Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Palladium catalyzed/silver tuned selective mono-/tetra-acetoxylation of *o*-carboranes via B-H activation

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Context

General information

1a-1i[1], 1k-1o[2] and 4a-4b[3] were synthesized according to literature methods. Other materials were purchased from J&K Scientific 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 NMR, ^{13}C{^{1}H} and ^{11}B{^{1}H} NMR spectra were recorded on the Bruker 600MHz or 400MHz instruments. All ^{1}H NMR and ^{13}C{^{1}H} NMR spectral 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. HRMS data were measured with ESI techniques.

Experimental

General procedure for palladium catalyzed selective tetra-acetoxylation of *o*-carboranes:

To a 10 mL dried flask were sequentially added *o*-carborane (0.25 mmol), AcOH (1 mL), Ac_2O (1ml), $Pd(OAc)_2$ (1.12 mg, 0.005 mmol) and PIDA (644 mg, 2 mmol) under argon atmosphere. After the reaction mixture was stirred at 100^{o}C for 12-24h until the palladium black was formed, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel.

Typical procedure for synthesis of 2a:

To a 10 ml dried flask were sequentially added 1, 2-Me_2-*o*-carborane (43 mg, 0.25 mmol), AcOH (1 ml), Ac_2O (1 ml), $Pd(OAc)_2$ (1.12 mg, 0.005 mmol) and PIDA (644 mg, 2 mmol) under argon atmosphere. After the reaction mixture was stirred at 100^{o}C for 12h, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=2:1 afford **2a** (83mg, 82% yield).

The experiment procedure for synthesis **2b**-**2o** is almost the same. The detailed experiment parameters for synthesis of **2a**-**2o** are shown in Table S1.

Table S1: Detailed reaction time and eluent for separate 2a-2o

Product	Reaction time	Eluent for column chromatography
2a	12h	petroleum ether/EtOAc=2:1
2b	24h	petroleum ether/EtOAc=2:1
2c	12h	petroleum ether/EtOAc=2:1
2d	12h	petroleum ether/EtOAc=2:1
2e	12h	petroleum ether/EtOAc=2:1
2f	21h	petroleum ether/EtOAc=2:1
2g	16h	petroleum ether/EtOAc=2:1
2h	16h	petroleum ether/EtOAc=2:1
2i	12h	petroleum ether/EtOAc=2:1
2j	12h	CH_2Cl_2/EtOAc=6:1
2k	12h	CH_2Cl_2/EtOAc=6:1
2l	12h	CH_2Cl_2/EtOAc=6:1
2m	12h	CH_2Cl_2/EtOAc=6:1
2n	14h	CH_2Cl_2/EtOAc=6:1
2o	12h	CH_2Cl_2/EtOAc=6:1

General procedure for palladium catalyzed selective mono-acetoxylation of *o*-carboranes:

To a 10 mL dried flask were sequentially added *o*-carborane (0.25 mmol), DCM (1 ml), AcOH (0.05ml), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), AgOAc (0.25 mmol, 41.5mg) and PIDA (161 mg, 0.5 mmol) under argon atmosphere. After the reaction mixture was stirred at 40^{o}C for 24-36h until the palladium black was formed, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, both isomers were separated by column chromatography on 200-300 mesh silica gel.

Typical procedure for synthesis of 3aa and 3ab:

To a 10 ml dried flask were sequentially added 1, 2-Me_2-*o*-carborane (43 mg, 0.25

mmol), DCM (1 ml), AcOH (0.05 ml), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), AgOAc (0.25 mmol, 41.5mg) and PIDA (161 mg, 0.5 mmol) under argon atmosphere. After the reaction mixture was stirred at 40^{o}C for 24h, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=30:1~20:1 afford 3aa and 3ab (34mg, 83% yield), and recovered 1, 2-Me_2-*o*-carborane (13mg, 70% conversion).

The experiment procedure for synthesis **3b-3h** is almost the same.The detailed parameters for synthesis of **3b-3h** are shown in Table S2.

Table S2: Detailed reaction time and eluent for separate both isomers of 3a-3h

Product	Reaction time	Eluent for column chromatography
3a	24h	petroleum ether/EtOAc=30:1~20:1
3b	24h	petroleum ether/EtOAc=30:1~20:1
3c	24h	petroleum ether/EtOAc=50:1~40:1
3d	24h	petroleum ether/EtOAc=30:1~20:1
3e	26h	petroleum ether/EtOAc=30:1
3f	24h	petroleum ether/EtOAc=40:1
3g	24h	petroleum ether/EtOAc=40:1
3h	36h	petroleum ether/EtOAc=40:1~30:1

Experiment procedure for synthesis of 4aa:

To a 10 mL dried flask were sequentially added 9-Me-1, 2-Me_2-*o*-carborane (46.5mg, 0.25 mmol), AcOH (1ml), Ac_2O (1ml), $Pd(OAc)_2$ (1.12 mg, 0.005 mmol) and PIDA (483 mg, 1.5 mmol) under argon atmosphere. After the reaction mixture was stirred at 100^{o}C for 10h, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with CH_2Cl_2/EtOAc=20:1 as eluent, and gave 4aa (27mg, 30% yield).

Experiment procedure for synthesis of 4bb:

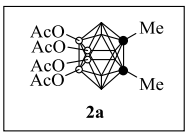
To a 10 mL dried flask were sequentially added 9, 12-Me_2-1, 2-Me_2-*o*-carborane

(50mg, 0.25 mmol), AcOH (1ml), Ac_2O (1ml), $Pd(OAc)_2$ (1.12 mg, 0.005 mmol) and PIDA (322 mg, 1.0 mmol) under argon atmosphere. After the reaction mixture was stirred at 100^oC for 10h, the reaction mixture was cooled to room temperature and extracted with ethyl acetate (60 ml). The organic phase was washed with water (3x10ml), $NaHCO_3$ (aq.) (3x10ml) and brine (3x10ml) in sequence, then dried over anhydrous Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography on 200-300 mesh silica gel with petroleum ether/EtOAc=10:1 as eluent, and gave 4bb (22mg, 28% yield).

Reference:

1. (a) Heying, T. L.; Ager, J. W.; Jr., Clark, S. L.; Alexander, R. P.; Papetti, S.; Reid, J. A.; Trotz, S. I.; *Inorg. Chem.* **1963**, *2*, 1097. (b) Paxson, T. E.; Kaloustian, M. K.; Tom, G. M.; Wiersema, R. J.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1972**, *94*, 4882. (c) Armstrong, A. F.; Valliant, J. F.; *Inorg. Chem.* **2007**, *46*, 2148.
2. Toppino, A.; Genady, A. R.; El-Zaria, M. E.; Reeve, J.; Mostofian, F.; Kent, J.; Valliant, J. F. *Inorg. Chem.* **2013**, *52*, 8743.
3. Zheng, Zhi.; Jiang, W.; Zinn, A. A.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1995**, *34*, 2095.

Spectroscopic data for products



2a

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.11 (s, 6H), 2.09(s, 6H), 2.08 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.6, 50.1, 22.3, 21.7; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 3.2 (2B, *BOAc*), -2.2 (2B, *BOAc*), -16.6 (4B), -19.2 (2B); HRMS: calculated for $C_{12}B_{10}H_{24}O_8Na$ (M^++Na) 427.2368; found: 427.2361.

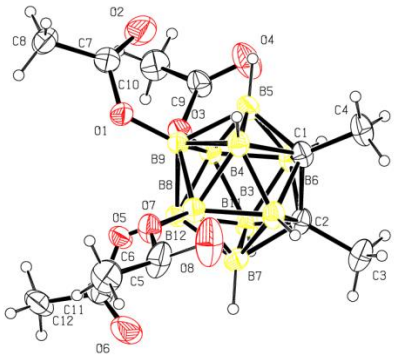
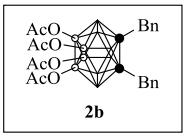
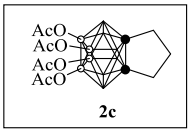


Figure S1. Molecular structure of 2a.



2b

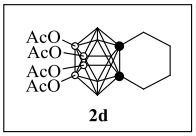
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.37-7.34 (m, 6H), 7.24-7.22 (m, 4H), 3.69 (s, 4H), 2.02 (s, 6H), 2.00 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.3, 134.3, 130.2, 128.9, 128.4, 60.2, 55.9, 39.4, 22.3, 22.2; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 3.7 (2B, *BOAc*), -1.8 (2B, *BOAc*), -17.7 (4B), -20.6 (2B); HRMS: calculated for $C_{24}B_{10}H_{32}O_8Na$ (M^++Na) 579.2977; found: 579.2986.



2c

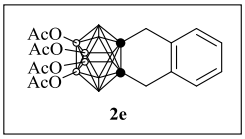
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.60-2.56 (m, 4H), 2.55-2.54 (m, 2H), 2.09 (s, 12H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.6, 61.7, 33.5, 32.9, 22.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 2.3 (2B, *BOAc*), -0.4 (2B, *BOAc*), -18.5

(4B), -20.1 (2B); HRMS: calculated for $C_{13}B_{10}H_{24}O_8Na$ (M^++Na) 439.2365; found: 439.2360.



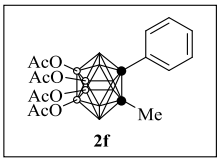
2d

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.51 (m, 4H), 2.08 (s, 12H), 1.62 (s, 4H); $^{13}C\{^1H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.0, 169.6, 49.5, 31.2, 22.3, 19.3; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 2.7 (2B, *BOAc*), -1.3 (2B, *BOAc*), -16.8 (4B), -22.8 (2B); HRMS: calculated for $C_{14}B_{10}H_{26}O_8Na$ (M^++Na) 453.2521; found: 453.2517.



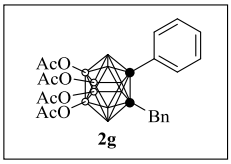
2e

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.24-7.23 (m, 2H), 7.08-7.06 (m, 2H), 3.78 (s, 4H), 2.12 (s, 6H), 2.08 (s, 6H); $^{13}C\{^1H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 169.9, 169.6, 128.8, 128.5, 127.8, 48.2, 36.1, 22.4, 22.3; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 3.2 (2B, *BOAc*), -1.7 (2B, *BOAc*), -16.9 (4B), -22.0 (2B); HRMS: calculated for $C_{18}B_{10}H_{26}O_8Na$ (M^++ Na) 501.2509; found: 501.2523.



2f

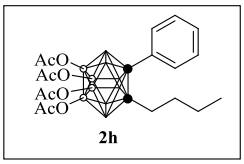
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.73-7.72 (d, *J*=6Hz, 2H), 7.47-7.45 (dd, *J*=6Hz, 1H), 7.40-7.37 (dd, *J*=6Hz, 2H), 2.12 (brs, 9H), 2.08 (s, 3H), 1.75 (s, 3H); $^{13}C\{^1H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.6, 169.5, 132.0, 131.0, 129.0, 128.6, 59.3, 53.6, 22.4, 21.4; $^{11}B\{^1H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.5 (1B, *BOAc*), 3.9 (1B, *BOAc*), -1.8 (2B, *BOAc*), -16.3 (4B), -21.0 (2B); HRMS: calculated for $C_{17}B_{10}H_{26}O_8Na$ (M^++ Na) 489.2505; found: 489.2523.



2g

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.81-7.80 (d, *J*=6Hz, 2H), 7.54-7.52 (dd, *J*=6Hz, 1H), 7.47-7.45 (dd, *J*=6Hz, 2H), 7.25-7.21 (m, 3H), 6.85-6.83 (m, 2H), 3.12 (s, 2H),

2.09 (s, 6H), 2.08 (s, 3H), 2.07 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.4, 134.6, 132.5, 131.3, 129.9, 129.2, 128.6, 128.2, 60.8, 58.3, 39.1, 22.4, 22.3; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.5 (2B, *BOAc*), -1.59 (2B, *BOAc*), -17.2 (4B), -22.2 (2B); HRMS: calculated for $C_{23}B_{10}H_{30}O_8Na$ (M^++Na) 565.2844; found: 565.2830.



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.71-7.70 (d, *J*=6Hz, 2H), 7.48-7.45 (dd, *J*=6Hz, 1H), 7.40-7.37 (dd, *J*=6Hz, 2H), 2.12 (s, 6H), 2.11 (s, 3H), 2.09 (s, 3H), 1.81-1.78 (m, 2H), 1.42-1.37 (m, 2H), 1.12-1.05 (m, 2H), 0.73-0.71 (t, *J*=6Hz, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.2, 169.6, 169.5, 132.1, 131.0, 129.0, 128.6, 60.7, 58.6, 32.9, 31.8, 22.4, 22.0, 13.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.3 (2B, *BOAc*), -1.8 (2B, *BOAc*), -17.4 (4B), -21.8 (2B); HRMS: calculated for $C_{20}B_{10}H_{32}O_8Na$ (M^++Na) 531.2994; found: 531.2986.

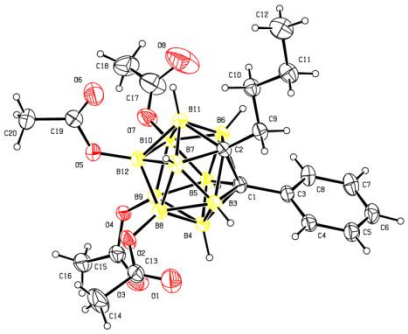
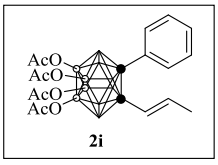
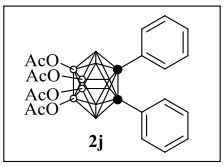


Figure S2. Molecular structure of 2h.



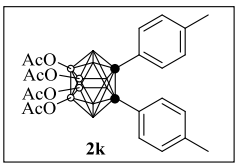
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.66-7.65 (d, *J*=6Hz, 2H), 7.44-7.42 (dd, *J*=6Hz, 1H), 7.35-7.33 (dd, *J*=6Hz, 2H), 6.18-6.12 (m, 1H), 5.27-5.24 (m, 1H), 2.13 (s, 6H), 2.12 (s, 3H), 2.09 (s, 3H), 1.53-1.52 (d, *J*=6Hz, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 169.6, 169.5, 139.6, 131.9, 130.8, 128.7, 128.6, 120.8, 59.8, 57.7, 22.4,

22.3, 17.8; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.5 (2B, *BOAc*), -1.9 (2B, *BOAc*), -16.6 (4B), -21.5 (2B); HRMS: calculated for $C_{19}B_{10}H_{28}O_8Na$ (M^++Na) 515.2667; found: 515.2680.



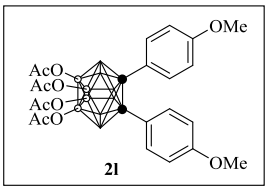
2j

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.51-7.49 (m, 4H), 7.25-7.22 (m, 2H), 7.14-7.12 (m, 4H), 2.16 (s, 6H), 2.12 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.2, 169.5, 131.5, 130.6, 128.4, 128.3, 61.9, 22.5, 22.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.2 (2B, *BOAc*), -1.9 (2B, *BOAc*), -15.9 (4B), -21.9 (2B); HRMS: calculated for $C_{22}B_{10}H_{28}O_8Na$ (M^++Na) 551.2672; found: 551.2680.



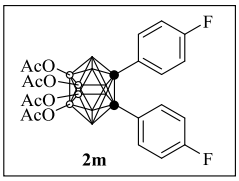
2k

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.39-7.38 (d, *J*=6Hz, 4H), 6.94-6.93 (d, *J*=6Hz, 4H), 2.22 (s, 6H), 2.16 (6H), 2.12 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.3, 169.5, 140.9, 131.4, 129.1, 125.6, 62.3, 22.5, 22.4, 20.9; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.1 (2B, *BOAc*), -2.0 (2B, *BOAc*), -15.9 (4B), -21.9 (2B); HRMS: calculated for $C_{24}B_{10}H_{32}O_8Na$ (M^++Na) 579.2969; found: 579.2993.



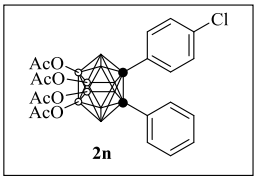
2l

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.42-7.41 (d, *J*=6Hz, 4H), 6.63-6.62 (d, *J*=6Hz, 4H), 3.71 (s, 6H), 2.15 (s, 6H), 2.12 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): 170.3, 169.6, 161.1, 133.1, 120.5, 113.6, 62.9, 60.2, 55.3, 22.5, 22.4; $\delta^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.9 (2B, *BOAc*), -2.2 (2B, *BOAc*), -15.9 (4B), -21.8 (2B); HRMS: calculated for $C_{24}B_{10}H_{32}O_{10}Na$ (M^++Na) 611.2872; found: 611.2891.



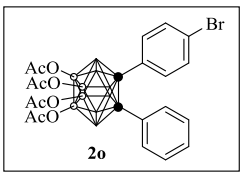
2m

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.52-7.50 (m, 4H), 6.87-6.84 (m, 4H), 2.17 (s, 6H), 2.13 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.2, 169.5, 164.7, 163.1, 133.7, 133.6, 124.3, 115.8, 115.7, 61.2, 22.5, 22.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.4 (2B, *BOAc*), -1.9 (2B, *BOAc*), -15.8 (4B), -21.9 (2B); HRMS: calculated for $C_{22}B_{10}H_{27}O_8F_2$ (M^++H) 565.2656; found: 565.2672.



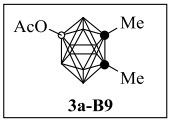
2n

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.52-7.51 (d, *J*=6Hz, 2H), 7.44-7.43 (d, *J*=6Hz, 2H), 7.28-7.26 (dd, *J*=6Hz, 1H), 7.19-7.16 (dd, *J*=6Hz, 2H), 7.12-7.11 (d, *J*=6Hz, 2H), 2.17 (s, 6H), 2.13 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.2, 169.5, 137.2, 132.7, 131.5, 130.9, 128.7, 128.6, 128.1, 127.1, 61.9, 60.9, 22.5, 22.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.3 (2B, *BOAc*), -1.8 (2B, *BOAc*), -15.9 (4B), -21.9 (2B); HRMS: calculated for $C_{22}B_{10}H_{28}O_8Cl$ (M^++H) 563.2458; found: 563.2470.



2o

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.52-7.51 (d, *J*=6Hz, 2H), 7.37-7.36 (d, *J*=6Hz, 2H), 7.29-7.27 (dd, *J*=6Hz, 3H), 7.19-7.17 (dd, *J*=6Hz, 2H), 2.17 (s, 6H), 2.13 (s, 6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.2, 169.5, 132.9, 131.7, 131.5, 130.9, 128.6, 128.1, 127.6, 125.6, 61.9, 60.9, 22.5, 22.4; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.4 (2B, *BOAc*), -1.8 (2B, *BOAc*), -15.9 (4B), -22.0 (2B); HRMS: calculated for $C_{22}B_{10}H_{27}O_8BrNa$ (M^++Na) 629.1755; found: 629.1785.



3a-B9

^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.06 (s, 3H), 2.04 (s, 3H), 2.01 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 69.0, 61.9, 23.7, 22.6, 21.2; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 9.5 (1B, *BOAc*), -5.8 (1B), -10.3 (4B), -10.9 (2B), -11.8 (2B); HRMS: calculated for $C_6B_{10}H_{19}O_2$(M^++H) 231.23827; found: 231.23854.

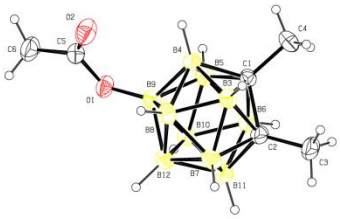
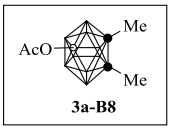


Figure S3. Molecular structure of 3aa (3a-B9).



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.08 (s, 3H), 2.04 (6H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.5, 68.5, 22.9, 22.5; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.7 (1B, *BOAc*), -5.6 (2B), -9.2 (1B), -10.4 (2B), -11.8 (2B), -13.3 (1B), -14.3 (1B); HRMS: calculated for $C_6B_{10}H_{19}O_2$(M^++H) 231.23827; found: 231.23827.

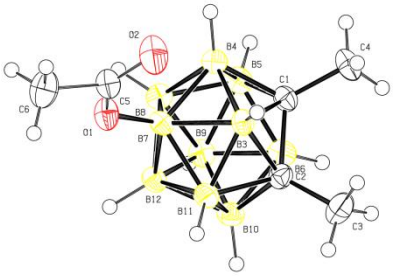
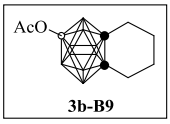
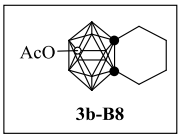


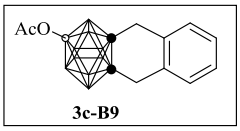
Figure S4. Molecular structure of 3ab (3a-B8).



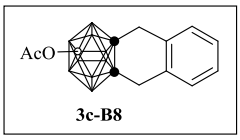
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.44-2.43 (m, 4H), 2.09 (s, 3H), 1.59-1.58 (m, 4H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 68.6, 61.6, 32.9, 30.9, 22.6, 19.9, 19.3; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 8.8 (1B, *BOAc*), -6.3 (1B), -9.9 (2B), -10.5 (2B), -11.9 (2B), -13.7 (2B); HRMS: calculated for $C_8B_{10}H_{21}O_2$ (M^++H) 257.25392; found: 257.25394.



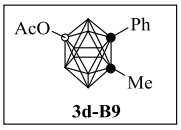
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 2.44-2.43 (m, 4H), 2.09 (s, 3H), 1.59-1.58 (m, 4H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.5, 68.2, 32.6, 22.6, 19.6; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.8 (1B, *BOAc*), -6.2 (2B), -10.6 (2B), -12.0 (4B), -17.7 (1B); HRMS: calculated for $C_8B_{10}H_{21}O_2$ (M^++H) 257.25392; found: 257.25385.



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.25-7.23 (m, 2H), 7.07 (brs, 2H), 3.77 (s, 2H), 3.72 (s, 2H), 2.04 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 129.2, 128.8, 128.7, 127.7, 127.6, 66.8, 60.3, 37.8, 35.7, 22.6; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 9.3 (1B, *BOAc*), -5.7 (2B), -10.3 (4B), -11.9 (2B), -12.6 (1B); HRMS: calculated for $C_{12}B_{10}H_{21}O_2$ (M^++H) 305.25392; found: 305.25391.

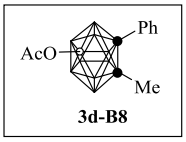


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.24-7.22 (m, 2H), 7.06-7.05 (m, 2H), 3.72 (s, 2H), 3.71 (s, 2H), 2.07 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.4, 128.8, 128.7, 127.6, 66.4, 37.3, 22.5; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.3 (1B, *BOAc*), -5.7 (2B), -10.6 (2B), -11.9 (2B), -12.7 (2B), -16.8 (1B); HRMS: calculated for $C_{12}B_{10}H_{21}O_2$ (M^++H) 305.25392; found: 305.25400.

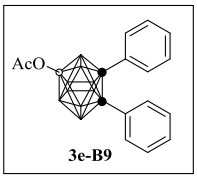


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.64-7.63 (d, *J*= 6Hz, 2H), 7.47-7.46 (dd, *J*=6Hz, 1H), 7.40-7.38 (dd, *J*=6Hz, 2H), 2.02 (s, 3H), 1.72 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.0, 131.0, 130.7, 130.6, 128.9, 77.9, 65.7, 22.6, 20.9; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 10.1 (1B, *BOAc*), -3.9 (2B), -10.1 (3B), -10.8 (2B), -11.8 (1B), -12.3 (1B); HRMS: calculated for $C_{11}B_{10}H_{21}O_2$ (M^++H) 293.25392; found:

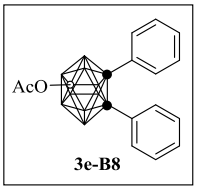
293.25378.



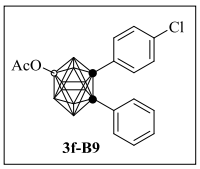
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.69-7.68 (d, *J*= 6Hz, 2H), 7.47-7.45 (dd, *J*= 6Hz, 1H), 7.40-7.38 (dd, *J*= 6Hz, 2H), 2.12 (s, 3H), 1.70 (s, 3H); ^{13}C{^{1}H} NMR (150MHz, $CDCl_3$, *ppm*): δ 170.6, 131.4, 130.7, 130.3, 128.9, 72.3, 22.9, 22.6; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.0 (1B, *BOAc*), -3.7 (1B), -4.9 (1B), -10.2 (2B), -11.5 (3B), -13.2 (1B), -16.2 (1B); HRMS: calculated for $C_{11}B_{10}H_{21}O_2$ (M^++H) 293.25392; found: 293.25397.



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.45-7.40 (m, 4H), 7.25-7.21 (2H), 7.15-7.12 (m, 4H), 2.06 (s, 3H); ^{13}C{^{1}H} NMR (150MHz, $CDCl_3$, *ppm*): δ 170.0, 130.9, 130.5, 130.3, 130.2, 129.1, 128.3, 128.2, 80.9, 73.7, 22.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 11.8 (1B, *BOAc*), -3.3 (2B), -9.9 (2B), -11.0 (3B), -13.4 (2B); HRMS: calculated for $C_{16}B_{10}H_{23}O_2$ (M^++H) 355.26957; found: 355.26974.

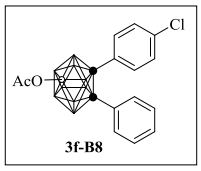


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.46-7.45 (m, 4H), 7.24-7.22 (m, 2H), 7.15-7.12 (m, 4H), 2.16 (s, 3H); ^{13}C{^{1}H} NMR (150MHz, $CDCl_3$, *ppm*): δ 170.6, 130.9, 130.3, 130.1, 128.3, 80.3, 22.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.9 (1B, *BOAc*), -2.9 (2B), -9.9 (2B), -11.2 (2B), -12.3 (1B), -13.5 (1B), -17.3 (1B); HRMS: calculated for $C_{16}B_{10}H_{23}O_2$ (M^++H) 355.26957; found: 355.26956.

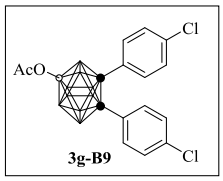


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.45-7.44 (d, *J*=6Hz, 2H), 7.34-7.33 (d, *J*=6Hz,

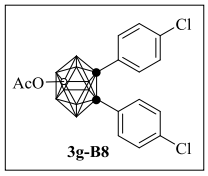
2H), 7.29-7.27 (dd, *J*=6Hz, 1H), 7.18-7.16 (dd, *J*=6Hz, 2H), 7.12-7.11 (d, *J*=6Hz, 2H), 2.06 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.0, 136.8, 131.8, 130.9, 130.6, 128.9, 128.8, 128.6, 128.5, 79.8, 73.8, 22.6; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 11.9 (1B, *BOAc*), -3.1 (2B), -10.0 (2B), -10.9 (3B), -13.6 (2B); HRMS: calculated for $C_{16}B_{10}H_{22}O_2Cl$ (M^++H) 389.23060; found: 389.23102.



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.46-7.45 (d, *J*=6Hz, 2H), 7.39-7.38 (d, *J*=6Hz, 2H), 7.28-7.26 (dd, *J*=6Hz, 1H), 7.18-7.16 (dd, *J*=6Hz, 2H), 7.12-7.11 (d, *J*=6Hz, 2H), 2.16 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.6, 136.8, 132.1, 131.8, 130.9, 130.6, 129.9, 128.6, 128.5, 80.4, 79.2, 22.7; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 5.0 (1B, *BOAc*), -2.8 (2B), -9.9 (2B), -11.1 (3B), -13.4 (1B), -17.3 (1B); HRMS: calculated for $C_{16}B_{10}H_{22}O_2Cl$ (M^++H) 389.23060; found: 389.23077.

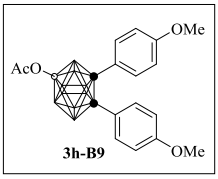


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.38-7.37 (d, *J*=6Hz, 2H), 7.34-7.33 (d, *J*=6Hz, 2H), 7.16-7.15 (d, *J*=6Hz, 4H), 2.06 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.0, 137.1, 132.2, 132.1, 131.8, 128.8, 128.7, 127.6, 79.9, 72.8, 22.6; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 12.0 (1B, *BOAc*), -3.1 (2B), -9.9 (2B), -10.8 (3B), -13.6 (2B); HRMS: calculated for $C_{16}B_{10}H_{21}Cl_2O_2$ (M^++H) 423.19163; found: 423.19153.

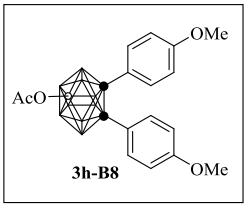


^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.40-7.38 (d, *J*=12Hz, 4H), 7.16-7.14 (d, *J*=12Hz, 4H), 2.15 (s, 3H); $^{13}C\{^{1}H\}$ NMR (150MHz, $CDCl_3$, *ppm*): δ 170.6, 137.1, 132.1, 128.8, 128.5, 79.3, 22.7; $^{11}B\{^{1}H\}$ NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.7 (1B, *BOAc*),

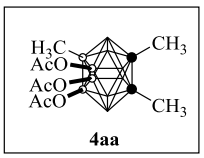
-3.1 (2B), -10.3 (2B), -11.6 (2B), -12.8 (1B), -13.7 (1B), -17.8 (1B); HRMS: calculated for $C_{16}B_{10}H_{21}Cl_2O_2$ (M^++H) 423.19163; found: 423.19186.



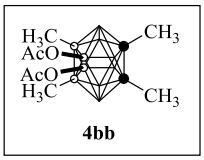
^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.37-7.35 (d, *J*=12Hz, 2H), 7.33-7.31 (d, *J*=12Hz, 2H), 6.65-6.63 (d, *J*=12Hz, 2H), 6.64-6.62 (d, *J*=12Hz, 2H), 3.72 (s, 6H), 2.05 (s, 3H); ^{13}C{^{1}H} NMR (150MHz, $CDCl_3$, *ppm*): δ 170.1, 160.9, 160.8, 132.5, 132.1, 122.8, 121.4, 113.6, 113.5, 81.8, 74.4, 55.3, 22.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 11.6 (1B, *BOAc*), -3.7 (2B), -9.9 (2B), -11.4 (3B), -13.4 (2B); HRMS: calculated for $C_{18}B_{10}H_{27}O_4$ (M^++H) 415.29070; found: 415.29068.



^{1}H NMR (600MHz, $CDCl_3$, *ppm*): δ 7.38-7.37 (d, *J*=6Hz, 4H), 6.64-6.63 (d, *J*=6Hz, 4H), 3.72 (s, 6H), 2.15 (s, 3H); ^{13}C{^{1}H} NMR (150MHz, $CDCl_3$, *ppm*): δ 170.8, 160.9, 132.4, 132.1, 122.6, 113.5, 81.2, 55.3, 22.7; ^{11}B{^{1}H} NMR (192 MHz, $CDCl_3$, *ppm*): δ 4.6 (1B, *BOAc*), -3.4 (2B), -10.1 (1B), -11.5 (2B), -13.9 (2B), -17.6 (2B); HRMS: calculated for $C_{18}B_{10}H_{27}O_4$ (M^++H) 415.29070; found: 415.29071.



^{1}H NMR (400MHz, $CDCl_3$, *ppm*): δ 2.07 (s, 9H), 2.05 (s, 3H), 0.17 (3H); ^{13}C{^{1}H} NMR (100MHz, $CDCl_3$, *ppm*): δ 170.2, 169.6, 53.4, 53.3, 29.7, 22.7, 22.3, 22.2; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$, *ppm*): δ 4.9 (1B, *BOAc*), 0.1 (1B, *BOAc*), -0.7 (2B, *BCH*$_3$), -15.5 (3B), -17.9 (3B); HRMS: calculated for $C_{11}B_{10}H_{25}O_6$ (M^++H) 361.26488; found: 361.26486.



4bb

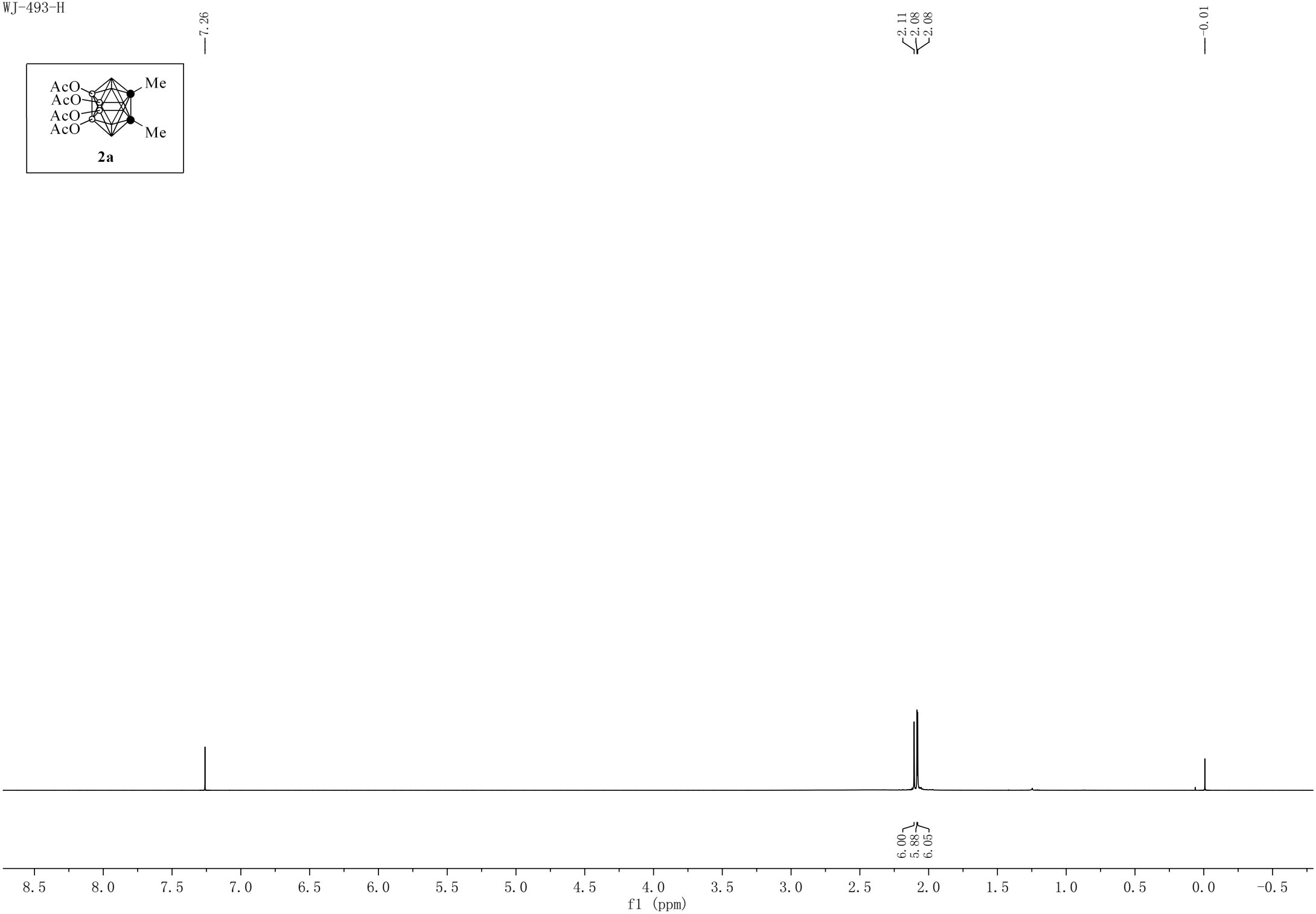
^{1}H NMR (400MHz, $CDCl_3$, *ppm*): δ 2.08 (s, 6H), 2.04 (s, 6H), 0.07 (s, 6H); $^{13}C\{^{1}H\}$ NMR (100MHz, $CDCl_3$, *ppm*): δ 170.5, 57.0, 33.2, 22.3, 22.2; $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$, *ppm*): δ 1.7 (2B, *BOAc*), 1.1 (2B, *BCH_3*), -13.4 (4B), -16.6 (2B); HRMS: calculated for $C_{10}B_{10}H_{25}O_4$ (M^++H) 317.27505; found: 317.27505.

Copies of ^{1}H NMR, $^{13}C\{^{1}H\}$ NMR , $^{11}B\{^{1}H\}$ and ^{11}B NMR

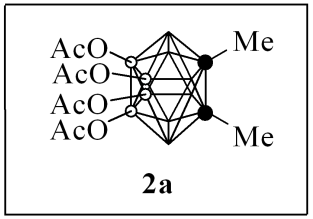
WJ-493-H

AcO
AcO
AcO
AcO
Me
Me

2a


7.26
2.11
2.08
2.08
0.01
6.00
5.88
6.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-493-C



2a

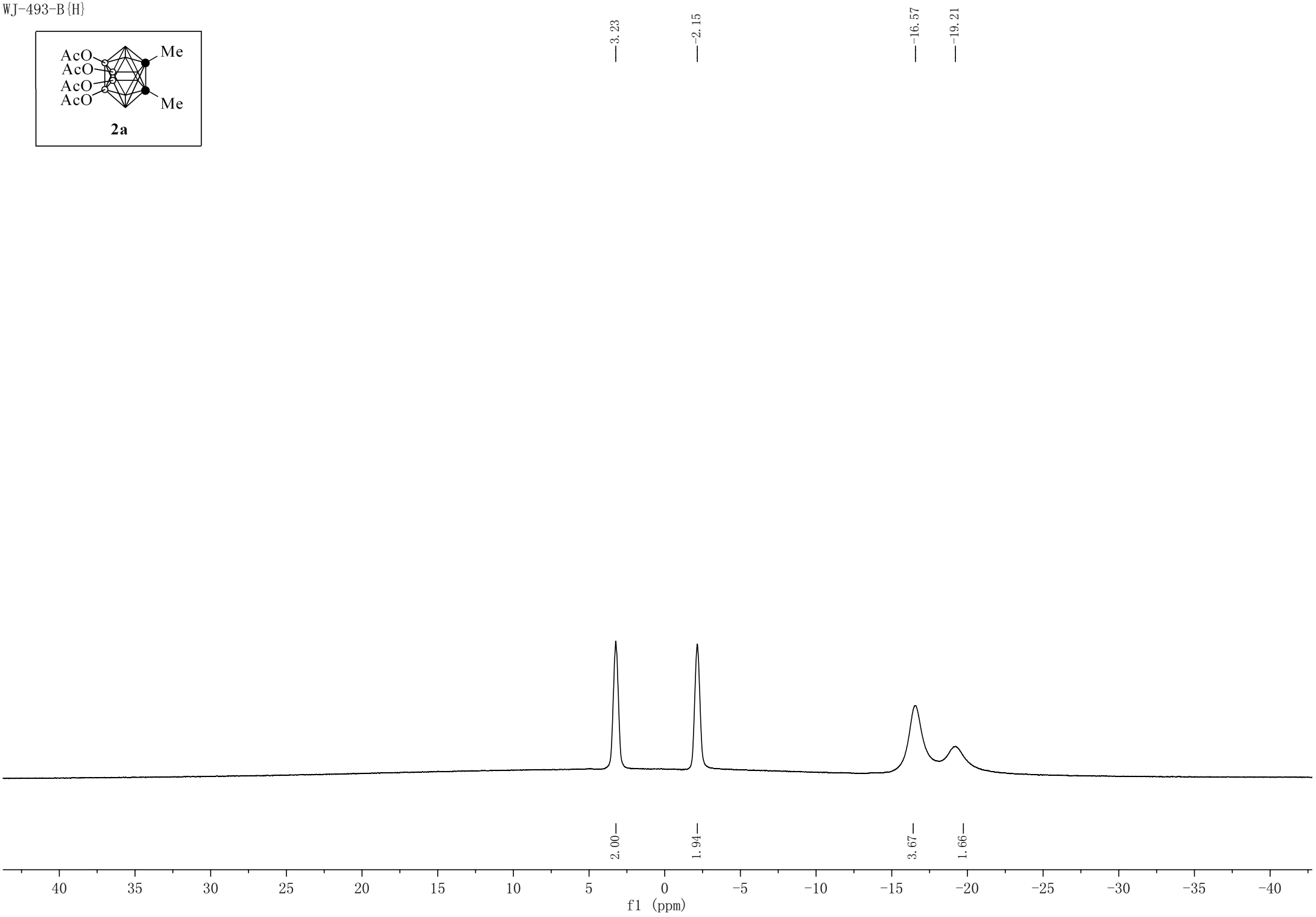
170.07
169.55
77.21
77.00
76.79
50.11
22.32
21.71
-0.03

f1 (ppm)

WJ-493-B{H}

AcO
AcO
AcO
AcO
Me
Me

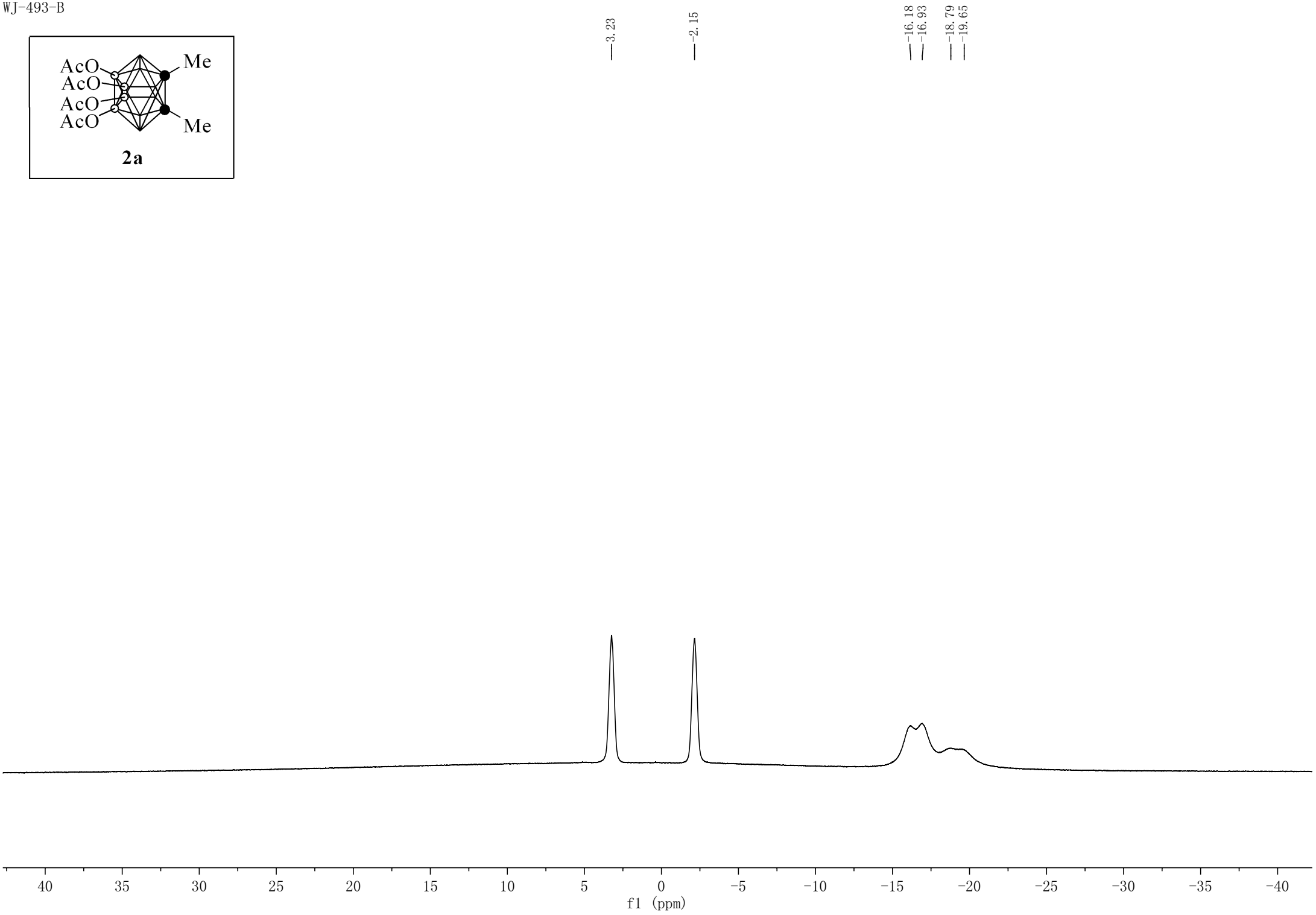
2a


3.23
2.15
16.57
19.21
2.00
1.94
3.67
1.66
40
35
30
25
20
15
10
5
0
−5
−10
−15
−20
−25
−30
−35
−40
f1 (ppm)

