Supporting Information

Serendipitous Synthesis of Phenanthrene Derivatives by Exploiting Electrocyclization During Thermolysis of Diels-Alder Intermediate Dihydro Dibenzothiophene *S, S*-dioxides

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1. General information

All melting points were uncorrected. Solvents were dried by standard procedures. The progression of all the reaction was monitored by TLC using hexanes/ethyl acetate mixture. Column chromatography was carried out on Silica gel (230-400 mesh, Merck) by increasing polarity. ^{1}H, ^{13}C NMR and DEPT 135 spectra were recorded on Bruker Avance III 300 MHz spectrometer in $CDCl_3$ solvent. Proton chemical shifts are reported in ppm (δ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$ δ 7.26 ppm). NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet), coupling constants (Hz) and integration. ^{13}C Chemical shifts are reported in ppm (δ) from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$ δ 77.06 ppm). HRMS were recorded on Xevo G2S QTOF (ESI) instrument.

2. General procedure for cyclopentadienones & benzo[*b*]thiophene *S, S*-dioxides

Procedure for the preparation of cyclopentadienones 1a-j [1, 2]

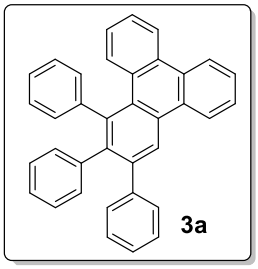
To a mixture of diarylethanedione/acenaphthoquinone and 1,3-disubstituted propanone, ethanol was added and refluxed for 15 min. At the refluxing condition, an ethanolic solution of KOH was added dropwise via addition funnel. The reaction mixture immediately became violet, and a black precipitate was formed. After that, it was cooled to 0 °C for 15 minutes. The solid obtained was filtered and dried to afford cyclopentadienone **1a-j**[1,2] as a deep purple/green/black solid.

Procedure for the preparation of benzo[*b*]thiophene *S, S*-dioxides 2a-c, 8a/b, 11a/b, 13[3-6]

The required benzo[*b*]thiophene *S, S*-dioxides were prepared adopting the published procedure. To a solution of benzo[*b*]thiophene in DCM at 0 °C, a pre-formed mixture of 33% H_2O_2 and HCOOH (1:2 mixture) was slowly added. The reaction mixture was then stirred at rt for 10-12 h. It was then poured into saturated aqueous solution of $NaHCO_3$ and extracted with DCM and dried (Na_2SO_4). Removal of solvent under vacuo followed by trituration with methanol furnished benzo[*b*]thiophene *S, S*-dioxide **2a-c, 8a/b, 11a/b, 13**[3-6] as a colorless/pale yellow solid. 5-Nitro benzo[*b*]thiophene *S, S*-dioxide **2d** was prepared via oxidation of the corresponding 5-nitro benzo[*b*]thiophene using *m*-CPBA in DCM.

3. Experimental Procedures and Analytical data

1,2,3-Triphenyltriphenylene (3a)

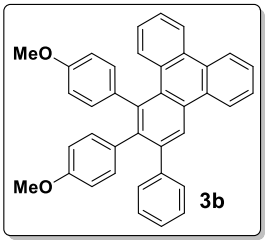


To a solution of cyclopentadienone **1a** (0.23 g, 0.60 mmol) in nitrobenzene (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), the solvent was completely removed by vacuum distillation (90-100 °C, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 100% hexane) afforded triphenylene **3a** (0.17 g, 60%) as a colorless solid. **Mp** 156-158 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.69-8.57 (m, 4H), 7.69-7.66 (m, 2H), 7.58 (d, J = 8.4 Hz, 1H), 7.44 (t, J = 7.2 Hz, 1H), 7.22-7.17 (m, 8H), 7.10-6.98 (m, 6H), 6.85-6.83 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 143.1, 142.2, 140.6, 140.0, 139.6, 131.6, 131.5, 131.4, 130.7, 130.5, 130.2, 130.1, 129.9, 129.8, 128.5, 128.2, 127.5, 127.4, 126.8, 126.4, 126.3, 125.6, 125.1, 124.2, 123.7, 123.2, 123.1 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 131.6, 131.5, 130.1, 129.9, 128.2, 127.5, 127.5, 127.4, 126.8, 126.4, 126.3, 125.6, 125.1, 124.2, 123.7, 123.2, 123.1 ppm; **HRMS (ESI)**: *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{24}$ 457.1956; Found 457.1958.

Diels-Alder reaction of 1a with benzo[*b*]thiophene *S, S*-dioxide 2a in diphenyl ether

The Diels-Alder reaction of cyclopentadienone **1a** (0.23 g, 0.60 mmol) with benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) in diphenyl ether (5 mL) at reflux for 36 h followed by workup and column chromatographic purification (Silica gel, eluent: 100% hexane) to furnish triphenylene **3a** (0.16 g, 46%) as a colorless solid.

1,2-Bis(4-methoxyphenyl)-3-phenyltriphenylene (3b)



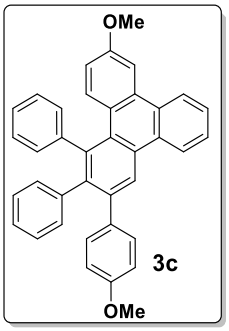
To a solution of cyclopentadienone **1b** (0.27 g, 0.60 mmol) in nitrobenzene (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. Subsequent removal solvent in vacuo (90-100 °C, 10-20 mm Hg) followed by column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) furnished

triphenylene **3b** (0.20 g, 65%) as a colorless solid. **Mp** 198-200 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.64-8.53 (m, 4H), 7.65-7.57 (m, 3H), 7.43 (t, J = 7.8 Hz, 1H), 7.22-7.19 (m, 5H), 7.03 (t, J = 7.8 Hz, 1H), 6.95 (d, J = 8.1 Hz, 2H), 6.71 (t, J = 8.4 Hz, 4H), 6.54 (d, J = 8.4 Hz, 2H), 3.79 (s, 3H), 3.71 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.0, 157.4, 142.5, 140.8, 140.4, 139.5, 135.5, 132.6, 132.4, 131.4, 130.6, 130.5, 130.4, 130.1, 129.9, 129.8, 128.8, 127.6, 127.4, 127.3, 126.3, 126.3, 125.1, 123.9, 123.6, 123.1, 123.1, 113.8, 112.5, 55.2, 55.0 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$): δ 132.6, 132.5, 130.1, 129.8, 127.6, 127.4, 127.3, 126.3, 125.1, 123.9, 123.6, 123.1, 123.1, 113.8, 112.5, 55.2, 55.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_2$ 517.2168; Found 517.2172.

Diels-Alder reaction of 1b with benzo[*b*]thiophene *S, S*-dioxide 2a in diphenyl ether

The Diels-Alder reaction of cyclopentadienone **1b** (0.27 g, 0.60 mmol) with benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) in diphenyl ether (5 mL) at reflux for 36 h followed by workup and column chromatographic purification (Silica gel, 5% ethyl acetate in hexane) yielded triphenylene **3b** (0.11 g, 36%) as a colorless solid.

10-Methoxy-3-(4-methoxyhenyl)-1,2-diphenyltriphenylene (3c)



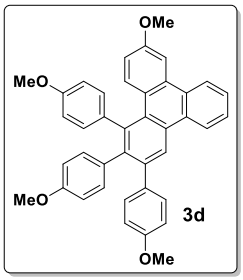
To a solution of cyclopentadienone **1c** (0.27 g, 0.60 mmol) in nitrobenzene (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. Subsequent removal solvent in vacuo (90-100 °C, 10-20 mm Hg) followed by column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave triphenylene **3c** (0.19 g, 62%) as a colorless solid. **Mp** 216-218 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.67-8.64 (m, 2H), 8.54-8.52 (m, 1H), 7.97 (d, J = 2.4 Hz, 1H), 7.66-7.63 (m, 2H), 7.44 (d, J = 9.3 Hz, 1H) 7.16-7.10 (m, 3H), 7.07-7.03 (m, 4H), 6.98-6.96 (m, 3H), 6.82-6.79 (m, 2H), 6.74 (d, J = 8.7 Hz, 2H), 6.60 (dd, J_1 = 9.3 Hz, J_2 = 2.7 Hz, 1H), 3.92 (s, 3H), 3.78 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.1, 157.8, 143.2, 140.6, 140.2, 139.3, 138.8, 134.7, 133.0, 131.6, 131.5, 131.4, 131.1, 130.2, 129.7, 128.4, 128.2, 127.5, 127.3, 126.9, 126.2, 125.5, 124.2, 124.1, 123.7, 123.2, 113.1, 112.9, 106.0, 55.3, 55.2 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 131.6, 131.5, 131.4, 131.1, 128.2, 127.5, 127.3, 126.9, 126.2, 125.5, 124.2,

123.7, 123.2, 113.1, 113.0, 106.0, 55.3, 55.2 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_2$ 517.2168; Found 517.2156.

Diels-Alder reaction of 1c with benzo[*b*]thiophene *S, S*-dioxide 2a in diphenyl ether

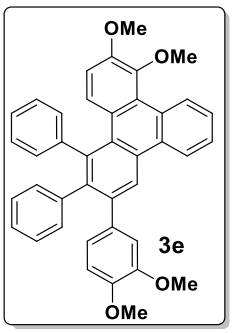
The Diels-Alder reaction of cyclopentadienone **1b** (0.27 g, 0.60 mmol) with benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) in diphenyl ether (5 mL) at reflux for 36 h followed by workup and column chromatographic purification (Silica gel, 5% ethyl acetate in hexane) to produce triphenylene **3c** (0.09 g, 30%) as a colorless solid.

10-Methoxy-1,2,3-tris(4-methoxyphenyl)triphenylene (3d)



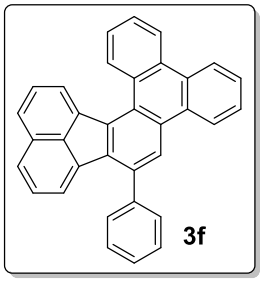
To a solution of cyclopentadienone **1d** (0.30 g, 0.60 mmol) in $PhNO_2$ (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. Subsequent removal solvent in vacuo (90-100 ℃, 10-20 mm Hg) followed by column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave triphenylene **3d** (0.22 g, 64%) as a colorless solid. **Mp** 132-134 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.57-8.54 (m, 1H), 8.52 (s, 1H), 8.47-8.44 (m, 1H), 7.88 (d, J = 2.7 Hz, 1H), 7.57-7.54 (m, 2H), 7.40 (d, J = 9.2 Hz, 1H), 7.01 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 6.70-6.61 (m, 7H), 6.47 (d, J = 8.7 Hz, 2H), 3.85 (s, 3H), 3.72 (s, 6H), 3.64 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.0, 158.0, 157.7, 157.3, 140.5, 139.6, 138.7, 135.7, 135.0, 133.0, 132.6, 132.6, 132.5, 131.3, 131.1, 130.3, 130.2, 129.6, 128.7, 127.4, 127.2, 124.4, 124.0, 123.7, 123.2, 113.8, 113.1, 113.1, 112.5, 106.0, 55.3, 55.2, 55.1 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 132.5, 132.5, 131.3, 131.1, 127.4, 127.2, 124.0, 123.7, 123.2, 113.8, 113.1, 113.1, 112.5, 106.0, 55.3, 55.2, 55.1 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{32}O_4$ 577.2379; Found 577.2379.

3-(3,4-Dimethoxyphenyl)-9,10-dimethoxy-1,2 diphenyltriphenylene (3e)



The triphenylene **3e** was prepared as per the above-mentioned procedure using cyclopentadienone **1h** (0.30 g, 0.60 mmol) and benzo[*b*]thiophene *S*, *S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ at reflux followed by workup and column chromatographic purification (Silica gel, eluent: 10% ethyl acetate in hexane). 0.11 g (44%); **Mp** 254-256 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.46-9.45 (m, 1H), 8.58-8.56 (m, 2H), 7.61-7.58 (m, 2H), 7.36 (d, J = 9.3 Hz, 1H), 7.11-7.10 (m, 3H), 7.02-6.95 (m, 5H), 6.88-6.84 (m, 1H), 6.80-6.76 (m, 3H), 6.63 (d, J = 9.6 Hz, 1H), 6.44 (d, J = 1.8 Hz, 1H), 3.86 (s, 6H), 3.78 (s, 3H), 3.48 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 151.1, 147.6, 147.5, 146.3, 143.1, 140.3, 139.4, 138.6, 134.8, 131.6, 131.5, 130.8, 130.6, 129.2, 128.9, 128.4, 128.1, 127.3, 127.0, 126.3, 126.1, 125.5, 125.5, 123.8, 123.3, 122.1, 113.7, 110.4, 110.2, 59.8, 56.0, 55.7, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{32}O_4$ 577.2379; Found 577.2379.

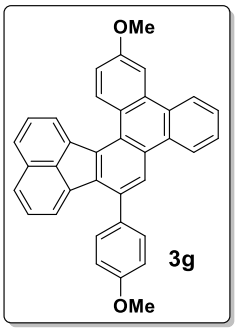
6-Phenylacenaphtho[1,2-*a*]triphenylene (3f)



The Diels-Alder reaction of cyclopentadienone **1f** (0.26 g, 0.60 mmol) with benzo[*b*]thiophene *S*, *S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ (5 mL) at reflux followed by workup and column chromatographic purification (Silica gel, eluent: 100% hexane) afforded acenaphthene fused triphenylene **3f** (0.19 g, 75%) as a fluorescent green solid. **Mp** 184-186 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.45 (d, J = 7.8 Hz, 1H), 8.74 (d, J = 7.2 Hz, 1H), 8.61 (d, J = 8.7 Hz, 3H), 8.41 (s, 1H), 7.87-7.58 (m, 12H), 7.38 (t, J = 7.8 Hz, 1H), 7.22 (d, J = 7.2 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.1, 137.9, 137.6, 137.1, 136.3, 136.1, 132.6, 131.0, 130.6, 130.3, 130.2, 129.9, 129.4, 128.9, 128.8, 128.3, 128.0, 128.0, 127.6, 127.6, 127.4, 127.3, 127.2, 127.1, 125.4, 123.9, 123.8, 123.6, 123.6, 123.4, 123.3 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 129.4, 128.9, 128.8, 128.0, 128.0, 127.6, 127.6, 127.4, 127.3, 127.2, 127.1, 125.4, 123.9, 123.8,

123.6, 123.6, 123.4, 123.3 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{34}H_{20}$ 429.1643; Found 429.1645.

15-Methoxy-6-(4-methoxyphenyl)acenaphtho[1,2-*a*]triphenylene (3g)

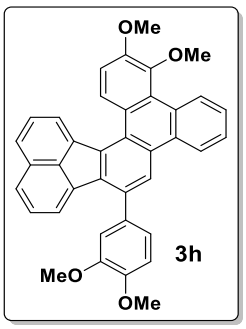


The Diels-Alder reaction of cyclopentadienone **1g** (0.25 g, 0.60 mmol) with benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ (5 mL) at reflux followed by column chromatographic purification (Silica gel, eluent: 5% ethyl acetate in hexane) furnished triphenylene **3g** (0.18 g, 63%) as a fluorescent green solid. **Mp** 190-192°C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.29 (d, J = 8.7 Hz, 1H), 8.64 (d, J = 7.2 Hz, 1H), 8.54-8.49 (m, 2H), 8.32 (s, 1H), 7.97 (s, 1H), 7.81-7.74 (m, 2H), 7.66-7.50 (m, 6H), 7.35 (t, J = 7.8 Hz, 1H), 7.25 (d, J = 6.9 Hz, 1H) 7.16-7.10 (m, 2H), 4.05 (s, 3H), 3.95 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.4, 138.2, 137.3, 136.6, 136.6, 135.3, 133.5, 132.6, 130.6, 130.5, 130.4, 129.9, 129.7, 128.6, 128.3, 127.6, 127.2, 127.1, 127.0, 123.9, 123.8, 123.6, 123.3, 114.1, 113.5, 106.3, 55.5, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 130.5, 130.4, 127.6, 127.2, 127.1, 127.0, 123.9, 123.8, 123.6, 123.3, 114.1, 106.3, 55.5, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{24}O_2$ 489.1855; Found 489.1852.

Diels-Alder reaction of 1h with benzo[*b*]thiophene *S, S*-dioxide 2a

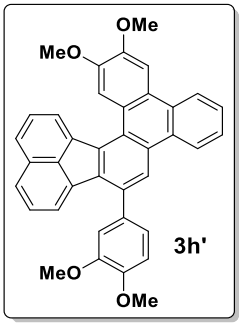
To a solution of cyclopentadienone **1h** (0.29 g, 0.60 mmol) in nitrobenzene (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), the solvent was removed by vacuum distillation (90-100 °C, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 10% ethyl acetate in hexane) gave triphenylene **3h** as a yellow solid. Further elution of the column (eluent: 12% ethyl acetate in hexane) afforded **3h'** also as a yellow solid.

6-(3,4-Dimethoxyphenyl)-15,16-dimethoxyacenaphtho[1,2-*a*]triphenylene (3h)



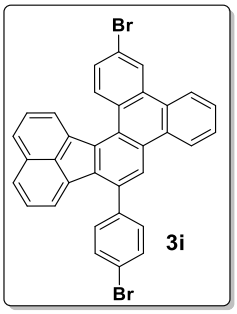
0.10 g (30%); **Mp** 236-238 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.48-9.45 (m, 1H), 9.14 (d, *J* = 9 Hz, 1H), 8.61 (d, *J* = 7.2 Hz, 1H), 8.57-8.48 (m, 1H), 8.29-8.19 (m, 2H), 7.80-7.74 (m, 2H), 7.61-7.52 (m, 3H), 7.38-7.18 (m, 4H), 7.09 (d, *J* = 7.8 Hz, 1H), 4.07 (s, 3H), 4.03 (s, 3H), 3.92 (s, 3H), 3.88 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 149.0, 148.8, 146.8, 138.1, 137.3, 136.9, 136.5, 135.2, 133.8, 133.4, 132.7, 131.3, 130.4, 129.9, 129.3, 129.0, 128.9, 128.6, 127.7, 127.5, 127.4, 127.2, 127.0, 125.4, 125.0, 124.7, 123.6, 123.4, 123.2, 121.4, 112.6, 111.5, 110.7, 59.7, 56.3, 56.1 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 128.6, 127.7, 127.5, 127.2, 127.0, 125.0, 123.6, 123.4, 123.2, 121.4, 112.6, 111.4, 110.7, 59.7, 56.3, 56.1 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_4$ 549.2066; Found 549.2064.

6-(3,4-Dimethoxyphenyl)-14,15-dimethoxyacenaphtho[1,2-*a*]triphenylene (3h')



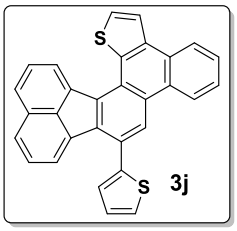
0.15 g (45%); **Mp** 264-266 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.83 (s, 1H), 8.69 (d, *J* = 7.2 Hz, 1H), 8.53 (d, *J* = 7.8 Hz, 1H), 8.43 (d, *J* = 7.8 Hz, 1H), 8.36 (s, 1H), 7.92 (s, 1H), 7.79-7.73 (m, 2H), 7.62-7.48 (m, 3H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.26-7.24 (m, 3H), 7.08 (d, *J* = 7.8 Hz, 1H), 4.14 (s, 3H), 4.01 (s, 3H), 3.91 (s, 3H), 3.90 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 149.7, 149.0, 148.8, 147.3, 138.1, 137.2, 136.6, 136.6, 135.2, 133.8, 132.6, 130.1, 130.0, 130.0, 129.6, 127.9, 127.8, 127.5, 127.2, 127.1, 126.8, 126.6, 125.6, 124.0, 123.9, 123.8, 123.6, 123.6, 122.8, 121.4, 112.6, 111.5, 110.7, 104.9, 56.1, 56.0 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 127.8, 127.5, 127.2, 127.1, 126.8, 126.6, 124.0, 123.9, 123.8, 123.6, 122.8, 121.4, 112.6, 111.4, 110.7, 104.9, 56.1, 56.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_4$ 549.2066; Found 549.2067.

15-Bromo-6-(4-bromophenyl)acenaphtho[1,2-*a*]triphenylene (3i)



The Diels-Alder reaction of cyclopentadienone **1i** (0.31 g, 0.60 mmol) with benzo[*b*]thiophene *S*, *S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ (5 mL) at reflux for 12 h followed by workup and column chromatographic purification on silica gel (eluent: 100% hexane) furnished acenaphthene based triphenylene **3i** (0.25 g, 70%) as a fluorescent green solid. **Mp** 272-274 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.30 (d, J = 8.4 Hz, 1H), 8.73 (s, 1H), 8.65 (d, J = 6.9 Hz, 1H), 8.55 (m, 2H), 8.34 (s, 1H), 7.89-7.59 (m, 9H), 7.44 (t, J = 7.5 Hz, 1H), 7.28-7.25 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 139.9, 137.5, 137.1, 136.1, 135.9, 132.9, 132.6, 132.0, 131.0, 130.5, 130.5, 130.4, 130.0, 129.0, 128.5, 128.3, 128.0, 127.8, 127.8, 127.7, 127.5, 127.3, 126.5, 124.0, 123.8, 123.5, 123.4, 122.3, 122.2 ppm; **HRMS (ESI)** *m/z*: $[M-2Br]^+$ Calcd for $C_{34}H_{18}Br_2$ 426.1409; Found 426.1408.

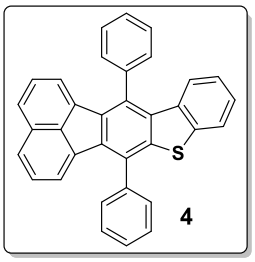
9-(Thiophen-2-yl)acenaphtho[1',2':7,8]phenanthrol[9,10-*b*]thiophene (3j)



The Diels-Alder reaction of cyclopentadienone **1j** (0.22 g, 0.60 mmol) with benzo[*b*]thiophene *S*, *S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ (5 mL) at reflux for 12 h followed by workup and column chromatographic purification on silica gel (eluent: 100% hexane) gave thieno[*b*]phenanthrene **3j** (0.17 g, 69%) as a yellow solid. **Mp** 170-172 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.48 (d, J = 7.2 Hz, 1H), 8.64 (m, 2H), 8.35-8.32 (m, 1H), 8.00 (d, J = 5.4 Hz, 1H), 7.90 (d, J = 8.1 Hz, 1H), 7.84 (d, J = 8.1 Hz, 1H), 7.71-7.56 (m, 5H), 7.48-7.36 (m, 3H), 7.30-7.27 (m, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.8, 138.3, 137.4, 136.5, 135.8, 135.0, 133.5, 132.4, 129.8, 129.8, 129.5, 128.9, 128.4, 127.8, 127.7, 127.6, 127.4, 127.3, 127.3, 127.2, 126.9, 126.6, 126.0, 125.5, 124.7, 124.2, 124.1, 123.8, 122.9 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 127.8, 127.7, 127.6, 127.4, 127.4, 127.3, 127.2, 126.9, 126.6, 126.0, 124.7,

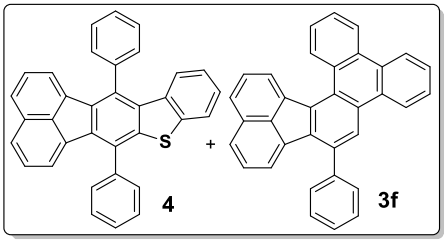
124.2, 124.1, 123.8, 122.9 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{30}H_{16}S_2$ 441.0772; Found 441.0775.

Diels-Alder reaction of 1f with benzo[*b*]thiophene *S, S*-dioxide 2a in NMP



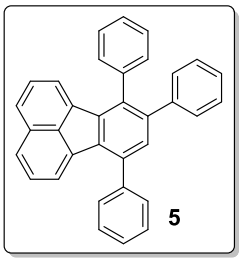
To a solution of cyclopentadienone **1f** (0.26 g, 0.60 mmol) in *N*-methyl-2-pyrrolidone (NMP) (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), it was poured over ice water (15 mL) containing HCl (0.5 mL). The solid obtained was filtered and dried. The crude product was triturated with methanol (10 mL) to furnish known 7,13-diphenylbenzo[*b*]fluorantheno[8,9-*d*]thiophene **4**[14] (0.25 g, 68%) as a yellow solid.

Diels-Alder reaction of 1f with benzo[*b*]thiophene *S, S*-dioxide 2a in DEG



To a solution of cyclopentadienone **1f** (0.26 g, 0.60 mmol) in diethylene glycol (DEG) (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), it was poured over ice water (15 mL). The solid obtained was filtered and dried. The crude product was triturated with methanol (10 mL) to afford a 1:1 (based on ^{1}H NMR integration) mixture of 7,13-diphenylbenzo[*b*]fluorantheno[8,9-*d*]thiophene **4** and 6-phenylacenaphtho[1,2-*a*]triphenylene **3f** (0.19 g, 75%) as a yellow solid.

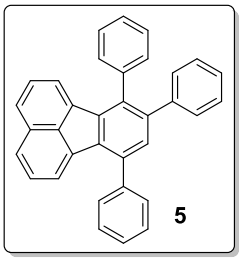
Diels-Alder reaction of 1f with phenyl acetylene



To a solution of cyclopentadienone **3a** (0.35 g, 0.98 mmol) in nitrobenzene (5 mL), phenyl acetylene (0.10 g, 0.98 mmol) was added and refluxed for 6 h. After completion of reaction

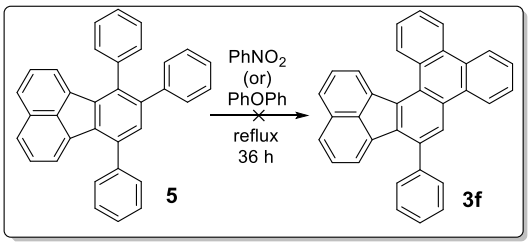
(TLC), the solvent was removed by vacuum distillation (90-100 °C, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 100% hexane) afforded 7,8,10-triphenylfluoranthene **5** (0.25 g, 68%) as a colorless solid. **Mp** 182-184 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.75 (d, J = 6.9 Hz, 4H), 7.61-7.55 (m, 3H), 7.41-7.32 (m, 9H), 7.30-7.19 (m, 5H), 6.72 (d, J = 6.9 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.0, 140.8, 139.4, 138.3, 138.0, 136.6, 136.2, 136.0, 135.7, 133.2, 131.2, 130.4, 130.0, 129.7, 129.2, 128.7, 128.5, 127.9, 127.7, 127.6, 127.3, 126.7, 126.4, 123.4, 123.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{34}H_{22}$ 431.1800; Found 431.1799.

Diels-Alder reaction of 1f with benzo[*b*]thiophene *S, S*-dioxide 2a using K_2CO_3



To a solution of cyclopentadienone **1f** (0.26 g, 0.60 mmol) in nitrobenzene (5 mL), benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol), K_2CO_3 (0.17 g, 1.21 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent by vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) afforded 7,8,10-triphenylfluoranthene **5** (0.15 g, 59%) as a colorless solid.

Thermal electrocyclization of compound 5

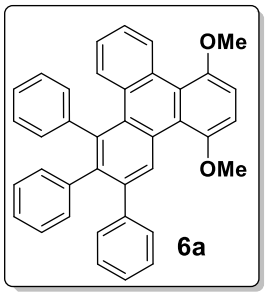


To a solution of 7,8,10-triphenylfluoranthene **5** in PhOPh or $PhNO_2$ (5 mL**)** at reflux for 26 h. The reaction was monitered by TLC, and the starting material was recovered.

Diels-Alder reaction of 1f with benzo[*b*]thiophene *S, S*-dioxide 2a

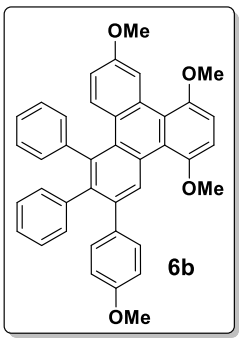
A solution of cyclopentadienone **1f** (0.26 g, 0.60 mmol) with benzo[*b*]thiophene *S, S*-dioxide **2a** (0.10 g, 0.60 mmol) in $PhNO_2$ (5 mL) was stirred at 150 °C in an (oil bath) for 36 h. Subsequent removal of solvent followed by column chromatographic purification (Silica gel, eluent: 100% hexane) to give 7,13-diphenylbenzo[*b*]fluorantheno[8,9-*d*]thiophene **4**[14] (0.05 g, 20%) as a yellow solid.

5,8-Dimethoxy-1,2,3-triphenyltriphenylene (6a)



To a solution of cyclopentadienone **1a** (0.17 g, 0.44 mmol) in $PhNO_2$ (5 mL), 4,7-dimethoxybenzo[*b*]thiophene *S, S*-dioxide **2b** (0.10 g, 0.44 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent by vacuum distillation (90-100 °C, 10-20 mm Hg) followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) furnished triphenylene **6a** (0.19 g, 82%) as a colorless solid. **Mp** 214-216 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.54 (s, 1H), 9.10 (d, J = 8.4 Hz, 1H), 7.45 (d, J = 8.1 Hz, 1H), 7.28-7.24 (m, 1H), 7.16-6.87 (m, 16H), 6.79-6.77 (m, 2H), 4.00 (s, 3H), 3.96 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 152.4, 151.7, 142.8, 140.2, 140.0, 138.2, 132.1, 131.7, 130.6, 130.5, 130.5, 130.2, 129.7, 129.5, 128.3, 127.9, 127.4, 127.1, 126.8, 126.0, 125.6, 125.5, 124.8, 123.7, 122.9, 111.1, 110.9, 56.9, 56.6 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 132.1, 131.7, 130.2, 129.8, 128.3, 127.8, 127.0, 126.8, 126.0, 125.6, 125.5, 124.8, 111.1, 110.9, 56.9, 56.6 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_2$ 516.2168; Found 516.2166.

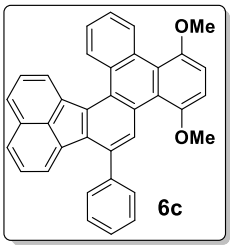
5,8,10-Trimethoxy-3-(4-methoxyphenyl)-1,2-diphenyltriphenylene (6b)



To a solution of cyclopentadienone **1c** (0.20 g, 0.44 mmol) in nitrobenzene (5 mL), 4,7-dimethoxybenzo[*b*]thiophene *S, S*-dioxide **2b** (0.10 g, 0.44 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent by vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) furnished triphenylene **6b** (0.21 g, 82%) as a colorless solid. **Mp** 202-204 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.49 (s, 1H), 8.68 (s, 1H), 7.35 (d, J = 9.3 Hz, 1H), 7.24-6.95 (m, 13H), 6.78-6.77 (m, 2H), 6.70 (d, J = 8.4 Hz, 2H), 6.54-6.50 (m, 1H), 4.00 (s, 3H), 3.96 (s, 3H), 3.83

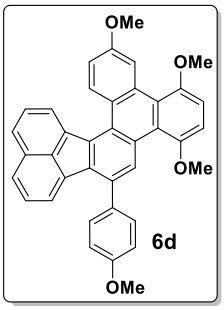
(s, 3H), 3.75 (s, 3H) ppm; **$^{13}C\{^{1}H\}$ NMR** (75 MHz, $CDCl_3$): δ 157.8, 156.9, 152.5, 151.8, 143.1, 140.5, 140.0, 138.7, 137.6, 135.4, 132.1, 131.9, 131.7, 131.2, 130.9, 129.7, 129.3, 128.6, 127.8, 126.9, 125.9, 125.4, 124.5, 123.6, 123.4, 112.9, 112.9, 111.3, 111.0, 110.2, 56.9, 56.8, 55.1 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$): δ 132.1, 131.7, 131.2, 131.0, 128.6, 127.9, 126.9, 125.9, 125.4, 112.9, 111.3, 111.0, 110.2, 56.9, 56.8, 55.1 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{32}O_4$ 577.2379; Found 577.2377.

1,4-Dimethoxy-6-phenylacenaphtho[1,2-*a*]triphenylene (6c)



To a solution of cyclopentadienone **1f** (0.16 g, 0.44 mmol) in $PhNO_2$ (5 mL), 4,7-dimethoxybenzo[*b*]thiophene *S, S*-dioxide **2b** (0.10 g, 0.44 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent by vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded acenaphthene fused triphenylene **6c** (0.20 g, 91%) as a yellow solid. **Mp** 194-196 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.37 (s, 1H), 9.26-9.17 (m, 2H), 8.60 (d, J =7.2 Hz, 1H), 7.81-7.74 (m, 4H), 7.62-7.45 (m, 6H), 7.35 (t, J = 8.1 Hz, 1H), 7.26 (d, J = 6.6 Hz, 1H), 7.14-7.07 (m, 2H), 4.03 (s, 3H), 3.98 (s, 3H) ppm; **$^{13}C\{^{1}H\}$ NMR** (75 MHz, $CDCl_3$): δ 152.5, 152.0, 141.8, 138.3, 136.8, 136.6, 136.3, 134.6, 132.8, 130.2, 130.0, 129.9, 129.7, 129.4, 128.7, 128.7, 128.2, 128.0, 127.7, 127.6, 127.3, 127.2, 126.9, 125.6, 123.5, 123.1, 122.9, 122.8, 111.5, 110.9, 56.8, 56.6 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 129.4, 128.8, 128.7, 128.6, 128.0, 127.7, 127.6, 127.3, 127.2, 126.9, 125.6, 123.5, 123.1, 111.5, 110.9, 56.8, 56.6 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{24}O_2$ 489.1855; Found 489.1852.

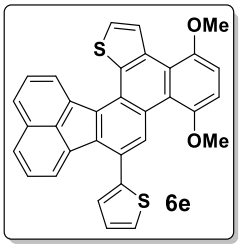
1,4,15-Trimethoxy-6-(4-methoxyphenyl)acenaphtho[1,2-*a*]triphenylene (6d)



To a solution of cyclopentadienone **1g** (0.18 g, 0.44 mmol) in $PhNO_2$ (5 mL), 4,7-dimethoxybenzo[*b*]thiophene *S, S*-dioxide **2b** (0.10 g, 0.44 mmol) was added and refluxed for

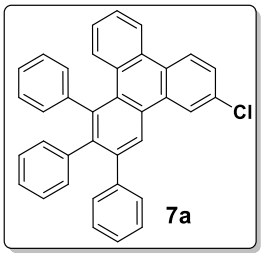
12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 10% ethyl acetate in hexane) gave triphenylene **6d** (0.17 g, 69%) as a yellow solid. **Mp** 238-240 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.32 (s, 1H), 9.14 (d, J = 9 Hz, 1H), 8.76 (d, J = 2.4 Hz, 1H), 8.55 (d, J = 7.2 Hz, 1H), 7.79-7.69 (m, 4H), 7.50 (t, J = 7.8 Hz, 1H), 7.39-7.32 (m ,2H), 7.14-7.08 (m, 5H), 4.05 (s, 3H), 4.00 (s, 6H), 3.97 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.3, 158.5, 152.6, 152.1, 138.5, 136.8, 136.4, 135.7, 134.2, 133.9, 132.8, 131.1, 130.5, 129.9, 129.4, 129.4, 129.3, 129.0, 127.6, 127.3, 126.8, 126.7, 124.1, 123.9, 123.3, 123.0, 122.8, 114.0, 113.7, 111.7, 111.2, 111.0, 56.8 (2C), 55.4 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 130.5, 129.4, 129.0, 127.6, 127.3, 126.8, 126.7, 123.2, 123.0, 114.0, 113.7, 111.7, 111.2, 111.0, 56.8, 56.7, 55.4 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{28}O_4$ 549.2066; Found 549.2065.

4,7-Dimethoxy-9-(thiophen-2-yl)acenaphtho[1'2':7,8]phenanthrol[9,10-*b*]thiophene (6e)



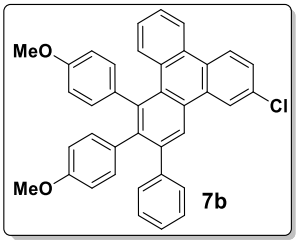
To a solution of cyclopentadienone **1j** (0.16 g, 0.44 mmol) in $PhNO_2$ (5 mL), 4,7-dimethoxybenzo[*b*]thiophene *S, S*-dioxide **2b** (0.10 g, 0.44 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded thieno[*b*]phenanthrene **6e** (0.16 g, 70%) as a yellow solid. **Mp** 120-122 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.45 (s, 1H), 9.31 (d, J = 7.2 Hz, 1H), 8.60 (d, J = 5.7 Hz, 1H), 7.87-7.79 (m, 2H), 7.64-7.41 (m, 6H), 7.27-7.25 (m, 1H), 7.41 (s, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 152.4, 150.8, 142.8, 136.9, 136.1, 135.7, 134.3, 133.8, 132.3, 132.2, 129.9, 128.9, 128.3, 127.6, 127.6, 127.5, 127.4, 127.1, 126.9, 126.4, 125.7, 124.2, 123.6, 122.3, 121.9, 121.5, 110.2, 109.0, 56.8, 56.2 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 132.2, 128.3, 127.6, 127.5, 127.4, 127.1, 126.9, 125.7, 124.2, 123.6, 122.3, 110.2, 109.0, 56.8, 56.2 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{32}H_{22}O_2S_2$ 501.0983; Found 501.0986.

6-Chloro-1,2,3-triphenyltriphenylene (7a)



To a solution of cyclopentadienone **1a** (0.19 g, 0.50 mmol) in nitrobenzene (5 mL), 5-chlorobenzo[*b*]thiophene *S, S*-dioxide **2c** (0.10 g, 0.50 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) led to triphenylene **7a** (0.17 g, 69%) as a colorless solid. **Mp** 230-232 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.59 (s, 1H), 8.56 (s, 1H), 8.52-8.45 (m, 2H), 7.60-7.52 (m, 2H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.25-7.15 (m, 8H), 7.05-6.96 (m, 6H), 6.80-6.79 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 142.8, 141.9, 141.2, 140.9, 139.8, 133.5, 131.5, 131.4, 131.3, 130.7, 130.1, 130.0, 129.9, 129.6, 128.9, 128.2, 127.7, 127.6, 126.9, 126.6, 126.5, 126.4, 125.7, 125.4, 124.8, 124.2, 123.4, 123.0 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 131.5, 131.4, 130.0, 128.3, 127.6, 126.9, 126.6, 126.5, 126.4, 125.7, 125.4, 124.8, 124.2, 123.4, 123.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{23}Cl$ 491.1567; Found 491.1569.

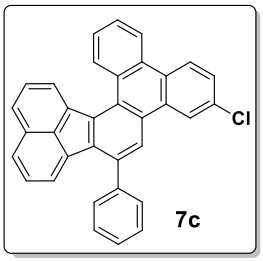
6-Chloro-1,2-bis(4-methoxyphenyl)-3-phenyltriphenylene (7b)



To a solution of cyclopentadienone **1b** (0.22 g, 0.50 mmol) in $PhNO_2$ (5 mL), 5-chlorobenzo[*b*]thiophene *S, S*-dioxide **2c** (0.10 g, 0.50 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) furnished triphenylene **7b** (0.18 g, 65%) as a colorless solid. **Mp** 210-212 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.57 (d, *J* = 1.8 Hz, 1H), 8.52-8.44 (m, 3H), 7.59-7.55 (m, 2H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.25-7.15 (m, 5H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.74-6.67 (m, 4H), 6.54 (d, *J* = 8.4 Hz, 2H), 3.79 (s, 3H), 3.70 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.1, 157.4, 142.2, 141.1, 139.6, 135.2, 133.4, 132.5, 132.4, 132.1, 131.4, 130.6, 130.4, 130.0, 129.9, 129.4, 129.2, 128.9, 127.7, 127.5, 126.5, 126.4, 125.4, 124.8, 123.9, 123.4, 122.9, 113.8, 112.5, 55.2, 55.0 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 132.5, 132.4, 130.0,

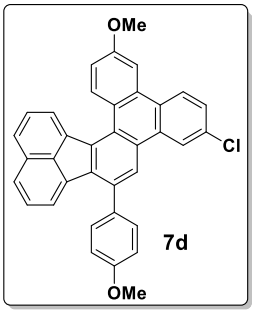
129.9, 127.7, 127.5, 126.5, 126.4, 125.4, 124.8, 124.0, 123.4, 122.9, 113.8, 112.5, 55.2, 55.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{27}ClO_2$ 551.1778; Found 551.1777.

3-Chloro-6-phenylacenaphtho[1,2-*a*]triphenylene (7c)



To a solution of cyclopentadienone **1f** (0.18 g, 0.50 mmol) in $PhNO_2$ (5 mL), 5-chlorobenzo[*b*]thiophene *S, S*-dioxide **2c** (0.10 g, 0.50 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) afforded acenaphthene incorporated triphenylene **7c** (0.21 g, 91%) as a fluorescent green solid. **Mp** 200-202 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.39 (d, J = 7.8 Hz, 1H), 8.68 (d, J = 6.9 Hz, 1H), 8.51-8.48 (m, 3H), 8.26 (s, 1H), 7.86-7.52 (m, 11H), 7.36 (t, J = 7.8 Hz, 1H), 7.20 (d, J = 6.9 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 140.8, 137.9, 137.8, 137.7, 136.1, 133.6, 132.6, 131.7, 130.3, 130.1, 129.9, 129.5, 129.4, 129.3, 128.9, 128.8, 128.6, 128.5, 128.2, 128.1, 127.7, 127.6, 127.4, 127.3, 127.2, 125.7, 124.8, 123.9, 123.7, 123.5, 123.4 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 129.3, 128.9, 128.8, 128.2, 128.1, 127.7, 127.6, 127.4, 127.3, 127.2, 125.7, 124.8, 123.9, 123.6, 123.5, 123.4 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{34}H_{19}Cl$ 463.1254; Found 463.1255.

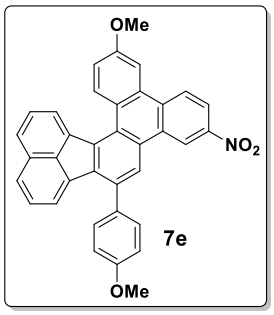
3-Chloro-15-methoxy-6-(4-methoxyphenyl)acenaphtho[1,2-*a*]triphenylene (7d)



To a solution of cyclopentadienone **1g** (0.21 g, 0.50 mmol) in $PhNO_2$ (5 mL), 5-chlorobenzo[*b*]thiophene *S, S*-dioxide **2c** (0.10 g, 0.50 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave acenaphthene fused triphenylene **7d** (0.19 g, 85%) as a yellow solid. **Mp** 212-

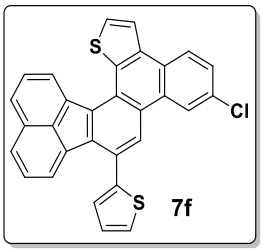
214 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.19 (d, *J* = 9 Hz, 1H), 8.56 (d, *J* = 7.2 Hz, 1H), 8.37-8.32 (m, 2H), 8.12 (s, 1H), 7.83 (s, 1H), 7.76 (t, *J* = 8.7 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.51-7.46 (m, 2H), 7.33 (t, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 6.8 Hz, 1H), 7.12-7.06 (m, 3H), 4.00 (s, 3H), 3.93 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.5, 159.5, 137.9, 137.8, 136.7, 136.4, 135.3, 133.6, 133.2, 132.6, 132.1, 131.9, 130.4, 129.9, 128.5, 128.4, 128.2, 127.6, 127.3, 127.2, 124.8, 123.8, 123.6, 123.5, 123.2, 114.1, 113.7, 106.2, 55.5, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 130.4, 127.6, 127.3, 127.2, 124.8, 123.8, 123.6, 123.5, 114.1, 113.7, 106.2, 55.5, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{23}ClO_2$ 523.1465; Found 523.1459.

15-Methoxy-6-(4-methoxyphenyl)-3-nitroacenaphtho[1,2-a]triphenylene (7e)



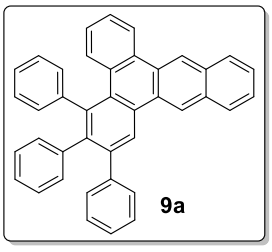
To a solution of cyclopentadienone **1g** (0.20 g, 0.47 mmol) in $PhNO_2$ (5 mL), 5-nitrobenzo[*b*]thiophene *S, S*-dioxide **2d** (0.10 g, 0.47 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded acenaphthene fused triphenylene **7e** (0.16 g, 65%) as a yellow solid. **Mp** 234-236 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.15-9.12 (m, 2H), 8.47 (d, *J* = 7.2 Hz, 1H), 8.40 (d, *J* = 9 Hz, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 8.1 (s, 1H), 7.79-7.73 (m, 3H), 7.54-7.43 (m, 3H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.23-7.13 (m, 2H), 7.04 (d, *J* = 8.1 Hz, 2H), 4.00 (s, 3H), 3.92 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.6, 159.6, 146.6, 138.4, 137.5, 137.1, 136.0, 135.4, 134.3, 132.6, 131.0, 130.8, 130.5, 130.4, 129.9, 128.3, 128.3, 127.7, 127.6, 127.5, 127.2, 124.5, 124.4, 123.9, 123.8, 123.7, 120.7, 119.6, 115.4, 114.2, 107.0, 55.6, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 130.5, 130.4, 127.7, 127.6, 127.5, 127.2, 124.4, 123.9, 123.8, 123.7, 120.7, 119.6, 115.4, 114.2, 107.0, 55.6, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{36}H_{23}NO_4$ 534.1705; Found 534.1702.

6-Chloro-9-(thiopheny-2-yl)acenaphtho[1'2':7,8]phenanthrol[9,10-*b*]thiophene (7f)



To a solution of cyclopentadienone **1j** (0.18 g, 0.50 mmol) in $PhNO_2$ (5 mL), 5-chlorobenzo[*b*]thiophene *S, S*-dioxide **2c** (0.10 g, 0.50 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) to give thieno[*b*]phenanthrene **7f** (0.16 g, 69%) as a yellow solid. **Mp** 240-242 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.33 (d, J = 7.2 Hz, 1H), 8.46 (s, 1H), 8.40 (s, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.83-7.74 (m, 3H), 7.57 (t, J = 7.8 Hz, 1H), 7.49-7.47 (m, 3H), 7.36 (t, J = 7.8 Hz, 1H), 7.28-7.26 (m, 2H), 7.21-7.19 (m, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.5, 138.9, 136.7, 136.3, 135.5, 135.1, 133.7, 132.5, 132.4, 132.4, 130.8, 129.8, 128.7, 128.0, 127.8, 127.8, 127.6, 127.5, 127.4, 127.2, 127.2, 126.9, 126.2, 125.8, 125.6, 124.9, 124.5, 124.1, 123.9, 122.7 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 128.0, 127.8, 127.7, 127.6, 127.5, 127.4, 127.2, 126.9, 126.2, 125.6, 124.9, 124.5, 124.1, 123.9, 122.7 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{30}H_{15}ClS_2$ 475.0382; Found 475.0378.

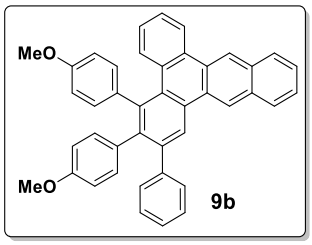
2,3,4-Triphenylbenzo[*f*]tetraphene (9a)



To a solution of cyclopentadienone **1a** (0.18 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) led to naphthalene fused phenanthrene **9a** (0.17 g, 71%) as a colorless solid. **Mp** 246-248 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.07 (s, 1H), 9.03 (s, 1H), 8.78 (s, 1H), 8.65 (d, J = 8.4 Hz, 1H), 8.06 (dd, J_1 = 8.4 Hz, J_2 = 6.6 Hz, 2H), 7.59-7.54 (m, 2H), 7.49-7.40 (m, 2H), 7.26-6.82 (m, 14H), 6.81-6.79 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 142.8, 142.2, 140.9, 140.8, 139.9, 139.7, 132.5, 132.4, 131.8, 131.5, 131.5, 131.0, 130.5, 130.0, 129.2, 128.9, 128.6, 128.2,

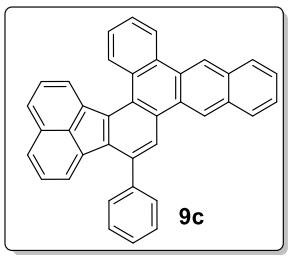
128.0, 127.6, 126.8, 126.6, 126.4, 126.3, 126.2, 126.0, 125.6, 125.5, 124.4, 123.7, 122.5, 121.7 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 131.6, 131.5, 130.2, 130.1, 128.2, 128.2, 127.6, 126.9, 126.7, 126.5, 126.3, 126.2, 126.1, 125.6, 125.5, 124.6, 123.7, 122.5, 121.8,ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{26}$ 507.2113; Found 507.2111.

3,4-Bis(4-methoxyphenyl)-2-phenylbenzo[*f*]tetraphene (9b)



To a solution of cyclopentadienone **1b** (0.21 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave naphthalene fused phenanthrene **9b** (0.18 g, 70%) as a colorless solid. **Mp** 208-210 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.05 (d, J = 7.8 Hz, 2H), 8.76 (s, 1H), 8.66 (d, J = 8.1 Hz, 1H), 8.07 (dd, J_1 = 8.7 Hz, J_2 = 6.9 Hz, 2H), 7.60-7.52 (m, 3H), (t, J = 7.5 Hz, 1H), 7.28-7.25 (m, 5H), 7.06-6.96 (m, 3H), 6.76-6.71 (m, 4H), 6.56 (d, J = 8.7 Hz, 2H), 3.81 (s, 3H), 3.73 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.1, 157.4, 142.5, 141.1, 140.7, 139.6, 135.3, 132.5, 132.4, 132.3, 131.8, 131.1, 130.8, 130.2, 130.1, 129.2, 129.2, 128.8, 128.2, 128.1, 127.7, 126.5, 126.3, 126.1, 126.0, 125.6, 124.3, 123.7, 122.4, 121.7, 113.8, 112.5, 55.2, 55.0 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 132.5, 130.2, 130.1, 128.2, 127.7, 126.5, 126.3, 126.1, 126.0, 125.6, 124.3, 123.7, 122.4, 121.7, 113.8, 112.5, 55.2, 55.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{42}H_{30}O_2$ 567.2324; Found 567.2329.

