

Supporting Information

Concise synthesis of succinimide-fused densely 1,3-cyclohexadienes via Co-catalyzed [2+2+2] cycloaddition of 1,6-diynes and maleimides

Kaili Cen,^{#a} Ziyi Zhai,^{#a} Yuan Liu,^{#c} Jiahao Wei,^d Mixia Ouyang,^a Guojun He,^a Shuyu Huang,^a Feng Zhao,^{*b} and Jinhui Cai^{*a}

^a College of Chemistry and Chemical Engineering, University of South China, Hengyang 421001, Hunan, China

^b Hunan Provincial Key Laboratory for Synthetic Biology of Traditional Chinese Medicine, School of Pharmaceutical Sciences, Hunan University of Medicine, Huaihua 418000, Hunan, China;

^c Chuanshan College University of South China, Hengyang 421001, Hunan, China

^d School of Pharmaceutical Science, University of South China, Hengyang 421001, Hunan, China

E-mail: jinhuicai@usc.edu.cn; zhaofenghnmu@163.com

[#]These authors contributed equally to this work

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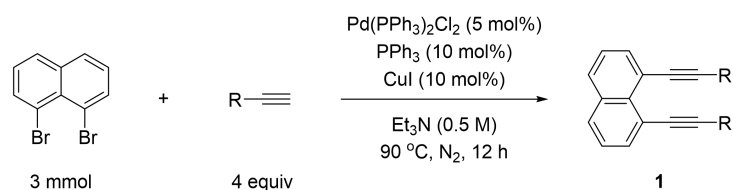
1. General Information

Unless otherwise noted, all experiments were carried out under nitrogen atmosphere. Reactions were monitored by thin-layer chromatography (TLC). TLC was performed using Huanghai 8±0.2 μm precoated glass plates (0.25 mm) and visualized by UV fluorescence quenching and KMnO₄. Huanghai silica gel (particle size 200 – 300 mesh) was used for chromatography. ¹H NMR spectra were recorded at room temperature on a Bruker ADVANCE III 500 MHz spectrometer and were reported relative to residual CDCl₃ (δ 7.26 ppm). ¹³C NMR spectra were recorded on a Bruker ADVANCE III 500 MHz spectrometer (125 MHz) and were reported relative to CDCl₃ (δ 77.16 ppm). ¹⁹F NMR spectra were recorded on a Bruker ADVANCE III 500 MHz spectrometer (471 MHz). Data for ¹H NMR were reported as chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration) using standard abbreviations for multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Data for ¹³C NMR and ¹⁹F NMR were reported in terms of chemical shifts (δ ppm). High resolution mass spectra (HRMS) were obtained by use of a Bruker Compact TOF mass spectrometer in electrospray ionization mode (ESI⁺). Single crystal diffraction data were collected using a Bruker SMART APEXII diffractometer.

Unless otherwise noted, all reagents were purchased commercially from Adamas, Innochem, Alfa, Energy Chemical, Sigma-Aldrich and used without further purification. Petroleum ether (PE) (60 ~ 90 °C), ethyl acetate (EA) and dichloromethane (DCM) were used as eluent for silica gel chromatography. Dry solvents were purchased commercially.

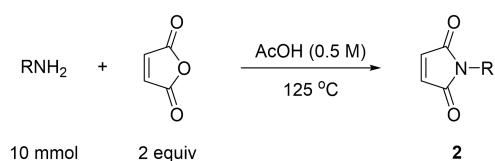
2. Preparation of Substrates

a) Synthesis of 1,6-Diynes **1**



According to the related literature,^[1-3] a mixture of 1,8-dibromonaphthalene (0.86 g, 3 mmol, 1 equiv), alkyne (12 mmol, 4 equiv), Pd(PPh₃)₂Cl₂ (105.6 mg, 0.15 mmol, 5 mol%), PPh₃ (78.6 mg, 0.3 mmol, 10 mol%), CuI (58.0 mg, 0.3 mmol, 10 mol%), and triethylamine (6 mL) were added to a 50 mL Schlenk-tube which was equipped with a stirring bar under nitrogen atmosphere, the obtained mixture was stirred at 90 °C for 12 h. After the reaction was finished, the solution was concentrated under the reduced pressure to give the crude product which was subsequently purified by flash column chromatography, furnishing the corresponding 1,6-diynes **1** smoothly.

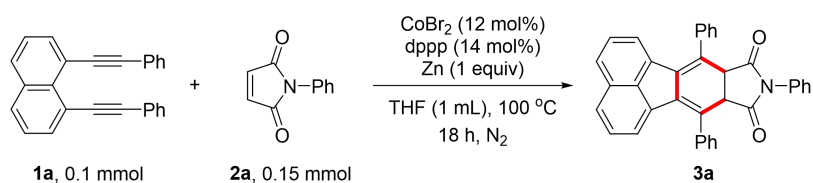
b) Synthesis of Maleimides **2**



According to the related literature,^[4] a mixture of aniline (10 mmol, 1 equiv), maleic anhydride (1.96 g, 20 mmol, 2 equiv), and acetic acid (20 mL) were added to a 100 mL round-bottom flask which was equipped with a stirring bar under air atmosphere, the solution was refluxed at 125 °C until the starting material was completely consumed. After the reaction was finished, the solution was cooled to room temperature and concentrated under the reduced pressure to give the mixture which was subsequently extracted by EA (15 mL x 3). Next, the combined organic phase was washed by water and dried over Na₂SO₄, followed by filtering. The filtrates was concentrated under the reduced pressure which was subsequently purified by flash column chromatography, furnishing the corresponding maleimides derivatives **2**.

3. General Procedure A: Concise Synthesis of Succinimide-Fused Densely 1,3-Cyclohexadienes via Co-Catalyzed [2+2+2]

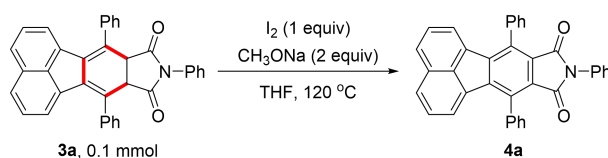
Cycloaddition of 1,6-Diynes and Maleimides



A mixture of 1,6-diynes **1a** (32.8 mg, 0.1 mmol, 1 equiv), *N*-phenylmaleimide **2a**

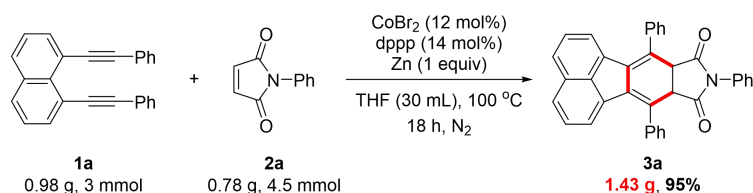
(26.0 mg, 0.15 mmol, 1.5 equiv), CoBr₂ (2.6 mg, 0.012 mmol, 12 mol%), dppp (5.9 mg, 0.014 mmol, 14 mol%), Zn (6.5 mg, 0.1 mmol, 1 equiv), and THF (1 mL) was added to a 10 mL Schlenk-tube under N₂ atmosphere, and the mixture was stirred at 100 °C for 18 hours. After the reaction was completed, the mixture was diluted with EA (20 mL) and filtered. The filtrates were concentrated under reduced pressure to give a crude residue which was purified by flash column chromatography to provide the desired product **3a** (48.1 mg) in 96% yield as a green oil. $R_f = 0.3$ (PE : EA = 5 : 1).

4. General Procedure B: Synthesis of 7, 9, 11-Triaryl-8*H*-Acenaphtho [1,2-*f*] Isoindole -8,10(9*H*)-Diones



A mixture of 7,9,11-triphenyl-7a,10a-dihydro-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione **3a** (50.5 mg, 0.1 mmol, 1 equiv), I₂ (25.6 mg, 0.1 mmol, 1 equiv), CH₃ONa (11.2 mg, 0.2 mmol, 2 equiv), and THF (1 mL) was added to a 10 mL Schlenk-tube under air atmosphere, and the mixture was stirred at 120 °C for 12 hours. After the reaction was completed, the mixture was diluted with EA (20 mL) and filtered. The filtrates were concentrated under reduced pressure to give a crude residue which was purified by flash column chromatography to provide the desired product **4a** (37.5 mg) in 75% yield as a green solid. $R_f = 0.4$ (PE : EA = 5 : 1).

5. Gram-scale Reaction

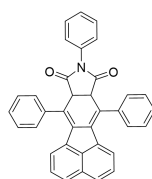


A mixture of 1,6-diynes **1a** (0.98 g, 3 mmol, 1 equiv), *N*-phenylmaleimide **2a** (0.78 g, 4.5 mmol, 1.5 equiv), CoBr₂ (77.8 mg, 0.36 mmol, 12 mol%), dppp (173.1 mg, 0.42 mmol, 14 mol%), Zn (192.0 mg, 3 mmol, 1 equiv), and THF (30 mL) was added to a

100 mL Schlenk-tube under N₂ atmosphere, and the mixture was stirred at 100 °C for 18 hours. After the reaction was completed, the mixture was diluted with EA (60 mL) and filtered. The filtrates were concentrated under reduced pressure to give a crude residue which was purified by flash column chromatography to provide the desired product **3a** (1.43 g) in 95% yield as a yellow solid. $R_f = 0.3$ (PE : EA = 5 : 1).

6. Spectra Data of 3a-3aj, 4a-4h, and 5.

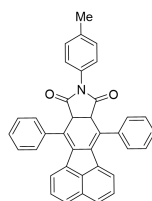
7,9,11-Triphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3a)



Following the general procedure A, the desired product **3a** (48.1 mg) was obtained in 96% yield as a green oil. $R_f = 0.3$ (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.66 – 7.45 (m, 12H), 7.41 – 7.36 (m, 2H), 7.33 – 7.28 (m, 1H), 7.25 (d, $J = 7.9$ Hz, 2H), 7.23 – 7.19 (m, 2H), 6.69 (d, $J = 7.3$ Hz, 2H), 4.38 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 175.0, 139.2, 138.9, 135.2, 134.3, 132.0, 131.0, 129.24, 129.16, 128.5, 127.8, 126.7, 126.3, 125.2, 120.0, 49.4; HRMS (ESI⁺) m/z calc'd for C₃₆H₂₄NO₂⁺ [M+H]⁺: 502.1802, found 502.1804.

7,11-Diphenyl-9-(*p*-tolyl)-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3b)

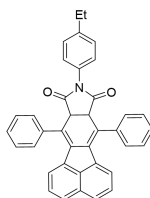


Following the general procedure A, the desired product **3b** (46.4 mg) was obtained in 90% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.66 – 7.52 (m, 10H), 7.51 – 7.47 (m, 2H), 7.25 – 7.19 (m, 4H), 7.17 – 7.12 (m, 2H), 6.69 (d, $J = 7.2$ Hz, 2H), 4.42 (s, 2H), 2.34 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 175.1, 139.3, 139.0, 138.6, 135.3, 134.4, 131.1, 129.9, 129.34, 129.26, 128.5, 127.8, 126.8, 126.1, 125.2, 120.0, 49.5, 21.3; HRMS (ESI⁺)

m/z calc'd for $C_{37}H_{26}NO_2^+$ $[M+H]^+$: 516.1958, found 516.1962.

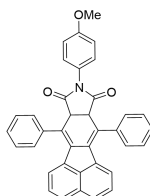
9-(4-Ethylphenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3c)



Following the general procedure A, the desired product **3c** (49.7 mg) was obtained in 94% yield as a green solid. $R_f = 0.3$ (PE : EA = 10 : 1).

1H NMR (500 MHz, $CDCl_3$) δ 7.65 – 7.43 (m, 12H), 7.23 – 7.19 (m, 4H), 7.15 (d, $J = 8.2$ Hz, 2H), 6.67 (d, $J = 7.2$ Hz, 2H), 4.38 (s, 2H), 2.61 (q, $J = 7.6$ Hz, 2H), 1.18 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.1, 144.8, 139.3, 138.9, 135.2, 134.3, 131.0, 129.5, 129.2, 128.7, 128.5, 127.8, 126.8, 126.2, 125.2, 120.0, 49.5, 28.7, 15.5; HRMS (ESI⁺) m/z calc'd for $C_{38}H_{28}NO_2^+$ $[M+H]^+$: 530.2115, found 530.2115.

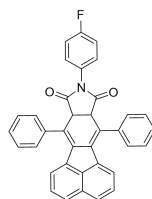
9-(4-Methoxyphenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3d)



Following the general procedure A, the desired product **3d** (50.4 mg) was obtained in 95% yield as a green oil. $R_f = 0.2$ (PE : EA = 10 : 1).

1H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.44 (m, 12H), 7.21 (t, $J = 7.2$ Hz, 2H), 7.18 – 7.13 (m, 2H), 6.89 (d, $J = 8.8$ Hz, 2H), 6.67 (dd, $J = 7.1, 1.9$ Hz, 2H), 4.37 (s, 2H), 3.74 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.2, 159.4, 139.3, 139.0, 135.2, 134.3, 131.0, 129.3, 128.5, 127.8, 127.5, 126.8, 125.2, 124.6, 120.0, 114.5, 55.6, 49.4; HRMS (ESI⁺) m/z calc'd for $C_{37}H_{26}NO_3^+$ $[M+H]^+$: 532.1907, found 532.1904.

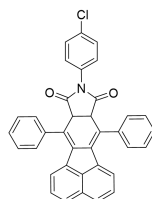
9-(4-Fluorophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3e)



Following the general procedure A, the desired product **3e** (47.2 mg) was obtained in 91% yield as a green solid. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.56 – 7.38 (m, 12H), 7.18 – 7.11 (m, 4H), 7.02 – 6.96 (m, 2H), 6.60 (d, $J = 7.3$ Hz, 2H), 4.32 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.9, 162.1 (d, $J = 246.3$ Hz), 139.3, 138.8, 135.2, 134.4, 131.0, 129.3, 128.6, 128.1 (d, $J = 8.8$ Hz), 127.9 (d, $J = 2.5$ Hz), 127.8, 126.5, 125.3, 120.0, 116.2 (d, $J = 22.5$ Hz), 49.4; ^{19}F NMR (471 MHz, CDCl_3) δ -112.36; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{23}\text{FNO}_2^+$ [M+H]⁺: 520.1707, found 520.1705.

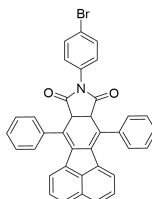
9-(4-Chlorophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3f)



Following the general procedure A, the desired product **3f** (45.5 mg) was obtained in 85% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.66 – 7.45 (m, 12H), 7.38 – 7.33 (m, 2H), 7.24 – 7.19 (m, 4H), 6.68 (dd, $J = 7.2, 1.8$ Hz, 2H), 4.40 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.8, 139.3, 138.8, 135.1, 134.5, 134.2, 131.0, 130.4, 129.4, 129.3, 128.6, 127.8, 127.5, 126.5, 125.3, 120.1, 49.4; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{23}\text{ClNO}_2^+$ [M+H]⁺: 536.1412, found 536.1415.

9-(4-Bromophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3g)

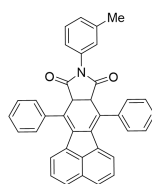


Following the general procedure A, the desired product **3g** (46.7 mg) was obtained in

81% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.61 – 7.47 (m, 14H), 7.24 – 7.20 (m, 2H), 7.18 – 7.13 (m, 2H), 6.68 (d, $J = 7.3$ Hz, 2H), 4.40 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.7, 139.3, 138.8, 135.1, 134.5, 132.4, 131.1, 131.0, 129.3, 128.6, 127.8, 127.7, 126.5, 125.3, 122.3, 120.1, 49.5; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{23}\text{BrNO}_2^+$ $[\text{M}+\text{H}]^+$: 580.0907, found 580.0905.

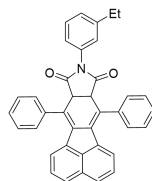
7,11-Diphenyl-9-(*m*-tolyl)-7a,10a-dihydro-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (3h)



Following the general procedure A, the desired product **3h** (41.2 mg) was obtained in 80% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 – 7.46 (m, 12H), 7.28 (t, $J = 7.8$ Hz, 1H), 7.24 – 7.20 (m, 2H), 7.13 (d, $J = 7.6$ Hz, 1H), 7.08 – 7.02 (m, 2H), 6.68 (d, $J = 7.2$ Hz, 2H), 4.42 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.1, 139.3, 139.2, 138.9, 135.3, 134.4, 131.9, 131.1, 129.5, 129.3, 129.0, 128.5, 127.8, 127.0, 126.8, 125.2, 123.5, 120.0, 49.5, 21.5; HRMS (ESI⁺) m/z calc'd for $\text{C}_{37}\text{H}_{26}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 516.1958, found 516.1960.

9-(3-Ethylphenyl)-7,11-diphenyl-7a,10a-dihydro-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (3i)

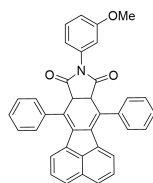


Following the general procedure A, the desired product **3i** (49.2 mg) was obtained in 93% yield as a green oil. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.65 – 7.45 (m, 12H), 7.29 (t, $J = 7.8$ Hz, 1H), 7.23 – 7.18 (m, 2H), 7.14 (d, $J = 7.6$ Hz, 1H), 7.09 – 7.02 (m, 2H), 6.67 (dd, $J = 7.2, 2.1$ Hz, 2H), 4.39 (s, 2H), 2.61 (q, $J = 7.5$ Hz, 2H), 1.24 – 1.12 (m, 3H); ^{13}C NMR (125 MHz,

CDCl₃) δ 175.1, 145.4, 139.3, 138.9, 135.2, 134.4, 131.9, 131.0, 129.3, 129.1, 128.5, 128.2, 127.8, 126.8, 125.8, 125.2, 123.7, 120.0, 49.5, 28.7, 15.2; HRMS (ESI⁺) m/z calc'd for C₃₈H₂₈NO₂⁺ [M+H]⁺: 530.2115, found 530.2118.

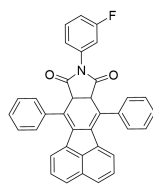
9-(3-Methoxyphenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3j)



Following the general procedure A, the desired product **3j** (51.0 mg) was obtained in 96% yield as a green oil. R_f = 0.2 (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.65 – 7.45 (m, 12H), 7.29 (t, J = 8.2 Hz, 1H), 7.25 – 7.20 (m, 2H), 6.89 – 6.83 (m, 2H), 6.81 – 6.78 (m, 1H), 6.71 – 6.67 (m, 2H), 4.40 (s, 2H), 3.74 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 174.9, 160.1, 139.3, 138.9, 135.2, 134.4, 133.0, 131.0, 129.9, 129.3, 128.5, 127.8, 126.7, 125.2, 120.0, 118.6, 114.6, 112.0, 55.5, 49.5; HRMS (ESI⁺) m/z calc'd for C₃₇H₂₆NO₃⁺ [M+H]⁺: 532.1907, found 532.1911.

9-(3-Fluorophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3k)

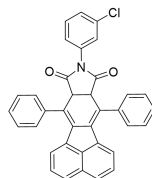


Following the general procedure A, the desired product **3k** (45.7 mg) was obtained in 88% yield as a green solid. R_f = 0.3 (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.55 – 7.40 (m, 12H), 7.31 – 7.25 (m, 1H), 7.17 – 7.12 (m, 2H), 7.04 – 6.94 (m, 3H), 6.61 (dd, J = 7.2, 1.8 Hz, 2H), 4.35 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 174.7, 162.7 (d, J = 245.0 Hz), 139.3, 138.8, 135.1, 134.6, 133.2 (d, J = 10.0 Hz), 131.1, 130.3 (d, J = 8.8 Hz), 129.4, 128.9, 128.7, 127.8, 126.4, 125.4, 121.9 (d, J = 2.5 Hz), 120.1, 115.6 (d, J = 21.3 Hz), 113.9 (d, J = 25.0 Hz), 49.5; ¹⁹F NMR (471 MHz, CDCl₃) δ -111.00; HRMS (ESI⁺) m/z calc'd for C₃₆H₂₃FNO₂⁺

[M+H]⁺: 520.1707, found 520.1709.

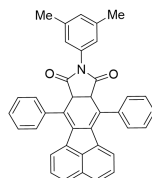
9-(3-Chlorophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3l)



Following the general procedure A, the desired product **3l** (50.8 mg) was obtained in 95% yield as a green oil. $R_f = 0.2$ (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.66 – 7.46 (m, 12H), 7.34 – 7.27 (m, 3H), 7.24 – 7.20 (m, 2H), 7.19 – 7.15 (m, 1H), 6.69 (d, $J = 7.2$ Hz, 2H), 4.40 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 174.6, 139.3, 138.8, 135.1, 134.7, 134.5, 133.0, 131.0, 130.1, 129.3, 128.7, 128.6, 127.8, 126.5, 126.4, 125.3, 124.4, 120.0, 49.4; HRMS (ESI⁺) m/z calc'd for C₃₆H₂₃ClNO₂⁺ [M+H]⁺: 536.1412, found 536.1412.

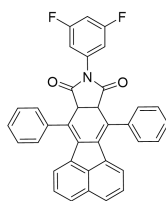
9-(3,5-Dimethylphenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3m)



Following the general procedure A, the desired product **3m** (50.3 mg) was obtained in 95% yield as a green oil. $R_f = 0.3$ (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.68 – 7.43 (m, 12H), 7.21 (t, $J = 7.7$ Hz, 2H), 6.94 (s, 1H), 6.84 (s, 2H), 6.67 (d, $J = 7.2$ Hz, 2H), 4.38 (s, 2H), 2.27 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 175.1, 139.3, 139.0, 135.3, 134.3, 131.7, 131.0, 130.4, 129.2, 128.5, 127.8, 126.8, 125.2, 124.1, 120.0, 49.5, 21.3; HRMS (ESI⁺) m/z calc'd for C₃₈H₂₈NO₂⁺ [M+H]⁺: 530.2115, found 530.2112.

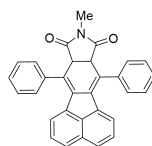
9-(3,5-Difluorophenyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3n)



Following the general procedure A, the desired product **3n** (27.9 mg) was obtained in 52% yield as a green oil. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.64 – 7.48 (m, 12H), 7.25 – 7.20 (m, 2H), 6.96 – 6.88 (m, 2H), 6.81 – 6.75 (m, 1H), 6.68 (d, $J = 7.3$ Hz, 2H), 4.42 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.3, 162.9 (dd, $J = 247.5, 13.8$ Hz), 139.3, 138.7, 135.1, 134.7, 133.9 (dd, $J = 12.5, 0.8$ Hz), 131.1, 129.4, 128.7, 127.9, 126.2, 125.4, 120.1, 109.8, 109.7 (dd, $J = 21.3, 6.3$ Hz), 109.6, 104.1 (dd, $J = 26.3, 0.8$ Hz), 49.4; ^{19}F NMR (471 MHz, CDCl_3) δ -108.08; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{36}\text{H}_{22}\text{F}_2\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 538.1613, found 538.1618.

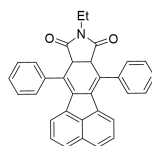
9-Methyl-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3o)



Following the general procedure A, the desired product **3o** (38.2 mg) was obtained in 87% yield as a green solid. $R_f = 0.2$ (PE : EA = 3 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.64 – 7.47 (m, 12H), 7.24 – 7.18 (m, 2H), 6.65 (d, $J = 7.3$ Hz, 2H), 4.29 (s, 2H), 3.01 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.9, 139.2, 139.1, 135.3, 133.9, 131.0, 129.2, 128.5, 127.8, 126.7, 125.1, 119.9, 49.0, 25.4; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{31}\text{H}_{22}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 440.1645, found 440.1642.

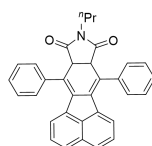
9-Ethyl-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3p)



Following the general procedure A, the desired product **3p** (38.1 mg) was obtained in 84% yield as a green solid. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.66 – 7.46 (m, 12H), 7.25 – 7.19 (m, 2H), 6.67 (d, $J = 7.2$ Hz, 2H), 4.27 (s, 2H), 3.58 (q, $J = 7.1$ Hz, 2H), 1.16 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.7, 139.3, 139.1, 135.3, 133.9, 131.1, 129.2, 128.5, 127.8, 126.9, 125.1, 119.9, 49.1, 34.3, 13.2; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{32}\text{H}_{24}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 454.1802, found 454.1804.

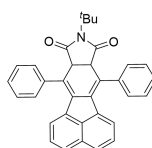
7,11-Diphenyl-9-propyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3q)



Following the general procedure A, the desired product **3q** (42.0 mg) was obtained in 90% yield as a green solid. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 – 7.46 (m, 12H), 7.25 – 7.20 (m, 2H), 6.68 (d, $J = 7.3$ Hz, 2H), 4.28 (s, 2H), 3.49 (t, $J = 7.3$ Hz, 2H), 1.63 – 1.57 (m, 2H), 0.89 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.9, 139.2, 139.1, 135.3, 133.8, 131.0, 129.2, 128.5, 127.8, 126.9, 125.1, 119.9, 49.0, 40.9, 21.2, 11.4; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{33}\text{H}_{26}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 468.1958, found 468.1954.

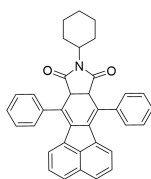
9-(tert-Butyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3r)



Following the general procedure A, the desired product **3r** (34.2 mg) was obtained in 71% yield as a green oil. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 – 7.44 (m, 12H), 7.24 – 7.18 (m, 2H), 6.67 (d, $J = 7.2$ Hz, 2H), 4.11 (s, 2H), 1.52 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3) δ 177.1, 139.3, 135.4, 134.0, 131.1, 129.2, 128.4, 127.8, 127.5, 125.1, 119.9, 58.9, 50.0, 28.6; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{34}\text{H}_{28}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 482.2115, found 482.2116.

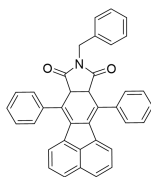
9-Cyclohexyl-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3s)



Following the general procedure A, the desired product **3s** (43.1 mg) was obtained in 85% yield as a green oil. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 – 7.41 (m, 12H), 7.22 – 7.17 (m, 2H), 6.64 (d, $J = 7.3$ Hz, 2H), 4.19 (s, 2H), 4.01 – 3.89 (m, 1H), 2.14 – 2.03 (m, 2H), 1.77 (d, $J = 13.3$ Hz, 2H), 1.64 – 1.52 (m, 3H), 1.29 – 1.10 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 176.0, 139.24, 139.16, 135.3, 133.9, 131.0, 129.2, 128.4, 127.8, 127.1, 125.1, 119.9, 52.2, 49.1, 29.0, 26.0, 25.0; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{30}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 508.2271, found 508.2273.

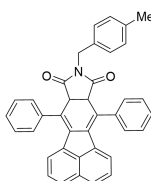
9-Benzyl-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3t)



Following the general procedure A, the desired product **3t** (46.4 mg) was obtained in 90% yield as a green oil. $R_f = 0.2$ (PE : EA = 3 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.57 – 7.45 (m, 8H), 7.41 (d, $J = 7.2$ Hz, 4H), 7.36 – 7.32 (m, 2H), 7.27 – 7.22 (m, 3H), 7.18 (t, $J = 7.7$ Hz, 2H), 6.60 (d, $J = 7.2$ Hz, 2H), 4.63 (s, 2H), 4.23 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.5, 139.2, 139.0, 135.8, 135.3, 134.0, 131.0, 129.23, 129.19, 128.7, 128.5, 128.1, 127.8, 126.8, 125.1, 119.9, 49.2, 42.9; HRMS (ESI⁺) m/z calc'd for $\text{C}_{37}\text{H}_{26}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 516.1958, found 516.1958.

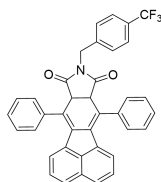
9-(4-Methylbenzyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3u)



Following the general procedure A, the desired product **3u** (46.6 mg) was obtained in 88% yield as a green oil. $R_f = 0.2$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 – 7.40 (m, 12H), 7.31 – 7.25 (m, 2H), 7.22 (t, $J = 7.7$ Hz, 2H), 7.09 (d, $J = 7.8$ Hz, 2H), 6.64 (d, $J = 7.3$ Hz, 2H), 4.63 (s, 2H), 4.25 (s, 2H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.5, 139.2, 139.0, 137.8, 135.3, 134.0, 132.9, 131.0, 129.3, 129.22, 129.16, 128.4, 127.7, 126.8, 125.1, 119.9, 49.1, 42.6, 21.2; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_2^+$ [M+H]⁺: 530.2115, found 530.2111.

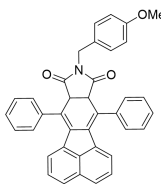
7,11-Diphenyl-9-(4-(trifluoromethyl)benzyl)-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3v)



Following the general procedure A, the desired product **3v** (47.2 mg) was obtained in 81% yield as a green solid. $R_f = 0.2$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 – 7.50 (m, 10H), 7.48 (d, $J = 8.1$ Hz, 2H), 7.45 – 7.41 (m, 4H), 7.22 (t, $J = 7.7$ Hz, 2H), 6.65 (dd, $J = 7.2, 1.7$ Hz, 2H), 4.69 (s, 2H), 4.30 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.4, 139.5, 139.2, 138.8, 135.1, 134.1, 131.0, 130.4 (q, $J = 32.5$ Hz), 129.6, 129.2, 128.6, 127.8, 126.4, 125.7 (q, $J = 3.6$ Hz), 125.2, 124.1 (q, $J = 270.0$ Hz), 120.0, 49.1, 42.3; ^{19}F NMR (471 MHz, CDCl_3) δ -62.55; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{25}\text{F}_3\text{NO}_2^+$ [M+H]⁺: 584.1832, found 584.1836.

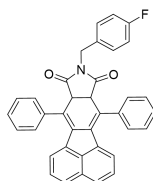
9-(4-Methoxybenzyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3w)



Following the general procedure A, the desired product **3w** (49.1 mg) was obtained in 90% yield as a green oil. $R_f = 0.3$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 – 7.41 (m, 12H), 7.33 (d, $J = 8.5$ Hz, 2H), 7.22 (t, $J = 7.7$ Hz, 2H), 6.81 (d, $J = 8.5$ Hz, 2H), 6.64 (d, $J = 7.2$ Hz, 2H), 4.61 (s, 2H), 4.24 (s, 2H), 3.76 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.5, 159.4, 139.2, 139.0, 135.3, 133.9, 131.0, 130.7, 129.1, 128.4, 128.1, 127.7, 126.8, 125.1, 119.8, 114.0, 55.3, 49.1, 42.3; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_3^+$ $[\text{M}+\text{H}]^+$: 546.2064, found 546.2061.

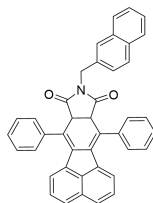
9-(4-Fluorobenzyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3x)



Following the general procedure A, the desired product **3x** (34.6 mg) was obtained in 65% yield as a green oil. $R_f = 0.2$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.60 – 7.47 (m, 8H), 7.45 – 7.40 (m, 4H), 7.37 – 7.32 (m, 2H), 7.21 (t, $J = 7.7$ Hz, 2H), 6.95 (t, $J = 8.5$ Hz, 2H), 6.62 (d, $J = 7.3$ Hz, 2H), 4.61 (s, 2H), 4.27 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.5, 162.6 (d, $J = 245.0$ Hz), 139.2, 138.9, 135.2, 134.1, 131.7 (d, $J = 3.3$ Hz), 131.3 (d, $J = 8.8$ Hz), 131.0, 129.2, 128.5, 127.8, 126.6, 125.2, 119.9, 115.6 (d, $J = 21.3$ Hz), 49.2, 42.1; ^{19}F NMR (471 MHz, CDCl_3) δ -113.88; HRMS (ESI⁺) m/z calc'd for $\text{C}_{37}\text{H}_{25}\text{FNO}_2^+$ $[\text{M}+\text{H}]^+$: 534.1864, found 534.1866.

9-(Naphthalen-2-ylmethyl)-7,11-diphenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3y)

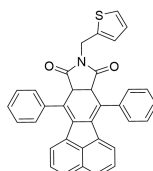


Following the general procedure A, the desired product **3y** (53.7 mg) was obtained in 95% yield as a green oil $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 8.26 (d, $J = 8.3$ Hz, 1H), 7.81 (dd, $J = 26.5, 8.0$ Hz, 2H), 7.61 – 7.39 (m, 16H), 7.21 (t, $J = 7.7$ Hz, 2H), 6.62 (d, $J = 7.1$ Hz, 2H), 5.15 (s, 2H), 4.27 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.7, 139.2, 138.9, 135.2, 134.0,

133.8, 131.4, 131.0, 130.8, 129.1, 128.9, 128.7, 128.39, 128.35, 127.7, 126.72, 126.69, 125.9, 125.3, 125.1, 123.8, 119.9, 49.2, 40.7; HRMS (ESI⁺) *m/z* calc'd for C₄₁H₂₈NO₂⁺ [M+H]⁺: 566.2115, found 566.2117.

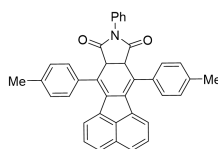
7,11-Diphenyl-9-(thiophen-2-ylmethyl)-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3z)



Following the general procedure A, the desired product **3z** (38.0 mg) was obtained in 73% yield as a green oil. *R_f* = 0.4 (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.62 – 7.41 (m, 12H), 7.24 – 7.16 (m, 3H), 7.10 – 7.05 (m, 1H), 6.93 – 6.86 (m, 1H), 6.66 – 6.60 (m, 2H), 4.83 (s, 2H), 4.26 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 175.0, 139.2, 138.9, 137.1, 135.3, 134.0, 131.0, 129.2, 128.5, 127.8, 126.9, 126.60, 126.59, 126.2, 125.1, 119.9, 49.1, 36.9; HRMS (ESI⁺) *m/z* calc'd for C₃₅H₂₄NO₂S⁺ [M+H]⁺: 522.1522, found 522.1518.

9-Phenyl-7,11-di-*p*-tolyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3aa)

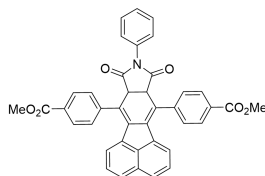


Following the general procedure A, the desired product **3aa** (47.6 mg) was obtained in 90% yield as a green oil. *R_f* = 0.4 (PE : EA = 5 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.61 (d, *J* = 8.2 Hz, 2H), 7.51 – 7.32 (m, 11H), 7.30 – 7.24 (m, 4H), 6.79 (d, *J* = 7.2 Hz, 2H), 4.41 (s, 2H), 2.49 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 175.2, 139.3, 138.3, 135.9, 135.4, 134.3, 132.0, 131.0, 130.0, 129.2, 128.5, 127.8, 126.7, 126.3, 125.1, 120.0, 49.6, 21.6; HRMS (ESI⁺) *m/z* calc'd for C₃₈H₂₈NO₂⁺ [M+H]⁺: 530.2115, found 530.2113.

Dimethyl

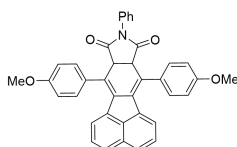
4,4'-(8,10-dioxo-9-phenyl-7a,9,10,10a-tetrahydro-8H-acenaphtho[1,2-f]isoindole-7,11-diyl)dibenzoate (**3ab**)



Following the general procedure A, the desired product **3ab** (30.2 mg) was obtained in 49% yield as a green oil. $R_f = 0.2$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 8.31 – 8.26 (m, 4H), 7.77 – 7.63 (m, 6H), 7.48 – 7.44 (m, 2H), 7.41 – 7.37 (m, 1H), 7.31 – 7.27 (m, 4H), 6.72 (d, $J = 7.2$ Hz, 2H), 4.51 (s, 2H), 4.02 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.6, 167.0, 143.7, 139.4, 134.8, 134.6, 131.8, 131.1, 130.7, 130.3, 129.3, 128.8, 127.9, 126.2, 125.83, 125.77, 120.2, 52.4, 49.1; HRMS (ESI⁺) m/z calc'd for $\text{C}_{40}\text{H}_{28}\text{NO}_6^+$ [M+H]⁺: 618.1911, found 618.1911.

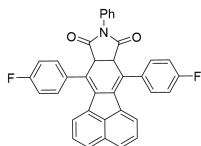
7,11-Bis(4-methoxyphenyl)-9-phenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (**3ac**)



Following the general procedure A, the desired product **3ac** (43.8 mg) was obtained in 78% yield as a green solid. $R_f = 0.2$ (PE : EA = 3 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 (d, $J = 8.2$ Hz, 2H), 7.58 – 7.46 (m, 4H), 7.43 (t, $J = 7.7$ Hz, 2H), 7.35 (t, $J = 7.4$ Hz, 1H), 7.28 (t, $J = 7.7$ Hz, 4H), 7.14 – 7.07 (m, 4H), 6.82 (d, $J = 7.2$ Hz, 2H), 4.40 (s, 2H), 3.92 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.3, 159.7, 139.2, 135.5, 134.5, 132.0, 131.08, 131.07, 129.2, 128.5, 127.8, 126.4, 126.3, 125.1, 119.9, 114.6, 55.4, 49.7; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_4^+$ [M+H]⁺: 562.2013, found 562.2009.

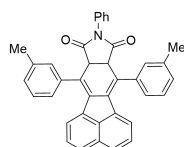
7,11-Bis(4-fluorophenyl)-9-phenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (**3ad**)



Following the general procedure A, the desired product **3ad** (44.0 mg) was obtained in 82% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 (d, $J = 8.2$ Hz, 2H), 7.60 – 7.31 (m, 7H), 7.29 – 7.21 (m, 8H), 6.68 (d, $J = 7.2$ Hz, 2H), 4.38 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.9, 162.9 (d, $J = 246.3$ Hz), 139.3, 135.0, 134.9, 131.8, 134.7 (d, $J = 2.5$ Hz), 131.8, 131.1, 129.3, 128.7, 127.9, 126.3, 125.6 (d, $J = 21.3$ Hz), 125.5, 120.0, 116.4 (d, $J = 21.3$ Hz), 49.4; ^{19}F NMR (471 MHz, CDCl_3) δ -112.78; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{22}\text{F}_2\text{NO}_2^+$ [M+H]⁺: 538.1613, found 538.1616.

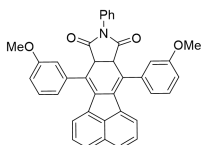
9-Phenyl-7,11-di-*m*-tolyl-7a,10a-dihydro-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (3ae)



Following the general procedure A, the desired product **3ae** (31.7 mg) was obtained in 60% yield as a green solid. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 (d, $J = 8.2$ Hz, 2H), 7.49 – 7.39 (m, 4H), 7.38 – 7.31 (m, 2H), 7.30 – 7.22 (m, 5H), 7.14 (d, $J = 8.1$ Hz, 2H), 7.06 – 7.01 (m, 2H), 6.78 (d, $J = 6.6$ Hz, 2H), 4.43 (s, 2H), 3.85 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.0, 160.3, 140.2, 139.3, 135.1, 134.4, 132.0, 131.1, 130.4, 129.3, 128.6, 127.9, 126.5, 126.4, 125.3, 120.2, 114.3, 55.5, 49.6; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_2^+$ [M+H]⁺: 530.2115, found 530.2118.

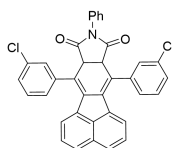
7,11-Bis(3-methoxyphenyl)-9-phenyl-7a,10a-dihydro-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (3af)



Following the general procedure A, the desired product **3af** (49.9 mg) was obtained in 89% yield as a green oil. $R_f = 0.2$ (PE : EA = 5 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.62 (d, $J = 8.2$ Hz, 2H), 7.50 – 7.39 (m, 4H), 7.37 – 7.31 (m, 1H), 7.30 – 7.23 (m, 5H), 7.22 – 7.09 (m, 3H), 7.04 – 7.01 (m, 2H), 6.78 (d, $J = 6.8$ Hz, 2H), 4.43 (s, 2H), 3.85 (s, 6H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.0, 160.3, 140.2, 139.3, 135.1, 134.5, 132.1, 131.1, 130.4, 129.3, 128.6, 127.9, 126.6, 126.4, 125.3, 120.3, 114.3, 55.5, 49.6; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_4^+$ $[\text{M}+\text{H}]^+$: 562.2013, found 562.2010.

