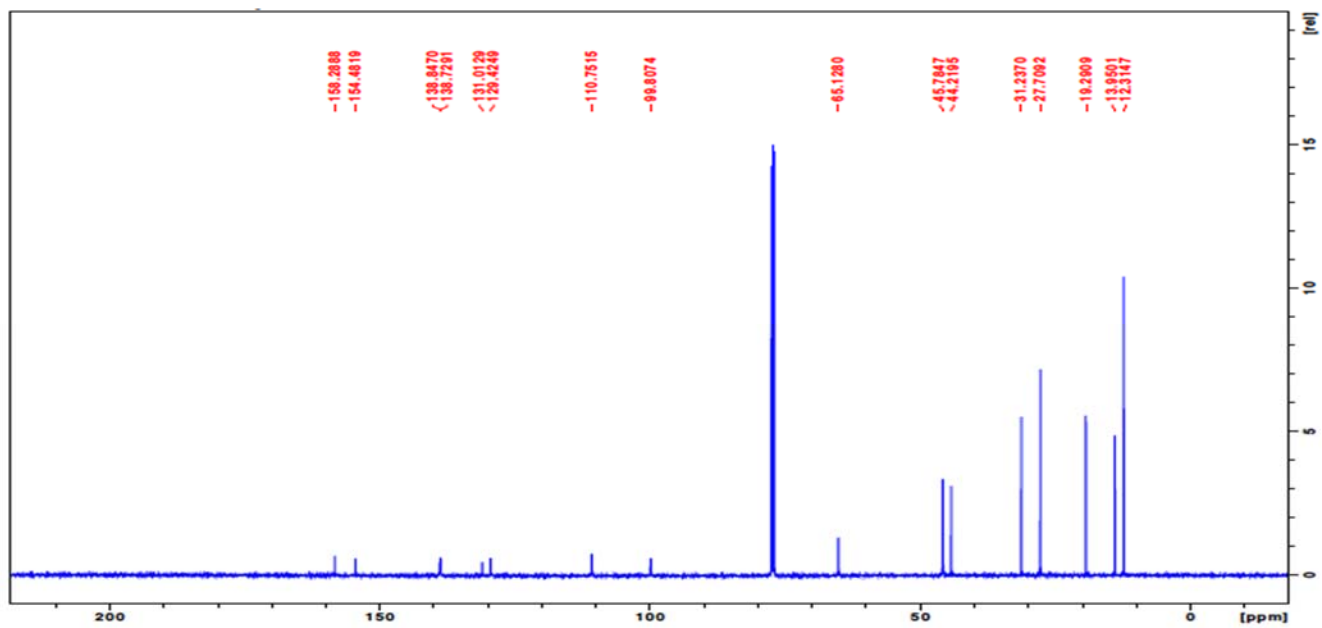


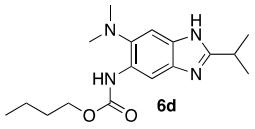
154.2635
153.1255
139.1470
136.6889
130.0547
129.1810
127.4020
111.0931
99.6333
77.4792
76.9706
65.1103
45.7900
36.0919
31.2616
19.3206
13.9831
[rel]
200
150
100
50
0
[ppm]



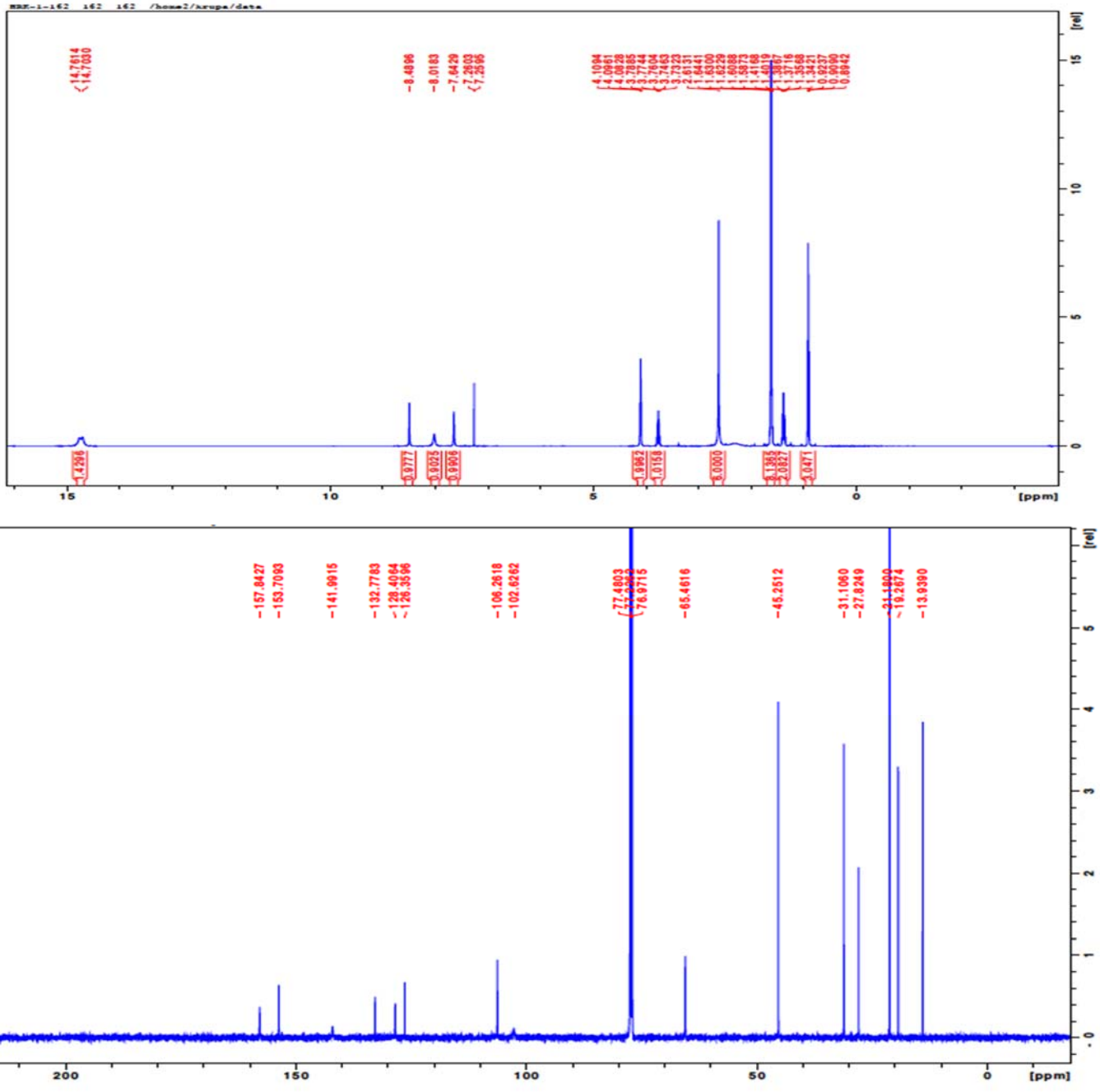
6c

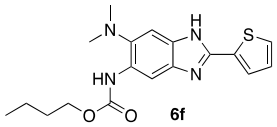




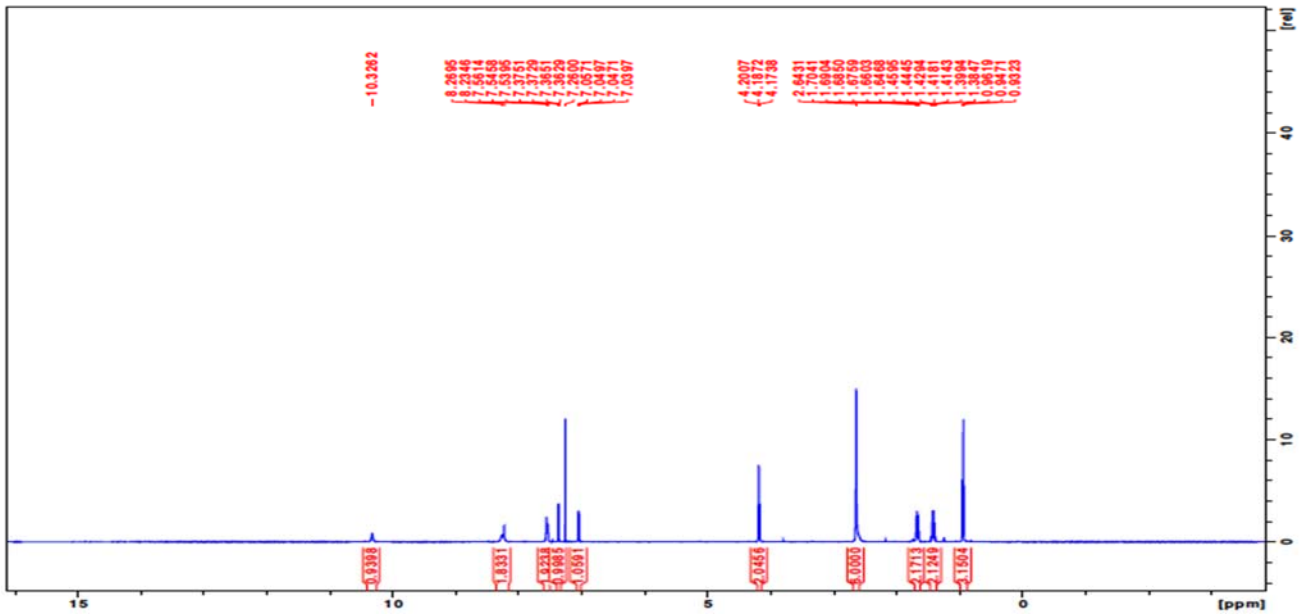


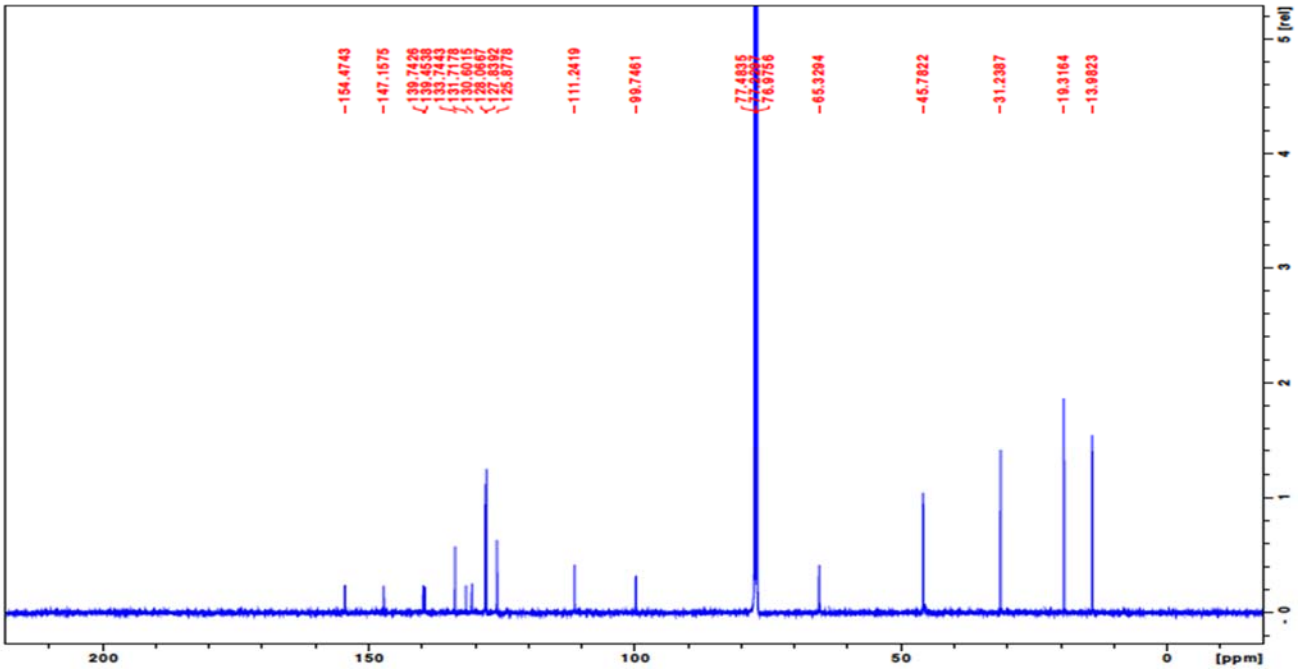
6d

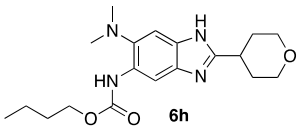




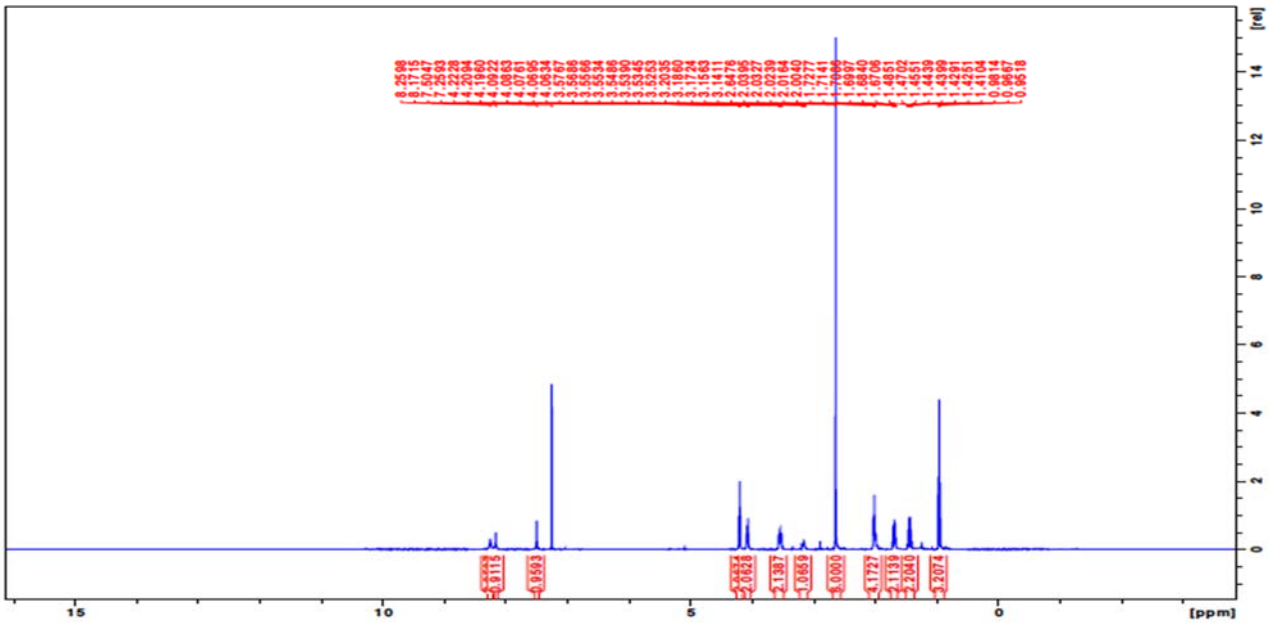
6f

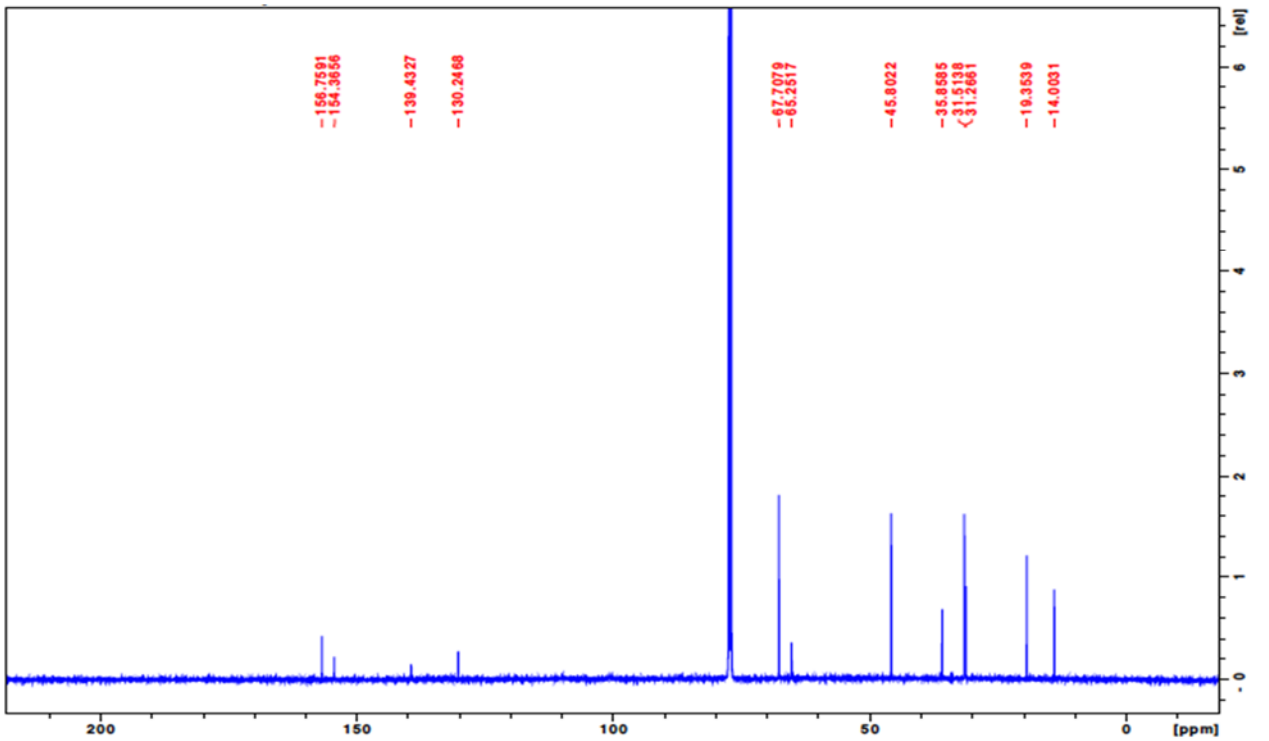

10.3262
8.2695
8.2346
7.5614
7.5458
7.5396
7.3751
7.3729
7.3651
7.3629
7.2600
7.0571
7.0497
7.0471
7.0097
4.2007
4.1872
4.1738
2.6431
1.7041
1.6904
1.6850
1.6759
1.6603
1.6468
1.4596
1.4445
1.4284
1.4181
1.4143
1.3994
1.3847
0.9819
0.9471
0.9023
0.9398
1.8331
1.9234
0.9985
0.9591
2.0456
5.0007
2.1713
2.1249
3.1504
15
10
5
0
[ppm]
[rel]