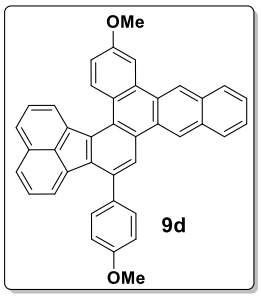
18-Phenylacenaphtho[1,2-*c*]benzo[*f*]tetraphene (9c)



To a solution of cyclopentadienone **1f** (0.16 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) furnished

naphthalene fused phenanthrene **9c** (0.16 g, 72%) as a fluorescent green solid. **Mp** 236-238 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.31 (d, J = 7.8 Hz, 1H), 9.00 (s, 1H), 8.94 (s, 1H), 8.69-8.66 (m, 2H), 8.48 (s, 1H), 8.08-8.00 (m, 2H), 7.85-7.77 (m, 4H), 7.69-7.53 (m, 9H), 7.37 (t, J = 8.1 Hz, 1H), 7.19 (d, J = 6.9 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.1, 137.8, 137.4, 136.3, 136.2, 132.7, 132.6, 132.4, 131.4, 131.0, 130.0, 129.7, 129.4, 129.0, 128.9, 128.8, 128.3, 128.2, 128.1, 128.0, 127.6, 127.4, 127.2, 127.1, 126.1, 125.9, 124.2, 123.9, 123.8, 123.3, 122.5, 121.9 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$):δ 129.4, 129.3, 129.0, 128.8, 128.3, 128.2, 128.1, 128.0, 127.6, 127.4, 127.2, 127.1, 126.1, 125.9, 124.2, 123.9, 123.8, 123.3, 122.5, 121.9 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{22}$ 479.1800; Found 479.1797.

9-Methoxy-18-(4-methoxyphenyl)acenaphtho[1,2-*c*]benzo[*f*]tetraphene (9d)



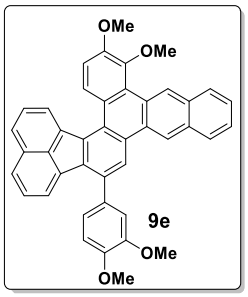
To a solution of cyclopentadienone **1g** (0.19 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation (90-100 °C, 10-20 mm Hg) followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded naphthalene fused phenanthrene **9d** (0.20 g, 82%) as a yellow solid. **Mp** 258-260 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.18 (d, J = 9.0 Hz, 1H), 8.89 (s, 2H), 8.61 (d, J = 7.2 Hz, 1H), 8.41 (s, 1H), 8.06-7.97 (m, 3H), 7.78 (dd, J_1 = 8.1 Hz, J_2 = 4.3 Hz, 2H), 7.68 (d, J = 8.7 Hz, 2H), 7.39-7.34 (m, 2H), 7.27-7.19 (m, 2H), 7.18-7.10 (m, 2H), 4.08 (s, 3H), 3.96 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.6, 159.5, 138.0, 137.6, 136.7, 136.5, 135.5, 133.0, 132.8, 132.7, 132.3, 130.5, 130.1, 129.9, 129.2, 128.8, 128.6, 128.2, 128.1, 127.2, 127.1, 126.9, 126.2, 126.1, 124.0, 123.6, 123.5, 123.2, 122.5, 122.0, 114.8, 114.2, 113.4, 107.5, 55.6, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 139.5, 128.2, 128.1, 127.6, 127.2, 127.2, 126.9, 126.2, 126.1, 124.0, 123.6, 123.2, 122.5, 122.0, 114.2, 113.4, 107.5, 55.6, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{26}O_2$ 539.2011; Found 539.2018.

Diels-Alder reaction of cyclopentadienone 1h with naphtho[2,3-*b*]thiophene *S, S*-dioxide

To a solution of cyclopentadienone **1h** (0.22 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by

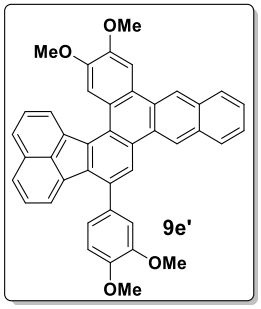
subsequent column chromatographic purification on silica gel (eluent: 10% ethyl acetate in hexane) gave naphthalene fused phenanthrene **9e** (0.08 g, 28%) as a yellow solid. Further elution of the column (eluent: 12% ethyl acetate in hexane) led to the isolation of positionally isomeric naphthalene fused phenanthrene **9e'** (0.14 g, 53%) also as a yellow solid.

18-(3,4-dimethoxyphenyl)-9,10-dimethoxyacenaphtho[1,2-*c*]benzo[*f*]tetraphene (9e)



Mp 210-212 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.98 (s, 1H), 8.96 (d, J = 9 Hz, 1H), 8.83 (s, 1H), 8.52 (t, J = 7.8 Hz, 1H), 8.34 (s, 1H), 8.00-7.92 (m, 2H), 7.87-7.66 (m, 3H), 7.51-7.44 (m, 2H), 7.33-7.05 (m, 6H), 4.02 (s, 3H), 4.00 (s, 3H), 3.87 (s, 3H), 3.81 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 153.2, 149.0, 148.9, 147.3, 138.0, 137.6, 137.1, 136.5, 135.4, 133.8, 133.4, 132.8, 132.5, 132.3, 130.9, 130.0, 129.9, 129.4, 128.9, 128.4, 127.9, 127.7, 127.2, 127.2, 126.5, 125.8, 125.7, 125.1, 124.8, 123.6, 123.4, 123.3, 121.8, 121.4, 112.7, 111.5, 110.8, 59.5, 56.3, 56.1 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{42}H_{30}O_4$ 599.2222; Found 599.2225.

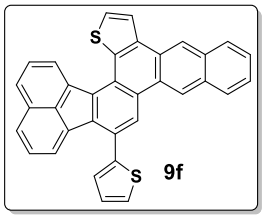
18-(3,4-Dimethoxyphenyl)-8,9-dimethoxyacenaphtho[1,2-*c*]benzo[*f*]tetraphene (9e')



Mp 178-180 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.89 (s, 1H), 8.76 (s, 1H), 8.68 (s, 1H), 8.62 (d, J = 7.2 Hz, 1H), 8.42 (s, 1H), 8.02-7.94 (m, 3H), 7.78-7.70 (m, 2H), 7.50-7.45 (m, 3H), 7.35-7.15 (m, 3H), 7.06 (d, J = 7.8 Hz, 1H), 3.99 (s, 3H), 3.88 (s, 3H), 3.88 (s, 3H), 3.85 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 149.8, 149.0, 148.9, 147.6, 138.0, 137.5, 136.9, 136.5, 135.3, 133.8, 132.8, 132.4, 132.1, 130.5, 130.0, 128.9, 128.6, 128.4, 128.2, 127.8, 127.5, 127.1, 126.8, 126.1, 125.8, 125.6, 124.0, 123.9, 123.8, 123.5, 122.6, 121.4, 121.0, 112.7, 111.5, 111.1, 105.9, 56.2, 56.1, 56.0 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$): δ 128.2, 127.8, 127.5, 127.1, 126.8, 126.1, 125.8, 125.6, 124.0, 123.9, 123.8, 123.5, 122.6, 121.4, 121.0, 112.7, 111.5,

111.1, 105.9, 56.2, 56.1, 56.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{42}H_{30}O_4$ 599.2222; Found 599.2219.

17-(Thiophen-2-yl)acenaphtho[1',2':3,4]tetrapheno[5,6-*b*]thiophene (9f)

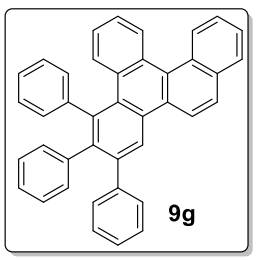


To a solution of cyclopentadienone **1j** (0.14 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) provided thieno[*b*]anthracene **9f** (0.17 g, 74%) as a yellow solid. **Mp** 248-250 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.54 (d, J = 7.2 Hz, 1H), 8.98 (s, 1H), 8.72 (s, 1H), 8.71 (s, 1H), 8.03-7.98 (m, 3H), 7.85-7.73 (m, 2H), 7.61 (t, J = 7.8 Hz, 1H), 7.52-7.41 (m, 4H), 7.38-7.16 (m, 4H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.8, 138.6, 137.4, 136.4, 135.7, 135.2, 133.5, 132.6, 132.4, 131.9, 130.2, 129.9, 128.6, 128.5, 128.4, 127.8, 127.7, 127.5, 127.3, 127.2, 126.9, 126.3, 126.1, 125.8, 125.6, 124.7, 124.1, 123.7, 123.5, 123.4, 122.1 ppm; **DEPT-135 NMR** (75 MHz, $CDCl_3$): δ 128.5, 127.8, 127.7, 127.5, 127.3, 127.2, 126.9, 126.3, 126.1, 125.8, 124.7, 124.1, 123.7, 123.5, 123.4, 122.1 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{34}H_{18}S_2$ 491.0928; Found 491.0927.

Diels-Alder reaction of cyclopentadienone 1a with naphthothiophene *S, S*-dioxide 8b

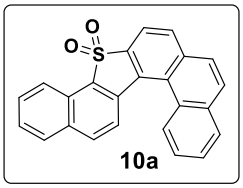
To a solution of cyclopentadienone **1a** (0.18 g, 0.46 mmol) in nitrobenzene (5 mL), naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol)) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 100% hexane) offered benzo chrysene **9g** (0.11 g, 49%) as a colorless solid. Further elution of the column (eluent: 5% ethyl acetate in hexane) led to the isolation of thiophene *S, S*-dioxide tethered hetero helicene **10** (0.03 g, 20%) as a brown solid.

1,2,3-Triphenylbenzo[*g*]chrysene (9g)



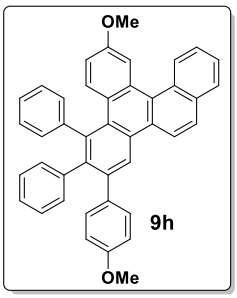
Mp 172-174 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.94 (d, *J* = 8.1 Hz, 1H), 8.72 (d, *J* = 8.1 Hz, 1H), 8.65 (s, 1H), 8.60 (d, *J* = 9 Hz, 1H), 8.00 (t, *J* = 8.7 Hz, 2H), 7.67-7.58 (m, 3H), 7.40 (t, *J* = 7.8 Hz, 1H), 7.26-7.16 (m, 10H), 7.06-6.98 (m, 4H), 6.82-6.81 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 142.6, 142.4, 140.9, 140.3, 139.9, 138.9, 133.7, 131.7, 131.6, 131.3, 131.1, 130.7, 130.1, 129.8, 129.5, 129.1, 128.8, 128.2, 127.8, 127.8, 127.6, 126.9, 126.4, 126.1, 126.0, 125.7, 124.9, 124.2, 120.7 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 131.9, 131.7, 130.1, 129.5, 129.1, 128.5, 128.2, 127.8, 127.6, 126.9, 126.4, 126.4, 126.1, 126.0, 125.7, 124.9, 124.2, 120.7 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{26}$ 507.2113; Found 507.2111.

Naphtho[1,2-*b*]phenanthro[4,3-*d*]thiophene *S, S*-dioxide (10a)



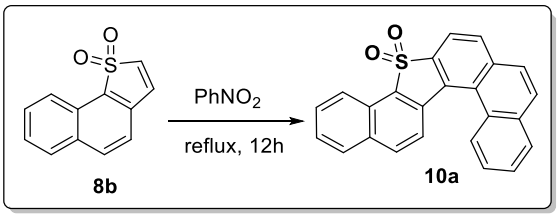
Mp 140-142 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.77 (d, *J* =8.1 Hz, 1H), 8.50 (d, *J* = 8.1 Hz, 1H), 8.28 (d, *J* = 8.7 Hz, 1H), 8.03-7.84 (m, 6H), 7.78-7.60 (m, 4H), 7.51 (t, *J* = 7.8 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 139.0, 137.1, 134.1, 133.7, 133.1, 132.8, 132.4, 130.6, 129.8, 129.4, 128.9, 128.9, 128.8, 128.6, 128.6, 128.3, 128.0, 126.6, 125.1, 123.8, 122.2, 118.3 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 132.8, 130.6, 129.8, 129.4, 128.9, 128.6, 128.6, 128.3, 128.0, 126.6, 125.1, 123.8, 122.2, 118.3 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{24}H_{14}O_2S$ 367.0793; Found 367.0792.

Diels-Alder reaction of cyclopentadienone 1c with naphthothiophene *S, S*-dioxide 8b



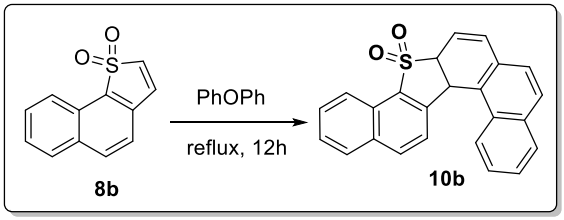
To a solution of cyclopentadienone **1c** (0.18 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 2-3% ethyl acetate in hexane) offered a complex mixture of compounds. Further elution of the column (eluent: 5% ethyl acetate in hexane) led to the isolation of thiophene *S, S*-dioxide tethered hetero helicene **10a** (0.05 g, 30%) as a brown solid.

Self-Diels-Alder reaction of naphthothiophene *S, S*-dioxide 8b in $PhNO_2$



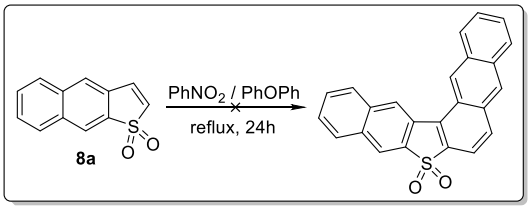
To a solution of naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol) in nitrobenzene (5 mL), was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave thiophene *S, S*-dioxide tethered hetero helicene **10a** (0.06 g, 68%) as a brown solid.

Self-Diels-Alder reaction of naphthothiophene *S, S*-dioxide 8b in PhOPh



The self-Diels-Alder reaction of naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol) in diphenyl ether (5 mL) at reflux for 12 h followed by workup and column chromatographic purification on silica gel (eluent: 4% ethyl acetate in hexane) offered dihydro thiophene *S, S*-dioxide **10b** (0.04 g, 55%)as a colorless solid. **Mp** 194-196 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.60 (d, J = 8.4 Hz, 1H), 8.12 (d, J = 8.4 Hz, 1H), 7.97-7.59 (m, 7H), 7.32 (d, J = 8.4 Hz, 1H), 6.71-6.66 (m, 2H), 6.32 (d, J = 7.8 Hz, 1H), 5.71 (d, J = 7.5 Hz, 1H), 4.72 (d, J = 7.2 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 138.2, 134.3, 133.8, 133.5, 132.3, 132.1, 131.1, 129.2, 129.1, 129.0, 128.5, 127.8, 126.3, 126.1, 123.1, 122.8, 120.2, 64.9, 36.3 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 134.3, 131.1, 129.2, 129.1, 129.0, 128.5, 127.8, 127.5, 126.3, 126.1, 123.1, 122.8, 120.2, 64.9, 36.3 ppm.

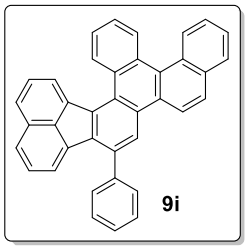
Attempted self-Diels-Alder reaction of naphthothiophene *S, S*-dioxide 8a



A solution of naphtho[2,3-*b*]thiophene *S, S*-dioxide **8a** (0.10 g, 0.46 mmol) in $PhNO_2$ / PhOPh (5 mL) was refluxed for 24 h. The usual workup followed by column chromatographic

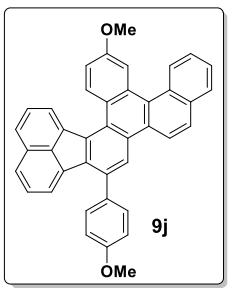
purification on silica gel (eluent: 7% ethyl acetate in hexane) failed to produce the expected hetero helicene, instead the starting material **8a** was recovered unchanged.

12-Phenylacenaphtho[1,2-*a*]benzo[*p*]chrysene (9i)



To a solution of cyclopentadienone **1f** (0.16 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by column chromatographic purification on silica gel (eluent: 100% hexane) to afford fluoranthene fused chrysene **9i** (0.18 g, 84%) as a fluorescent green solid. **Mp** 240-242 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.51(d, J = 7.8 Hz, 1H), 9.04 (d, J = 8.4 Hz, 1H), 8.85-8.82 (m, 2H), 8.52 (d, J = 9.0 Hz, 1H), 8.41 (s, 1H), 8.02-7.95 (m, 2H), 7.87 (d, J = 8.1 Hz, 1H), 7.82-7.77 (m, 3H), 7.71-7.55 (m, 8H), 7.39 (t, J = 7.8 Hz, 1H), 7.24 (d, J = 6.9 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.2, 137.9, 137.7, 137.1, 136.4, 135.5, 133.6, 132.8, 130.5, 130.3, 129.9, 129.4, 129.2, 128.8, 128.2, 128.1, 128.1, 128.0, 127.9, 127.9, 127.7, 127.5, 127.5, 127.3, 127.1, 126.2, 126.0, 125.4, 124.0, 123.9, 123.6, 123.5, 120.8 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 129.4, 129.2, 128.8, 128.2, 128.1, 128.1, 128.0, 127.9, 127.7, 127.5, 127.5, 127.3, 127.1, 126.2, 126.0, 125.4, 124.0, 123.6, 123.5, 120.8 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{38}H_{22}$ 479.1800; Found 479.1803.

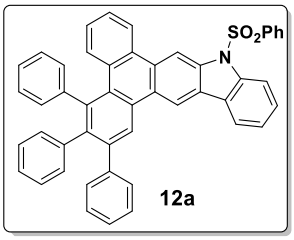
3-Methoxy-12-(4-methoxyphenyl)acenaphtho[1,2-*a*]benzo[*p*]chrysene (9j)



To a solution of cyclopentadienone **1g** (0.19 g, 0.46 mmol) in $PhNO_2$ (5 mL), naphtho[1,2-*b*]thiophene *S, S*-dioxide **8b** (0.10 g, 0.46 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave acenaphtho based benzo chrysene **9j** (0.14 g, 78%) as a fluorescent green solid.

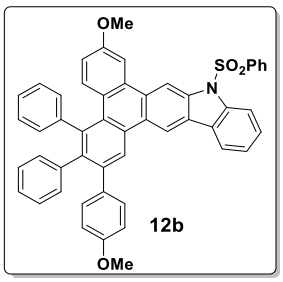
Mp 190-192 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.39 (d, J = 9 Hz, 1H), 9.09 (d, J = 8.4 Hz, 1H), 8.78 (d, J = 7.2 Hz, 1H), 8.52 (d, J = 9 Hz, 1H), 8.36 (s, 1H), 8.32 (d, J = 1.8 Hz, 1H), 8.02-7.98 (m, 2H), 7.83 (dd, J_1 =8.1 Hz, J_2 =8.4 Hz, 2H), 7.70-7.58 (m, 5H), 7.40 (t, J = 7.8 Hz, 1H), 7.32-7.28 (m, 1H), 7.16 (d, J = 8.4 Hz, 3H), 4.02 (s, 3H), 4.00 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.5, 158.8, 137.9, 137.3, 136.8, 136.7, 134.8, 133.6, 133.5, 132.8, 132.0, 130.5, 130.1, 129.9, 129.8, 129.7, 128.6, 128.3, 128.0, 127.7, 127.6, 127.5, 127.3, 127.2, 127.0, 126.2, 125.9, 124.4, 123.9, 123.6, 123.4, 121.0, 114.1, 113.3, 112.0, 55.6, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 130.5, 129.7, 128.3, 128.0, 127.7, 127.5, 127.3, 127.2, 127.0, 126.2, 125.9, 123.6, 123.4, 121.0, 114.1, 113.3, 112.0, 55.6, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{40}H_{26}O_2$ 539.2011; Found 539.2010.

2,3,4-Triphenyl-10-(phenylsulfonyl)-10*H*-phenanthro[9,10-b]carbazole (12a)



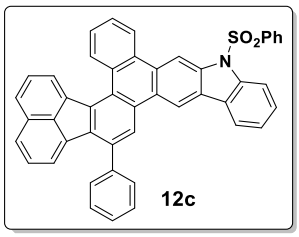
To a solution of cyclopentadienone **1a** (0.10 g, 0.25 mmol) in nitrobenzene (5 mL), 9-(phenylsulfonyl)-9*H*-thieno[2,3-*b*]carbazole *S*, *S*-dioxide **11a** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), the solvent was removed by vacuum distillation (90-100 ℃, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded phenanthrene fused carbazole **12a** (0.12 g, 65%) as a pale brown solid. **Mp** >300 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.53 (s, 1H), 9.04 (s, 1H), 8.74-8.69 (m, 2H), 8.36 (d, J = 8.4 Hz, 1H), 8.04 (d, J = 7.5 Hz, 1H), 7.85 (d, J = 7.8 Hz, 2H), 7.52 (t, J = 8.1 Hz, 3H), 7.47-7.36 (m, 2H), 7.29-7.13 (m, 10H), 7.05-6.93 (m, 6H), 6.79-6.77 (m, 2H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 142.9, 142.3, 140.8, 140.6, 139.9, 139.7, 138.4, 137.7, 133.9, 131.7, 131.6, 131.5, 130.7, 130.6, 130.4, 130.1, 129.1, 128.4, 128.2, 128.1, 127.6, 127.0, 126.9, 126.8, 126.6, 126.4, 125.7, 125.5, 124.5, 123.9, 123.8, 120.6, 115.5, 114.8, 108.9 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 131.6, 131.5, 130.1, 129.1, 128.2, 128.1, 127.6, 126.9, 126.8, 126.6, 126.5, 126.4, 125.6, 125.5, 124.5, 123.9, 123.8, 120.6, 115.5, 114.8, 108.9 ppm; **HRMS (ESI)** *m/z* : $[M+H]^+$ Calcd for $C_{48}H_{31}NO_2S$ 686.2154; Found 686.2161.

2-Methoxy-7-(4-methoxyphenyl)-5,6-diphenyl-10-(phenylsulfonyl)-10*H*-phenanthro[9,10-*b*]carbazole (12b)



To a solution of cyclopentadienone **1c** (0.11 g, 0.25 mmol) in $PhNO_2$ (5 mL), 9-(phenylsulfonyl)-9*H*-thieno[2,3-*b*]carbazole *S, S*-dioxide **11a** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 10% ethyl acetate in hexane) provided phenanthrene fused carbazole **12b** (0.17 g, 87%) as a pale brown solid. **Mp** 254-256 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.42 (s, 1H), 9.02 (s, 1H), 8.63 (s, 1H), 8.32 (d, *J* = 8.1 Hz, 1H), 8.10 (s, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.42-7.36 (m, 3H), 7.27-7.21 (m, 2H), 7.11-6.93 (m, 10H), 6.74-6.71 (m, 4H), 6.59 (d, *J* = 9.3 Hz, 1H), 3.94 (s, 3H), 3.74 (s, 3H) ppm**; ^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 158.2, 158.0, 143.1, 140.6, 140.1, 139.7, 139.5, 138.9, 138.3, 137.7, 134.7, 133.9, 133.3, 131.6, 131.5, 131.2, 130.3, 129.8, 129.2, 128.2, 128.1, 127.3, 126.9, 126.8, 126.6, 126.5, 126.3, 125.5, 124.4, 124.3, 124.0, 120.5, 115.5, 114.9, 113.2, 113.1, 109.0, 106.9, 55.4, 55.2 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 131.6, 131.5, 131.2, 129.1, 128.3, 128.1, 126.9, 126.5, 126.3, 125.5, 124.4, 124.0, 120.6, 115.5, 114.9, 113.2, 113.1, 109.0, 106.0, 55.4, 55.2 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{50}H_{35}NO_4S$ 746.2365; Found 746.2365.

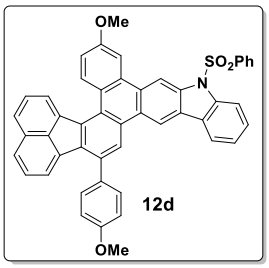
19-Phenyl-12-(phenylsulfonyl)-12H-acenaphtho[1',2':3,4]phenanthro[9,10-*b*]carbazole (12c)



To a solution of cyclopentadienone **1f** (0.09 g, 0.25 mmol) in $PhNO_2$ (5 mL), 9-(phenylsulfonyl)-9*H*-thieno[2,3-*b*]carbazole *S, S*-dioxide **11a** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) gave phenanthrene fused carbazole **12c** (0.14 g, 81%) as a yellow

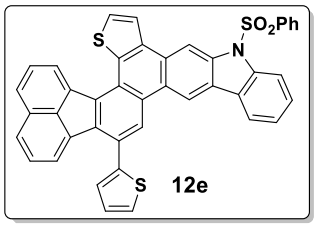
solid. **Mp** >300 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.52 (s, 1H), 9.40 (d, *J* = 8.1 Hz, 1H), 8.94 (s, 1H), 8.76 (d, *J* = 8.1 Hz, 2H), 8.71 (d, *J* = 7.2 Hz, 2H), 8.40 (s, 1H), 8.36 (d, *J* = 8.1 Hz, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.88-7.74 (m, 7H), 7.65-7.50 (m, 3H), 7.42-7.25 (m, 6H), 7.15 (d, *J* = 7.2 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.1, 139.7, 138.2, 137.9, 137.7, 137.7, 137.1, 136.2, 136.2, 133.9, 132.7, 131.2, 130.5, 130.2, 129.9, 129.5, 129.1, 128.9, 128.8, 128.3, 128.1, 127.6, 127.5, 127.4, 127.2, 127.1, 126.8, 126.6, 126.5, 125.8, 124.4, 124.2, 124.0, 123.4, 120.5, 115.5, 114.9, 109.0 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 129.4, 129.1, 128.9, 128.8, 128.3, 128.1, 127.6, 127.5, 127.2, 127.1, 126.5, 125.8, 124.4, 124.2, 124.0, 123.4, 120.5, 115.4, 114.9, 109.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{46}H_{27}NO_2S$ 658.1841; Found 658.1835.

9-Methoxy-19-(4-methoxyphenyl)-12-(phenylsulfonyl)-12H-acenaphtho[1',2':3,4]phenanthro[9,10-*b*]carbazole (12d)



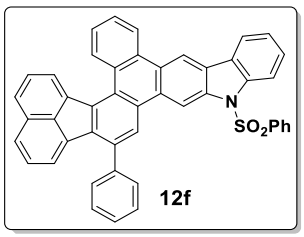
To a solution of cyclopentadienone **1g** (0.11 g, 0.25 mmol) in $PhNO_2$ (5 mL), 9-(phenylsulfonyl)-9*H*-thieno[2,3-*b*]carbazole *S*, *S*-dioxide **11a** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 10% ethyl acetate in hexane) furnished phenanthrene incorporated carbazole **12d** (0.14 g, 79%) as a yellow solid. **Mp** 270-272 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.46 (s, 1H), 9.32 (d, *J* = 9.0 Hz, 1H), 8.98 (s, 1H), 8.67 (d, *J* = 7.2 Hz, 1H), 8.39-8.35 (m, 2H), 8.17 (d, *J* = 2.4 Hz, 1H), 8.06 (d, *J* = 7.5 Hz, 1H), 7.87-7.77 (m, 4H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.59-7.51 (m, 2H), 7.44-7.35 (m, 3H), 7.31-7.15 (m, 6H), 4.14 (s, 3H), 3.98 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.7, 159.6, 139.7, 138.2, 138.1, 137.7, 137.4, 136.7, 136.5, 135.4, 133.9, 133.6, 133.0, 132.7, 130.5, 130.0, 130.0, 129.7, 129.1, 128.1, 127.8, 127.7, 127.3, 127.1, 127.0, 126.6, 126.6, 124.4, 123.7, 123.6, 123.5, 123.3, 120.5, 115.5, 114.9, 114.2, 113.6, 109.2, 107.2, 55.7, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 130.5, 129.1, 128.1, 127.7, 127.3, 127.1, 126.6, 124.5, 123.7, 123.6, 123.3, 120.5, 115.5, 114.9, 114.2, 113.6, 109.2, 107.2, 55.7, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{48}H_{31}NO_4S$ 718.2052; Found 718.2054.

11-(Phenylsulfonyl)-18-(thiophen-2-yl)-11*H*-acenaphtho[1',2':5,6] thieno[3',2':3,4]naphtho[2,1-*b*]carbazole (12e)



To a solution of cyclopentadienone **1j** (0.09 g, 0.25 mmol) in $PhNO_2$ (5 mL), 9-(phenylsulfonyl)-9*H*-thieno[2,3-*b*]carbazole *S, S*-dioxide **11a** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) furnished fluoranthene fused carbazole **12e** (0.12 g, 69%) as a yellow solid. **Mp** 184-186 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.43 (d, J = 7.2 Hz, 1H), 9.16 (s, 1H), 9.01 (s, 1H), 8.64 (s, 1H), 8.33 (d, J = 8.4 Hz, 1H), 8.16 (d, J = 5.7 Hz, 1H), 8.04 (d, J = 7.2 Hz, 1H), 7.90-7.80 (m, 4H), 7.67 (t, J = 7.8 Hz, 1H), 7.62-7.57 (m, 2H), 7.51 (t, J = 7.8 Hz, 1H), 7.43-7.38 (m, 4H), 7.31-7.25 (m, 4H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.8, 139.6, 138.3, 138.1, 137.7, 136.4, 135.6, 135.1, 133.9, 133.7, 132.4, 129.8, 129.8, 129.1, 128.7, 128.4, 128.0, 127.9, 127.7, 127.6, 127.5, 127.4, 127.3, 126.6, 126.5, 126.1, 126.1, 125.2, 124.8, 124.4, 124.3, 123.8, 123.5, 120.5, 115.5, 115.4, 109.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 129.1, 128.0, 127.9, 127.7, 127.6, 127.5, 127.4, 127.3, 126.6, 126.5, 126.1, 124.8, 124.4, 124.3, 123.8, 123.5, 120.5, 115.5, 115.4, 109.4 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{42}H_{23}NO_2S_3$ 670.0969; Found 670.0972.

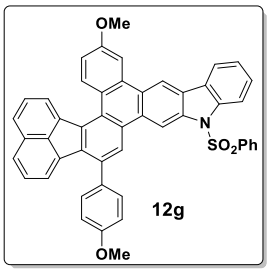
2-Phenyl-18-(phenylsulfonyl)-18*H*-acenaphtho[1',2':5,6]phenanthro[9,10-*b*]carbazole (12f)



To a solution of cyclopentadienone **1f** (0.09 g, 0.25 mmol) in $PhNO_2$ (5 mL), 5-(phenylsulfonyl)-5*H*-thieno[3,2-*b*]carbazole *S, S*-dioxide **11b** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded fluoranthene fused carbazole **12f** (0.12 g, 74%) as a

yellow solid. **Mp** 266-268 °C; **^{1}H NMR (300 MHz**, $CDCl_3$): δ 9.48 (s, 1H), 9.39 (d, J = 7.8 Hz, 1H), 9.02 (s, 1H), 8.68 (d, J = 7.2 Hz, 1H), 8.63 (d, J = 8.4 Hz, 1H), 8.55 (s, 1H), 8.35 (d, J = 8.1 Hz, 1H), 8.08 (d, J = 7.8 Hz, 1H), 7.84- 7.76 (m, 5H), 7.72-7.61 (m, 4H), 7.56-7.51 (m, 3H), 7.45-7.34 (m, 4H), 7.27-7.19 (m, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 140.9, 139.7, 138.6, 138.1, 137.9, 137.5, 136.3, 136.1, 133.9, 132.7, 131.0, 130.9, 130.4, 129.9, 129.4, 129.1, 129.1, 128.9, 128.6, 128.2, 128.2, 128.1, 127.7, 127.5, 127.4, 127.2, 126.6, 125.4, 124.4, 123.9, 123.7, 123.6, 123.5, 120.4, 115.6, 114.4, 109.6 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 129.4, 129.1, 128.9, 128.2, 128.2, 128.1, 127.7, 127.5, 127.2, 126.6, 125.4, 124.4, 123.9, 123.7, 123.6, 123.5, 120.4, 115.6, 114.4, 109.6 ppm**; HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{46}H_{27}NO_2S$ 658.1841; Found 658.1843.

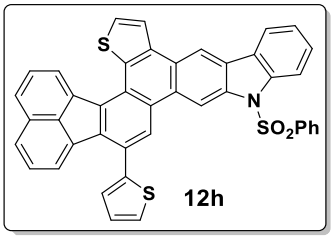
11-Methoxy-2-(4-methoxyphenyl)-18-(phenylsulfonyl)-18*H*-acenaphtho[1',2':5,6]phenanthro[9,10-*b*]carbazole (12g)



To a solution of cyclopentadienone **1g** (0.11 g, 0.25 mmol) in $PhNO_2$ (5 mL), 5-(phenylsulfonyl)-5*H*-thieno[3,2-*b*]carbazole *S*, *S*-dioxide **11b** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) offered fluoranthene fused carbazole **12g** (0.14 g, 75%) as a yellow solid. M**p** 146-148 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.38 (s, 1H), 9.18 (d, J = 8.7 Hz, 1H), 8.83 (s, 1H), 8.55 (d, J = 7.2 Hz, 1H), 8.41 (s, 1H), 8.28 (d, J = 8.1 Hz, 1H), 8.01 (d, J = 7.2 Hz, 1H), 7.94 (s, 1H), 7.76-7.66 (m, 6H), 7.48-7.44 (m, 2H), 7.43-7.32 (m, 6H), 7.30-7.06 (m, 6H), 4.01 (s, 3H), 3.93 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 159.6, 159.5, 139.6, 138.6, 138.0, 137.6, 136.9, 136.5, 135.3, 133.9, 133.3, 132.7, 132.6, 131.4, 130.7, 130.6, 130.1, 129.9, 129.1, 128.8, 128.8, 128.4, 128.0, 127.7, 127.2, 127.1, 127.1, 126.6, 126.3, 124.4, 123.9, 123.6, 123.4, 123.0, 120.4, 115.5, 114.3, 113.0, 109.6, 106.8, 55.6, 55.5 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 130.6, 129.1, 128.0, 127.7, 127.2, 127.2, 127.1, 126.6, 124.4, 123.9, 123.6, 123.4, 120.4, 115.5, 114.3, 113.0, 109.6, 106.8, 55.6, 55.5 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{48}H_{31}NO_4S$ 718.2052; Found 718.2051.

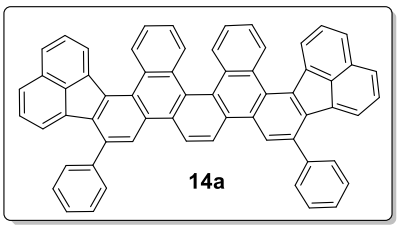
17-(Phenylsulfonyl)-2-(thiophen-2-yl)-17*H*

acenaphtho[1',2':5,6]thieno[3',2':3,4]naphtho[1,2-*b*]carbazole (12h)



To a solution of cyclopentadienone **1j** (0.09 g, 0.25 mmol) in $PhNO_2$ (5 mL), 5-(phenylsulfonyl)-5*H*-thieno[3,2-*b*]carbazole *S, S*-dioxide **11b** (0.10 g, 0.25 mmol) was added and refluxed for 12 h. After completion of reaction (TLC), removal of solvent through vacuum distillation followed by subsequent column chromatographic purification on silica gel (eluent: 5% ethyl acetate in hexane) afforded fluoranthene fused carbazole **12h** (0.11 g, 63%) as a yellow solid. **Mp** 260-262 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.55 (s, 1H), 9.45 (d, J = 7.2 Hz, 1H), 8.77 (s, 1H), 8.74 (s, 1H), 8.36 (d, J = 8.1 Hz, 1H), 8.09-8.05 (m, 2H), 7.86-7.82 (m, 3H), 7.69 (t, J = 7.5 Hz, 1H), 7.62-7.52 (m, 3H), 7.49-7.35 (m, 6H), 7.34-7.26 (m, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): δ 141.6, 139.9, 138.6, 137.8, 137.6, 137.3, 136.4, 135.7, 135.1, 133.9, 133.1, 132.5, 130.2, 129.9, 129.6, 129.1, 128.8, 128.3, 128.0, 127.8, 127.7, 127.6, 127.5, 127.2, 127.1, 126.8, 126.6, 126.4, 126.2, 126.1, 125.6, 124.7, 124.4, 124.4, 124.0, 122.9, 120.6, 115.6, 114.9, 110.0 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): δ 133.9, 129.1, 128.3, 128.0, 127.8, 127.7, 127.6, 127.5, 127.2, 126.6, 126.2, 124.7, 124.4, 124.0, 122.9, 120.6, 115.6, 114.9, 110.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{42}H_{23}NO_2S_3$ 670.0969; Found 670.0961.

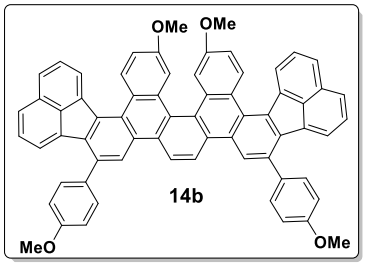
4,9-Diphenyldiacenaphtho[1,2-c:1',2'-*m*]dibenzo[*f,j*]picene (14a)



To a solution of cyclopentadienone **1f** (0.28 g, 0.79 mmol) in $PhNO_2$ (5 mL), benzo[2,1-*b*:3,4-*b'*]dithiophene *S, S*-tetraoxide **13** (0.10 g, 0.39 mmol) was added and refluxed for 24 h. After completion of reaction (TLC), the solvent was removed by vacuum distillation (90-100 ℃, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 100% hexane) gave dibenzo picene **14a** (0.24 g, 78%) as a green fluorescent solid. **Mp** 230-232 ℃; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 9.4 (d, J = 8.1 Hz, 1H), 8.68 (d, J = 7.2 Hz, 1H), 8.47 (s, 1H),

8.35-8.29 (m, 3H), 8.04 (d, *J* = 7.2 Hz, 1H), 7.82-7.70 (m, 9H), 7.62-7.49 (m, 11H), 7.44-7.30 (m, 5H), 7.16 (d, *J* = 6.9 Hz, 1H), 6.76 (d, *J* = 6.9 Hz, 1H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): *δ* 141.2, 140.9, 140.4, 140.0, 139.5, 138.5, 138.2, 138.0, 137.7, 137.1, 136.7, 136.4, 136.1, 136.0, 133.3, 132.7, 131.6, 131.5, 131.3, 131.1, 130.5, 130.0, 129.8, 128.8, 128.6, 128.5, 128.3, 128.0, 127.9, 127.7, 127.6, 127.6, 127.4, 127.2, 127.1, 126.8, 125.3, 125.0, 123.9, 123.7, 123.5, 123.4, 123.1, 121.0 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): *δ* 131.3, 130.5, 129.5, 129.3, 129.1, 128.8, 128.8, 128.6, 128.4, 128.3, 128.0, 127.9, 127.7, 127.6, 127.6, 127.4, 127.2, 127.1, 126.8, 125.3, 125.0, 123.9, 123.7, 123.5, 123.4, 123.1, 121.0 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{62}H_{34}$ 779.2739; Found 779.2756.

18,21-Dimethoxy-4,9-bis(4-methoxyphenyl)diacenaphtho[1,2-*c*:1',2'-*m*]dibenzo[*f,j*]picene (14b)



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To a solution of cyclopentadienone **1g** (0.33 g, 0.79 mmol) in $PhNO_2$ (5 mL), benzo[2,1-*b*:3,4-*b'*]dithiophene *S, S*-tetraoxide **13** (0.10 g, 0.39 mmol) was added and refluxed for 24 h. After completion of reaction (TLC), the solvent was removed by vacuum distillation (90-100 °C, 10-20 mm Hg). Subsequent column chromatographic purification on silica gel (eluent: 20% ethyl acetate in hexane) furnished dibenzo picene **14b** (0.25 g, 73%) as a yellow solid. **Mp** 222-224 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): *δ* 9.24 (d, *J* = 9 Hz, 1H), 8.61 (d, *J* = 7.2 Hz, 1H), 8.35-8.31 (m, 2H), 8.24 (s, 1H), 7.79-7.71 (m, 4H), 7.63 (dd, J_1 = 8.7 Hz, J_2 = 8.4 Hz, 4H), 7.52-7.44 (m, 3H), 7.39-7.29 (m, 5H), 7.23-7.21 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 4H) 6.90 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 7.2 Hz, 1H), 4.00 (s, 3H), 3.93 (s, 3H), 3.91 (s, 3H), 3.76 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (75 MHz, $CDCl_3$): *δ* 159.4, 159.4, 158.9, 140.7, 139.9, 138.9, 137.8, 137.2, 136.8, 136.6, 136.6, 136.2, 136.1, 135.9, 135.3, 133.6, 133.2, 133.1, 132.7, 132.7, 132.7, 131.6, 130.5, 130.4, 130.0, 129.8, 129.6, 129.6, 129.3, 128.9, 128.2, 127.7, 127.6, 127.2, 127.1, 127.0, 126.7, 124.9, 123.9, 123.6, 123.5, 123.4, 123.3, 123.0, 114.2, 114.1, 113.5, 106.3, 55.6, 55.5, 55.4, 55.2 ppm; **DEPT 135-NMR** (75 MHz, $CDCl_3$): *δ* 131.5, 130.5, 130.3, 129.6, 127.7, 127.6, 127.2, 126.9, 126.7, 124.9, 123.9, 123.6, 123.5, 123.3, 123.0, 114.1, 112.5, 106.3, 55.6, 55.5, 55.4, 55.2 ppm; **HRMS (ESI)** *m/z*: $[M+H]^+$ Calcd for $C_{66}H_{42}O_4$ 899.3161; Found 899.3159.

4. References

1. Karunakaran, J.; Mohanakrishnan, A. K. *Org. Lett.* **2018**, *20*, 966.
2. Iniesta, J.; Matsumoto, T.; Thiemann, T. *J. Chem. Res.***2008**, *2*, 109.
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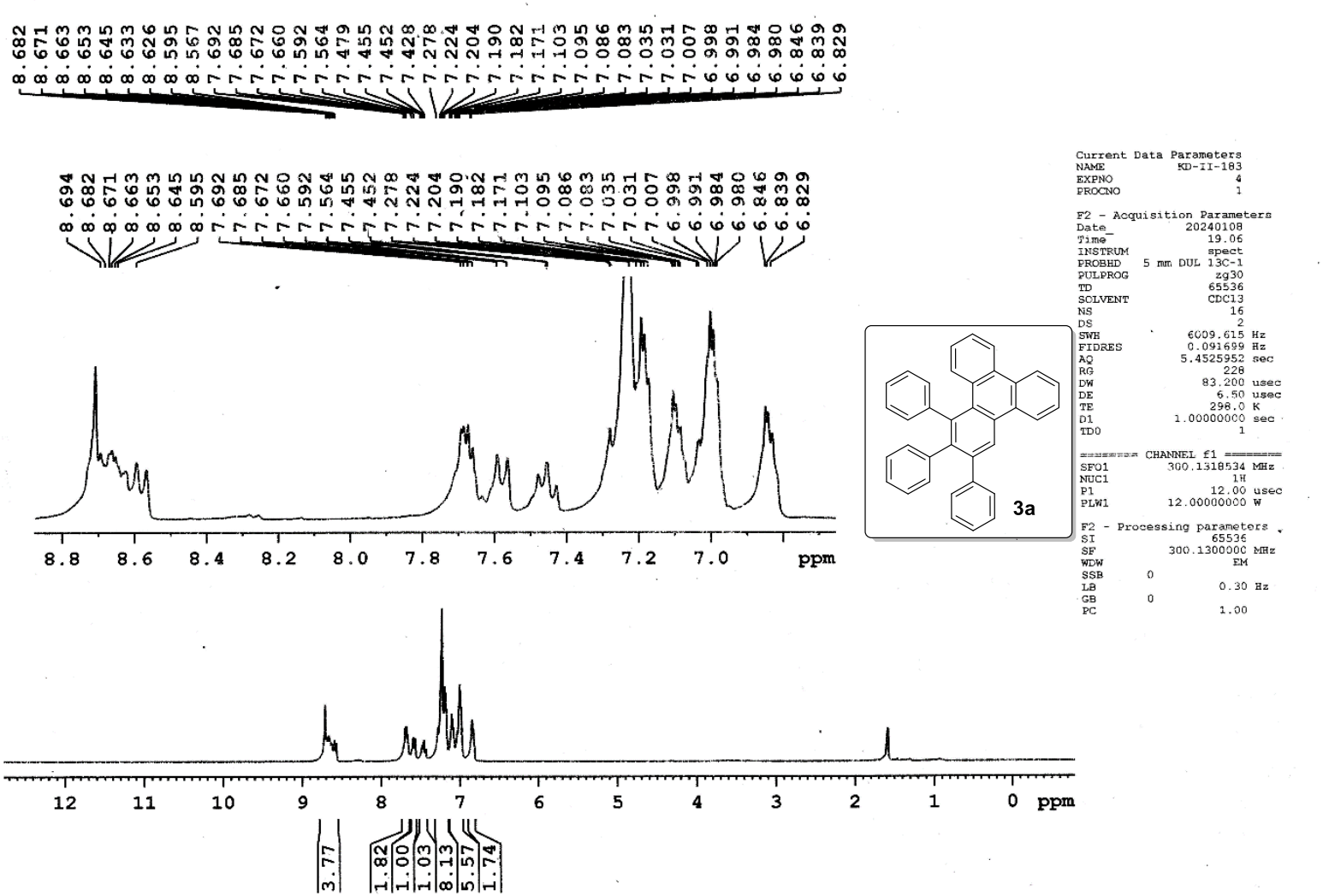
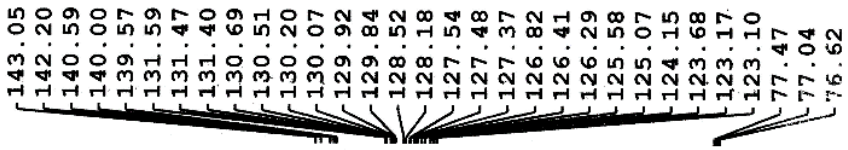
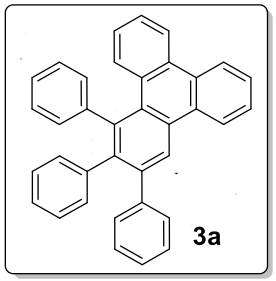
5. ^{1}H, ^{13}C {^{1}H}, DEPT-135 (selected) NMR & HRMS (selected) spectra of benzo phenanthrenes **3a-i** and thieno[*b*]phenanthrene **3j**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3a**



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Current Data Parameters
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EXPNO                         3
PROCNO                        1

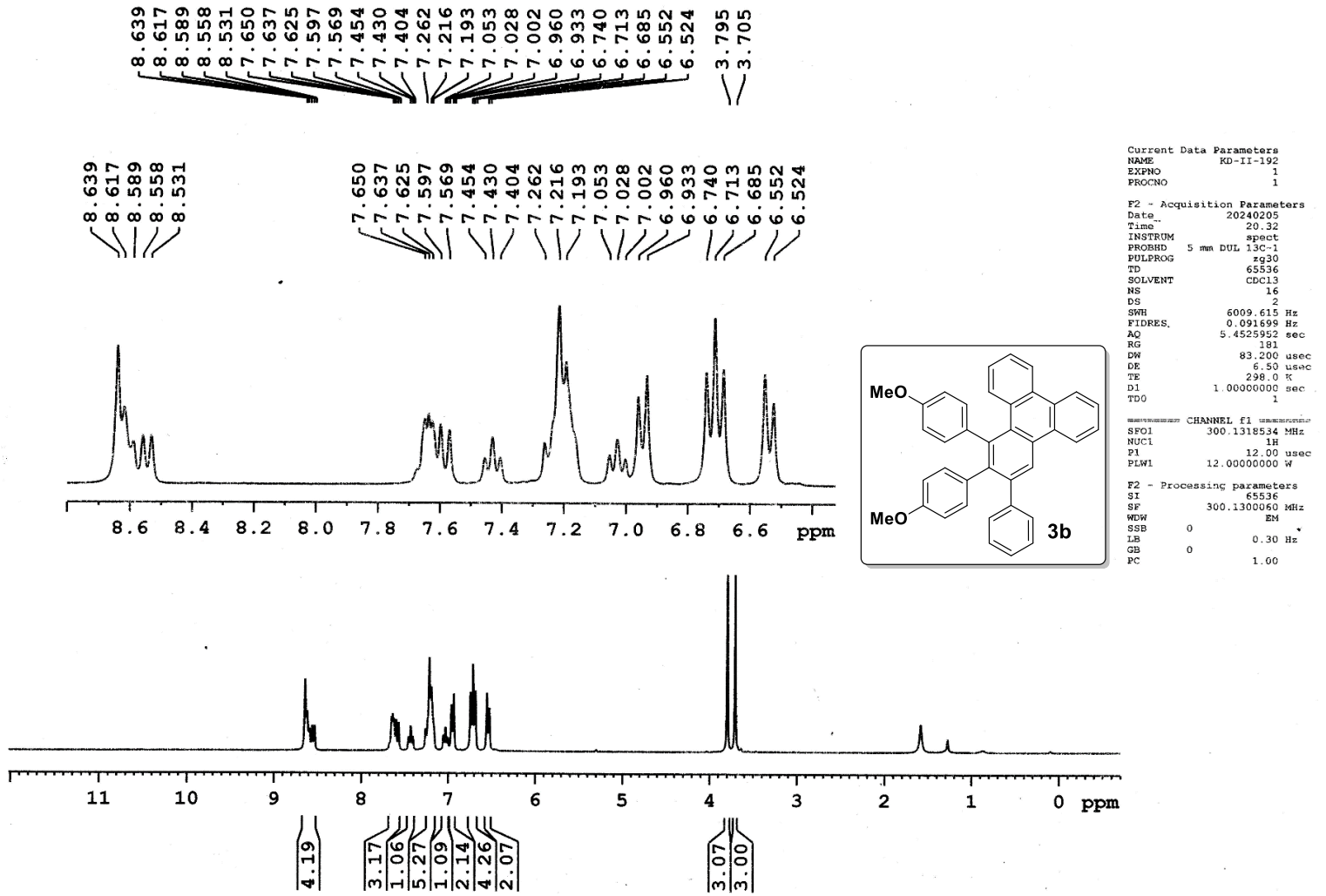
F2 - Acquisition Parameters
Date_                  20240108
Time                      19.03
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         1290
DW                       27.733 usec
DE                         6.50 usec
TE                        300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                48.00000000 W