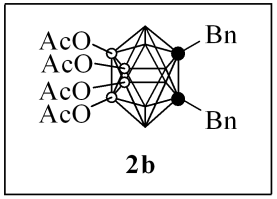
WJ-493-B

AcO
AcO
AcO
AcO
Me
Me

2a


3.23
2.15
16.18
16.93
18.79
19.65
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

WJ-599-H



2b

7.37 7.37 7.37 7.36 7.35 7.35 7.35 7.34 7.34 7.34 7.26 7.24 7.23 7.22 7.22

3.69

2.02 2.00

-0.01

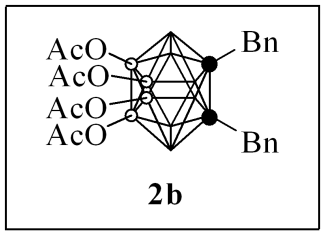
impurity in petroleum ether

6.00 3.94 3.85 5.80 6.06

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

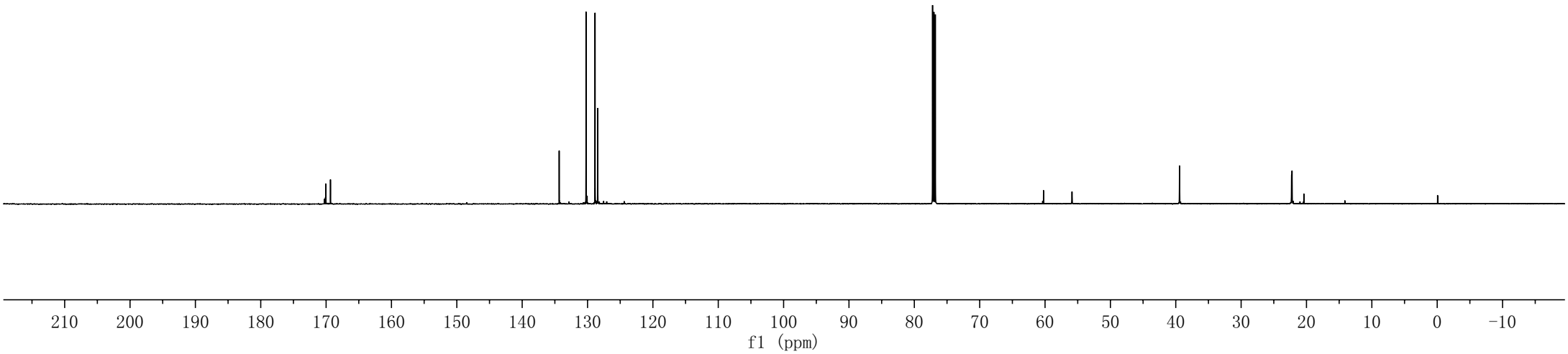
f1 (ppm)

WJ-599-C



2b

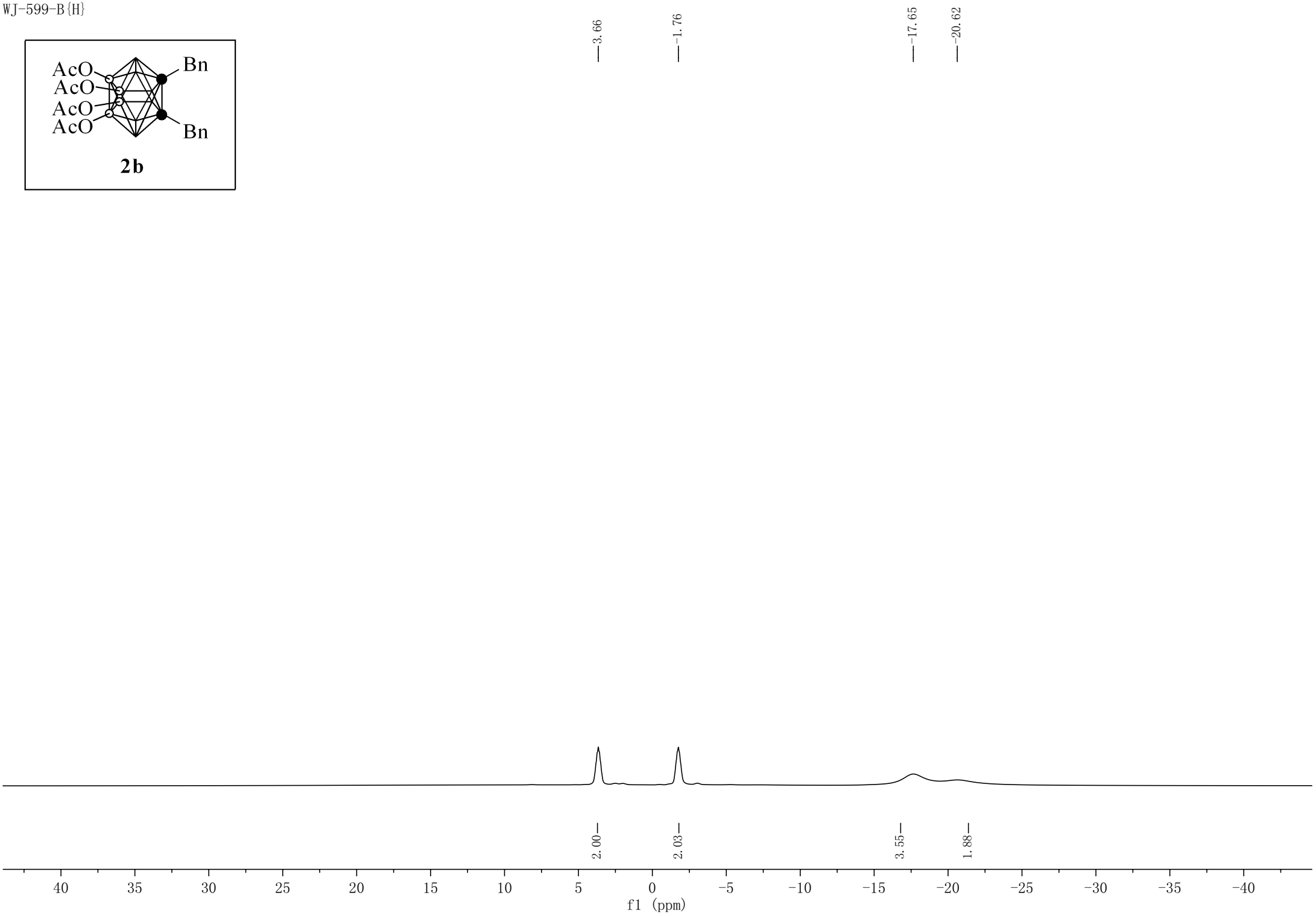
170.05
169.33
134.34
130.23
128.85
128.44
77.21
77.00
76.79
60.23
55.87
39.42
22.28
22.21



WJ-599-B{H}

AcO
AcO
AcO
AcO
Bn
Bn

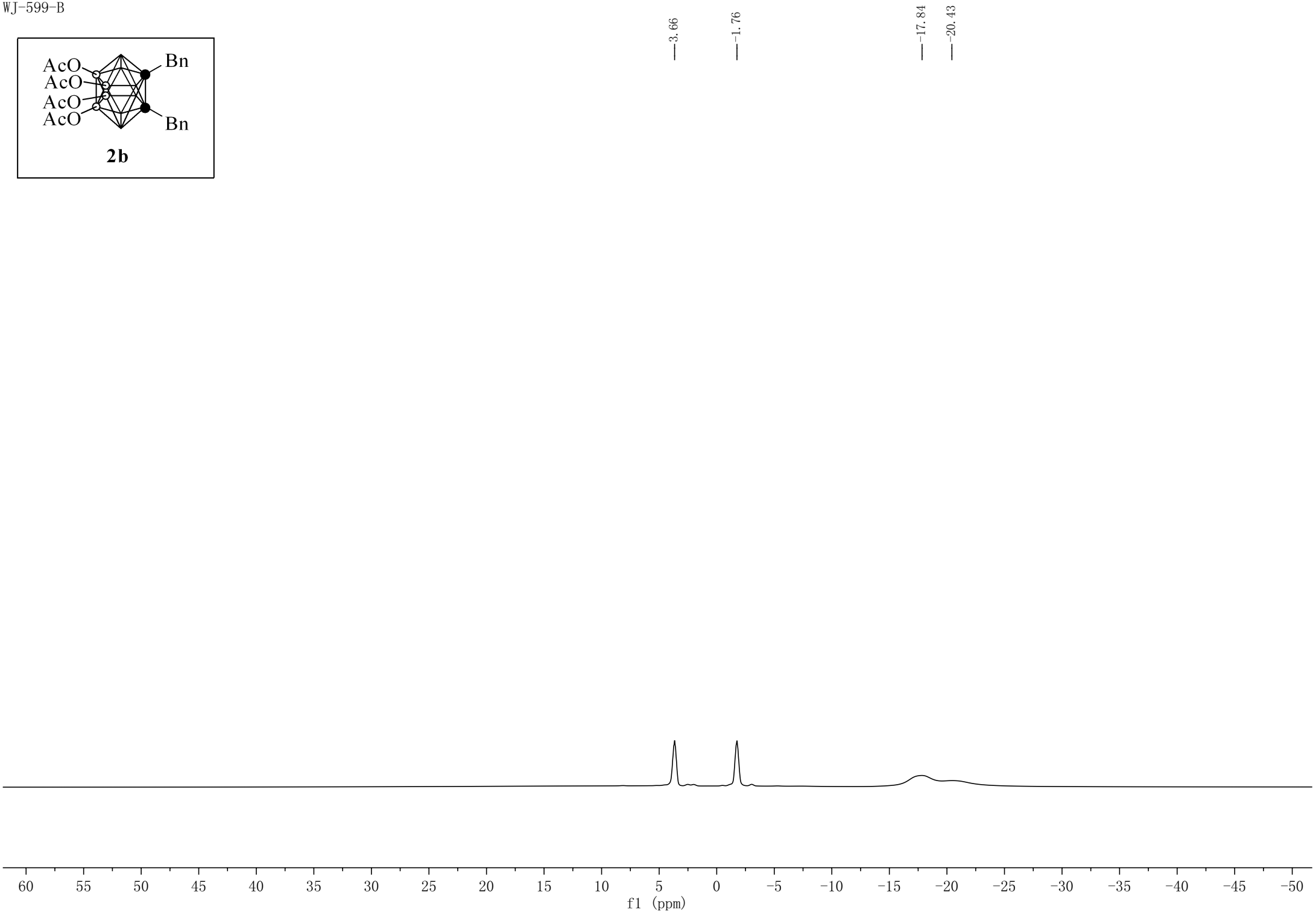
2b


3.66
1.76
17.65
20.62
2.00
2.03
3.55
1.88
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

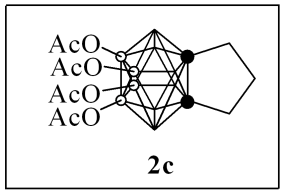
WJ-599-B

AcO
AcO
AcO
AcO
Bn
Bn

2b


3.66
1.76
17.84
20.43
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
f1 (ppm)

WJ-522-H



2c

7.26

2.60
2.59
2.58
2.56
2.55
2.55
2.54
2.54
2.09

0.01

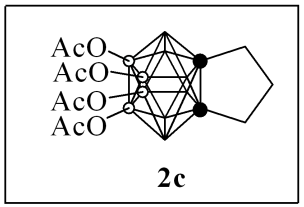
4.30
2.37

12.00

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

WJ-522-C



2c

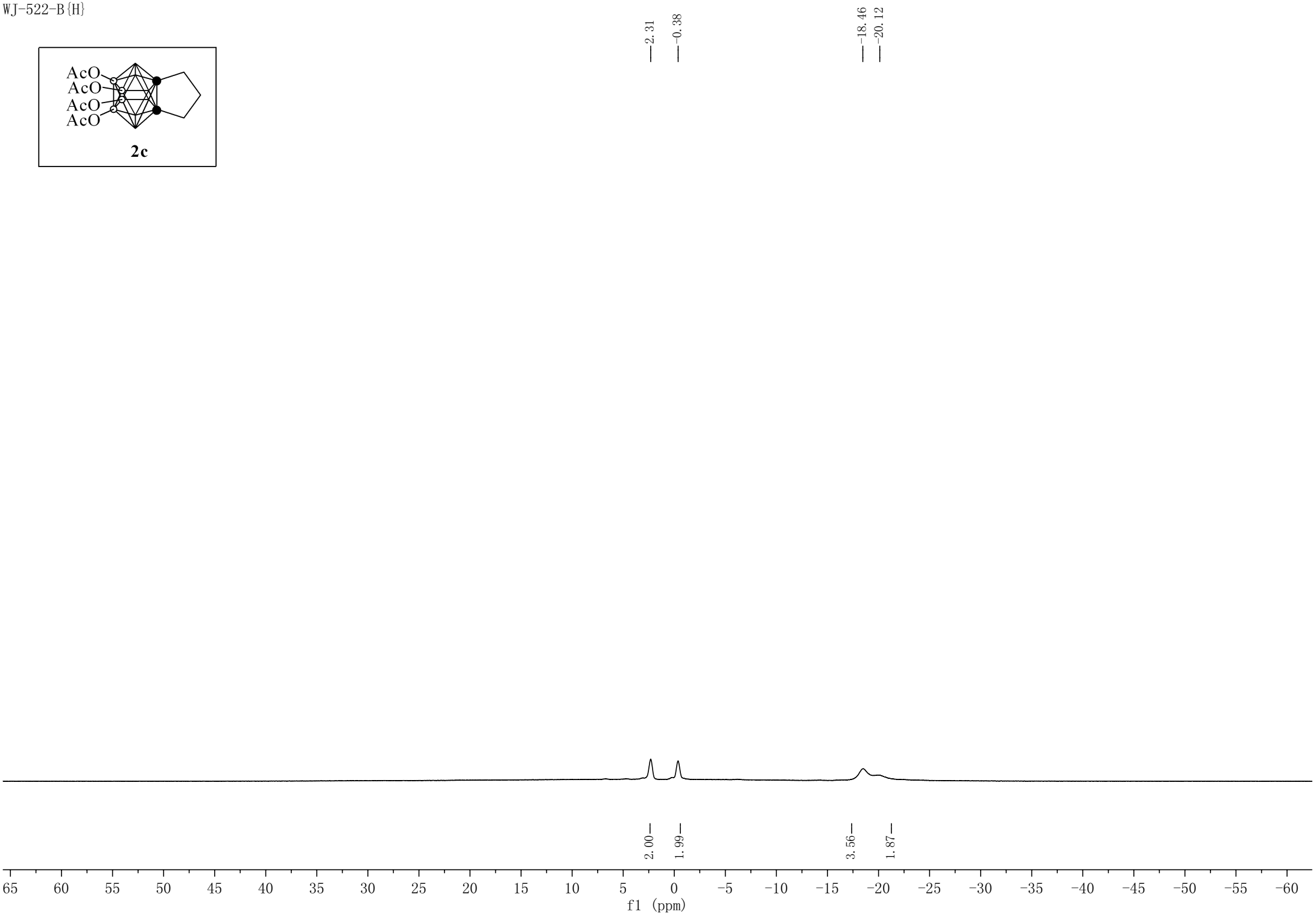
170.10
169.60
77.21
77.00
76.79
61.71
33.52
32.93
22.35
-0.03

f1 (ppm)

WJ-522-B{H}

AcO
AcO
AcO
AcO

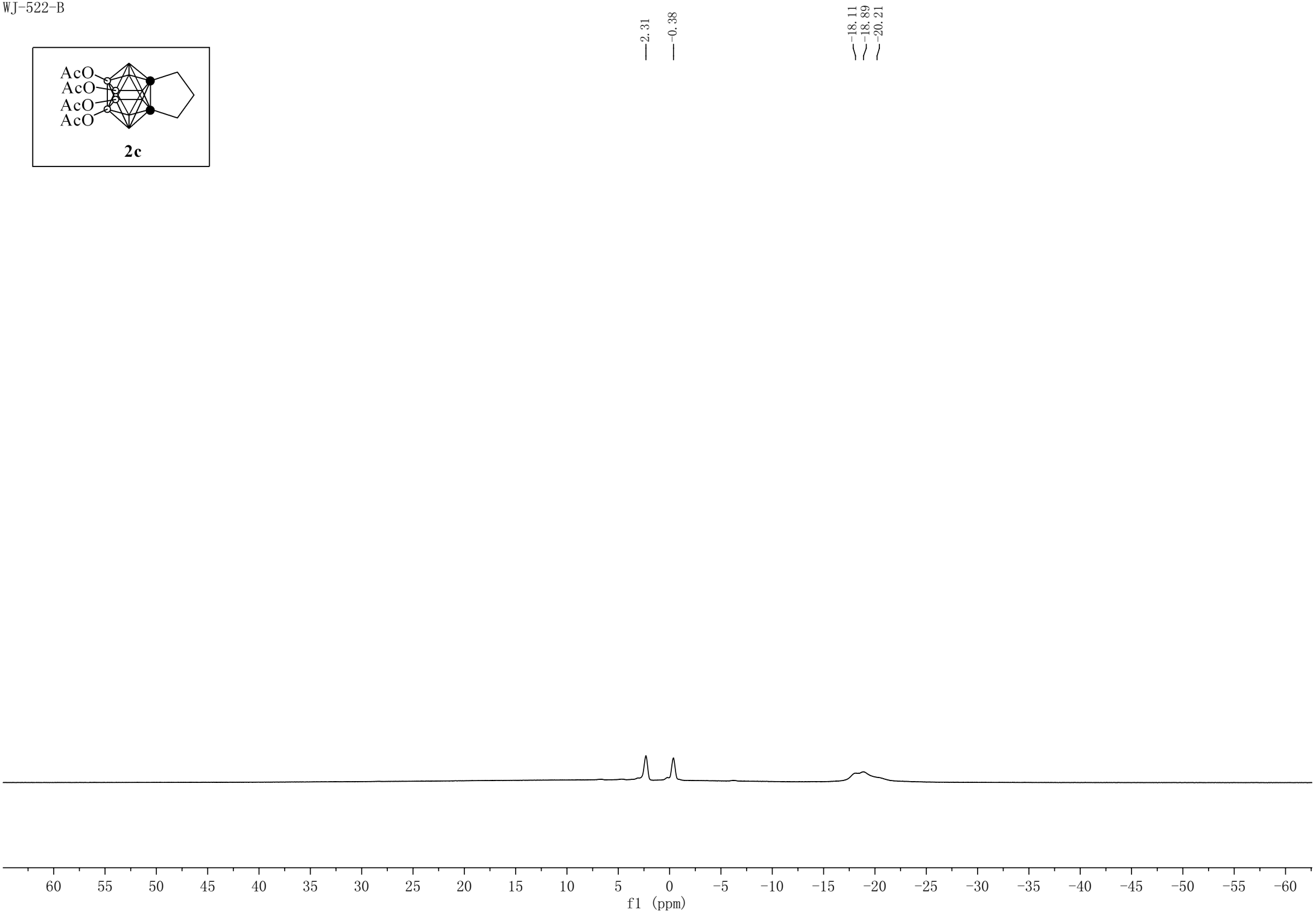
2c


2.31
-0.38
-18.46
-20.12
2.00
1.99
3.56
1.87
65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

WJ-522-B

AcO
AcO
AcO
AcO

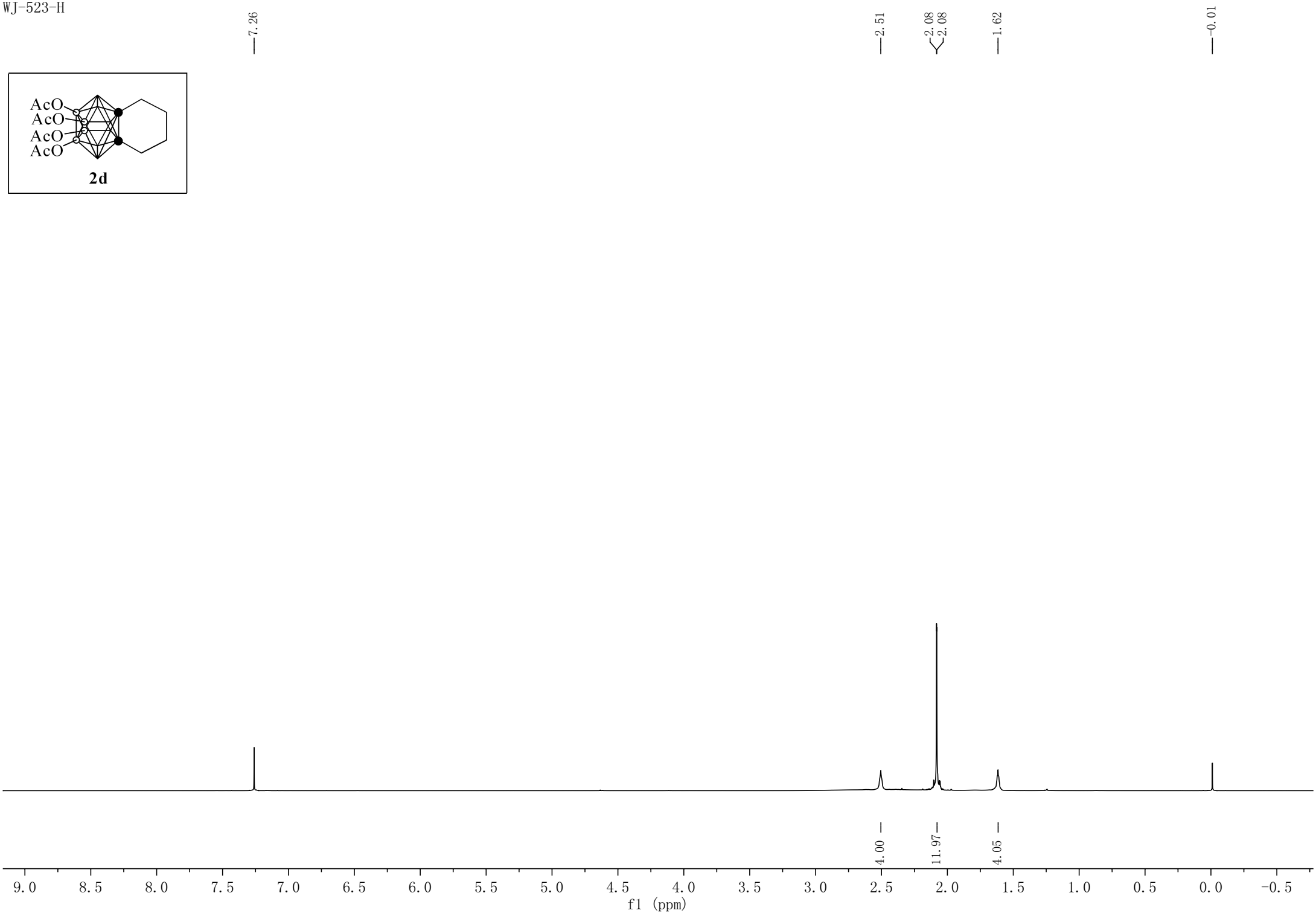
2c


2.31
0.38
18.11
18.89
20.21
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
f1 (ppm)

WJ-523-H

AcO
AcO
AcO
AcO

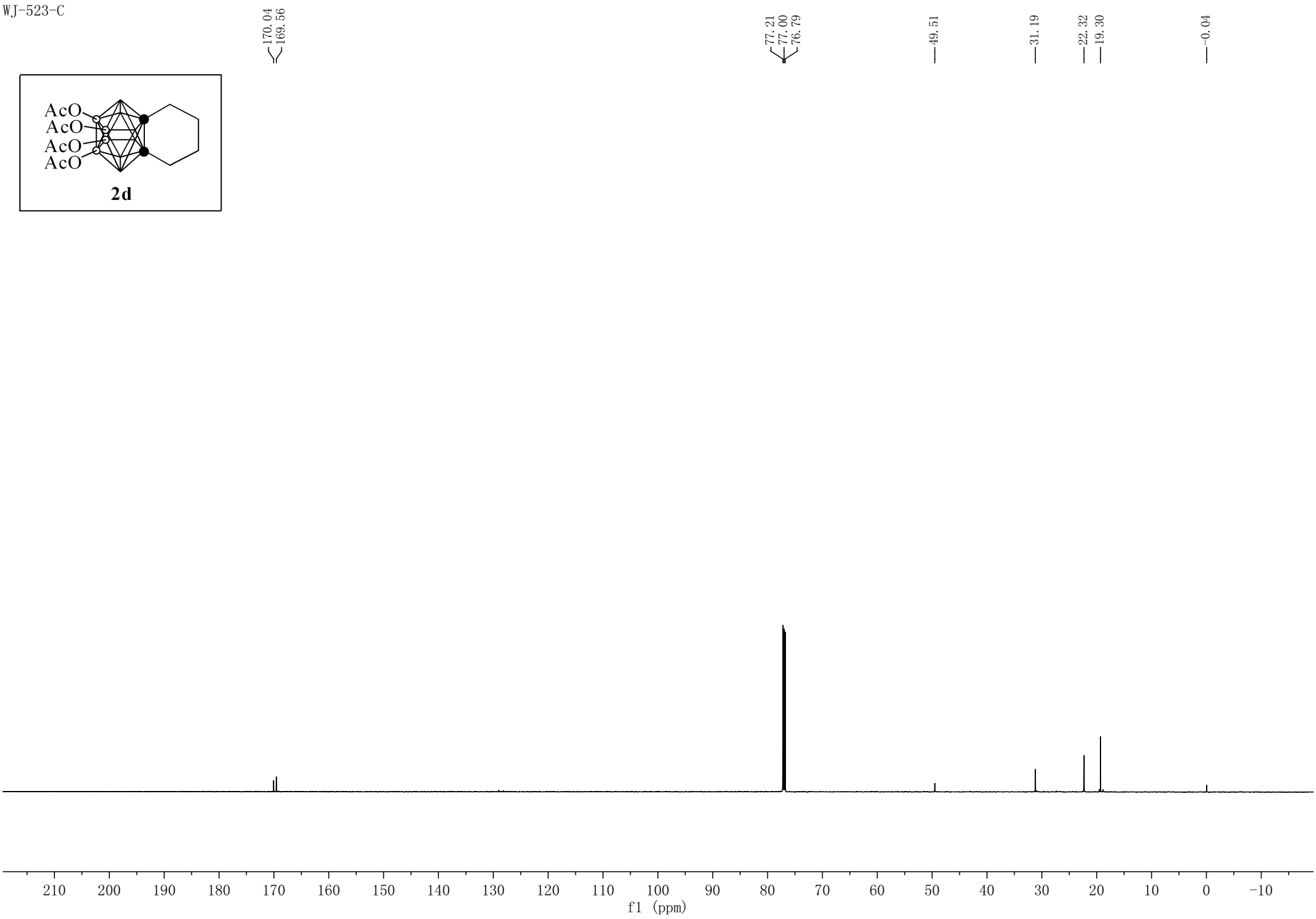
2d


7.26
2.51
2.08
2.08
1.62
0.01
4.00
11.97
4.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-523-C

AcO
AcO
AcO
AcO

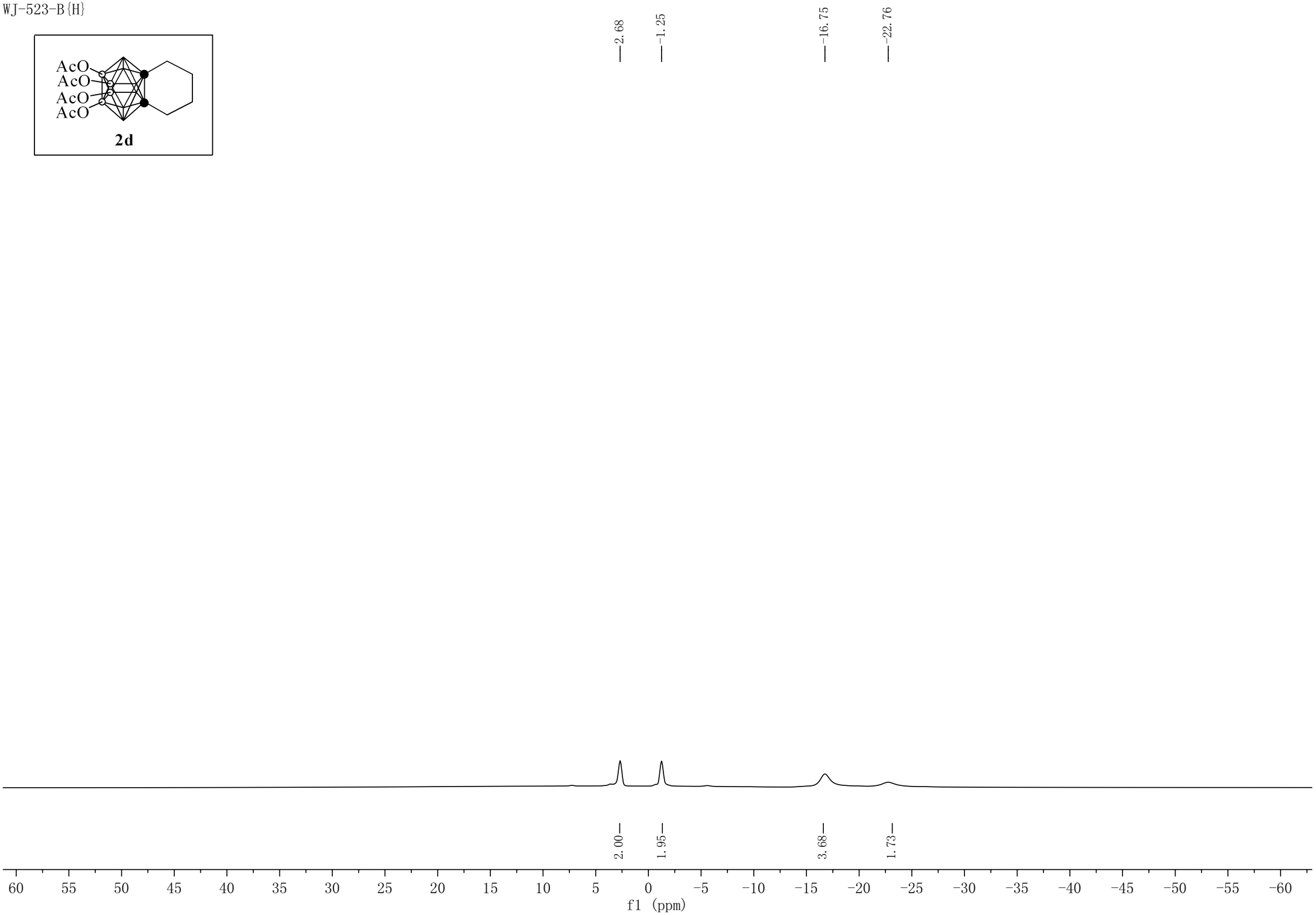
2d


170.04
169.56
77.21
77.00
76.79
49.51
31.19
22.32
19.30
-0.04
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

WJ-523-B{H}

AcO
AcO
AcO
AcO

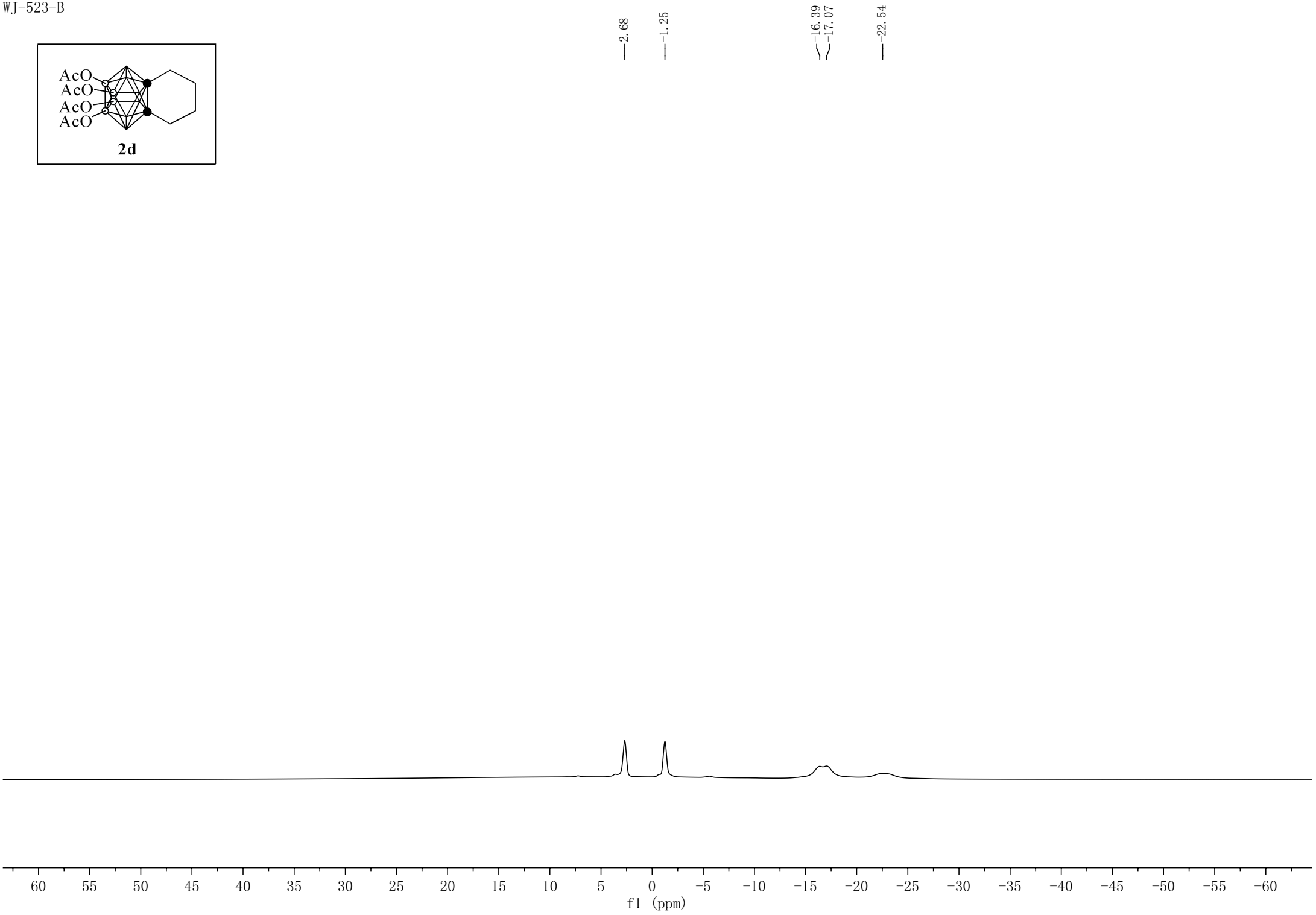
2d


2.68
1.25
16.75
22.76
2.00
1.95
3.68
1.73
60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

WJ-523-B

AcO
AcO
AcO
AcO

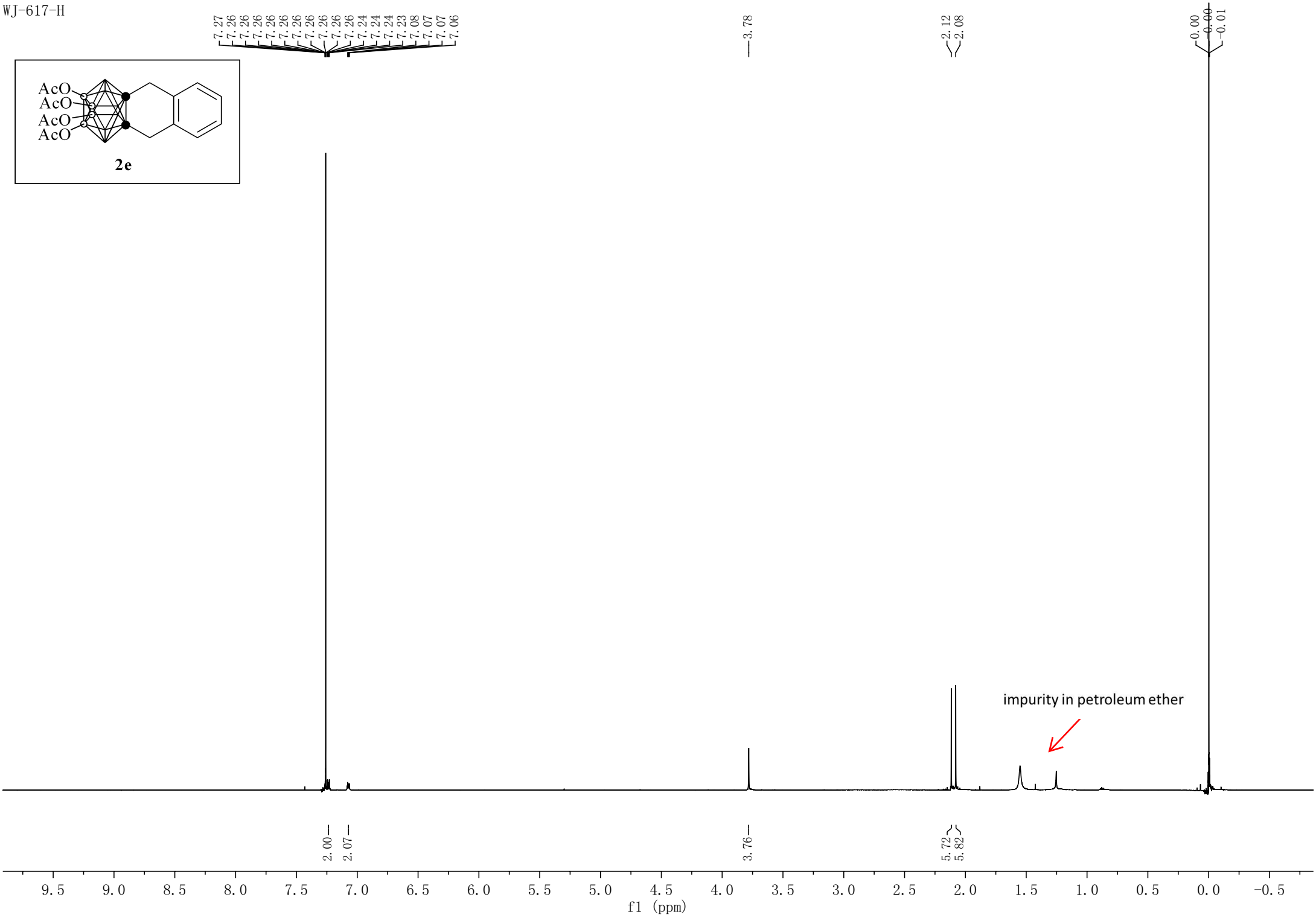
2d


2.68
1.25
16.39
17.07
22.54
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
f1 (ppm)

WJ-617-H

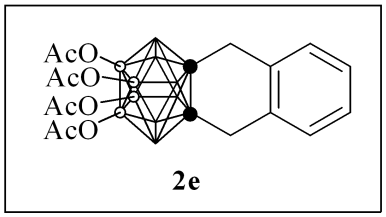
AcO
AcO
AcO
AcO

2e

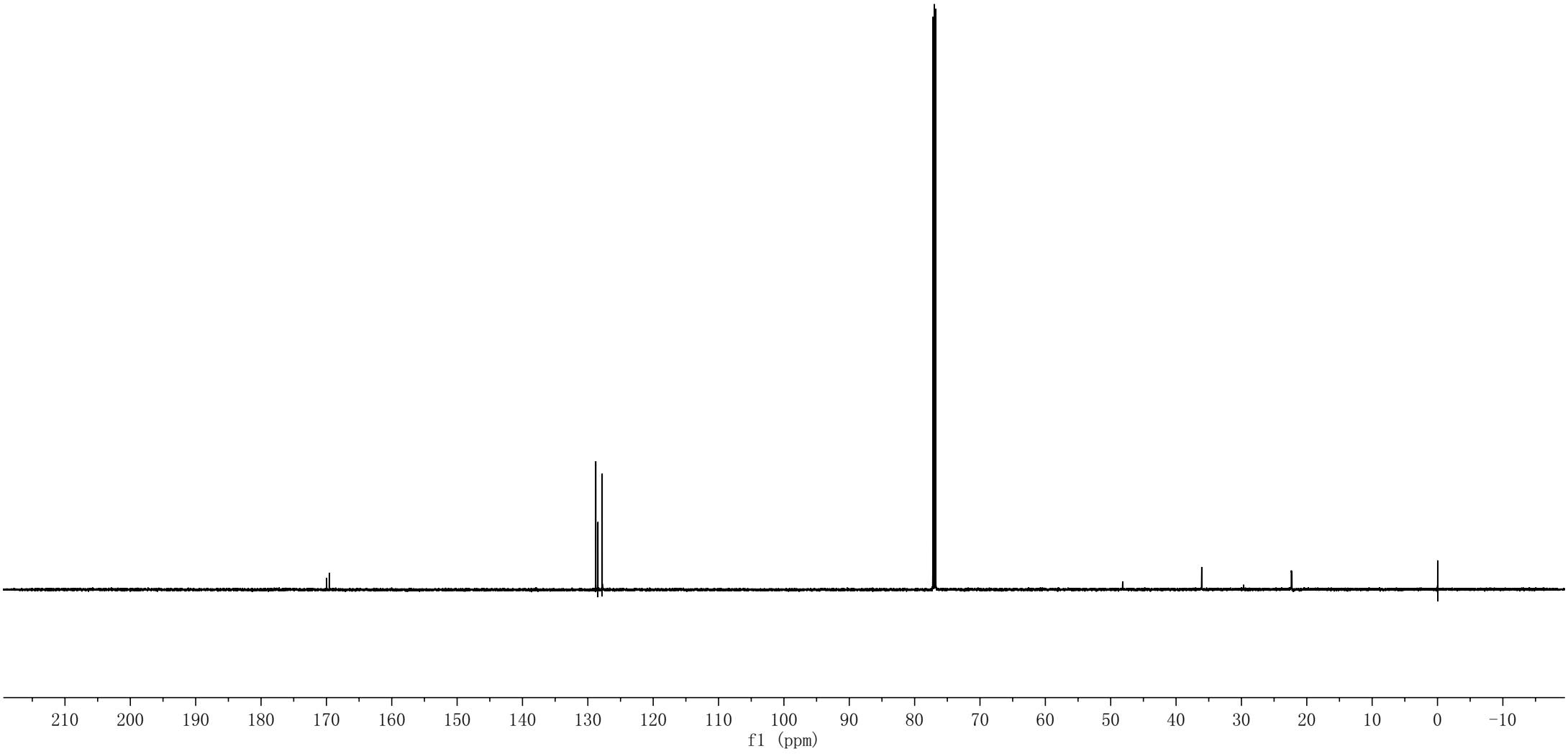

7.27
7.26
7.26
7.26
7.26
7.26
7.26
7.26
7.26
7.26
7.24
7.24
7.24
7.23
7.08
7.07
7.07
7.06
3.78
2.12
2.08
0.00
0.00
-0.01
impurity in petroleum ether
2.00
2.07
3.76
5.72
5.82
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-617-C