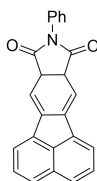
7,11-Bis(3-chlorophenyl)-9-phenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3ag)



Following the general procedure A, the desired product **3ag** (37.6 mg) was obtained in 66% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.61 (d, $J = 8.2$ Hz, 2H), 7.50 – 7.36 (m, 9H), 7.31 (t, $J = 7.4$ Hz, 1H), 7.24 (dd, $J = 15.8, 7.8$ Hz, 5H), 6.68 (d, $J = 7.2$ Hz, 2H), 4.37 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 174.7, 140.6, 139.3, 135.2, 134.9, 134.7, 131.8, 131.1, 130.6, 129.4, 128.82, 128.79, 126.3, 125.7, 125.3, 120.3, 49.1; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{36}\text{H}_{22}\text{Cl}_2\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 570.1022, found 570.1024.

9-phenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3ah)

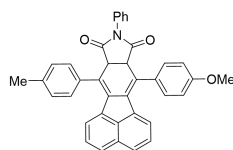


Following the general procedure A, the desired product **3ah** (8.4 mg) was obtained in 24% yield as a green oil. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.74 (d, $J = 8.1$ Hz, 2H), 7.63 (d, $J = 7.0$ Hz, 2H), 7.58 – 7.54 (m, 2H), 7.50 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 7.31 (d, $J = 7.5$ Hz, 2H), 6.58 (s, 2H), 4.24 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 177.0, 139.2, 136.9, 135.6, 131.9, 131.5, 129.4, 129.0, 128.3, 126.7, 125.5, 117.1, 111.5, 41.7; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{24}\text{H}_{16}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 350.1176, found 350.1174.

7-(4-Methoxyphenyl)-9-phenyl-11-(p-tolyl)-7a,10a-dihydro-8H-acenaphtho[1,2-f]i

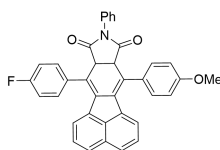
soindole-8,10(9H)-dione (3ai)



Following the general procedure A, the desired product **3ai** (46.3 mg) was obtained in 85% yield as a green solid. $R_f = 0.2$ (PE : EA = 3 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.63 (d, $J = 8.2$ Hz, 2H), 7.59 – 7.26 (m, 13H), 7.14 – 7.08 (m, 2H), 6.84 (d, $J = 7.2$ Hz, 1H), 6.80 (d, $J = 7.2$ Hz, 1H), 4.43 (d, $J = 10.3$ Hz, 1H), 4.40 (d, $J = 10.3$ Hz, 1H), 3.93 (s, 3H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.3, 175.1, 159.7, 139.2, 138.2, 135.8, 135.43, 135.41, 134.4, 134.3, 132.0, 131.1, 130.0, 129.2, 128.5, 127.8, 126.7, 126.4, 126.3, 125.10, 125.08, 119.94, 119.88, 114.6, 55.4, 49.7, 49.6, 21.6; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{38}\text{H}_{28}\text{NO}_3^+$ $[\text{M}+\text{H}]^+$: 546.2064, found 546.2061.

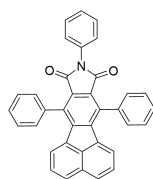
7-(4-Fluorophenyl)-11-(4-methoxyphenyl)-9-phenyl-7a,10a-dihydro-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (3aj)



Following the general procedure A, the desired product **3aj** (45.0 mg) was obtained in 82% yield as a green solid. $R_f = 0.2$ (PE : EA = 3 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.64 (dd, $J = 8.1, 5.0$ Hz, 2H), 7.61 – 7.42 (m, 6H), 7.40 – 7.34 (m, 1H), 7.33 – 7.25 (m, 6H), 7.17 – 7.08 (m, 2H), 6.83 (d, $J = 7.2$ Hz, 1H), 6.71 (d, $J = 7.2$ Hz, 1H), 4.43 (d, $J = 10.4$ Hz, 1H), 4.39 (d, $J = 10.4$ Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.2, 175.1, 162.8 (d, $J = 246.3$ Hz), 159.7, 139.2, 135.3, 135.1, 135.0, 134.9 (d, $J = 2.5$ Hz), 134.3, 131.9, 131.1, 130.9, 129.2, 128.6, 127.9, 127.8, 126.8, 126.3, 125.4, 125.3, 125.2, 120.0, 119.9, 116.4 (d, $J = 21.3$ Hz), 114.7, 55.4, 49.5, 49.4; ^{19}F NMR (471 MHz, CDCl_3) δ -112.98; HRMS (ESI $^+$) m/z calc'd for $\text{C}_{37}\text{H}_{25}\text{FNO}_3^+$ $[\text{M}+\text{H}]^+$: 550.1813, found 550.1809.

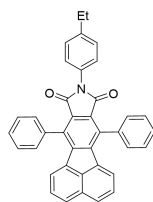
7,9,11-Triphenyl-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (4a)



Following the general procedure B, the desired product **4a** (37.4 mg) was obtained in 75% yield as a green solid. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.88 (d, $J = 8.1$ Hz, 2H), 7.66 – 7.58 (m, 10H), 7.44 – 7.35 (m, 6H), 7.30 – 7.27 (m, 1H), 6.94 (d, $J = 7.1$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.7, 143.5, 136.1, 135.6, 134.6, 133.4, 131.8, 129.7, 128.93, 128.91, 128.82, 128.75, 128.2, 127.7, 126.8, 125.8; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{22}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 500.1645, found 500.1646.

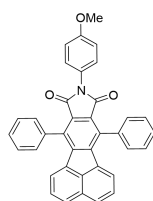
9-(4-Ethylphenyl)-7,11-diphenyl-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (4b)



Following the general procedure B, the desired product **4b** (48.0 mg) was obtained in 91% yield as a green solid. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.88 (d, $J = 8.1$ Hz, 2H), 7.66 – 7.60 (m, 10H), 7.44 – 7.39 (m, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 6.96 (d, $J = 7.2$ Hz, 2H), 2.63 (q, $J = 7.6$ Hz, 2H), 1.20 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.8, 143.9, 143.4, 136.0, 135.7, 134.6, 133.4, 129.7, 129.3, 128.92, 128.89, 128.8, 128.7, 128.2, 127.8, 126.6, 125.7, 28.7, 15.7; HRMS (ESI⁺) m/z calc'd for $\text{C}_{38}\text{H}_{26}\text{NO}_2^+$ $[\text{M}+\text{H}]^+$: 528.1958, found 528.1955.

9-(4-Methoxyphenyl)-7,11-diphenyl-8H-acenaphtho[1,2-f]isoindole-8,10(9H)-dione (4c)

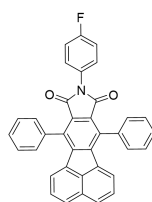


Following the general procedure B, the desired product **4c** (45.5 mg) was obtained in

86% yield as a green solid. $R_f = 0.1$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.88 (d, $J = 8.1$ Hz, 2H), 7.66 – 7.60 (m, 10H), 7.45 – 7.40 (m, 2H), 7.31 (d, $J = 8.8$ Hz, 2H), 6.96 (d, $J = 7.2$ Hz, 2H), 6.90 (d, $J = 8.9$ Hz, 2H), 3.79 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.9, 158.9, 143.4, 136.0, 135.6, 134.6, 133.4, 129.7, 128.9, 128.8, 128.7, 128.2, 128.0, 127.8, 125.7, 124.6, 114.1, 55.6; HRMS (ESI⁺) m/z calc'd for $\text{C}_{37}\text{H}_{24}\text{NO}_3^+$ $[\text{M}+\text{H}]^+$: 530.1751, found 530.1752.

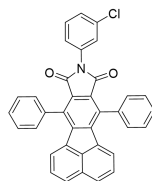
9-(4-Fluorophenyl)-7,11-diphenyl-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (4d)



Following the general procedure, the desired product **4d** (40.8 mg) was obtained in 79% yield as a green solid. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.88 (d, $J = 8.1$ Hz, 2H), 7.67 – 7.58 (m, 10H), 7.45 – 7.35 (m, 4H), 7.10 – 7.03 (m, 2H), 6.95 (d, $J = 7.1$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.6, 161.8 (d, $J = 245.0$ Hz), 143.6, 136.2, 135.6, 134.5, 133.4, 129.7, 129.0, 128.89, 128.87, 128.8, 128.5 (d, $J = 8.8$ Hz), 128.3, 127.8 (d, $J = 2.5$ Hz), 127.6, 125.8, 115.7 (d, $J = 22.5$ Hz); ^{19}F NMR (471 MHz, CDCl_3) δ -113.84; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{21}\text{FNO}_2^+$ $[\text{M}+\text{H}]^+$: 518.1551, found 518.1547.

9-(3-Chlorophenyl)-7,11-diphenyl-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (4e)

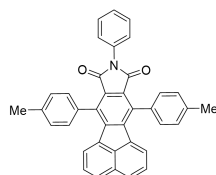


Following the general procedure B, the desired product **4e** (45.3 mg) was obtained in 85% yield as a green solid. $R_f = 0.2$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.89 (d, $J = 8.2$ Hz, 2H), 7.67 – 7.58 (m, 10H), 7.50 – 7.47 (m 1H), 7.45 – 7.40 (m, 2H), 7.36 – 7.26 (m, 3H), 6.94 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.3, 143.7, 136.2, 135.5, 134.5, 134.3, 133.5, 133.0,

129.73, 129.65, 129.0, 128.94, 128.88, 128.85, 128.3, 127.8, 127.5, 126.7, 125.9, 124.7; HRMS (ESI⁺) *m/z* calc'd for C₃₆H₂₁ClNO₂⁺ [M+H]⁺: 534.1255, found 534.1253.

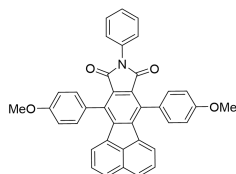
9-Phenyl-7,11-di-*p*-tolyl-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (4f)



Following the general procedure B, the desired product **4f** (50.1 mg) was obtained in 95% yield as a green solid. *R_f* = 0.3 (PE : EA = 10 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.85 (d, *J* = 8.1 Hz, 2H), 7.50 – 7.47 (m, 4H), 7.45 – 7.39 (m, 8H), 7.38 – 7.33 (m, 2H), 7.28 – 7.24 (m, 1H), 7.03 (d, *J* = 7.1 Hz, 2H), 2.53 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 166.7, 143.5, 138.5, 136.2, 134.7, 133.4, 132.5, 131.9, 129.7, 129.6, 128.8, 128.62, 128.61, 128.2, 127.8, 127.6, 126.7, 125.7, 21.7; HRMS (ESI⁺) *m/z* calc'd for C₃₈H₂₆NO₂⁺ [M+H]⁺: 528.1958, found 528.1953.

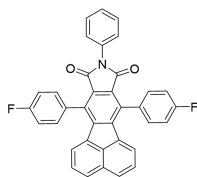
7,11-Bis(4-methoxyphenyl)-9-phenyl-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (4g)



Following the general procedure, the desired product **4g** (40.8 mg) was obtained in 73% yield as a green solid. *R_f* = 0.1 (PE : EA = 10 : 1).

¹H NMR (500 MHz, CDCl₃) δ 7.88 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 8.5 Hz, 4H), 7.47 – 7.43 (m, 2H), 7.42 – 7.35 (m, 4H), 7.31 – 7.27 (m, 1H), 7.15 (d, *J* = 8.5 Hz, 4H), 7.08 (d, *J* = 7.1 Hz, 2H), 3.96 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 166.8, 160.0, 143.8, 135.9, 134.8, 133.4, 131.9, 130.3, 129.7, 128.72, 128.67, 128.2, 127.9, 127.7, 127.6, 126.8, 125.7, 114.3, 55.5; HRMS (ESI⁺) *m/z* calc'd for C₃₈H₂₆NO₄⁺ [M+H]⁺: 560.1856, found 560.1856.

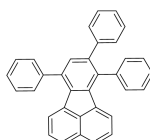
7,11-Bis(4-fluorophenyl)-9-phenyl-8*H*-acenaphtho[1,2-*f*]isoindole-8,10(9*H*)-dione (4h)



Following the general procedure, the desired product **4h** (47.1 mg) was obtained in 88% yield as a green solid. $R_f = 0.3$ (PE : EA = 10 : 1).

^1H NMR (500 MHz, CDCl_3) δ 7.92 (d, $J = 8.1$ Hz, 2H), 7.64 – 7.55 (m, 4H), 7.50 – 7.45 (m, 2H), 7.42 – 7.37 (m, 4H), 7.36 – 7.30 (m, 5H), 7.02 (d, $J = 7.1$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 166.6, 163.2 (d, $J = 247.5$ Hz), 143.7, 135.1, 134.3, 133.4, 131.7, 131.3 (d, $J = 3.8$ Hz), 130.8 (d, $J = 7.5$ Hz), 129.8, 129.0, 128.9, 128.3, 127.9 (d, $J = 2.5$ Hz), 126.7, 125.7, 116.2, 116.0; ^{19}F NMR (471 MHz, CDCl_3) δ -112.70; HRMS (ESI⁺) m/z calc'd for $\text{C}_{36}\text{H}_{20}\text{F}_2\text{NO}_2$ [M+H]⁺: 536.1457, found 536.1456.

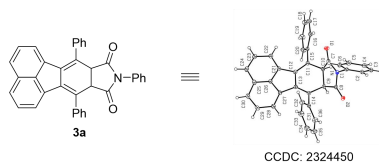
7,8,10-Triphenylfluorene (**5**)



Following the general procedure A with the addition of phenylacetylene (1.5 equiv), the desired product **5** (40.3 mg) was obtained in 91% yield as a green oil. $R_f = 0.3$ (PE).

^1H NMR (500 MHz, CDCl_3) δ 7.72 – 7.64 (m, 4H), 7.54 – 7.50 (m, 2H), 7.49 -7.45 (m, 1H), 7.37 – 7.30 (m, 8H), 7.26 – 7.20 (m, 3H), 7.17 – 7.10 (m, 3H), 6.70 (d, $J = 7.1$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 141.1, 140.9, 139.4, 138.4, 138.0, 136.7, 136.3, 136.0, 135.8, 133.3, 131.7, 131.3, 130.4, 130.1, 129.8, 129.3, 128.8, 128.6, 128.0, 127.9, 127.74, 127.67, 127.6, 127.4, 126.8, 126.5, 123.5, 123.0; HRMS (ESI⁺) m/z calc'd for $\text{C}_{34}\text{H}_{23}$ [M+H]⁺: 431.1794, found 431.1793.

7. X-ray Crystallography Data of **3a**



CCDC: 2324450

Table 1 Crystal data and structure refinement for **3a.**

Identification code	2324450
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Empirical formula	C ₃₆ H ₂₃ NO ₂
Formula weight	501.55
Temperature/K	170.00
Crystal system	triclinic
Space group	P1
a/Å	10.7902(4)
b/Å	10.8352(3)
c/Å	11.7820(4)
α/°	110.9550(10)
β/°	95.1980(10)
γ/°	105.6050(10)
Volume/Å ³	1211.94(7)
Z	2
ρ _{calc} /g/cm ³	1.374
μ/mm ⁻¹	0.085
F(000)	524.0
Crystal size/mm ³	0.42 × 0.32 × 0.26
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.256 to 55.034
Index ranges	-14 ≤ h ≤ 14, -14 ≤ k ≤ 13, -15 ≤ l ≤ 15
Reflections collected	30695
Independent reflections	9997 [R _{int} = 0.0335, R _{sigma} = 0.0337]
Data/restraints/parameters	9997/3/703
Goodness-of-fit on F ²	1.029
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0390, wR ₂ = 0.0936
Final R indexes [all data]	R ₁ = 0.0483, wR ₂ = 0.1007
Largest diff. peak/hole / e Å ⁻³	0.22/-0.20
Flack parameter	0.2(4)

8. References

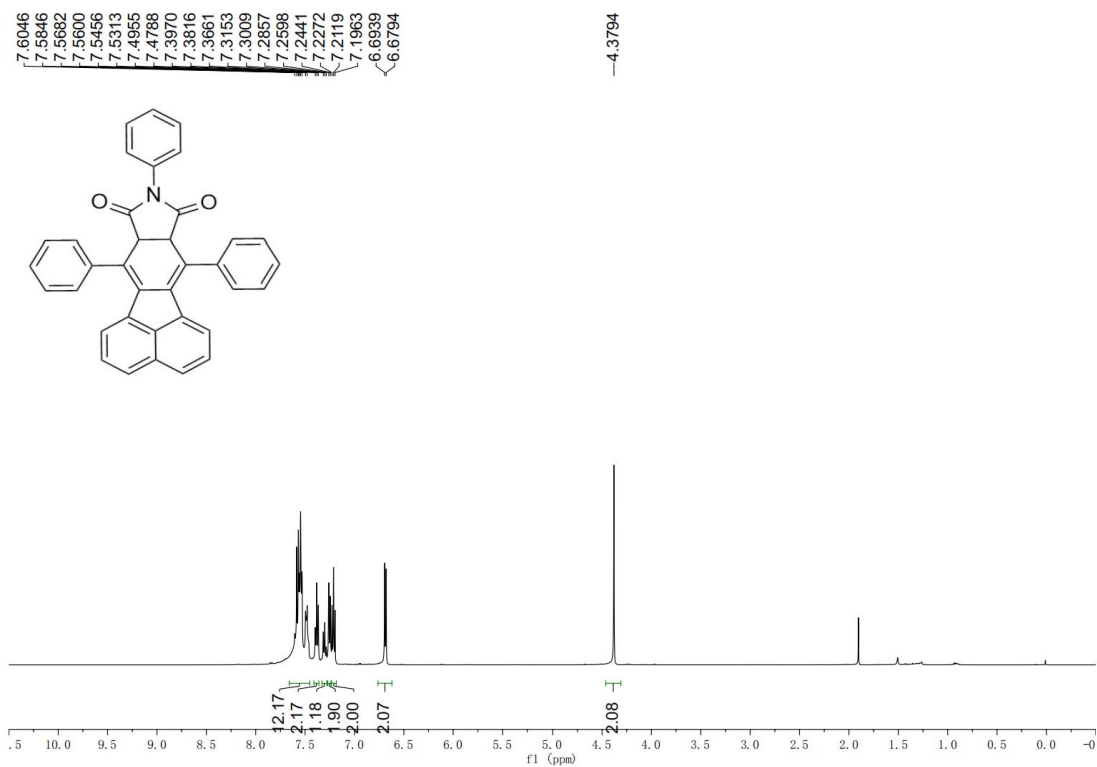
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2. T. Sawano, K. Takamura, T. Yoshikawa, K. Mruata, M. Koga, R. Yamada, T. Saito, K. Tabata, Y. Ishii, W. Kashihara, T. Nishihara, K. Tanabe, T. Suzuki and R.

Takeuchi, Synthesis of azafluoranthenes by iridium-catalyzed [2+2+2] cycloaddition and evaluation of their fluorescence properties, *Org. Biomol. Chem.*, 2023, **21**, 323.

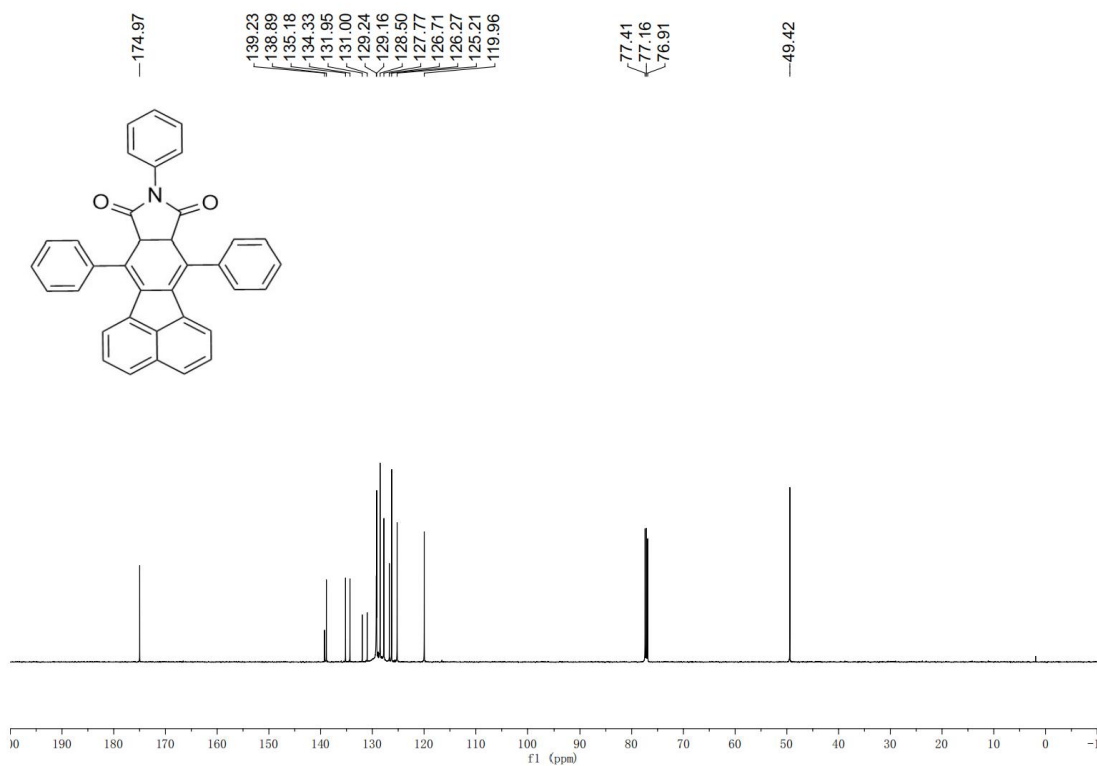
3. R. Abe, Y. Nagashima, J. Tanaka and K. Tanaka, Room temperature fluoranthene synthesis through cationic Rh(I)/H₈-BINAP-catalyzed [2+2+2] cycloaddition: unexpected acceleration due to noncovalent interactions, *ACS Catal.*, 2023, **13**, 1604.

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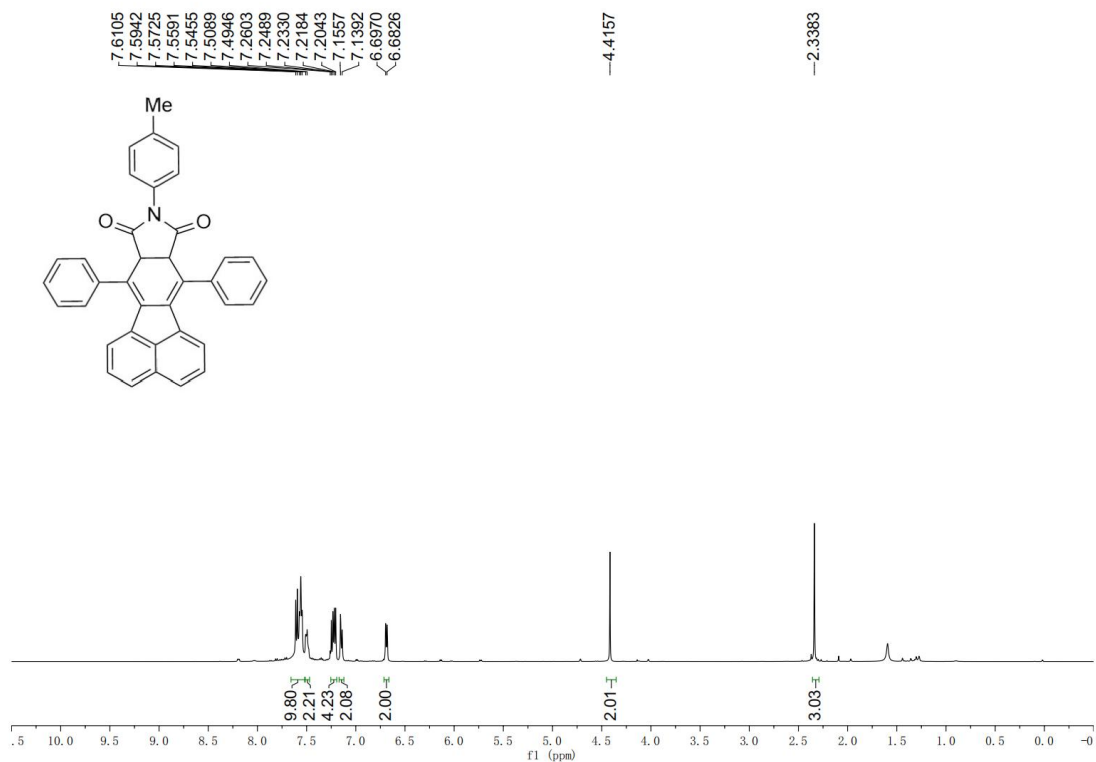
9. NMR Spectra



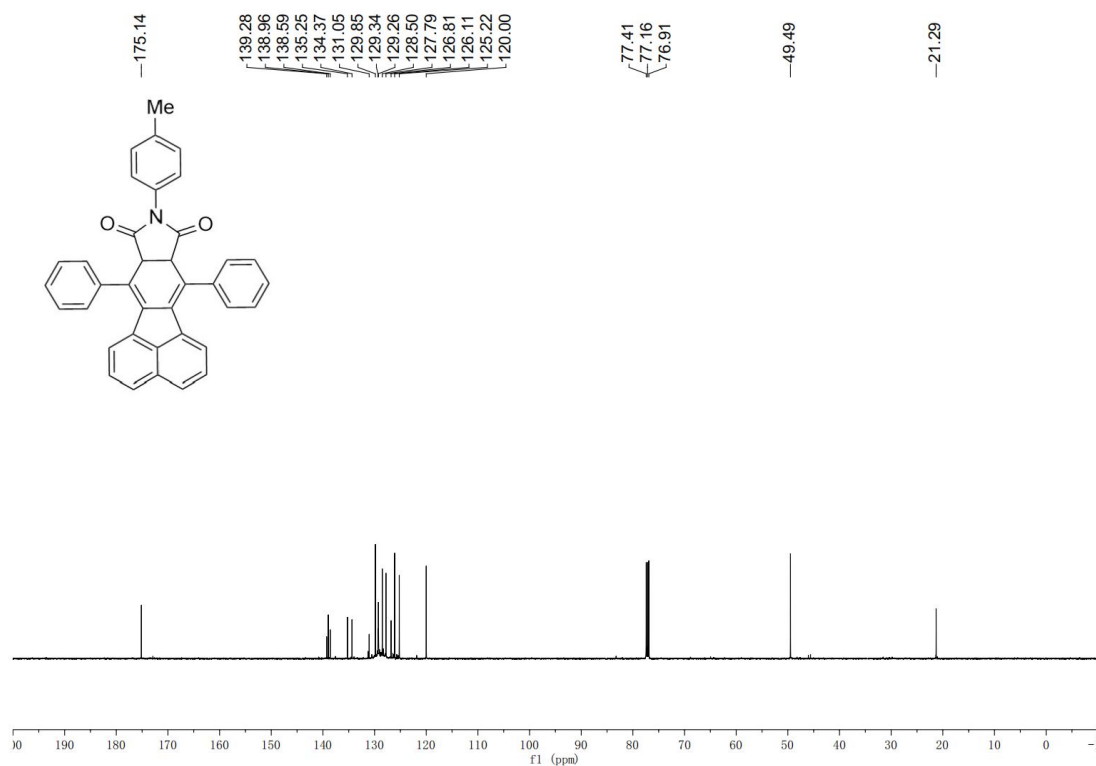
^1H NMR (500 MHz, CDCl_3) of **3a**.



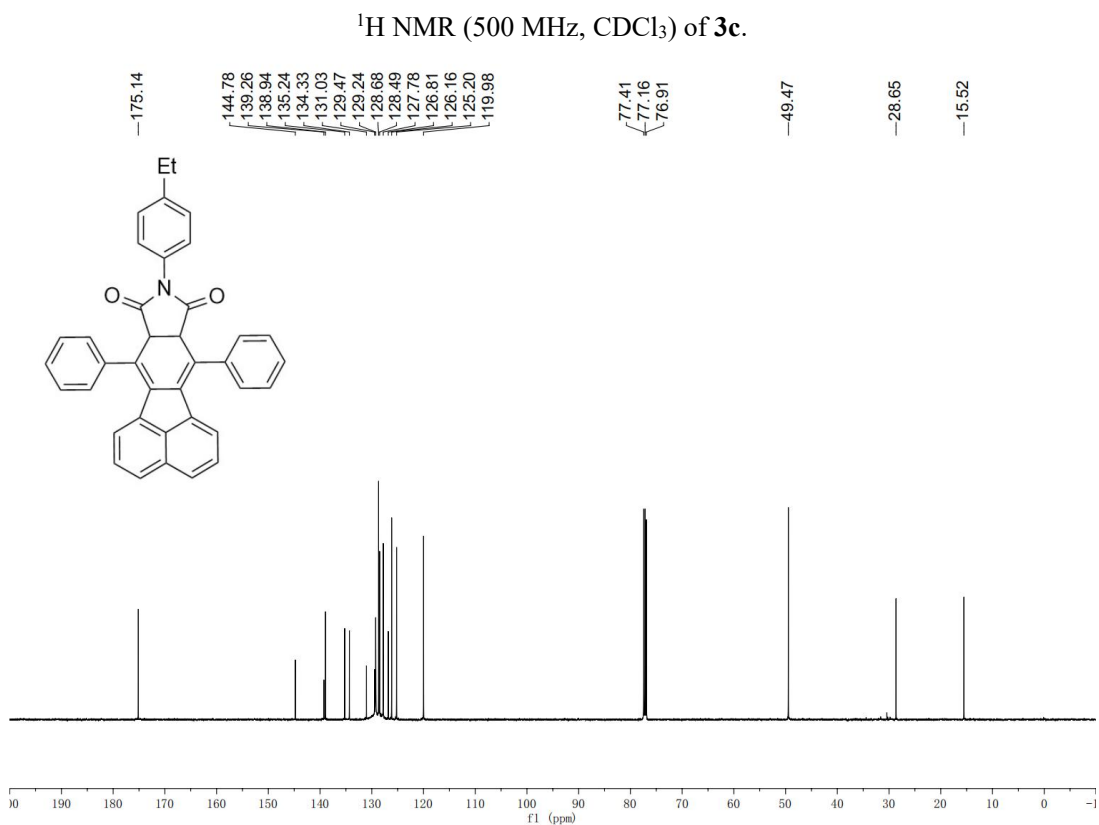
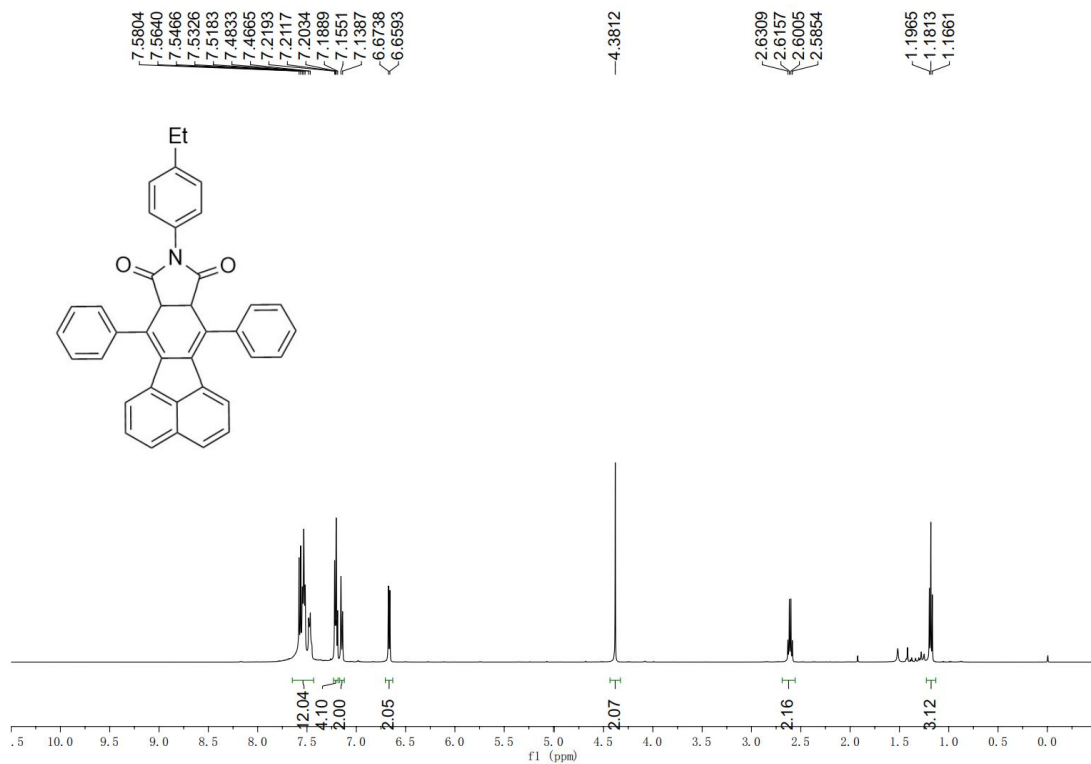
^{13}C NMR (125 MHz, CDCl_3) of **3a**.

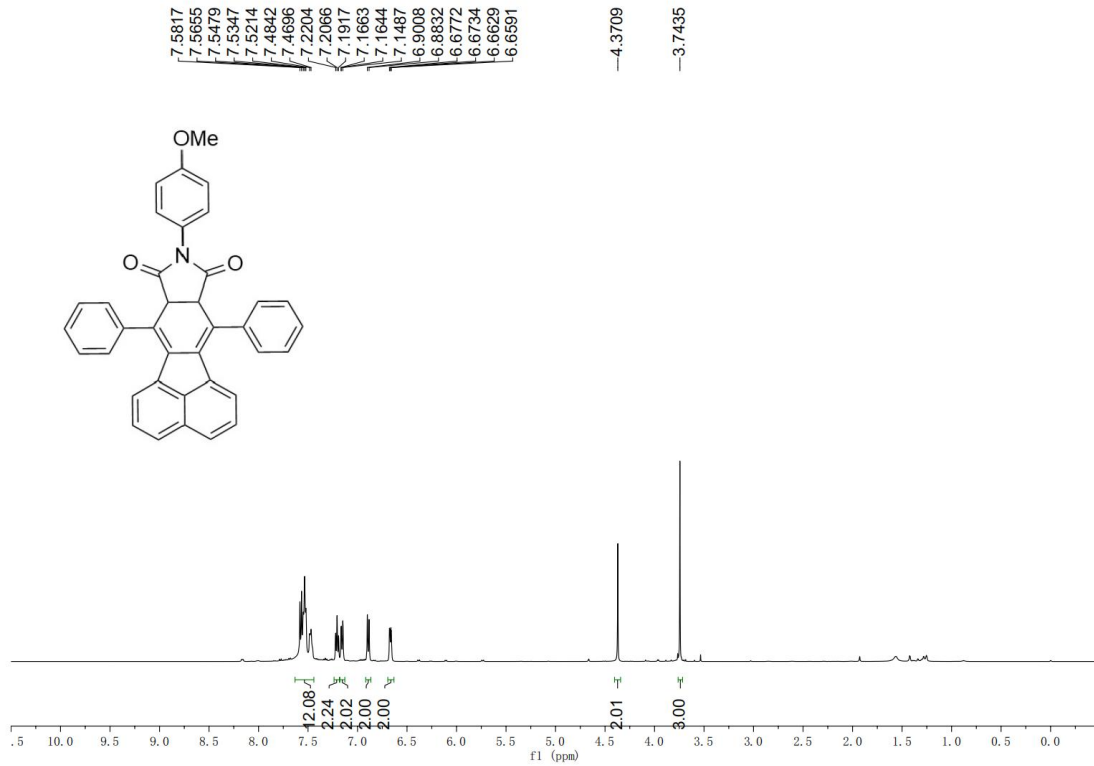


¹H NMR (500 MHz, CDCl₃) of **3b**.

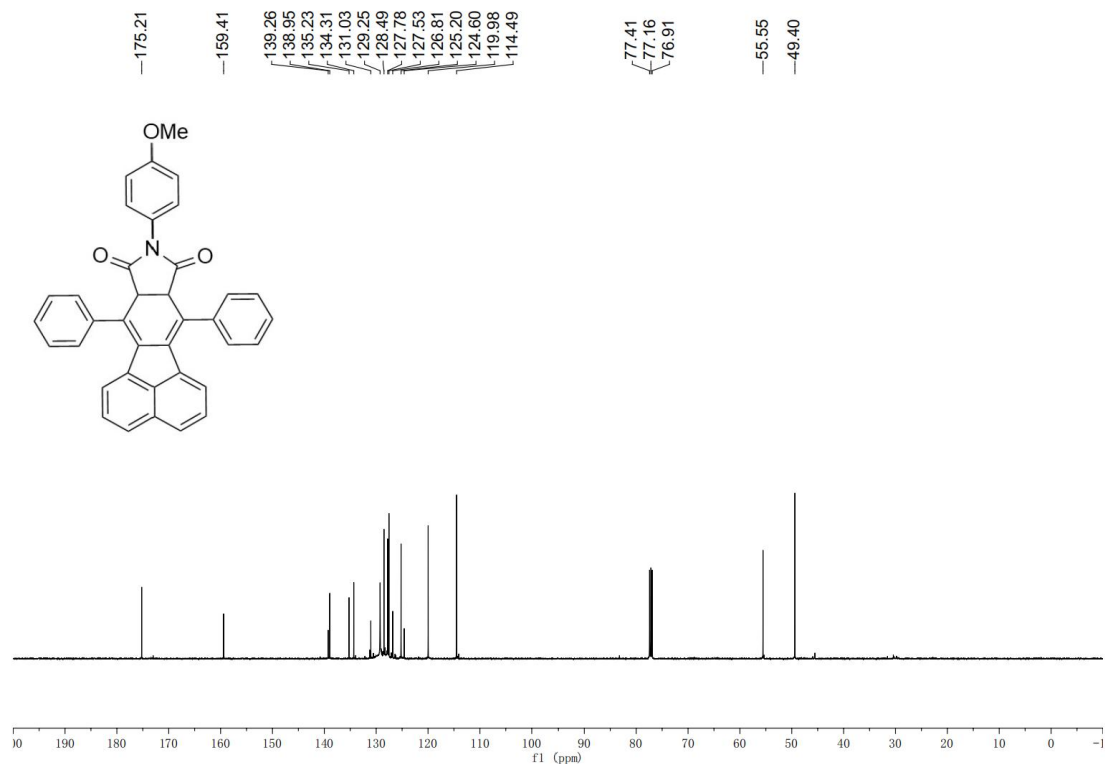


¹³C NMR (125 MHz, CDCl₃) of **3b**.