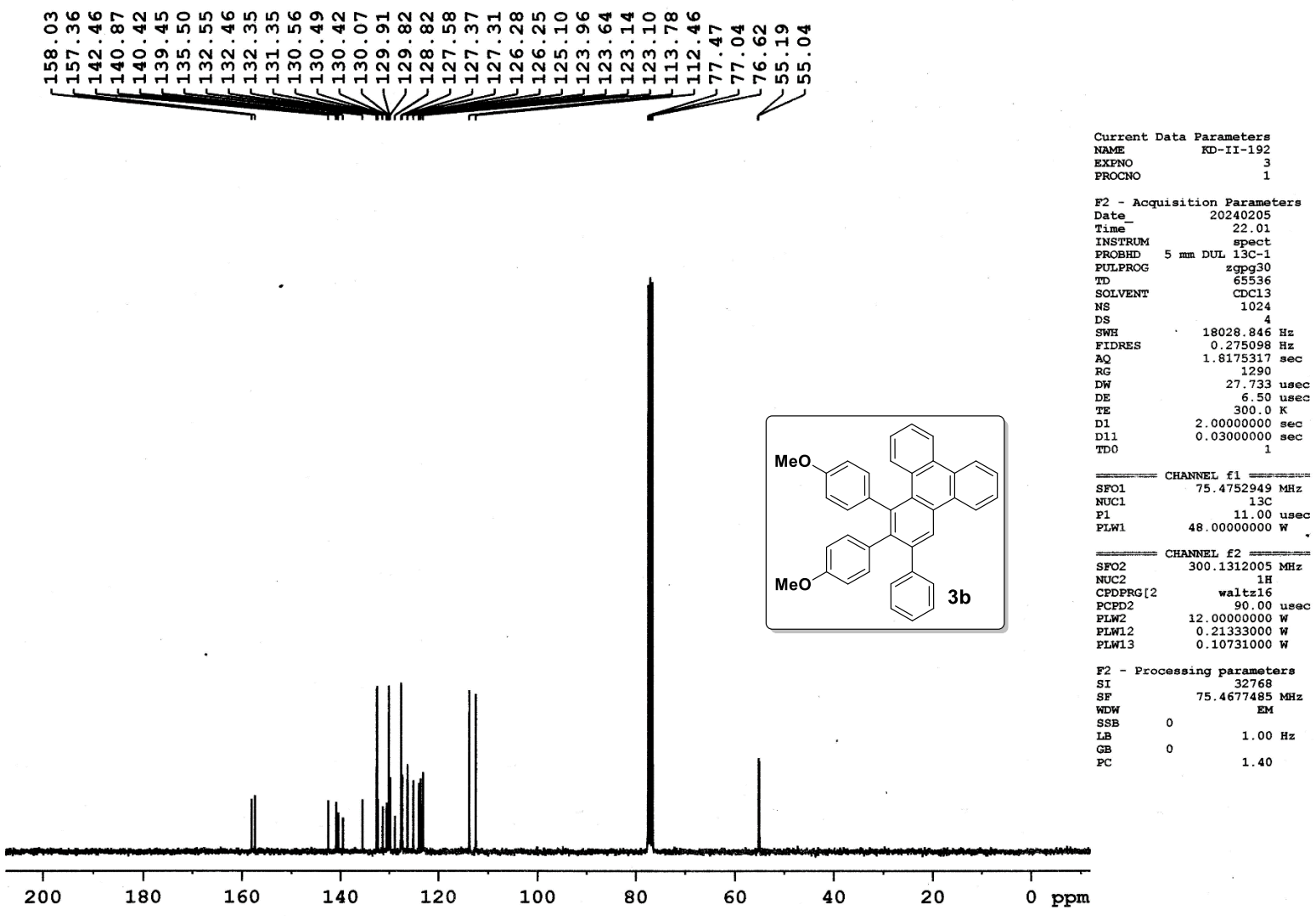
======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W
PLW13                0.10731000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677485 MHz
WDW                          EM
SSB                   0
LB                        1.00 Hz
GB                    0
PC                        1.40
```


$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3a**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3b**



Current Data Parameters

NAME	KD-II-192
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20240205
Time	22.01
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	18028.846 Hz
FIDRES	0.275098 Hz
AQ	1.8175317 sec
RG	1290
DW	27.733 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

CHANNEL f1

SFO1	75.4752949 MHz
NUC1	13C
P1	11.00 usec
PLW1	48.00000000 W

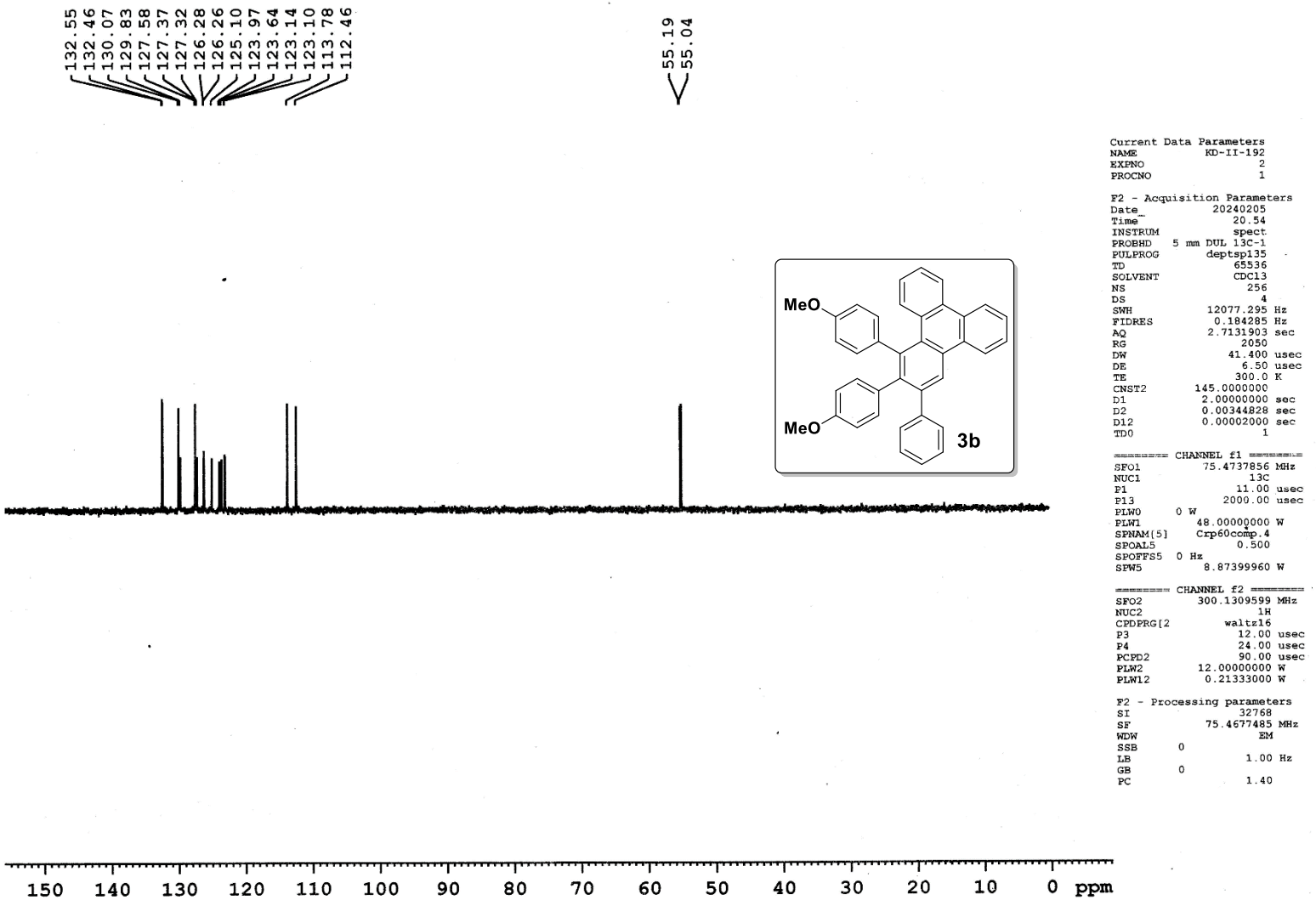
CHANNEL f2

SFO2	300.1312005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.21333000 W
PLW13	0.10731000 W

F2 - Processing parameters

SI	32768
SF	75.4677485 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3b**




```
Current Data Parameters
NAME                  KD-II-192
EXPNO                         2
PROCNO                        1

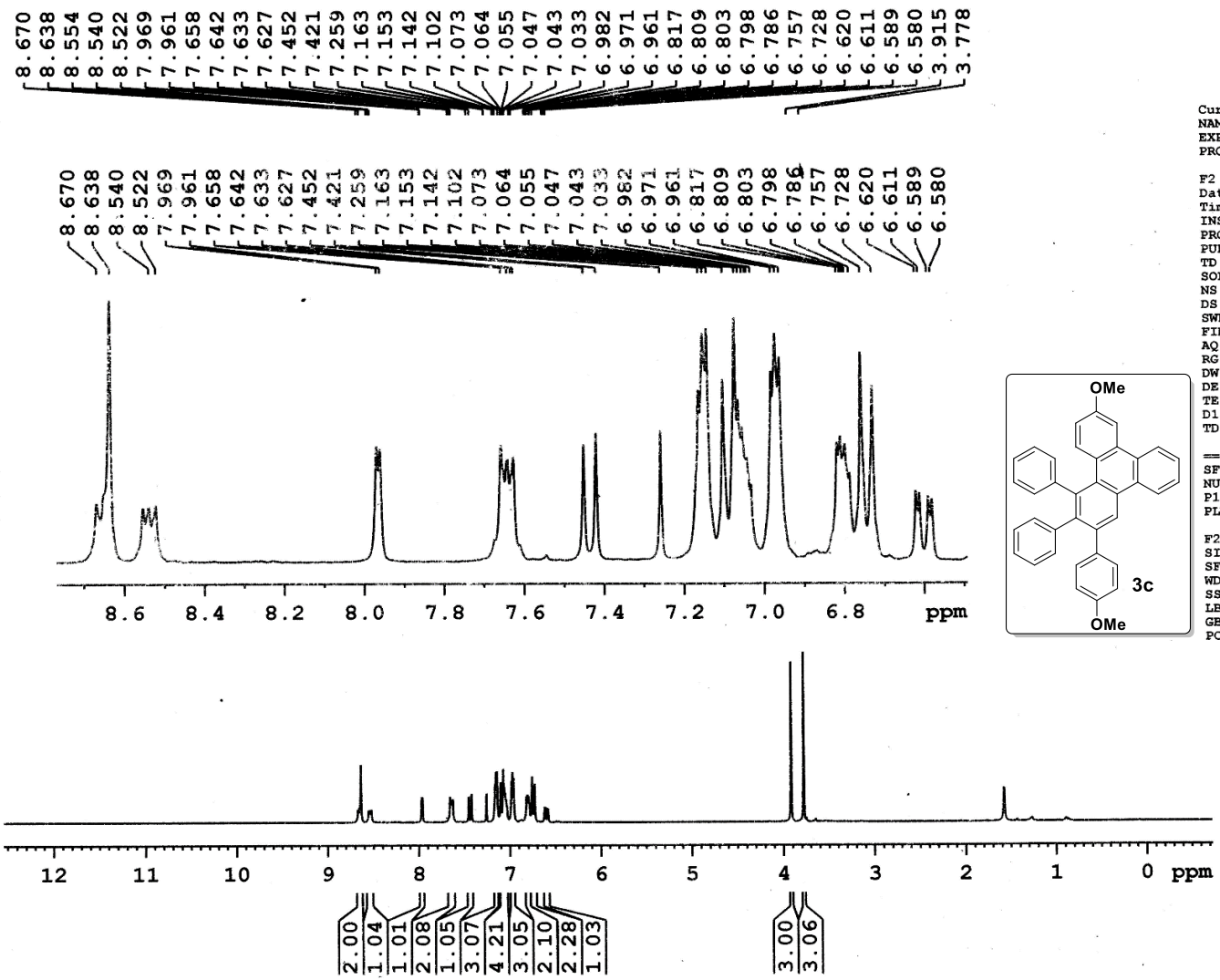
F2 - Acquisition Parameters
Date_              20240205
Time                   20.54
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG          deptsp135
TD                     65536
SOLVENT                CDCl3
NS                       256
DS                         4
SWH              12077.295 Hz
FIDRES            0.184285 Hz
AQ                2.7131903 sec
RG                      2050
DW                 41.400 usec
DE                  6.50 usec
TE                 300.0 K
CNST2            145.0000000
D1                 2.00000000 sec
D2                 0.00344828 sec
D12                0.00002000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             75.4737856 MHz
NUC1                     13C
P1                    11.00 usec
P13                 2000.00 usec
PLW0        0 W
PLW1            48.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5                0.500
SPOFFS5     0 Hz
SPW5             8.87399960 W

======== CHANNEL f2 ========
SFO2            300.1309599 MHz
NUC2                      1H
CPDPRG[2              waltz16
P3                    12.00 usec
P4                    24.00 usec
PCPD2                 90.00 usec
PLW2            12.00000000 W
PLW12            0.21333000 W

F2 - Processing parameters
SI                        32768
SF                75.4677485 MHz
WDW                          EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **3b**



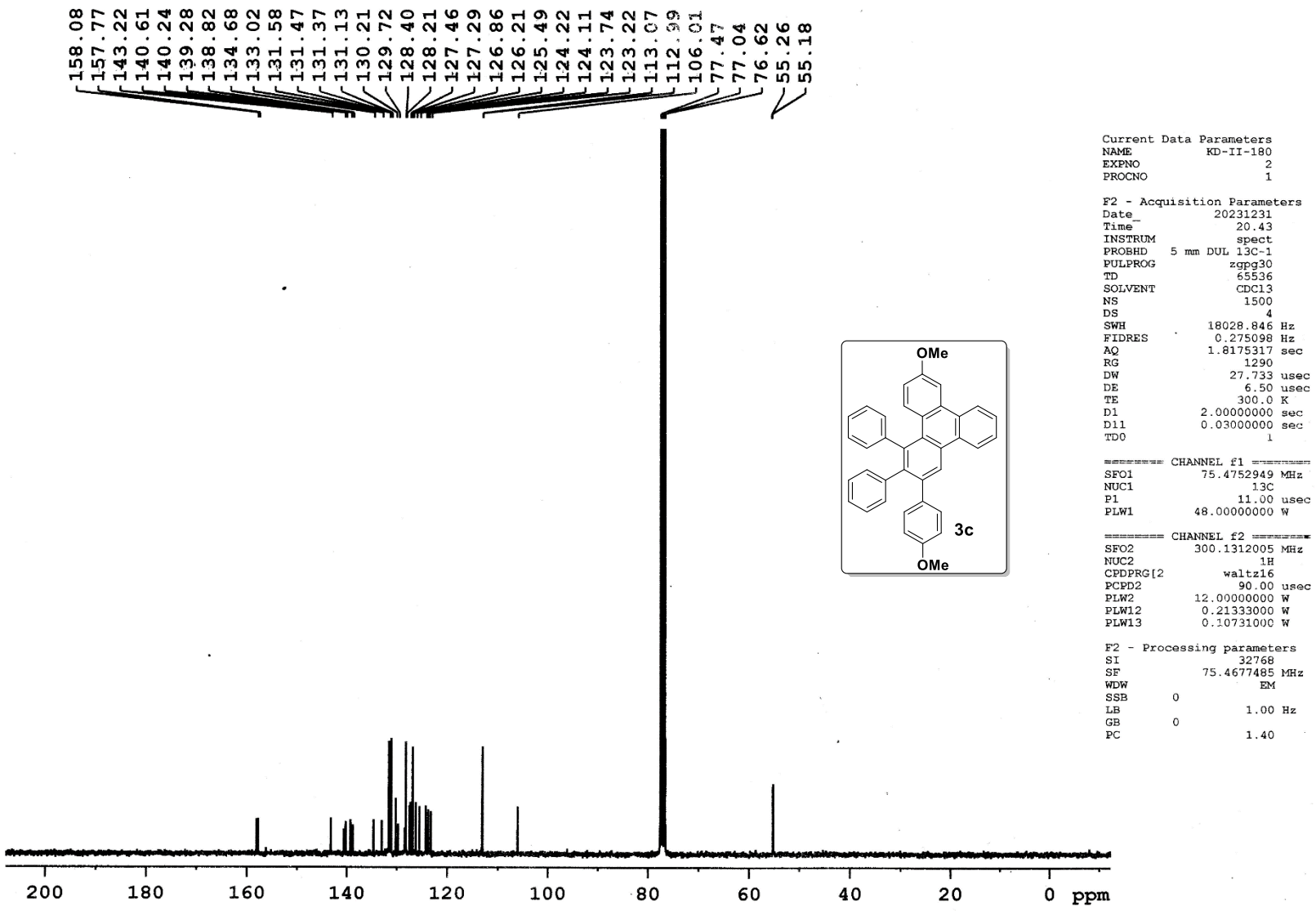

```
Current Data Parameters
NAME                  KD-II-180
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20231231
Time                      22.47
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                 6009.615 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                          256
DW                     83.200 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1             300.1318534 MHz
NUC1                       1H
P1                     12.00 usec
PLW1            12.00000000 W

F2 - Processing parameters
SI                        65536
SF               300.1300070 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3c**



Current Data Parameters
NAME KD-II-180
EXPNO 2
PROCNO 1

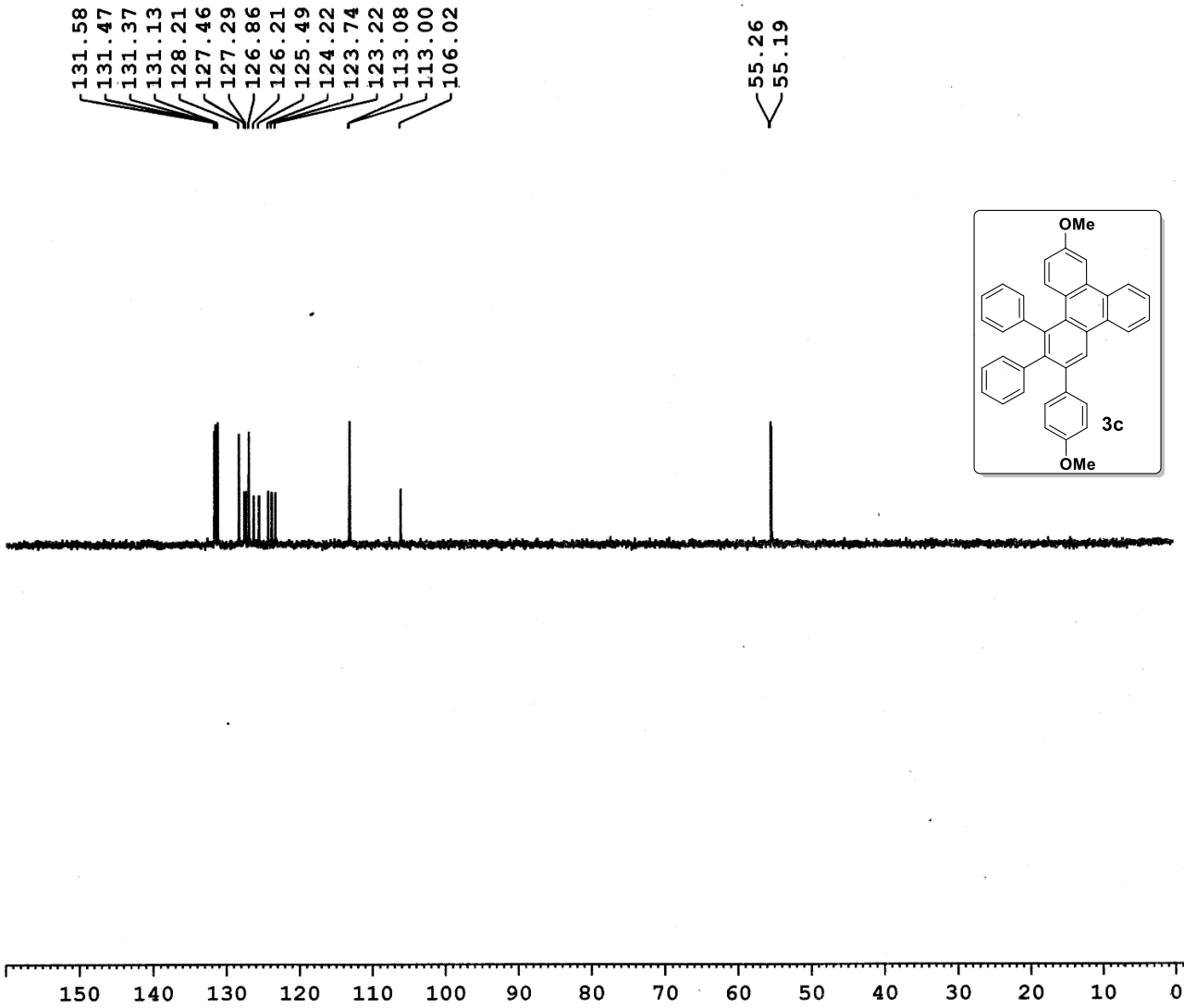
F2 - Acquisition Parameters
Date_ 20231231
Time 20.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 1290
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 11.00 usec
PLW1 48.00000000 W

======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.21333000 W
PLW13 0.10731000 W

F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3c**




```
Current Data Parameters
NAME                  KD-II-180
EXPNO                         3
PROCNO                        1

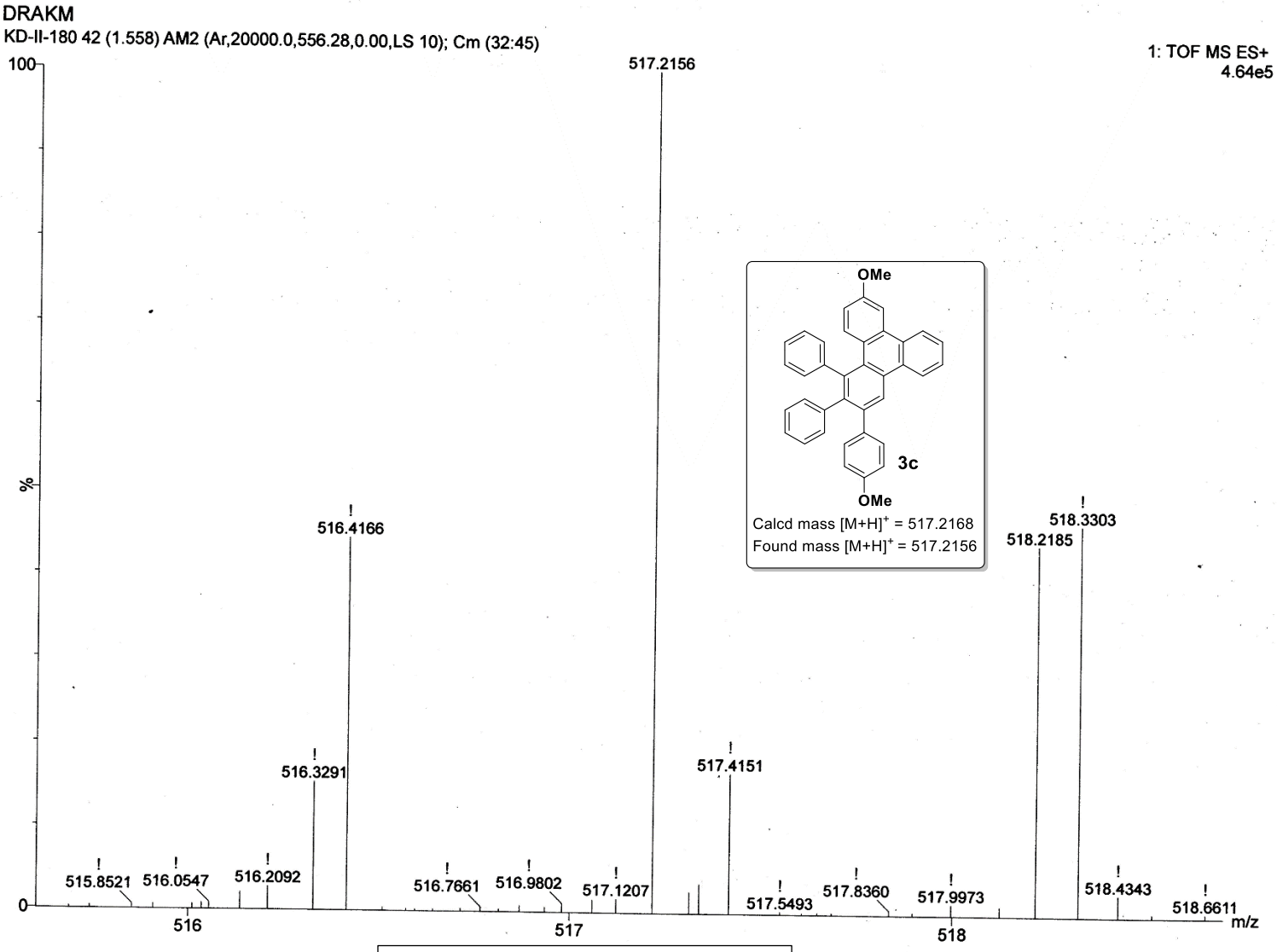
F2 - Acquisition Parameters
Date_                  20231231
Time                      22.44
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG              deptsp135
TD                       65536
SOLVENT                  CDCl3
NS                         500
DS                           4
SWH                  12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                        2050
DW                      41.400 usec
DE                       6.50 usec
TE                      300.0 K
CNST2                145.0000000
D1                     2.00000000 sec
D2                     0.00344828 sec
D12                    0.00002000 sec
TD0                           1

========== CHANNEL f1 =========
SFO1                75.4737856 MHz
NUC1                       13C
P1                       11.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1               48.00000000 W
SPNAM[5]           Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5                8.87399960 W

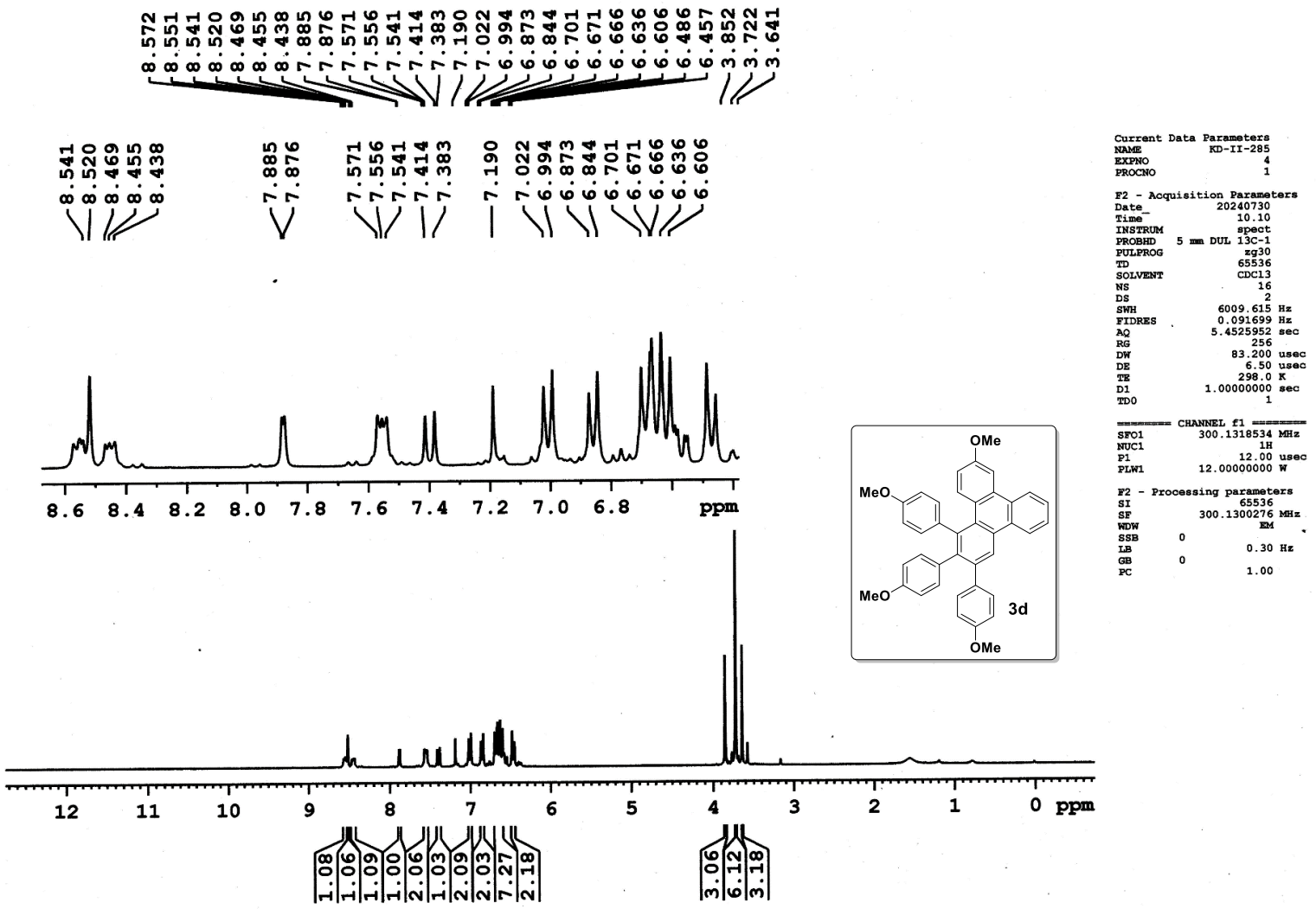
========== CHANNEL f2 =========
SFO2               300.1309599 MHz
NUC2                       1H
CPDPRG[2                waltz16
P3                       12.00 usec
P4                       24.00 usec
PCPD2                    90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677485 MHz
WDW                           EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

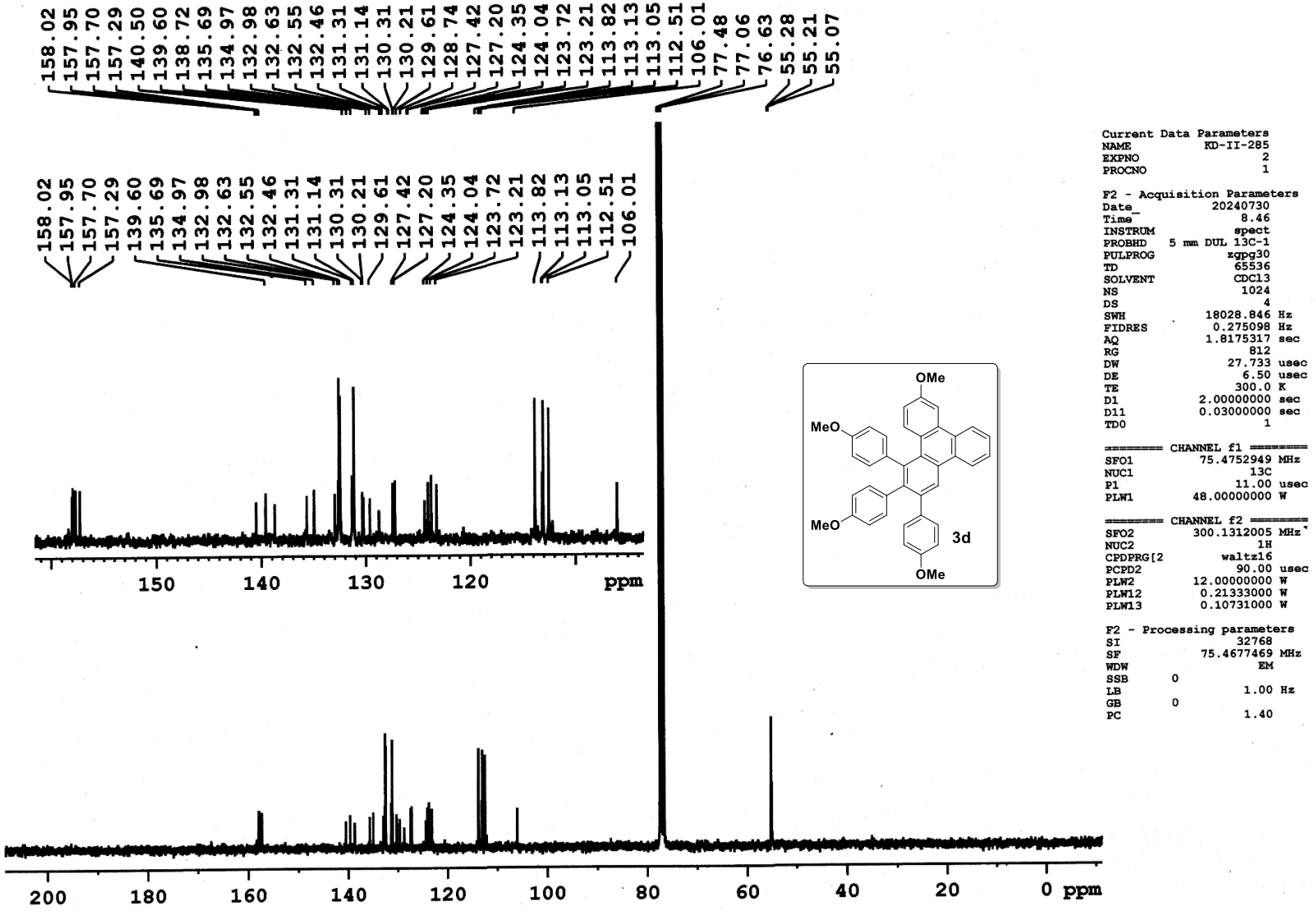
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **3c**



HRMS spectrum of compound **3c**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3d**

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Current Data Parameters
NAME                  KD-II-285
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240730
Time                       8.46
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                        812
DW                     27.733 usec
DE                      6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

========== CHANNEL f1 ==========
SFO1               75.4752949 MHz
NUC1                       13C
P1                     11.00 usec
PLW1              48.00000000 W

========== CHANNEL f2 ==========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                   90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                         32768
SF                75.4677469 MHz
WDW                           EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```
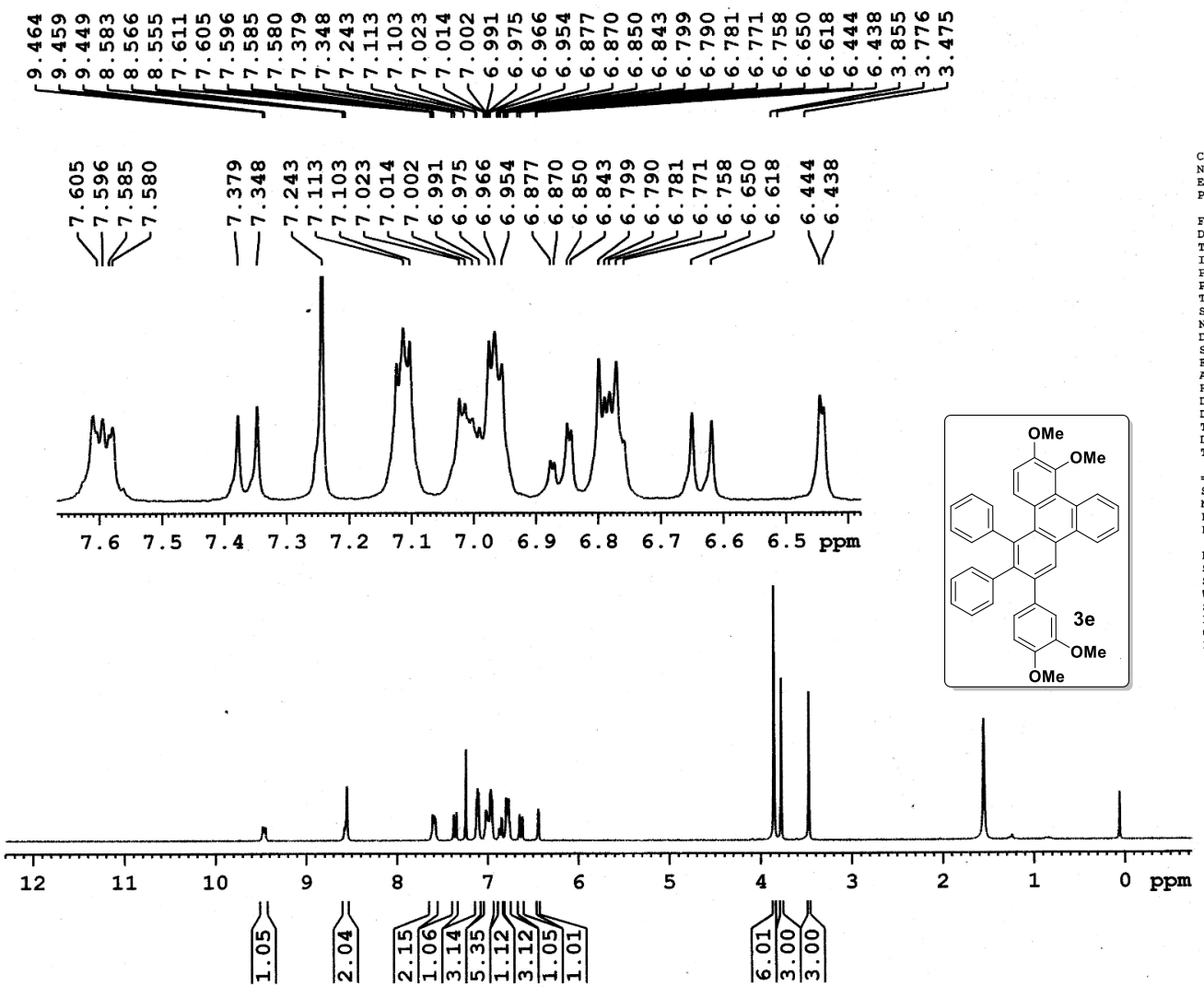
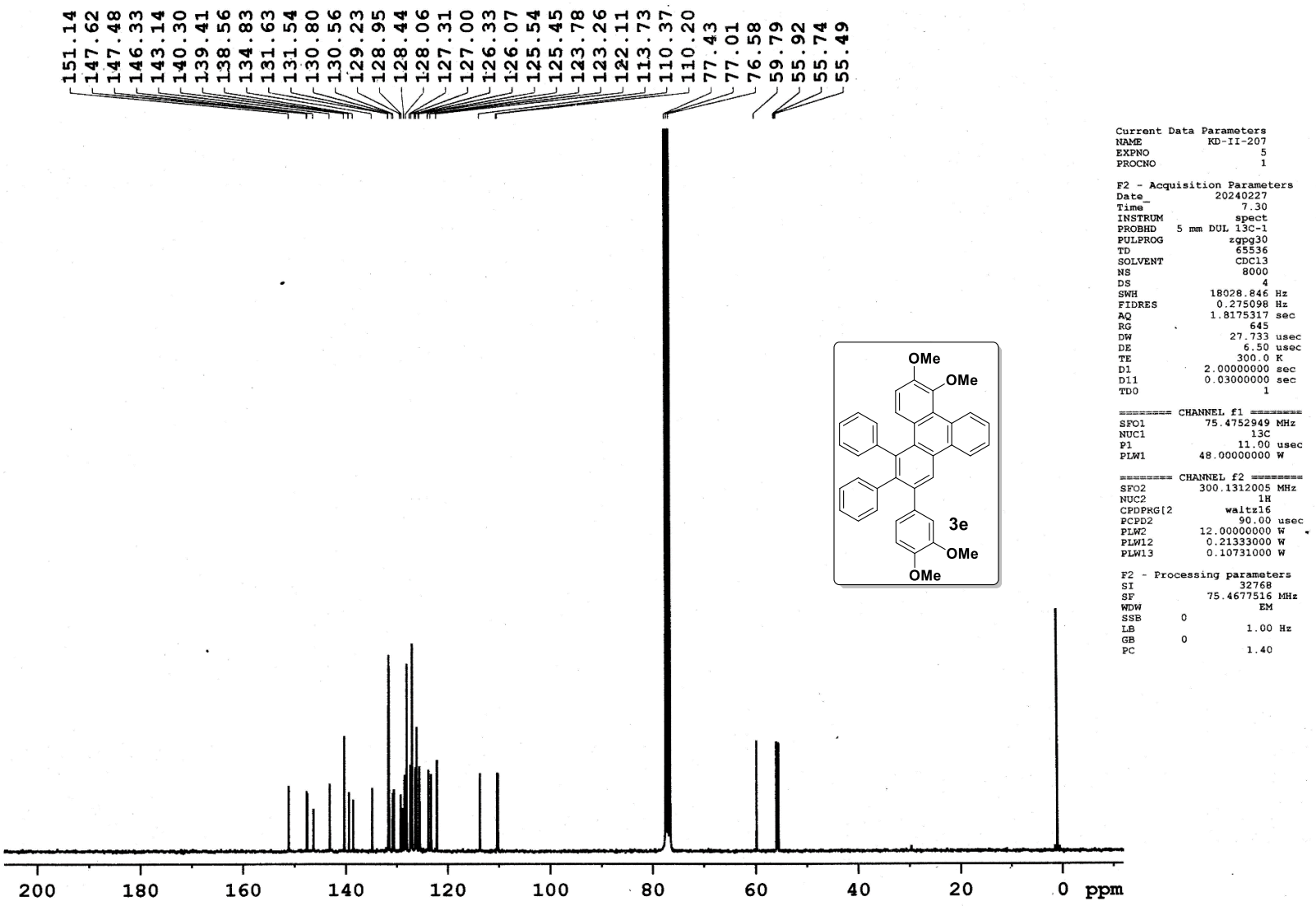

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3d**



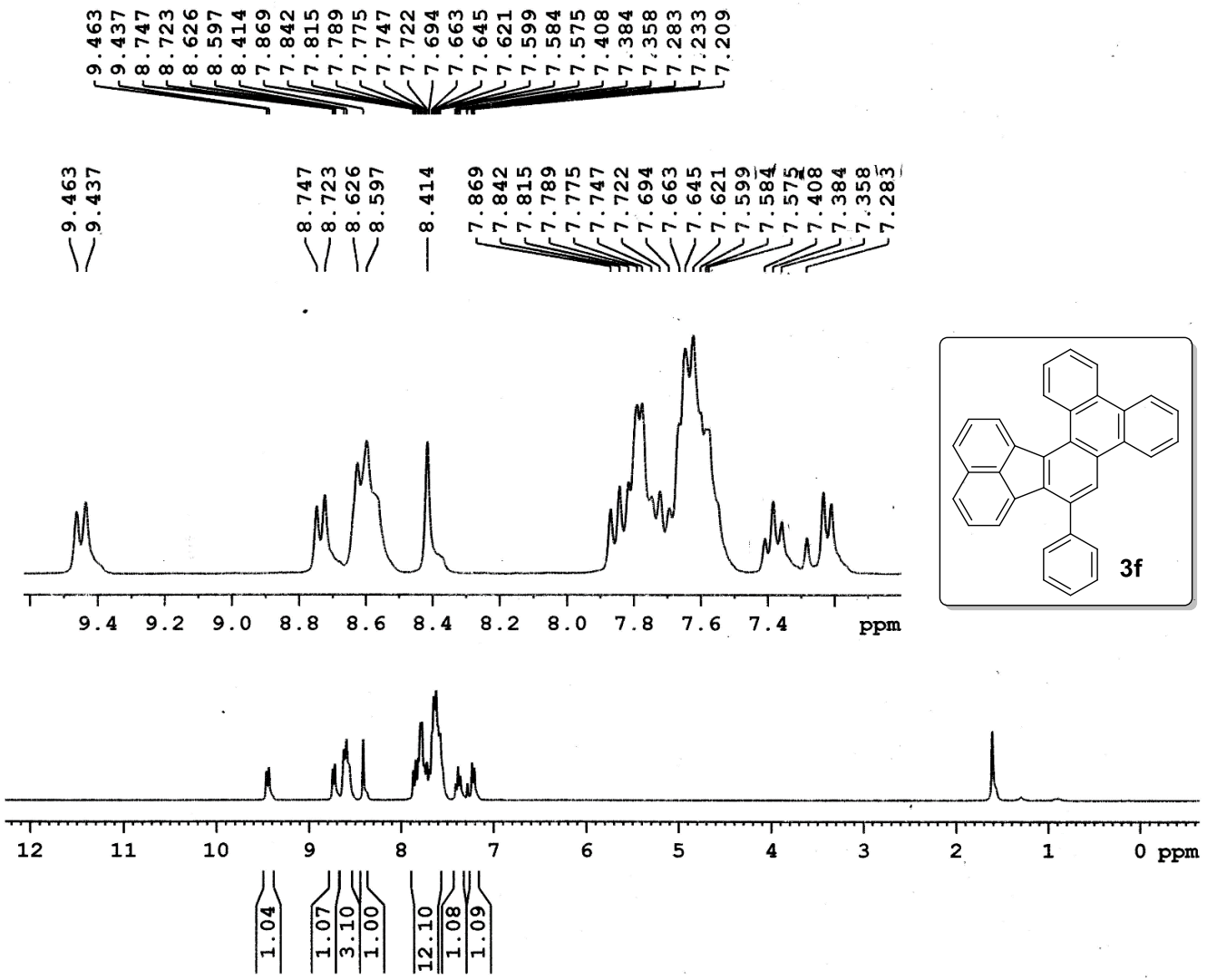

```
Current Data Parameters
NAME                  KD-II-207
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240223
Time                      17.24
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.091699 Hz
AQ                     5.4525952 sec
RG                          406
DW                       83.200 usec
DE                        6.50 usec
TE                       298.0 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1            300.1318534 MHz
NUC1                       1H
P1                      12.00 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                        65536
SF              300.1300115 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                        1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3e**



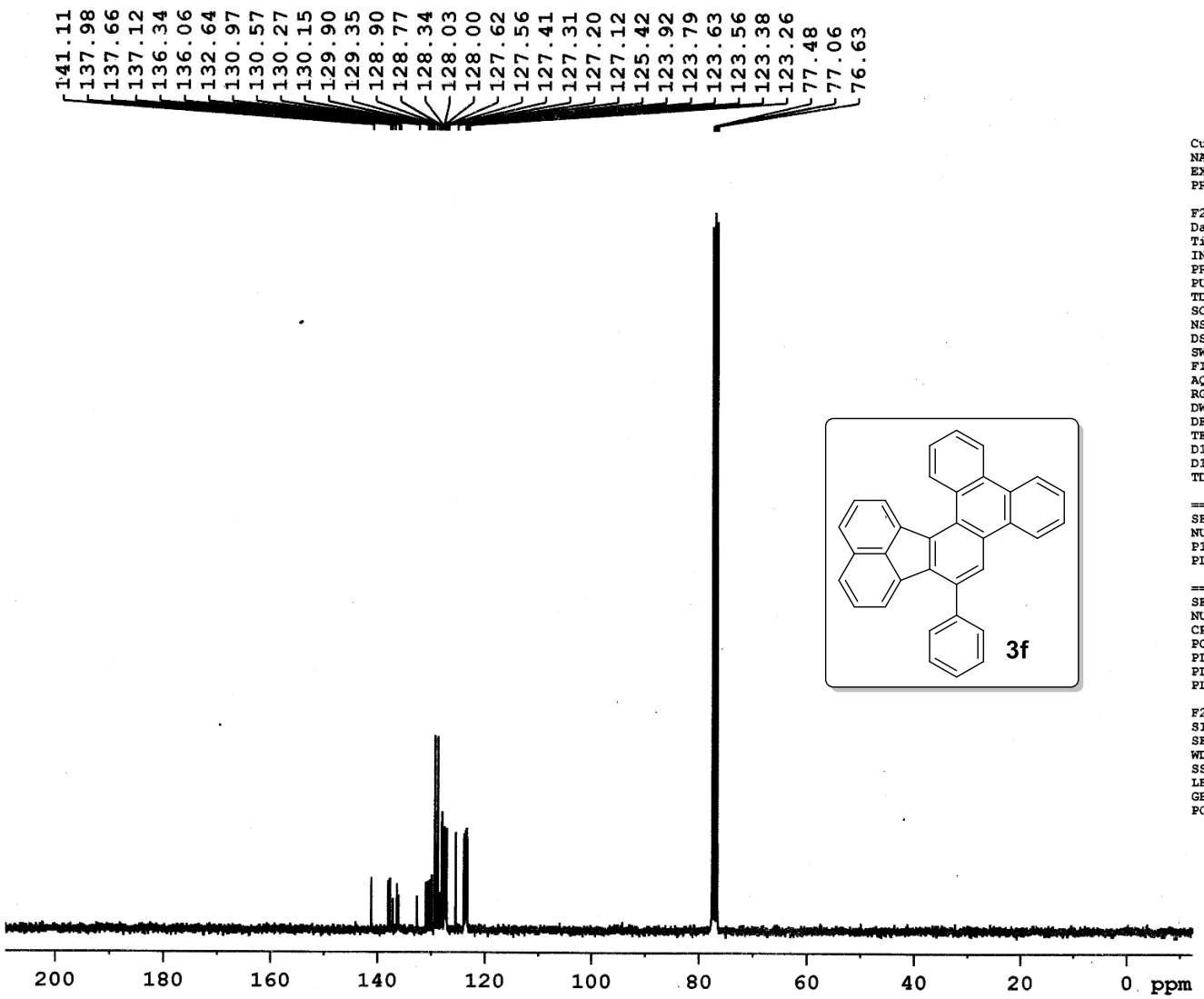

