Supplementary Material

Synthesis of aryl benzamides containing a pyrimidine moiety as possible HIV-1 NNRTIs

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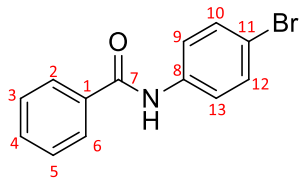
General method for the reaction of benzoic acid (4) with aniline (5) or aminopyridine (7) derivatives

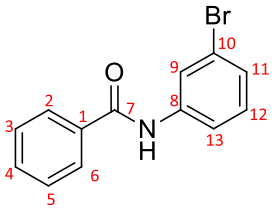
Benzoic acid **4** (1.00 g, 8.20 mmol) was placed in a round-bottomed flask, followed by addition of excess thionyl chloride (2 mL). The mixture was stirred and refluxed at 80 °C in an oil bath for 24 h. After cooling the mixture to room-temperature, excess thionyl chloride was evaporated leaving a pale-yellow benzoyl chloride residue which was dissolved in distilled acetonitrile (4 mL), followed by slow addition of an ice-cold mixture of the appropriate aniline (**5**) or aminopyridine (**7**) and pyridine or triethylamine (3 mL) in dry acetonitrile (10 mL). The mixture was stirred at 0 °C for 30 min, warmed to room temperature and stirred for 8-24 h. Excess solvent was removed *in vacuo* and the residue dissolved in ethyl acetate (50 mL). The organic mixture was washed with aqueous saturated $NaHCO_3$ or aqueous K_2CO_3 (2 × 50 mL), saturated brine solution (50 mL) and the organic layer was separated and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product purified by normal silica gel chromatography eluting with 20-30% EtOAc/hexane. Recrystallization was performed using hot ethyl acetate and cold hexane where necessary to afford compounds **6** and **8**.

N-(4-Bromophenyl)benzamide (6a)[1]



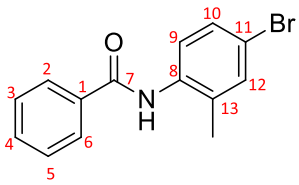
4-Bromoaniline **5a** (1. 02 g, 8.20 mmol) was reacted as per the general method for 18 h in the presence of pyridine. A precipitate formed that was recovered by filtration to give compound **6a** as a white solid (1.17 g, 83% yield). Rf= 0.4 (7:3 EtOAc/hexane); M.p. = 209-211 °C (lit.[1] 204 °C); IR (ν/cm^{-1}) 3331 (NH, br), 1545 (C=O, str); ^{1}H NMR (300 MHz, DMSO-d_6) δ_H 10.38 (s, 1H, NH), 7.98 – 7.93 (m, 2H, H2 and H6), 7.81 – 7.75 (m, 2H, H9 and H13), 7.64 – 7.51 (m, 5H, H3, H4, H5, H10, and H12); ^{13}C NMR (75 MHz, DMSO-d_6) δ_C 165.6 (C7), 138.5 (C8), 134.7 (C1), 131.7 (C3 and C5), 131.4 (C4), 128.4 (C2 and C6) , 127.6 (C10 and 12), 122.2 (C9 and C13), 115.3 (C11).

N-(3-Bromophenyl)benzamide (6b)[2]



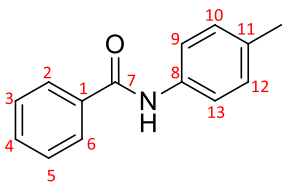
3-Bromoaniline **5b** (0.900 mL, 8.20 mmol) was reacted using the general method for 24 h in the presence of triethylamine. Compound **6b** was obtained as a white solid product (2.10 g, 93% yield). Rf= 0.4 (7:3 EtOAc/hexane); M.p. = 124-126 °C; IR (ν/cm^{-1}) 3282 (NH, br), 1649 (C=O, str); ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 8.00 (s, 1H, NH), 7.90 (t, J = 1.9 Hz, 1H, H9), 7.82 (m, 2H, H2 and H6), 7.58 – 7.51 (m, 2H, H4 and H11), 7.49 – 7.42 (m, 2H, H3 and H5), 7.29 – 7.16 (m, 2H, H12 and H13); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 165.9 (C7), 139.2 (C8), 134.5 (C1), 132.1 (C4), 130.3 (C12), 128.8 (C3 and C5), 127.5 (C13), 127.1 (C3 and C5), 123.2 (C9), 122.7 (C10), 118.7 (C11).

N-(4-Bromo-2-methylphenyl)benzamide (6c)[3]



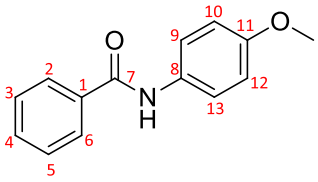
4-Bromo-2-methylaniline **5c** (1. 350 g, 8.20 mmol) was reacted using the general method for 24 h in the presence of pyridine. Compound **6c** was obtained as a white solid product (1.22 g, 89% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 174-176 °C (lit.[3] 177-178 °C); IR (ν/cm^{-1}) 3257 (NH, br), 1587 (C=O, str), ^{1}H NMR (300 MHz, DMSO) δ_H 10.00 (s, 1H, NH), 8.03 – 7.96 (m, 2H, H2 and H6), 7.64 – 7.49 (m, 4H, H3, H4, H5, and H12), 7.41 (dd, J = 2.2, 8.5 Hz, 1H, H10), 7.32 (d, J = 8.5 Hz, 1H, H9), 2.24 (s, 3H, H-CH_3); ^{13}C NMR (75 MHz, DMSO) δ_C 165.3 (C7), 136.5 (C-8), 135.9 (C13), 134.2 (C1), 132.7 (C12), 131.6 (C4), 128.7 (C9), 128.4 (C3 and C5), 127.7 (C2 and C6), 118.2 (C11), 17.6 (C-CH_3).

N-(p-Tolyl)benzamide (6d)[3]



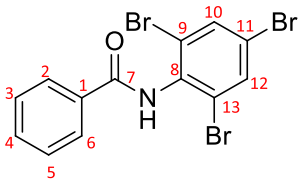
4-Methylaniline **5d** (0.880 g, 8.20 mmol) was reacted using the general method in the presence of triethylamine for 14 h. Compound **6d** was obtained as a white solid (1.31 g, 72% yield). Rf= 0.4 (7:3 EtOAc/hexane); M.p. = 162-164 °C (lit.[3] 160-161 °C); IR (ν/cm^{-1}) 3310 (NH, br), 1645 (C=O, str); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ_H 10.19 (s, 1H, NH), 7.98 – 7.94 (m, 2H, H2 and H6), 7.68 (d, J = 8.4 Hz, 2H, H9 and H13), 7.61 – 7.50 (m, 3H, H3, H4, and H5), 7.16 (d, J = 8.3 Hz, 2H, H10 and H12), 2.29 (s, 3H, H-CH$_3$); ^{13}C NMR (101 MHz, DMSO-d$_6$) δ_C 165.8 (C7), 137.1 (C8), 135.5 (C1), 133.1 (C11), 131.9 (C4), 129.4 (C10 and C12), 128.8 (C3 and C5), 128.1 (C2 and C6), 120.8 (C9 and C13), 21.0 (C-CH$_3$).

N-(4-Methoxyphenyl)benzamide (6e)[1]



4-Methoxyaniline **5e** (0.990 g, 8.20 mmol) was reacted according to the general method for 22 h in the presence of triethylamine. Compound **6e** was obtained as a white solid (1.36 g, 73% yield). Rf= 0.4 (7:3 EtOAc/hexane); M.p. = 162-164 °C (lit.[1] 140-160 °C); IR (ν/cm^{-1}) 3330 (NH, br), 1646 (C=O, str), 1174 (C-O); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ_H 10.16 (s, 1H, NH), 7.98 – 7.93 (m, 2H, H2 and H6), 7.72 – 7.67 (d, J = 12.1 Hz, 2H, H9 and H13), 7.61 – 7.50 (m, 3H, H3, H4, and H5), 6.91– 6.94 (d, J = 9.0 Hz, 2H, H10 and H12), 3.75 (s, 3H, H-OCH$_3$); ^{13}C NMR (101 MHz, DMSO-d$_6$) δ_C 165.6 (C7), 156.0 (C11), 135.5 (C1), 132.7 (C8), 131.8 (C4), 128.8 (C3 and C5), 128.0 (C2 and C6), 122.4 (C9 and C13), 114.2 (C10 and C12), 56.6 (C-OCH$_3$).

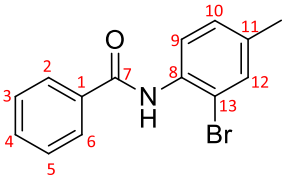
N-(2-Bromo-4-methylphenyl)benzamide (6f)



2,4,6-Tribromoaniline **5f** (2.72 g, 8.20 mmol) was reacted according to the general method for 10 h in the presence of triethylamine. Compound **6f** was obtained as a white solid (1.04 g, 29% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 207-209 °C; IR (ν/cm^{-1}) 3227 (NH, br), 1652 (C=O, str) ; ^{1}H NMR (400 MHz, DMSO-d$_6$) δ_H 10.38 (s, 1H, NH), 8.09 – 7.98 (m, 4H, H2, H6, H10,

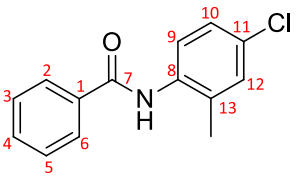
and H12), 7.54-7.62 (dt, *J* = 30.4, 7.3 Hz, 3H, H3, H4, and H5); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 165.5 (C7), 136.3 (C8), 134.8 (C10 and C12), 133.8 (C1), 132.6 (C4), 129.0 (C3 and C5), 128.2 (C2 and C6), 126.1 (C9 and C13), 121.7 (C11).

N-(2-Bromo-4-methylphenyl)benzamide (6g)[4]



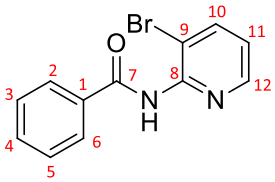
2-Bromo-4-methylaniline **5g** (1.52 g, 8.20 mmol) was reacted according to the general method for 24 h in the presence of triethylamine. Compound **6g** was obtained as a white solid (1.88 g, 74% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 150-152 °C (lit.[4] 148-150 °C); IR (*v*/cm^{-1}) 3247 (NH, br), 1647 (C=O); ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 8.37 (m, 2H, H9 and NH), 7.93 (dt, *J*= 6.8, 1,4 Hz, 2H, H2 and H6), 7.44-7.52 (m, 4H, H3, H4, H5, H12), 7.17 (m, 1H, H10), 2.32 (s, 3H, H-CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 162.5 (C7), 135.4 (C10), 134.7 (C1), 133.2 (C8), 132.5 (C12), 132.1 (C4), 129.1 (C10), 128.9 (C3 and C5), 127.1 (C1 and C6), 121.6 (C9), 113.7 (C13), 20.6 (C-CH_3).

N-(4-Chloro-2-methylphenyl)benzamide (6h)[5]



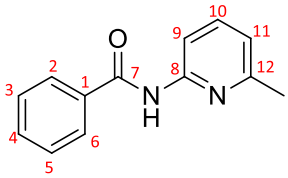
4-Chloro-2-methyloaniline **5h** (1.16 g, 8.2 mmol) was reacted according to the general method for 16 h in the presence of triethylamine. Compound **6h** was obtained as a white solid (1.26 g, 63% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 170-173 °C; IR (*v*/cm^{-1}) 3249 (NH, br), 1648 (C=O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 9.96 (s, 1H, NH), 7.99 (d, *J* = 7.2 Hz, 2H, H2 and H6), 7.57 (dt, *J* = 7.2, 27.4 Hz, H4, H5, and H6), 7.40 (d, *J* = 8.4 Hz, 2H, H10 and H12), 7.29 (dd, *J* = 2.4, 8.4 Hz, 1H, H9), 3.41 (s, 3H, H-CH_3); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 165.9 (C7), 136.6 (C8), 135.9 (C11), 134.7 (C1), 132.1 (C4), 130.3 (C12), 128.9 (C3 and C5), 128.6 (C2 and C6), 128.1 (C10), 126.3 (C9), 18.2 (C-CH_3).

N-(3-Bromopyridin-2-yl)benzamide (8b) [6]



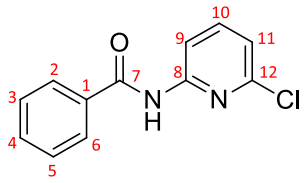
2-Amino-3-bromopyridine **7b** (1.40 g, 8.20 mmol) was reacted as per the general method for 24 h in the presence of pyridine. Compound **8b** was obtained as a dark brown solid product (0.50 g, 22% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 117-119 °C (lit.[6] 89-90 °C); IR (ν/cm^{-1}) 3284 (NH, str), 1641 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 9.03 (s, 1H, NH), 8.18 (d, J = 3.9 Hz, 1H, H12), 7.76 (dd, J = 7.7, 36.8 Hz, 3H, H2, H6, and H10), 7.38 (dt, J = 7.4, 36.7 Hz, 3H, H3, H4, and H5), 6.83 (dd, J = 4.7, 7.9 Hz, 1H, H9); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.4 (C7), 148.9 (C8), 147.3 (C12), 141.6 (C10), 133.9 (C1), 132.2 (C4), 128.7 (C3 and C5), 127.6 (C2 and C6), 122.0 (C11), 114.2 (C9).

N-(6-Methylpyridin-2-yl)benzamide (8c) [7]



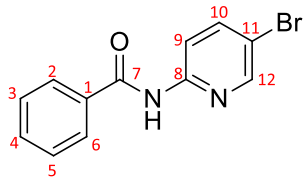
2-Amino-6-methylpyridine **7c** (0.890 g, 8.20 mmol) was reacted for 9 h in the presence of triethylamine. Compound **8c** was obtained as a white solid (1.27 g, 73% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 97-99 °C (lit.[7] 86-88 °C); IR (ν/cm^{-1}) 3321 (NH, br), 1670 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 9.13 (s, 1H, NH), 8.22 (d, J = 8.3 Hz, 1H, H9), 7.91 (d, J = 7.5 Hz, 2H, H2 and H6), 7.62 (t, J = 7.9 Hz, 1H, H10), 7.51 (t, J = 7.3 Hz, 1H, H4), 7.43 (t, J = 7.4 Hz, 2H, H3 and H5), 2.35 (s, 3H, H-CH3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.9 (C7), 156.8 (C8), 151.0 (C12), 138.8 (C10), 134.4 (C1), 132.1 (C4), 128.7 (C3 and C5), 127.3 (C2 and C6), 119.4 (C11), 111.1 (C9), 23.8 (C-CH_3); HRMS (ES+) calculated for $C_{13}H_{12}N_2O$ $[M+H]^+$: 213.1023, found: 213.1036.

N-(6-Chloropyridin-2-yl)benzamide (8d) [8]



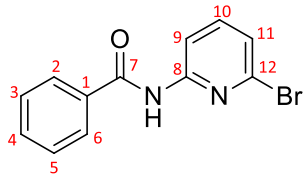
2-Amino-6-chloropyridine **7d** (1.05 g, 8.20 mmol) was reacted for 16 h in the presence of triethylamine. Compound **8d** was obtained as a white solid product (0.960 g, 50% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 99-101 °C (lit.[8] 46-48 °C); IR (ν/cm^{-1}) 3408 (NH, br) 1674 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.73 (s, 1H NH), 8.31 (d, *J* = 8.2 Hz, 1H, H9), 7.92 – 7.87 (m, 2H, H2 and H6), 7.68 (t, *J* = 8.0 Hz, 1H, H10), 7.54 (t, *J* = 7.4 Hz, 1H, H4), 7.46 (t, *J* = 7.5 Hz, 2H, H3 and H5), 7.06 (d, *J* = 7.7 Hz, 1H, H11); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.7 (C7), 151.4 (C8), 148.9 (C12), 141.0 (C10), 133.7 (C1), 132.5 (C4), 128.9 (C3 and C5), 127.3 (C2 and C6), 119.8 (C11), 112.2 (C3).

N-(5-Bromopyridin-2-yl)benzamide (8e) [7]



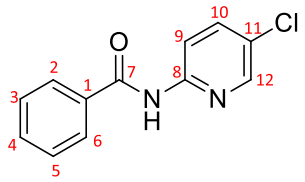
2-Amino-5-bromopyridine **7e** (1.38 g, 8.20 mmol g) was reacted with benzoyl chloride (8.20 mmol) for 22 h in the presence of pyridine. Compound **8e** was obtained as a white solid (1.28 g, 56% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 123-128 °C (lit.[7] 119-120 °C); IR (ν/cm^{-1}) 3230 (NH, br), 1676 (C=O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 11.00 (s, 1H, NH), 8.52 (d, *J* = 2.0 Hz, 1H, H12), 8.20 (d, *J* = 8.9 Hz, 1H, H9), 8.07 (dd, *J* = 8.9, 2.5 Hz, 1H, H10), 8.05 – 8.00 (m, 2H, H2 and H6), 7.63 – 7.57 (m, 1H, H4), 7.52 (t, *J* = 7.5 Hz, 2H, H3 and H5); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 166.6 (C7), 151.7 (C8), 148.9 (C12), 141.0 (C10), 134.3 (C1), 132.5 (C4), 128.8 (C3 and C5), 128.5 (C2 and C6), 116.8 (C9), 114.4 (C11).

N-(6-Bromopyridin-2-yl)benzamide (8f) [9]



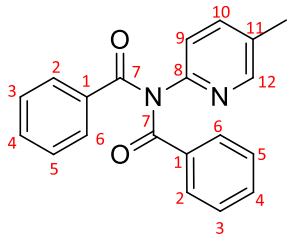
2-Amino-6-bromopyridine **7f** (1.42 g, 8.20 mmol) was reacted for 8 h in the presence of pyridine and the product was recrystallized from ethyl acetate and hexane after column chromatographic purification. Compound **8f** was obtained as a white solid (1.73 g, 76% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 98-100 °C (lit.[9] 86-87 °C); IR (ν/cm^{-1}) 3416 (NH, br), 1673 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.60 (s, 1H, NH), 8.38 (d, J = 8.2 Hz, 1H, H9), 7.92 (d, J = 7.4 Hz, 2H, H2 and H6), 7.66 – 7.57 (m, 2H, H4 and H10), 7.52 (t, J = 7.5 Hz, 2H, H3 and H5), 7.27 (d, J = 7.7 Hz, 1H, H11); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.5 (C7), 151.6 (C8), 140.7 (C10), 139.2 (C12), 133.7 (C1), 132.6 (C4), 128.9 (C3 and C5), 127.2 (C2 and C6), 123.7 (C11), 112.5 (C9).

N-(5-Chloropyridin-2-yl)benzamide (8g)[8]



2-Amino-5-chloropyridine **7g** (1.01 g, 8.2 mmol) was reacted for 22 h in the presence of pyridine. Compound **8g** was obtained as a white solid product (1.25 g, 65% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 128-130 °C (lit.[8] 121-124 °C); IR (ν/cm-1) 3231 (NH, br), 1675 (C=O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 11.01 (s, 1H, NH), 8.46 – 8.44 (m, 1H, H12), 8.27 – 8.23 (m, 1H, H9), 8.05 – 8.01 (m, 2H, H2 and H6), 7.97 (dd, J = 8.9, 2.7 Hz, 1H, H10), 7.63 – 7.58 (m, 1H, H4), 7.52 (t, J = 7.5 Hz, 2H, H3 and H5); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 166.6 (C7), 151.3 (C8), 146.7 (C12), 138.3 (C10), 134.3 (C1), 132.5 (C4), 128.8 (C3 and C5), 128.5 (C2 and C6), 126.0 (C11), 116.3 (C9).

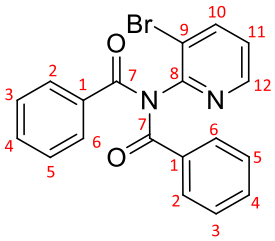
N-Benzoyl-N-(5-methylpyridin-2-yl)benzamide (9a)



2-Amino-5-methylpyridine **7a** (0.890 g, 8.20 mmol) was reacted as per the general method in the presence of triethylamine for 10 h. Compound **9a** was obtained as a white solid (1.27 g,

98% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 167-172 °C; IR (ν/cm^{-1}) 1689 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.22 (d, *J* = 2.1 Hz, 1H, H12), 7.80 – 7.75 (m, 4H, H2 and H6), 7.51 (dd, *J* = 8.1, 2.0 Hz, 1H, H10), 7.45 (t, *J* = 7.4 Hz, 2H, H4), 7.34 (t, *J* = 7.7 Hz, 4H, H3 and H5), 7.15 (d, *J* = 8.1 Hz, 1H, H9), 2.29 (s, 3H, H-CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 173.1 (C7), 151.3 (C8), 149.6 (C12), 138.9 (C10), 134.7 (C1), 132.4 (C4), 132.2 (C11), 129.3 (C3 and C5), 128.5 (C2 and C6), 121.6 (C9), 18.0 (C-*C*H$_3$); HRMS (ES+) calculated for $C_{20}H_{17}N_2O_2$ $[M+H]^+$: 317.1285, found: 317.1275.

N-Benzoyl-N-(3-bromopyridin-2-yl)benzamide (9b)



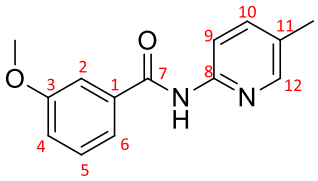
2-Amino-3-bromopyridine **7b** (1.40 g, 8.20 mmol) was reacted as per the general method in the presence of triethylamine for 24 h. Compound **9b** was obtained as a dark brown solid (0.0344 g, 22% yield). Rf= 0.3 (7:3 EtOAc/hexane); M.p. = 197-199 °C; IR (ν/cm^{-1}) 1680 (C=O, str); ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 8.31 (dd, *J* = 4.7, 1.5 Hz, 1H, H12), 7.98 (dd, *J* = 8.0, 1.5 Hz, 1H, H10), 7.85 – 7.79 (m, 4H, H2 and H6), 7.50 – 7.43 (m, 2H, H4), 7.40 – 7.32 (m, 4H, H3 and H5), 7.13 – 7.08 (m, 1H, H11); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 172.5 (C7), 152.3 (C8), 148.1 (C12), 142.4 (C10), 134.5 (C1), 132.6 (C4), 129.1 (C3 and C5), 128.6 (C2 and C6), 124.2 (C11), 119.0 (C9); HRMS (ES+) calculated for $C_{19}H_{14}BrN_2O_2$ $[M+H]^+$: 381.0233, found: 381.0233.

General method for the reaction of 3-methoxybenzoic acid (10) with aminopyridines (7)

3-Methoxybenzoic acid **10** (1.00 g, 6.60 mmol) was placed in a round-bottomed flask, followed by addition of excess thionyl chloride (2 mL). The mixture was stirred and refluxed at 80 °C in an oil bath for 24 h. After cooling the mixture to room temperature, excess thionyl chloride was evaporated leaving a pale-yellow benzoyl chloride residue which was dissolved in distilled acetonitrile (4 mL), followed by slow addition of an ice-cold mixture of the appropriate aminopyridine (**7**) and pyridine (3 mL) in dry acetonitrile (10 mL). The mixture was stirred at 0 °C for 30 min, warmed to room temperature and stirred for 11-18 h. Excess solvent was removed *in vacuo* and the residue dissolved in ethyl acetate (50 mL). The organic mixture was

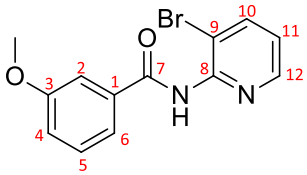
washed with aqueous saturated $NaHCO_3$ or aqueous K_2CO_3 (2 × 50 mL), saturated brine solution (50 mL) and the organic layer was separated and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product purified by normal silica gel chromatography eluting with 20-30% EtOAc/hexane. Recrystallization was performed using ethyl acetate and hexane where necessary to afford compounds **11**.

3-Methoxy-N-(5-methylpyridin-2-yl)benzamide (11a)



2-Amino-5-methylpyridine **7a** (0.710 g, 6.60 mmol) was reacted for 13 h as per the general method. Compound **11a** was obtained as a white solid (1.30 g, 81% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 100-102 °C; IR (ν/cm^{-1}) 3225 (NH, br), 1670 (C=O, str), 1213 (C-O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.98 (s, 1H, NH), 8.29 (d, J = 8.5 Hz, 1H, H9), 8.00 (d, J = 2.4 Hz, 1H, H12), 7.57 (dd, J = 2.4, 8.6 Hz, 1H, H10), 7.49 – 7.42 (m, 2H, H2 and H6), 7.37 (t, J = 7.9 Hz, 1H, H5), 7.09 (ddd, J = 1.1, 2.6, 8.2 Hz, 1H, H4), 3.85 (s, 3H, OCH_3), 2.28 (s, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.6 (C7), 159.9 (C3), 149.4 (C8), 147.8 (C12), 139.0 (C10), 135.9 (C1), 129.7 (C5), 129.3 (C11), 119.1 (C6), 118.5 (C4), 113.8 (C9), 112.3 (C2), 55.5 (C-O*C*H$_3$), 17.8 (C-*C*H$_3$); HRMS (ES+) calculated for $C_{14}H_{15}N_2O_2$ $[M+H]^+$: 243.1128, found: 243.1141.

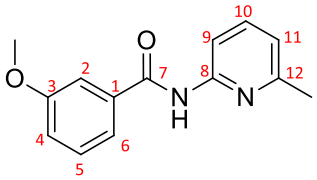
N-(3-Bromopyridin-2-yl)-3-methoxybenzamide (11b)



2-Amino-3-bromopyridine **7b** (1.12 g, 6.60 mmol) was reacted for 14 h. Compound **11b** was obtained as a cream white solid (0.97 g, 48% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 136-138 °C; IR (ν/cm^{-1}) 3183 (NH, br), 1651 (C=O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.66 (s, 1H, NH), 8.46 (s, 1H, H12), 7.93 (d, J = 7.8 Hz, 1H, H10), 7.53 – 7.46 (m, 2H, H2 and H6), 7.40 (t, J = 7.9 Hz, 1H, H5), 7.13 – 7.01 (m, 2H, H4 and H11), 3.87 (s, 3H, OCH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 164.8 (C7), 160.0 (C3), 148.7 (C8), 147.5 (C12), 141.5, 135.6 (C1), 129.9 (C5), 121.6

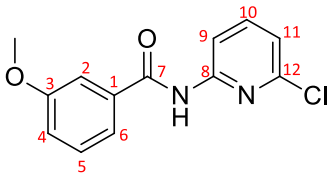
(C9), 119.2 (C6), 118.6 (C4), 112.9 (C2), 55.5 (OCH_3); HRMS (ES+) calculated for $C_{13}H_{12}BrN_2O_2$ $[M+H]^+$: 307.0077, found: 307.0077.

3-Methoxy-N-(6-methylpyridin-2-yl)benzamide (11c)



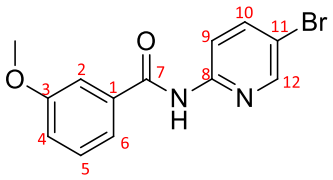
2-Amino-6-methylpyridine **7c** (0.710 g, 6.60 mmol) was reacted for 13 h. Compound **11c** was obtained as a white solid (1.38 g, 88% yield). Rf= 0.2 (70:30 EtOAc/hexane); M.p. = 57-59 °C; IR (ν/cm^{-1}) 3182 (NH, br), 1666 (C=O, str), 1227 (C-O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.76 (s, 1H, NH), 8.12 (d, *J* = 8.2 Hz, 1H, H9), 7.56 (t, *J* = 7.9 Hz, 1H, H10), 7.41 – 7.36 (m, 2H, H2 and H6), 7.28 (t, *J* = 7.9 Hz, 1H, H5), 7.00 (dd, *J* = 8.1, 2.4 Hz, 1H, H4), 6.84 (d, *J* = 7.5 Hz, 1H, H11), 3.76 (s, 3H, H-OCH_3), 2.34 (s, 3H, H-CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.6 (C7), 159.9 (C3), 156.8 (C8), 150.8 (12), 138.8 (C10), 135.8 (C1), 129.7 (C5), 119.5 (C6), 119.0 (C11), 118.6 (C4), 112.3 (C2), 111.1 (C9), 55.5 (OCH_3), 23.9 (C-*C*H_3).

N-(6-Chloropyridin-2-yl)-3-methoxybenzamide (11d)



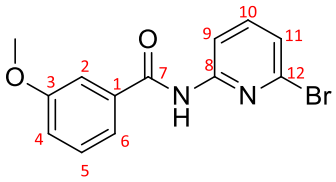
2-Amino-6-chloropyridine **7d** (0.86 g, 6.60 mmol) was reacted for 11 h, purified by column chromatography and recrystallized from toluene and hexane. Compound **11d** was obtained as a white solid (1.13 g, 65% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 100-102 °C (lit.[3] 66-67 °C), IR (ν/cm^{-1}) 3390 (NH, br), 1640 (C=O, str), 1188 (C-O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.73 (s, 1H, NH), 8.28 (d, *J* = 8.2 Hz, 1H, H9), 7.65 (t, *J* = 8.0 Hz, 1H, H10), 7.43 – 7.38 (m, 2H, H2 and H6), 7.32 (t, *J* = 7.9 Hz, 1H, H5), 7.07 – 7.01 (m, 2H, H4 and H11), 3.80 (s, 3H, OCH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.5 (C7), 159.9 (C3), 151.4 (C8), 148.9 (C12), 141.0 (C10), 135.1 (C1), 129.8 (C5), 119.8 (C11), 119.1 (C6), 118.8 (C4), 112.4 (C2), 112.2 (C9), 55.4 (OCH_3); HRMS (ES+) Calculated for $C1_3H_{11}ClN_2O_2$ $[M+H]^+$: 263.0582, found: 263.0580.

N-(5-Bromopyridin-2-yl)-3-methoxybenzamide (11e)



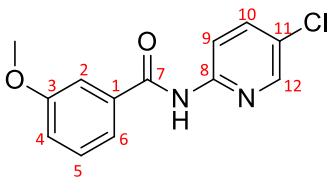
2-Amino-5-bromopyridine **7e** (1.12 g, 6.60 mmol) was reacted for 18 h. Compound **11e** was obtained as a white solid (1.66 g, 99% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 61-63°C; IR (ν/cm^{-1}) 3232 (NH, br), 1671 (C=O, str), 1213 (C-O, str); ^{1}H NMR (500 MHz, $CDCl_3$) δ_H 8.75 (s, 1H, NH), 8.35 – 8.33 (m, 1H, H9), 8.29 – 8.26 (m, 1H, H12), 7.84 (dd, J = 2.4, 8.9 Hz, 1H, H10), 7.47 – 7.36 (m, 3H, H2, H5, and H6), 7.11 (ddd, J = 1.2, 2.6, 8.1 Hz, 1H, H4), 3.87 (s, 3H, OCH_3); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C 165.6 (C7), 160.1 (C3), 150.3 (C8), 148.8 (C12), 140.9 (C10), 135.4 (C1), 129.9 (C5), 119.0 (C6), 118.7 (C4), 115.4 (C9), 114.8 (C11), 112.5 (C2), 55.5 (OCH_3); HRMS (ES+) calculated for $C_{13}H_{12}BrN_2O_2$ $[M+H]^+$: 307.0077, found: 307.0088.

