Supporting Information

Palladium catalyzed selective arylation of *o*-carboranes via B(4)-H activation: amide induced regioselectivity reversal

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Context

General information

1a, 1r-1za were synthesized according to literature method[1,2]. Other materials were purchased from Acros, J&K and Aladdin, and used as received unless otherwise specified. All reactions under standard conditions were monitored by thin-layer chromatography (TLC) on gel F254 plates. The silica gel (200-300 meshes) is used for column chromatography, and the distillation range of petroleum ether is 60-90 ^{o}C. ^{1}H, ^{13}C{^{1}H} and ^{11}B{^{1}H} NMR spectra were recorded on the Bruker 600MHz instruments. All ^{1}H NMR and ^{13}C{^{1}H} NMR spectra data are reported in ppm relative to tetramethylsilane (TMS) as internal standard, and ^{11}B{^{1}H} NMR spectra data are referenced to external BF_3•Et_2O (Due to 1J(^{11}B-^{1}H) coupling, the resonances corresponding to the B-H bonds display a broad peaks with low intensity, therefore, the B-H protons were not reported in the NMR data). HRMS data were measured with ESI techniques.

The calculations were performed using the DFT program Dmol3 in Materials Studio (Accelrys, San Diego, CA), in which the physical wave functions were expanded in terms of numerical basis sets. The double numerical basis set with polarisation function (DNP)[3,4], that is comparable to the 6-31G** basis set, was utilised during the calculations[5]. The core electrons were treated with DFT semicore pseudopotentials. The exchange-correlation energy was calculated using the PBE and GGA methods[6]. Special point sampling integration over the Brillouin zone was employed using Monkhorst-Pack schemes with a 5 × 5 × 1 k-point mesh[7]. A Fermi smearing of 0.005 Ha and a global orbital cutoff of 7 Å were employed. The convergence criteria for the geometric optimization and energy calculation were set as follows: (1) self-consistent field tolerance of 1.0×10^{-6} Ha/atom, (2) energy tolerance of 1.0×10^{-5} Ha/atom, (3) maximum force tolerance of 0.002 Ha/Å, and (4) maximum displacement tolerance of 0.005 Å.

Experimental

Typical procedure for synthesis of 1a:

To a 100 ml dried flask were sequentially added 9-I-*o*-carborane (1.35 g, 5 mmol), benzamide (1.817 g, 15mmol), $Pd_2(dba)_3$ (0.115 g, 0.125 mmol), Davephos (0.098 g,

0.25 mmol), K_3PO_4 (5.31 g, 25 mmol) and toluene (20 ml) under argon atmosphere. The reaction mixture was stirred at 100 ^oC for 2h, the color of system was changed from purple to orange. Then the mixture was filtered through a short silica gel column using ethyl acetate as eluent. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=8:1-5:1 (v/v) as eluent and afforded **1a** (1.32 g, 50 % yield).

Typical procedure for synthesis of 2a:

To a 10 ml dried flask were sequentially added 9-benzamide-*o*-carborane (52.6 mg, 0.20 mmol), THF (1 ml), $PhB(OH)_2$ (48.6 mg, 0.40 mmol), $Pd(MeCN)_4(BF_4)_2$ (8.8 mg, 0.02 mmol) ,$Cu(OAc)_2$ (72.8 mg, 0.40 mmol) and Cyclohexanecarboxylic acid (25.6 mg, 0.2 mmol) under an argon atmosphere. After the reaction mixture was stirred at 40 ^oC for 24h, the reaction mixture was cooled to room temperature and filtered through a short silica gel column using ethyl acetate as eluent. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=5:1-2:1 (v/v) as eluent to gave **2a** with 74 % yield (50 mg).

Typical procedure for synthesis of 3a:

To a 10 ml dried flask were sequentially added 2a (67.8 mg, 0.20 mmol), 1,4-dioxane (1 ml), $Pd(OAc)_2$ (4.6 mg, 0.02 mmol), AgOAc (66.7 mg, 0.40 mmol) and K_2CO_3 (27.6 mg, 0.2 mmol) under an argon atmosphere. After the reaction mixture was stirred at 100 ^oC for 24h, the reaction mixture was cooled to room temperature and filtered through a short silica gel column using ethyl acetate as eluent. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=8:1 (v/v) as eluent to gave the **3a** with 52% yield (24 mg).

Table S1. Calculated Mulliken charge

Mulliken charge	9-benzamide-*o*-carborane	*o*-carborane
B4	-0.010	0.024
H4	0.023	-0.000
B5	0.004	0.025
H5	0.034	0.001
B8	-0.072	-0.031
H8	-0.007	-0.012
B10	-0.075	-0.030
H10	-0.006	-0.011
B12	-0.063	-0.012
H12	-0.015	-0.018

Table S2. Detailed calculated Mulliken charge

9-benzamide-*o*-carborane:

	charge	spin
N (1)	-0.427	0.000
O (2)	-0.456	0.000
C (3)	-0.262	0.000
C (4)	-0.260	0.000
H (5)	0.171	0.000
H (6)	0.175	0.000
C (7)	0.419	0.000
B (8)	0.084	0.000
H (9)	0.010	0.000
B (10)	0.004	0.000
H (11)	0.034	0.000
B (12)	-0.010	0.000
H (13)	0.023	0.000
B (14)	0.087	0.000
H (15)	0.010	0.000
B (16)	0.016	0.000
H (17)	0.001	0.000
B (18)	-0.075	0.000
H (19)	-0.006	0.000
B (20)	0.415	0.000
B (21)	-0.072	0.000
H (22)	-0.007	0.000
B (23)	0.014	0.000
H (24)	0.000	0.000
B (25)	-0.063	0.000
H (26)	-0.015	0.000
H (27)	0.198	0.000
C (28)	-0.083	0.000
C (29)	-0.079	0.000
C (30)	-0.106	0.000
C (31)	-0.052	0.000
C (32)	-0.061	0.000
C (33)	-0.082	0.000
H (34)	0.085	0.000
H (35)	0.085	0.000
H (36)	0.086	0.000
H (37)	0.114	0.000
H (38)	0.086	0.000

o-carborane:

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Mulliken atomic charges:
          charge    spin
H (  1)  -0.018   0.000
C (  2)  -0.252   0.000
C (  3)  -0.254   0.000
H (  4)   0.171   0.000
H (  5)   0.171   0.000
B (  6)   0.098   0.000
H (  7)   0.008   0.000
B (  8)   0.027   0.000
H (  9)  -0.000   0.000
B ( 10)   0.022   0.000
H ( 11)  -0.000   0.000
B ( 12)   0.098   0.000
H ( 13)   0.008   0.000
B ( 14)   0.024   0.000
H ( 15)  -0.000   0.000
B ( 16)  -0.031   0.000
H ( 17)  -0.012   0.000
B ( 18)  -0.012   0.000
B ( 19)  -0.030   0.000
H ( 20)  -0.011   0.000
B ( 21)   0.025   0.000
H ( 22)   0.001   0.000
B ( 23)  -0.013   0.000
H ( 24)  -0.018   0.000
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Reference:

1. Mukhin, S. N.; Kabytaev, K. Z.; Zhigareva, G. G.; Glukhov, I. V.; Starikova, Z. A.; Bregadze, V. I.; Beletskaya, I. P. *Organometallics* **2008**, *27*, 5937.

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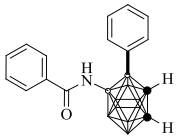
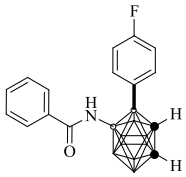
Spectroscopic data for products



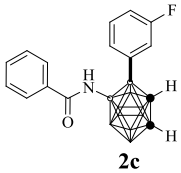
2a

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.60 (m, 4H), 7.42-7.39 (dd, 1H, *J* = 6 Hz), 7.34-7.28 (m, 5H), 5.69 (s, 1H), 3.74 (s, 1H), 3.72 (s, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 135.8, 134.1, 132.9, 131.1, 128.9, 128.3, 128.0, 127.0, 52.3, 47.6; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -3.7 (2B), -9.8 (2B), -14.2 (1B), -17.4 (3B); HRMS: calculated for $C_{15}B_{10}H_{22}NO^{+}$ $(M+H)^{+}$ 340.2699, found 340.2706.



2b

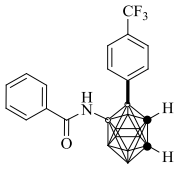
^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.61-7.58 (m, 4H), 7.43-7.41 (dd, 1H, *J* = 6 Hz,), 7.35-7.33 (dd, 2H, *J* = 6Hz), 6.99-6.96 (dd, 2H, *J* = 6 Hz,), 5.68 (s, 1H), 3.74 (s, 1H), 3.71 (s, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.4, 164.5-162.8 (d, *J* = 248 Hz), 136.1-136.0 (d, *J* = 8 Hz), 135.7, 131.2, 128.4, 127.0, 115.2-115.0 (d, *J* = 21 Hz), 52.6, 47.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -3.9 (2B), -9.7 (2B), -14.3 (1B), -16.9 (3B); HRMS: calculated for $C_{15}H_{19}B_{10}FNO^{-}$ $(M-H)^{-}$ 358.2387, found 358.2408.



2c

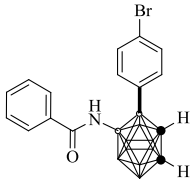
^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.61-7.60 (d, 2H, *J* = 6 Hz), 7.43-7.41 (dd, 1H, *J* = 6 Hz), 7.37-7.33 (m, 3H), 7.30-7.23 (m, 2H), 7.01-6.99 (m, 1H), 5.70 (s, 1H), 3.77 (s, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.4, 163.3-161.7 (d, *J* = 246 Hz), 135.7, 131.2, 129.8-129.7 (m, *J* = 6 Hz), 128.4, 127.0, 120.6-120.4 (d, *J* = 20 Hz), 115.9-115.8 (d, *J* = 20 Hz), 52.4, 47. 8; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5

(1B), -2.3 (1B), -4.4 (2B), -9.8 (2B), -14.2 (1B), -17.0 (3B); HRMS: calculated for $C_{15}H_{21}B_{10}FNO^+$ $(M+H)^+$ 358.2605, found 358.2608.



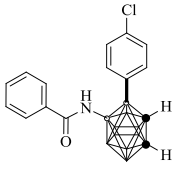
2d

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.75-7.73 (d, 2H, *J* = 6 Hz), 7.63-7.61 (m, 2H), 7.54-7.53 (d, 2H, *J* = 6 Hz), 7.44-7.41 (dd, 1H, *J* = 6 Hz), 7.36-7.33 (dd, 2H, *J* = 6 Hz), 5.70 (s, 1H), 3.76 (s, 2H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 135.5, 134.4, 131.3, 131.2-130.6 (m, *J* = 30 Hz), 128.4, 127.0, 124.7-124.6 (d, *J* = 3 Hz), 52.4, 47.6; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -4.6 (2B), -9.8 (2B), -14.4 (1B), -17.0 (3B); HRMS: calculated for $C_{16}B_{10}H_{21}NOF_3^+$ $(M+H)^+$ 408.2573, found 408.2574.



2e

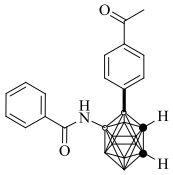
^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.60 (m, 2H), 7.49-7.47 (m, 2H), 7.42-7.41 (m, 2H), 7.36-7.30 (m, 3H), 5.69 (s, 1H), 3.73 (s, 1H), 3.71 (s, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 135.8, 134.1, 131.2, 131.1, 129.0, 128.4, 127.0, 123.9, 52.4, 47.6; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -4.0 (2B), -9.8 (2B), -14.3 (1B), -16.9 (3B); HRMS: calculated for $C_{15}B_{10}H_{19}NOBr^-$ $(M-H)^-$ 418.1586, found 418.1603.



2f

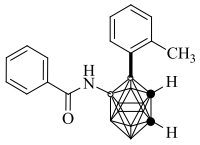
^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.61 (d, 2H, *J* = 6 Hz), 7.55-7.54 (d, 2H, *J* = 6 Hz), 7.44-7.41(dd, 1H, *J* = 6 Hz), 7.36-7.33 (dd, 2H, *J* = 6 Hz), 7.27-7.25 (m, 2H), 5.68 (s, 1H), 3.73 (s, 1H), 3.70 (s, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 135.6, 135.5, 135.4, 131.2, 128.4, 128.2, 127.0, 52.5, 47.6; $^{11}B\{^1H\}$ NMR (192

MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -4.1 (2B), -9.8 (2B), -14.4 (1B), -17.0 (3B); HRMS: calculated for $C_{15}H_{19}B_{10}ClNO^-$ $(M-H)^-$ 374.2091, found 374.2134.



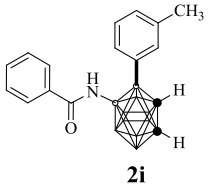
2g

^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.83-7.82 (d, 2H, *J* = 6 Hz), 7.69-7.68 (d, 2H, *J* = 6 Hz), 7.60-7.59 (d, 2H, *J* = 6 Hz), 7.42-7.40 (dd, 1H, *J* = 6 Hz), 7.34-7.31 (dd, 2H, *J* = 6 Hz), 5.73 (s, 1H), 3.88 (s, 1H), 3.82 (s, 1H), 2.54 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 198.3, 169.3, 137.1, 135.6, 134.2, 131.3, 128.4, 127.6, 127.0, 52.5, 47.8, 26.7; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.4 (1B), -2.4 (1B), -4.5 (2B), -9.8 (2B), -14.4 (1B), -16.9 (3B); HRMS: calculated for $C_{17}B_{10}H_{24}NO_2^+$ $(M+H)^+$ 382.2805, found 382.2806.



2h

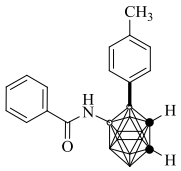
^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.63-7.62 (d, 1H, *J* = 6 Hz), 7.59-7.58 (d, 2H, *J* = 6 Hz), 7.41-7.39 (dd, 1H, *J* = 6 Hz), 7.33-7.31 (dd, 2H, *J* = 6 Hz), 7.22-7.19 (m, 1H), 7.14-7.12 (dd, 2H, *J* = 6 Hz), 5.70 (s, 1H), 3.94 (s, 1H), 3.71 (s, 1H), 2.57 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.1, 142.2, 136.7, 135.9, 131.1, 131.0, 128.8, 128.3, 127.0, 125.5, 52.0, 47.4, 23.8; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.6 (1B), -3.1 (1B), -4.1 (2B), -9.6 (2B), -13.9 (1B), -17.0 (3B); HRMS: calculated for $C_{16}B_{10}H_{24}NO^+$ $(M+H)^+$ 354.2856, found 354.2859.



2i

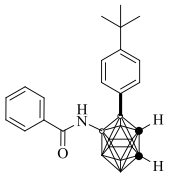
^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.61 (d, 2H, *J* = 6 Hz), 7.42-7.40 (m, 3H), 7.35-7.32 (dd, 2H, *J* = 6 Hz), 7.21-7.18 (dd, 1H, *J* = 6 Hz), 7.15-7.14 (m, 1H), 5.70 (s, 1H), 3.73 (s, 2H), 2.31 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.4, 137.4, 135.9, 134.8, 131.1, 129.7, 128.3, 128.0, 127.0, 52.3, 47.7, 21.4; $^{11}B\{^1H\}$

NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.4 (1B), -2.5 (1B), -3.8 (2B), -9.8 (2B), -14.2 (1B), -16.8 (3B); HRMS: calculated for $C_{16}B_{10}H_{24}NO^+$ $(M+H)^+$ 354.2856, found 354.2858.



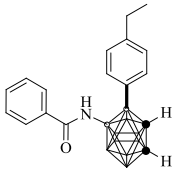
2j

^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.63-7.61 (d, 2H, *J* = 6 Hz), 7.51-7.50 (d, 2H, *J* = 6 Hz), 7.42-7.40 (dd, 1H, *J* = 6 Hz), 7.35-7.32 (dd, 2H, *J* = 6 Hz), 7.12-7.11 (d, 2H, *J* = 6 Hz), 5.71 (s, 1H), 3.73 (s, 1H), 3.69 (s, 1H), 2.32 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.4, 138.8, 135.8, 135.7, 134.1, 131.1, 128.9, 128.3, 127.0, 52.4, 47.7, 21.3; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.4 (1B), -2.4 (1B), -3.6 (2B), -9.9 (2B), -14.3 (1B), -17.0 (3B); HRMS: calculated for $C_{16}H_{22}B_{10}NO^-$ $(M-H)^-$ 354.2637, found 354.2657.



2k

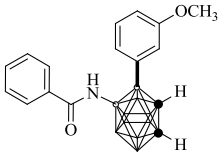
^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.64-7.62 (d, 2H, *J* = 6 Hz), 7.56-7.55 (d, 2H, *J* = 6 Hz), 7.43-7.40 (dd, 1H, *J* = 6 Hz), 7.35-7.33 (dd, 4H, *J* = 6 Hz), 5.70 (s, 1H), 3.72 (s, 1H), 3.70 (s, 1H), 1.29 (s, 9H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 151.9, 135.9, 134.0, 132.7, 131.1, 128.3, 127.1, 125.0, 52.4, 47.5, 34.6, 31.2; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 1.6 (1B), -7.2 (1B), -8.3 (2B), -14.6 (2B), -19.0 (1B), -21.6 (3B); HRMS: calculated for $C_{19}B_{10}H_{30}NO^+(M+H)^+$396.3325, found 396.3327.



2l

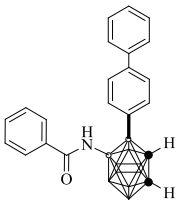
^1H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.61 (d, 2H, *J* = 6 Hz), 7.54-7.53 (d, 2H, *J* = 6 Hz), 7.42-7.40 (dd, 1H, *J* = 6 Hz), 7.35-7.32 (dd, 2H, *J* = 6 Hz), 7.15-7.14 (d, 2H, *J* = 6 Hz), 5.71 (s, 1H), 3.74 (s, 1H), 3.70 (s, 1H), 2.64-2.60 (m, 2H), 1.23-1.20 (t, 3H,

J = 6 Hz); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.4, 145.1, 135.8, 134.2, 132.9, 131.1, 128.3, 127.7, 127.1, 52.4, 47.7, 28.6, 15.3; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -3.5 (2B), -9.8 (2B), -14.2 (1B), -16.9 (3B); HRMS: calculated for $C_{17}B_{10}H_{26}NO^+$ $(M+H)^+$ 368.3012, found 368.3016.



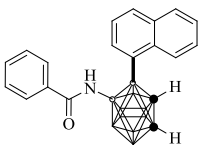
2m

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.64-7.63 (d, 2H, *J* = 6 Hz), 7.43-7.40 (dd, 1H, *J* = 6 Hz), 7.35-7.32 (dd, 2H, *J* = 6 Hz), 7.24-7.21 (dd, 1H, *J* = 6 Hz), 7.19-7.18 (m, 2H), 6.87-6.86 (m, 1H), 5.71 (s, 1H), 3.74 (s, 3H), 3.72 (brs, 2H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.2, 159.1, 135.8, 131.1, 129.2, 128.3, 127.0, 126.3, 119.5, 114.6, 55.1, 52.3, 47.5; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.3 (1B), -3.8 (2B), -9.7 (2B), -14.1 (1B), -16.8 (3B); HRMS: calculated for $C_{16}B_{10}H_{22}NO_2^-$ $(M-H)^-$ 370.2786, found 370.2817.



2n

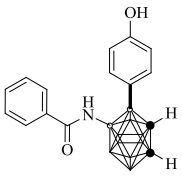
^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.70-7.69 (d, 2H, *J* = 6 Hz), 7.65-7.63 (d, 2H, *J* = 6 Hz), 7.57-7.56 (d, 2H, *J* = 6 Hz), 7.54-7.53 (d, 2H, *J* = 6 Hz), 7.44-7.41 (m, 3H), 7.35-7.32 (m, 3H), 5.74 (s, 1H), 3.76 (s, 2H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 141.6, 140.7, 135.8, 134.6, 131.1, 128.8, 128.4, 127.5, 127.1, 127.0, 126.7, 52.5, 47.6; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.4 (1B), -2.4 (1B), -3.8 (2B), -9.8 (2B), -14.3 (1B), -17.1 (3B); HRMS: calculated for $C_{21}B_{10}H_{26}NO^+(M+H)^+$ 416.3012, found 416.3020.



2o

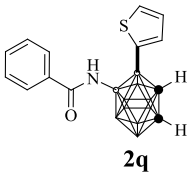
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 8.74-8.73 (d, 1H, *J* = 6 Hz), 7.95-7.94 (d, 1H, *J*

= 6 Hz), 7.85-7.82 (dd, 2H, *J* = 6 Hz,), 7.52-7.49 (m, 3H), 7.47-7.45 (dd, 1H, *J* = 6 Hz), 7.42-7.40 (dd, 1H, *J* = 6 Hz), 7.35-7.33 (dd, 1H, *J* = 6 Hz), 7.25-7.23 (dd, 2H, *J* = 6 Hz), 5.70 (s, 1H), 3.92 (s, 1H), 3.70 (s, 1H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 136.1, 135.8, 133.8, 131.0, 130.2, 129.2, 128.2, 127.1, 127.0, 126.2, 125.4, 125.2, 52.6, 47.3; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.6 (1B), -2.8 (1B), -4.1 (2B), -9.5 (2B), -13.5 (1B), -16.7 (3B); HRMS: calculated for $C_{19}H_{22}B_{10}NO^-$ $(M\text{-}H)^-$ 390.2637, found 390.2665.



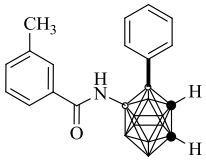
2p

^{1}H NMR (600 MHz, DMSO, *ppm*): δ 9.36 (s, 1H), 7.74-7.73 (d, 2H, *J* = 6 Hz), 7.45-7.43 (dd, 1H, *J* = 6 Hz), 7.38-7.35 (m, 5H), 6.65-6.64 (d, 2H, *J* = 6 Hz), 4.96 (s, 2H); $^{13}C\{^{1}H\}$ NMR (150 MHz, DMSO, *ppm*): δ 170.3, 168.3, 157.7, 135.8, 134.9, 130.7, 127.9, 127.5, 114.7, 53.2, 48.3; $^{11}B\{^{1}H\}$ NMR (192 MHz, DMSO, *ppm*): δ 6.2 (1B), -3.4 (3B), -10.2 (2B), -14.3 (1B), -16.9 (3B); HRMS: calculated for $C_{15}H_{20}B_{10}NO_2^-$ $(M\text{-}H)^-$ 356.2430, found 356.2453.



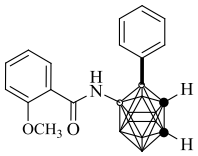
2q

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.65-7.64 (d, 2H, *J* = 6 Hz), 7.56-7.55 (m, 1H), 7.44-7.42 (dd, 1H, *J* = 6 Hz), 7.37-7.34 (d, 2H, *J* = 6 Hz), 7.30-7.29 (m, 1H), 7.26-7.24 (dd, 1H, *J* = 6 Hz), 5.71 (s, 1H), 3.69 (s, 1H), 3.66 (s, 1H); $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.3, 135.8, 132.4, 132.1, 131.2, 128.4, 127.1, 125.8, 52.6, 47.1; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.3 (1B), -2.4 (1B), -5.8 (2B), -9.6 (2B), -14.0 (1B), -16.9 (3B); HRMS: calculated for $C_{13}H_{18}B_{10}NOS^-$ $(M\text{-}H)^-$ 346.2045, found 346.2073.



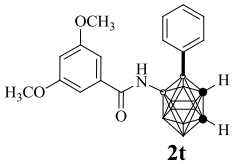
2r

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.61 (d, 2H, *J* = 6 Hz), 7.44 (s, 1H), 7.37 (brs, 1H), 7.34-7.28 (m, 3H), 7.22-7.21 (m, 2H), 5.68 (s, 1H), 3.73 (s, 1H), 3.71 (s, 1H), 2.32 (s, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.5, 138.1, 135.8, 134.2, 131.8, 128.9, 128.2, 128.0, 127.9, 124.0, 52.4, 47.5, 21.3; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.5 (1B), -2.4 (1B), -3.7 (2B), -9.8 (2B), -14.3 (1B), -17.0 (3B); HRMS: calculated for $C_{16}H_{22}B_{10}NO^-$ $(M-H)^-$ 354.2637, found 354.2657.



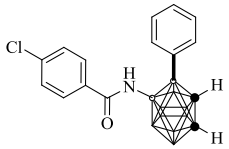
2s

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 8.13-8.11 (m, 1H), 7.64-7.63 (d, 2H, *J* = 6 Hz), 7.49 (s, 1H), 7.35-7.30 (m, 4H), 7.00-6.97 (m, 1H), 6.82-6.81 (d, 1H, *J* = 6 Hz), 3.70 (brs, 2H), 3.56-3.55 (brs, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 166.9, 157.6, 134.2, 133.0, 132.5, 132.4, 128.8, 128.0, 121.1, 111.4, 55.6, 52.0, 47.5; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.7 (1B), -2.6 (1B), -4.3 (2B), -9.6 (2B), -14.0 (1B), -16.8 (3B); HRMS: calculated for $C_{16}H_{22}B_{10}NO_2^-$ $(M-H)^-$ 370.2587, found 370.2621.



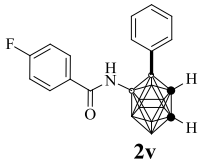
2t

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.62-7.60 (d, 2H, *J* = 6 Hz), 7.34-7.29 (m, 3H), 6.74 (brs, 2H), 6.49 (brs, 1H), 5.64 (s, 1H), 3.75 (s, 6H), 3.72-3.68 (brs, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 169.1, 160.6, 138.1, 134.1, 129.0, 128.1, 104.9, 103.4, 55.5, 52.3, 47.6; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.3 (1B), -2.5 (1B), -3.8 (2B), -9.8 (2B), -14.3 (1B), -17.0 (3B); HRMS: calculated for $C_{17}H_{24}B_{10}NO_3^-$ $(M-H)^-$ 400.2692, found 400.2720.



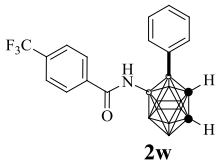
2u

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.61-7.59 (d, 2H, *J* = 6 Hz), 7.54-7.53 (d, 2H, *J* = 6 Hz), 7.35-7.29 (m, 5H), 5.63 (s, 1H), 3.74 (brs, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 168.3, 137.3, 134.2, 134.1, 129.0, 128.6, 128.5, 128.1, 52.4, 47.8; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.3 (1B), -2.4 (1B), -3.7 (2B), -9.9 (2B), -14.4 (1B), -17.0 (3B); HRMS: calculated for $C_{15}H_{19}B_{10}ClNO^{-}$(M-H)$^{-}$ 374.2091, found 374.2123.



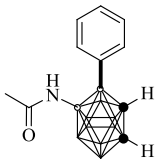
2v

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.61-7.60 (m, 4H), 7.35-7.29 (m, 3H), 7.01-6.98 (dd, 2H, *J* = 6 Hz), 5.62 (s, 1H), 3.73 (brs, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 168.3, 165.3-163.7 (d, *J* = 250Hz), 134.1, 129.4-129.3 (d, *J* = 9 Hz), 129.0, 128.1, 115.4-115.2 (d, *J* = 22 Hz), 52.4, 47.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.4 (1B), -2.4 (1B), -3.8 (2B), -9.8 (2B), -14.4 (1B), -17.0 (3B); HRMS: calculated for $C_{15}H_{19}B_{10}FNO^{-}$ (M-H)$^{-}$ 358.2387, found 358.2400.



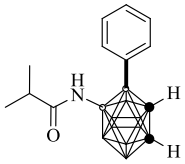
2w

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.70-7.69 (d, 2H, *J* = 6 Hz), 7.62-7.61 (d, 2H, *J* = 6 Hz), 7.59-7.58 (d, 2H, *J* = 6 Hz), 7.36-7.30 (m, 3H), 5.67 (s, 1H), 3.75 (s, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 168.1, 139.2, 134.1, 133.1-132.4 (m, *J* = 30 Hz), 129.1, 128.1, 127.5, 125.4 (m, *J* = 3Hz), 52.4, 47.9; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.3 (1B), -2.4 (1B), -3.7 (2B), -9.8 (2B), -14.3 (1B), -16.9 (3B); HRMS: calculated for $C_{16}H_{19}B_{10}F_3NO^{-}$ (M-H)$^{-}$ 408.2355, found 408.2373.



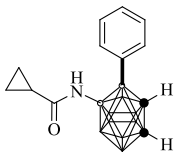
2x

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.59-7.58 (d, 2H, *J* = 6 Hz), 7.37-7.31 (m, 3H), 5.00 (s, 1H), 3.70 (s, 1H), 3.65 (s, 1H), 1.85 (s, 3H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 172.4, 134.2, 133.7, 128.9, 128.0, 52.3, 47.4, 25.0; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.1 (1B), -2.5 (1B), -3.8 (2B), -9.9 (2B), -14.4 (1B), -17.0 (3B); HRMS: calculated for $C_{10}B_{10}H_{20}NO^{+}$ $(M+H)^{+}$ 278.2543, found 278.2546.



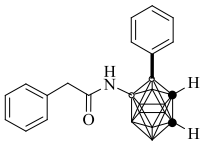
2y

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.57-7.56 (d, 2H, *J* = 6 Hz), 7.35-7.29 (m, 3H), 4.94 (s, 1H), 3.70 (s, 1H), 3.67 (s, 1H), 2.20-2.14 (m, 1H), 1.01-0.99 (d, 3H, *J* = 6 Hz), 0.95-0.94 (d, 3H, *J* = 6 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 179.3, 134.2, 128.9, 127.9, 52.3, 47.3, 36.8, 19.8, 19.5; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.3 (1B), -2.5 (1B), -3.7 (2B), -9.9 (2B), -14.5 (1B), -17.2 (3B); HRMS: calculated for $C_{12}H_{22}B_{10}NO^{-}$ $(M-H)^{-}$ 306.2637, found 306.2655.



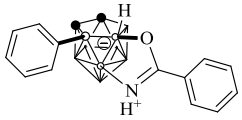
2z

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.59-7.58 (d, 2H, *J* = 6 Hz), 7.37-7.31 (m, 3H), 5.17 (s, 1H), 3.68 (s, 1H), 3.64 (s, 1H), 1.19-1.15 (m, 1H), 0.88 (s, 1H), 0.81 (s, 1H), 0.62-0.56 (m, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 175.6, 134.3, 128.9, 128.0, 52.3, 47.2, 16.0, 7.5, 7.3; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 6.2 (1B), -2.5 (1B), -3.9 (2B), -9.9 (2B), -14.4 (1B), -17.1 (3B); HRMS: calculated for $C_{12}H_{20}B_{10}NO^{-}$ $(M-H)^{-}$ 304.2481, found 304.2500.



2za

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 7.47-7.45 (d, 2H, *J* = 6 Hz), 7.37-7.34 (dd, 1H, *J* =6 Hz), 7.29-7.26 (dd, 2H, *J* = 6 Hz), 7.20-7.19 (m, 3H), 6.99-6.98 (m, 2H), 4.97 (s, 1H), 3.66 (s, 1H), 3.63 (s, 1H), 3.44-3.42 (m, 2H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 173.4, 135.3, 134.0, 129.3, 128.9, 128.7, 128.0, 126.9, 52.2, 47.7, 45.0; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 1.4 (1B), -7.5 (1B), -8.8 (2B), -14.9 (2B), -19.3 (1B), -22.5 (3B); HRMS: calculated for $C_{16}H_{22}B_{10}NO^-$ $(M-H)^-$ 354.2637, found 354.2660.



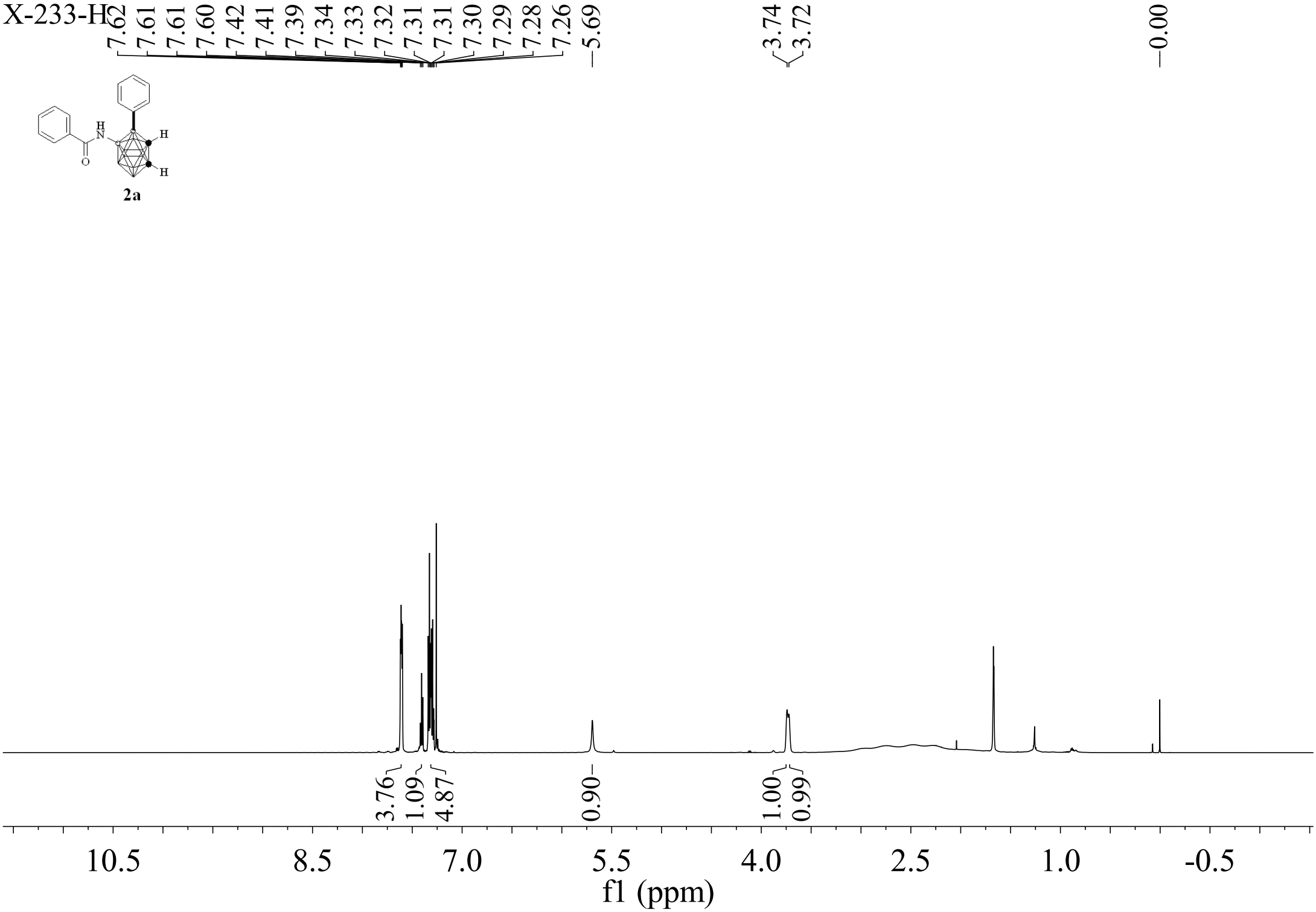
3a

^{1}H NMR (600 MHz, $CDCl_3$, *ppm*): δ 8.09 (brs, 1H), 7.80-7.79 (d, 2H, *J* = 6 Hz), 7.71-7.66 (m, 3H), 7.54-7.52 (d, 2H, *J* = 6 Hz), 7.32-7.29 (m, 3H), 2.37 (s, 1H), 2.07 (s, 1H); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, *ppm*): δ 179.1, 135.2, 134.6, 129.4, 128.2, 127.5, 127.3, 125.3, 42.5, 38.0. ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ -8.3 (1B), -9.7 (1B), -11.9 (1B), -18.6 (1B), -19.9 (1B), -20.5 (1B), -25.7 (1B), -29.6 (1B), -43.3 (1B); HRMS: calculated for $C_{15}H_{19}B_9NO^-$ $(M-H)^-$ 328.2310, found 328.2328.