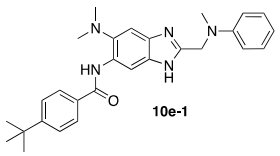

154.4743
147.1575
139.7426
139.4538
133.7443
131.7178
130.6015
128.0667
127.8392
125.8778
111.2419
99.7461
77.4835
77.1653
76.8756
65.3294
45.7822
31.2387
19.3164
13.9823
200
150
100
50
0
[ppm]
[rel]



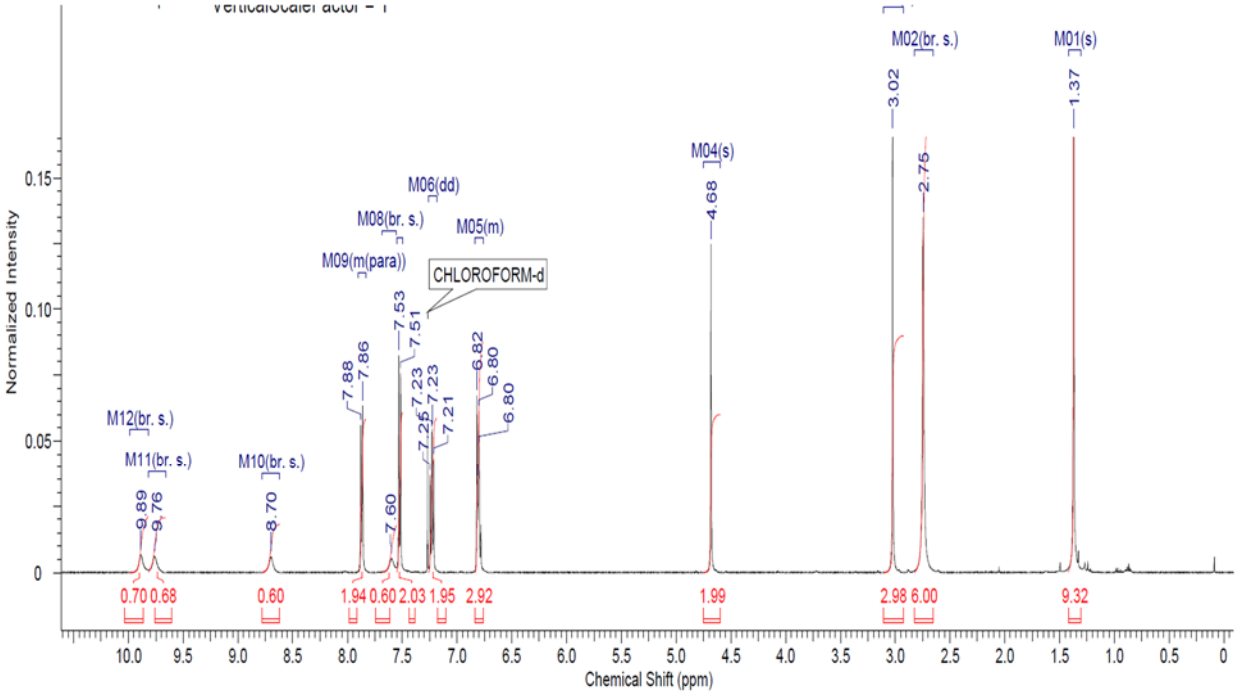
6h

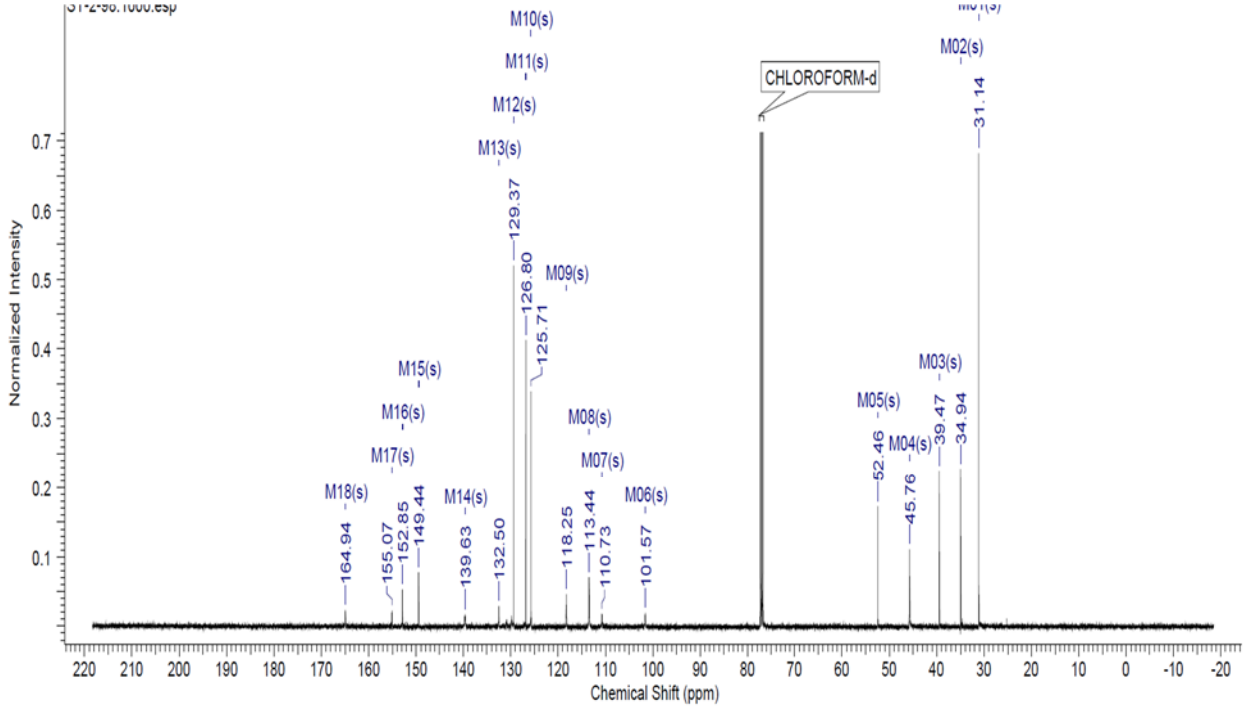


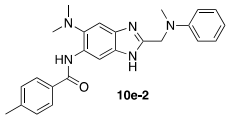


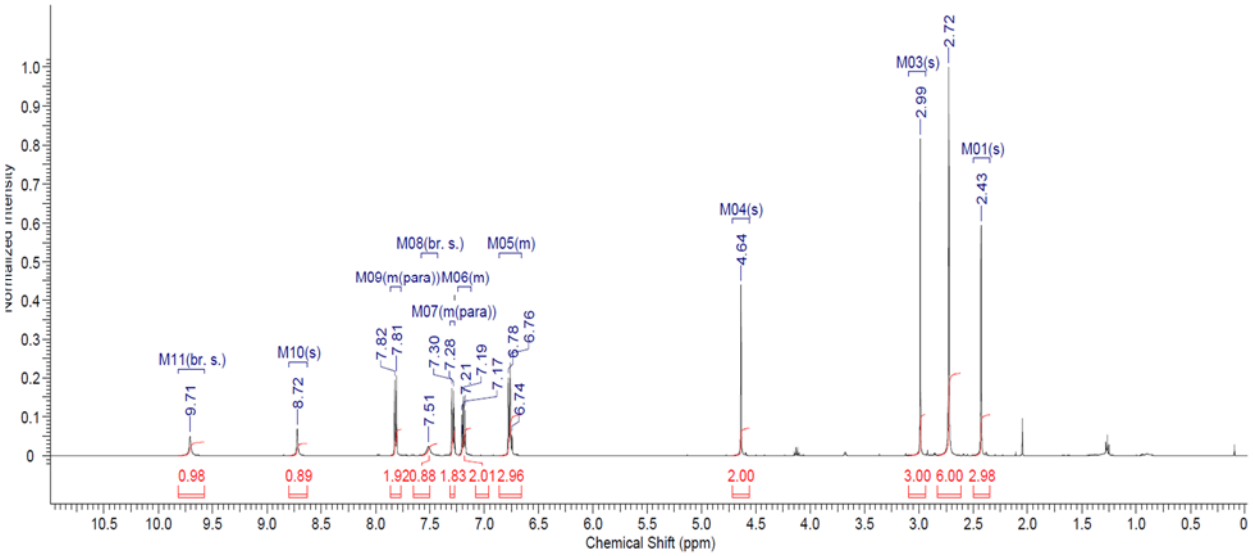


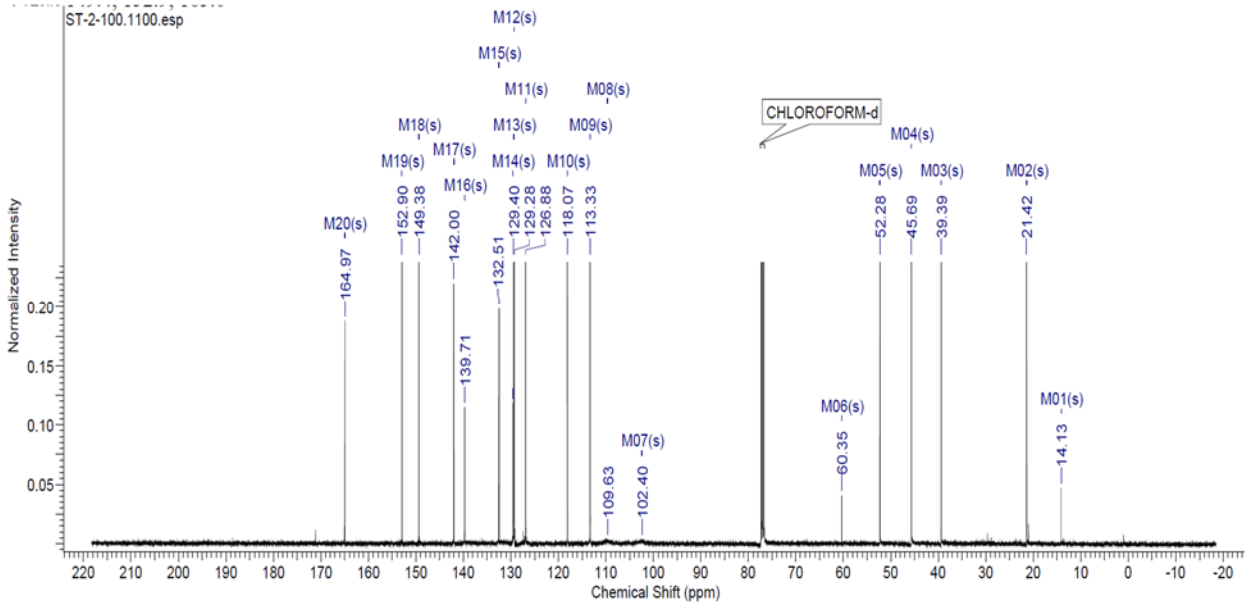
10e-1

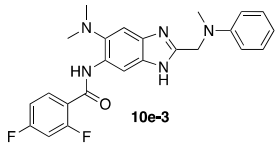

M12(br. s.)
M11(br. s.)
M10(br. s.)
M08(br. s.)
M06(dd)
M05(m)
M09(m(para))
CHLOROFORM-d
M04(s)
M02(br. s.)
M01(s)
9.89
9.76
8.70
7.88
7.86
7.60
7.53
7.51
7.23
7.23
7.25
7.21
6.82
6.80
6.80
4.68
3.02
2.75
1.37
0.70 0.68
0.60
1.94 0.60 2.03 1.95 2.92
1.99
2.98 6.00
9.32
Normalized Intensity
Chemical Shift (ppm)


M10(s)
M11(s)
M12(s)
M13(s)
M09(s)
M15(s)
M16(s)
M17(s)
M18(s)
M14(s)
M08(s)
M07(s)
M06(s)
M05(s)
M04(s)
M03(s)
M02(s)
CHLOROFORM-d
129.37
126.80
125.71
164.94
155.07
152.85
149.44
139.63
132.50
118.25
113.44
110.73
101.57
52.46
45.76
39.47
34.94
31.14
Normalized Intensity
Chemical Shift (ppm)