N-(6-Bromopyridin-2-yl)-3-methoxybenzamide (11f)



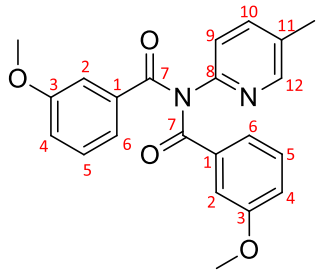
2-Amino-6-bromopyridine **7f** (1.10 g, 6.60 mmol) was reacted for 12 h and recrystallized from toluene and hexane after column chromatographic purification. Compound **11f** was obtained as a white solid (1.13 g, 56% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 95-97 °C, IR (ν/cm^{-1}) 3394 (NH, br), 1644 (C=O,str), 1166 (C-O, str); ^{1}H NMR (300 MHz, $CDCl_3$) δ_H 8.74 (s, 1H, NH), 8.34 (d, J = 8.2 Hz, 1H, H11), 7.58 (t, J = 8.0 Hz, 1H, H10), 7.46 – 7.40 (m, 2H, H2 and H6), 7.39 – 7.32 (m, 1H, H5), 7.25 – 7.19 (m, 1H, H9), 7.11 – 7.05 (m, 1H, H4), 3.84 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 165.5 (C7), 160.0 (C3), 151.6 (C8), 140.7 (C10), 139.3 (C12), 135.0 (C1), 129.8 (C5), 123.7 (C9), 119.1 (C6), 118.9 (C4), 112.5 (C2), 112.4 (C11) , 55.5 (OCH_3); HRMS (ES+) calculated for $C_{13}H_{12}BrN_2O_2$ $[M+H]^+$: 307.0077, found: 307.0070.

N-(5-Chloropyridin-2-yl)-3-methoxybenzamide (11g)



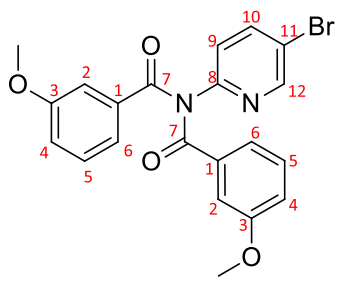
2-Amino-5-chloropyridine **7g** (0.840 g, 6.60 mmol) was reacted for 24 h and recrystallized after column chromatography purification. Compound **11g** was obtained as a white solid (1.07 g, 62% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 84-86 °C (lit.[3] 96-97 °C); IR (ν/cm^{-1}) 3218 (NH, br), 1674 (C=O str), 1218 (C-O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.76 (s, 1H, NH), 8.38 (d, J = 8.9 Hz, 1H, H9), 8.18 (d, J = 1.8 Hz, 1H, H12), 7.71 (dd, J = 8.9, 2.0 Hz, 1H, H10), 7.48 – 7.35 (m, 3H, H2, H5, and H6), 7.13 – 7.08 (m, 1H, H4), 3.86 (s, 3H, H-OCH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 165.5 (C7), 160.0 (C3), 149.8 (C8), 146.5 (C12), 138.1 (C10), 135.3 (C1), 129.9 (C5), 126.9 (C11), 119.0 (C6), 118.7 (C5), 114.8 (C9), 112.5 (C2), 55.5 (OCH_3).

3-Methoxy-N-(3-methoxybenzoyl)-N-(5-methylpyridin-2-yl)benzamide (12a)



2-Amino-5-methylpyridine **7a** (0.71 g, 6.5 mmol) was reacted for 13 h as per the general method, except triethylamine was used as a base. Compound **12a** was obtained as a white solid (0.97 g, 78% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 128-130 °C; IR (ν/cm^{-1}) 1681 (C=O, str), 1207 (C-O, str); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 8.23 – 8.21 (m, 1H, H12), 7.53 (dd, J = 8.1, 2.1 Hz, 1H, H10), 7.34 (d, J = 7.3 Hz, 4H, H2 and H6), 7.23 (t, J = 7.9 Hz, 2H, H5), 7.16 (d, J = 8.1 Hz, 1H, H9), 6.99 (dd, J = 8.0, 2.9 Hz, 2H, H4), 3.76 (s, 6H, 2 x OCH_3), 2.30 (s, 3H, H-CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ_C 172.9 (C7), 159.6 (C3), 151.3 (C8), 149.6 (C12), 138.9 (C10), 136.0 (C1), 132.1 (C11), 129.5 (C5), 121.6 (C9), 121.4 (C6), 119.0 (C4), 113.9 (C2), 55.4 (OCH_3), 18.0 (CH_3); HRMS (ES+) calculated for $C_{22}H_{21}N_2O_4$ $[M+H]^+$: 377.1496, found: 377.1505.

N-(5-Bromopyridin-2-yl)-3-methoxy-N-(3-methoxybenzoyl)benzamide (12e)

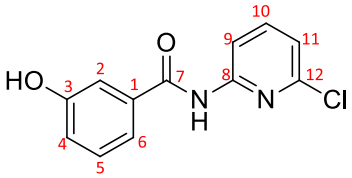


2-Amino-5-bromopyridine **7e** (1.12 g, 6.60 mmol) was reacted for 18 h as per the general method, except triethylamine was used as a base. Compound **12e** was obtained as a white solid (1.43 g, 99% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p= 138-140 °C; IR (*v*/cm^{-1}) 1678 (C=O, str), 1208 (C-O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 8.47 (d, *J* = 2.3 Hz, 1H, H12), 8.20 (dd, *J* = 8.6, 2.5 Hz, 1H, H10), 7.68 (d, *J* = 8.6 Hz, 1H, H9), 7.37 (d, *J* = 7.3 Hz, 4H, H5 and H6), 7.28 (d, *J* = 2.6 Hz, 2H, H2), 7.14 (dt, *J* = 7.3, 2.3 Hz, 2H, H4), 3.73 (s, 1H, H-OCH_3); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 172.6 (C7), 159.6 (C3), 152.6 (C8), 149.9 (C12), 141.9 (C10), 135.8 (C1), 130.5 (C5), 124.4 (C9), 121.8 (C11), 119.0 (C4), 118.9 (C6), 114.6 (C2), 55.8 (C-OCH_3); HRMS (ES+) calculated for $C_{21}H_{18}BrN_2O_4$ $[M+H]^+$: 441.0444, found: 441.0439.

General method for the reaction of 3-hydroxybenzoic acid with aminopyridines

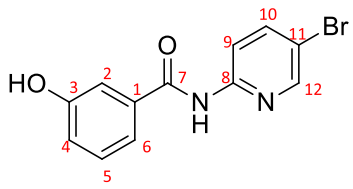
3-Hydroxybenzoic acid **13** (1.00 g, 7.20 mmol) was placed in a round-bottomed flask, followed by addition of excess thionyl chloride (2 mL). The mixture was stirred and refluxed at 80 °C in an oil bath for 24 h. After cooling the mixture to room-temperature, excess thionyl chloride was evaporated leaving a pale-yellow benzoyl chloride residue which was dissolved in distilled acetonitrile (4 mL), followed by slow addition of an ice-cold mixture of the appropriate aminopyridine (**7**) and pyridine (3 mL) in dry acetonitrile (10 mL). The mixture was stirred at 0 °C for 30 min, warmed to room temperature and stirred for 21-24 h. Excess solvent was removed *in vacuo* and the residue dissolved in ethyl acetate (50 mL). The organic mixture was washed with aqueous saturated $NaHCO_3$ or aqueous K_2CO_3 (50 × 2 mL) and saturated brine solution (50 mL) and the organic layer was separated and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product purified by normal silica gel chromatography eluting with 30% EtOAc/hexane. The impure product obtained after chromatography was further purified to give pure product **14** by extraction into ethyl acetate, leaving an insoluble residue behind.

N-(6-Chloropyridin-2-yl)-3-hydroxybenzamide (14d)



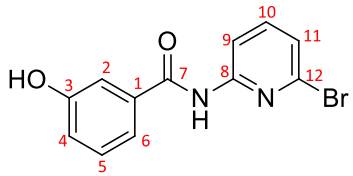
2-Amino-6-chloropyridine **7d** (0.930 g, 7.20 mmol) was reacted 24 h as per the general method. Compound **14d** was isolated as a light brown solid (0.92 g, 51% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 232-234 °C; IR (ν/cm^{-1}) 3392 (NH, br), 3118 (OH, br), 1690 (C=O, str), 1290 (C-O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 10.98 (s, 1H, NH), 9.76 (s, 1H, H-OH), 8.17 (d, J = 8.2 Hz, 1H, H9), 7.89 (t, J = 8.0 Hz, 1H, H10), 7.47 (d, J = 7.7 Hz, 1H, H6), 7.37 (s, 1H, H2), 7.32 – 7.23 (m, 2H, H5 and H11), 6.98 (dd, J = 8.0, 1.9 Hz, 1H, H4); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 166.2 (C7), 157.3 (C3), 152.3 (C8), 148.0 (C12), 141.6 (C10), 135.1 (C1), 129.4 (C5), 119.5 (C11), 119.2 (C4), 118.7 (C6), 115.0 (C2), 113.4 (C9); HRMS (ES+) calculated for $C_{12}H_{10}ClN_2O_2$ $[M+H]^+$: 249.0425, found: 249.0427.

N-(5-Bromopyridin-2-yl)-3-hydroxybenzamide (14e)



2-Amino-5-bromopyridine **7e** (1.25 g, 7.20 mmol) was reacted for 21 h. Compound **14e** was isolated as brown solid (1.21 g, 53% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 225-227 °C; IR (ν/cm^{-1}) 3397 (NH, br), 3065 (OH, br), 1686 (C=O, str), 1292 (C-O, str); ^{1}H NMR (400 MHz, DMSO-d_6) δ_H 10.85 (s, 1H, NH), 9.78 (s, 1H, H-OH), 8.51 (s, 1H, H12), 8.22 – 8.01 (m, 2H, H9 and H10), 7.51 – 7.26 (m, 3H, H2, H5, and H6), 7.02 – 6.95 (m, 1H, H4); ^{13}C NMR (101 MHz, DMSO-d_6) δ_C 166.2 (C7), 157.4 (C3), 151.2 (C8), 148.5 (C12), 140.5 (C10), 135.3 (C1), 129.4 (C5), 119.1 (C4), 118.6 (C6), 116.4 (C2), 114.9 (C9), 113.9 (C11); HRMS (ES+) calculated for $C_{12}H_{10}BrN_2O_2$ $[M+H]^+$: 292.9920, found: 292.9923.

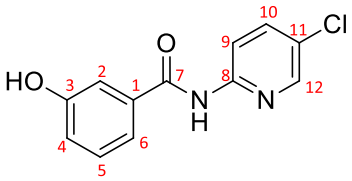
N-(6-Bromopyridin-2-yl)-3-hydroxybenzamide (14f)



2-Amino-6-bromopyridine **7f** (1.25 g, 7.20 mmol) was reacted for 23 h. Compound **14f** was isolated as a brown solid (0.60g, 28% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p. = 243-245 °C; IR (ν/cm^{-1}) 3393 (NH, br), 3115 (OH, br), 1689 (C=O str), 1289 (C-O); ^{1}H NMR (400 MHz, DMSO-

d$_6$) δ_H 10.98 (s, 1H, NH), 9.75 (s, 1H, OH), 8.19 (d, *J* = 8.1 Hz, 1H, H9), 7.78 (t, *J* = 7.9 Hz, 1H, H10), 7.47 (d, *J* = 7.7 Hz, 1H, H6), 7.41 – 7.35 (m, 2H, H2 and H11), 7.29 (t, *J* = 7.9 Hz, 1H, H5), 7.00 – 6.96 (m, 1H, H4); ^{13}C NMR (101 MHz, DMSO-d$_6$) δ_C 166.2 (C7), 157.3 (C3), 152.5 (C8), 141.2 (C10), 138.8 (C12), 135.0 (C1), 129.4 (C5), 123.4 (C11), 119.2 (C6), 118.7 (C4), 115.0 (C2), 113.7 (C9); HRMS (ES+) calculated for $C_{12}H_{10}BrN_2O_2$ [M+H]$^+$: 292.9920, found: 292.9919.

N-(5-Chloropyridin-2-yl)-3-hydroxybenzamide (14g)



2-Amino-5-chloropyridine **7g** (0.930 g, 7.20 mmol) was reacted for 21 h. Compound **14g** was isolated as a brown solid (0.82 g, 46% yield). Rf= 0.2 (7:3 EtOAc/hexane); M.p= 215-217 °C; IR (ν/cm^{-1}) 3396 (NH, br), 1689 (C=O, str), 1293 (C-O, str); ^{1}H NMR (400 MHz, DMSO-d$_6$) δ_H 10.81 (s, 1H, NH), 9.71 (s, 1H, OH), 8.37 (d, *J* = 2.4 Hz, 1H, H12), 8.15 (d, *J* = 8.9 Hz, 1H, H9), 7.89 (dd, *J* = 8.9, 2.6 Hz, 1H, H10), 7.40 (d, *J* = 7.9 Hz, 1H, H6), 7.32 – 7.28 (m, 1H, H2), 7.24 (t, *J* = 7.9 Hz, 1H, H5), 6.92 (dd, *J* = 8.0, 1.8 Hz, 1H, H4); ^{13}C NMR (101 MHz, DMSO-d$_6$) δ_C 166.6 (C7), 157.8 (C3), 151.3 (C8), 146.7 (C12), 138.2 (C10), 135.7 (C1), 129.9 (C5), 125.9 (C11), 119.5 (C4), 119.0 (C6), 116.3 (C9), 115.3 (C2); HRMS (ES+) calculated for $C_{12}H_{10}ClN_2O_2$ [M+H]$^+$: 249.0425, found: 249.0426.

NMR Spectra

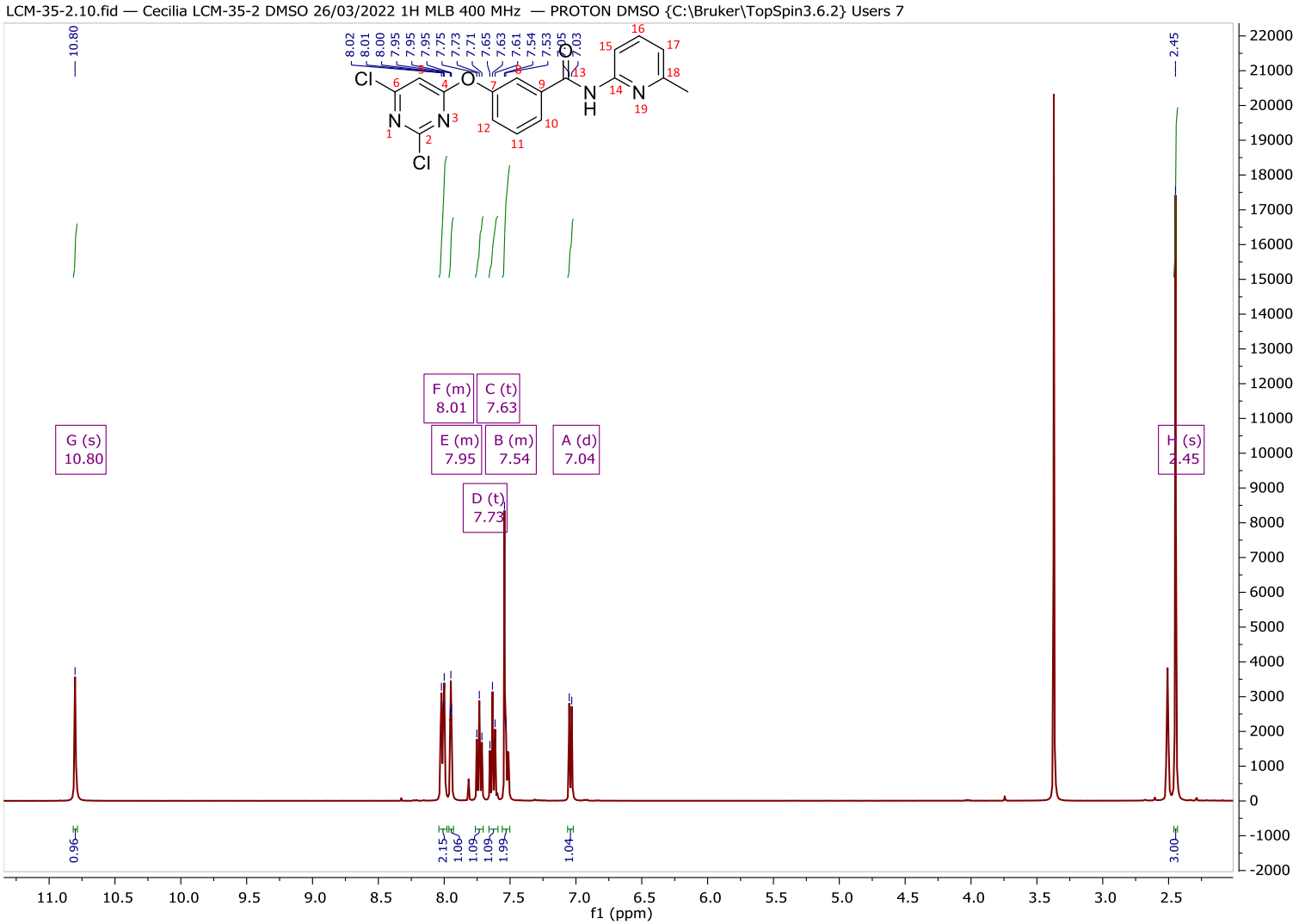


Fig. S1. ^{1}H NMR spectrum of compound **17c**.

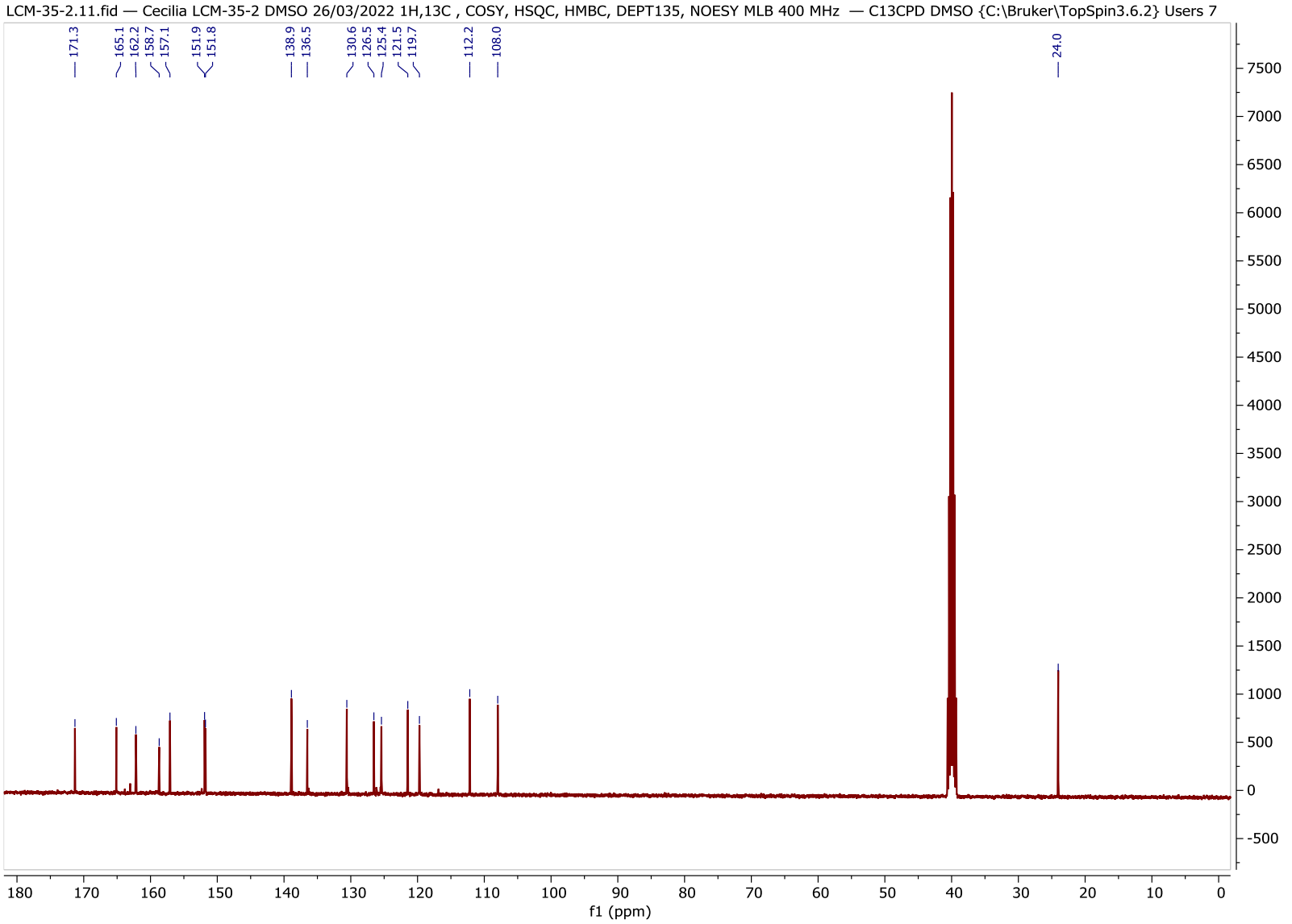


Fig. S2. ^{13}C NMR spectrum of compound **17c**.

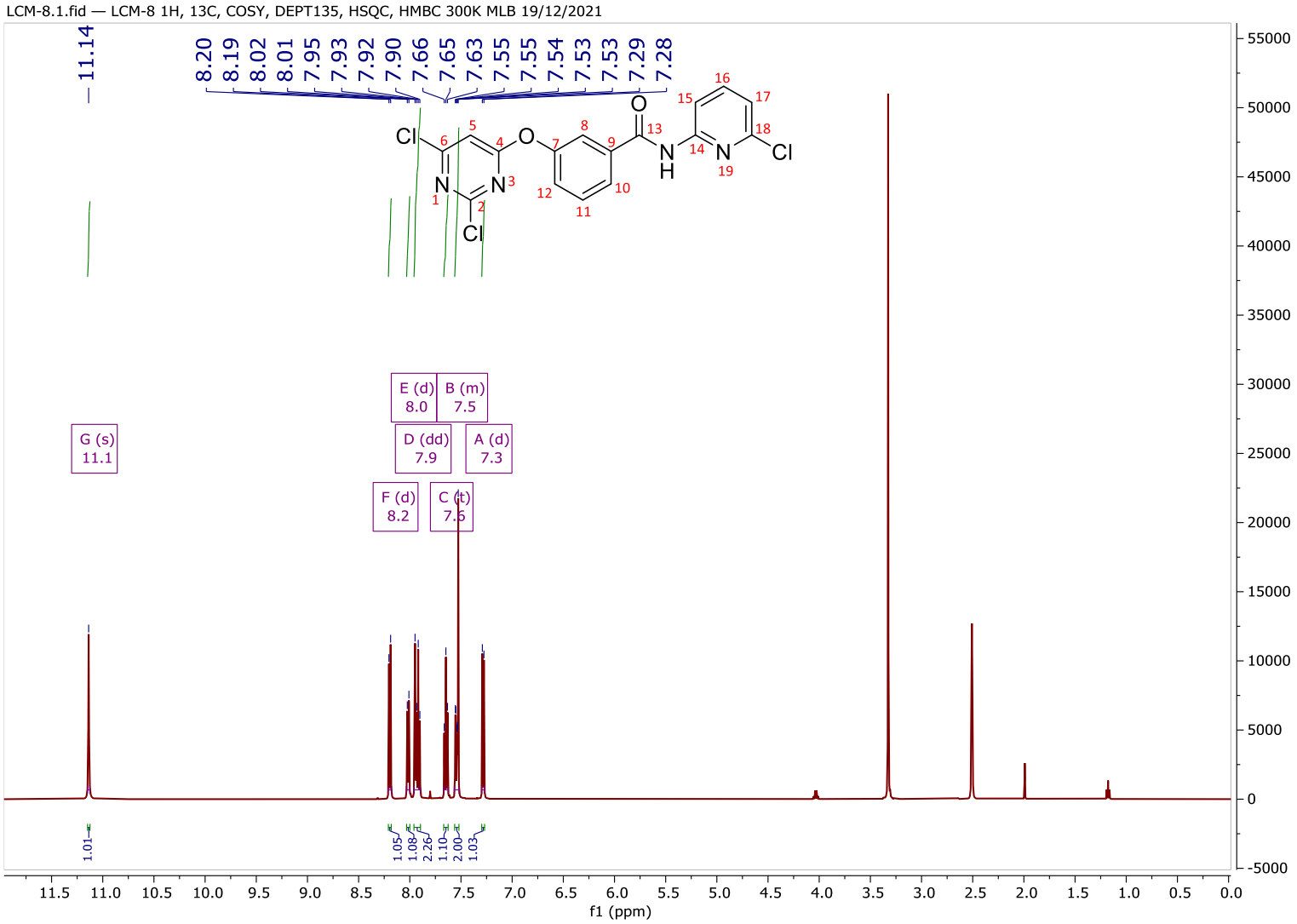


Fig. S3. ^{1}H NMR spectrum of compound **17d**.

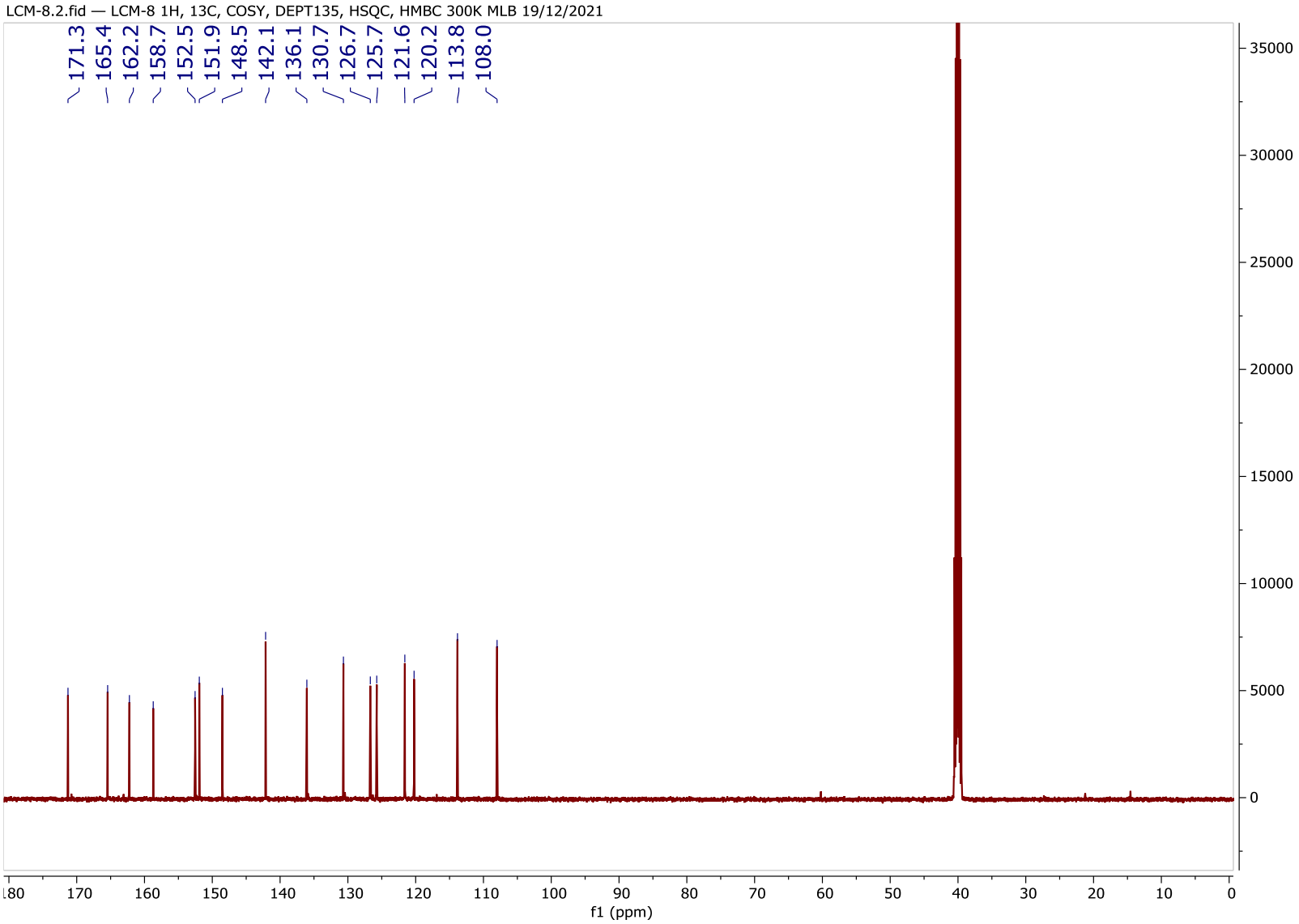


Fig. S4. ^{13}C NMR spectrum of compound **17d**.

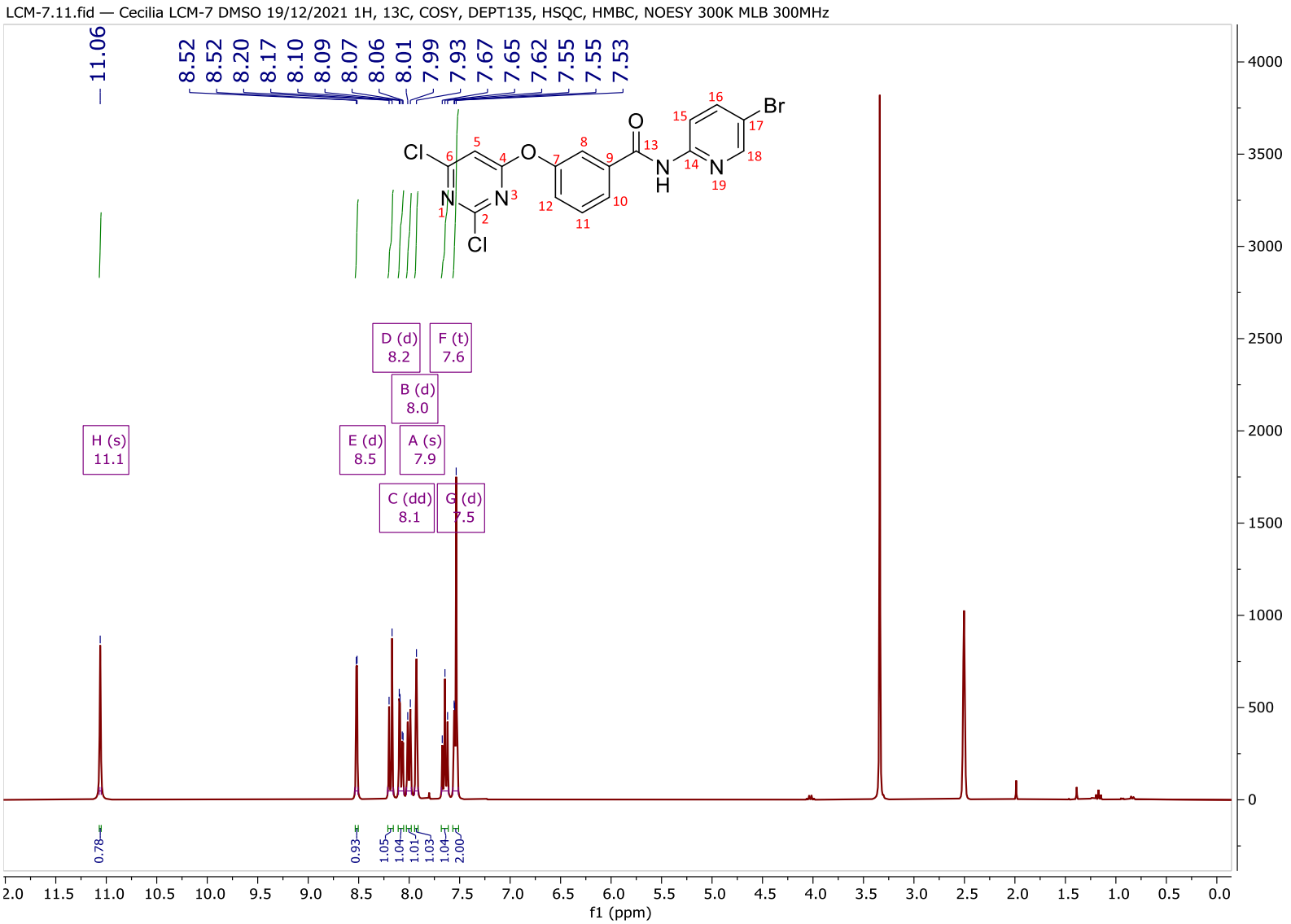


Fig. S5. ^{1}H NMR spectrum of compound **17e**.