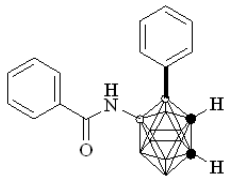
X-233-H

H
N
O
H
H

2a

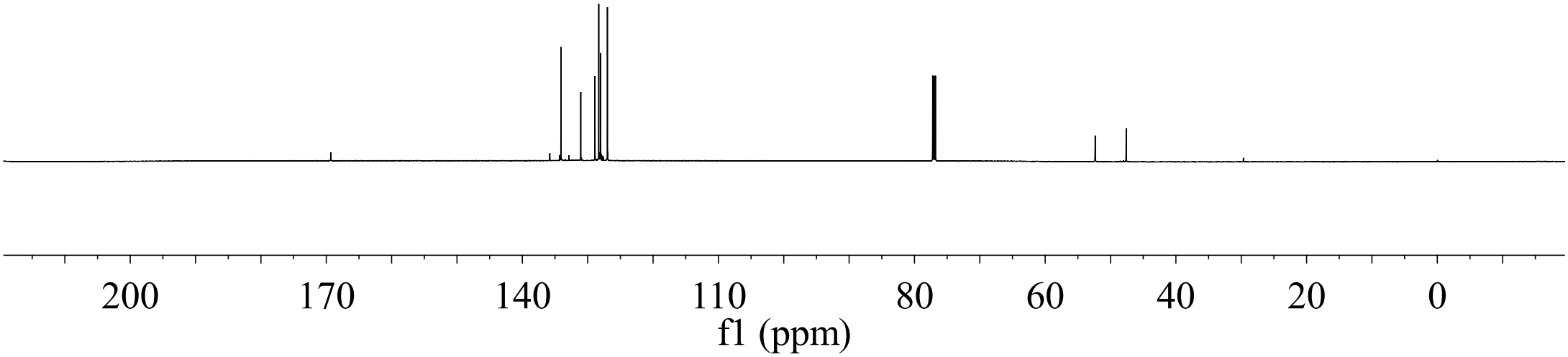

7.62
7.61
7.61
7.60
7.42
7.41
7.39
7.34
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.26
5.69
3.74
3.72
0.00
3.76
1.09
4.87
0.90
1.00
0.99
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-233-C



2a

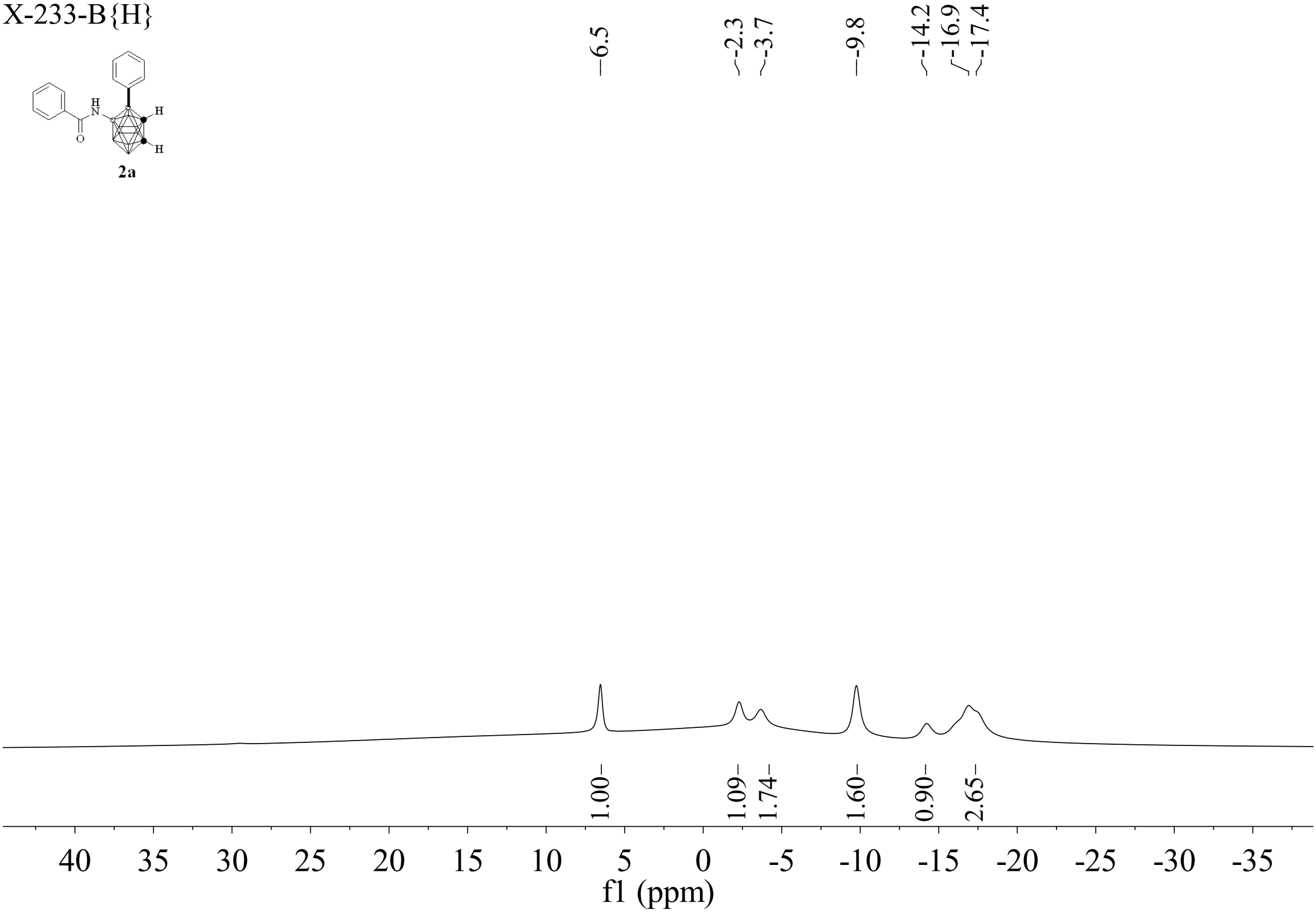
169.3
135.8
134.1
132.9
131.1
128.9
128.3
128.0
127.0
77.2
77.0
76.8
52.3
47.6
0.0



X-233-B{H}

H
N
O
H
H

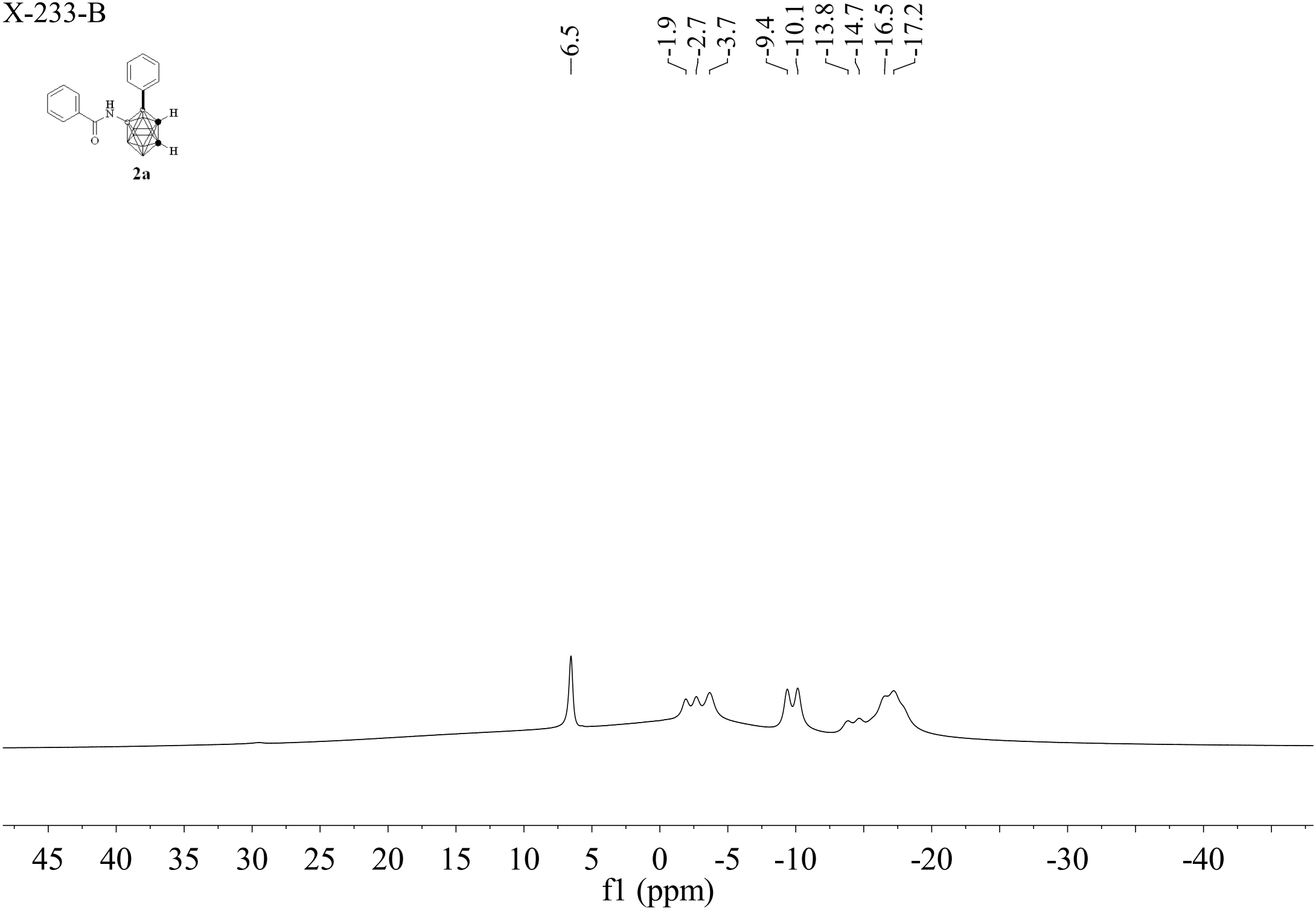
2a


-6.5
-2.3
-3.7
-9.8
-14.2
-16.9
-17.4
1.00
1.09
1.74
1.60
0.90
2.65
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

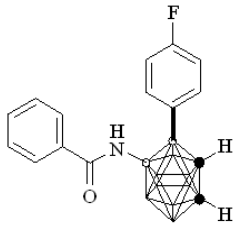
X-233-B

H
N
O
H
H

2a


-6.5
-1.9
-2.7
-3.7
-9.4
-10.1
-13.8
-14.7
-16.5
-17.2
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-270-H



2b

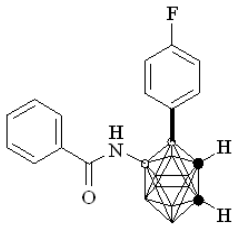
7.61 7.60 7.60 7.59 7.58 7.43 7.42 7.41 7.35 7.34 7.33 7.26 6.99 6.98 6.96 5.68 3.74 3.71 0.00

3.83 1.05 2.06 1.94 0.96 1.00 0.98

10.5 8.5 7.0 5.5 4.0 2.5 1.0 -0.5

f1 (ppm)

X-270-C



2b

169.4
164.5
162.8
136.1
136.1
135.7
131.2
128.4
127.0
115.2
115.0
77.2
77.0
76.8
52.6
47.7
0.0

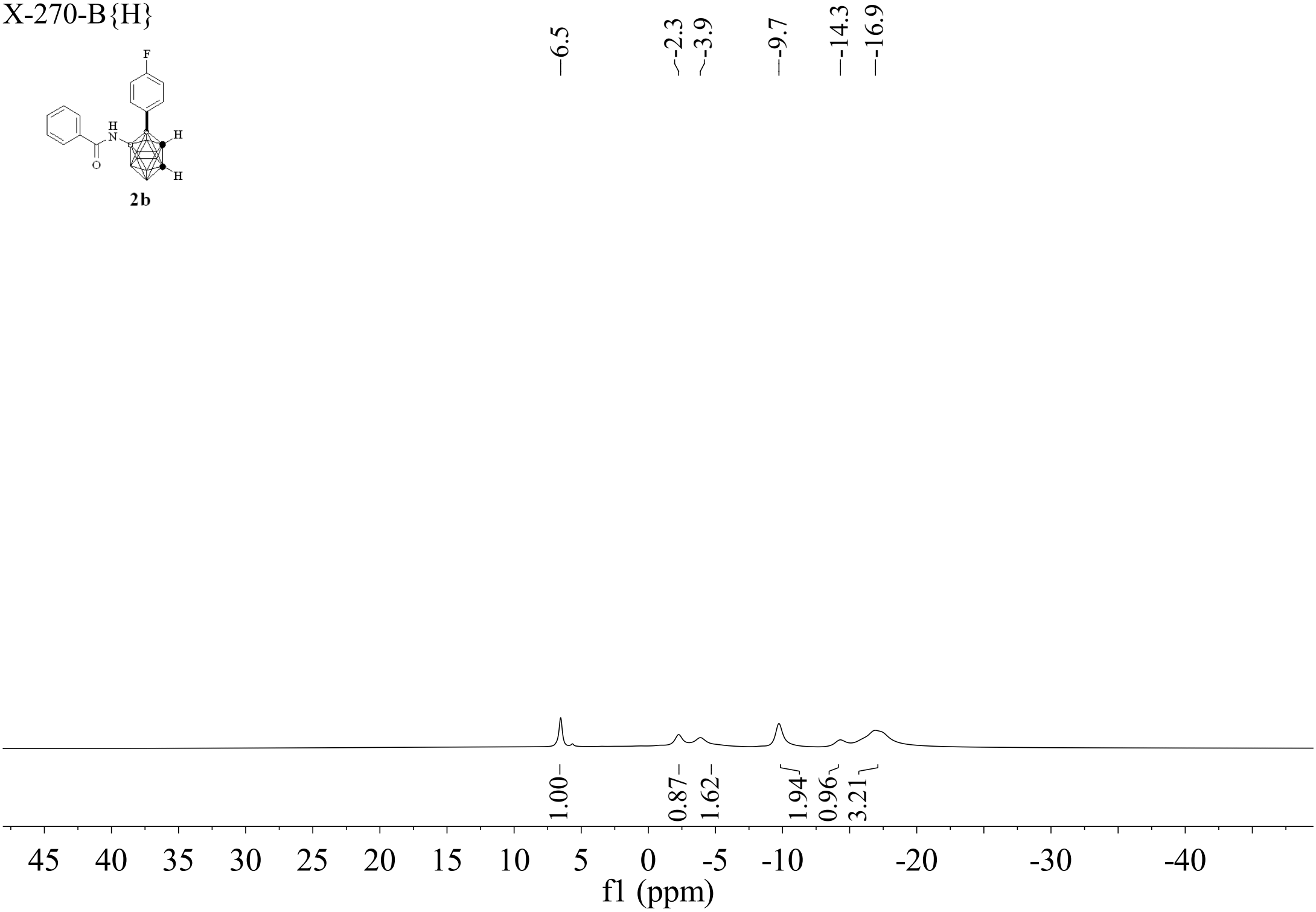
200 170 140 110 80 60 40 20 0

f1 (ppm)

X-270-B{H}

F
H
N
O
H
H

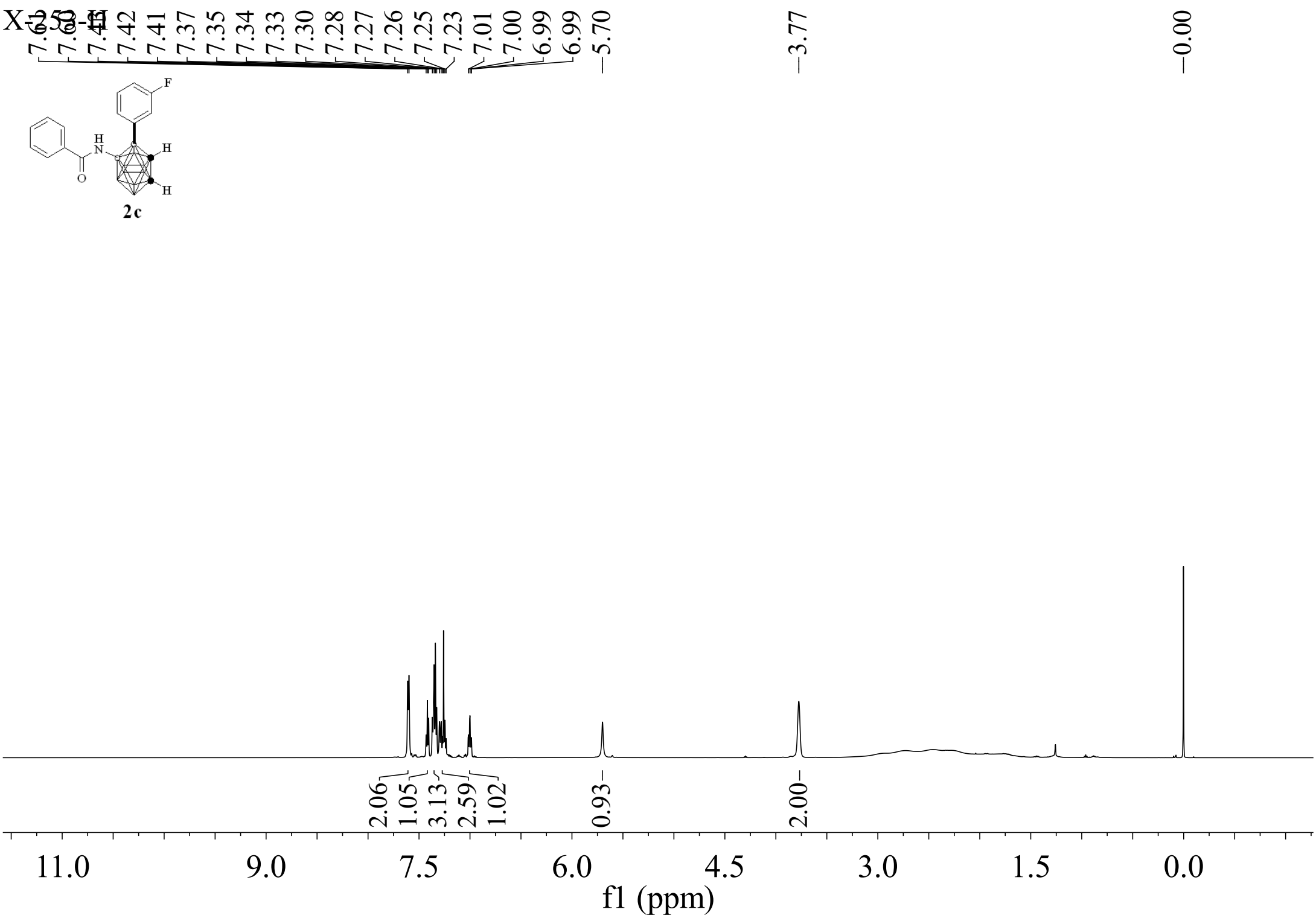
2b


-6.5
-2.3
-3.9
-9.7
-14.3
-16.9
1.00
0.87
1.62
1.94
0.96
3.21
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

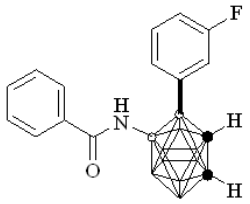
X-253-H

F
H
N
O
H
H

2c

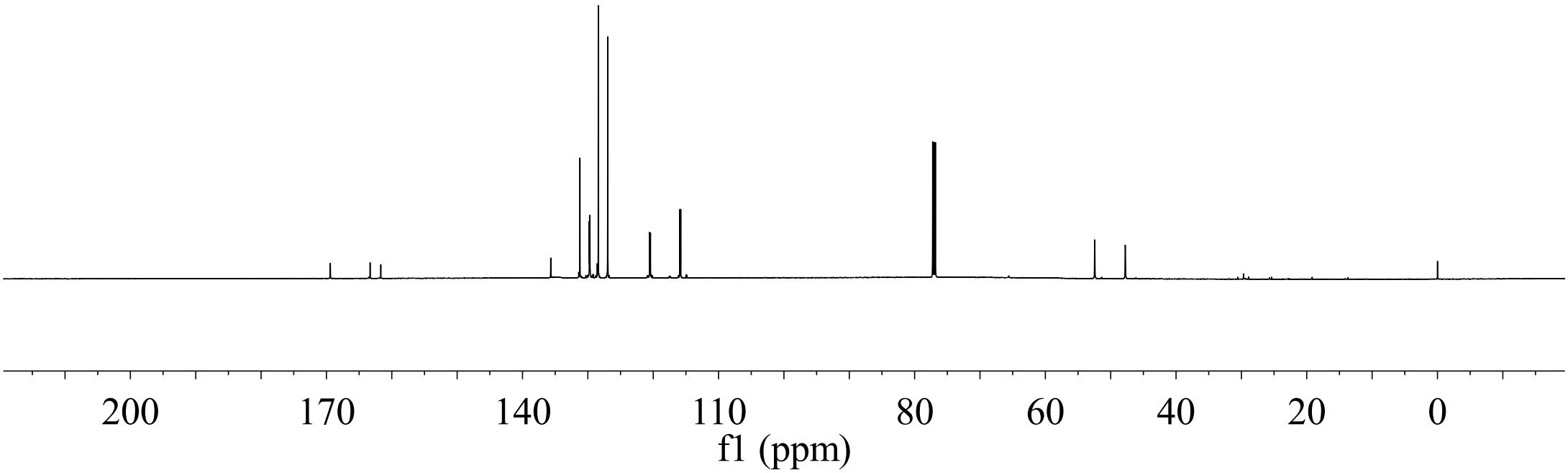

7.63
7.45
7.42
7.41
7.37
7.35
7.34
7.33
7.30
7.28
7.27
7.26
7.25
7.23
7.01
7.00
6.99
6.99
5.70
3.77
0.00
2.06
1.05
3.13
2.59
1.02
0.93
2.00
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-253-C



2c

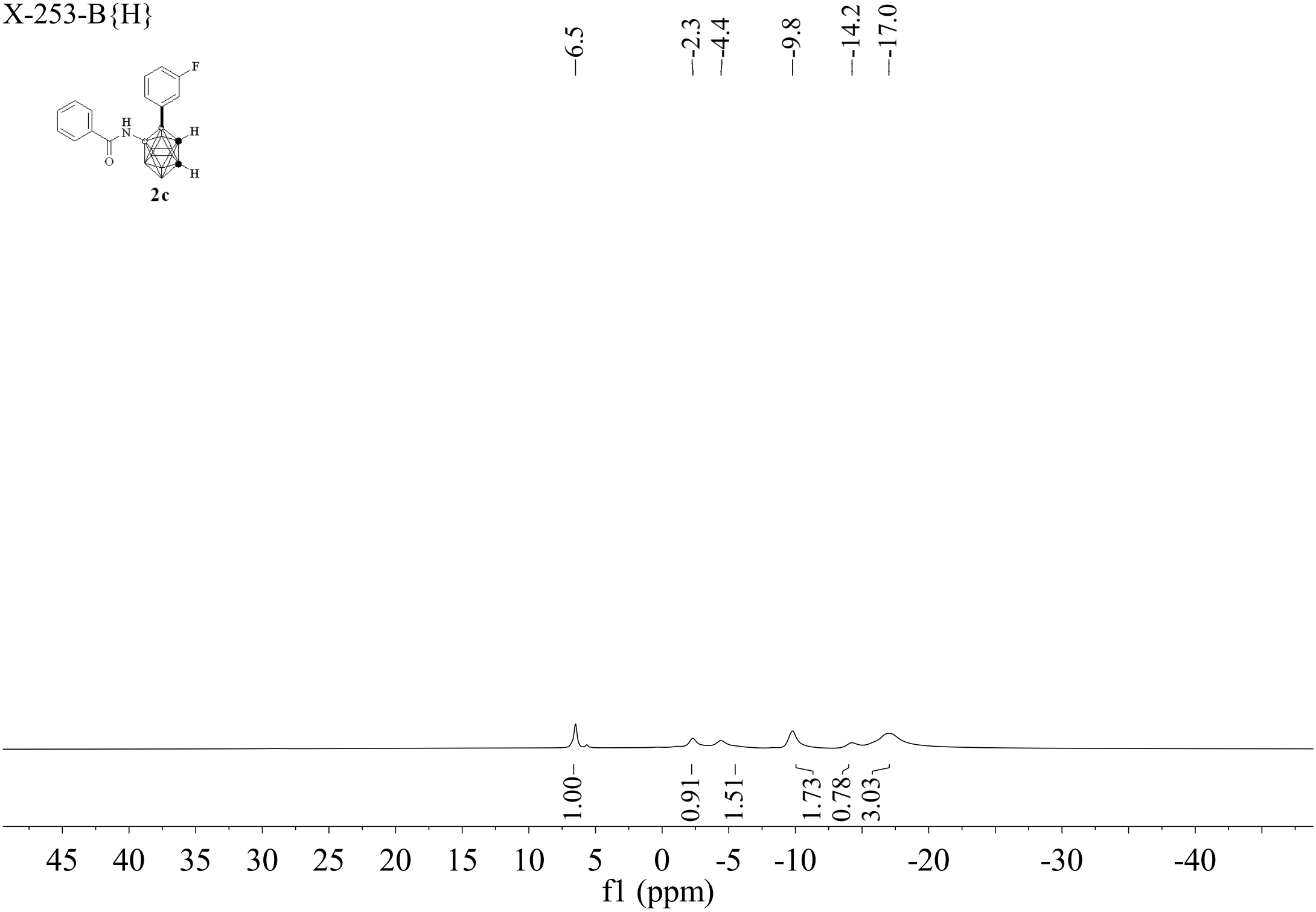
169.4
163.3
161.7
135.7
131.2
129.8
129.7
129.7
128.4
127.0
120.6
120.4
115.9
115.8
77.2
77.0
76.8
52.4
47.8
0.0



X-253-B{H}

F
H
N
O
H
H

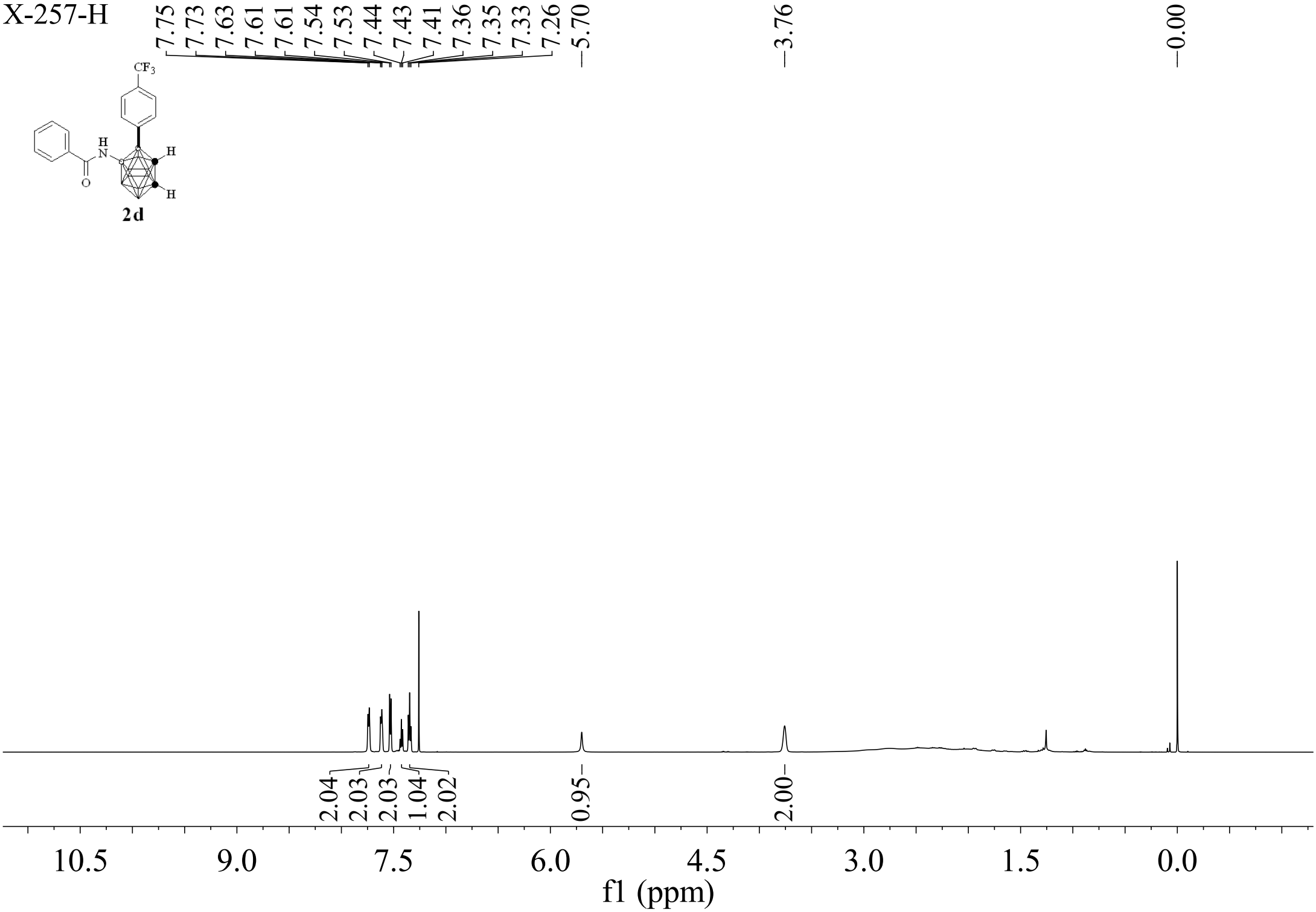
2c


-6.5
-2.3
-4.4
-9.8
-14.2
-17.0
1.00
0.91
1.51
1.73
0.78
3.03
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

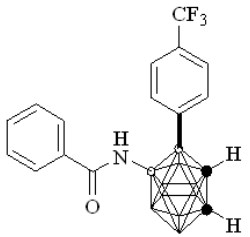
X-257-H

CF3
H
N
O
H
H

2d

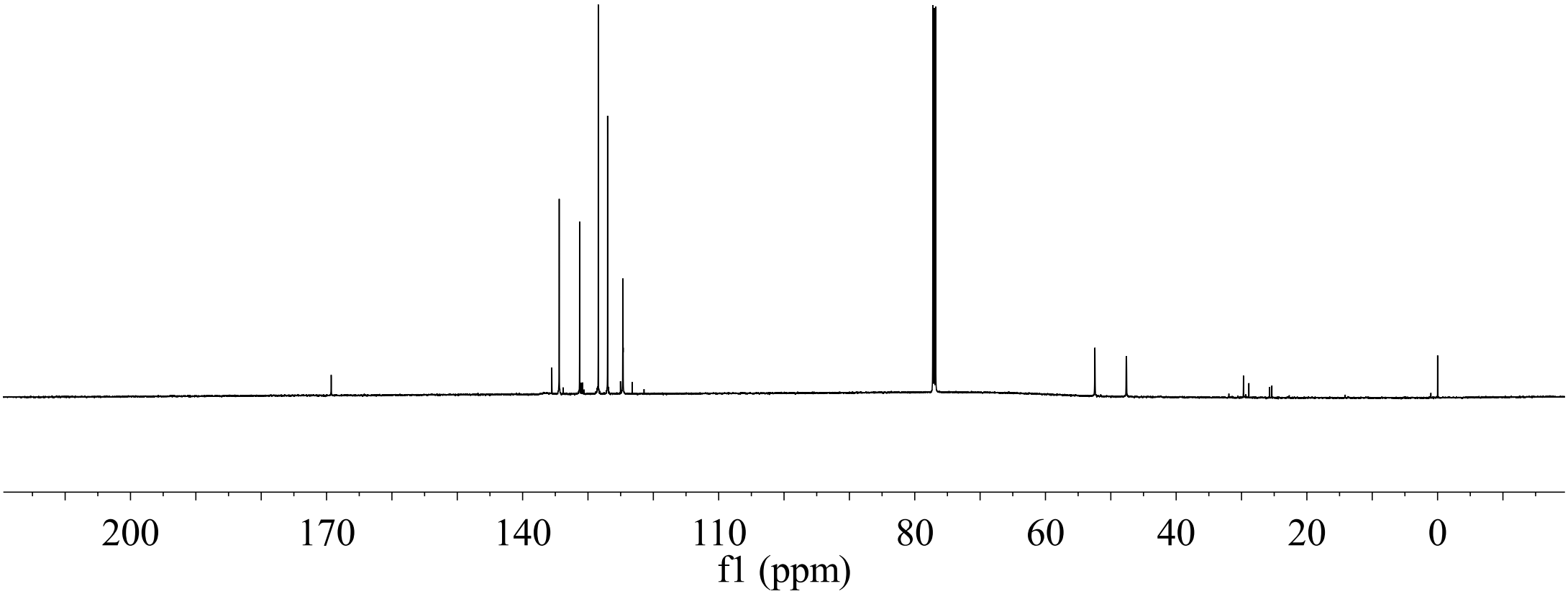

7.75
7.73
7.63
7.61
7.61
7.54
7.53
7.44
7.43
7.41
7.36
7.35
7.33
7.26
5.70
3.76
0.00
2.04
2.03
2.03
1.04
2.02
0.95
2.00
10.5
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-257-C



2d

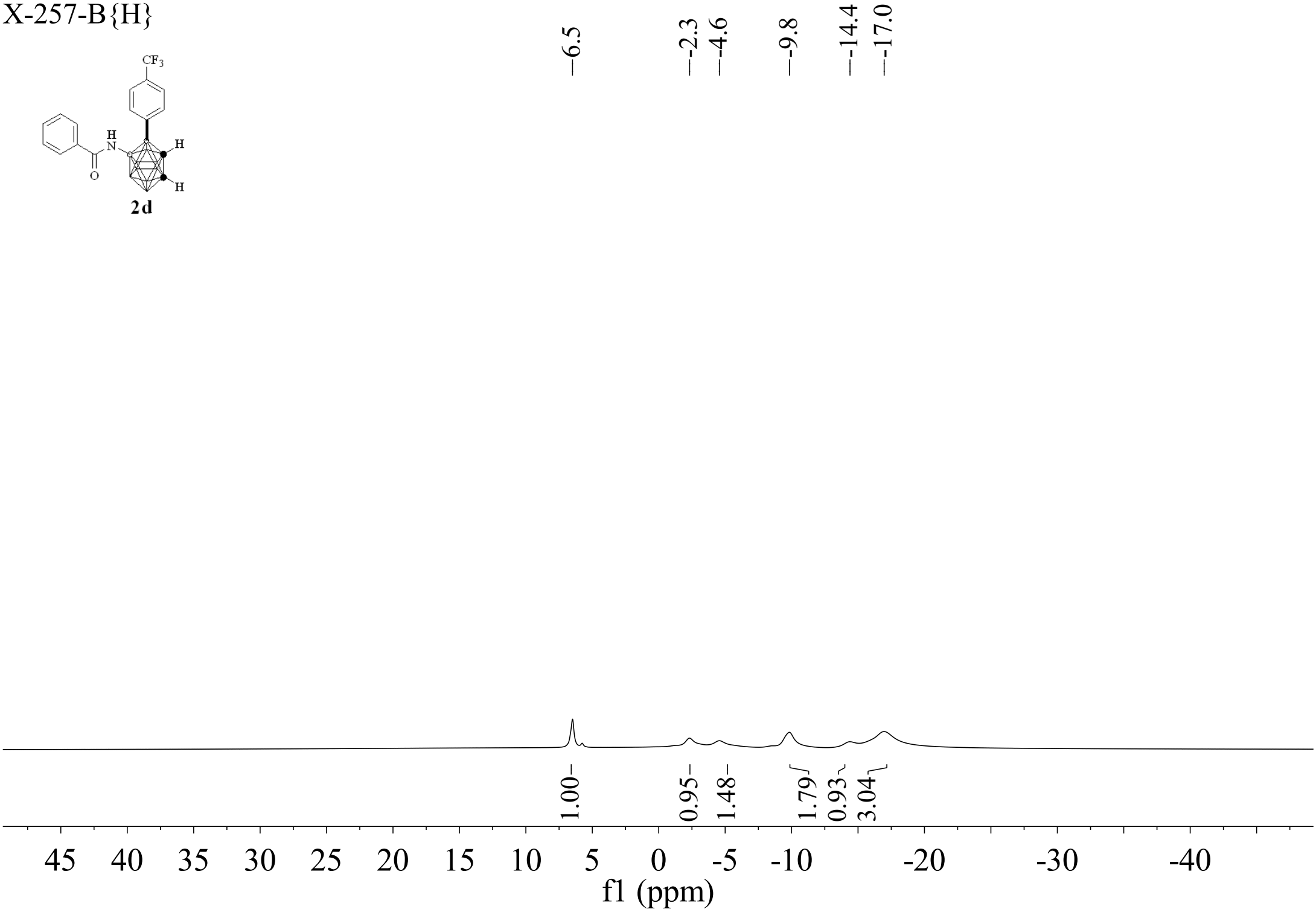
169.3
135.5
134.4
131.3
131.2
131.0
130.8
130.6
128.4
127.0
124.7
124.7
124.6
124.6
77.2
77.0
76.8
52.4
47.6