```
Current Data Parameters
NAME                  KD-II-207
EXPNO                         5
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240227
Time                       7.30
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         8000
DS                            4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                          645
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677516 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3e**



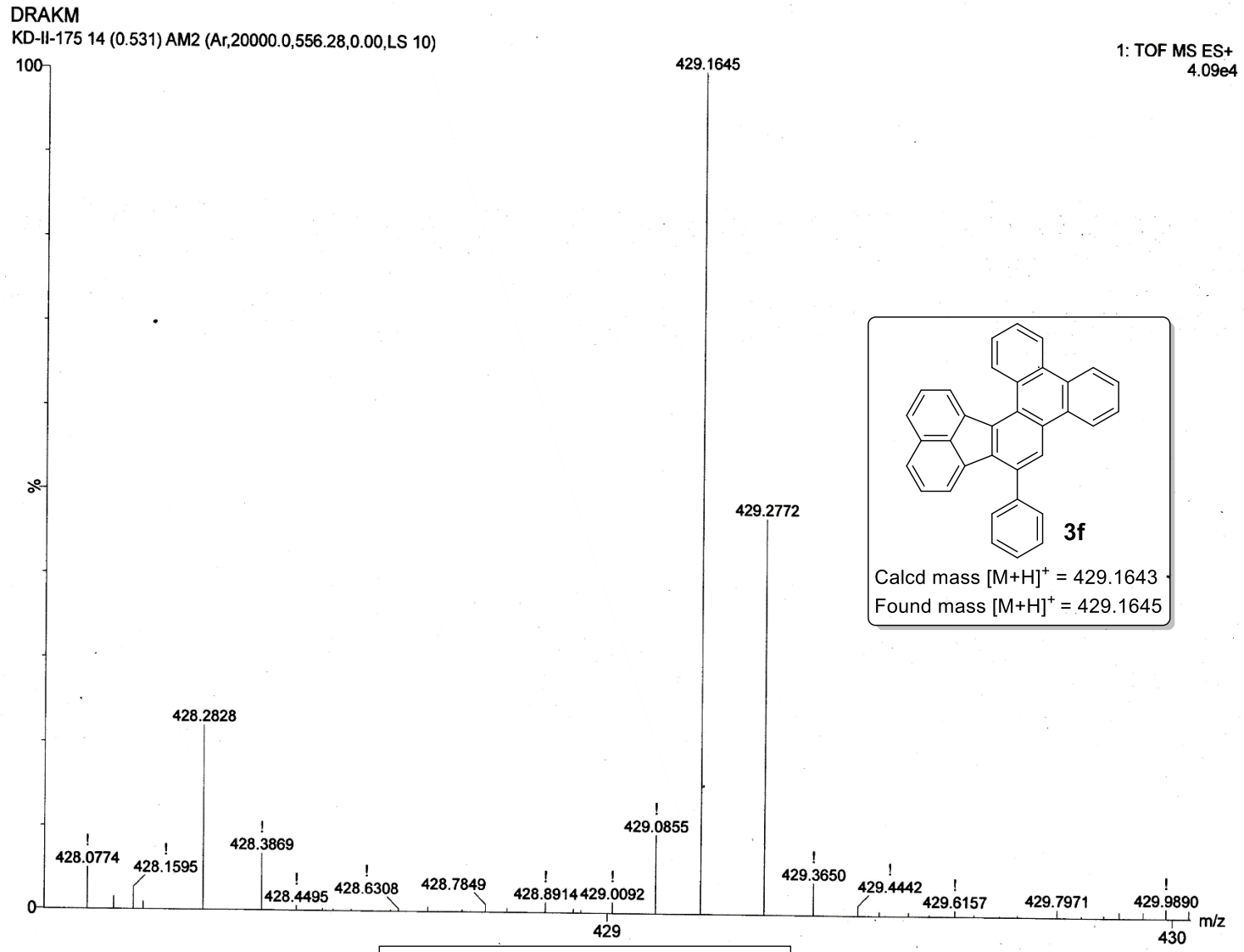

```
Current Data Parameters
NAME                  KD-II-175
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20231227
Time                   20.10
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.091699 Hz
AQ                5.4525952 sec
RG                      228
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                       1

========== CHANNEL f1 ==========
SFO1          300.1318534 MHz
NUC1                      1H
P1                 12.00 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                    65536
SF            300.1300000 MHz
WDW                      EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3f**

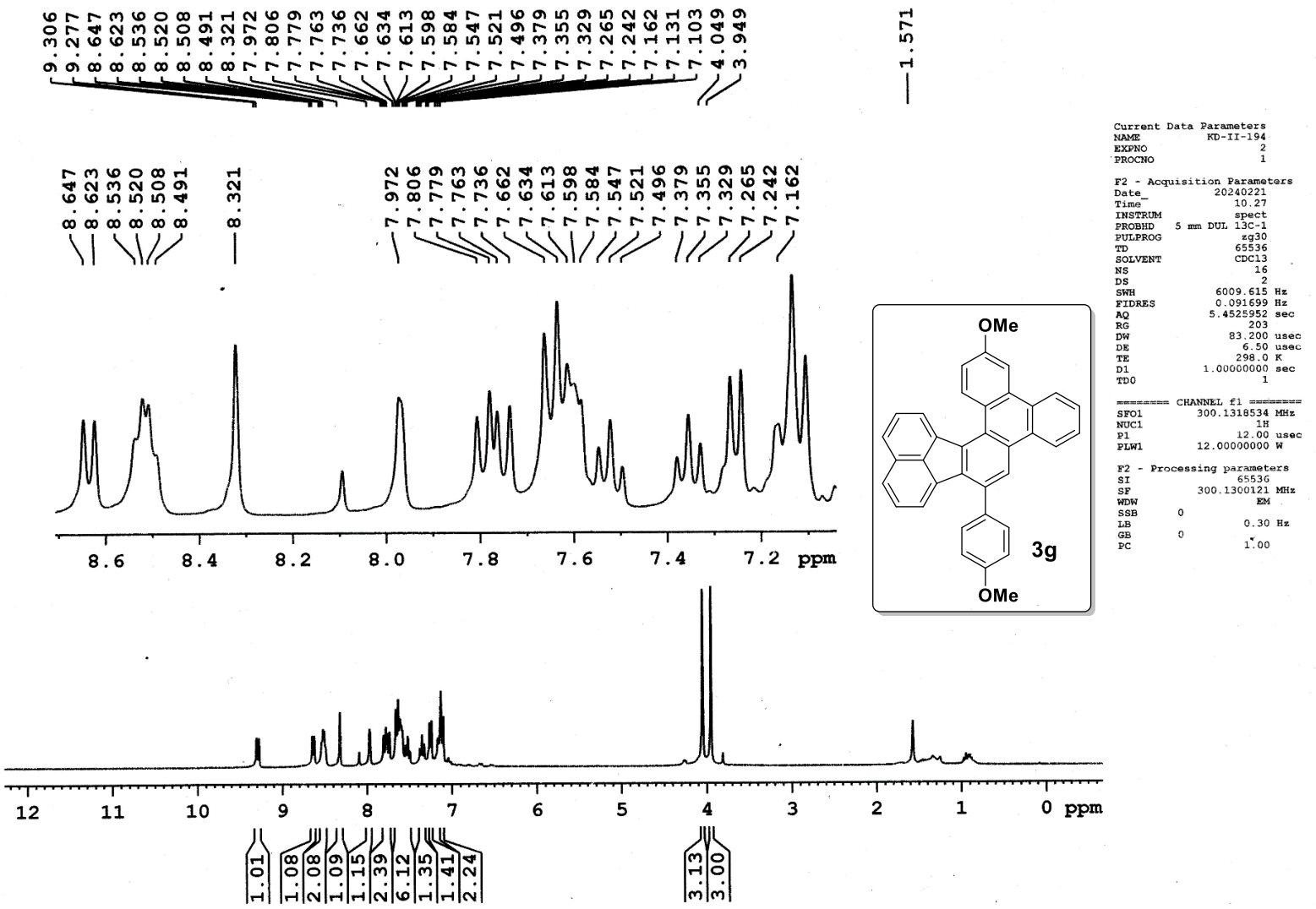



```
Current Data Parameters
NAME            KD-II-175
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20231227
Time                20.16
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175317 sec
RG                  1030
DW               27.733 usec
DE                6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 11.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W
PLW13         0.10731000 W

F2 - Processing parameters
SI                 32768
SF            75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

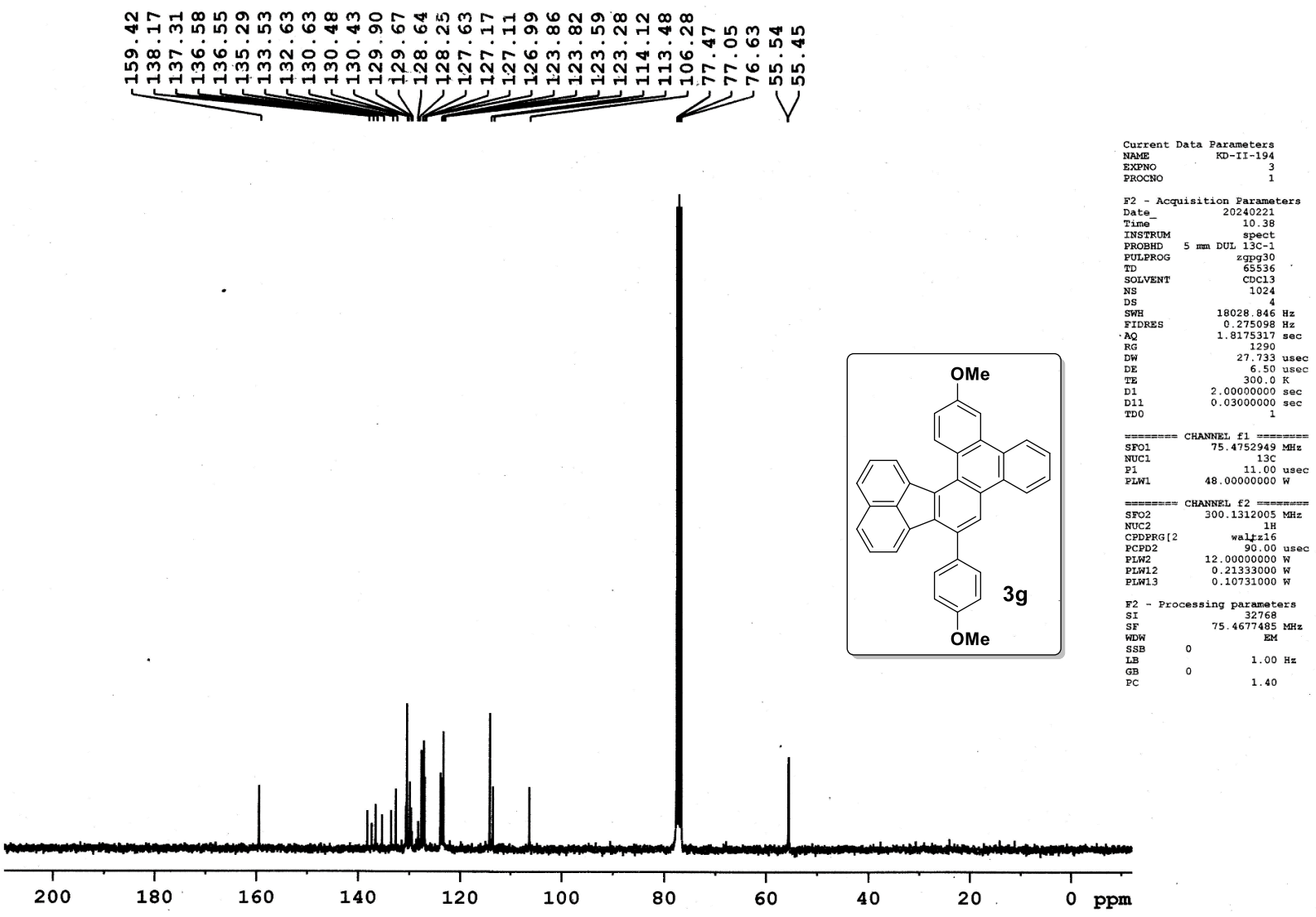
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3f**



HRMS spectrum of compound **3f**

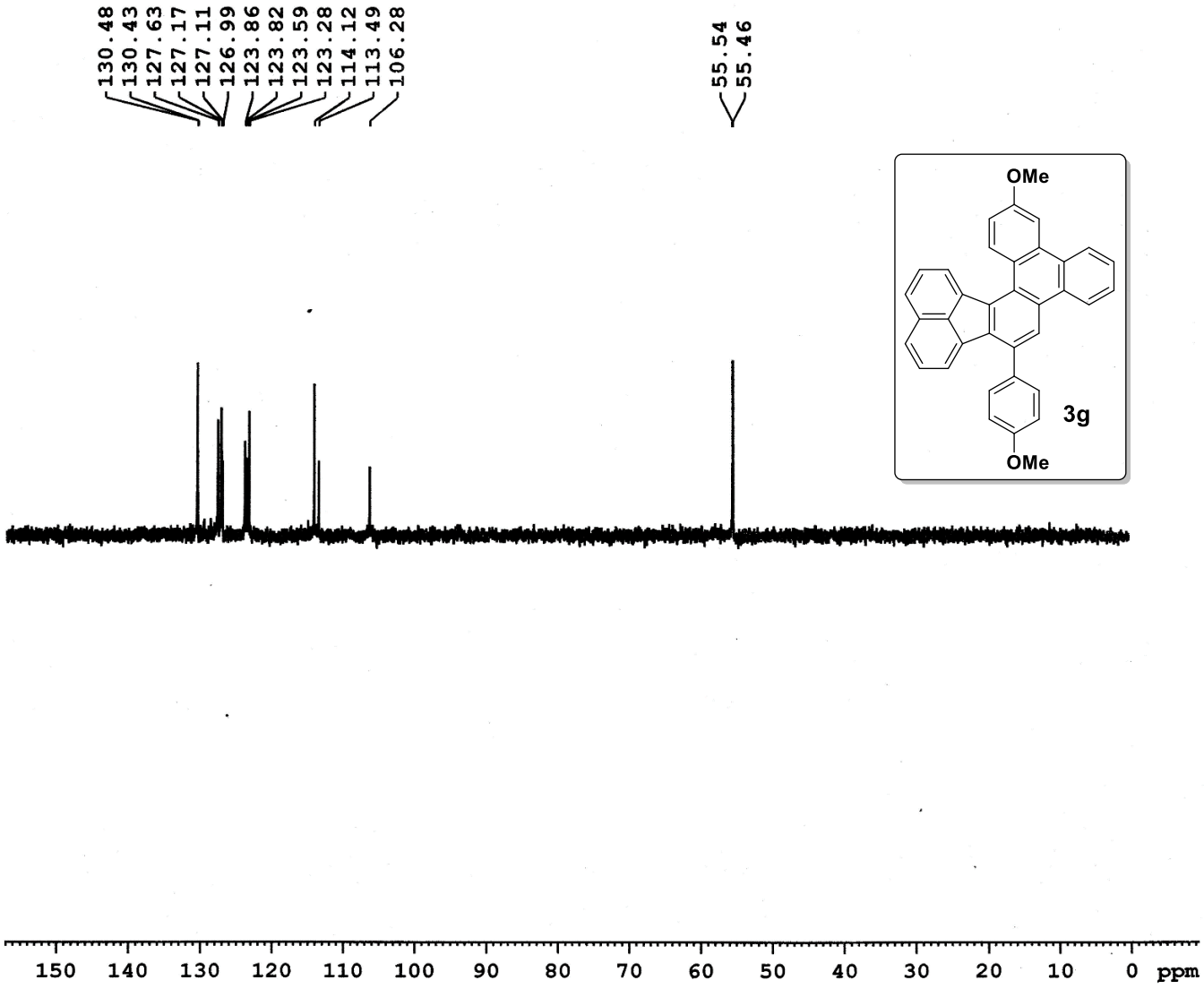


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3g**



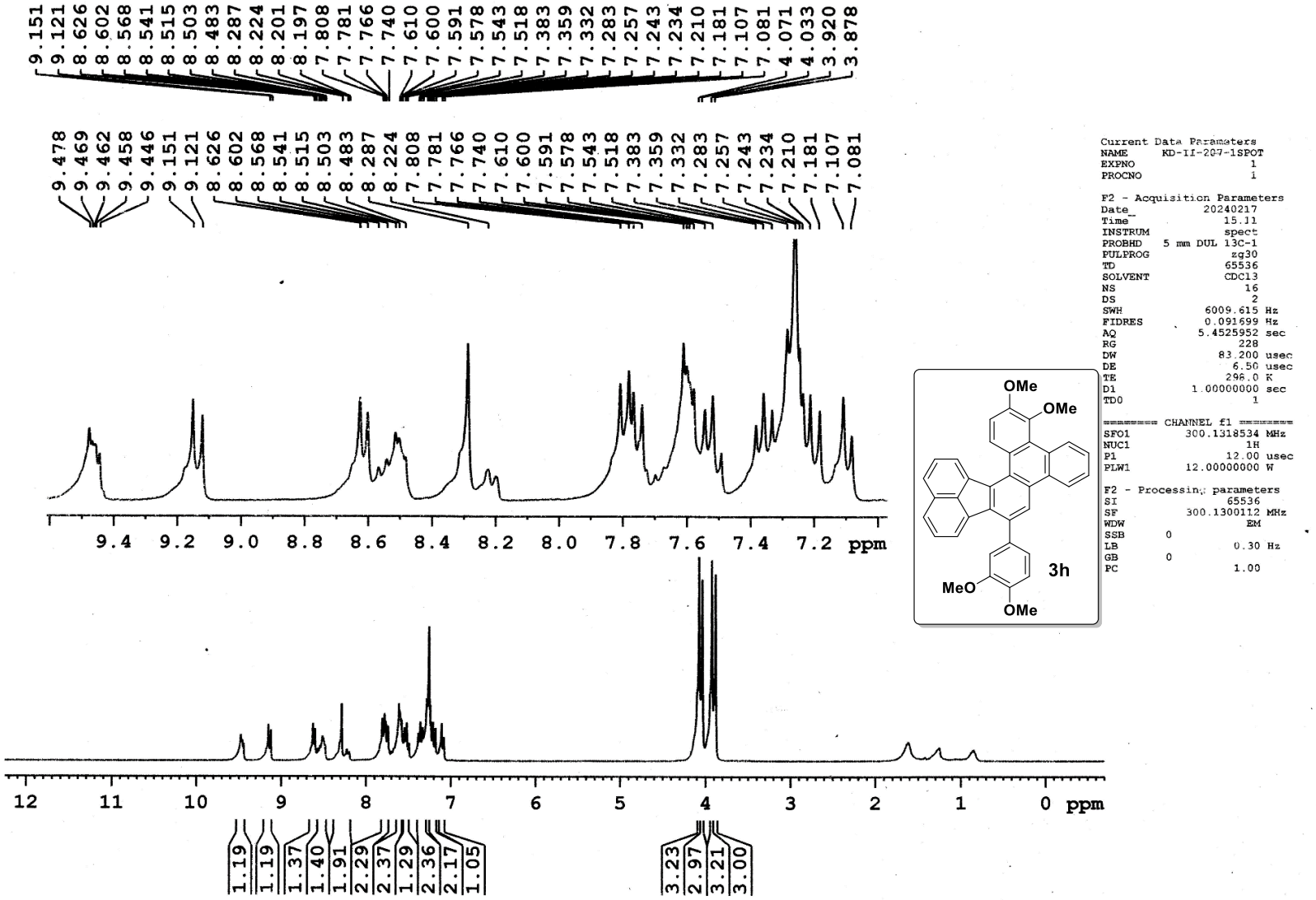

```
Current Data Parameters
NAME                  KD-II-194
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240221
Time                      10.38
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                       1290
DW                     27.733 usec
DE                      6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3g**

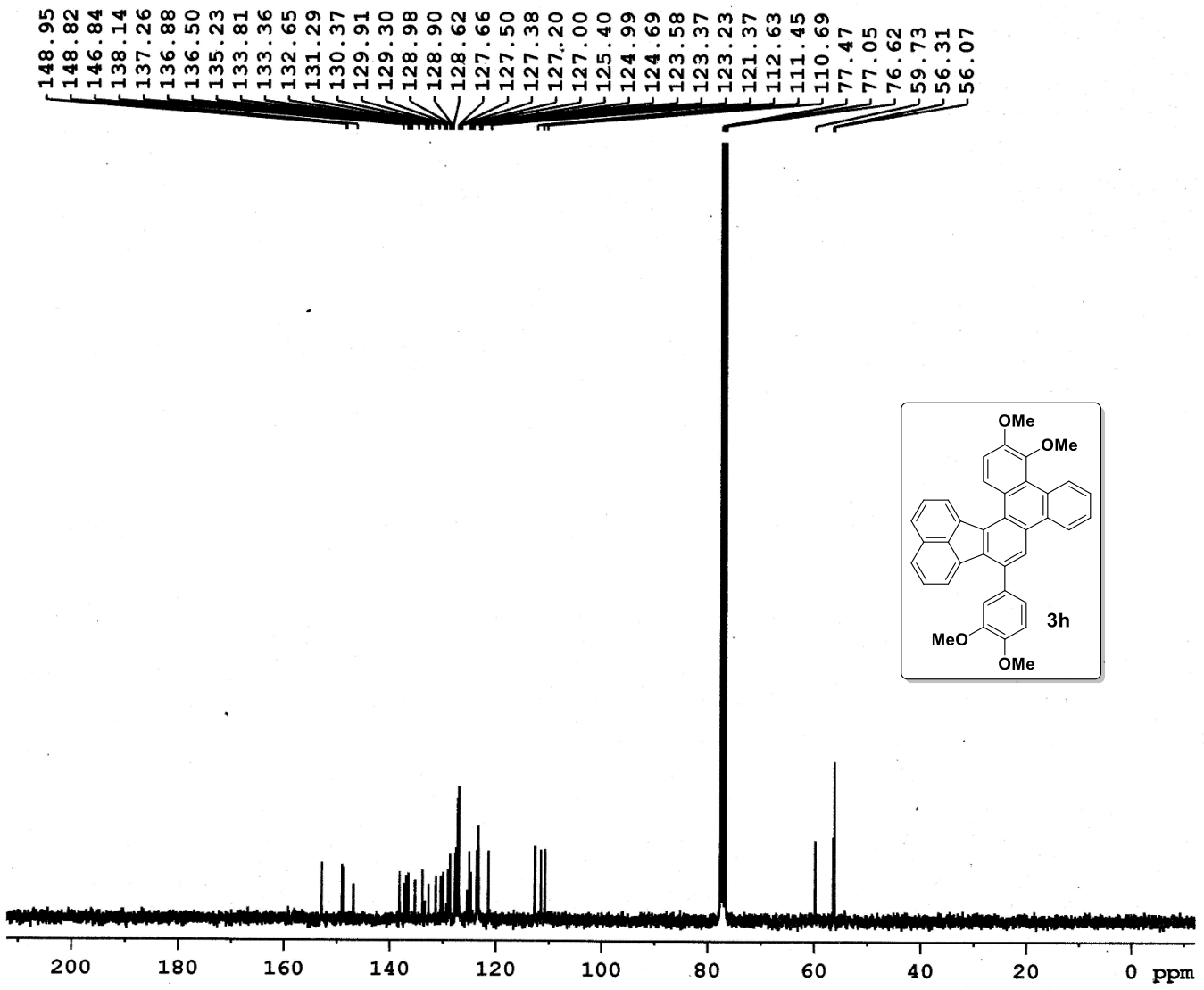



```
Current Data Parameters
NAME                  KD-II-194
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240221
Time                      11.57
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG              deptsp135
TD                        65536
SOLVENT                   CDCl3
NS                          256
DS                            4
SWH                 12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                         2050
DW                       41.400 usec
DE                        6.50 usec
TE                       300.0 K
CNST2               145.0000000
D1                    2.00000000 sec
D2                    0.00344828 sec
D12                   0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                75.4737856 MHz
NUC1                       13C
P1                       11.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1              48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5               8.87399960 W

======== CHANNEL f2 ========
SFO2              300.1309599 MHz
NUC2                       1H
CPDPRG[2                waltz16
P3                       12.00 usec
P4                       24.00 usec
PCPD2                    90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W

F2 - Processing parameters
SI                         32768
SF                 75.4677485 MHz
WDW                           EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **3g**

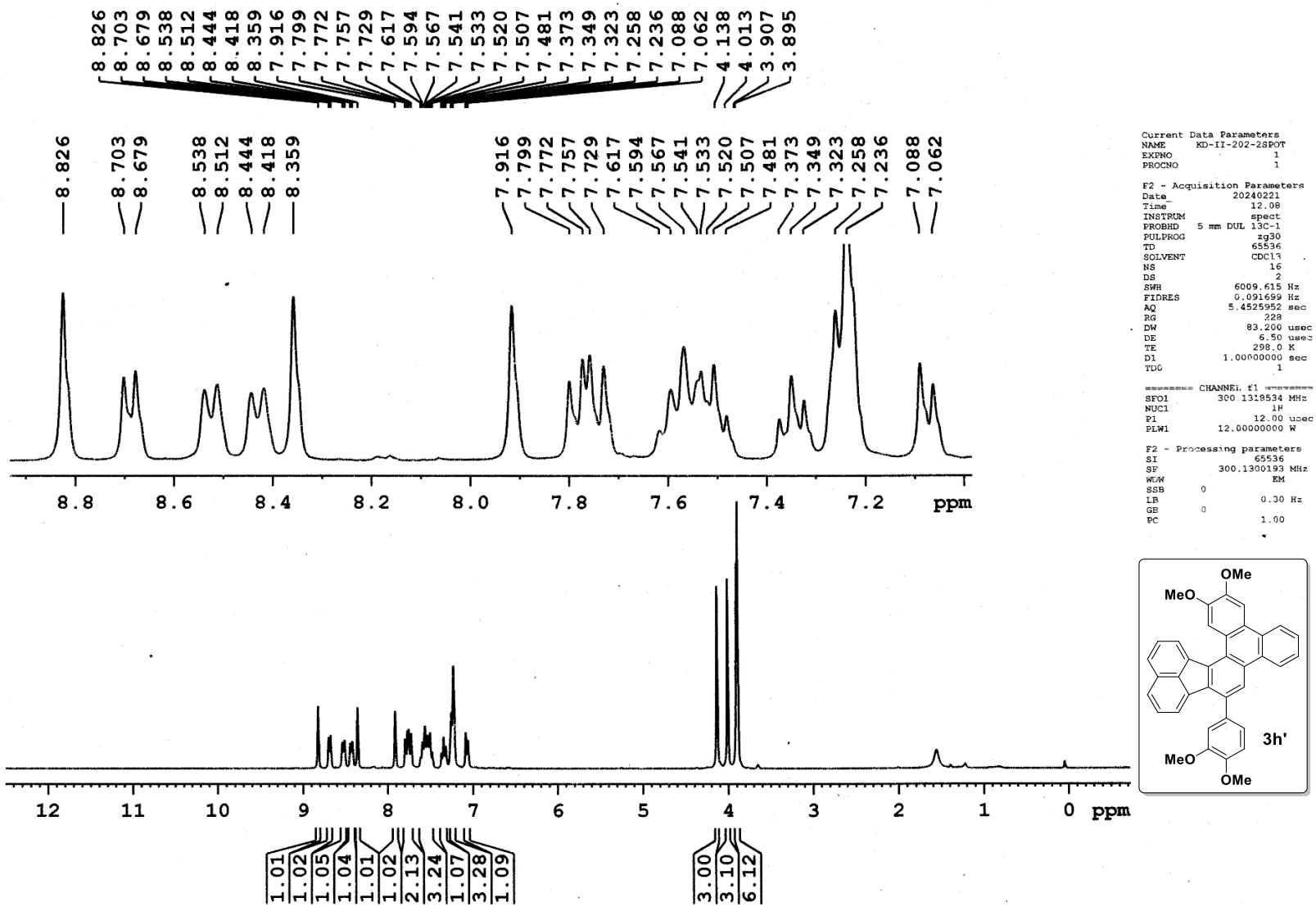


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3h**

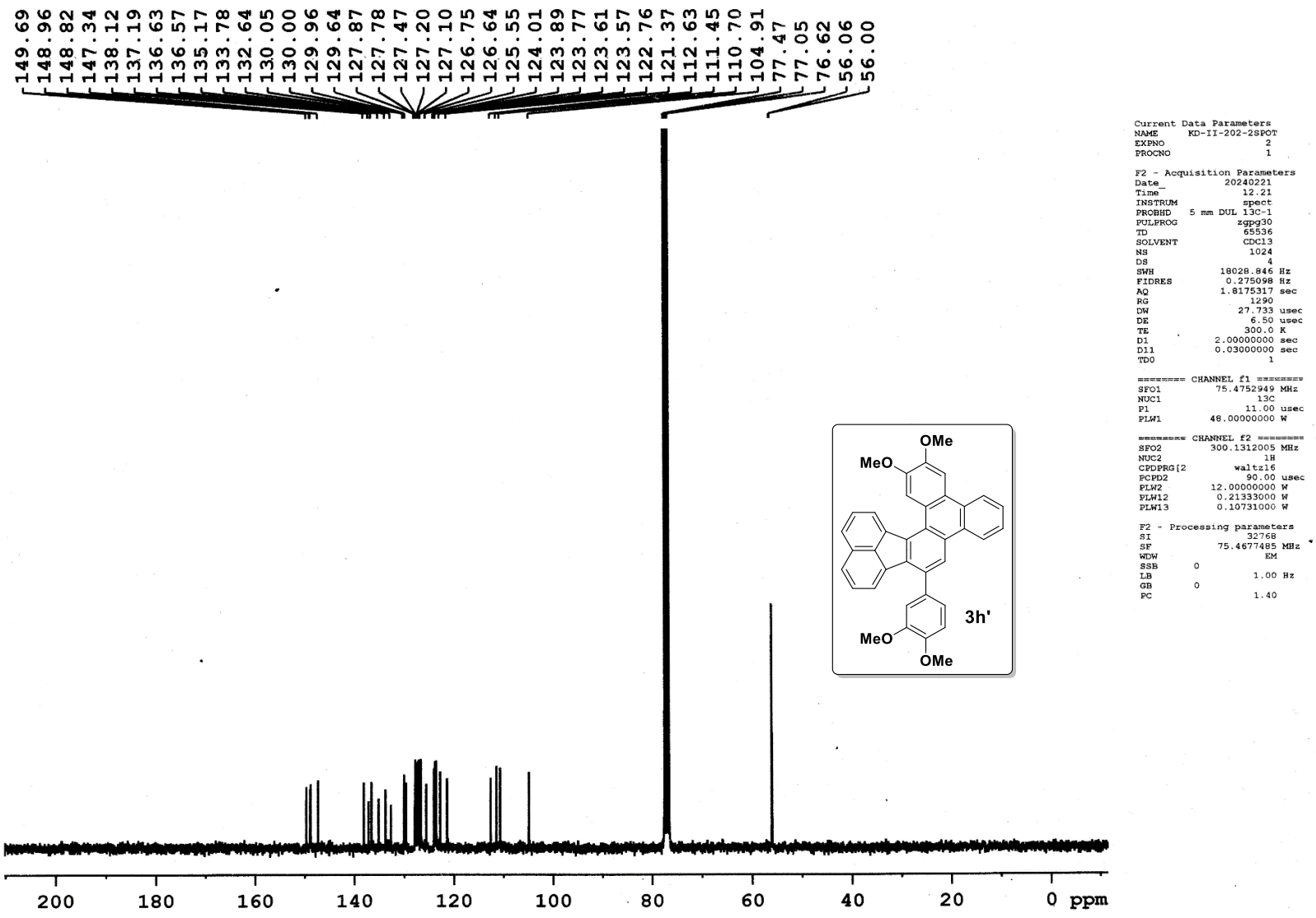



```
Current Data Parameters
NAME      KD-II-207-1SPOT
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20240217
Time               15.09
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              65536
SOLVENT         CDCl3
NS               1024
DS                  4
SWH         18028.846 Hz
FIDRES        0.275098 Hz
AQ          1.8175317 sec
RG                724
DW             27.733 usec
DE              6.50 usec
TE             300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                1

======== CHANNEL f1 ========
SFO1       75.4752949 MHz
NUC1              13C
P1             11.00 usec
PLW1       48.00000000 W

======== CHANNEL f2 ========
SFO2      300.1312005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2          90.00 usec
PLW2       12.00000000 W
PLW12       0.21333000 W
PLW13       0.10731000 W

F2 - Processing parameters
SI                32768
SF         75.4677485 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3h**

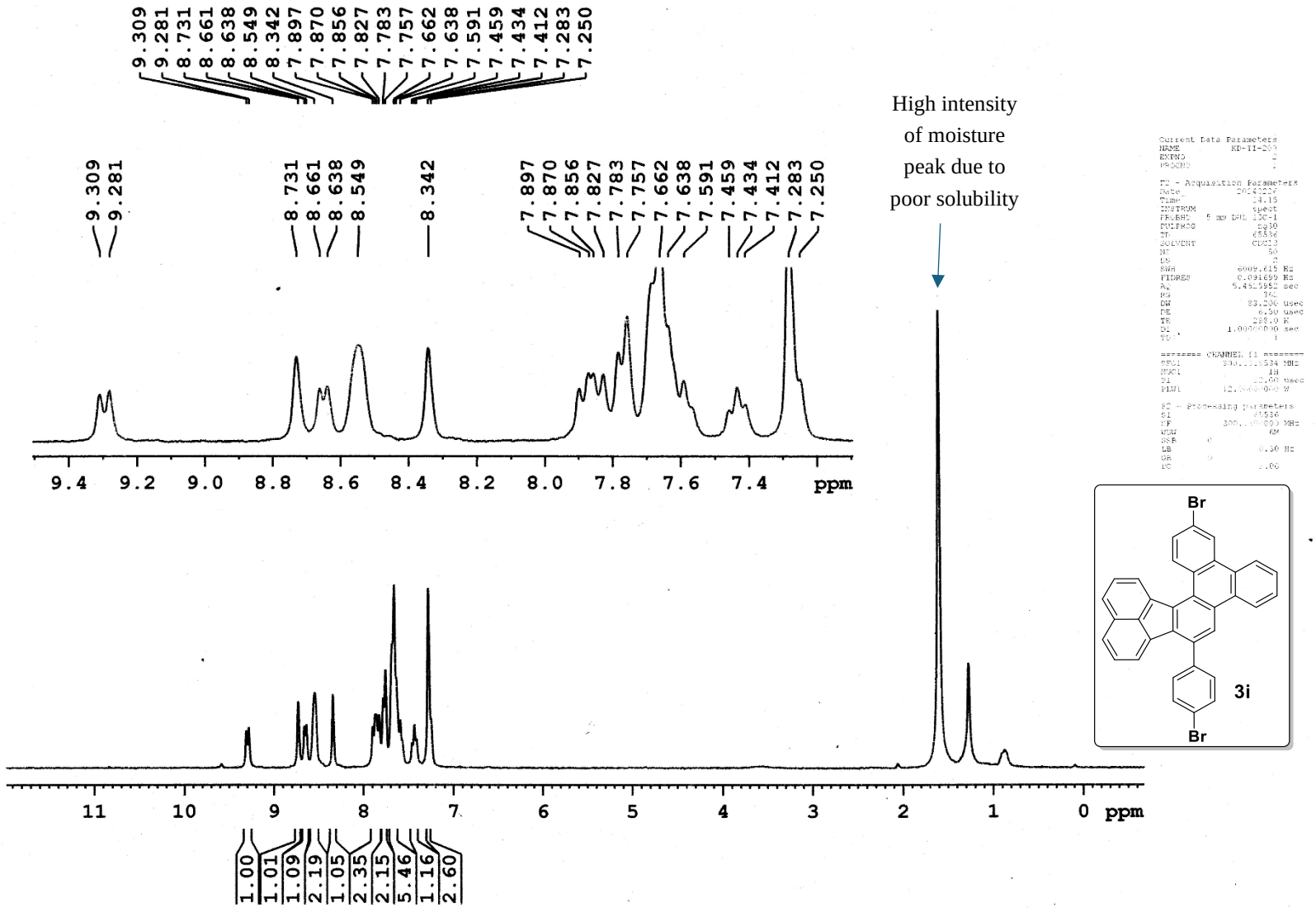


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3h'**

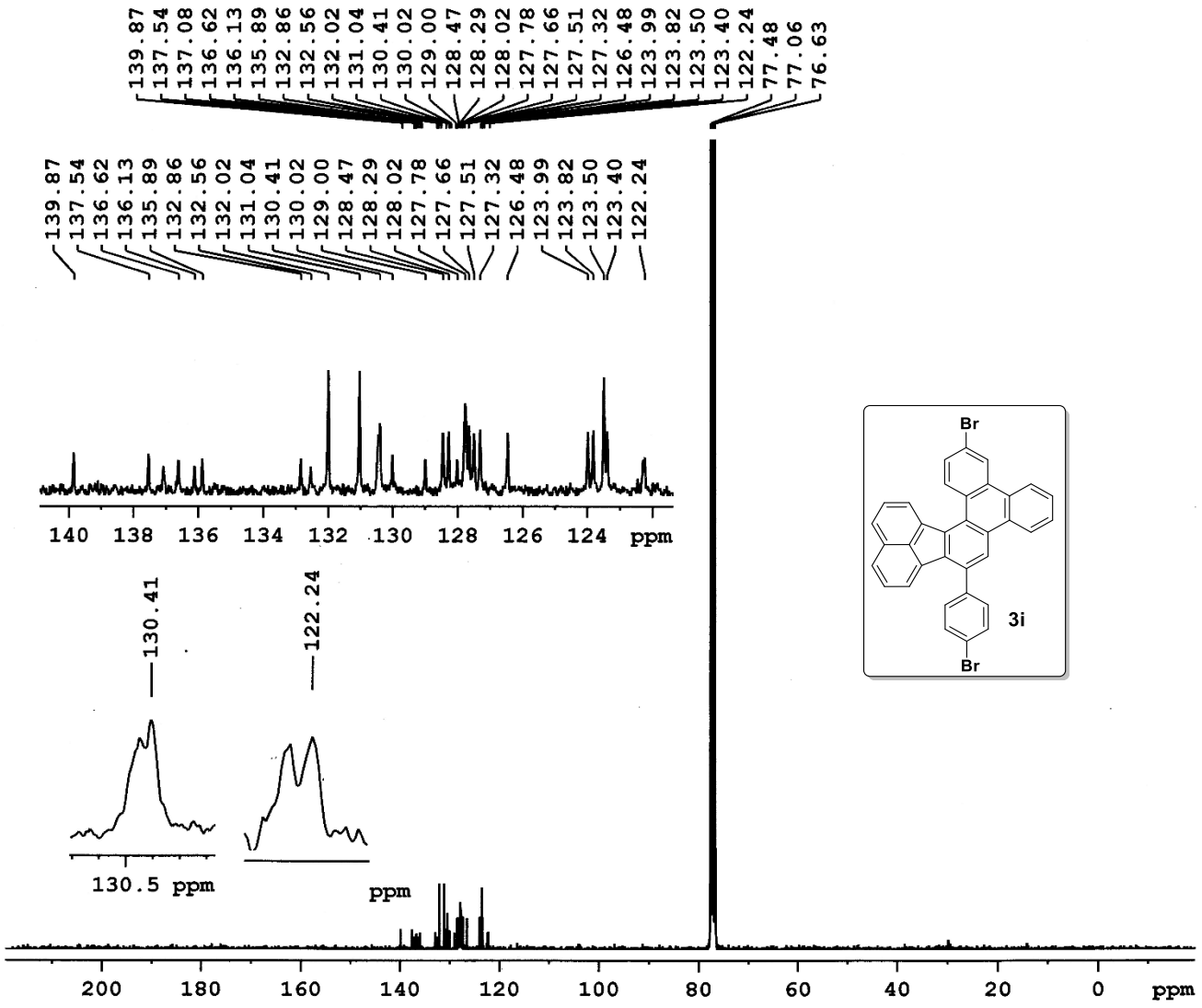



```
Current Data Parameters
NAME      KD-II-202-2SPOT
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240221
Time              12.21
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175317 sec
RG                1290
DW              27.733 usec
DE               6.50 usec
TE              300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI                 32768
SF          75.4677485 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3h'**

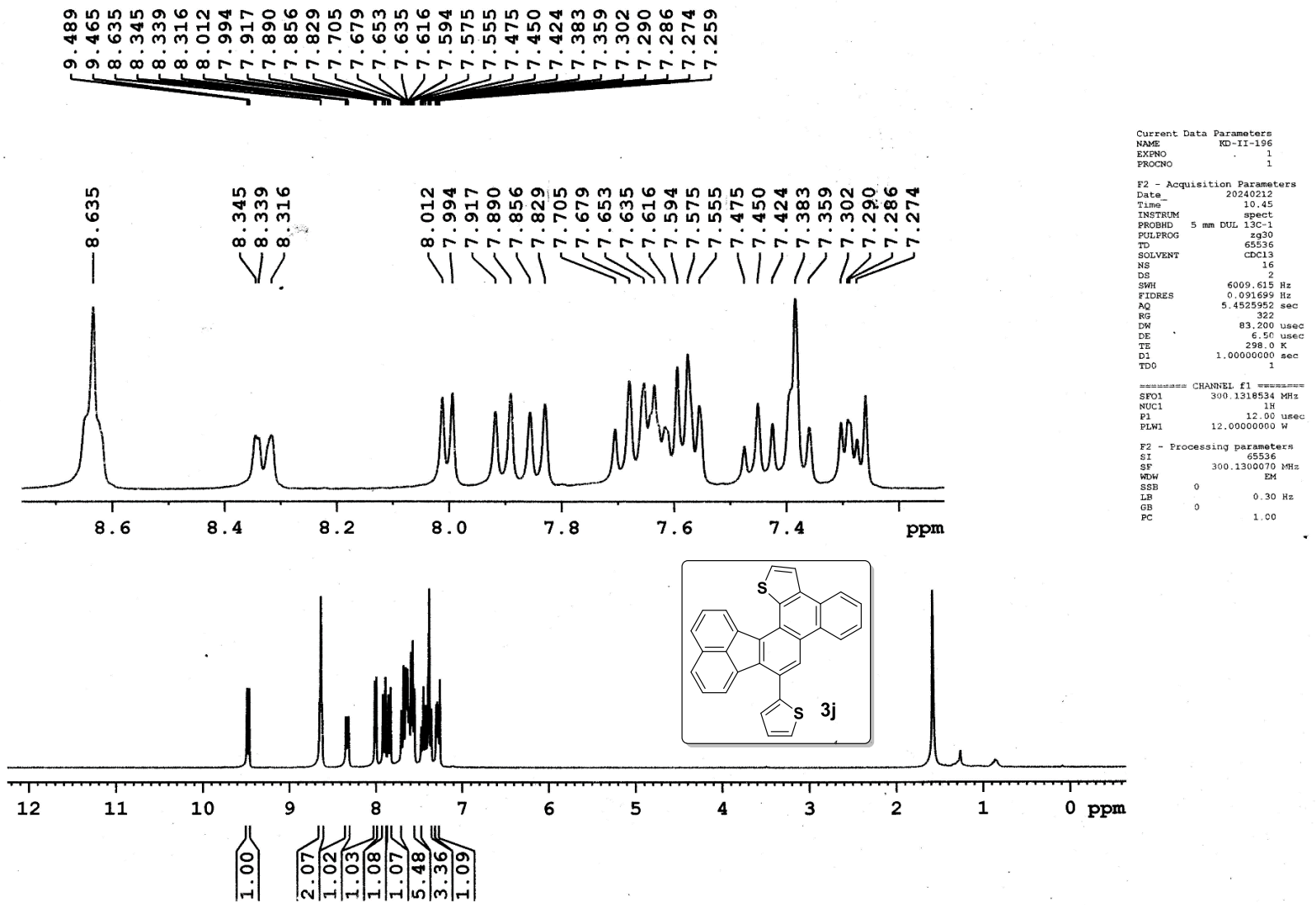


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3i**



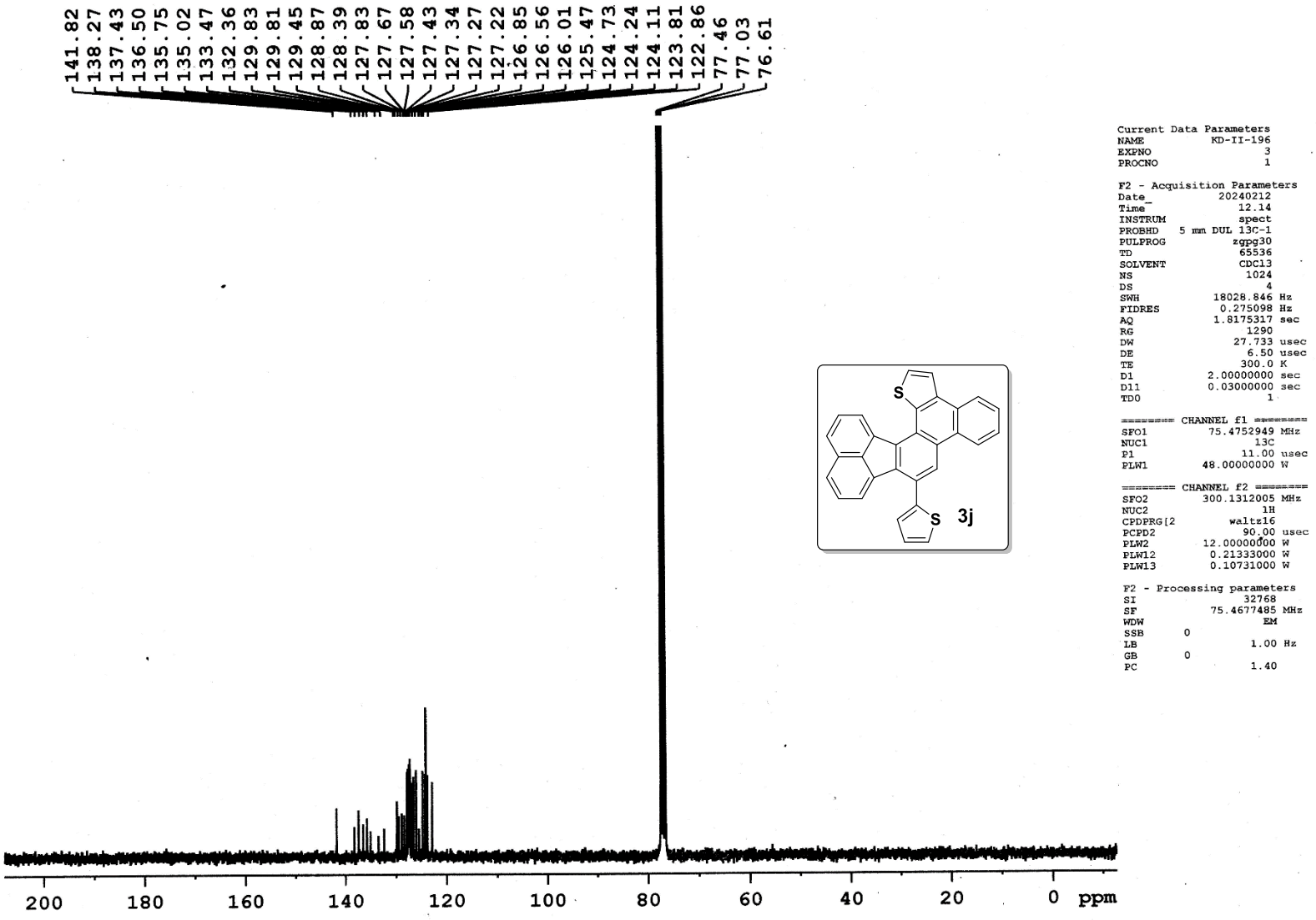

```
Current Data Parameters
NAME         KD-II-209-CC
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20241010
Time_              20.39
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                13000
DS                    4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175317 sec
RG                  912
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1               11.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W
PLW13         0.10731000 W

F2 - Processing parameters
SI                 32768
SF          75.4677463 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3i**




```
Current Data Parameters
NAME                  KD-II-196
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240212
Time                      10.45
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6009.615 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                         322
DW                     83.200 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                           1

========== CHANNEL f1 ========
SFO1               300.1318534 MHz
NUC1                        1H
P1                       12.00 usec
PLW1               12.00000000 W

F2 - Processing parameters
SI                       65536
SF                 300.1300070 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