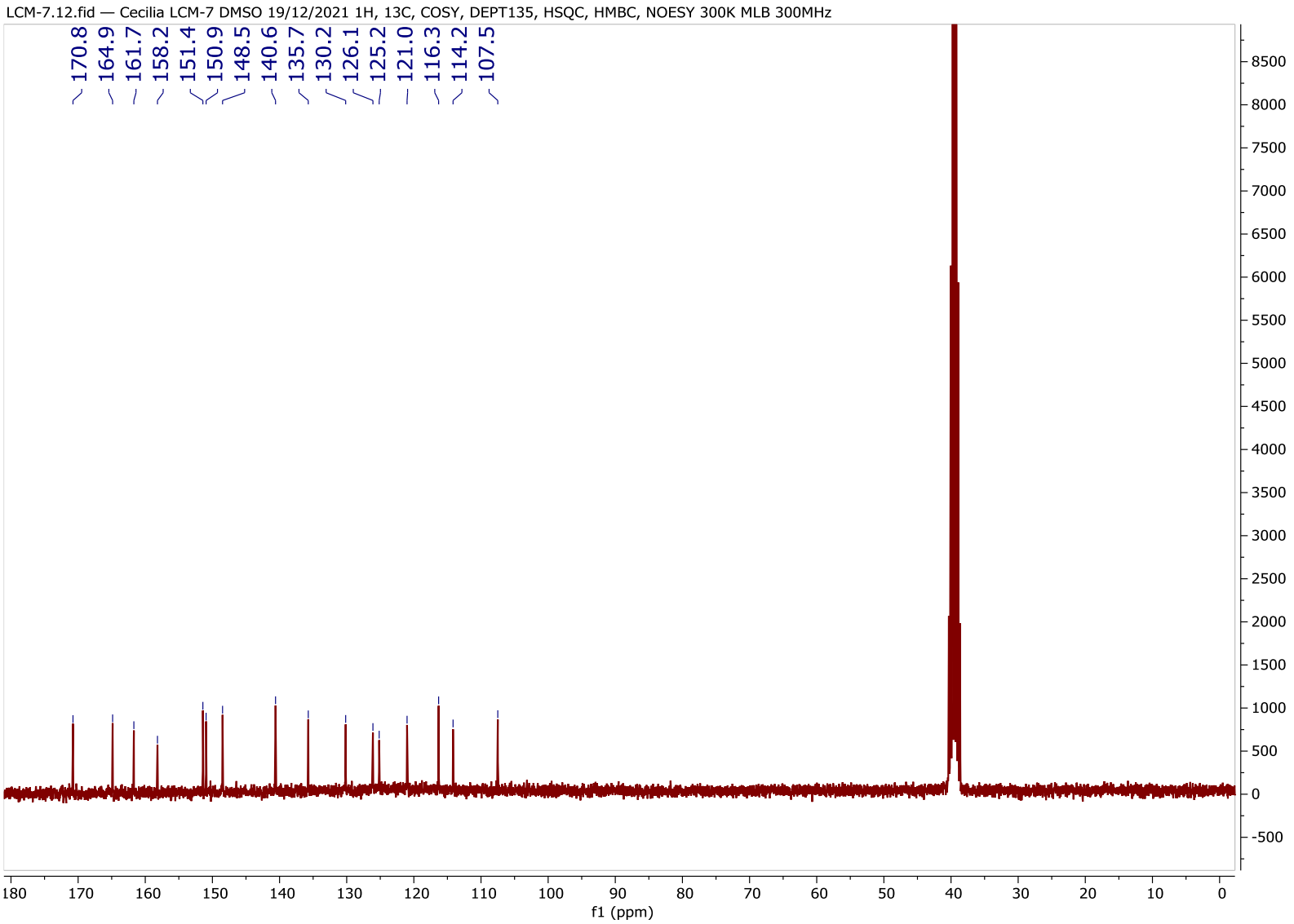


Fig. S6. ^{13}C NMR spectrum of compound **17e**.

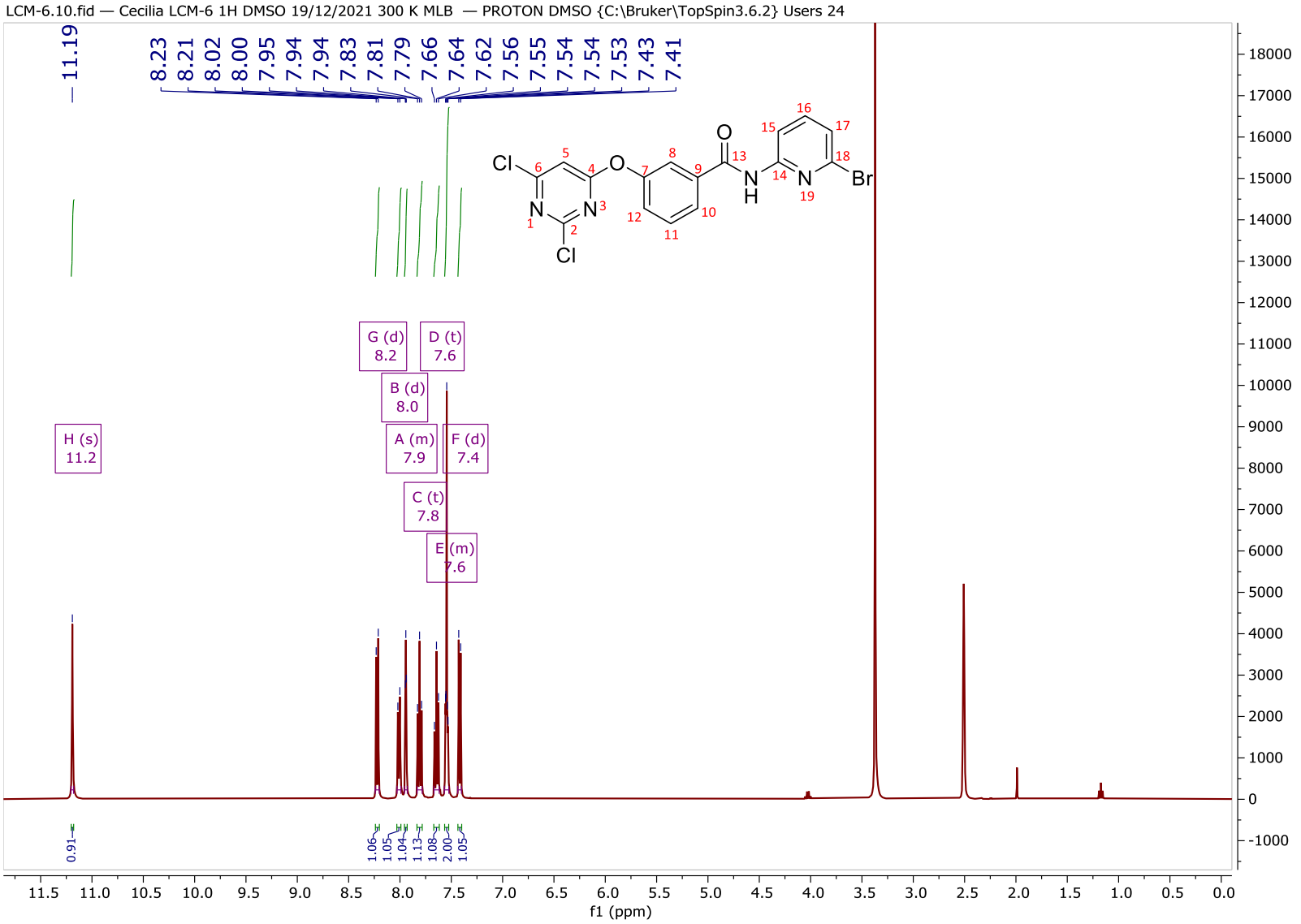


Fig. S7. ^{1}H NMR spectrum of compound **17f**.

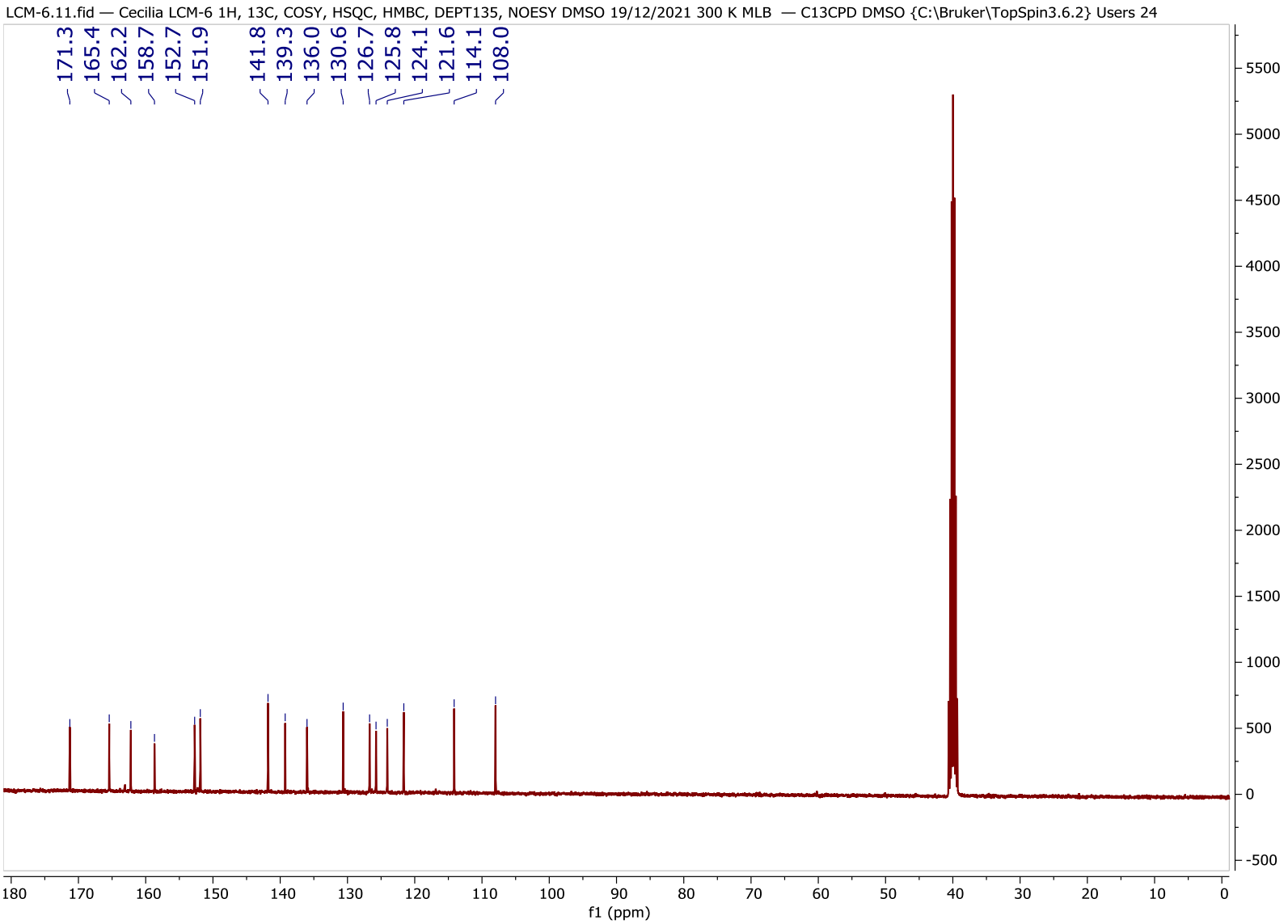


Fig. S8. ^{13}C NMR spectrum of compound **17f**.

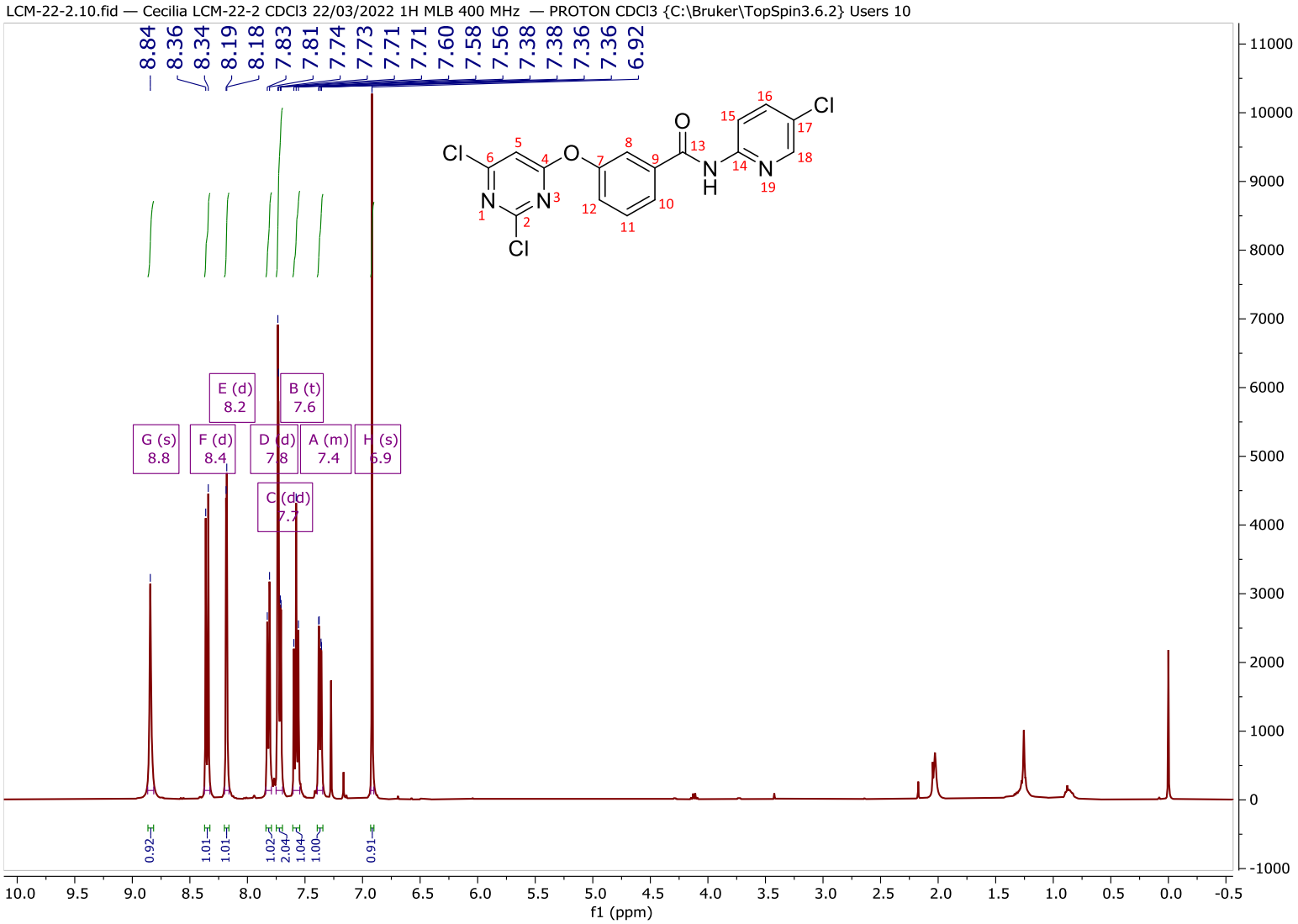


Fig. S9. ^{1}H NMR spectrum of compound **17g**.

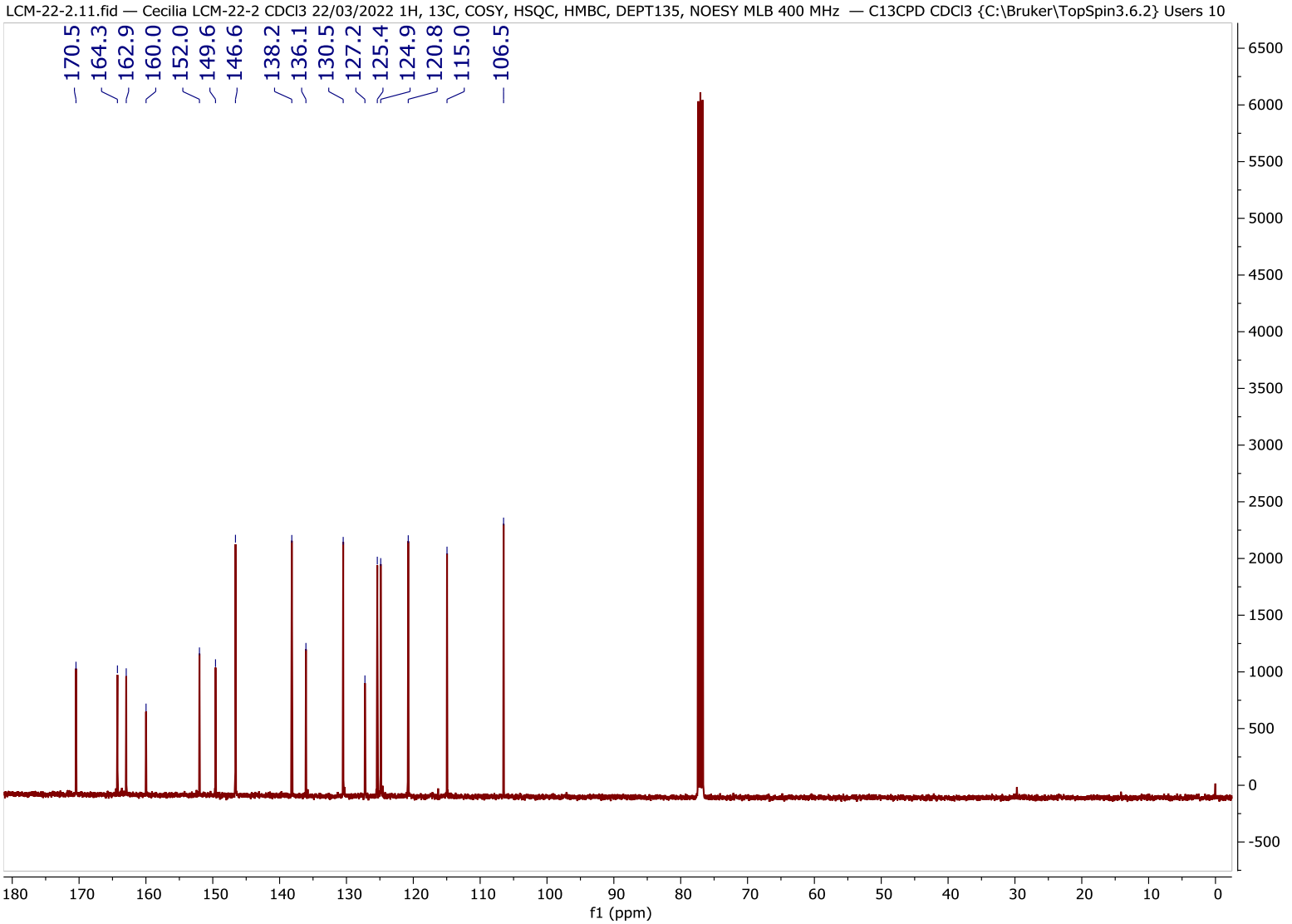


Fig. S10. ^{13}C NMR spectrum of compound **17g**.

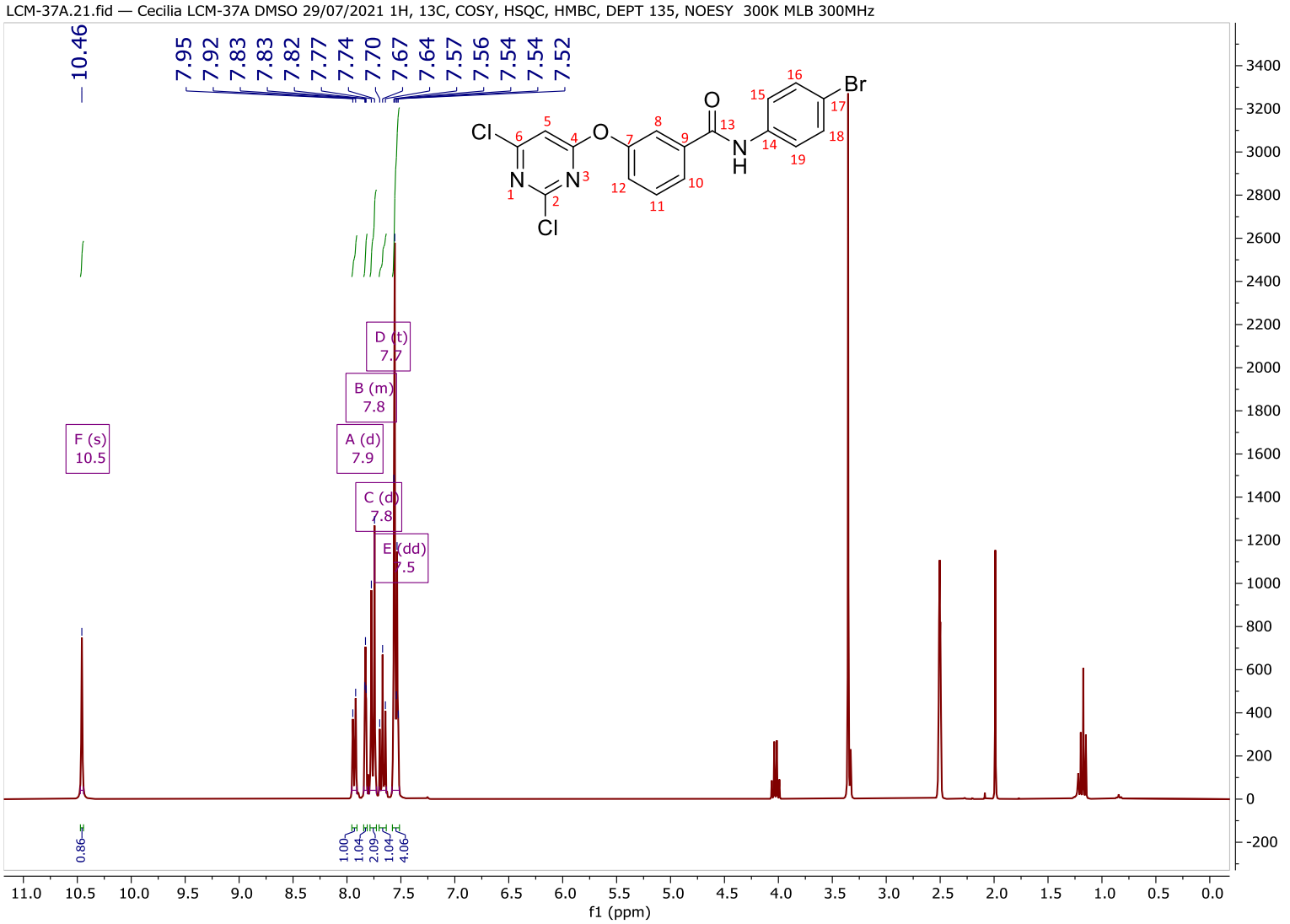


Fig. S11. ^{1}H NMR spectrum of compound **19a**.

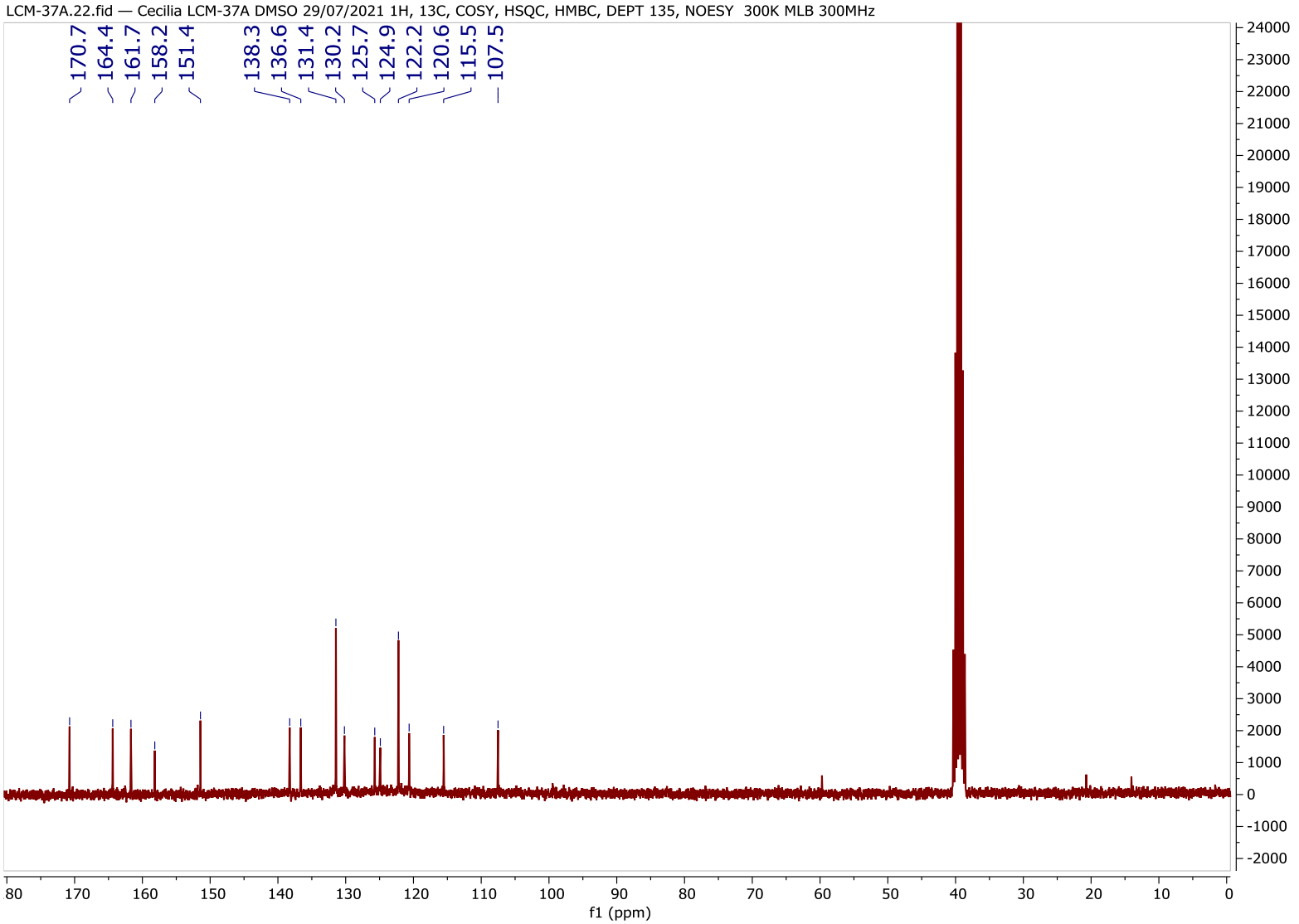


Fig. S12. ^{13}C NMR spectrum of compound **19a**.

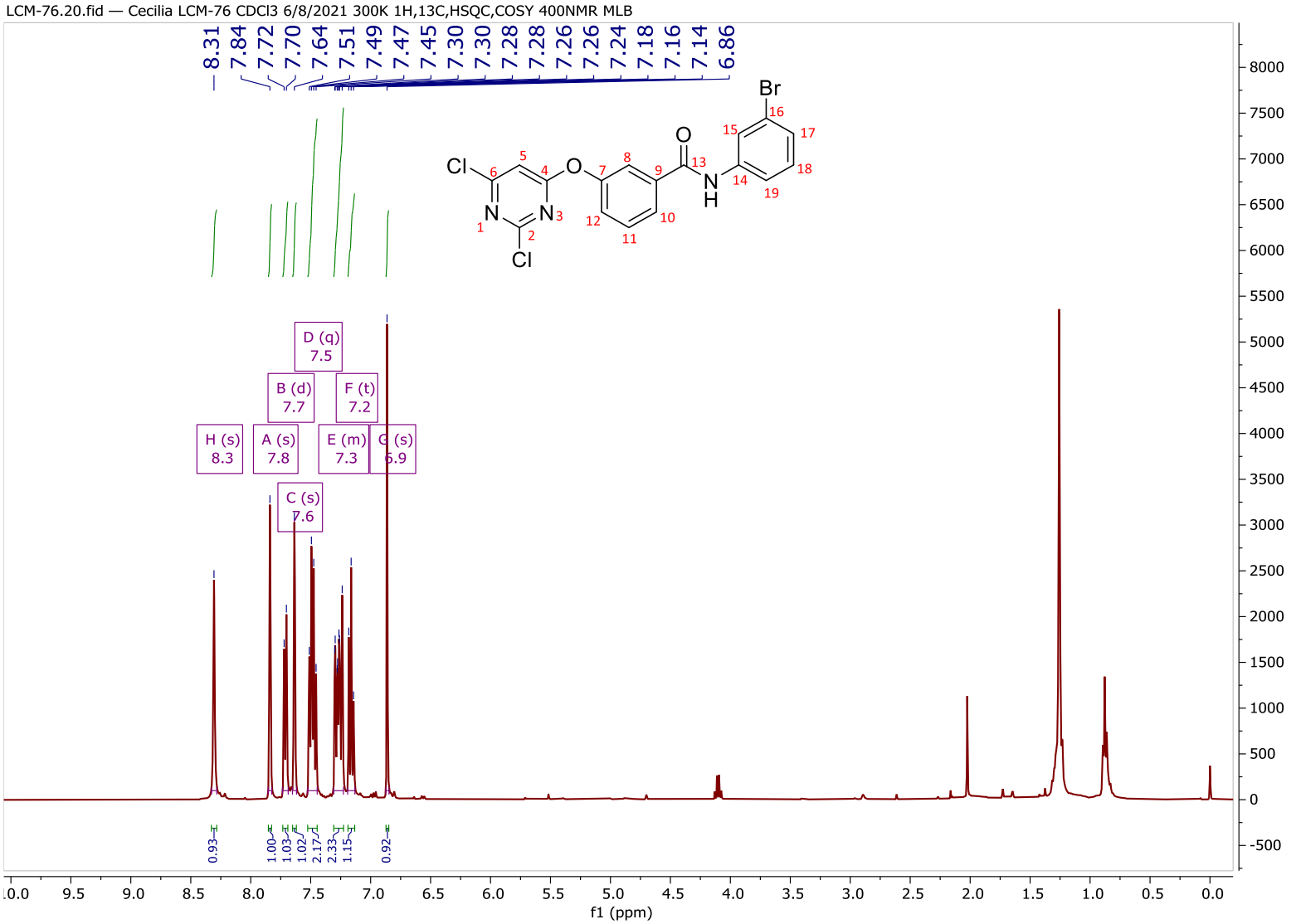


Fig. S13. ^{1}H NMR spectrum of compound **19b**.