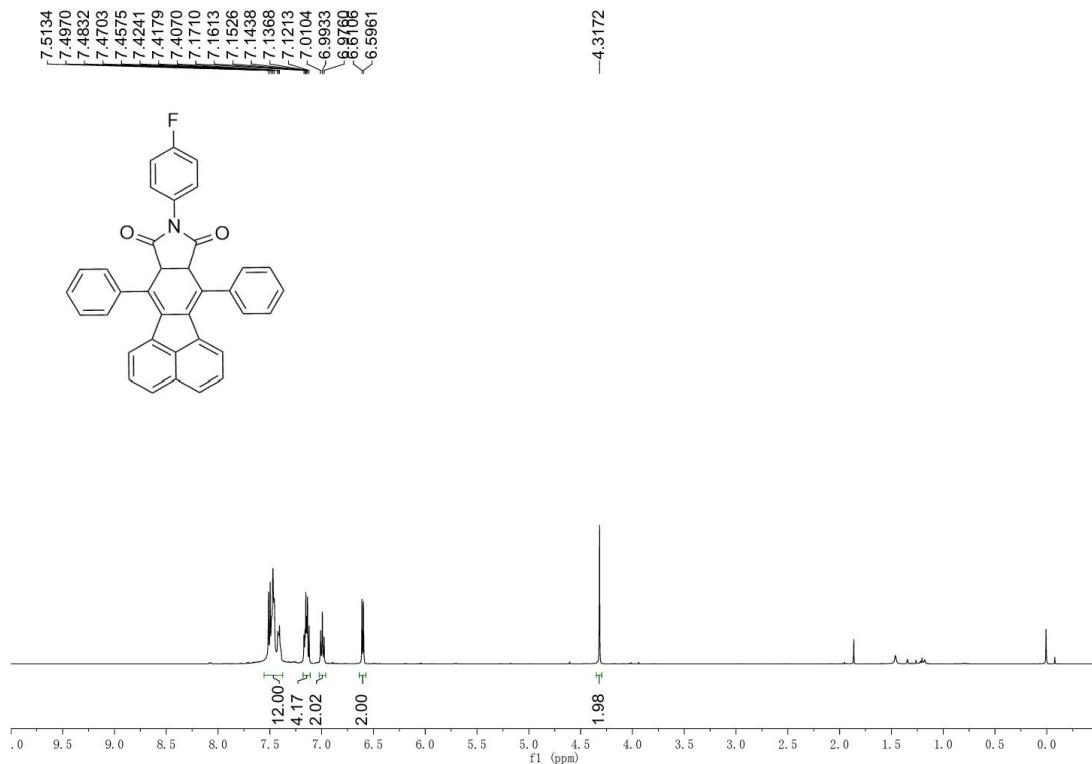




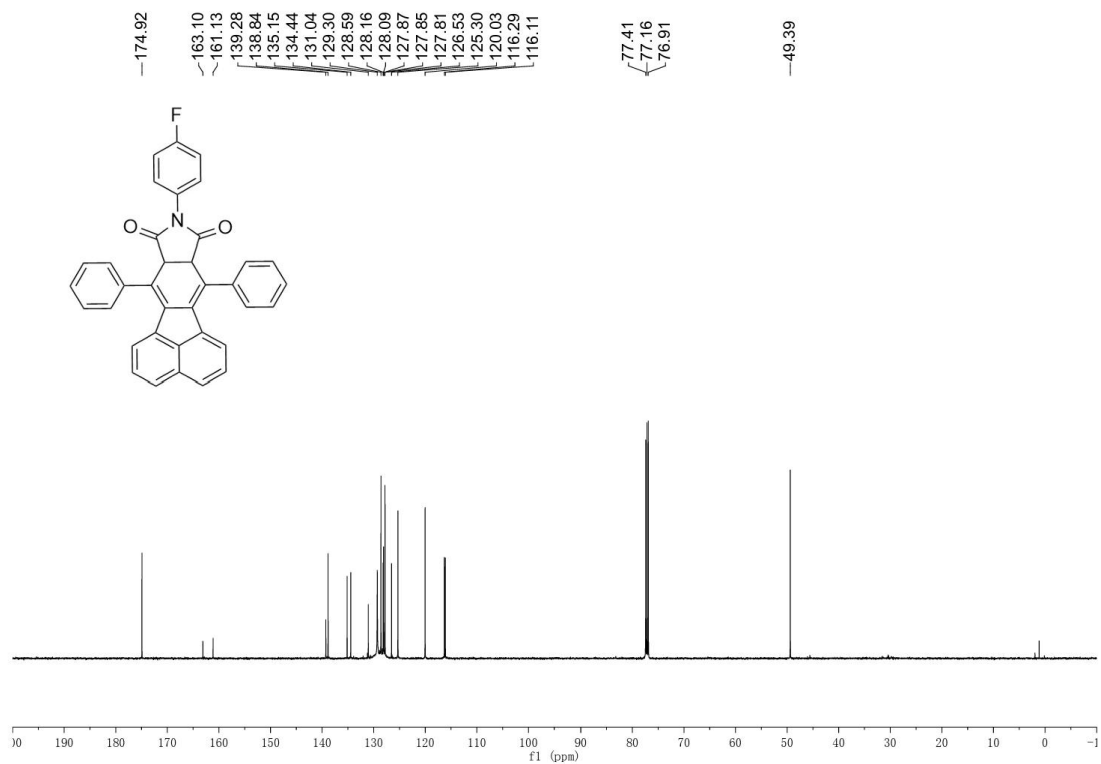
¹H NMR (500 MHz, CDCl₃) of **3d**.



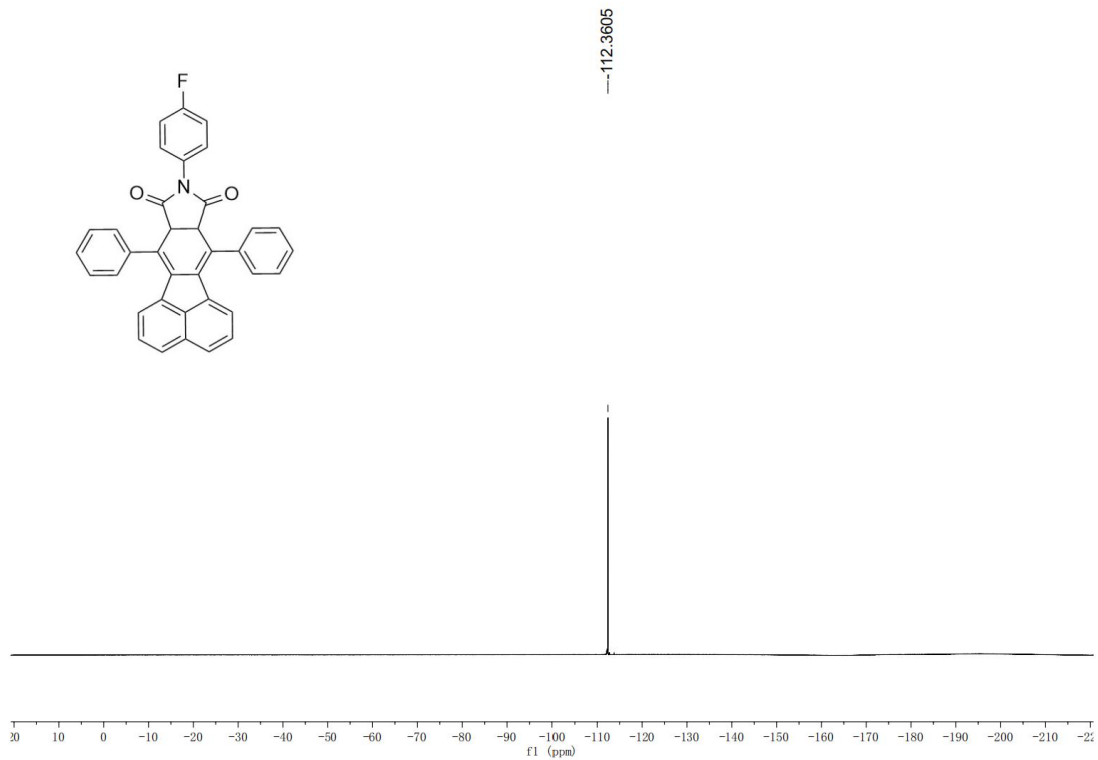
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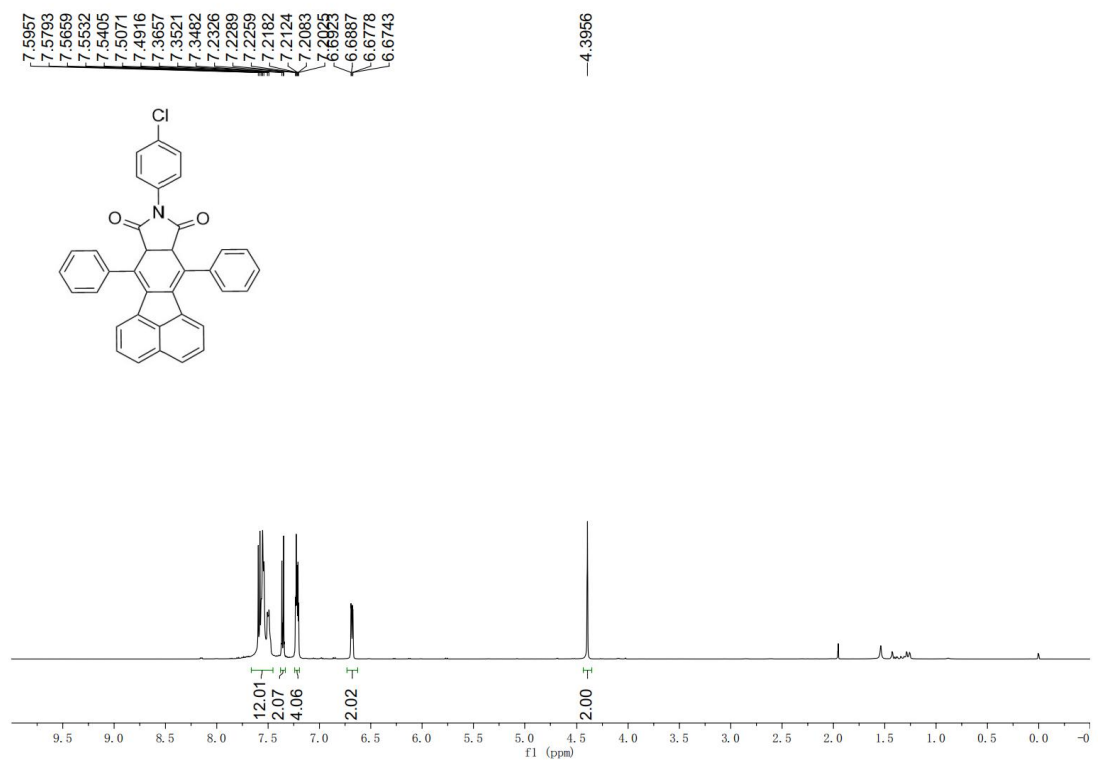
^1H NMR (500 MHz, CDCl_3) of **3e**.



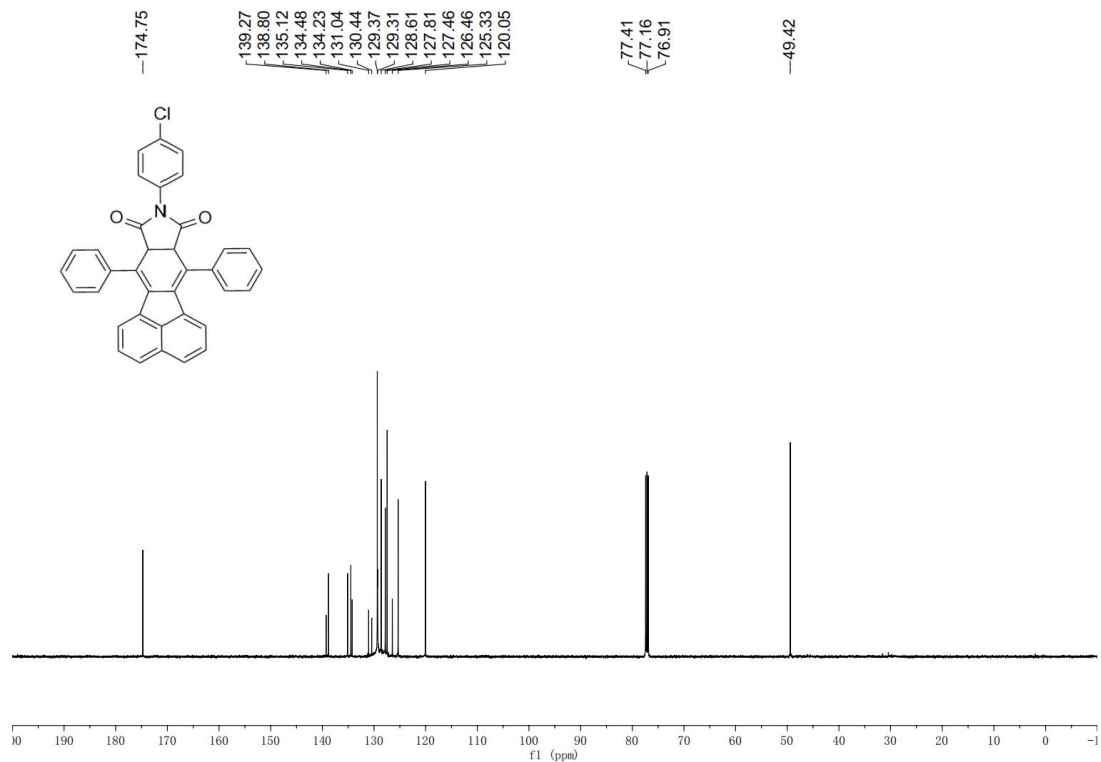
^{13}C NMR (125 MHz, CDCl_3) of **3e**.



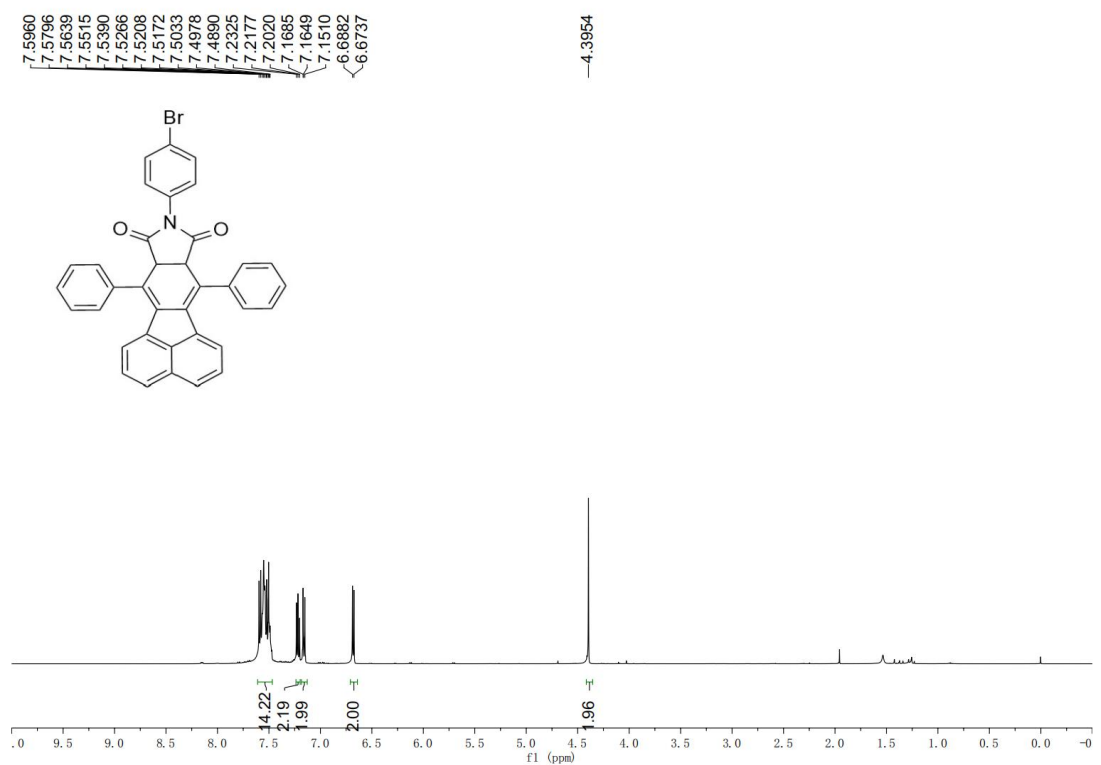
^{19}F NMR (471 MHz, CDCl_3) of **3e**.



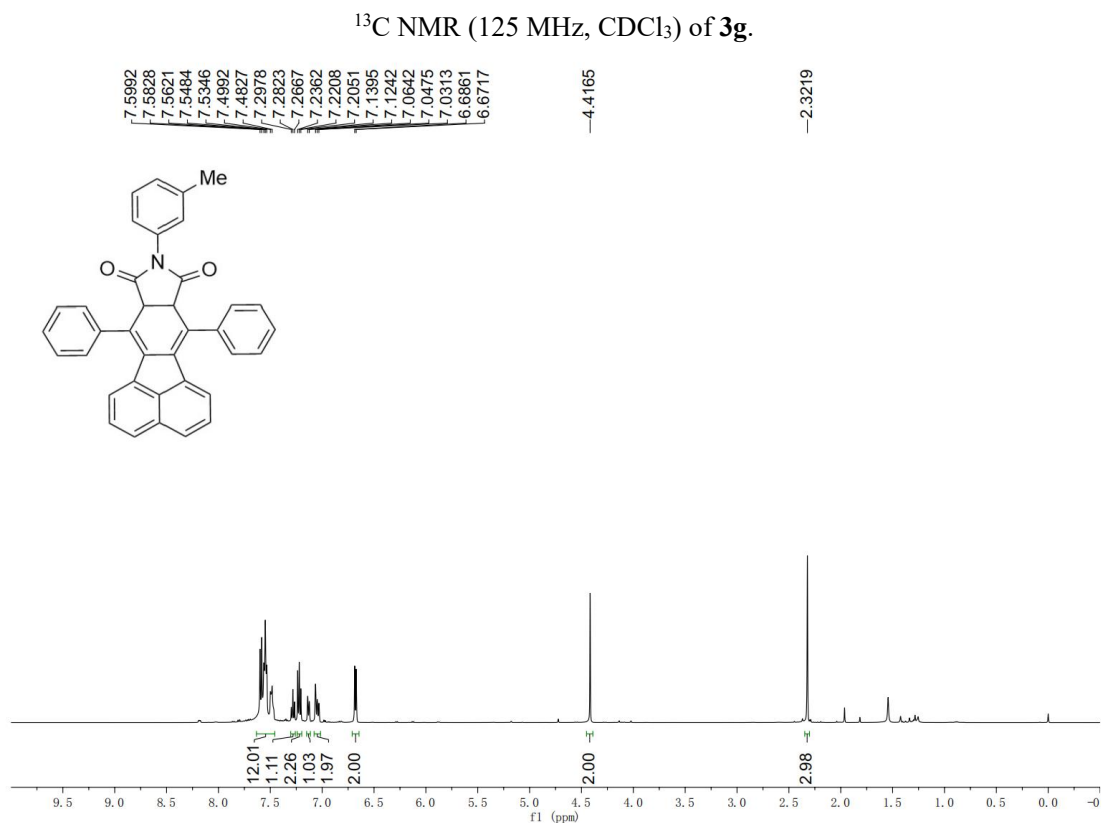
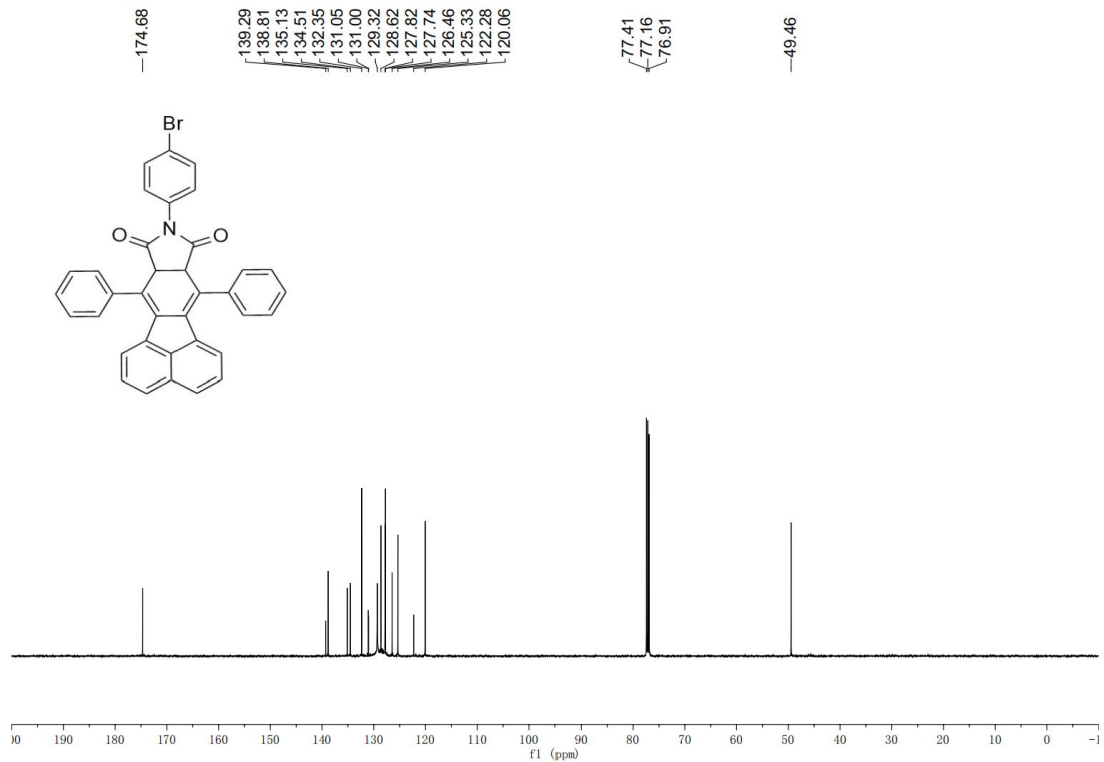
^1H NMR (500 MHz, CDCl_3) of **3f**.



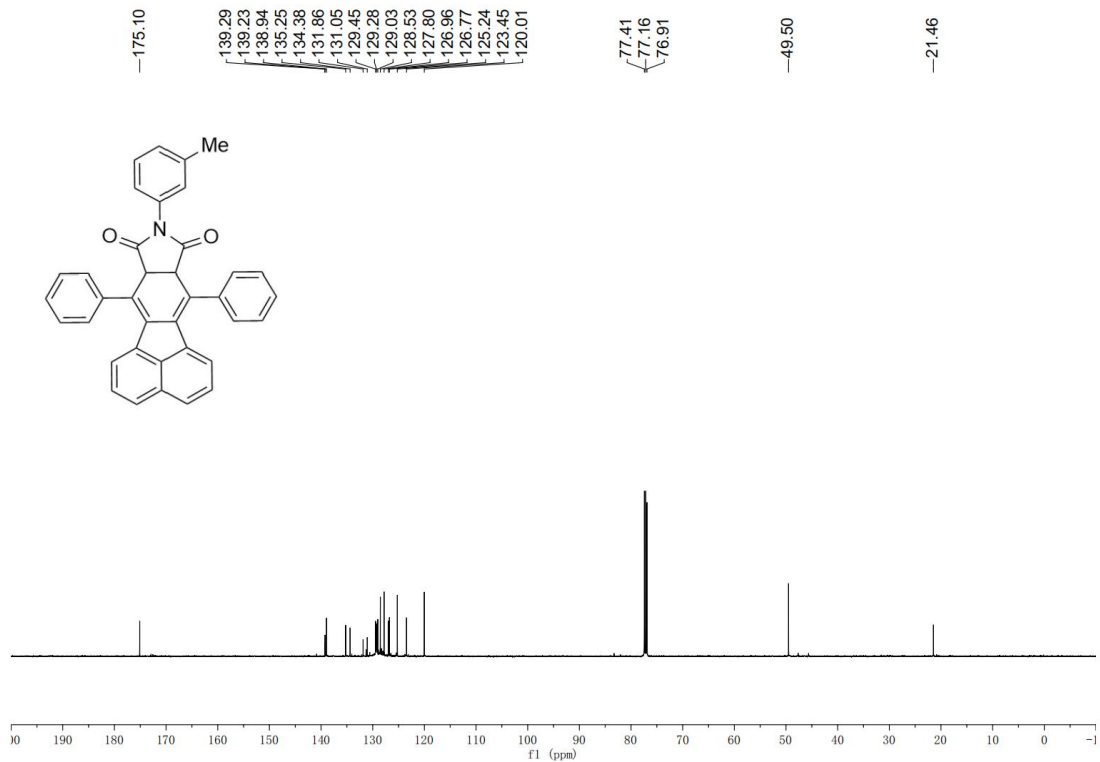
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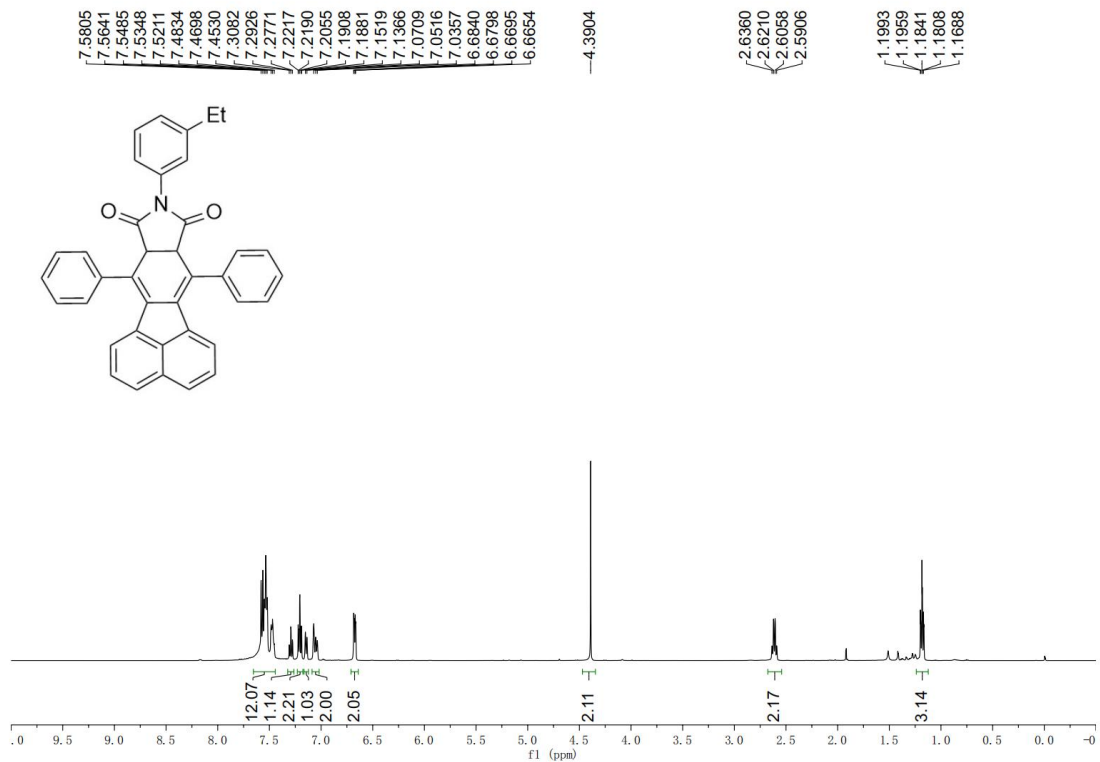
^1H NMR (500 MHz, CDCl_3) of **3g**.



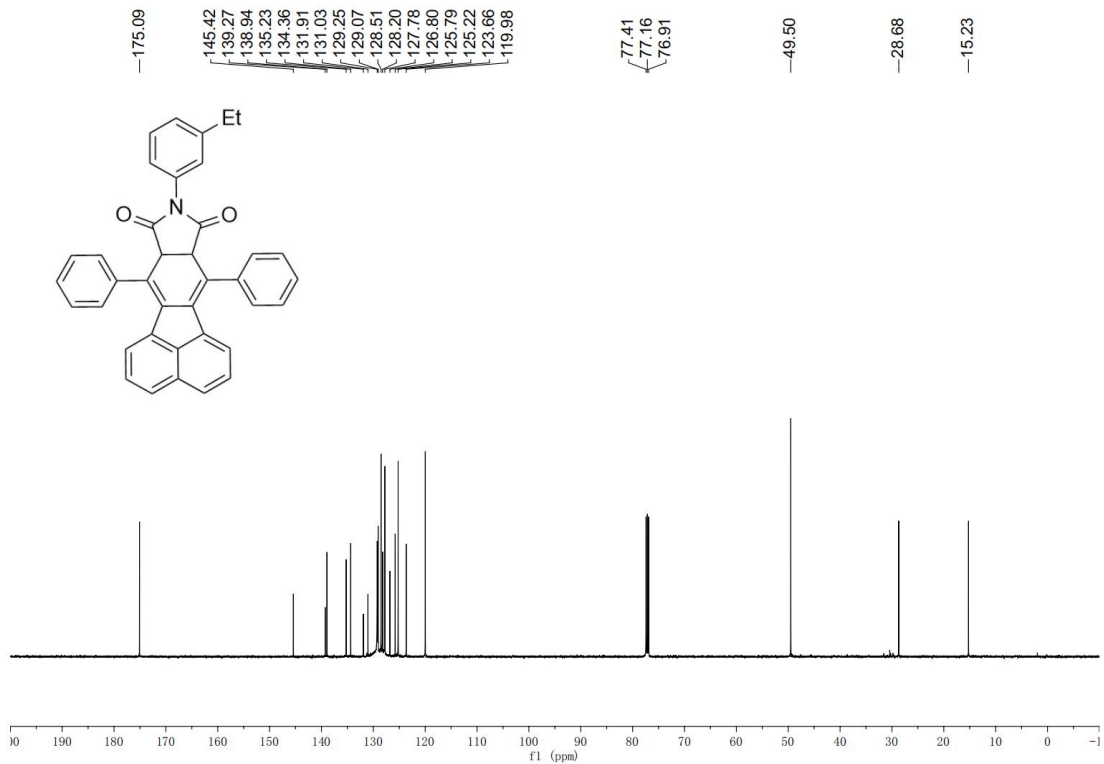
1H NMR (500 MHz, CDCl₃) of **3h**.



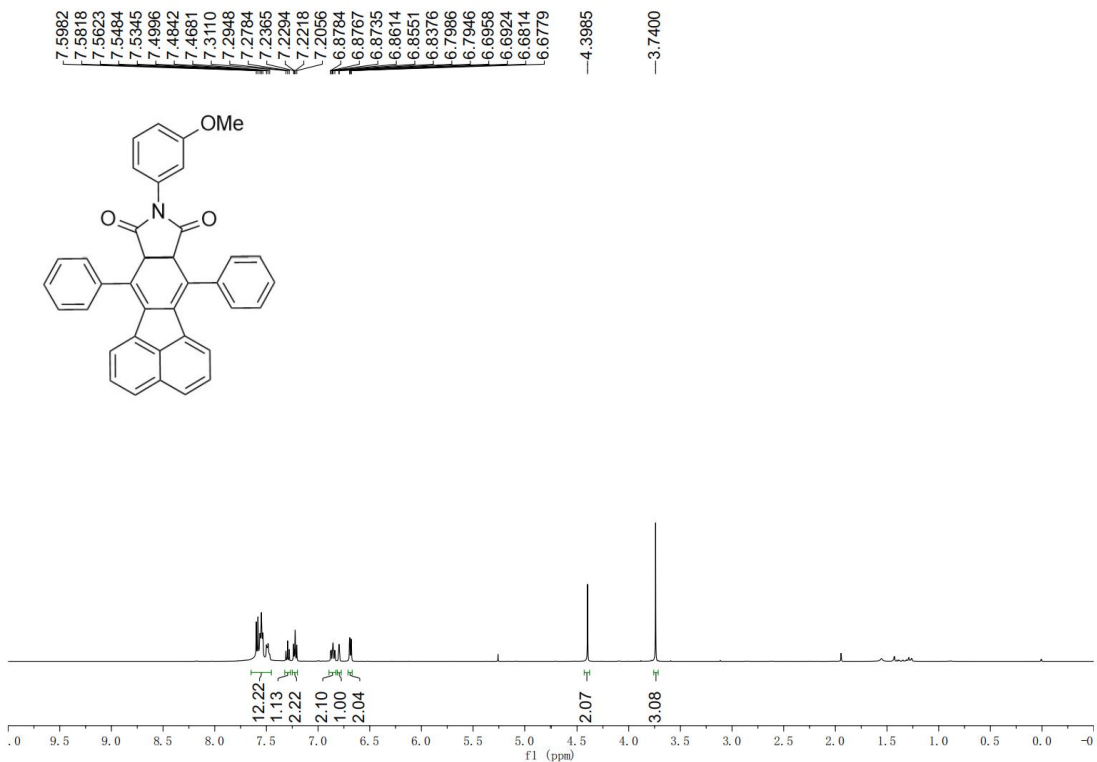
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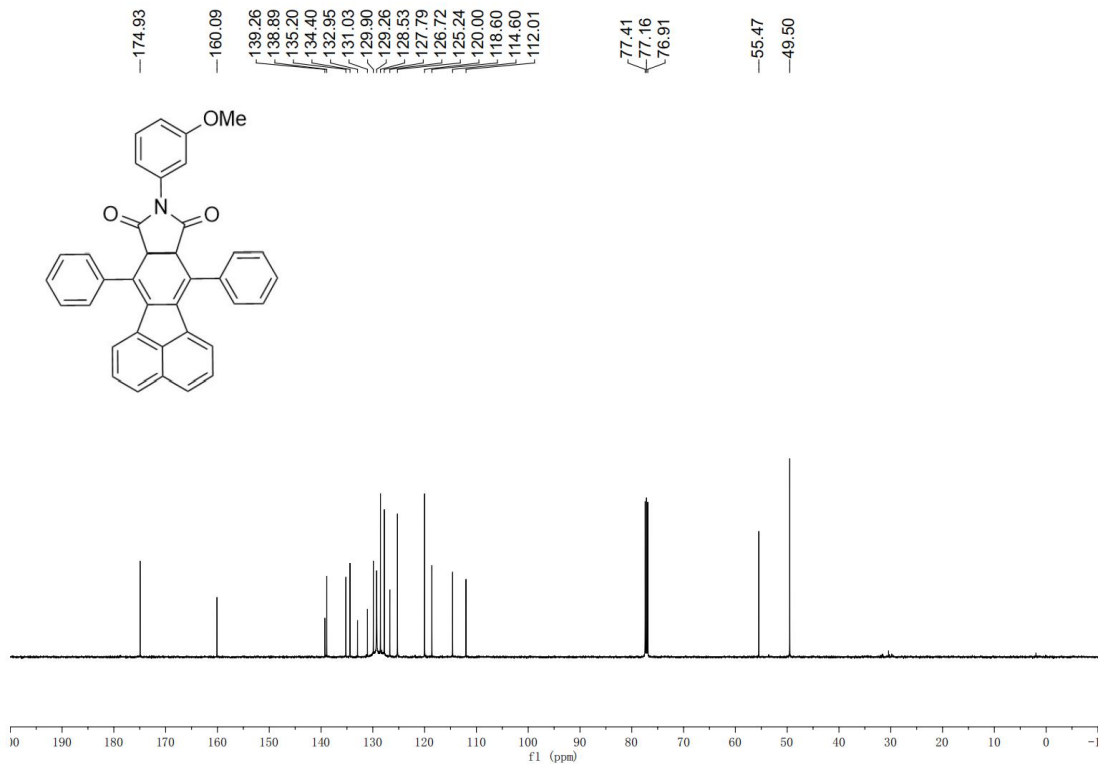
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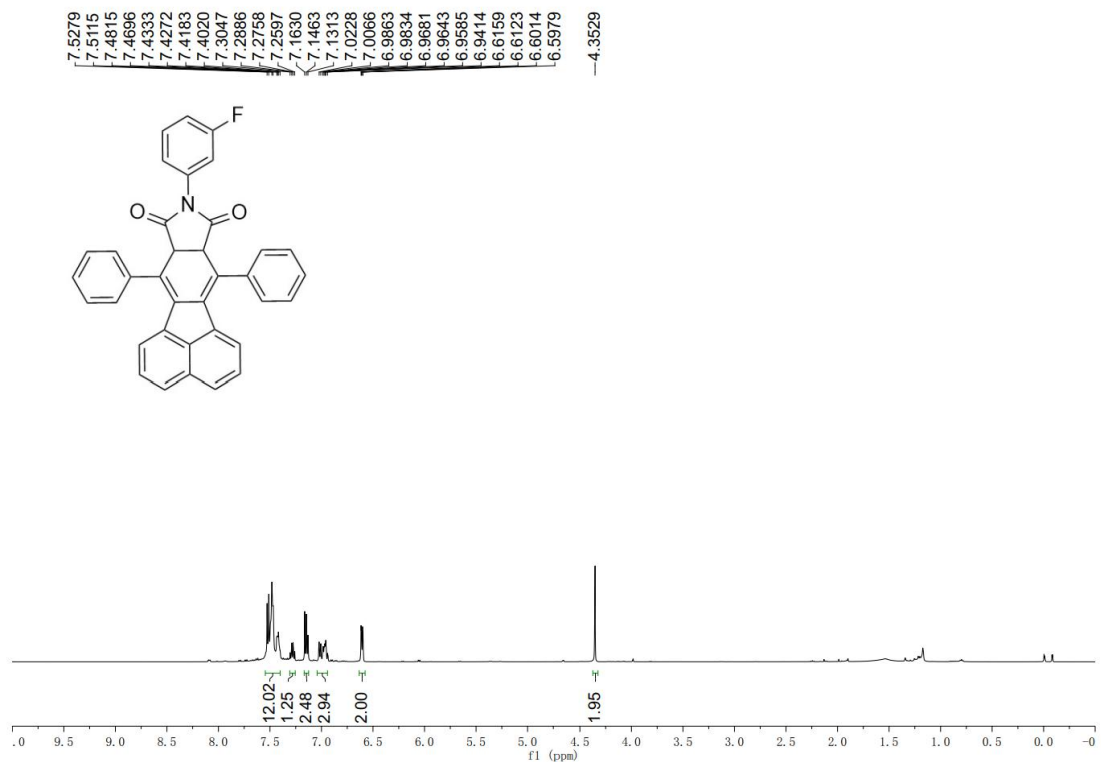
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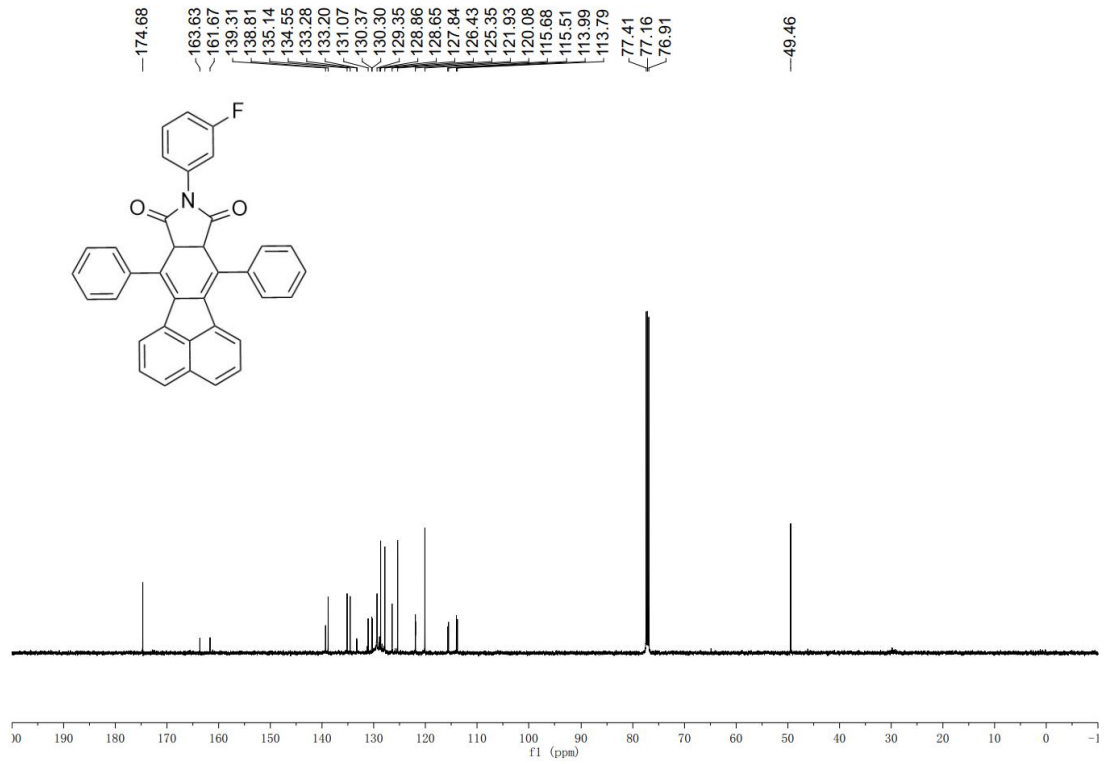
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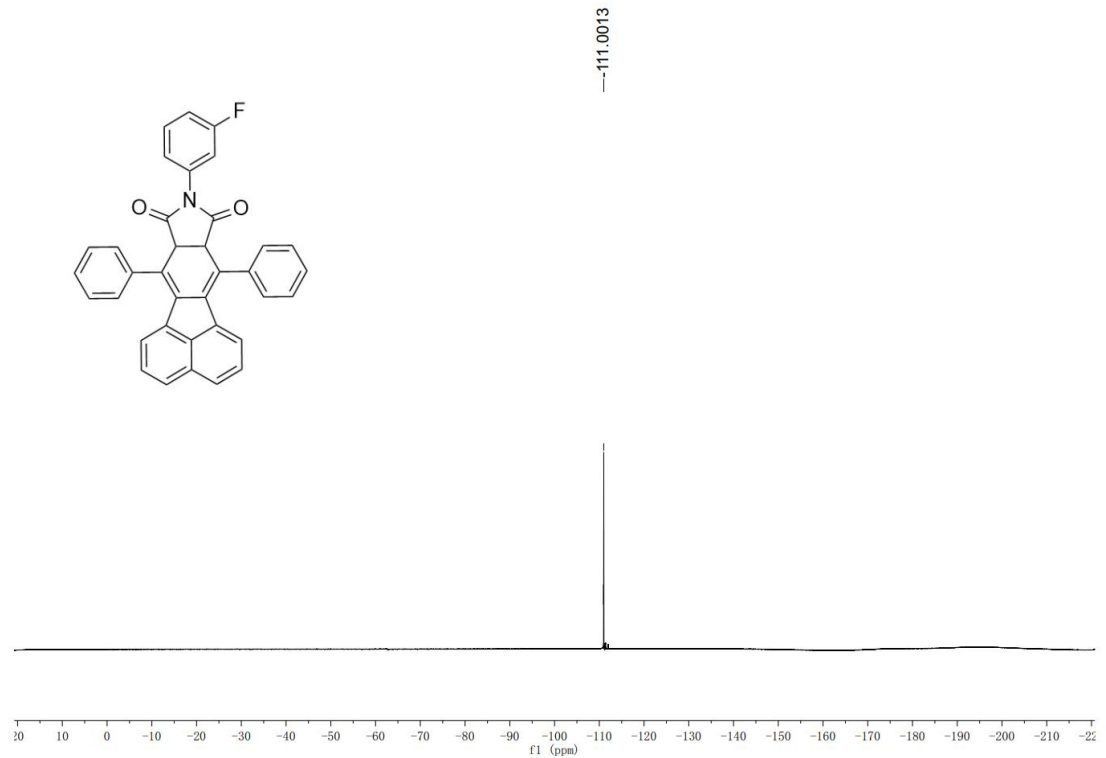
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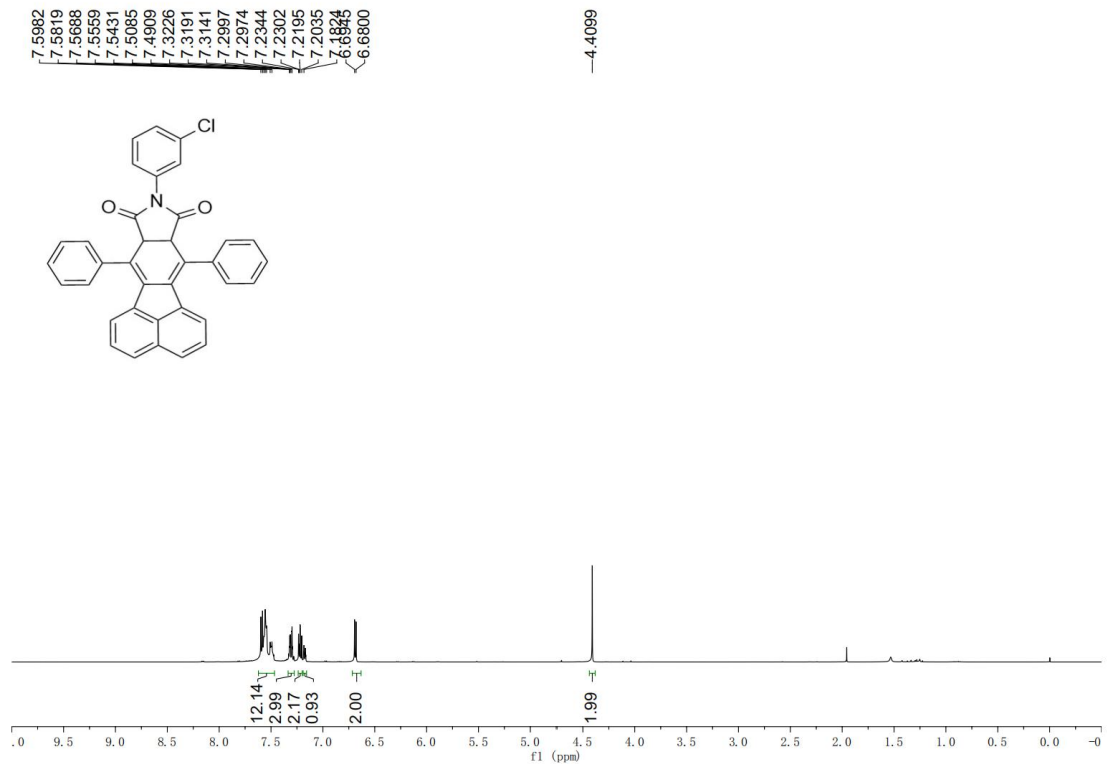
^1H NMR (500 MHz, CDCl_3) of **3k**.