X-257-B{H}

CF3
H
N
O
H
H

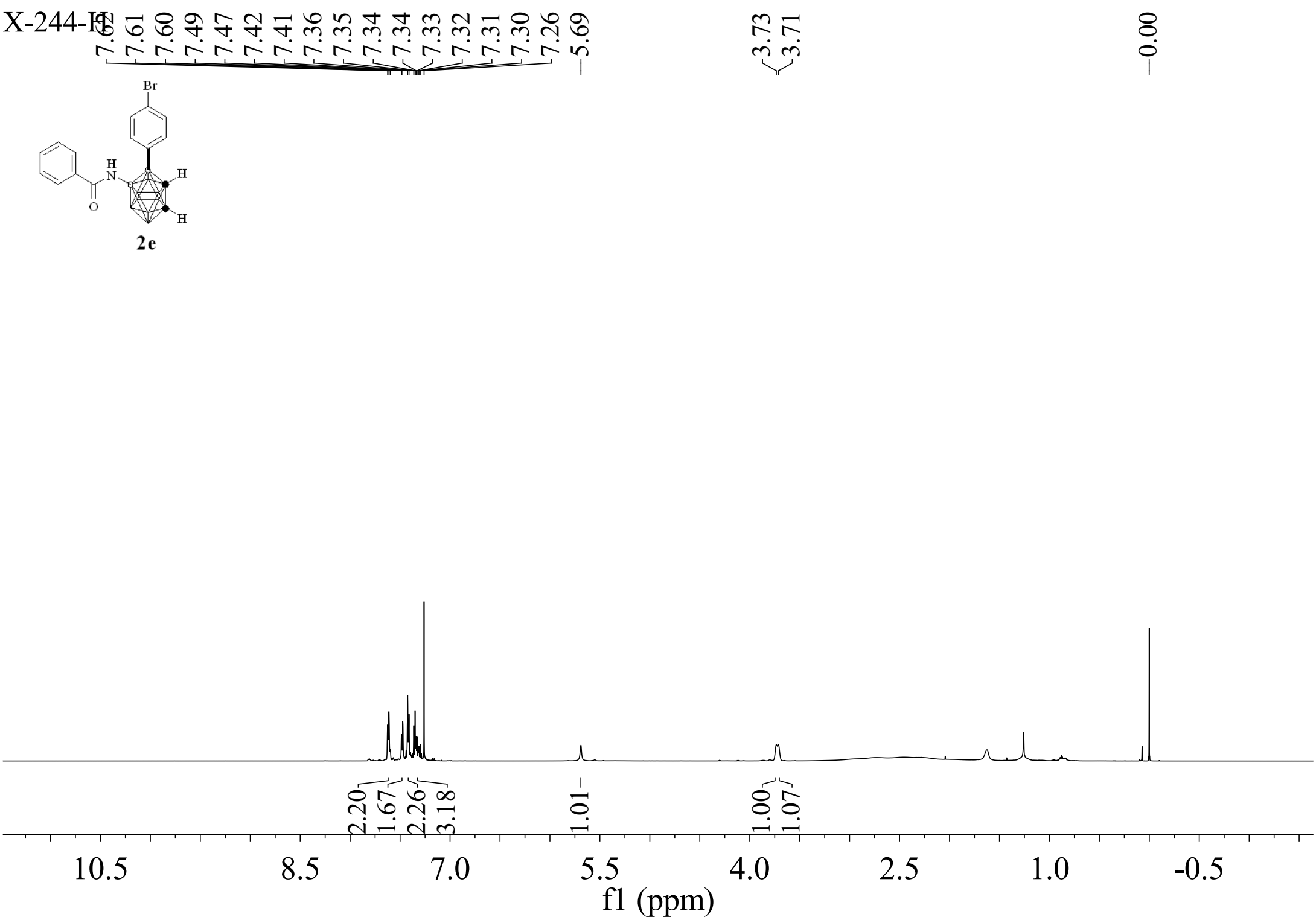
2d


-6.5
-2.3
-4.6
-9.8
-14.4
-17.0
1.00
0.95
1.48
1.79
0.93
3.04
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

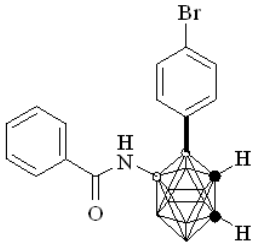
X-244-H1

Br
H
N
O
H
H

2e

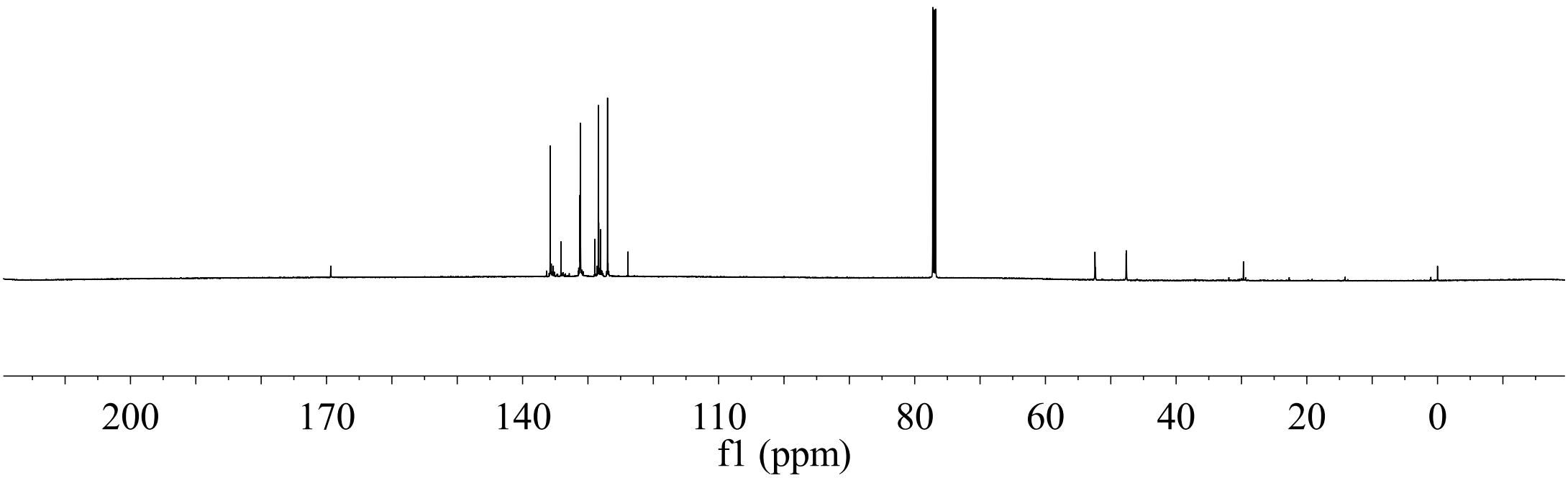

7.62
7.61
7.60
7.49
7.47
7.42
7.41
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.30
7.26
5.69
3.73
3.71
0.00
2.20
1.67
2.26
3.18
1.01
1.00
1.07
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-244-C



2e

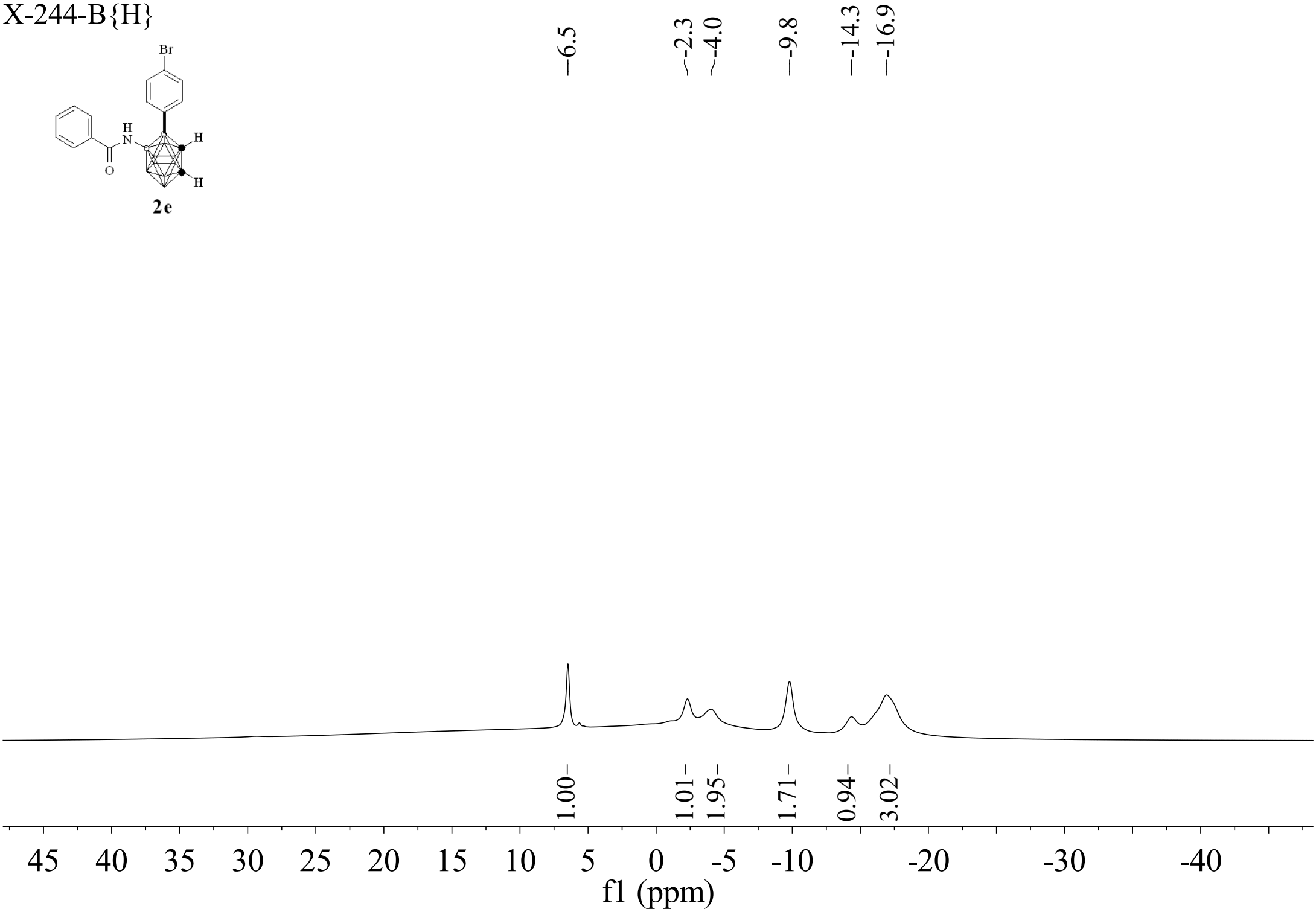
169.3
135.8
134.1
131.2
131.2
129.0
128.4
127.0
123.9
77.2
77.0
76.8
52.4
47.6
0.0



X-244-B{H}

Br
H
N
O
H
H

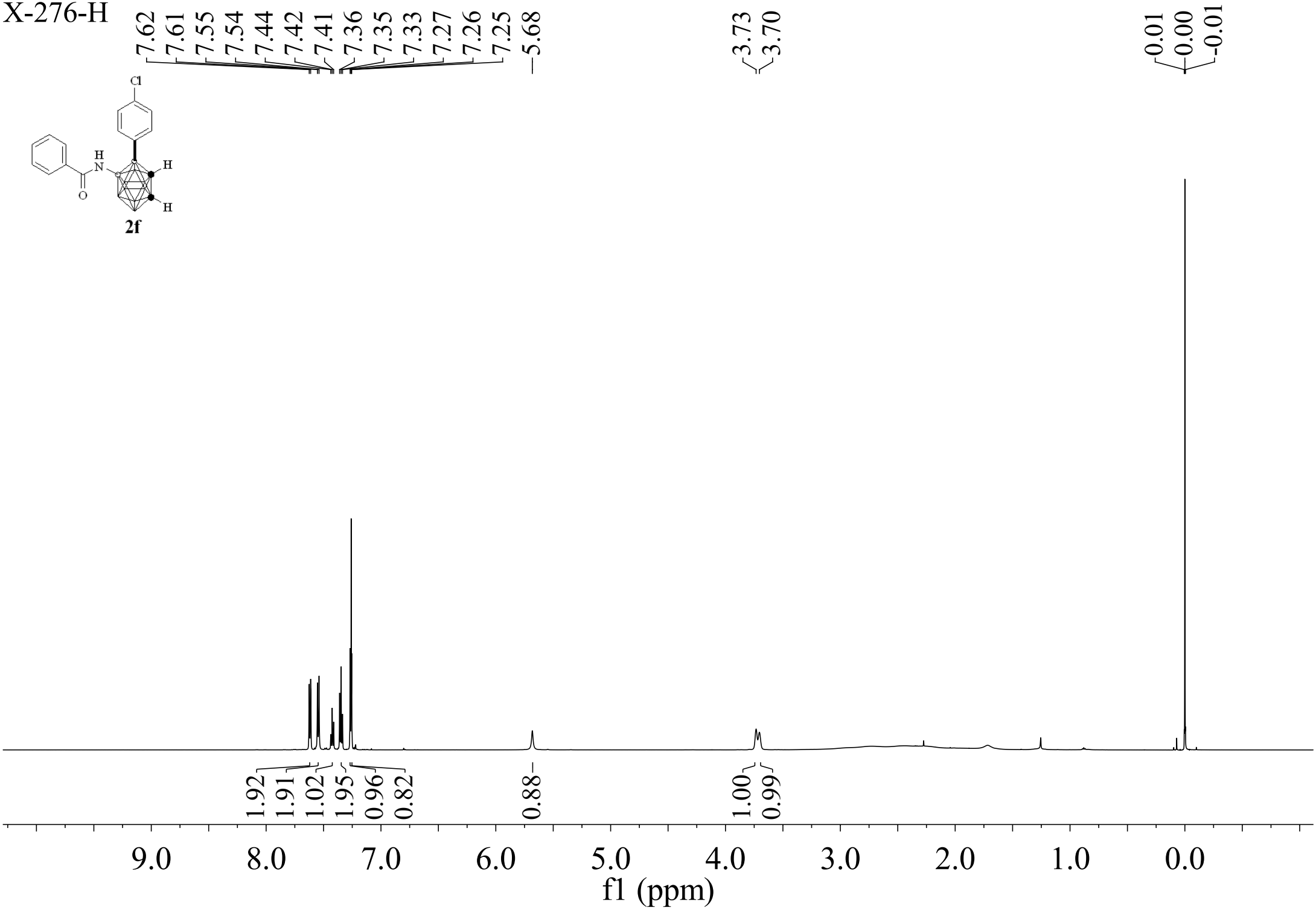
2e


-6.5
-2.3
-4.0
-9.8
-14.3
-16.9
1.00
1.01
1.95
1.71
0.94
3.02
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

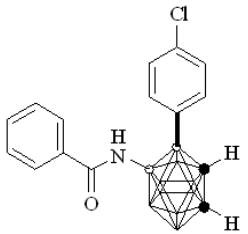
X-276-H

Cl
H
N
O
H
H

2f

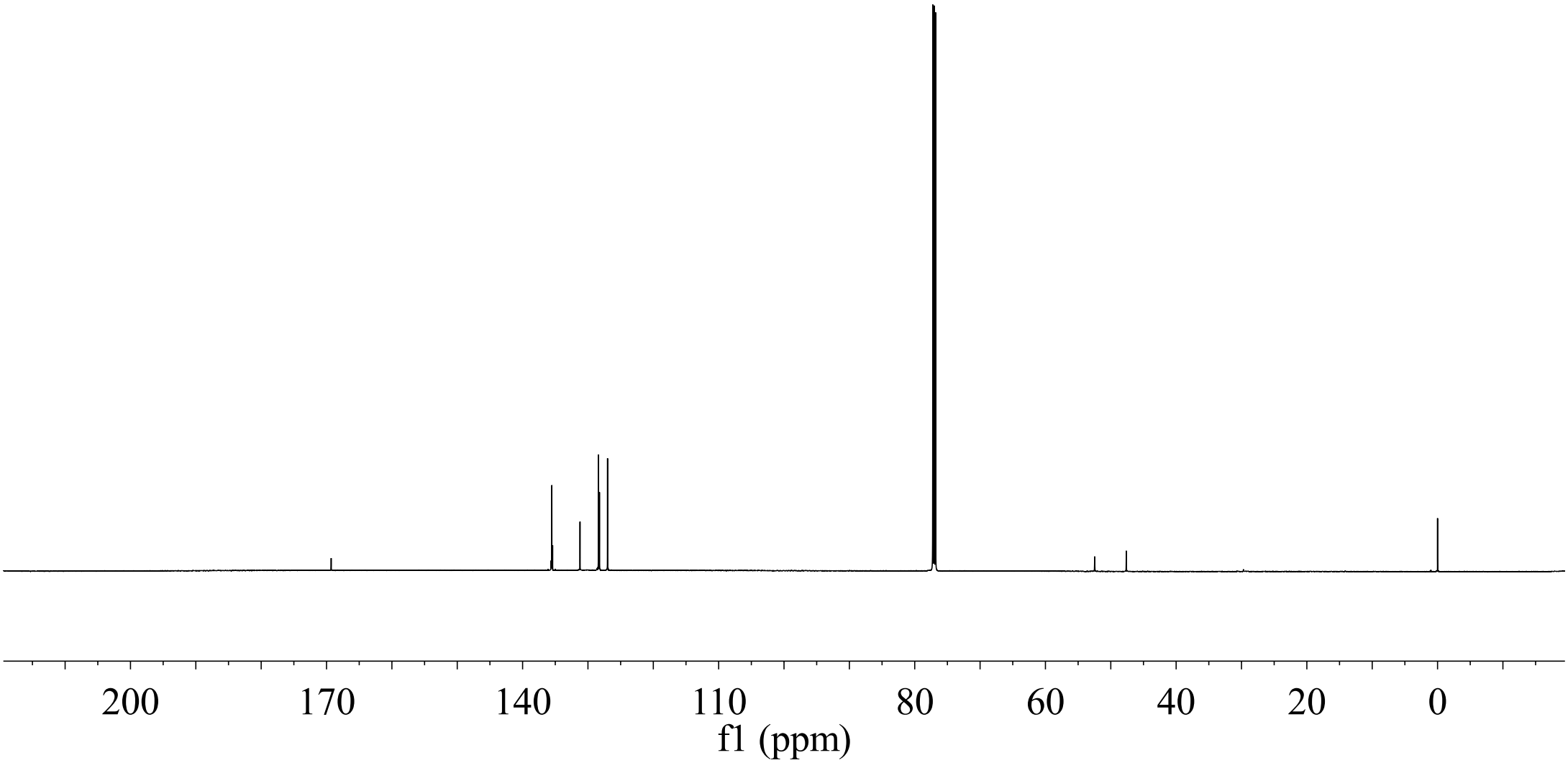

7.62
7.61
7.55
7.54
7.44
7.42
7.41
7.36
7.35
7.33
7.27
7.26
7.25
5.68
3.73
3.70
0.01
0.00
-0.01
1.92
1.91
1.02
1.95
0.96
0.82
0.88
1.00
0.99
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

X-276-C

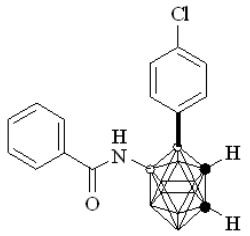


2f

169.3
135.6
135.5
135.4
131.2
128.4
128.2
127.0
77.2
77.0
76.8
52.5
47.6
0.0



X-276-B{H}



2f

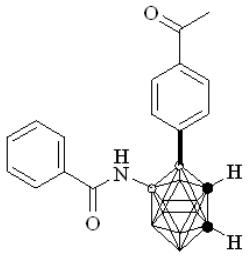
-6.5
-2.3
-4.1
-9.8
-14.4
-17.0

1.00
1.08
1.63
1.93
0.84
2.92

45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40

f1 (ppm)

X-282-H



2g

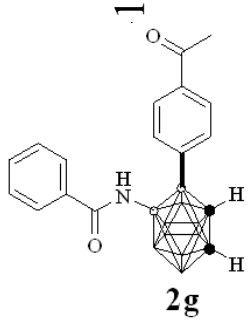
7.83
7.82
7.69
7.68
7.60
7.59
7.42
7.42
7.41
7.40
7.34
7.32
7.31
7.26
5.73
3.88
3.82
2.54
0.00

1.96
1.92
1.93
1.16
1.94
0.88
1.00
0.96
2.99

11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0

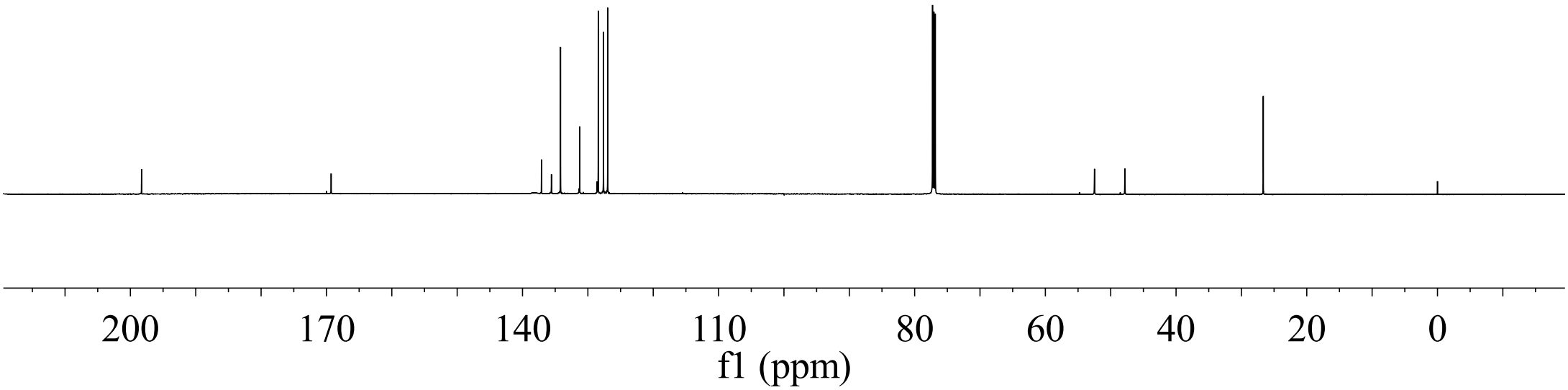
f1 (ppm)

X-282-C



2g

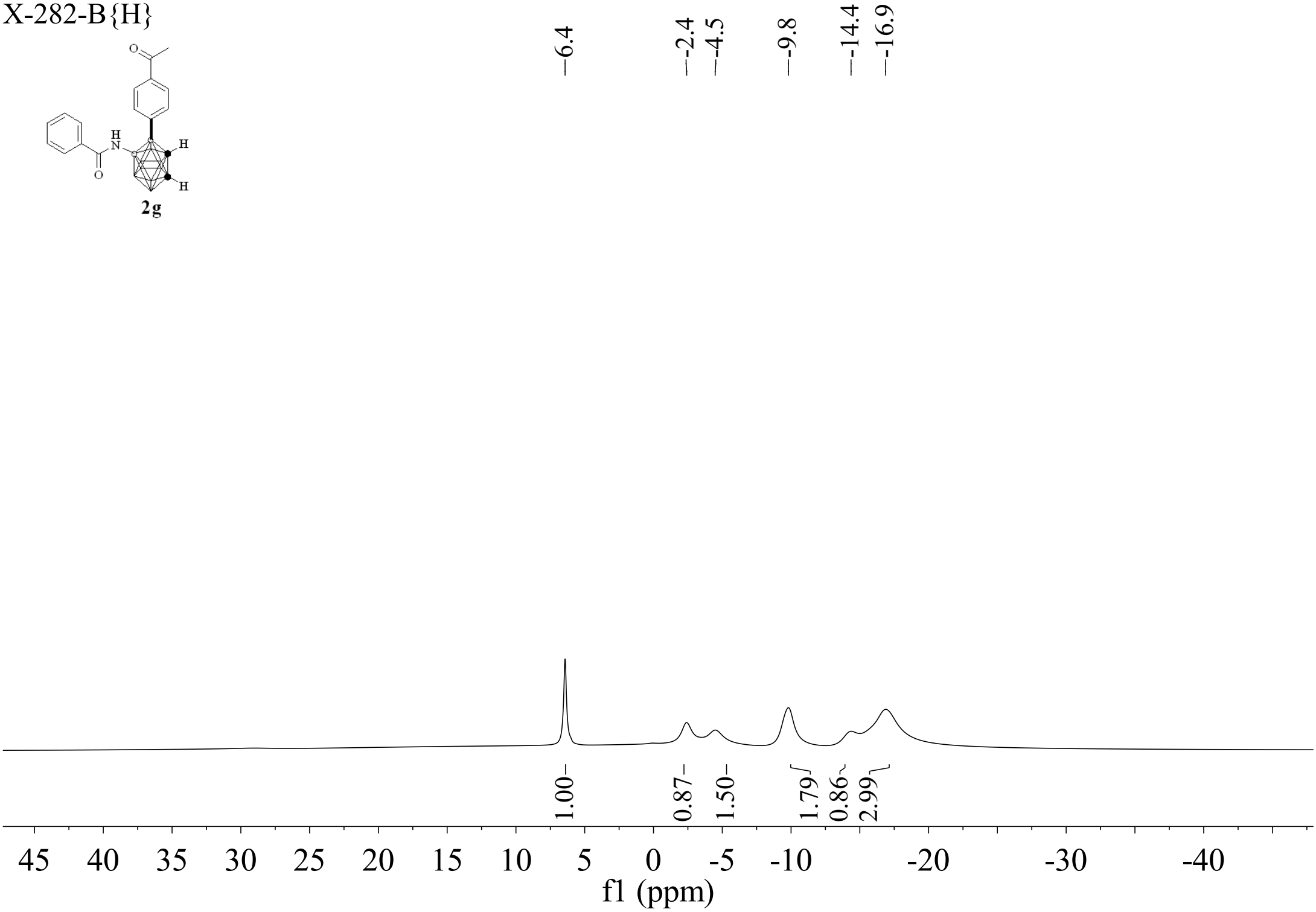
198.3
169.3
137.1
135.6
134.2
131.3
128.4
127.6
127.0
77.3
77.1
76.8
52.5
47.8
26.7
0.0

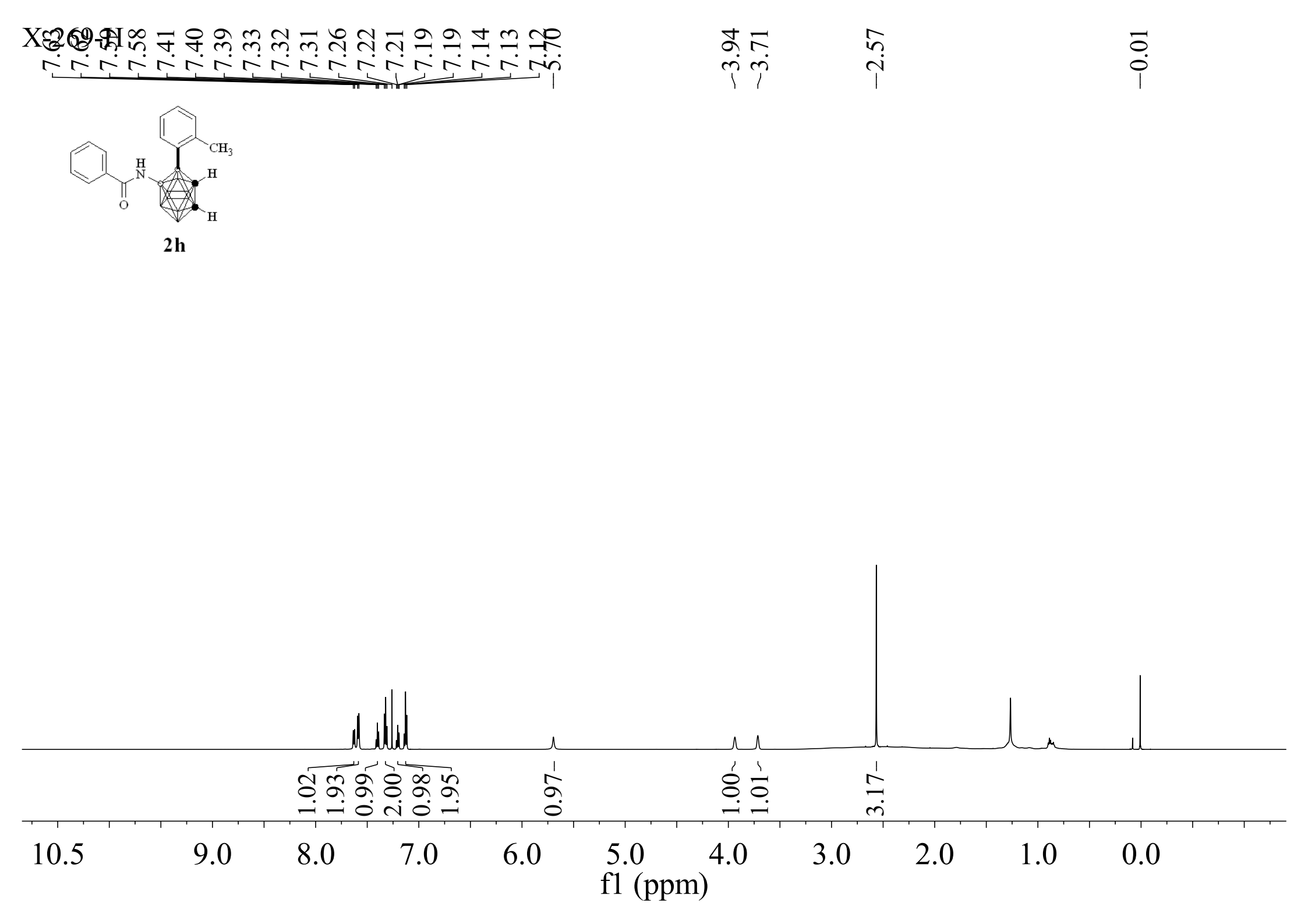


X-282-B{H}

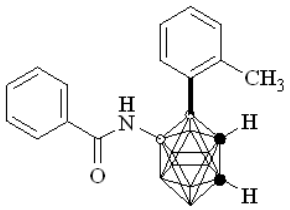
O
H
N
O
H
H

2g


-6.4
-2.4
-4.5
-9.8
-14.4
-16.9
1.00
0.87
1.50
1.79
0.86
2.99
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

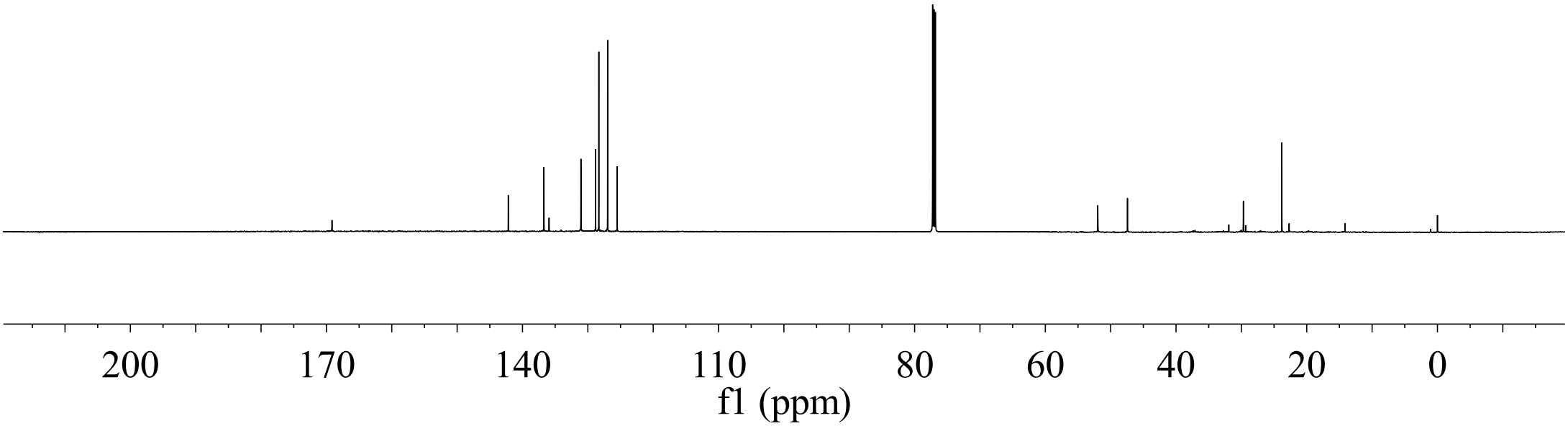

X3269-H
7.63
7.62
7.59
7.58
7.41
7.40
7.39
7.33
7.32
7.31
7.26
7.22
7.21
7.19
7.19
7.14
7.13
7.12
5.70
3.94
3.71
2.57
0.01
CH3
H
N
O
H
H
2h
1.02
1.93
0.99
2.00
0.98
1.95
0.97
1.00
1.01
3.17
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

X-269-C



2h

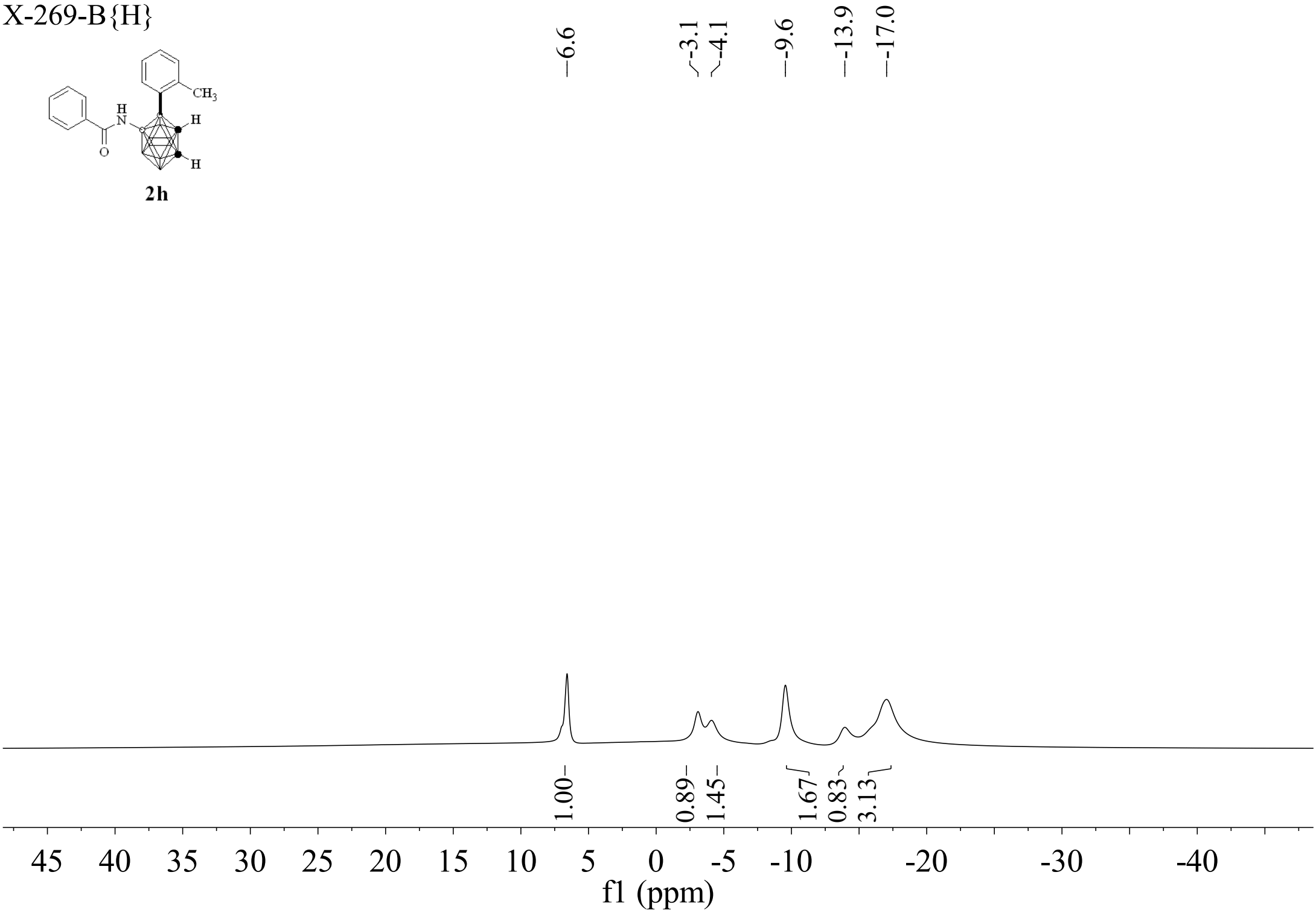
169.1
142.2
136.7
135.9
131.1
131.0
128.8
128.3
127.0
125.5
77.2
77.0
76.8
52.0
47.4
23.8
0.0