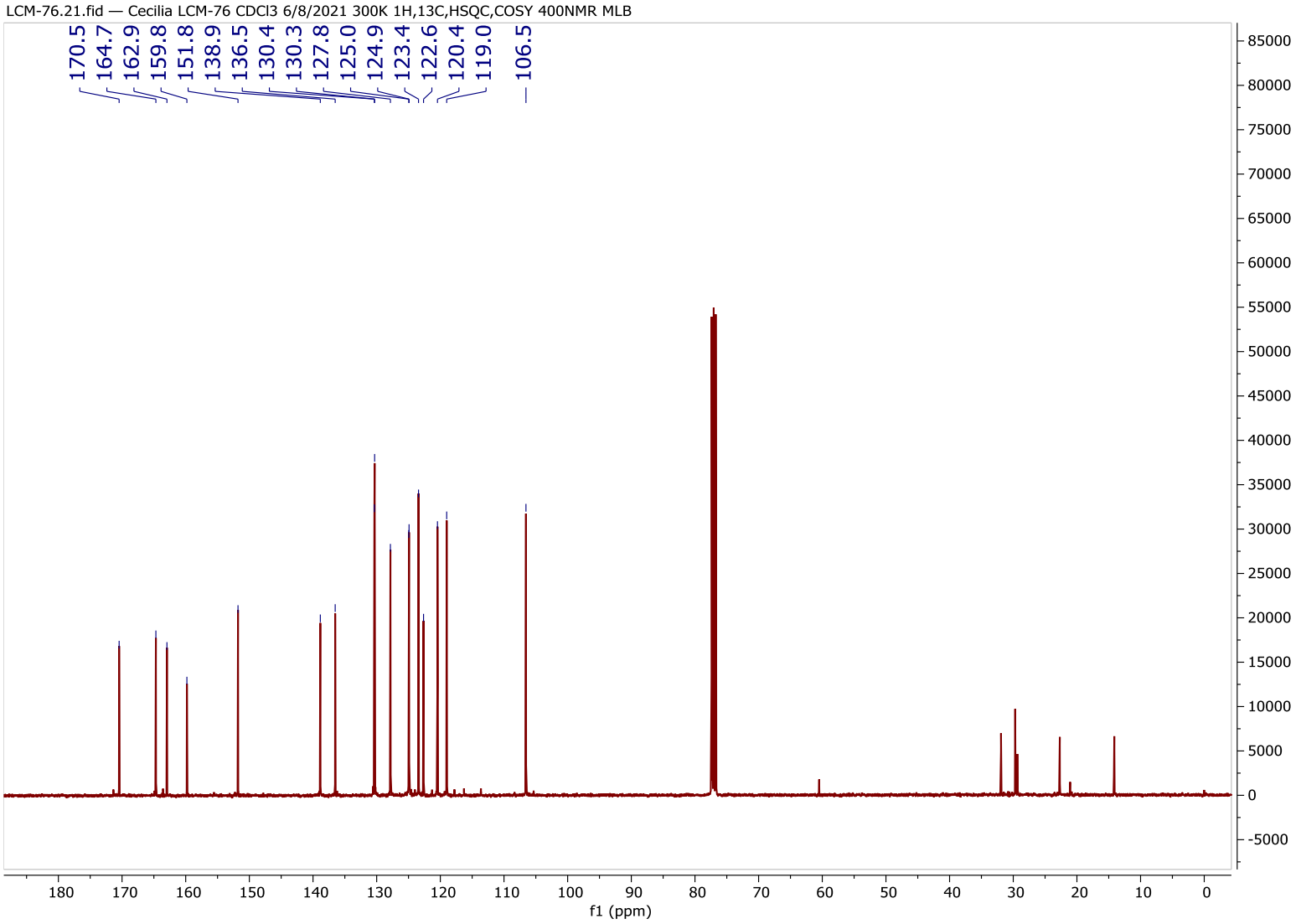


Fig. S14. ^{13}C NMR spectrum of compound **19b**.

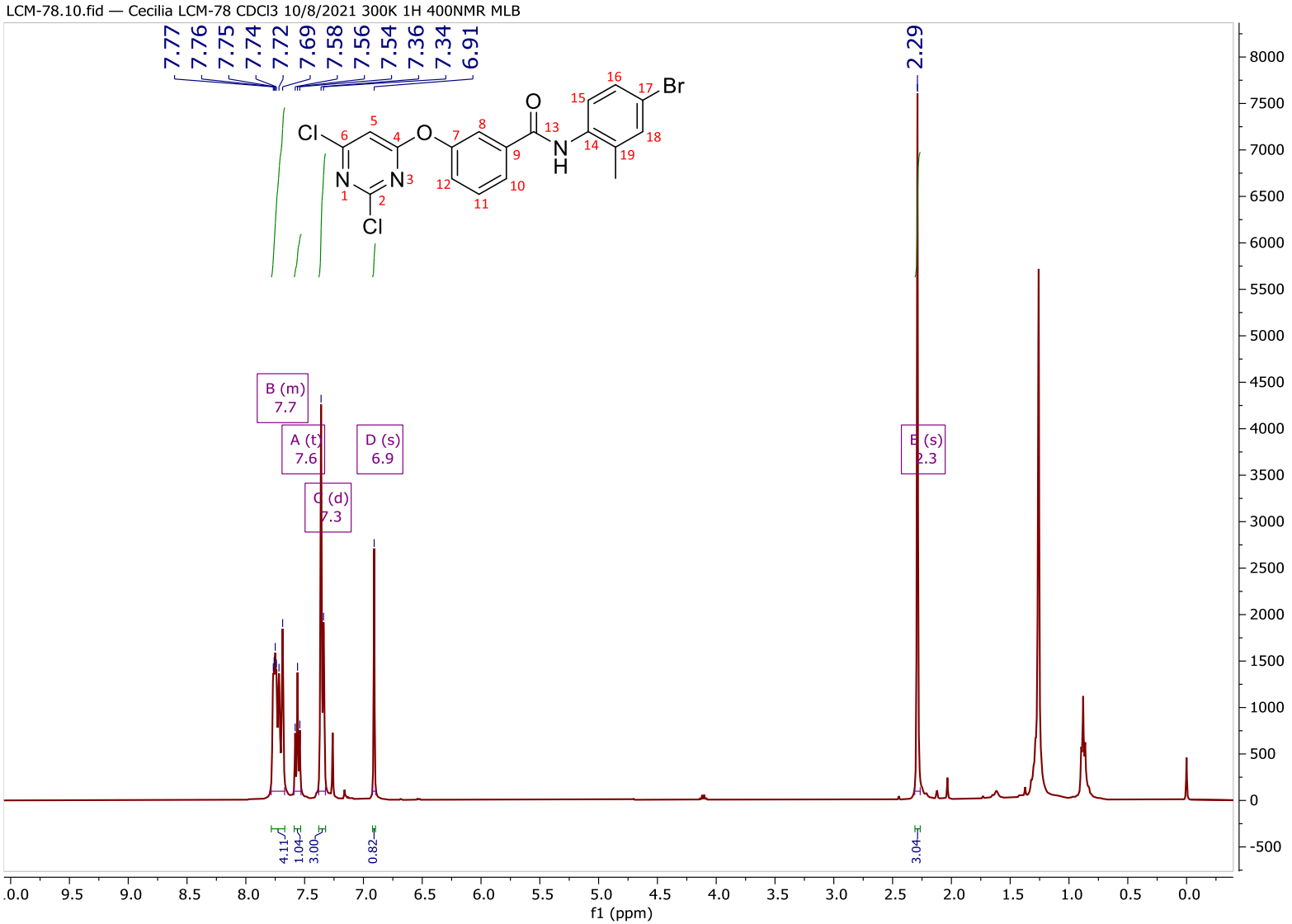


Fig. S15. ^{1}H NMR spectrum of compound **19c**.

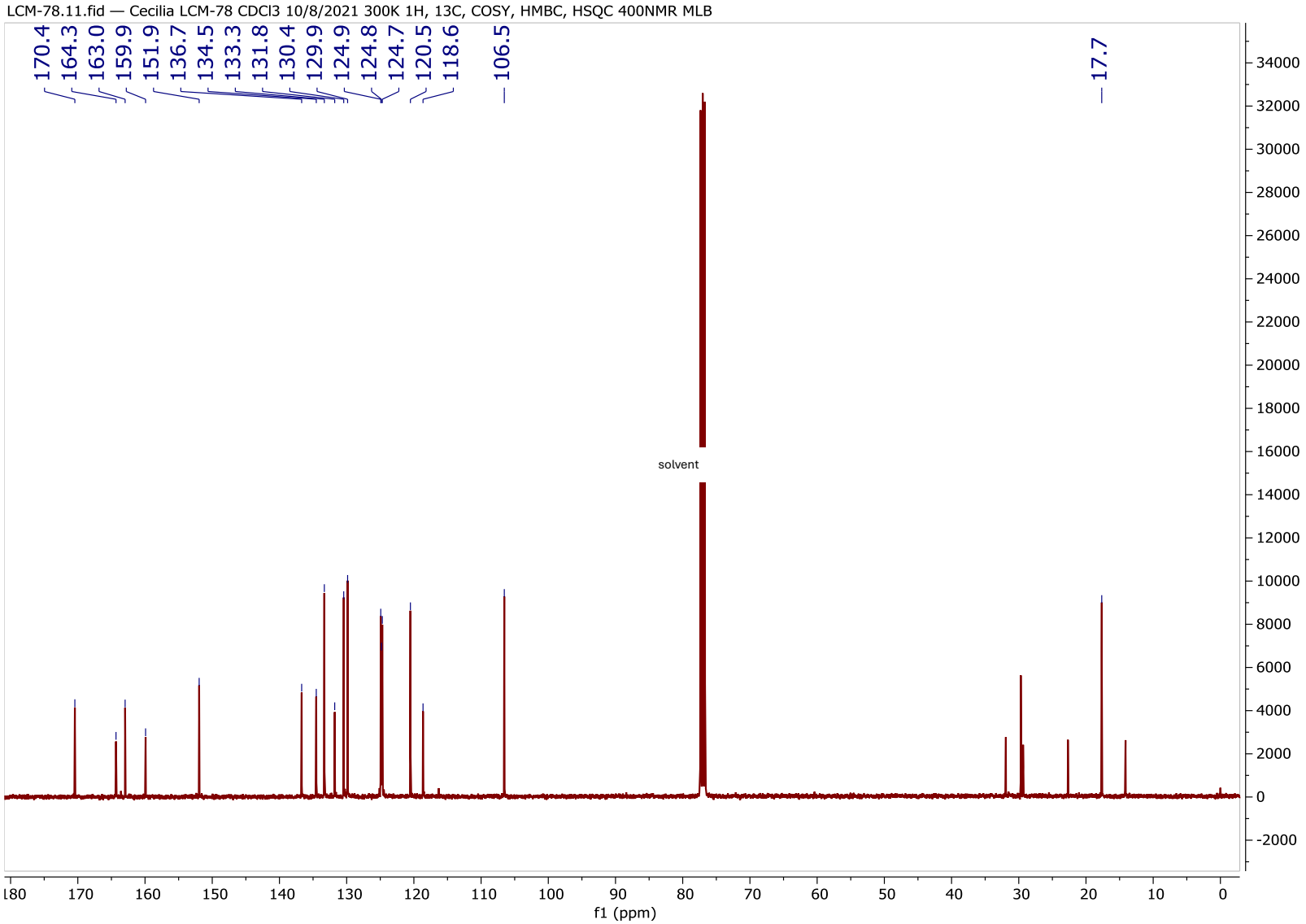


Fig. S16. ^{13}C NMR spectrum of compound **19c**.

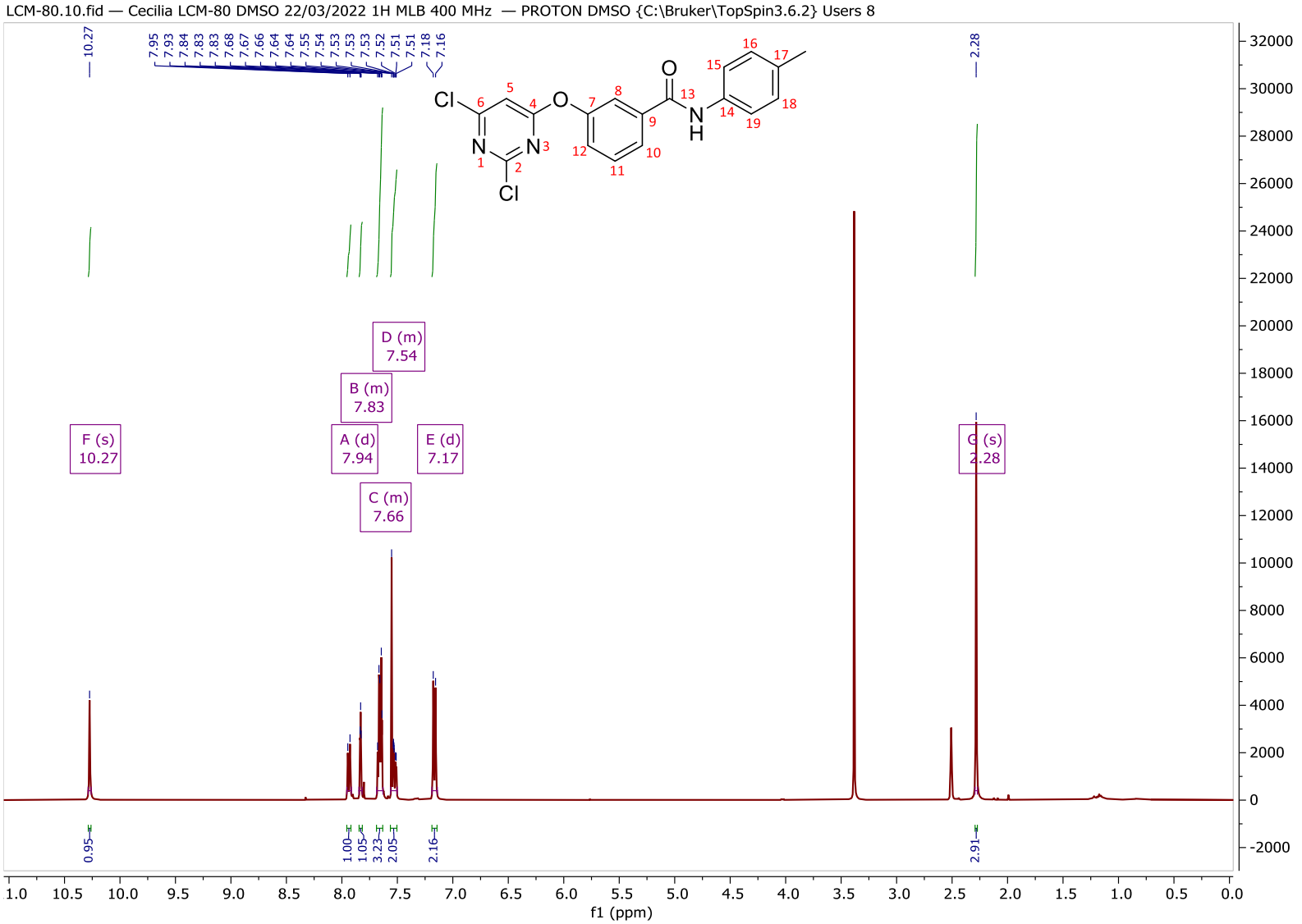


Fig. S17. ^{1}H NMR spectrum of compound **19d**.

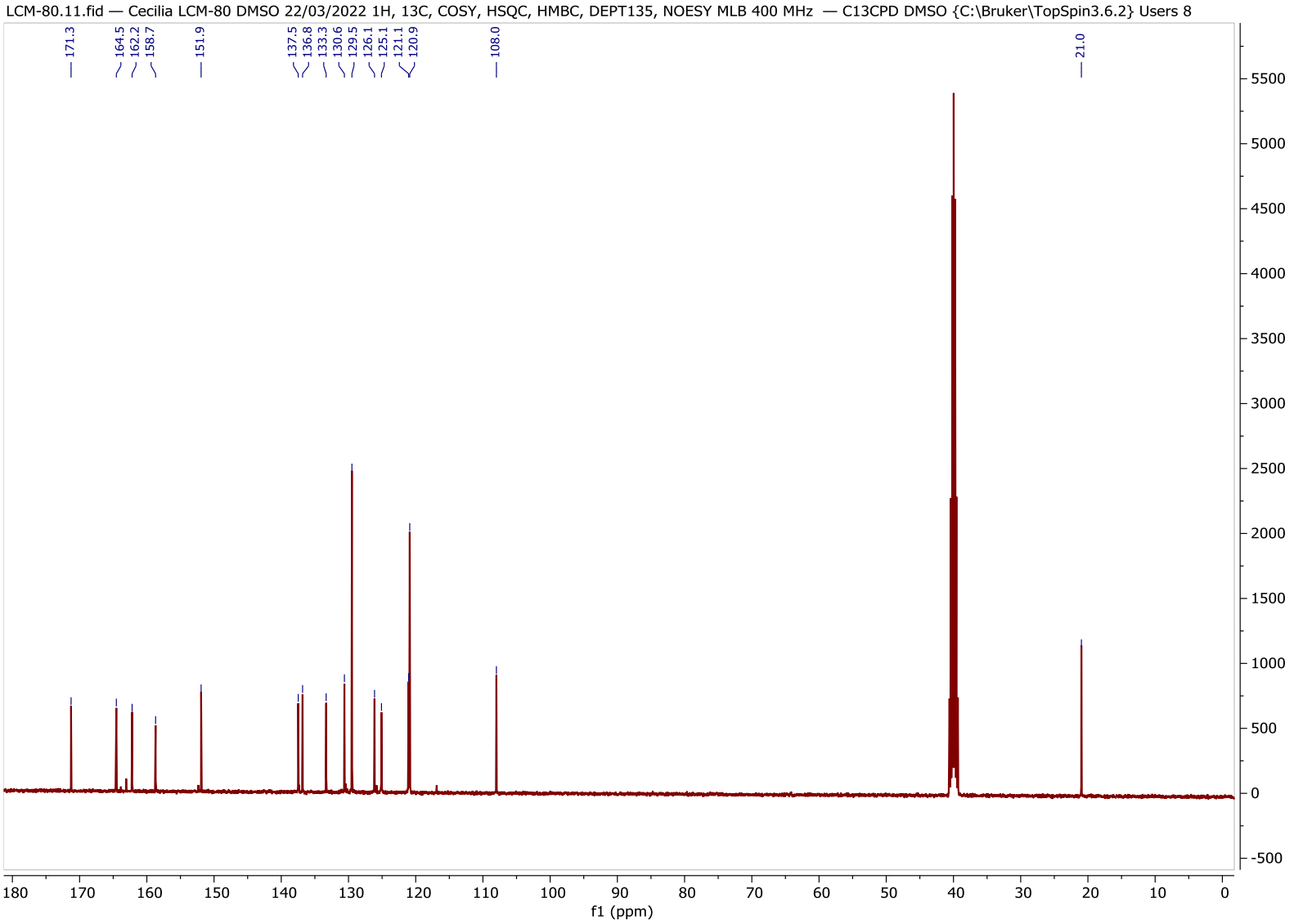


Fig. S18. ^{13}C NMR spectrum of compound **19d**.

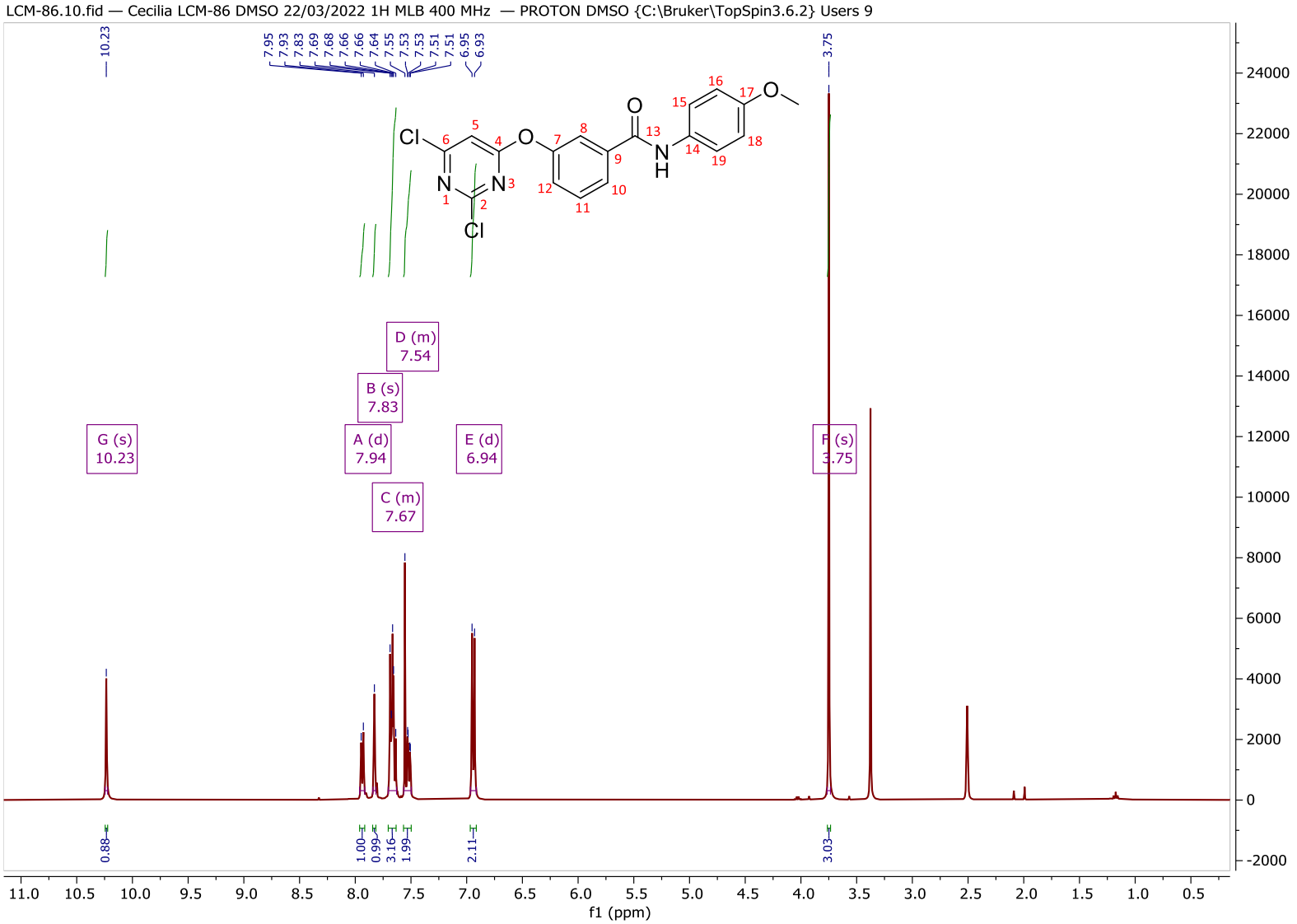


Fig. S19. ^{1}H NMR spectrum of compound **19e**.

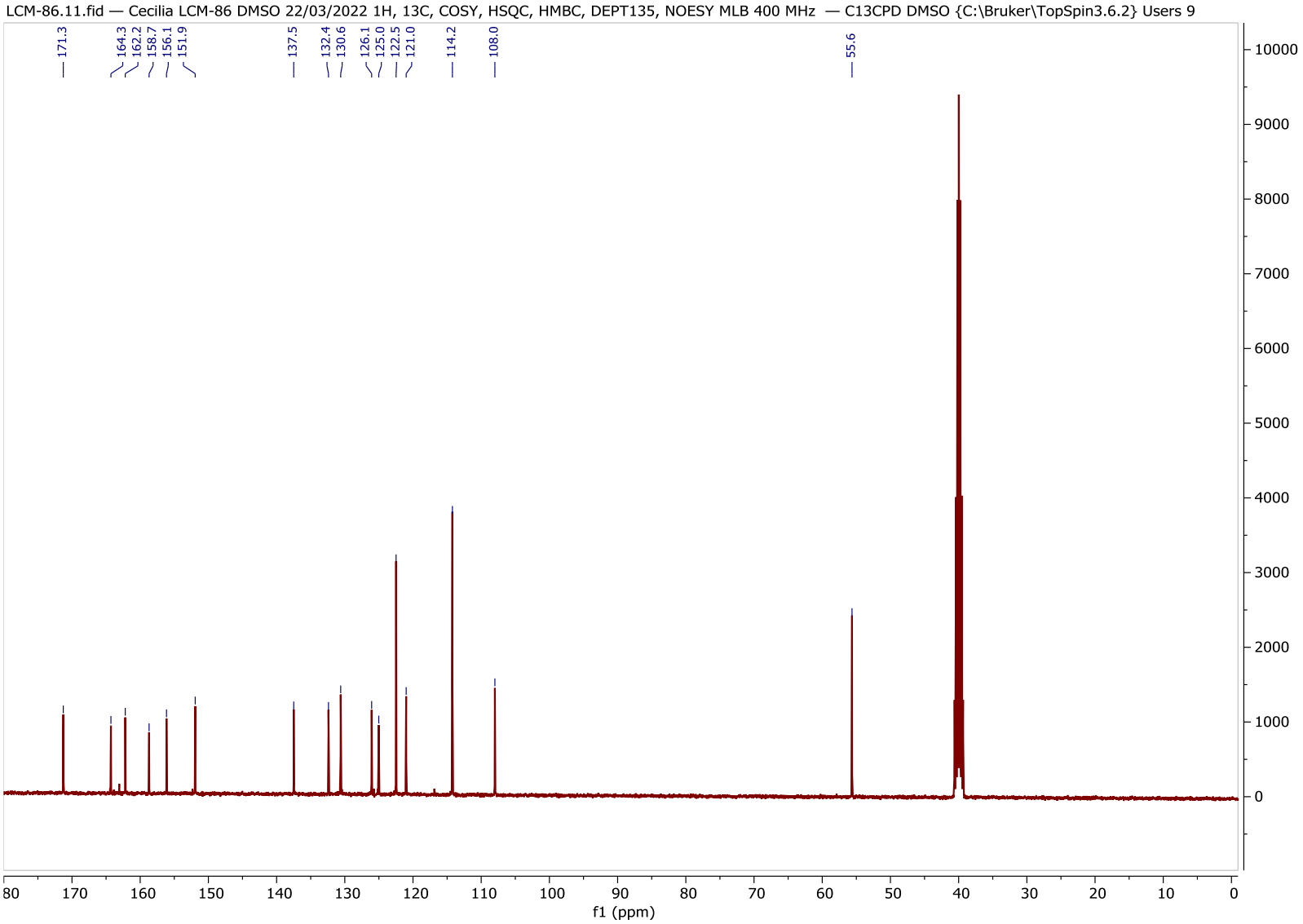


Fig. S20. ^{13}C NMR spectrum of compound **19e**.

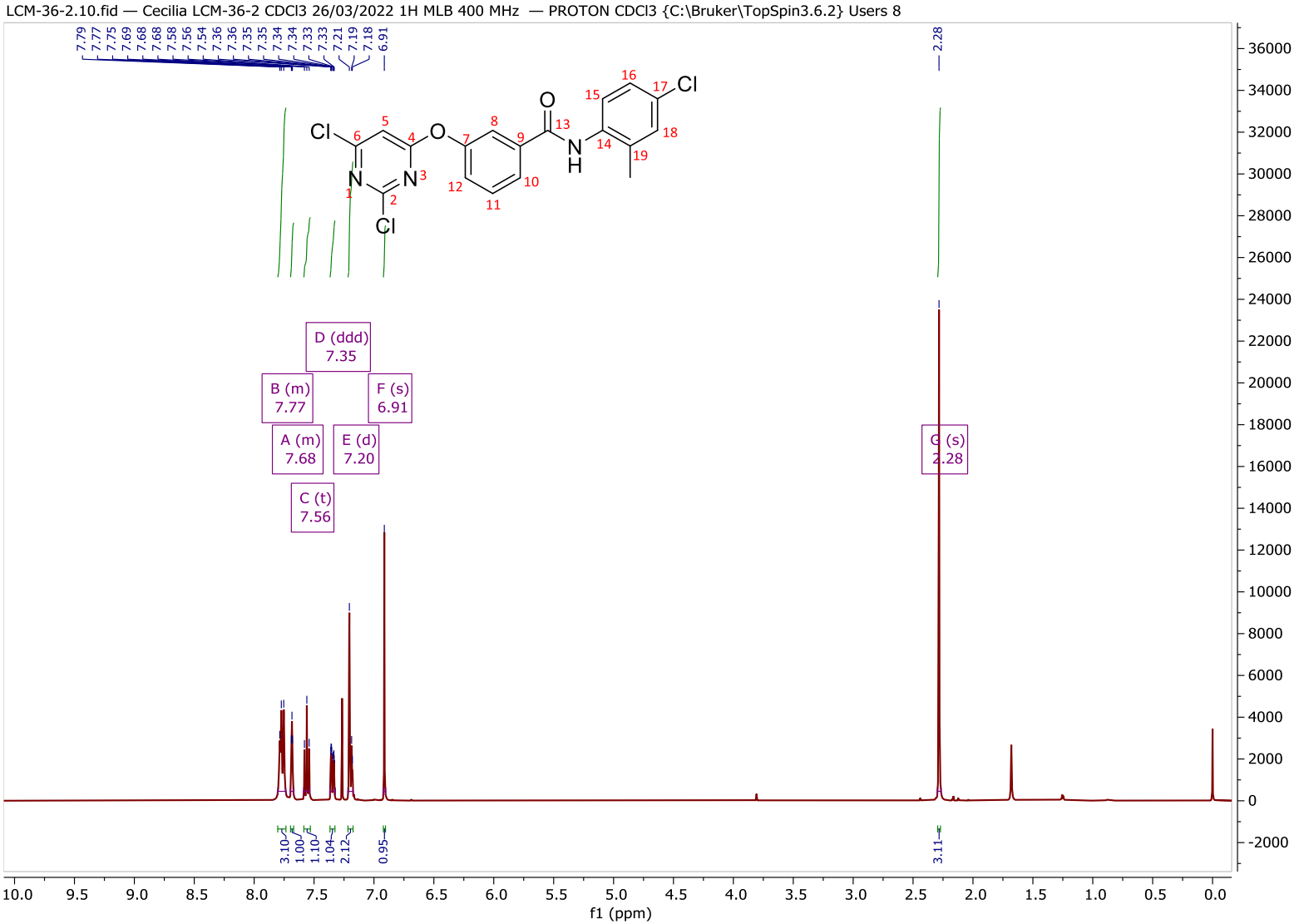


Fig. S21. ^{1}H NMR spectrum of compound **19h**.

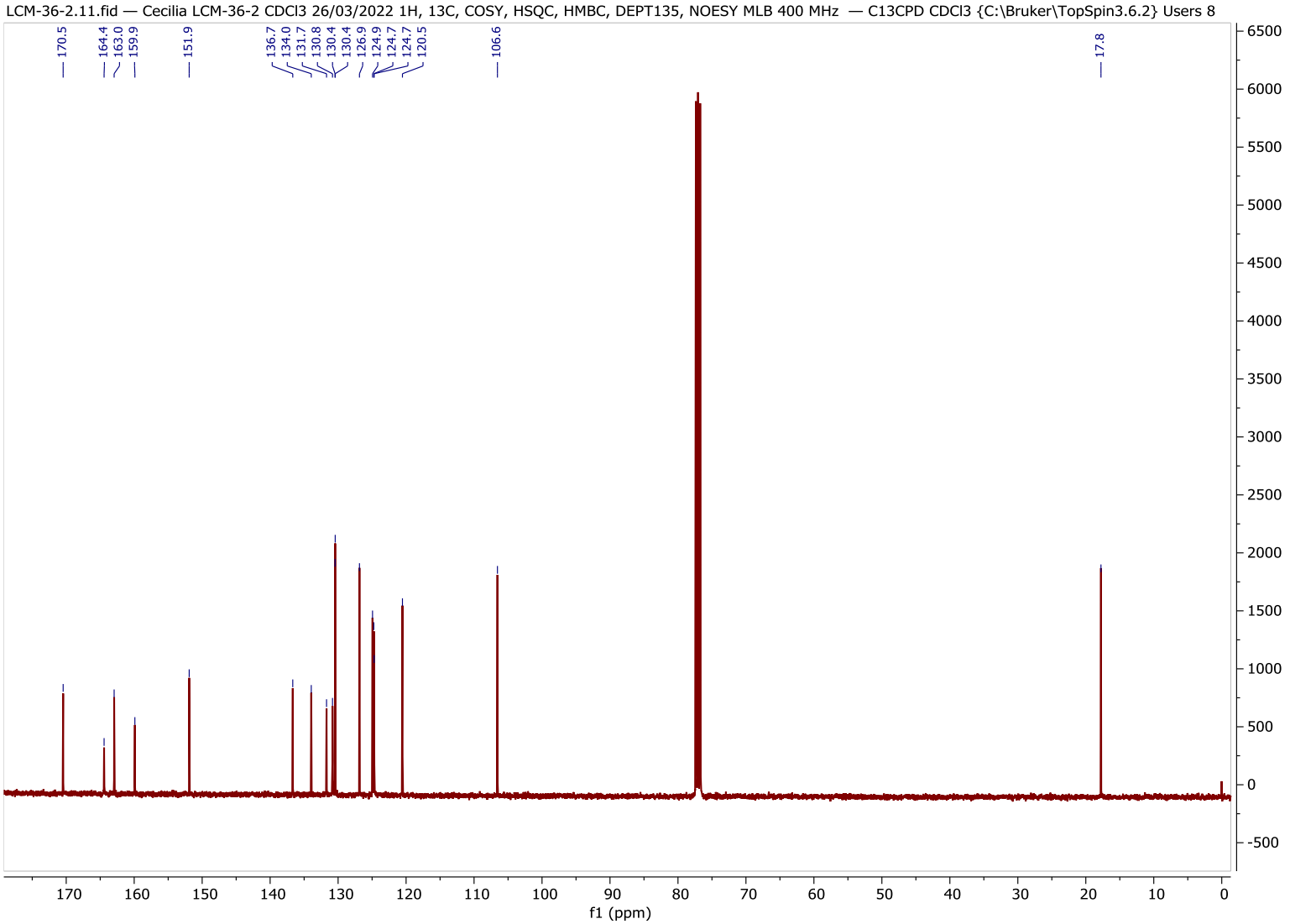


Fig. S22. ^{13}C NMR spectrum of compound **19h**.