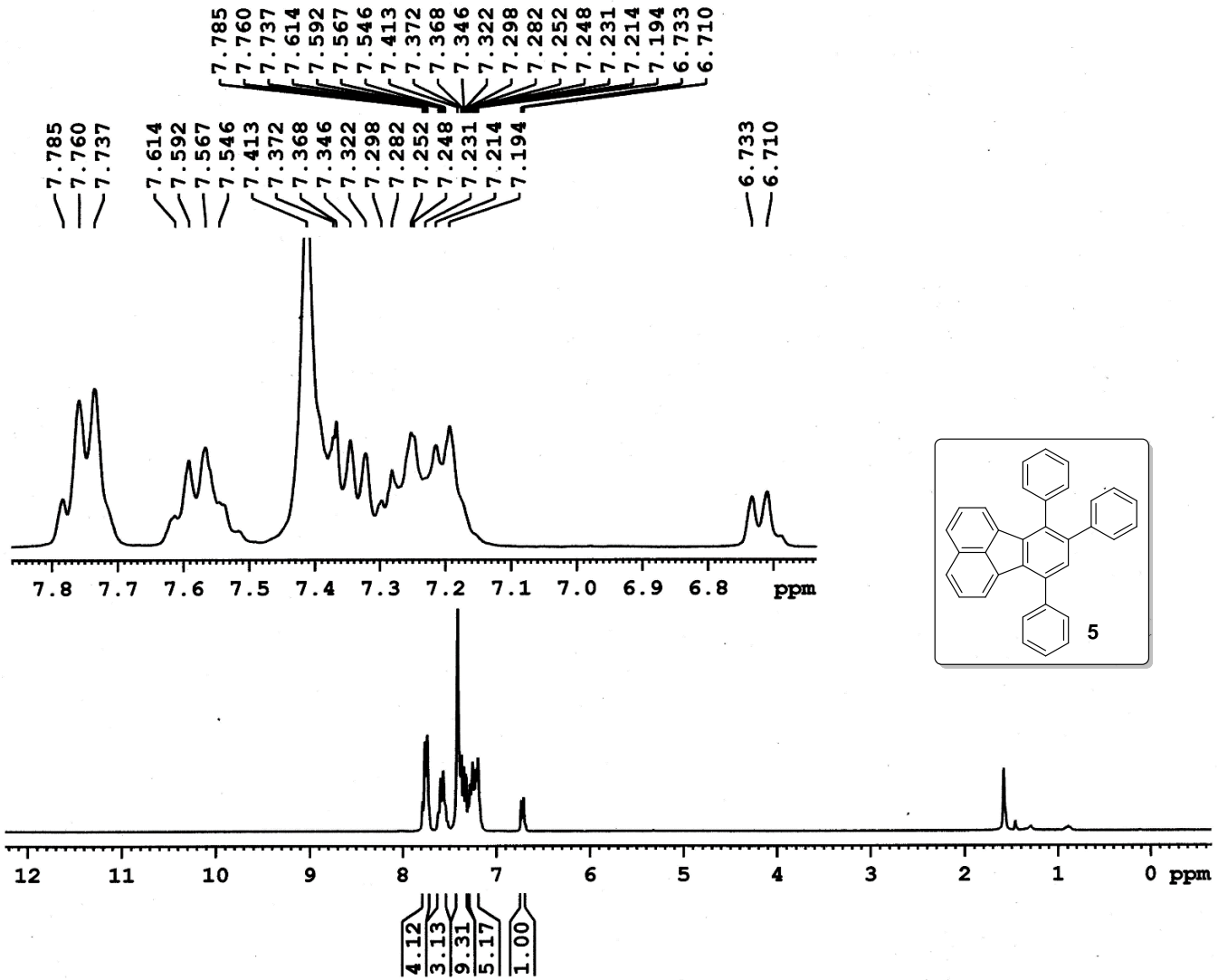
^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **3j**




```
Current Data Parameters
NAME                  KD-II-196
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240212
Time                      12.14
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                         1290
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB           0
LB                     1.00 Hz
GB            0
PC                        1.40
```

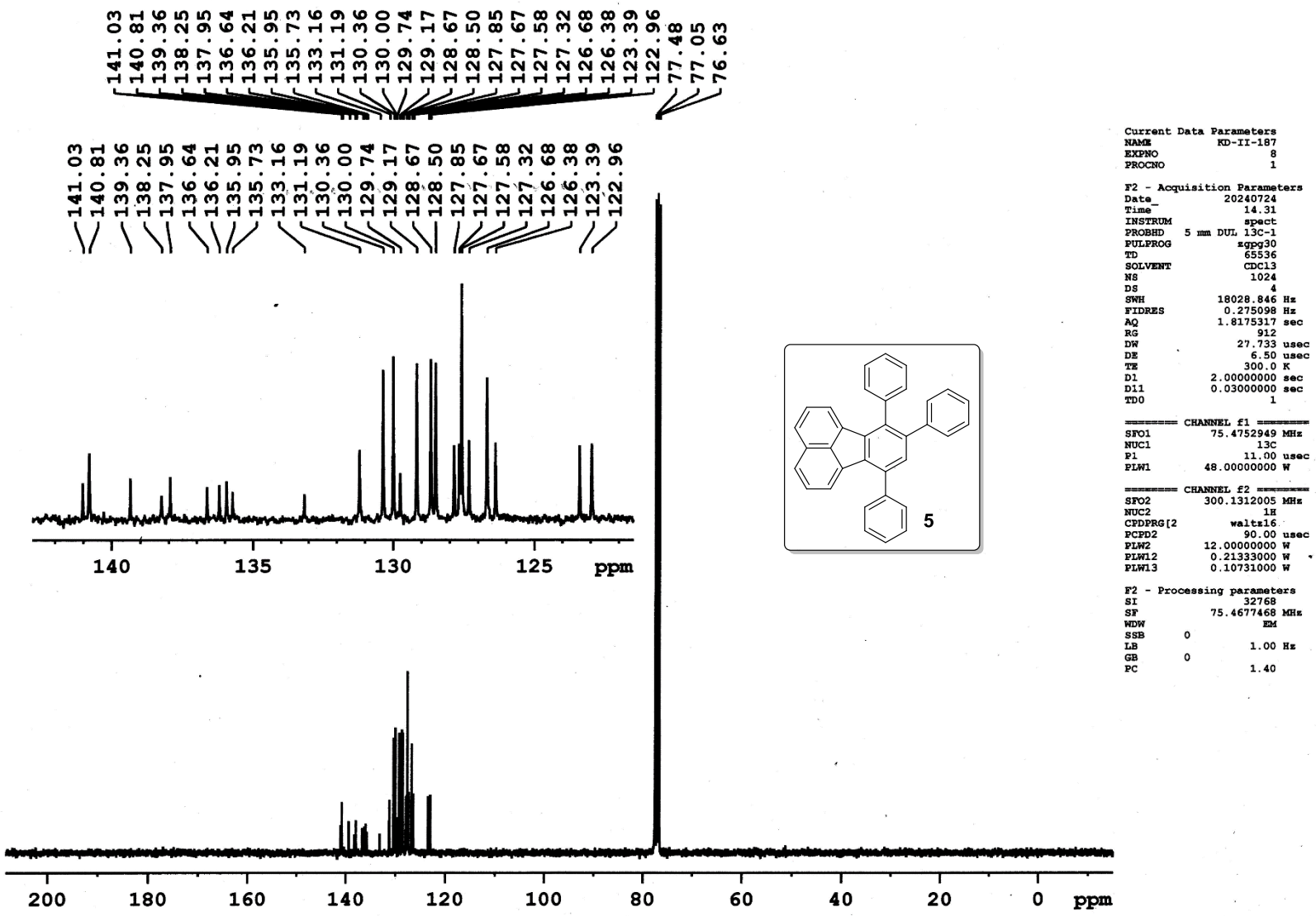
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **3j**

6. ^{1}H, ^{13}C {^{1}H} NMR & HRMS (selected) Spectra of compounds **4, 5** and **3f**



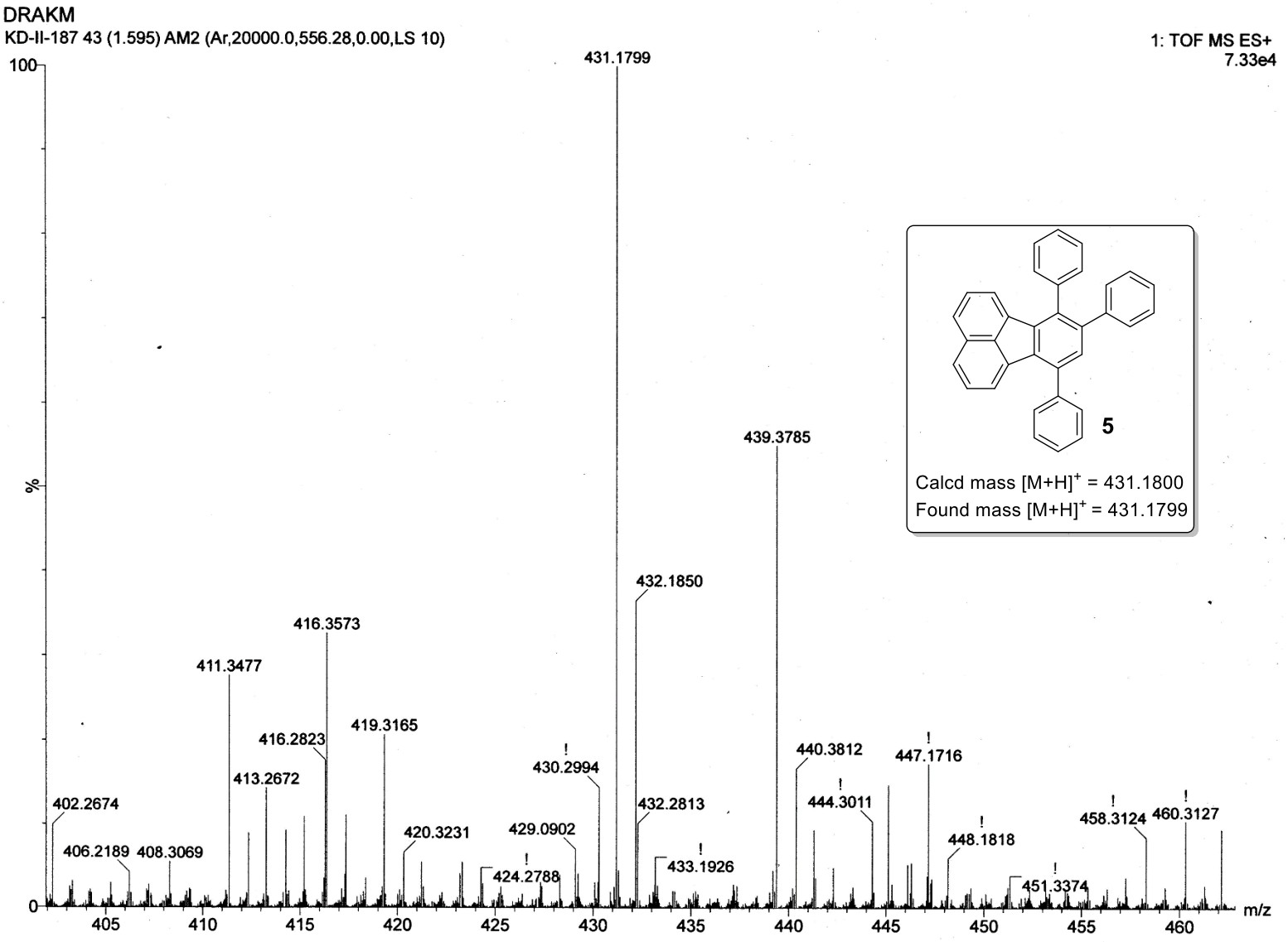

```
Current Data Parameters
NAME                  KD-II-187
EXPNO                         7
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240724
Time                      14.18
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                  6009.615 Hz
FIDRES               0.091699 Hz
AQ                   5.4525952 sec
RG                          256
DW                     83.200 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1            300.1318534 MHz
NUC1                       1H
P1                     12.00 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                       65536
SF              300.1300000 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **5**

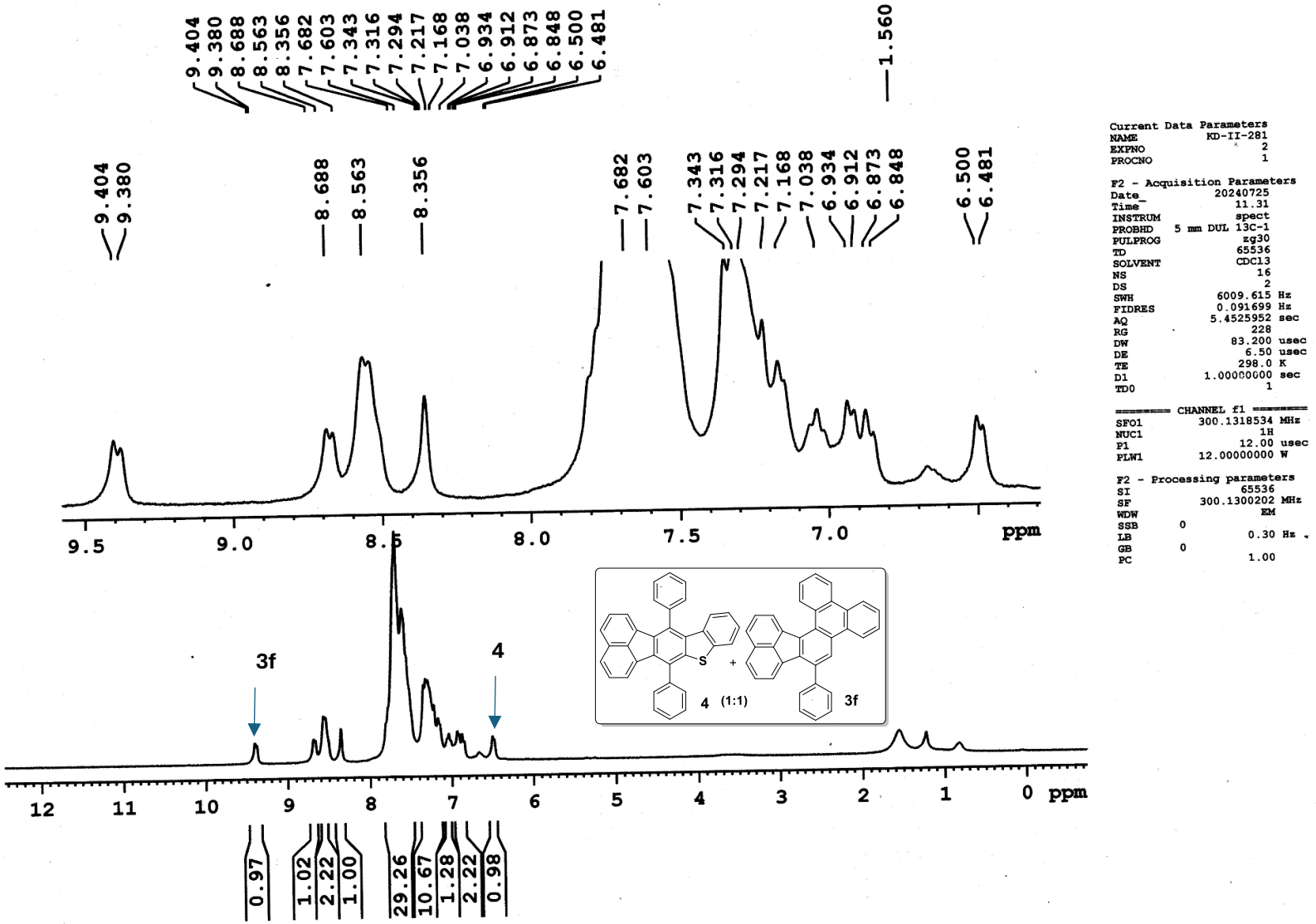



```
Current Data Parameters
NAME                  KD-II-187
EXPNO                         8
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240724
Time                      14.31
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                          1024
DS                             4
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                          912
DW                       27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                 48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W
PLW13                 0.10731000 W

F2 - Processing parameters
SI                         32768
SF                   75.4677468 MHz
WDW                           EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **5**



HRMS spectrum of compound **5**



Current Data Parameters
NAME KD-II-281
EXPNO 2
PROCNO 1

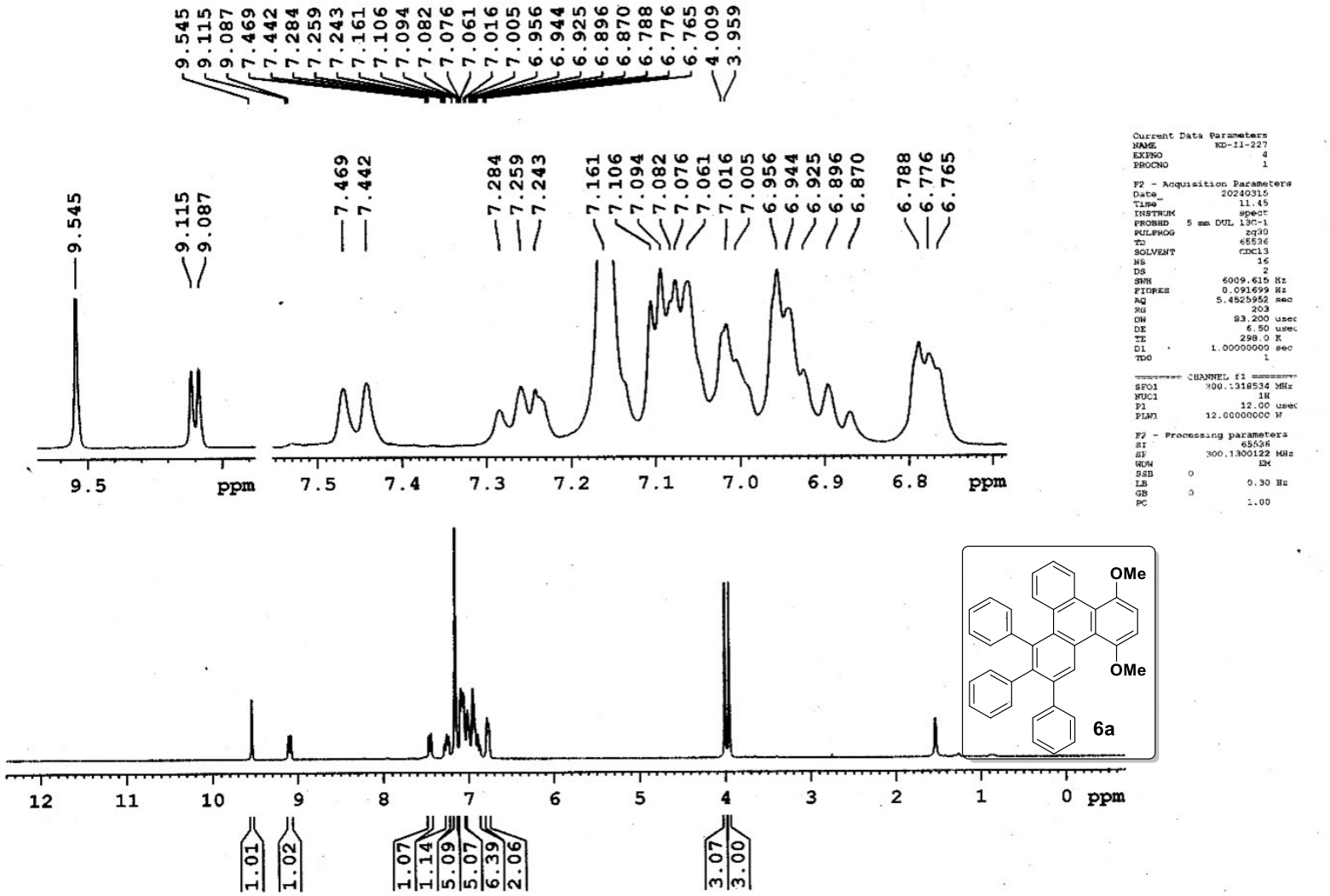
F2 - Acquisition Parameters
Date_ 20240725
Time 11.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 228
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 12.00 usec
PLW1 12.00000000 W

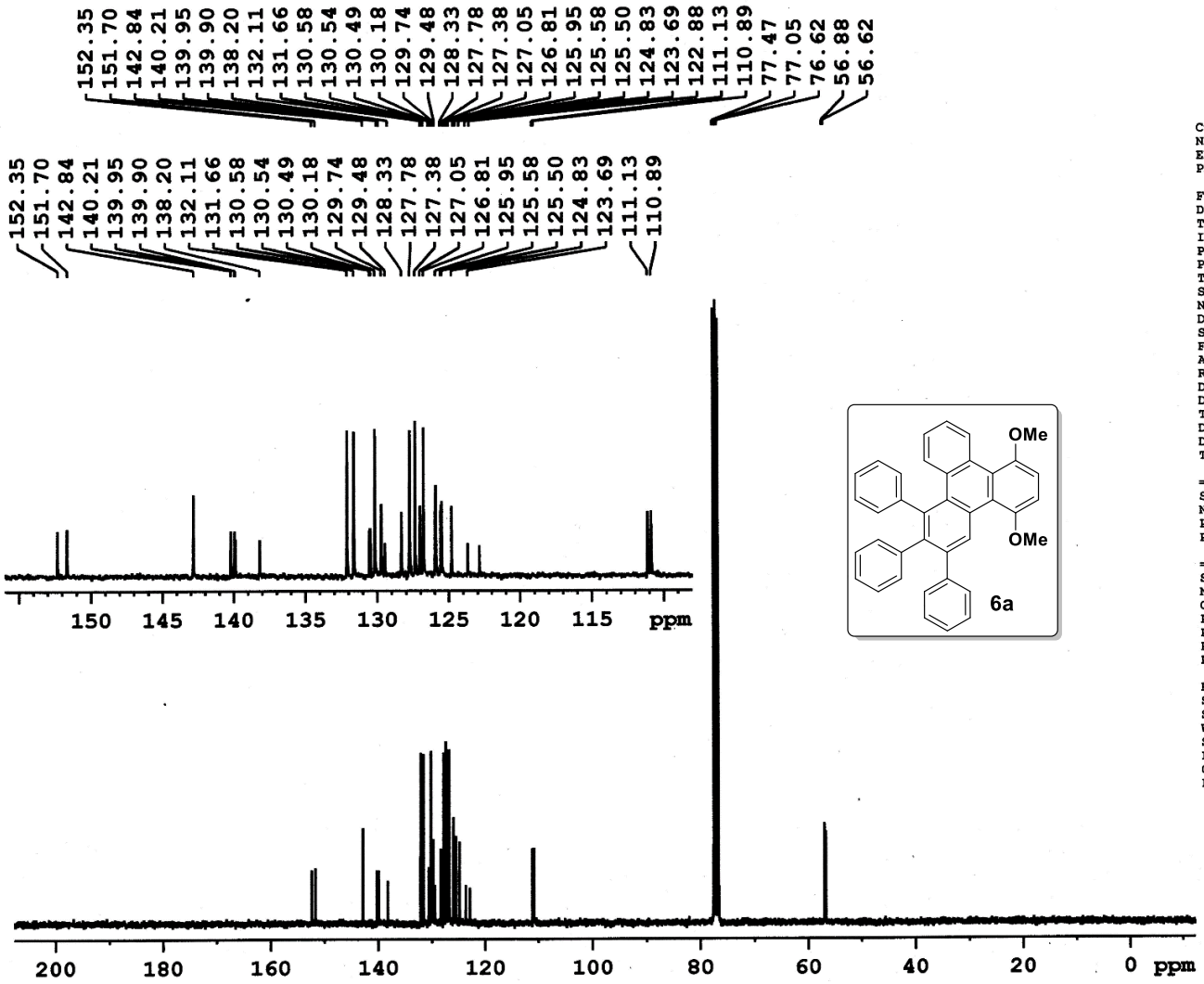
F2 - Processing parameters
SI 65536
SF 300.1300202 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of crude compounds of **4** & **3f**

7. ^{1}H, ^{13}C {^{1}H}, DEPT 135 (selected) NMR & HRMS (selected) spectra of compounds **6a-e** and **7a-f**

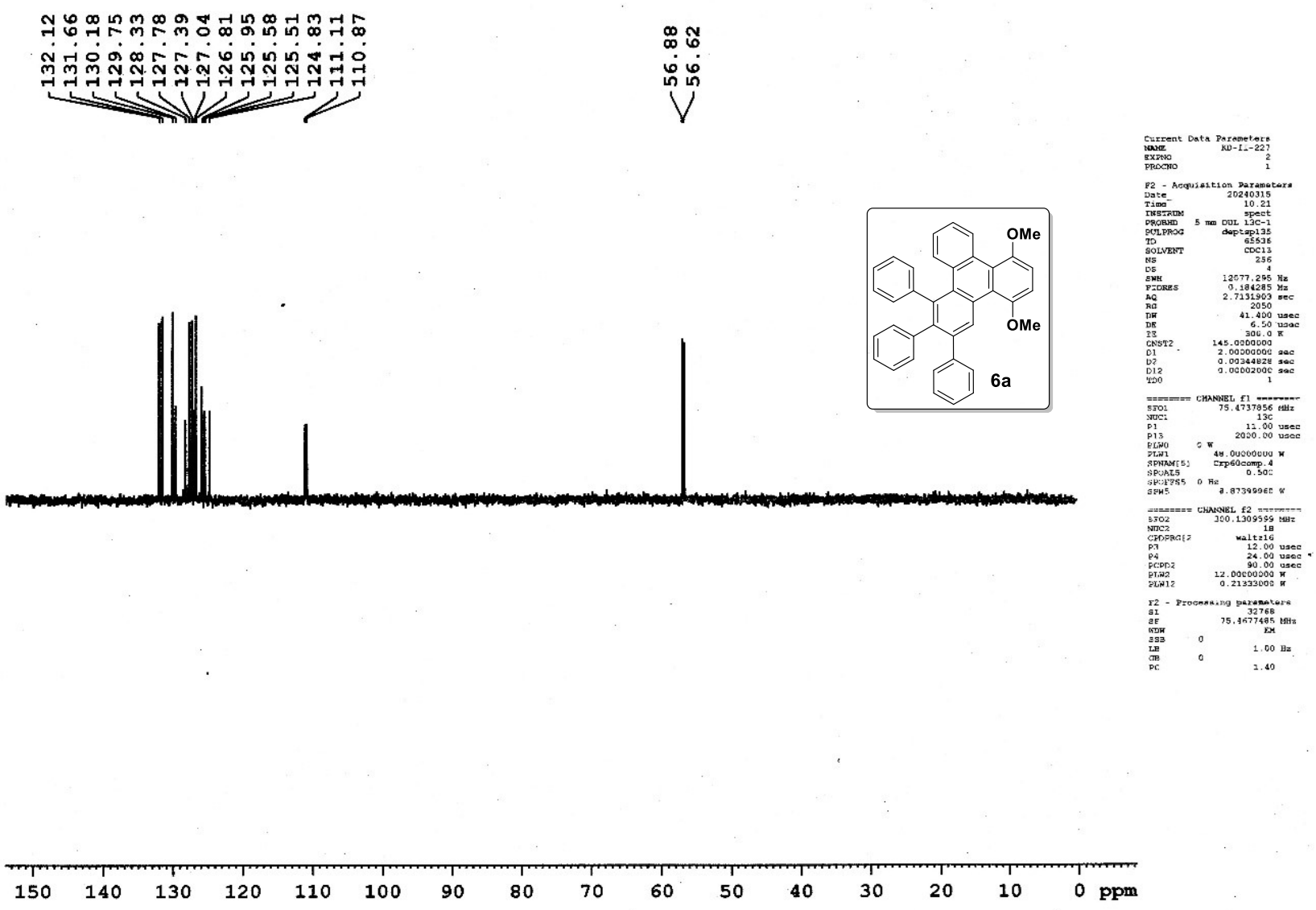


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **6a**




```
Current Data Parameters
NAME                  KD-II-227
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240315
Time                      11.43
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                         1290
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                       13C
P1                    11.00 usec
PLW1               48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                  90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB                   0
LB                     1.00 Hz
GB                    0
PC                         1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **6a**



Current Data Parameters
NAME KD-IL-227
EXPNO 2
PROCNO 1

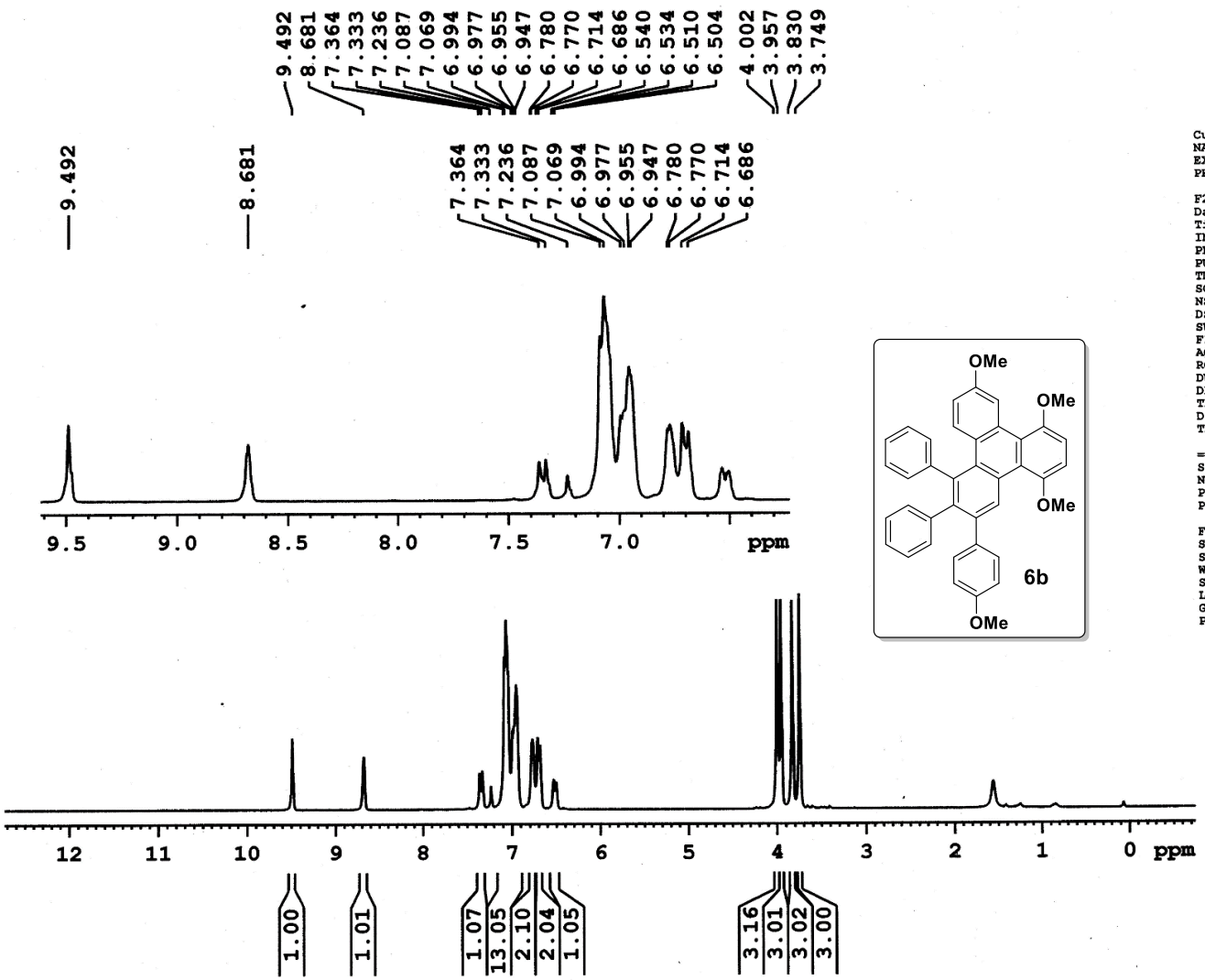
F2 - Acquisition Parameters
Date_ 20240315
Time 10.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 12077.295 Hz
FIDRES 0.184285 Hz
AQ 2.7131903 sec
RG 2050
DW 41.400 usec
DE 6.50 usec
TE 304.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4737856 MHz
NUC1 13C
P1 11.00 usec
P13 2020.00 usec
PLW0 0 W
PLW1 48.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.87399960 W

======== CHANNEL f2 ========
SFO2 300.1309599 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 12.00 usec
P4 24.00 usec
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.21333000 W

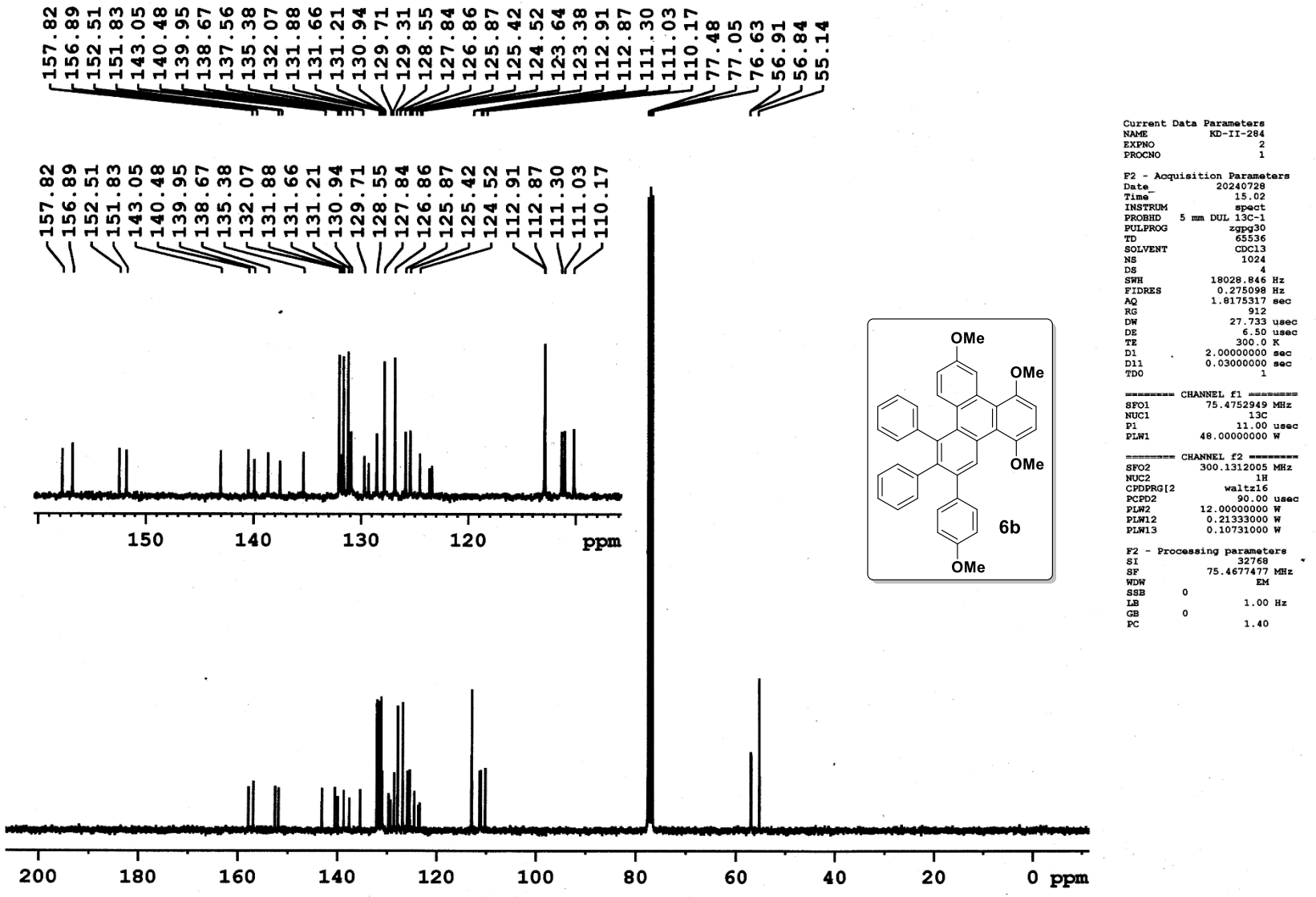
F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **6a**



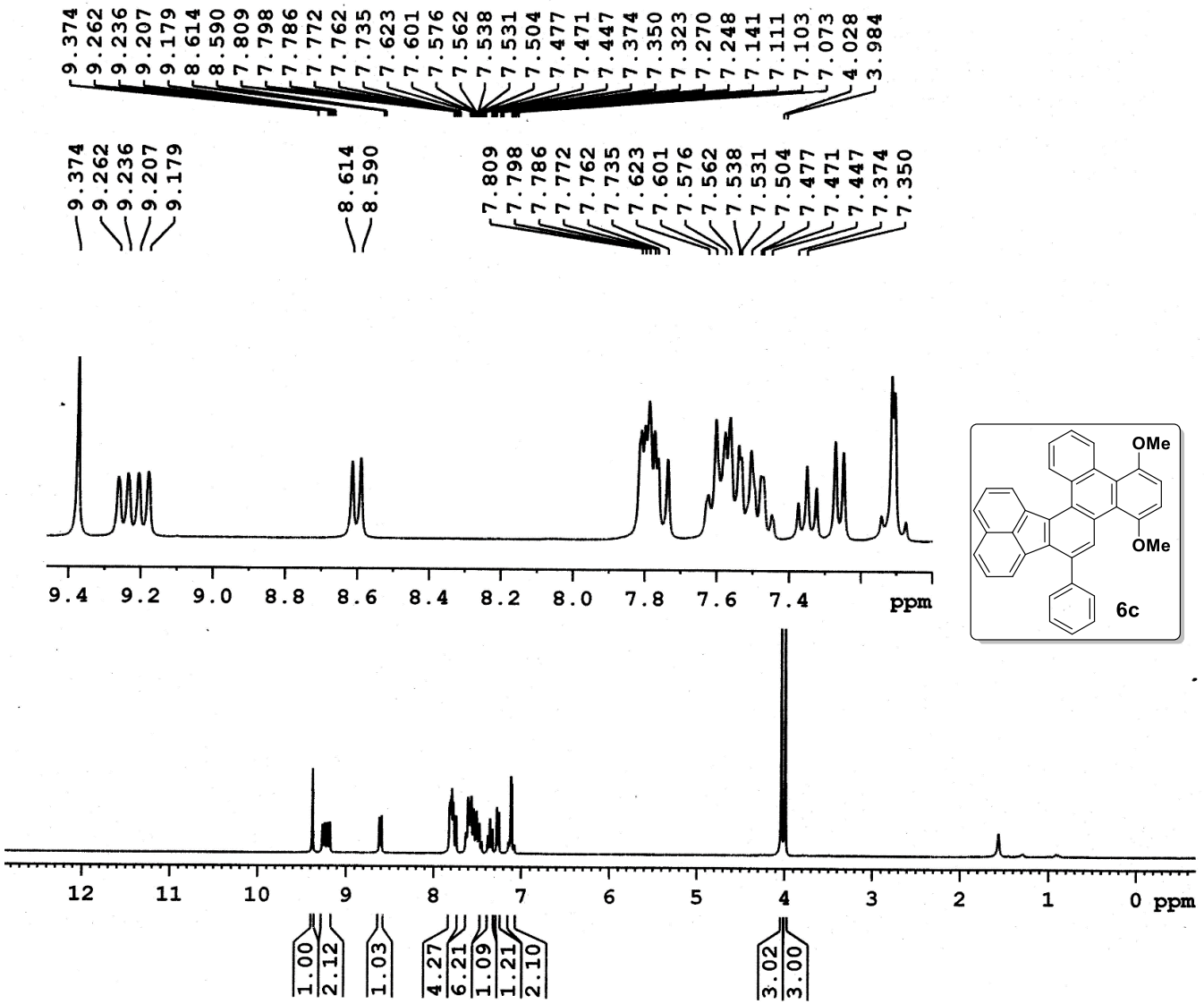

```
Current Data Parameters
NAME                  KD-II-284
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_              20240728
Time                   16.27
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6009.615 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                       181
DW                    83.200 usec
DE                     6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                        1

========== CHANNEL f1 ==========
SFO1               300.1318534 MHz
NUC1                       1H
P1                     12.00 usec
PLW1              12.00000000 W

F2 - Processing parameters
SI                     65536
SF                 300.1300145 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **6b**



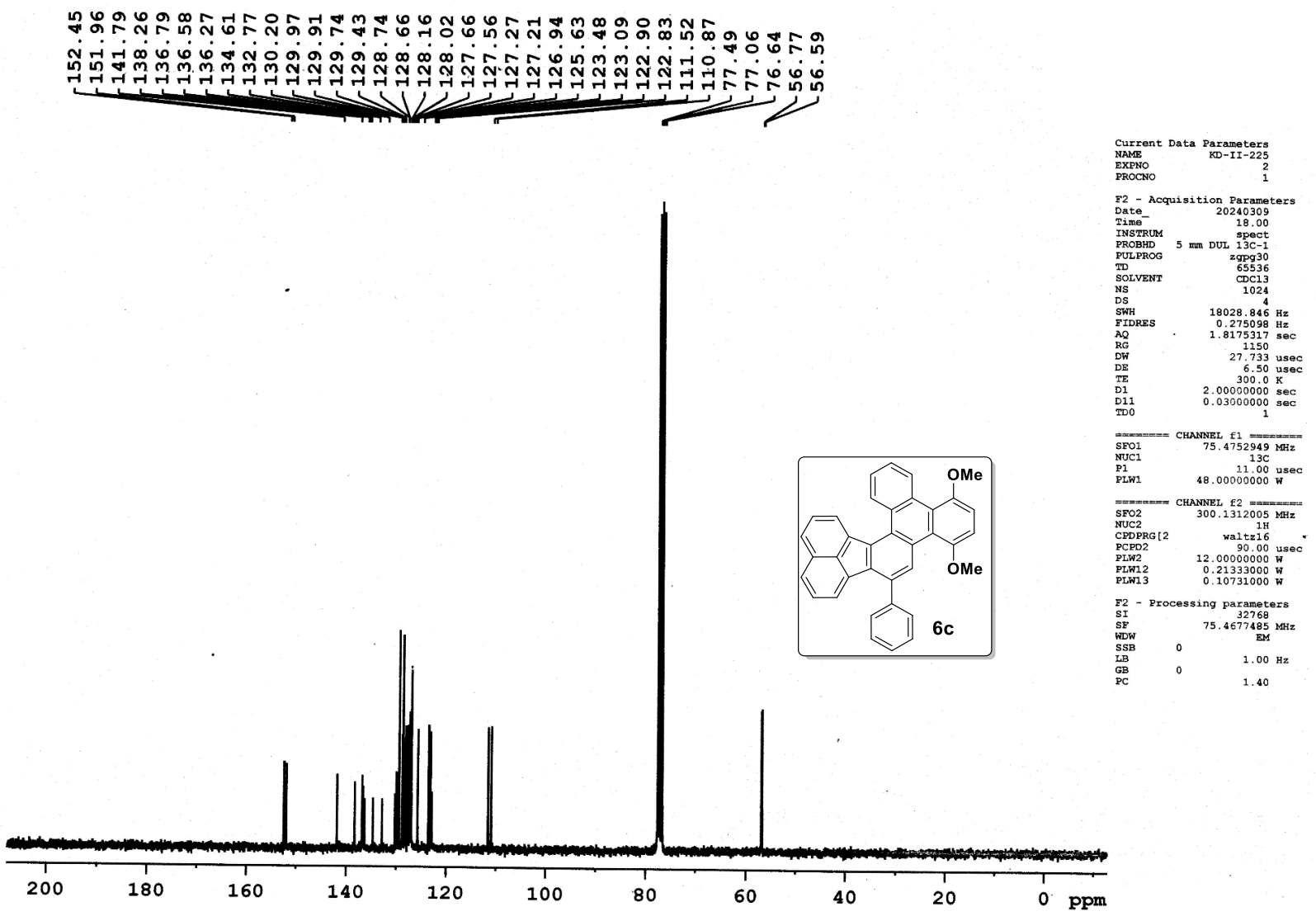

```
Current Data Parameters
NAME                  KD-II-284
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240728
Time                      15.02
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                   18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                          912
DW                       27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                 48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W
PLW13                 0.10731000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677477 MHz
WDW                          EM
SSB                   0
LB                        1.00 Hz
GB                    0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **6b**



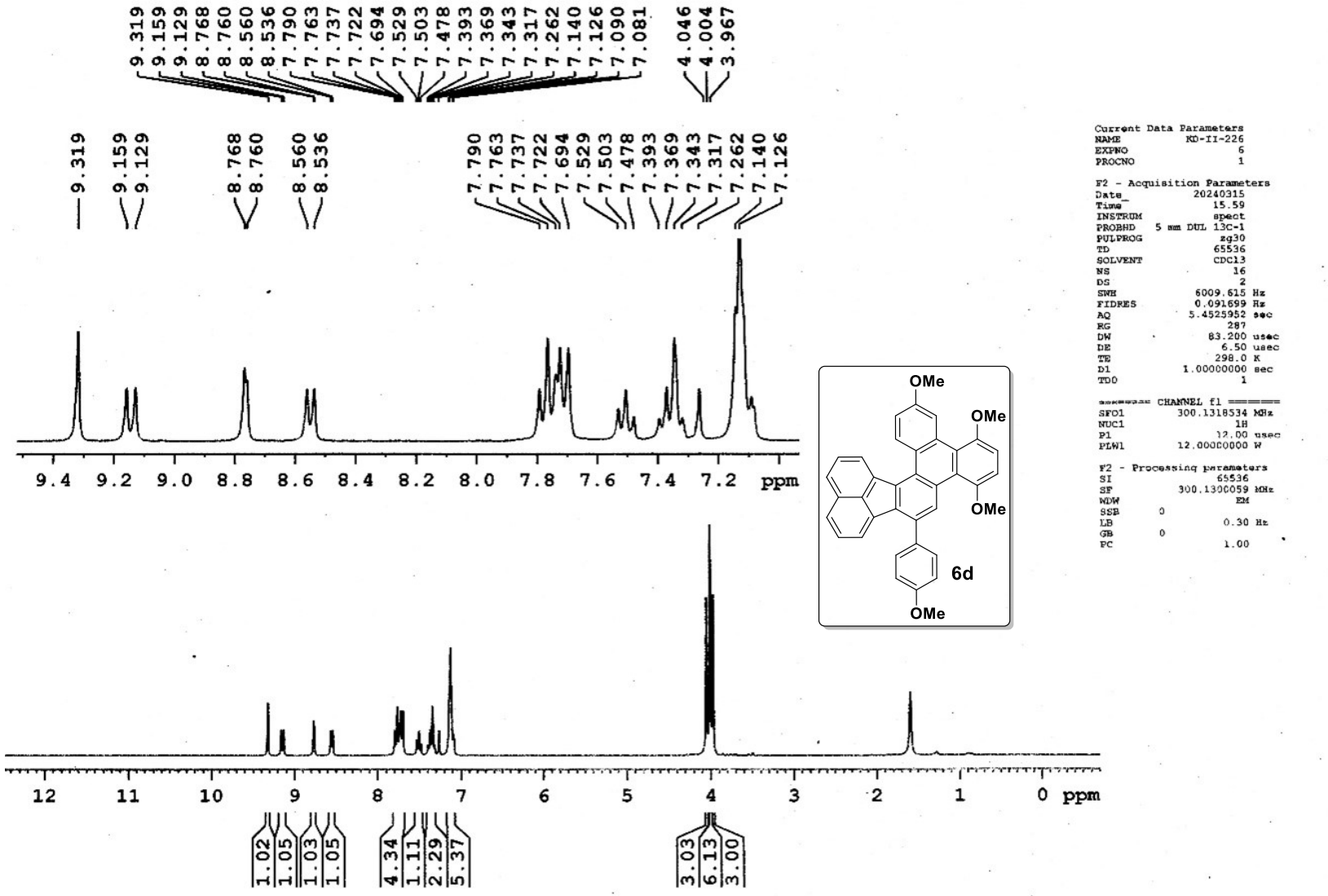

```
Current Data Parameters
NAME                  KD-II-225
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_              20240309
Time                      19.26
INSTRUM                   spect
PROBHD  5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                6009.615 Hz
FIDRES             0.091699 Hz
AQ                 5.4525952 sec
RG                          203
DW                    83.200 usec
DE                     6.50 usec
TE                     298.0 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1             300.1318534 MHz
NUC1                       1H
P1                      12.00 usec
PLW1            12.00000000 W

F2 - Processing parameters
SI                       65536
SF               300.1300100 MHz
WDW                        EM
SSB                   0
LB                      0.30 Hz
GB                    0
PC                      1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **6c**