X-269-B{H}

CH3
H
N
O
H
H

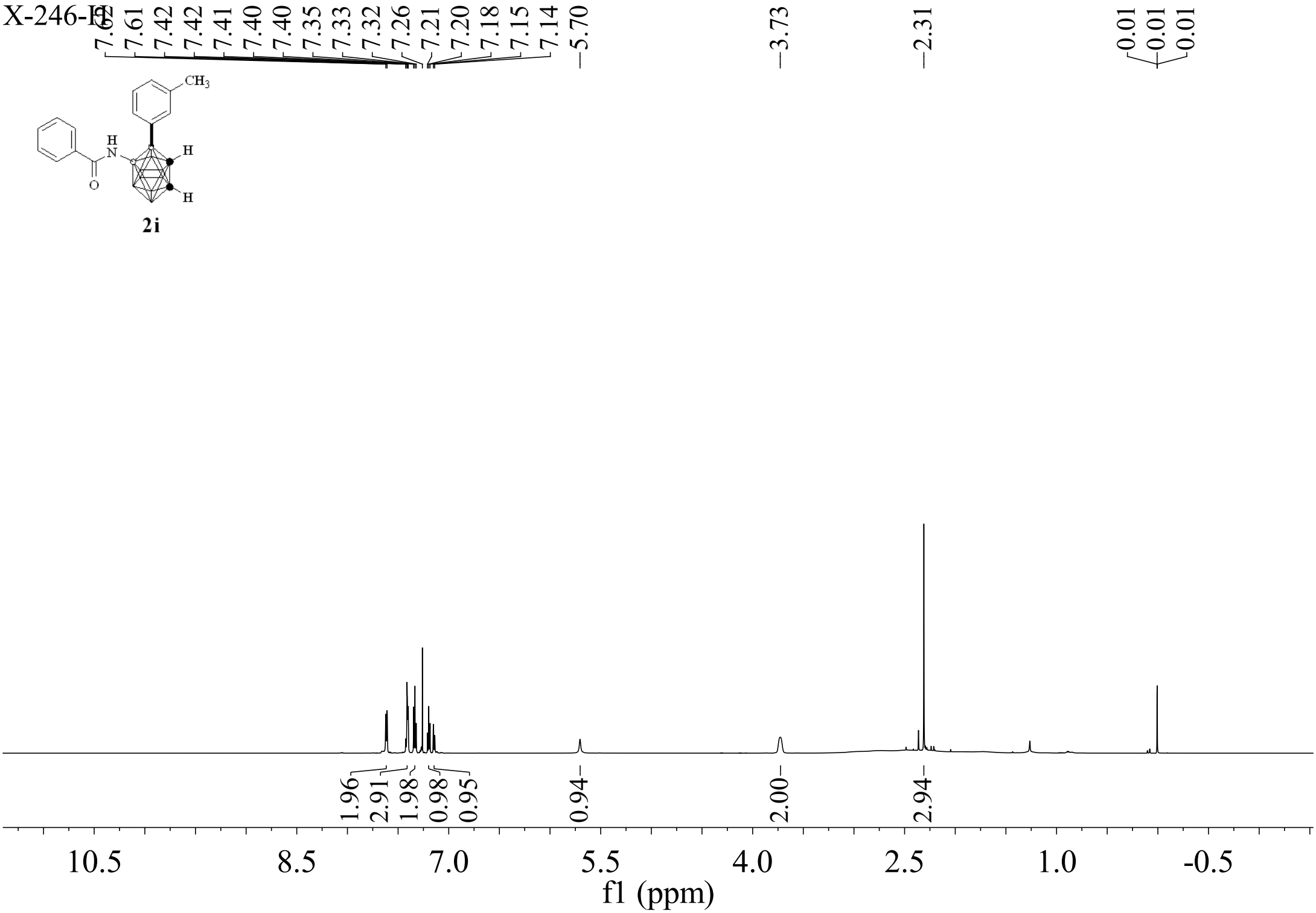
2h


-6.6
-3.1
-4.1
-9.6
-13.9
-17.0
1.00
0.89
1.45
1.67
0.83
3.13
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

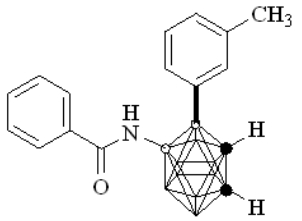
X-246-H

CH3
H
N
O
H
H

2i

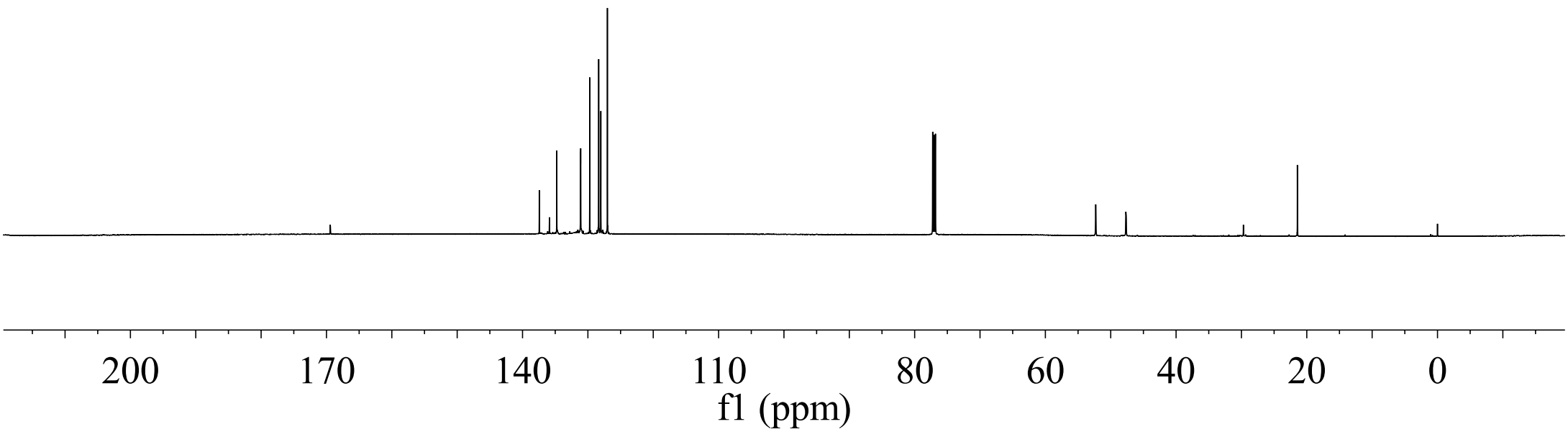

7.62
7.61
7.42
7.42
7.41
7.40
7.40
7.35
7.33
7.32
7.26
7.21
7.20
7.18
7.15
7.14
5.70
3.73
2.31
0.01
0.01
0.01
1.96
2.91
1.98
0.98
0.95
0.94
2.00
2.94
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-246-C



2i

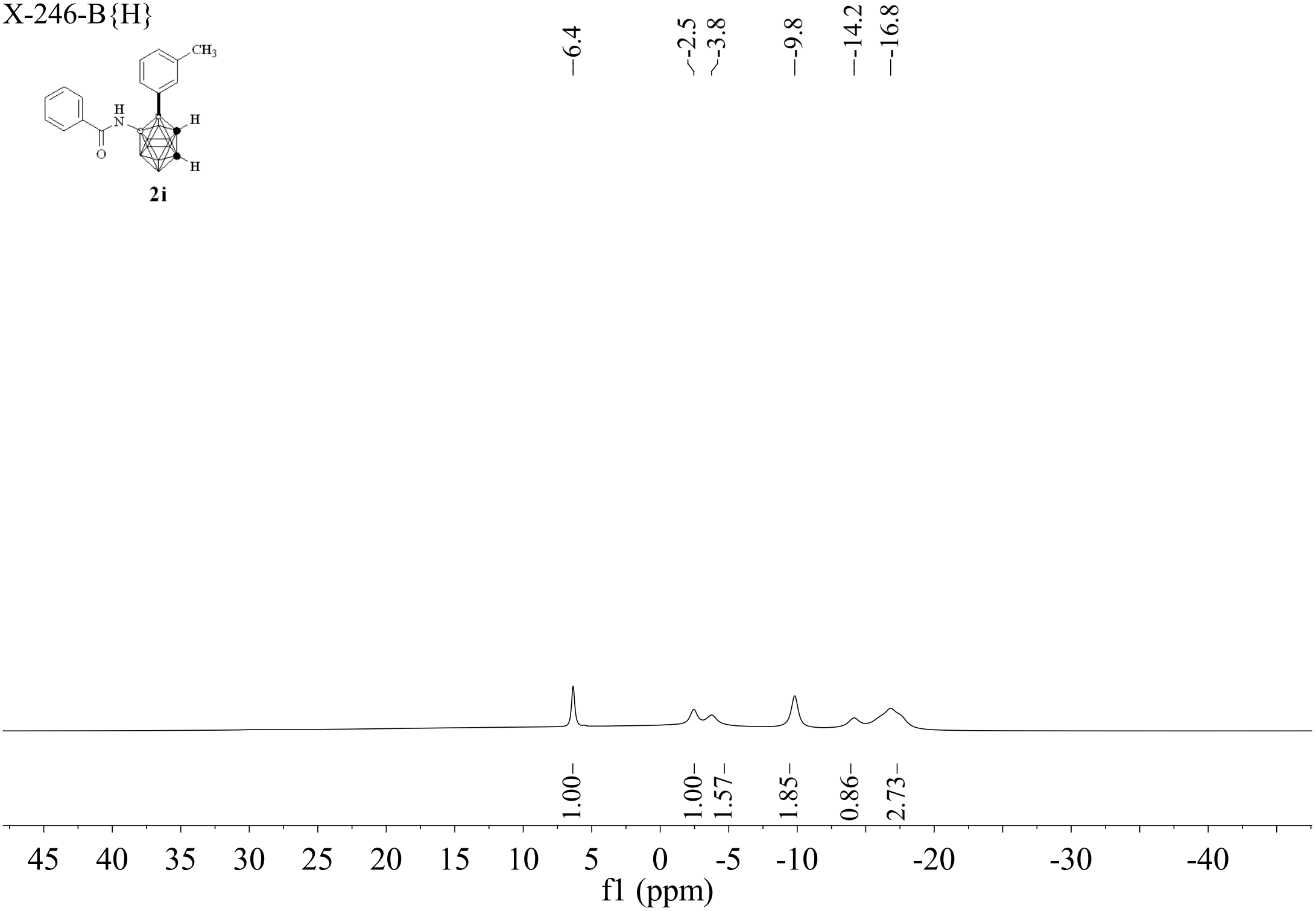
169.4
137.4
135.9
134.8
131.1
131.1
129.7
128.3
128.0
127.0
77.2
77.0
76.8
52.3
47.7
21.4
0.0



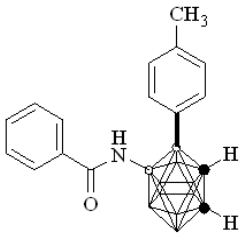
X-246-B{H}

CH3
H
N
O
H
H

2i


-6.4
-2.5
-3.8
-9.8
-14.2
-16.8
1.00
1.00
1.57
1.85
0.86
2.73
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-278-H



2j

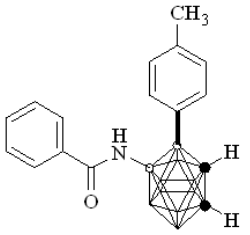
7.63
7.61
7.51
7.50
7.42
7.41
7.40
7.35
7.34
7.32
7.26
7.12
7.11
5.71
3.73
3.69
2.32
0.01

1.98
1.97
1.05
2.21
2.00
0.97
1.00
1.02
3.10

10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

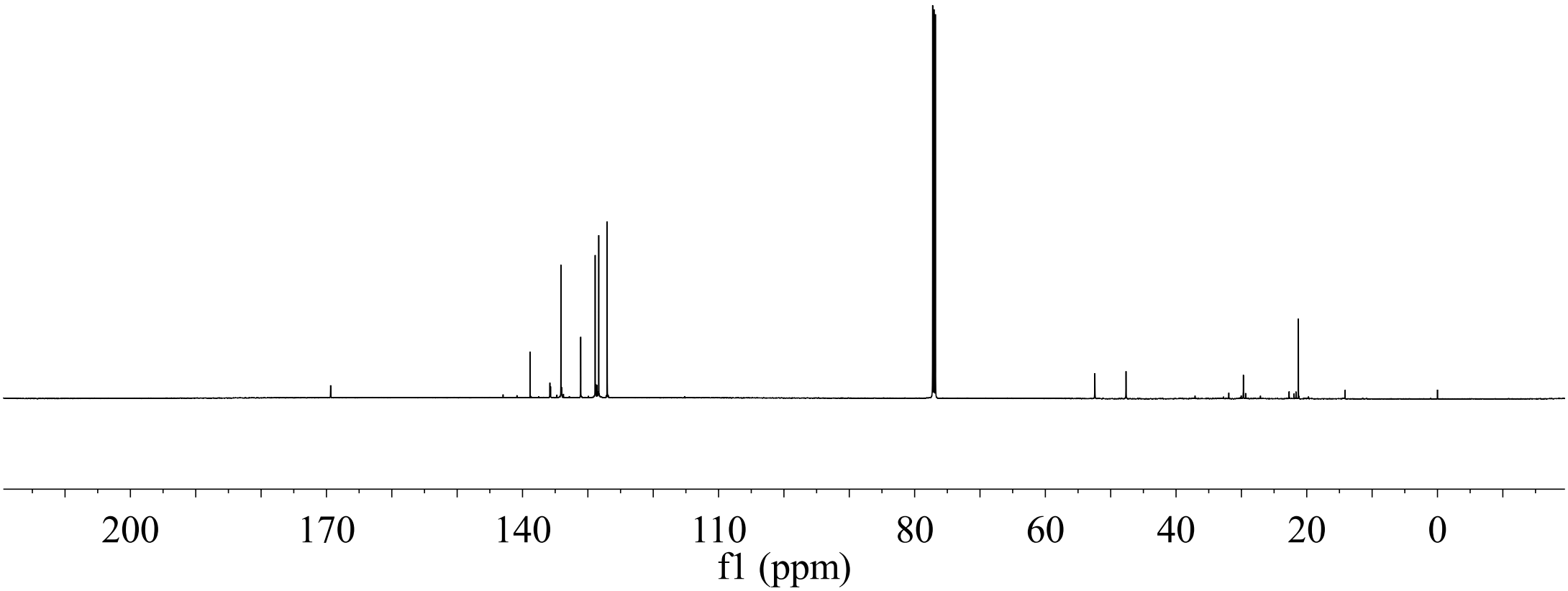
f1 (ppm)

X-278-C



2j

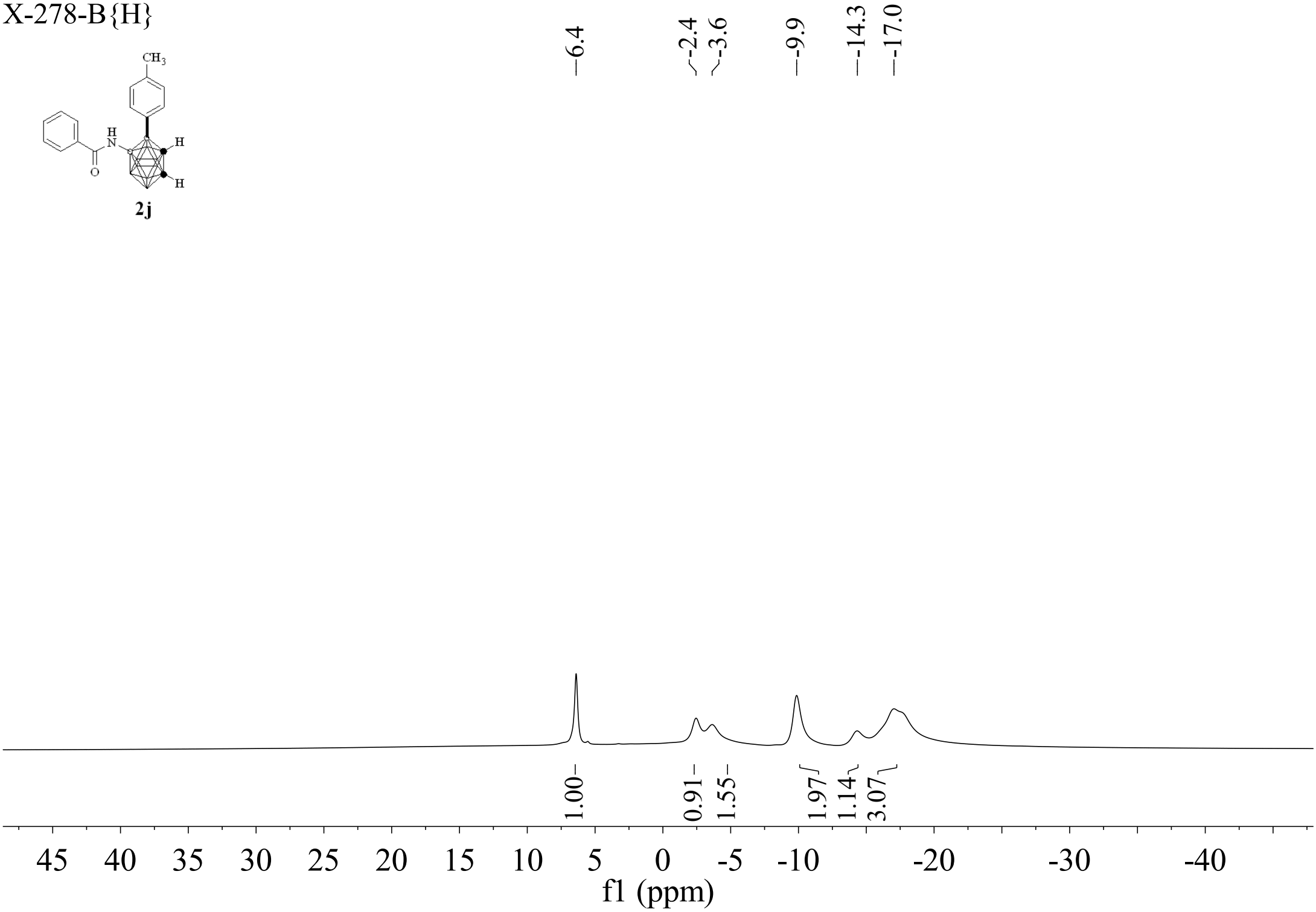
169.4
138.8
135.8
135.7
134.1
131.1
128.9
128.3
127.0
77.2
77.0
76.8
52.4
47.7
21.3
0.0



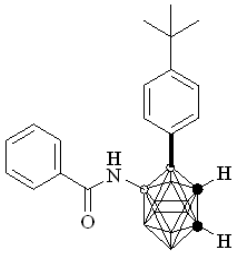
X-278-B{H}

CH3
H
N
O
H
H

2j


-6.4
-2.4
-3.6
-9.9
-14.3
-17.0
1.00
0.91
1.55
1.97
1.14
3.07
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-273-H



2k

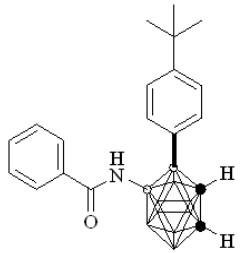
7.64
7.62
7.56
7.55
7.43
7.41
7.40
7.35
7.34
7.33
7.26
5.70
3.72
3.70
1.29
0.01

1.79
1.81
1.05
3.86
0.88
1.00
0.93
8.69

10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5

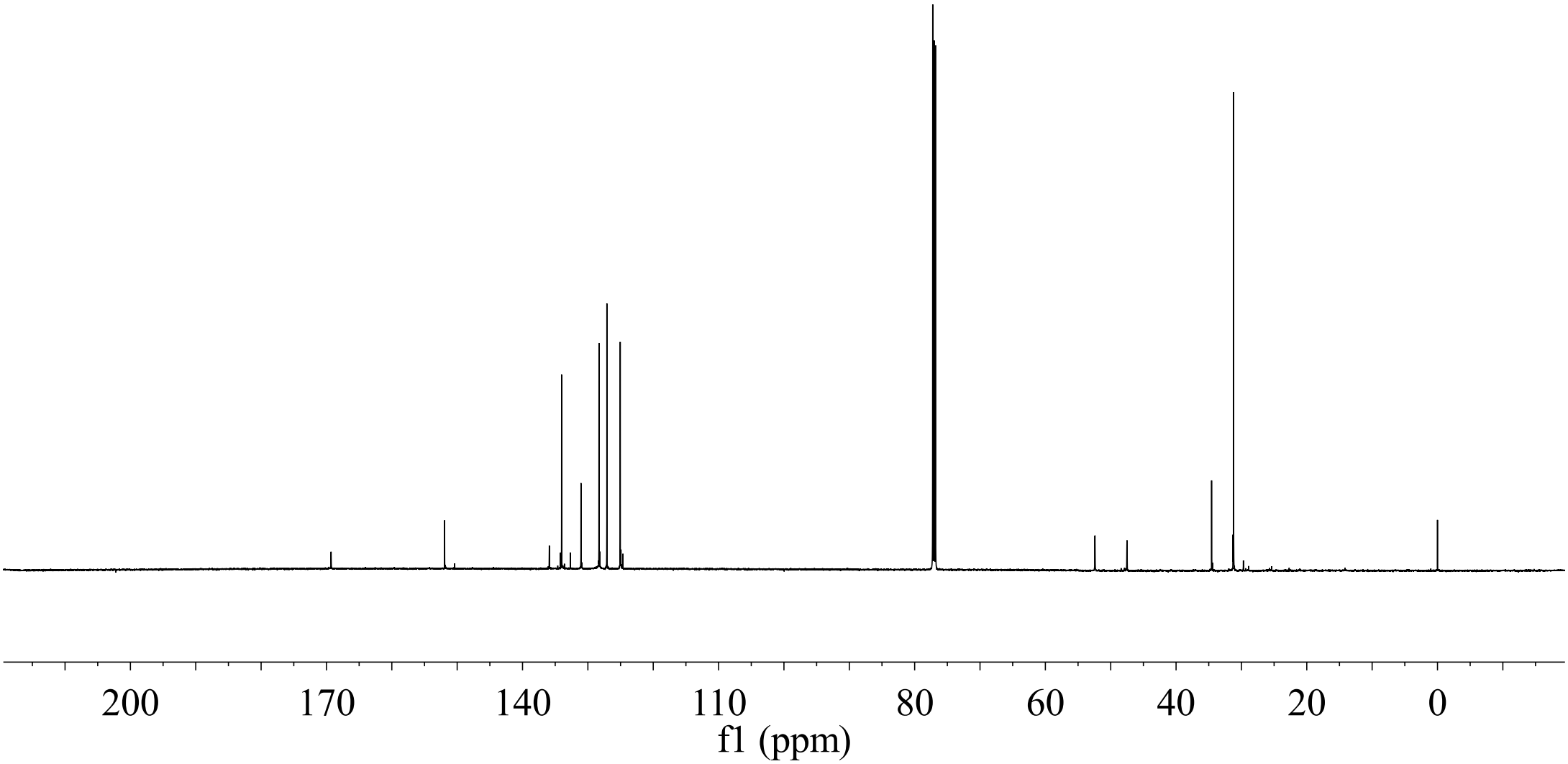
f1 (ppm)

X-273-C



2k

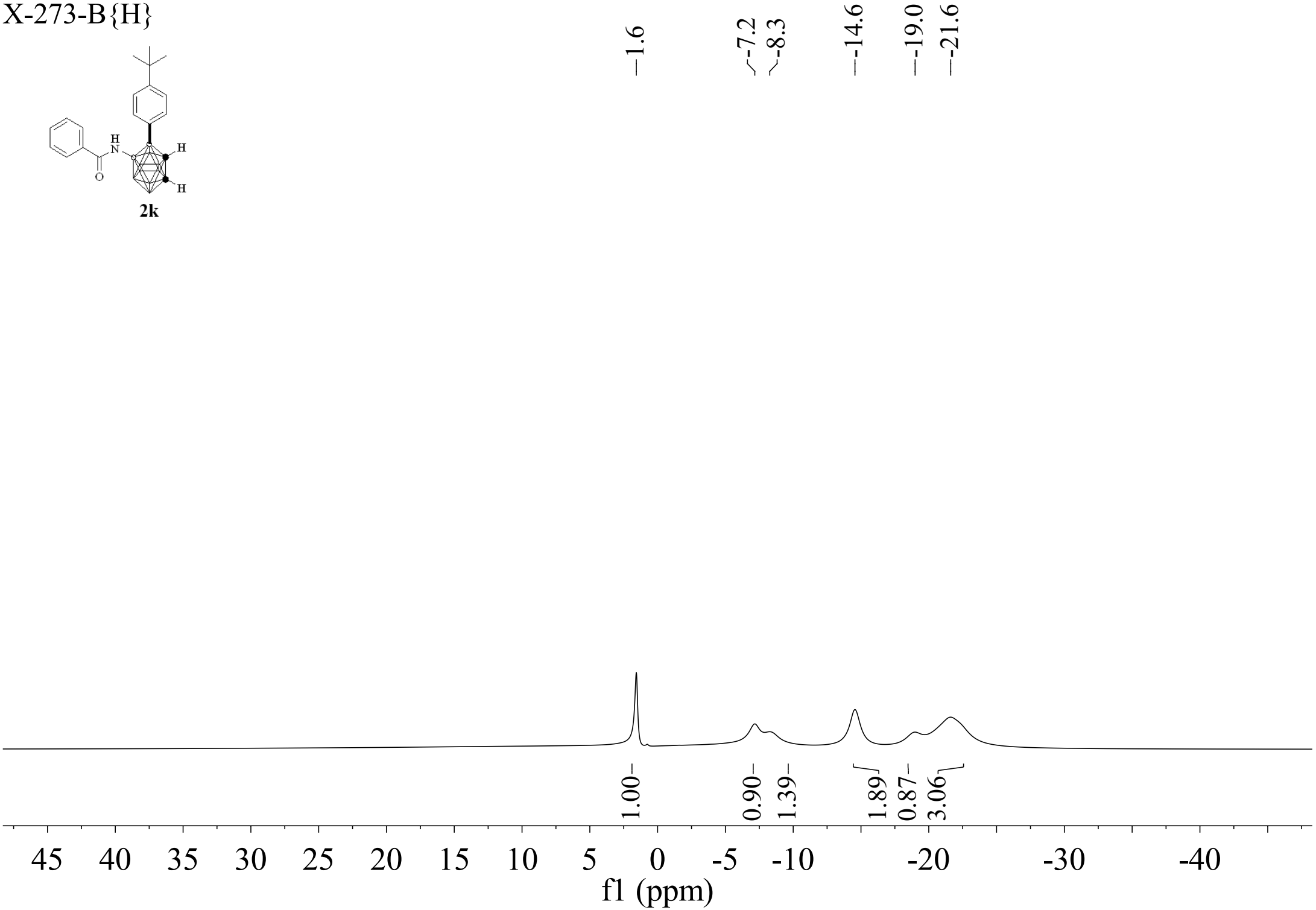
169.3
151.9
135.9
134.0
132.7
131.1
128.3
127.1
125.0
77.2
77.0
76.8
52.4
47.5
34.6
31.2
0.0



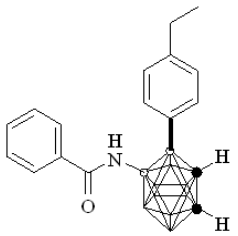
X-273-B{H}

H
N
O
H
H

2k


-1.6
-7.2
-8.3
-14.6
-19.0
-21.6
1.00
0.90
1.39
1.89
0.87
3.06
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-261-H



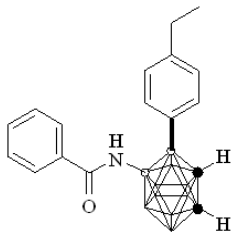
2l

7.62
7.61
7.54
7.53
7.42
7.41
7.40
7.35
7.33
7.32
7.26
7.15
7.14
5.71
3.74
3.70
2.64
2.62
2.61
2.60
1.23
1.21
1.20
0.01

1.97
1.99
1.07
2.11
2.00
0.97
1.00
1.00
2.39
3.26

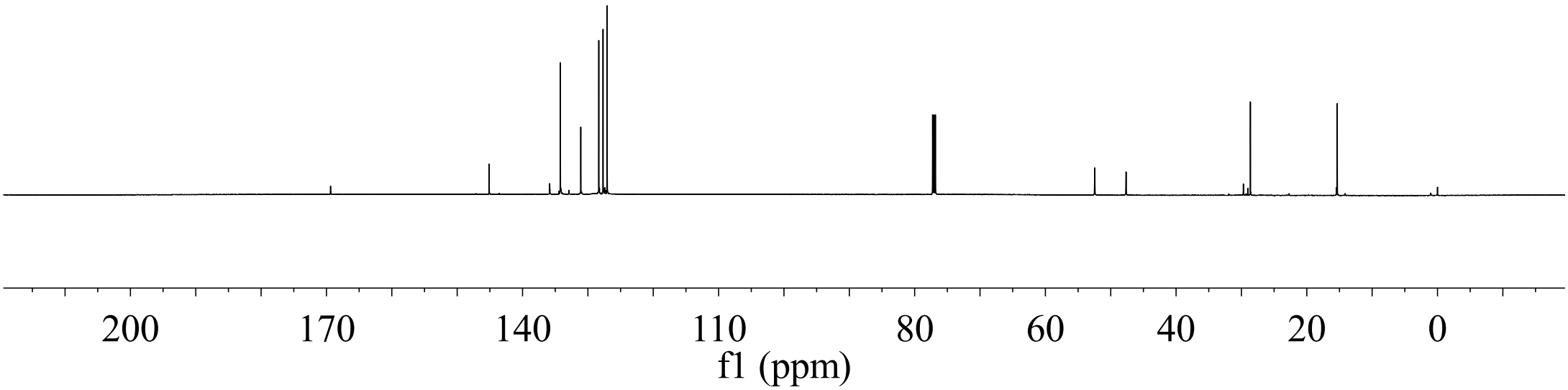
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-261-C



21

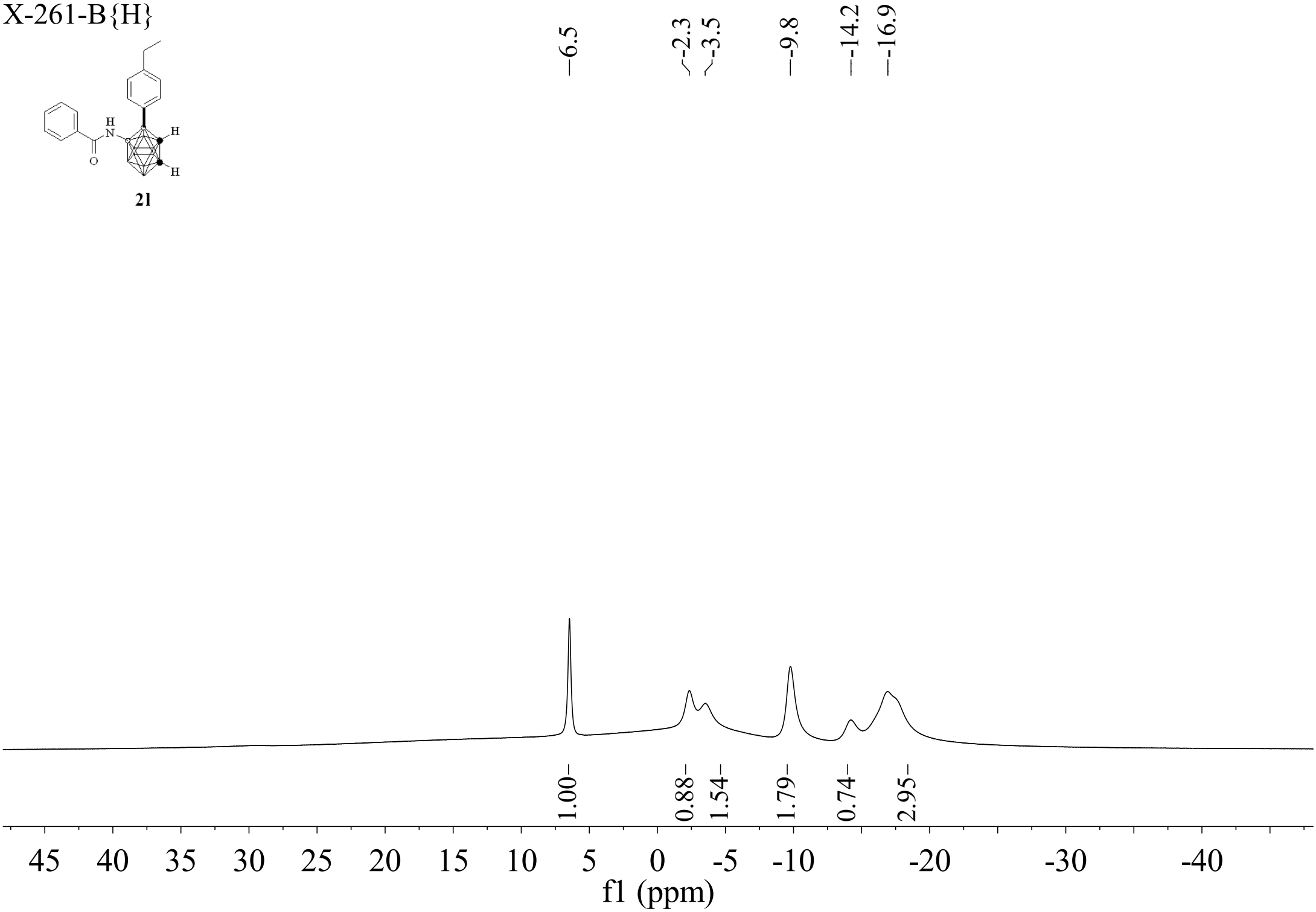
169.4
145.1
135.8
134.2
132.9
131.1
128.3
127.7
127.1
77.2
77.0
76.8
52.4
47.7
28.6
15.3
0.0



X-261-B{H}

H
N
O
H
H

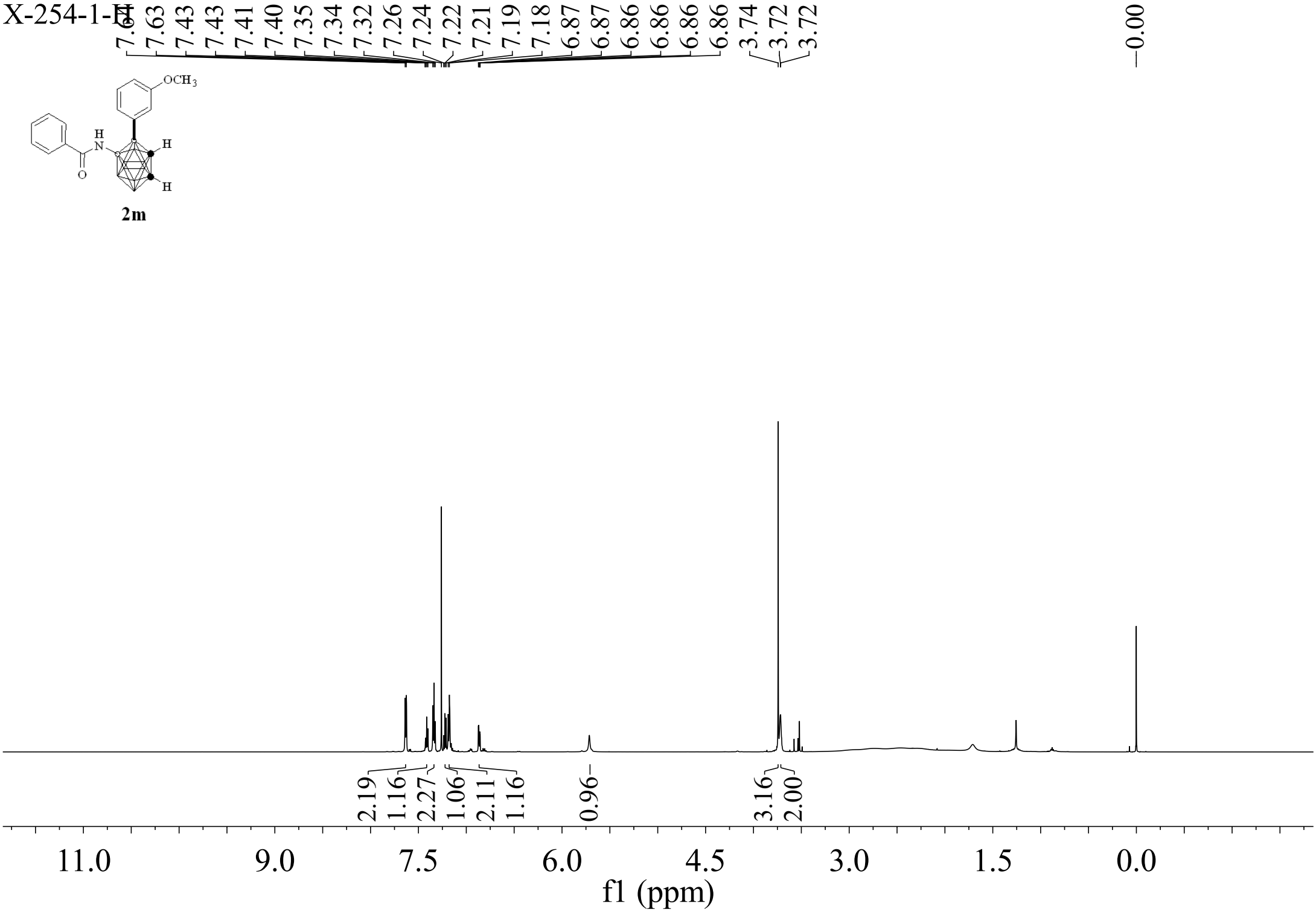
21


-6.5
-2.3
-3.5
-9.8
-14.2
-16.9
1.00
0.88
1.54
1.79
0.74
2.95
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