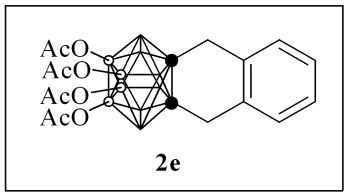
169.97
169.56
128.78
128.46
127.80
77.21
77.00
76.79
48.16
36.08
22.36
22.29
-0.02



2e



WJ-617-B{H}



2e

3.17
1.70
16.89
22.04

2.00
2.22
4.04
1.88

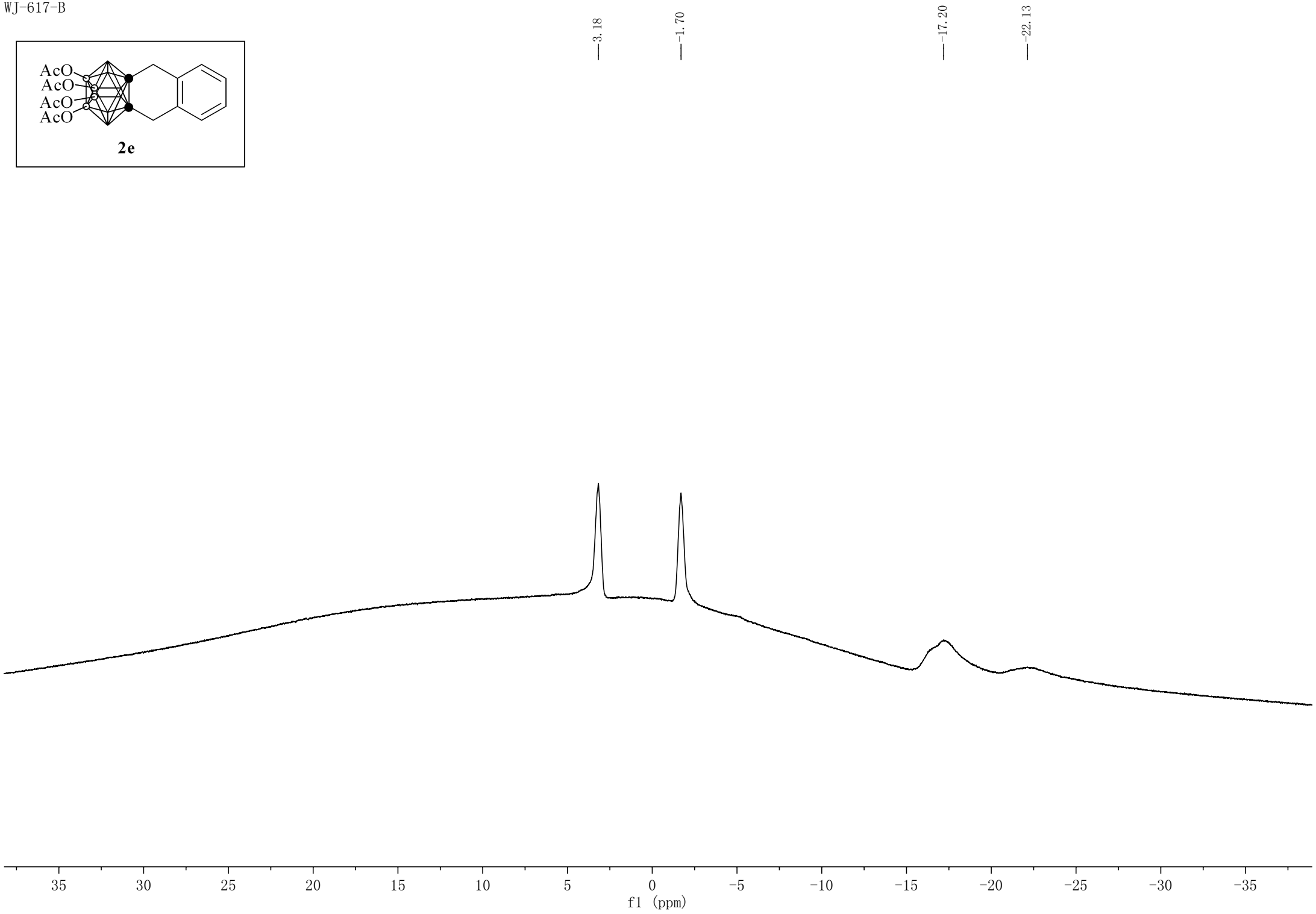
35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35

f1 (ppm)

WJ-617-B

AcO
AcO
AcO
AcO

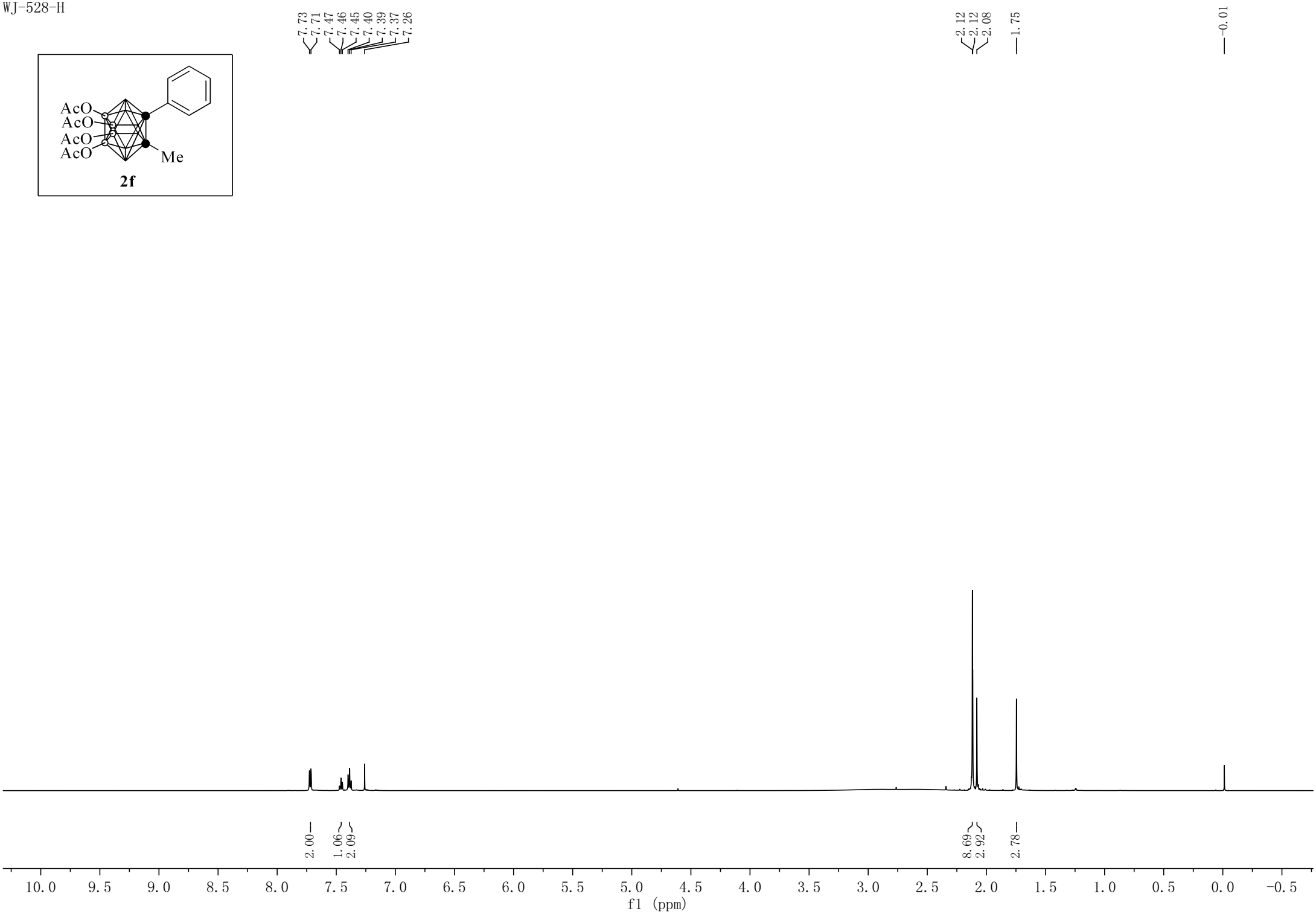
2e


3.18
-1.70
-17.20
-22.13
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

WJ-528-H

AcO
AcO
AcO
AcO
Me

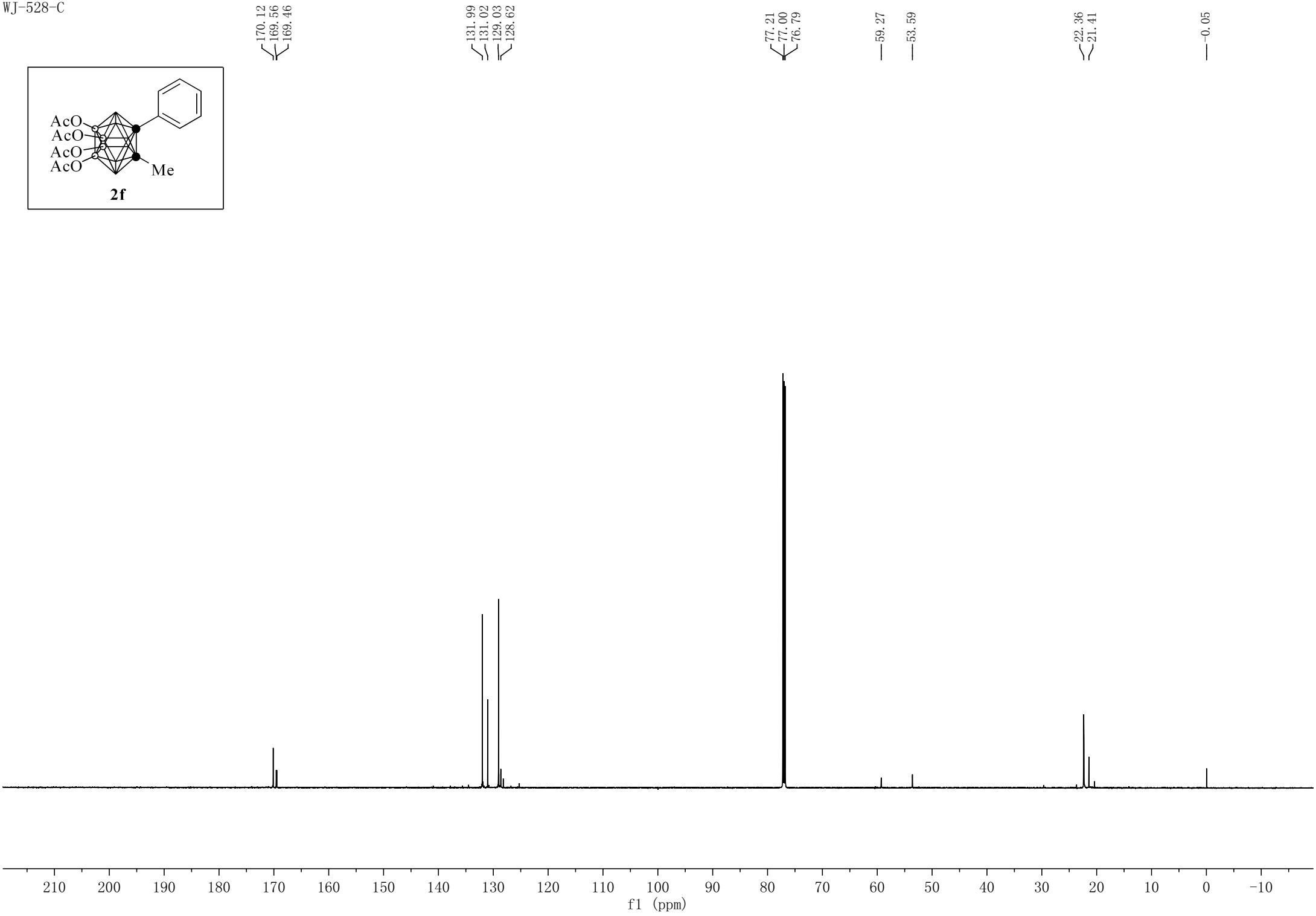
2f


7.73
7.71
7.47
7.46
7.45
7.40
7.39
7.37
7.26
2.12
2.12
2.08
1.75
0.01
2.00
1.06
2.09
8.69
2.92
2.78
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-528-C

AcO
AcO
AcO
AcO
Me

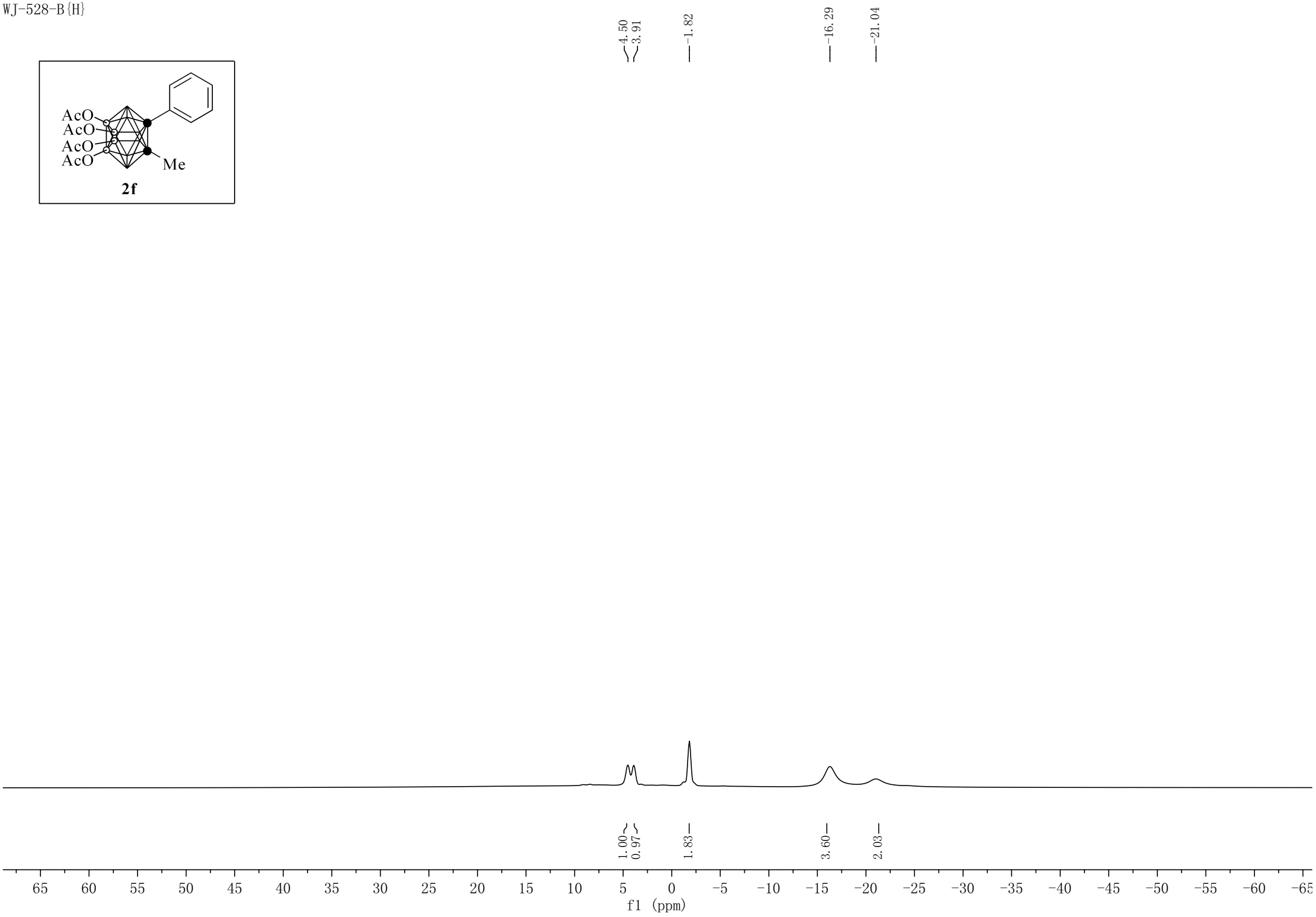
2f


170.12
169.56
169.46
131.99
131.02
129.03
128.62
77.21
77.00
76.79
59.27
53.59
22.36
21.41
-0.05
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

WJ-528-B{H}

AcO
AcO
AcO
AcO
Me

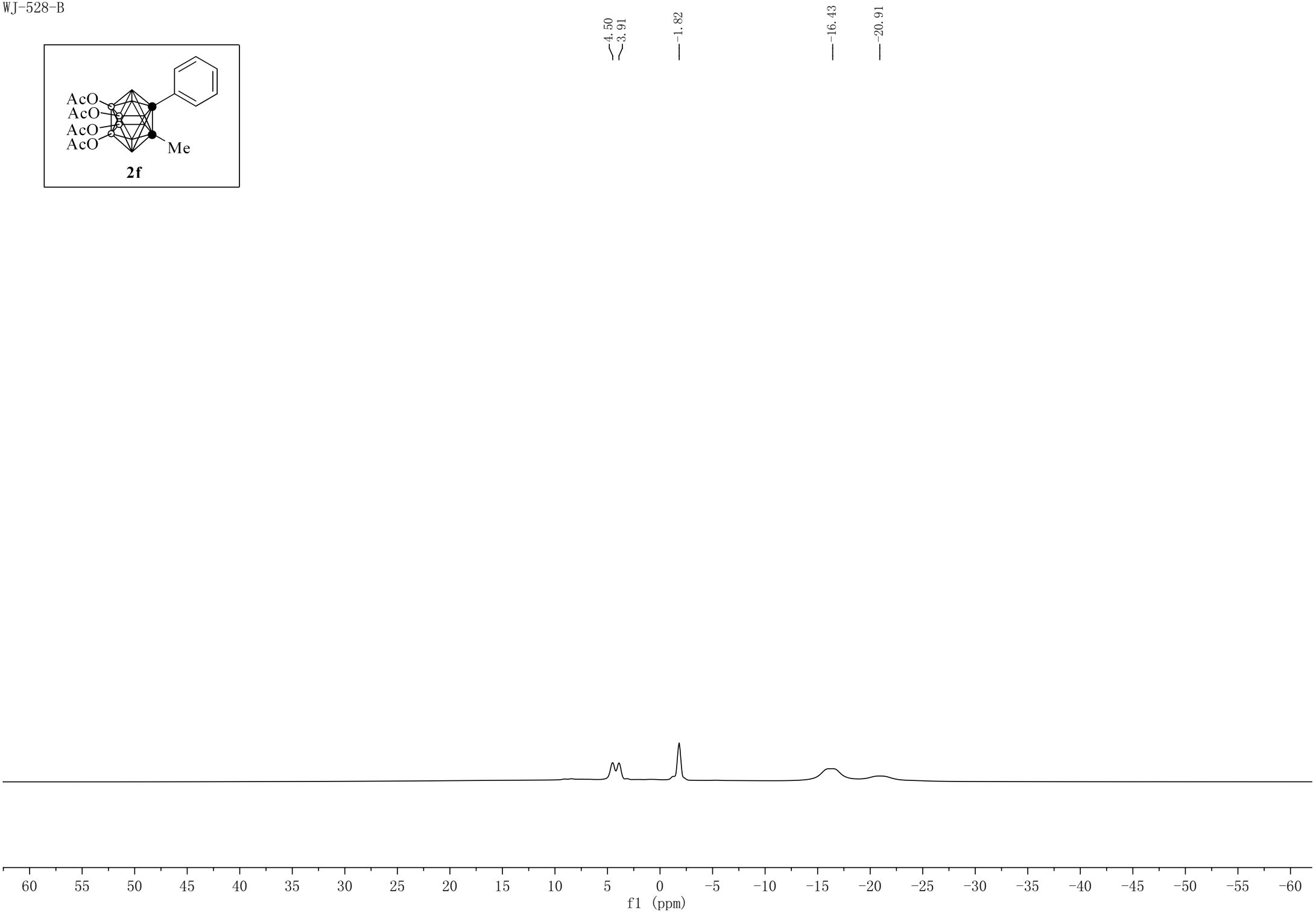
2f


4.50
3.91
1.82
16.29
21.04
1.00
0.97
1.83
3.60
2.03
65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65
f1 (ppm)

WJ-528-B

AcO
AcO
AcO
AcO
Me

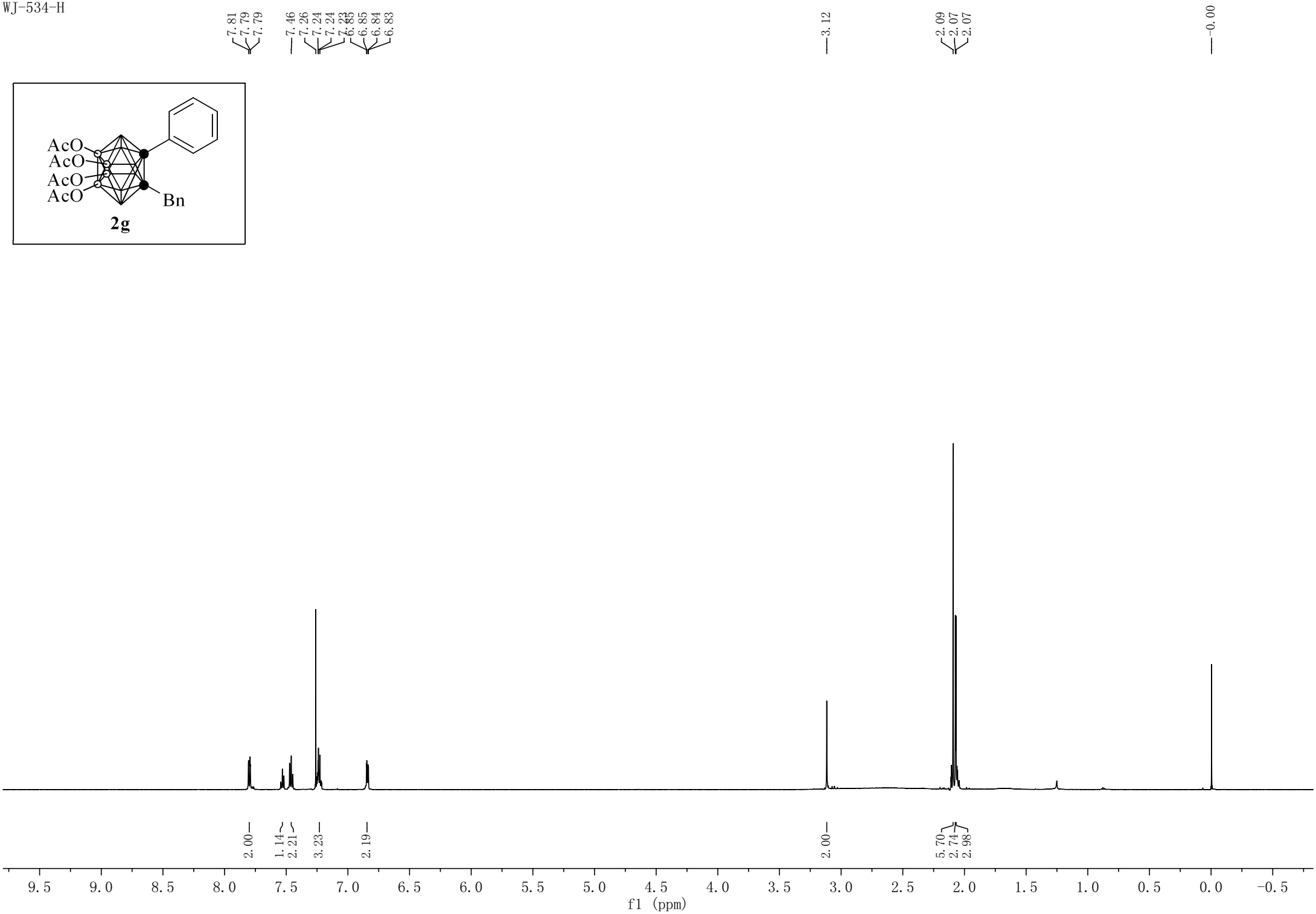
2f


4.50
3.91
-1.82
-16.43
-20.91
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
f1 (ppm)

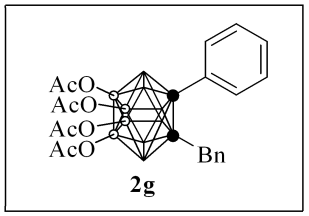
WJ-534-H

AcO
AcO
AcO
AcO
Bn

2g


7.81
7.79
7.79
7.46
7.26
7.24
7.24
7.23
6.85
6.84
6.83
3.12
2.09
2.07
2.07
0.00
2.00
1.14
2.21
3.23
2.19
2.00
5.70
2.74
2.98
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-534-C



2g

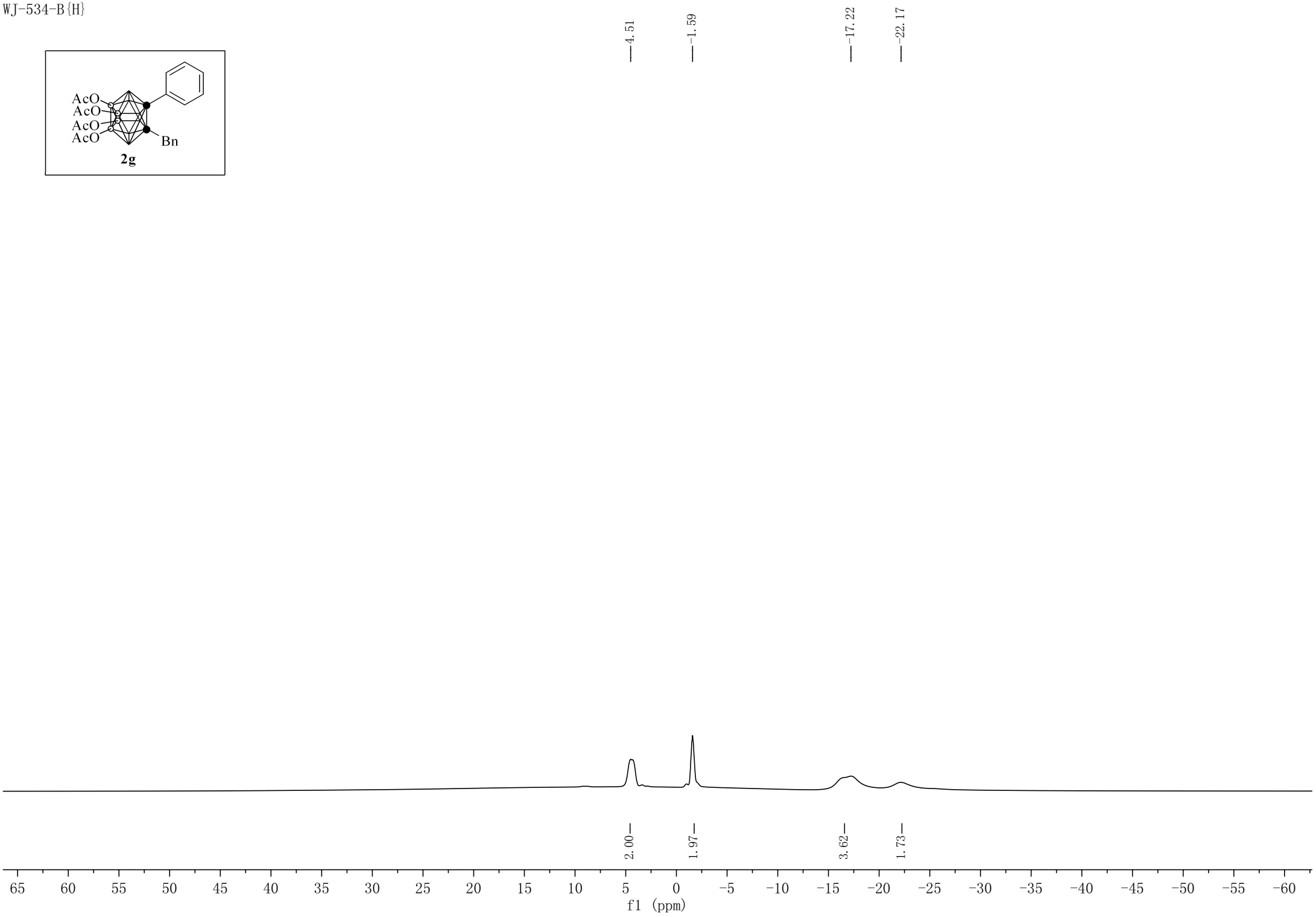
170.06
169.43
134.60
132.47
131.31
129.92
129.21
128.57
128.16
77.21
77.00
76.79
60.75
58.32
39.13
22.40
22.32
-0.03

f1 (ppm)

WJ-534-B{H}

AcO
AcO
AcO
AcO
Bn

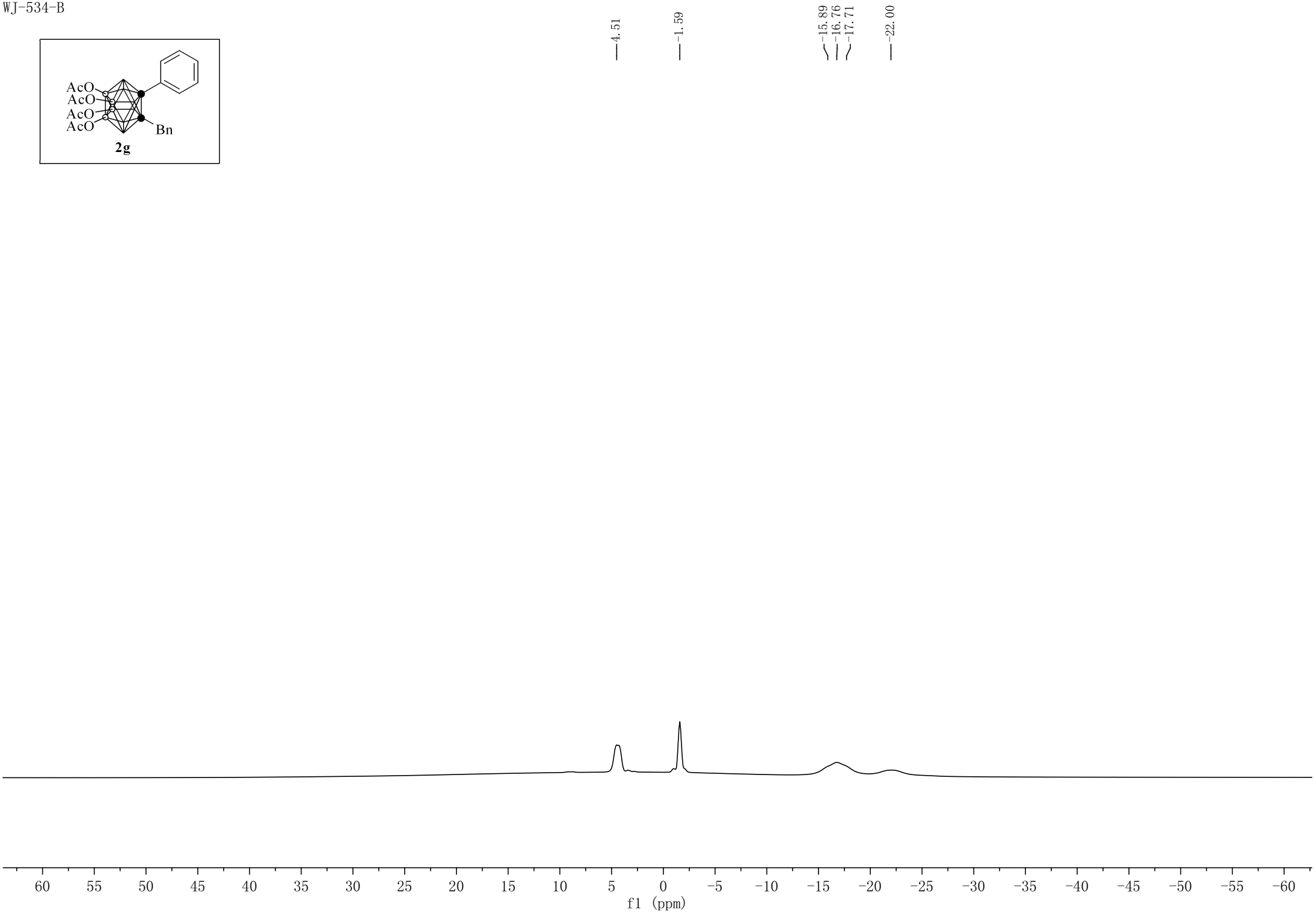
2g


4.51
1.59
17.22
22.17
2.00
1.97
3.62
1.73
65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

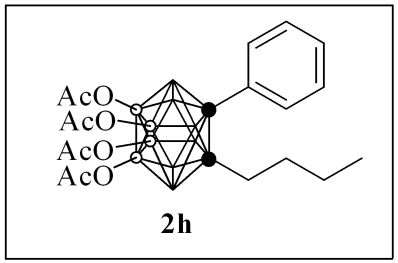
WJ-534-B

AcO
AcO
AcO
AcO
Bn

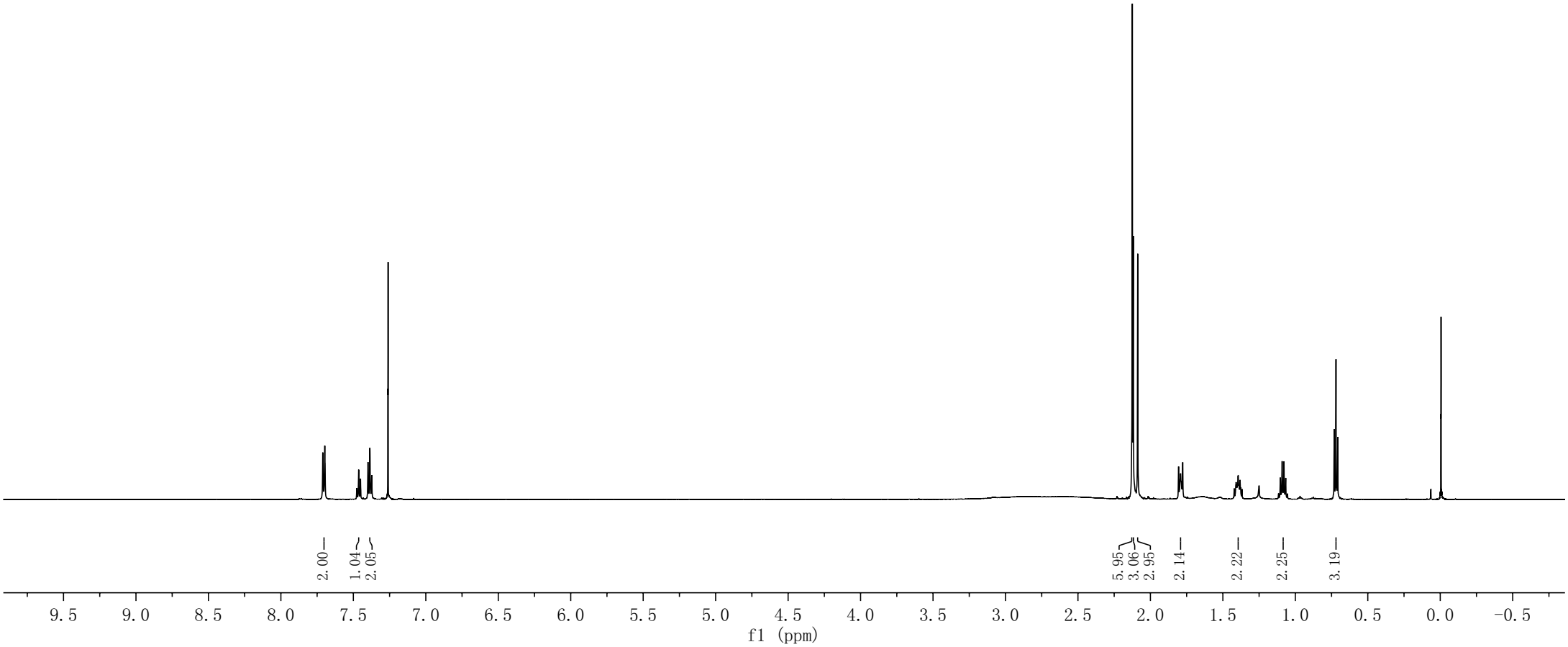
2g


4.51
1.59
15.89
16.76
17.71
22.00
60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

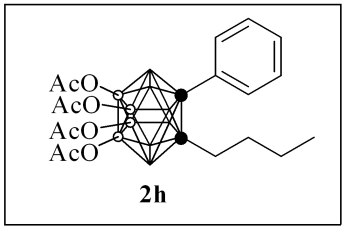
WJ-533-H


AcO
AcO
AcO
AcO

2h


7.71
7.70
7.48
7.46
7.45
7.40
7.39
7.37
7.26
7.26
2.12
2.12
2.09
1.81
1.79
1.78
1.41
1.39
1.38
1.10
1.09
0.73
0.72
0.71
-0.00
-0.01
2.00
1.04
2.05
5.95
3.06
2.95
2.14
2.22
2.25
3.19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-533-C



2h

170.15
169.59
169.48
132.08
131.03
129.01
128.55
77.21
77.00
76.79
60.69
58.58
32.88
31.77
22.42
22.00
13.41
-0.03

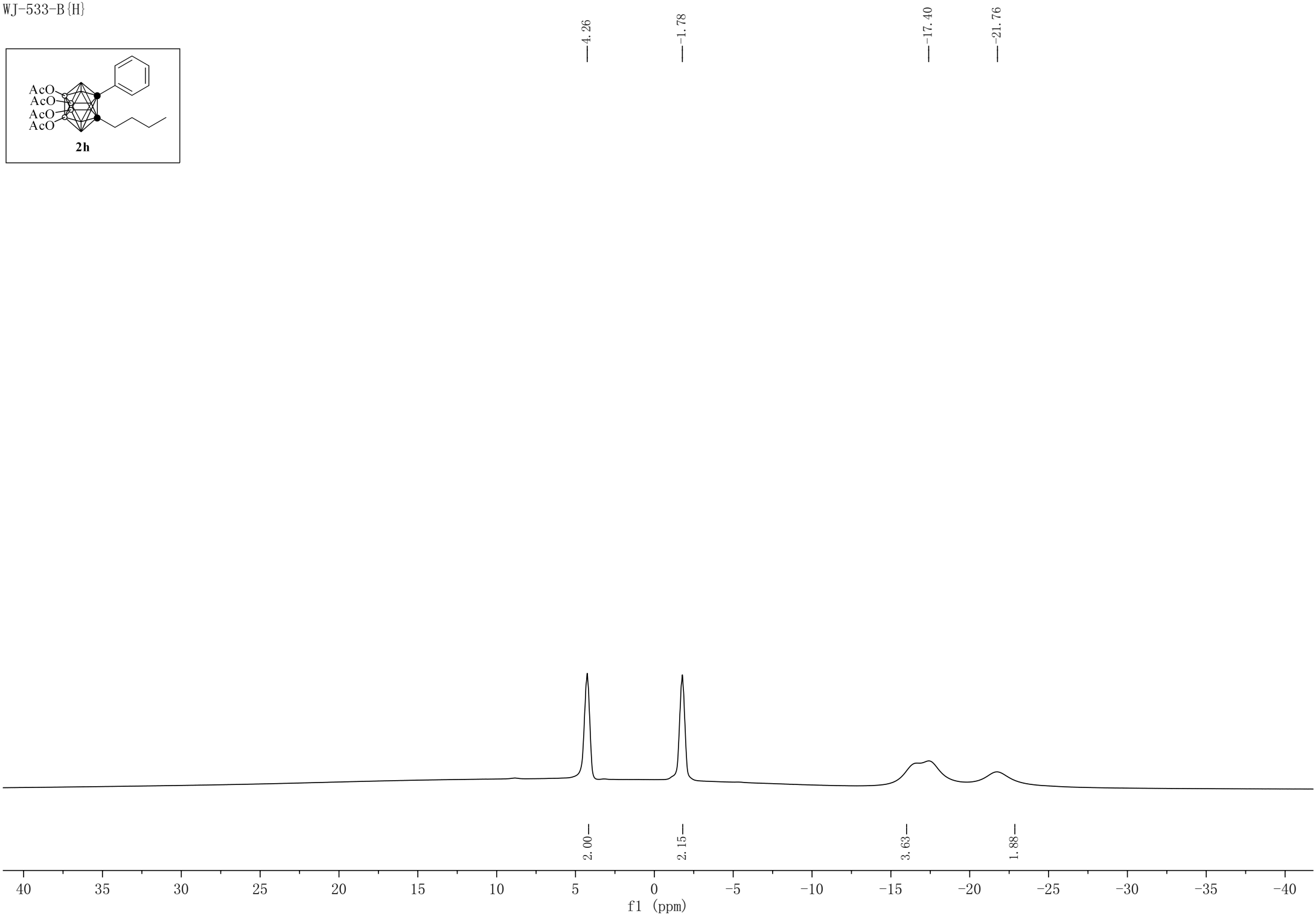
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