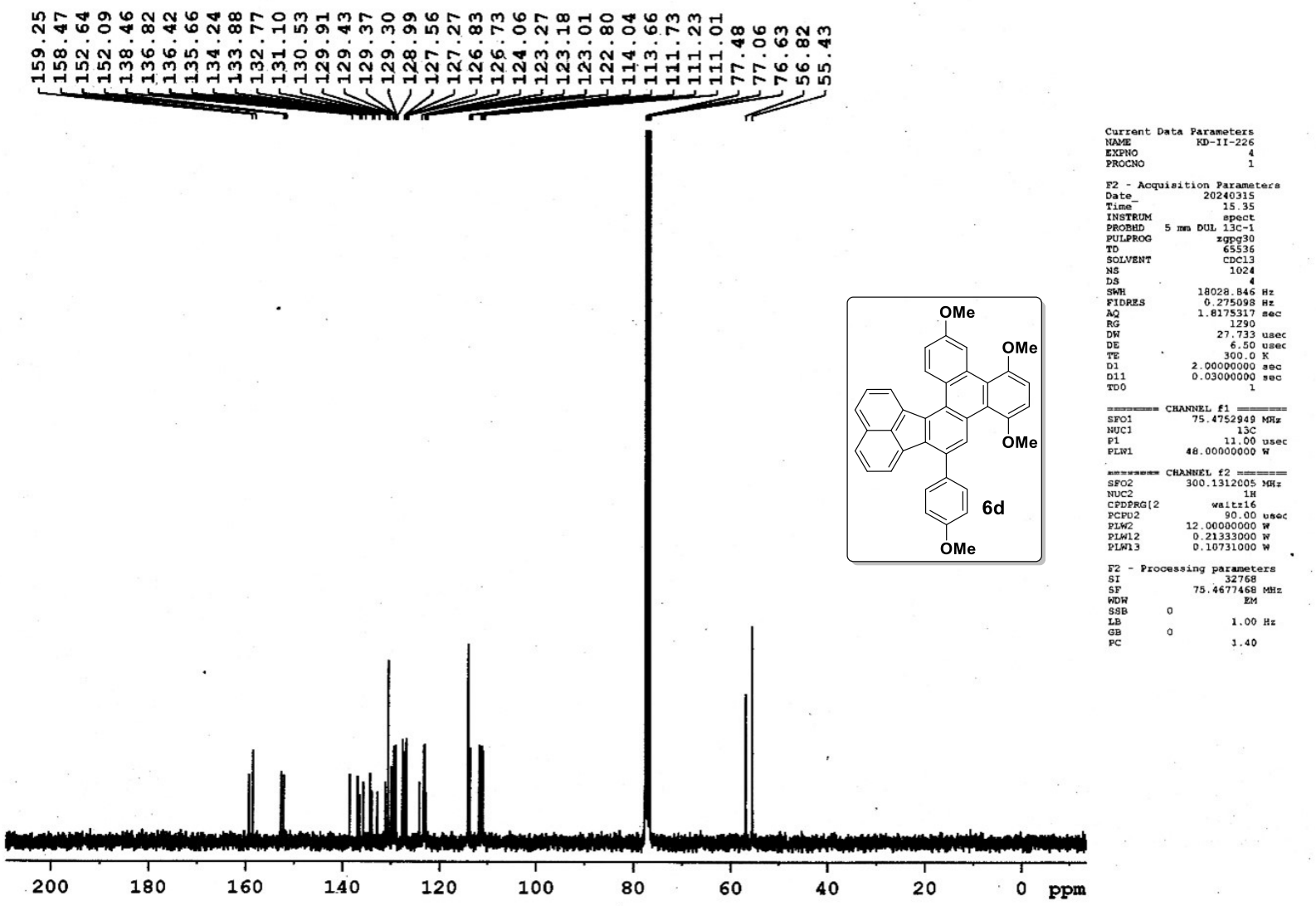



```
Current Data Parameters
NAME                  KD-II-225
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240309
Time                      18.00
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                         1150
DW                     27.733 usec
DE                      6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1               48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                      1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **6c**

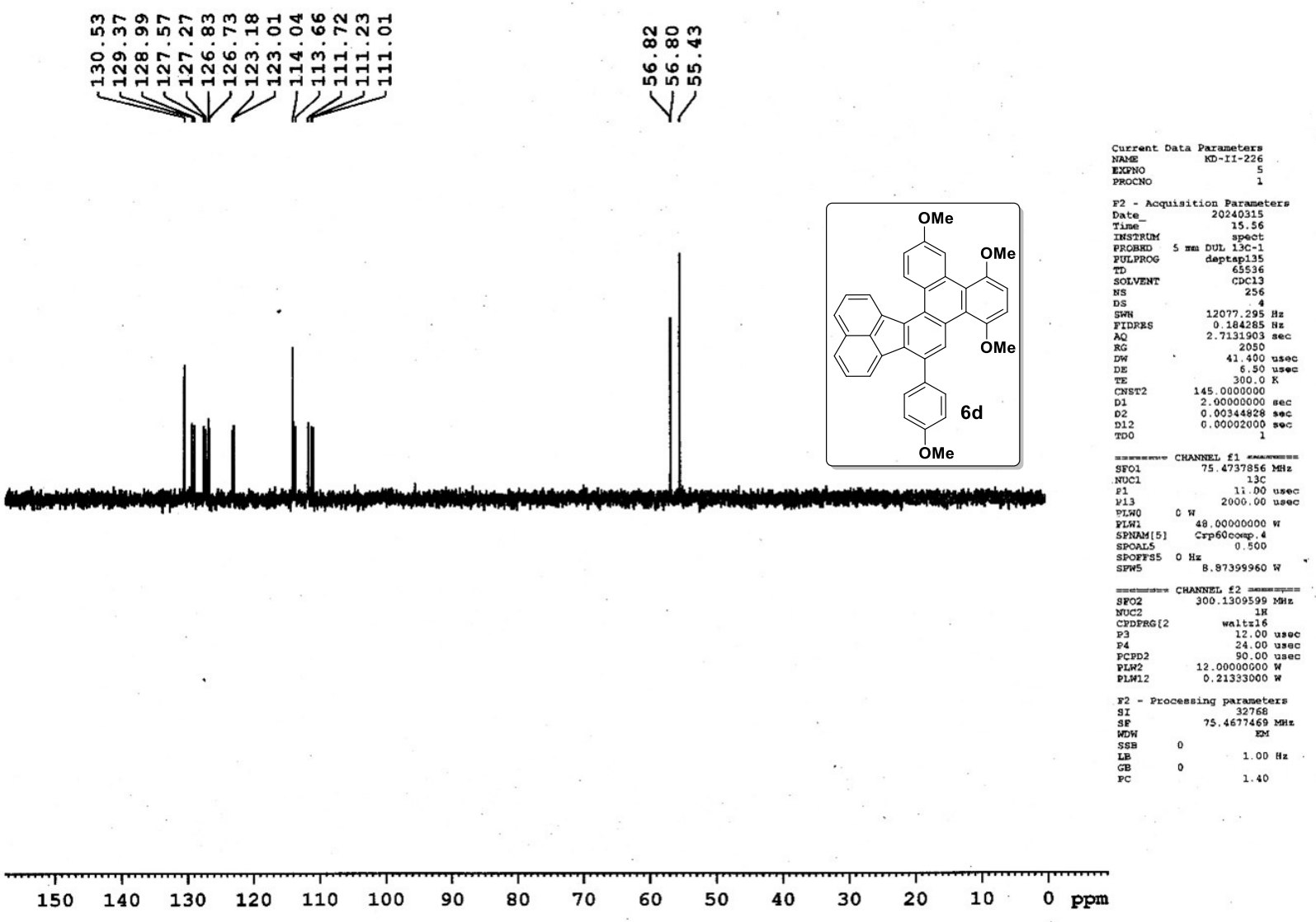


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **6d**



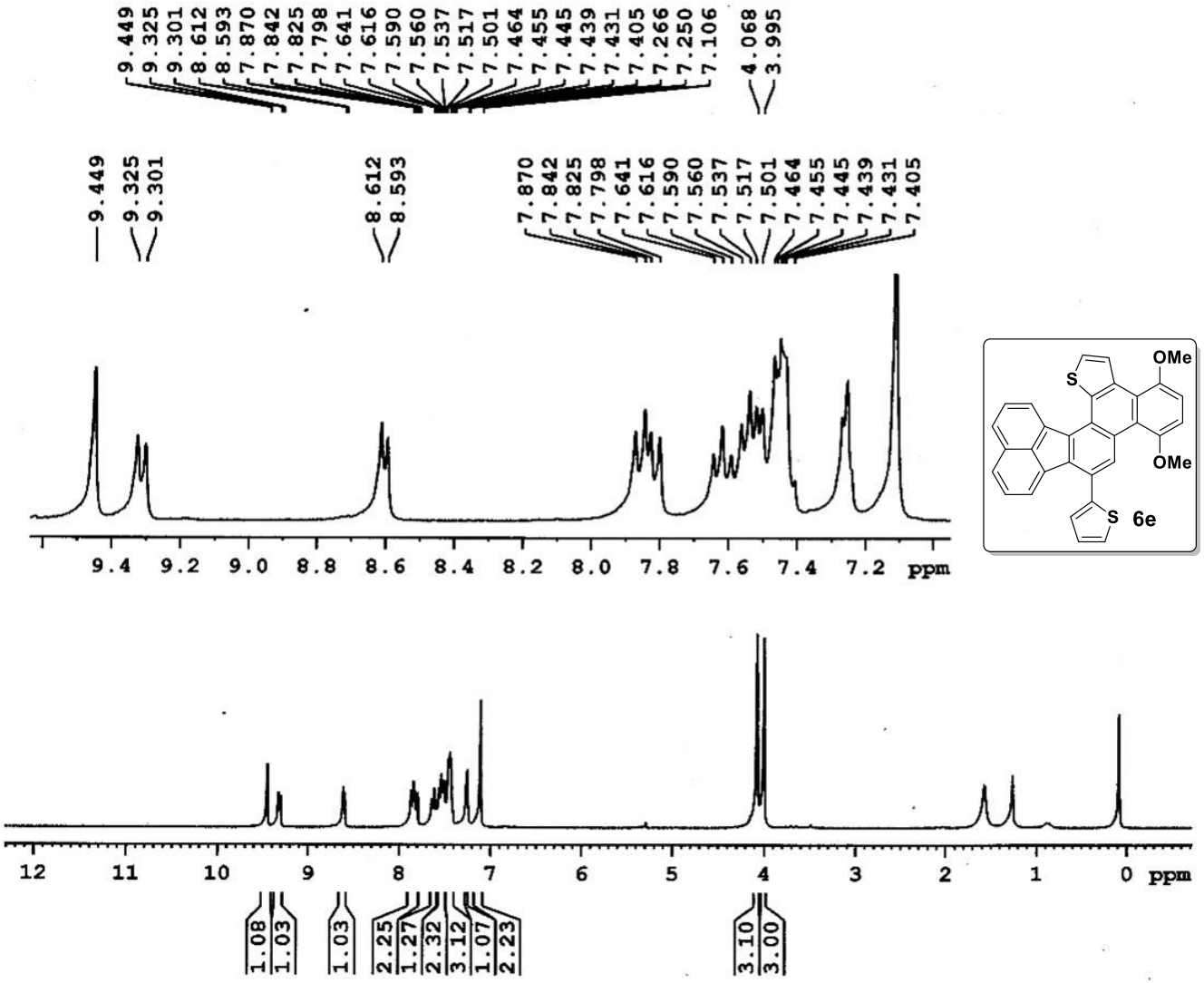

```
Current Data Parameters
NAME                  KD-II-226
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240315
Time                      15.35
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  18028.846 Hz
FIDRES                6.275098 Hz
AQ                    1.8175317 sec
RG                         1290
DW                       27.733 usec
DE                         6.50 usec
TE                        300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                 48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W
PLW13                 0.10731000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677468 MHz
WDW                          EM
SSB                   0
LB                        1.00 Hz
GB                    0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **6d**



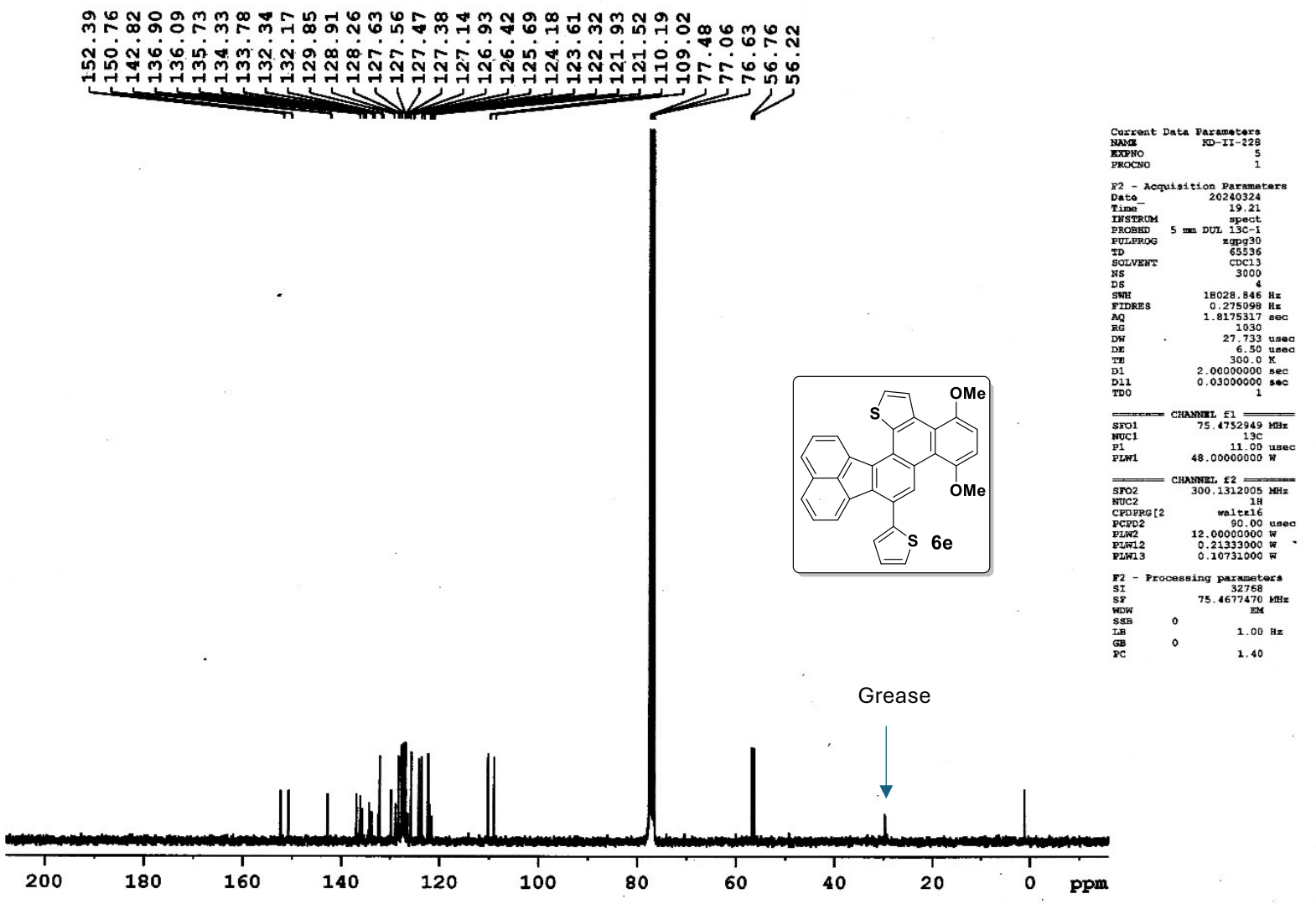

```
Current Data Parameters
NAME                 KD-II-226
EXPNO                        5
PROCNO                       1

F2 - Acquisition Parameters
Date_               20240315
Time                   15.56
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG             deptap135
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                 12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                         2050
DW                       41.400 usec
DE                        6.50 usec
TE                       300.0 K
CNST2               145.0000000
D1                    2.00000000 sec
D2                    0.00344828 sec
D12                   0.00002000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                75.4737856 MHz
NUC1                       13C
P1                       11.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1              48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                  0.500
SPOFFS5     0 Hz
SPW5               8.87399960 W

======== CHANNEL f2 ========
SFO2              300.1309599 MHz
NUC2                       1H
CPDPRG[2                waltz16
P3                       12.00 usec
P4                       24.00 usec
PCPD2                    90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677469 MHz
WDW                           EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **6d**



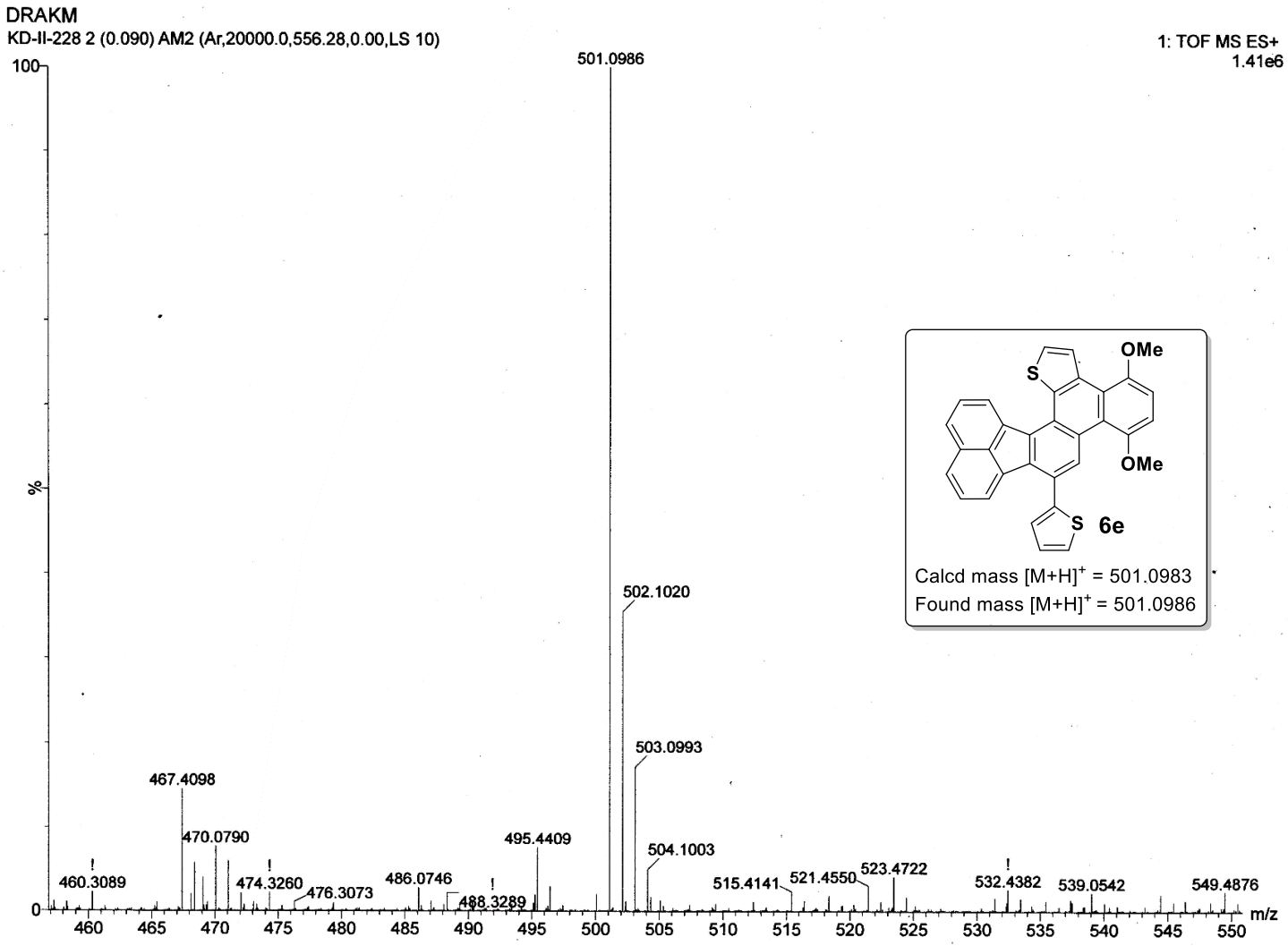

```
Current Data Parameters
NAME                  KD-II-228
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240323
Time                      14.19
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.091699 Hz
AQ                     5.4525952 sec
RG                          287
DW                       83.200 usec
DE                        6.50 usec
TE                       298.0 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 300.1318534 MHz
NUC1                         1H
P1                        12.00 usec
PLW1                 12.00000000 W

F2 - Processing parameters
SI                           65536
SF                   300.1300091 MHz
WDW                             EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **6e**

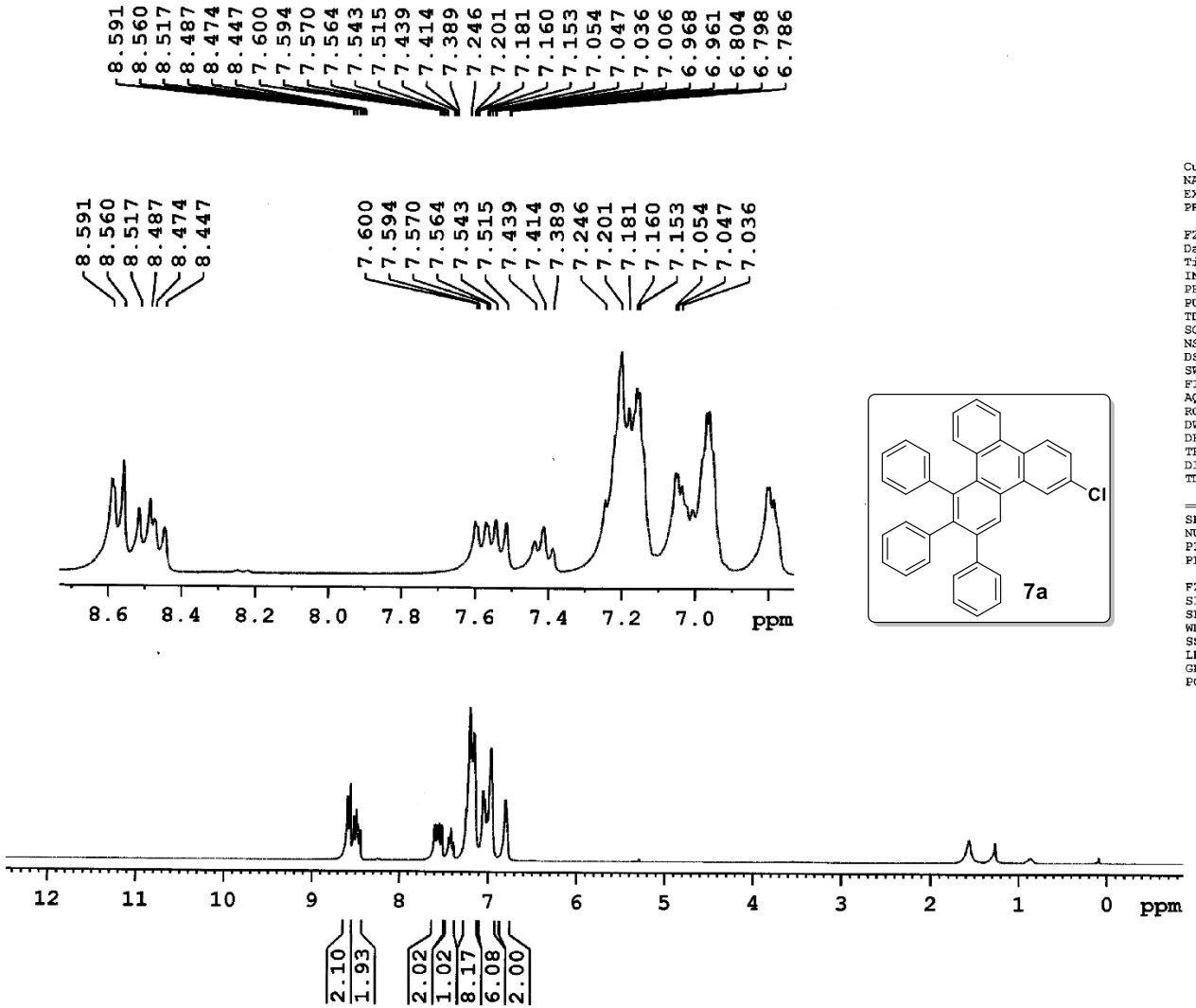



```
Current Data Parameters
NAME            KD-II-228
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240324
Time                19.21
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3000
DS                     4
SWH            18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175317 sec
RG                  1030
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                  11.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W
PLW13         0.10731000 W

F2 - Processing parameters
SI                  32768
SF            75.4677470 MHz
WDW                    EM
SSB          0
LB                  1.00 Hz
GB           0
PC                  1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **6e**



HRMS spectrum of compound **6e**



Current Data Parameters

NAME	KD-II-195-CC
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20241011
Time	16.48
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4525952 sec
RG	203
DW	83.200 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1

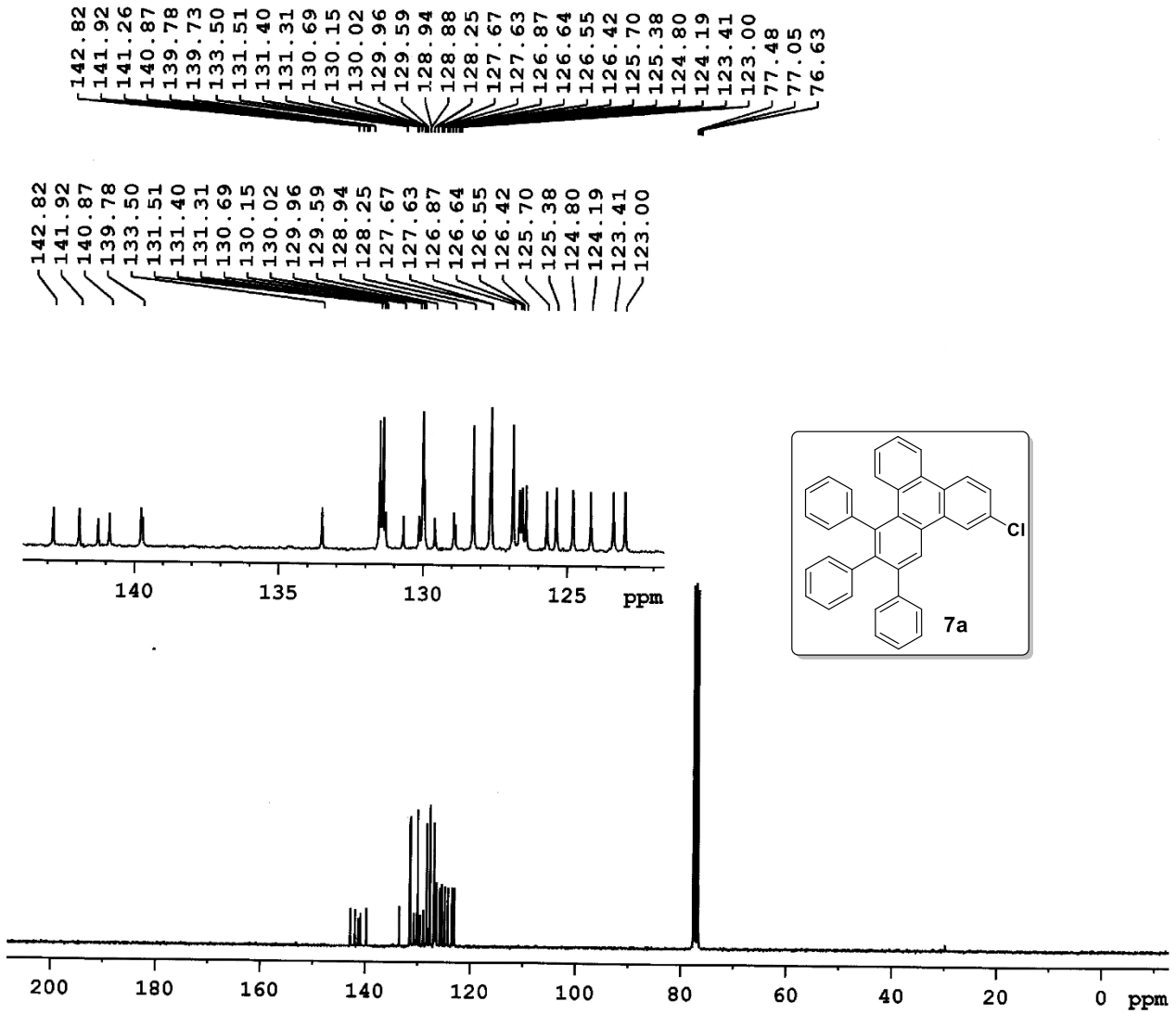
======== CHANNEL f1 ========

SFO1	300.1318534 MHz
NUC1	1H
P1	12.00 usec
PLW1	12.00000000 W

F2 - Processing parameters

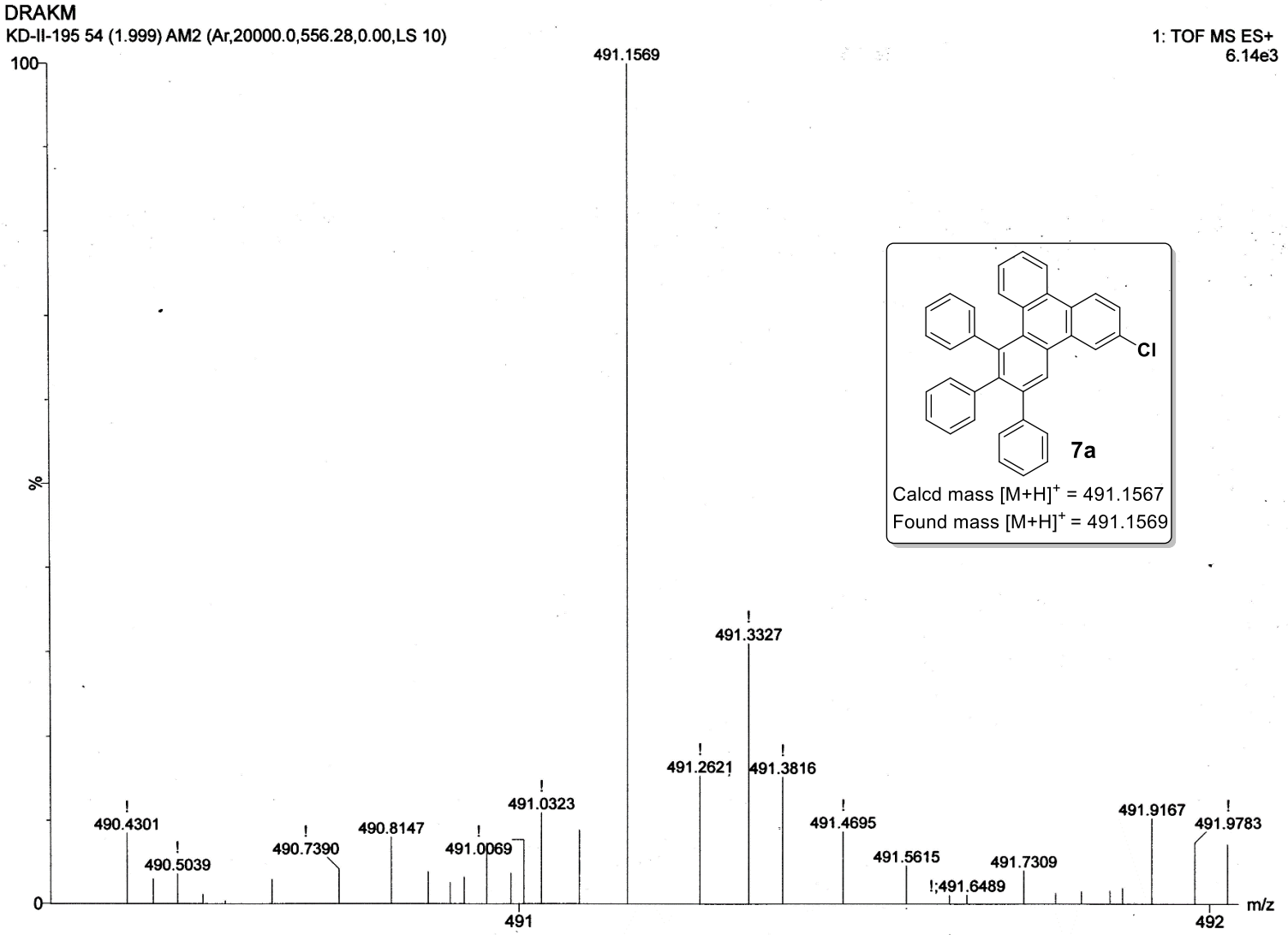
SI	65536
SF	300.1300105 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7a**

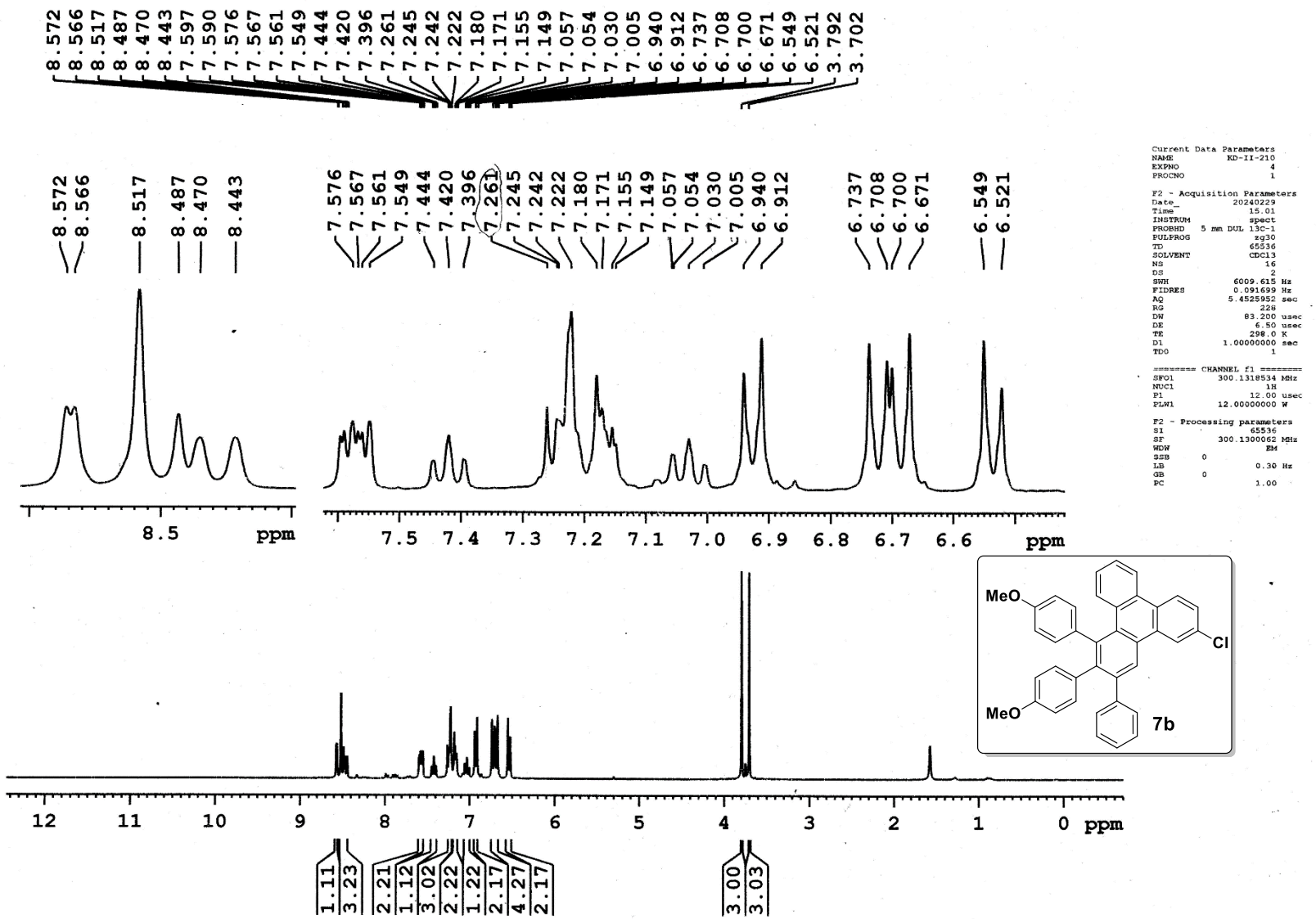



```
Current Data Parameters
NAME        KD-II-195-CC
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241011
Time              16.45
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                3000
DS                   4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175317 sec
RG                 912
DW              27.733 usec
DE               6.50 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1               13C
P1               11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI                32768
SF           75.4677479 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

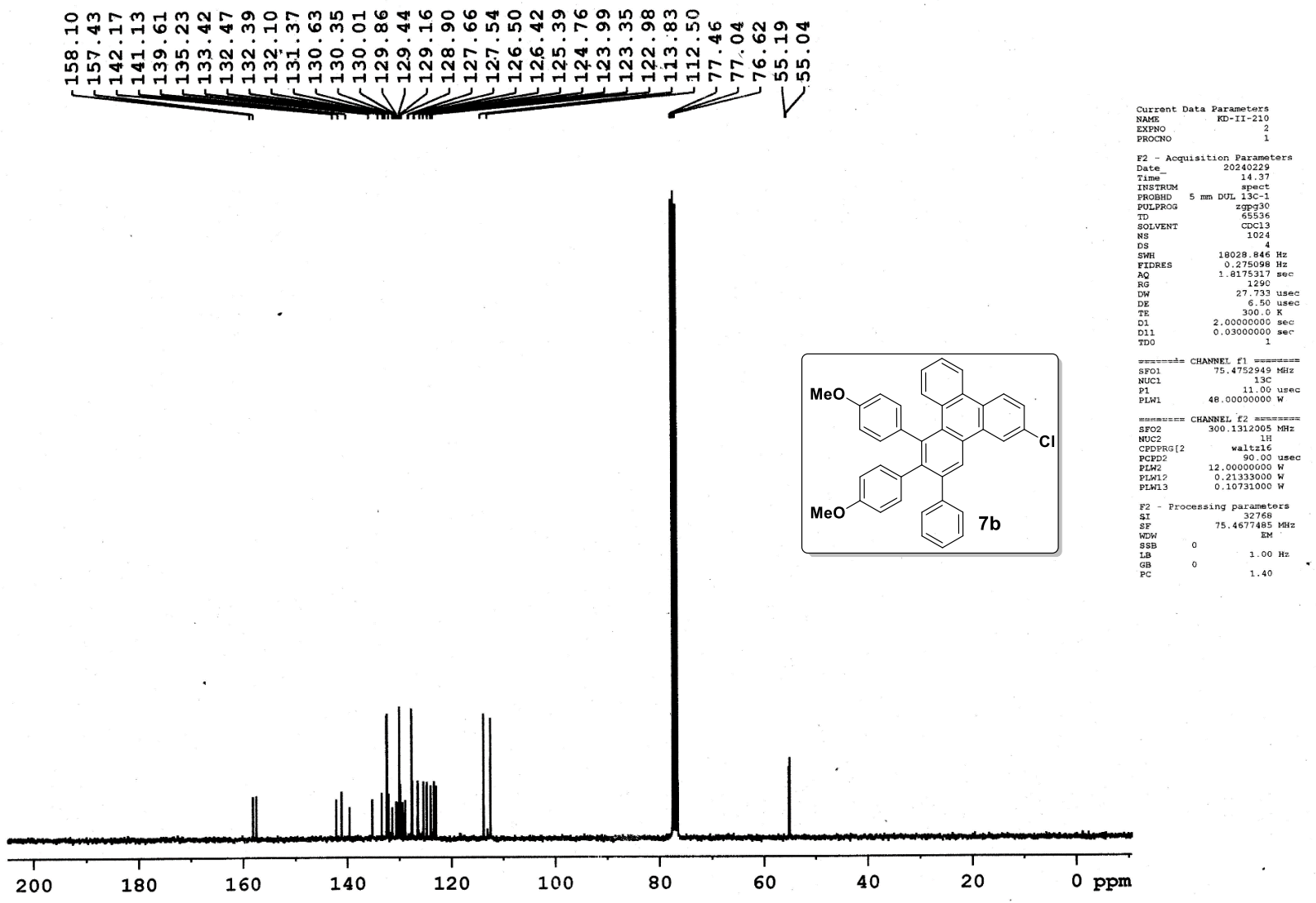
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7a**



HRMS spectrum of compound **7a**

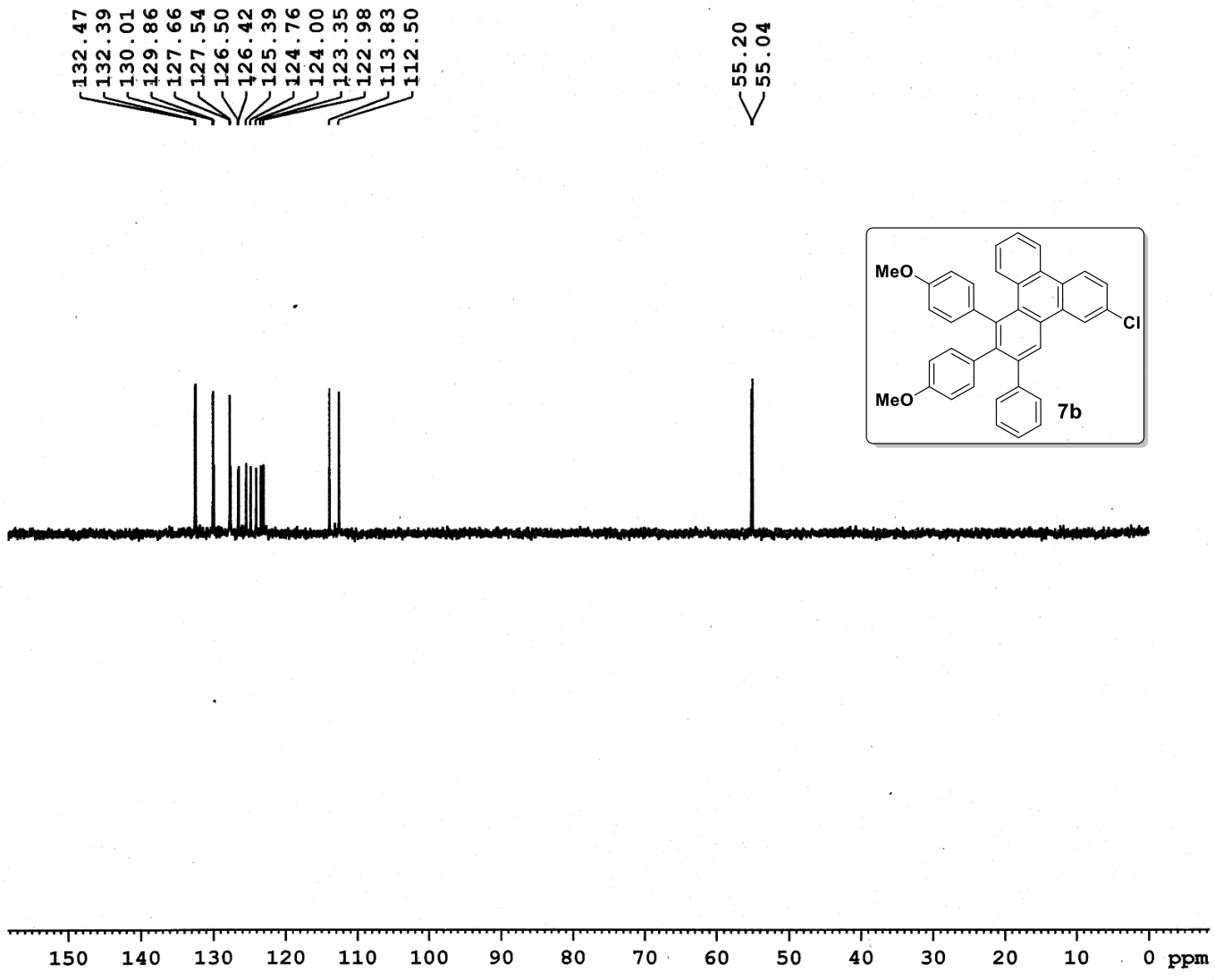


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7b**



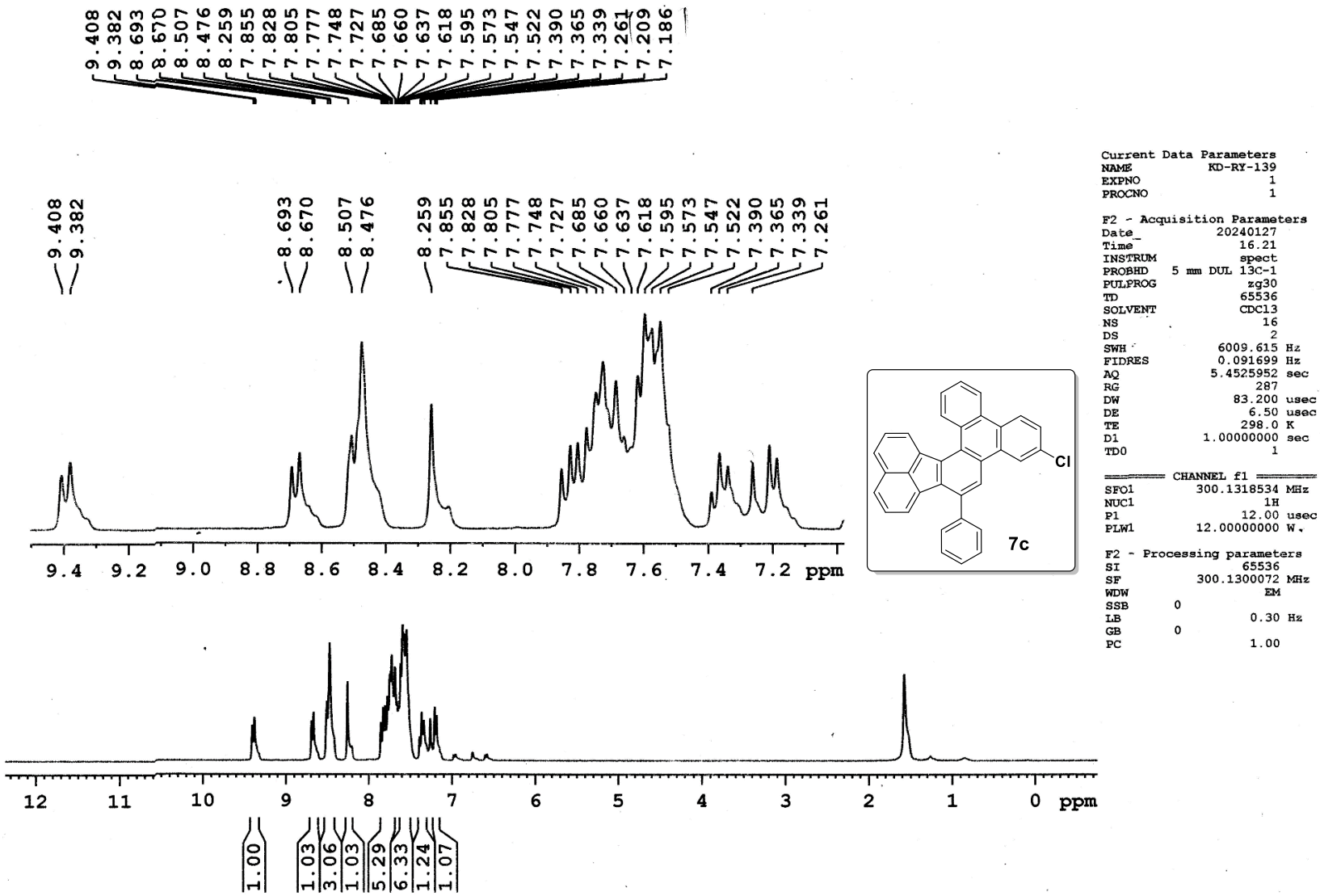

```
Current Data Parameters
NAME                  KD-II-210
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240229
Time                      14.37
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                         1290
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                75.4677485 MHz
WDW                         EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7b**

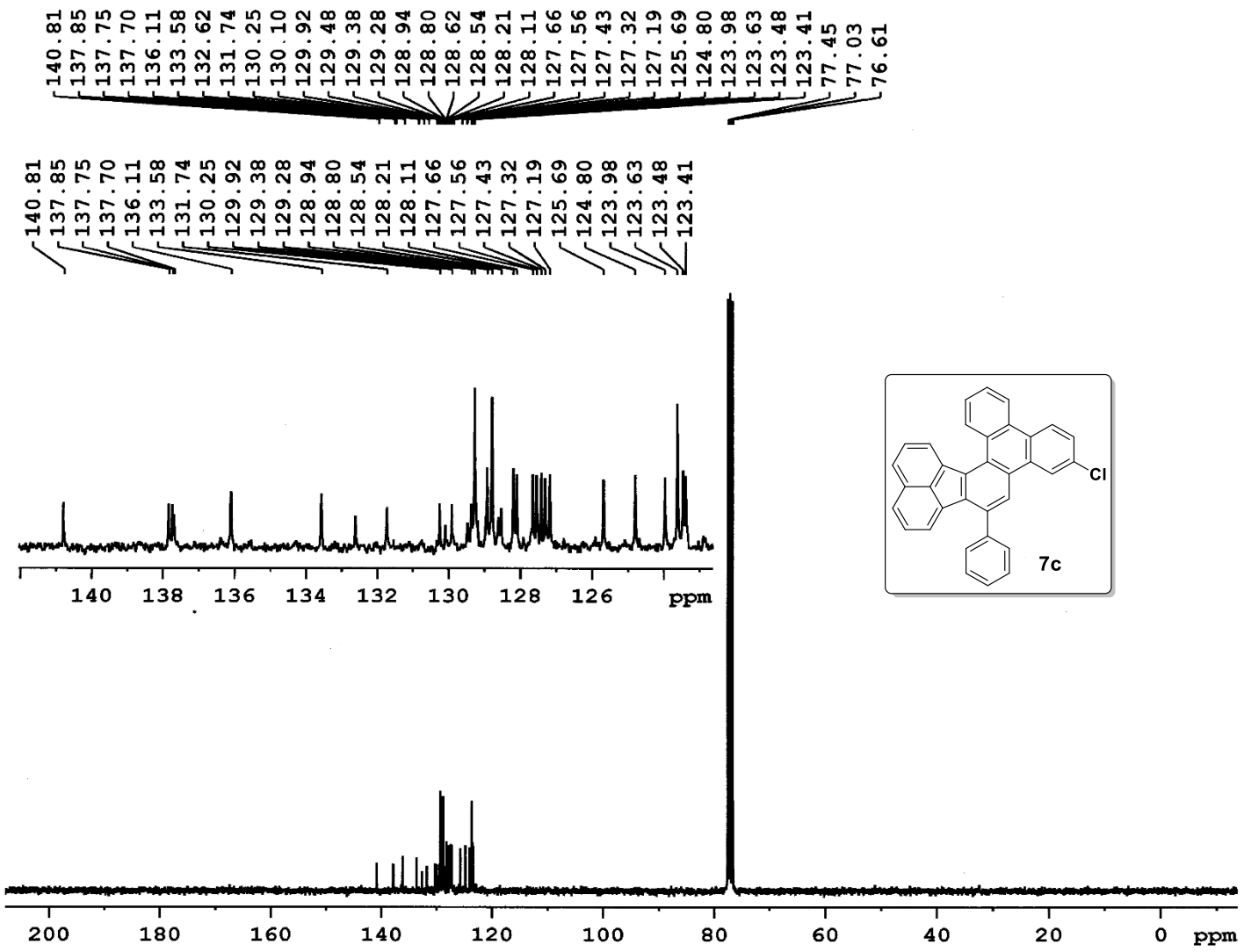



```
Current Data Parameters
NAME                  KD-II-210
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240229
Time_                     14.58
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG              deptsp135
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                         2050
DW                       41.400 usec
DE                        6.50 usec
TE                       300.0 K
CNST2                145.0000000
D1                     2.00000000 sec
D2                     0.00344828 sec
D12                    0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4737856 MHz
NUC1                        13C
P1                         11.00 usec
P13                      2000.00 usec
PLW0        0 W
PLW1                48.00000000 W
SPNAM[5]           Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5                 8.87399960 W

======== CHANNEL f2 ========
SFO2                 300.1309599 MHz
NUC2                        1H
CPDPRG[2                waltz16
P3                         12.00 usec
P4                         24.00 usec
PCPD2                      90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677485 MHz
WDW                          EM
SSB         0
LB                         1.00 Hz
GB          0
PC                         1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **7b**