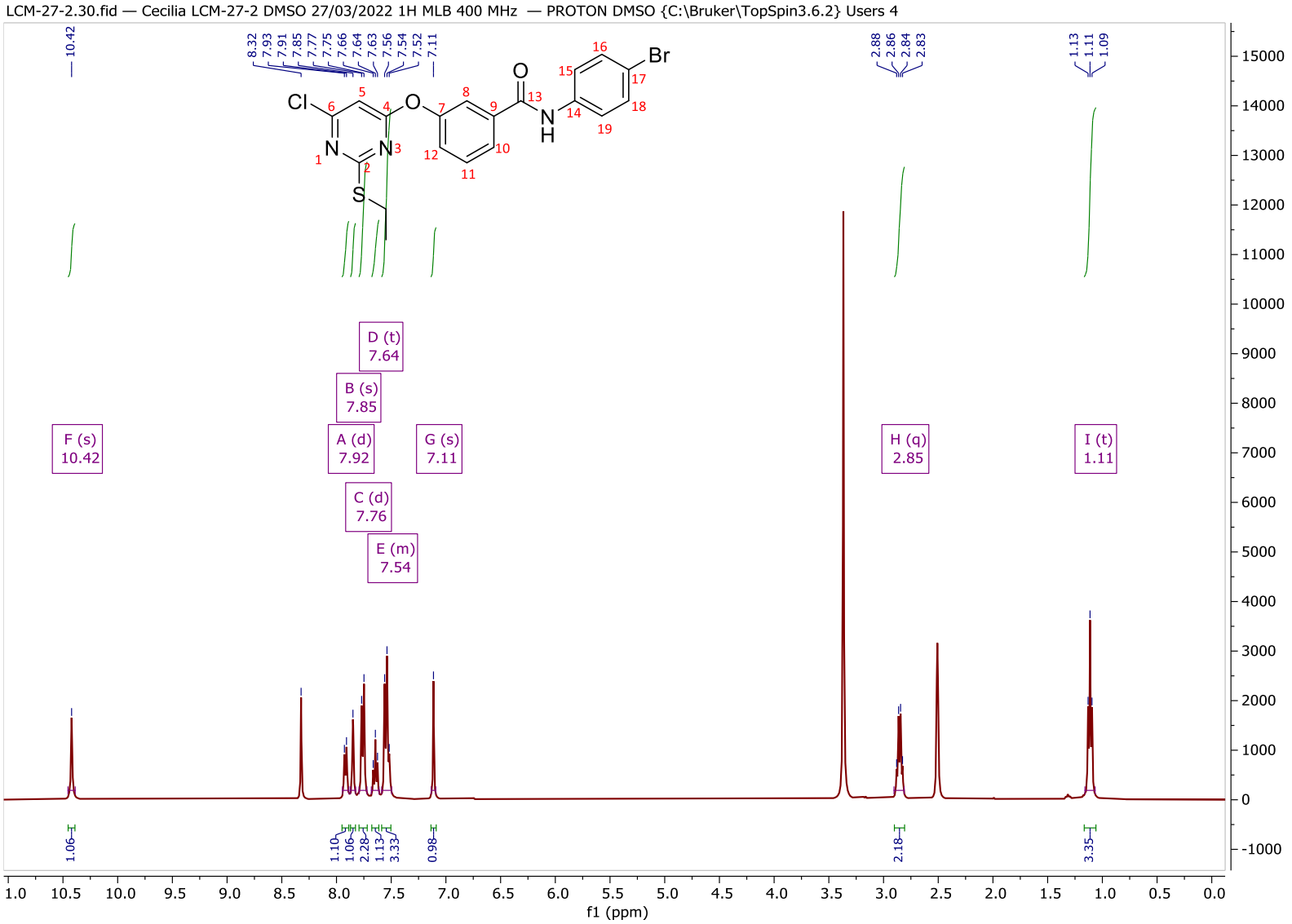


Fig. S23. ^{1}H NMR spectrum of compound **20a**.

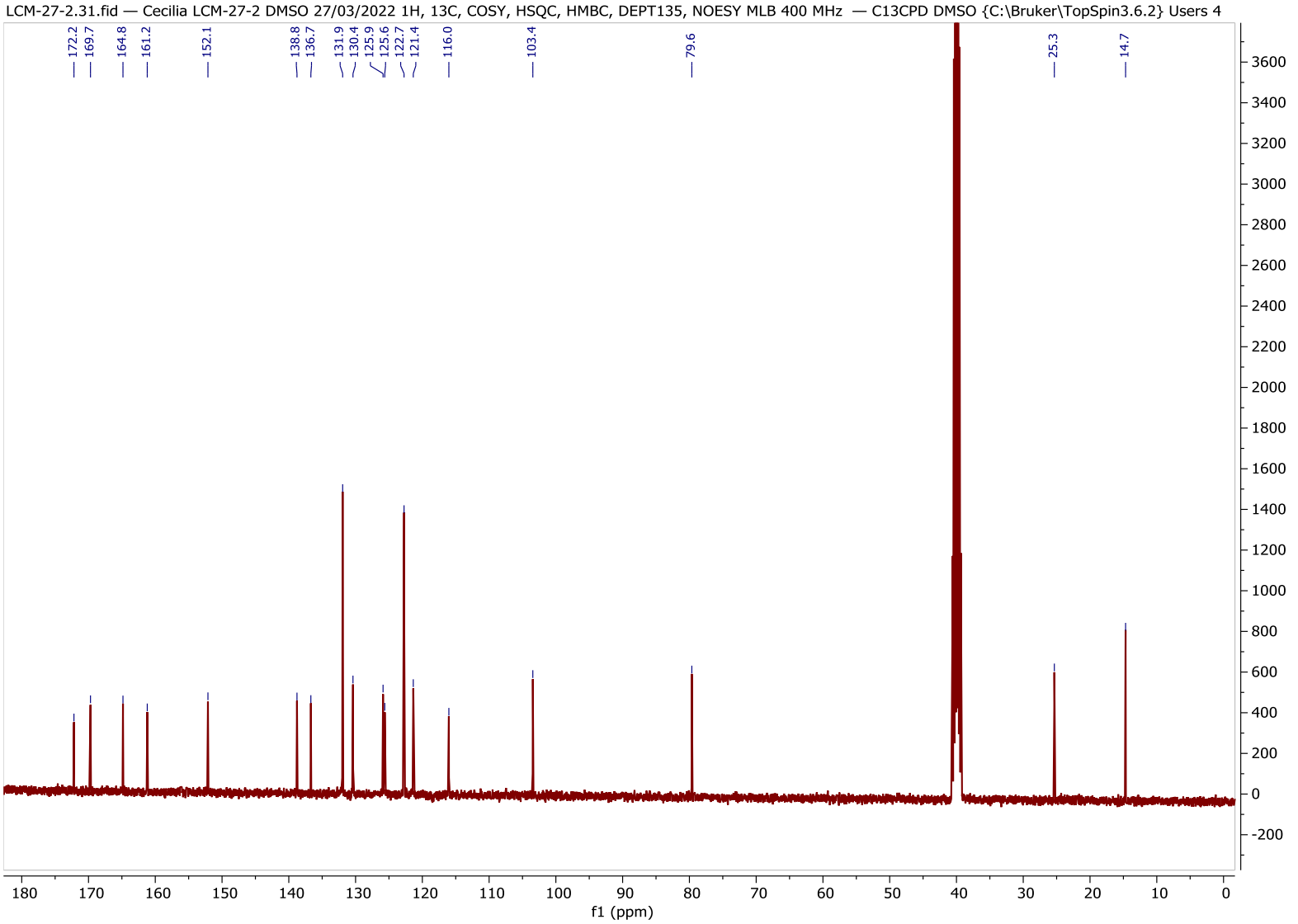


Fig. S24. ^{13}C NMR spectrum of compound **20a**.

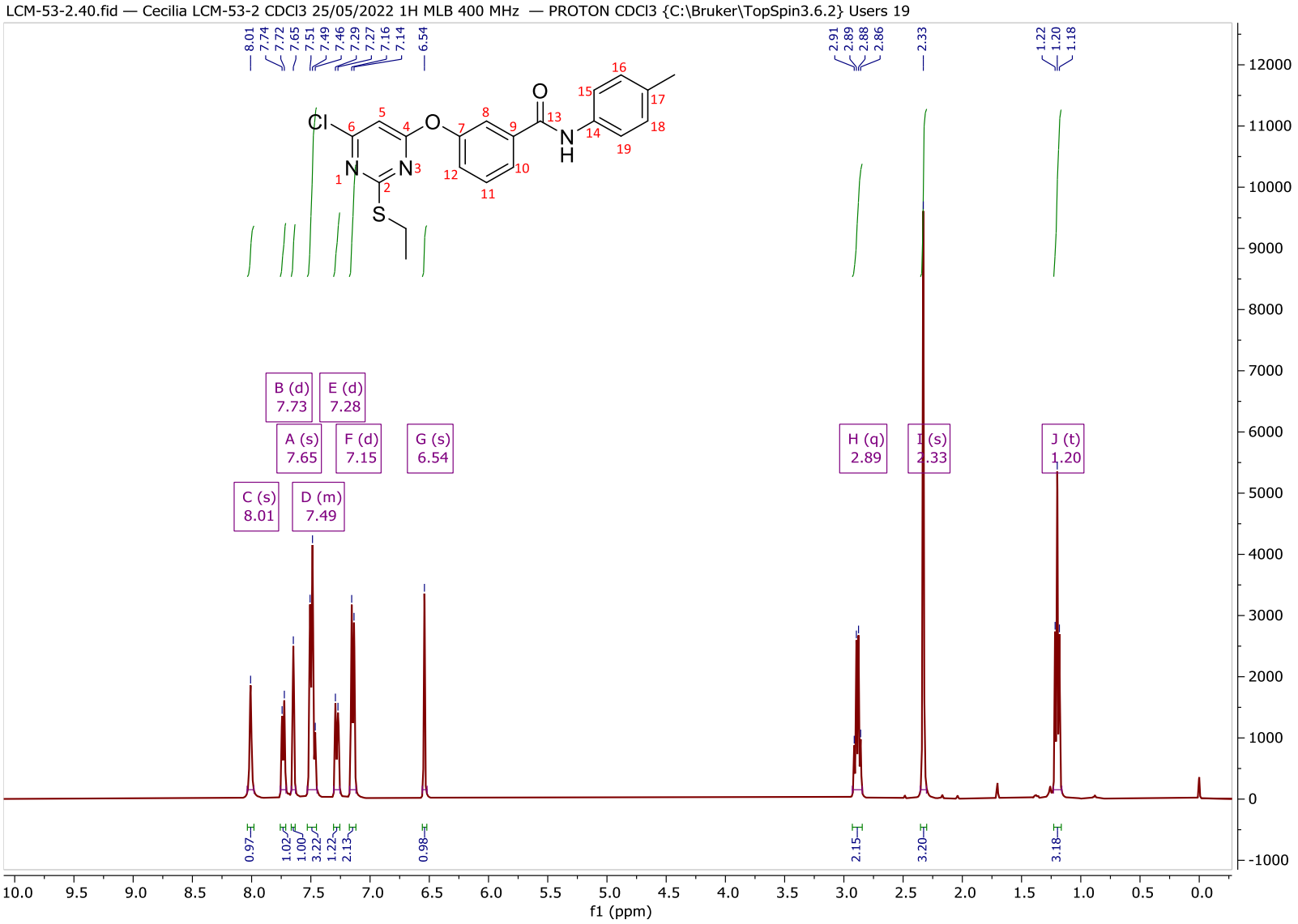


Fig. S25. ^{1}H NMR spectrum of compound **20d**.

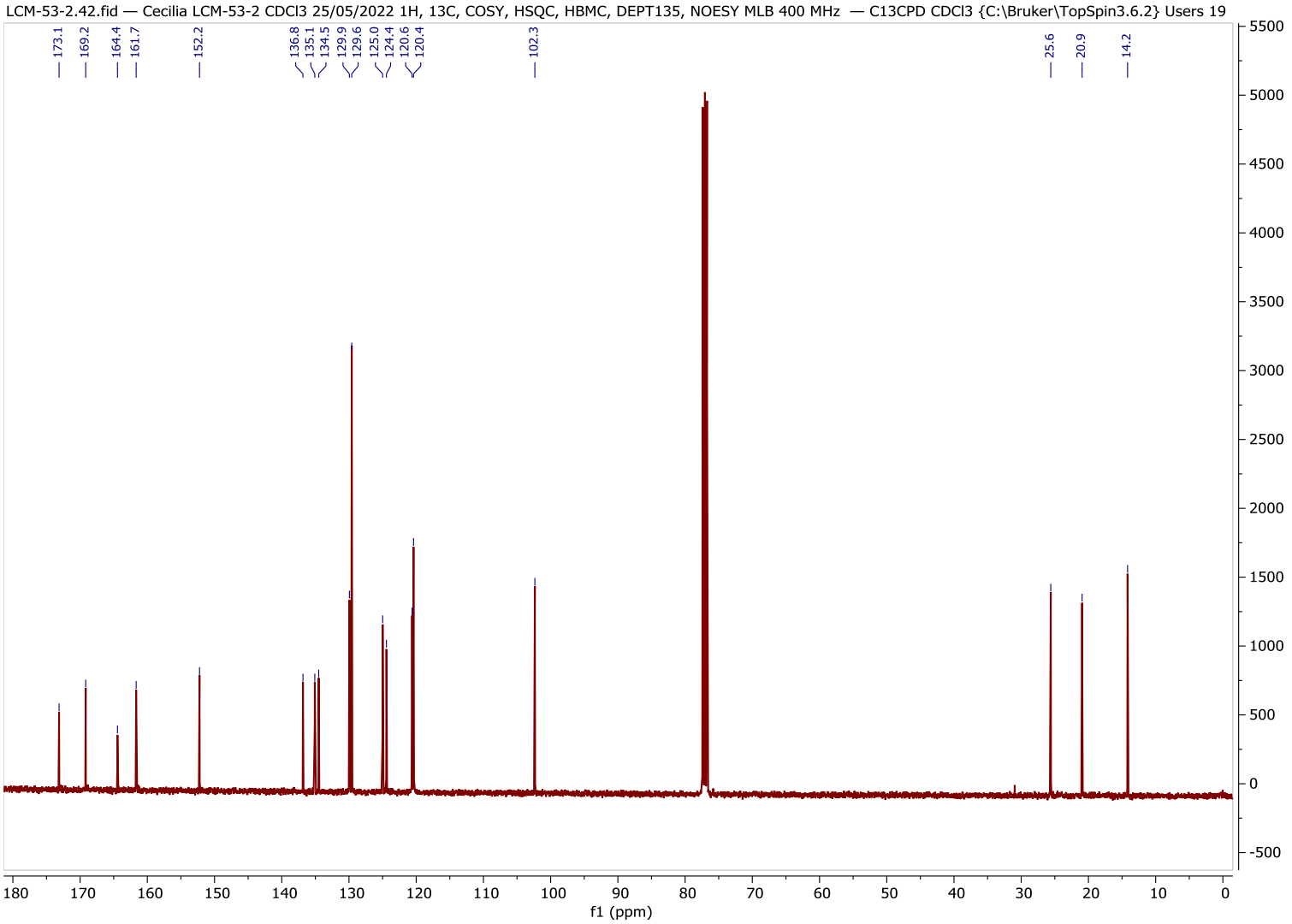


Fig. S26. ^{13}C NMR spectrum of compound **20d**.

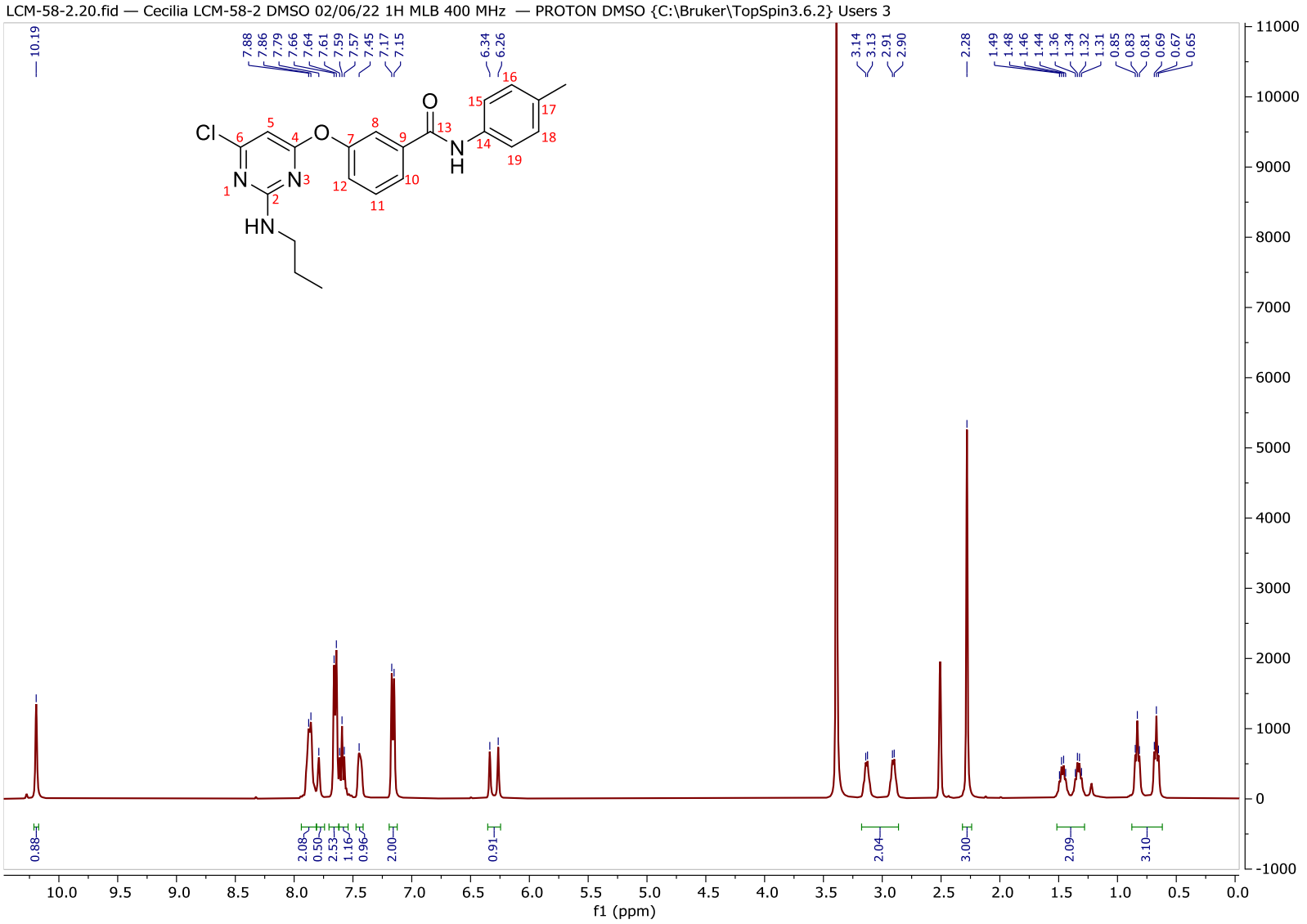


Fig. S27. ^{1}H NMR spectrum of compound **24**.

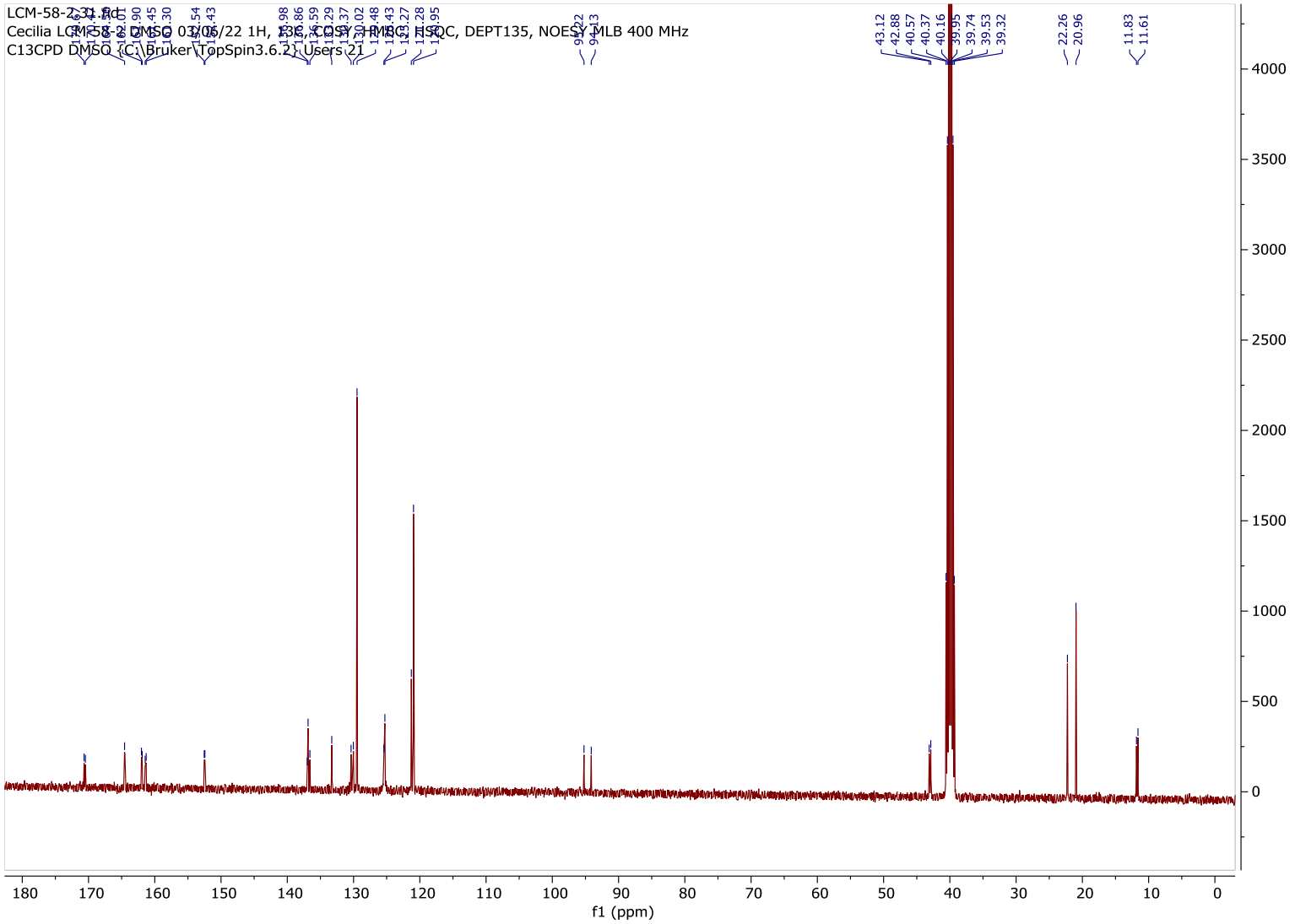


Fig. S28. ^{13}C NMR spectrum of compound **24**.

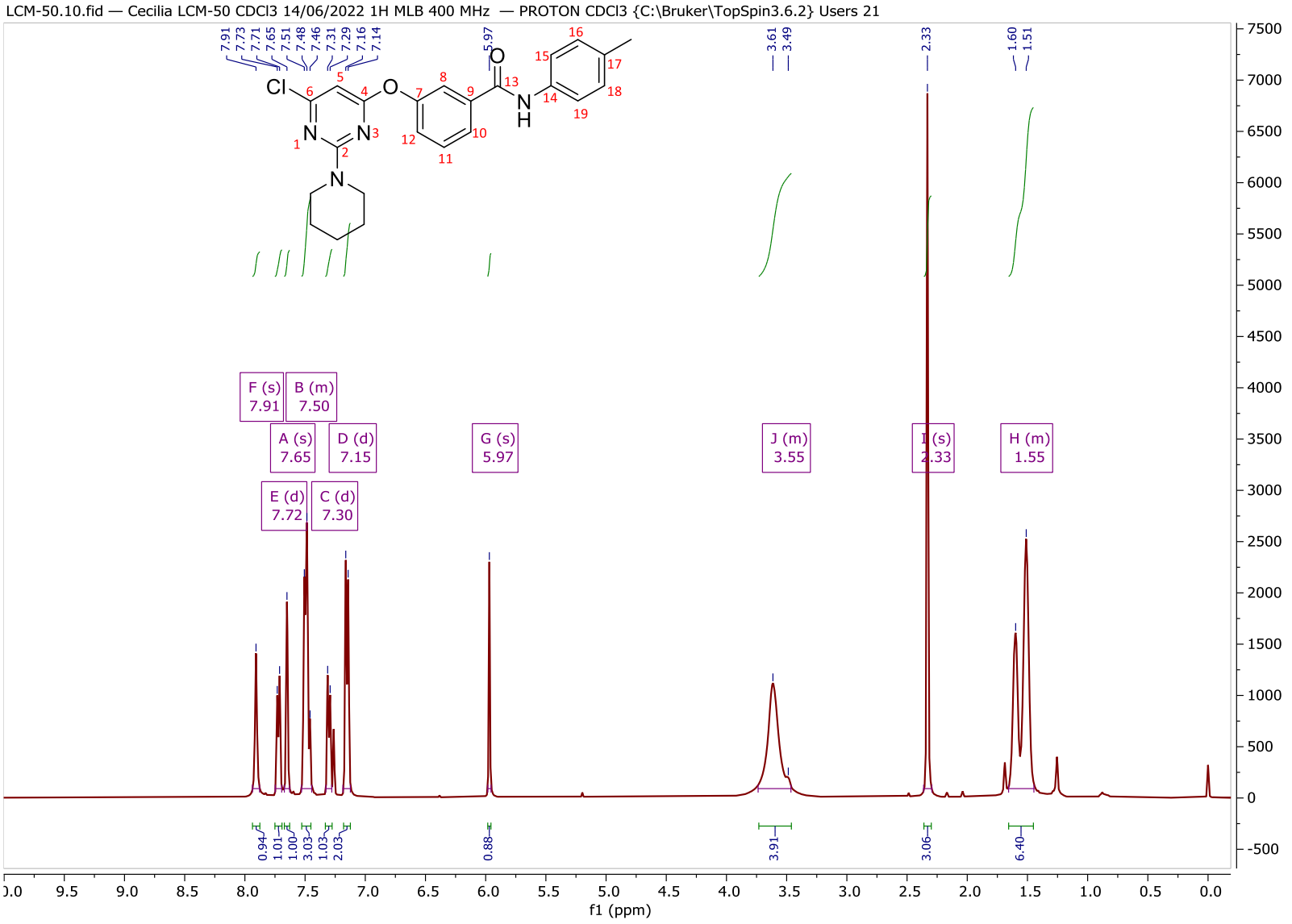


Fig. S29. ^{1}H NMR spectrum of compound **25**.