WJ-533-B{H}

AcO
AcO
AcO
AcO

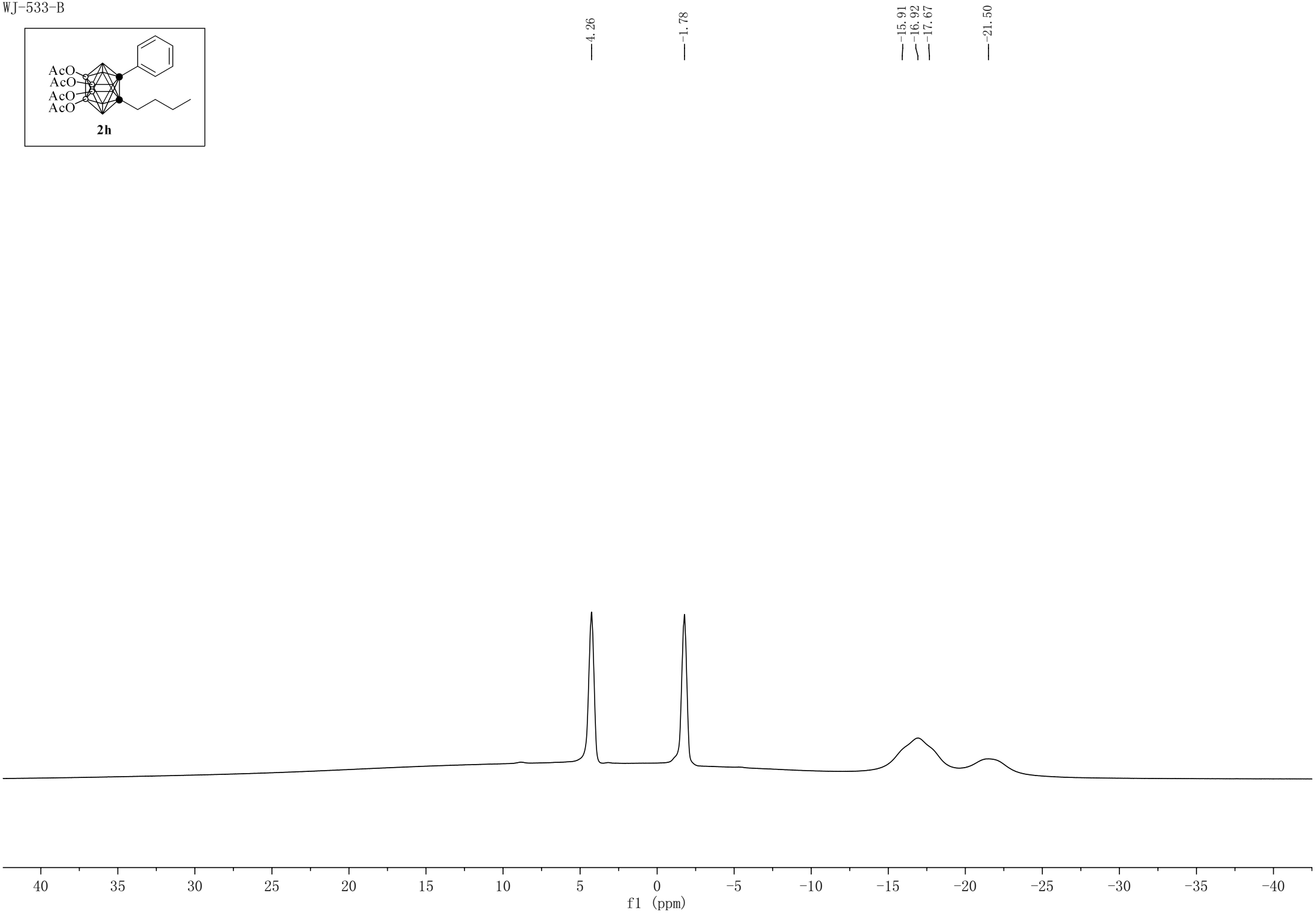
2h


4.26
1.78
17.40
21.76
2.00
2.15
3.63
1.88
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

WJ-533-B

AcO
AcO
AcO
AcO

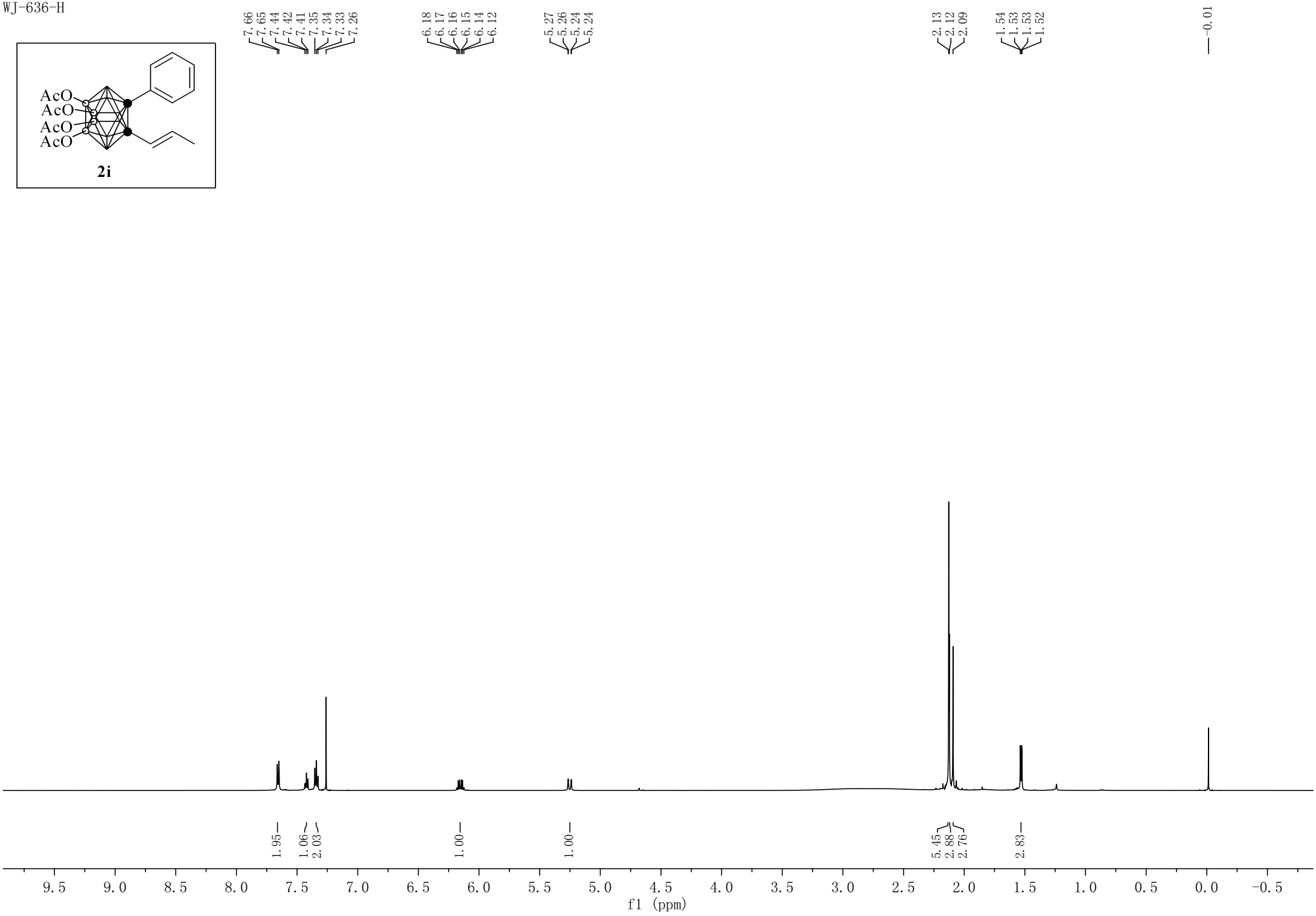
2h


4.26
1.78
15.91
16.92
17.67
21.50
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

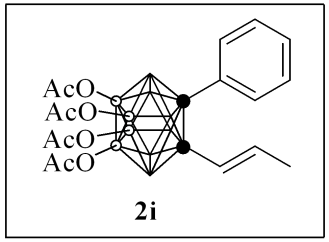
WJ-636-H

AcO
AcO
AcO
AcO

2i


7.66
7.65
7.44
7.42
7.41
7.35
7.34
7.33
7.26
6.18
6.17
6.16
6.15
6.14
6.12
5.27
5.26
5.24
5.24
2.13
2.12
2.09
1.54
1.53
1.53
1.52
-0.01
1.95
1.06
2.03
1.00
1.00
5.45
2.88
2.76
2.83
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-636-C



2i

170.13
169.56
169.48
139.63
131.90
130.76
128.70
128.60
120.84
77.21
77.00
76.79
59.81
57.68
22.43
22.37
17.75
-0.03

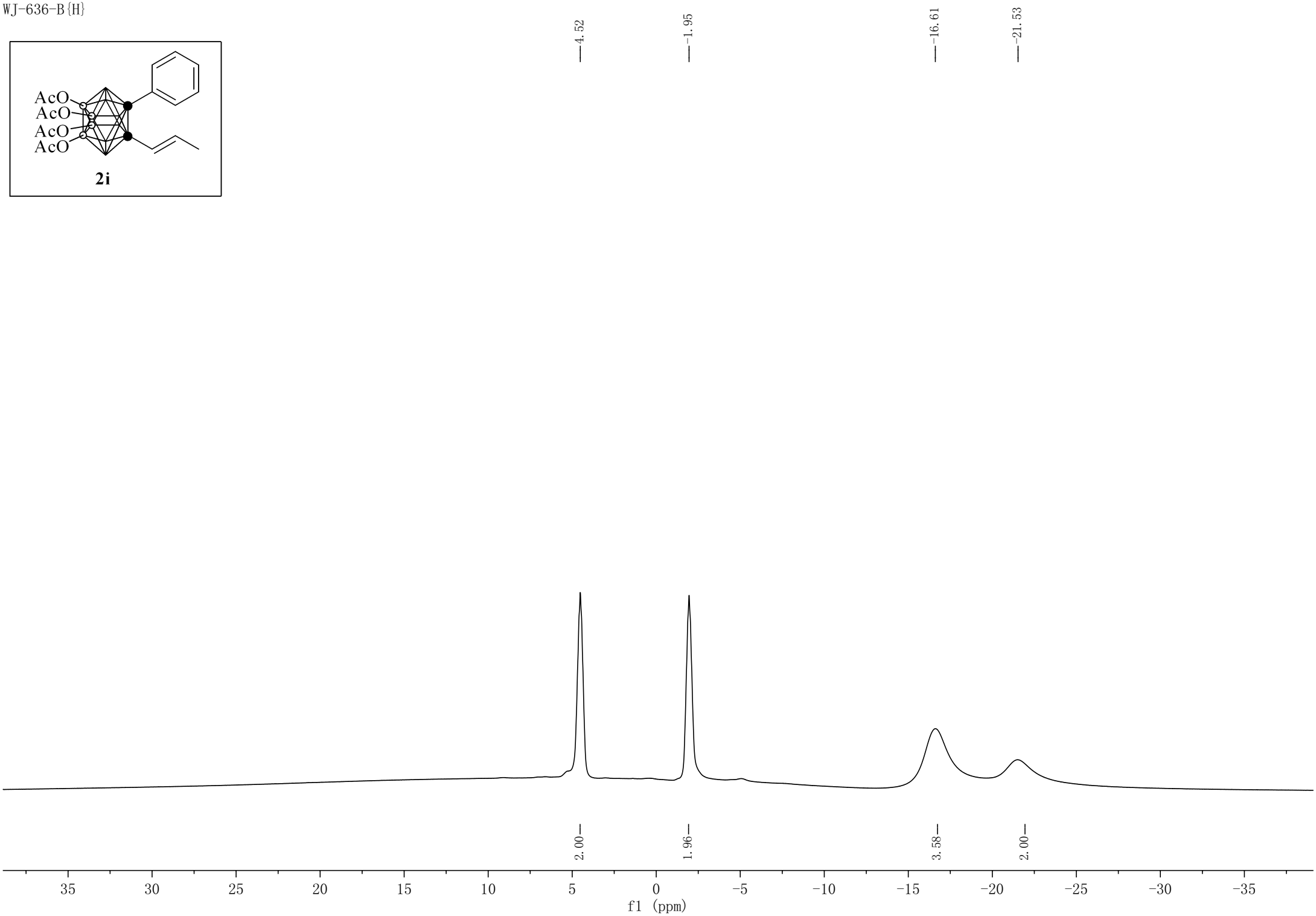
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

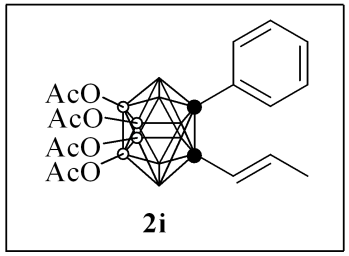
WJ-636-B{H}

AcO
AcO
AcO
AcO

2i


4.52
1.95
16.61
21.53
2.00
1.96
3.58
2.00
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

WJ-636-B



2i

4.51
-1.96
-16.69
-21.48

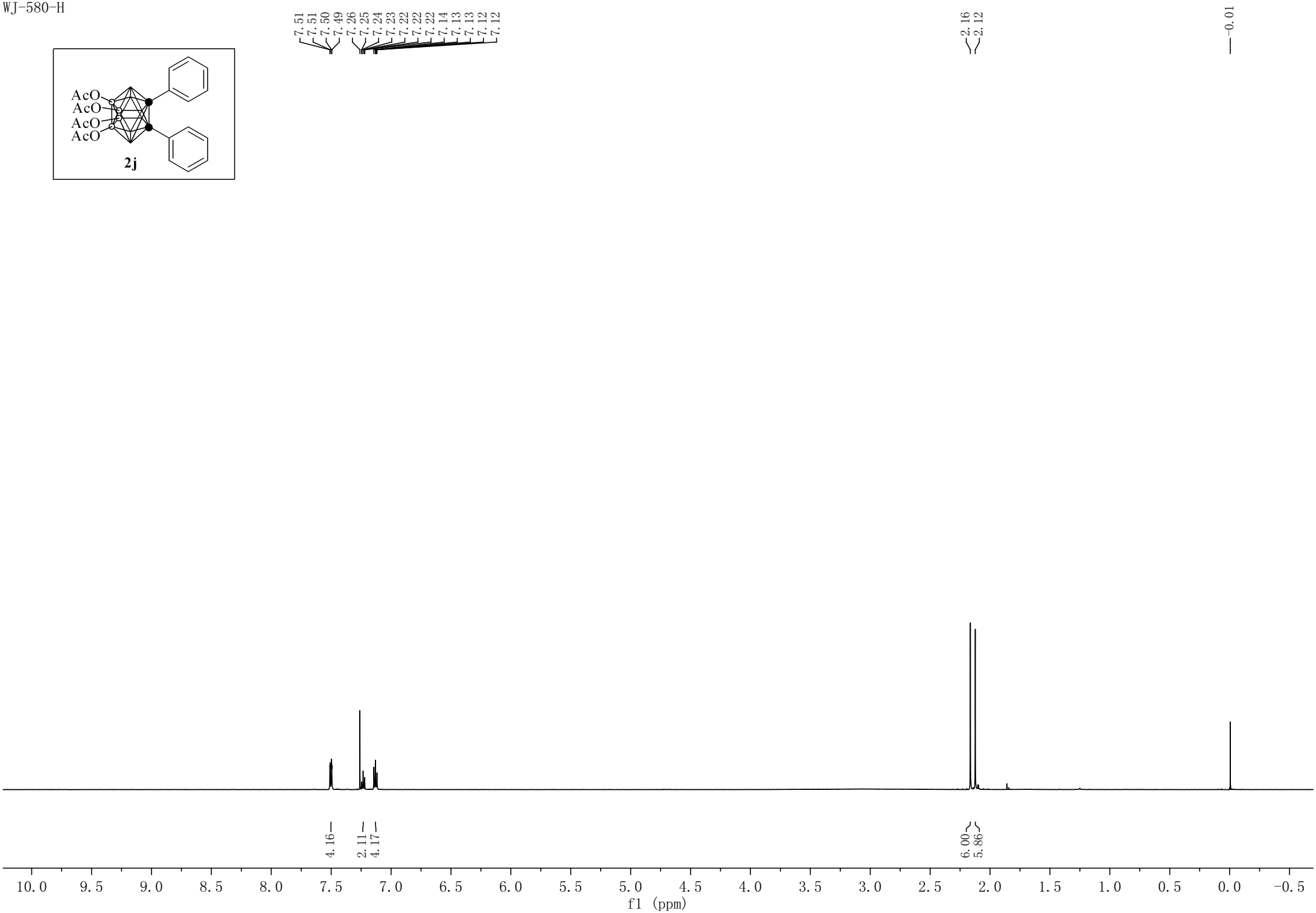
35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40

f1 (ppm)

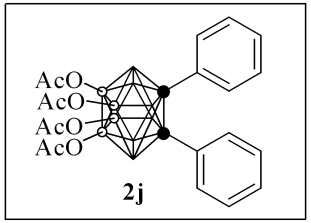
WJ-580-H

AcO
AcO
AcO
AcO

2j

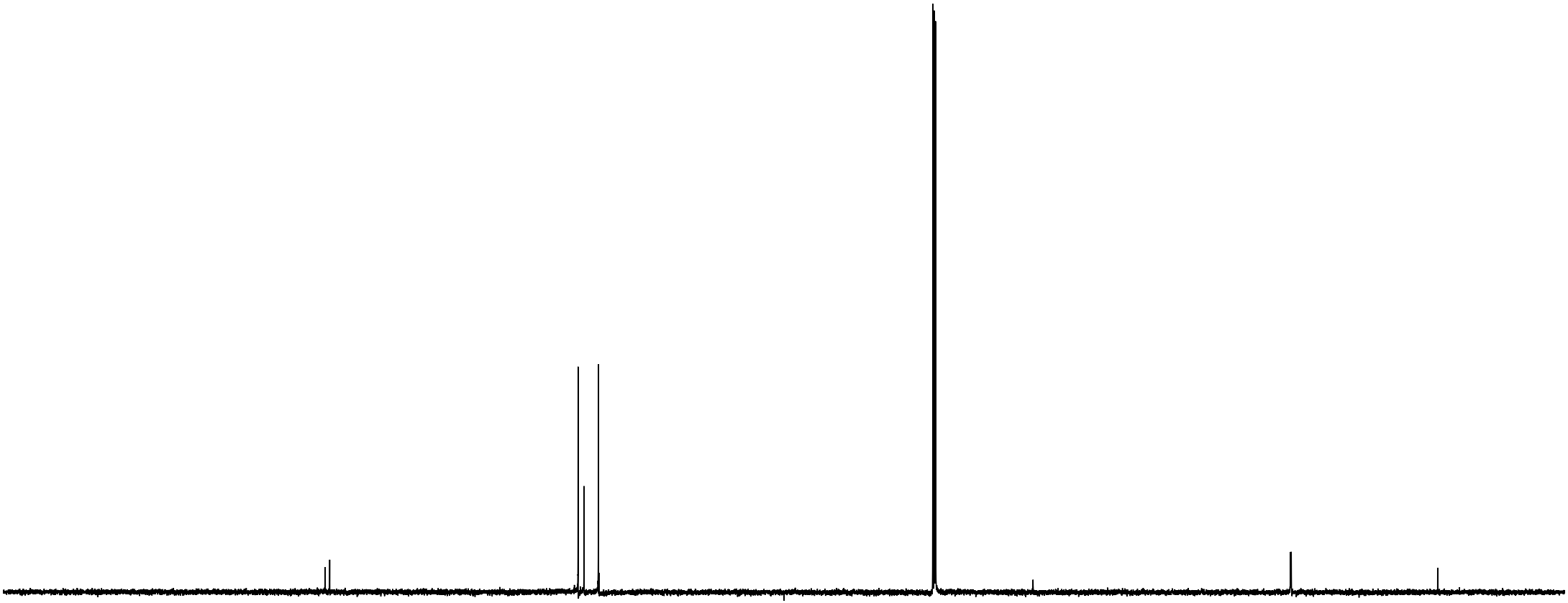

7.51
7.51
7.50
7.49
7.26
7.25
7.24
7.23
7.22
7.22
7.22
7.14
7.13
7.12
7.12
2.16
2.12
-0.01
4.16
2.11
4.17
6.00
5.86
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-580-C



2j

170.19
169.49
131.48
130.58
128.38
128.29
77.21
77.00
76.79
61.91
22.50
22.37
-0.03



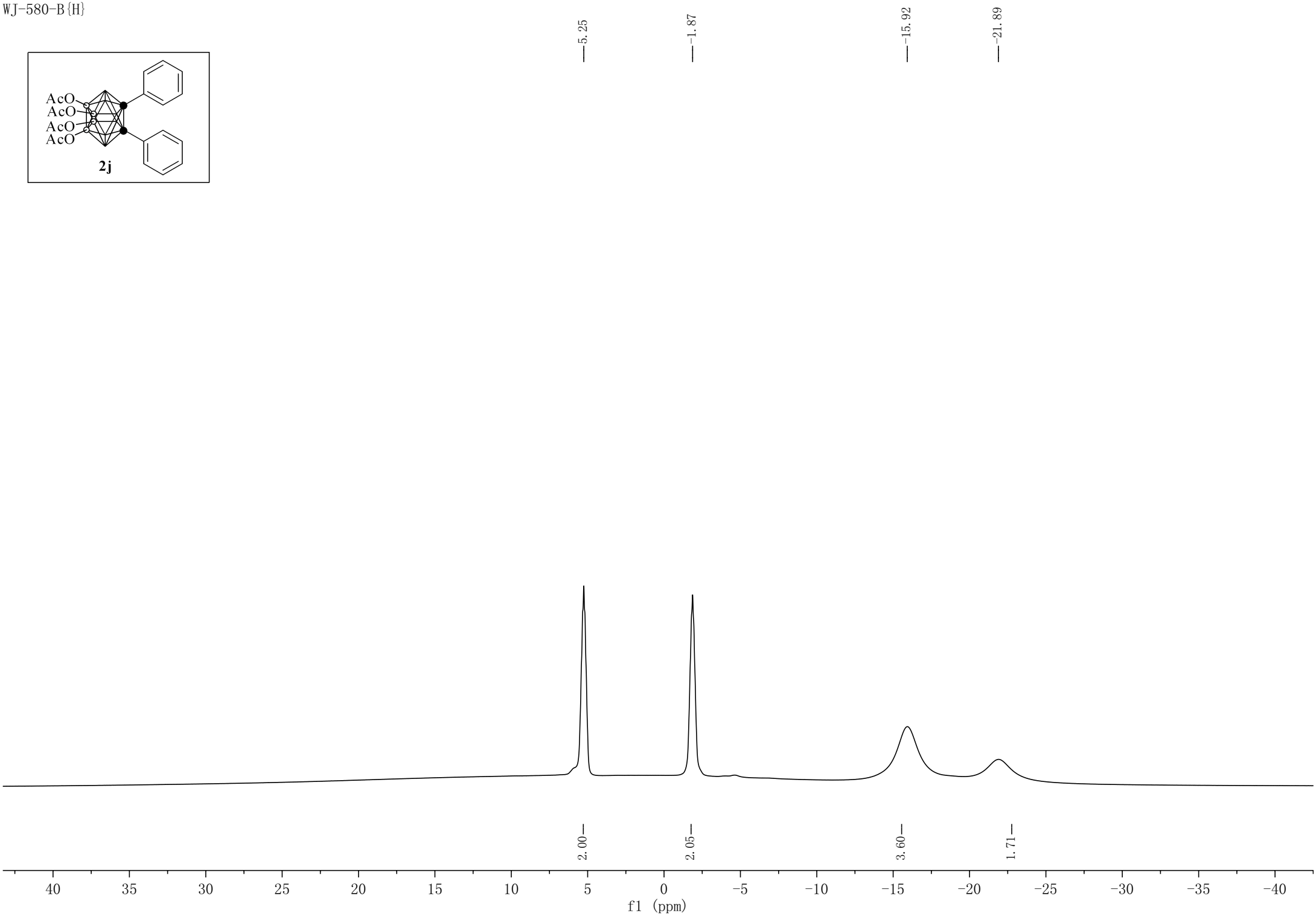
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

WJ-580-B{H}

AcO
AcO
AcO
AcO

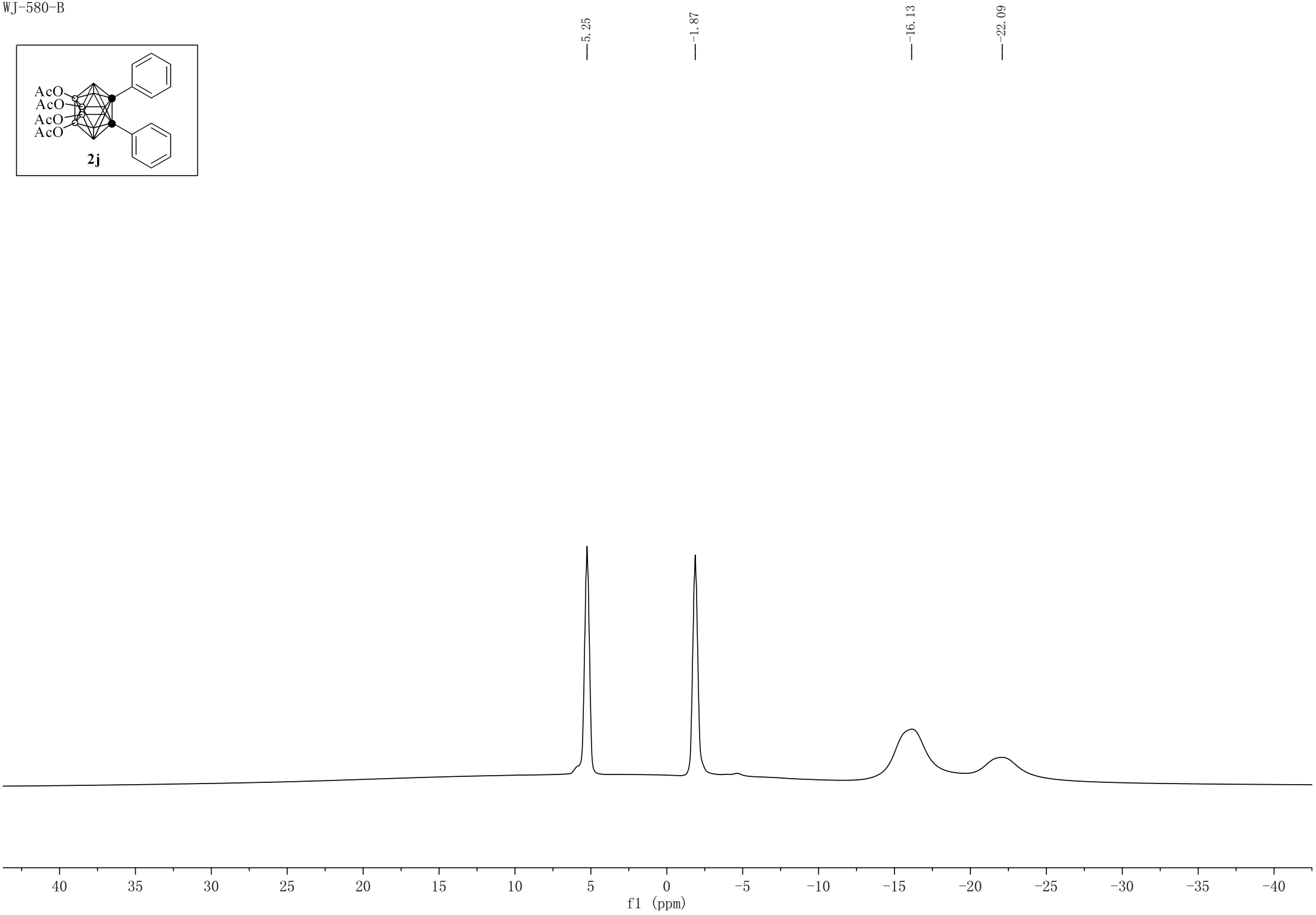
2j


5.25
1.87
15.92
21.89
2.00
2.05
3.60
1.71
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

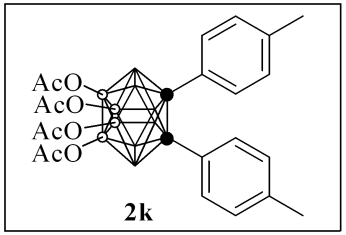
WJ-580-B

AcO
AcO
AcO
AcO

2j


5.25
1.87
16.13
22.09
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

WJ-655-H



2k

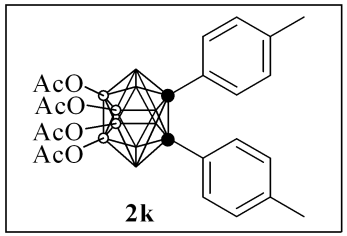
7.39
7.37
7.26
6.94
6.93
2.22
2.16
2.12
0.00

4.00
3.99
5.81
5.71
5.71

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

WJ-655-C



2k

170.25
169.52
140.90
131.40
129.09
125.58
77.21
77.00
76.79
62.28
22.52
22.40
20.99
-0.02

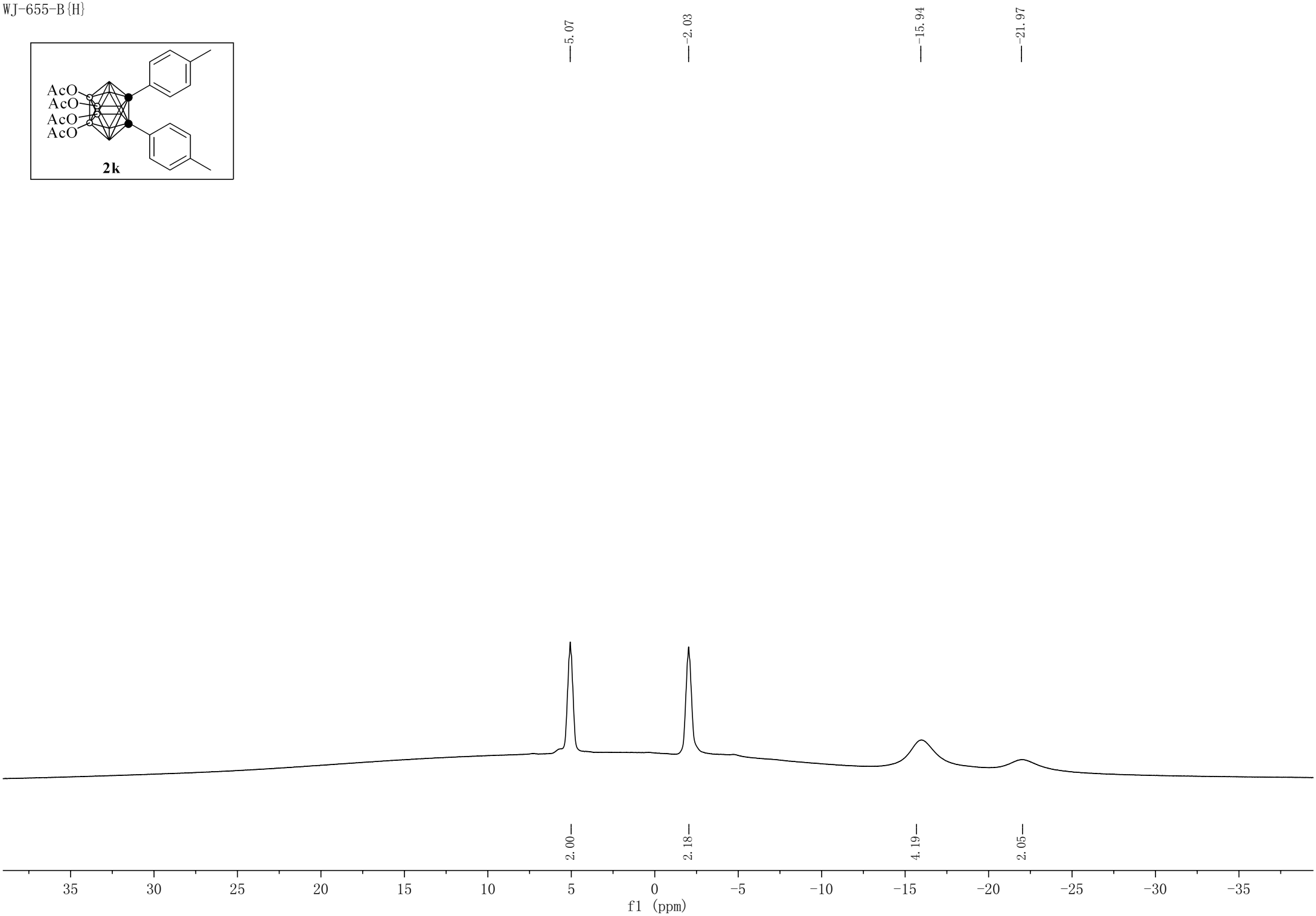
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

WJ-655-B{H}

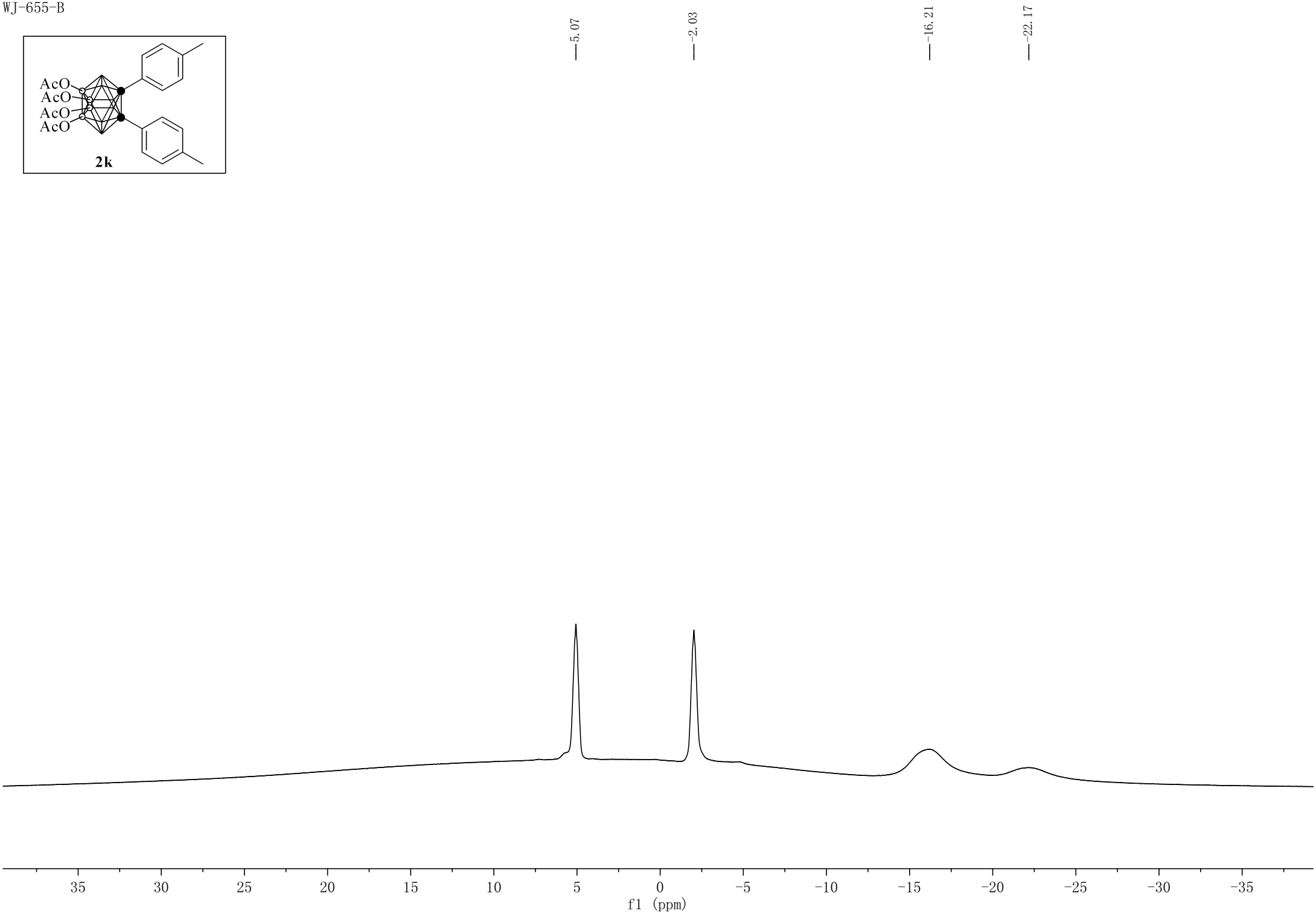
AcO
AcO
AcO
AcO

2k


5.07
2.03
-15.94
-21.97
2.00
2.18
4.19
2.05
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

WJ-655-B

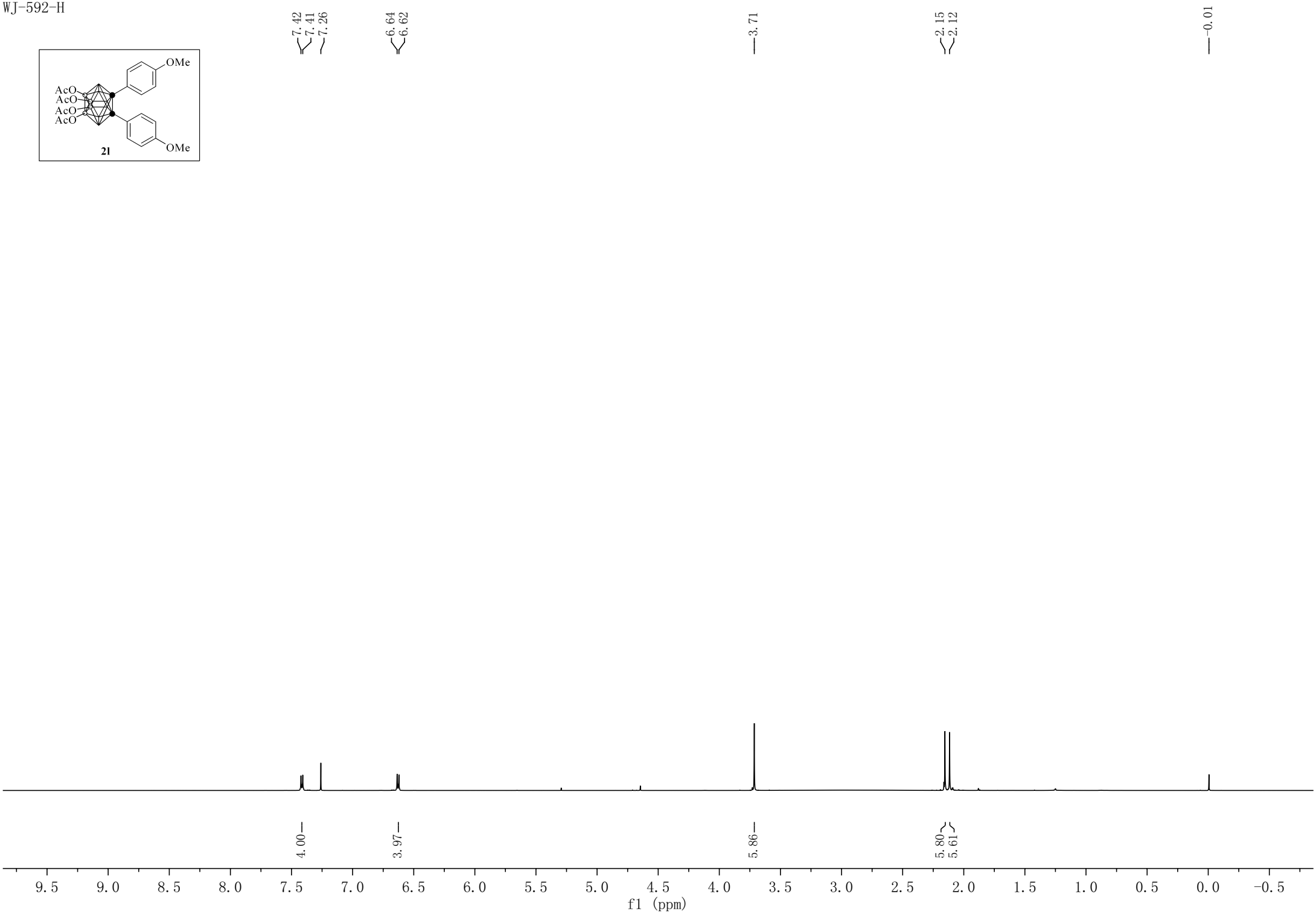
AcO
AcO
AcO
AcO
2k


5.07
2.03
16.21
22.17
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

WJ-592-H

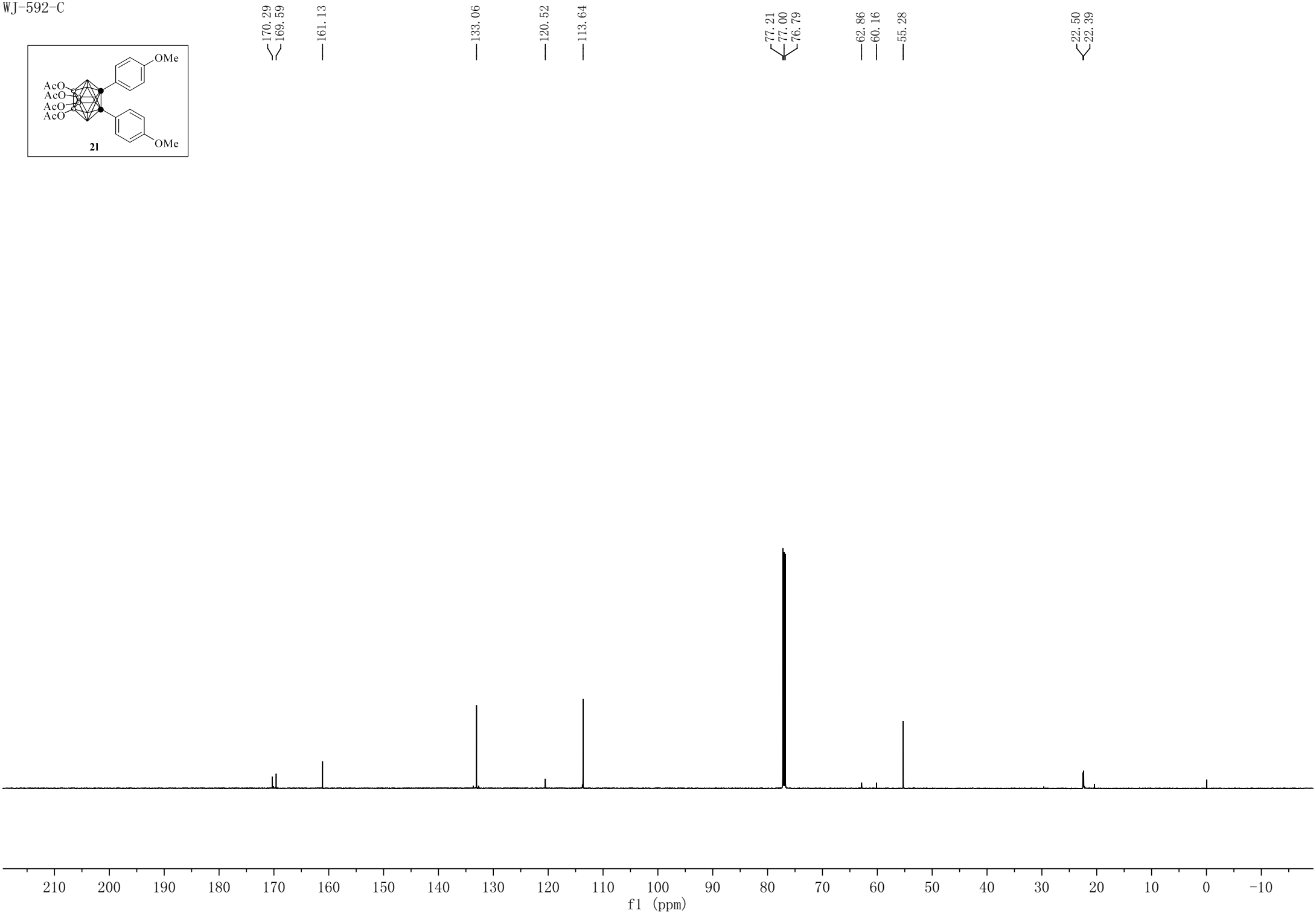
OMe
AcO
AcO
AcO
AcO
OMe

2l


7.42
7.41
7.26
6.64
6.62
3.71
2.15
2.12
0.01
4.00
3.97
5.86
5.80
5.61
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-592-C