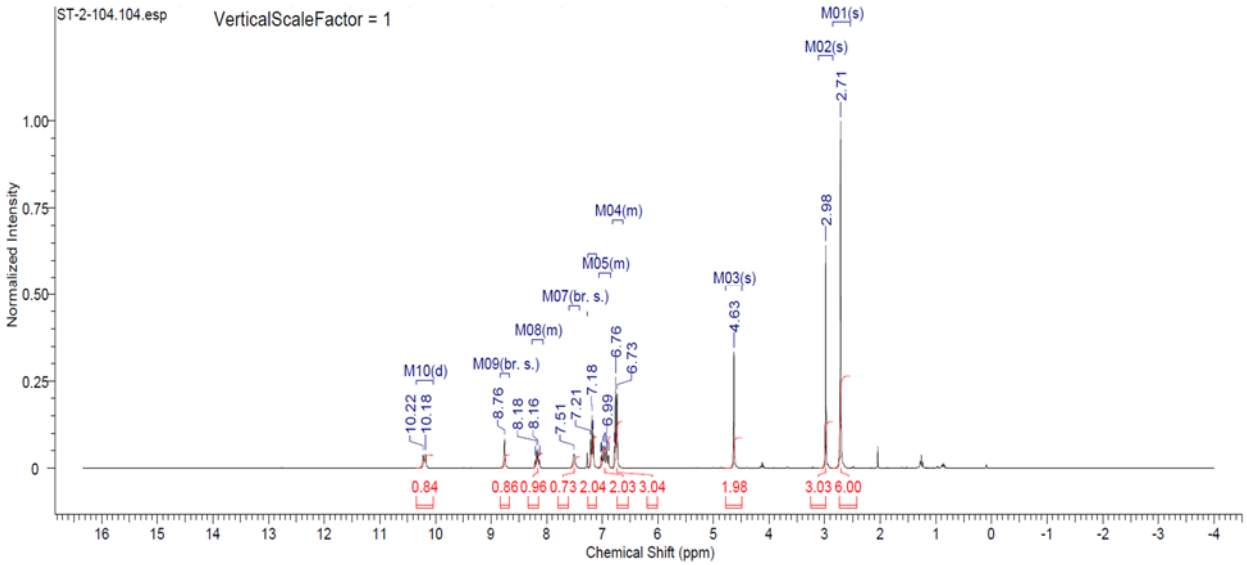

10e-2

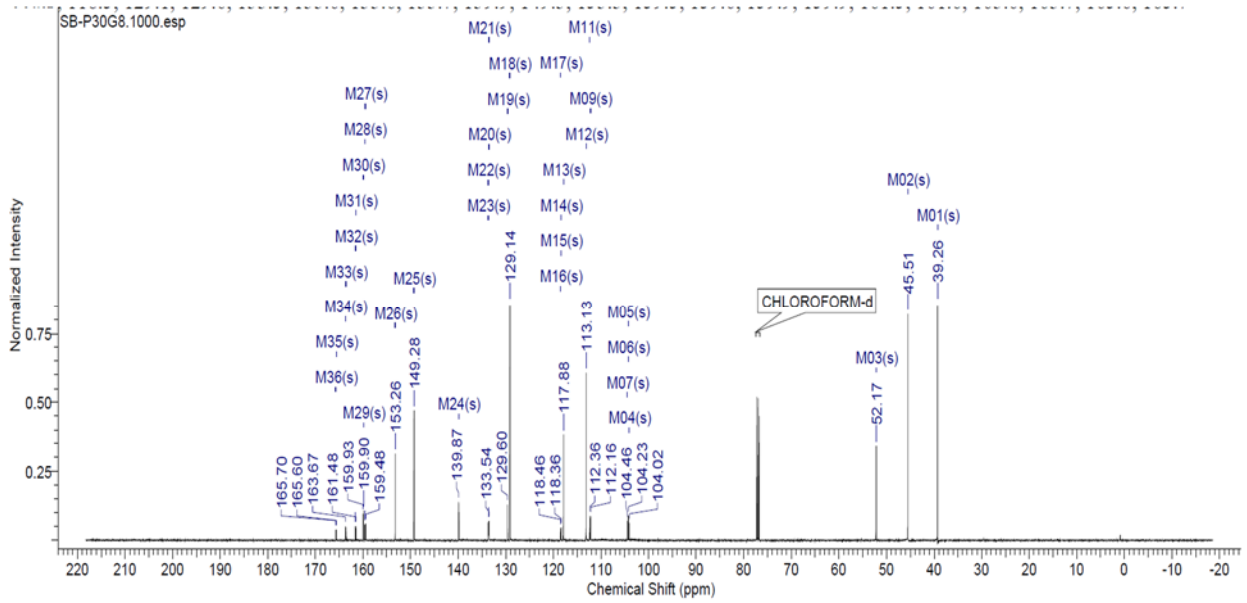

M11(br. s.)
M10(s)
M08(br. s.)
M05(m)
M09(m(para))
M06(m)
M07(m(para))
M04(s)
M03(s)
M01(s)
9.71
8.72
7.82
7.81
7.51
7.30
7.28
7.21
7.19
7.17
6.78
6.76
6.74
4.64
2.99
2.72
2.43
0.98
0.89
1.92
0.88
1.83
2.01
2.96
2.00
3.00
6.00
2.98
Chemical Shift (ppm)

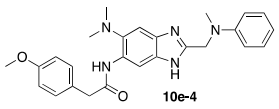

ST-2-100.1100.esp
M12(s)
M15(s)
M11(s)
M08(s)
M18(s)
M13(s)
M09(s)
M17(s)
M19(s)
M14(s)
M10(s)
M16(s)
M20(s)
CHLOROFORM-d
M04(s)
M05(s)
M03(s)
M02(s)
M06(s)
M07(s)
M01(s)
164.97
152.90
149.38
142.00
139.71
132.51
129.40
129.28
126.88
118.07
113.33
109.63
102.40
60.35
52.28
45.69
39.39
21.42
14.13
Normalized Intensity
Chemical Shift (ppm)

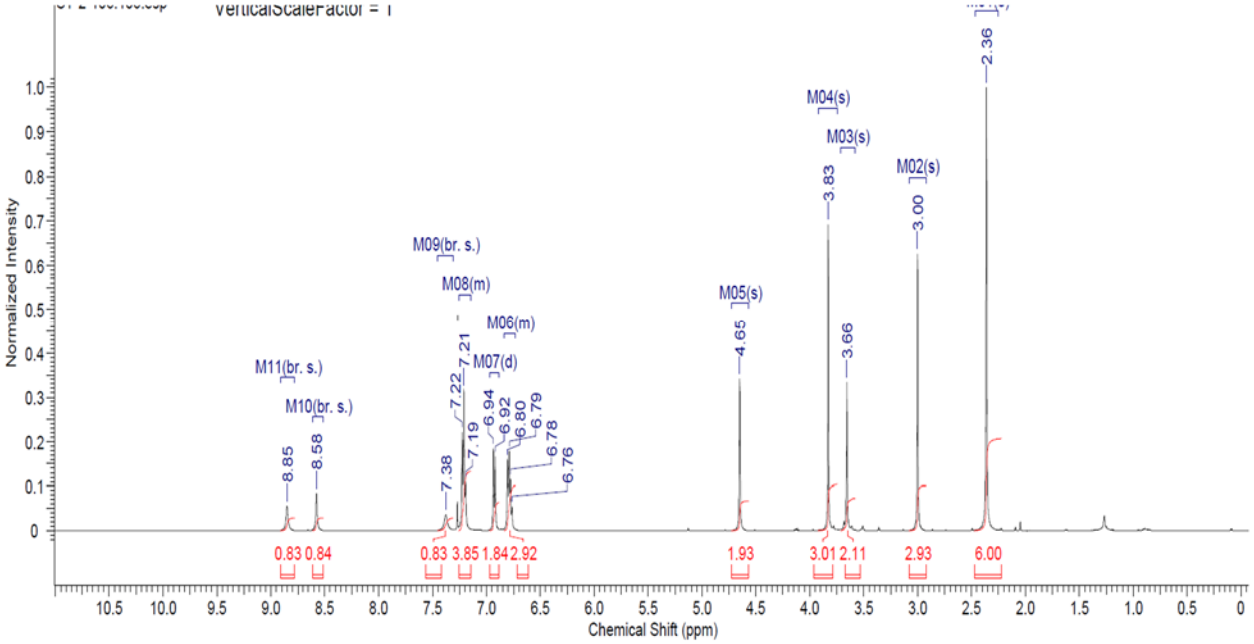


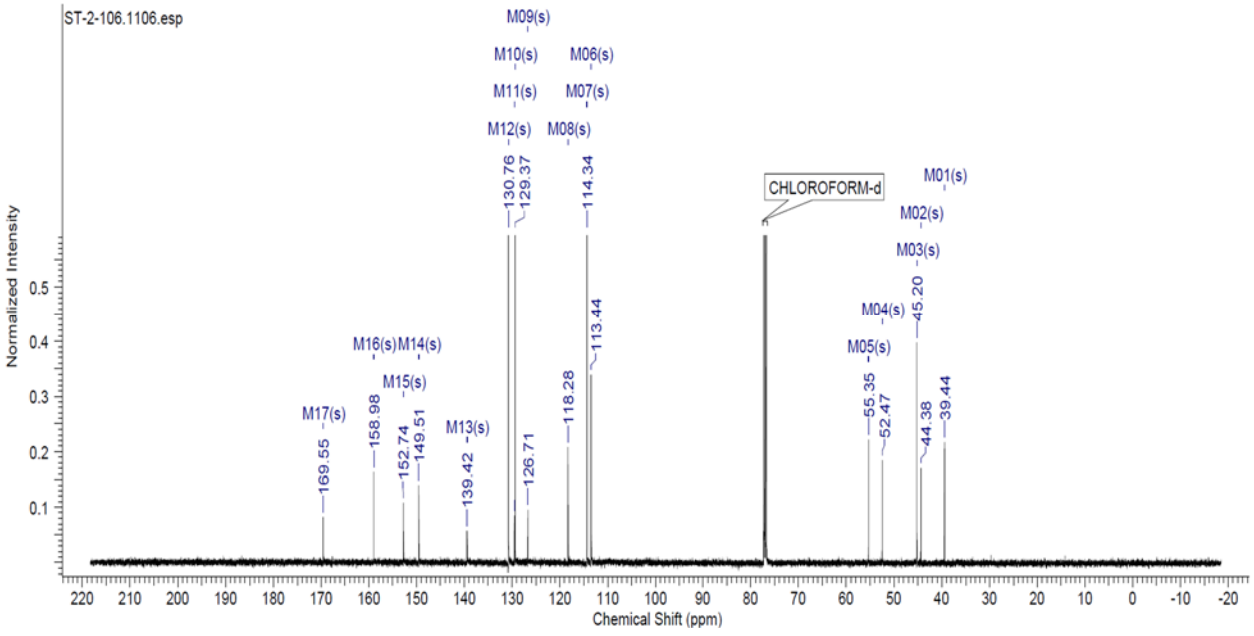
10e-3

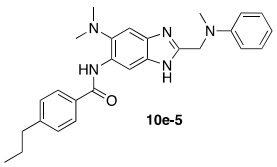

ST-2-104.104.esp
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
M01(s)
M02(s)
M03(s)
M04(m)
M05(m)
M07(br. s.)
M08(m)
M09(br. s.)
M10(d)
2.71
2.98
4.63
6.76
6.73
7.18
7.21
7.51
8.16
8.18
8.76
6.99
10.18
10.22
0.84
0.86
0.96
0.73
2.04
2.03
3.04
1.98
3.03
6.00


SB-P30G8.1000.esp
Normalized Intensity
Chemical Shift (ppm)
CHLOROFORM-d
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M06(s)
M07(s)
M09(s)
M11(s)
M12(s)
M13(s)
M14(s)
M15(s)
M16(s)
M17(s)
M18(s)
M19(s)
M20(s)
M21(s)
M22(s)
M23(s)
M24(s)
M25(s)
M26(s)
M27(s)
M28(s)
M29(s)
M30(s)
M31(s)
M32(s)
M33(s)
M34(s)
M35(s)
M36(s)
39.26
45.51
52.17
77.00
104.02
104.23
104.46
112.16
112.36
113.13
117.88
118.36
118.46
129.14
129.60
133.54
139.87
149.28
153.26
159.48
159.90
159.93
161.48
163.67
165.60
165.70

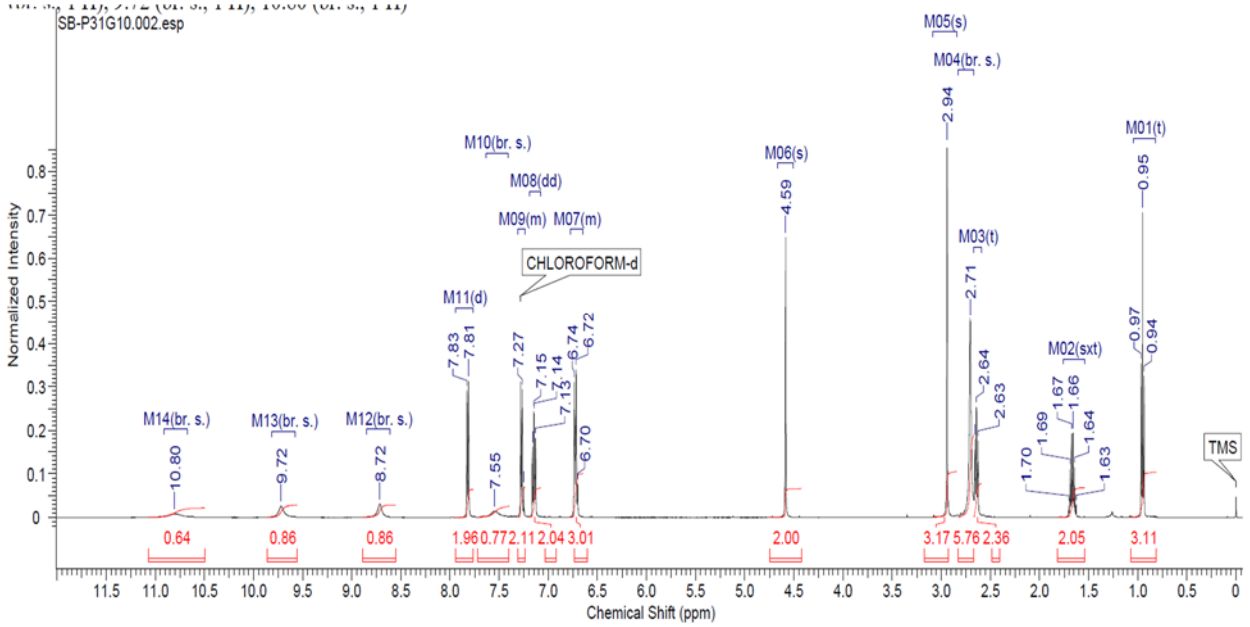

10e-4

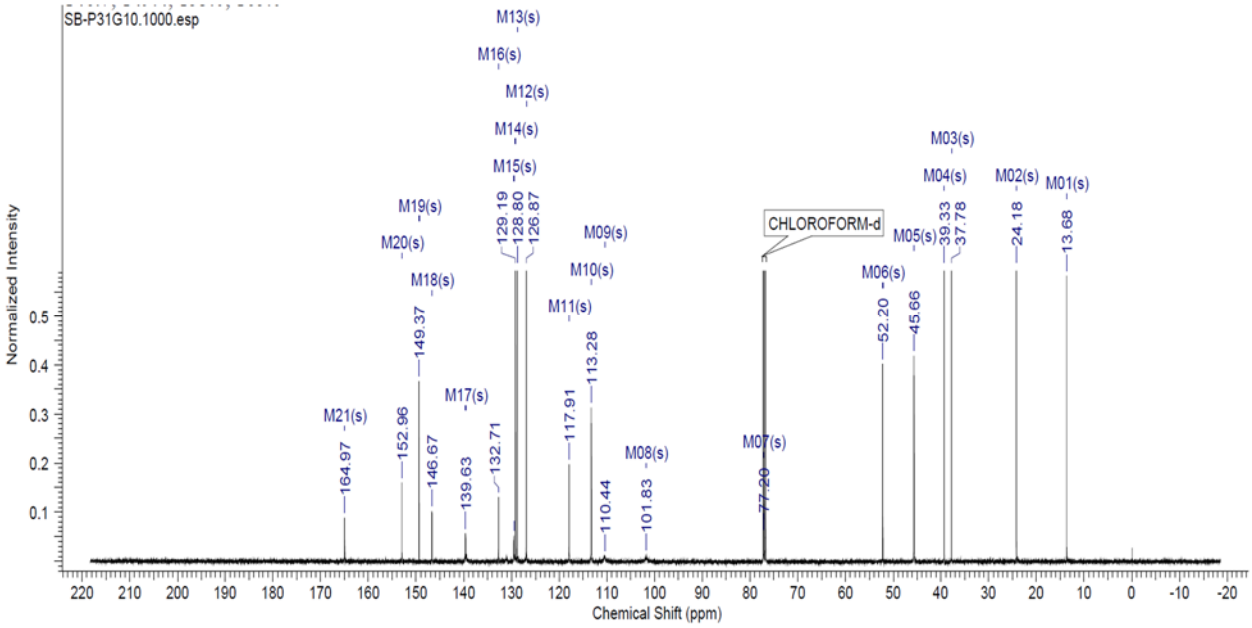

VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
M11(br. s.)
M10(br. s.)
M09(br. s.)
M08(m)
M06(m)
M07(d)
M05(s)
M04(s)
M03(s)
M02(s)
8.85
8.58
7.38
7.22
7.21
7.19
6.94
6.92
6.80
6.79
6.78
6.76
4.65
3.83
3.66
3.00
2.36
0.83 0.84 0.83 3.85 1.84 2.92 1.93 3.01 2.11 2.93 6.00

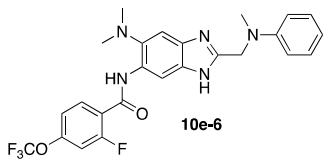

ST-2-106.1106.esp
Normalized Intensity
Chemical Shift (ppm)
M09(s)
M10(s)
M06(s)
M11(s)
M07(s)
M12(s)
M08(s)
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M16(s)
M14(s)
M15(s)
M17(s)
M13(s)
CHLOROFORM-d
130.76
129.37
114.34
113.44
118.28
126.71
169.55
158.98
152.74
149.51
139.42
45.20
55.35
52.47
44.38
39.44