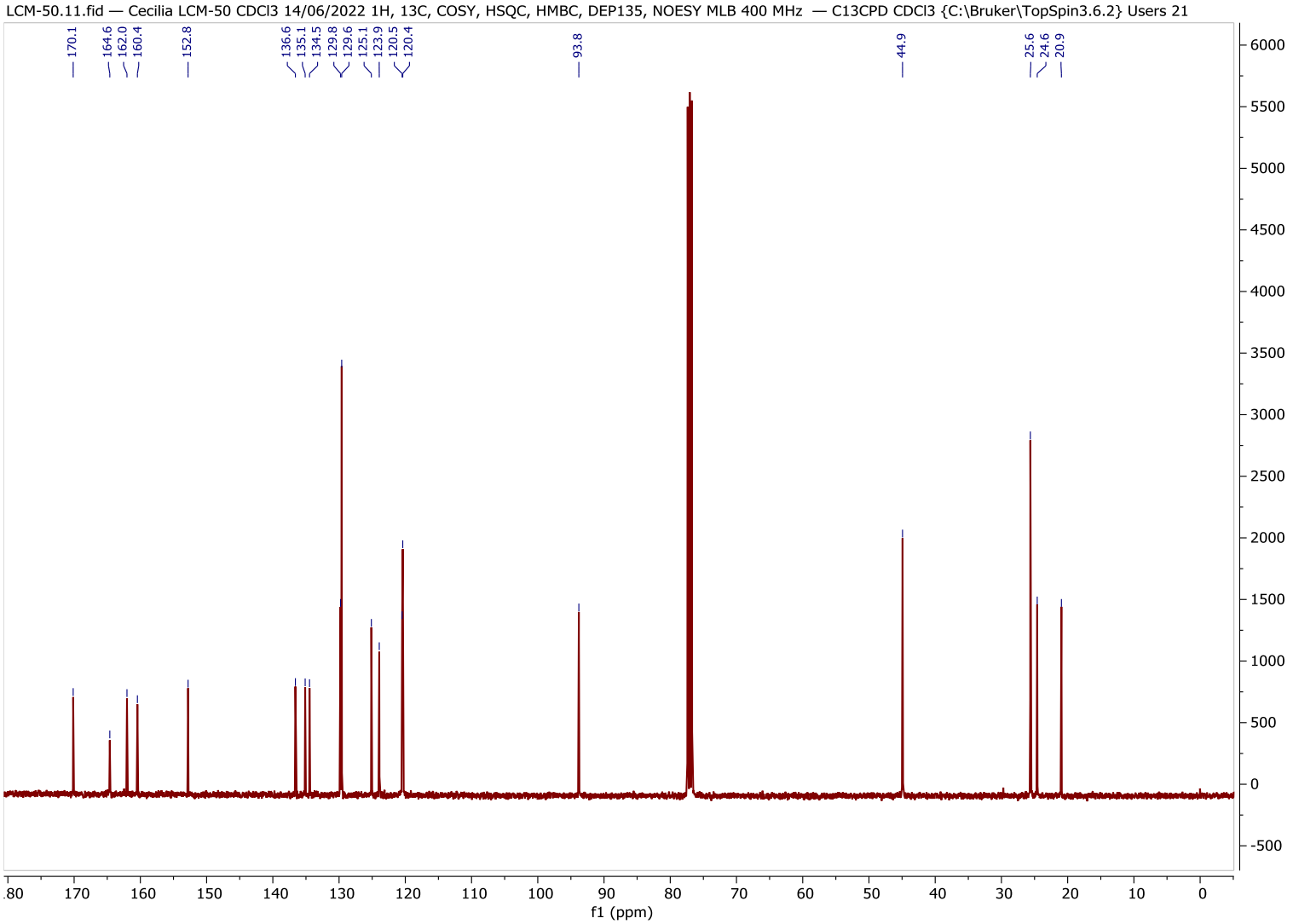


Fig. S30. ^{13}C NMR spectrum of compound **25.**

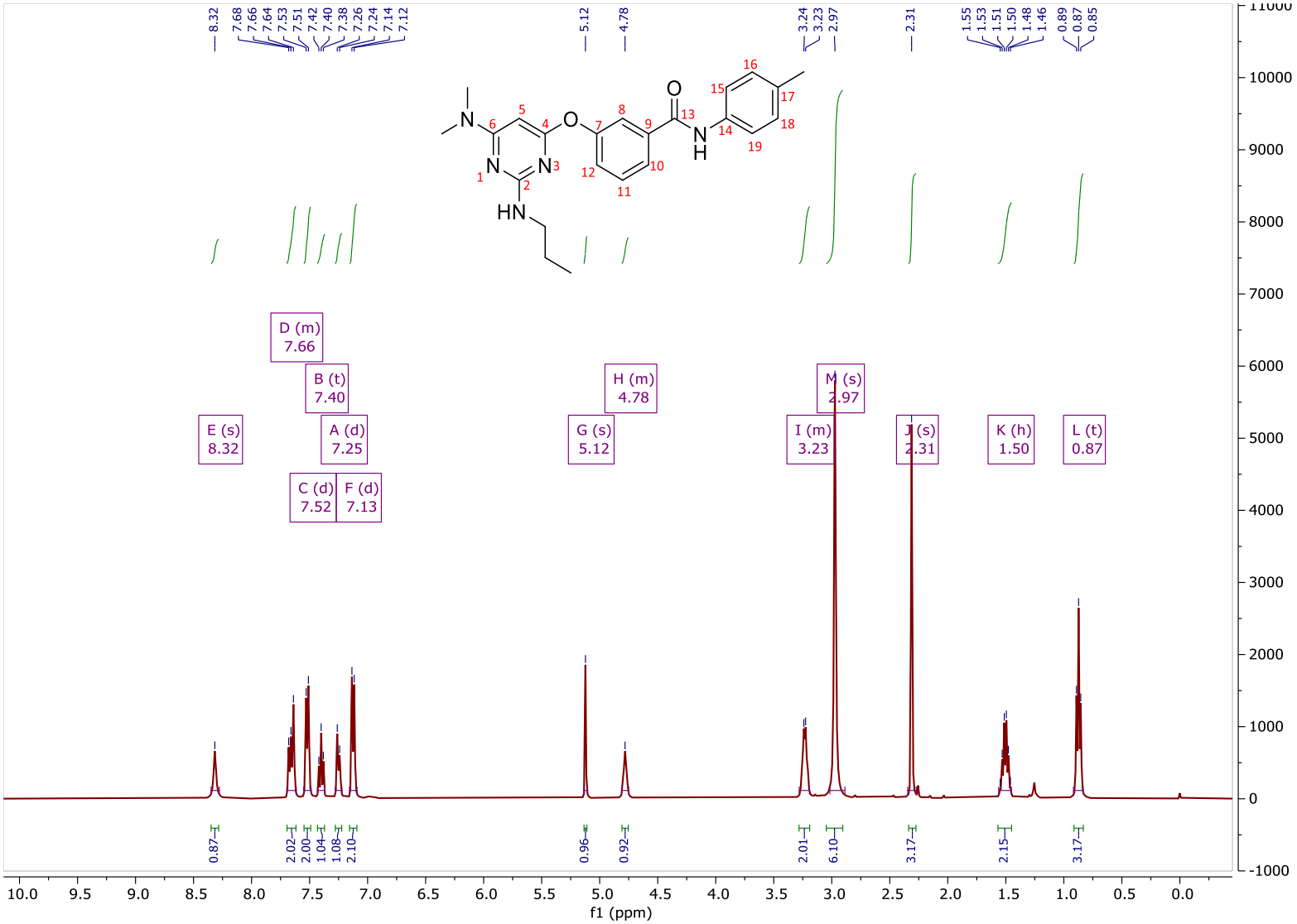


Fig. S31. ^{1}H NMR spectrum of compound **26**.

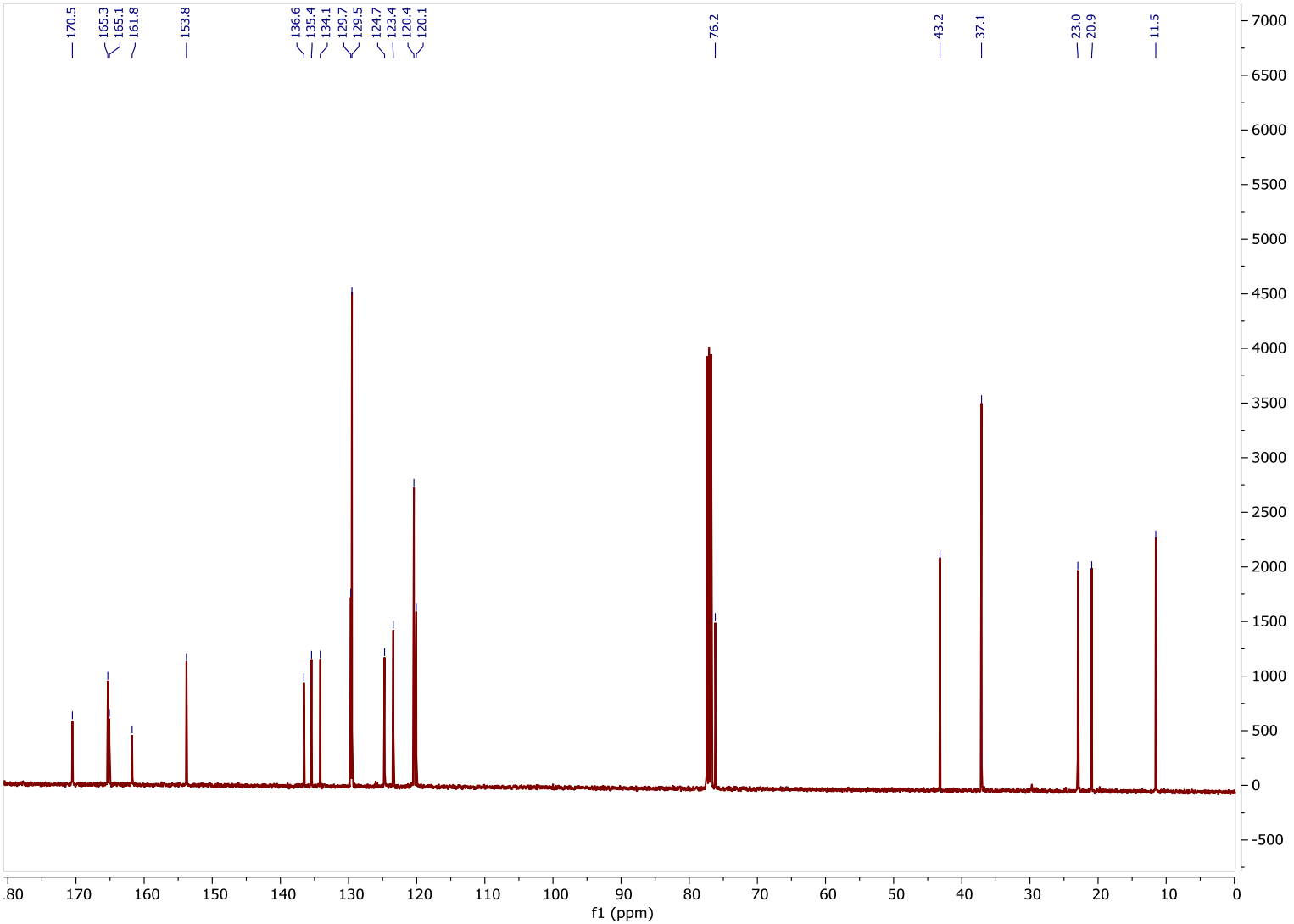


Fig. S32. ^{13}C NMR spectrum of compound **26**.

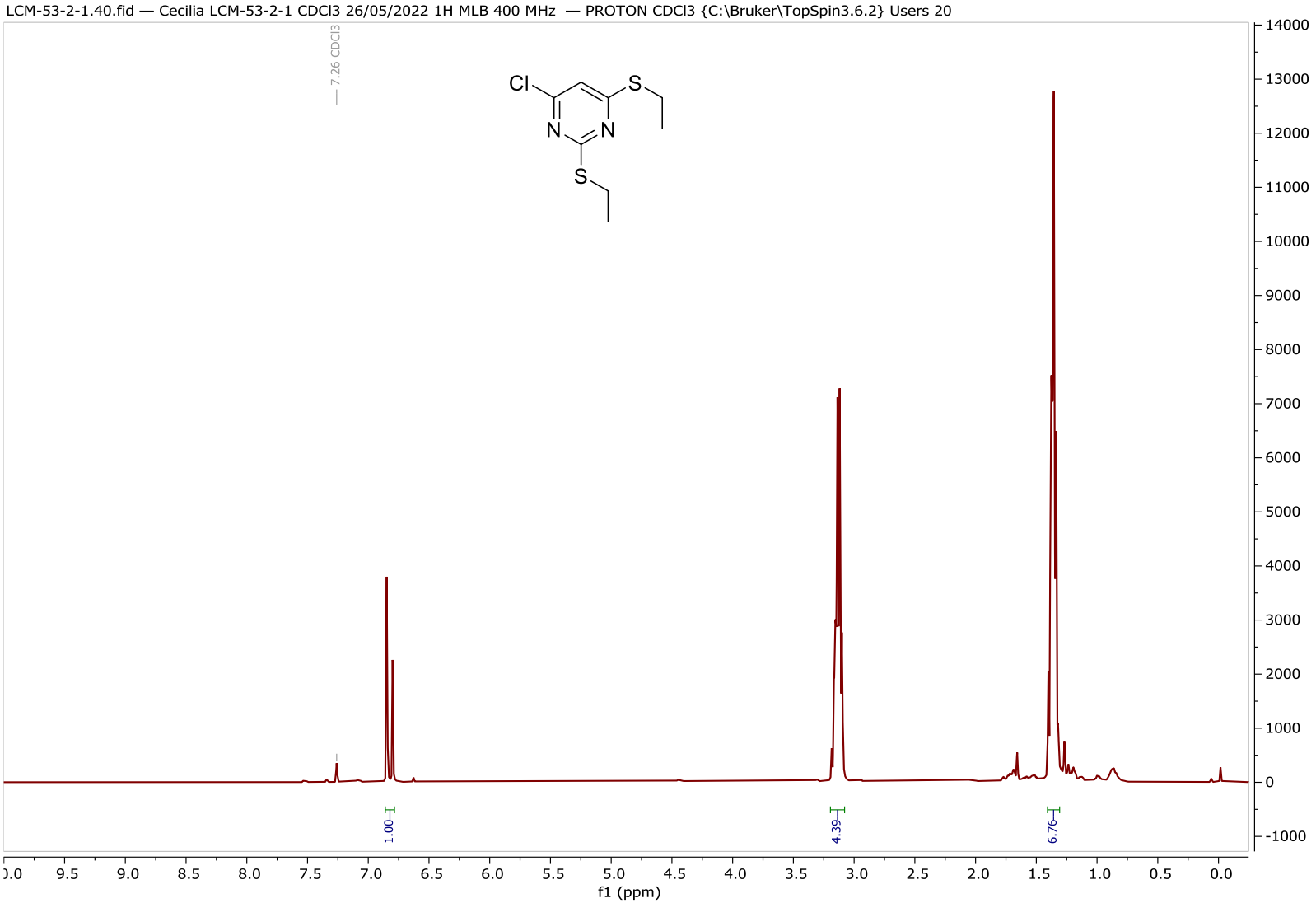


Fig. S33. ^{1}H NMR spectrum of compound **21**.

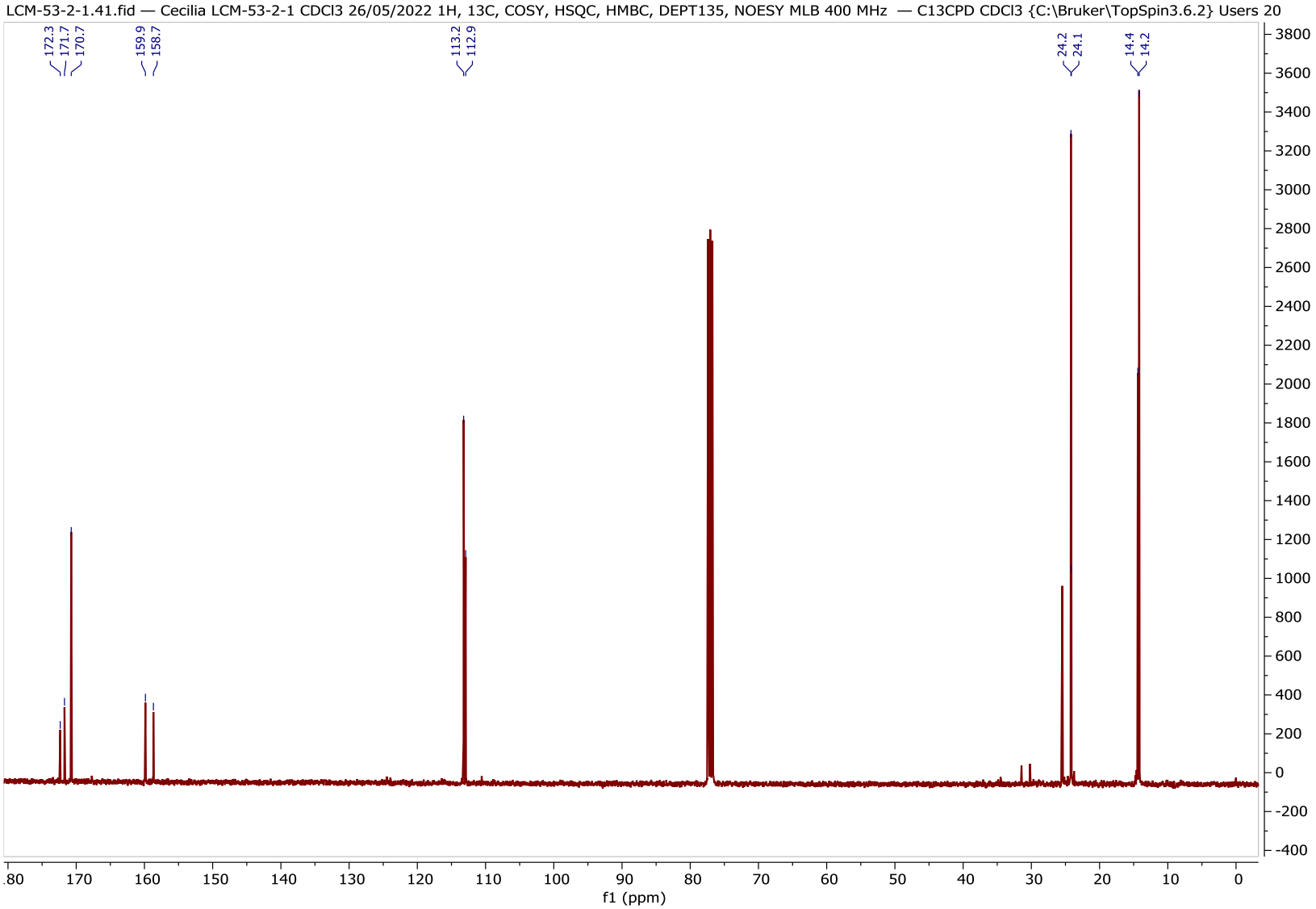


Fig. S34. ^{13}C NMR spectrum of compound **21**.

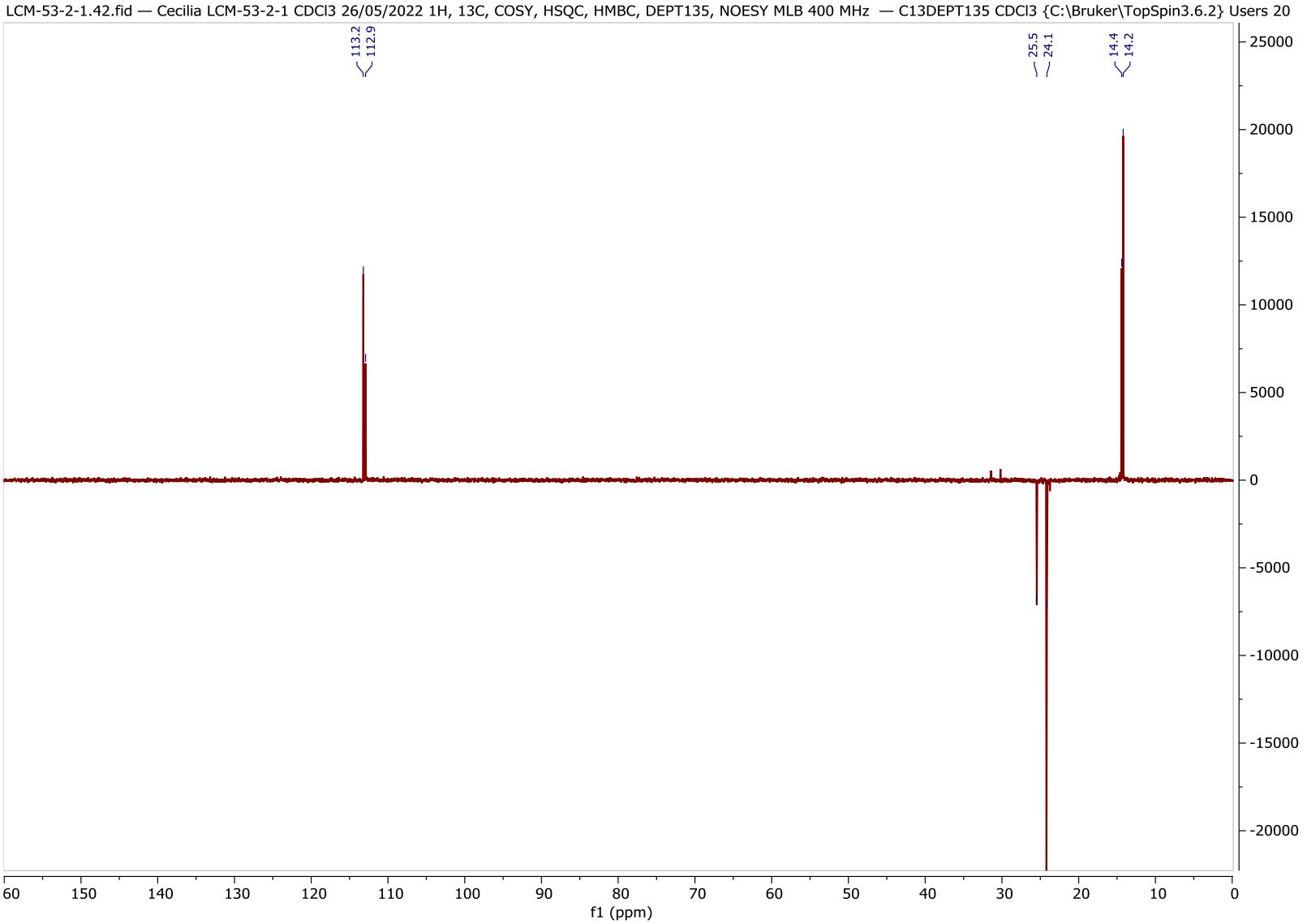


Fig. S35. DEPT spectrum of compound **21**

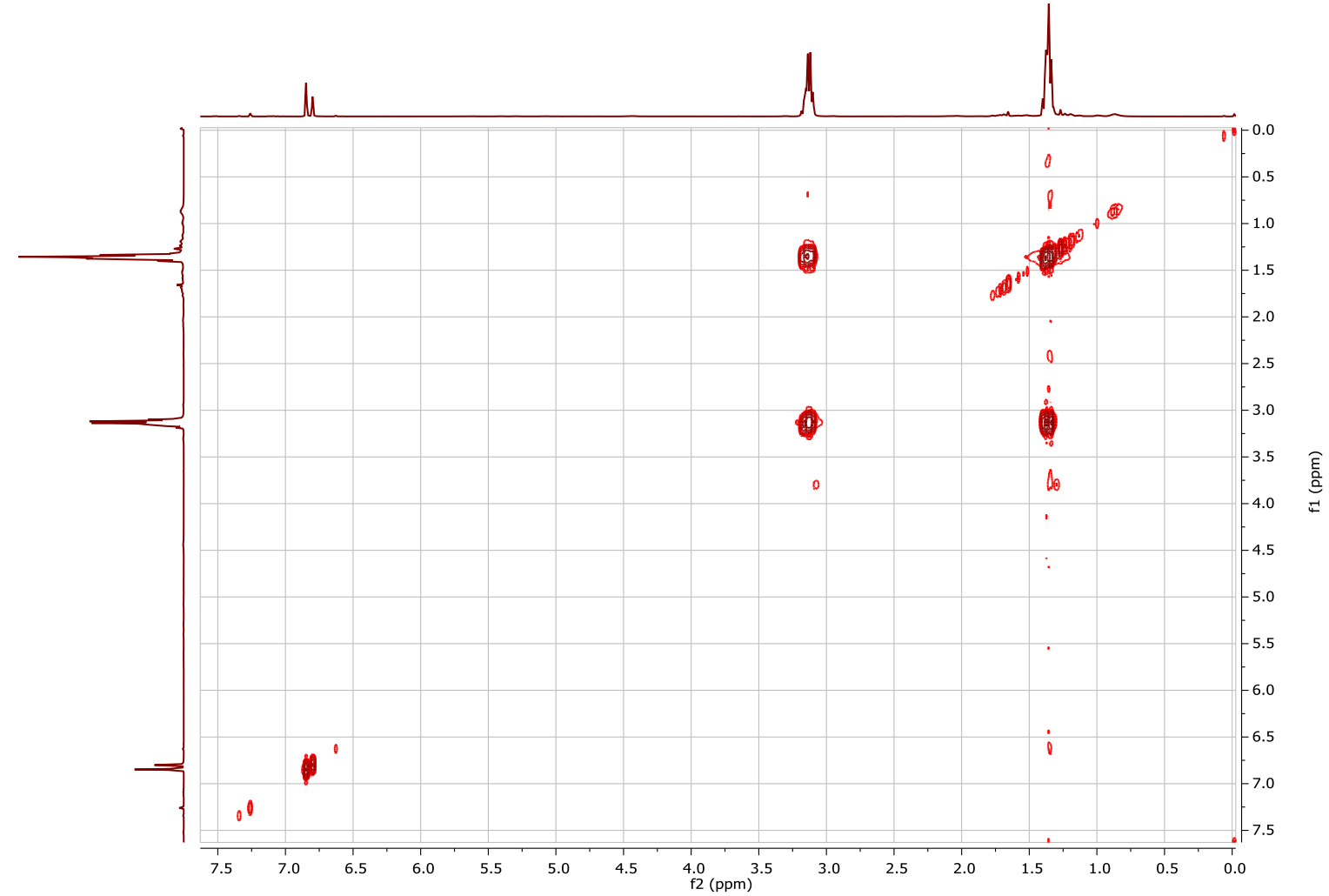


Fig. S36. COSY spectrum of compound **21**

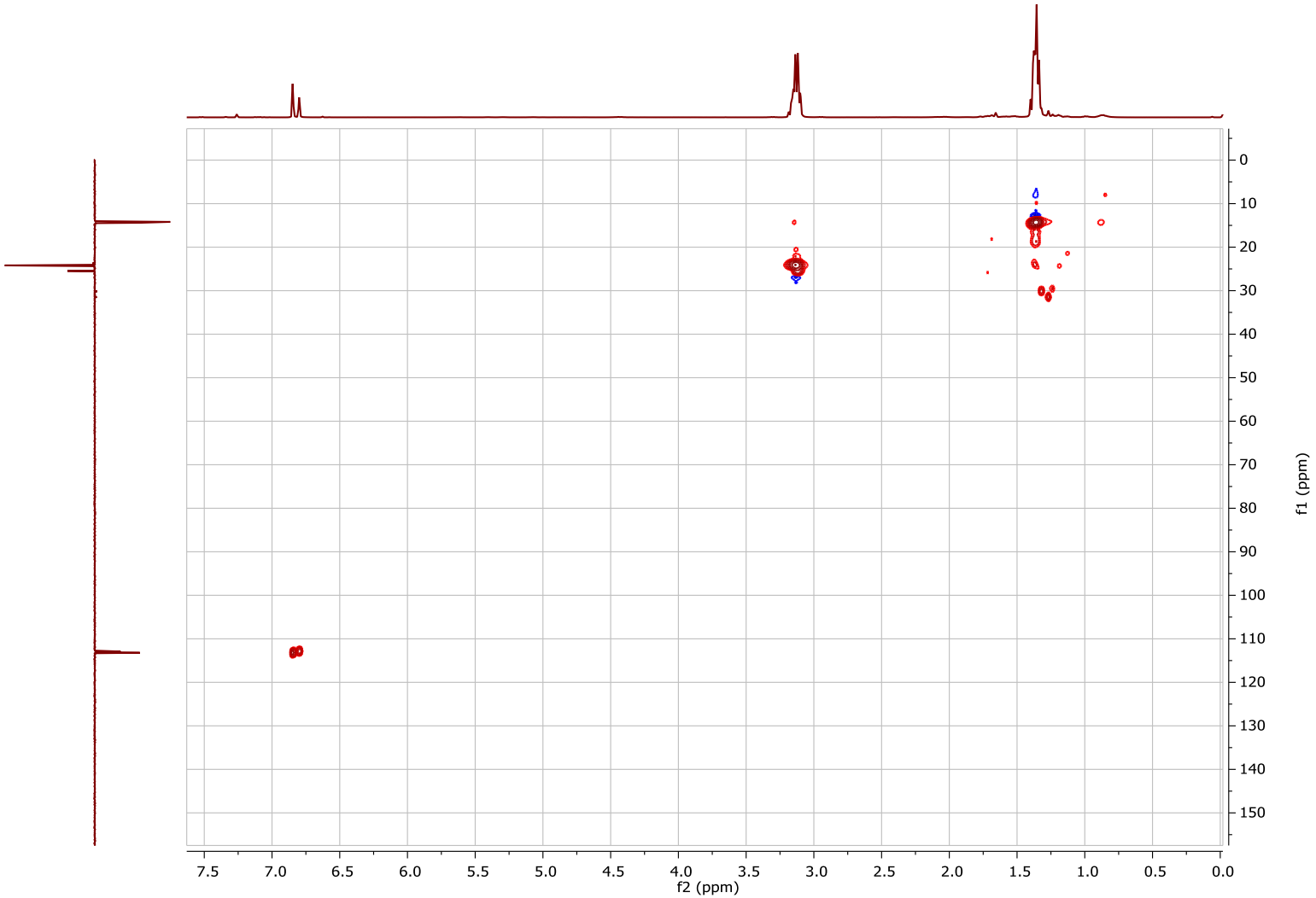


Fig. S37. HSQC spectrum of compound **21**

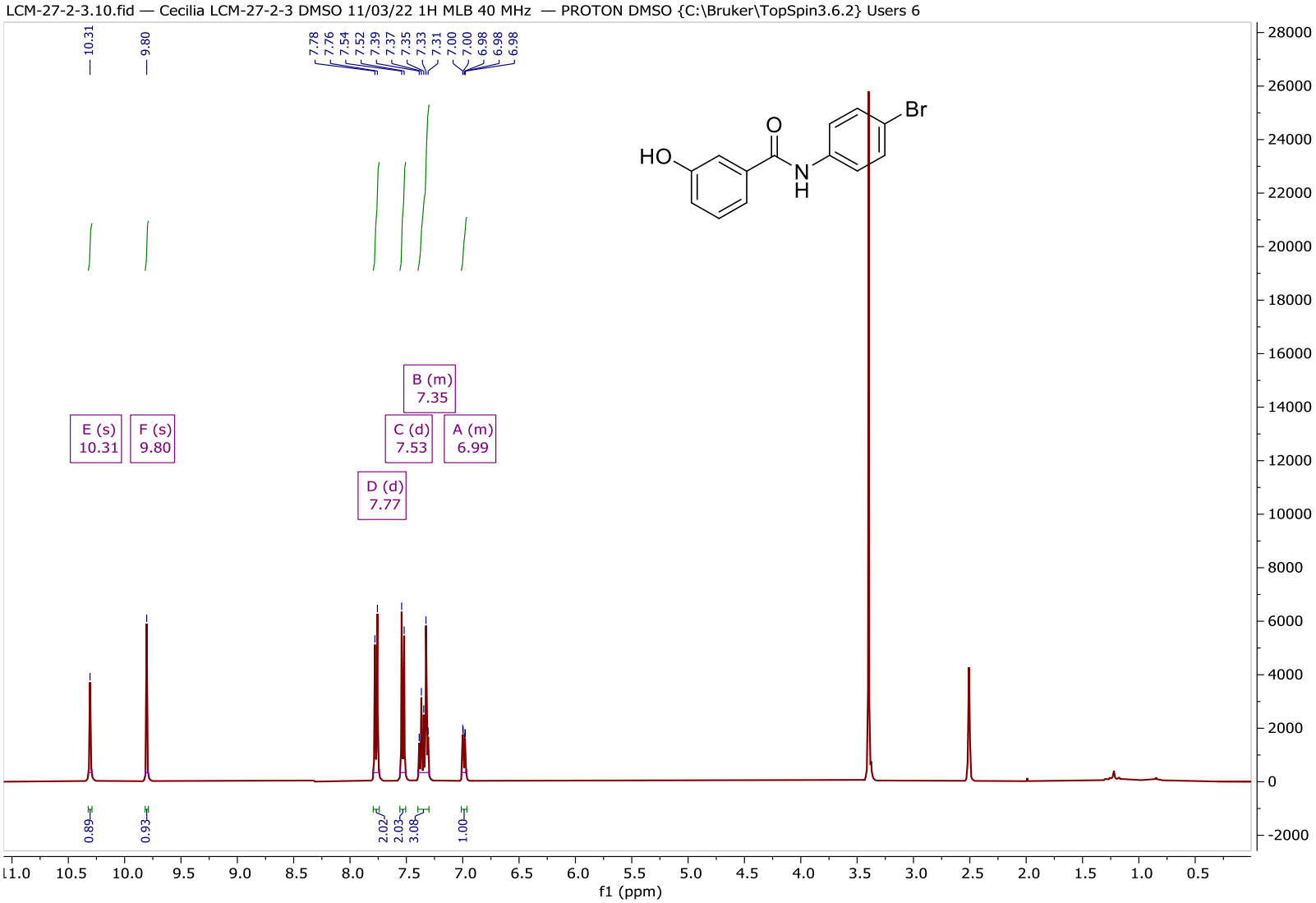


Fig. S38. ^{1}H NMR spectrum of compound **22**.