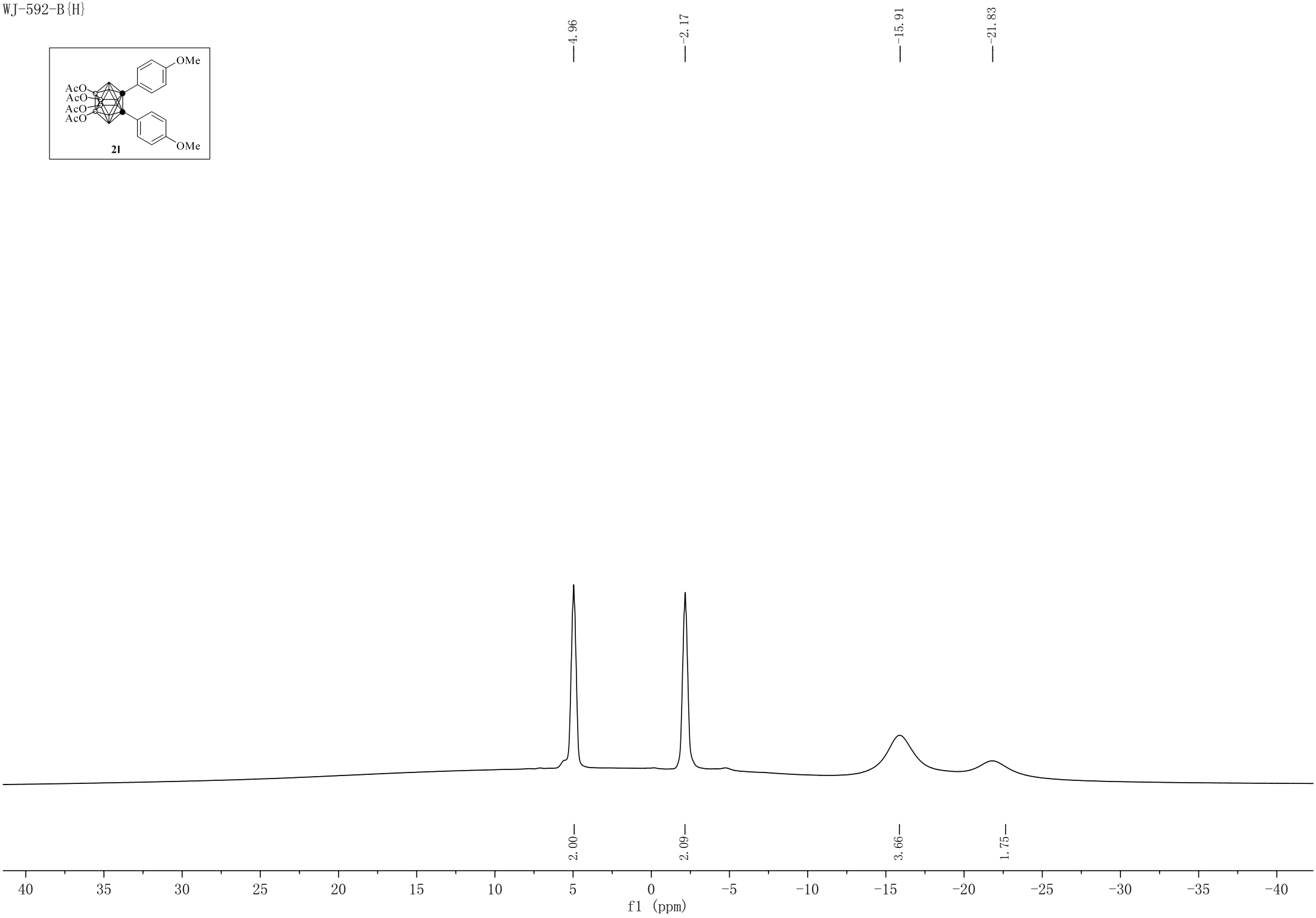
OMe
AcO
AcO
AcO
AcO
OMe
21


170.29
169.59
161.13
133.06
120.52
113.64
77.21
77.00
76.79
62.86
60.16
55.28
22.50
22.39
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

WJ-592-B{H}

OMe
AcO
AcO
AcO
AcO
OMe

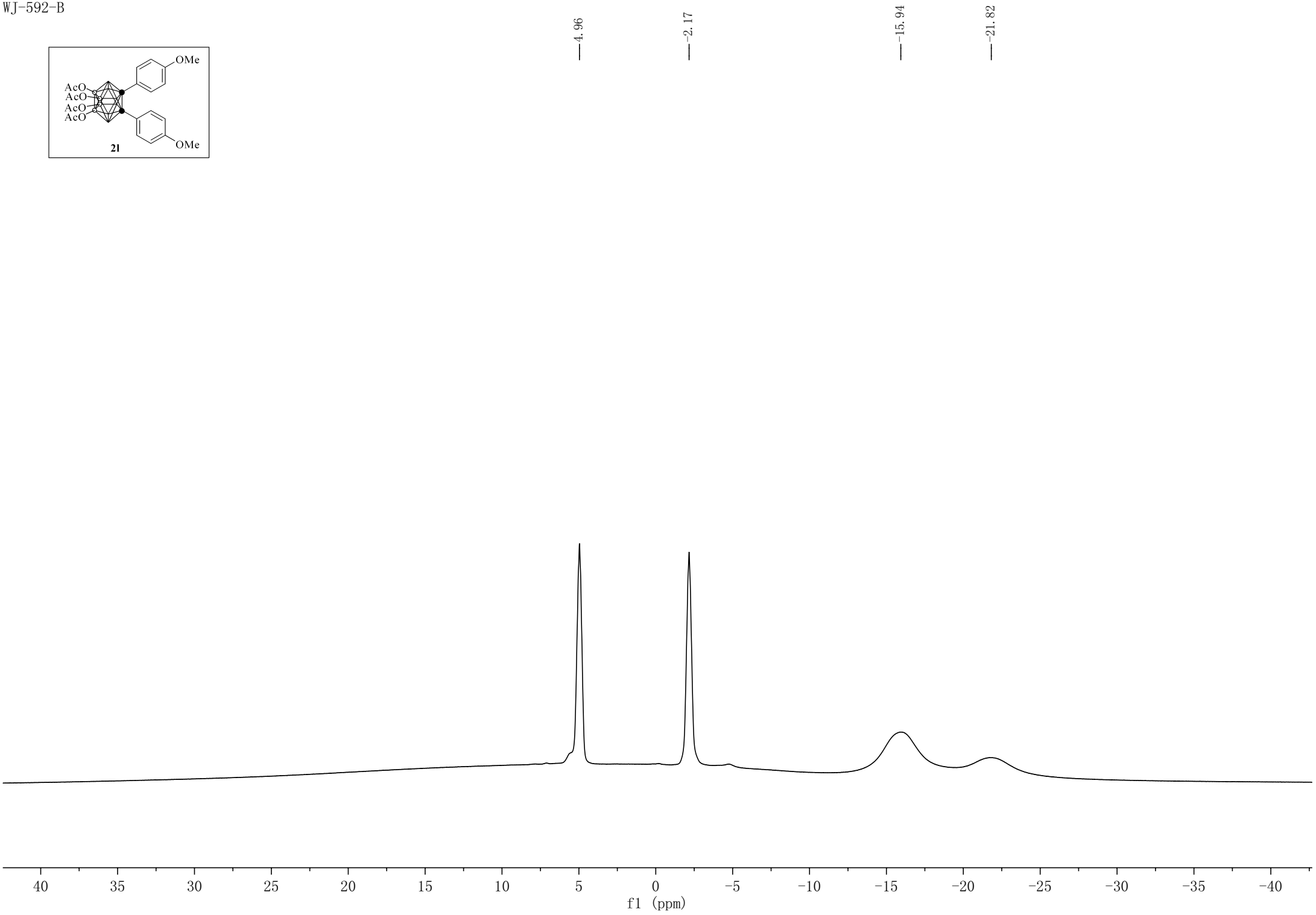
21


4.96
2.17
15.91
21.83
2.00
2.09
3.66
1.75
40
35
30
25
20
15
10
5
0
−5
−10
−15
−20
−25
−30
−35
−40
f1 (ppm)

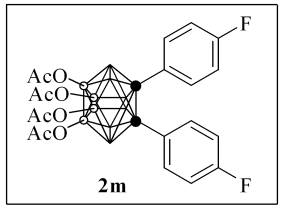
WJ-592-B

OMe
AcO
AcO
AcO
AcO
OMe

21


4.96
2.17
-15.94
-21.82
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

WJ-582-H



2m

7.52
7.51
7.51
7.50
7.50
7.26
6.87
6.86
6.84
2.17
2.13
0.00

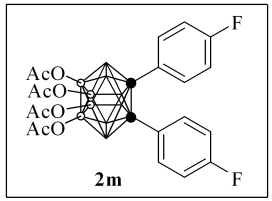
impurity in petroleum ether

4.00
4.07
5.57
5.69

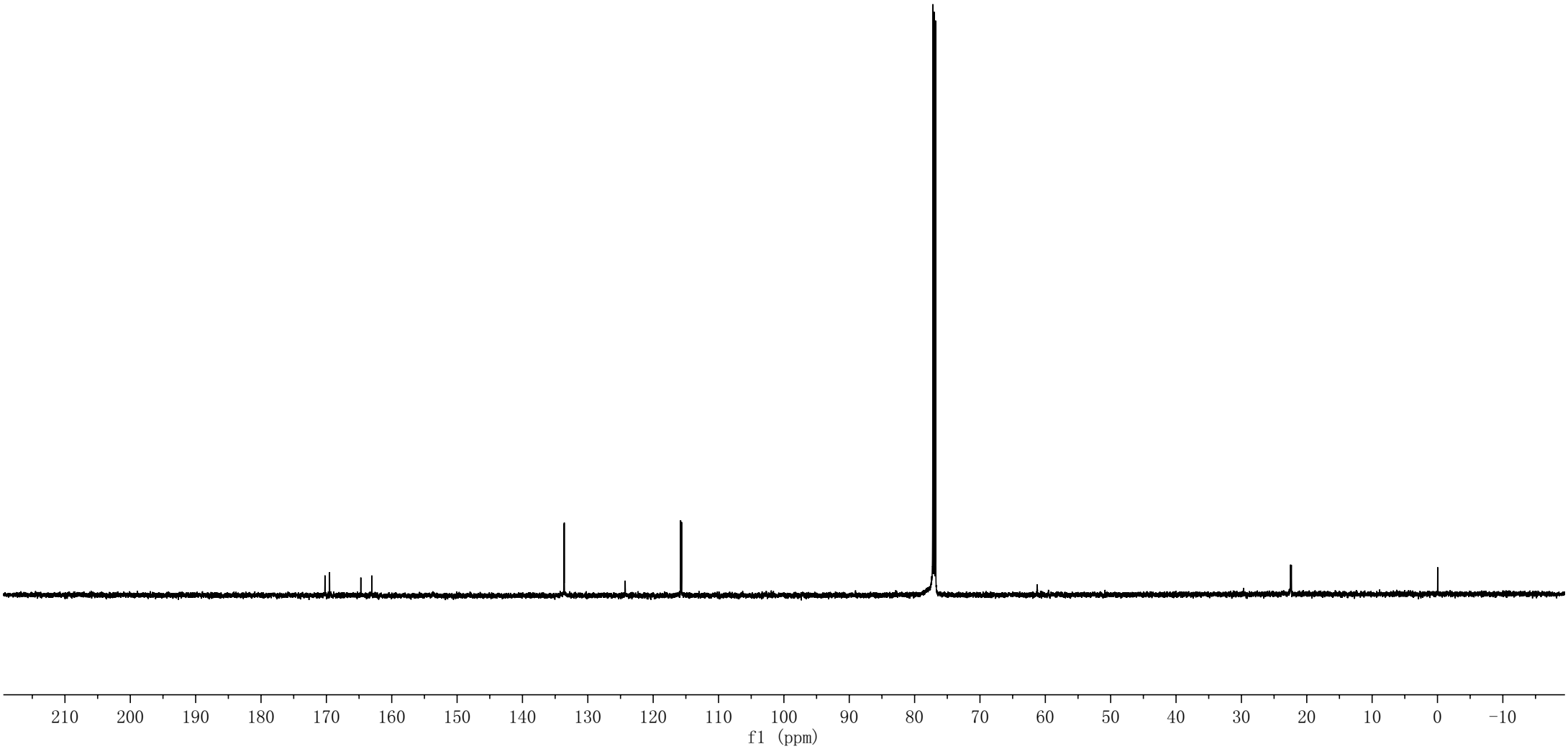
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

WJ-582-C



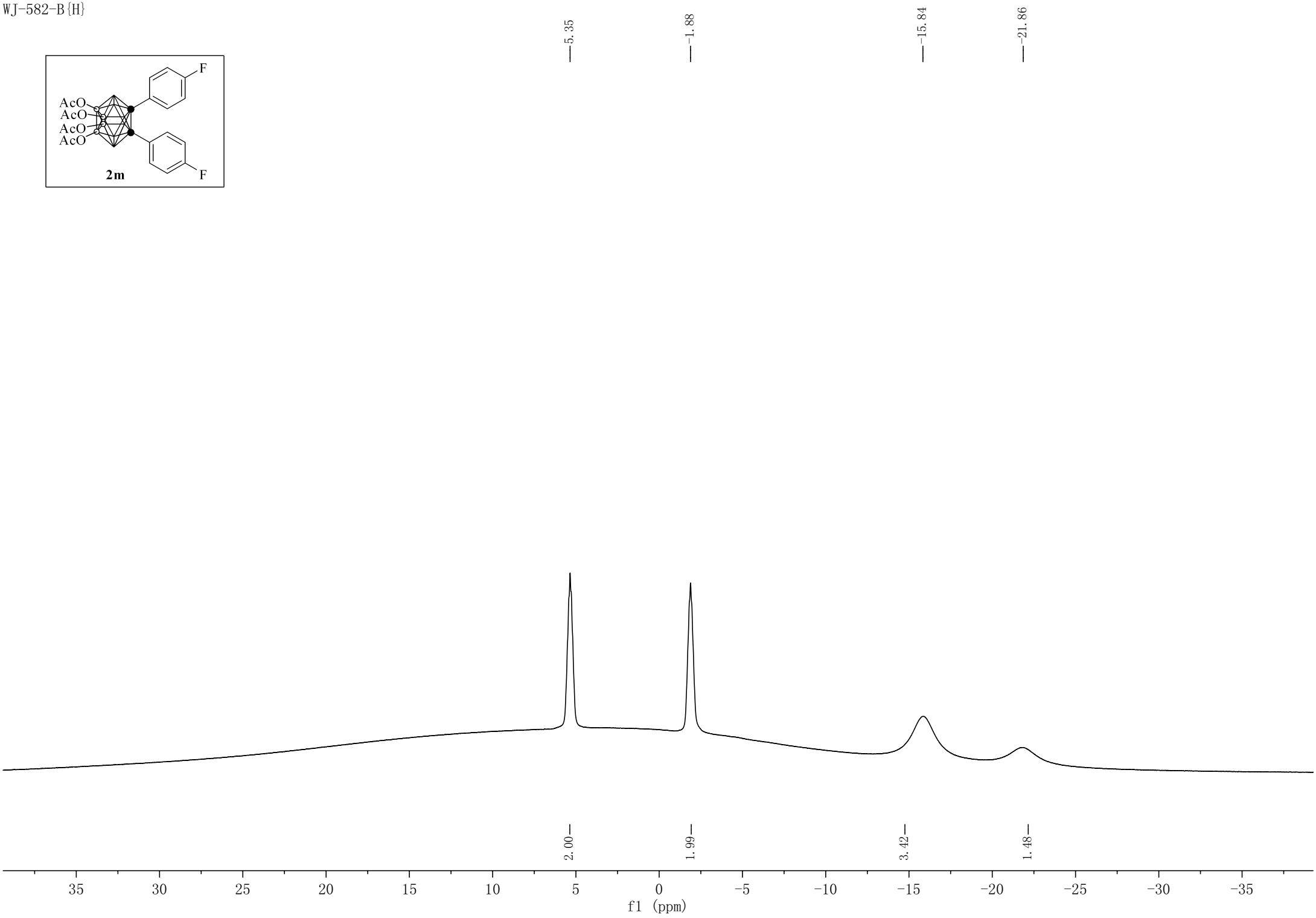
170.22
169.51
164.73
163.05
133.66
133.60
124.29
115.80
115.65
77.21
77.00
76.79
61.23
22.47
22.35
-0.03



WJ-582-B{H}

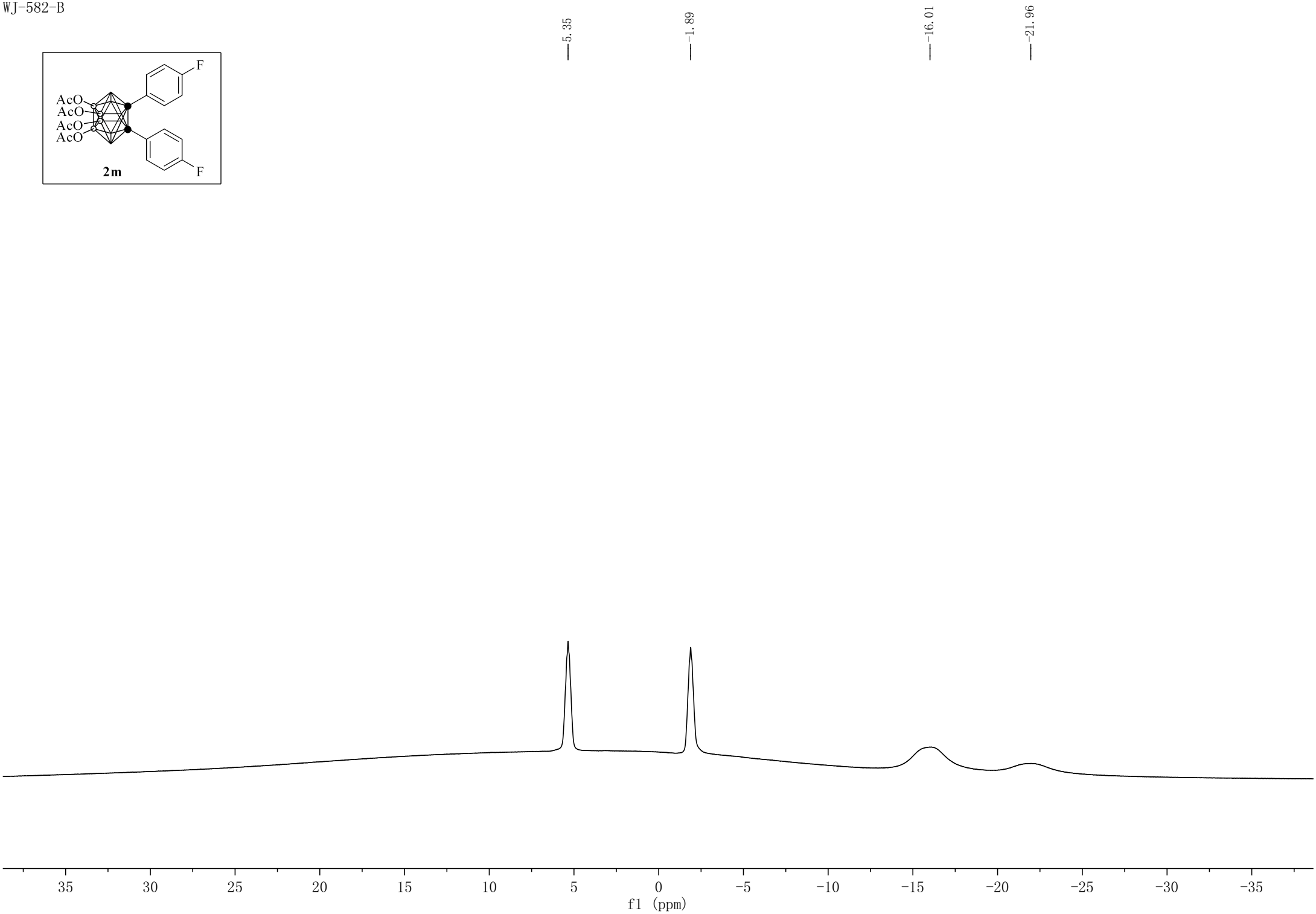
AcO
AcO
AcO
AcO
F
F

2m


5.35
1.88
15.84
21.86
2.00
1.99
3.42
1.48
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

WJ-582-B

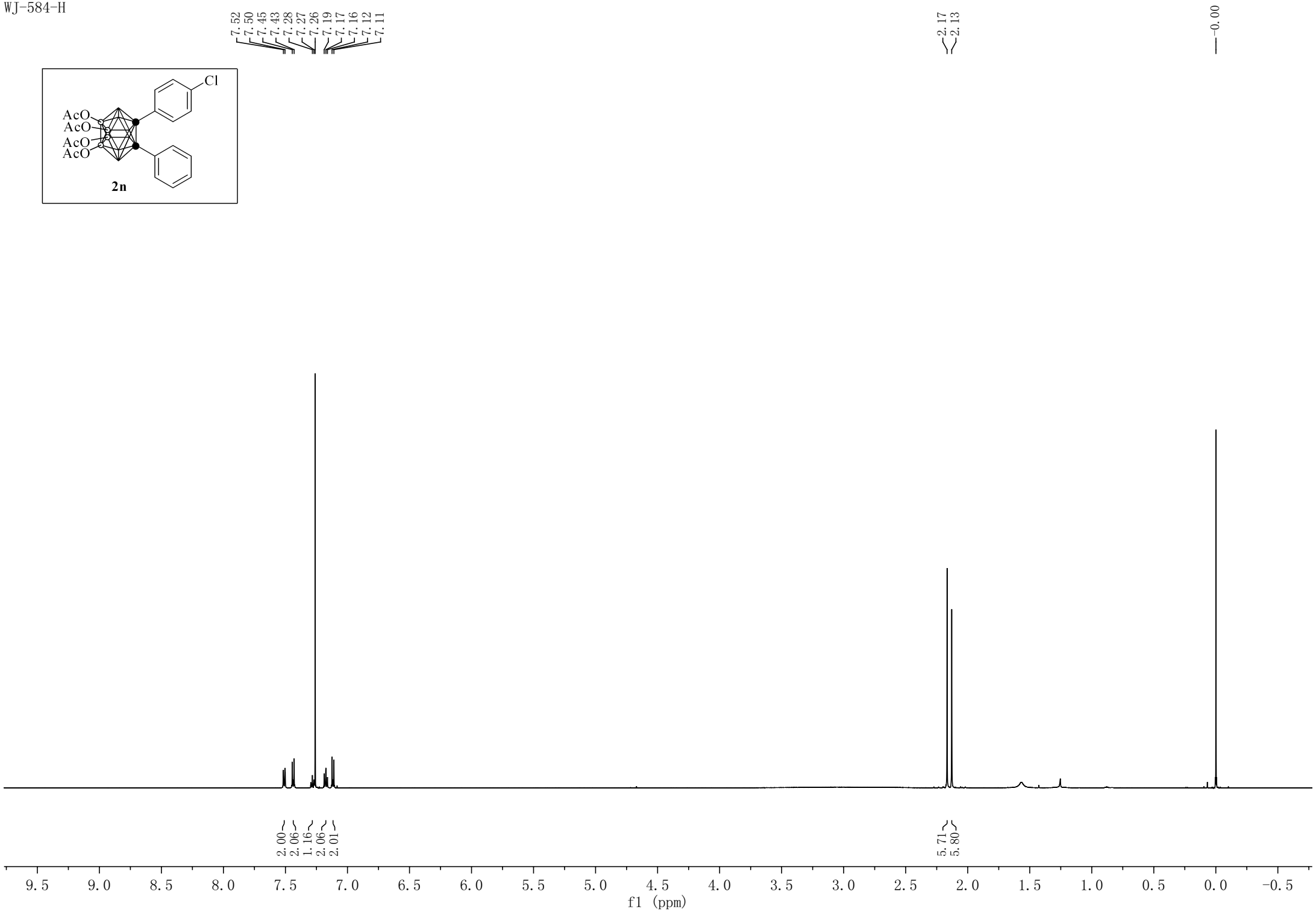
AcO
AcO
AcO
AcO
F
F
2m


5.35
1.89
16.01
21.96
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

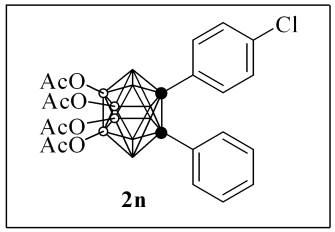
WJ-584-H

Cl
AcO
AcO
AcO
AcO

2n


7.52
7.50
7.45
7.43
7.28
7.27
7.26
7.19
7.17
7.16
7.12
7.11
2.17
2.13
0.00
2.00
2.06
1.16
2.06
2.01
5.71
5.80
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

WJ-584-C

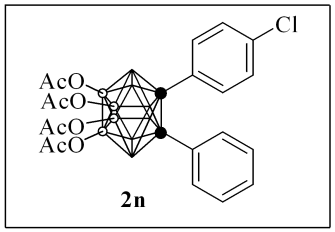


2n

170.20
169.48
137.21
132.72
131.49
130.87
128.66
128.61
128.10
127.08
77.21
77.00
76.79
61.98
60.92
22.48
22.36
-0.02

f1 (ppm)

WJ-584-B{H}



2n

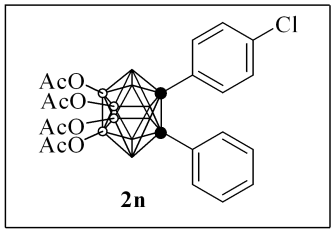
5.34
1.84
15.86
21.92

2.00
1.92
3.77
1.61

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

f1 (ppm)

WJ-584-B



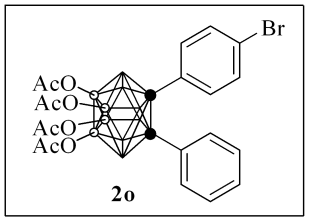
2n

5.34
-1.84
-15.94
-22.07

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

f1 (ppm)

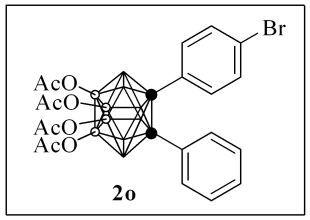
WJ-656-H



2o

f1 (ppm)

WJ-656-C



2o

170.21
169.49
132.91
131.65
131.49
130.89
128.63
128.07
127.62
125.61
77.21
77.00
76.79
61.91
60.97
22.48
22.37
-0.02

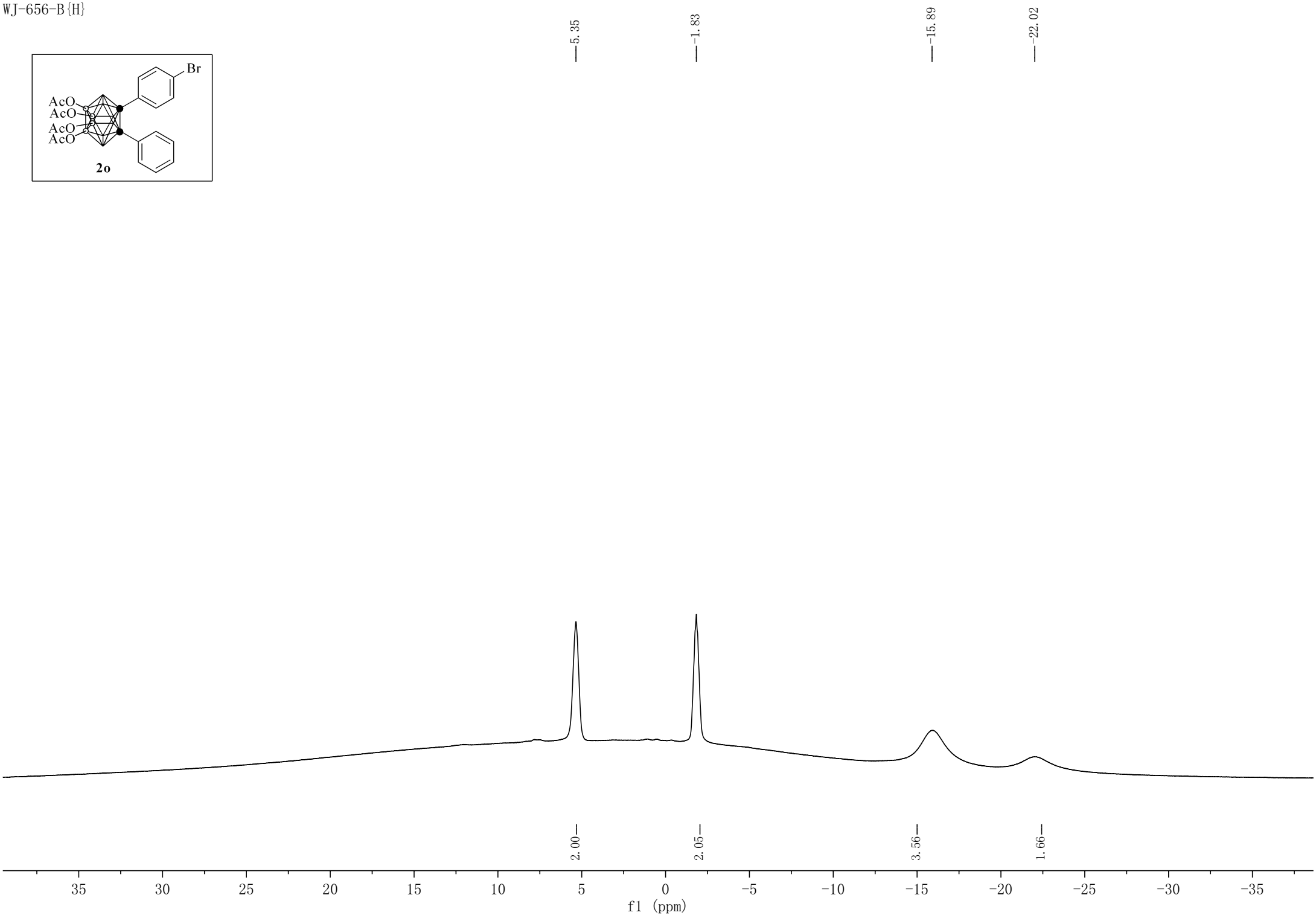
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

WJ-656-B{H}

Br
AcO
AcO
AcO
AcO

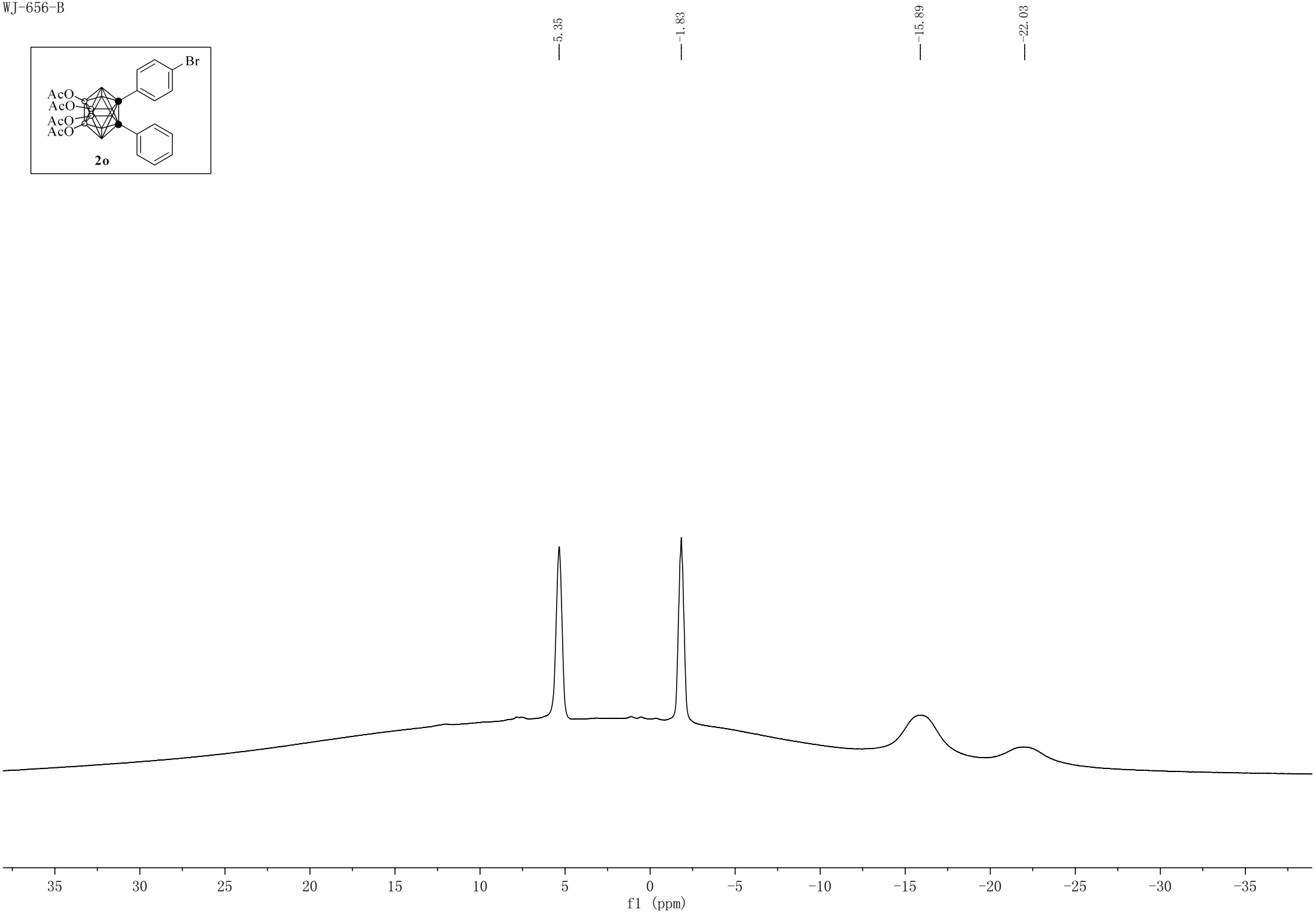
2o


5.35
1.83
15.89
22.02
2.00
2.05
3.56
1.66
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

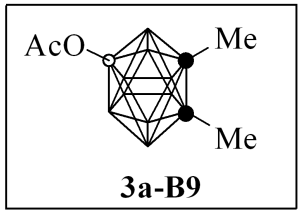
WJ-656-B

Br
AcO
AcO
AcO
AcO

2o


5.35
1.83
-15.89
-22.03
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

XT-400-B9-H



3a-B9

7.26

2.06
2.04
2.01

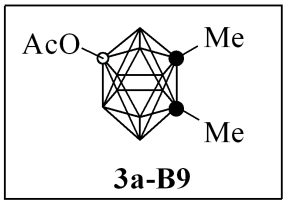
-0.01

3.00
3.31
3.18

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-400-B9-C



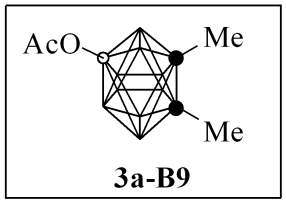
3a-B9

170.05
77.21
77.00
76.79
68.99
61.87
23.68
22.62
21.16
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

XT-400-B9-B{H}



3a-B9

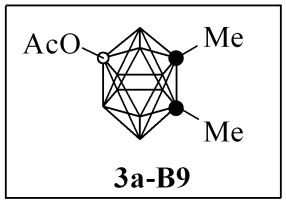
9.50
5.82
10.30
10.97
11.80

1.00
0.97
3.41
1.92
1.70

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

f1 (ppm)

XT-400-B9-B



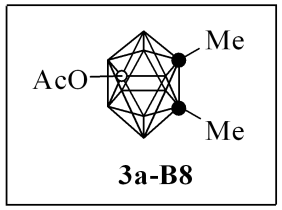
3a-B9

9.51
5.43
6.20
9.85
10.68
11.38
12.23

40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 −25 −30 −35 −40

f1 (ppm)

XT-400-B8-H



3a-B8

7.26

2.08
2.04

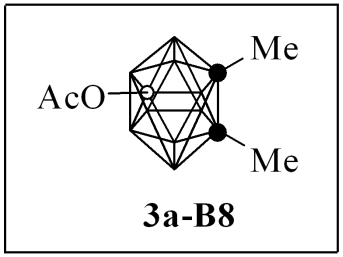
0.01

3.00
6.23

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-400-B8-C



3a-B8

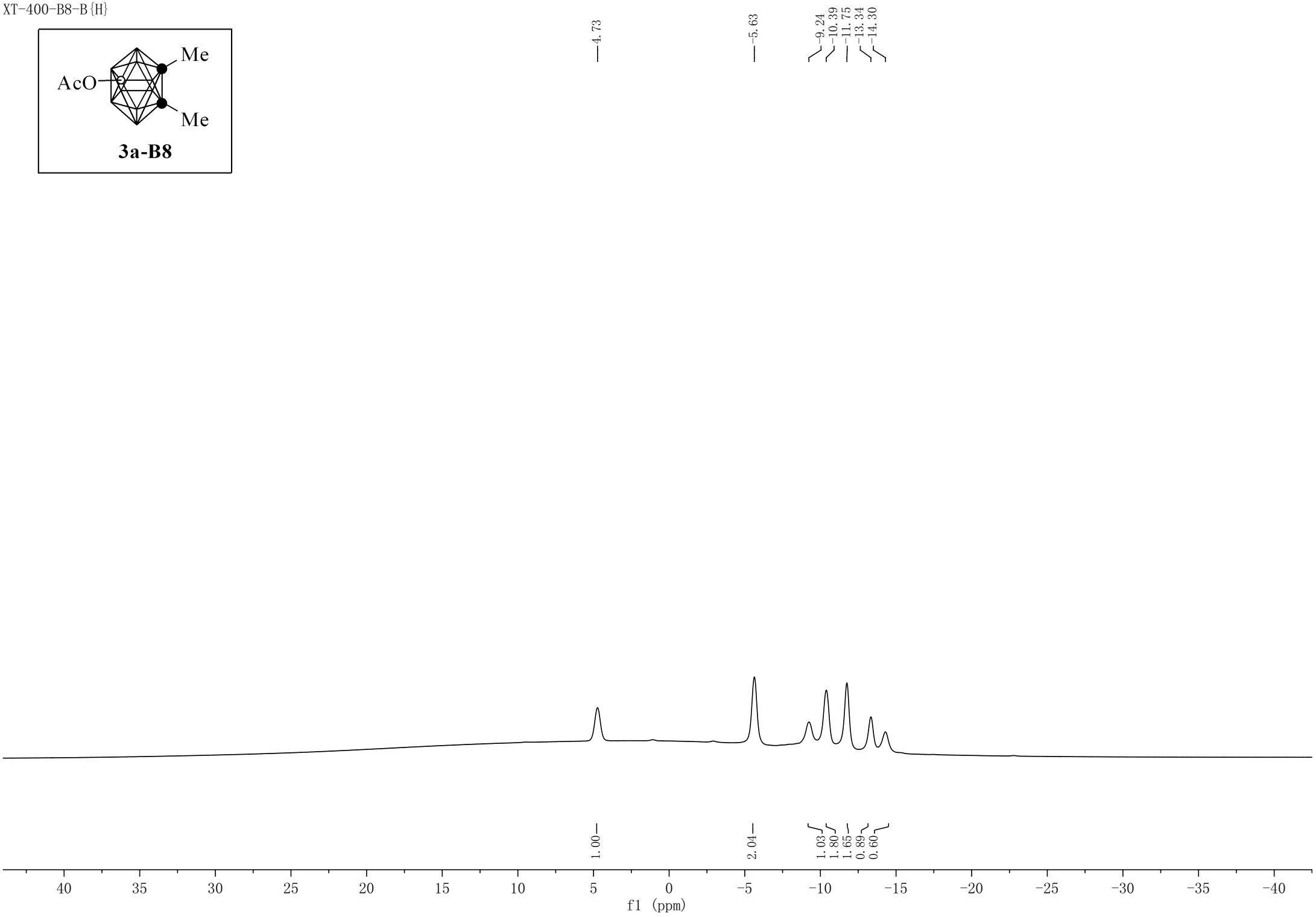
170.50
77.21
77.00
76.79
68.51
22.98
22.52
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