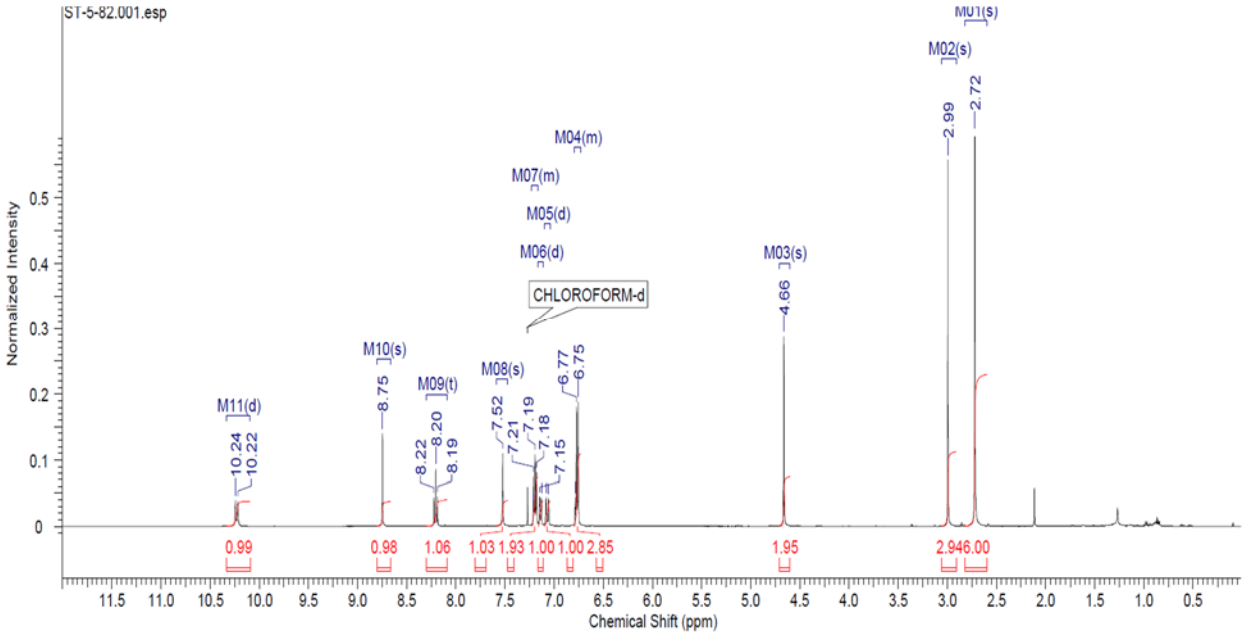


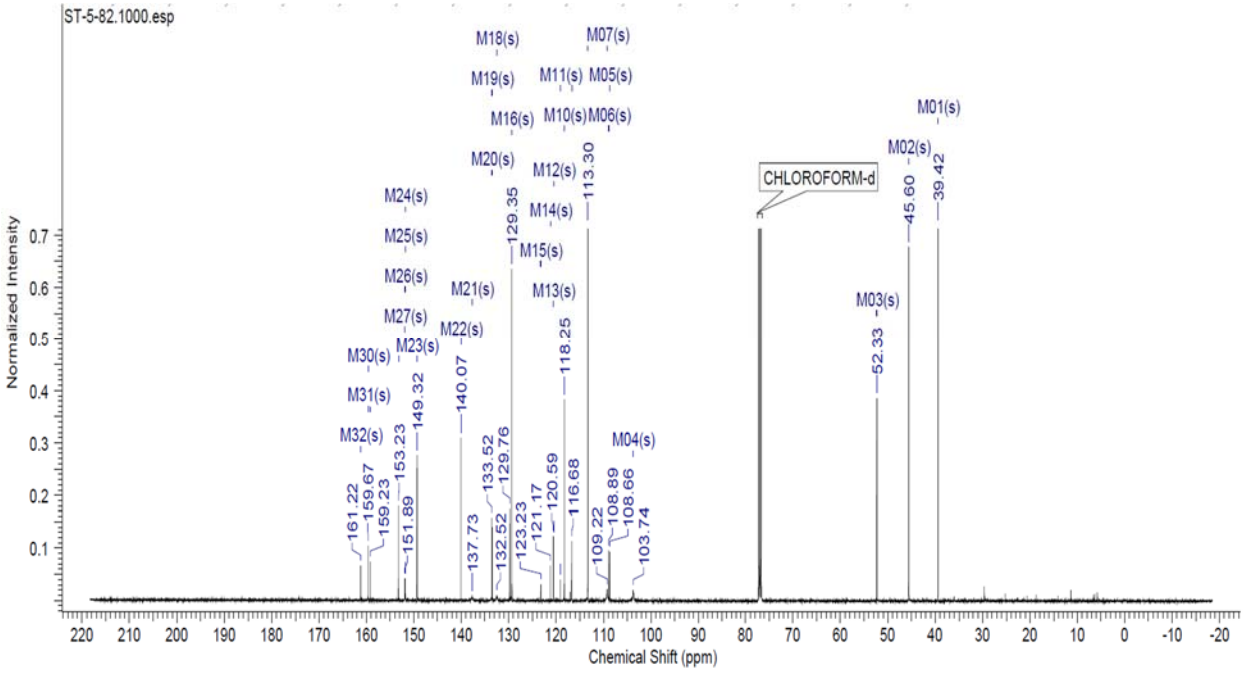
10e-5

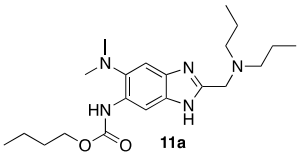

SB-P31G10.002.esp
Normalized Intensity
Chemical Shift (ppm)
M14(br. s.)
M13(br. s.)
M12(br. s.)
M11(d)
M10(br. s.)
M08(dd)
M09(m)
M07(m)
CHLOROFORM-d
M06(s)
M05(s)
M04(br. s.)
M03(t)
M02(sxt)
M01(t)
TMS
10.80
9.72
8.72
7.83
7.81
7.55
7.27
7.15
7.14
7.13
6.74
6.72
6.70
4.59
2.94
2.71
2.64
2.63
1.70
1.69
1.67
1.66
1.64
1.63
0.97
0.95
0.94
0.64
0.86
0.86
1.96
0.77
2.11
2.04
3.01
2.00
3.17
5.76
2.36
2.05
3.11


SB-P31G10.1000.esp
Normalized Intensity
Chemical Shift (ppm)
M21(s)
M20(s)
M19(s)
M18(s)
M17(s)
M16(s)
M15(s)
M14(s)
M13(s)
M12(s)
M11(s)
M10(s)
M09(s)
M08(s)
M07(s)
M06(s)
M05(s)
M04(s)
M03(s)
M02(s)
M01(s)
CHLOROFORM-d
164.97
152.96
149.37
146.67
139.63
132.71
129.19
128.80
126.87
117.91
113.28
110.44
101.83
77.20
52.20
45.66
39.33
37.78
24.18
13.68

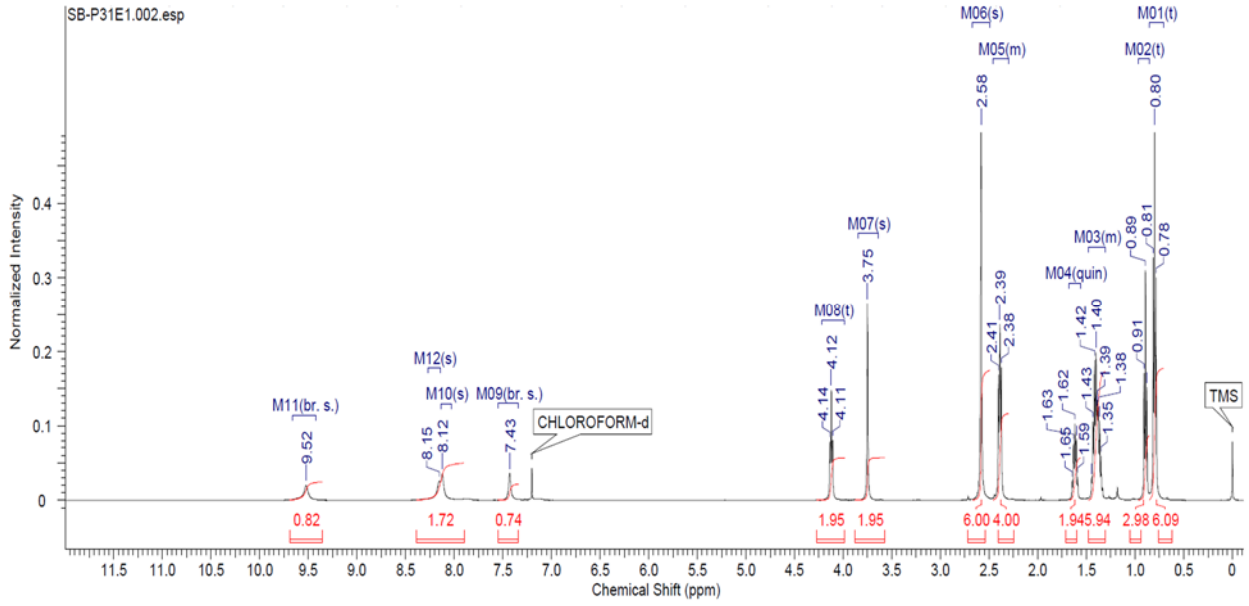

10e-6

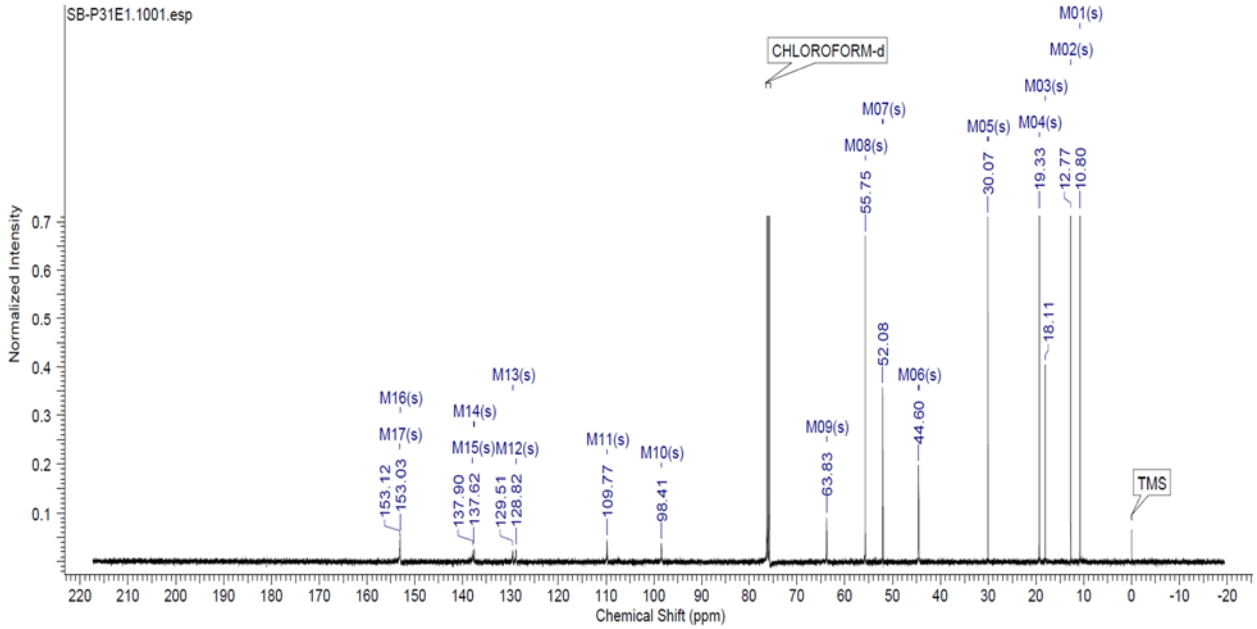

ST-5-82.001.esp
Normalized Intensity
Chemical Shift (ppm)
M01(s)
M02(s)
M03(s)
M04(m)
M05(d)
M06(d)
M07(m)
M08(s)
M09(t)
M10(s)
M11(d)
CHLOROFORM-d
2.99
2.72
4.66
8.75
8.22
8.20
8.19
7.52
7.21
7.19
7.18
7.15
6.77
6.75
10.24
10.22
0.99
0.98
1.06
1.03
1.93
1.00
1.00
2.85
1.95
2.94
6.00

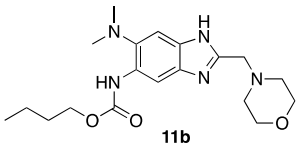

ST-5-82.1000.esp
Normalized Intensity
Chemical Shift (ppm)
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M06(s)
M07(s)
M10(s)
M11(s)
M12(s)
M13(s)
M14(s)
M15(s)
M16(s)
M18(s)
M19(s)
M20(s)
M21(s)
M22(s)
M23(s)
M24(s)
M25(s)
M26(s)
M27(s)
M30(s)
M31(s)
M32(s)
CHLOROFORM-d
113.30
129.35
118.25
140.07
149.32
153.23
159.67
159.23
161.22
151.89
137.73
133.52
129.76
132.52
123.23
121.17
120.59
116.68
109.22
108.89
108.66
103.74
52.33
45.60
39.42