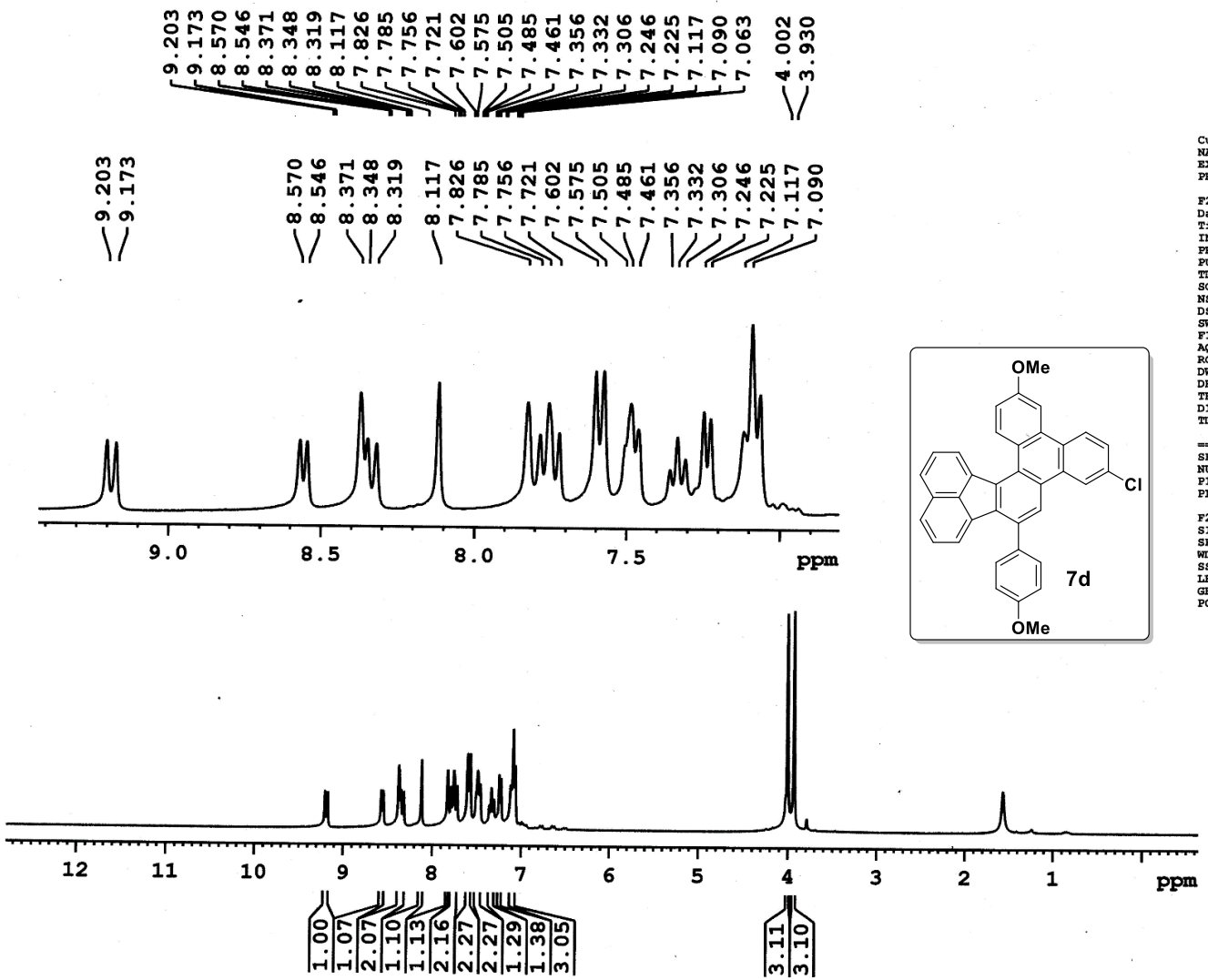


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7c**



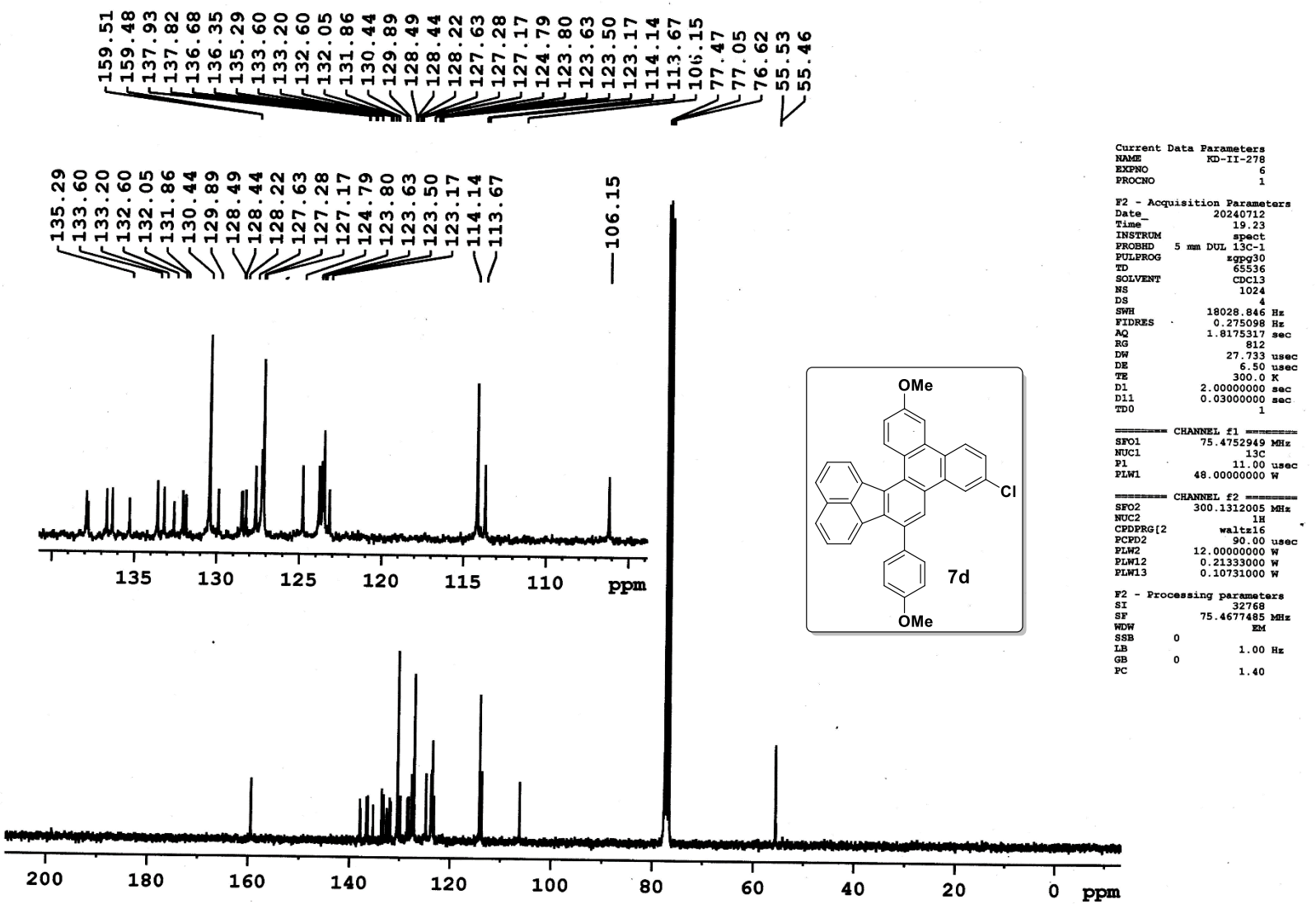

```
Current Data Parameters
NAME                  KD-RY-139
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240127
Time                      17.30
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                         1290
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                       13C
P1                     11.00 usec
PLW1               48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                  90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

F2 - Processing parameters
SI                         32768
SF                75.4677485 MHz
WDW                           EM
SSB          0
LB                      1.00 Hz
GB           0
PC                         1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7c**



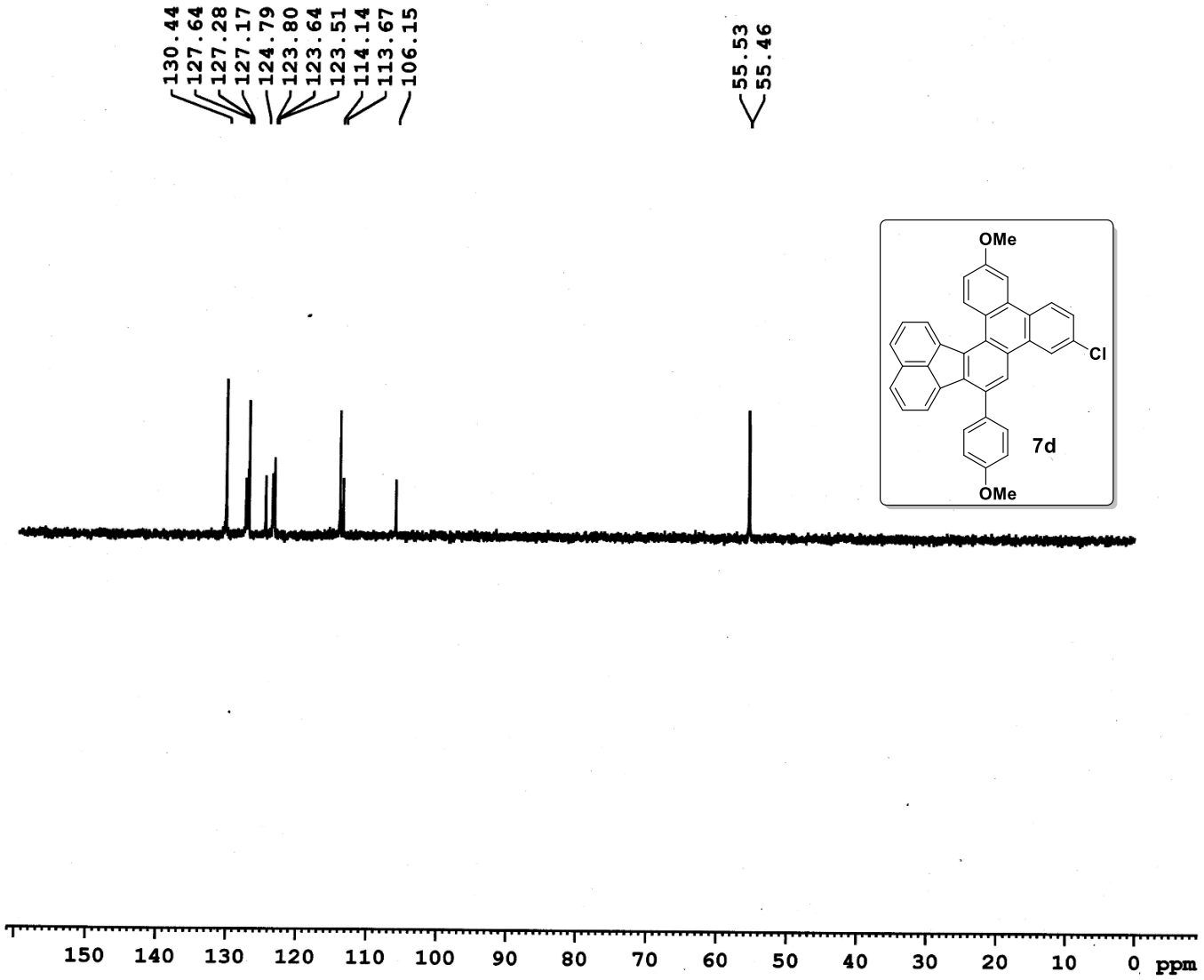

```
Current Data Parameters
NAME                  KD-II-278
EXPNO                         9
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240712
Time                      19.47
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6009.615 Hz
FIDRES                 0.091699 Hz
AQ                  5.4525952 sec
RG                         203
DW                      83.200 usec
DE                       6.50 usec
TE                      298.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                300.1318534 MHz
NUC1                        1H
P1                       12.00 usec
PLW1                12.00000000 W

F2 - Processing parameters
SI                       65536
SF                  300.1300167 MHz
WDW                         EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7d**



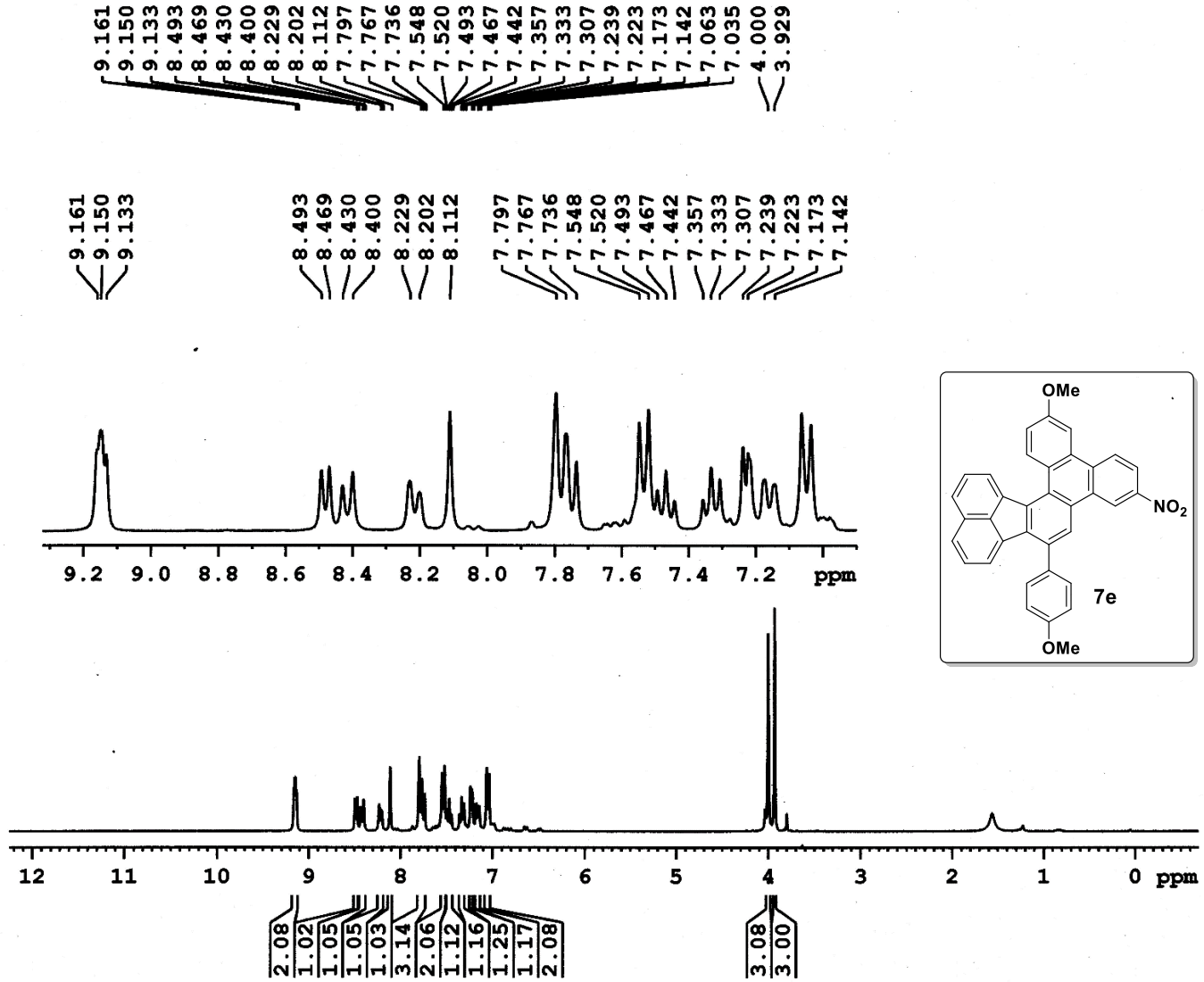

```
Current Data Parameters
NAME                  KD-II-278
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240712
Time                      19.23
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES                 0.275098 Hz
AQ                     1.8175317 sec
RG                          812
DW                        27.733 usec
DE                         6.50 usec
TE                        300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                  75.4752949 MHz
NUC1                         13C
P1                         11.00 usec
PLW1                  48.00000000 W

======== CHANNEL f2 ========
SFO2                  300.1312005 MHz
NUC2                         1H
CPDPRG[2                 waltz16
PCPD2                      90.00 usec
PLW2                  12.00000000 W
PLW12                  0.21333000 W
PLW13                  0.10731000 W

F2 - Processing parameters
SI                        32768
SF                    75.4677485 MHz
WDW                          EM
SSB                   0
LB                         1.00 Hz
GB                    0
PC                         1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7d**

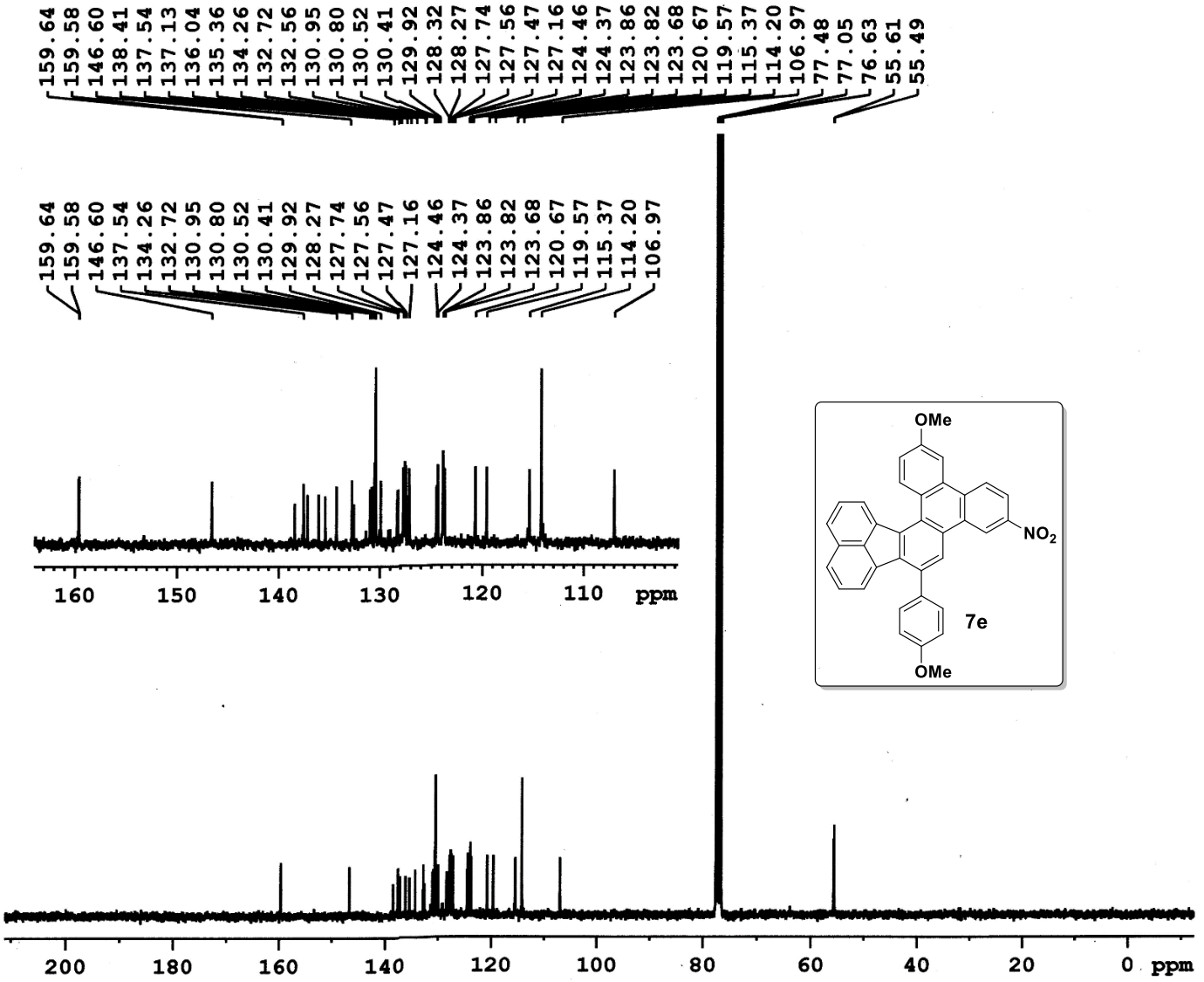



```
Current Data Parameters
NAME                  KD-II-278
EXPNO                         8
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240712
Time                      19.44
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG            deptsp135
TD                        65536
SOLVENT                   CDCl3
NS                          256
DS                            4
SWH                   12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                         2050
DW                       41.400 usec
DE                        6.50 usec
TE                       300.0 K
CNST2                145.0000000
D1                    2.00000000 sec
D2                    0.00344828 sec
D12                   0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4737856 MHz
NUC1                        13C
P1                        11.00 usec
P13                     2000.00 usec
PLW0       0 W
PLW1              48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                    0.500
SPOFFS5    0 Hz
SPW5               8.87399960 W

======== CHANNEL f2 ========
SFO2                300.1309599 MHz
NUC2                        1H
CPDPRG[2                waltz16
P3                        12.00 usec
P4                        24.00 usec
PCPD2                     90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677485 MHz
WDW                          EM
SSB        0
LB                        1.00 Hz
GB         0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **7d**

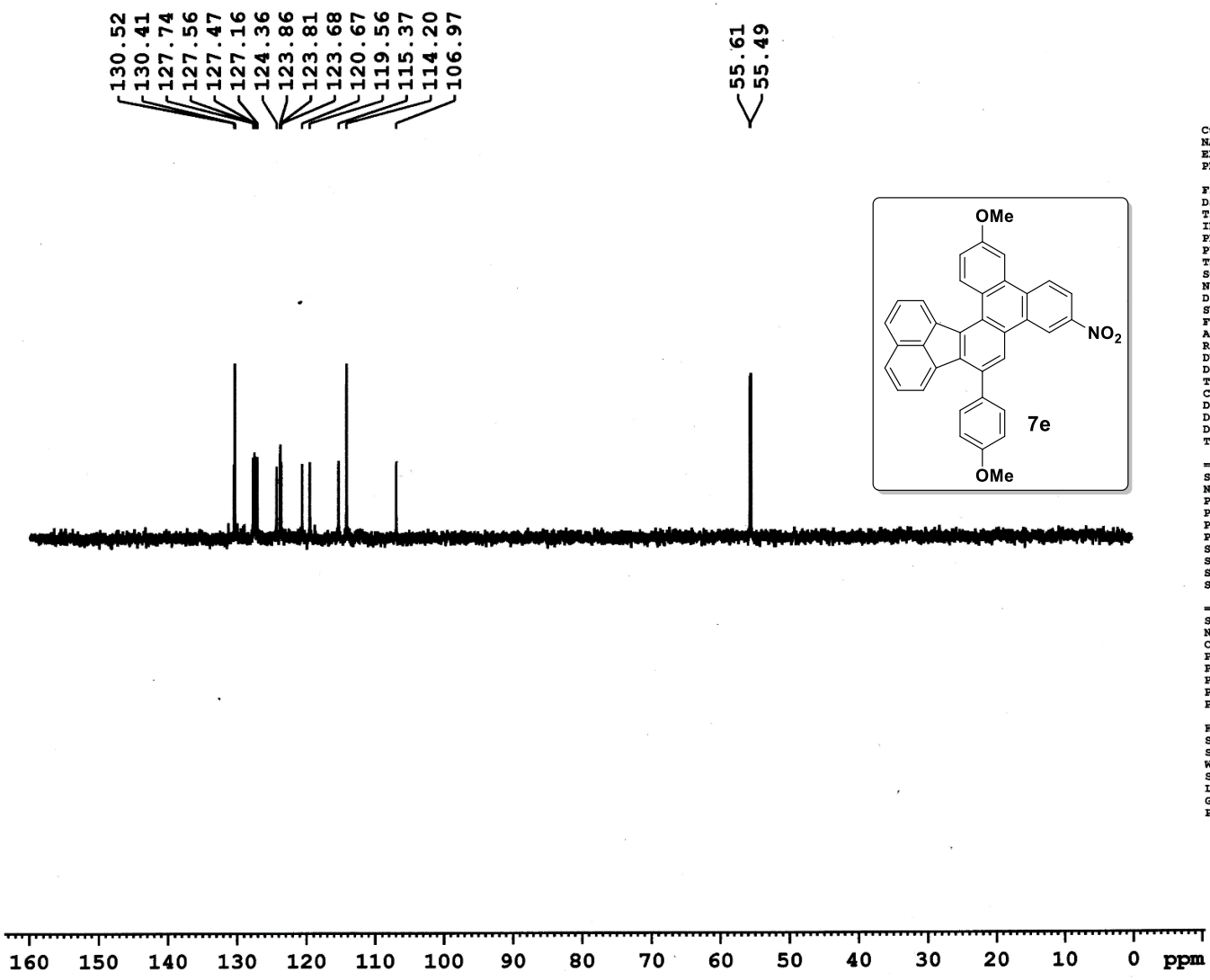


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7e**



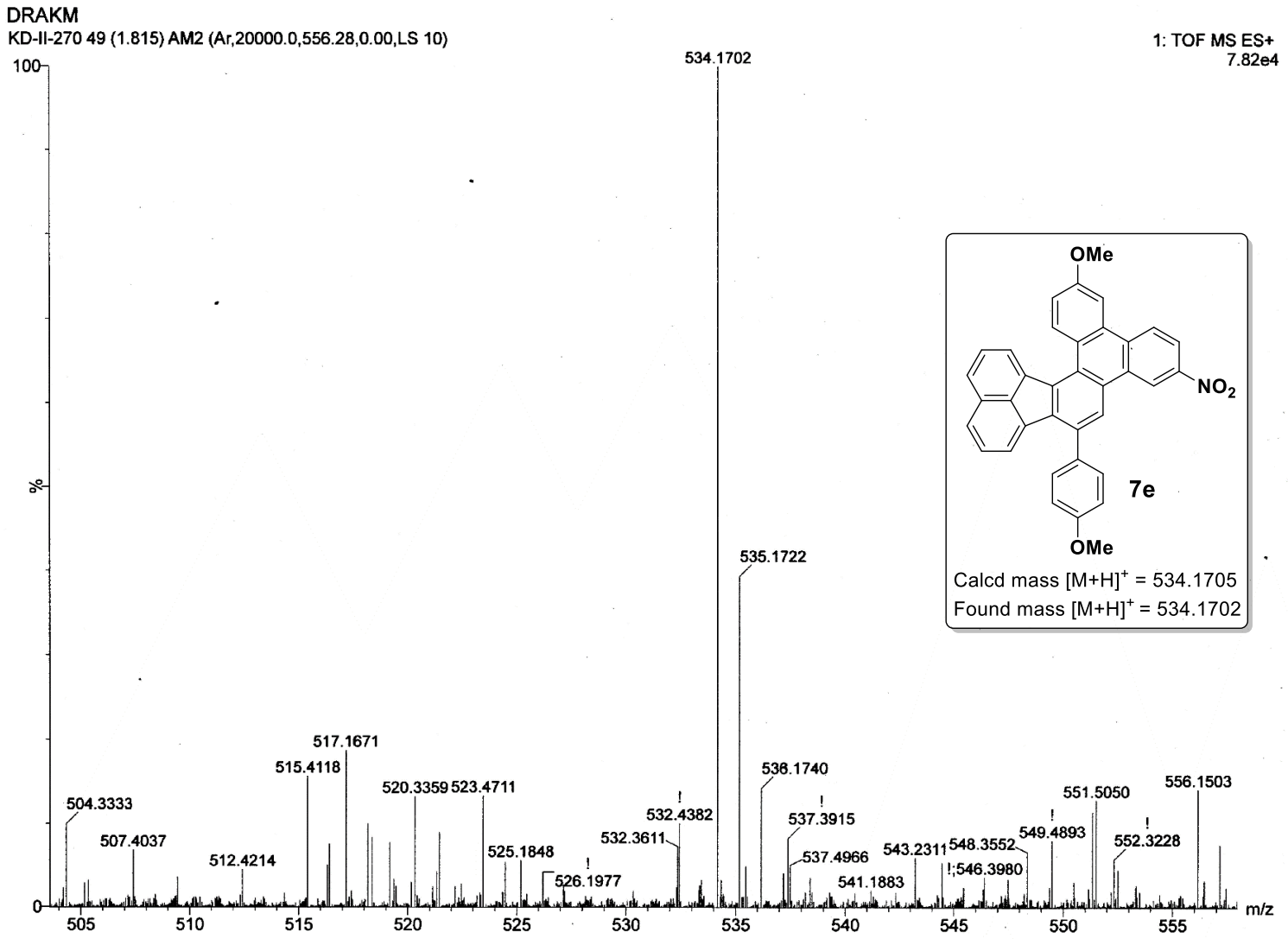

```
Current Data Parameters
NAME                 KD-II-270
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20240629
Time                   14.15
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG              zgpg30
TD                    65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                       812
DW                    27.733 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                       32768
SF                 75.4677473 MHz
WDW                        EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7e**

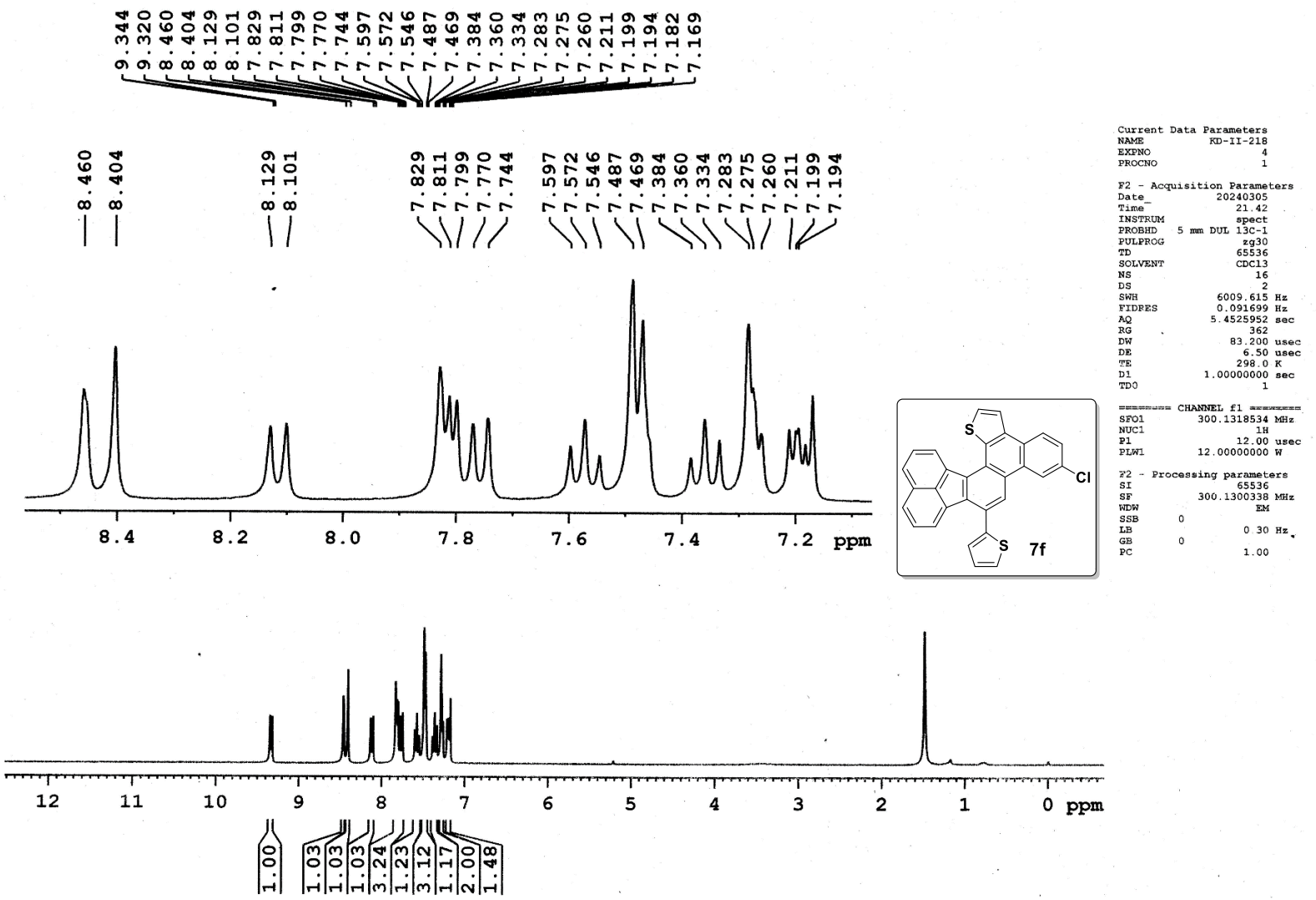



```
Current Data Parameters
NAME                  KD-II-270
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240629
Time                      14.36
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG              deptsp135
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  12077.295 Hz
FIDRES                0.184285 Hz
AQ                   2.7131903 sec
RG                        2050
DW                      41.400 usec
DE                       6.50 usec
TE                      300.0 K
CNST2               145.0000000
D1                    2.00000000 sec
D2                    0.00344828 sec
D12                   0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4737856 MHz
NUC1                        13C
P1                        11.00 usec
P13                     2000.00 usec
PLW0        0 W
PLW1           48.00000000 W
SPNAM[5]       Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5            8.87399960 W

======== CHANNEL f2 ========
SFO2                300.1309599 MHz
NUC2                        1H
CPDPRG[2                waltz16
P3                        12.00 usec
P4                        24.00 usec
PCPD2                     90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677476 MHz
WDW                          EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

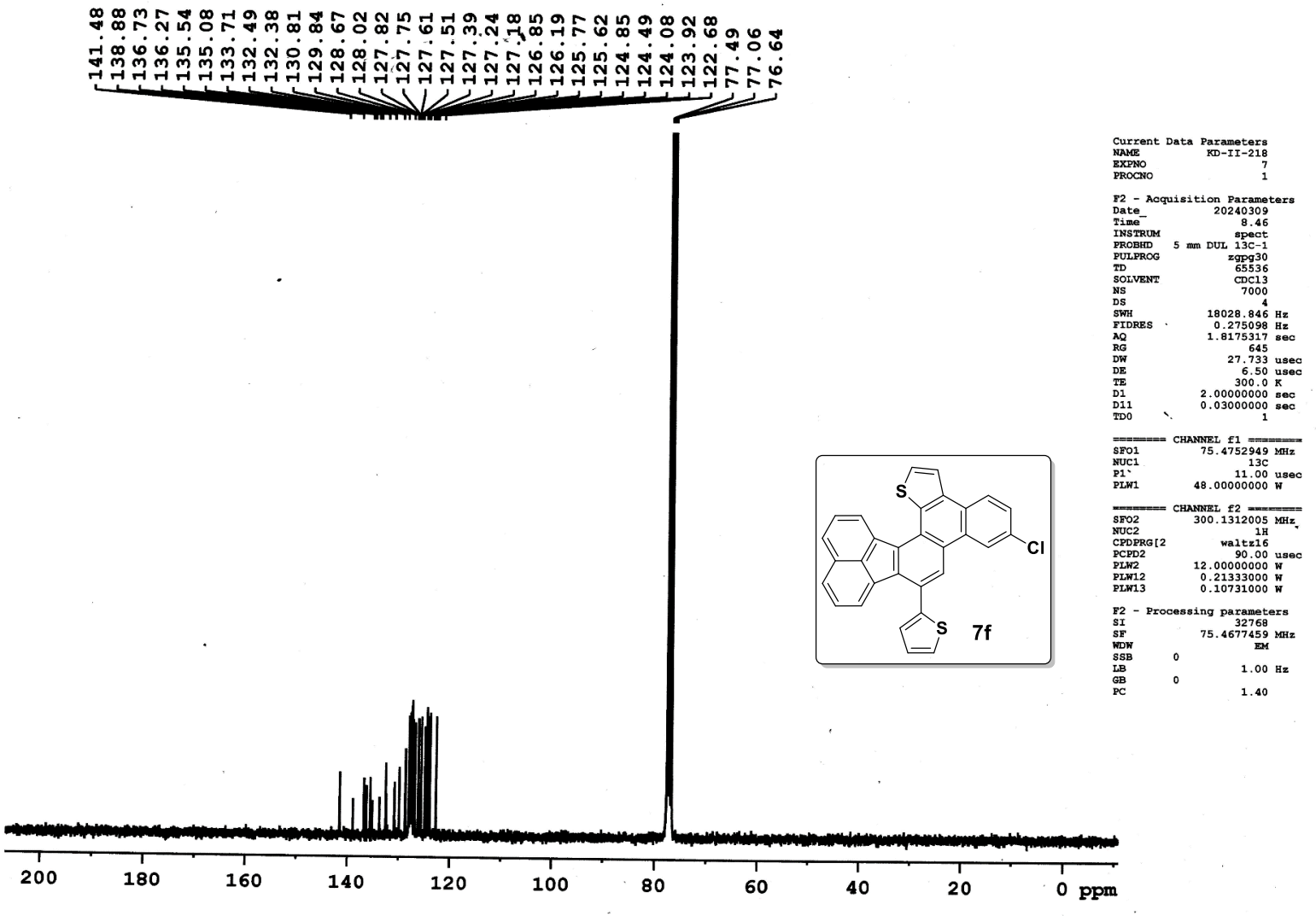
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **7e**



HRMS spectrum of compound **7e**

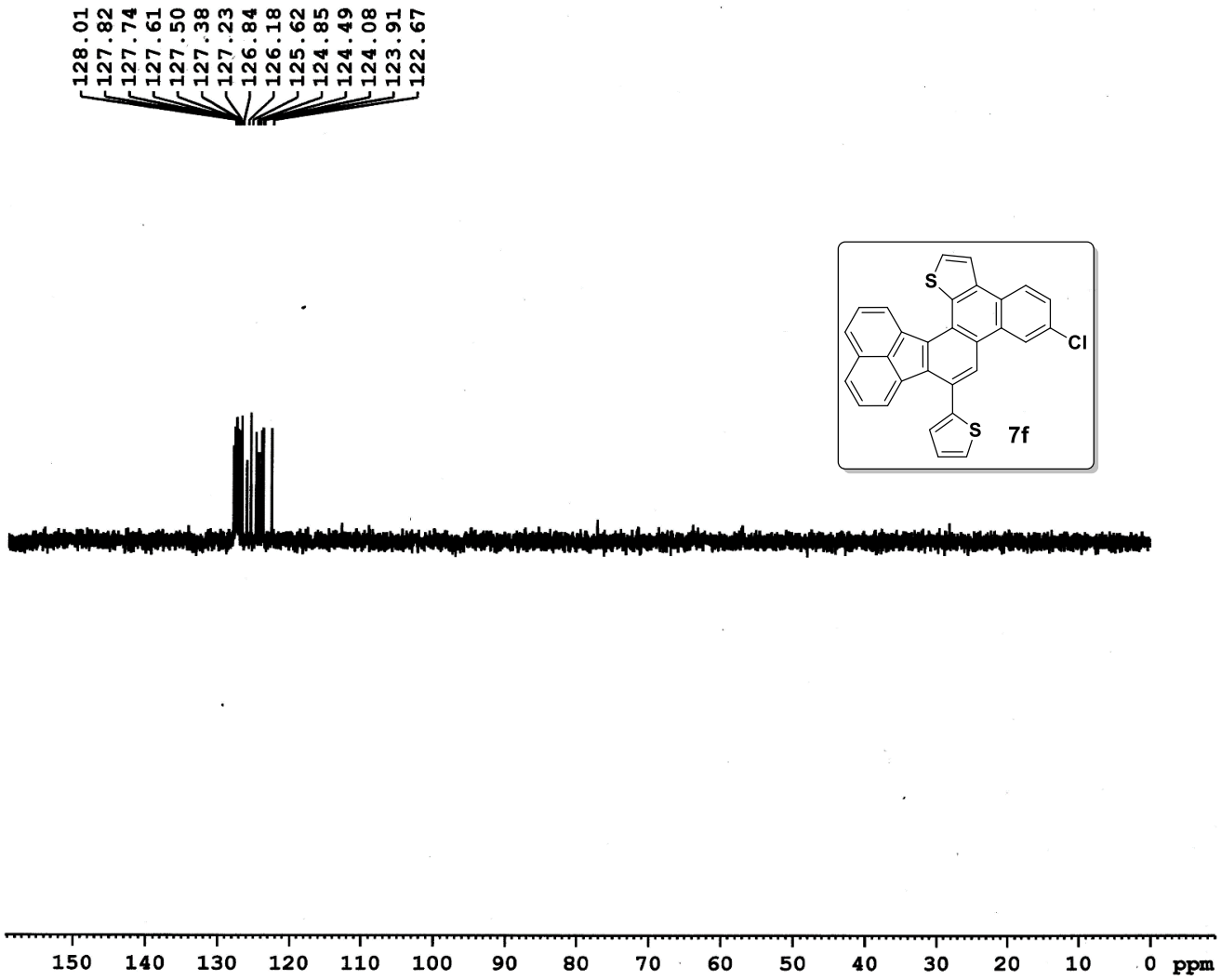


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **7f**




```
Current Data Parameters
NAME                  KD-II-218
EXPNO                         7
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240309
Time                       8.46
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                          65536
SOLVENT                   CDCl3
NS                           7000
DS                              4
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                            645
DW                       27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                       11.00 usec
PLW1                48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W
PLW13                0.10731000 W

F2 - Processing parameters
SI                         32768
SF                   75.4677459 MHz
WDW                           EM
SSB                0
LB                            1.00 Hz
GB                 0
PC                            1.40
```

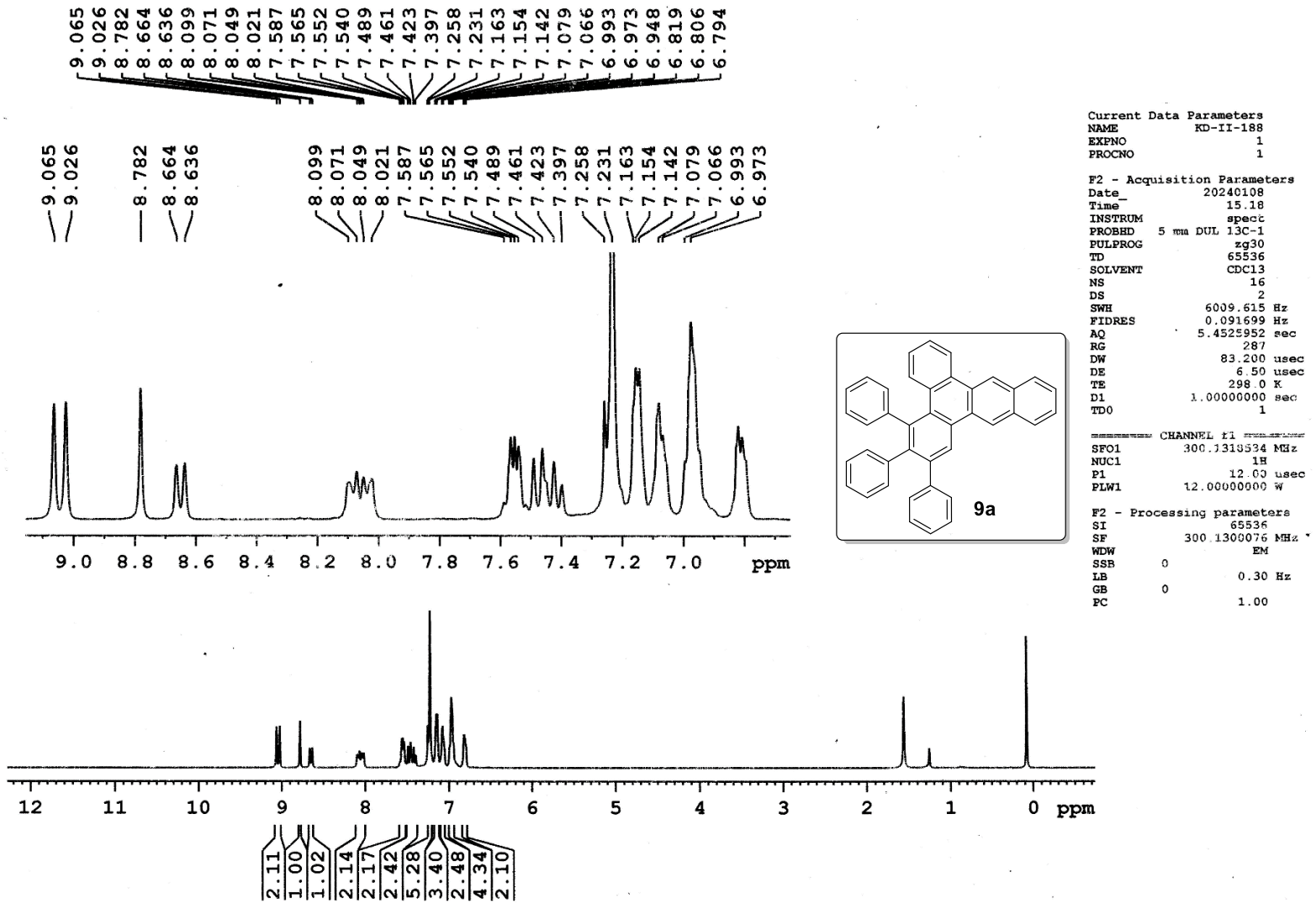
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **7f**



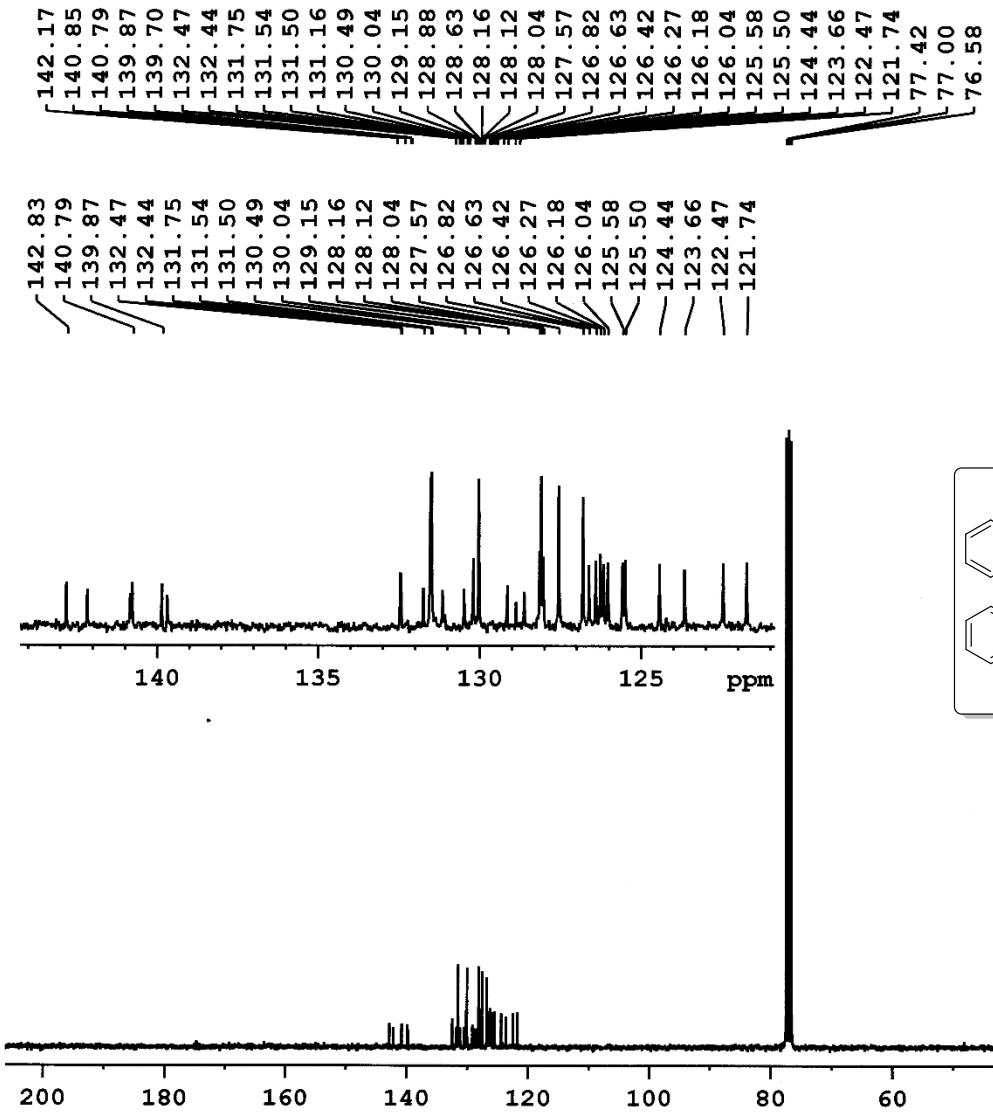

```
Current Data Parameters
NAME            KD-II-218
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240309
Time                1.16
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG         deptsp135
TD                  65536
SOLVENT             CDCl3
NS                   1000
DS                      4
SWH             12077.295 Hz
FIDRES`          0.184285 Hz
AQ              2.7131903 sec
RG                   2050
DW                 41.400 usec
DE                  6.50 usec
TE                 300.0 K
CNST2          145.0000000
D1               2.00000000 sec
D2               0.00344828 sec
D12              0.00002000 sec
TD0                     1

========= CHANNEL f1 ========
SFO1            75.4737856 MHz
NUC1                   13C
P1                   11.00 usec
P13                2000.00 usec
PLW0       0 W
PLW1         48.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5    0 Hz
SPW5          8.87399960 W

======== CHANNEL f2 ========
SFO2          300.1309599 MHz
NUC2                   1H
CPDPRG[2           waltz16
P3                   12.00 usec
P4                   24.00 usec
PCPD2                90.00 usec
PLW2          12.00000000 W
PLW12          0.21333000 W

F2 - Processing parameters
SI                   32768
SF              75.4677465 MHz
WDW                    EM
SSB        0
LB                    1.00 Hz
GB         0
PC                    1.40
```

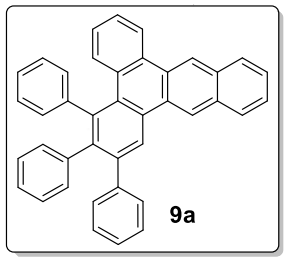
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **7f**