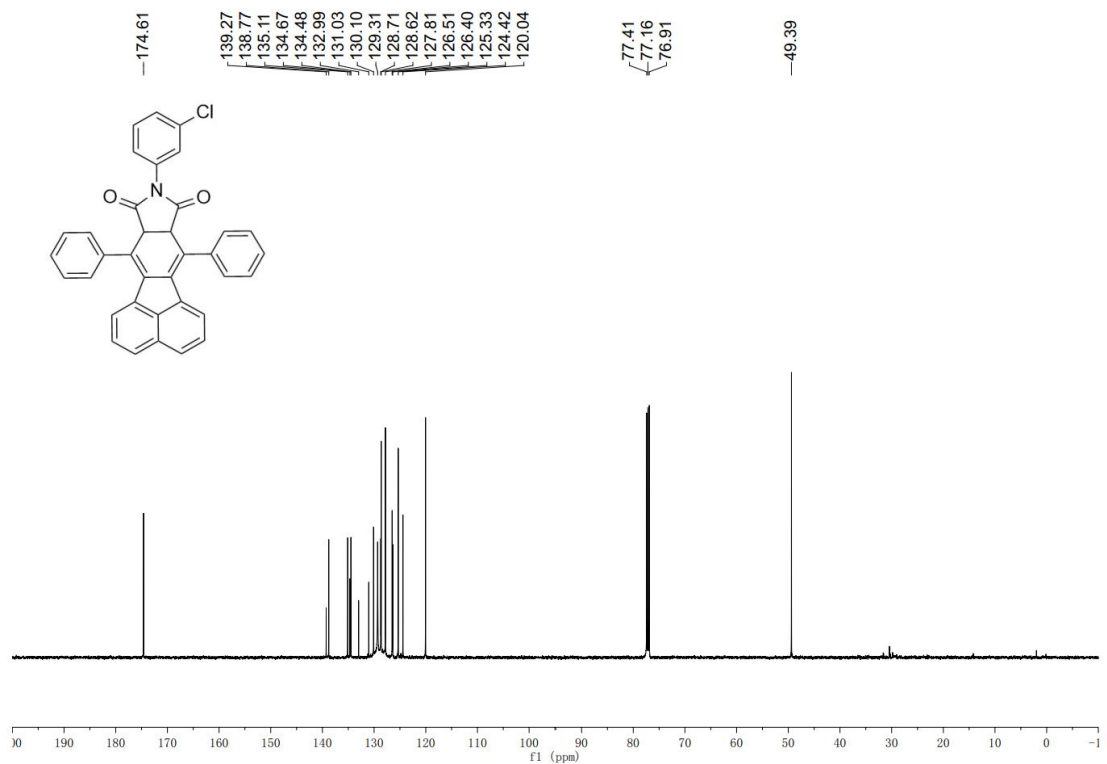
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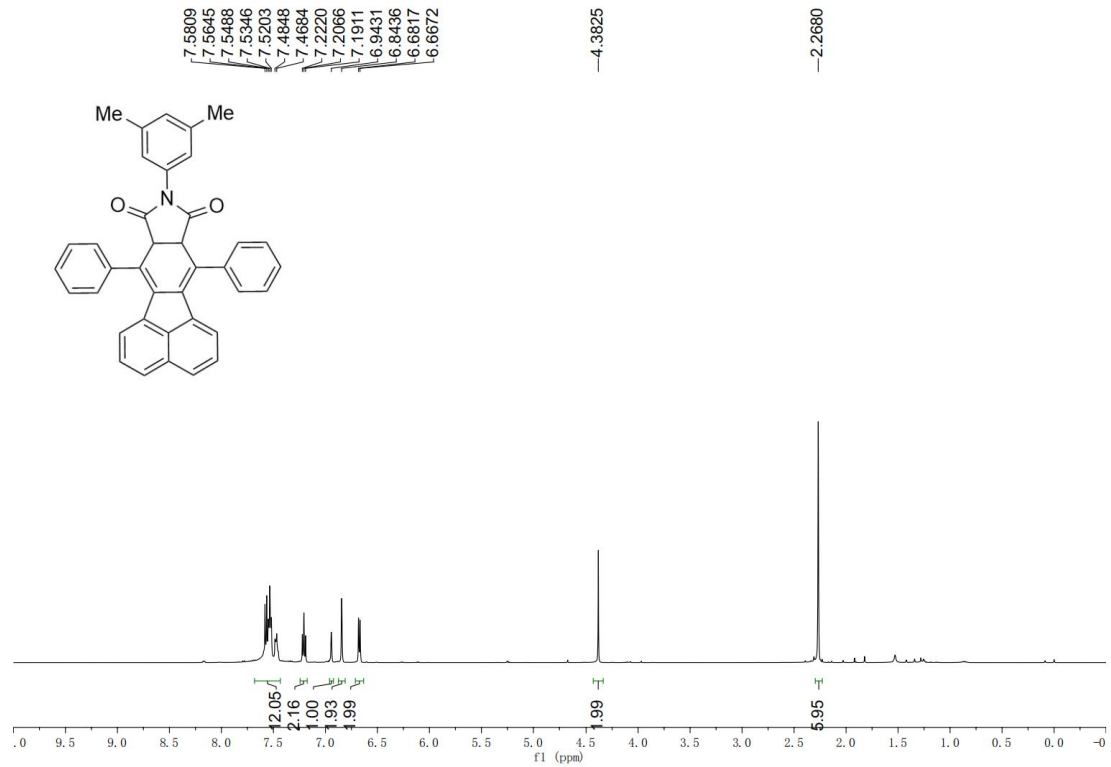
^{19}F NMR (471 MHz, CDCl_3) of 3k.



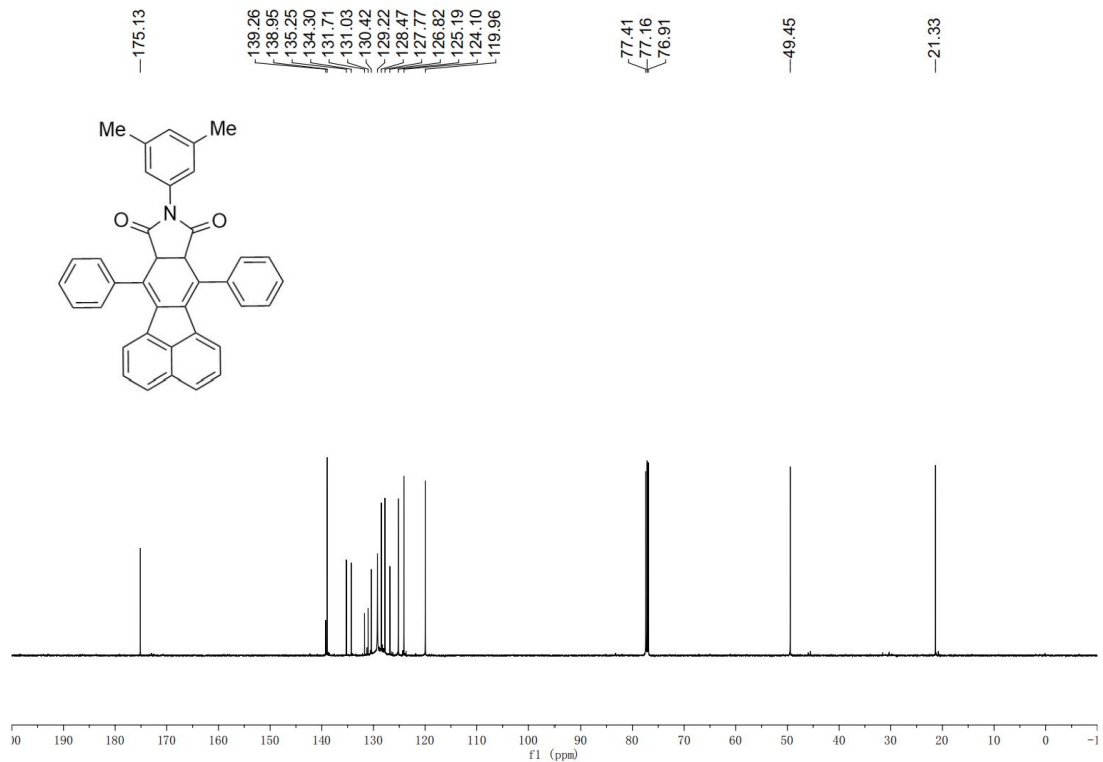
^1H NMR (500 MHz, CDCl_3) of **31**.



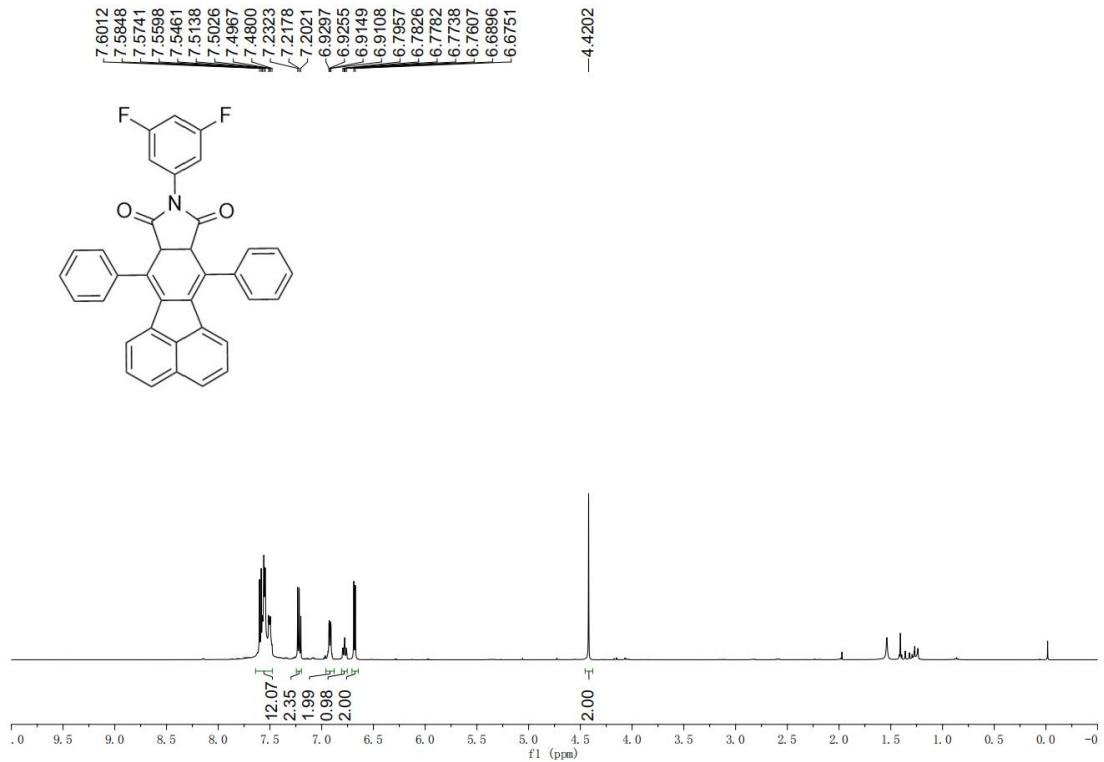
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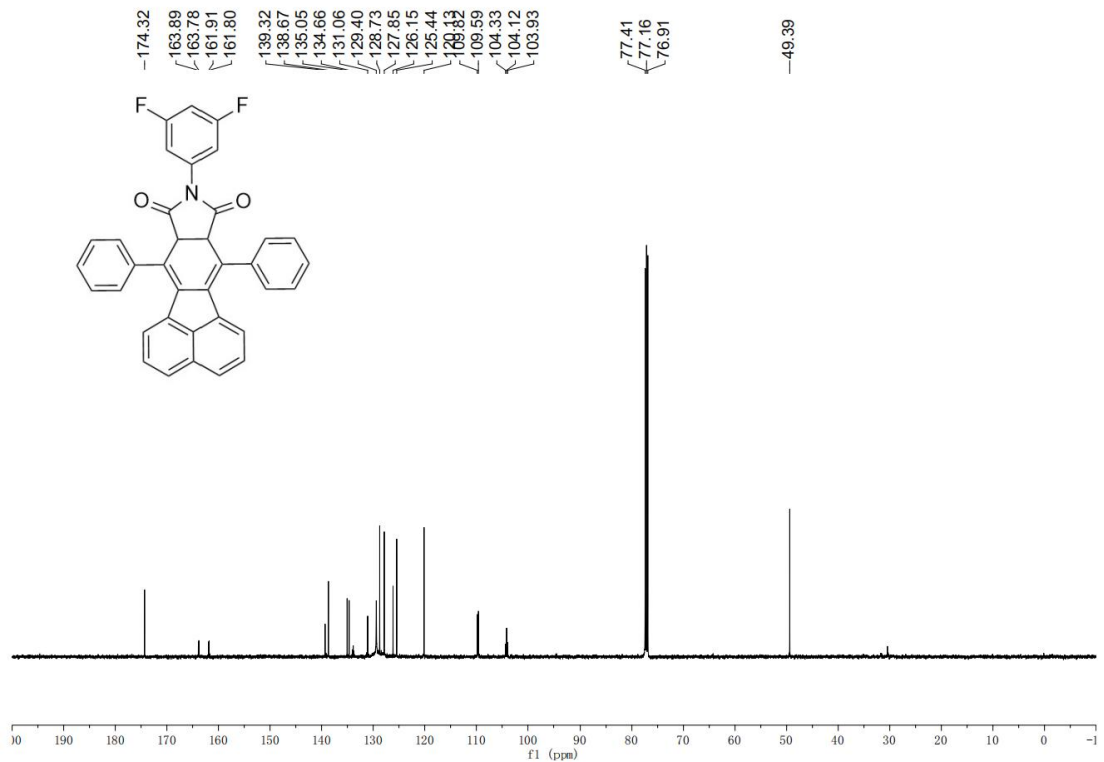
¹H NMR (500 MHz, CDCl₃) of **3m**.



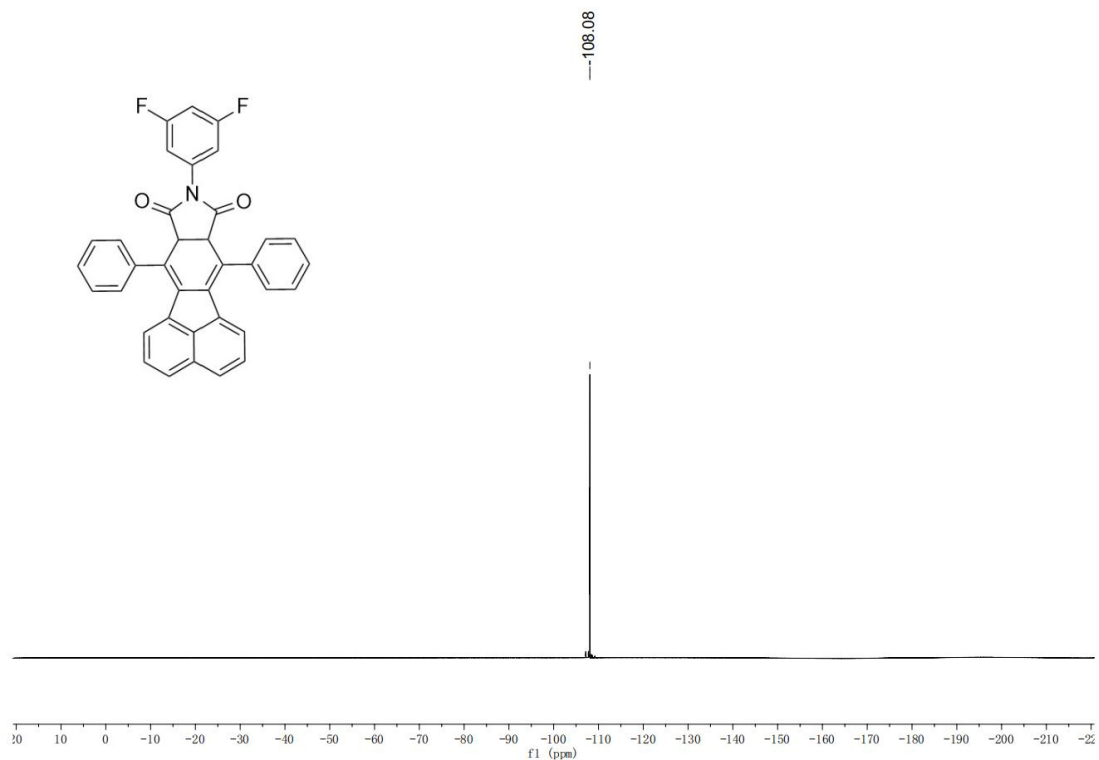
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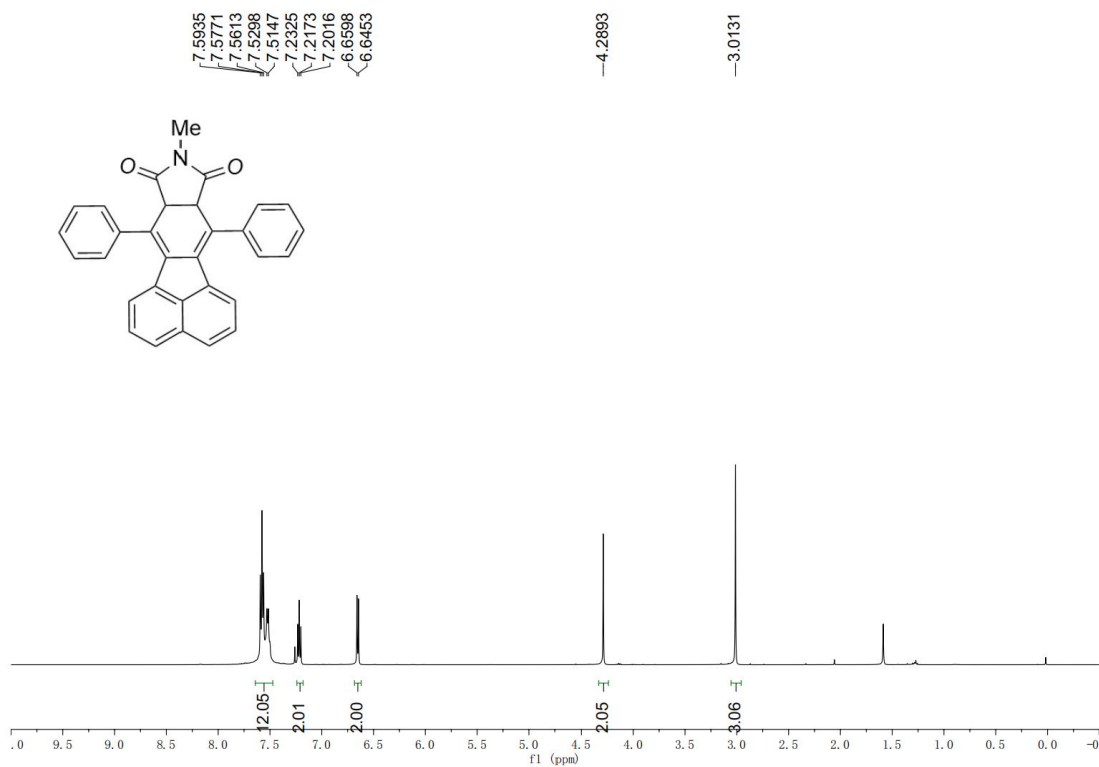
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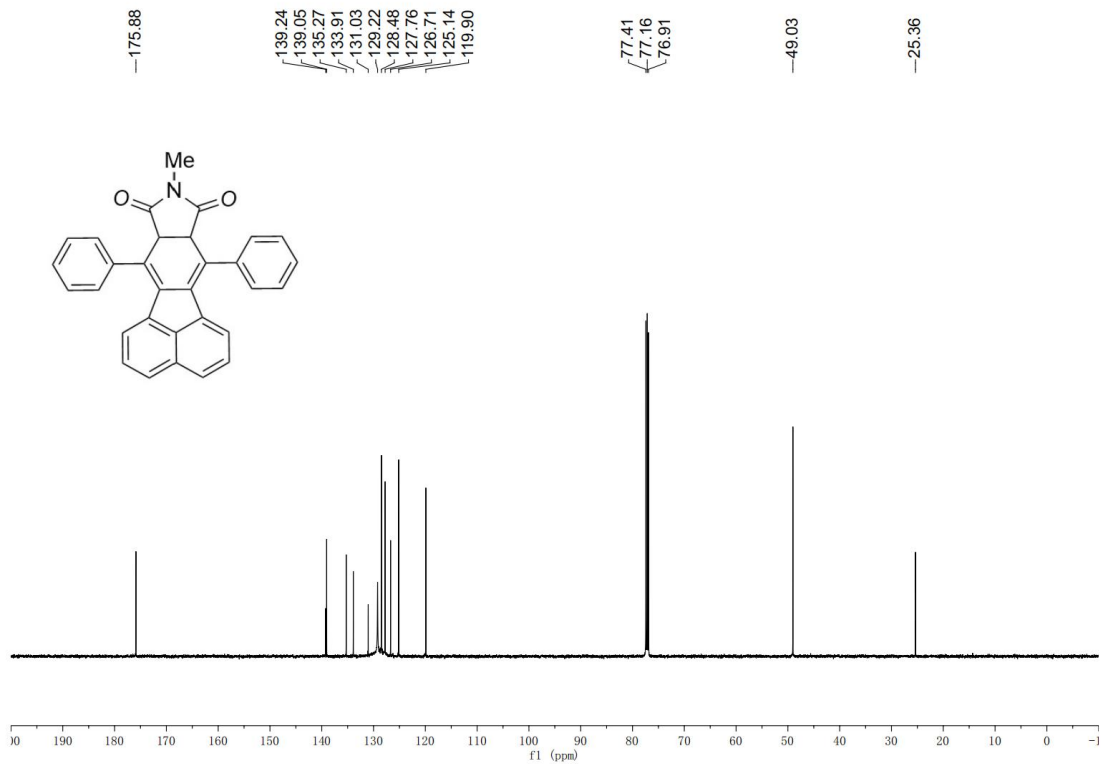
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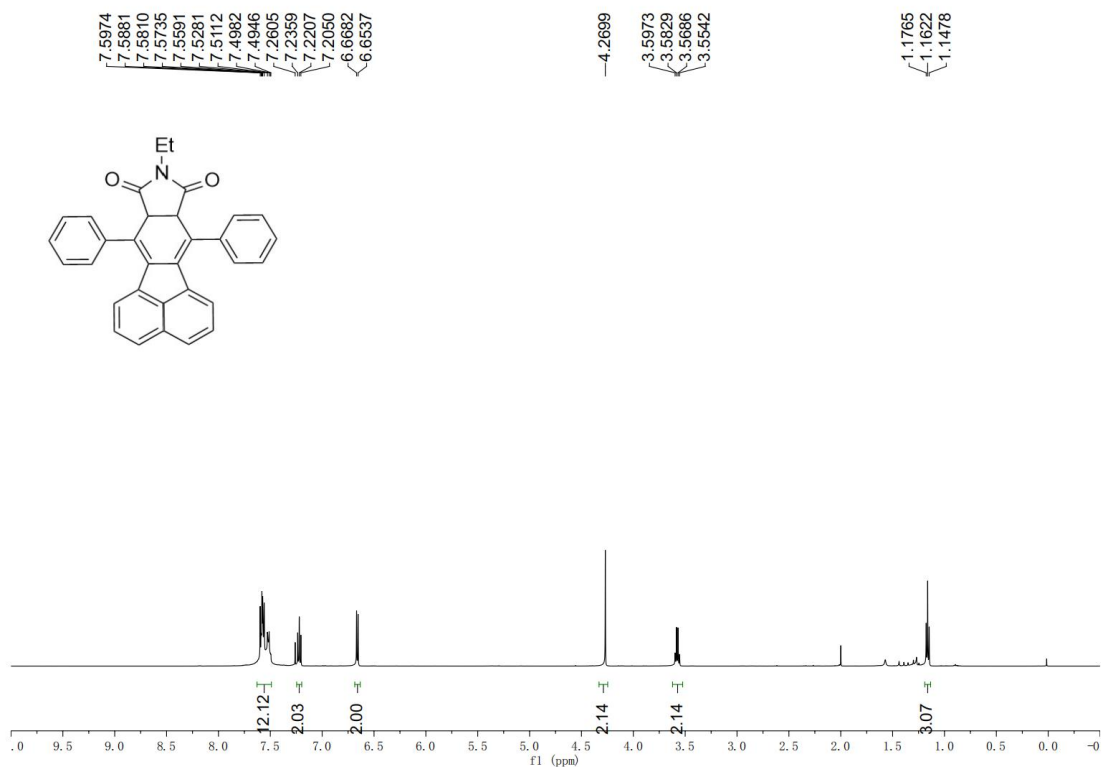
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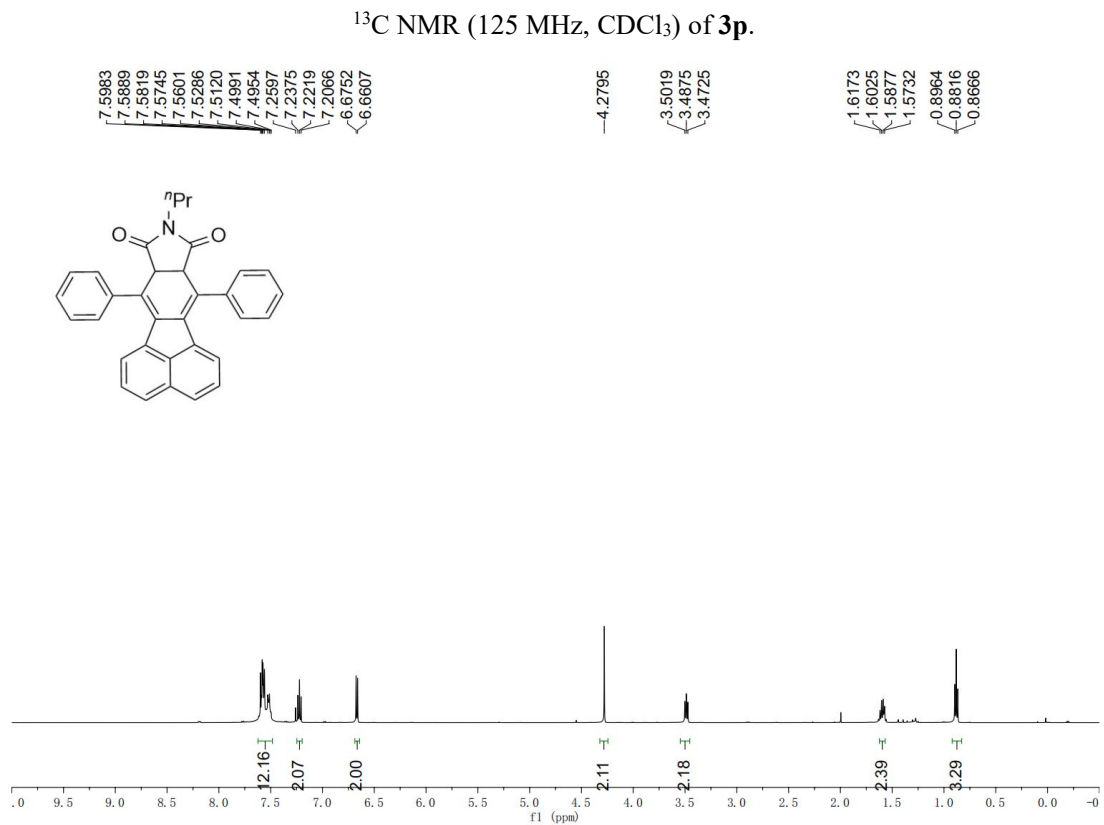
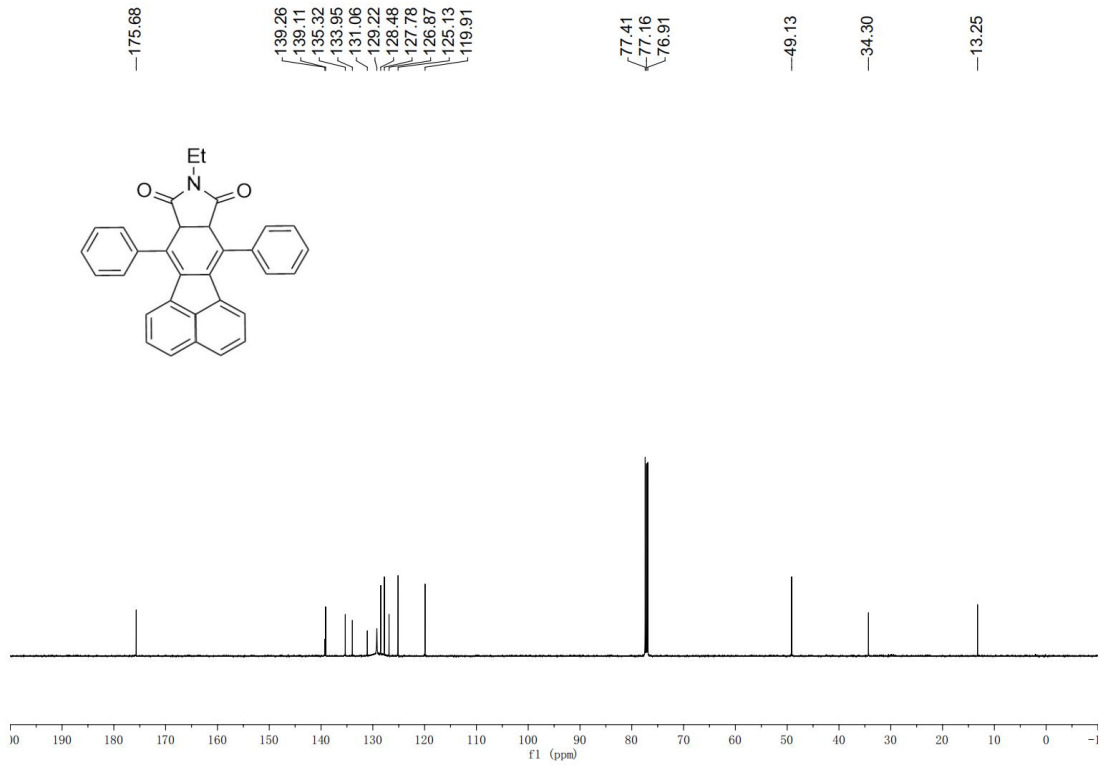
^1H NMR (500 MHz, CDCl_3) of **3o**.

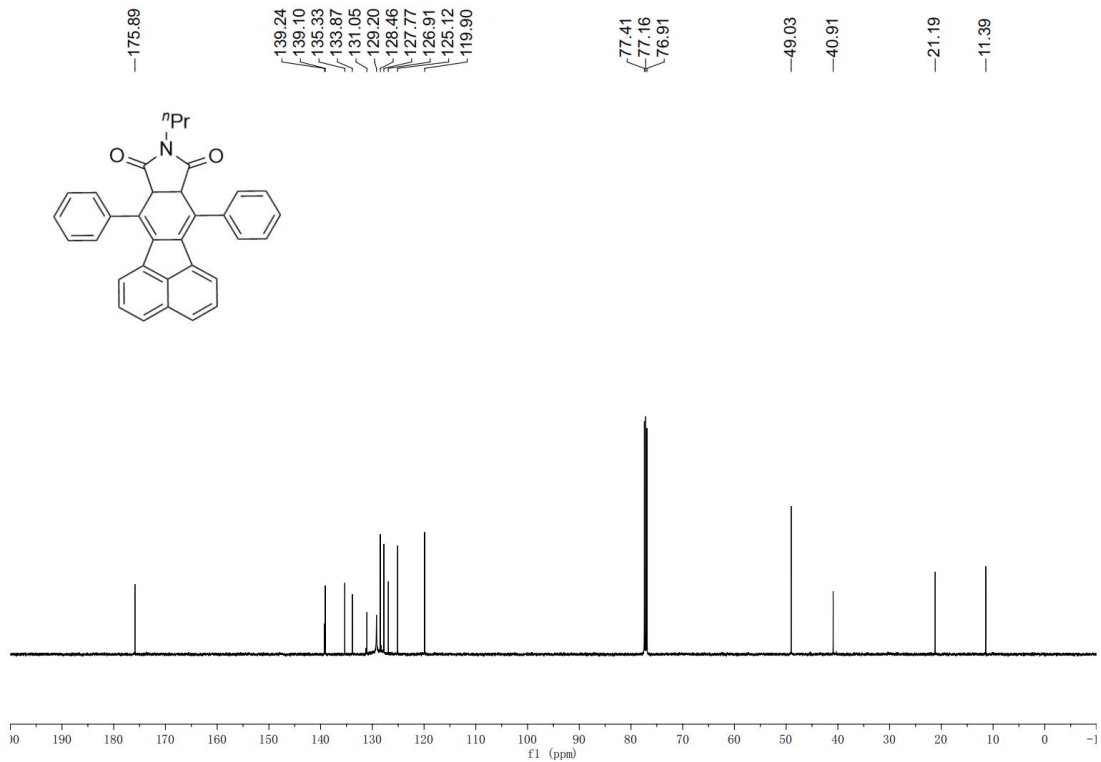


^{13}C NMR (125 MHz, CDCl_3) of **3o**.

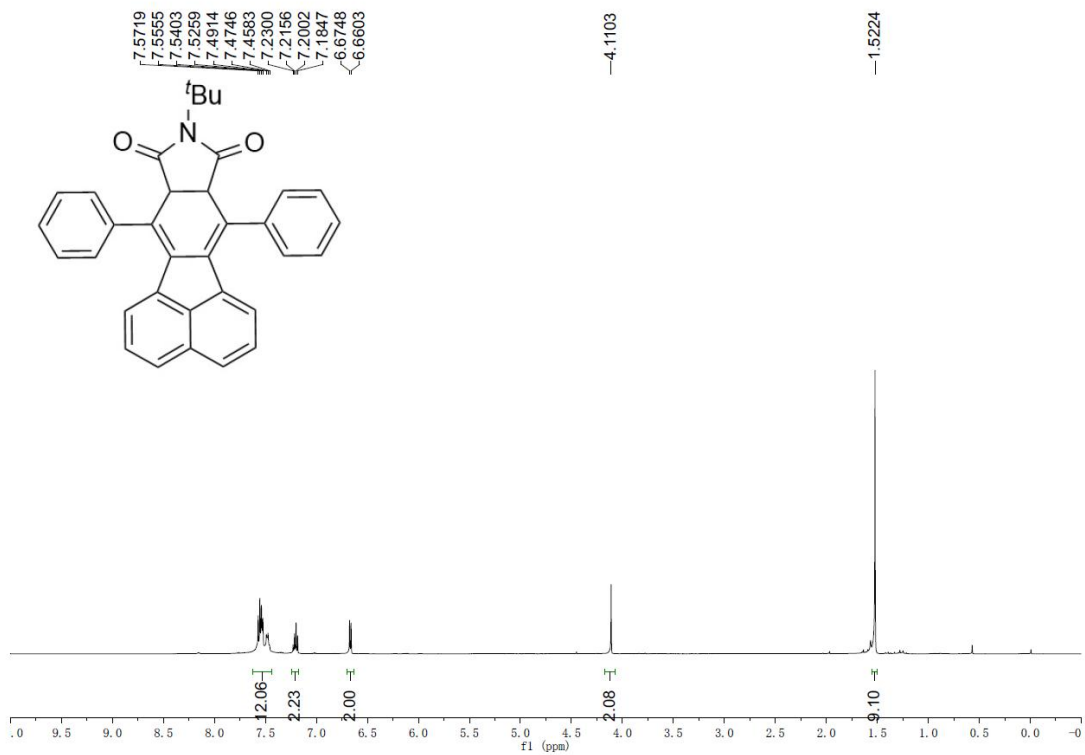


^1H NMR (500 MHz, CDCl_3) of **3p**.