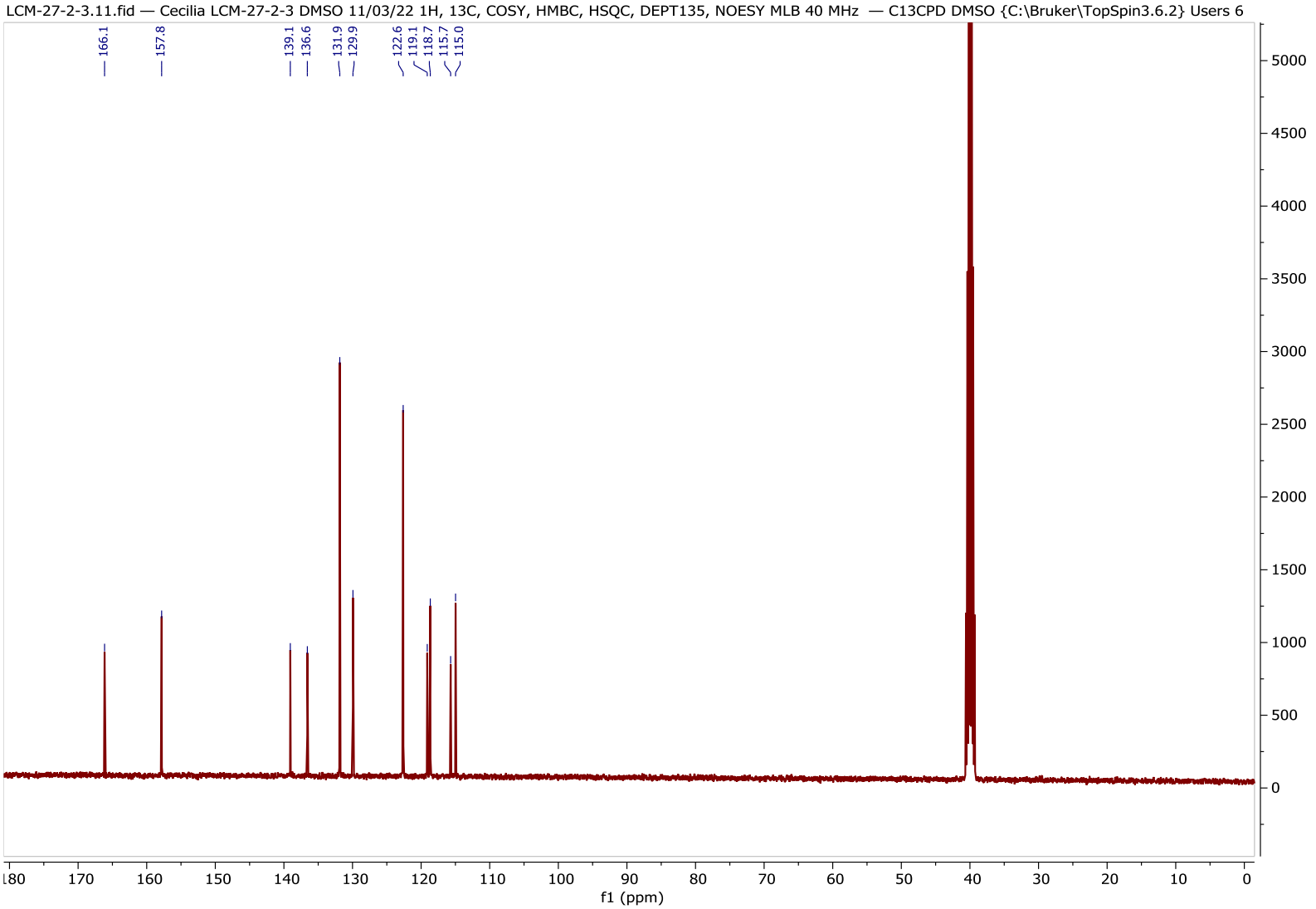


Fig. S39. ^{13}C NMR spectrum of compound **22**.

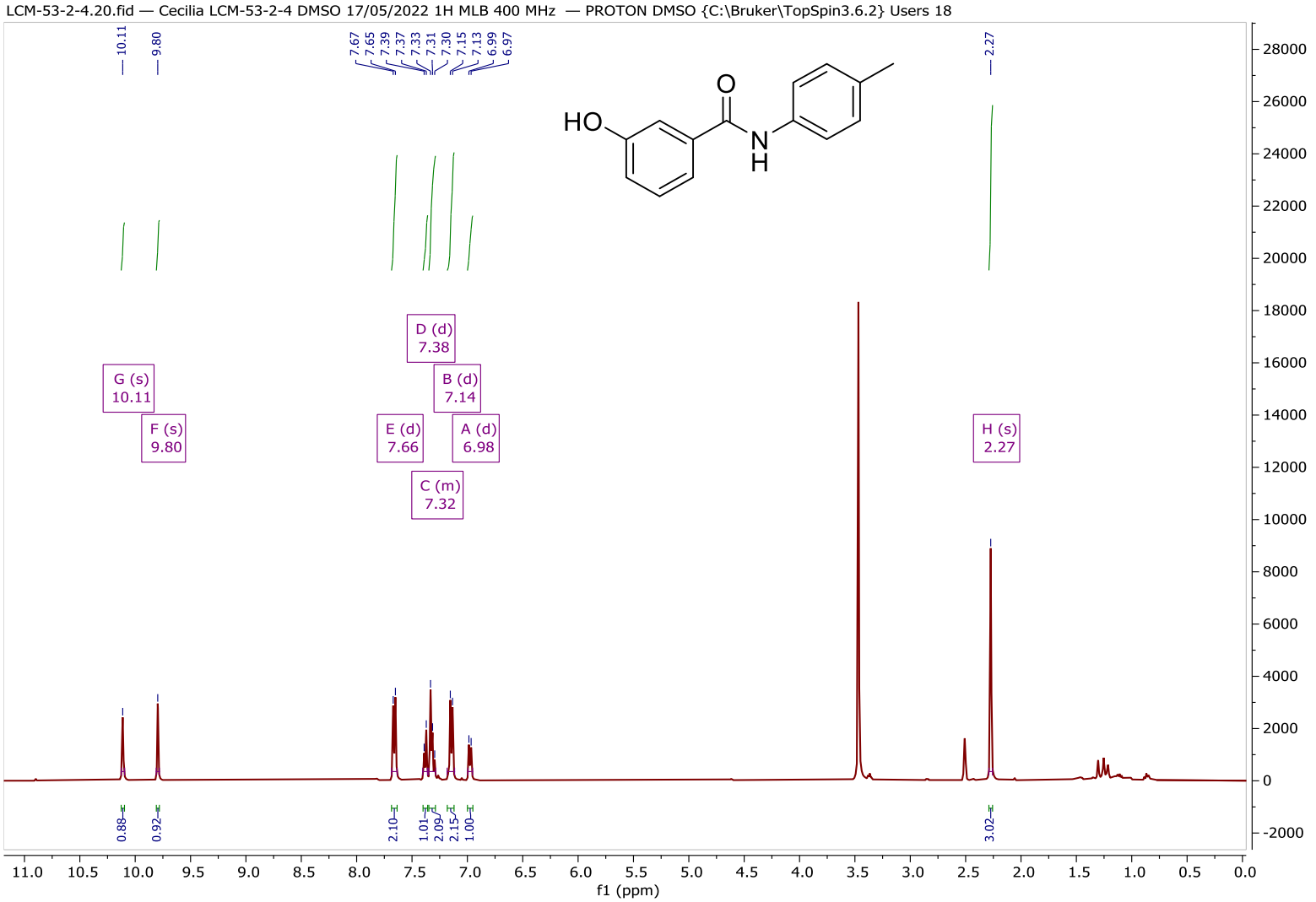


Fig. S40. ^{1}H NMR spectrum of compound **23**.

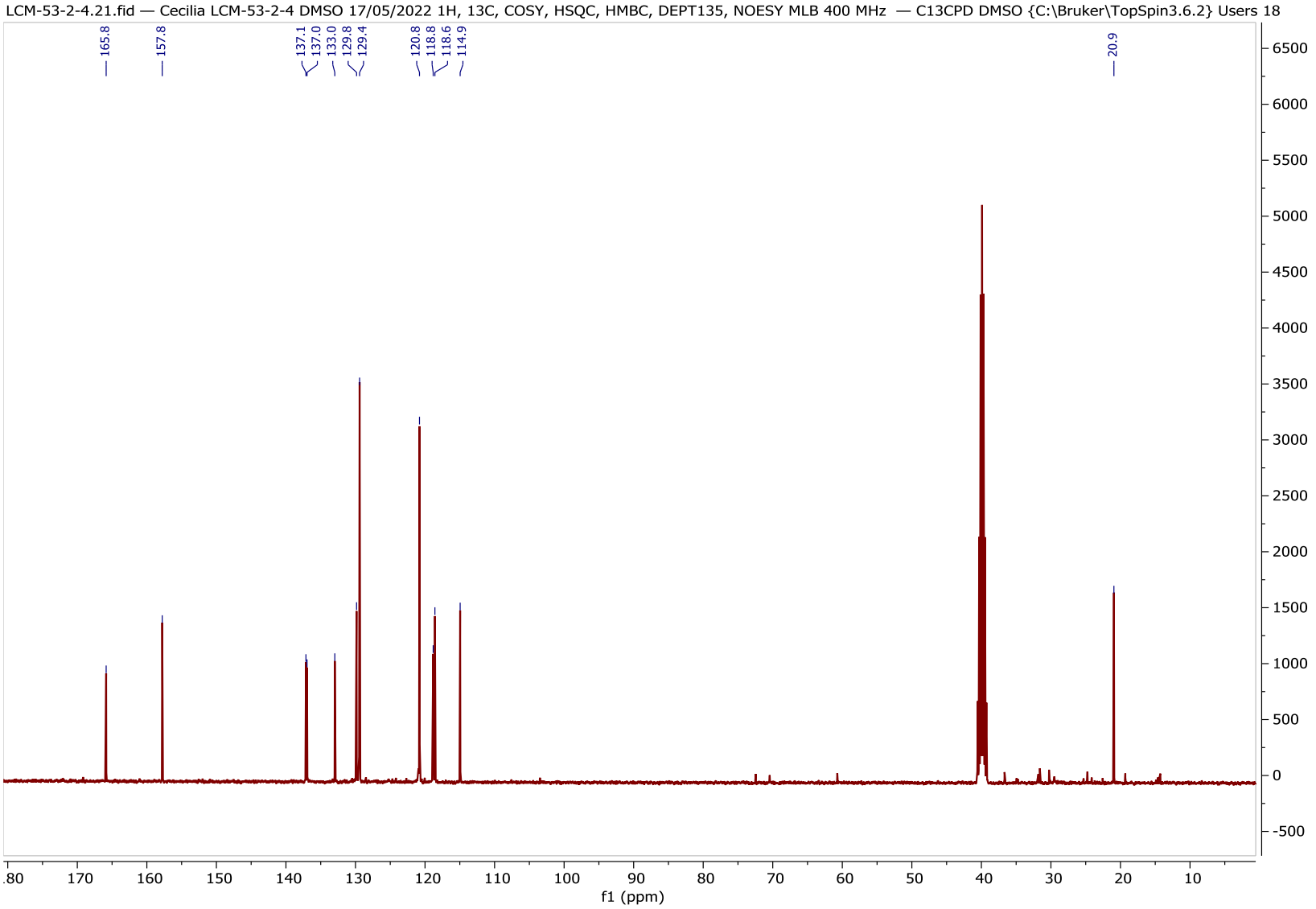


Fig. S41. ^{13}C NMR spectrum of compound **23**.

2. Crystallographic data for *N*-(5-bromopyridin-2-yl)-3-methoxy-N-(3-methoxybenzoyl)benzamide 12e

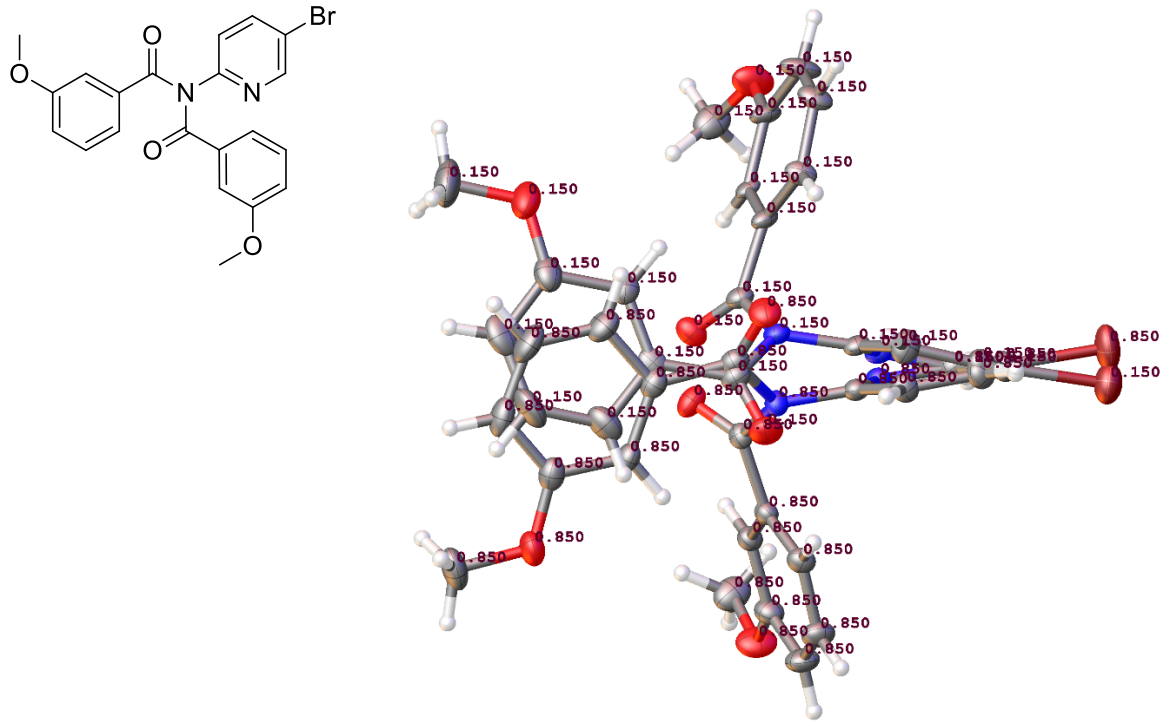


Table 1 Crystal data and structure refinement for compound 12e.

Identification code	22mv_MLB2Likhopotso_LCM_26_2_f
Empirical formula	$C_{21}H_{17}BrN_2O_4$
Formula weight	441.27
Temperature/K	173.00
Crystal system	monoclinic
Space group	*C*2/*c*
a/Å	21.3612(9)
b/Å	7.9993(4)
c/Å	22.9711(8)
α/°	90
β/°	102.2740(10)
γ/°	90
Volume/Å^3	3835.5(3)
Z	8
ρ_{calc}g/cm^3	1.528
μ/mm^{-1}	2.174
F(000)	1792.0
Crystal size/mm^3	0.225 × 0.151 × 0.074
Radiation	MoKα (λ = 0.71073)

2Θ range for data collection/°	5.454 to 56.64
Index ranges	-28 ≤ h ≤ 28, -10 ≤ k ≤ 10, -29 ≤ l ≤ 30
Reflections collected	59065
Independent reflections	4768 [R_{int} = 0.0395, R_{sigma} = 0.0207]
Data/restraints/parameters	4768/461/480
Goodness-of-fit on F^2	1.125
Final R indexes [I>=2σ (I)]	R_1 = 0.0387, wR_2 = 0.0833
Final R indexes [all data]	R_1 = 0.0533, wR_2 = 0.0886
Largest diff. peak/hole / e $Å^{-3}$	0.24/-0.27

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å²×10^3) for compound 12e. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	5157.0(3)	7791.1(9)	505.0(2)	47.69(15)
O1	4891.0(9)	1317(2)	3528.1(9)	40.8(5)
O2	6223.2(8)	6731(2)	3780.4(7)	27.9(4)
O3	7433.6(8)	8779(2)	2922.4(8)	32.5(4)
O4	8040.9(9)	3654(2)	4781.2(8)	37.4(5)
N1	6710.9(9)	6724(2)	2976.0(8)	20.8(4)
N2	5748.1(11)	7148(3)	2310.3(12)	21.9(6)
C1	6324.1(10)	5977(3)	3354.3(9)	20.1(4)
C2	6095.2(10)	4259(3)	3195.4(9)	20.0(4)
C3	5585.5(11)	3657(3)	3434.0(10)	21.8(5)
C4	5382.6(13)	2031(4)	3323.0(11)	27.2(5)
C5	5699.6(14)	979(3)	2991.8(12)	33.6(6)
C6	6198.0(19)	1587(4)	2760(2)	31.6(10)
C7	6400.7(13)	3232(4)	2851.8(12)	24.4(6)
C8	4500(2)	2361(5)	3798.8(16)	39.3(9)
C9	7250.6(19)	7679(5)	3216.1(15)	24.6(8)
C10	7617(2)	7244(4)	3825.3(14)	25.5(7)
C11	7668.1(11)	5620(3)	4039.4(10)	24.9(5)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 12e. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C12	8030.5(11)	5287(3)	4609.4(10)	28.3(5)
C13	8340.1(15)	6583(4)	4956.5(13)	34.6(7)
C14	8284.2(19)	8185(4)	4735.9(14)	39.6(9)
C15	7930.1(12)	8536(4)	4177.2(12)	32.3(6)
C16	8396.6(17)	3244(4)	5364.3(12)	46.3(8)
C17	6380.7(10)	6987(3)	2374.3(9)	20.3(4)
C18	6698.9(12)	7044(4)	1906.4(10)	25.5(5)
C19	6338(2)	7318(15)	1339.9(18)	28.3(15)
C20	5681(3)	7460(40)	1270(2)	26.9(4)
C21	5401(3)	7390(30)	1758(2)	25.2(4)
Br1A	5159.8(17)	7001(5)	502.7(14)	44.0(8)
O1A	4867(5)	13669(13)	3484(5)	42(3)
O2A	6229(4)	8269(12)	3770(4)	28(2)
O3A	7424(5)	6176(12)	2933(4)	36(2)
O4A	8062(6)	11327(12)	4781(4)	46(3)
N1A	6705(4)	8250(12)	2965(3)	20.7(17)
N2A	5737(5)	7766(18)	2300(7)	22(3)
C1A	6324(5)	9022(11)	3342(4)	21.3(19)
C2A	6094(6)	10732(11)	3170(5)	21(2)
C3A	5586(5)	11366(13)	3404(6)	18(2)
C4A	5369(6)	12976(14)	3289(6)	25(2)
C5A	5660(7)	14012(15)	2933(7)	36(3)
C6A	6167(7)	13402(19)	2717(8)	29(3)
C7A	6374(10)	11761(19)	2808(11)	24(4)
C8A	4581(15)	12760(40)	3887(10)	39.3(9)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 12e. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C9A	7259(11)	7350(40)	3209(8)	24.6(8)
C10A	7596(12)	7701(16)	3836(6)	22(3)
C11A	7681(6)	9332(15)	4041(5)	28(2)
C12A	8033(7)	9695(12)	4612(5)	34(3)
C13A	8329(10)	8372(17)	4952(7)	40(4)
C14A	8259(11)	6770(20)	4738(7)	46(4)
C15A	7895(7)	6387(18)	4185(6)	38(3)
C16A	8405(11)	11740(30)	5369(5)	62(6)
C17A	6369(5)	7961(18)	2366(4)	20(2)
C18A	6682(6)	7950(20)	1895(5)	31(3)
C19A	6326(14)	7540(100)	1338(9)	28.3(15)
C20A	5669(15)	7400(200)	1267(11)	26.9(4)
C21A	5394(17)	7370(150)	1758(10)	25.2(4)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 12e. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	41.31(19)	73.9(4)	23.66(15)	4.2(3)	-2.42(12)	9.3(3)
O1	42.6(11)	30.9(10)	54.2(12)	2.4(9)	22.4(9)	-10.5(9)
O2	25.7(8)	32.8(10)	26.5(8)	-10.1(7)	8.1(7)	-6.6(7)
O3	32.0(9)	29.3(10)	36.0(9)	2.3(8)	6.9(7)	-10.3(8)
O4	43.5(11)	41.5(11)	23.5(9)	3.9(8)	-1.5(8)	6.3(9)
N1	19.8(9)	22.4(10)	19.9(9)	0.8(7)	3.5(7)	-3.4(7)
N2	20.0(9)	21.0(16)	24.5(9)	-1.1(11)	3.9(7)	0.2(9)
C1	16.2(10)	23.2(11)	20.2(10)	1.1(8)	2.5(8)	-1.0(8)
C2	19.9(11)	18.8(11)	20.2(10)	2.4(8)	2.0(8)	0.3(8)
C3	23.5(11)	21.0(13)	21.5(11)	2.0(9)	6.1(9)	0.1(10)
C4	28.3(14)	22.5(16)	30.6(13)	4.3(13)	6.2(11)	-2.6(12)
C5	41.2(16)	19.2(12)	40.5(15)	-1.9(11)	9.1(12)	-3.7(11)
C6	37(3)	22.6(14)	36.9(17)	-3.6(12)	11.7(18)	3.5(14)
C7	24.1(14)	22.1(14)	27.7(13)	1.7(10)	7.1(11)	2.5(11)
C8	39.2(18)	40(2)	43.9(17)	-5.1(14)	20.4(15)	-12.0(15)
C9	20.9(9)	24(2)	29.7(10)	-4.0(10)	6.7(7)	-0.2(11)
C10	19.8(14)	30.2(16)	26.1(12)	-4.6(12)	4.3(10)	-1.3(15)
C11	21.7(12)	29.5(13)	22.2(11)	-5.3(10)	1.4(9)	-1.1(10)
C12	21.6(11)	38.5(15)	24.5(11)	-4.1(10)	4.3(9)	3.1(10)
C13	22.9(14)	53.9(19)	24.7(14)	-10.8(13)	-0.1(12)	-0.6(12)
C14	33.0(16)	49(2)	35.5(18)	-18.7(14)	3.3(15)	-10.6(13)
C15	28.3(13)	33.3(15)	35.3(14)	-9.4(12)	6.6(11)	-9.3(12)
C16	47.8(17)	62(2)	25.6(13)	9.9(13)	-0.4(12)	14.6(16)
C17	22.3(11)	16.1(12)	22.2(10)	-0.9(9)	4.1(8)	-0.5(9)
C18	23.0(11)	28.4(14)	26.3(11)	1.6(11)	7.7(9)	4.8(11)
C19	31.6(10)	30(4)	24.7(9)	2.0(10)	9.8(8)	2.3(12)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for compound 12e. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C20	30.0(10)	26.5(12)	21.9(8)	0.6(8)	0.7(7)	2.6(11)
C21	21.5(9)	25.2(12)	28.0(9)	-1.5(8)	3.5(7)	1.6(8)
Br1A	44.8(11)	56.7(18)	27.6(9)	-3.8(13)	1.6(7)	-3.0(15)
O1A	52(6)	33(5)	51(6)	3(4)	32(5)	13(4)
O2A	26(5)	29(5)	31(4)	7(3)	9(3)	6(4)
O3A	43(6)	27(4)	39(5)	-4(3)	10(4)	10(4)
O4A	58(7)	46(5)	30(5)	-2(4)	2(4)	-18(5)
N1A	22(3)	15(4)	25(3)	-2(3)	5(2)	-2(3)
N2A	21(3)	18(8)	28(3)	-3(5)	7(3)	-3(4)
C1A	22(5)	16(4)	26(4)	-3(3)	6(3)	-3(3)
C2A	26(5)	14(4)	23(5)	-5(3)	10(4)	-5(3)
C3A	20(5)	12(4)	21(6)	-6(4)	6(4)	-7(3)
C4A	33(6)	12(5)	32(6)	-5(5)	13(4)	-1(4)
C5A	42(7)	16(5)	55(8)	9(5)	23(6)	7(5)
C6A	28(7)	21(5)	41(8)	11(5)	12(6)	0(5)
C7A	19(8)	21(5)	34(8)	5(5)	12(7)	0(5)
C8A	39.2(18)	40(2)	43.9(17)	-5.1(14)	20.4(15)	-12.0(15)
C9A	20.9(9)	24(2)	29.7(10)	-4.0(10)	6.7(7)	-0.2(11)
C10A	13(6)	29(5)	28(4)	5(3)	11(3)	1(5)
C11A	24(6)	32(5)	28(4)	1(4)	8(4)	-5(5)
C12A	34(6)	42(5)	27(4)	6(3)	6(4)	-9(4)
C13A	32(7)	57(6)	33(7)	17(5)	13(6)	-1(6)
C14A	42(8)	54(6)	38(6)	21(6)	1(6)	13(6)
C15A	31(7)	42(6)	41(5)	14(5)	9(5)	11(6)
C16A	78(13)	78(12)	25(6)	-3(7)	-2(6)	-22(11)
C17A	21(3)	14(6)	25(3)	-3(4)	6(2)	-4(4)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 12e. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka*b*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C18A	26(4)	40(8)	29(3)	-4(5)	11(3)	-4(6)
C19A	31.6(10)	30(4)	24.7(9)	2.0(10)	9.8(8)	2.3(12)
C20A	30.0(10)	26.5(12)	21.9(8)	0.6(8)	0.7(7)	2.6(11)
C21A	21.5(9)	25.2(12)	28.0(9)	-1.5(8)	3.5(7)	1.6(8)

Table 4 Bond Lengths for compound 12e.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C20	1.892(9)	Br1A	C20A	1.892(11)
O1	C4	1.364(3)	O1A	C4A	1.364(6)
O1	C8	1.415(5)	O1A	C8A	1.416(7)
O2	C1	1.207(3)	O2A	C1A	1.207(5)
O3	C9	1.222(6)	O3A	C9A	1.224(8)
O4	C12	1.363(3)	O4A	C12A	1.360(6)
O4	C16	1.431(3)	O4A	C16A	1.432(6)
N1	C1	1.449(3)	N1A	C1A	1.448(5)
N1	C9	1.395(5)	N1A	C9A	1.397(6)
N1	C17	1.427(3)	N1A	C17A	1.427(5)
N2	C17	1.334(3)	N2A	C17A	1.334(5)
N2	C21	1.341(7)	N2A	C21A	1.341(9)
C1	C2	1.478(3)	C1A	C2A	1.479(6)
C2	C3	1.404(3)	C2A	C3A	1.403(6)
C2	C7	1.394(4)	C2A	C7A	1.392(6)
C3	C4	1.378(4)	C3A	C4A	1.375(6)
C4	C5	1.401(4)	C4A	C5A	1.400(6)
C5	C6	1.376(4)	C5A	C6A	1.373(7)
C6	C7	1.388(4)	C6A	C7A	1.387(6)

Table 4 Bond Lengths for compound 12e.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C9	C10	1.492(3)	C9A	C10A	1.494(5)
C10	C11	1.385(4)	C10A	C11A	1.386(7)
C10	C15	1.392(4)	C10A	C15A	1.392(6)
C11	C12	1.398(3)	C11A	C12A	1.396(6)
C12	C13	1.387(4)	C12A	C13A	1.386(6)
C13	C14	1.373(4)	C13A	C14A	1.373(6)
C14	C15	1.373(4)	C14A	C15A	1.375(7)
C17	C18	1.389(3)	C17A	C18A	1.389(6)
C18	C19	1.382(5)	C18A	C19A	1.382(7)
C19	C20	1.381(5)	C19A	C20A	1.381(7)
C20	C21	1.379(3)	C20A	C21A	1.379(5)

Table 5 Bond Angles for compound 12e.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C4	O1	C8	118.3(2)	C4A	O1A	C8A	118.9(16)
C12	O4	C16	117.6(2)	C12A	O4A	C16A	118.4(12)
C9	N1	C1	121.1(2)	C9A	N1A	C1A	121.0(10)
C9	N1	C17	119.9(2)	C9A	N1A	C17A	120.9(8)
C17	N1	C1	114.45(17)	C17A	N1A	C1A	114.4(8)
C17	N2	C21	117.5(3)	C17A	N2A	C21A	118.2(17)
O2	C1	N1	120.2(2)	O2A	C1A	N1A	119.2(8)
O2	C1	C2	123.9(2)	O2A	C1A	C2A	125.1(8)
N1	C1	C2	115.86(18)	N1A	C1A	C2A	115.6(7)
C3	C2	C1	117.8(2)	C3A	C2A	C1A	118.2(8)
C7	C2	C1	121.2(2)	C7A	C2A	C1A	123.1(9)
C7	C2	C3	121.0(2)	C7A	C2A	C3A	118.7(10)
C4	C3	C2	119.4(2)	C4A	C3A	C2A	121.5(9)

Table 5 Bond Angles for compound 12e.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
O1	C4	C3	124.5(3)	O1A	C4A	C3A	125.1(9)
O1	C4	C5	115.6(3)	O1A	C4A	C5A	115.4(9)
C3	C4	C5	119.9(2)	C3A	C4A	C5A	119.5(9)
C6	C5	C4	120.1(3)	C6A	C5A	C4A	118.9(10)
C5	C6	C7	121.2(3)	C5A	C6A	C7A	122.2(14)
C6	C7	C2	118.4(3)	C6A	C7A	C2A	119.1(13)
O3	C9	N1	120.7(2)	O3A	C9A	N1A	120.1(9)
O3	C9	C10	121.3(4)	O3A	C9A	C10A	120.6(11)
N1	C9	C10	117.9(4)	N1A	C9A	C10A	118.9(10)
C11	C10	C9	122.5(3)	C11A	C10A	C9A	120.5(14)
C11	C10	C15	119.9(2)	C11A	C10A	C15A	120.3(11)
C15	C10	C9	117.6(3)	C15A	C10A	C9A	118.7(14)
C10	C11	C12	119.8(2)	C10A	C11A	C12A	121.4(10)
O4	C12	C11	115.2(2)	O4A	C12A	C11A	116.9(9)
O4	C12	C13	125.0(2)	O4A	C12A	C13A	125.7(10)
C13	C12	C11	119.8(3)	C13A	C12A	C11A	117.4(11)
C14	C13	C12	119.5(3)	C14A	C13A	C12A	120.7(13)
C15	C14	C13	121.5(3)	C13A	C14A	C15A	122.3(14)
C14	C15	C10	119.5(3)	C14A	C15A	C10A	117.8(13)
N2	C17	N1	113.7(2)	N2A	C17A	N1A	114.8(9)
N2	C17	C18	124.2(2)	N2A	C17A	C18A	123.6(10)
C18	C17	N1	122.1(2)	C18A	C17A	N1A	121.6(9)
C19	C18	C17	117.8(3)	C19A	C18A	C17A	117.5(16)
C20	C19	C18	118.1(5)	C20A	C19A	C18A	118.0(16)
C19	C20	Br1	120.3(4)	C19A	C20A	Br1A	119.3(15)
C21	C20	Br1	119.1(6)	C21A	C20A	Br1A	119.4(17)
C21	C20	C19	120.5(7)	C21A	C20A	C19A	120(2)

Table 5 Bond Angles for compound 12e.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
N2	C21	C20	121.8(7)	N2A	C21A	C20A	120.4(19)

Table 6 Torsion Angles for compound 12e.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
Br1	C20	C21	N2	179.7(16)	Br1A	C20A	C21A	N2A	-177(10)
O1	C4	C5	C6	179.4(3)	O1A	C4A	C5A	C6A	-179.7(16)
O2	C1	C2	C3	19.6(3)	O2A	C1A	C2A	C3A	-19(2)
O2	C1	C2	C7	-156.3(2)	O2A	C1A	C2A	C7A	158.2(18)
O3	C9	C10	C11	-143.8(4)	O3A	C9A	C10A	C11A	145(3)
O3	C9	C10	C15	34.4(6)	O3A	C9A	C10A	C15A	-27(5)
O4	C12	C13	C14	-178.9(3)	O4A	C12A	C13A	C14A	179.1(19)
N1	C1	C2	C3	-162.78(19)	N1A	C1A	C2A	C3A	162.5(11)
N1	C1	C2	C7	21.2(3)	N1A	C1A	C2A	C7A	-20(2)
N1	C9	C10	C11	32.4(6)	N1A	C9A	C10A	C11A	-43(4)
N1	C9	C10	C15	-149.3(3)	N1A	C9A	C10A	C15A	146(3)
N1	C17	C18	C19	-179.5(6)	N1A	C17A	C18A	C19A	176(4)
N2	C17	C18	C19	1.3(7)	N2A	C17A	C18A	C19A	-7(5)
C1	N1	C9	O3	-152.7(3)	C1A	N1A	C9A	O3A	149(2)
C1	N1	C9	C10	31.1(4)	C1A	N1A	C9A	C10A	-23(4)
C1	N1	C17	N2	25.3(3)	C1A	N1A	C17A	N2A	-26.8(16)
C1	N1	C17	C18	-153.9(2)	C1A	N1A	C17A	C18A	151.0(14)
C1	C2	C3	C4	-176.4(2)	C1A	C2A	C3A	C4A	176.6(13)
C1	C2	C7	C6	174.5(3)	C1A	C2A	C7A	C6A	-173.8(18)
C2	C3	C4	O1	-179.6(2)	C2A	C3A	C4A	O1A	177.9(14)
C2	C3	C4	C5	2.2(4)	C2A	C3A	C4A	C5A	0(2)
C3	C2	C7	C6	-1.3(4)	C3A	C2A	C7A	C6A	3(3)
C3	C4	C5	C6	-2.3(4)	C3A	C4A	C5A	C6A	-1(3)

Table 6 Torsion Angles for compound 12e.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
C4	C5	C6	C7	0.5(6)	C4A	C5A	C6A	C7A	4(3)
C5	C6	C7	C2	1.3(5)	C5A	C6A	C7A	C2A	-5(4)
C7	C2	C3	C4	-0.4(3)	C7A	C2A	C3A	C4A	-1(2)
C8	O1	C4	C3	9.7(4)	C8A	O1A	C4A	C3A	7(3)
C8	O1	C4	C5	-172.0(3)	C8A	O1A	C4A	C5A	-174.7(18)
C9	N1	C1	O2	38.5(3)	C9A	N1A	C1A	O2A	-41(2)
C9	N1	C1	C2	-139.2(2)	C9A	N1A	C1A	C2A	137(2)
C9	N1	C17	N2	-131.2(2)	C9A	N1A	C17A	N2A	132(2)
C9	N1	C17	C18	49.5(3)	C9A	N1A	C17A	C18A	-50(3)
C9	C10	C11	C12	178.6(4)	C9A	C10A	C11A	C12A	-176(2)
C9	C10	C15	C14	-178.6(4)	C9A	C10A	C15A	C14A	173(2)
C10	C11	C12	O4	178.9(3)	C10A	C11A	C12A	O4A	-176.9(19)
C10	C11	C12	C13	-0.3(4)	C10A	C11A	C12A	C13A	4(3)
C11	C10	C15	C14	-0.3(6)	C11A	C10A	C15A	C14A	1(4)
C11	C12	C13	C14	0.3(4)	C11A	C12A	C13A	C14A	-2(3)
C12	C13	C14	C15	-0.2(5)	C12A	C13A	C14A	C15A	0(4)
C13	C14	C15	C10	0.3(6)	C13A	C14A	C15A	C10A	1(4)
C15	C10	C11	C12	0.4(6)	C15A	C10A	C11A	C12A	-4(3)
C16	O4	C12	C11	-179.1(2)	C16A	O4A	C12A	C11A	178.5(16)
C16	O4	C12	C13	0.2(4)	C16A	O4A	C12A	C13A	-3(3)
C17	N1	C1	O2	-117.8(2)	C17A	N1A	C1A	O2A	117.2(13)
C17	N1	C1	C2	64.5(2)	C17A	N1A	C1A	C2A	-64.2(14)
C17	N1	C9	O3	2.3(4)	C17A	N1A	C9A	O3A	-8(4)
C17	N1	C9	C10	-174.0(3)	C17A	N1A	C9A	C10A	180(2)
C17	N2	C21	C20	1(2)	C17A	N2A	C21A	C20A	-11(13)
C17	C18	C19	C20	-2.4(17)	C17A	C18A	C19A	C20A	10(11)
C18	C19	C20	Br1	-178.9(13)	C18A	C19A	C20A	Br1A	177(8)

Table 6 Torsion Angles for compound 12e.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
C18	C19	C20	C21	3(3)	C18A	C19A	C20A	C21A	-15(17)
C19	C20	C21	N2	-2(3)	C19A	C20A	C21A	N2A	15(19)
C21	N2	C17	N1	-179.7(10)	C21A	N2A	C17A	N1A	-175(6)
C21	N2	C17	C18	-0.4(10)	C21A	N2A	C17A	C18A	7(6)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 12e.