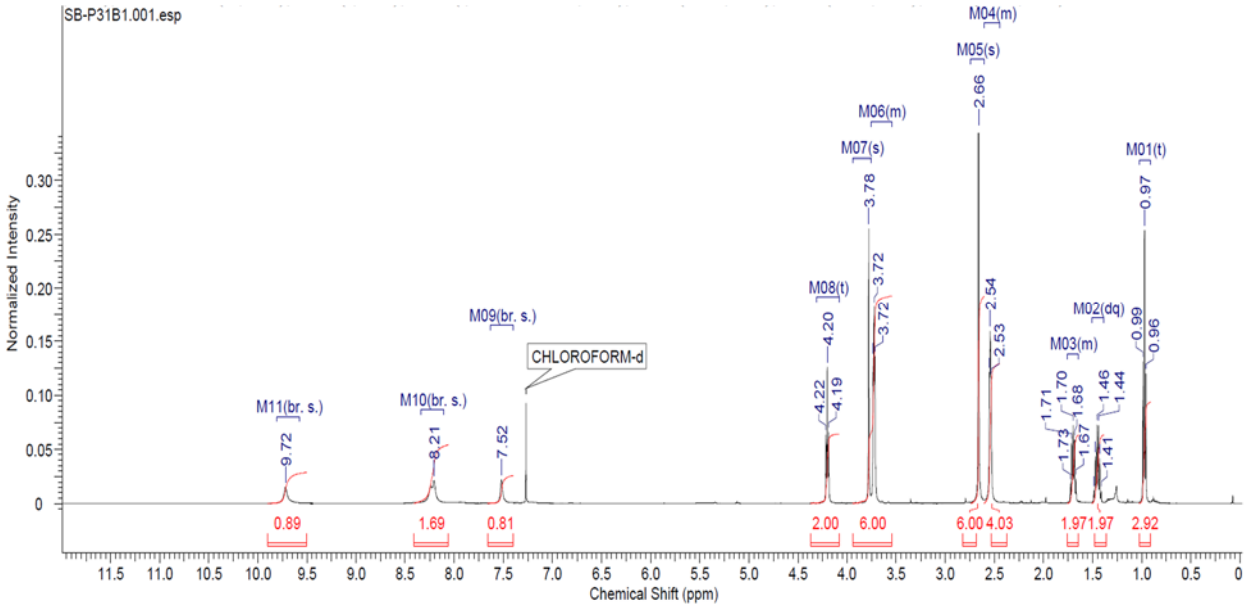
11a

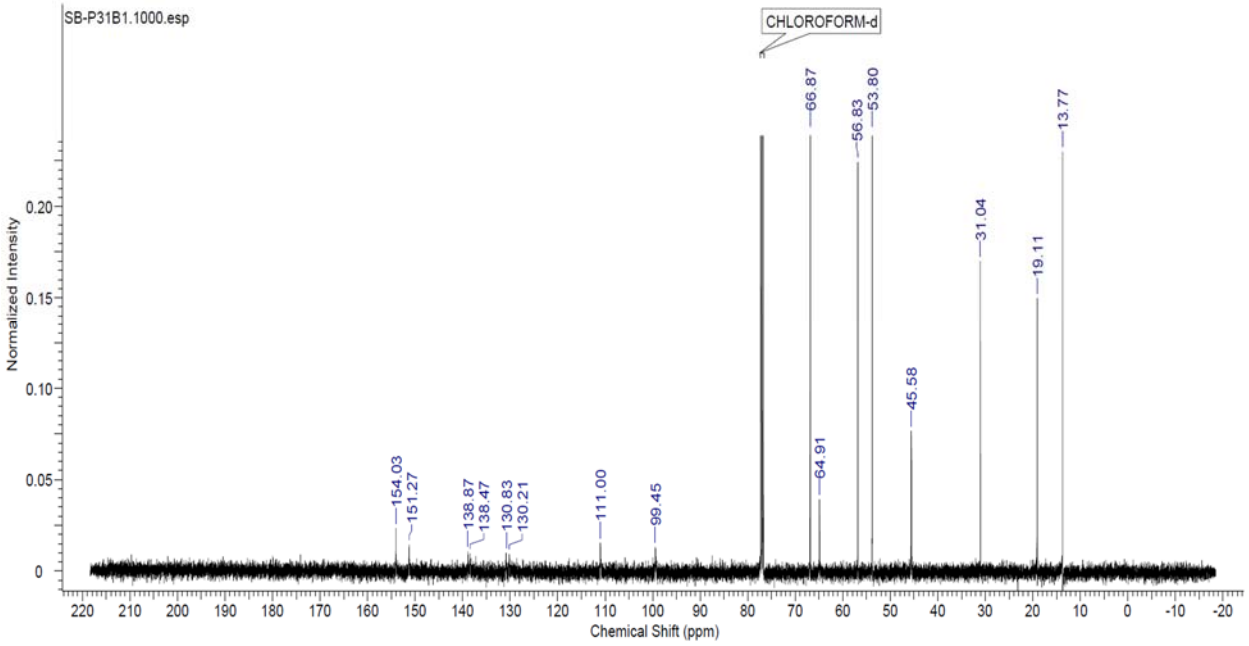

SB-P31E1.002.esp
Normalized Intensity
Chemical Shift (ppm)
M11(br. s.)
M12(s)
M10(s)
M09(br. s.)
CHLOROFORM-d
M08(t)
M07(s)
M06(s)
M05(m)
M04(quin)
M03(m)
M02(t)
M01(t)
TMS
9.52
8.15
8.12
7.43
4.14
4.12
4.11
3.75
2.58
2.41
2.39
2.38
1.63
1.62
1.65
1.59
1.43
1.42
1.40
1.39
1.38
1.35
0.91
0.89
0.81
0.80
0.78
0.82
1.72
0.74
1.95
1.95
6.00
4.00
1.94
5.94
2.98
6.09

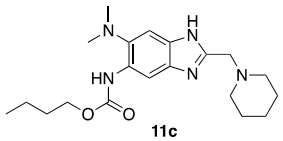

SB-P31E1.1001.esp
Normalized Intensity
Chemical Shift (ppm)
CHLOROFORM-d
M01(s)
M02(s)
M03(s)
M04(s)
M05(s)
M06(s)
M07(s)
M08(s)
M09(s)
M10(s)
M11(s)
M12(s)
M13(s)
M14(s)
M15(s)
M16(s)
M17(s)
TMS
153.12
153.03
137.90
137.62
129.51
128.82
109.77
98.41
63.83
55.75
52.08
44.60
30.07
19.33
18.11
12.77
10.80



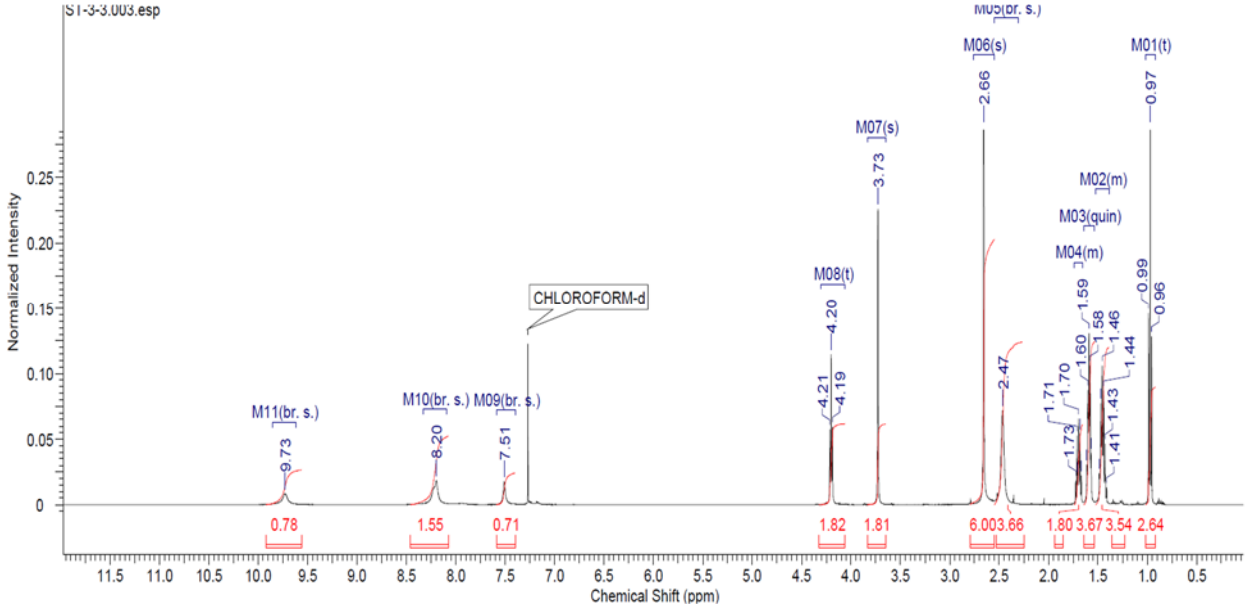
11b

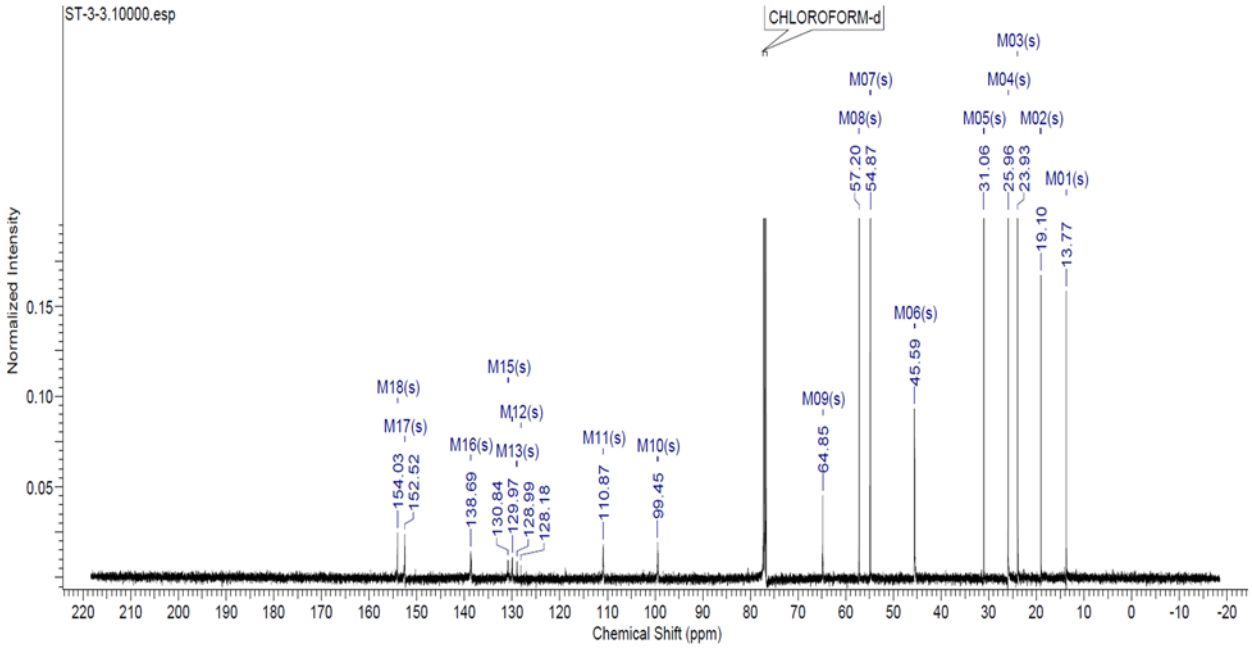

SB-P31B1.001.esp
Normalized Intensity
Chemical Shift (ppm)
M11(br. s.)
M10(br. s.)
M09(br. s.)
CHLOROFORM-d
M08(t)
M07(s)
M06(m)
M05(s)
M04(m)
M03(m)
M02(dq)
M01(t)
9.72
8.21
7.52
4.22
4.19
4.20
3.78
3.72
3.72
2.66
2.54
2.53
1.71
1.70
1.68
1.73
1.67
1.46
1.44
1.41
0.99
0.97
0.96
0.89
1.69
0.81
2.00
6.00
6.00
4.03
1.97
1.97
2.92

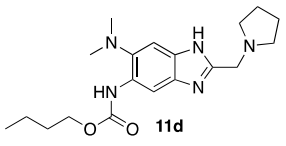

SB-P31B1.1000.esp
Normalized Intensity
Chemical Shift (ppm)
CHLOROFORM-d
154.03
151.27
138.87
138.47
130.83
130.21
111.00
99.45
66.87
64.91
56.83
53.80
45.58
31.04
19.11
13.77



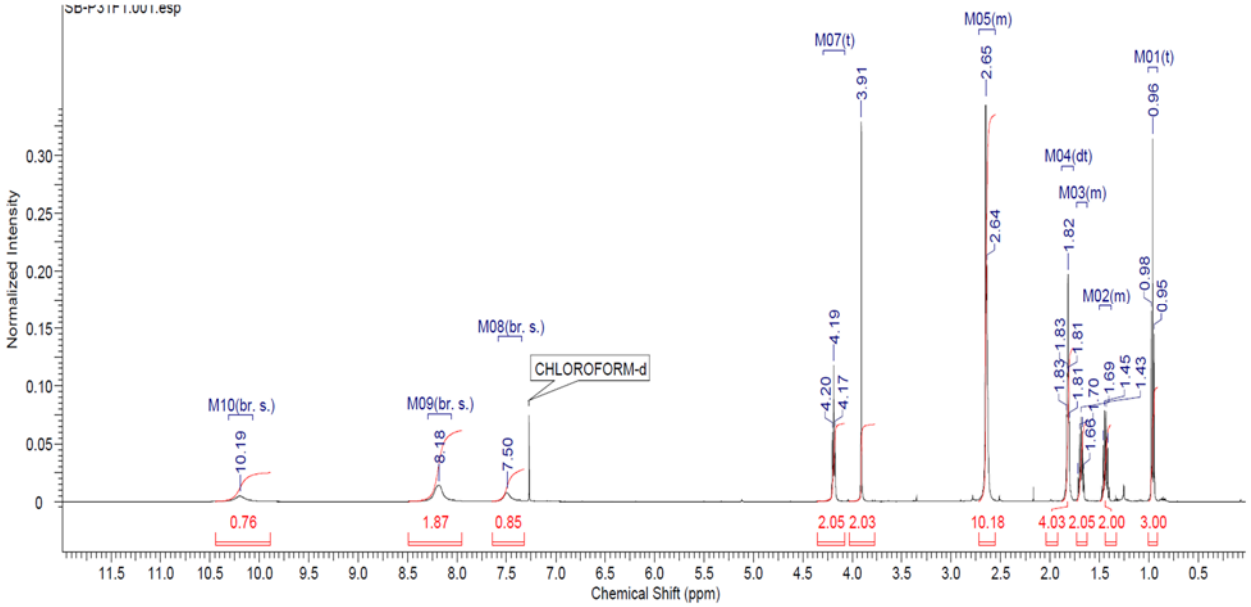
11c

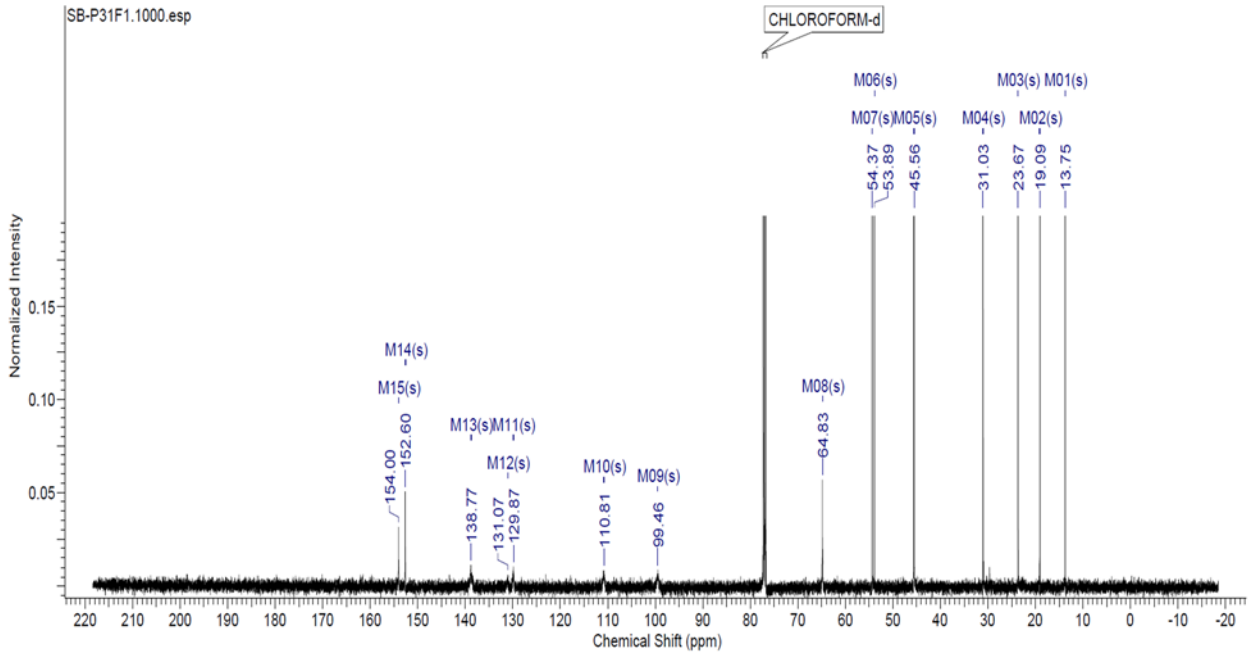

ST-3-3.003.esp
M05(br. s.)
M06(s)
M01(t)
M07(s)
M02(m)
M03(quin)
M04(m)
M08(t)
CHLOROFORM-d
M11(br. s.)
M10(br. s.)
M09(br. s.)
Normalized Intensity
Chemical Shift (ppm)

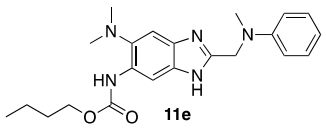

ST-3-3.10000.esp
CHLOROFORM-d
M03(s)
M07(s)
M04(s)
M08(s)
M05(s)
M02(s)
M01(s)
M06(s)
M09(s)
M18(s)
M15(s)
M17(s)
M12(s)
M16(s)
M13(s)
M11(s)
M10(s)
Normalized Intensity
Chemical Shift (ppm)