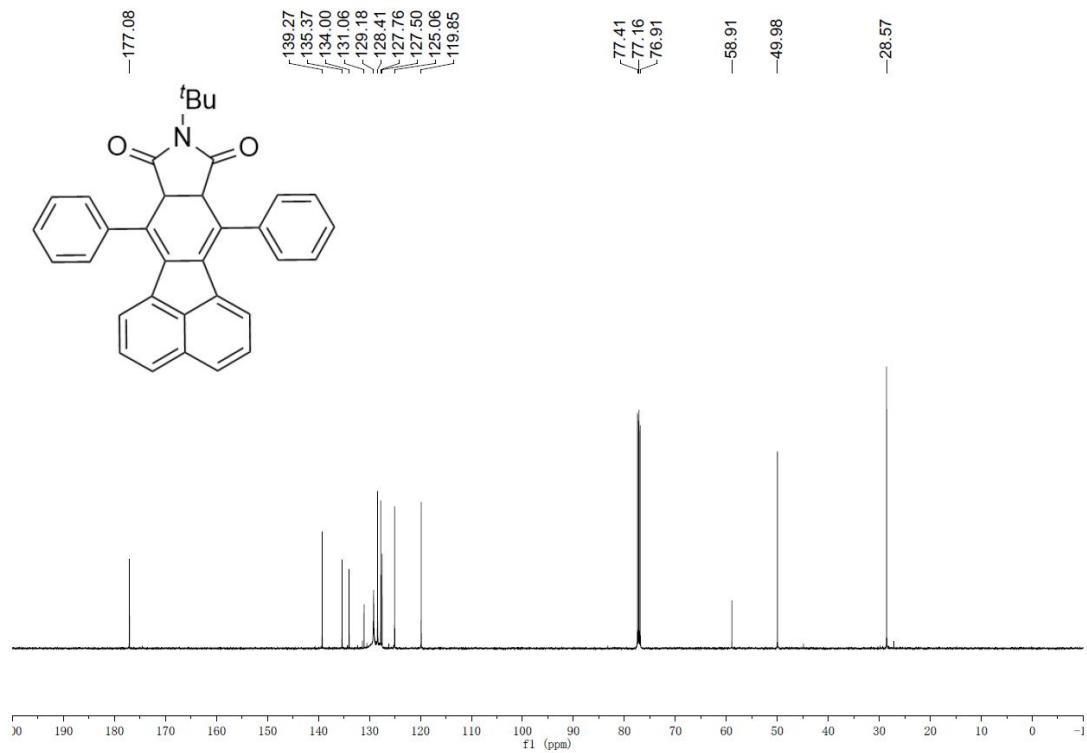




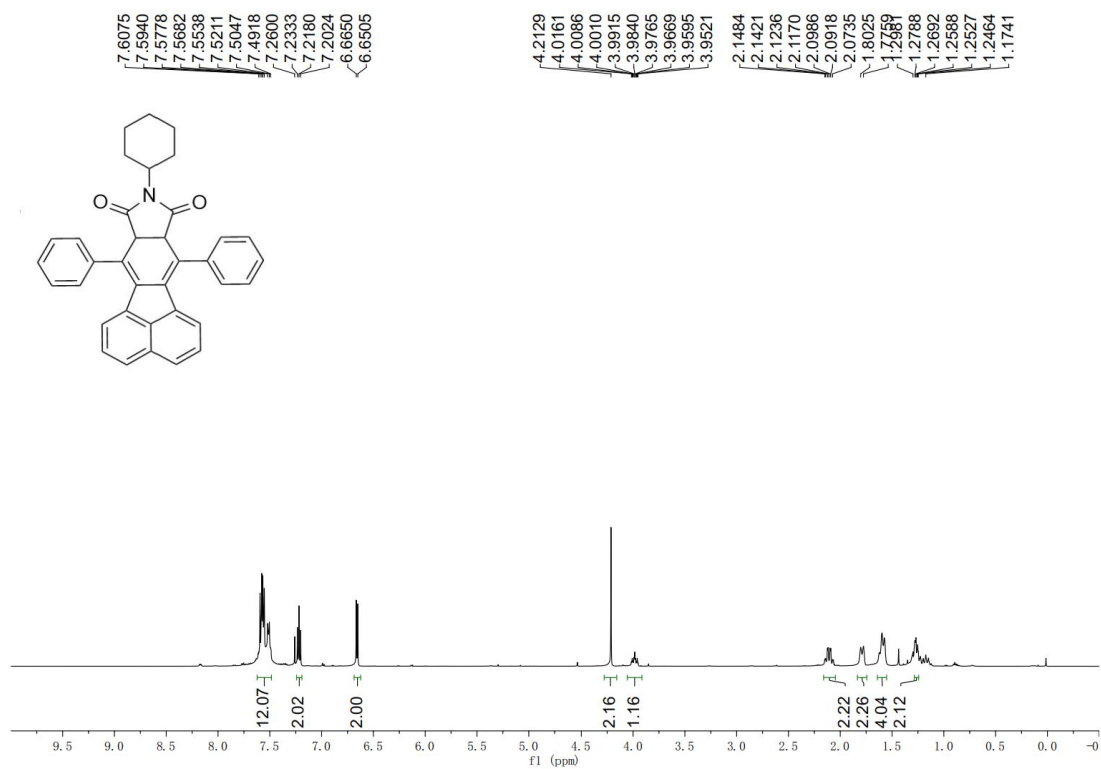
^{13}C NMR (125 MHz, CDCl_3) of **3q**.



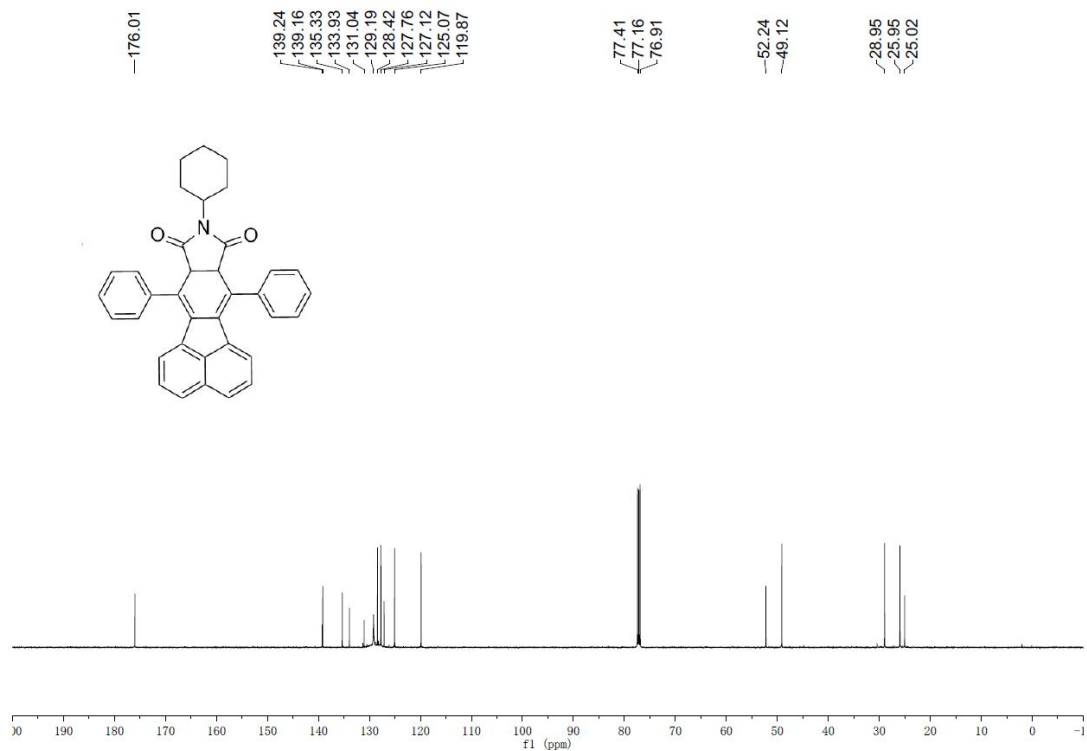
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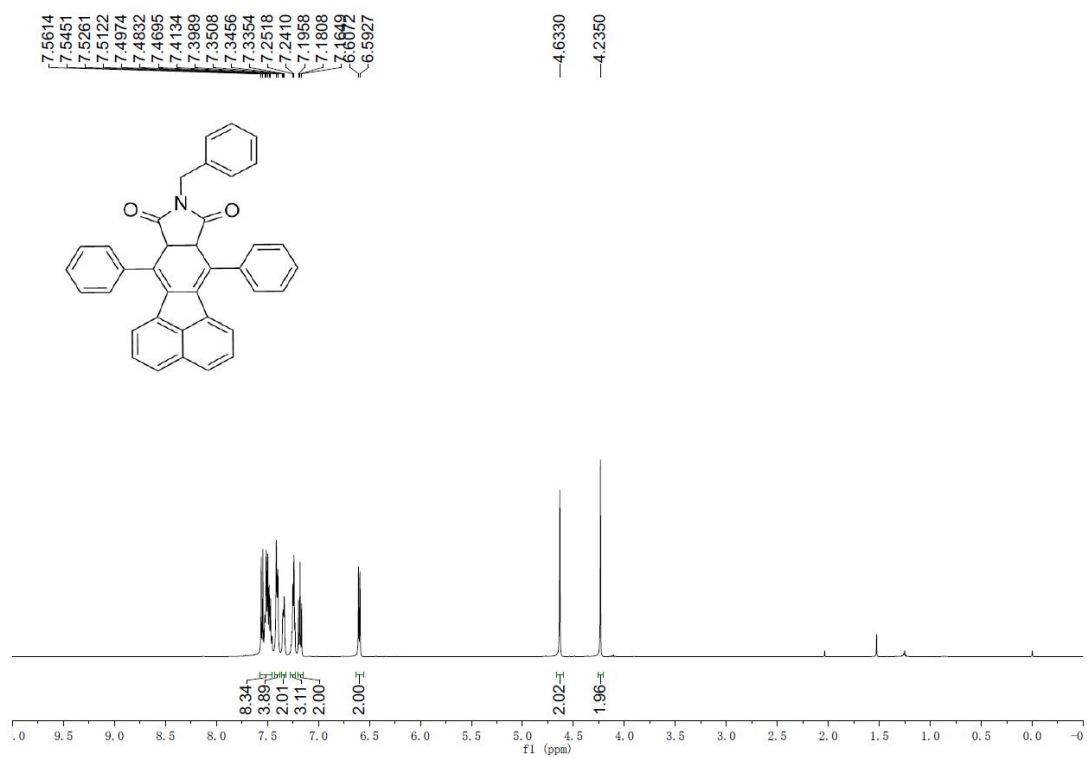
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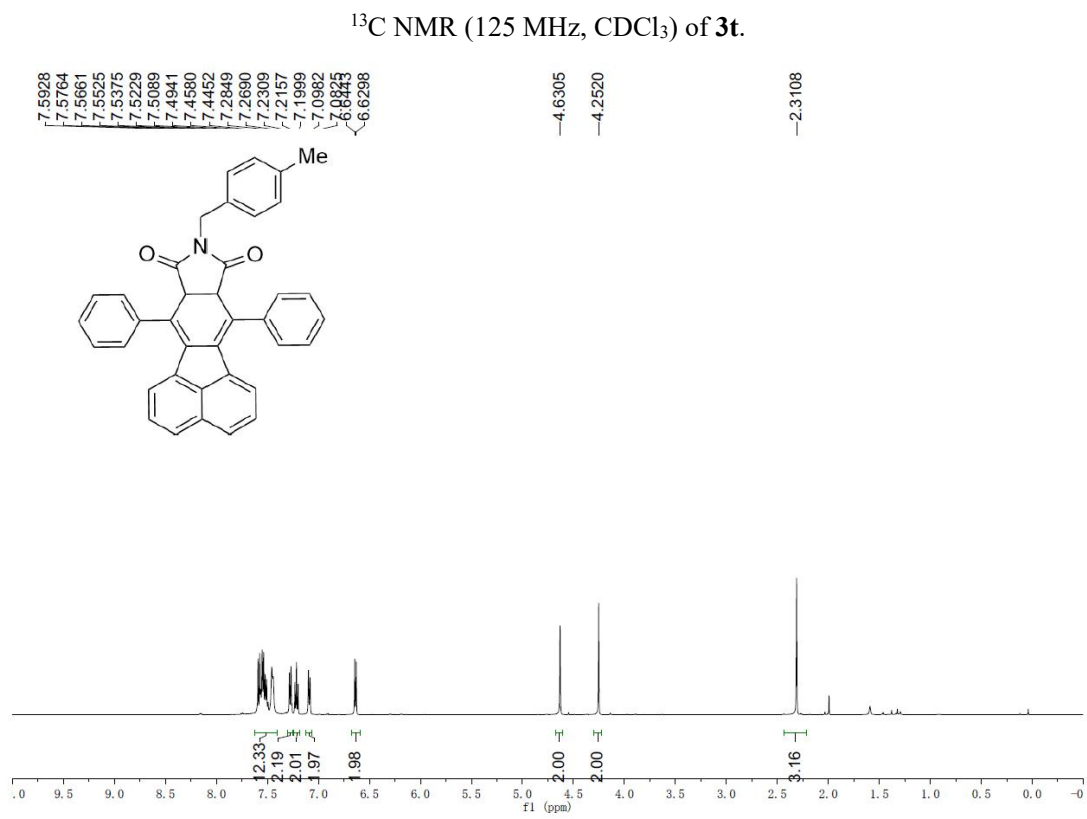
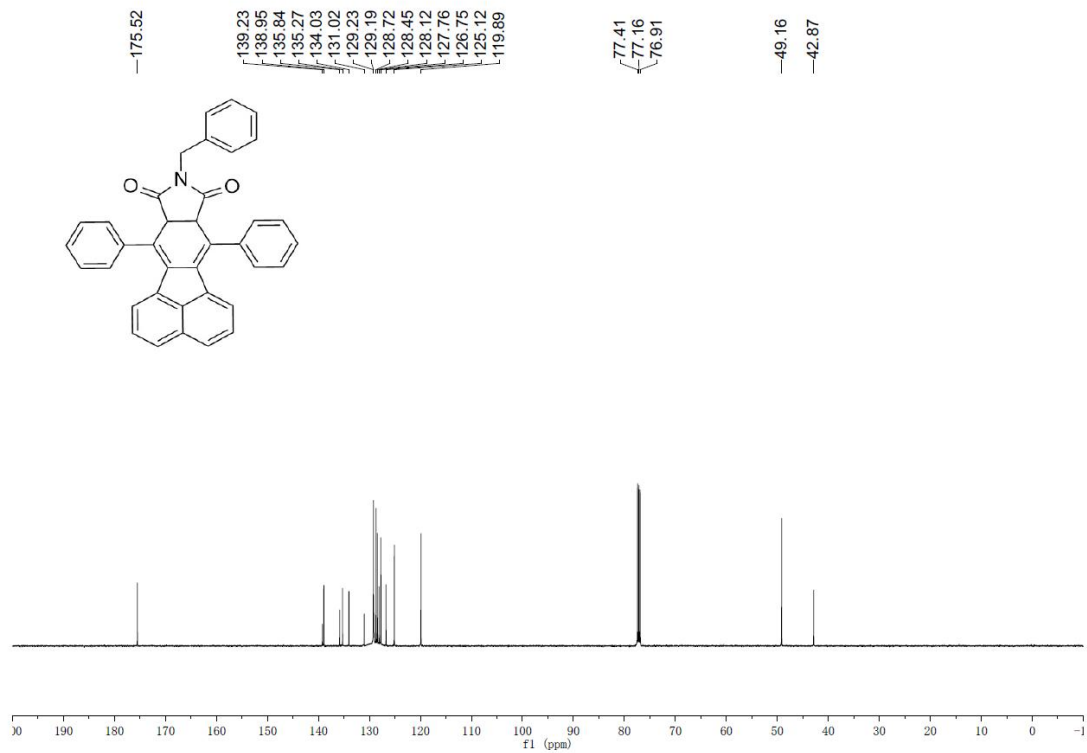
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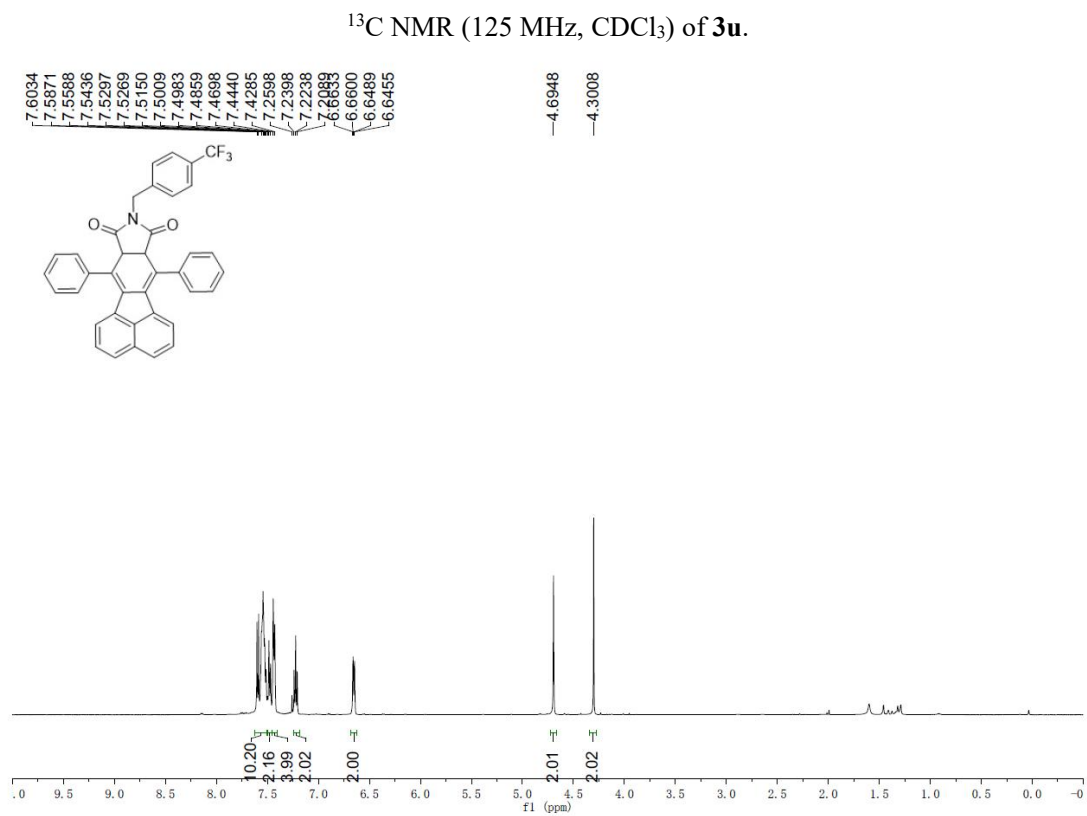
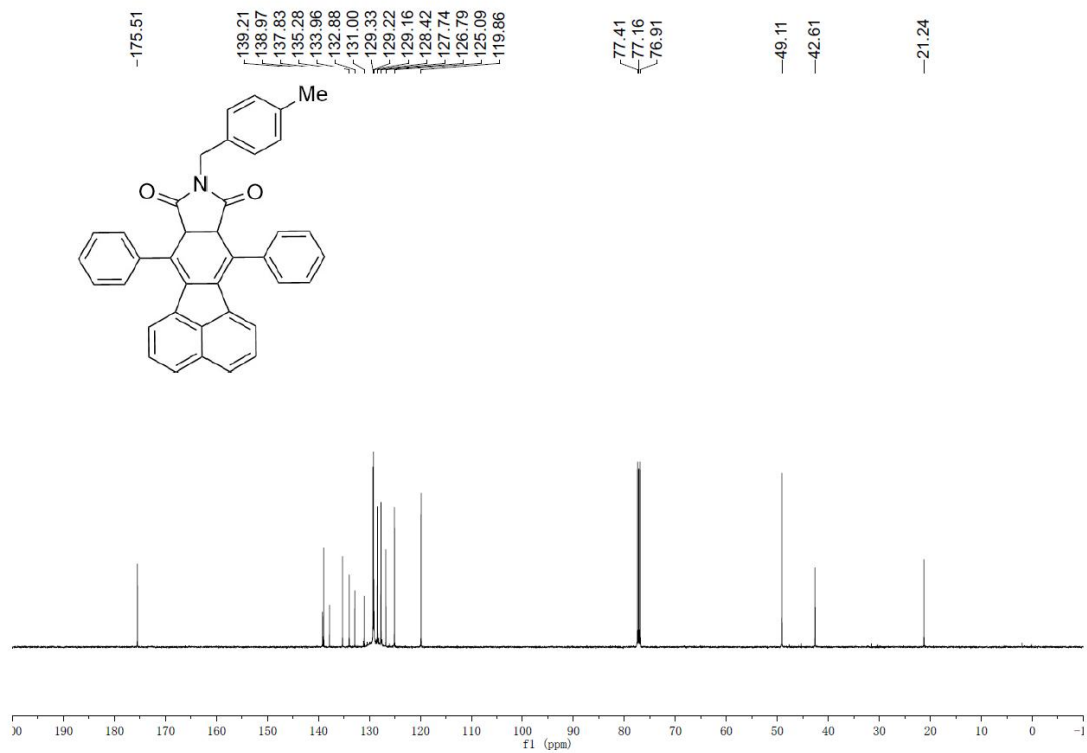


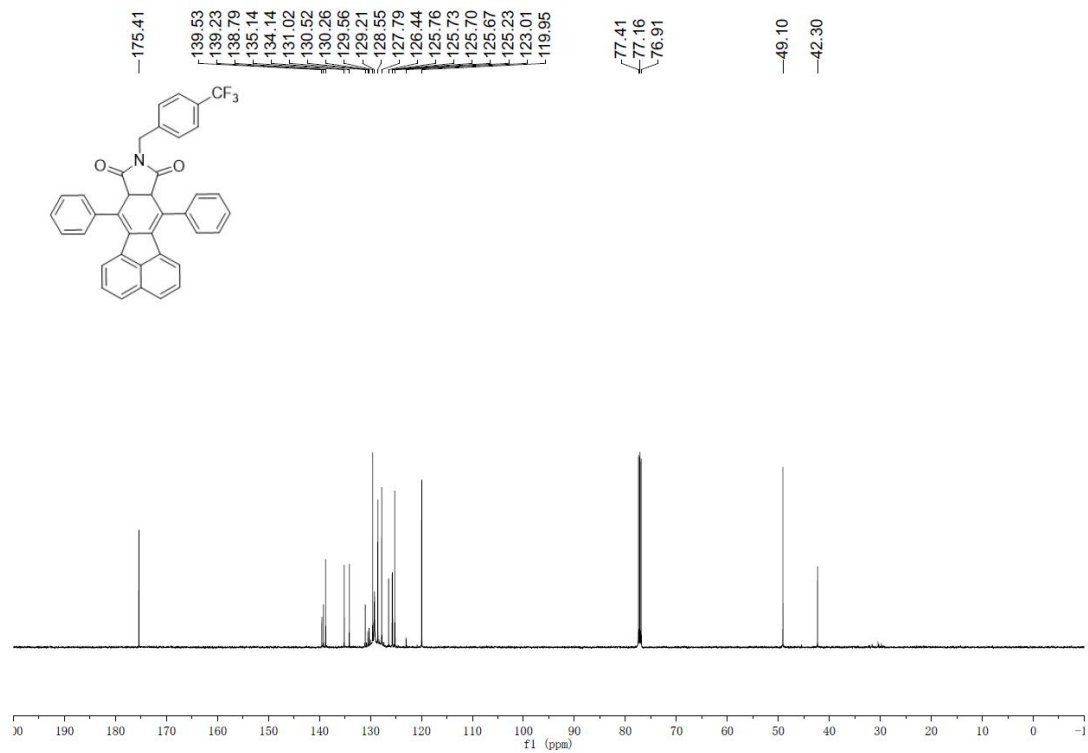
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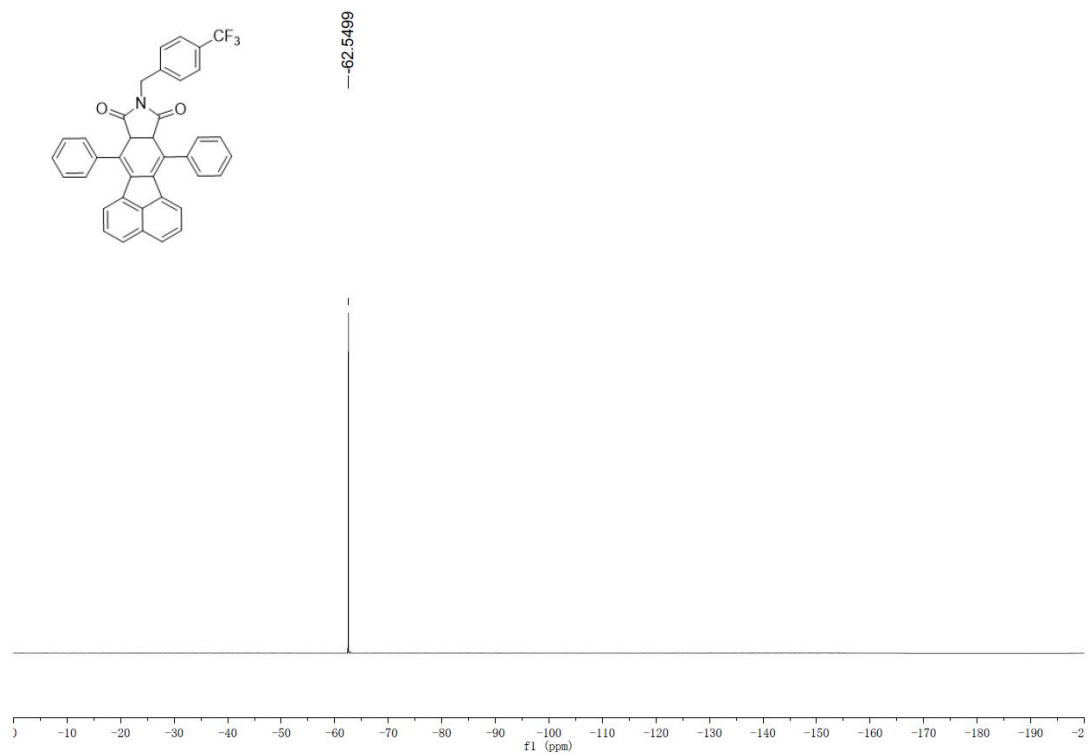
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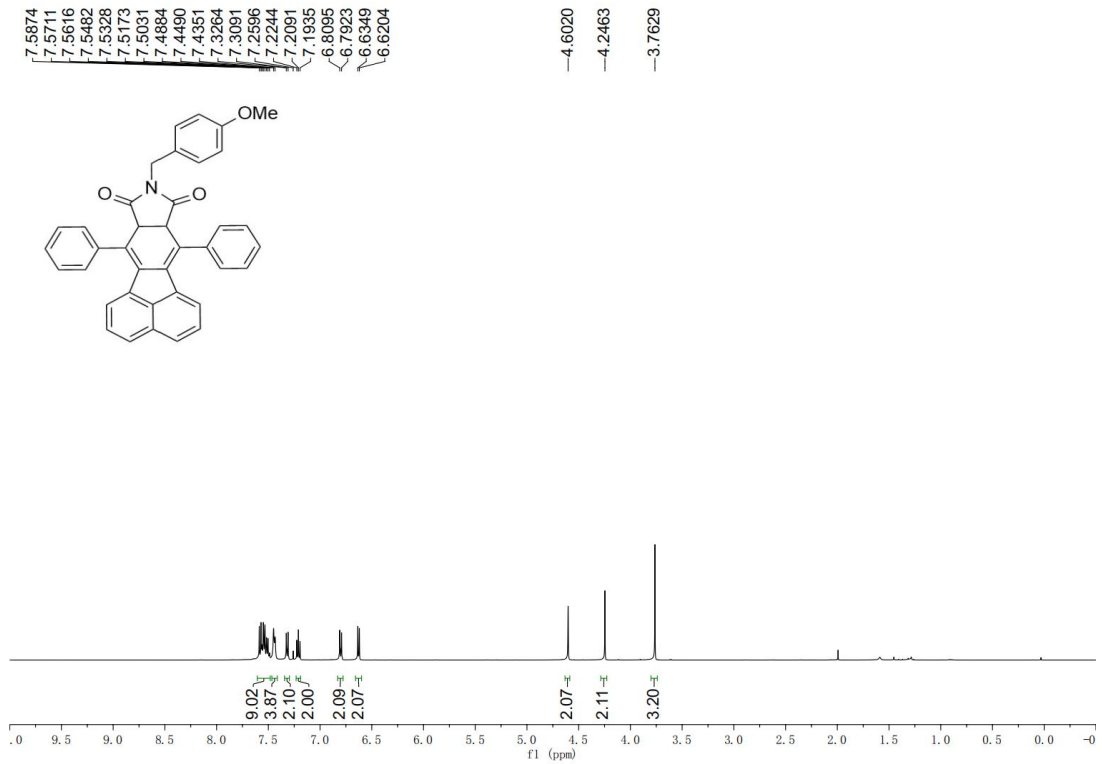




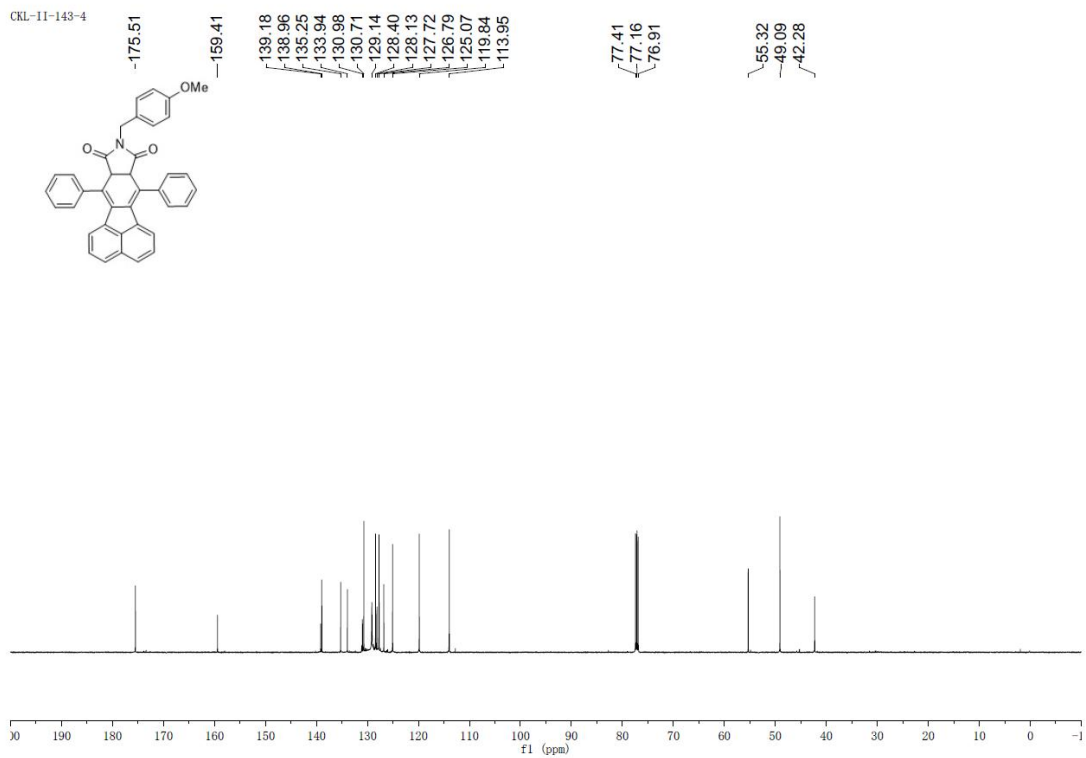
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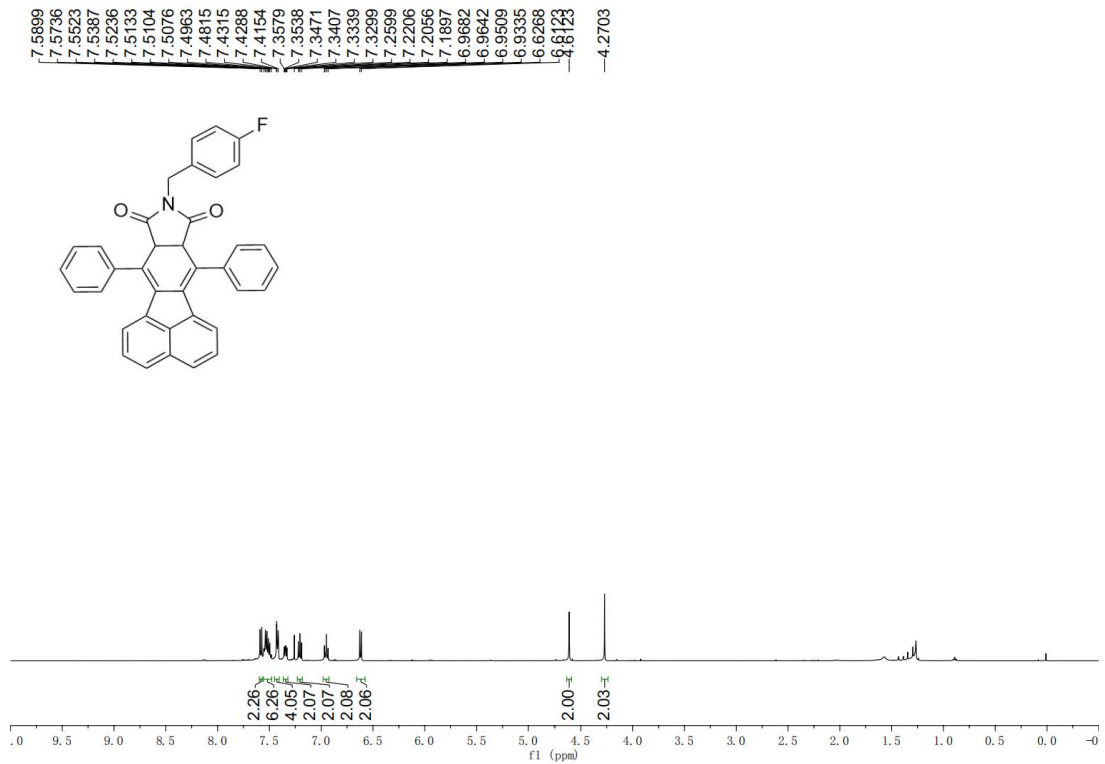
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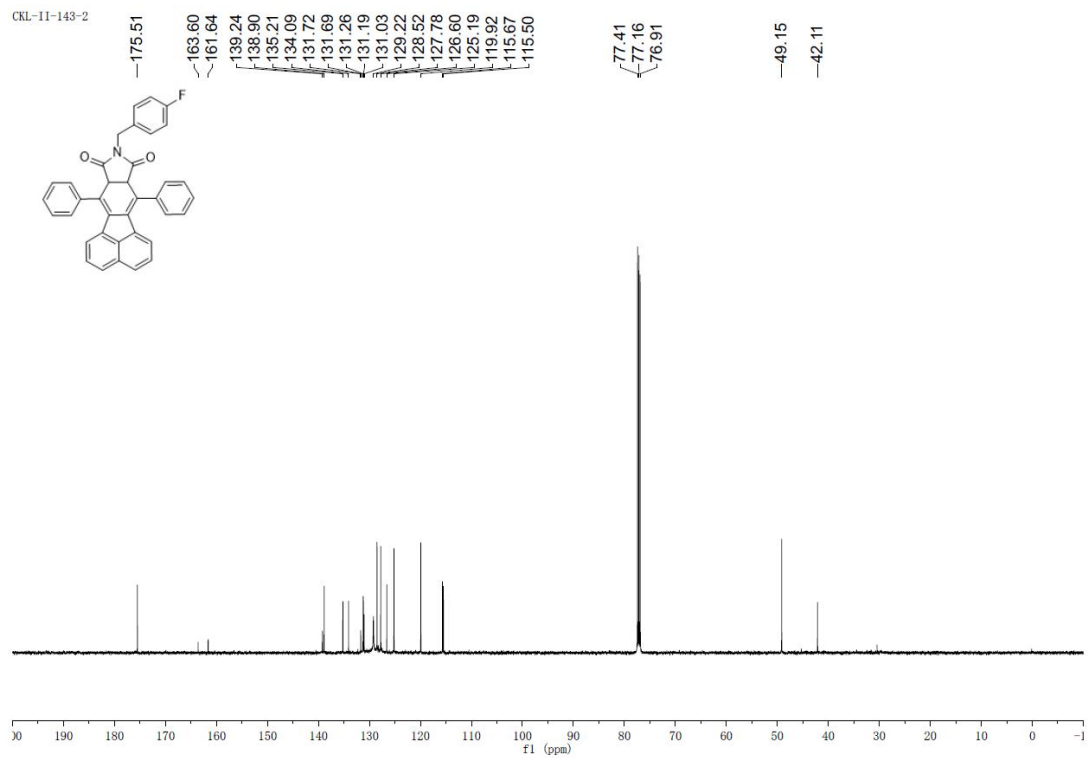
^1H NMR (500 MHz, CDCl_3) of **3w**.