Atom	*x*	*y*	*z*	U(eq)
H3	5382.39	4365.06	3669.95	26
H5	5570.25	-153.65	2927.04	40
H6	6407.37	865.72	2533.87	38
H7	6739.6	3649.36	2684.03	29
H8A	4323.5	3263.24	3523.92	59
H8B	4756.73	2840.72	4165.2	59
H8C	4148.45	1701.79	3894.77	59
H11	7457.16	4734.84	3799.41	30
H13	8588.69	6365.49	5343.59	41
H14	8495.49	9070.4	4975.43	48
H15	7898.69	9651.17	4031.94	39
H16A	8346.98	2051.19	5440.01	69
H16B	8235.71	3903.36	5661.16	69
H16C	8850.66	3498.16	5391.32	69
H18	7150.03	6899.23	1973.67	31
H19	6535.23	7410.49	1007.81	34
H21	4950.1	7518.96	1700.56	30
H3A	5388.71	10668.71	3647.6	21
H5A	5509.79	15119.48	2842.62	43

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 12e.

Atom	*x*	*y*	*z*	U(eq)
H6A	6382.98	14128.26	2498.07	35
H7A	6702.36	11344.86	2626.52	29
H8AA	4254.04	12004.05	3663.79	59
H8AB	4382.57	13531.85	4123.54	59
H8AC	4910.2	12096.58	4152.43	59
H11A	7496.23	10221.15	3787.95	33
H13A	8583.51	8579.7	5337.58	48
H14A	8469.63	5884.08	4980.22	55
H15A	7848.09	5266.04	4045.63	45
H16D	8856.91	11434.79	5412.65	93
H16E	8221.85	11129.95	5662.72	93
H16F	8371.83	12946.74	5434.47	93
H18A	7124.93	8214.91	1953.84	38
H19A	6527.02	7339.91	1013.76	34
H21A	4959.45	7041.45	1712.23	30

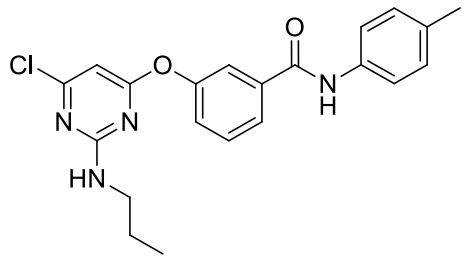
Table 8 Atomic Occupancy for compound 12e.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
Br1	0.8500(15)	O1	0.8500(15)	O2	0.8500(15)
O3	0.8500(15)	O4	0.8500(15)	N1	0.8500(15)
N2	0.8500(15)	C1	0.8500(15)	C2	0.8500(15)
C3	0.8500(15)	H3	0.8500(15)	C4	0.8500(15)
C5	0.8500(15)	H5	0.8500(15)	C6	0.8500(15)
H6	0.8500(15)	C7	0.8500(15)	H7	0.8500(15)
C8	0.8500(15)	H8A	0.8500(15)	H8B	0.8500(15)
H8C	0.8500(15)	C9	0.8500(15)	C10	0.8500(15)

Table 8 Atomic Occupancy for compound 12e.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
C11	0.8500(15)	H11	0.8500(15)	C12	0.8500(15)
C13	0.8500(15)	H13	0.8500(15)	C14	0.8500(15)
H14	0.8500(15)	C15	0.8500(15)	H15	0.8500(15)
C16	0.8500(15)	H16A	0.8500(15)	H16B	0.8500(15)
H16C	0.8500(15)	C17	0.8500(15)	C18	0.8500(15)
H18	0.8500(15)	C19	0.8500(15)	H19	0.8500(15)
C20	0.8500(15)	C21	0.8500(15)	H21	0.8500(15)
Br1A	0.1500(15)	O1A	0.1500(15)	O2A	0.1500(15)
O3A	0.1500(15)	O4A	0.1500(15)	N1A	0.1500(15)
N2A	0.1500(15)	C1A	0.1500(15)	C2A	0.1500(15)
C3A	0.1500(15)	H3A	0.1500(15)	C4A	0.1500(15)
C5A	0.1500(15)	H5A	0.1500(15)	C6A	0.1500(15)
H6A	0.1500(15)	C7A	0.1500(15)	H7A	0.1500(15)
C8A	0.1500(15)	H8AA	0.1500(15)	H8AB	0.1500(15)
H8AC	0.1500(15)	C9A	0.1500(15)	C10A	0.1500(15)
C11A	0.1500(15)	H11A	0.1500(15)	C12A	0.1500(15)
C13A	0.1500(15)	H13A	0.1500(15)	C14A	0.1500(15)
H14A	0.1500(15)	C15A	0.1500(15)	H15A	0.1500(15)
C16A	0.1500(15)	H16D	0.1500(15)	H16E	0.1500(15)
H16F	0.1500(15)	C17A	0.1500(15)	C18A	0.1500(15)
H18A	0.1500(15)	C19A	0.1500(15)	H19A	0.1500(15)
C20A	0.1500(15)	C21A	0.1500(15)	H21A	0.1500(15)

Crystallographic data for 3-((6-chloro-2-(propylamino)pyrimidin-4-yl)oxy)-*N*-(p-tolyl)benzamide 24



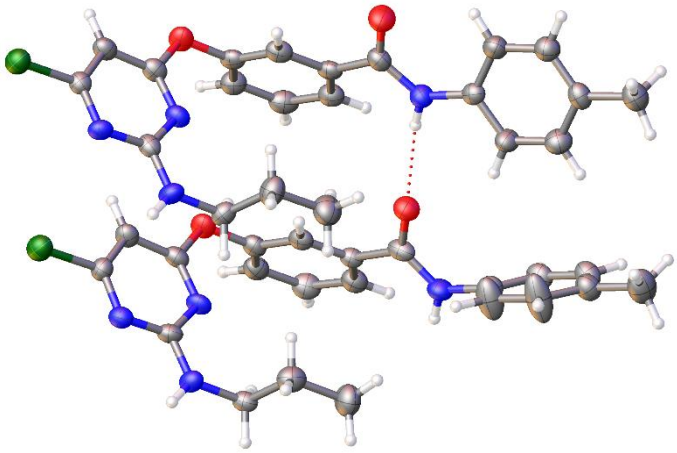


Table 1 Crystal data and structure refinement for compound 24.

Identification code	22mbB_MLB2_LCM_58_2_0f
Empirical formula	$C_{22}H_{25}ClN_4O_3$
Formula weight	428.91
Temperature/K	173.00
Crystal system	triclinic
Space group	*P*-1
a/Å	9.9951(12)
b/Å	11.5794(14)
c/Å	18.459(2)
α/°	92.121(6)
β/°	99.267(6)
γ/°	90.313(5)
Volume/Å^3	2106.9(5)
Z	4
ρ_{calc}g/cm^3	1.352
μ/mm^{-1}	1.868
F(000)	904.0
Crystal size/mm^3	0.139 × 0.028 × 0.016
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	4.854 to 129.984
Index ranges	-10 ≤ h ≤ 11, -13 ≤ k ≤ 13, -21 ≤ l ≤ 21
Reflections collected	46431
Independent reflections	7095 [R_{int} = 0.2403, R_{sigma} = 0.2189]
Data/restraints/parameters	7095/0/509
Goodness-of-fit on F^2	0.926
Final R indexes [I>=2σ (I)]	R_1 = 0.0967, wR_2 = 0.2126

Final R indexes [all data]	R_1 = 0.2660, wR_2 = 0.3115
Largest diff. peak/hole / e Å^{-3}	0.40/-0.57

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 24. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	5116(2)	8398.4(15)	-1034.0(10)	67.7(7)
O1	4432(6)	5592(4)	885(3)	62.3(15)
O2	5676(7)	6219(5)	3643(3)	76.6(18)
N1	2187(7)	9053(5)	832(3)	57.8(18)
N2	3587(6)	8703(4)	-29(3)	51.6(17)
N3	3307(7)	7311(5)	875(3)	52.3(17)
N4	3628(7)	6166(5)	4025(3)	55.1(17)
C1	1815(10)	8712(8)	2829(4)	95(3)
C2	2458(10)	9109(7)	2178(4)	78(3)
C3	1585(8)	8785(6)	1462(4)	56(2)
C4	3046(8)	8348(6)	554(4)	50(2)
C5	4404(8)	7967(6)	-289(4)	49(2)
C6	4762(8)	6896(6)	-9(4)	57(2)
C7	4138(9)	6642(6)	585(4)	55(2)
C8	3829(8)	5342(6)	1505(4)	51(2)
C9	2772(9)	4575(6)	1400(5)	62(2)
C10	2187(9)	4291(6)	2002(5)	68(2)
C11	2712(9)	4753(6)	2689(4)	61(2)
C12	3802(8)	5511(6)	2791(4)	52(2)
C13	4383(8)	5800(6)	2182(5)	59(2)
C14	4443(10)	5996(6)	3513(4)	59(2)
C15	3989(9)	6548(6)	4757(4)	51(2)
C16	2973(9)	6872(6)	5151(4)	61(2)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 24. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C17	3253(10)	7260(6)	5867(5)	71(2)
C18	4565(10)	7354(6)	6241(4)	63(2)
C19	5573(10)	6996(6)	5857(5)	69(2)
C20	5306(10)	6600(6)	5132(4)	68(2)
C21	4893(9)	7803(7)	7020(4)	84(3)
Cl2	148(2)	8423.5(15)	-1072.2(10)	70.4(7)
O3	-539(5)	5628(4)	850(3)	61.9(15)
O4	634(6)	6272(4)	3638(3)	65.0(16)
N5	-2824(7)	9053(5)	765(3)	54.7(17)
N6	-1418(7)	8708(5)	-90(3)	52.8(17)
N7	-1692(7)	7341(5)	826(3)	51.1(17)
N8	-1455(7)	6332(5)	3945(4)	61.8(18)
C22	-3115(10)	8975(7)	2792(4)	92(3)
C23	-2497(9)	9228(7)	2111(4)	76(3)
C24	-3402(8)	8829(5)	1415(4)	51(2)
C25	-1965(8)	8354(6)	492(4)	53(2)
C26	-559(8)	7983(6)	-328(4)	51(2)
C27	-201(8)	6928(6)	-43(4)	56(2)
C28	-837(8)	6674(6)	548(4)	51(2)
C29	-1136(8)	5365(6)	1465(4)	51(2)
C30	-2169(9)	4587(6)	1372(4)	63(2)
C31	-2737(9)	4273(6)	1973(5)	68(2)
C32	-2240(9)	4758(6)	2668(4)	63(2)
C33	-1186(8)	5540(5)	2750(4)	44.8(19)
C34	-617(8)	5833(5)	2142(4)	52(2)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 24. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C35	-603(10)	6072(6)	3476(5)	57(2)
C36	-1042(9)	6844(6)	4668(4)	60(2)
C37	-986(8)	6208(6)	5274(4)	63(2)
C38	-592(9)	6696(7)	5962(4)	66(2)
C39	-301(9)	7833(7)	6061(4)	72(3)
C40	-324(13)	8465(7)	5450(5)	134(5)
C41	-689(12)	7987(7)	4755(5)	113(4)
C42	115(11)	8364(7)	6827(5)	105(4)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 24. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	76.4(18)	56.4(12)	73.4(13)	9.1(10)	20.1(11)	3.3(11)
O1	84(5)	38(3)	67(3)	9(2)	17(3)	13(3)
O2	60(5)	92(4)	75(4)	-5(3)	4(3)	-12(4)
N1	56(5)	48(4)	74(4)	7(3)	25(4)	9(3)
N2	49(5)	38(4)	69(4)	2(3)	12(4)	5(3)
N3	54(5)	38(4)	63(4)	10(3)	2(3)	0(3)
N4	51(5)	54(4)	60(4)	4(3)	8(4)	-8(3)
C1	85(9)	112(7)	84(6)	4(5)	6(6)	-11(6)
C2	94(9)	71(6)	74(6)	3(5)	28(6)	-13(5)
C3	63(7)	41(4)	68(5)	2(4)	22(5)	-3(4)
C4	45(6)	49(5)	55(5)	10(4)	1(4)	-13(4)
C5	48(6)	44(5)	54(5)	3(4)	6(4)	-10(4)
C6	63(7)	44(5)	61(5)	1(4)	3(4)	7(4)
C7	63(7)	37(5)	64(5)	3(4)	2(5)	0(4)
C8	51(6)	33(4)	69(6)	12(4)	11(5)	8(4)
C9	64(7)	50(5)	72(6)	1(4)	10(5)	-1(4)
C10	57(7)	48(5)	95(7)	-4(5)	6(5)	-2(4)
C11	60(7)	49(5)	75(6)	10(4)	10(5)	2(4)
C12	44(6)	40(4)	71(6)	13(4)	8(5)	7(4)
C13	58(7)	40(4)	78(6)	4(4)	5(5)	-1(4)
C14	59(8)	53(5)	61(6)	7(4)	0(5)	1(5)
C15	53(7)	41(4)	61(5)	12(4)	14(5)	-2(4)
C16	48(7)	71(5)	61(5)	-5(4)	5(5)	-6(4)
C17	62(8)	68(6)	83(7)	1(5)	15(5)	-8(5)
C18	75(8)	51(5)	59(5)	7(4)	0(5)	-6(5)
C19	69(8)	64(5)	73(6)	5(4)	11(5)	1(5)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 24. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C20	67(8)	71(6)	67(6)	8(4)	11(5)	11(5)
C21	91(8)	83(6)	73(6)	5(5)	1(5)	0(5)
Cl2	79.7(19)	58.0(12)	77.2(14)	13.2(10)	21.6(12)	5.4(11)
O3	79(5)	41(3)	67(3)	13(2)	13(3)	12(3)
O4	57(5)	64(3)	72(4)	2(3)	5(3)	-7(3)
N5	57(5)	43(4)	67(4)	11(3)	20(4)	5(3)
N6	48(5)	45(4)	65(4)	7(3)	7(4)	-2(3)
N7	52(5)	31(3)	69(4)	13(3)	2(3)	5(3)
N8	45(5)	68(4)	72(5)	6(4)	5(4)	-2(4)
C22	109(10)	92(7)	79(6)	18(5)	25(6)	-5(6)
C23	69(8)	67(5)	92(6)	-2(5)	10(5)	-4(5)
C24	44(6)	38(4)	71(5)	9(4)	6(4)	-4(4)
C25	42(6)	43(5)	72(5)	6(4)	5(4)	4(4)
C26	55(7)	45(5)	53(5)	2(4)	13(4)	-5(4)
C27	68(7)	38(4)	61(5)	5(4)	8(4)	6(4)
C28	48(6)	39(5)	63(5)	8(4)	-4(4)	-1(4)
C29	55(7)	38(4)	60(5)	9(4)	3(4)	2(4)
C30	67(7)	49(5)	69(5)	1(4)	5(5)	7(4)
C31	65(7)	44(5)	92(6)	3(4)	7(5)	-14(4)
C32	64(7)	49(5)	74(6)	8(4)	5(5)	1(4)
C33	40(6)	33(4)	60(5)	5(3)	2(4)	-5(3)
C34	53(6)	37(4)	67(5)	13(4)	8(5)	3(4)
C35	43(7)	44(5)	81(7)	20(4)	3(5)	-8(4)
C36	67(7)	47(5)	68(5)	7(4)	12(5)	-1(4)
C37	66(7)	44(4)	79(6)	17(4)	9(5)	-7(4)
C38	64(7)	60(5)	72(6)	16(4)	2(5)	-16(4)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 24. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka*b*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C39	78(8)	63(6)	71(6)	6(5)	2(5)	2(5)
C40	266(17)	45(5)	76(7)	7(5)	-16(8)	-23(7)
C41	200(13)	52(6)	78(7)	11(5)	-6(7)	-14(6)
C42	143(11)	83(7)	86(7)	-6(5)	9(7)	10(6)

Table 4 Bond Lengths for compound 24.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C5	1.734(7)	Cl2	C26	1.734(7)
O1	C7	1.369(8)	O3	C28	1.365(7)
O1	C8	1.414(8)	O3	C29	1.406(8)
O2	C14	1.242(9)	O4	C35	1.243(9)
N1	C3	1.435(8)	N5	C24	1.445(8)
N1	C4	1.336(9)	N5	C25	1.327(9)
N2	C4	1.355(8)	N6	C25	1.357(9)
N2	C5	1.314(8)	N6	C26	1.318(9)
N3	C4	1.367(8)	N7	C25	1.354(8)
N3	C7	1.302(9)	N7	C28	1.307(9)
N4	C14	1.352(10)	N8	C35	1.336(9)
N4	C15	1.395(9)	N8	C36	1.436(9)
C1	C2	1.536(10)	C22	C23	1.523(10)
C2	C3	1.496(10)	C23	C24	1.503(9)
C5	C6	1.387(9)	C26	C27	1.374(9)
C6	C7	1.385(10)	C27	C28	1.389(10)
C8	C9	1.361(10)	C29	C30	1.352(10)
C8	C13	1.369(9)	C29	C34	1.364(9)
C9	C10	1.386(10)	C30	C31	1.385(10)

Table 4 Bond Lengths for compound 24.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C10	C11	1.379(10)	C31	C32	1.394(10)
C11	C12	1.381(10)	C32	C33	1.372(10)
C12	C13	1.395(10)	C33	C34	1.388(9)
C12	C14	1.473(10)	C33	C35	1.483(10)
C15	C16	1.387(10)	C36	C37	1.356(9)
C15	C20	1.385(10)	C36	C41	1.365(10)
C16	C17	1.366(10)	C37	C38	1.367(9)
C17	C18	1.382(11)	C38	C39	1.346(10)
C18	C19	1.379(11)	C39	C40	1.364(10)
C18	C21	1.496(10)	C39	C42	1.516(10)
C19	C20	1.383(10)	C40	C41	1.371(10)

Table 5 Bond Angles for compound 24.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C7	O1	C8	116.5(6)	C28	O3	C29	117.2(6)
C4	N1	C3	122.9(6)	C25	N5	C24	124.0(6)
C5	N2	C4	115.4(6)	C26	N6	C25	115.1(6)
C7	N3	C4	115.7(7)	C28	N7	C25	115.6(7)
C14	N4	C15	128.2(7)	C35	N8	C36	124.1(7)
C3	C2	C1	111.3(7)	C24	C23	C22	112.0(7)
N1	C3	C2	113.7(7)	N5	C24	C23	112.7(6)
N1	C4	N2	118.0(7)	N5	C25	N6	118.1(6)
N1	C4	N3	117.5(7)	N5	C25	N7	117.0(8)
N2	C4	N3	124.5(8)	N7	C25	N6	124.9(7)
N2	C5	Cl1	116.5(5)	N6	C26	Cl2	115.6(5)
N2	C5	C6	125.6(7)	N6	C26	C27	125.8(7)
C6	C5	Cl1	117.9(7)	C27	C26	Cl2	118.6(6)

Table 5 Bond Angles for compound 24.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
C7	C6	C5	112.9(7)	C26	C27	C28	113.1(7)
O1	C7	C6	116.2(8)	O3	C28	C27	116.0(7)
N3	C7	O1	118.1(7)	N7	C28	O3	118.5(7)
N3	C7	C6	125.7(7)	N7	C28	C27	125.5(6)
C9	C8	O1	117.2(7)	C30	C29	O3	118.2(7)
C9	C8	C13	123.1(8)	C30	C29	C34	121.5(8)
C13	C8	O1	119.5(7)	C34	C29	O3	120.1(7)
C8	C9	C10	118.4(8)	C29	C30	C31	119.5(8)
C11	C10	C9	119.7(8)	C30	C31	C32	119.9(8)
C10	C11	C12	121.3(8)	C33	C32	C31	119.6(8)
C11	C12	C13	118.7(7)	C32	C33	C34	119.7(7)
C11	C12	C14	123.9(8)	C32	C33	C35	122.0(7)
C13	C12	C14	117.4(8)	C34	C33	C35	118.3(7)
C8	C13	C12	118.8(7)	C29	C34	C33	119.8(7)
O2	C14	N4	122.2(8)	O4	C35	N8	121.5(8)
O2	C14	C12	120.9(9)	O4	C35	C33	120.8(8)
N4	C14	C12	116.8(8)	N8	C35	C33	117.7(8)
C16	C15	N4	118.8(8)	C37	C36	N8	121.1(7)
C20	C15	N4	124.5(8)	C37	C36	C41	118.7(8)
C20	C15	C16	116.7(8)	C41	C36	N8	120.2(7)
C17	C16	C15	122.0(8)	C36	C37	C38	121.0(7)
C16	C17	C18	121.9(9)	C39	C38	C37	121.1(7)
C17	C18	C21	122.7(9)	C38	C39	C40	117.7(8)
C19	C18	C17	116.2(8)	C38	C39	C42	120.4(7)
C19	C18	C21	121.1(9)	C40	C39	C42	121.8(8)
C18	C19	C20	122.6(9)	C39	C40	C41	122.0(8)
C19	C20	C15	120.6(8)	C36	C41	C40	119.4(8)

Table 6 Torsion Angles for compound 24.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
Cl1	C5	C6	C7	179.8(5)	Cl2	C26	C27	C28	-179.8(5)
O1	C8	C9	C10	178.2(6)	O3	C29	C30	C31	177.2(6)
O1	C8	C13	C12	-177.8(6)	O3	C29	C34	C33	-178.0(6)
N2	C5	C6	C7	1.3(11)	N6	C26	C27	C28	-0.5(11)
N4	C15	C16	C17	-179.6(6)	N8	C36	C37	C38	179.9(8)
N4	C15	C20	C19	179.7(6)	N8	C36	C41	C40	178.7(10)
C1	C2	C3	N1	-175.3(7)	C22	C23	C24	N5	-179.5(6)
C3	N1	C4	N2	-178.0(6)	C24	N5	C25	N6	-175.1(6)
C3	N1	C4	N3	2.6(11)	C24	N5	C25	N7	4.2(11)
C4	N1	C3	C2	84.8(9)	C25	N5	C24	C23	85.0(8)
C4	N2	C5	Cl1	-179.8(5)	C25	N6	C26	Cl2	180.0(5)
C4	N2	C5	C6	-1.3(11)	C25	N6	C26	C27	0.6(11)
C4	N3	C7	O1	-179.9(6)	C25	N7	C28	O3	-178.7(6)
C4	N3	C7	C6	0.0(11)	C25	N7	C28	C27	0.6(11)
C5	N2	C4	N1	-178.7(6)	C26	N6	C25	N5	179.1(7)
C5	N2	C4	N3	0.6(10)	C26	N6	C25	N7	-0.2(11)
C5	C6	C7	O1	179.3(6)	C26	C27	C28	O3	179.1(6)
C5	C6	C7	N3	-0.6(11)	C26	C27	C28	N7	-0.2(11)
C7	O1	C8	C9	105.2(7)	C28	O3	C29	C30	105.8(7)
C7	O1	C8	C13	-79.7(8)	C28	O3	C29	C34	-78.3(8)
C7	N3	C4	N1	179.3(7)	C28	N7	C25	N5	-179.6(7)
C7	N3	C4	N2	0.0(10)	C28	N7	C25	N6	-0.4(11)
C8	O1	C7	N3	-1.9(10)	C29	O3	C28	N7	-2.7(10)
C8	O1	C7	C6	178.3(7)	C29	O3	C28	C27	178.0(7)
C8	C9	C10	C11	-2.2(12)	C29	C30	C31	C32	-0.1(12)
C9	C8	C13	C12	-3.0(11)	C30	C29	C34	C33	-2.3(11)

Table 6 Torsion Angles for compound 24.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
C9	C10	C11	C12	1.0(12)	C30	C31	C32	C33	-0.3(12)
C10	C11	C12	C13	-0.6(11)	C31	C32	C33	C34	-0.6(11)
C10	C11	C12	C14	-177.4(7)	C31	C32	C33	C35	-179.1(7)
C11	C12	C13	C8	1.5(11)	C32	C33	C34	C29	1.9(11)
C11	C12	C14	O2	147.4(8)	C32	C33	C35	O4	143.3(7)
C11	C12	C14	N4	-31.6(10)	C32	C33	C35	N8	-37.1(10)
C13	C8	C9	C10	3.3(12)	C34	C29	C30	C31	1.4(12)
C13	C12	C14	O2	-29.4(10)	C34	C33	C35	O4	-35.3(10)
C13	C12	C14	N4	151.5(7)	C34	C33	C35	N8	144.3(7)
C14	N4	C15	C16	167.9(7)	C35	N8	C36	C37	-104.8(9)
C14	N4	C15	C20	-13.8(11)	C35	N8	C36	C41	74.8(11)
C14	C12	C13	C8	178.6(6)	C35	C33	C34	C29	-179.5(6)
C15	N4	C14	O2	-3.0(12)	C36	N8	C35	O4	-0.7(11)
C15	N4	C14	C12	176.0(6)	C36	N8	C35	C33	179.7(6)
C15	C16	C17	C18	0.1(11)	C36	C37	C38	C39	2.7(14)
C16	C15	C20	C19	-2.0(10)	C37	C36	C41	C40	-1.7(16)
C16	C17	C18	C19	-2.2(11)	C37	C38	C39	C40	-4.3(15)
C16	C17	C18	C21	178.6(7)	C37	C38	C39	C42	179.1(9)
C17	C18	C19	C20	2.2(11)	C38	C39	C40	C41	2.9(18)
C18	C19	C20	C15	0.0(11)	C39	C40	C41	C36	0.0(19)
C20	C15	C16	C17	2.0(11)	C41	C36	C37	C38	0.3(14)
C21	C18	C19	C20	-178.6(7)	C42	C39	C40	C41	179.5(11)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 24.

Atom	*x*	*y*	*z*	U(eq)
H1	1981.04	9710.53	618.99	69
H4	2760.09	6016.98	3877.64	66
H1A	901.51	9029.73	2798.62	142
H1B	2371.97	8986.11	3290.33	142
H1C	1759.83	7865.65	2815.21	142
H2A	2591.85	9958.08	2218.65	94
H2B	3358.38	8749.55	2193.35	94
H3A	1386.43	7945.42	1445.36	68
H3B	713.07	9195.07	1435.16	68
H6	5371.16	6390.4	-204.15	68
H9	2443.14	4243.99	926.06	75
H10	1427	3779.62	1942.19	81
H11	2314.69	4545.23	3100.91	74
H13	5147.84	6304.3	2236.15	71
H16	2055.71	6823.31	4915.25	73
H17	2524.03	7471.63	6115.89	85
H19	6486.23	7021.5	6100.74	82
H20	6032.69	6361.94	4888.89	82
H21A	5257	7176.49	7334.21	126
H21B	4067.72	8098.71	7181.46	126
H21C	5568.51	8427.56	7055.93	126
H5	-3055.59	9692.51	535.41	66
H8	-2322.62	6180.28	3802.13	74
H22A	-3405.8	8163.34	2769.58	138
H22B	-3899.86	9472.93	2811.13	138
H22C	-2438.68	9127.22	3232.17	138

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 24.

Atom	*x*	*y*	*z*	U(eq)
H23A	-2329.17	10070.09	2098.89	91
H23B	-1613.16	8835.68	2140.98	91
H24A	-3577.4	7989.11	1432.01	61
H24B	-4282.76	9227.05	1385.53	61
H27	418.46	6424.78	-231.83	67
H30	-2502.85	4259.37	897.56	75
H31	-3463.23	3727.31	1913.03	82
H32	-2628.29	4547.23	3081.58	76
H34	131.08	6356.02	2197.28	63
H37	-1224.64	5410.16	5220.27	76
H38	-522.94	6225.47	6376.54	79
H40	-79.54	9261.49	5507.92	161
H41	-696.94	8446.23	4338.69	136
H42A	764.67	8996.75	6813.16	158
H42B	537.51	7774.03	7154.25	158
H42C	-687.1	8664.44	7010.6	158

Table 8 Solvent masks information for compound 24.

Number	X	Y	Z	Volume	Electron count	Content
1	0.500	0.000	0.500	163.2	59.8	4 methanol

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