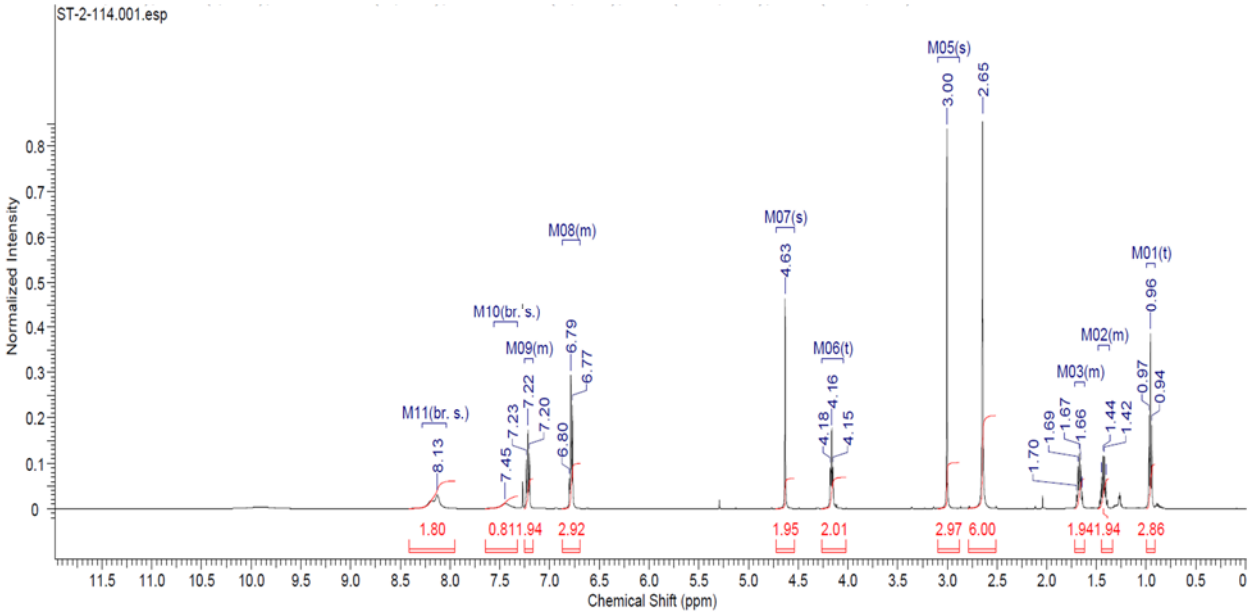
11d

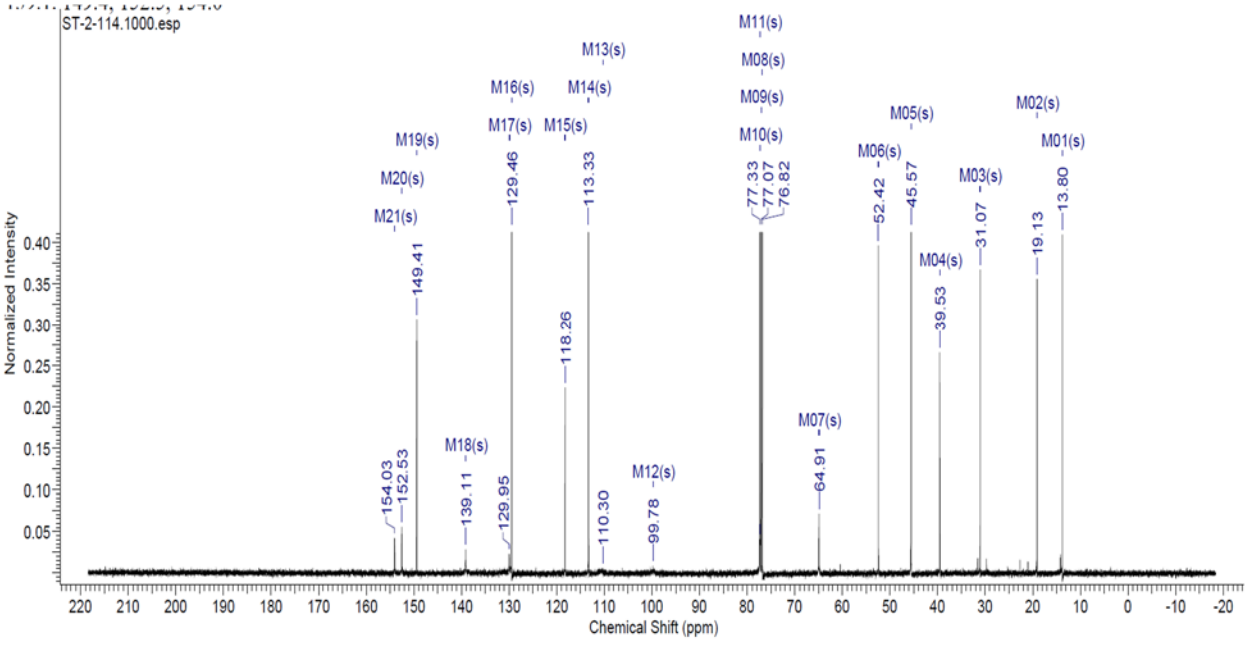

M10(br. s.)
M09(br. s.)
M08(br. s.)
CHLOROFORM-d
M07(t)
M05(m)
M04(dt)
M03(m)
M02(m)
M01(t)
Normalized Intensity
Chemical Shift (ppm)

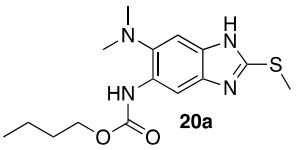

SB-P31F1.1000.esp
CHLOROFORM-d
M06(s)
M07(s)
M05(s)
M04(s)
M03(s)
M01(s)
M02(s)
M14(s)
M15(s)
M13(s)
M11(s)
M12(s)
M10(s)
M09(s)
M08(s)
Normalized Intensity
Chemical Shift (ppm)



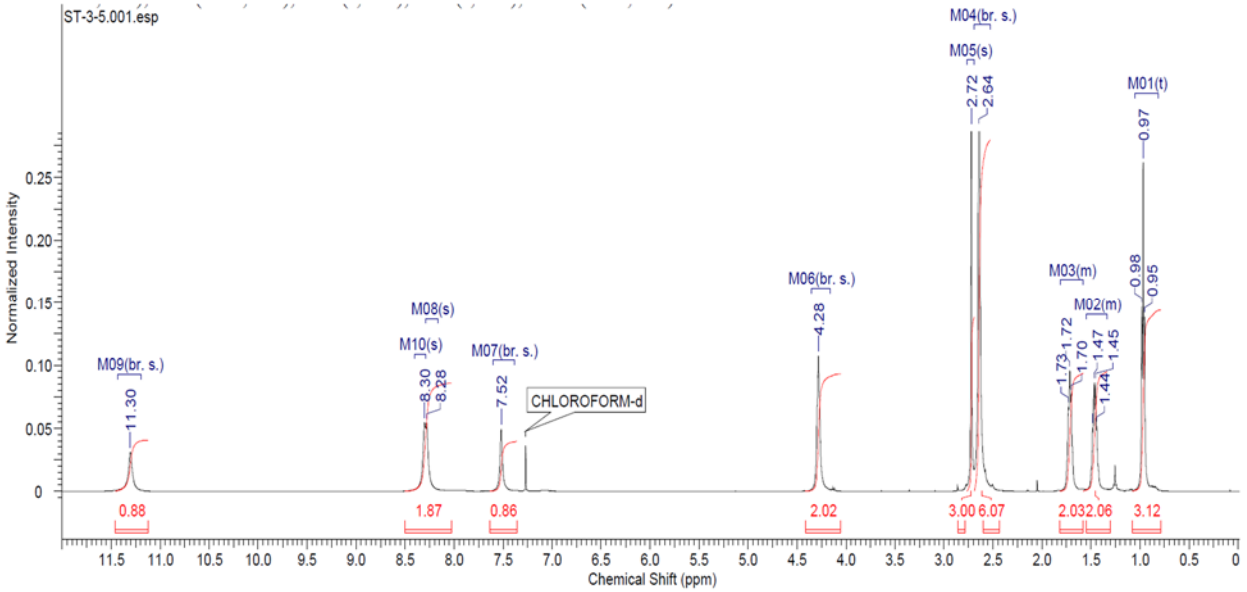
11e

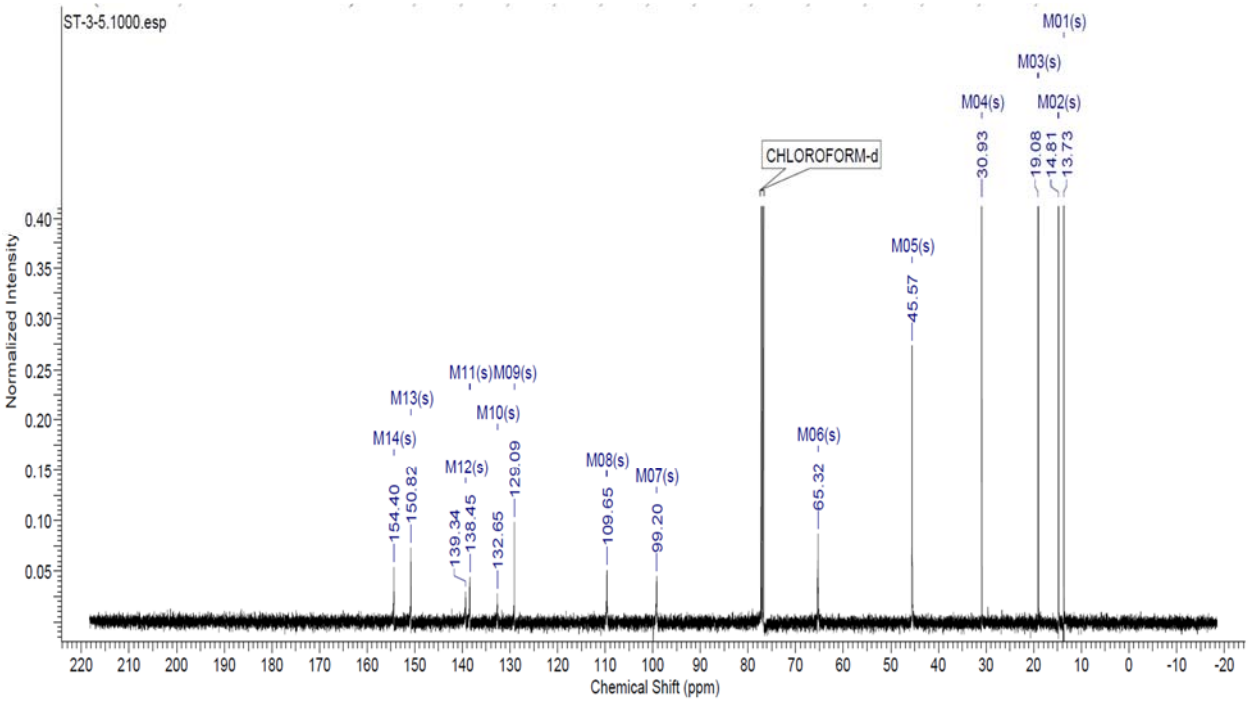

ST-2-114.001.esp
Normalized Intensity
Chemical Shift (ppm)
M05(s)
3.00
2.65
M08(m)
M07(s)
4.63
M01(t)
0.96
M10(br.'s.)
M09(m)
6.79
6.77
M06(t)
4.16
4.15
M02(m)
M03(m)
0.97
0.94
M11(br. s.)
8.13
7.45
7.23
7.22
7.20
6.80
4.18
1.70
1.69
1.67
1.66
1.44
1.42
1.80
0.81
1.94
2.92
1.95
2.01
2.97
6.00
1.94
1.94
2.86

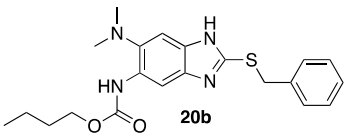

ST-2-114.1000.esp
Normalized Intensity
Chemical Shift (ppm)
M11(s)
M08(s)
M09(s)
M10(s)
M13(s)
M16(s)
M14(s)
M17(s)
M15(s)
M19(s)
M20(s)
M21(s)
129.46
113.33
149.41
118.26
77.33
77.07
76.82
M05(s)
M02(s)
M06(s)
M01(s)
52.42
45.57
M03(s)
M04(s)
31.07
19.13
13.80
39.53
M07(s)
64.91
M18(s)
M12(s)
154.03
152.53
139.11
129.95
110.30
99.78



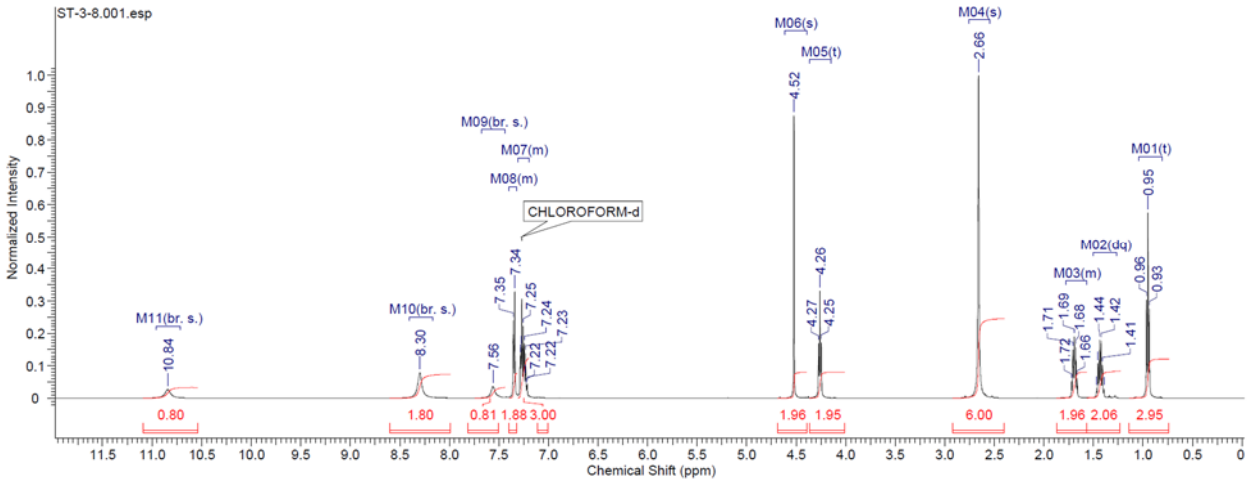
20a

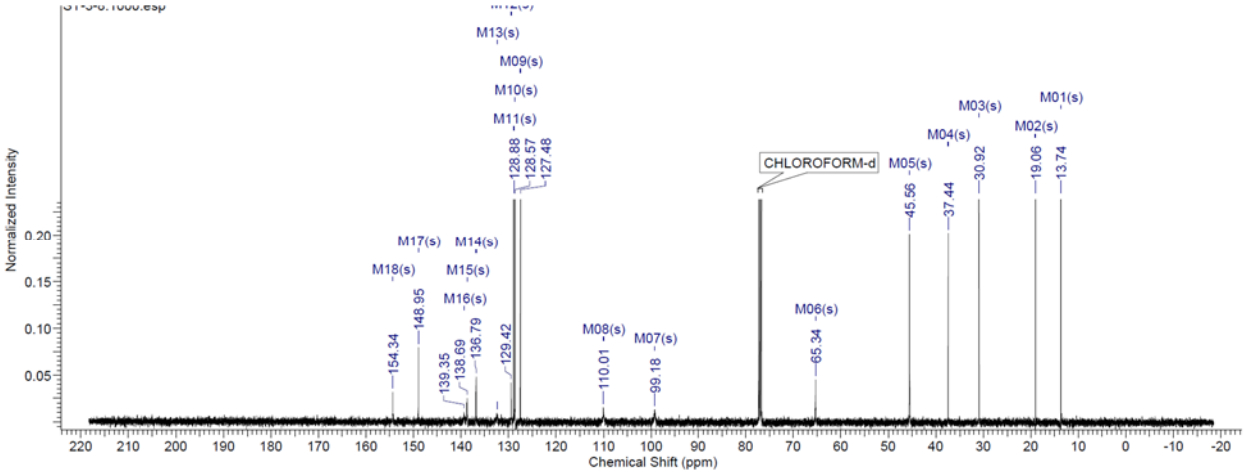

ST-3-5.001.esp
M04(br. s.)
M05(s)
M01(t)
M06(br. s.)
M03(m)
M02(m)
M08(s)
M10(s)
M09(br. s.)
M07(br. s.)
CHLOROFORM-d
11.30
8.30
8.28
7.52
4.28
2.72
2.64
1.73
1.72
1.70
1.47
1.45
1.44
0.98
0.97
0.95
0.88
1.87
0.86
2.02
3.00
6.07
2.03
2.06
3.12
Normalized Intensity
Chemical Shift (ppm)

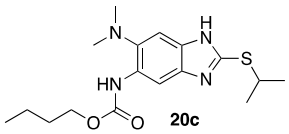

ST-3-5.1000.esp
M01(s)
M03(s)
M04(s)
M02(s)
CHLOROFORM-d
M05(s)
M11(s)
M09(s)
M13(s)
M10(s)
M14(s)
M12(s)
M08(s)
M07(s)
M06(s)
30.93
19.08
14.81
13.73
45.57
154.40
150.82
139.34
138.45
132.65
129.09
109.65
99.20
65.32
Normalized Intensity
Chemical Shift (ppm)