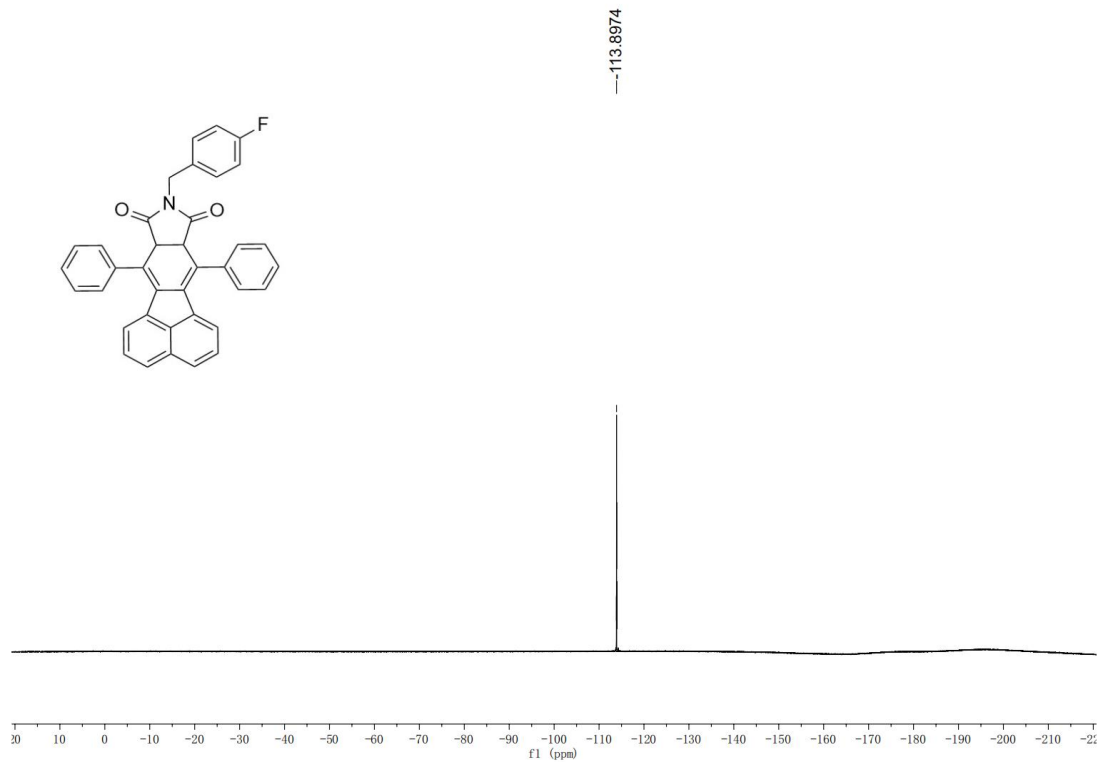
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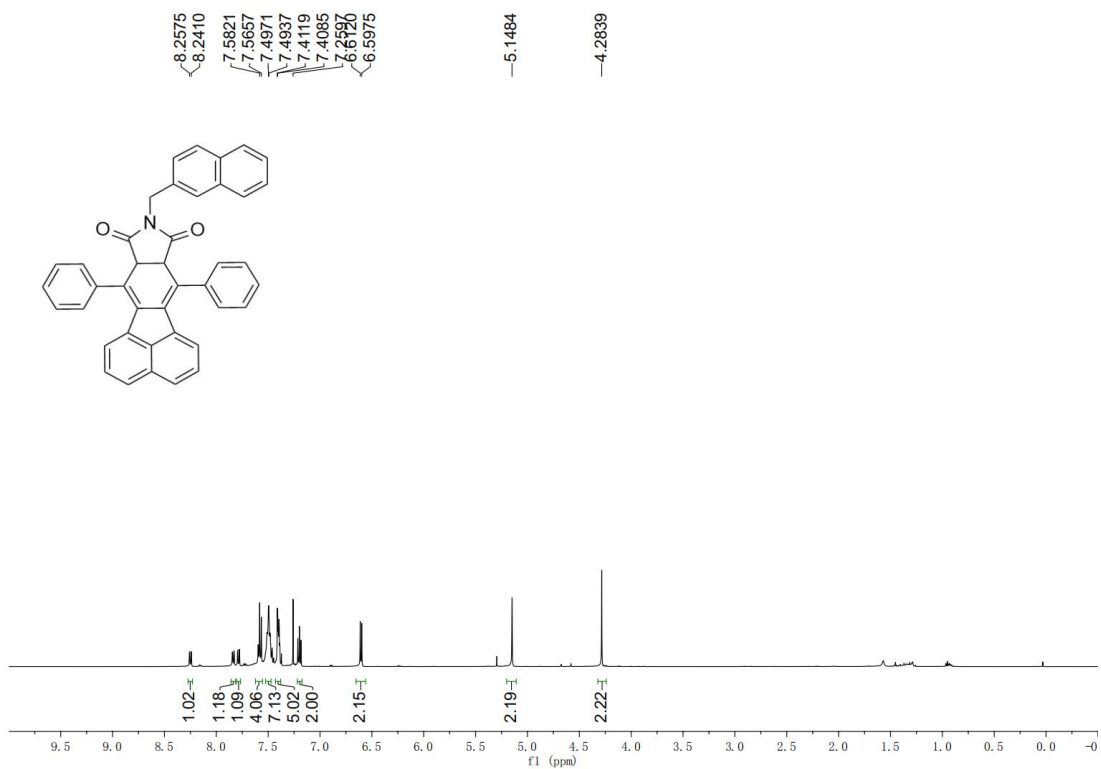
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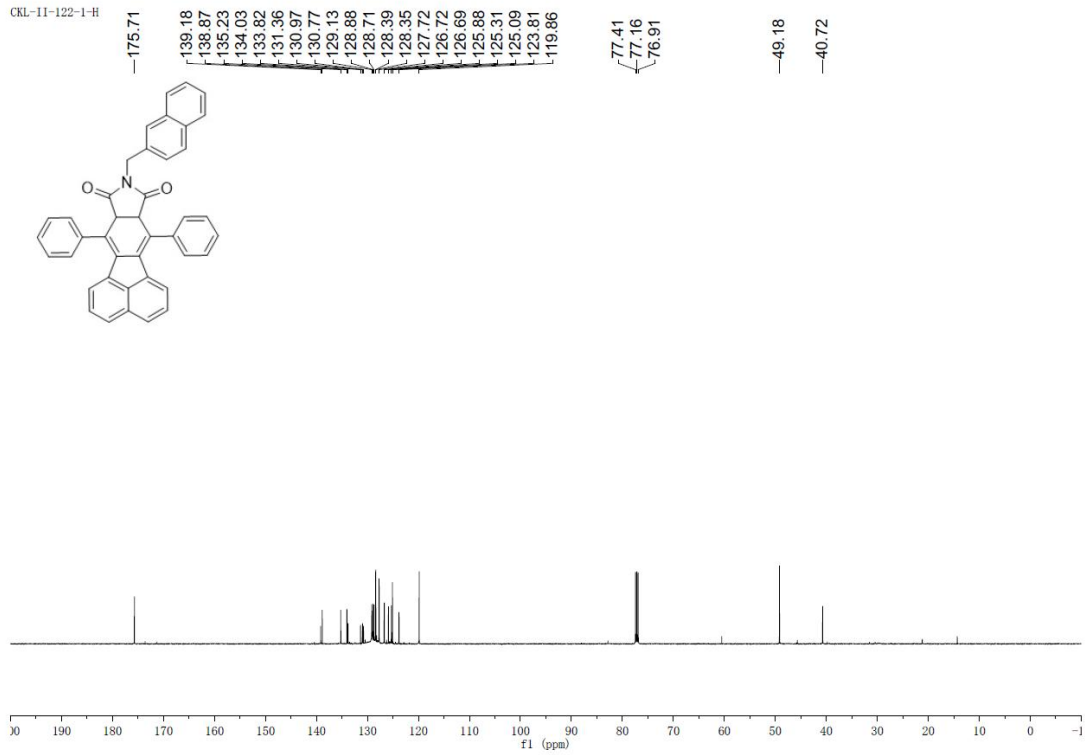
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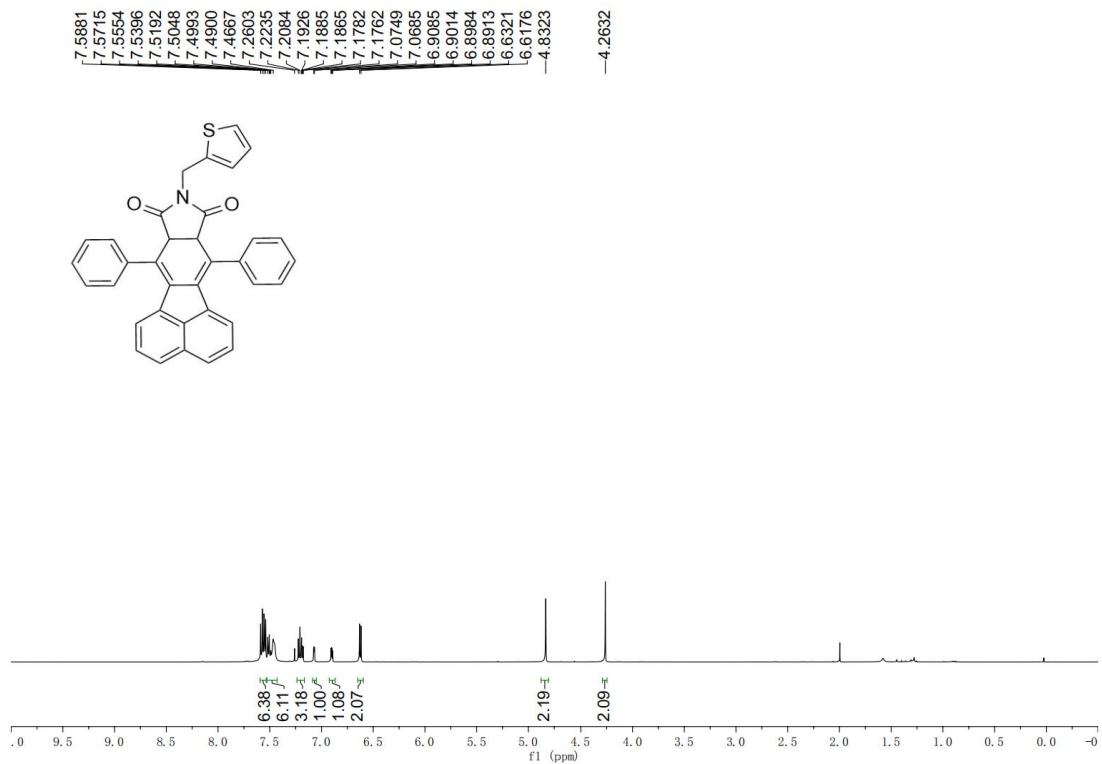
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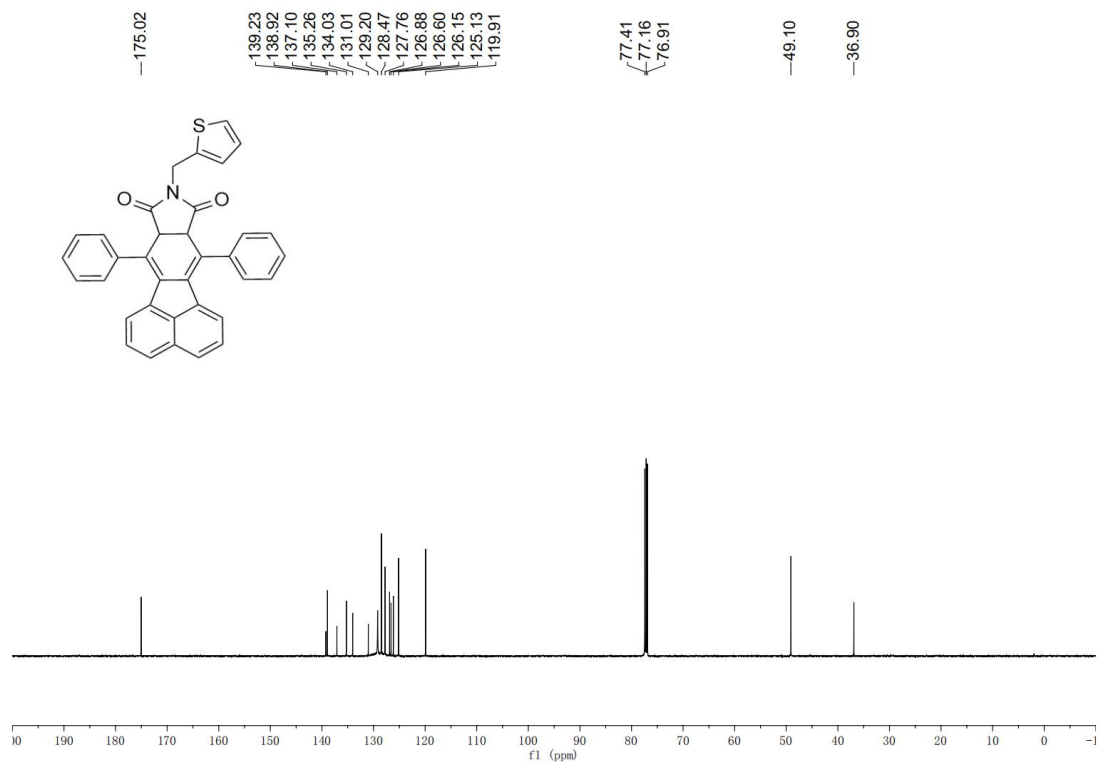
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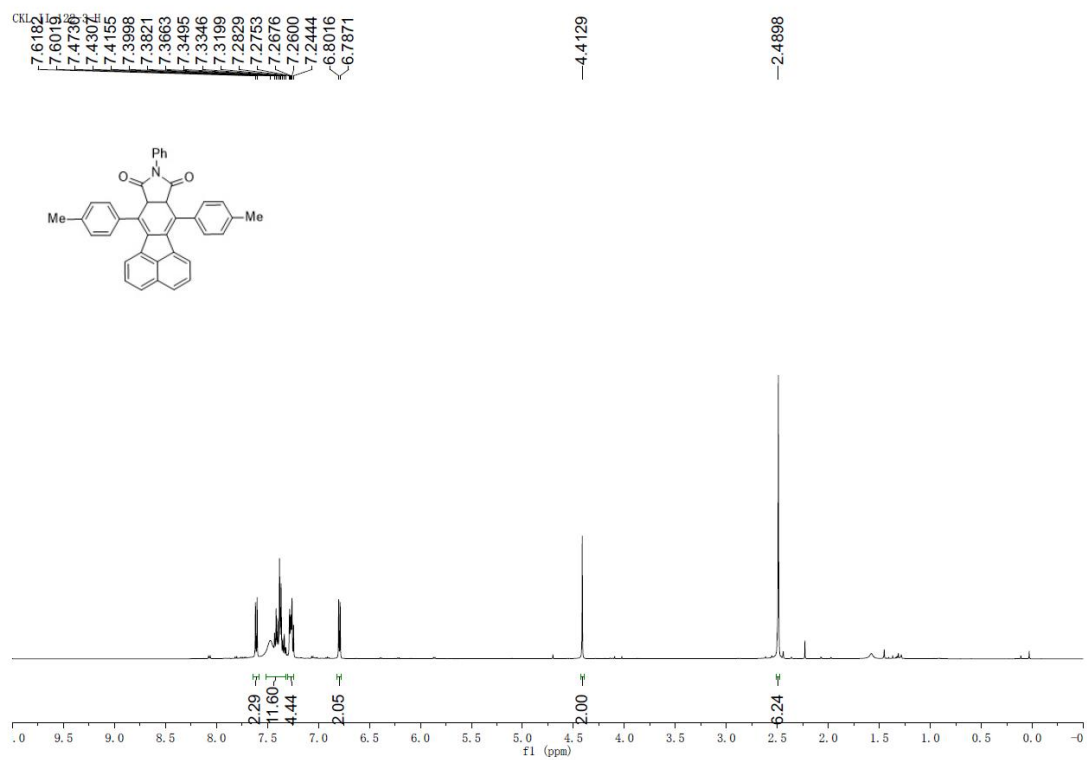
^{13}C NMR (125 MHz, CDCl_3) of **3y**.



^1H NMR (500 MHz, CDCl_3) of **3z**.

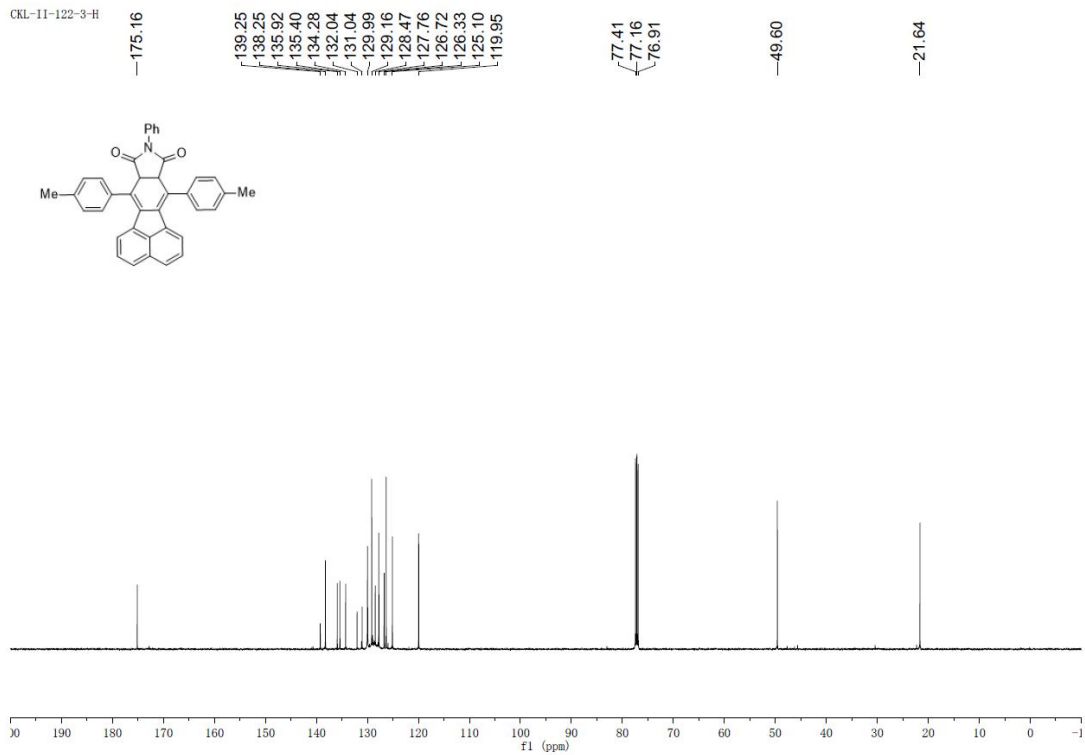


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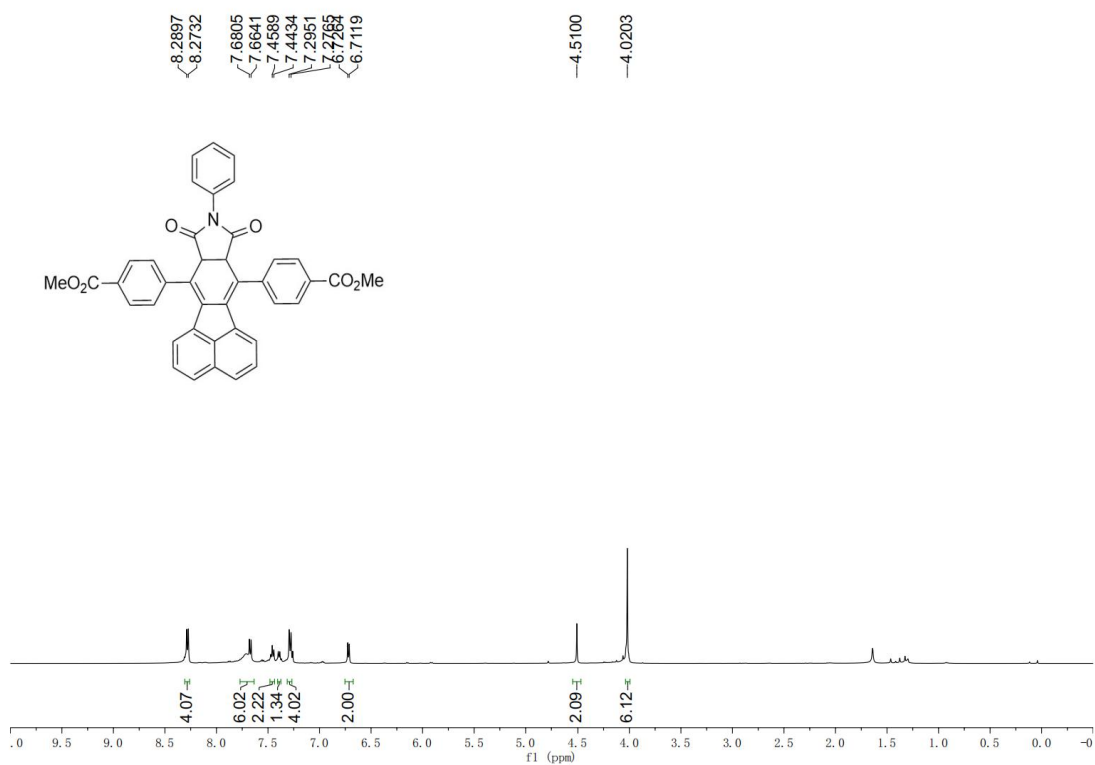