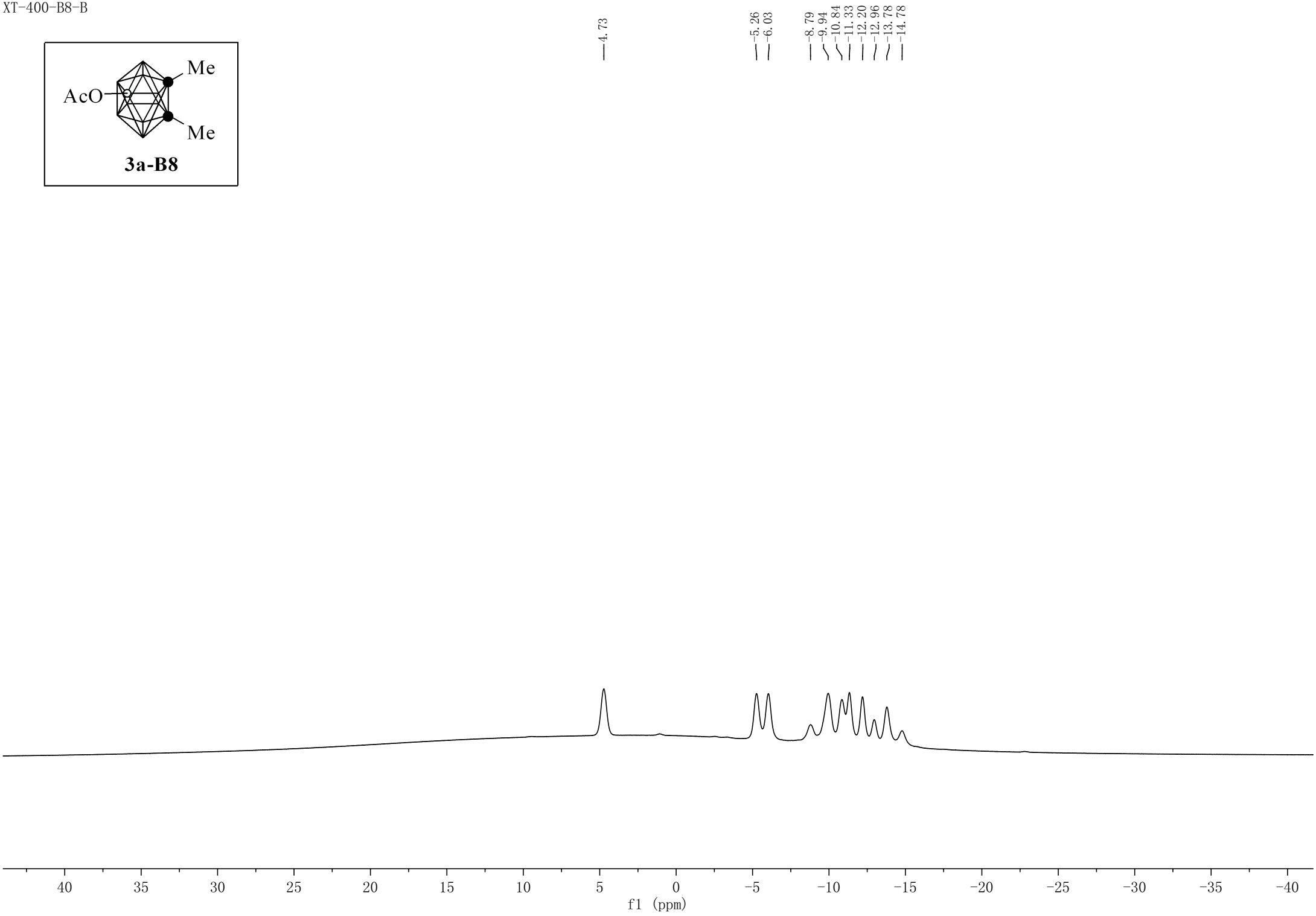
XT-400-B8-B{H}

3a-B8



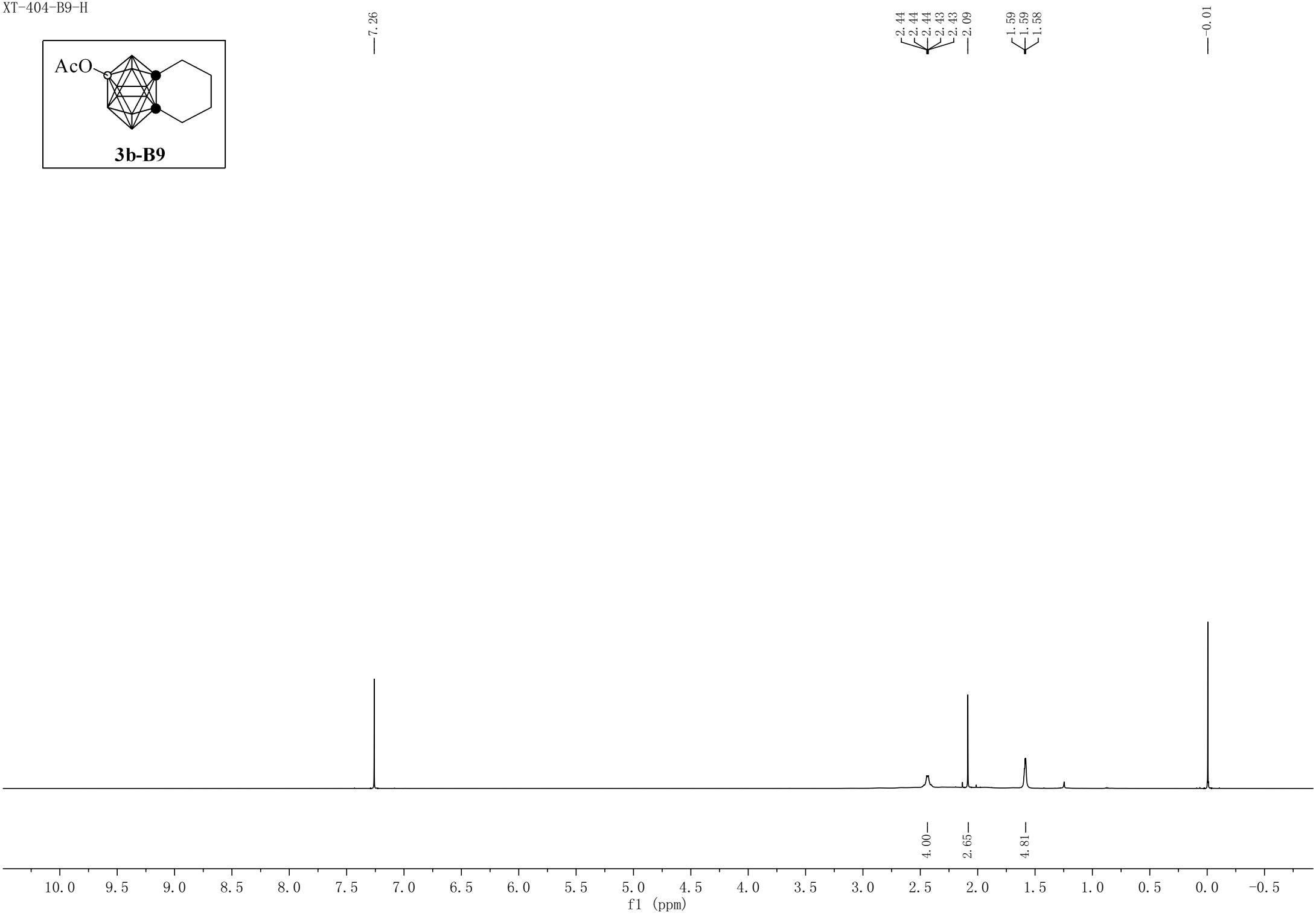
XT-400-B8-B

3a-B8

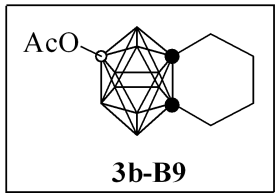


XT-404-B9-H

3b-B9



XT-404-B9-C



3b-B9

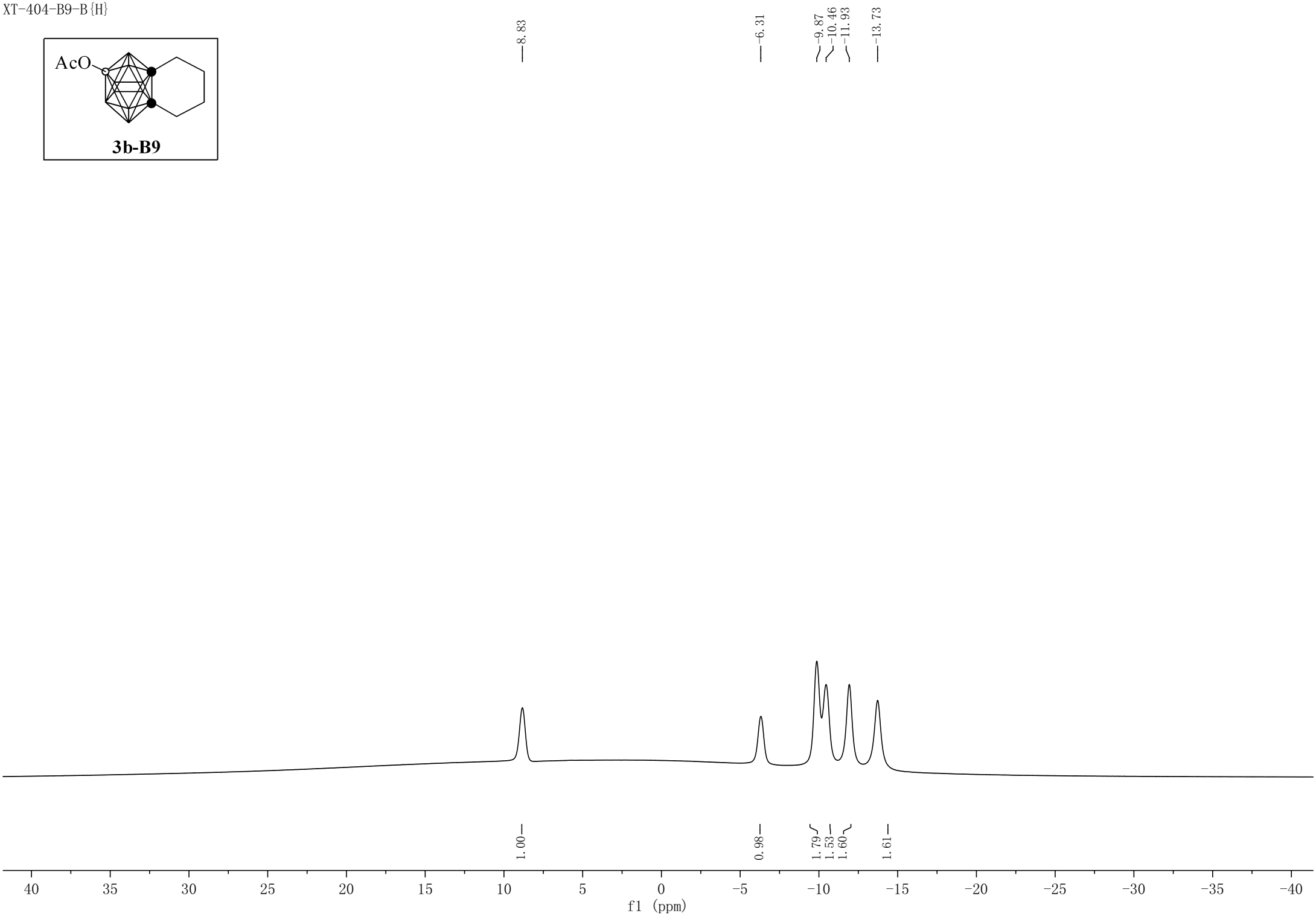
170.09
77.21
77.00
76.79
68.64
61.58
32.98
30.88
22.63
19.89
19.30
0.02

impurity in petroleum ether

f1 (ppm)

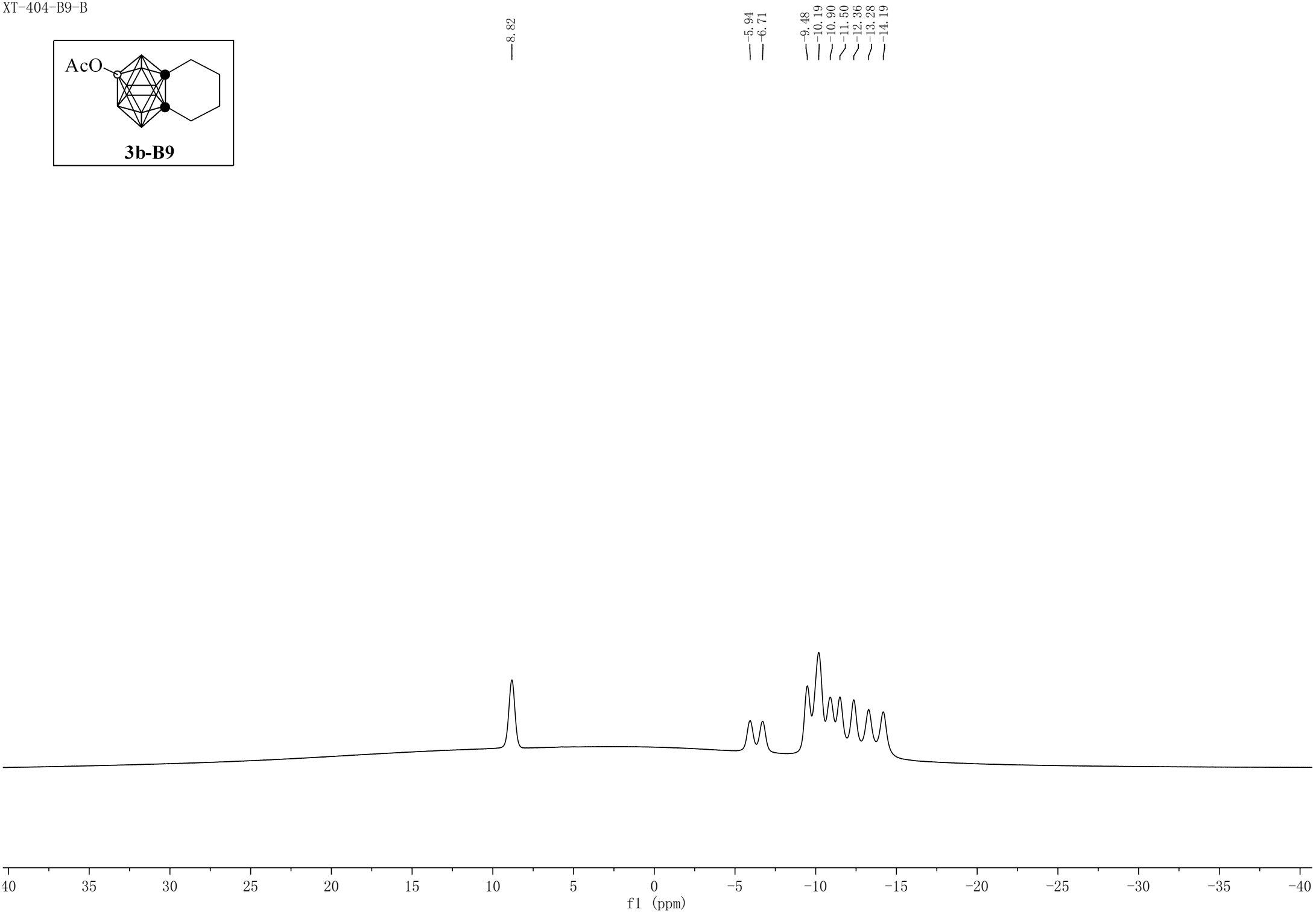
XT-404-B9-B{H}

AcO
3b-B9


8.83
6.31
9.87
10.46
11.93
13.73
1.00
0.98
1.79
1.53
1.60
1.61
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

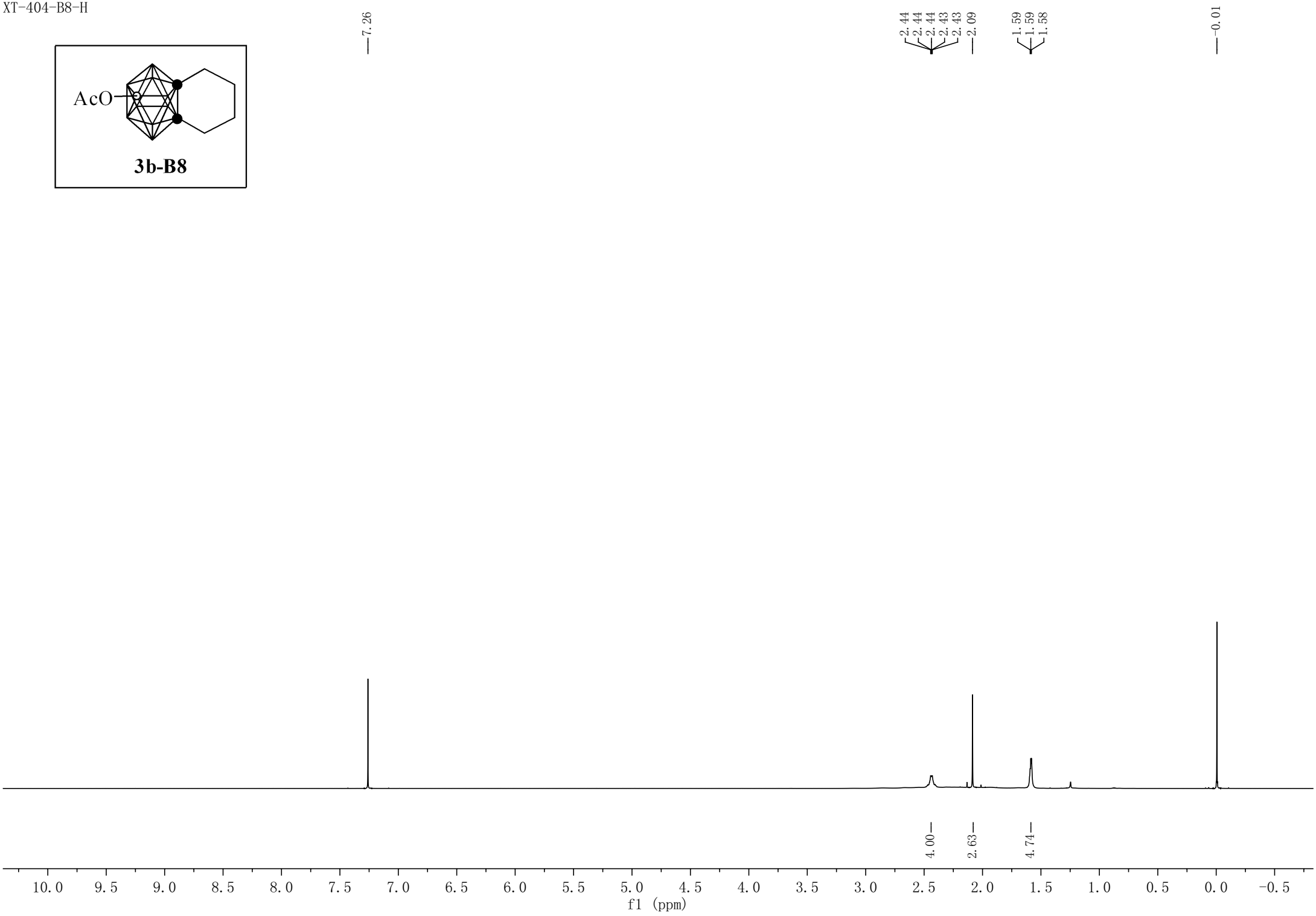
XT-404-B9-B

AcO
3b-B9

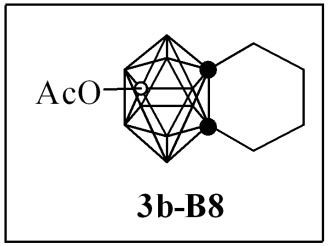

8.82
5.94
6.71
9.48
10.19
10.90
11.50
12.36
13.28
14.19
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-404-B8-H

AcO
3b-B8


7.26
2.44
2.44
2.44
2.43
2.43
2.09
1.59
1.59
1.58
0.01
4.00
2.63
4.74
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

XT-404-B8-C



3b-B8

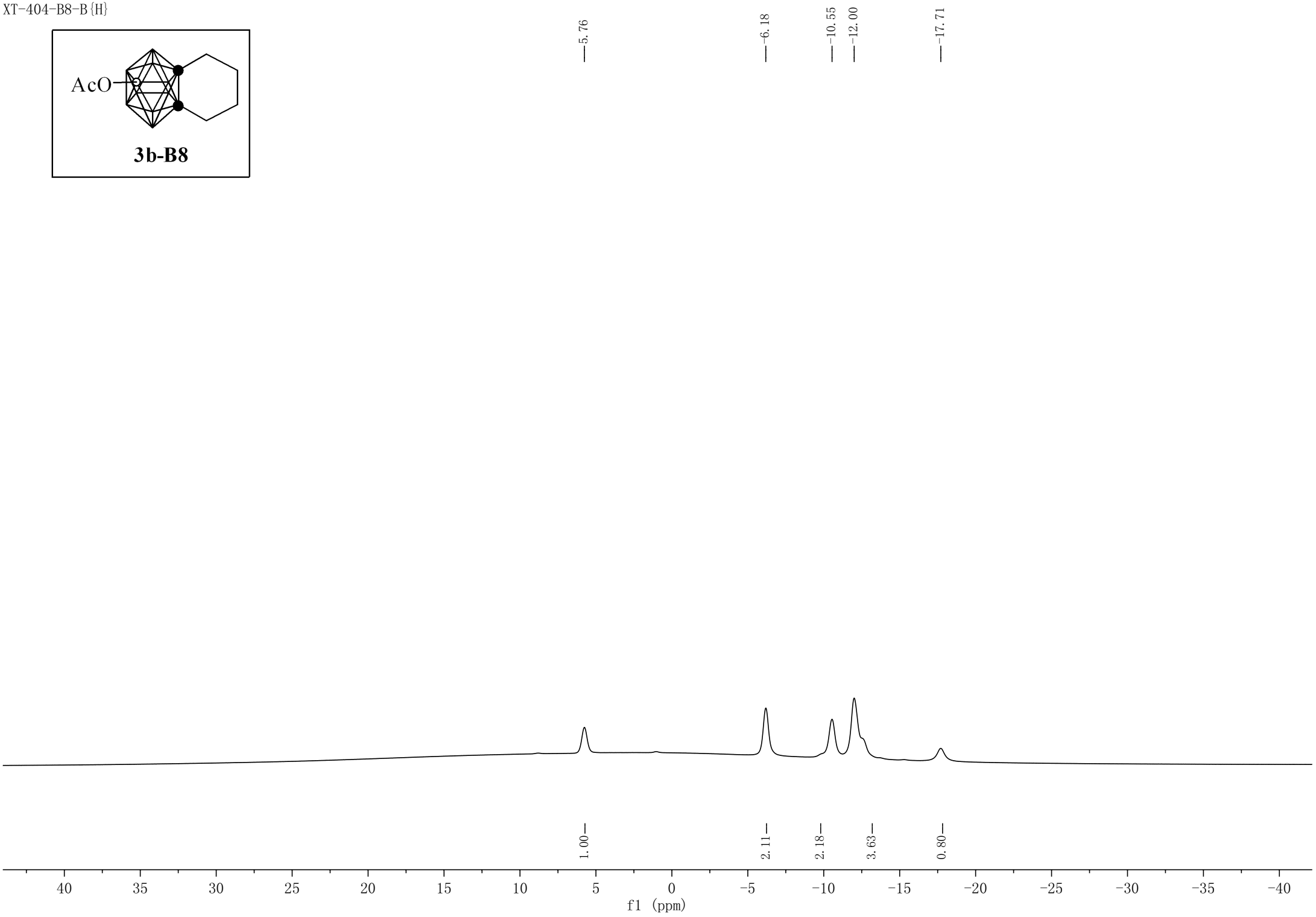
170.51
77.21
77.00
76.79
68.19
32.55
22.56
19.56
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

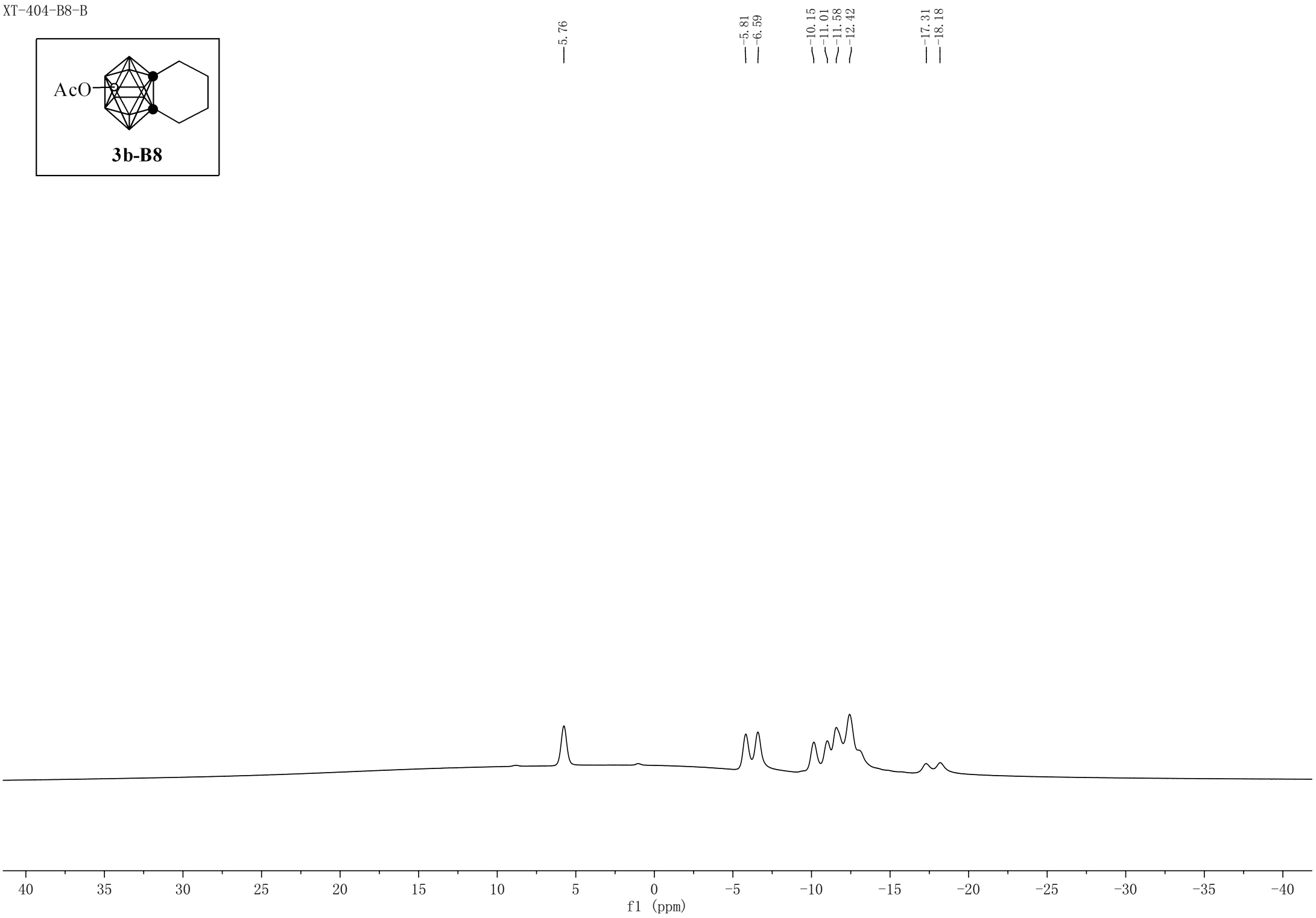
XT-404-B8-B{H}

AcO
3b-B8

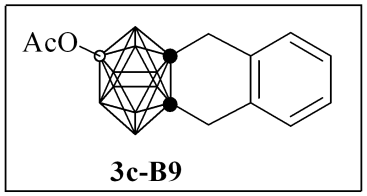

5.76
6.18
10.55
12.00
17.71
1.00
2.11
2.18
3.63
0.80
40
35
30
25
20
15
10
5
0
−5
−10
−15
−20
−25
−30
−35
−40
f1 (ppm)

XT-404-B8-B

AcO
3b-B8


5.76
5.81
6.59
10.15
11.01
11.58
12.42
17.31
18.18
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-457-B9-H



3c-B9

7.26
7.25
7.24
7.24
7.23
7.07

3.77
3.72

2.04

-0.00

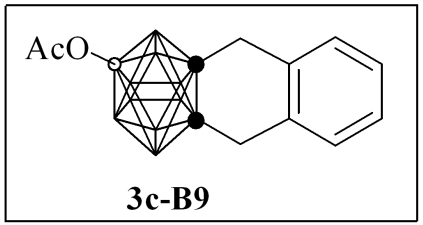
impurity in petroleum ether

2.00
2.18
2.11
2.12
2.91

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-457-B9-C

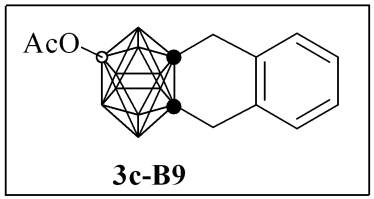


3c-B9

170.12
129.21
128.81
128.66
127.71
127.67
77.21
77.00
76.79
66.82
60.26
37.84
35.72
22.63
-0.02

f1 (ppm)

XT-457-B9-B{H}



3c-B9

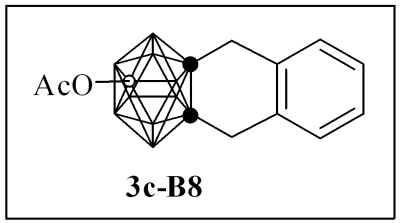
9.31
-5.72
-10.34
-11.85
-12.59

1.00
1.76
3.79
1.63
1.22

40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 −25 −30 −35 −40

f1 (ppm)

XT-457-B8-H



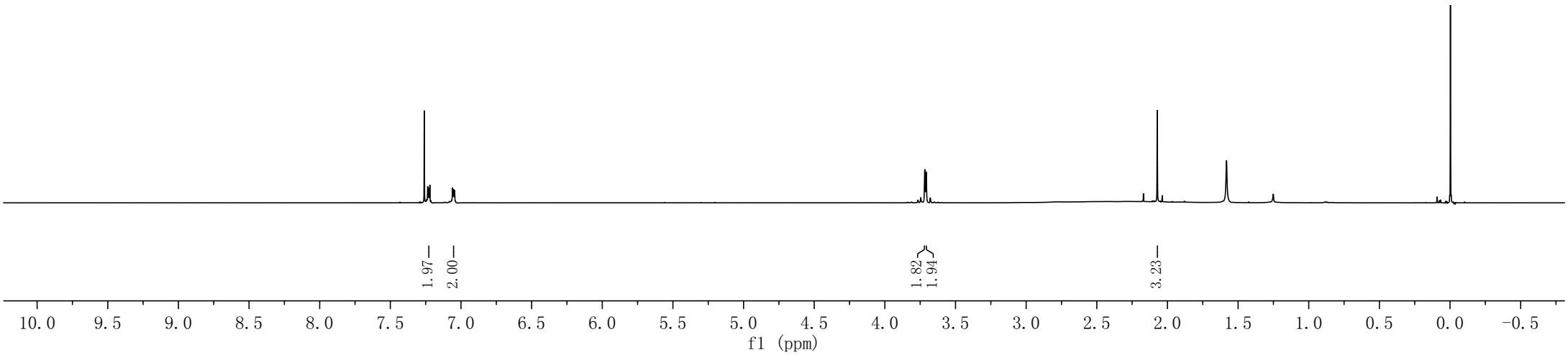
7.26
7.24
7.23
7.23
7.22
7.06
7.05
7.05
7.05

3.72
3.71

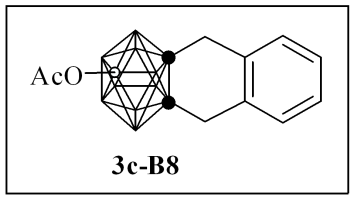
2.07

1.58

0.00



XT-457-B8-C



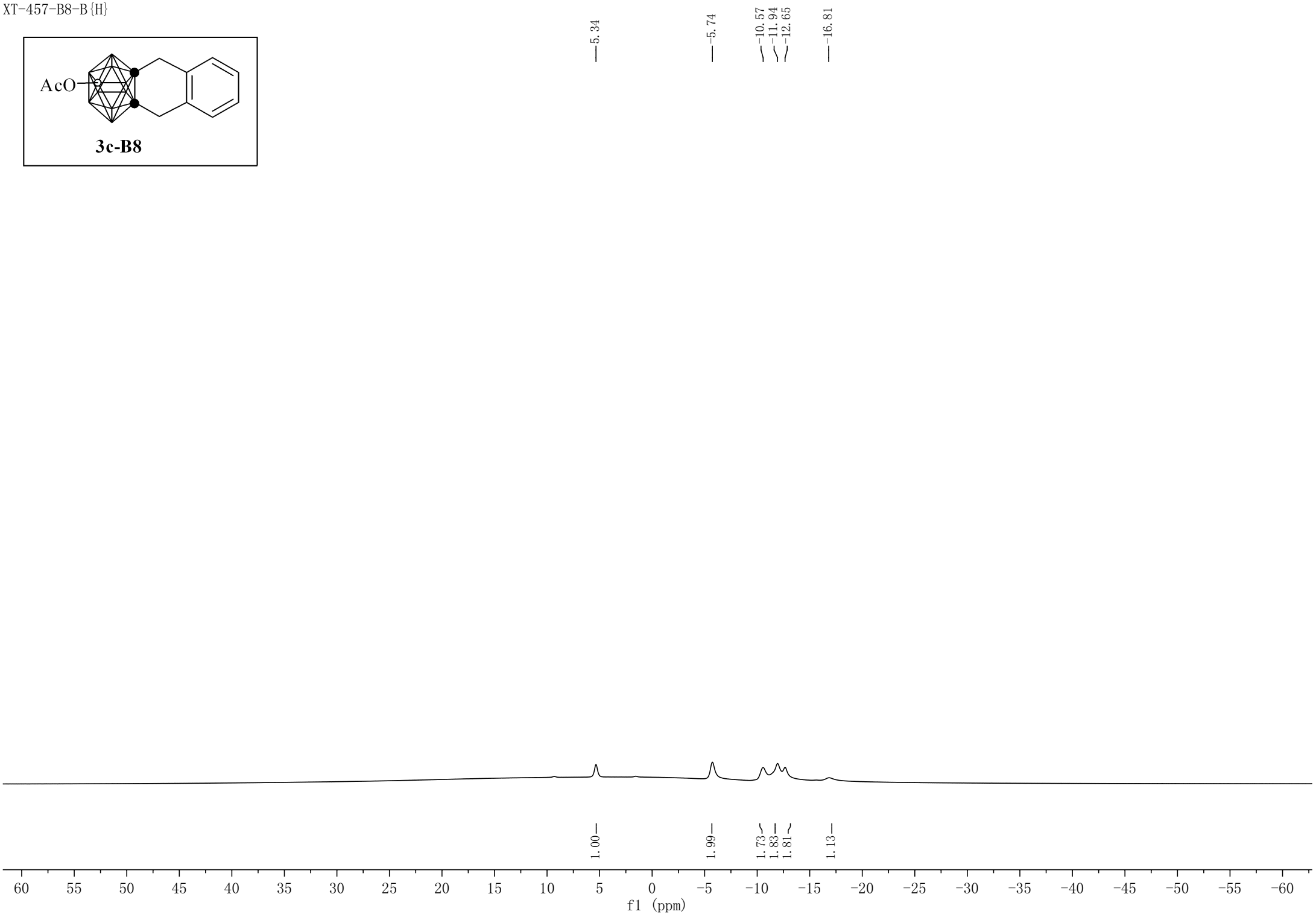
3c-B8

170.44
128.83
128.65
127.64
77.21
77.00
76.79
66.39
37.31
22.51
-0.01

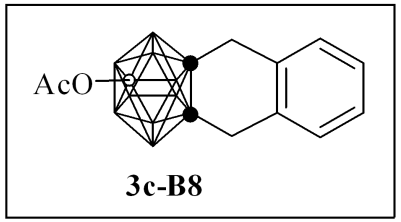
f1 (ppm)

XT-457-B8-B{H}

AcO
3c-B8

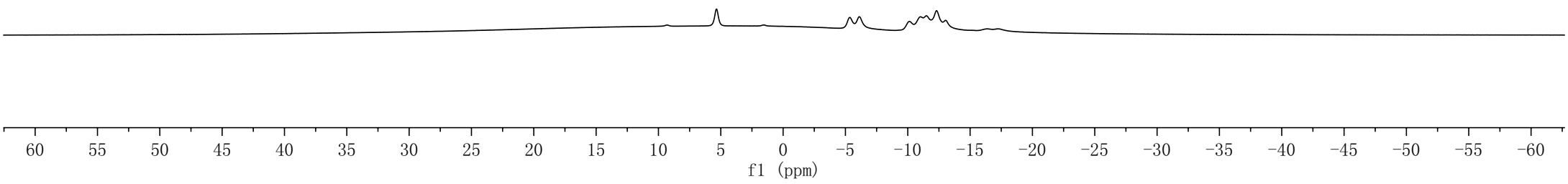

5.34
5.74
10.57
11.94
12.65
16.81
1.00
1.99
1.73
1.83
1.81
1.13
60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

XT-457-B8-B

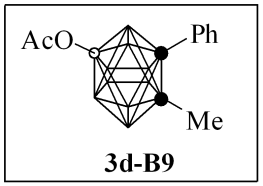


3c-B8

5.35
5.34
6.12
10.13
11.02
11.49
12.31
13.04
16.40
17.26



XT-410-B9-H



3d-B9

7.64
7.62
7.47
7.46
7.44
7.41
7.39
7.38
7.26

2.02
1.72
0.00

unknown impurities

impurity in petroleum ether

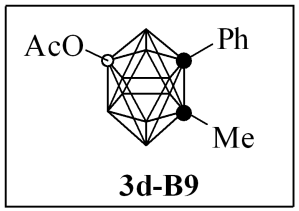
2.00
1.05
2.01

3.11
3.02

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-410-B9-C



3d-B9

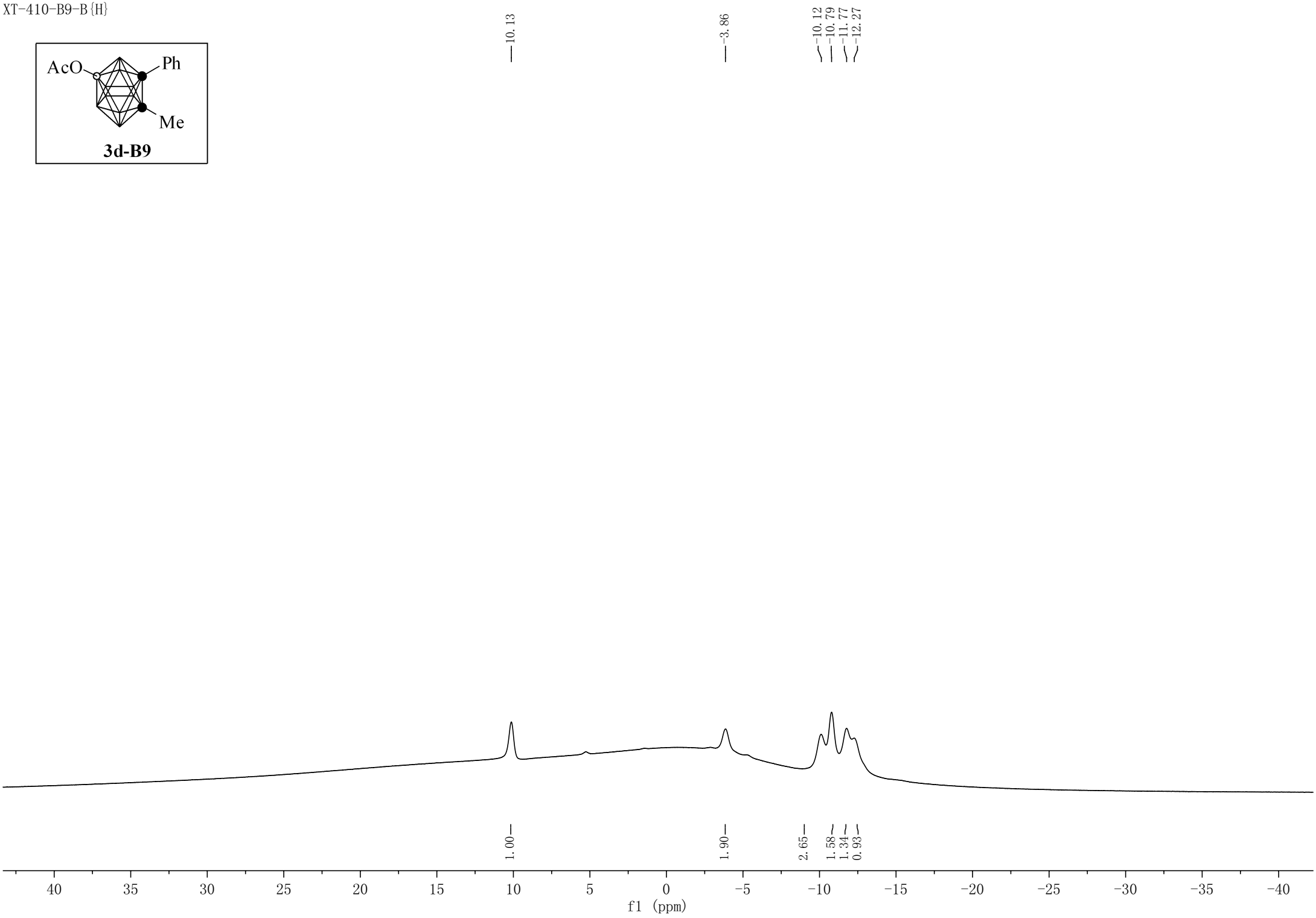
170.04
131.04
130.68
130.59
128.92
77.93
77.21
77.00
76.79
65.65
22.63
20.95
-0.01

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

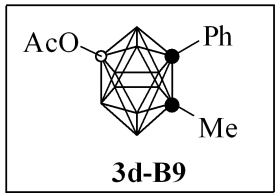
f1 (ppm)

XT-410-B9-B{H}

3d-B9

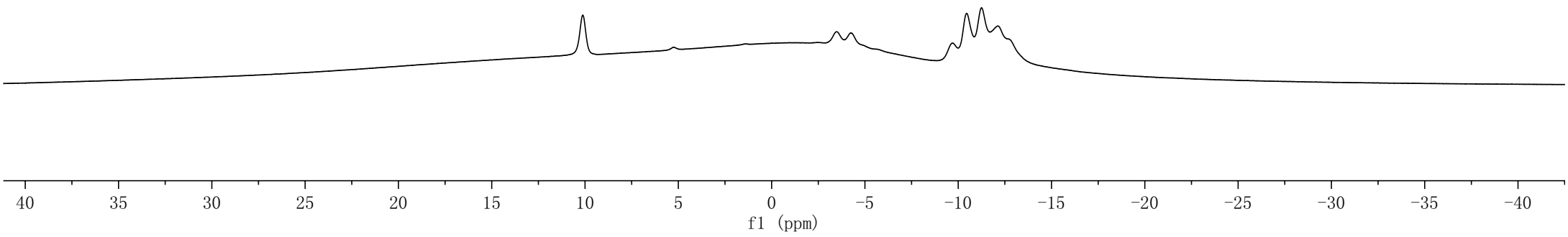


XT-410-B9-B

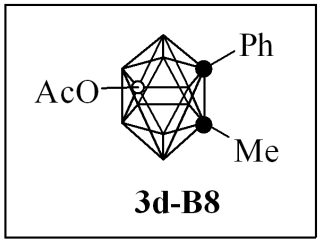


3d-B9

10.12
-3.48
-4.26
-9.68
-10.45
-11.26
-12.12
-12.75



XT-410-B8-H



7.69
7.68
7.47
7.46
7.45
7.41
7.39
7.38
7.26

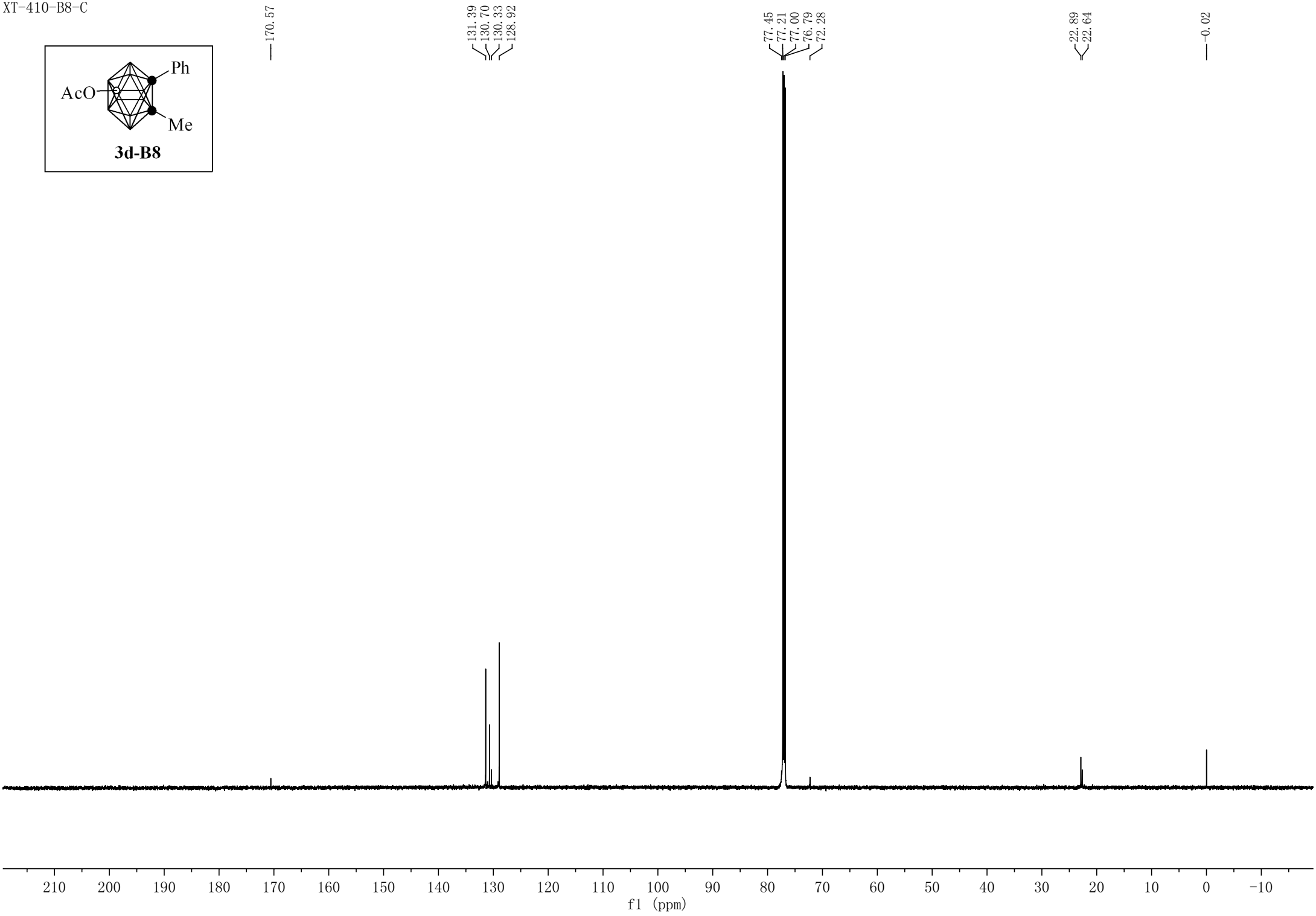
2.12
1.70
0.00

2.00
1.13
2.22
2.92
3.13

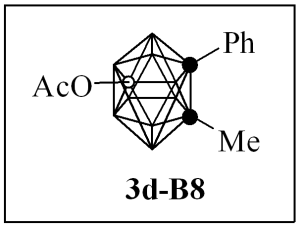
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