N
H
N
S
HN
O
O

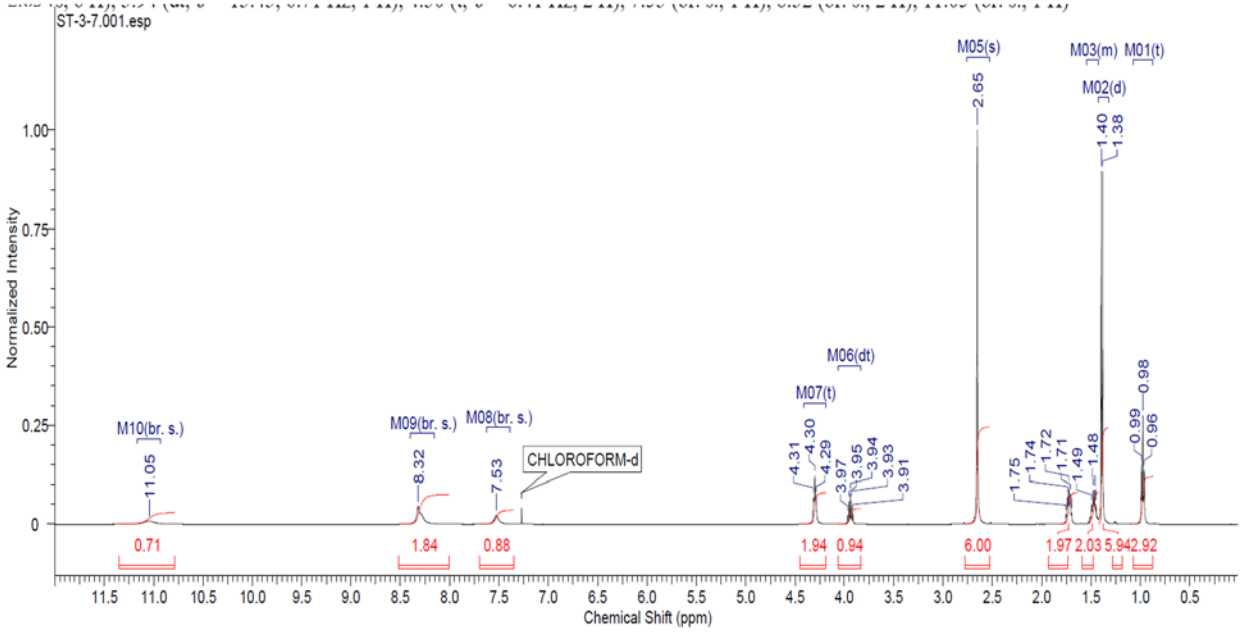
20b

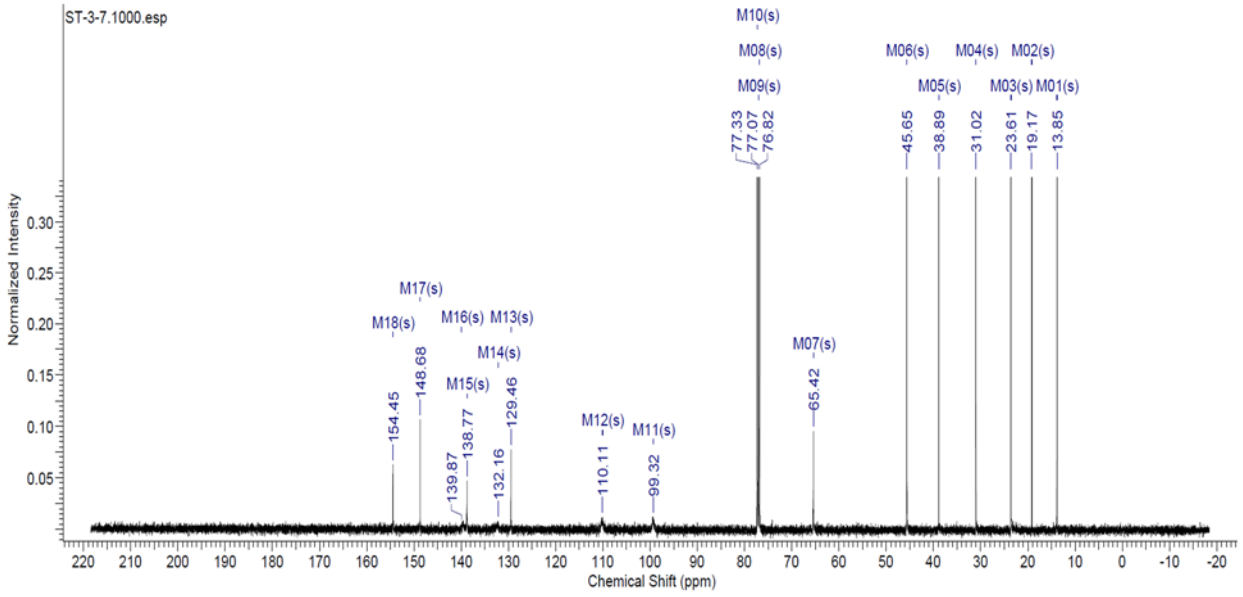

ST-3-8.001.esp
Normalized Intensity
Chemical Shift (ppm)
M11(br. s.)
10.84
M10(br. s.)
8.30
M09(br. s.)
M07(m)
M08(m)
CHLOROFORM-d
7.56
7.35
7.34
7.25
7.24
7.23
7.22
7.22
M06(s)
M05(t)
4.52
4.27
4.26
4.25
M04(s)
2.66
M01(t)
0.95
M02(dq)
M03(m)
0.96
0.93
1.71
1.69
1.68
1.66
1.72
1.44
1.42
1.41
0.80
1.80
0.81
1.88
3.00
1.96
1.95
6.00
1.96
2.06
2.95

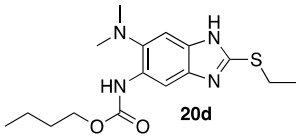

Normalized Intensity
Chemical Shift (ppm)
M13(s)
M09(s)
M10(s)
M11(s)
128.88
128.57
127.48
CHLOROFORM-d
M05(s)
M04(s)
M03(s)
M02(s)
M01(s)
45.56
37.44
30.92
19.06
13.74
M17(s)
M14(s)
M18(s)
M15(s)
M16(s)
148.95
154.34
139.35
138.69
136.79
129.42
M08(s)
M07(s)
110.01
99.18
M06(s)
65.34



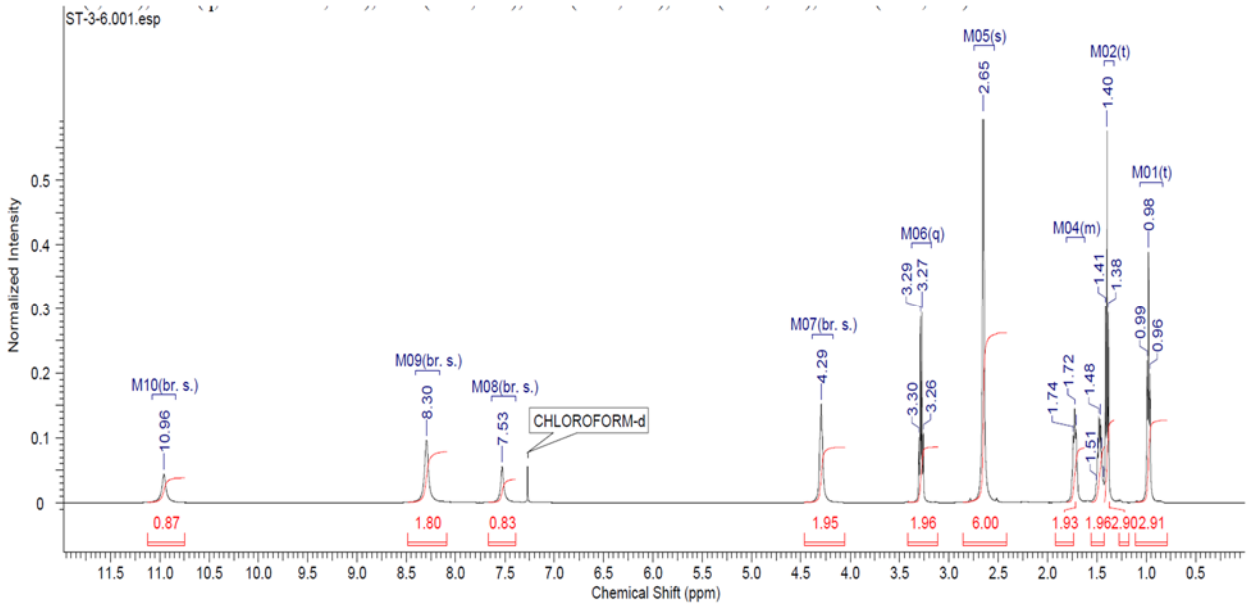
20c

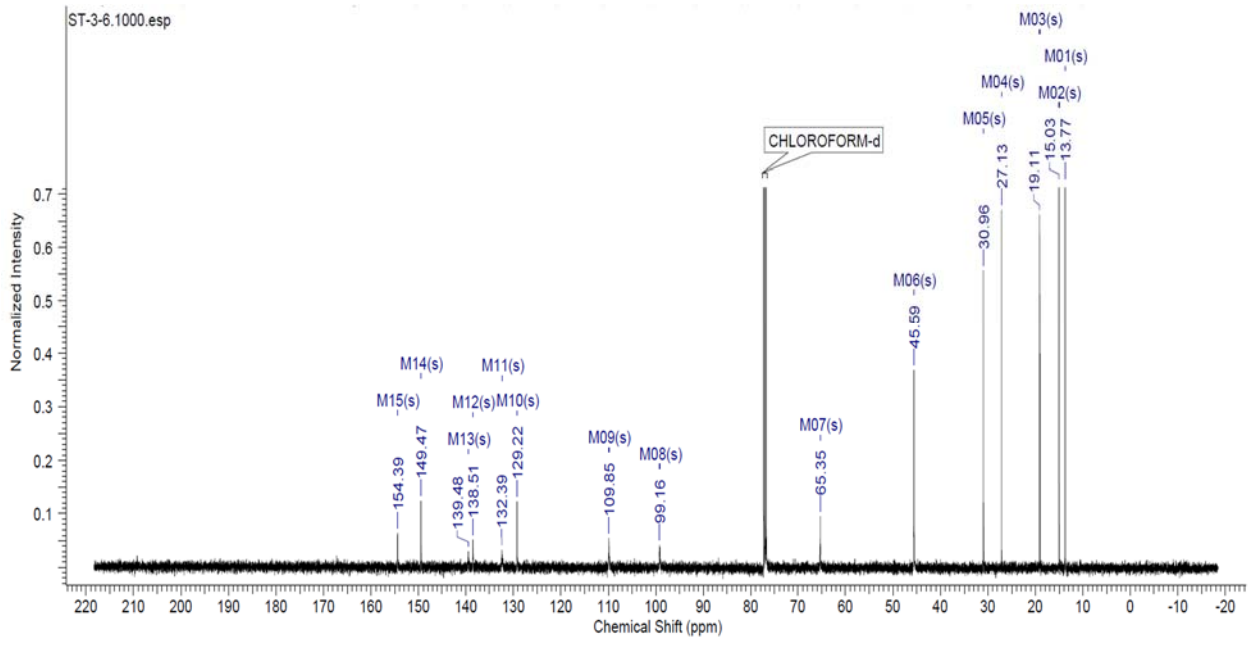

ST-3-7.001.esp
Normalized Intensity
Chemical Shift (ppm)
M10(br. s.)
11.05
M09(br. s.)
8.32
M08(br. s.)
7.53
CHLOROFORM-d
M07(t)
M06(dt)
4.31
4.30
4.29
3.97
3.95
3.94
3.93
3.91
M05(s)
2.65
M03(m)
M01(t)
M02(d)
1.40
1.38
1.75
1.74
1.72
1.71
1.49
1.48
0.99
0.98
0.96
0.71
1.84
0.88
1.94
0.94
6.00
1.97
2.03
5.94
2.92

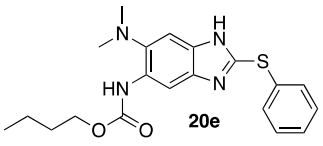

ST-3-7.1000.esp
Normalized Intensity
Chemical Shift (ppm)
M10(s)
M08(s)
M09(s)
77.33
77.07
76.82
M06(s)
M04(s)
M02(s)
M05(s)
M03(s)
M01(s)
45.65
38.89
31.02
23.61
19.17
13.85
M07(s)
65.42
M17(s)
M18(s)
M16(s)
M13(s)
M14(s)
M15(s)
154.45
148.68
139.87
138.77
132.16
129.46
M12(s)
M11(s)
110.11
99.32



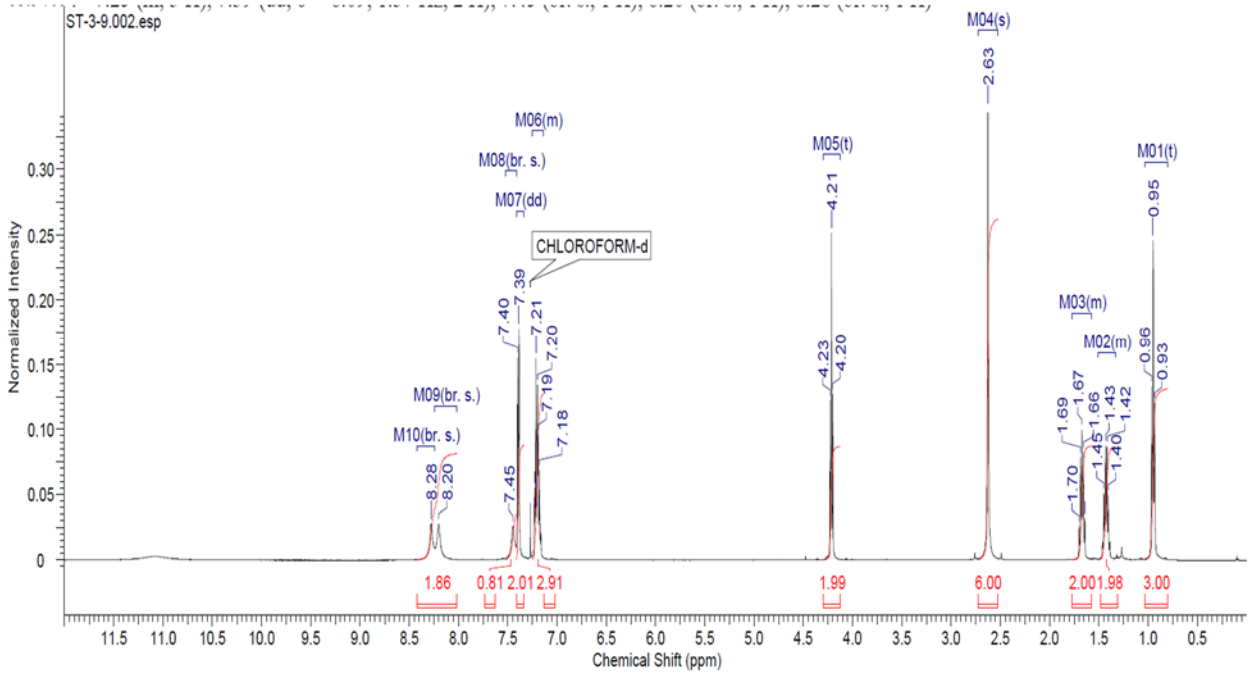
20d

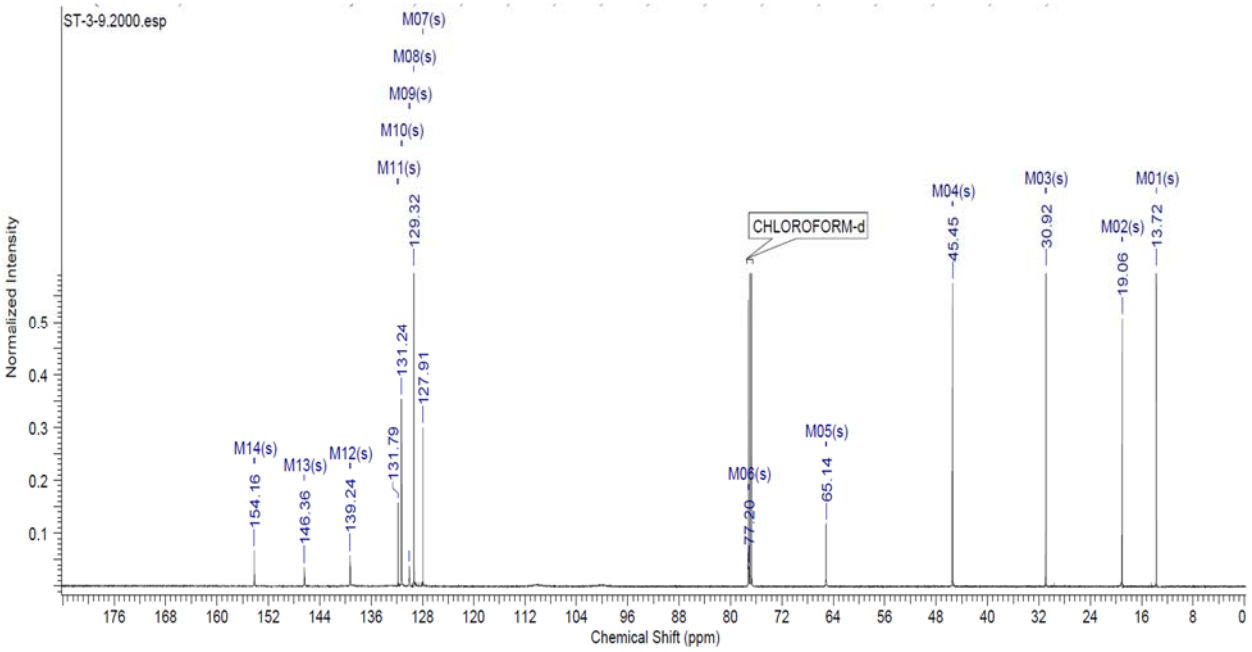

ST-3-6.001.esp
M10(br, s.)
10.96
M09(br, s.)
8.30
M08(br, s.)
7.53
CHLOROFORM-d
M07(br, s.)
4.29
M06(q)
3.29
3.27
3.30
3.26
M05(s)
2.65
M04(m)
1.74
1.72
1.48
1.51
M02(t)
1.40
1.41
1.38
M01(t)
0.98
0.99
0.96
0.87
1.80
0.83
1.95
1.96
6.00
1.93
1.96
2.90
2.91
Normalized Intensity
Chemical Shift (ppm)