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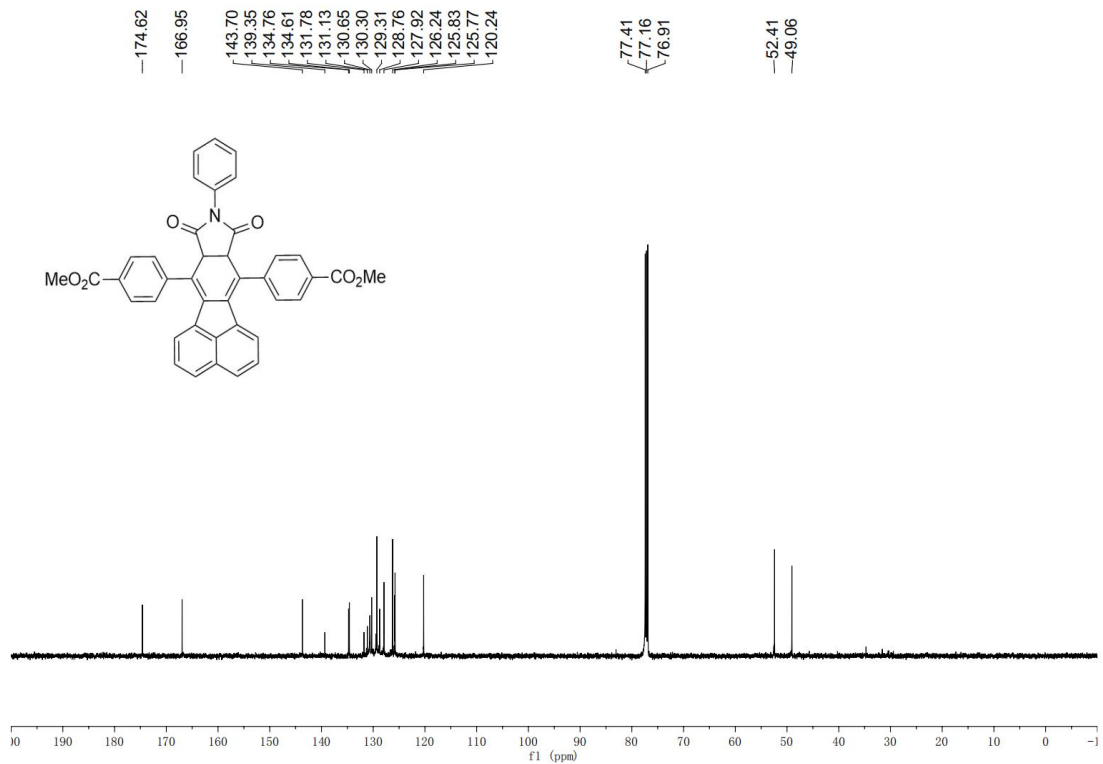
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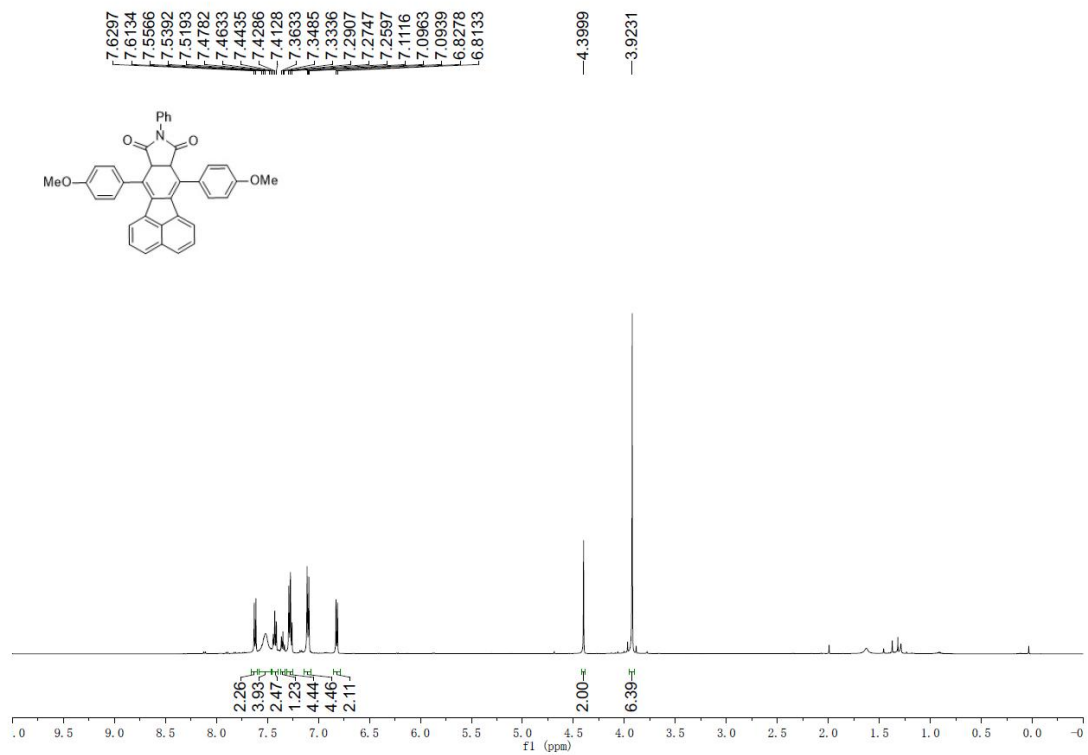
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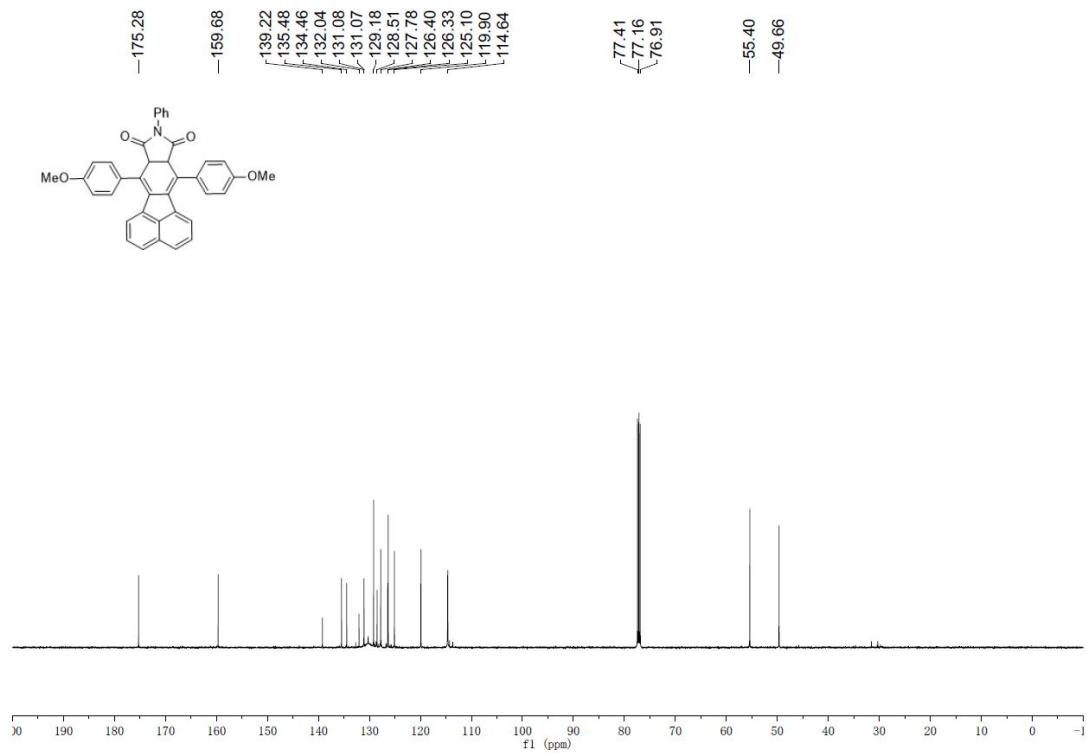
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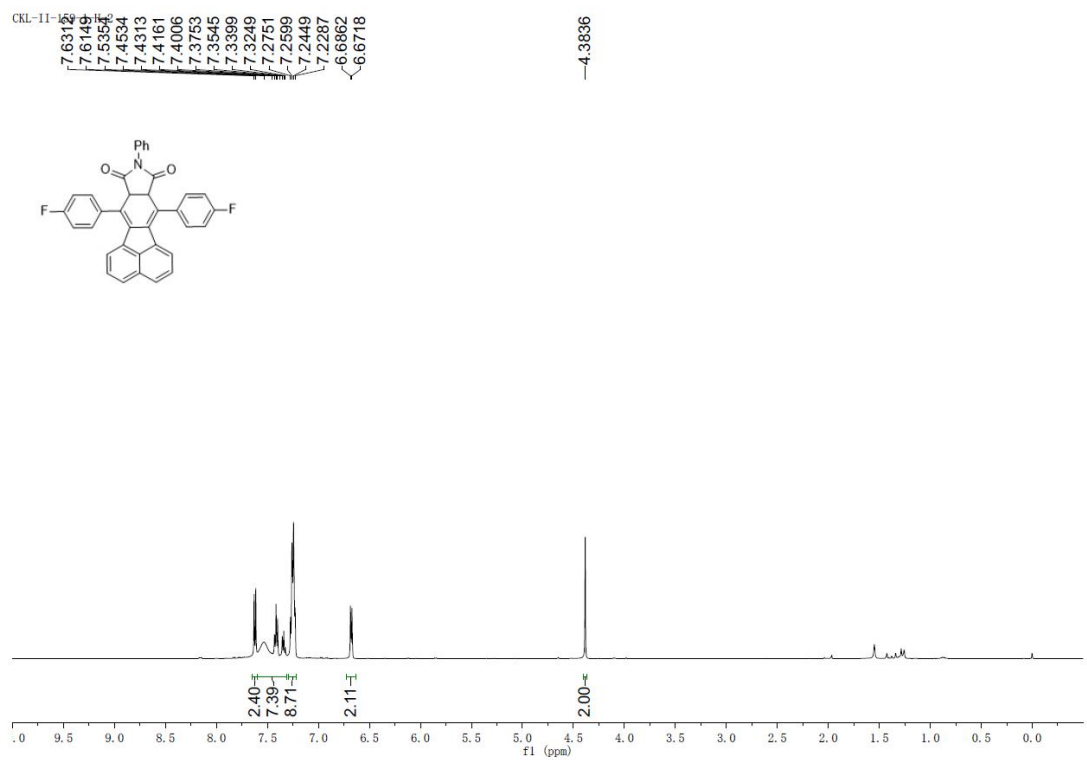
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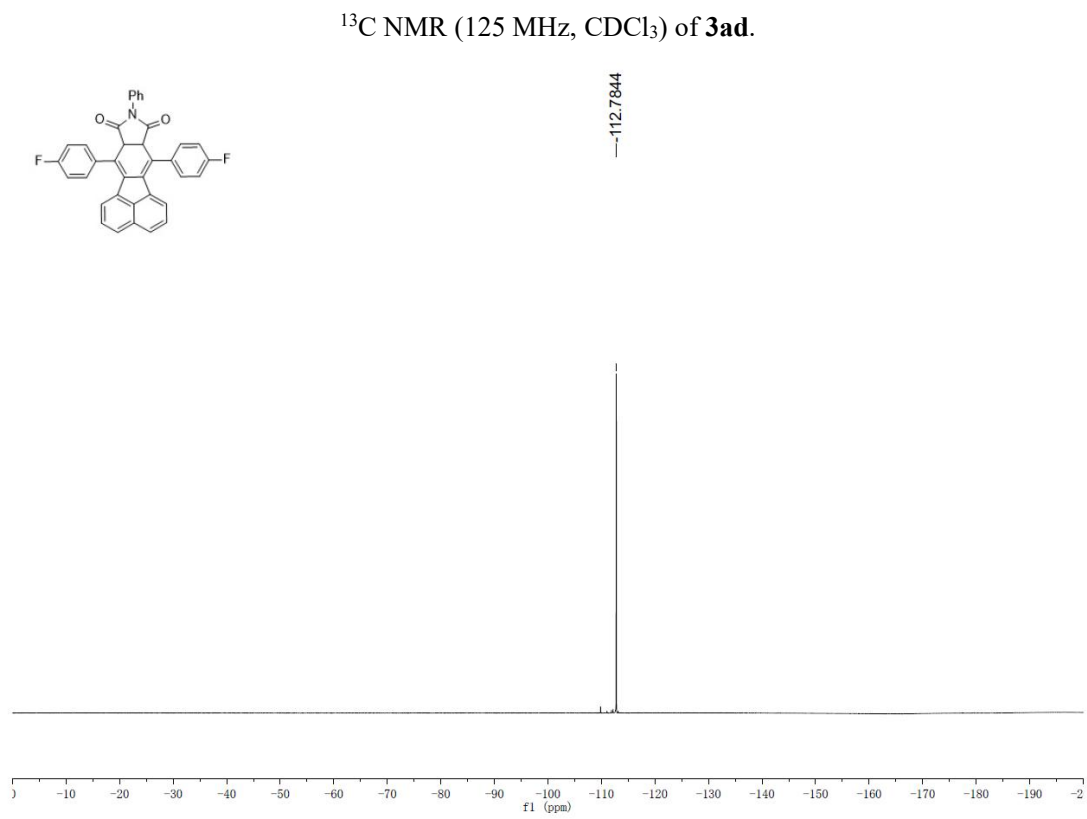
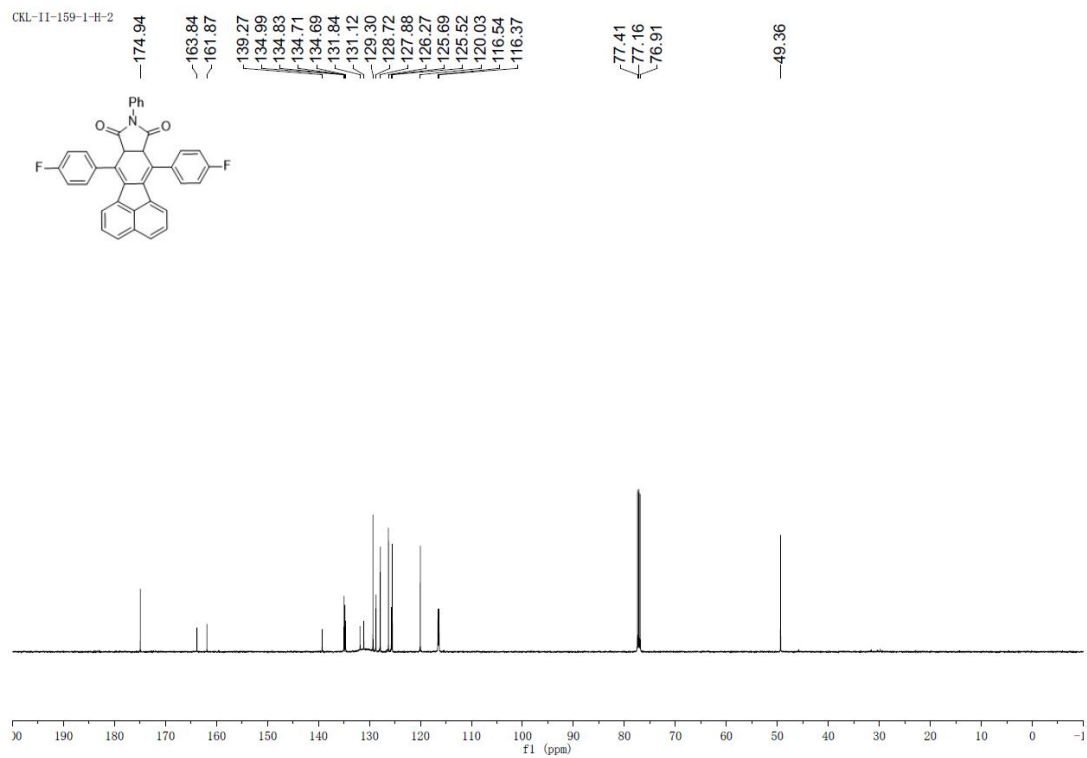
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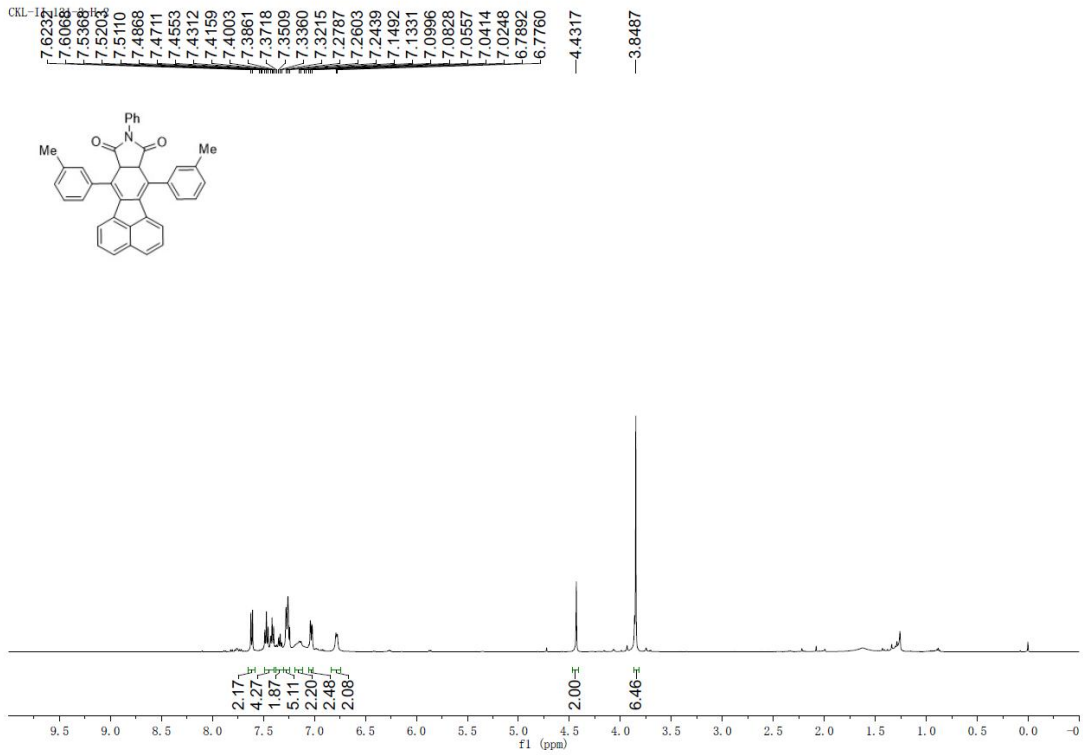
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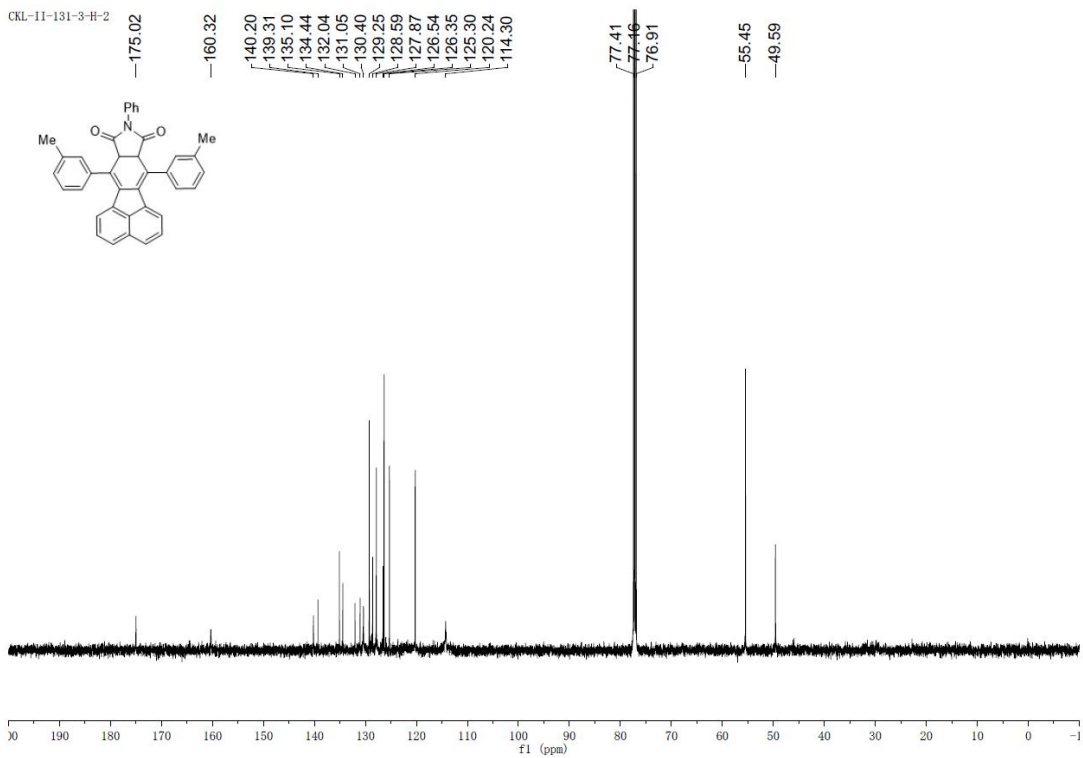
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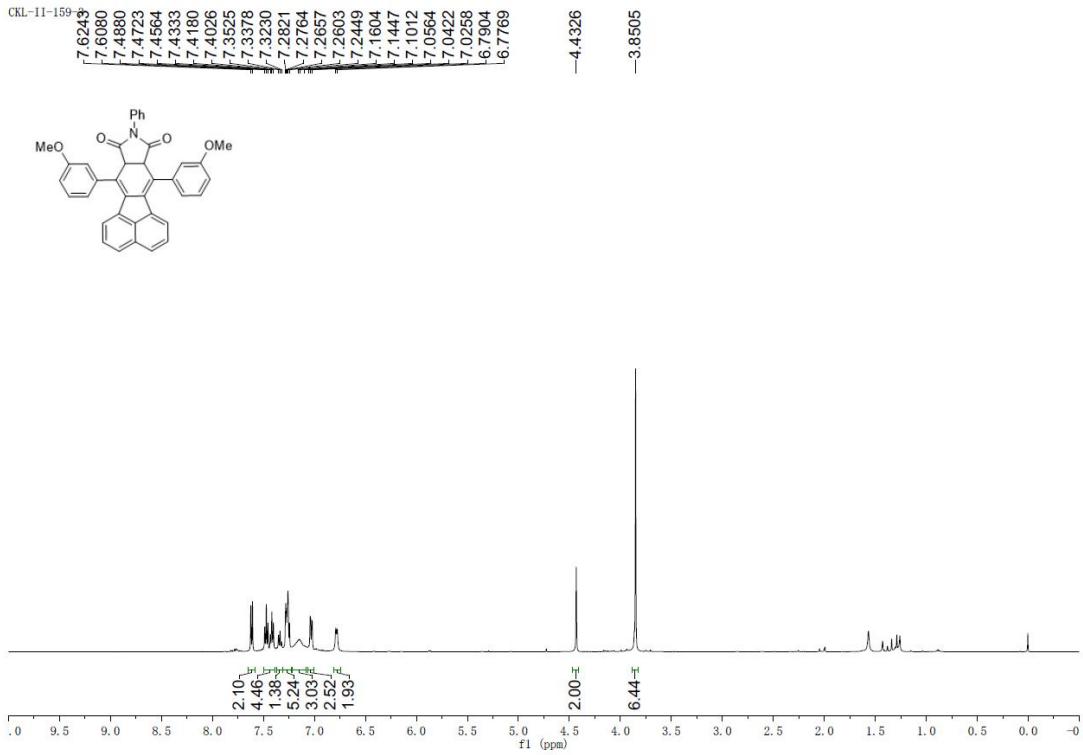
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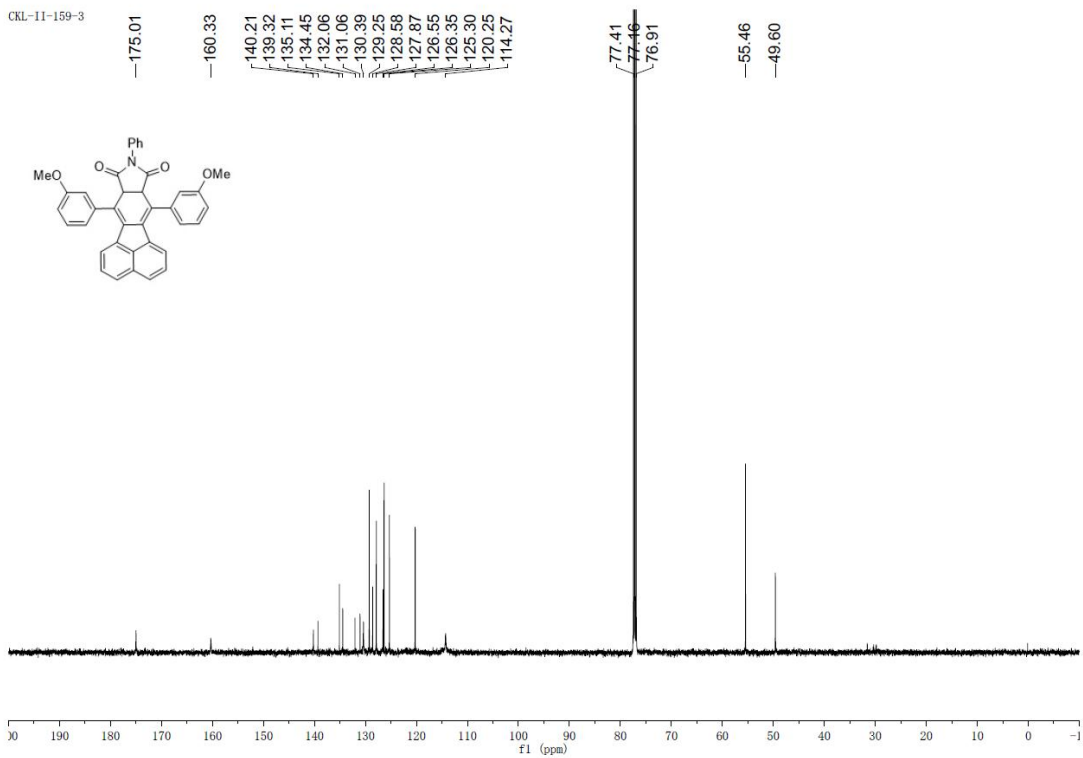
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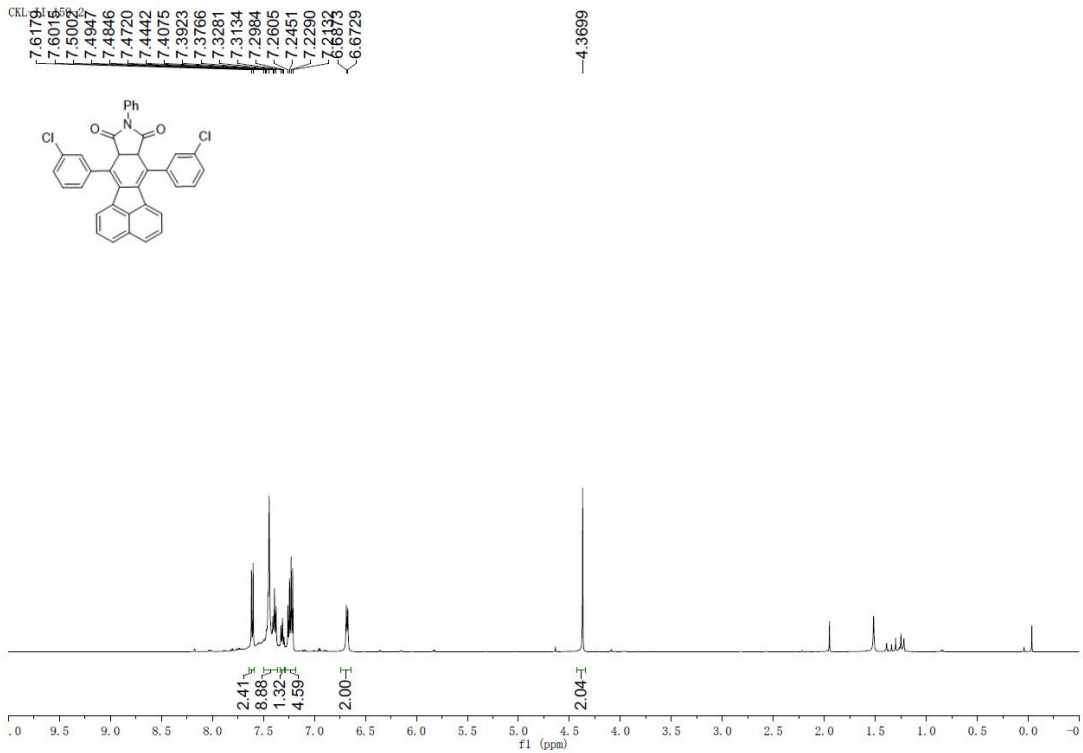
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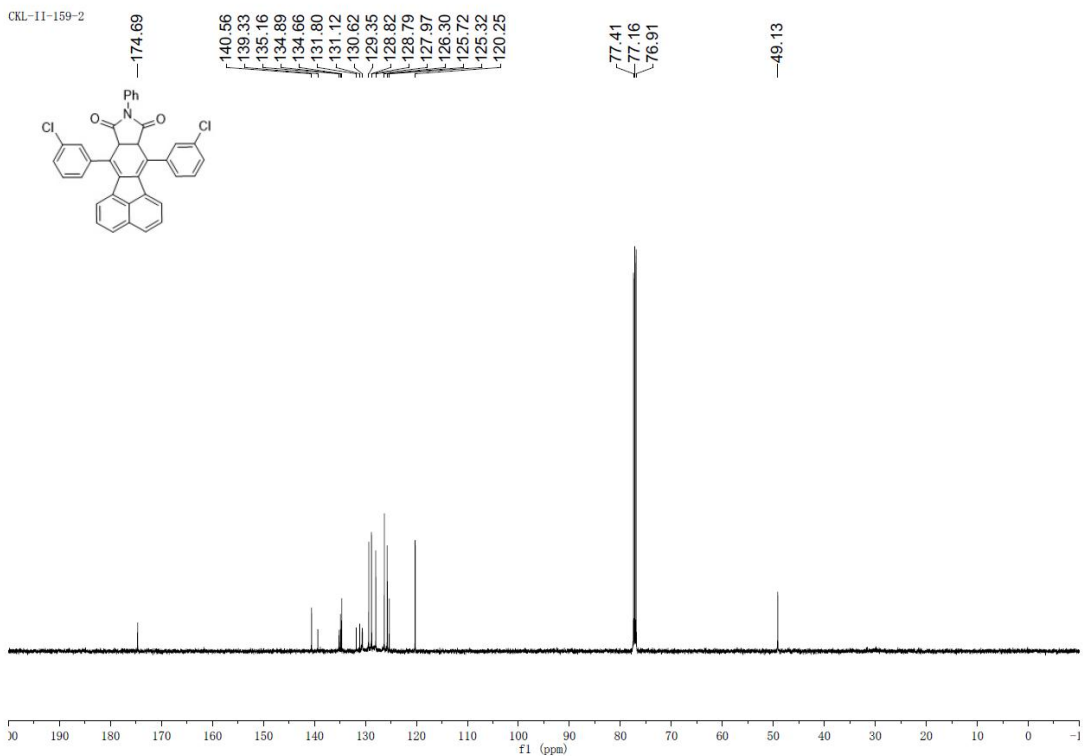
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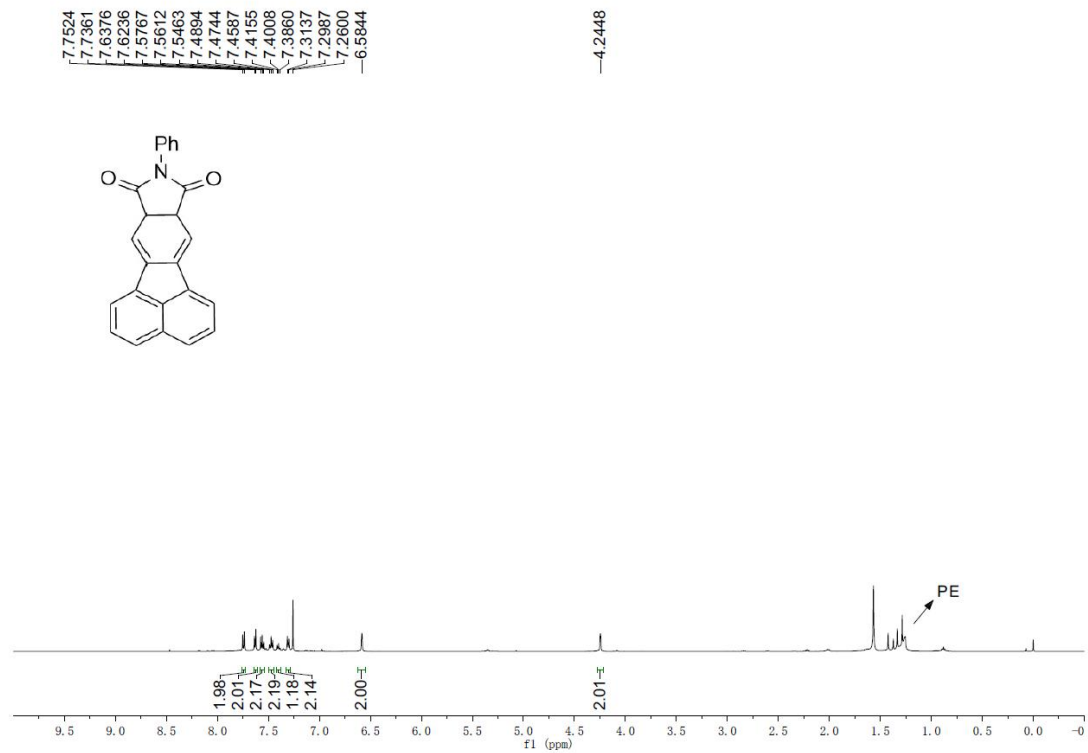
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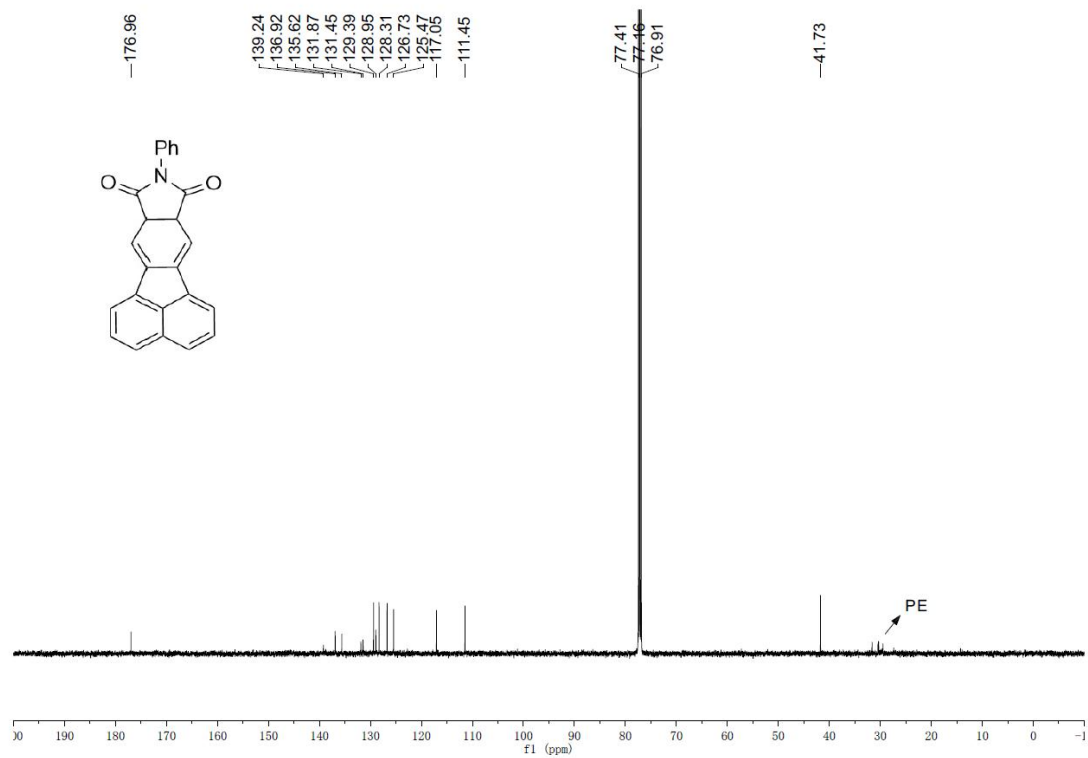
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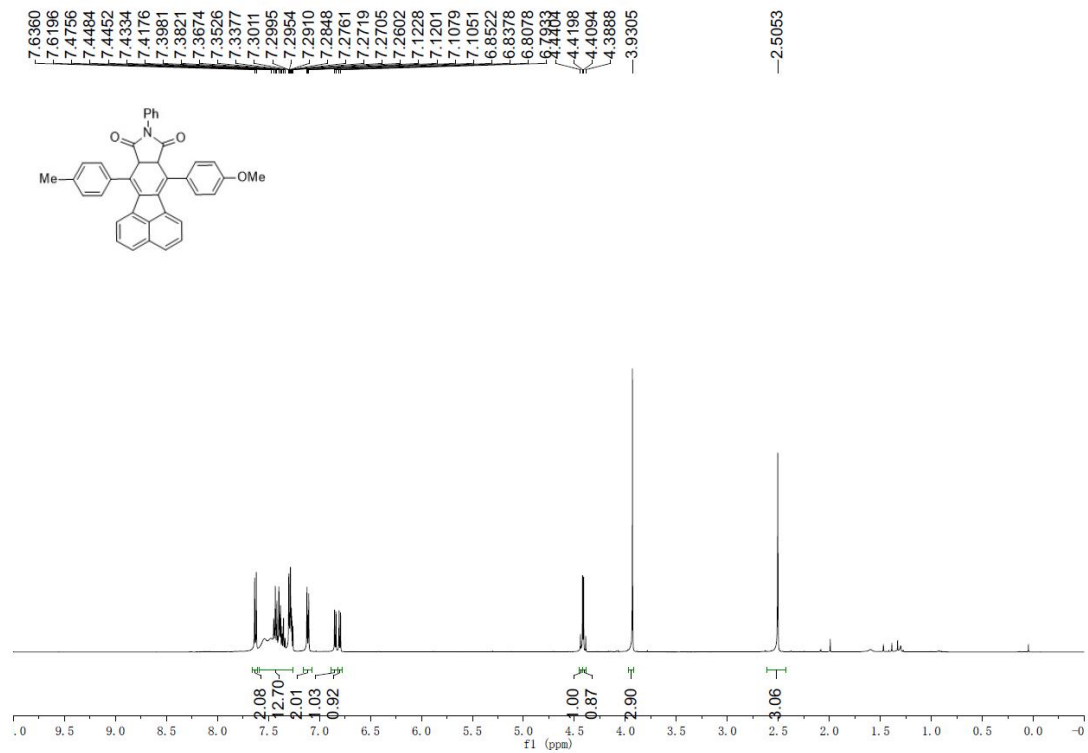
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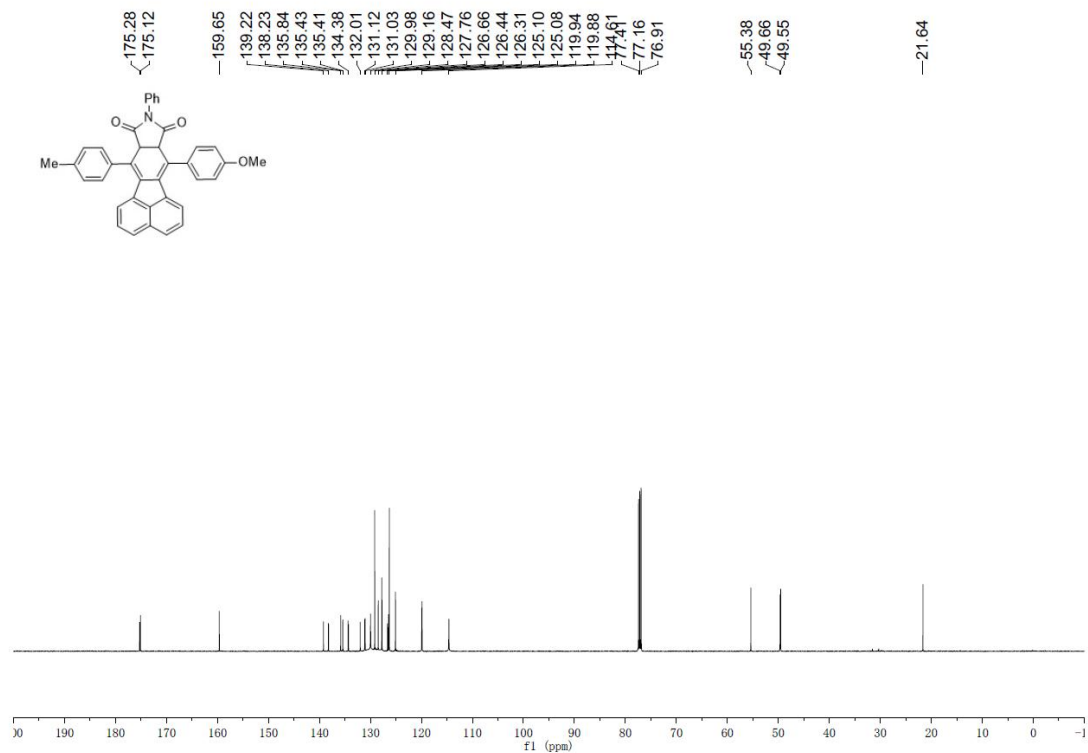
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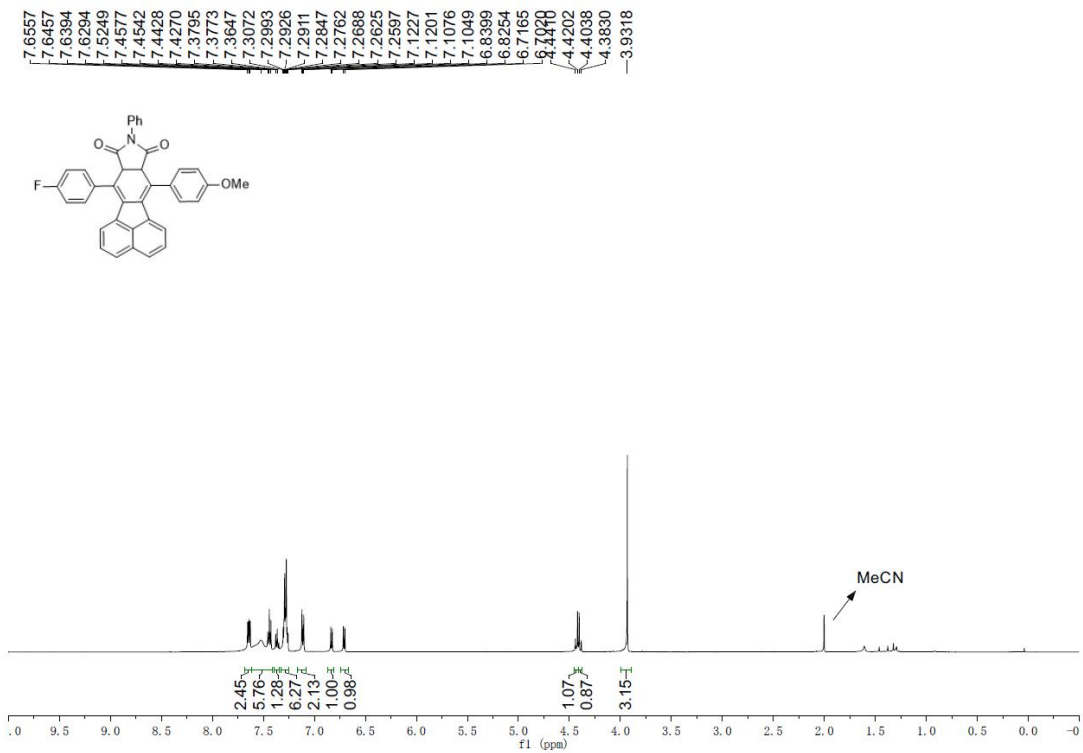
^{13}C NMR (125 MHz, CDCl_3) of **3ah**.



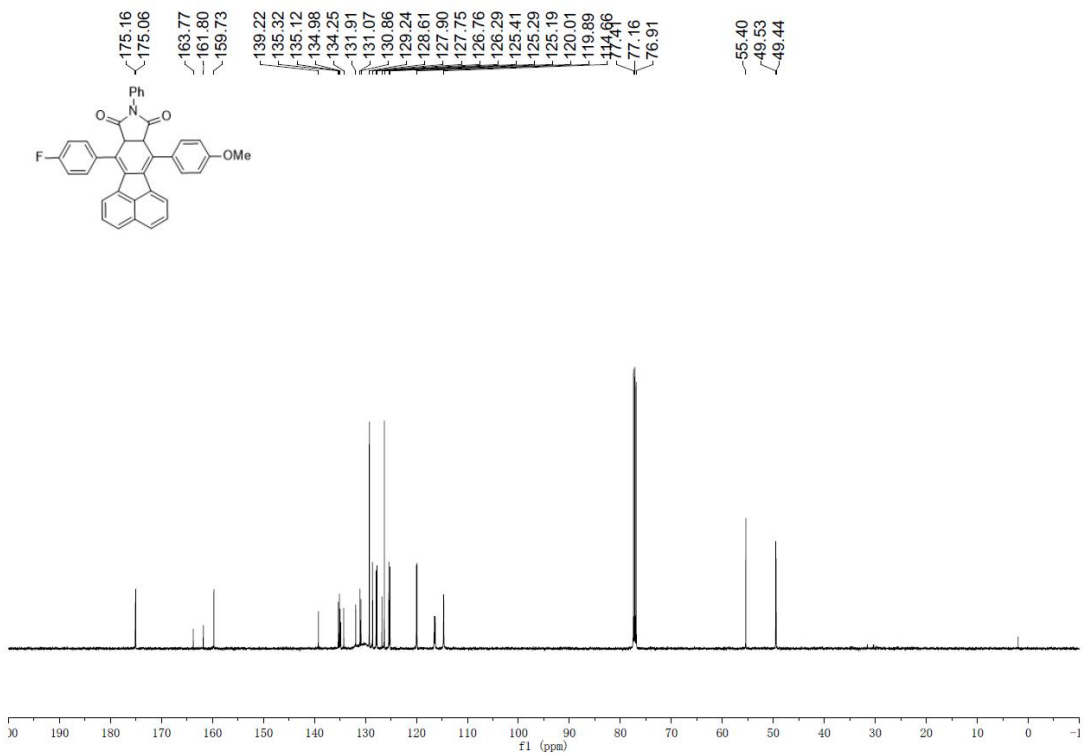
^1H NMR (500 MHz, CDCl_3) of **3ai**.



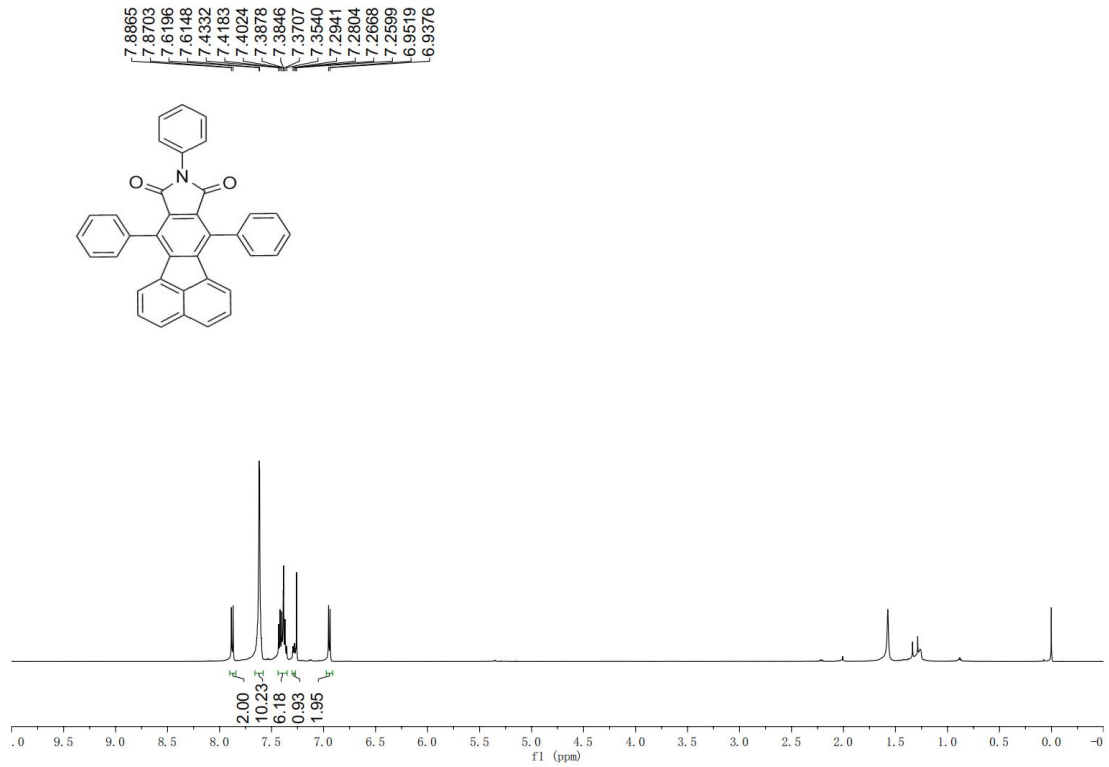
^{13}C NMR (125 MHz, CDCl_3) of **3ai**.



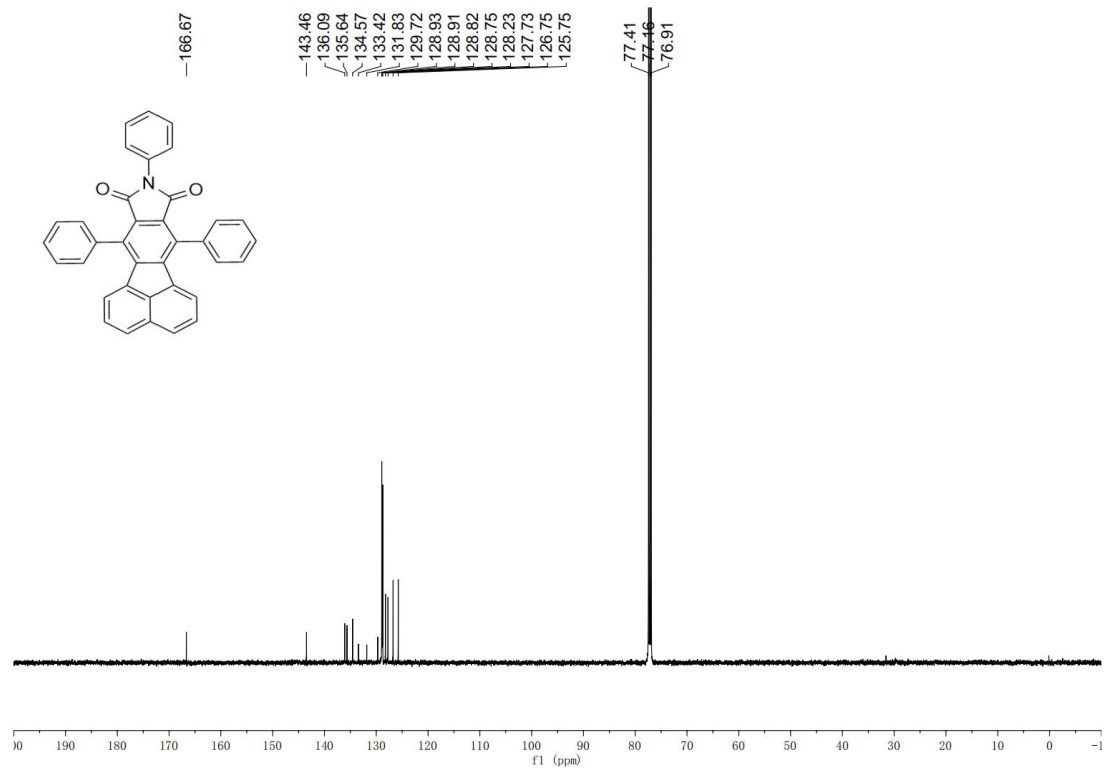
^1H NMR (500 MHz, CDCl_3) of **3aj**.



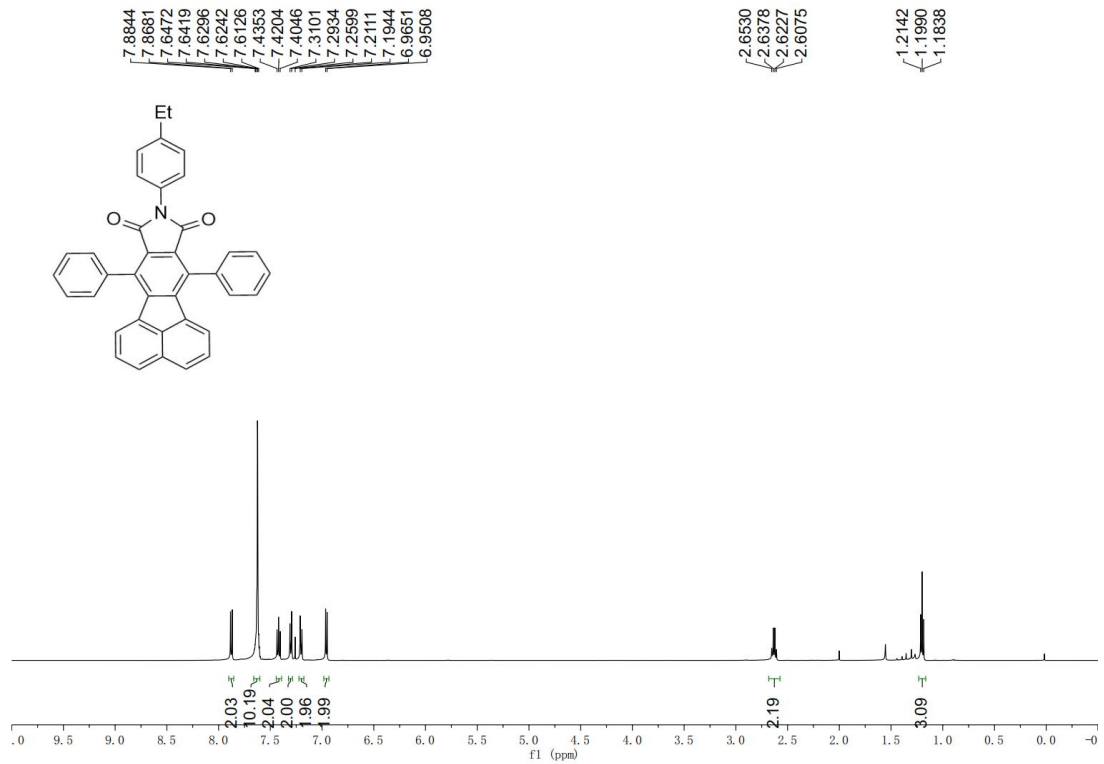
^{13}C NMR (125 MHz, CDCl_3) of **3aj**.



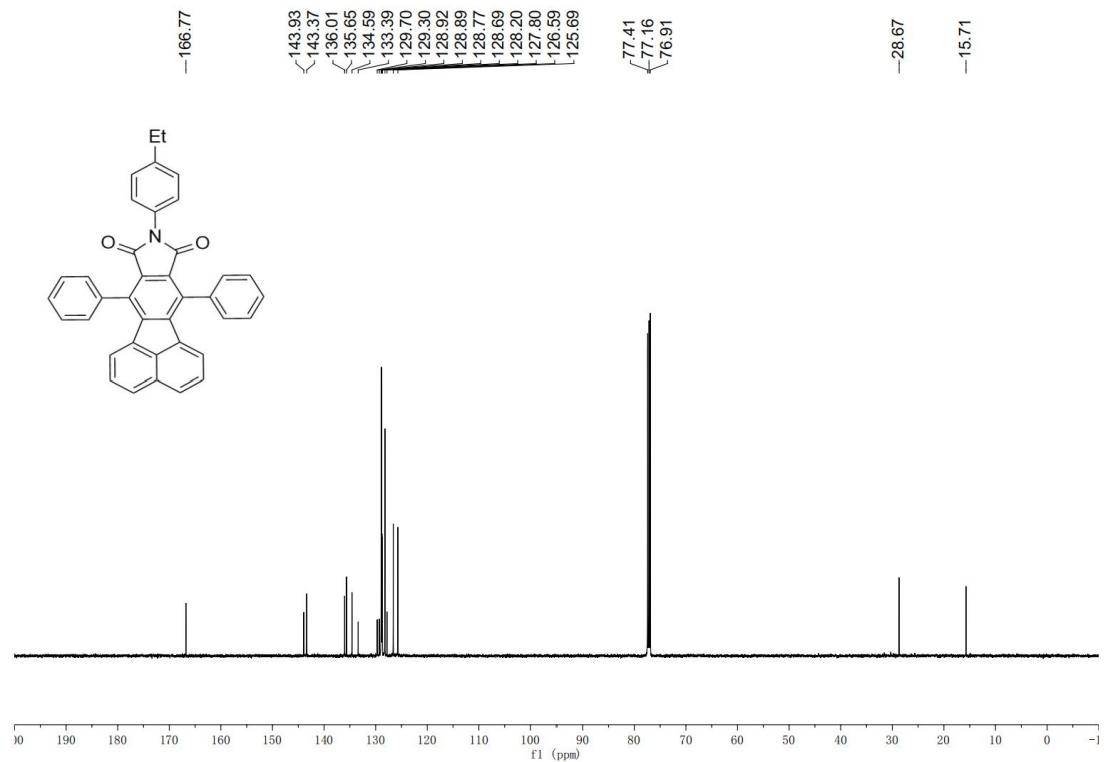
^1H NMR (500 MHz, CDCl_3) of **4a**.



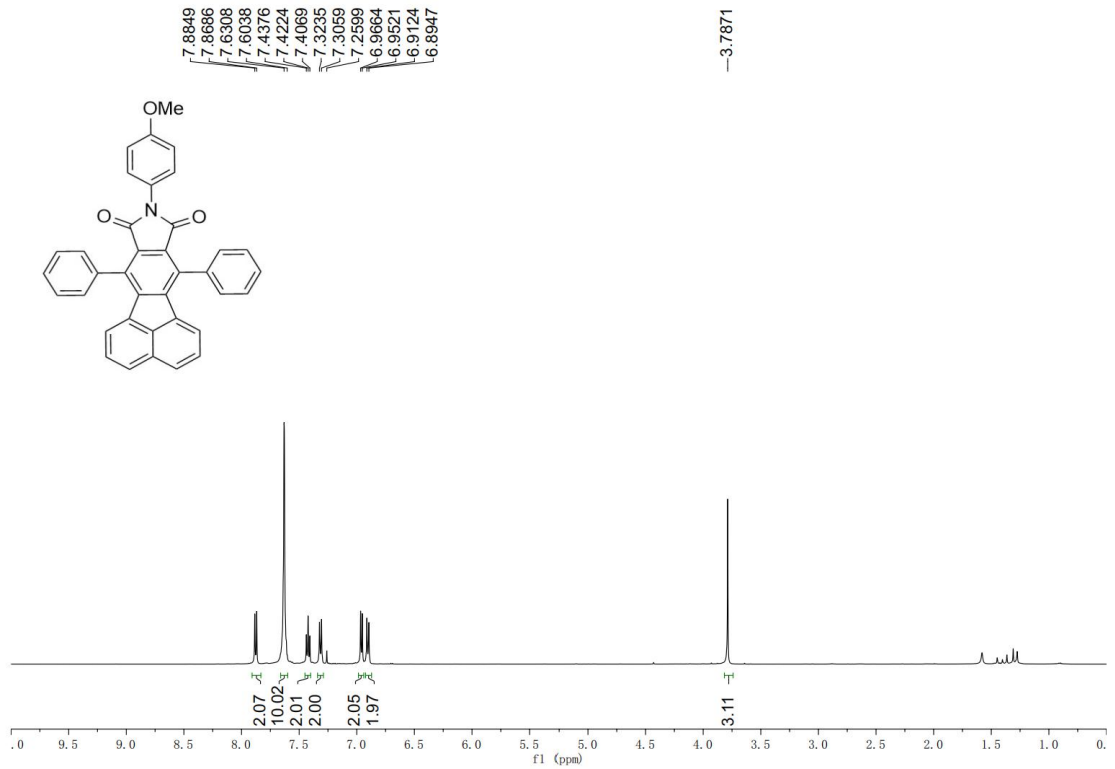
^{13}C NMR (125 MHz, CDCl_3) of **4a**.