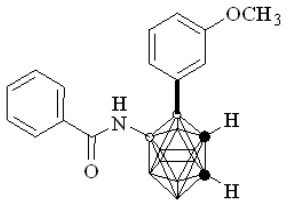
X-254-1-H

OCH3
H
N
O
H
H

2m

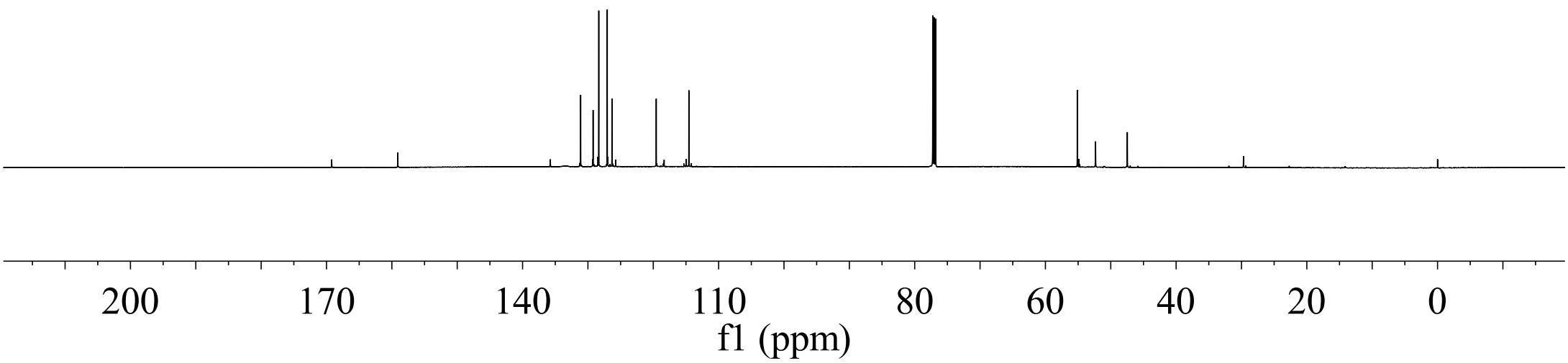

7.64
7.63
7.43
7.43
7.41
7.40
7.35
7.34
7.32
7.26
7.24
7.22
7.21
7.19
7.18
6.87
6.87
6.86
6.86
6.86
6.86
3.74
3.72
3.72
0.00
2.19
1.16
2.27
1.06
2.11
1.16
0.96
3.16
2.00
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-254-1-C



2m

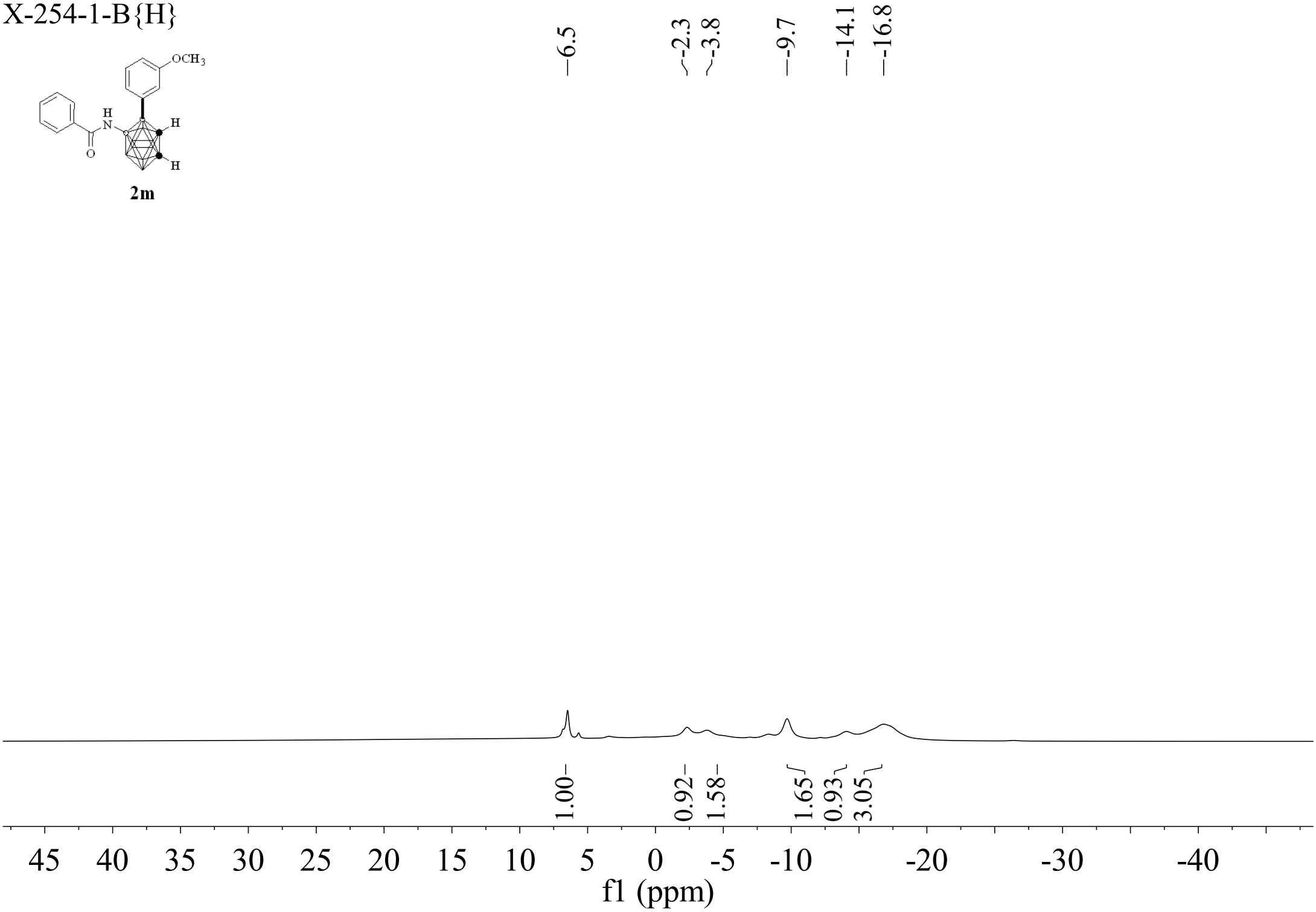
169.2
159.1
135.8
131.1
129.2
128.3
127.0
126.3
119.5
114.6
77.2
77.0
76.8
55.1
52.3
47.5

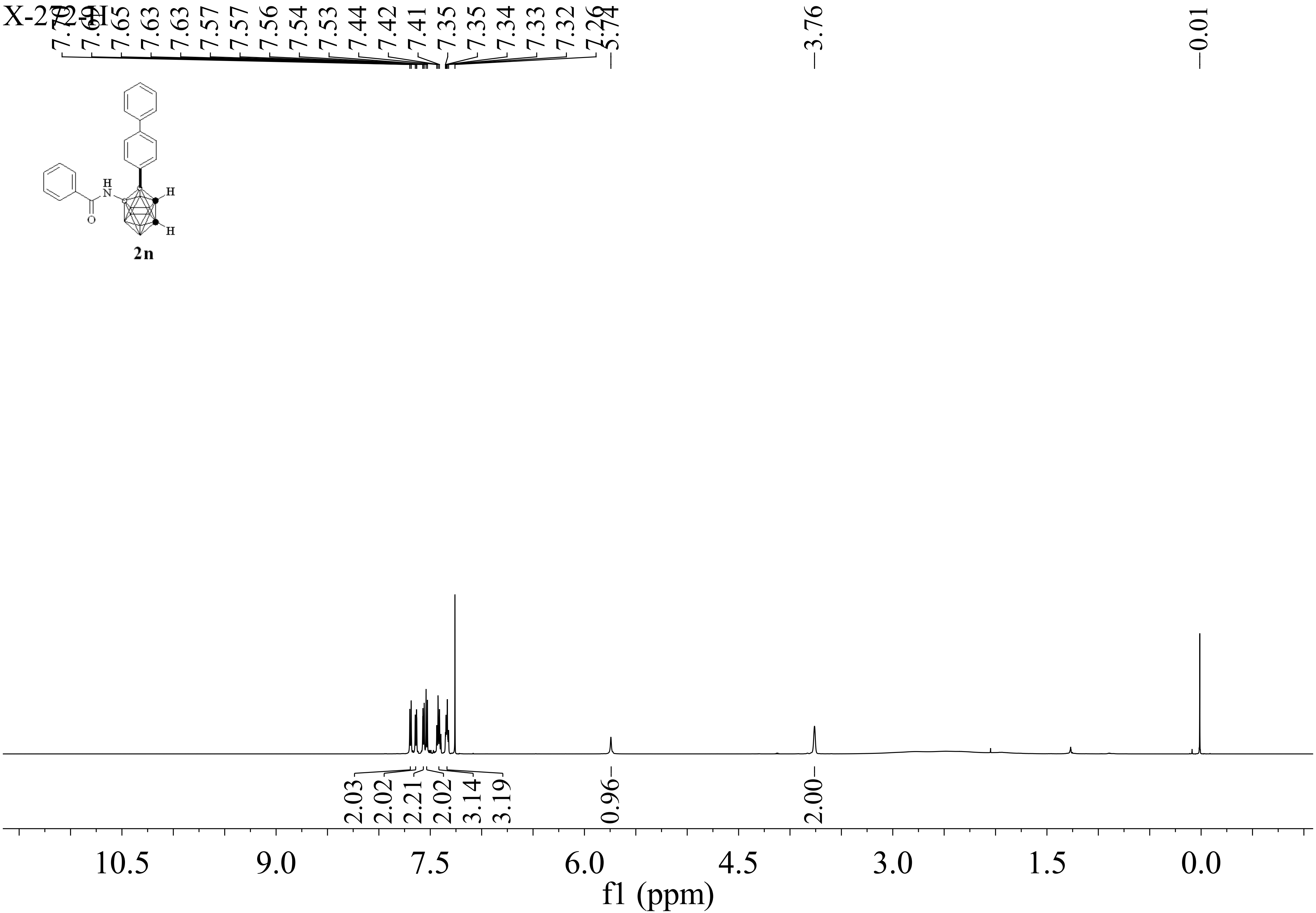


X-254-1-B{H}

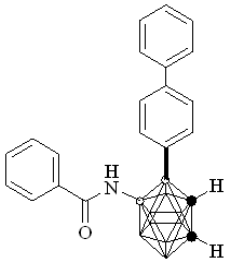
OCH3
H
N
O
H
H

2m


-6.5
-2.3
-3.8
-9.7
-14.1
-16.8
1.00
0.92
1.58
1.65
0.93
3.05
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

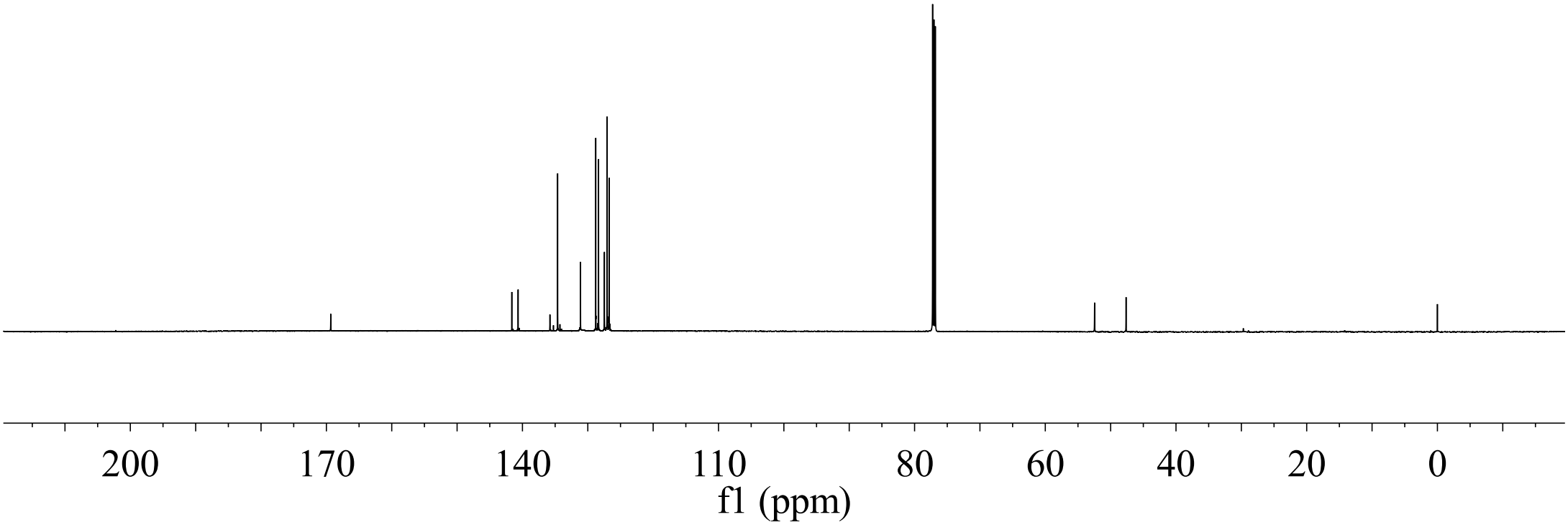

X-272-H
7.72 7.69 7.65 7.63 7.63 7.57 7.57 7.56 7.54 7.53 7.44 7.42 7.41 7.35 7.35 7.34 7.33 7.32 7.26 5.74
3.76
-0.01
2n
2.03 2.02 2.21 2.02 3.14 3.19
0.96
2.00
10.5 9.0 7.5 6.0 4.5 3.0 1.5 0.0
f1 (ppm)

X-272-C



2n

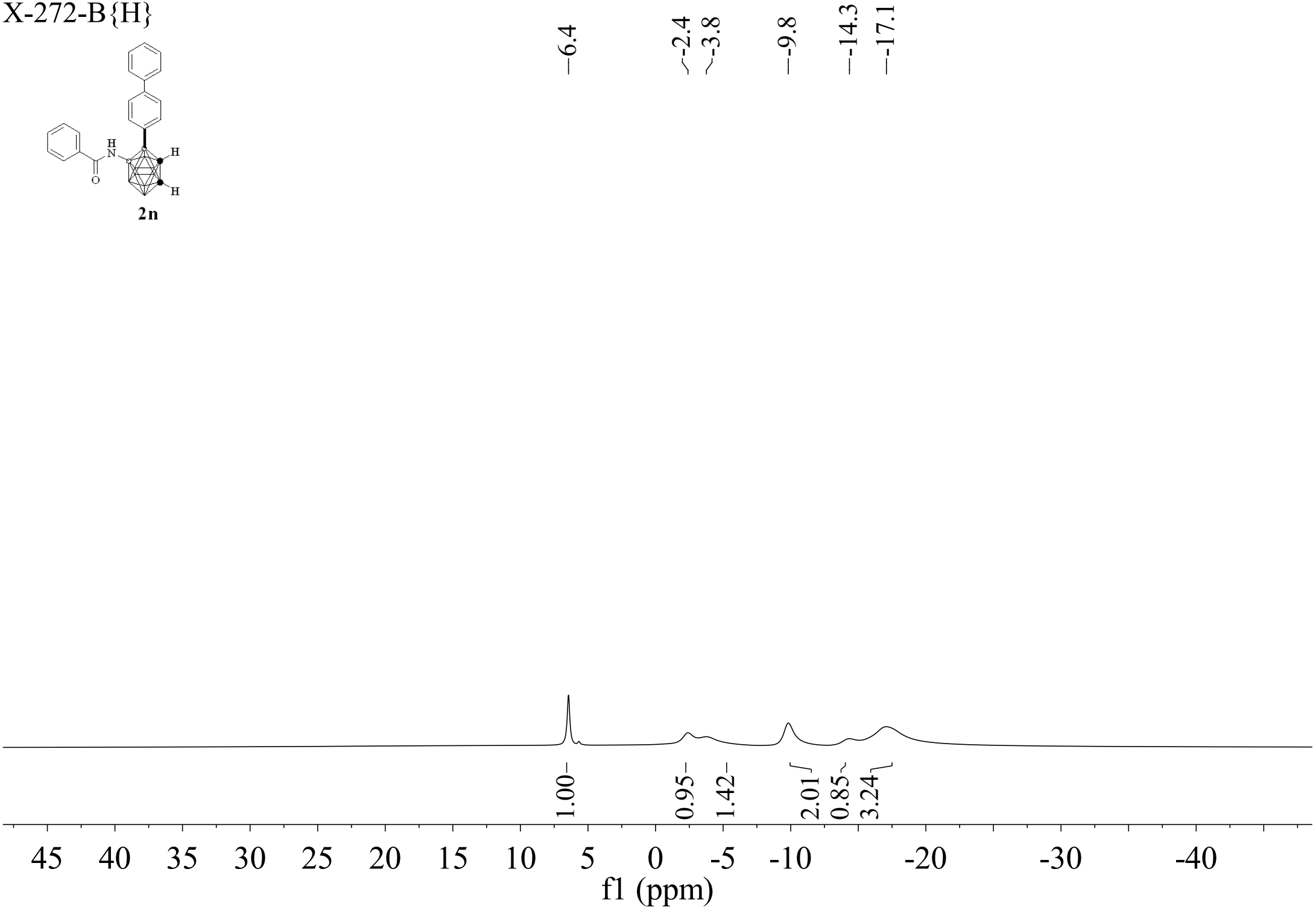
169.3
141.6
140.7
135.8
134.6
131.1
128.8
128.4
127.5
127.1
127.0
126.7
77.2
77.0
76.8
52.5
47.6
0.0



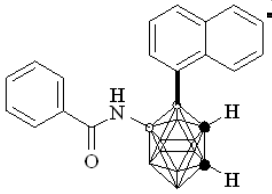
X-272-B{H}

H
N
O
H
H

2n


-6.4
-2.4
-3.8
-9.8
-14.3
-17.1
1.00
0.95
1.42
2.01
0.85
3.24
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-274-H



2o

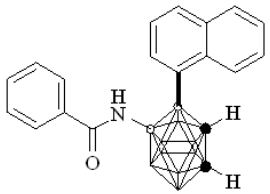
8.74
8.73
7.95
7.94
7.85
7.84
7.82
7.51
7.50
7.49
7.47
7.46
7.42
7.41
7.40
7.34
7.26
7.24
7.23
5.70
3.92
3.70
0.00

1.02
1.04
1.95
2.85
1.00
1.19
0.97
1.80
1.06
1.00
1.01

13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

f1 (ppm)

X-274-C



2o

169.3
136.1
135.8
133.8
131.0
130.2
129.2
128.2
127.1
127.0
126.2
125.4
125.2
77.2
77.0
76.8
52.6
47.3
0.0

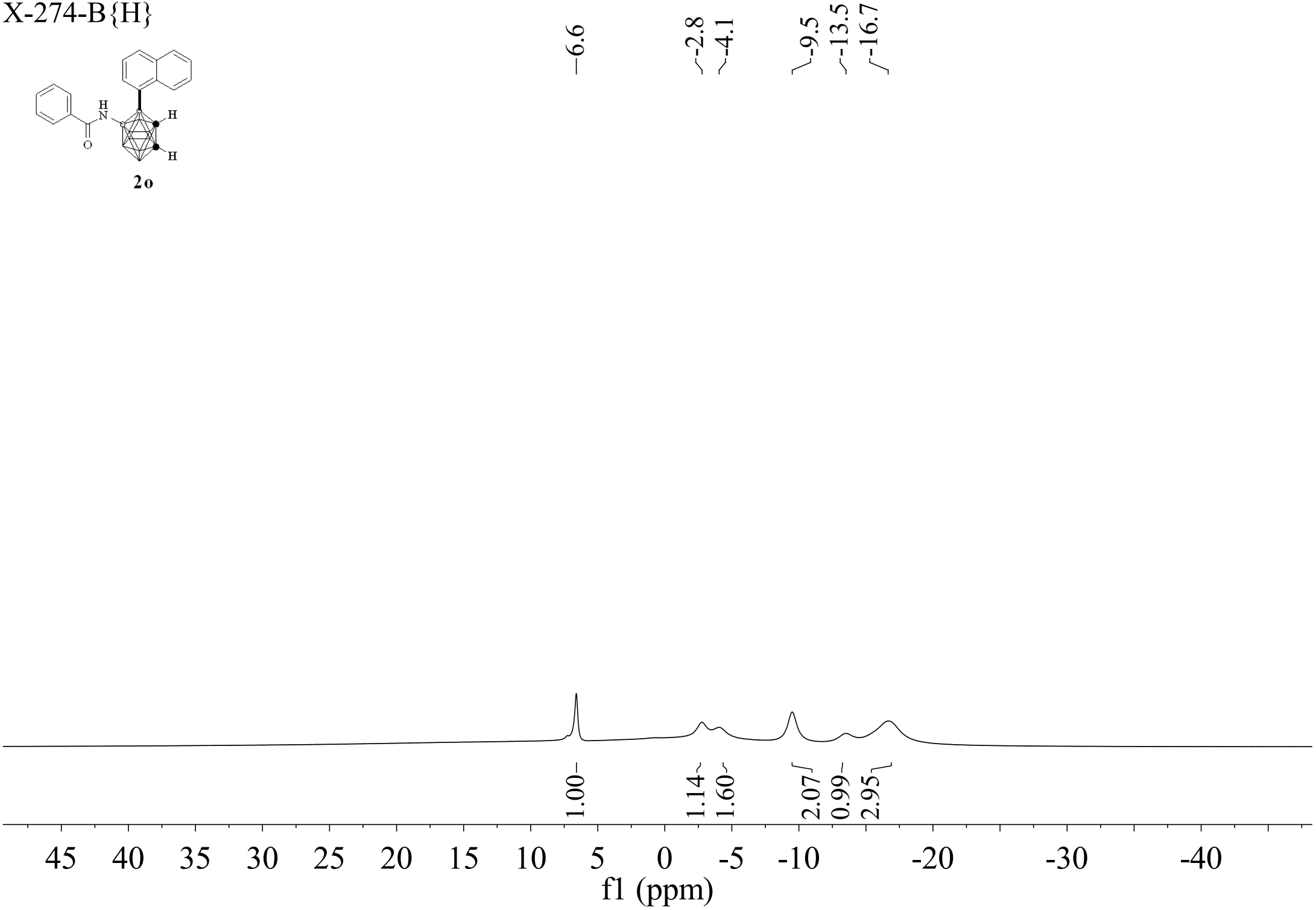
200 170 140 110 80 60 40 20 0

f1 (ppm)

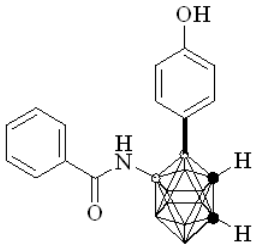
X-274-B{H}

H
N
O
H
H

2o


-6.6
-2.8
-4.1
-9.5
-13.5
-16.7
1.00
1.14
1.60
2.07
0.99
2.95
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-345-H



2p

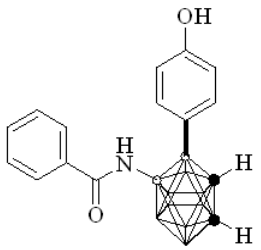
9.36
7.74
7.73
7.45
7.44
7.43
7.38
7.37
7.36
7.35
6.65
6.64
4.96
2.50

0.95
1.96
1.16
4.94
1.93
2.00

10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0

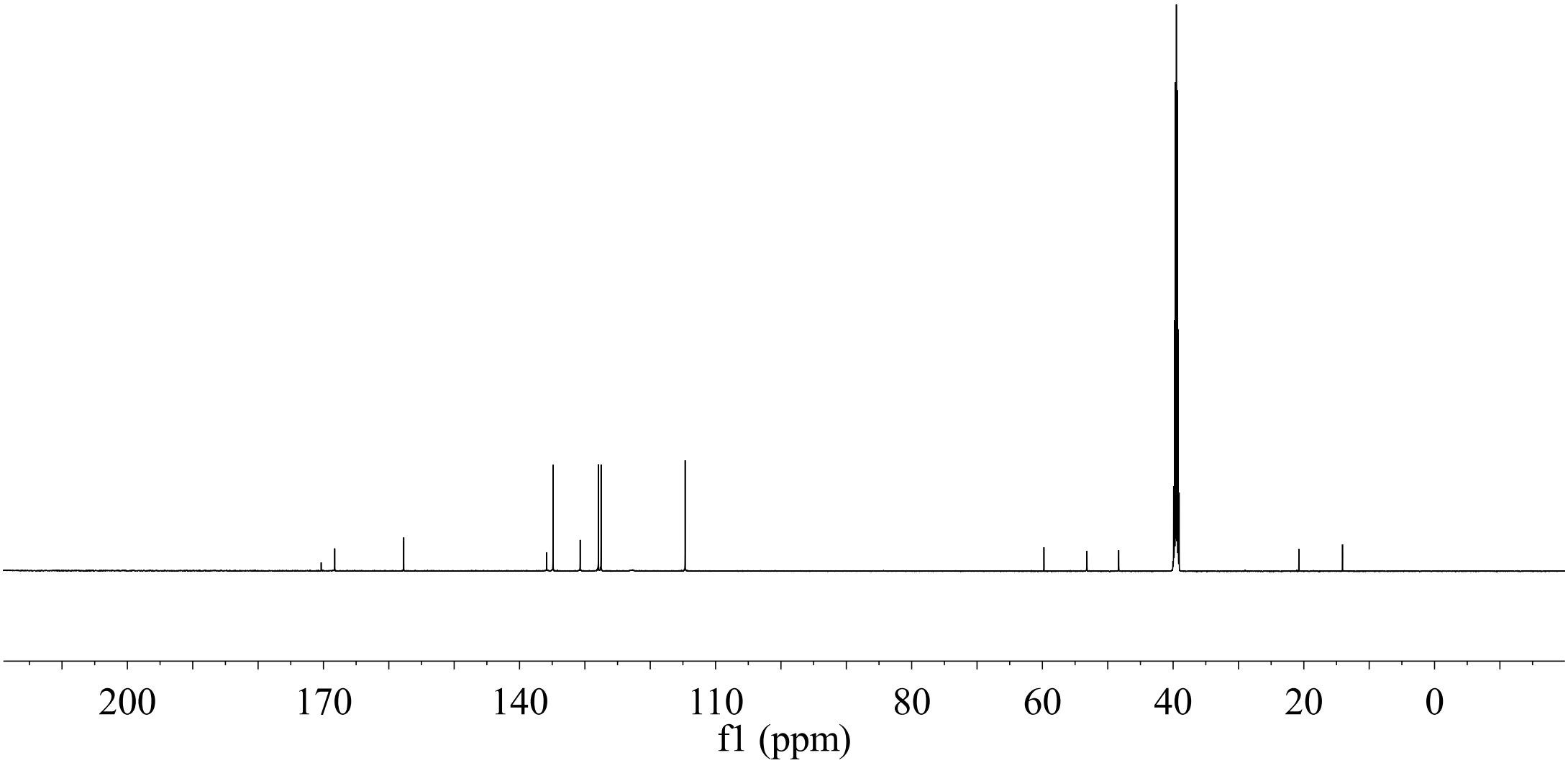
f1 (ppm)

X-345-C



2p

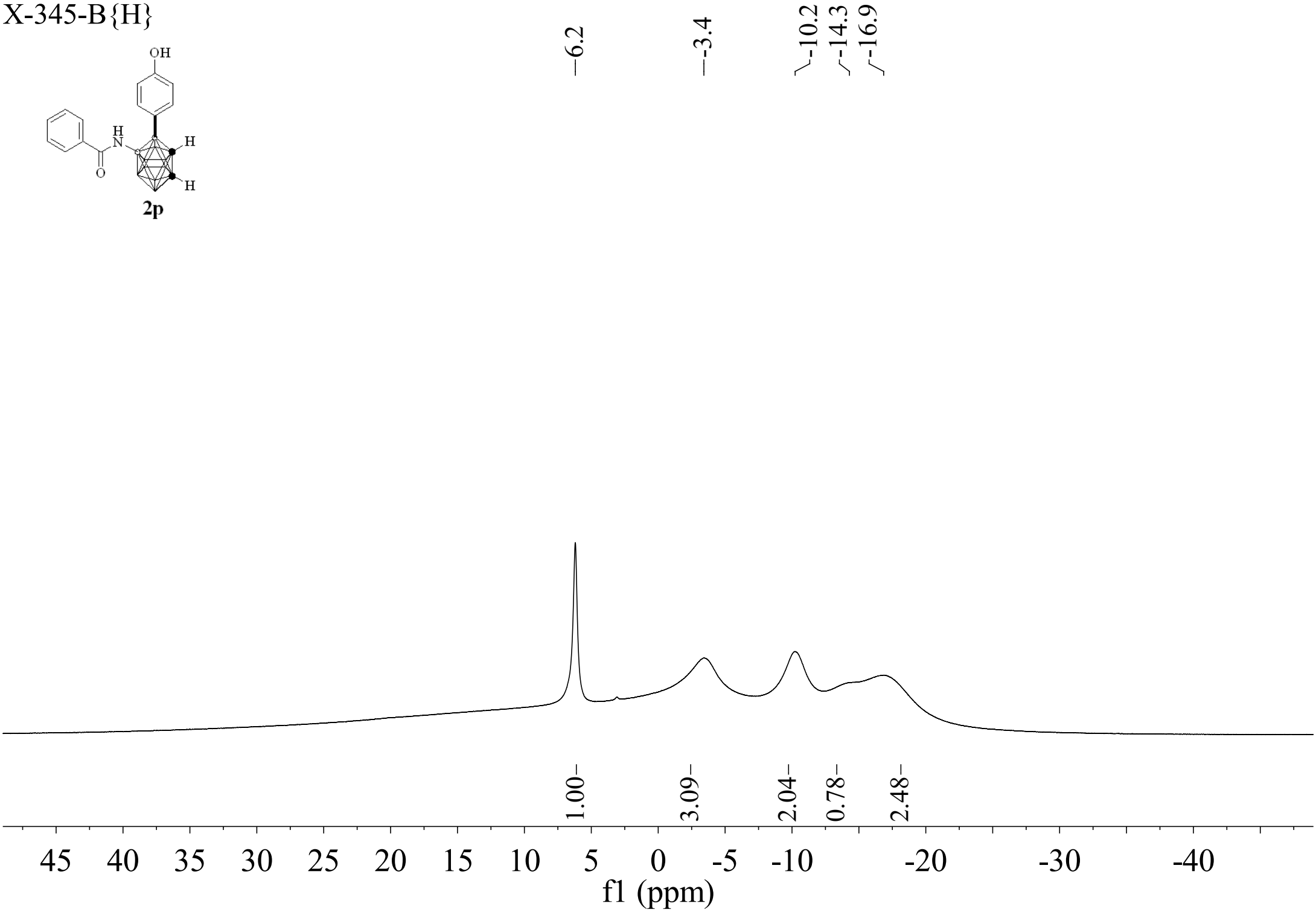
170.3
168.3
157.7
135.8
134.9
130.7
127.9
127.5
114.7
53.2
48.3
39.9
39.8
39.6
39.5
39.4
39.2
39.1



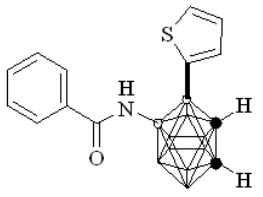
X-345-B{H}

OH
H
N
O
H
H

2p


-6.2
-3.4
-10.2
-14.3
-16.9
1.00
3.09
2.04
0.78
2.48
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-280-H



2q

7.65
7.64
7.56
7.55
7.44
7.43
7.42
7.37
7.35
7.34
7.30
7.30
7.29
7.29
7.26
7.25
7.24
5.71
3.69
3.66
0.00

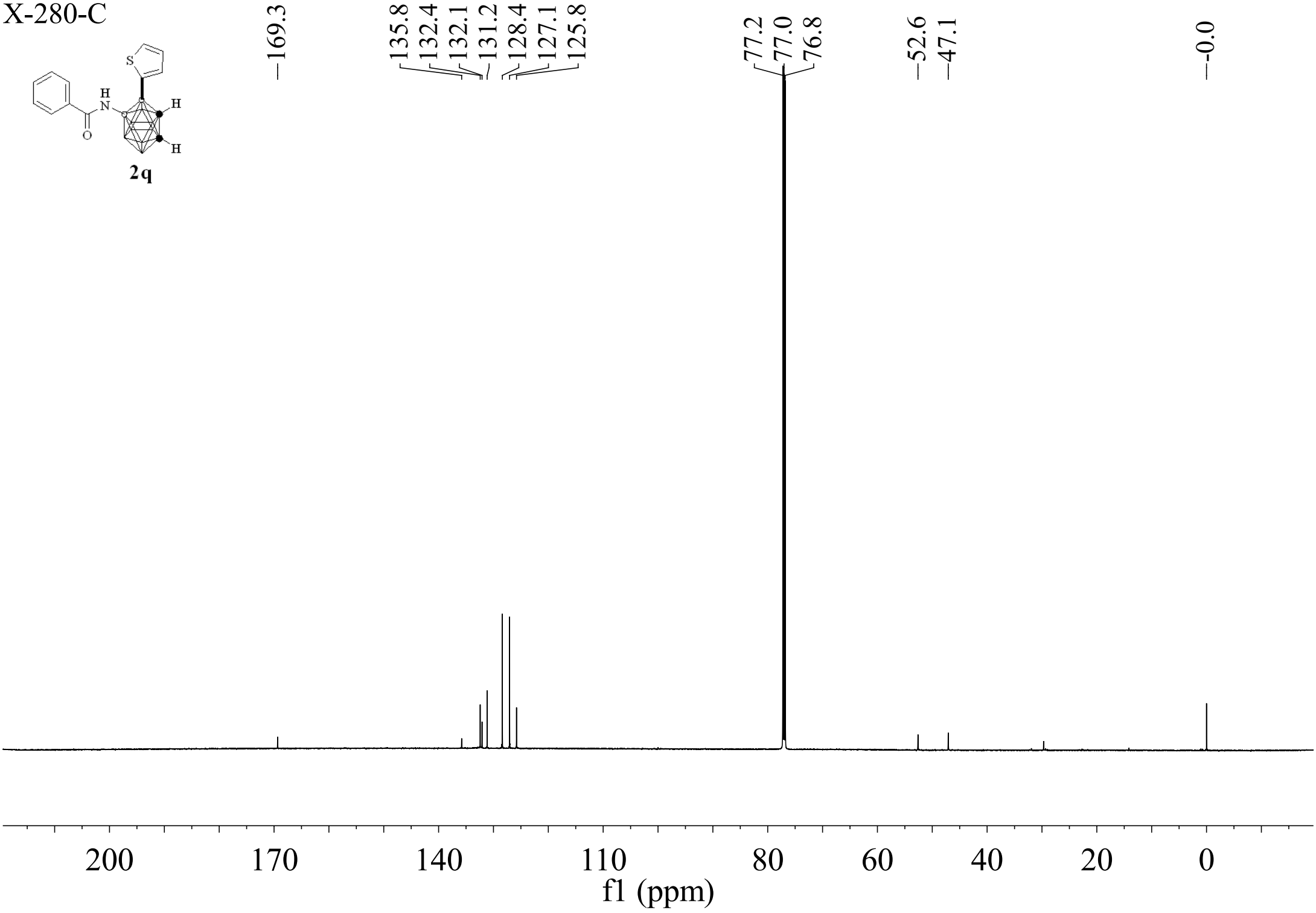
1.97
1.00
1.09
2.04
1.07
0.95
1.07
1.00
1.04

11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-280-C

S
H
N
O
H
H

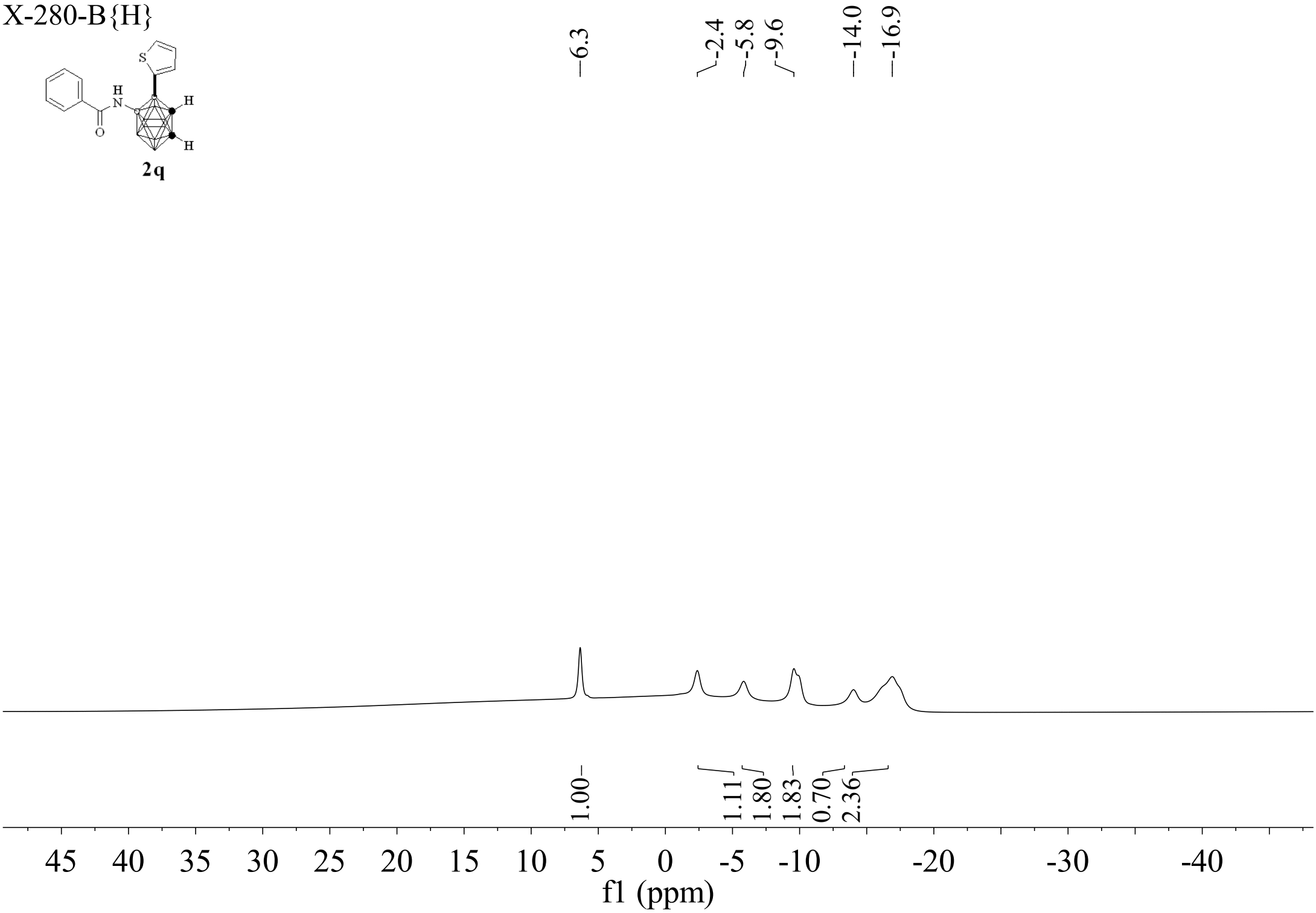
2q


169.3
135.8
132.4
132.1
131.2
128.4
127.1
125.8
77.2
77.0
76.8
52.6
47.1
0.0
200
170
140
110
80
60
40
20
0
f1 (ppm)