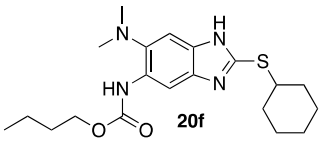

ST-3-6.1000.esp
M03(s)
M01(s)
M04(s)
M02(s)
M05(s)
CHLOROFORM-d
27.13
19.11
15.03
13.77
30.96
M06(s)
45.59
M14(s)
M15(s)
M12(s)
M11(s)
M10(s)
M13(s)
M09(s)
M08(s)
M07(s)
154.39
149.47
139.48
138.51
132.39
129.22
109.85
99.16
65.35
Normalized Intensity
Chemical Shift (ppm)



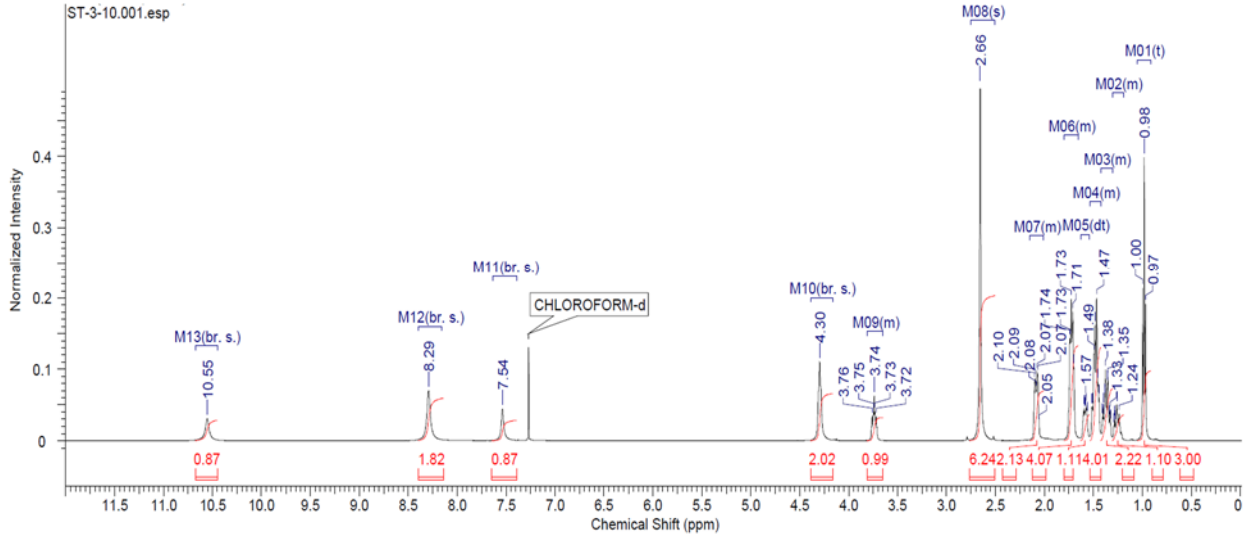
20e

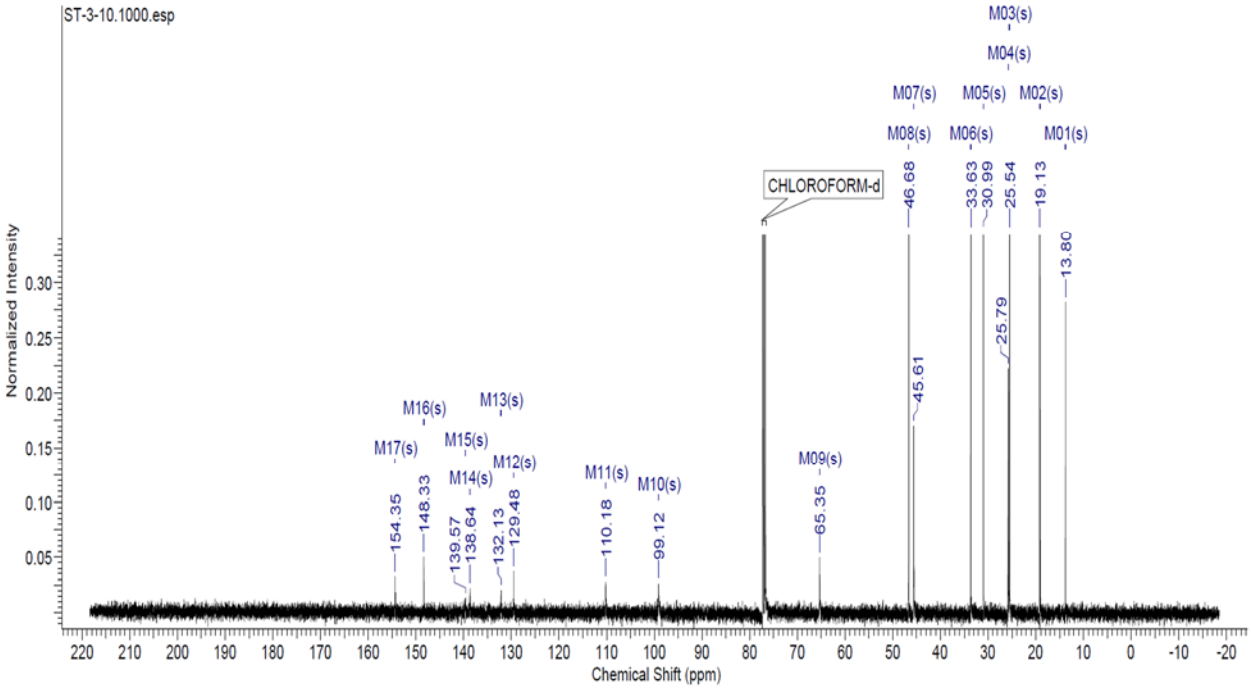

ST-3-9.002.esp
M04(s)
2.63
M06(m)
M08(br. s.)
M07(dd)
M05(t)
4.21
M01(t)
0.95
CHLOROFORM-d
7.40
7.39
7.21
7.20
7.19
7.18
4.23
4.20
M03(m)
M02(m)
0.96
0.93
M09(br. s.)
M10(br. s.)
8.28
8.20
7.45
1.69
1.67
1.66
1.43
1.42
1.40
1.70
1.86
0.81
2.01
2.91
1.99
6.00
2.00
1.98
3.00
Normalized Intensity
Chemical Shift (ppm)

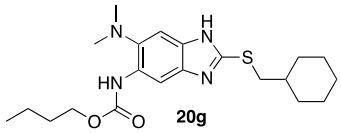

ST-3-9.2000.esp
M07(s)
M08(s)
M09(s)
M10(s)
M11(s)
129.32
131.24
127.91
131.79
M14(s)
154.16
M13(s)
146.36
M12(s)
139.24
CHLOROFORM-d
M06(s)
77.20
M05(s)
65.14
M04(s)
45.45
M03(s)
30.92
M02(s)
19.06
M01(s)
13.72
Normalized Intensity
Chemical Shift (ppm)



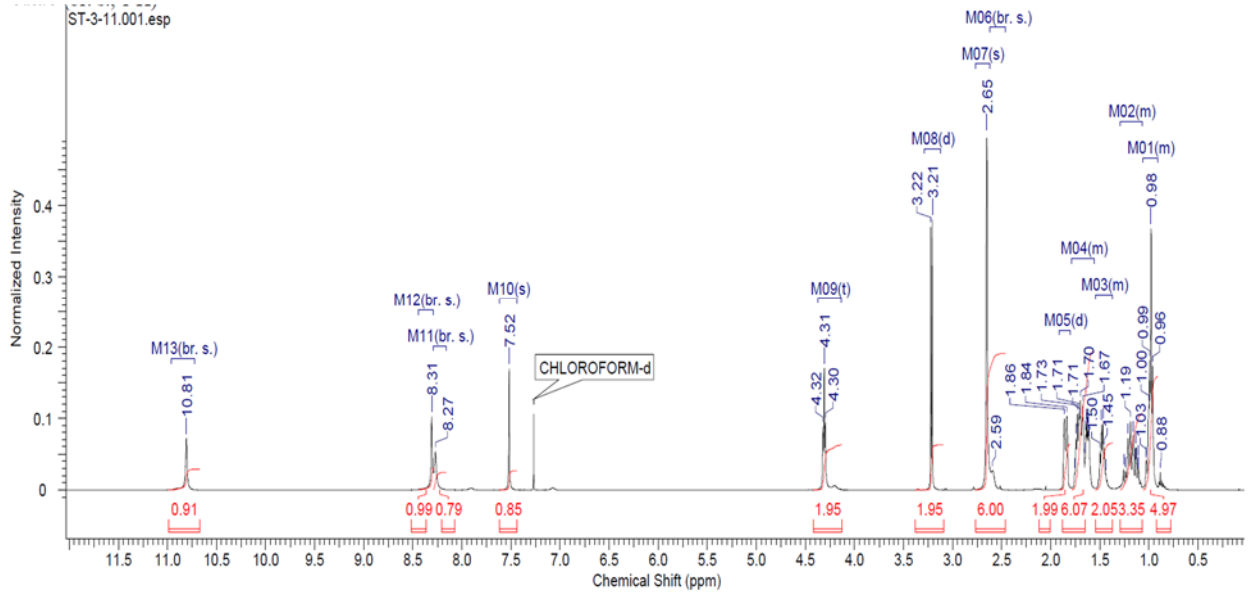
20f

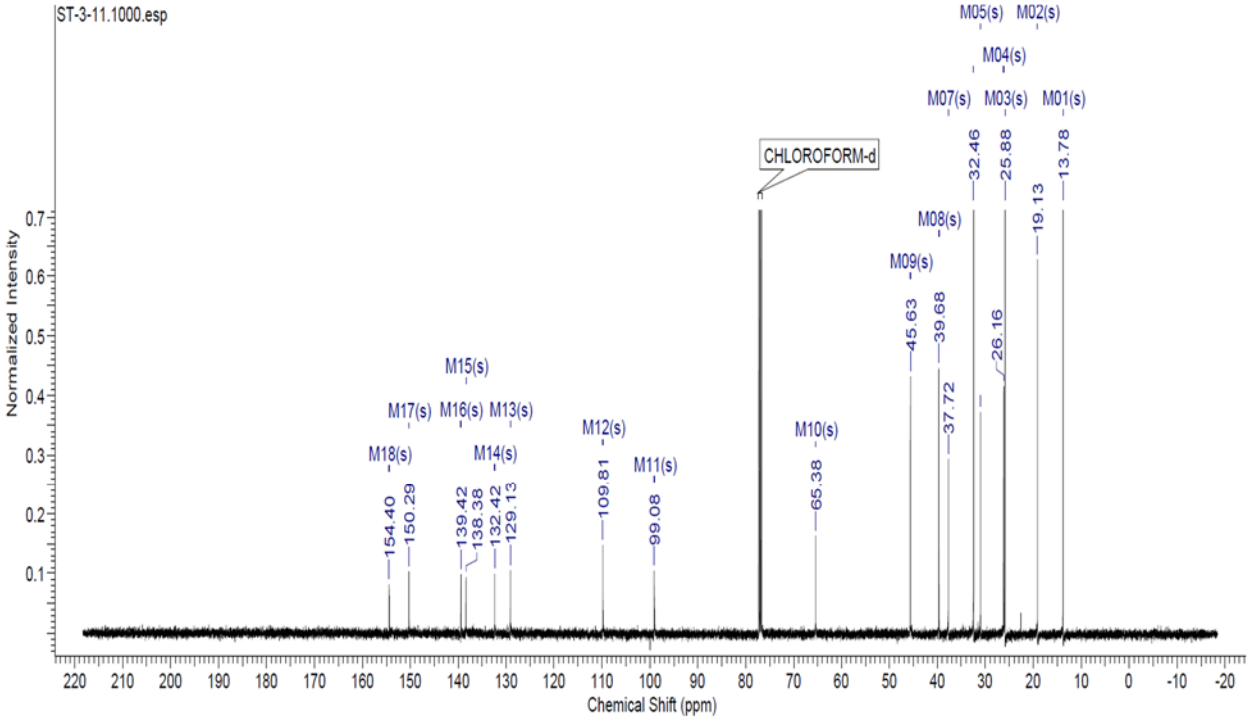

ST-3-10.001.esp
Normalized Intensity
Chemical Shift (ppm)
M13(br. s.)
M12(br. s.)
M11(br. s.)
CHLOROFORM-d
M10(br. s.)
M09(m)
M08(s)
M01(t)
M02(m)
M06(m)
M03(m)
M04(m)
M07(m)
M05(dt)
0.87
1.82
0.87
2.02
0.99
6.24
2.13
4.07
1.11
4.01
2.22
1.10
3.00


ST-3-10.1000.esp
Normalized Intensity
Chemical Shift (ppm)
CHLOROFORM-d
M03(s)
M04(s)
M07(s)
M05(s)
M02(s)
M08(s)
M06(s)
M01(s)
M16(s)
M13(s)
M17(s)
M15(s)
M12(s)
M14(s)
M11(s)
M10(s)
M09(s)
154.35
148.33
139.57
138.64
132.13
129.48
110.18
99.12
65.35
46.68
45.61
33.63
30.99
25.54
25.79
19.13
13.80



20g


ST-3-11.001.esp
Normalized Intensity
Chemical Shift (ppm)
M13(br. s.)
10.81
M12(br. s.)
M11(br. s.)
8.31
8.27
M10(s)
7.52
CHLOROFORM-d
M09(t)
4.32
4.31
4.30
M08(d)
3.22
3.21
M06(br. s.)
M07(s)
2.65
2.59
M02(m)
M01(m)
0.98
M04(m)
M03(m)
M05(d)
1.86
1.84
1.73
1.71
1.71
1.70
1.67
1.50
1.45
1.19
1.03
1.00
0.99
0.96
0.88
0.91
0.99
0.79
0.85
1.95
1.95
6.00
1.99
6.07
2.05
3.35
4.97


ST-3-11.1000.esp
Normalized Intensity
Chemical Shift (ppm)
M05(s)
M02(s)
M04(s)
M07(s)
M03(s)
M01(s)
32.46
25.88
13.78
CHLOROFORM-d
M08(s)
19.13
M09(s)
45.63
39.68
26.16
37.72
M15(s)
M17(s)
M16(s)
M13(s)
M18(s)
M14(s)
M12(s)
M10(s)
M11(s)
154.40
150.29
139.42
138.38
132.42
129.13
109.81
99.08
65.38