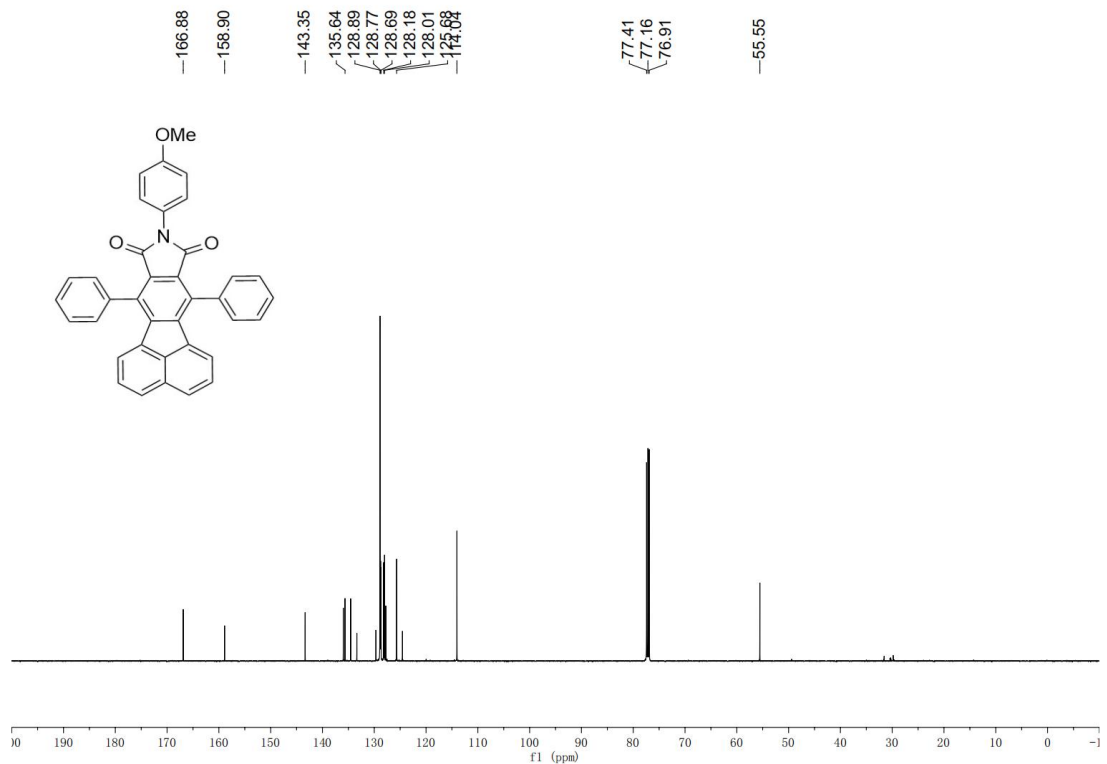
^1H NMR (500 MHz, CDCl_3) of **4b**.



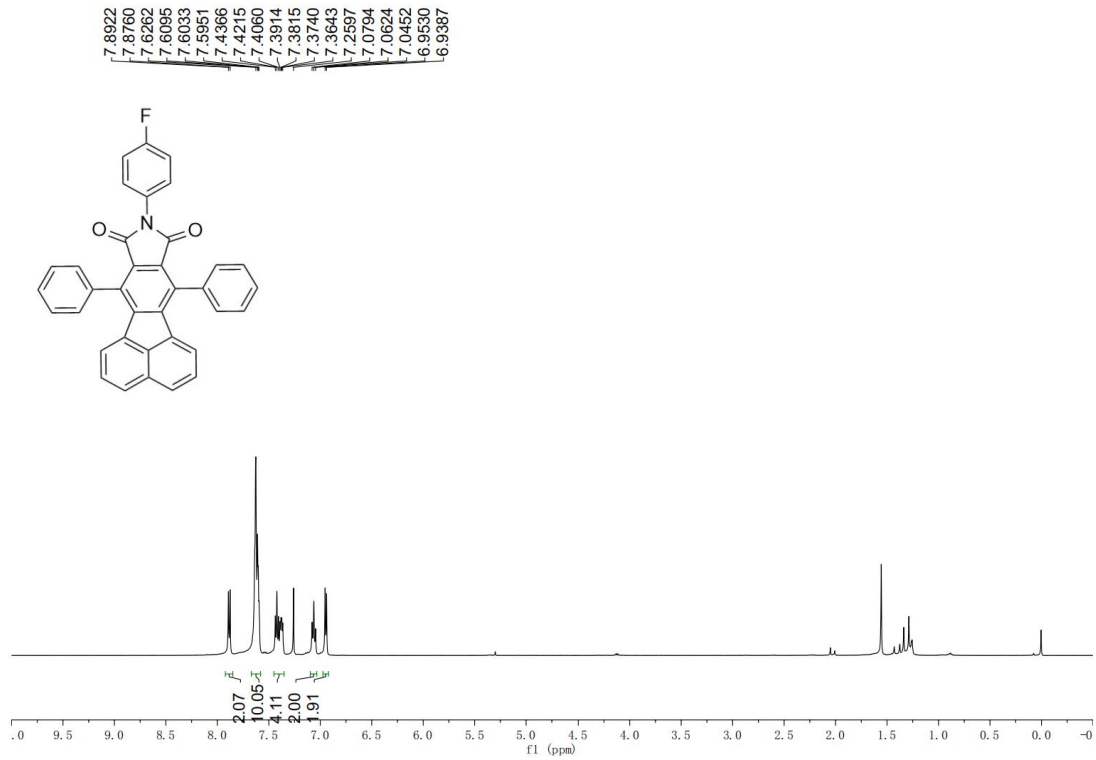
^{13}C NMR (125 MHz, CDCl_3) of **4b**.



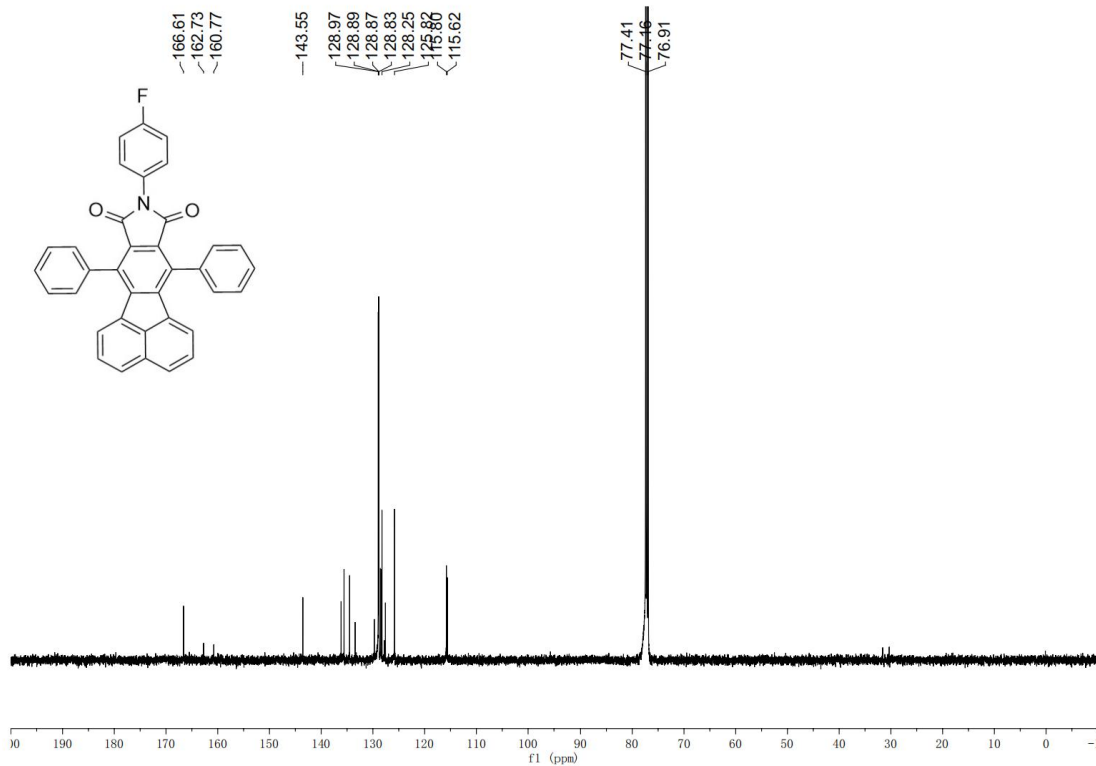
$^1\text{H NMR}$ (500 MHz, CDCl_3) of **4c**.



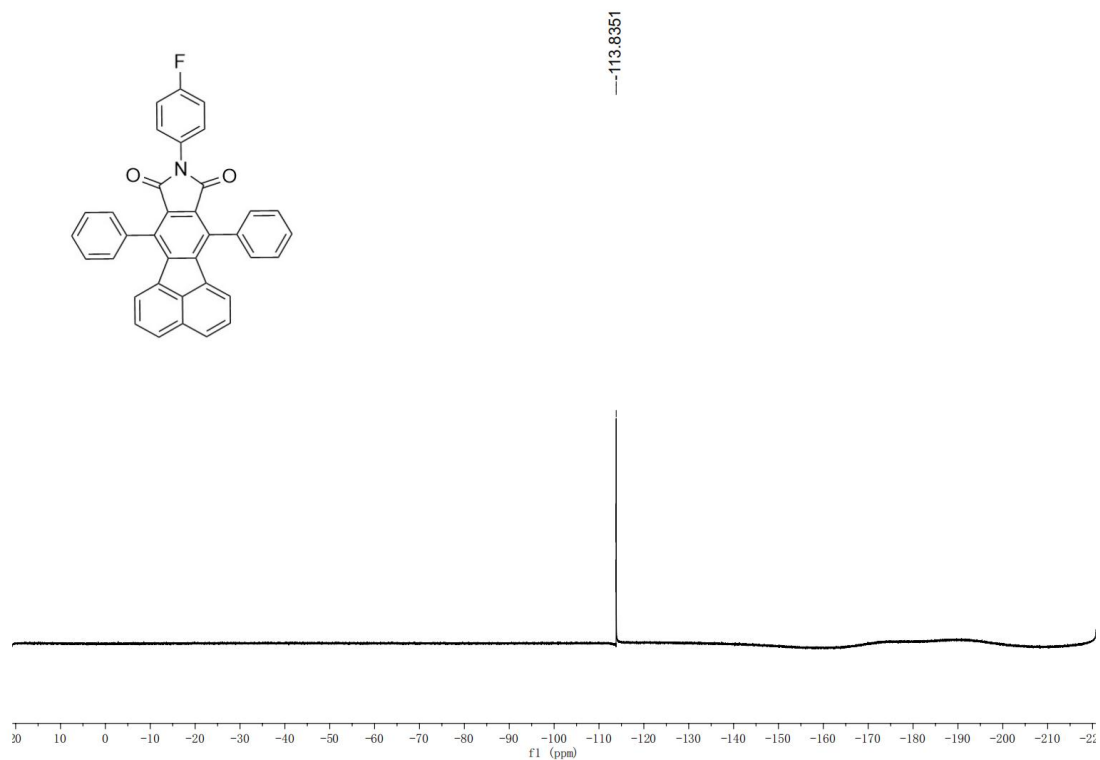
$^{13}\text{C NMR}$ (125 MHz, CDCl_3) of **4c**.



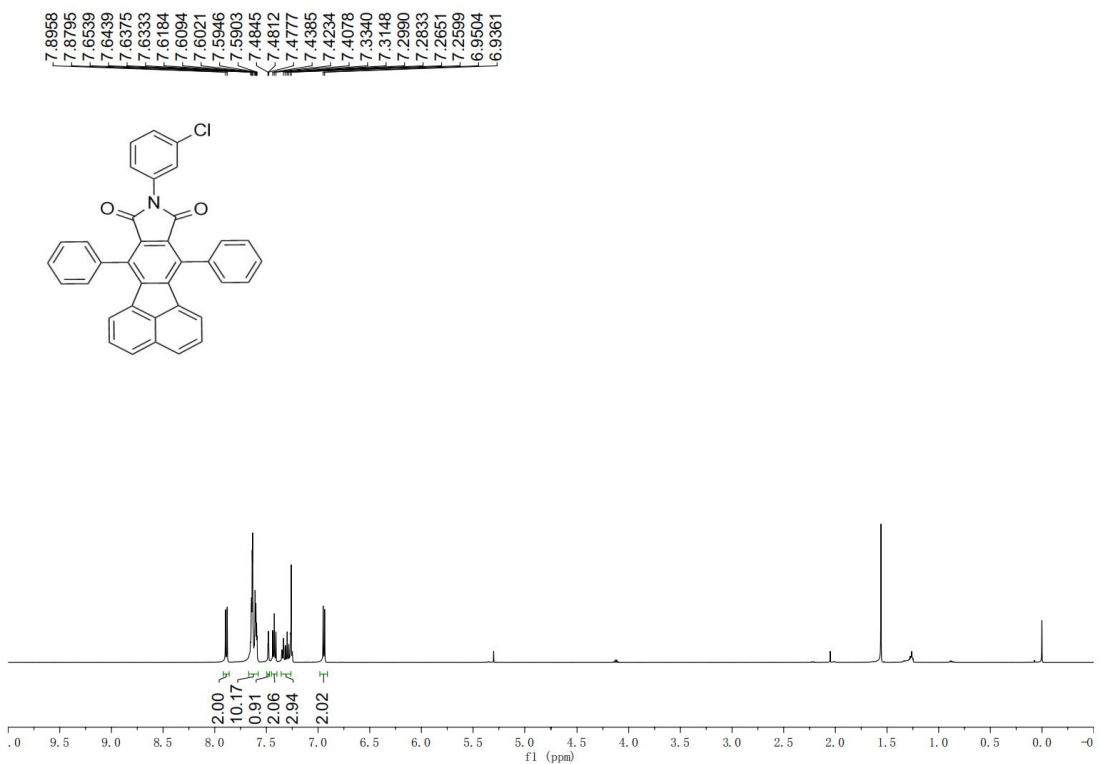
^1H NMR (500 MHz, CDCl_3) of **4d**.



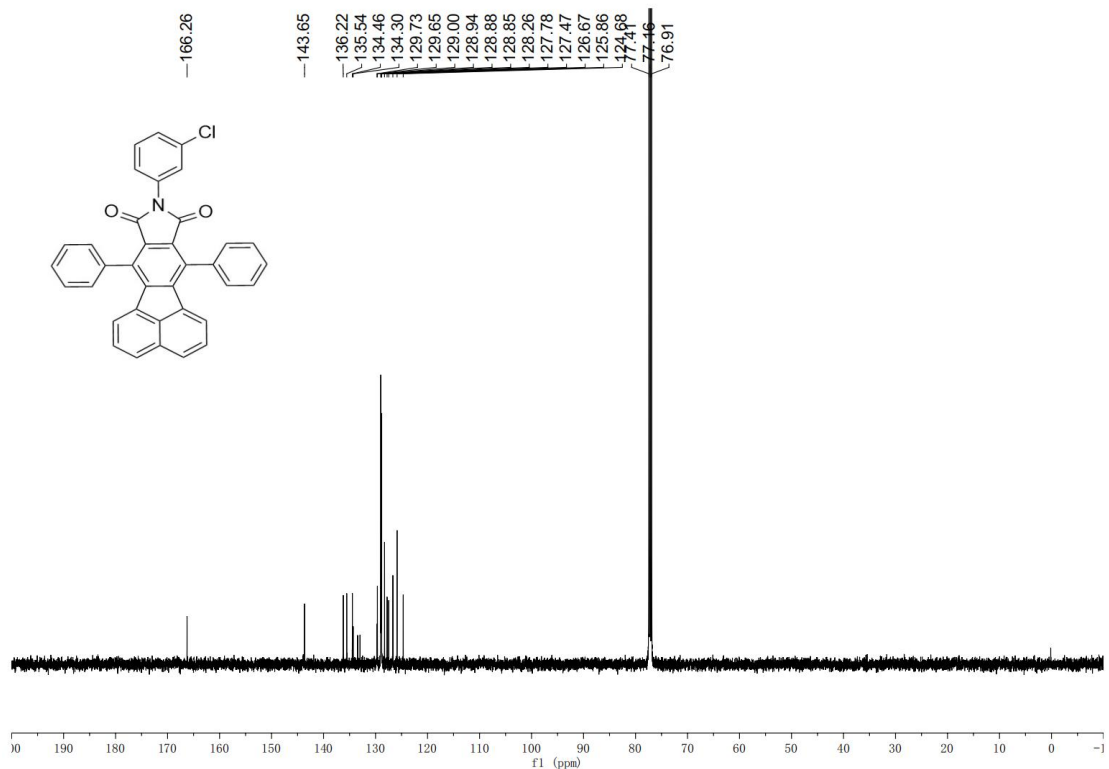
^{13}C NMR (125 MHz, CDCl_3) of **4d**.



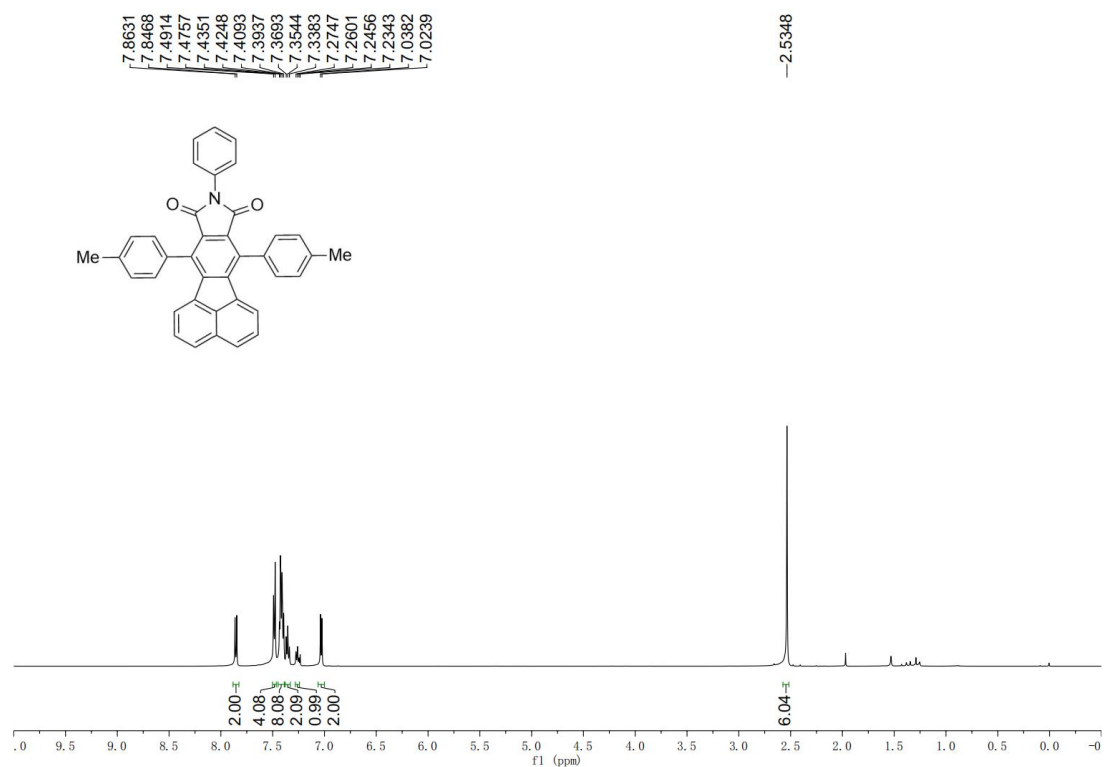
^{19}F NMR (471 MHz, CDCl_3) of **4d**.



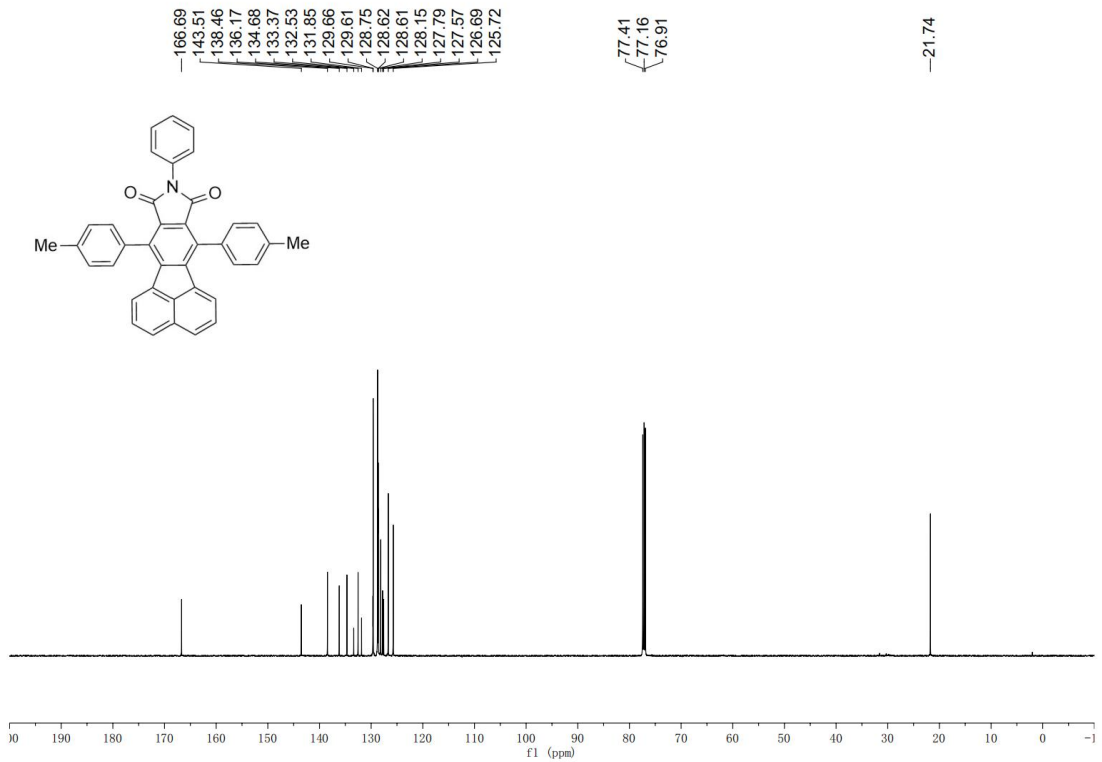
^1H NMR (500 MHz, CDCl_3) of **4e**.



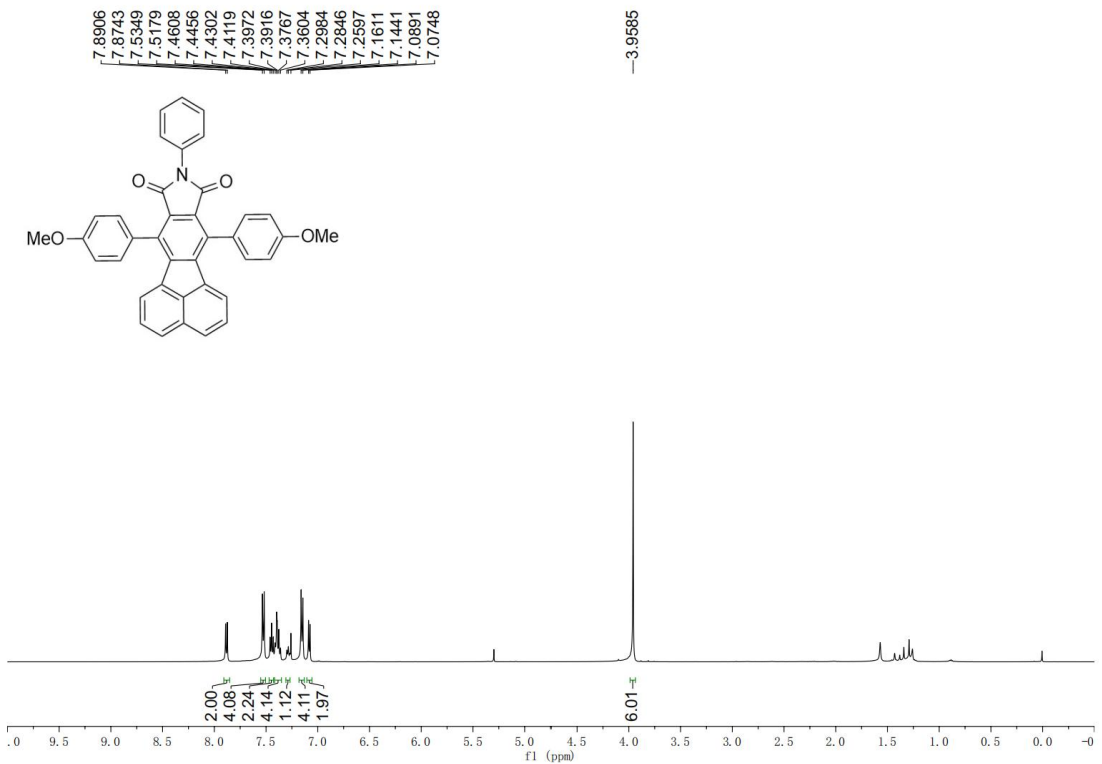
^{13}C NMR (125 MHz, CDCl_3) of **4e**.



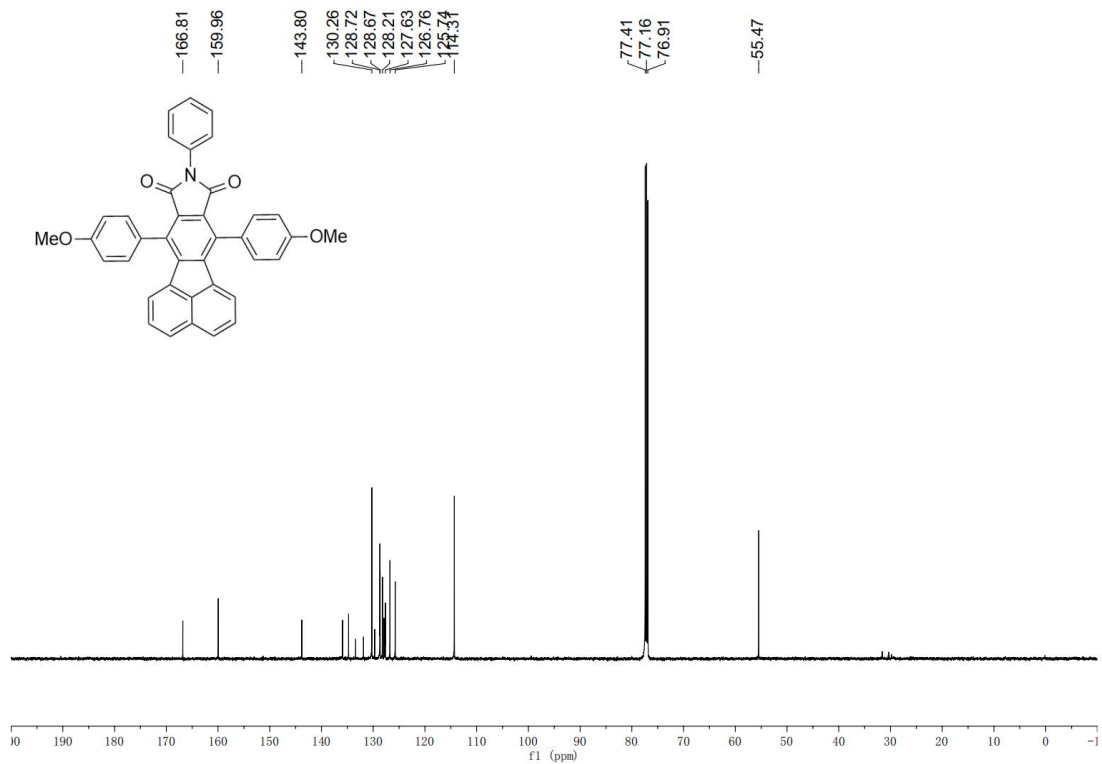
^1H NMR (500 MHz, CDCl_3) of **4f**.



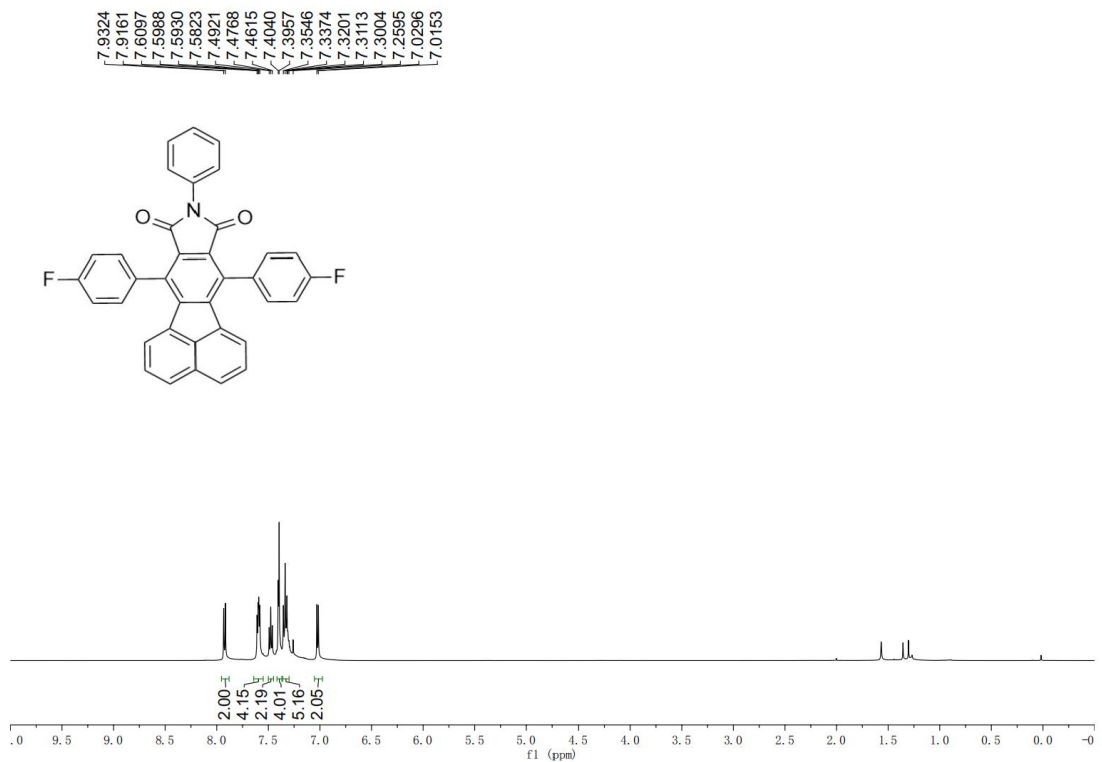
^{13}C NMR (125 MHz, CDCl_3) of **4f**.



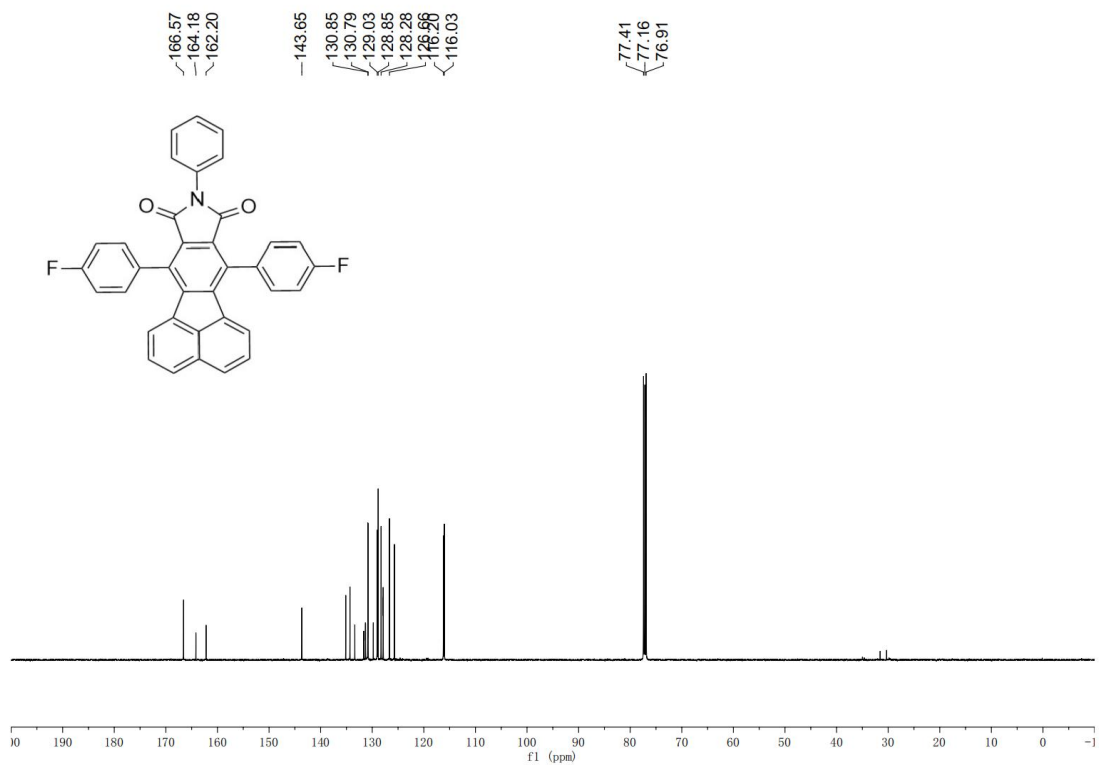
^1H NMR (500 MHz, CDCl_3) of **4g**.



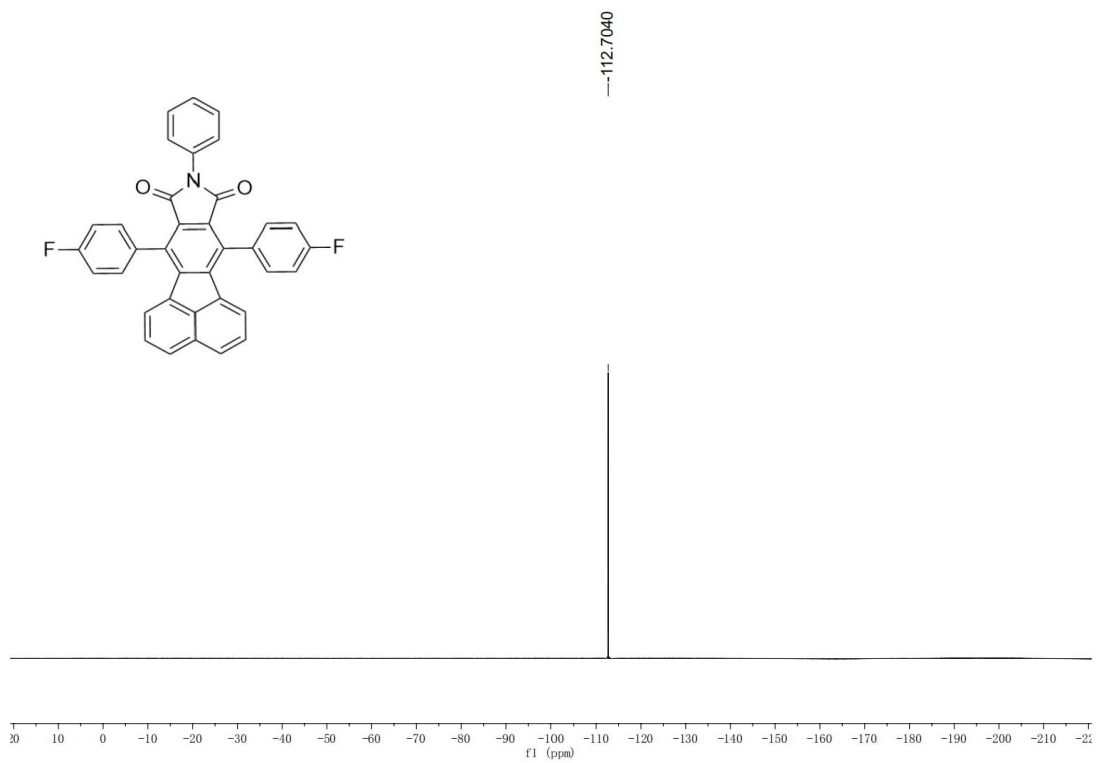
¹³C NMR (125 MHz, CDCl₃) of 4g.



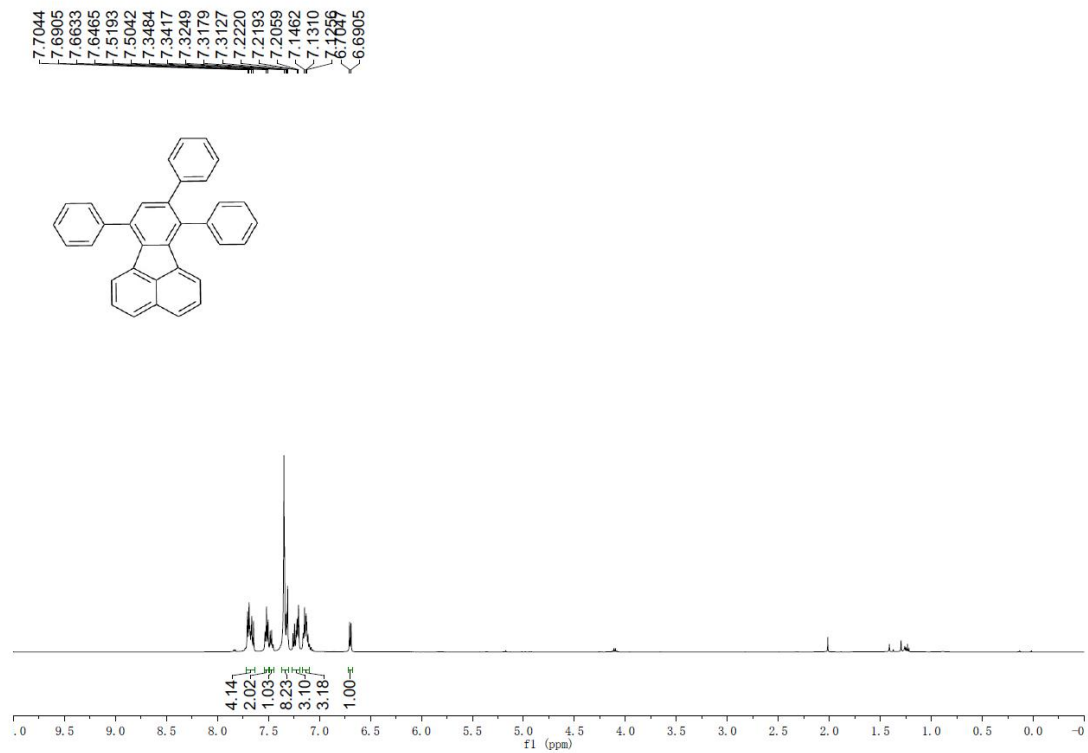
¹H NMR (500 MHz, CDCl₃) of 4h.



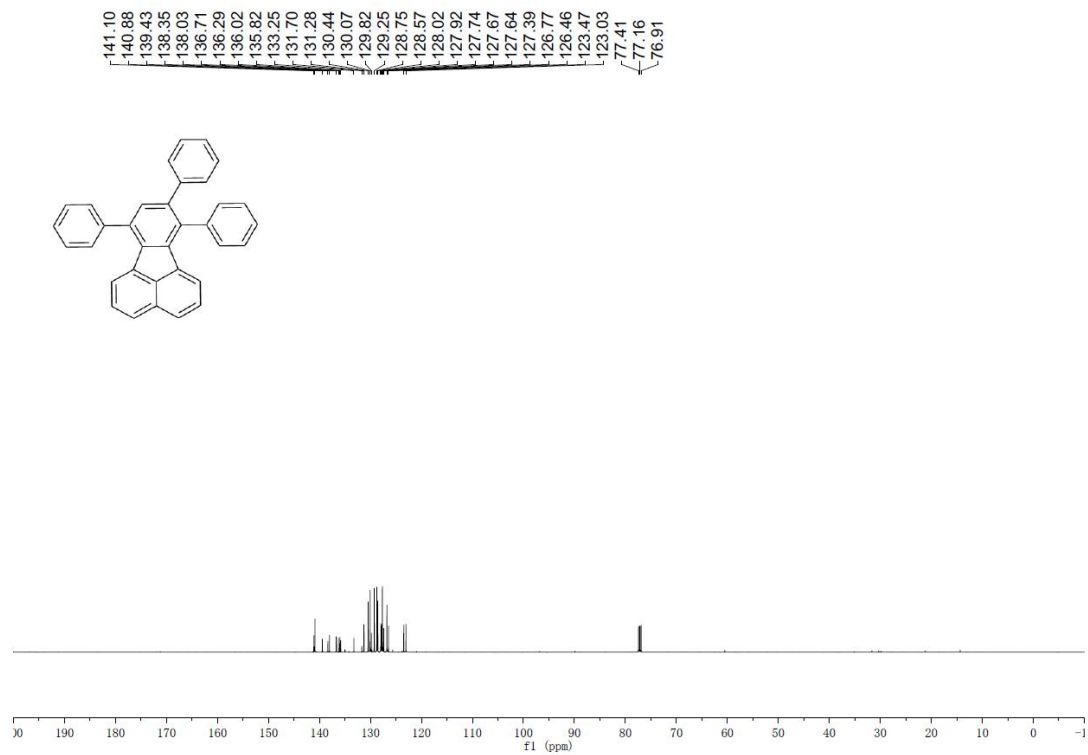
^{13}C NMR (125 MHz, CDCl_3) of **4h**.



^{19}F NMR (471 MHz, CDCl_3) of **4h**.



^1H NMR (500 MHz, CDCl_3) of **5**.



^{13}C NMR (125 MHz, CDCl_3) of **5**.