XT-410-B8-C

3d-B8



XT-410-B8-B{H}



3d-B8

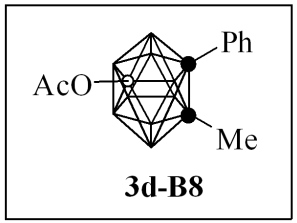
5.01
3.73
4.92
10.20
11.52
13.18
16.20

1.00
1.14
1.04
1.84
2.54
0.90
0.77

40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 −25 −30 −35 −40

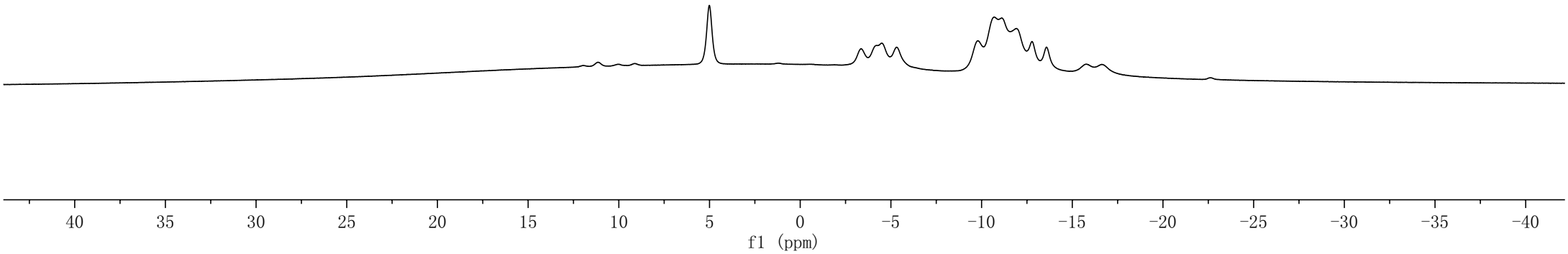
f1 (ppm)

XT-410-B8-B

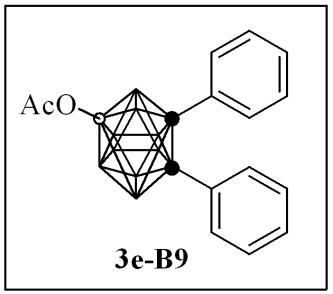


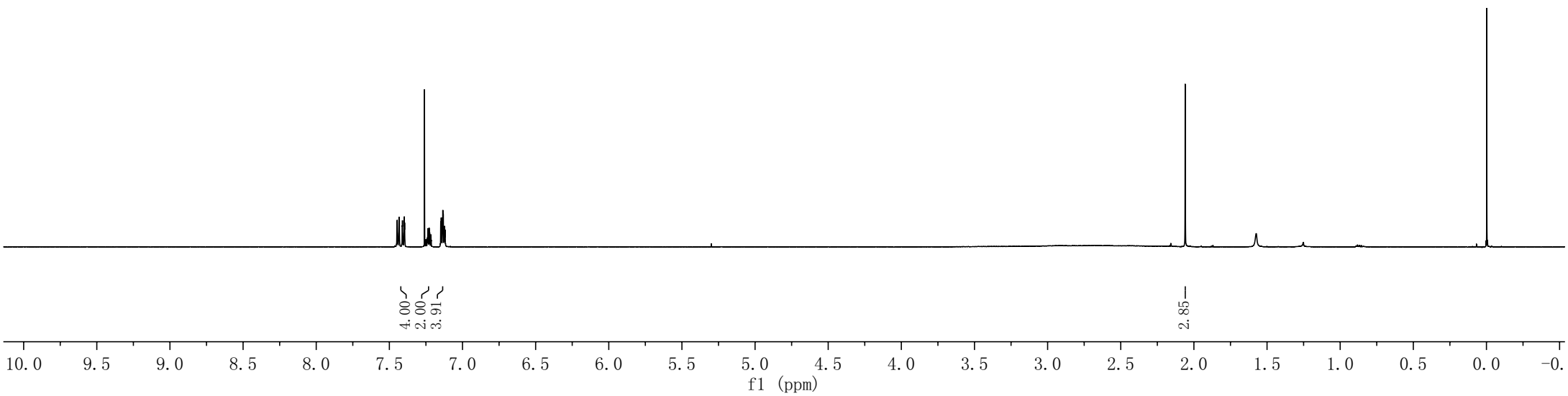
3d-B8

5.01
3.35
4.15
4.49
5.32
9.79
10.69
11.11
11.91
12.78
13.57
15.80
16.61

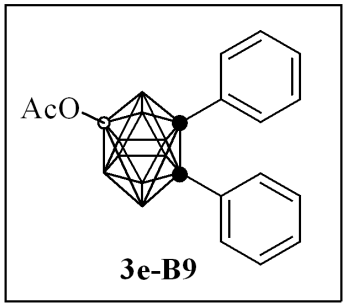


XT-428-B9-H


AcO
3e-B9


7.45
7.45
7.44
7.43
7.43
7.41
7.41
7.40
7.40
7.40
7.26
7.25
7.25
7.24
7.24
7.24
7.23
7.23
7.23
7.22
7.22
7.22
7.22
7.21
7.15
7.15
7.14
7.14
7.13
7.13
7.13
7.12
7.12
2.06
0.00
4.00
2.00
3.91
2.85
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

XT-428-B9-C

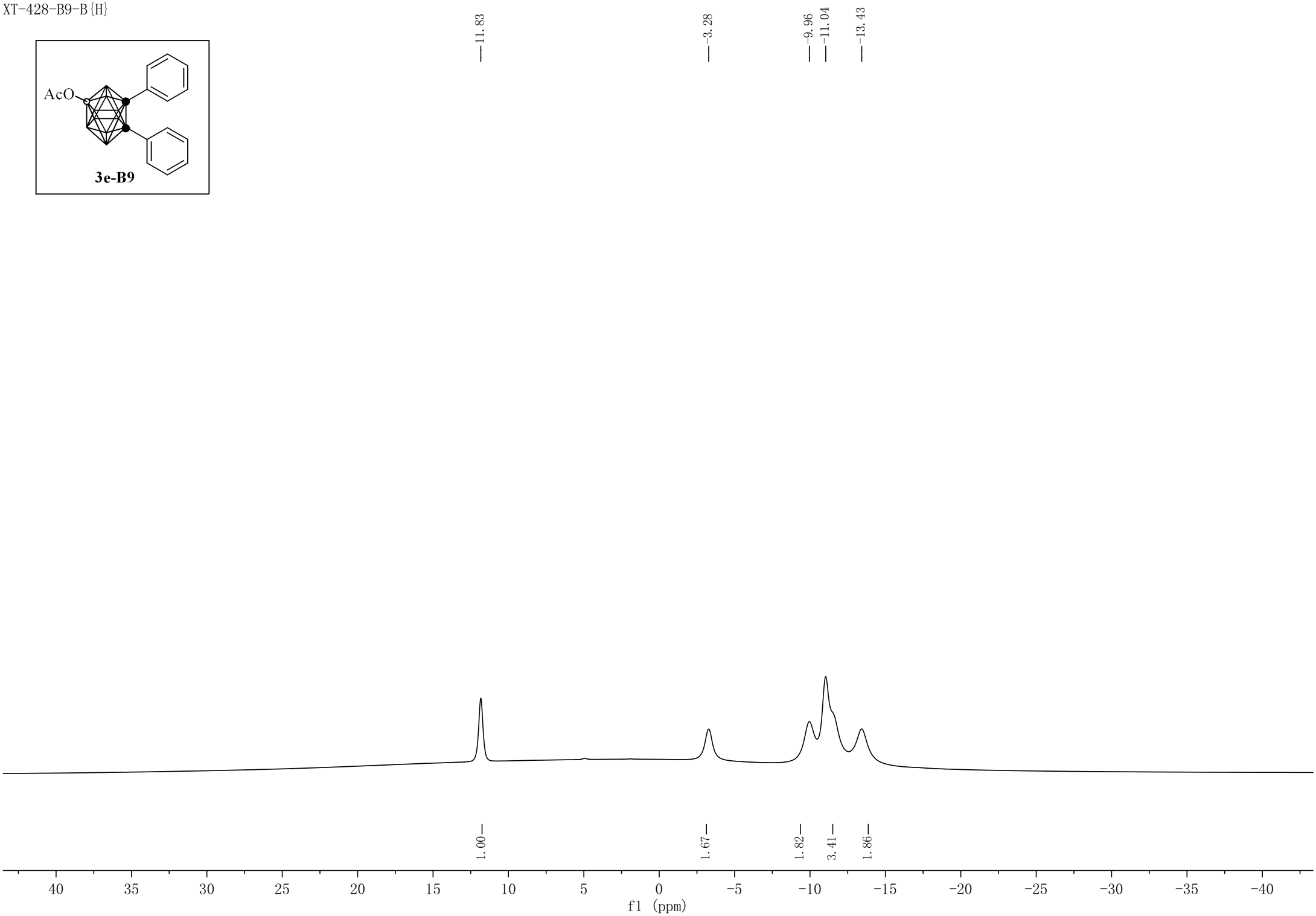


170.03
130.99
130.54
130.27
130.25
129.10
128.30
128.27
80.96
77.21
77.00
76.79
73.70
22.68
-0.02

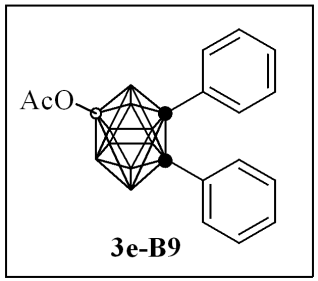
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

XT-428-B9-B{H}

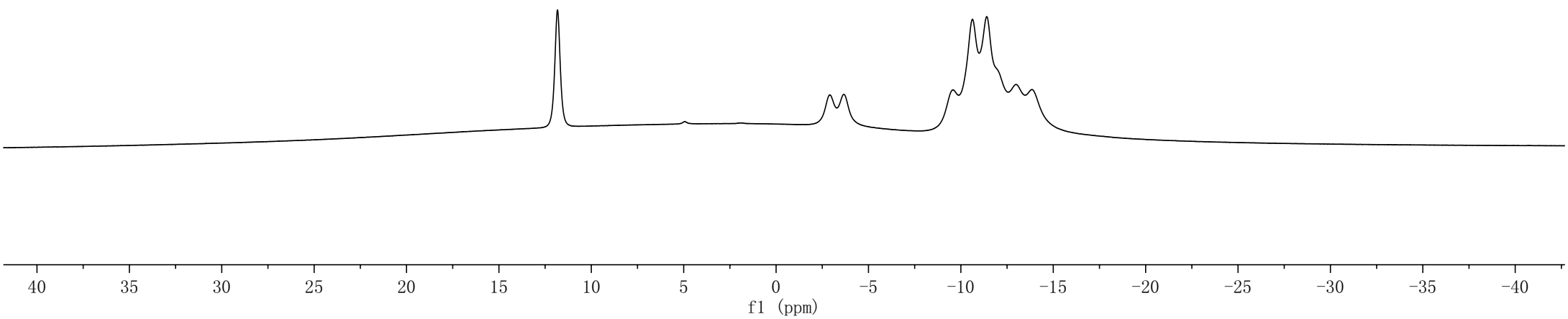
AcO
3e-B9


11.83
3.28
9.96
11.04
13.43
1.00
1.67
1.82
3.41
1.86
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

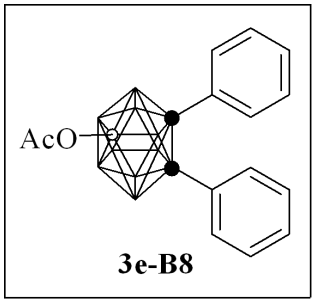
XT-428-B9-B



11.83
2.90
3.67
9.58
10.63
11.40
12.99
13.86



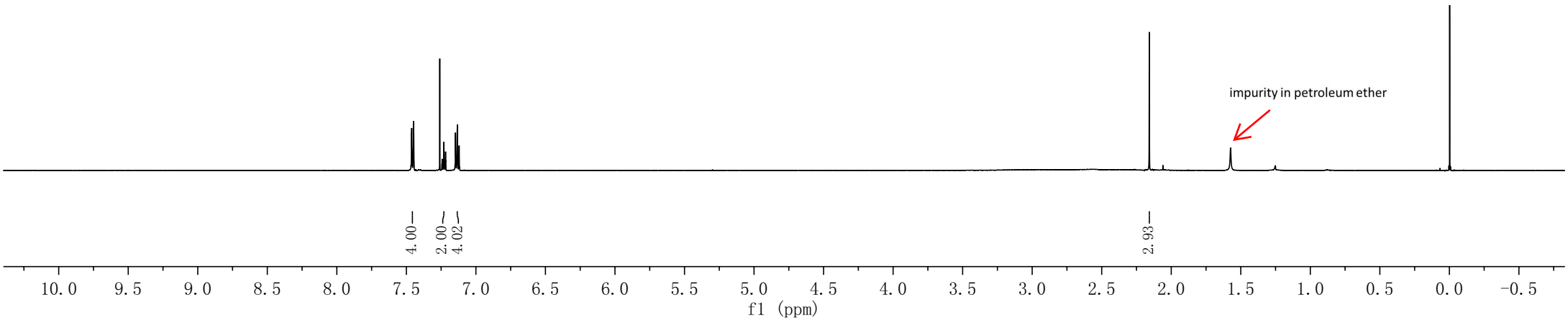
XT-428-B8-H



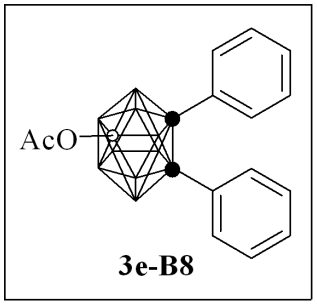
7.46
7.46
7.45
7.45
7.45
7.26
7.24
7.23
7.22
7.22
7.22
7.15
7.14
7.13
7.12

2.16

0.00



XT-428-B8-C

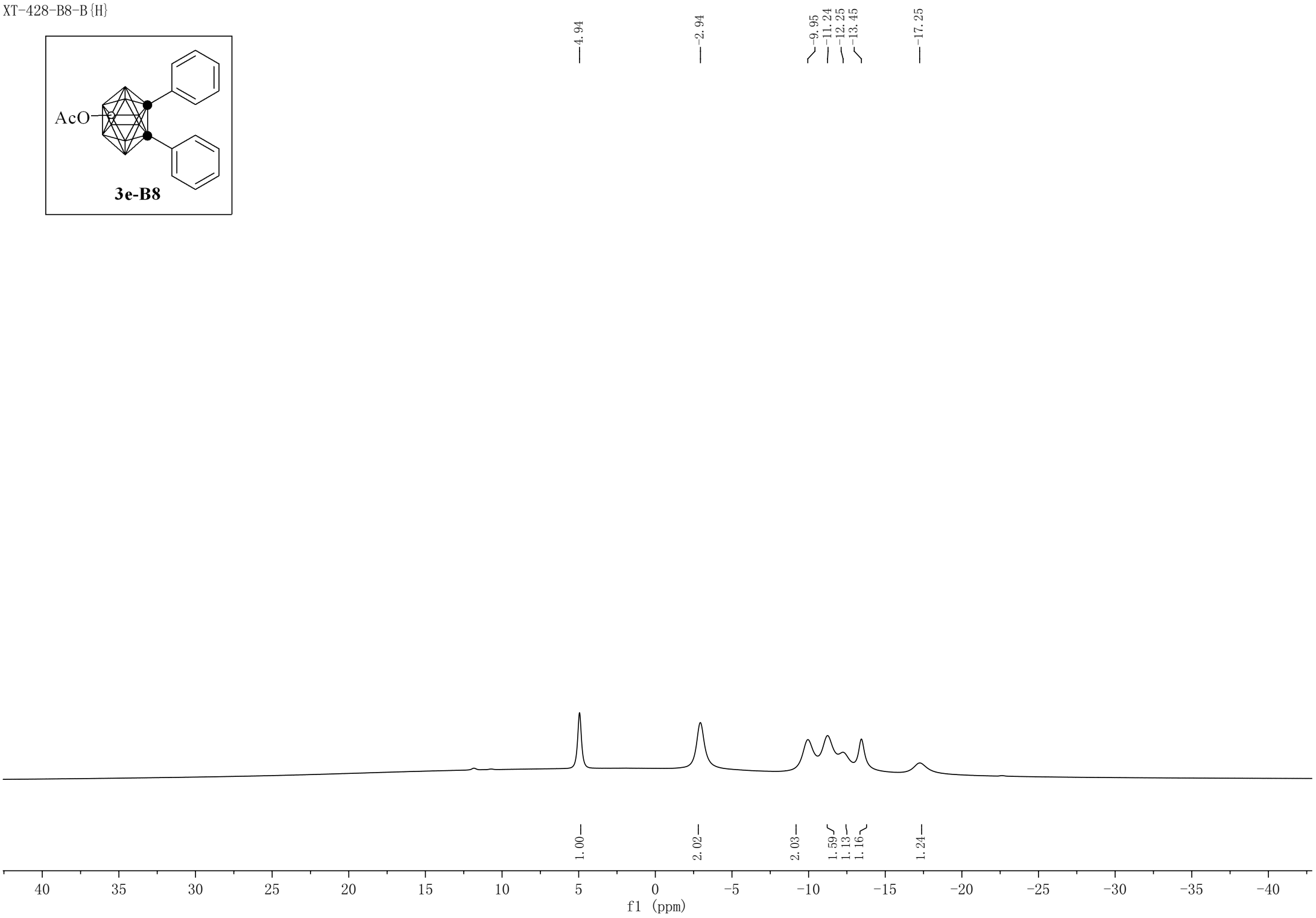


170.61
130.90
130.27
130.07
128.28
80.31
77.21
77.00
76.79
22.73
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

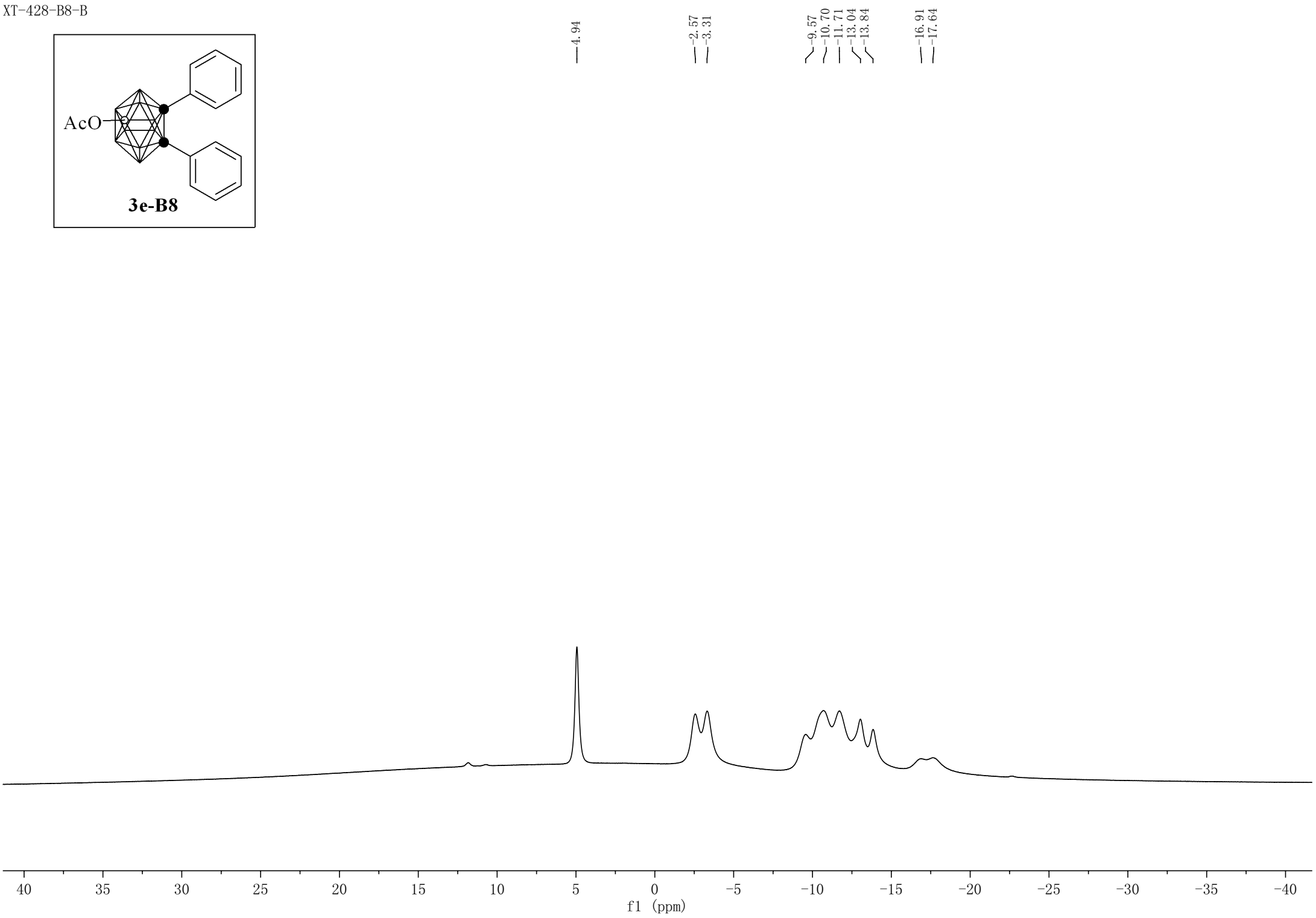
XT-428-B8-B{H}

AcO
3e-B8

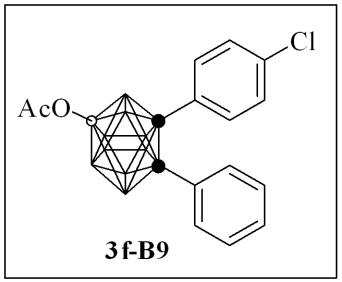

4.94
2.94
9.95
11.24
12.25
13.45
17.25
1.00
2.02
2.03
1.59
1.13
1.16
1.24
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-428-B8-B

AcO
3e-B8


4.94
2.57
3.31
9.57
10.70
11.71
13.04
13.84
16.91
17.64
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

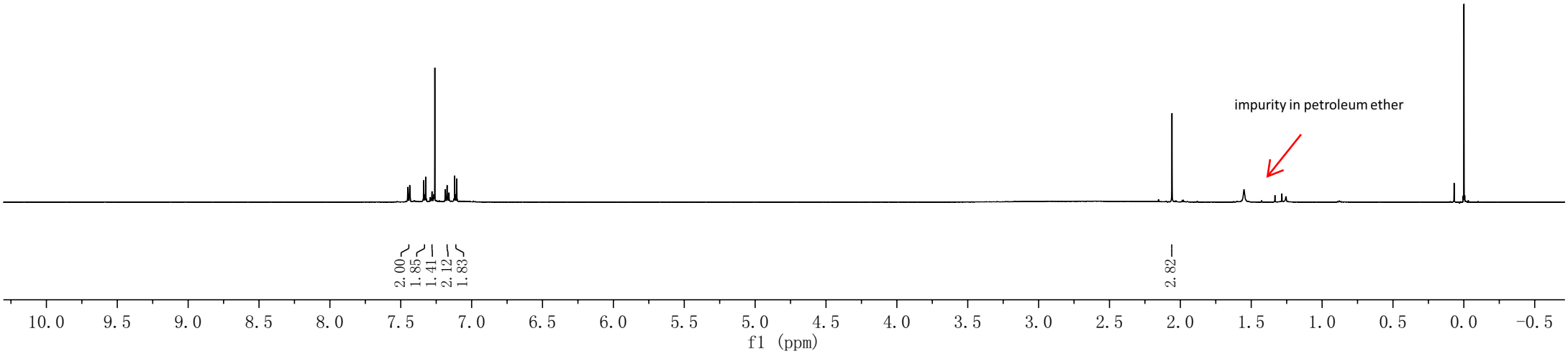
XT-414-B9-H



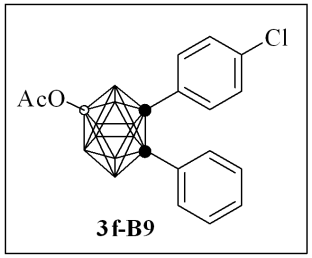
7.45
7.44
7.34
7.32
7.29
7.28
7.27
7.26
7.19
7.17
7.16
7.12
7.11

2.06

0.07
0.00



XT-414-B9-C



3f-B9

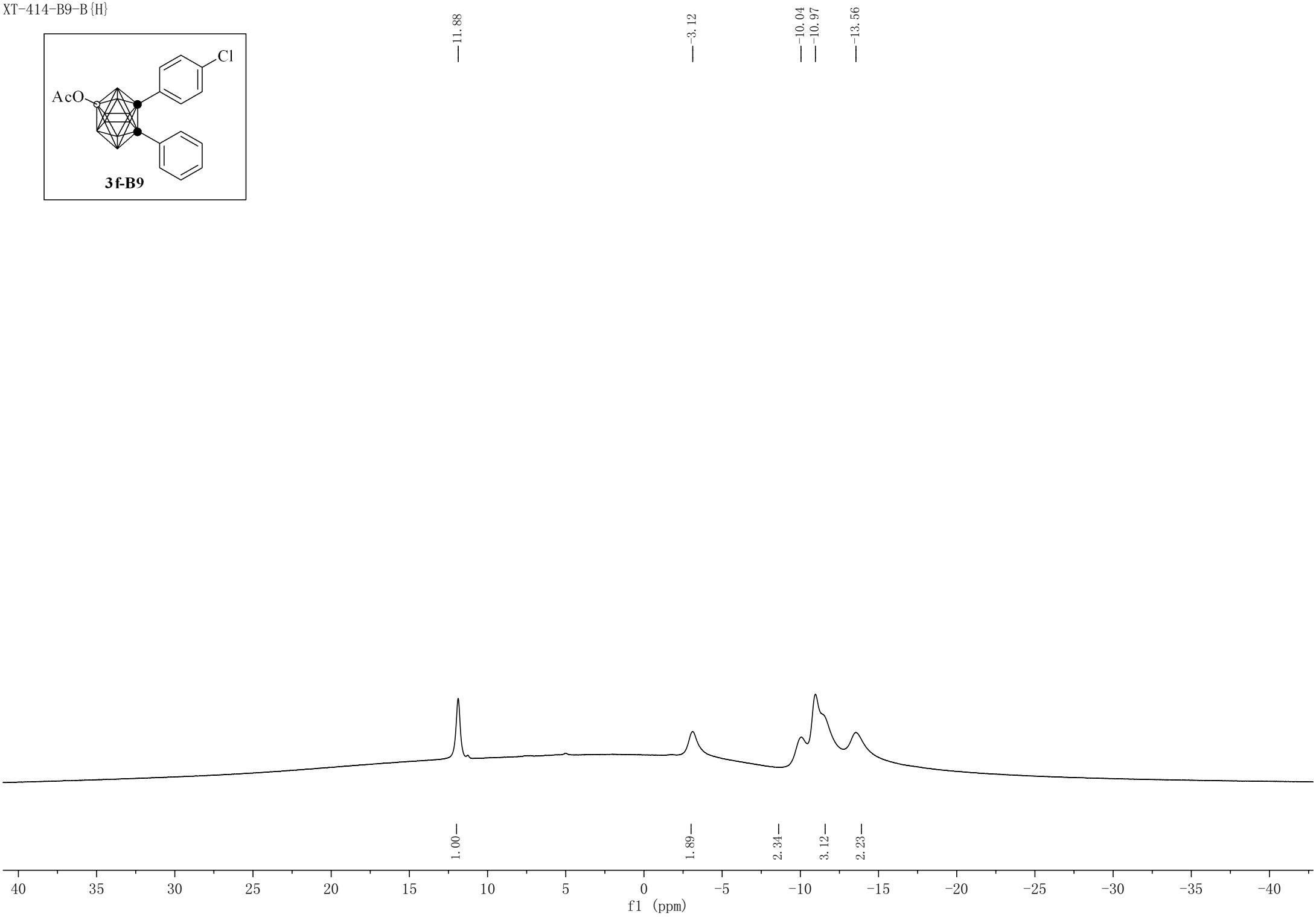
170.00
136.84
131.78
130.99
130.56
128.98
128.89
128.54
128.52
79.84
77.21
77.00
76.79
73.80
22.61
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

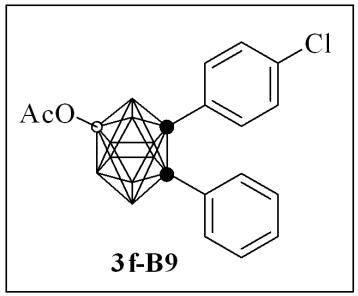
f1 (ppm)

XT-414-B9-B{H}

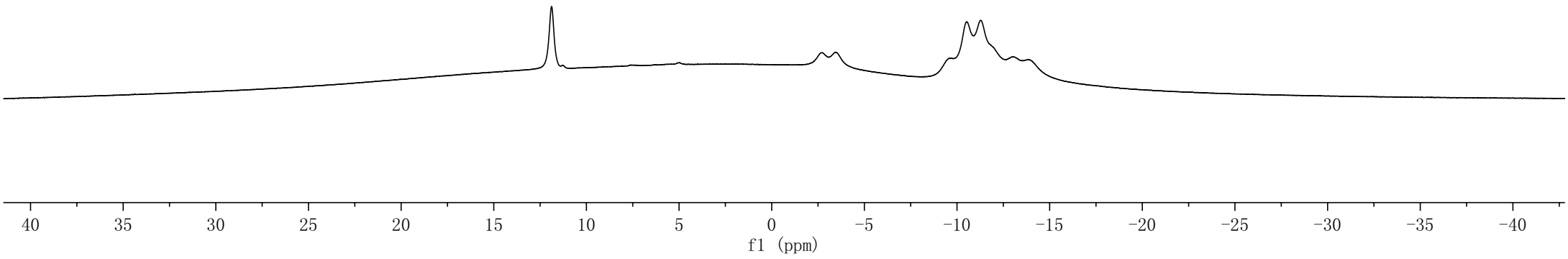
Cl
AcO
3f-B9


11.88
3.12
-10.04
-10.97
-13.56
1.00
1.89
2.34
3.12
2.23
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

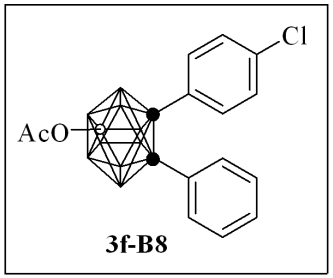
XT-414-B9-B



11.88
2.68
3.45
9.58
10.52
11.28
13.03
13.85



XT-414-B8-H



7.47
7.45
7.39
7.38
7.29
7.27
7.26
7.19
7.17
7.16
7.12
7.11

2.16

0.00

impurity in petroleum ether

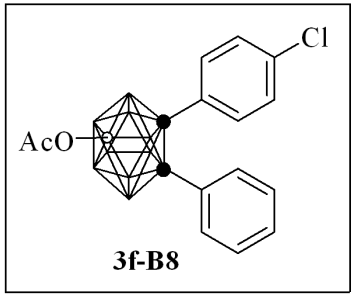
2.00
1.81
1.01
2.27
2.06

2.87

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

f1 (ppm)

XT-414-B8-C



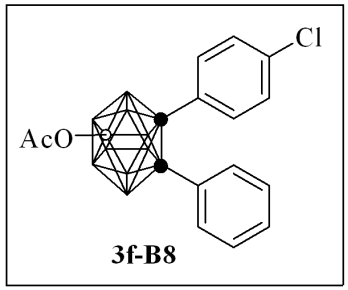
3f-B8

170.59
136.84
132.14
131.78
130.90
130.55
129.87
128.55
128.50
80.43
79.20
77.21
77.00
76.79
22.68
-0.02

impurity in petroleum ether

f1 (ppm)

XT-414-B8-B{H}



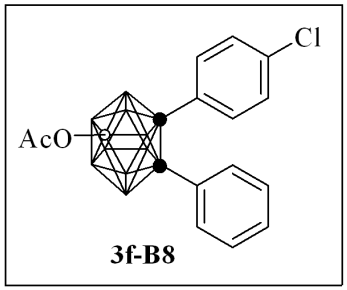
5.01
2.81
-9.91
-11.14
-13.35
-17.31

1.00
1.85
1.80
2.80
1.13
0.92

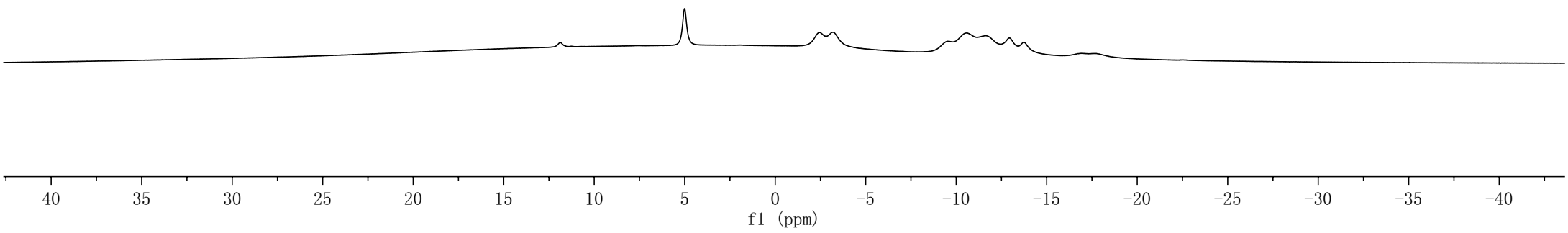
40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40

f1 (ppm)

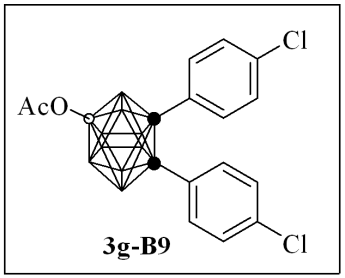
XT-414-B8-B



5.01
2.45
3.19
9.55
10.56
11.63
12.96
13.74
17.00
17.65



XT-455-B9-H



7.38
7.37
7.35
7.33
7.26
7.16
7.15

2.06

0.00

impurity in petroleum ether

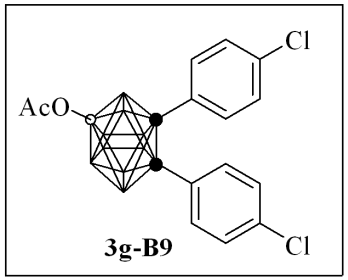
2.00
1.87
3.70

2.99

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-455-B9-C



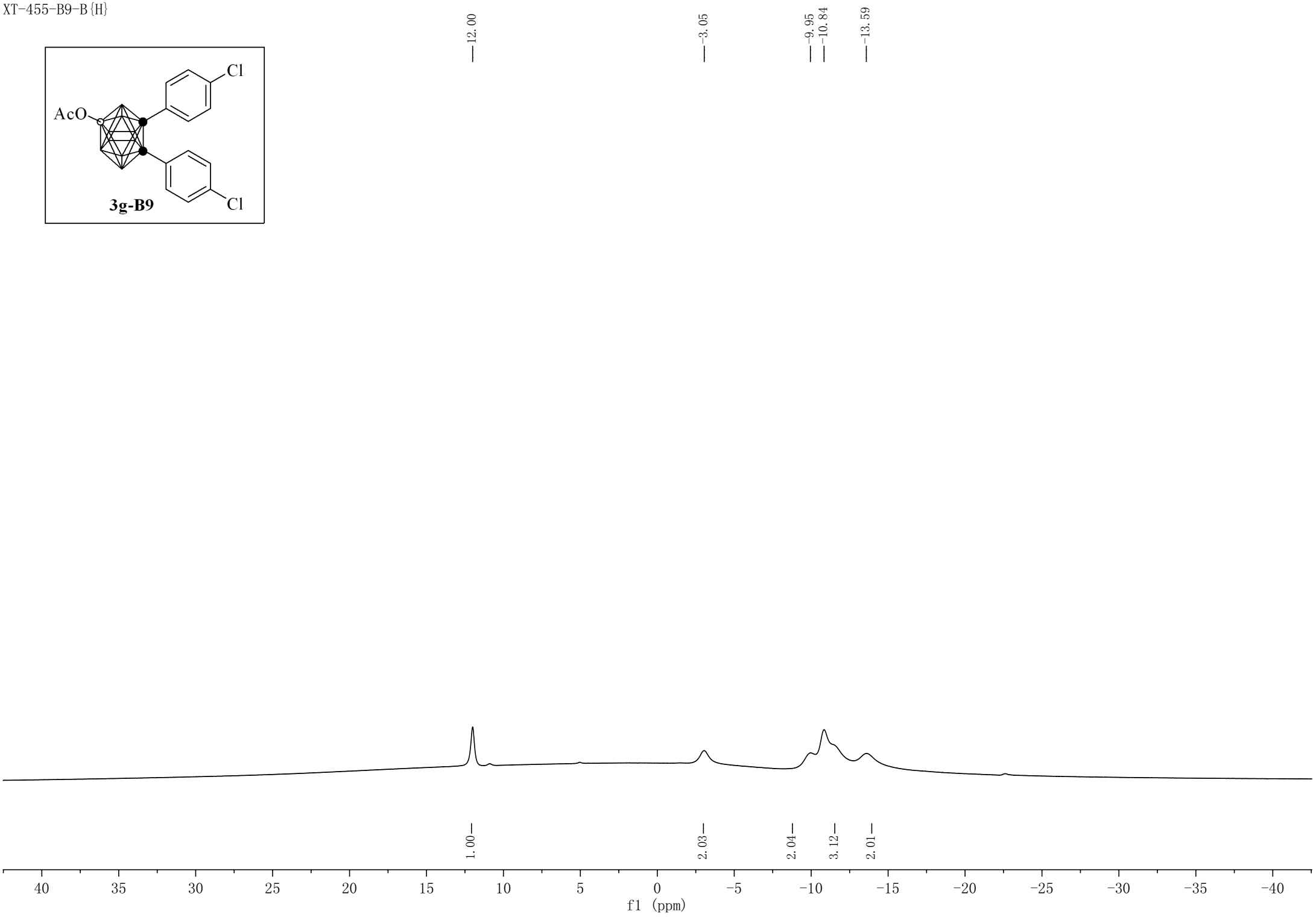
169.99
137.13
132.21
132.12
131.76
128.80
128.78
128.71
127.59
79.97
77.21
76.79
72.76
22.60
-0.02

impurity in petroleum ether

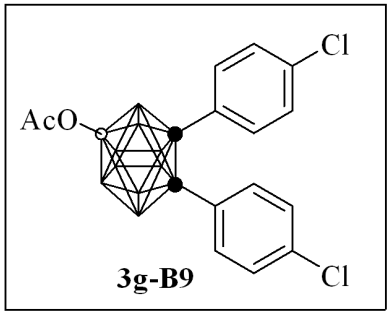
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

XT-455-B9-B{H}

Cl
AcO
3g-B9
Cl

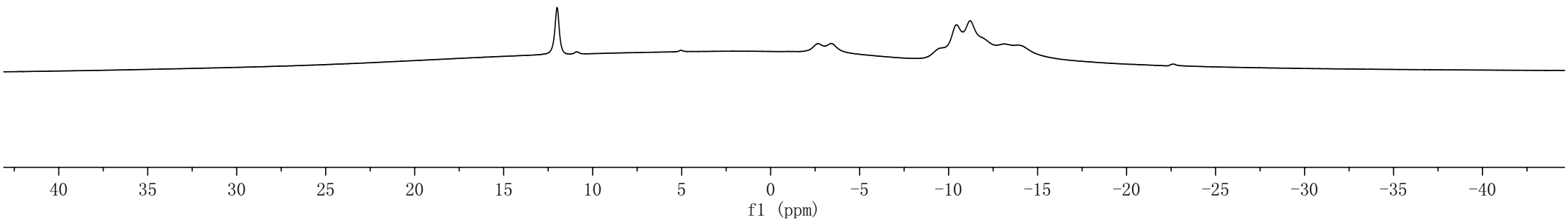

12.00
3.05
9.95
10.84
13.59
1.00
2.03
2.04
3.12
2.01
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-455-B9-B