X-280-B{H}

S
H
N
O
H
H

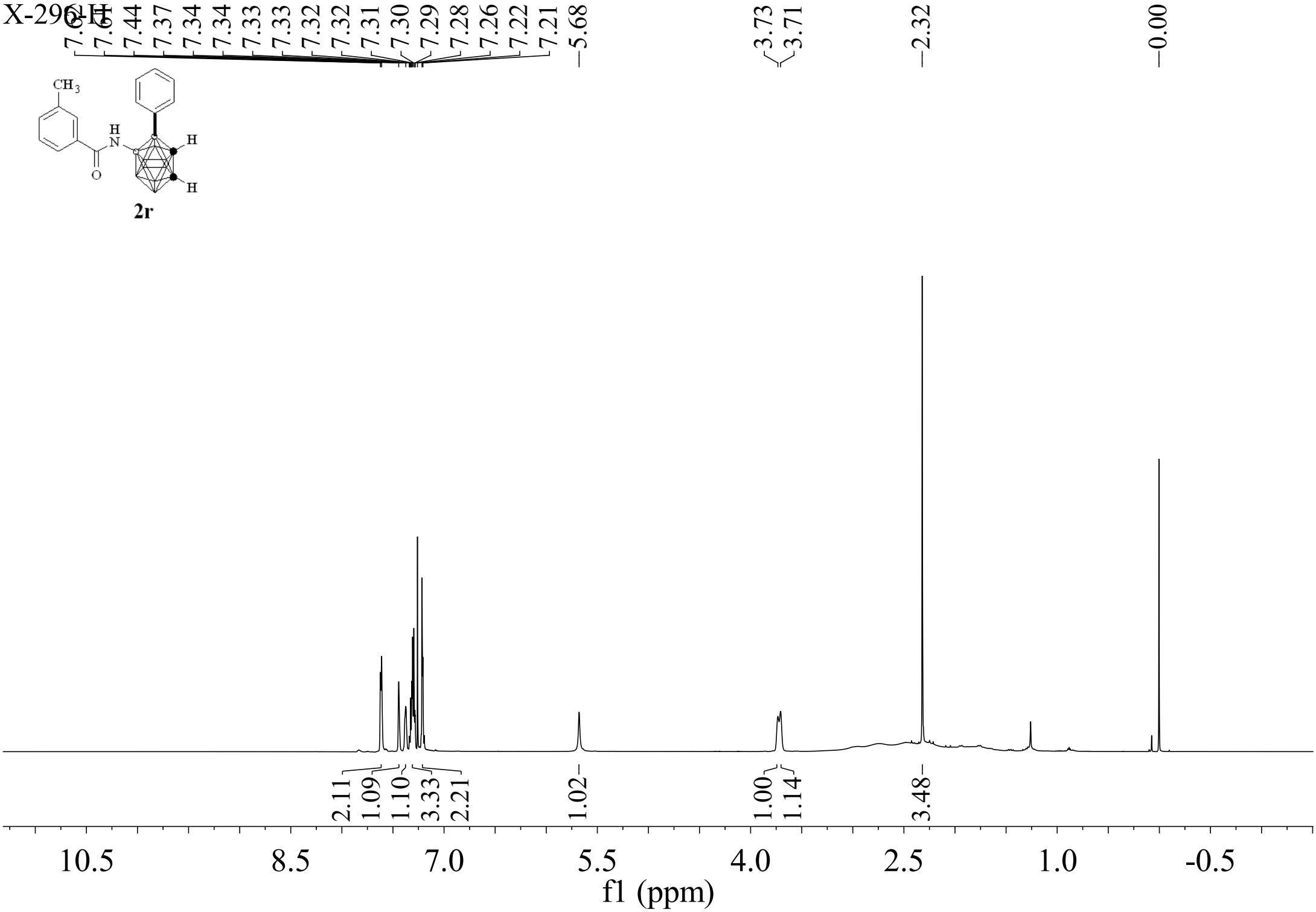
2q


-6.3
-2.4
-5.8
-9.6
-14.0
-16.9
1.00
1.11
1.80
1.83
0.70
2.36
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

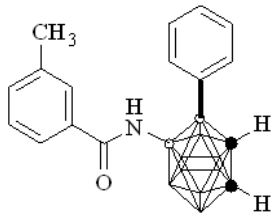
X-296-1H

CH3
H
N
O
H
H

2r

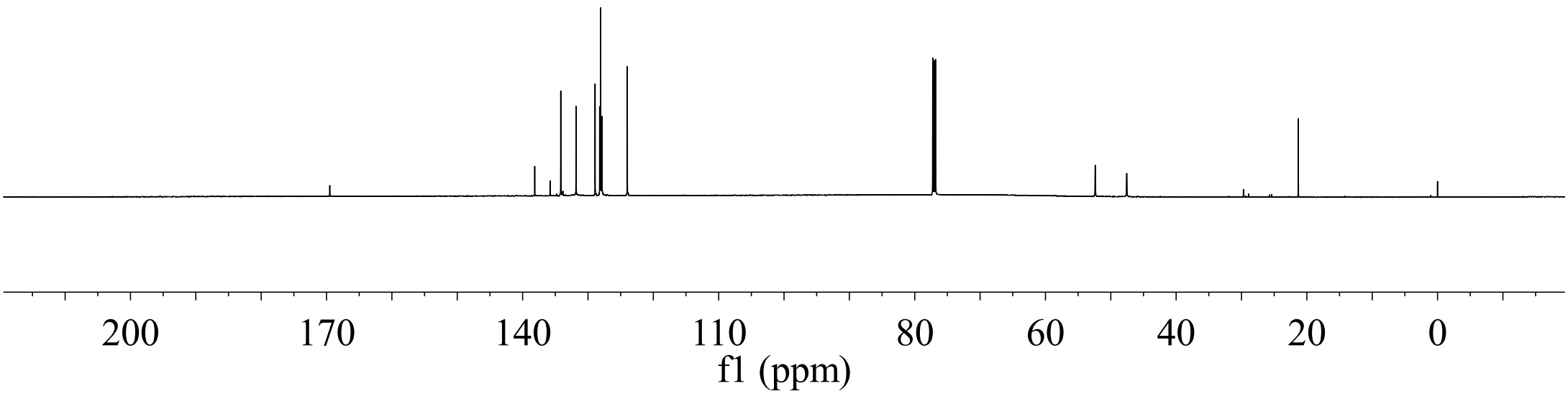

7.61
7.44
7.37
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.29
7.28
7.26
7.22
7.21
5.68
3.73
3.71
2.32
0.00
2.11
1.09
1.10
3.33
2.21
1.02
1.00
1.14
3.48
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-296-C



2r

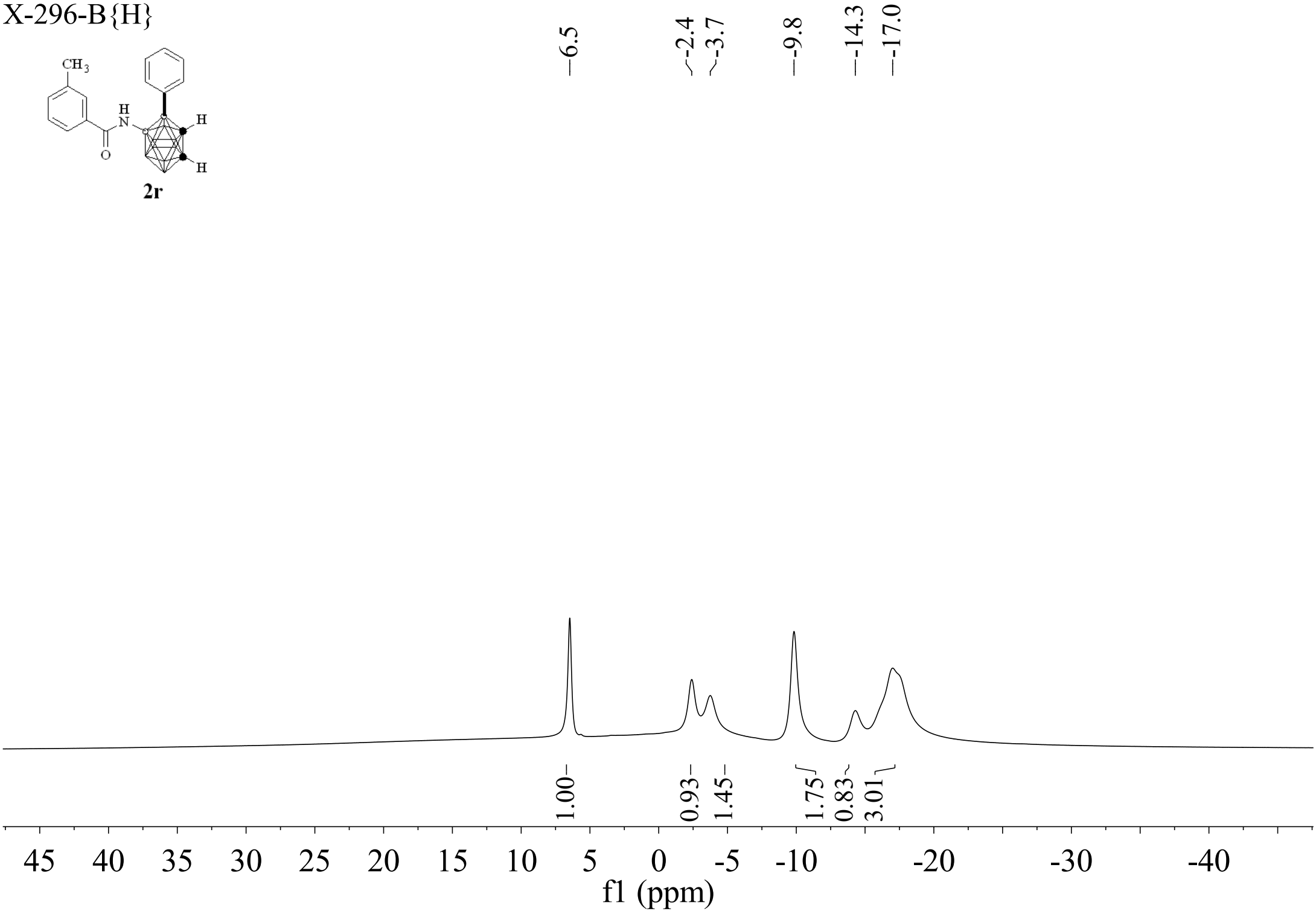
169.5
138.1
135.8
134.2
131.8
128.9
128.2
128.0
127.9
124.0
77.2
77.0
76.8
52.4
47.5
21.3
0.0



X-296-B{H}

CH3
H
N
O
H
H

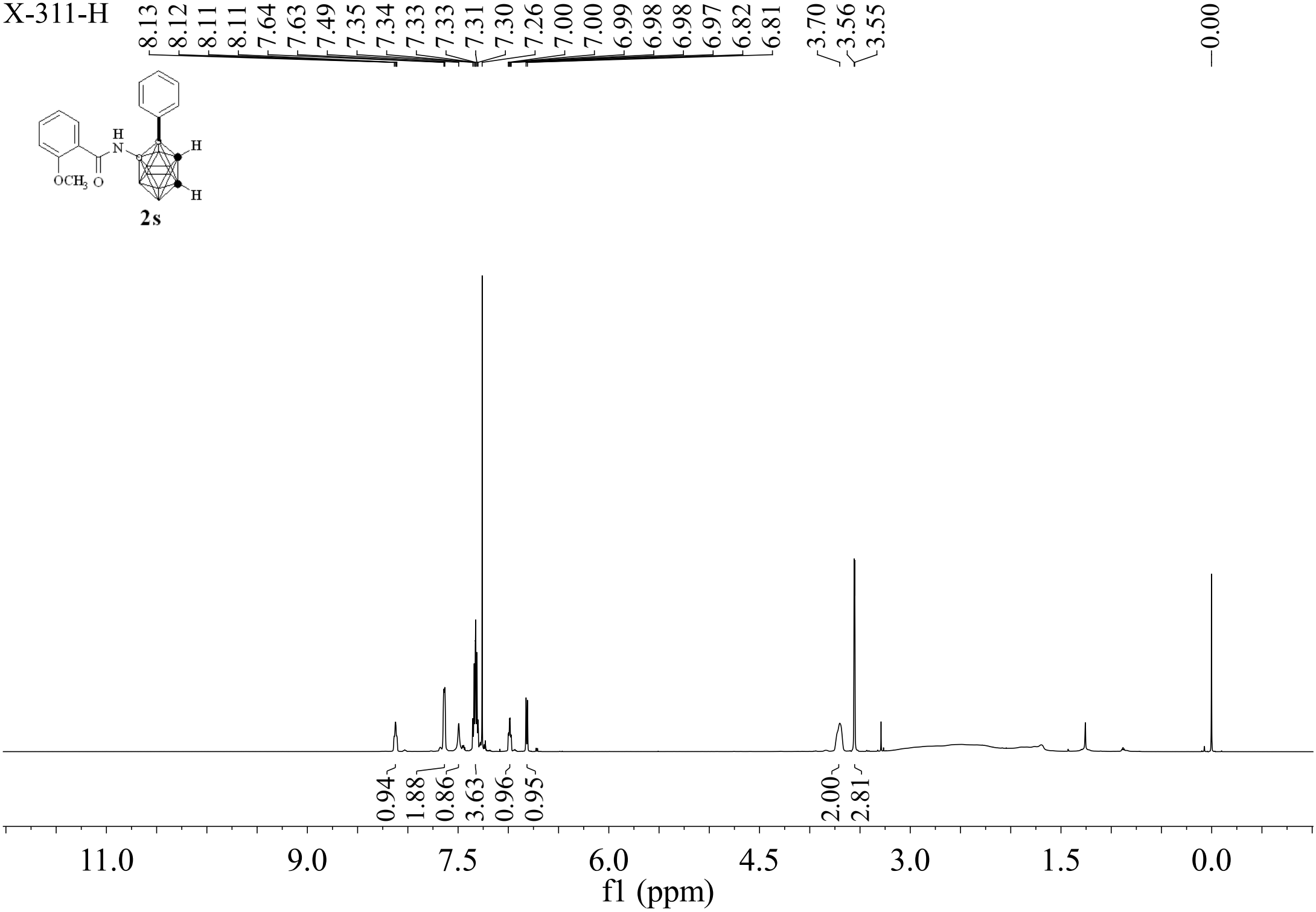
2r


-6.5
-2.4
-3.7
-9.8
-14.3
-17.0
1.00
0.93
1.45
1.75
0.83
3.01
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

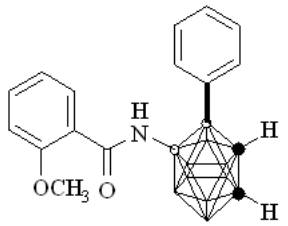
X-311-H

H
N
OCH3 O
H
H

2s

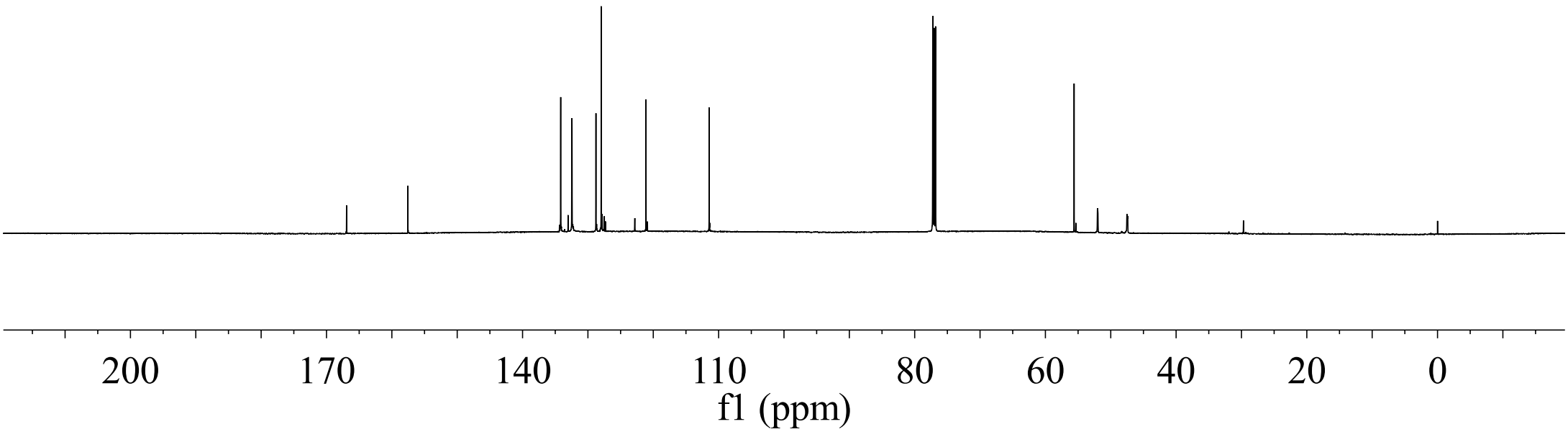

8.13
8.12
8.11
8.11
7.64
7.63
7.49
7.35
7.34
7.33
7.33
7.31
7.30
7.26
7.00
7.00
6.99
6.98
6.98
6.97
6.82
6.81
3.70
3.56
3.55
0.00
0.94
1.88
0.86
3.63
0.96
0.95
2.00
2.81
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-311-C



2s

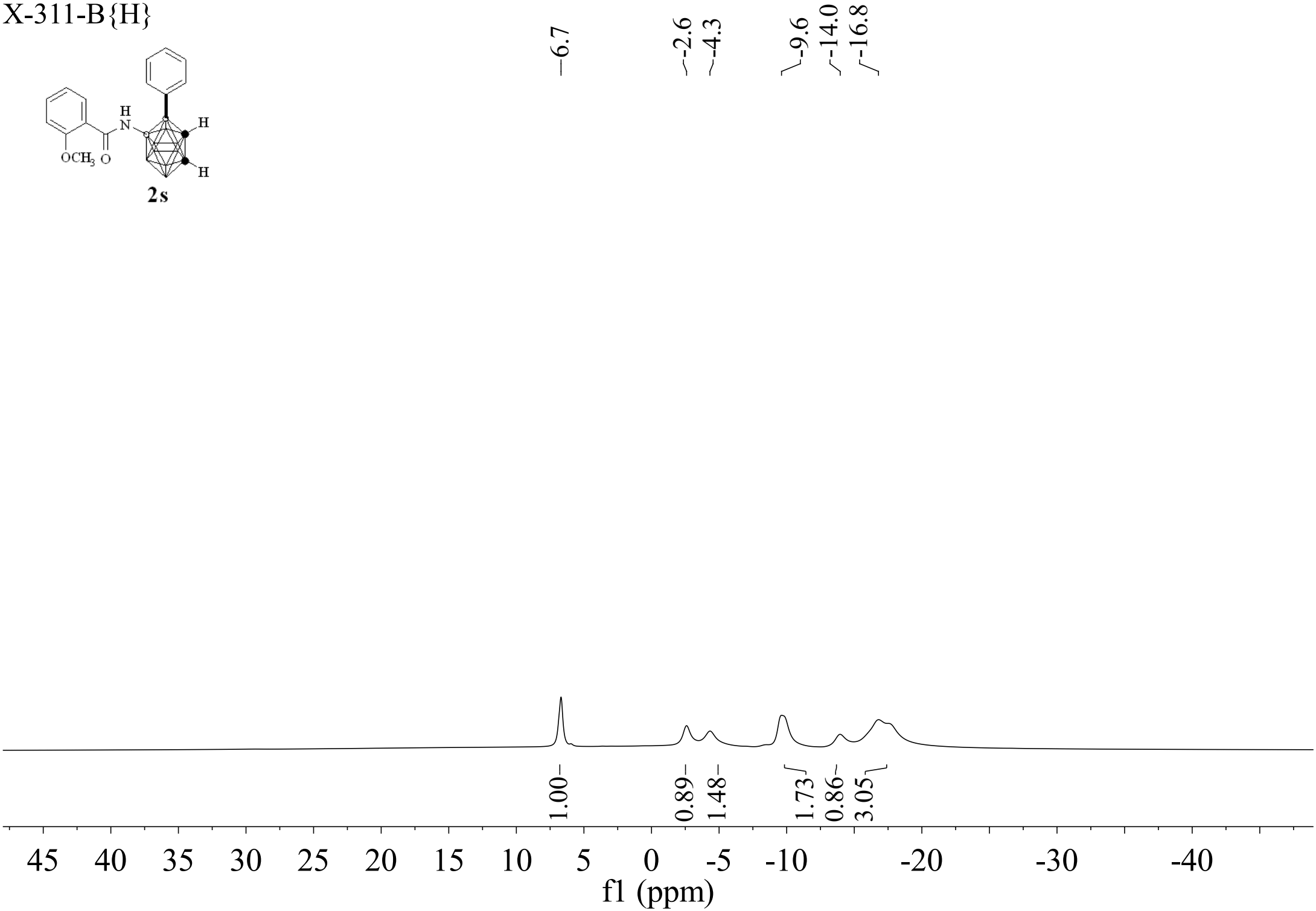
166.9
157.6
134.2
133.0
132.5
132.4
128.8
128.0
121.1
111.4
77.2
77.0
76.8
55.6
52.0
47.5



X-311-B{H}

H
N
H
H
OCH3 O

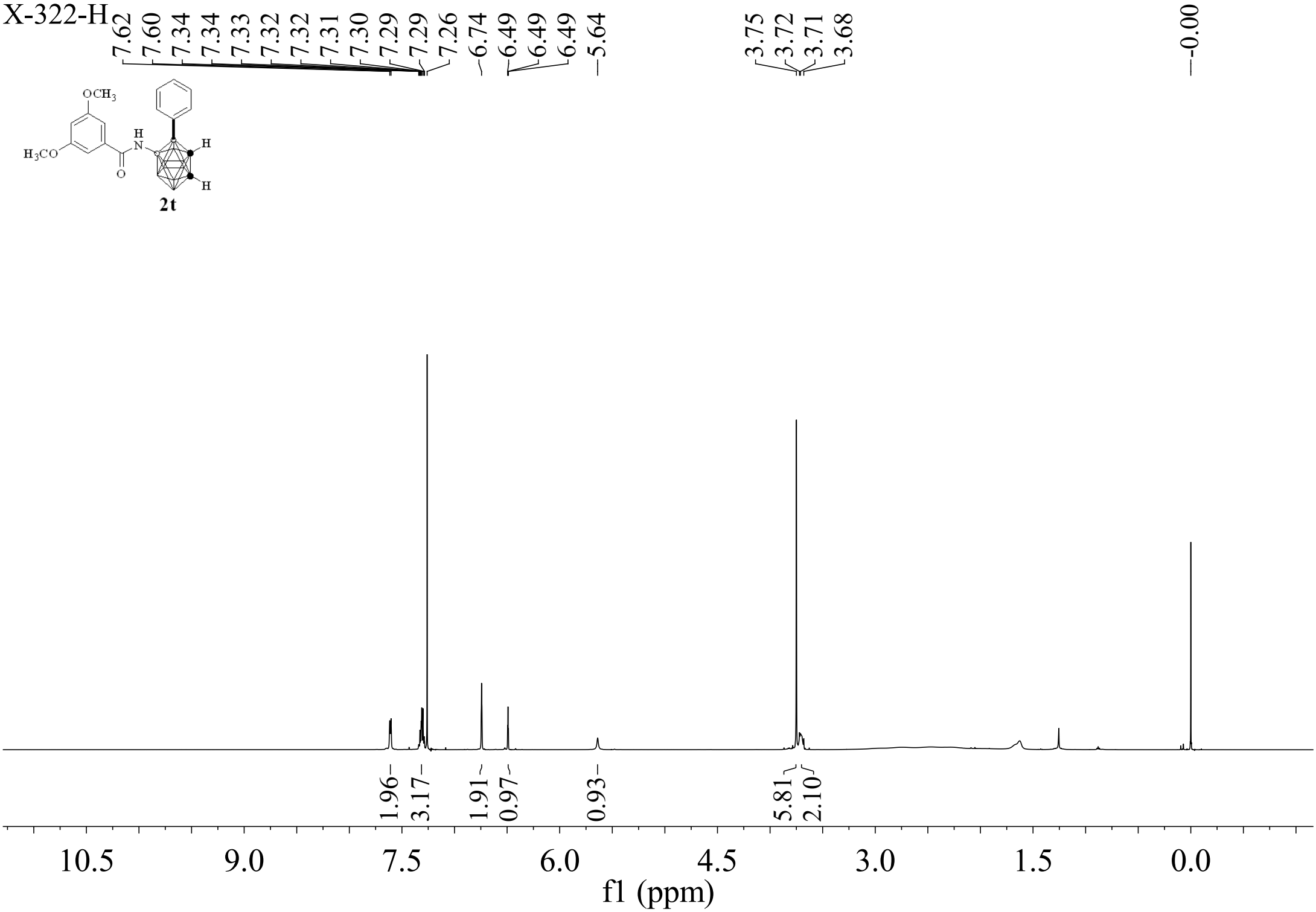
2s


-6.7
-2.6
-4.3
-9.6
-14.0
-16.8
1.00
0.89
1.48
1.73
0.86
3.05
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

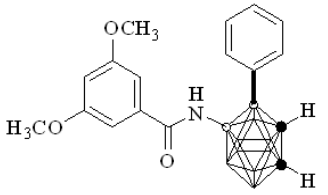
X-322-H

OCH3
H3CO
H
N
O
H
H

2t

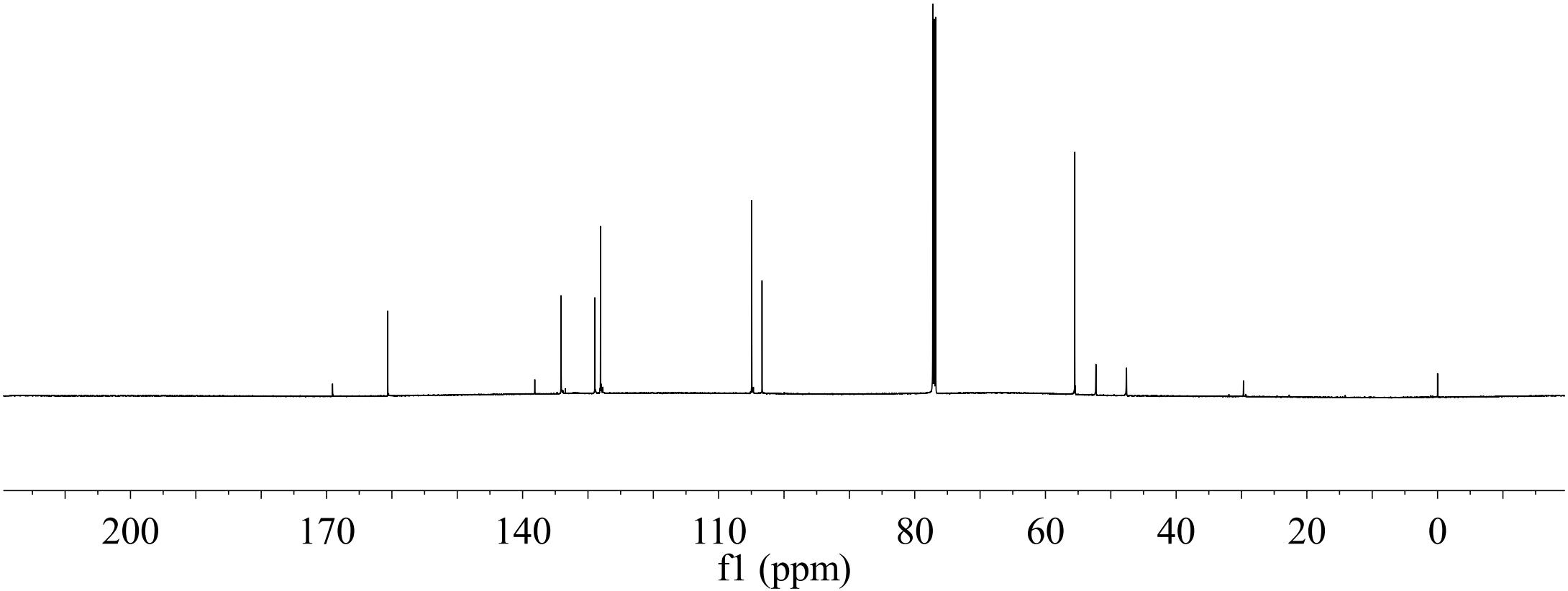

7.62
7.60
7.34
7.34
7.33
7.32
7.32
7.31
7.30
7.29
7.29
7.26
6.74
6.49
6.49
6.49
5.64
3.75
3.72
3.71
3.68
0.00
1.96
3.17
1.91
0.97
0.93
5.81
2.10
10.5
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-322-C



2t

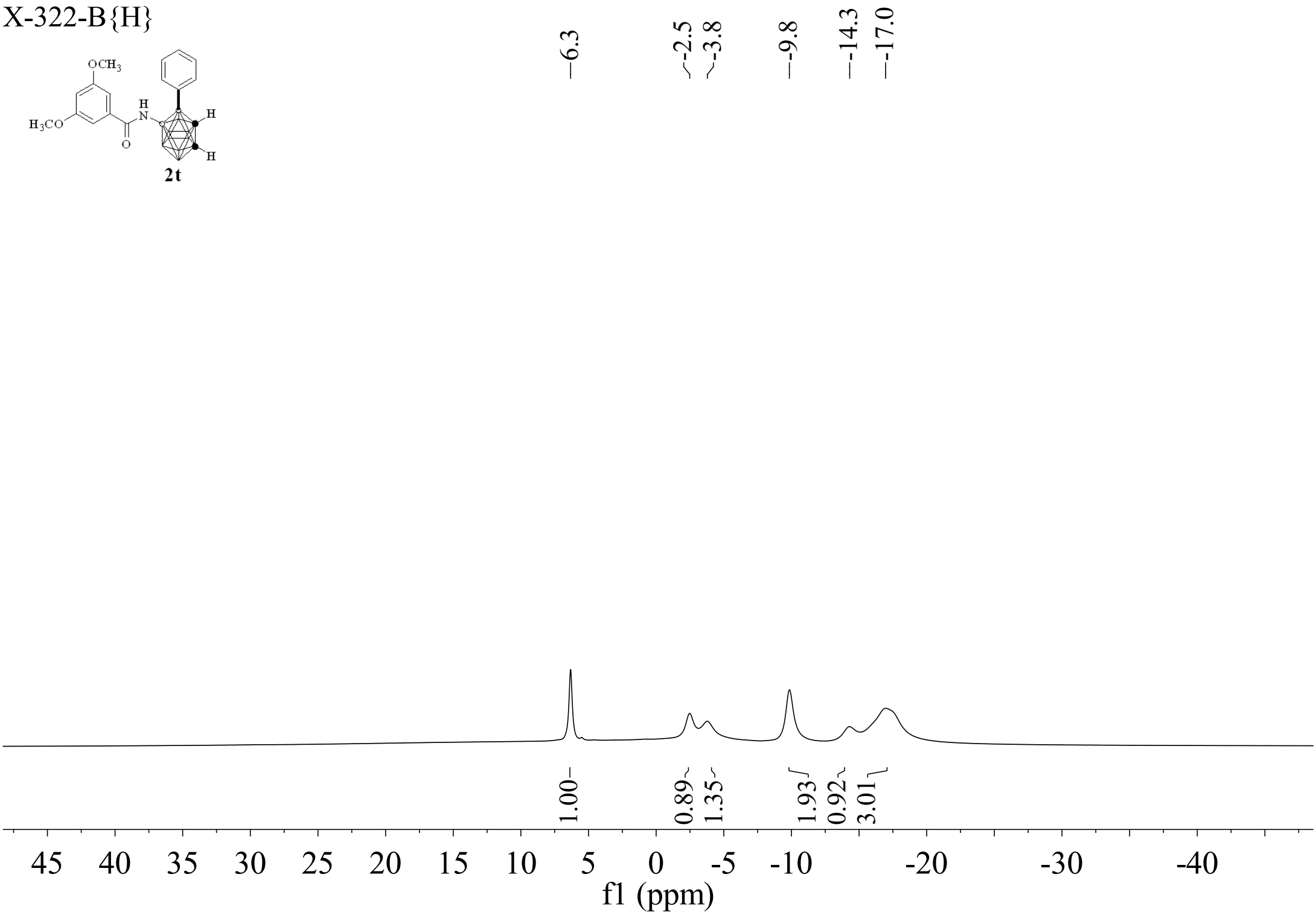
169.1
160.6
138.1
134.1
129.0
128.1
104.9
103.4
77.2
77.0
76.8
55.5
52.3
47.6
0.0



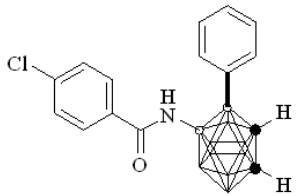
X-322-B{H}

OCH3
H3CO
H
N
O
H
H

2t


-6.3
-2.5
-3.8
-9.8
-14.3
-17.0
1.00
0.89
1.35
1.93
0.92
3.01
45 40 35 30 25 20 15 10 5 0 -5 -10 -20 -30 -40
f1 (ppm)

X-300-H



2u

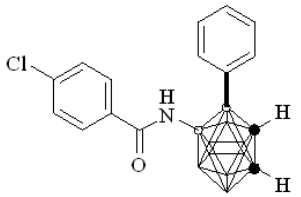
7.61 7.59 7.54 7.53 7.35 7.34 7.32 7.31 7.30 7.29 7.26 5.63 3.74 0.00

1.94 1.99 5.15 0.92 2.00

9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

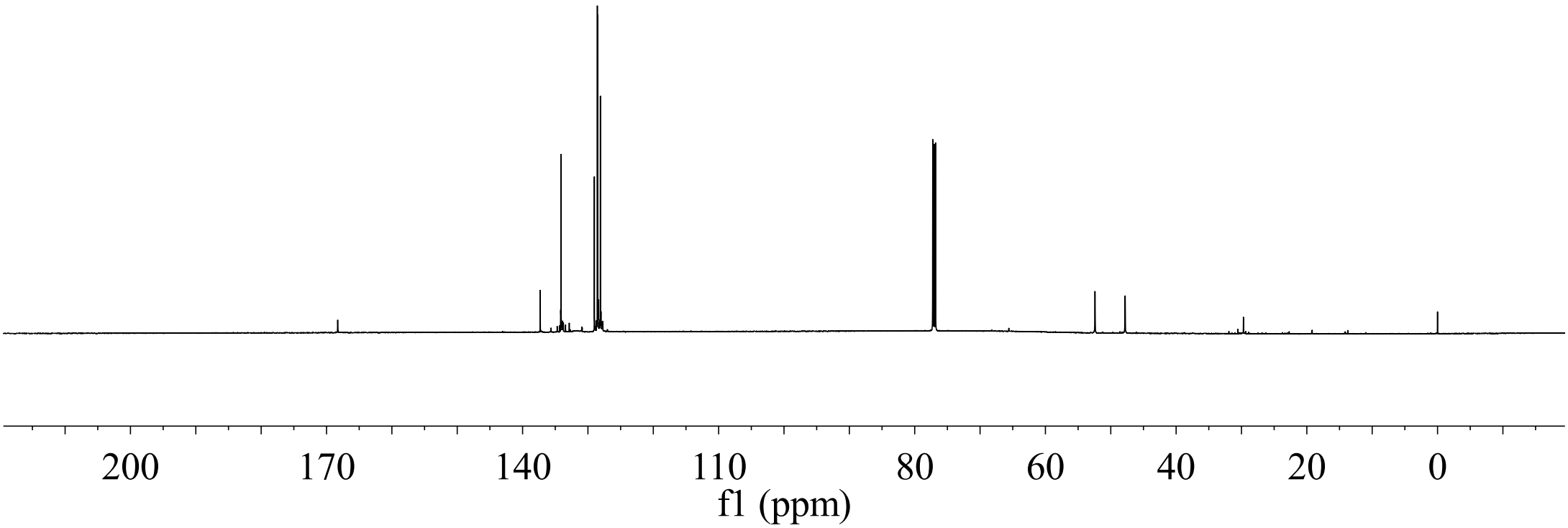
f1 (ppm)