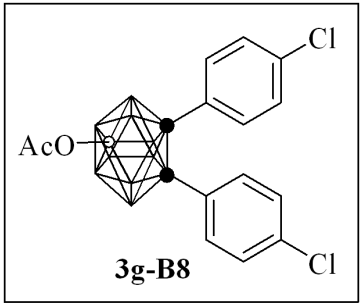


3g-B9

12.00
2.68
3.40
10.44
11.20
13.12
14.04



XT-455-B8-H



3g-B8

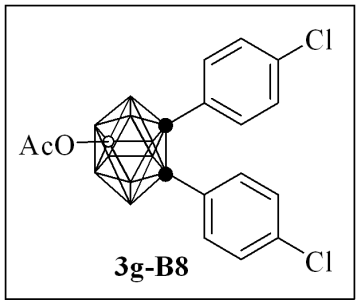
7.40
7.38
7.26
7.16
7.14
2.15
0.00

4.00
3.94
3.25

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-455-B8-C



3g-B8

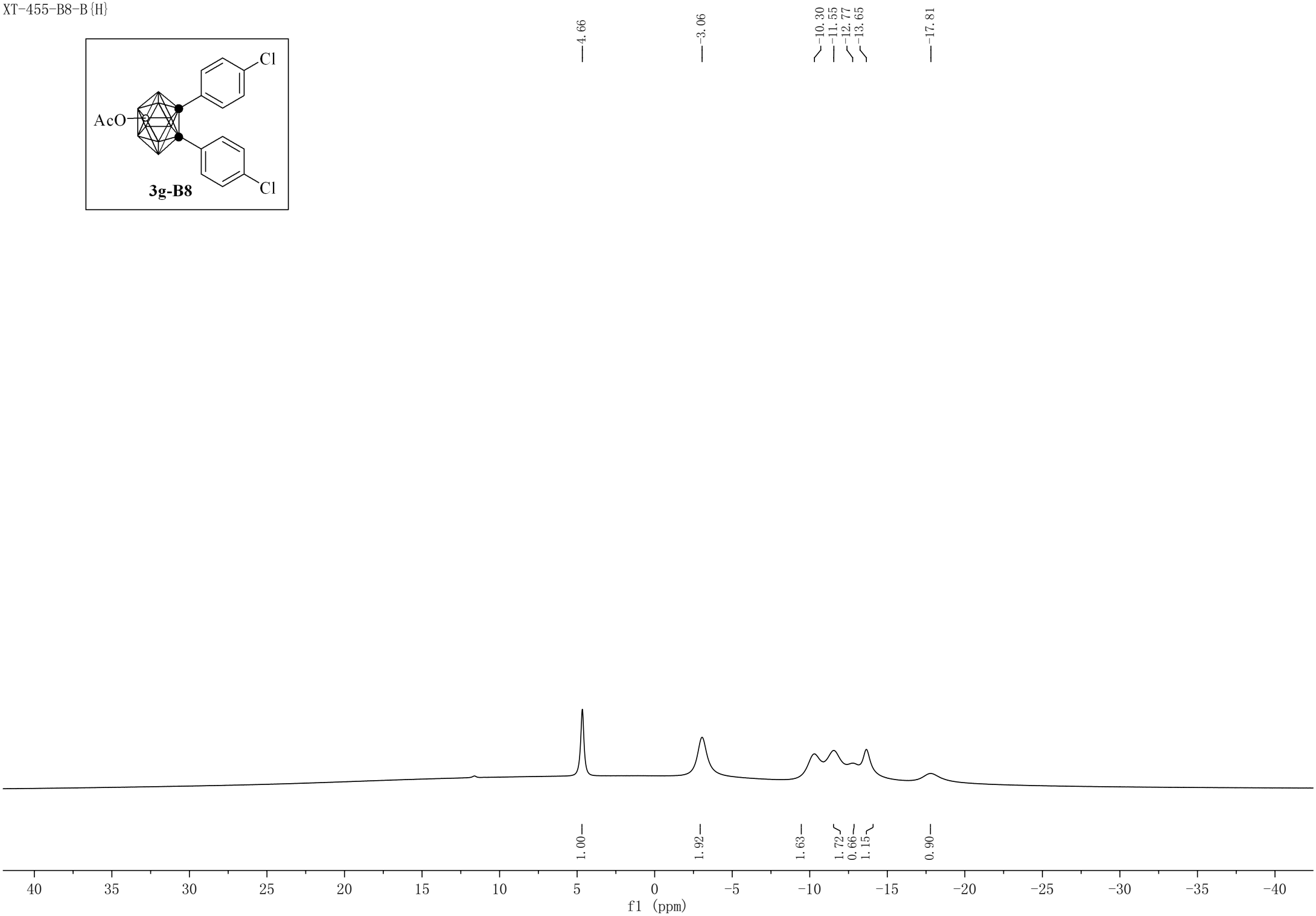
170.60
137.14
132.12
128.77
128.53
79.32
77.21
77.00
76.79
22.67
-0.02

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

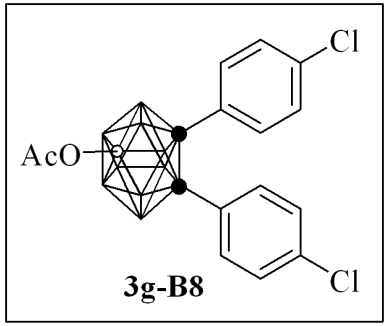
f1 (ppm)

XT-455-B8-B{H}

3g-B8

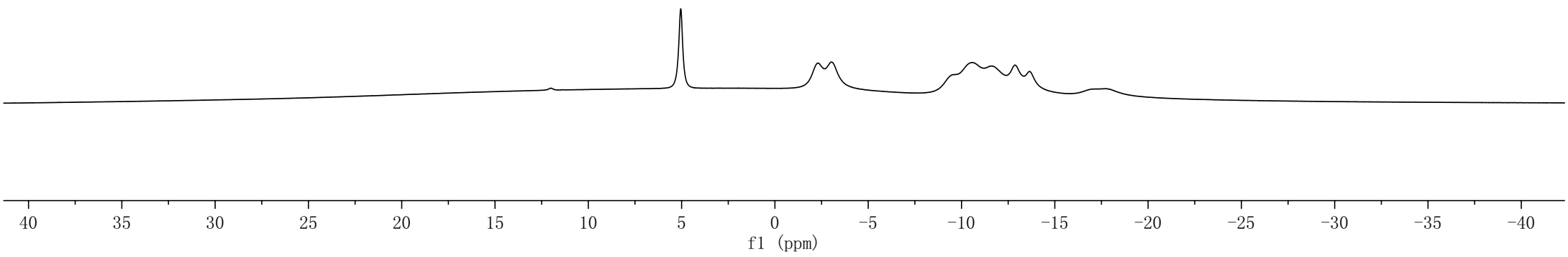


XT-455-B8-B

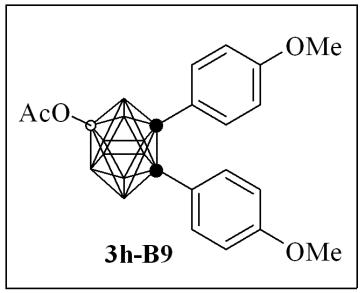


3g-B8

5.05
2.30
3.04
10.58
11.59
12.86
13.65
17.68



XT-491-B9-H



3h-B9

7.37
7.35
7.33
7.31
7.26
6.65
6.64
6.63
6.63
3.72
2.05
0.00

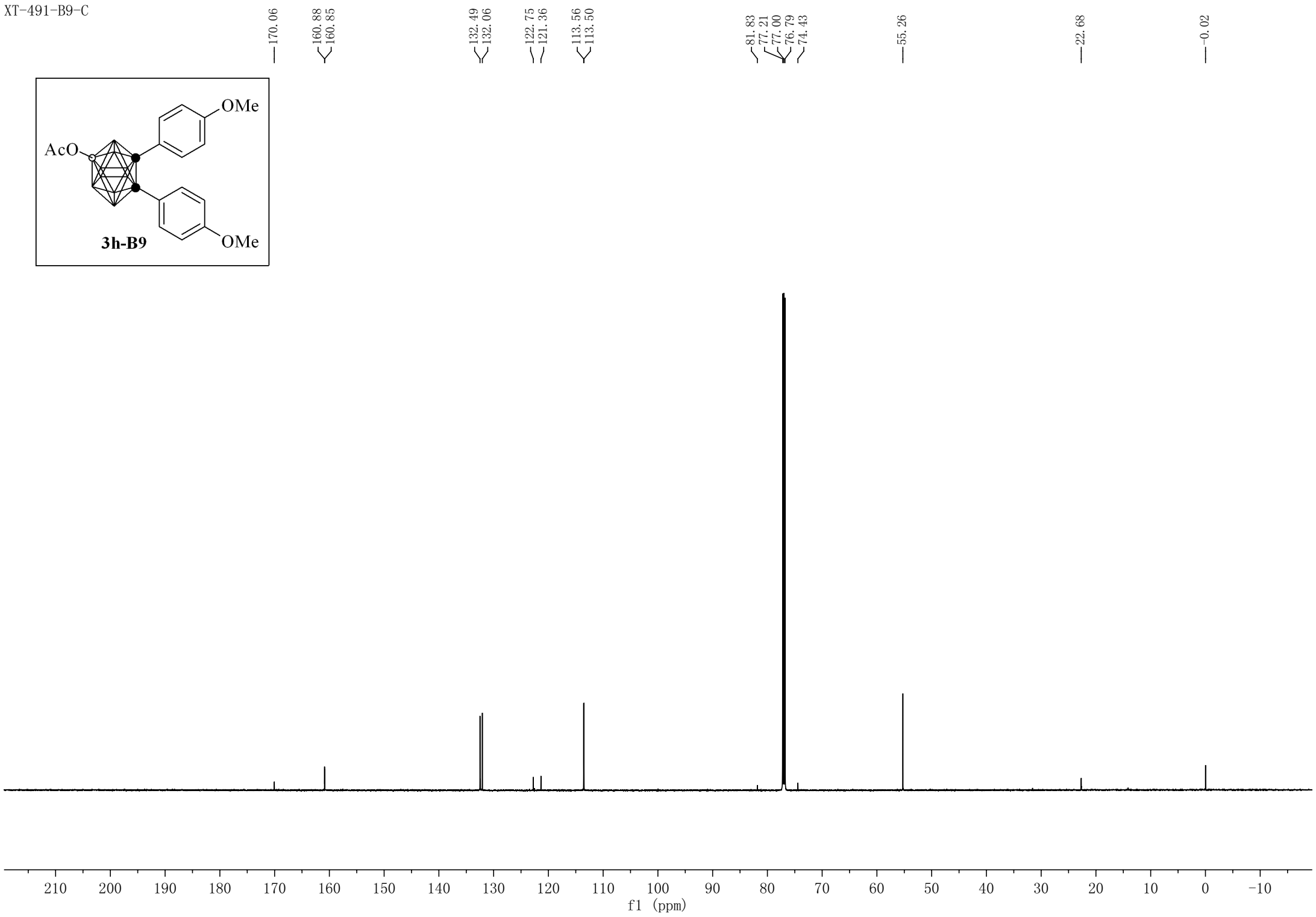
2.00
1.80
2.08
1.89
6.11
2.84

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

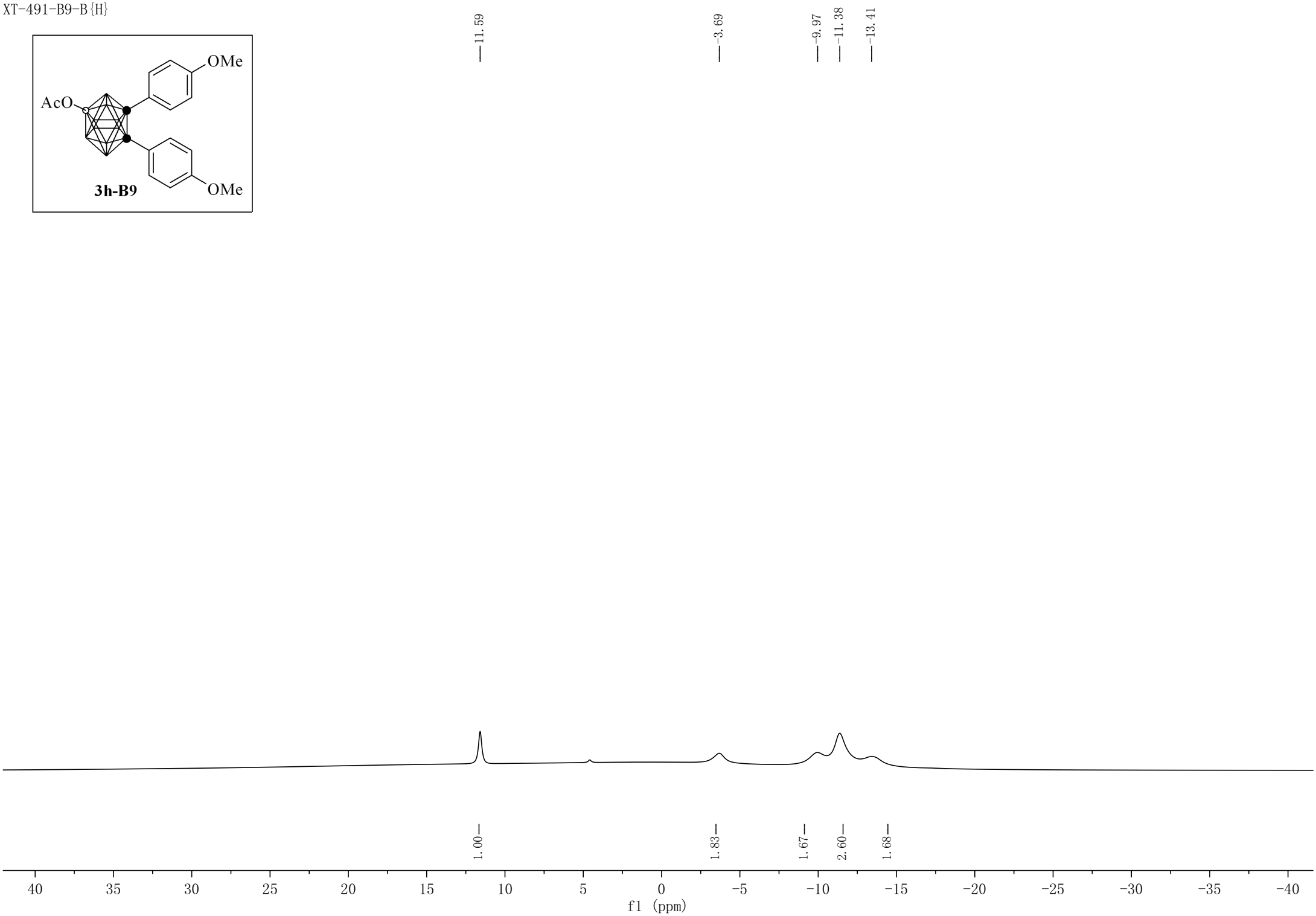
XT-491-B9-C

3h-B9



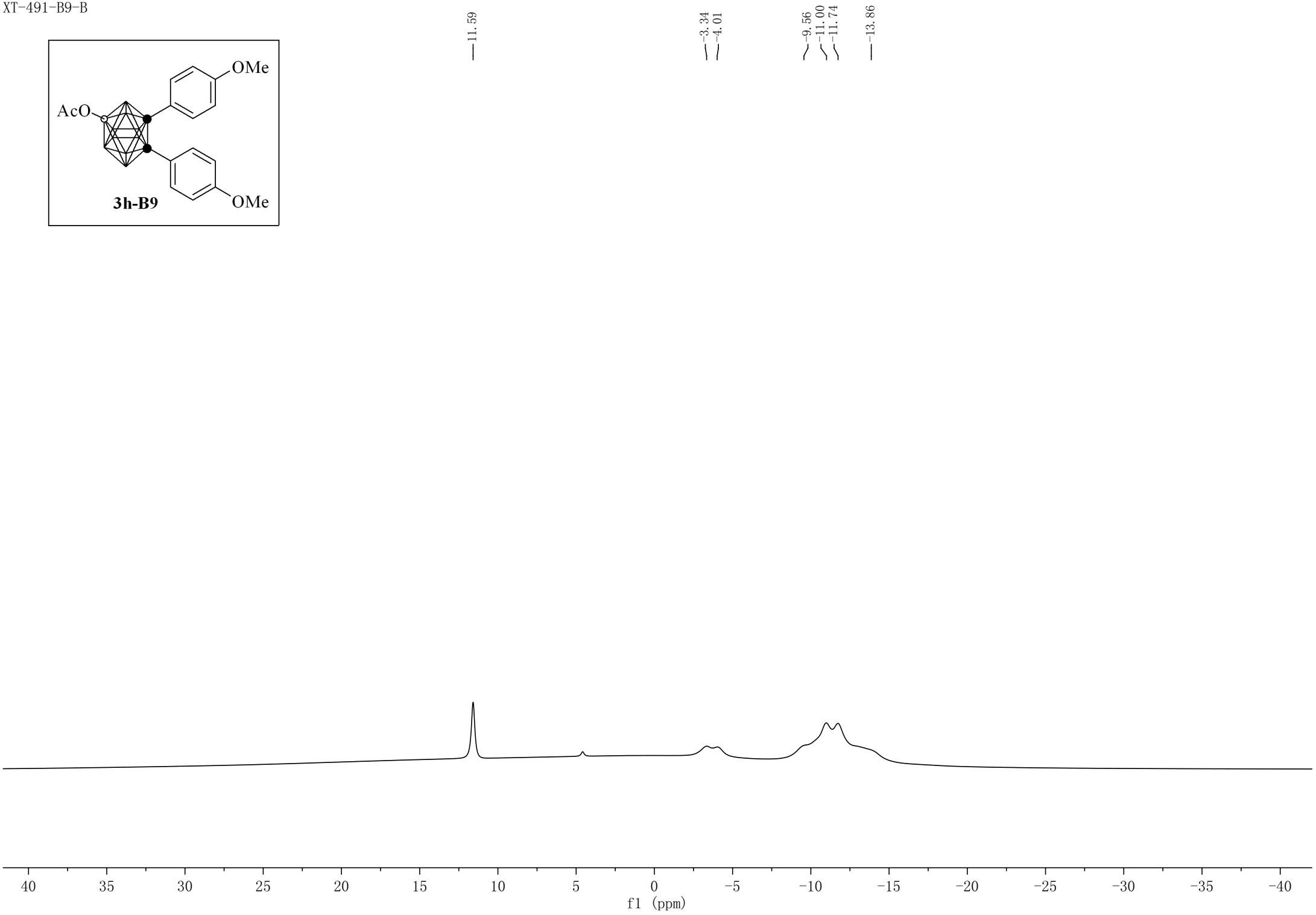
XT-491-B9-B{H}

3h-B9

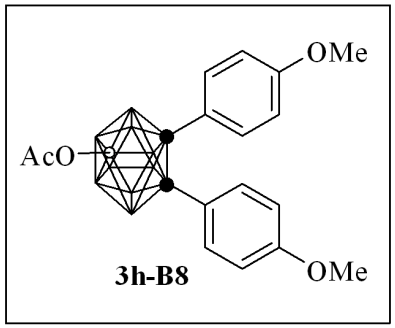


XT-491-B9-B

OMe
AcO
3h-B9
OMe


11.59
3.34
4.01
9.56
11.00
11.74
13.86
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-491-B8-H



3h-B8

7.38
7.37
7.26
6.64
6.63
3.72
2.15
0.00

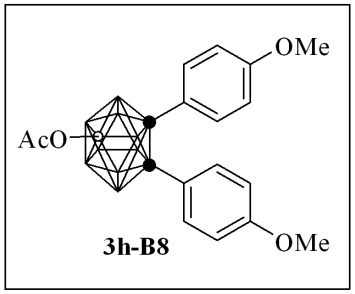
impurity in petroleum ether

4.00
4.04
6.38
2.85

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)

XT-491-B8-C



3h-B8

170.81
160.86
132.43
132.07
122.56
113.51
81.16
77.21
77.00
76.79
55.27
22.69
-0.02

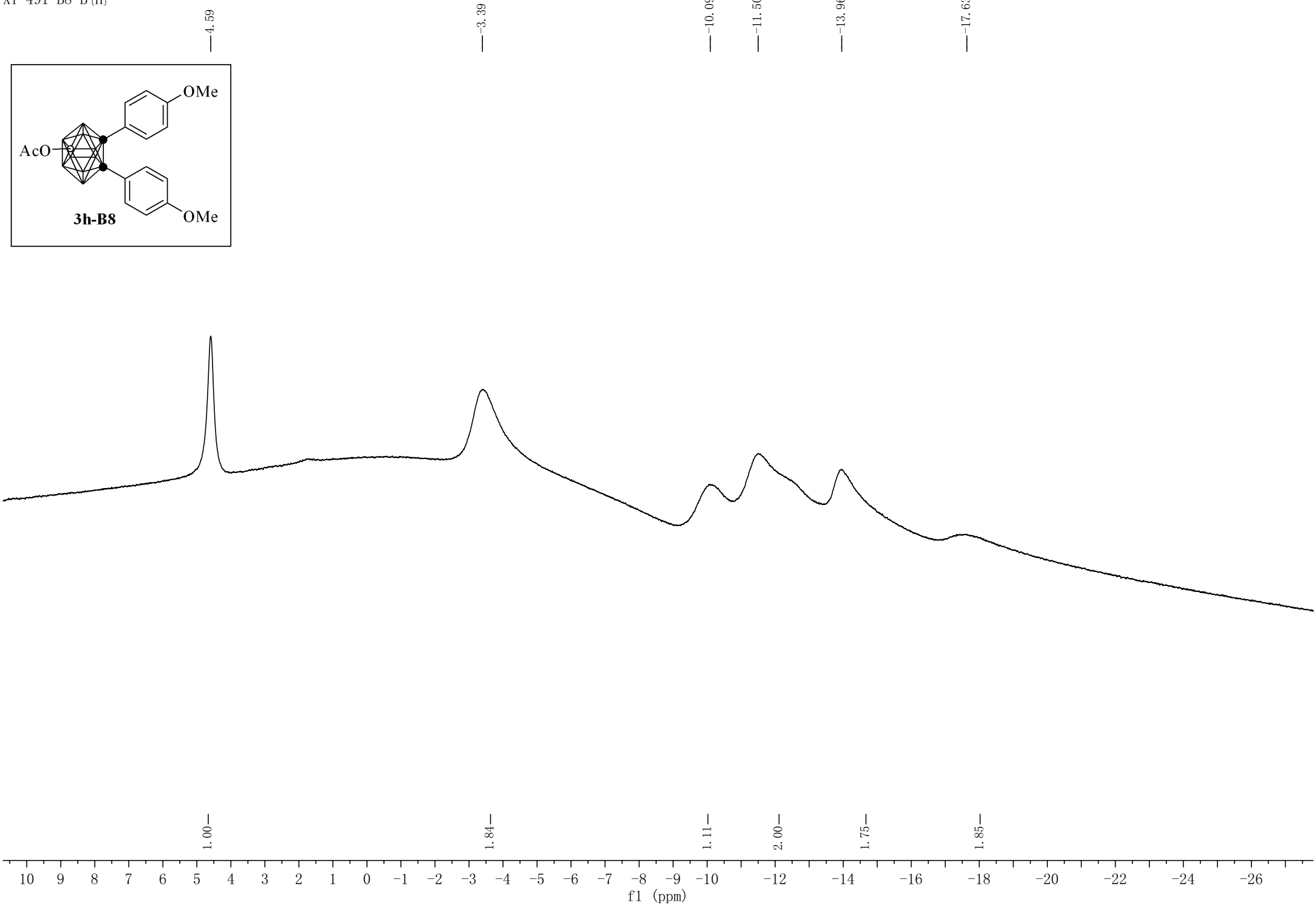
impurity in petroleum ether

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

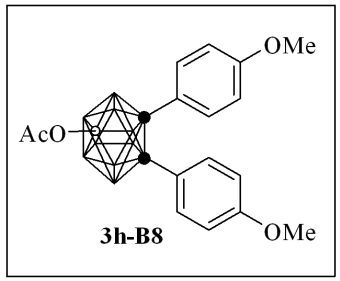
f1 (ppm)

XT-491-B8-B{H}

3h-B8



XT-491-B8-B



3h-B8

4.59
3.39
-10.09
-11.50
-13.96
-17.63

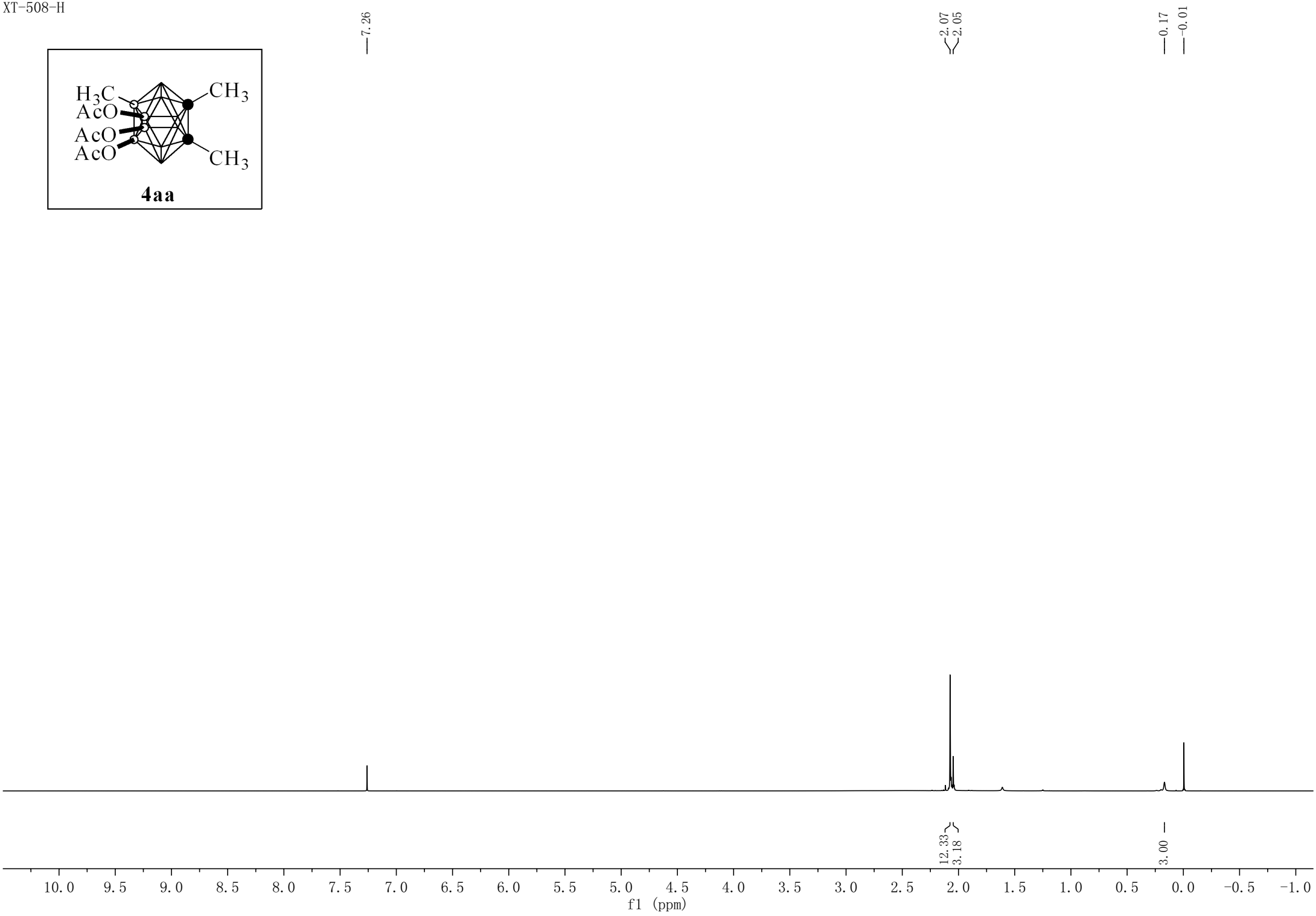
10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5 -6 -7 -8 -9 -10 -12 -14 -16 -18 -20 -22 -24

f1 (ppm)

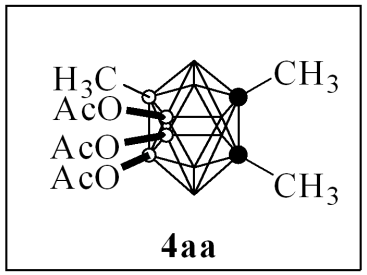
XT-508-H

H3C
AcO
AcO
AcO
CH3
CH3

4aa


7.26
2.07
2.05
0.17
-0.01
12.33
3.18
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

XT-508-C



4aa

170.24
169.55
77.32
77.20
77.00
76.68
53.41
53.30
29.68
22.69
22.31
21.21
-0.02

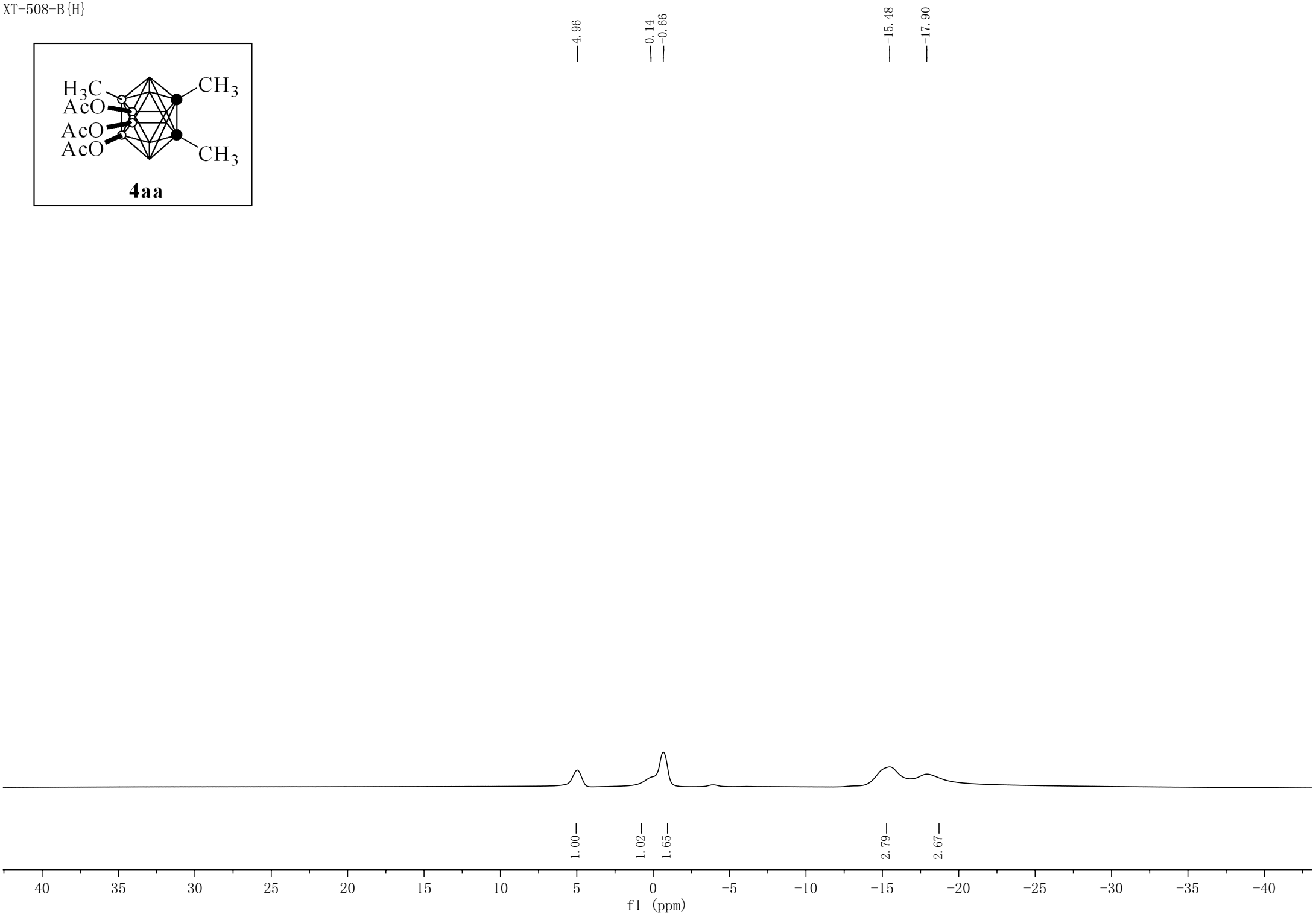
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

XT-508-B{H}

H3C
AcO
AcO
AcO
CH3
CH3

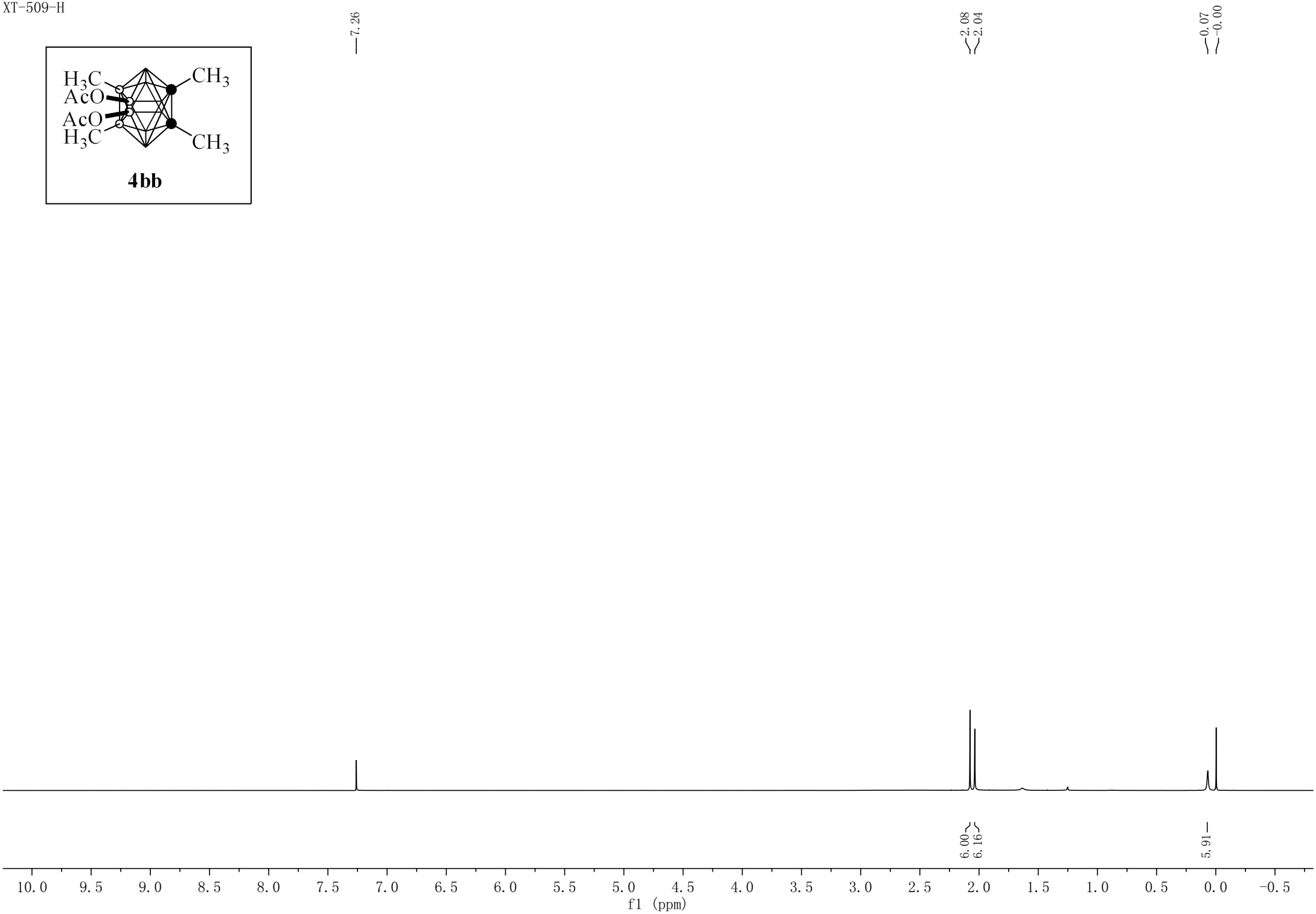
4aa


4.96
0.14
-0.66
-15.48
-17.90
1.00
1.02
1.65
2.79
2.67
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)

XT-509-H

H3C
AcO
AcO
H3C
CH3
CH3

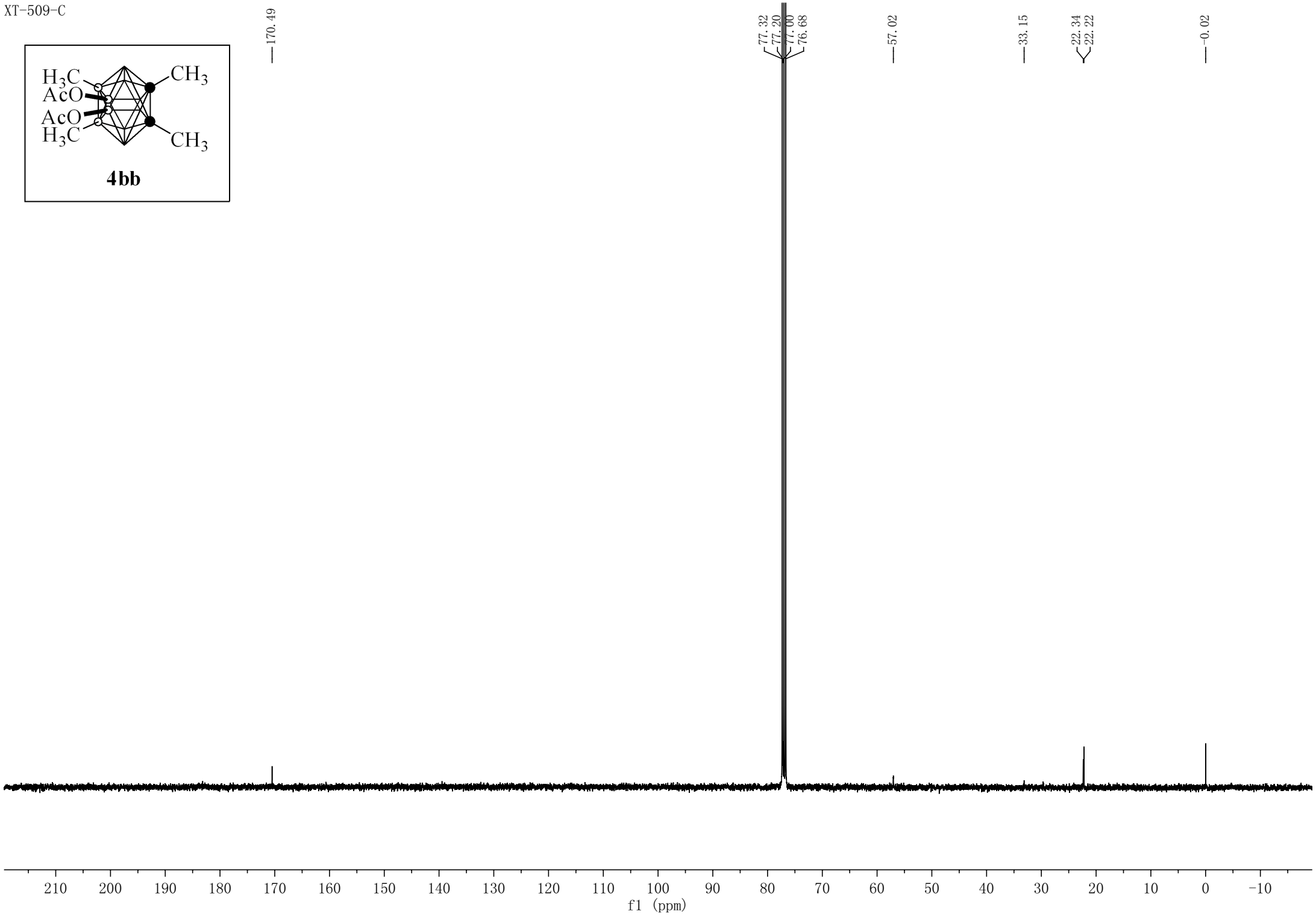
4bb


7.26
2.08
2.04
0.07
0.00
6.00
6.16
5.91
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

XT-509-C

H3C
AcO
AcO
H3C
CH3
CH3

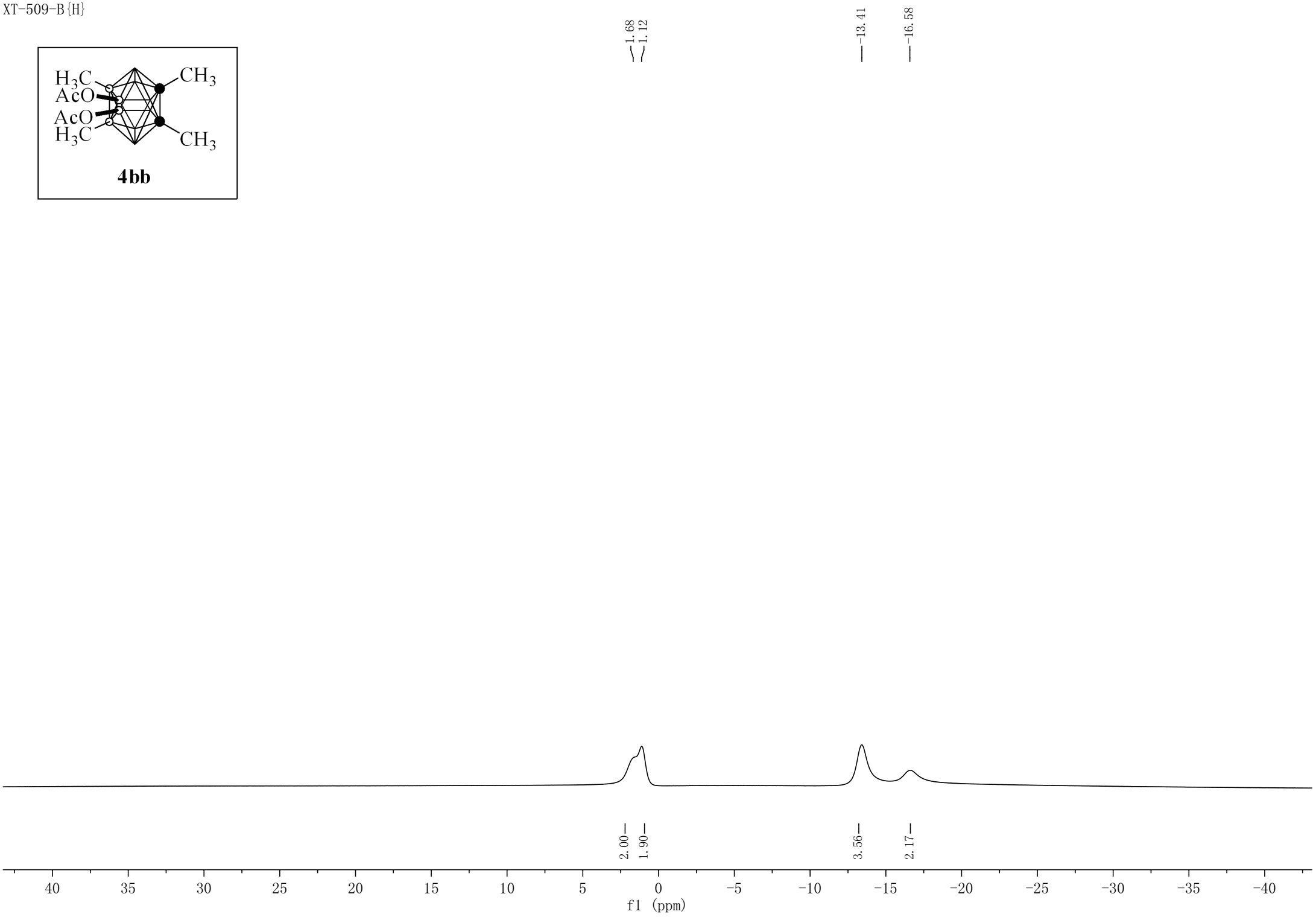
4bb


170.49
77.32
77.20
77.00
76.68
57.02
33.15
22.34
22.22
0.02
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

XT-509-B{H}

H3C
AcO
AcO
H3C
CH3
CH3

4bb


1.68
1.12
-13.41
-16.58
2.00
1.90
3.56
2.17
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
f1 (ppm)