X-300-C



2u

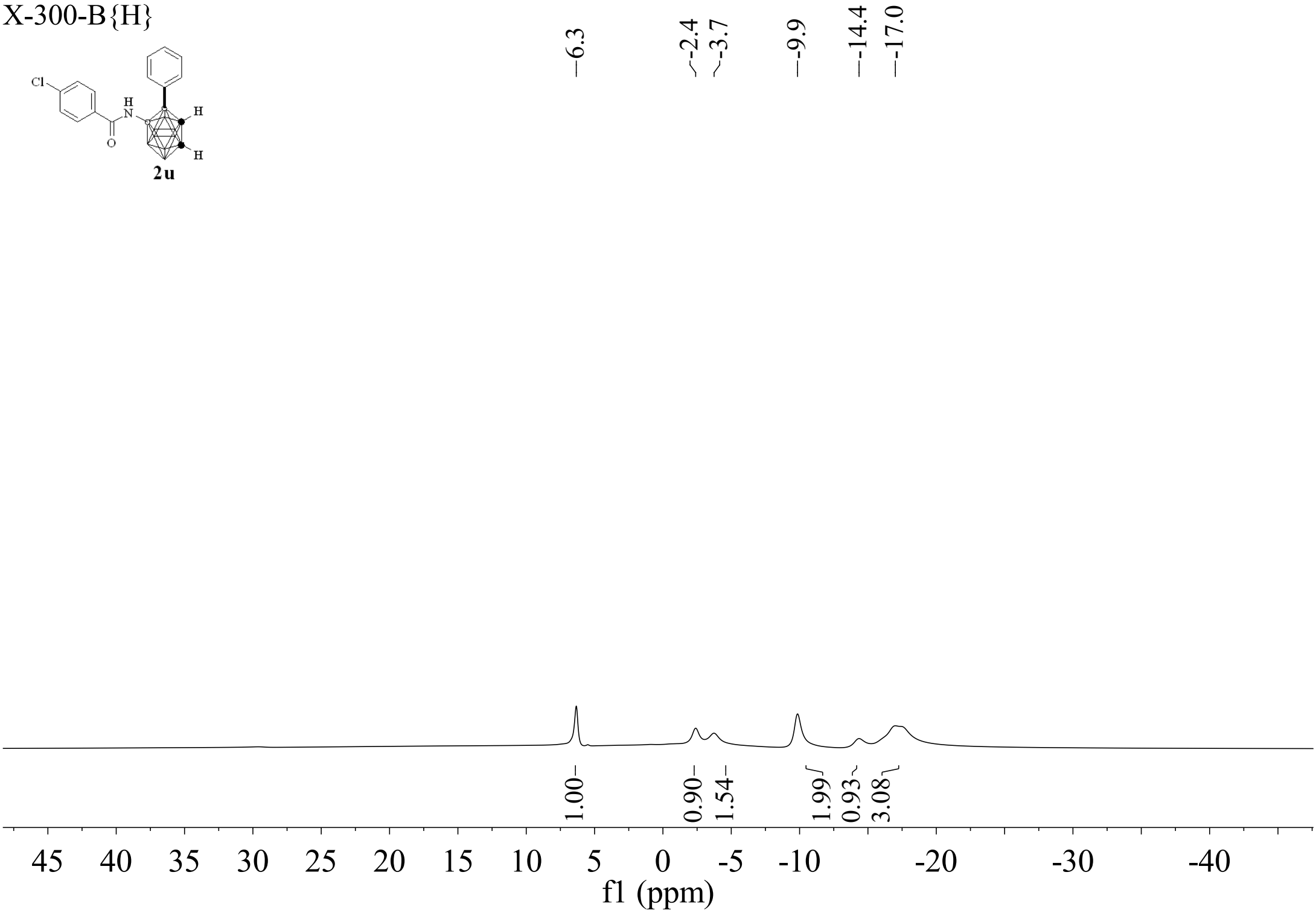
168.3
137.3
134.2
134.1
129.0
128.6
128.5
128.1
77.2
77.0
76.8
52.4
47.8
0.0



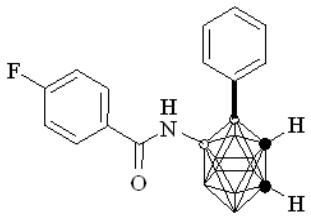
X-300-B{H}

Cl
H
N
O
H
H

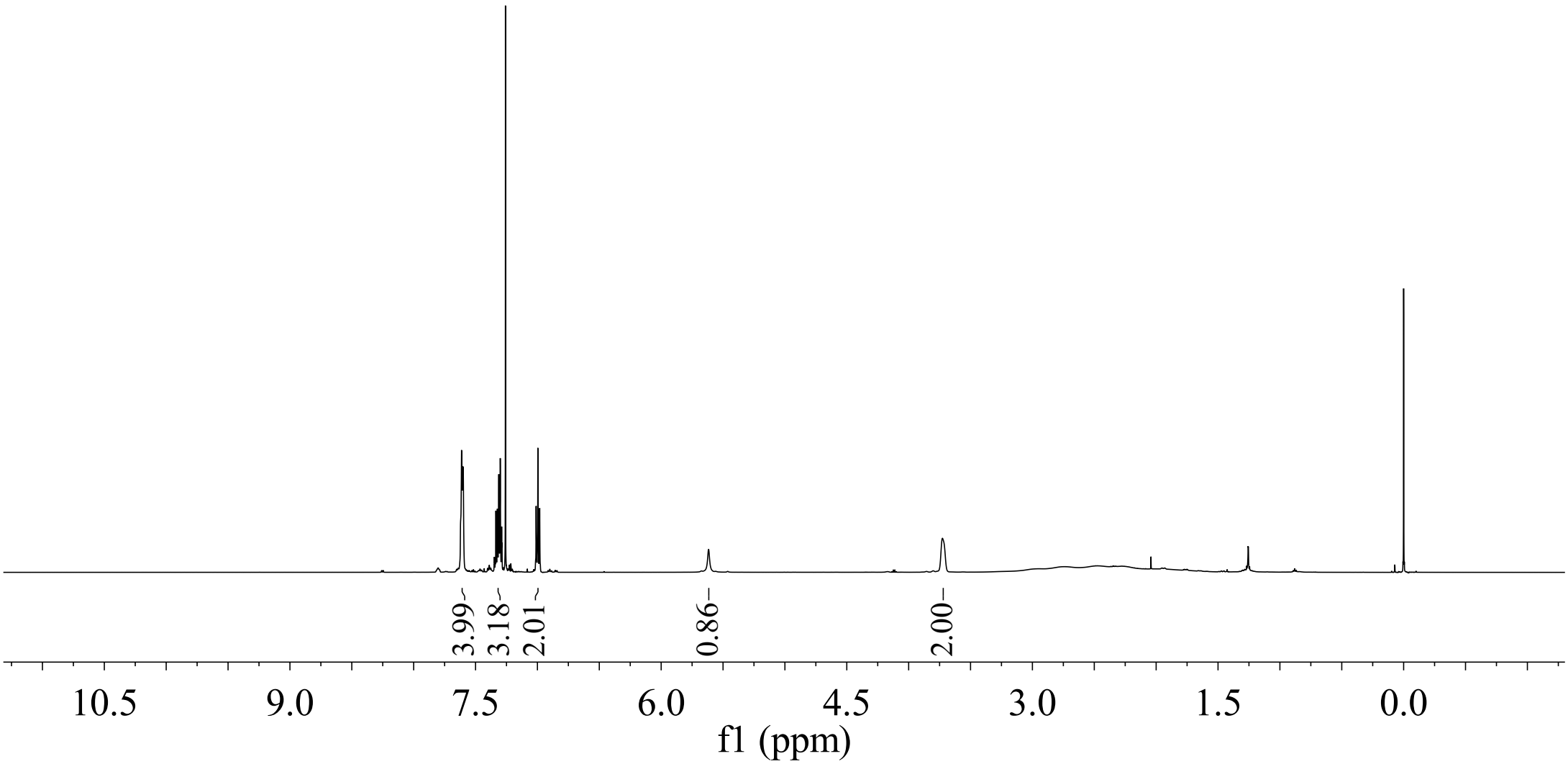
2u


-6.3
-2.4
-3.7
-9.9
-14.4
-17.0
1.00
0.90
1.54
1.99
0.93
3.08
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

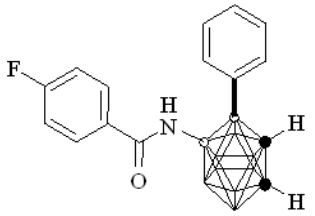
X-317-H


F
H
N
O
H
H

2v

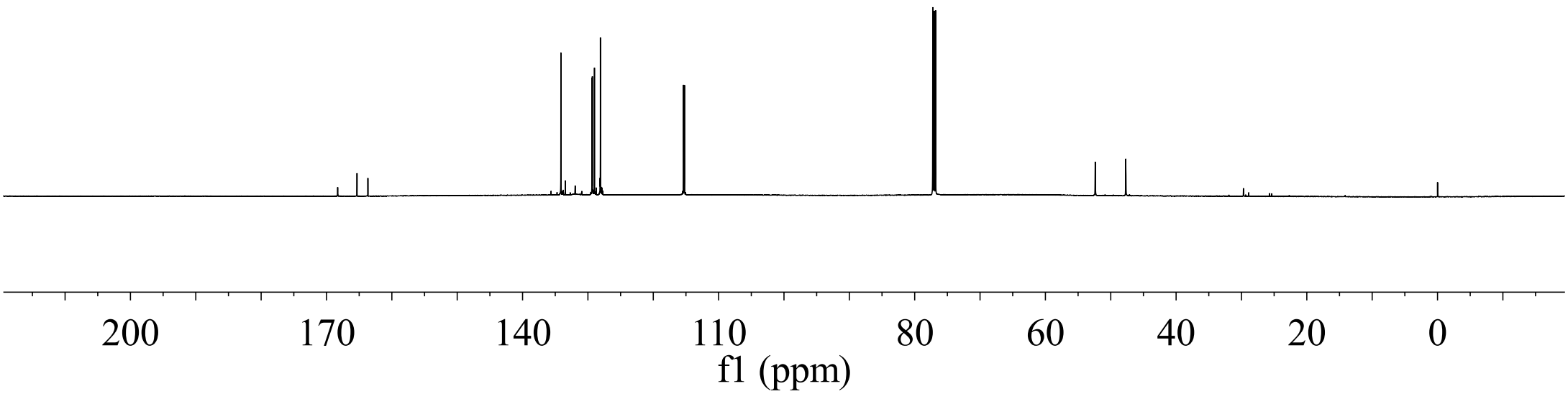

7.61
7.60
7.60
7.60
7.35
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.29
7.26
7.01
7.00
6.98
5.82
3.73
0.00
3.99
3.18
2.01
0.86
2.00
10.5
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-317-C



2v

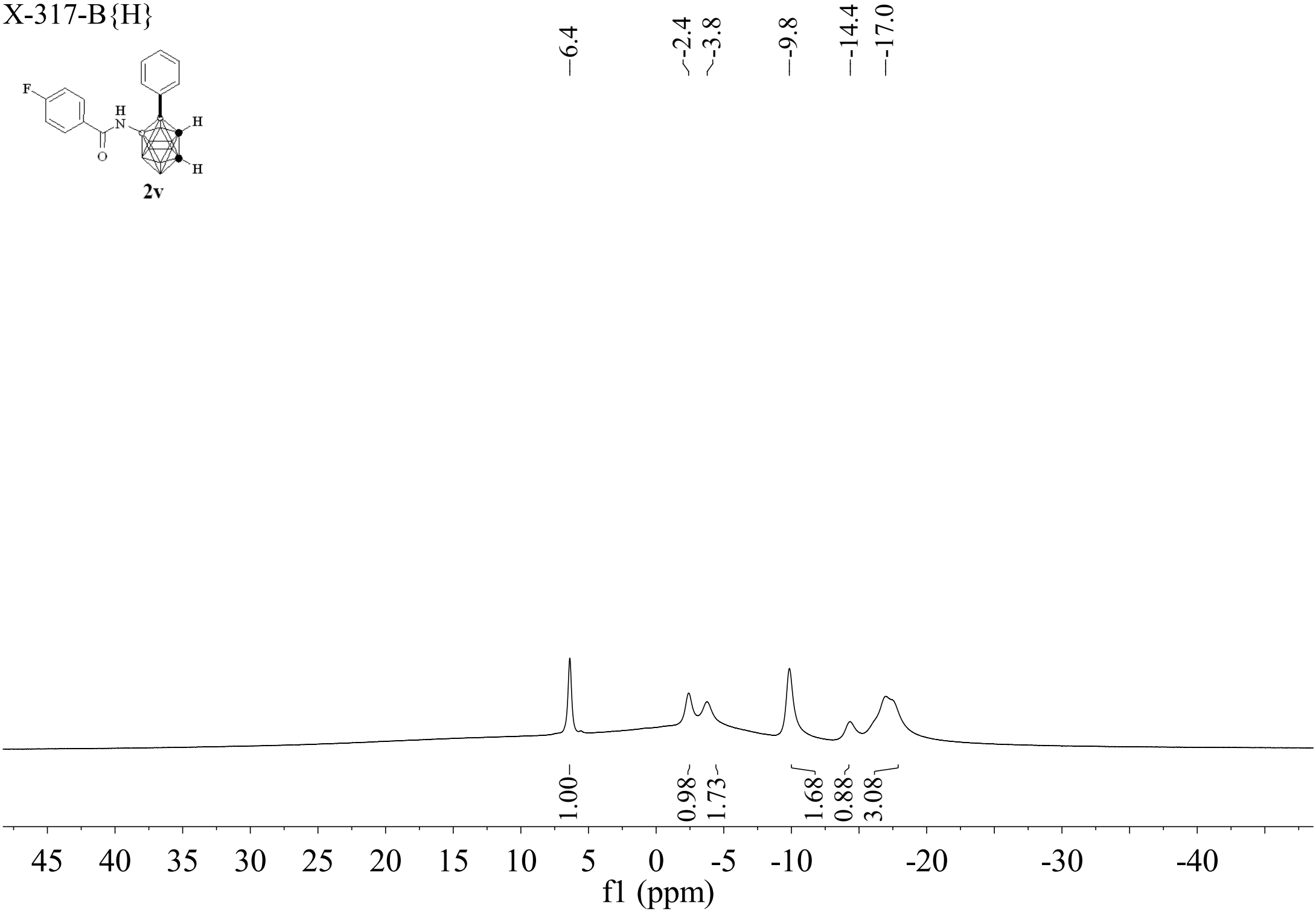
168.3
165.3
163.7
134.1
129.4
129.3
129.0
128.1
115.4
115.2
77.2
77.0
76.8
52.4
47.7
0.0



X-317-B{H}

F
H
N
O
H
H

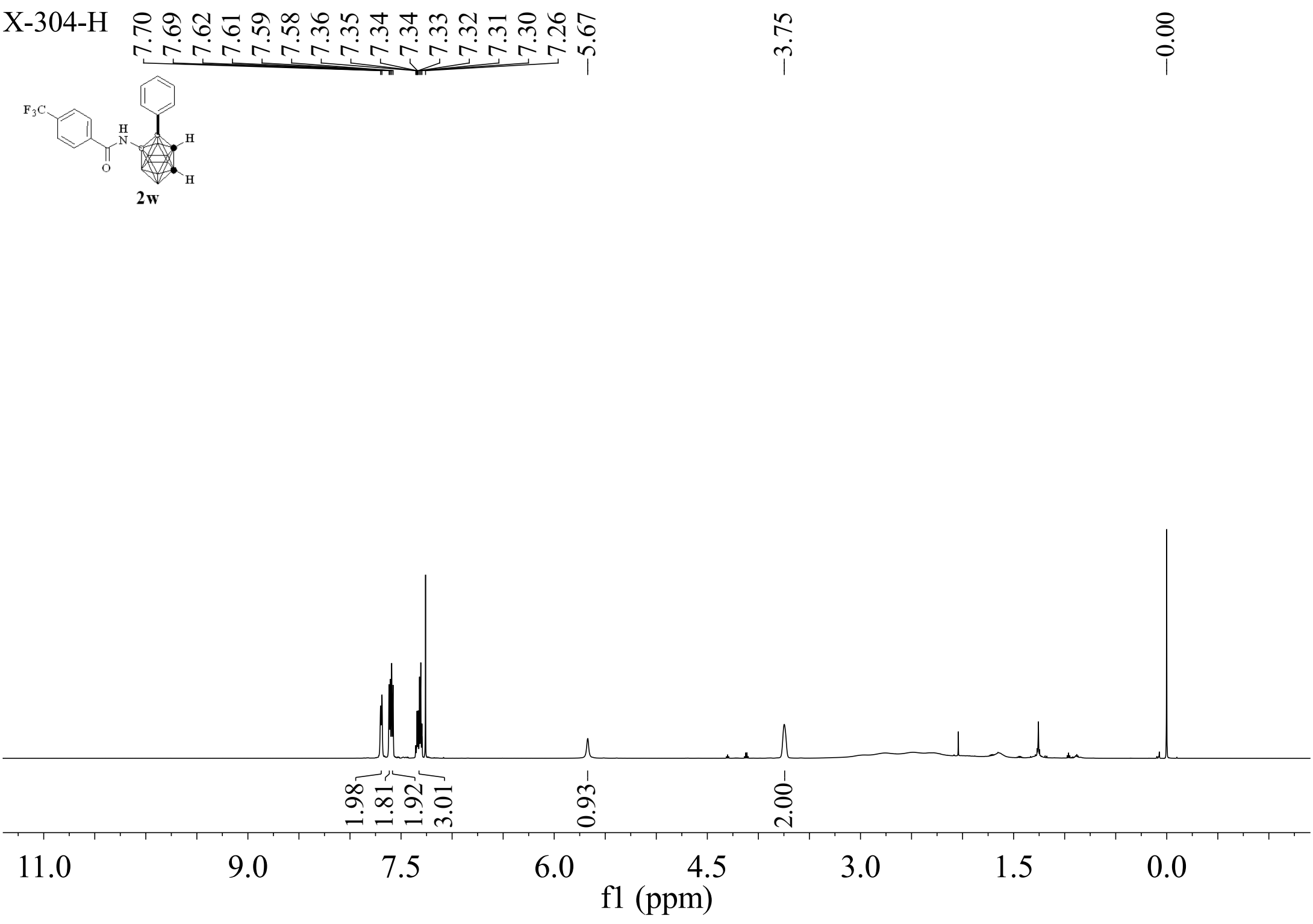
2v


-6.4
-2.4
-3.8
-9.8
-14.4
-17.0
1.00
0.98
1.73
1.68
0.88
3.08
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

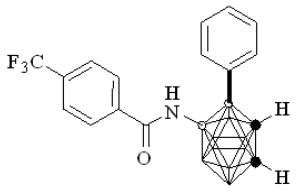
X-304-H

F3C
H
N
O
H
H

2w

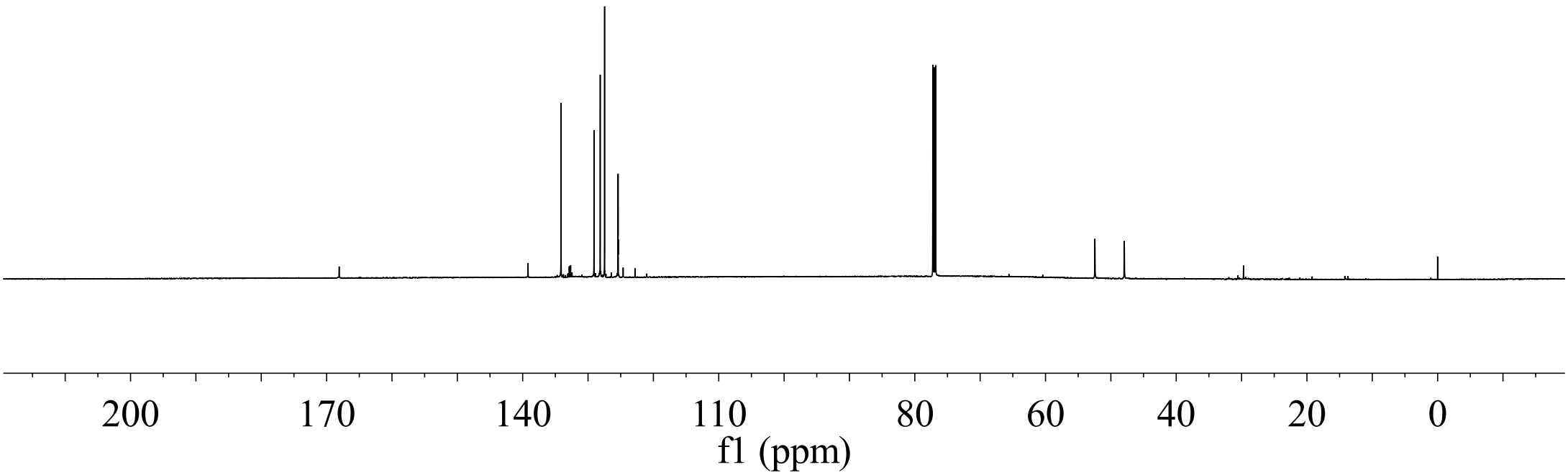

7.70
7.69
7.62
7.61
7.59
7.58
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.30
7.26
5.67
3.75
0.00
1.98
1.81
1.92
3.01
0.93
2.00
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-304-C



2w

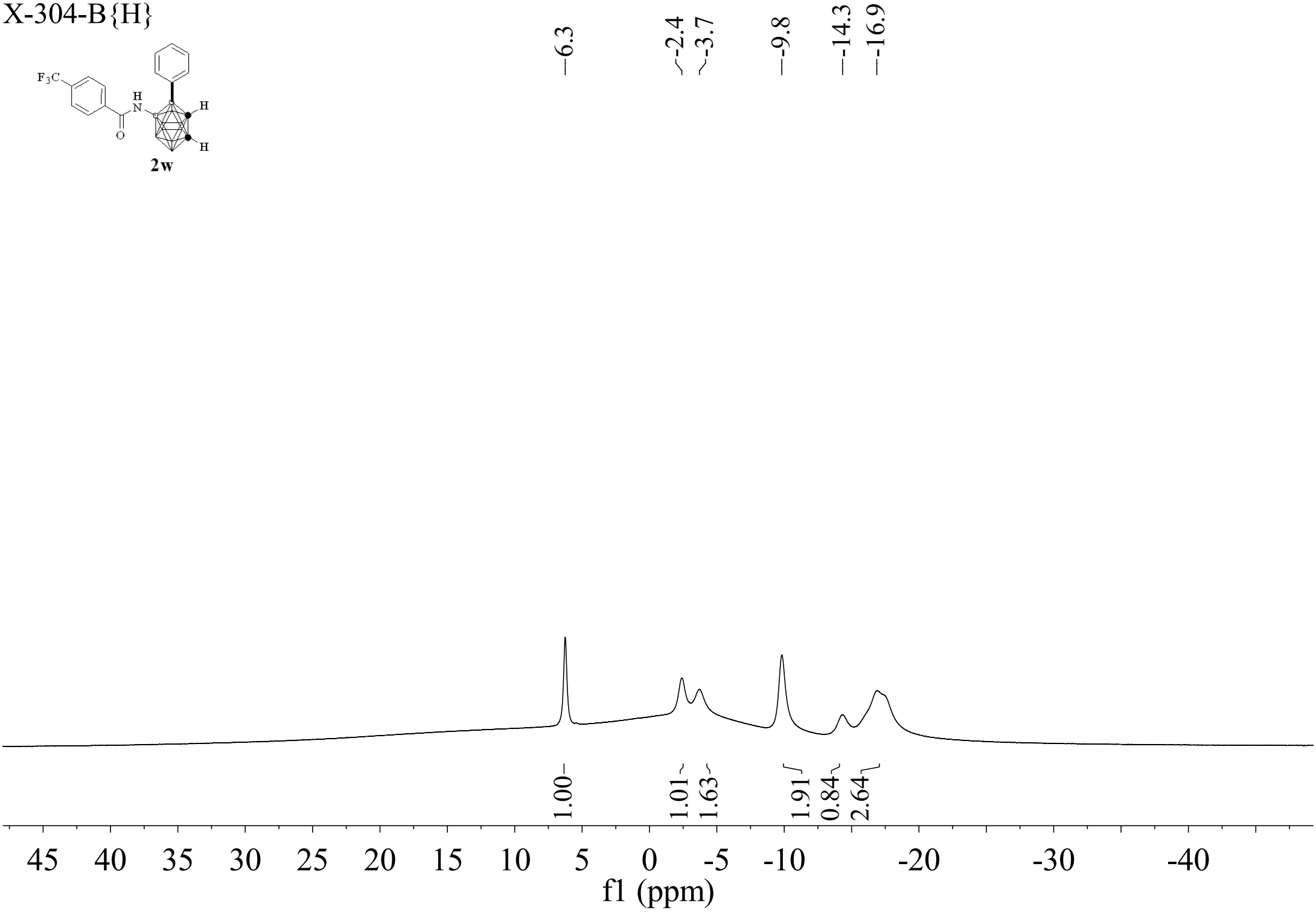
168.1
139.2
134.1
133.1
132.9
132.7
132.4
129.1
128.1
127.5
125.4
125.4
125.4
125.4
77.2
77.0
76.8
52.4
47.9
0.0



X-304-B{H}

F3C
H
N
O
H
H

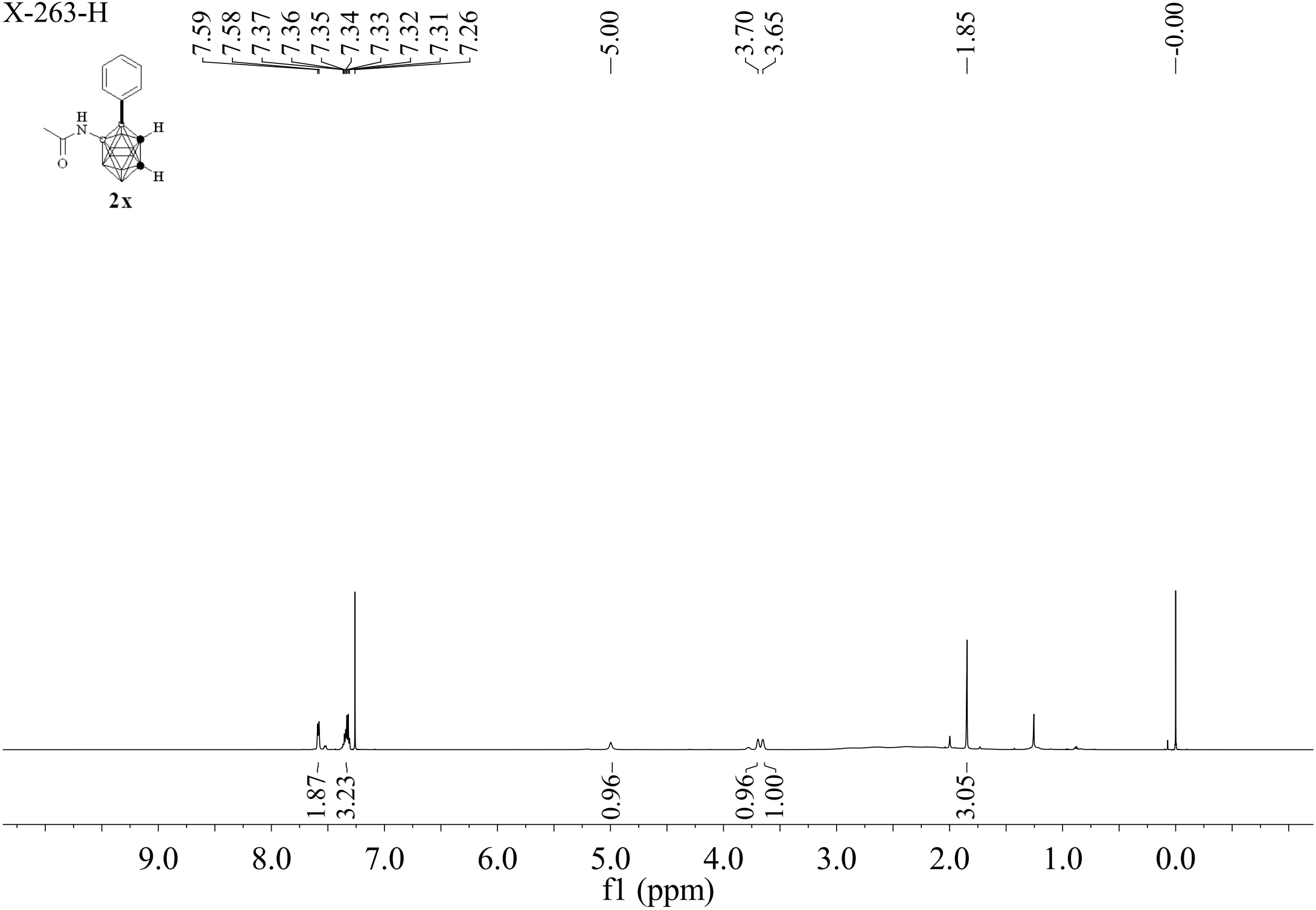
2w


-6.3
-2.4
-3.7
-9.8
-14.3
-16.9
1.00
1.01
1.63
1.91
0.84
2.64
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

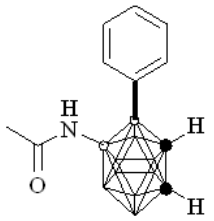
X-263-H

H
N
O
H
H

2x


7.59
7.58
7.37
7.36
7.35
7.34
7.33
7.32
7.31
7.26
5.00
3.70
3.65
1.85
0.00
1.87
3.23
0.96
0.96
1.00
3.05
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

X-263-C



2x

172.4

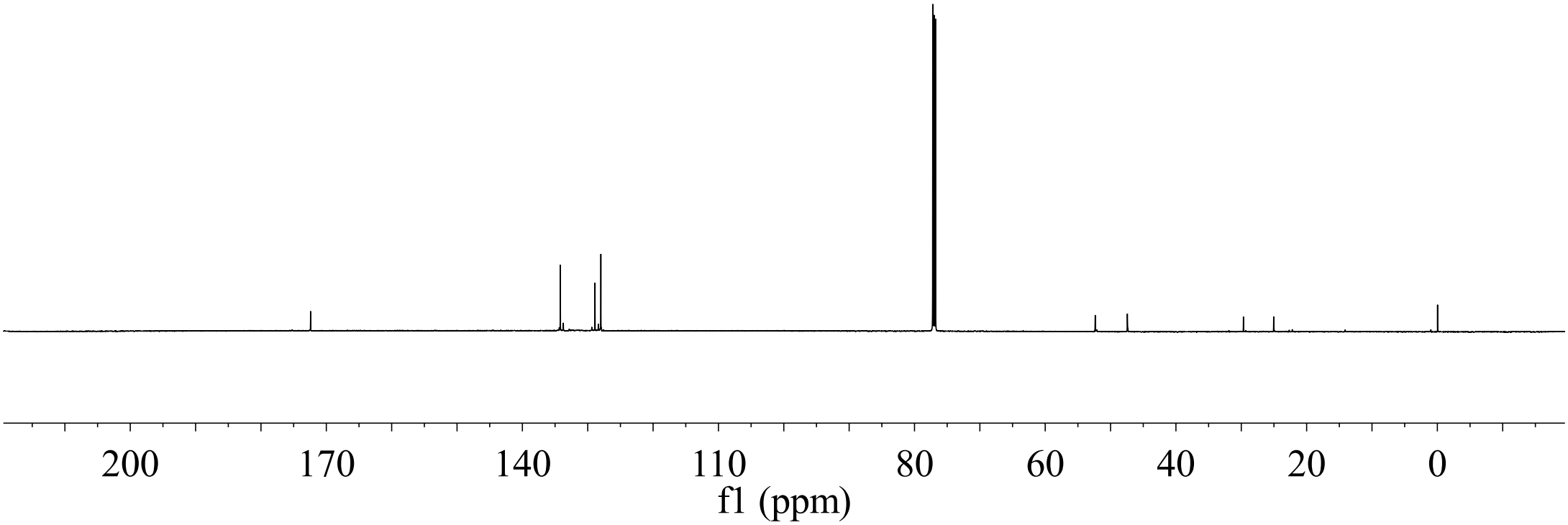
134.2
133.7
128.9
128.0

77.2
77.0
76.8

52.3
47.4

25.0

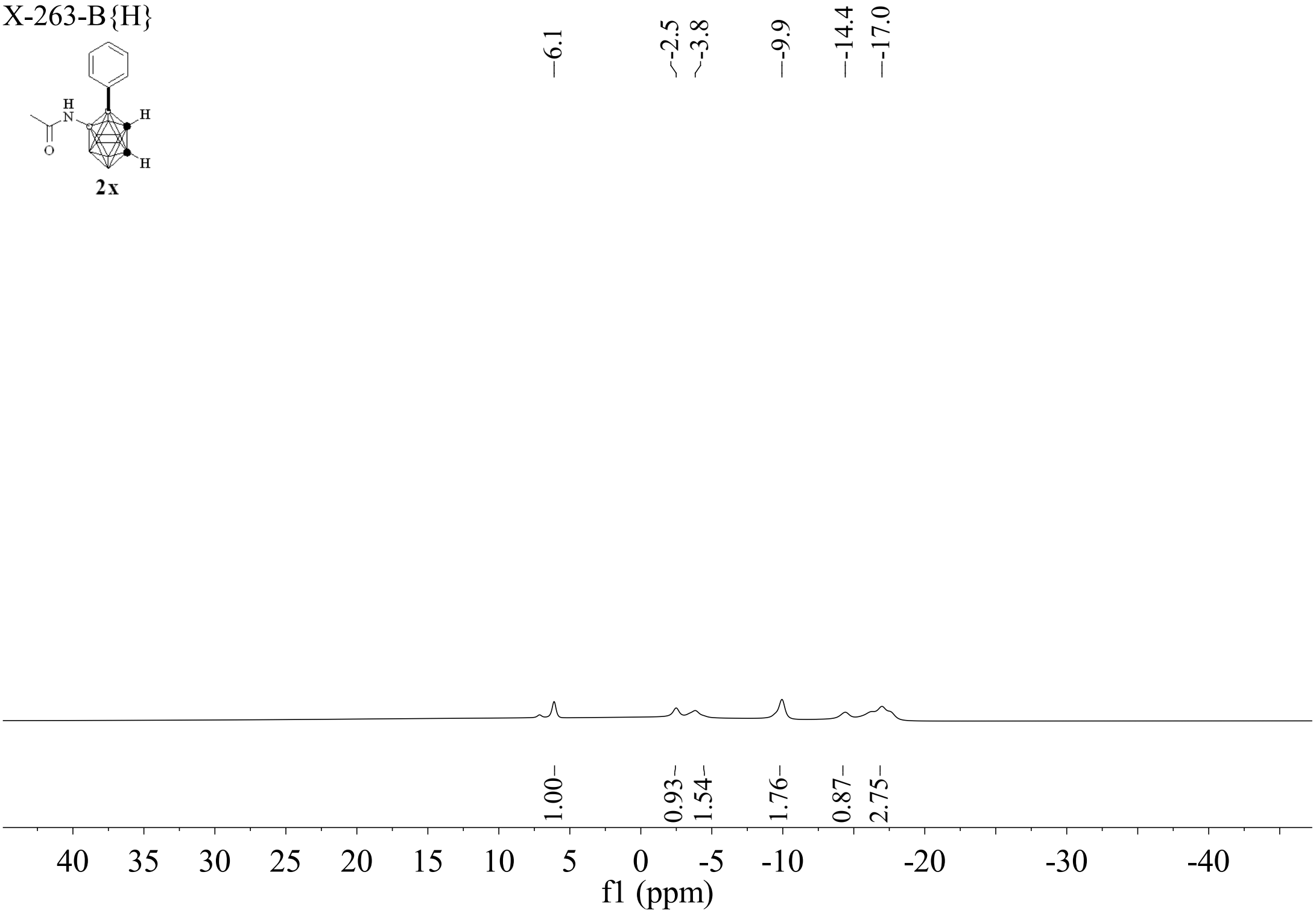
0.0



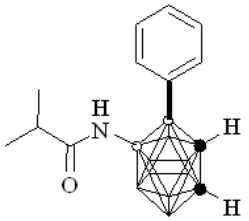
X-263-B{H}

H
N
O
H
H

2x

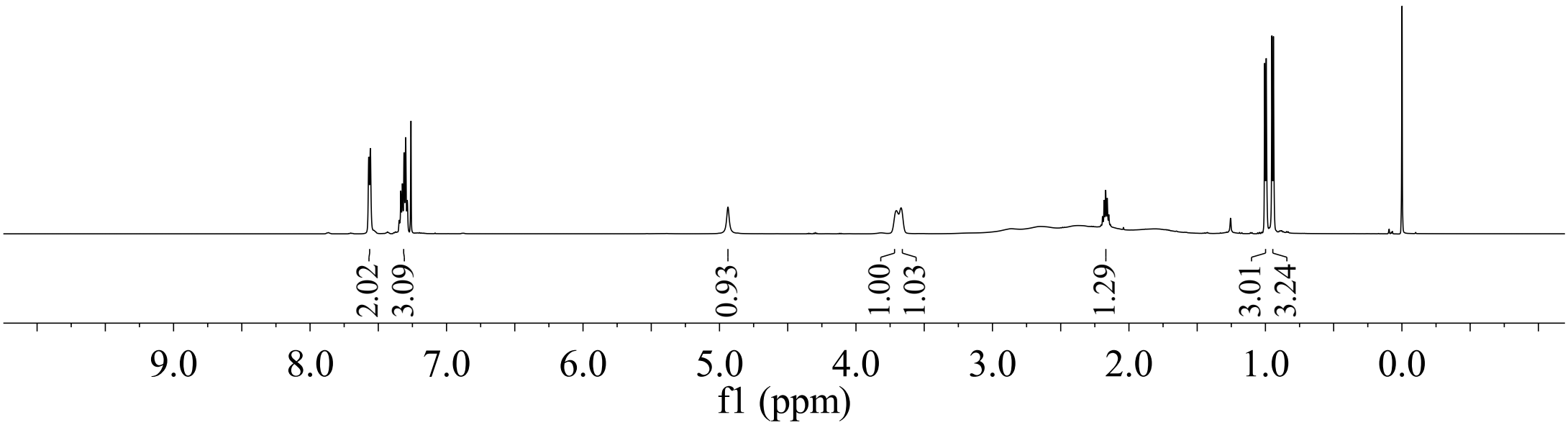

-6.1
-2.5
-3.8
-9.9
-14.4
-17.0
1.00
0.93
1.54
1.76
0.87
2.75
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

X-306-H

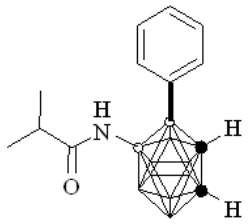


2y

7.57 7.56 7.35 7.34 7.32 7.31 7.30 7.29 7.26 4.94 3.70 3.67 2.20 2.19 2.18 2.17 2.16 2.15 2.14 1.01 0.99 0.95 0.94 0.00



X-306-C



2y

179.3

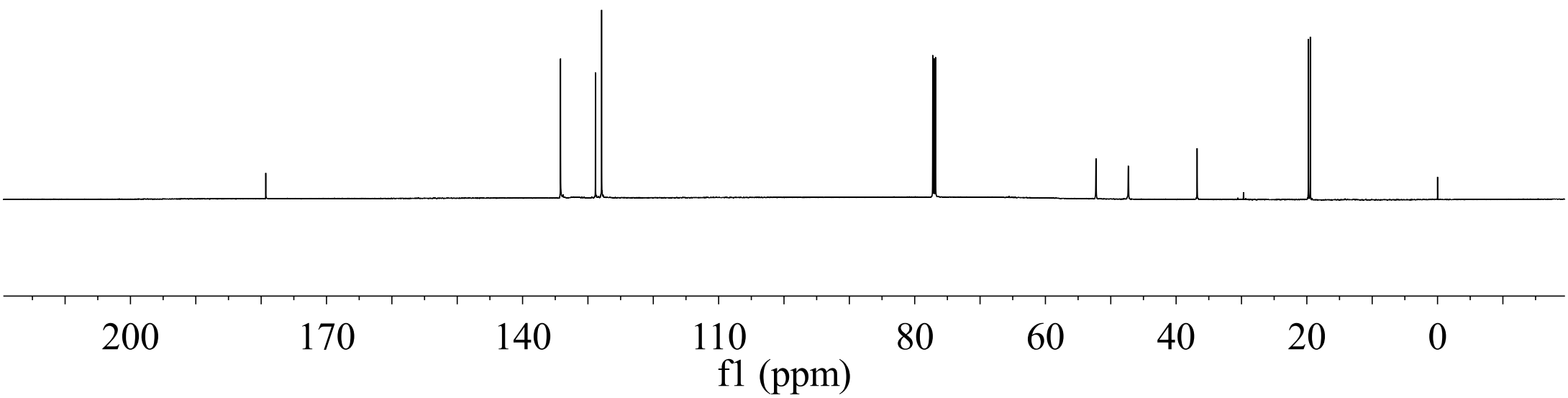
134.2
128.9
127.9

77.2
77.0
76.8

52.3
47.3
36.8

19.8
19.5

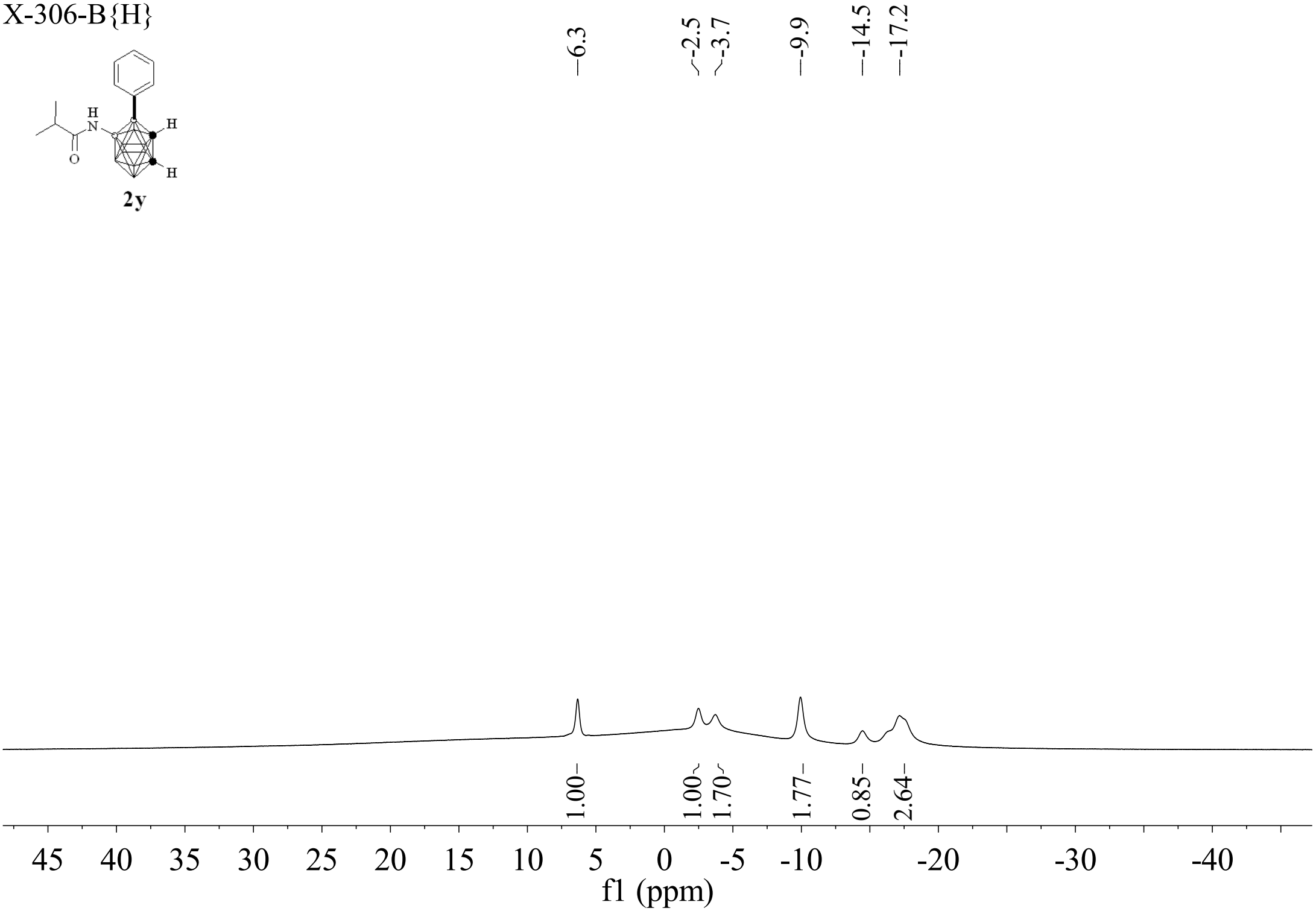
0.0



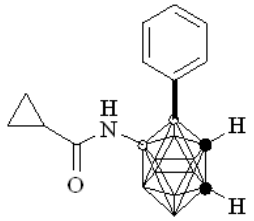
X-306-B{H}

H
N
O
H
H

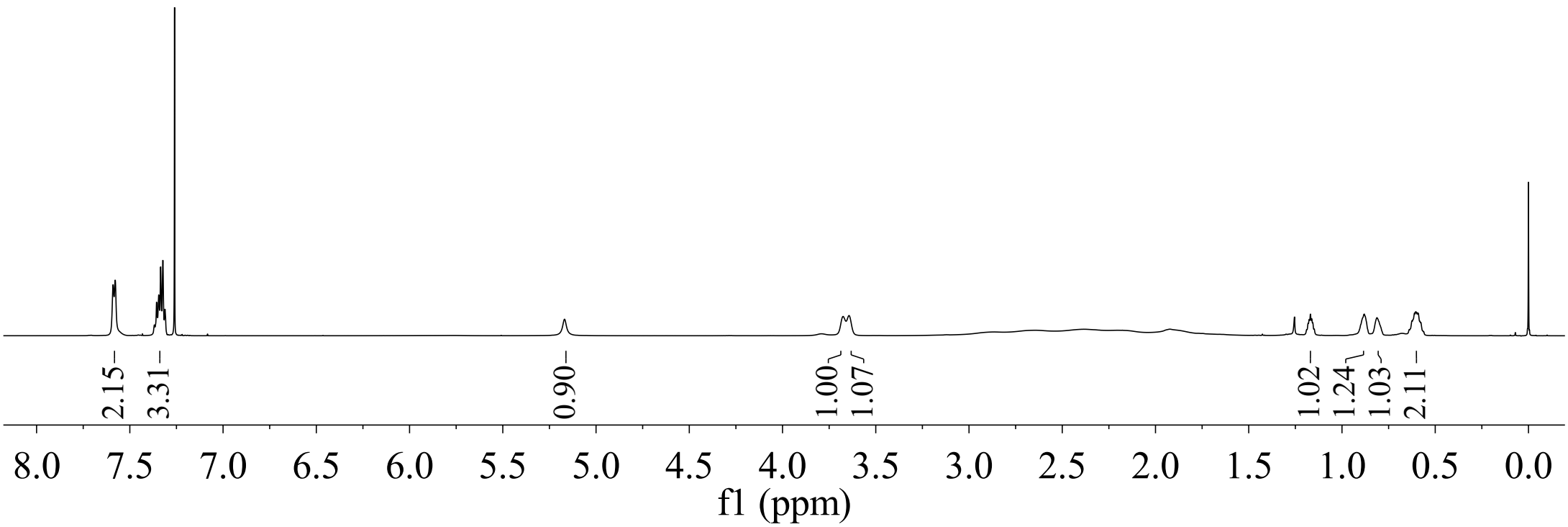
2y


-6.3
-2.5
-3.7
-9.9
-14.5
-17.2
1.00
1.00
1.70
1.77
0.85
2.64
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

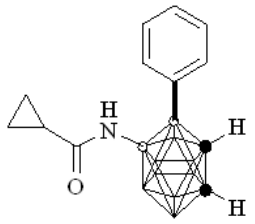
X-316-H


H
N
O
H
H

2z

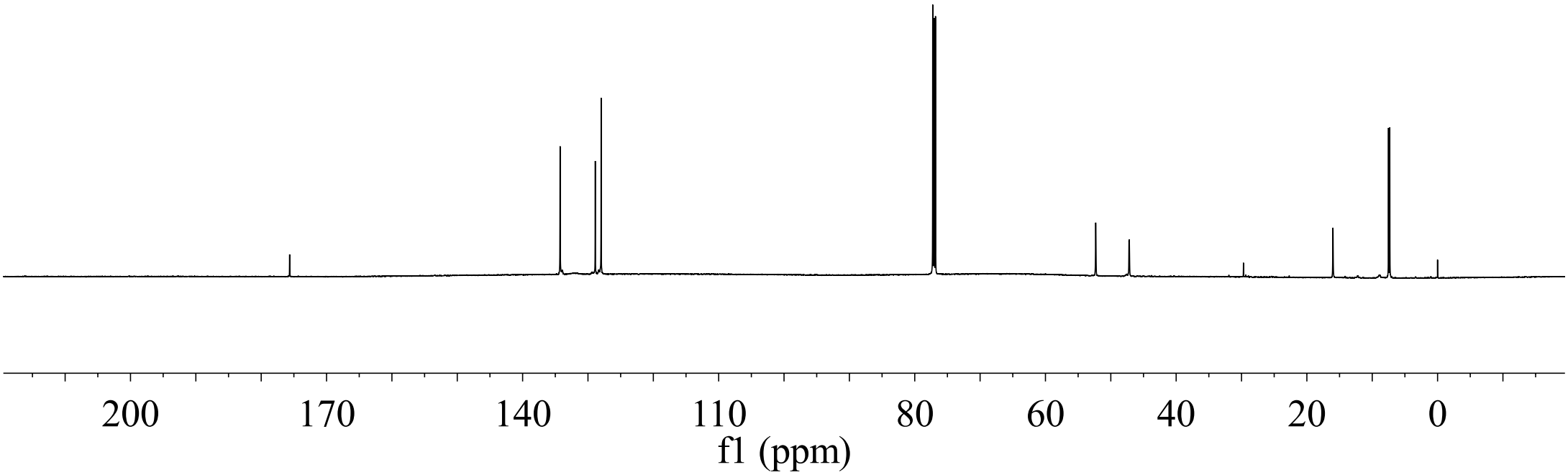

7.58
7.58
7.37
7.36
7.34
7.33
7.32
7.31
7.26
5.17
3.68
3.64
1.19
1.18
1.18
1.17
1.16
1.16
1.15
0.88
0.81
0.62
0.61
0.61
0.60
0.60
0.59
0.58
0.58
0.00
2.15
3.31
0.90
1.00
1.07
1.02
1.24
1.03
2.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

X-316-C



2z

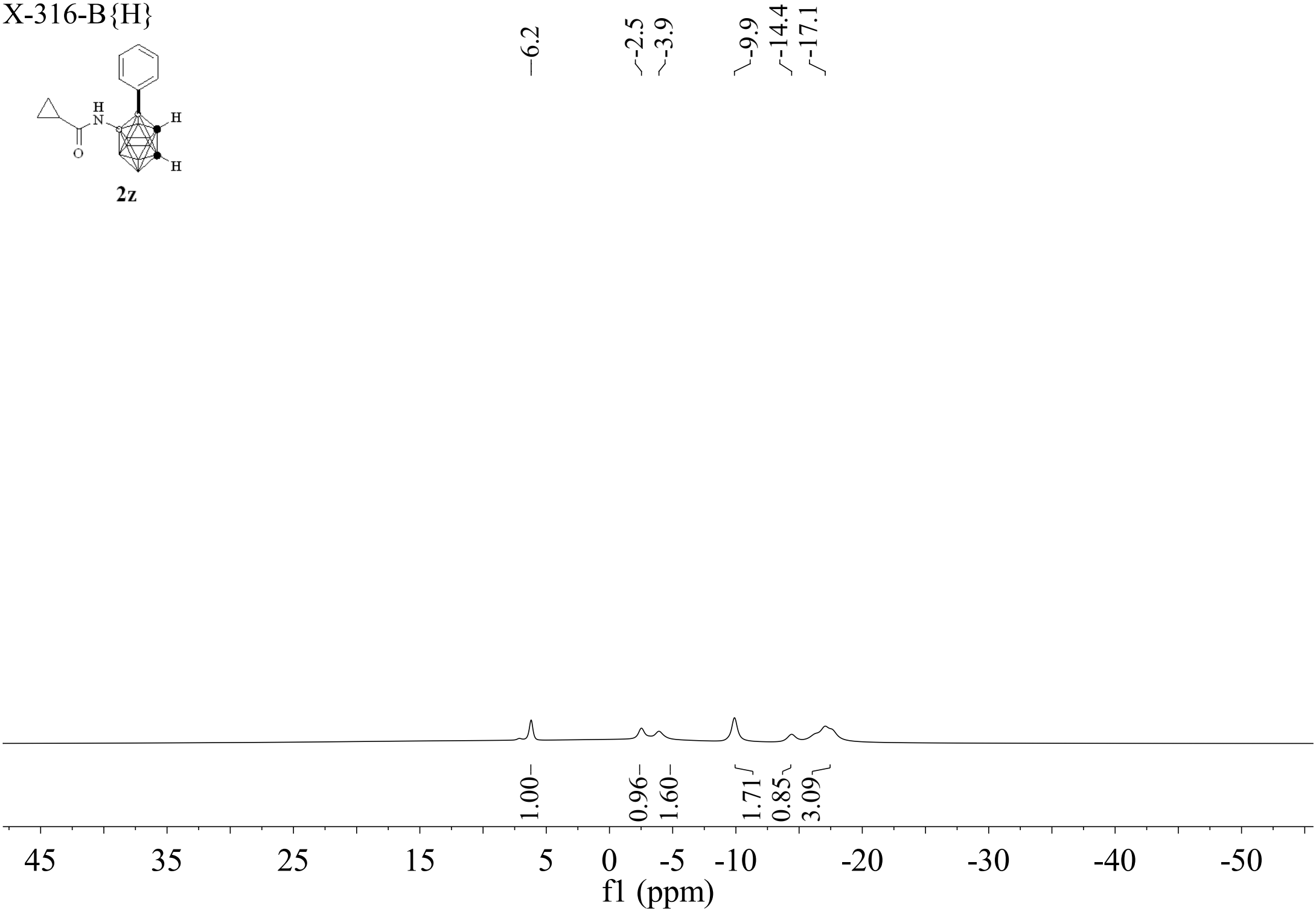
175.6
134.3
128.9
128.0
77.2
77.0
76.8
52.3
47.2
16.0
7.5
7.3
0.0



X-316-B{H}

H
N
O
H
H

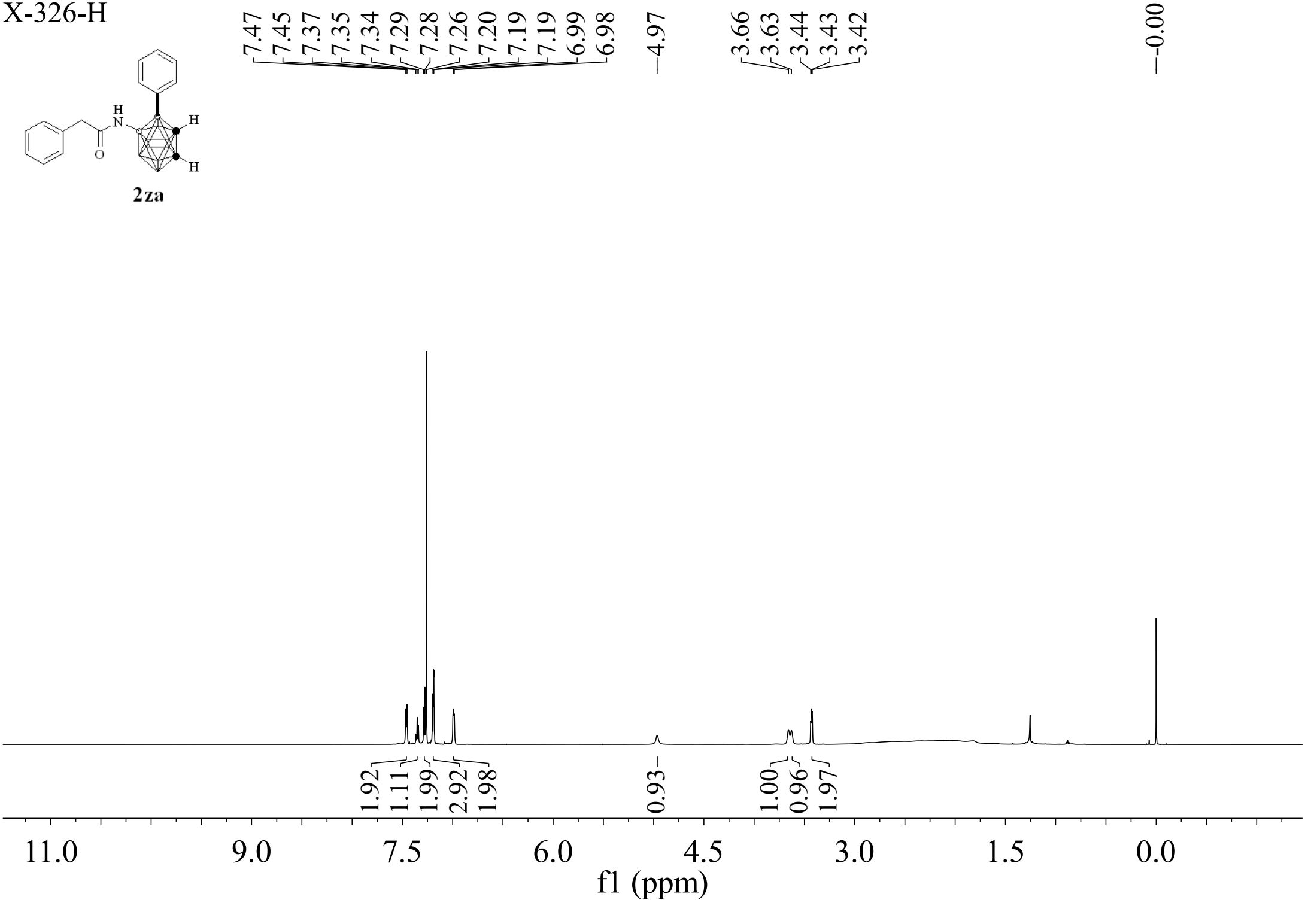
2z


6.2
-2.5
-3.9
-9.9
-14.4
-17.1
1.00
0.96
1.60
1.71
0.85
3.09
45
35
25
15
5
0
-5
-10
-20
-30
-40
-50
f1 (ppm)

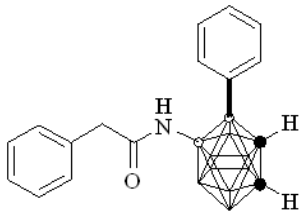
X-326-H

H
N
O
H
H

2za


7.47
7.45
7.37
7.35
7.34
7.29
7.28
7.26
7.20
7.19
7.19
6.99
6.98
4.97
3.66
3.63
3.44
3.43
3.42
0.00
1.92
1.11
1.99
2.92
1.98
0.93
1.00
0.96
1.97
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

X-326-C



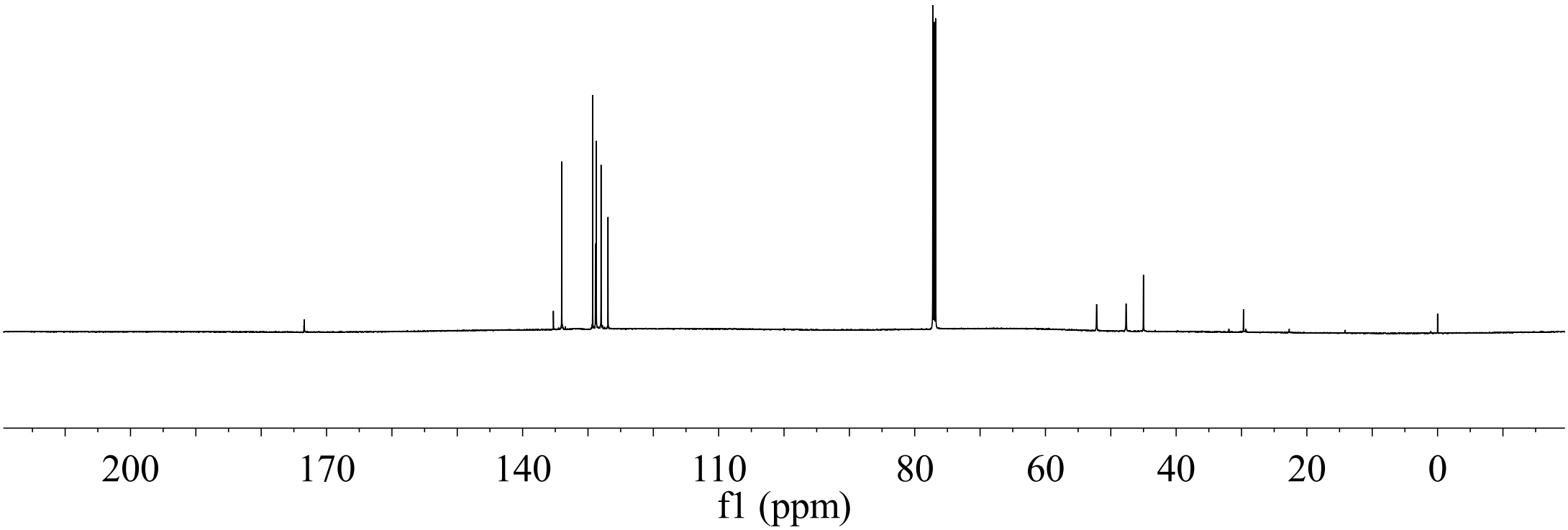
2za

173.4

135.3
134.0
129.3
128.9
128.7
128.0
126.9

77.2
77.0
76.8

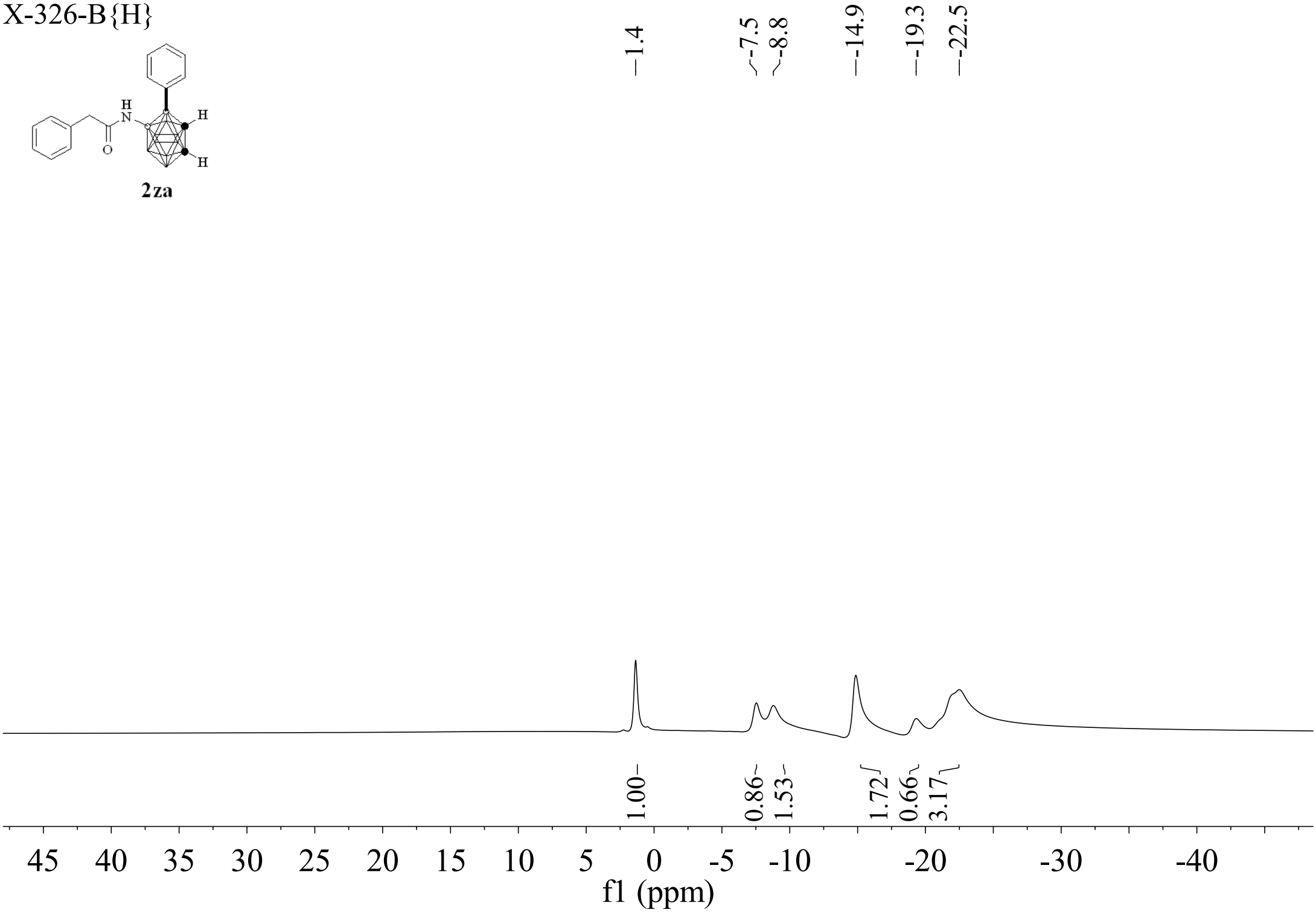
52.2
47.7
45.0



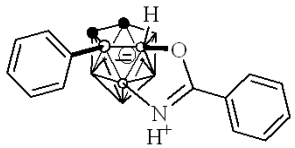
X-326-B{H}

H
N
O
H
H

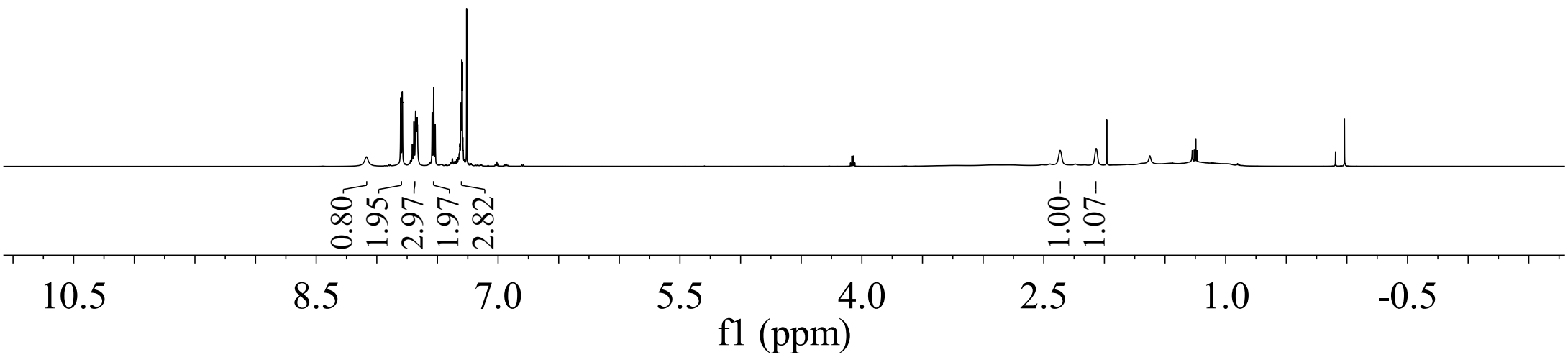
2za


-1.4
-7.5
-8.8
-14.9
-19.3
-22.5
1.00
0.86
1.53
1.72
0.66
3.17
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

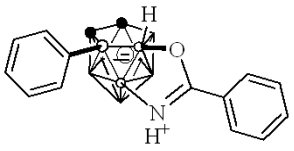
X-363-H


H
O
N
H+

3a


8.03
7.83
7.79
7.71
7.69
7.68
7.68
7.67
7.66
7.54
7.53
7.52
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.26
2.37
2.07
0.80
1.95
2.97
1.97
2.82
1.00
1.07
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

X-363-C



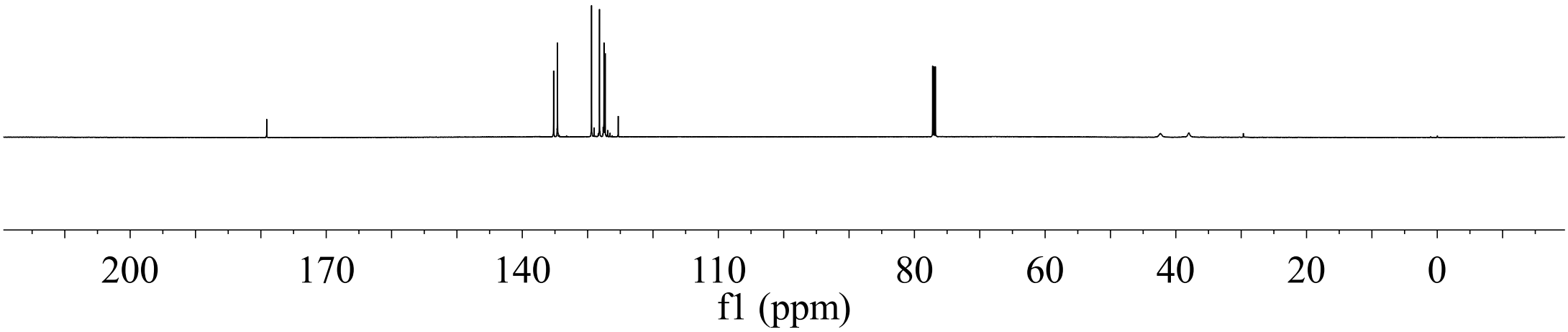
3a

179.1

135.2
134.6
129.4
128.2
127.5
127.3
125.3

77.2
77.0
76.8

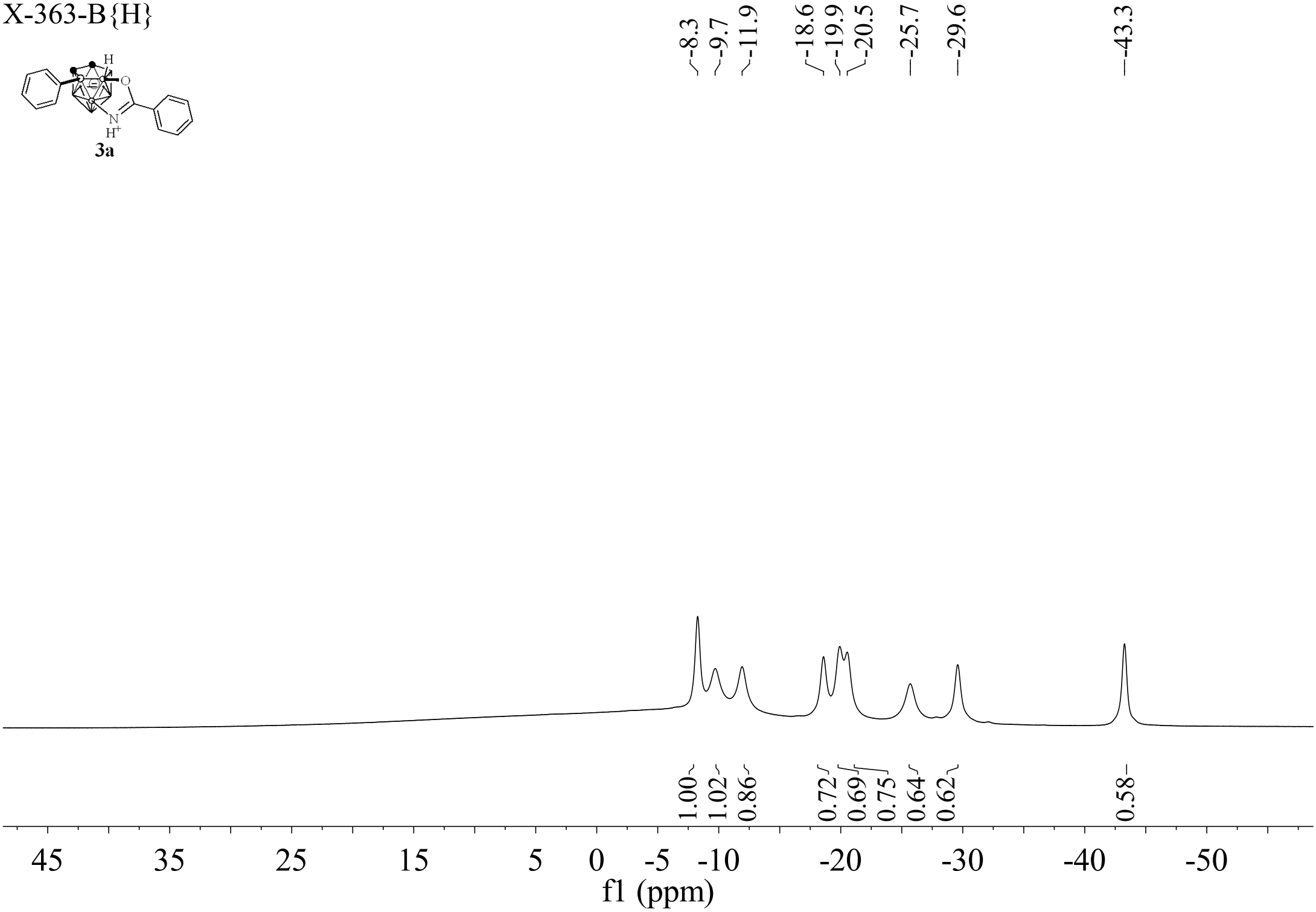
42.4
38.0



X-363-B{H}

H
O
N
H+

3a


-8.3
-9.7
-11.9
-18.6
-19.9
-20.5
-25.7
-29.6
-43.3
1.00
1.02
0.86
0.72
0.69
0.75
0.64
0.62
0.58
45
35
25
15
5
0
-5
-10
-20
-30
-40
-50
f1 (ppm)