8. ^{1}H, ^{13}C {^{1}H}, DEPT 135 (selected) NMR & HRMS (selected) spectra of phenanthrenes **9a-j** and hetero helicene **10a** & **10b**



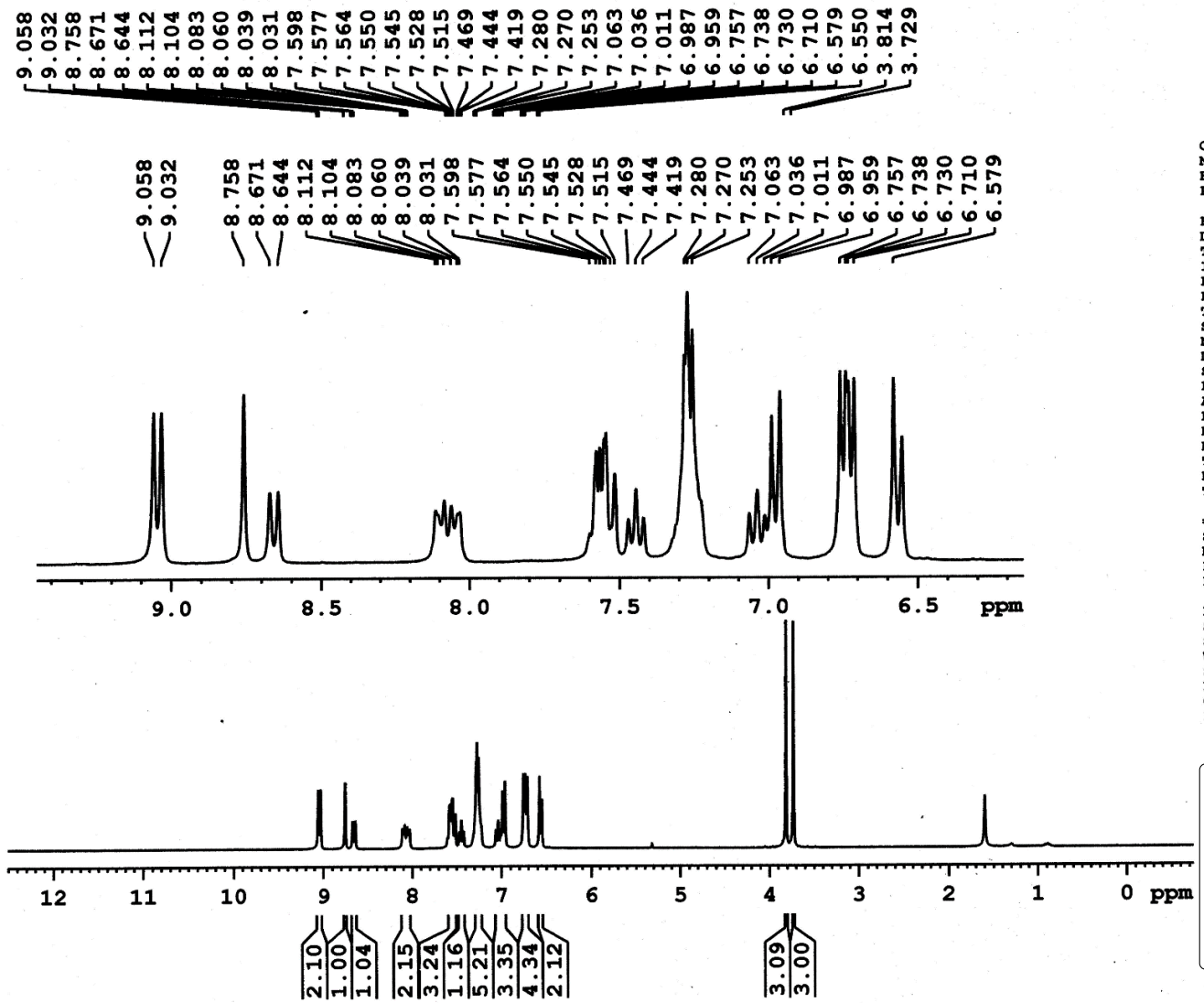
^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9a**



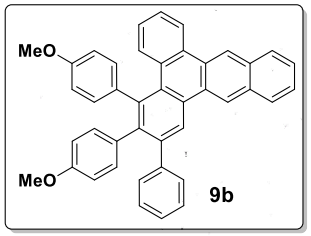



```
Current Data Parameters
NAME                  KD-II-188
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240108
Time                      16.27
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS .                          4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                         1030
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677509 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                         1.40
```

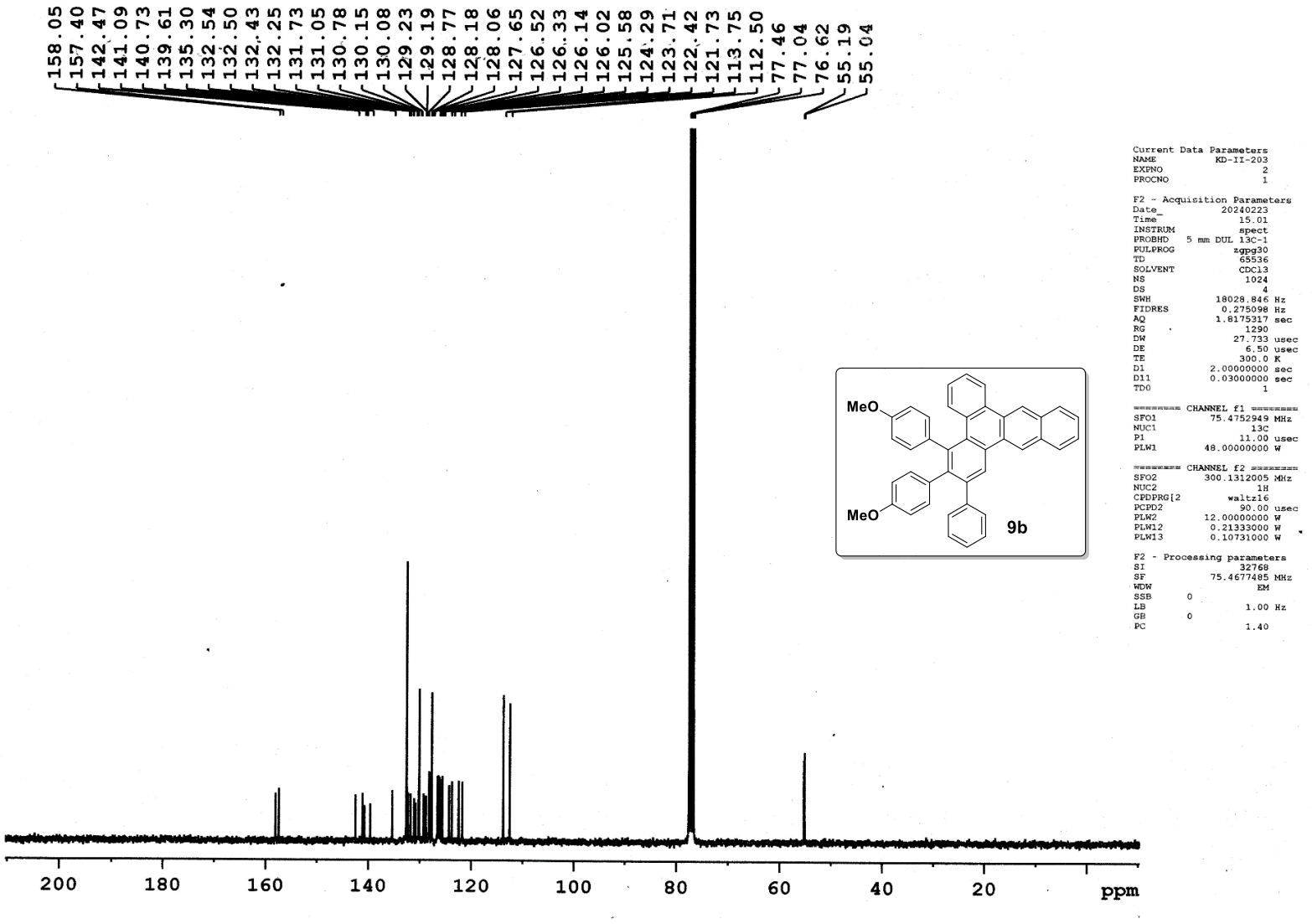
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9a**




```
Current Data Parameters
NAME                  KD-II-203
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240223
Time                      13.52
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    6009.615 Hz
FIDRES                 0.091699 Hz
AQ                     5.4525952 sec
RG                          203
DW                     83.200 usec
DE                      6.50 usec
TE                      298.0 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 300.1318534 MHz
NUC1                         1H
P1                      12.00 usec
PLW1                12.00000000 W

F2 - Processing parameters
SI                        65536
SF                   300.1300000 MHz
WDW                          EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

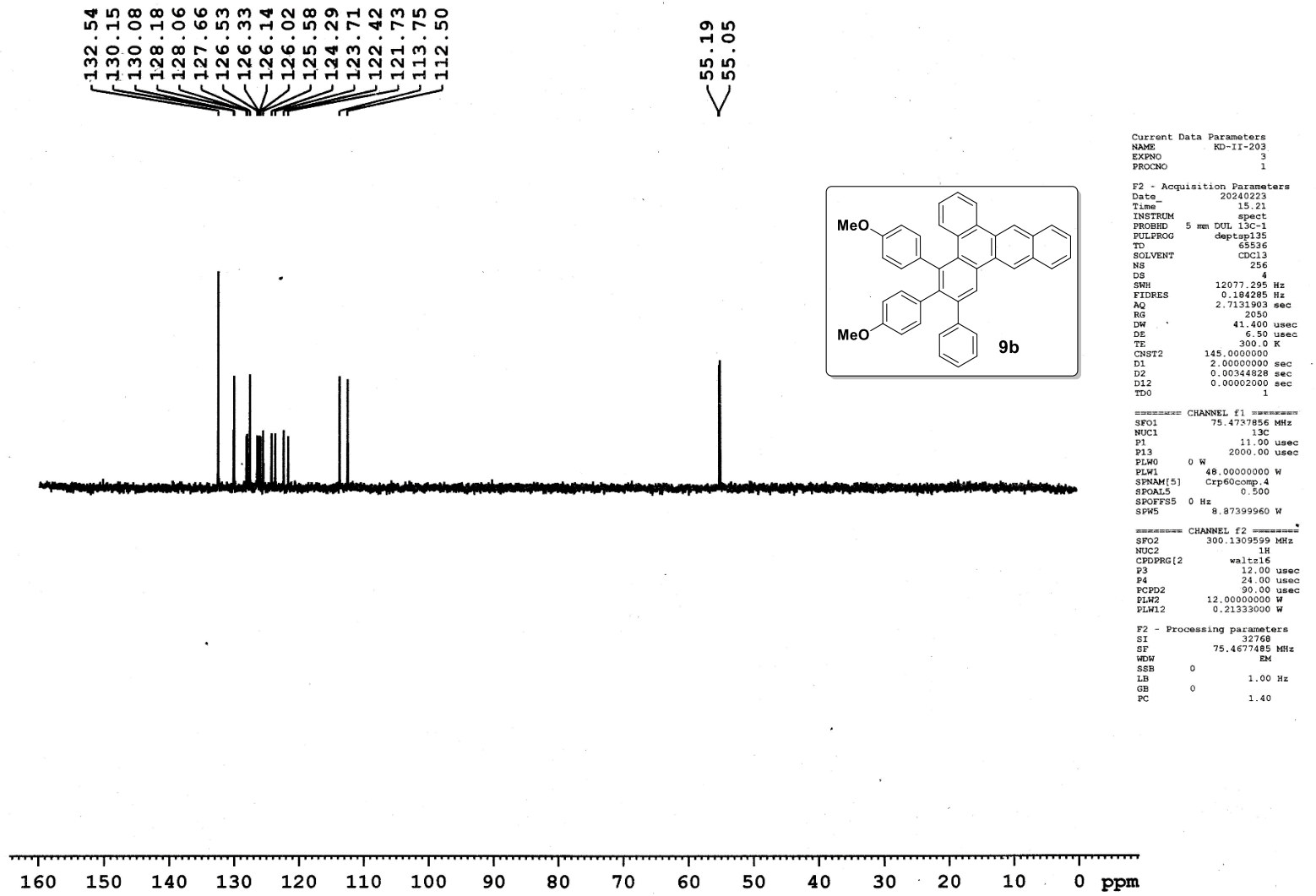


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9b**



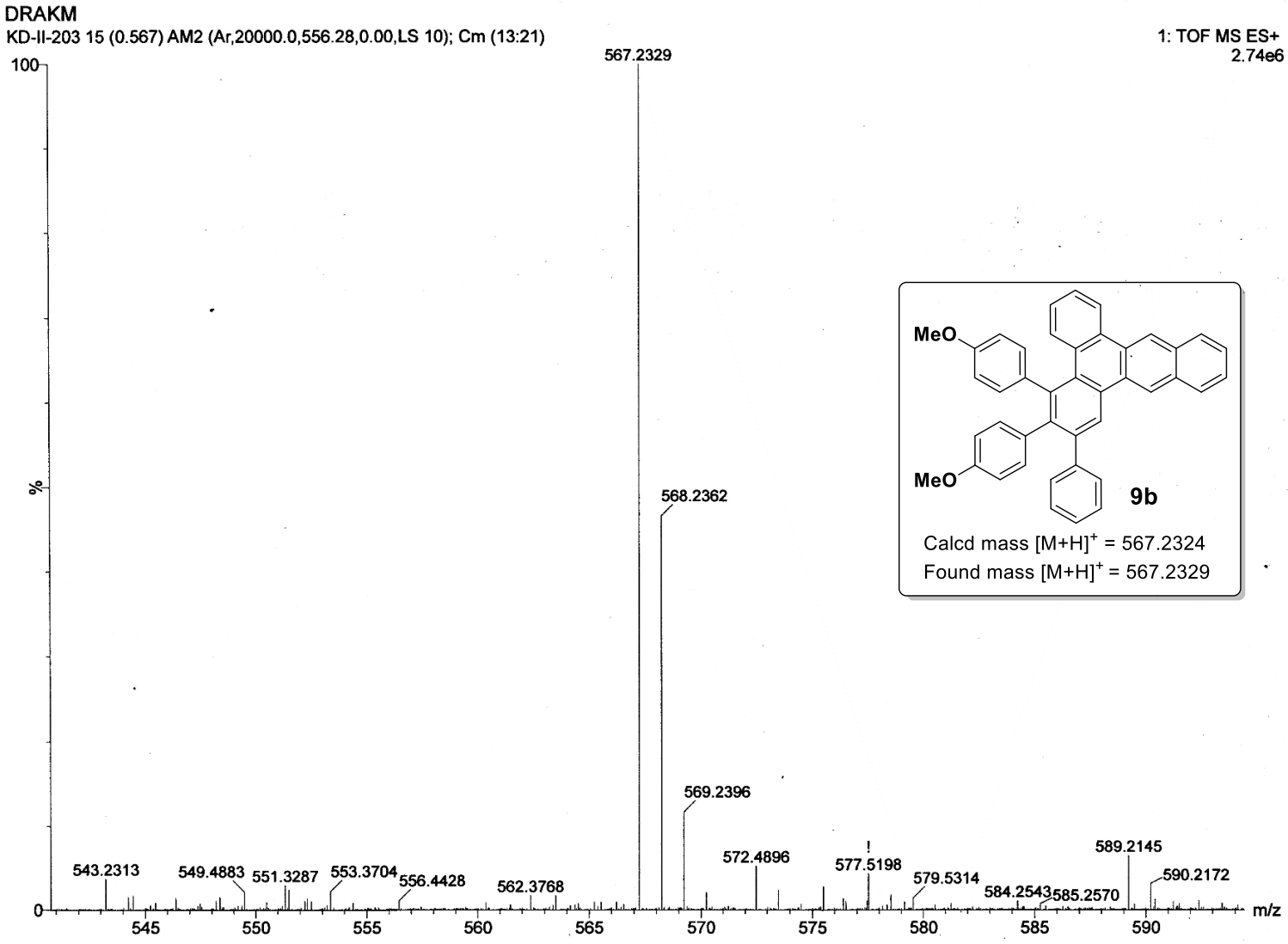

```
Current Data Parameters
NAME                  KD-II-203
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240223
Time                      15.01
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                   18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175217 sec
RG                         1290
DW                       27.733 usec
DE                         6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                         11.00 usec
PLW1                 48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                      90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W
PLW13                 0.10731000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677485 MHz
WDW                          EM
SSB                   0
LB                         1.00 Hz
GB                    0
PC                         1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9b**

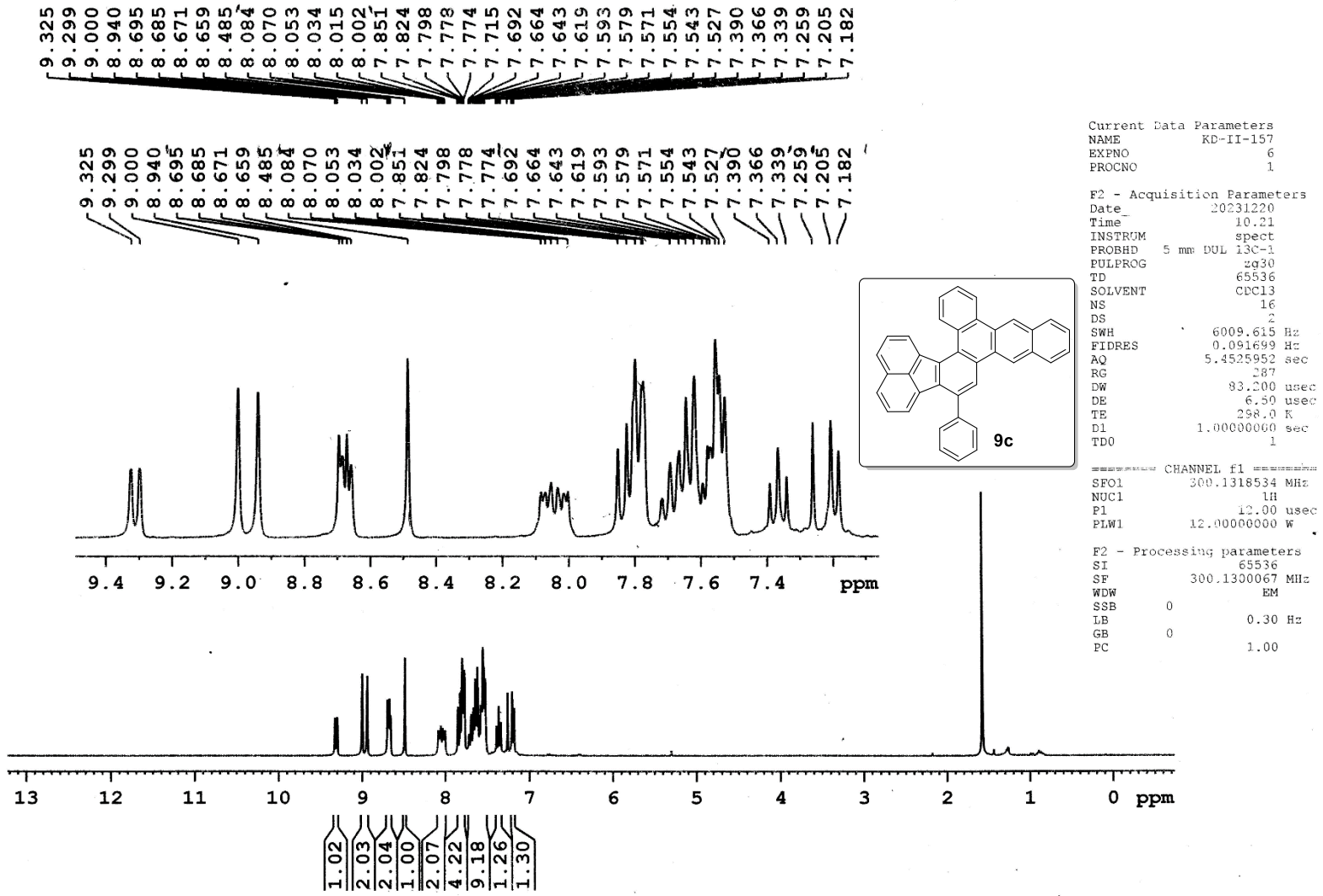



```
Current Data Parameters
NAME                  KD-II-203
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_               20240223
Time                      15.21
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG              deptsp135
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                         2050
DW                       41.400 usec
DE                        6.50 usec
TE                       300.0 K
CNST2                145.0000000
D1                     2.00000000 sec
D2                     0.00344828 sec
D12                    0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4737856 MHz
NUC1                        13C
P1                        11.00 usec
P13                     2000.00 usec
PLW0       0 W
PLW1                 48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                    0.500
SPOFFS5    0 Hz
SPW5                  8.87399960 W

======== CHANNEL f2 ========
SFO2                 300.1309599 MHz
NUC2                        1H
CPDPRG[2                waltz16
P3                        12.00 usec
P4                        24.00 usec
PCPD2                     90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W

F2 - Processing parameters
SI                        32768
SF                   75.4677485 MHz
WDW                          EM
SSB        0
LB                        1.00 Hz
GB         0
PC                        1.40
```

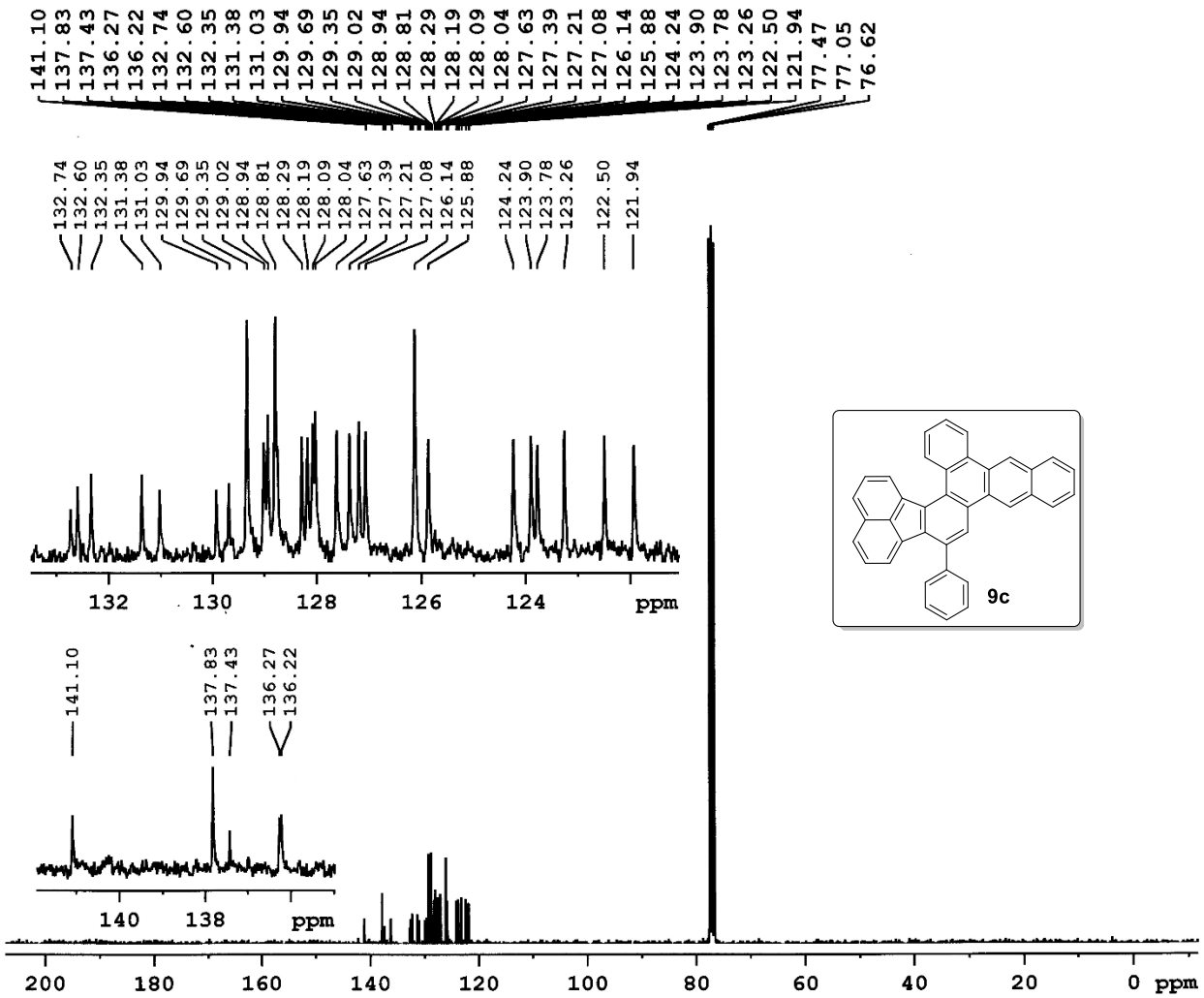
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **9b**



HRMS spectrum of compound **9b**

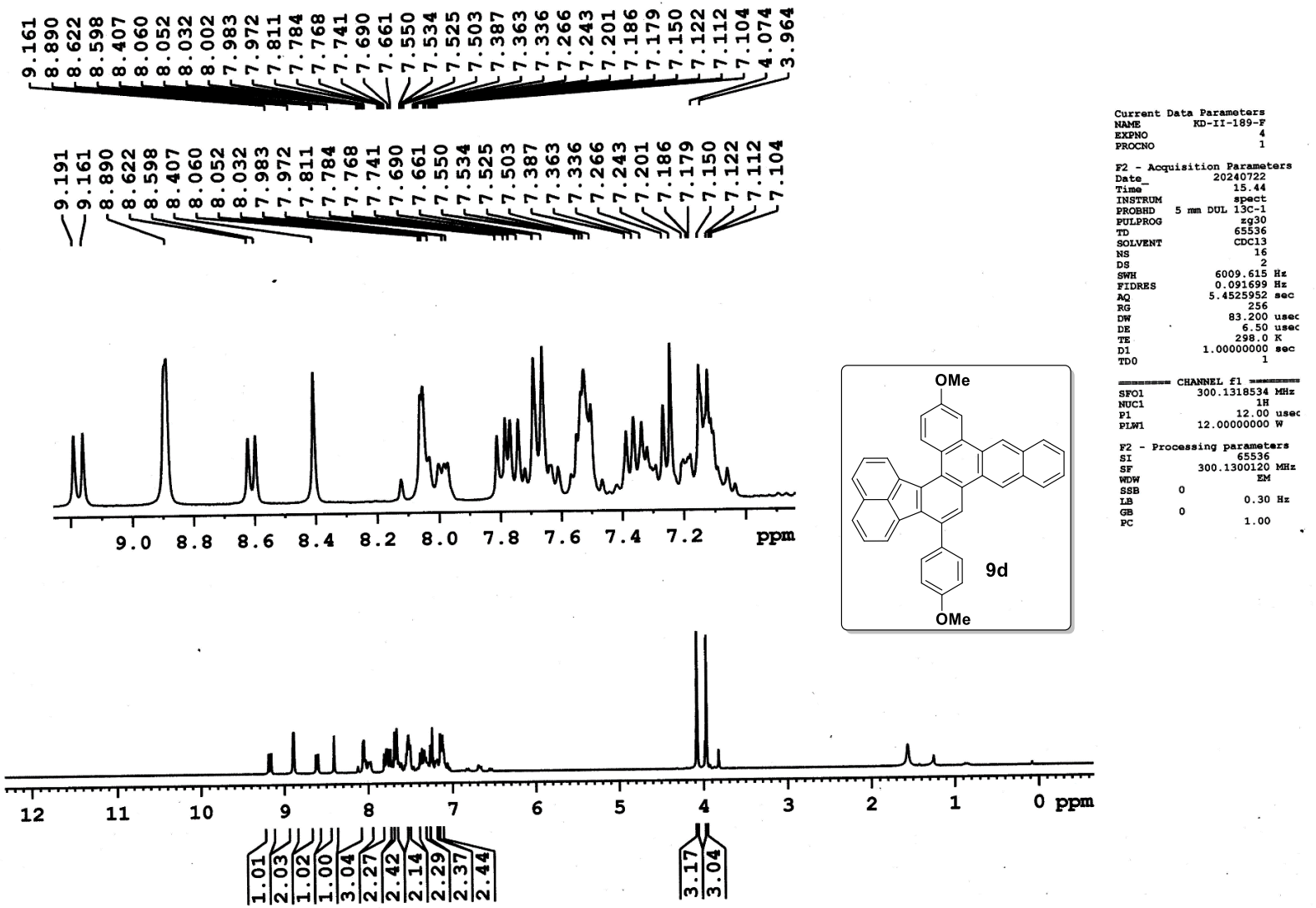


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9c**



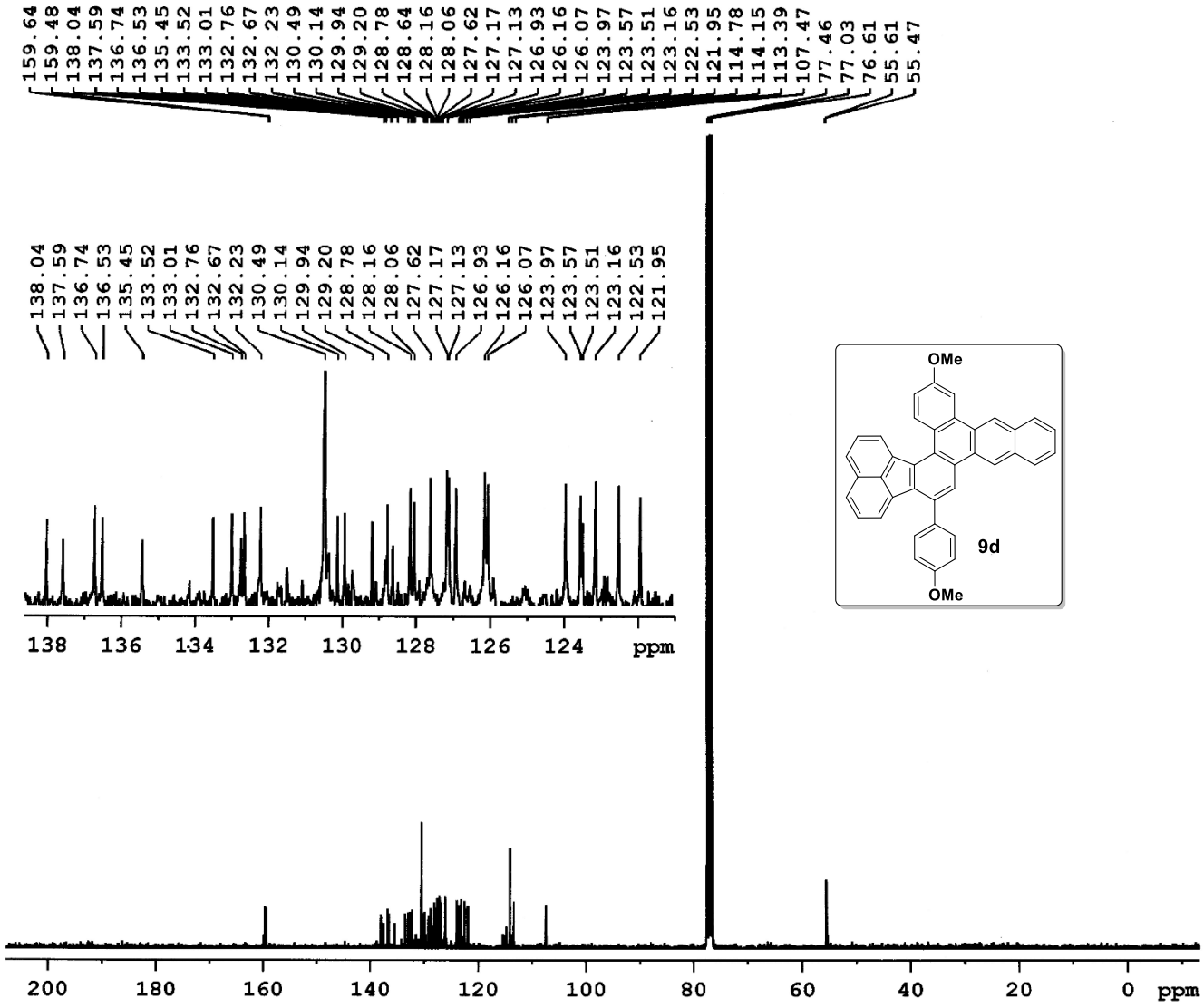

```
Current Data Parameters
NAME                  KD-II-157
EXPNO                         7
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20231220
Time                      16.10
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                          645
DW                     27.733 usec
DE                      6.50 usec
TE                      300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9c**



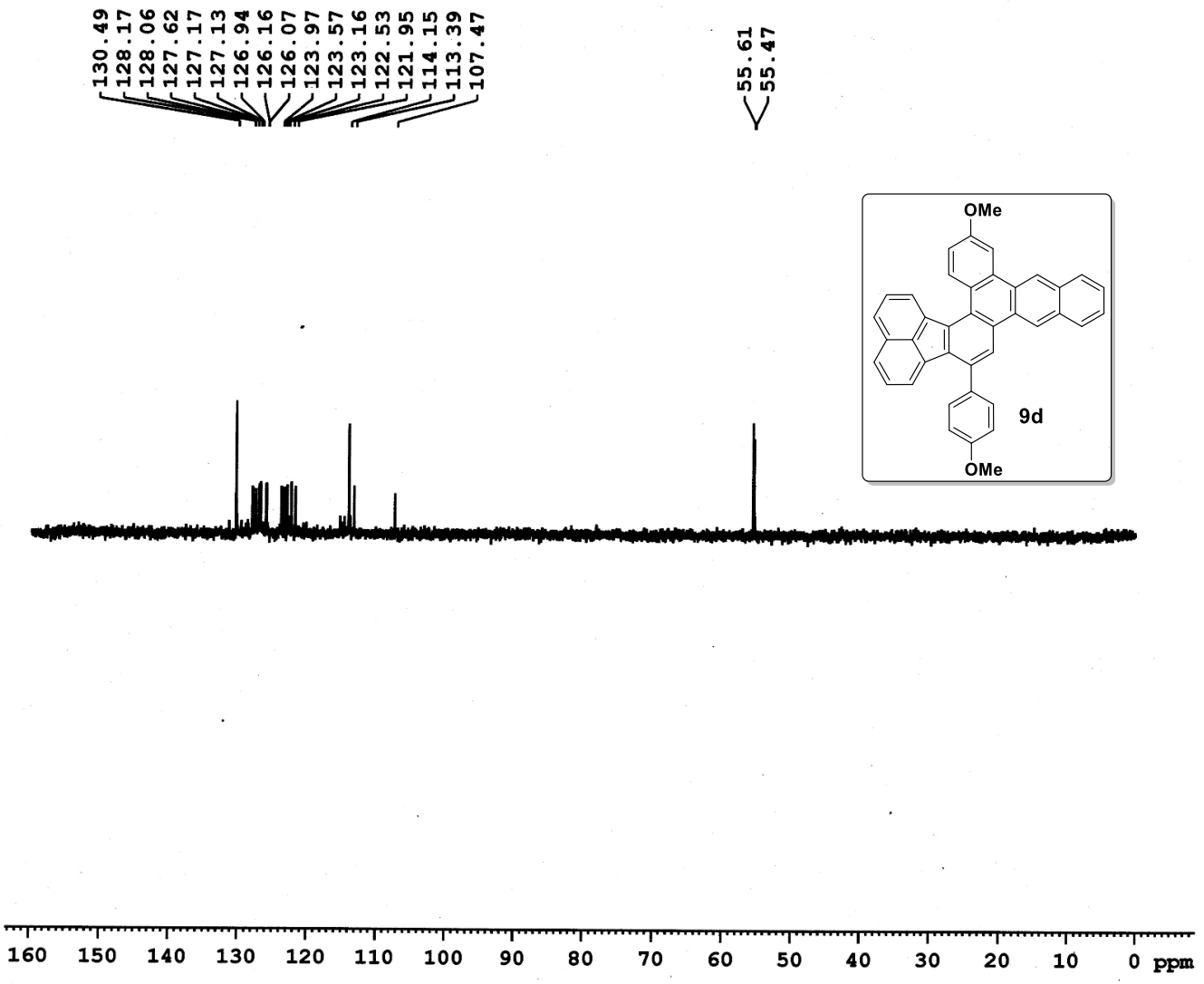

```
Current Data Parameters
NAME          KD-II-189-F
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240722
Time                15.44
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4525952 sec
RG                   256
DW              83.200 usec
DE               6.50 usec
TE              298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1                   1H
P1                12.00 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           300.1300120 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9d**



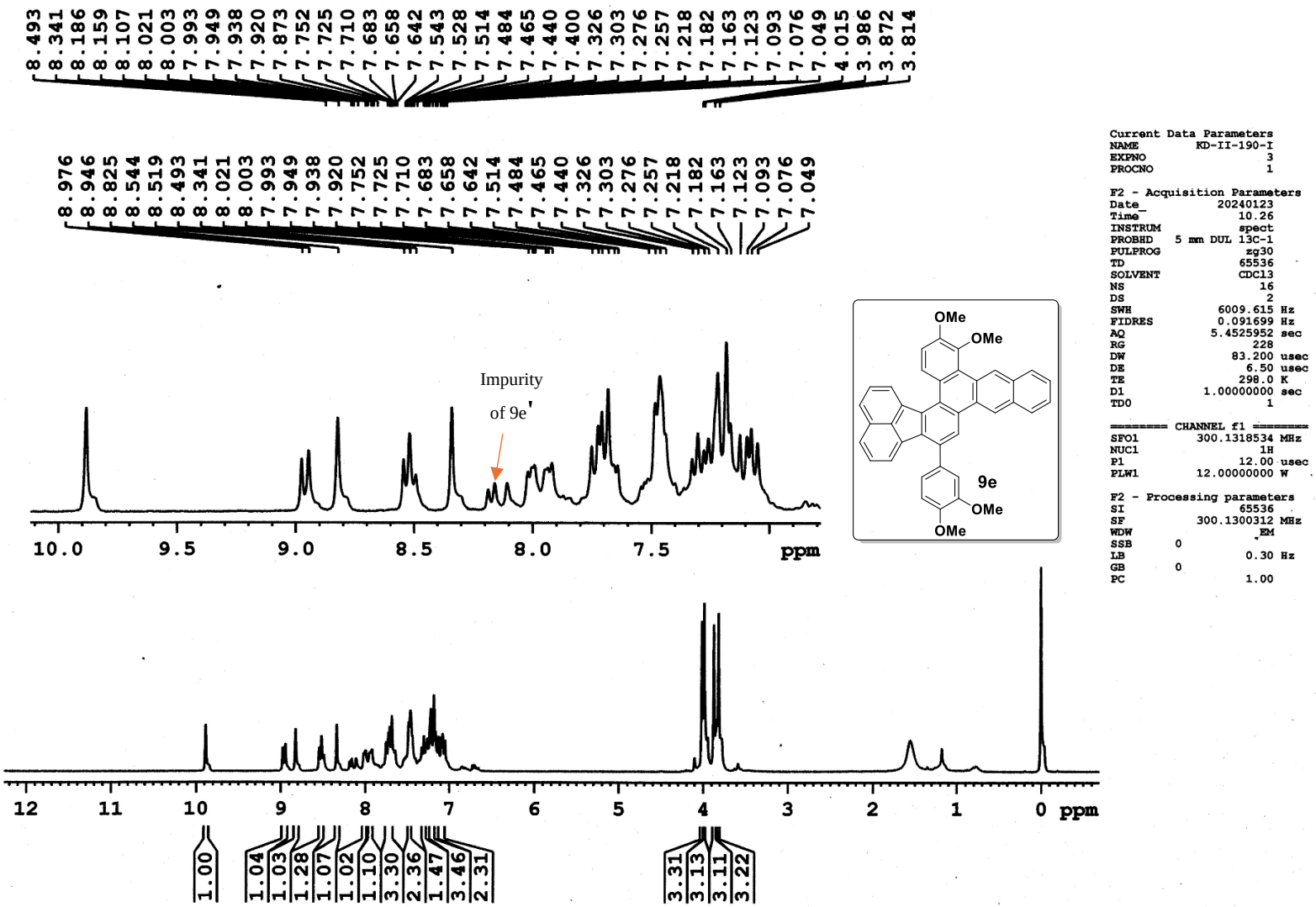

```
Current Data Parameters
NAME                 KD-II-189-F
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_               20240722
Time                   15.20
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                18028.846 Hz
FIDRES                 0.275098 Hz
AQ                     1.8175317 sec
RG                       912
DW                      27.733 usec
DE                       6.50 usec
TE                     300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                75.4752949 MHz
NUC1                       13C
P1                      11.00 usec
PLW1                48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                   90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W
PLW13                0.10731000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677485 MHz
WDW                           EM
SSB              0
LB                      1.00 Hz
GB               0
PC                      1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9d**



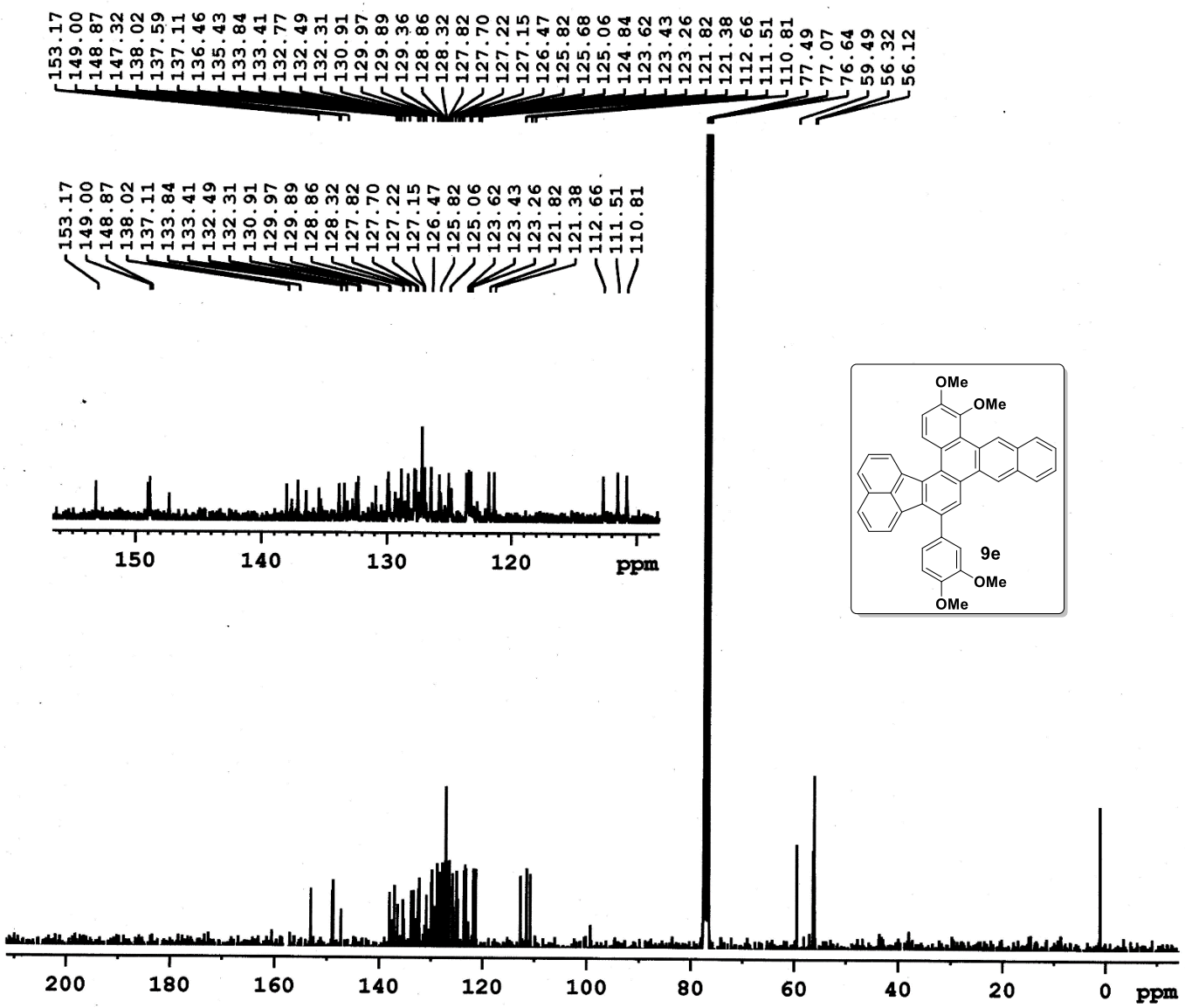

```
Current Data Parameters
NAME                 KD-II-189-F
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_               20240722
Time                    15.41
INSTRUM                 spect
PROBHD     5 mm DUL 13C-1
PULPROG             deptsp135
TD                      65536
SOLVENT                 CDCl3
NS                        256
DS                          4
SWH                 12077.295 Hz
FIDRES                0.184285 Hz
AQ                    2.7131903 sec
RG                       2050
DW                     41.400 usec
DE                      6.50 usec
TE                     300.0 K
CNST2                145.0000000
D1                     2.00000000 sec
D2                     0.00344828 sec
D12                    0.00002000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                75.4737856 MHz
NUC1                       13C
P1                       11.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1           48.00000000 W
SPNAM[5]       Crp60comp.4
SPOAL5                   0.500
SPOFFS5     0 Hz
SPW5            8.87399960 W

======== CHANNEL f2 ========
SFO2               300.1309599 MHz
NUC2                       1H
CPDPRG[2               waltz16
P3                       12.00 usec
P4                       24.00 usec
PCPD2                    90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677485 MHz
WDW                           EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **9d**



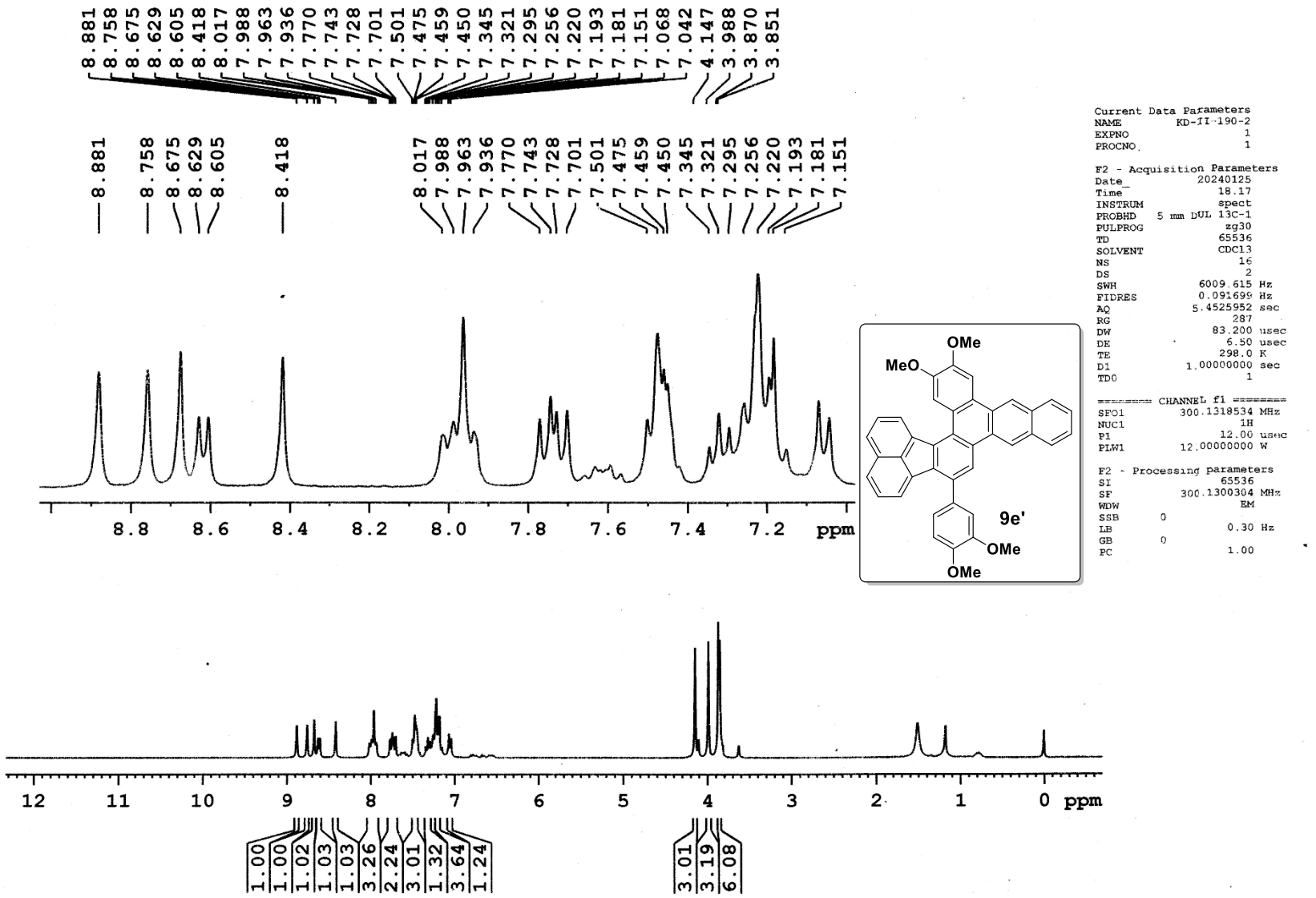

```
Current Data Parameters
NAME          KD-II-190-I
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240123
Time                10.26
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    228
DW                 83.200 usec
DE                  6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                  1H
P1                 12.00 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                  65536
SF            300.1300312 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9e**




```
Current Data Parameters
NAME          KD-II-190-I
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240123
Time                11.35
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ            1.8175317 sec
RG                   645
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 11.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W
PLW13         0.10731000 W

F2 - Processing parameters
SI                 32768
SF            75.4677468 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9e**



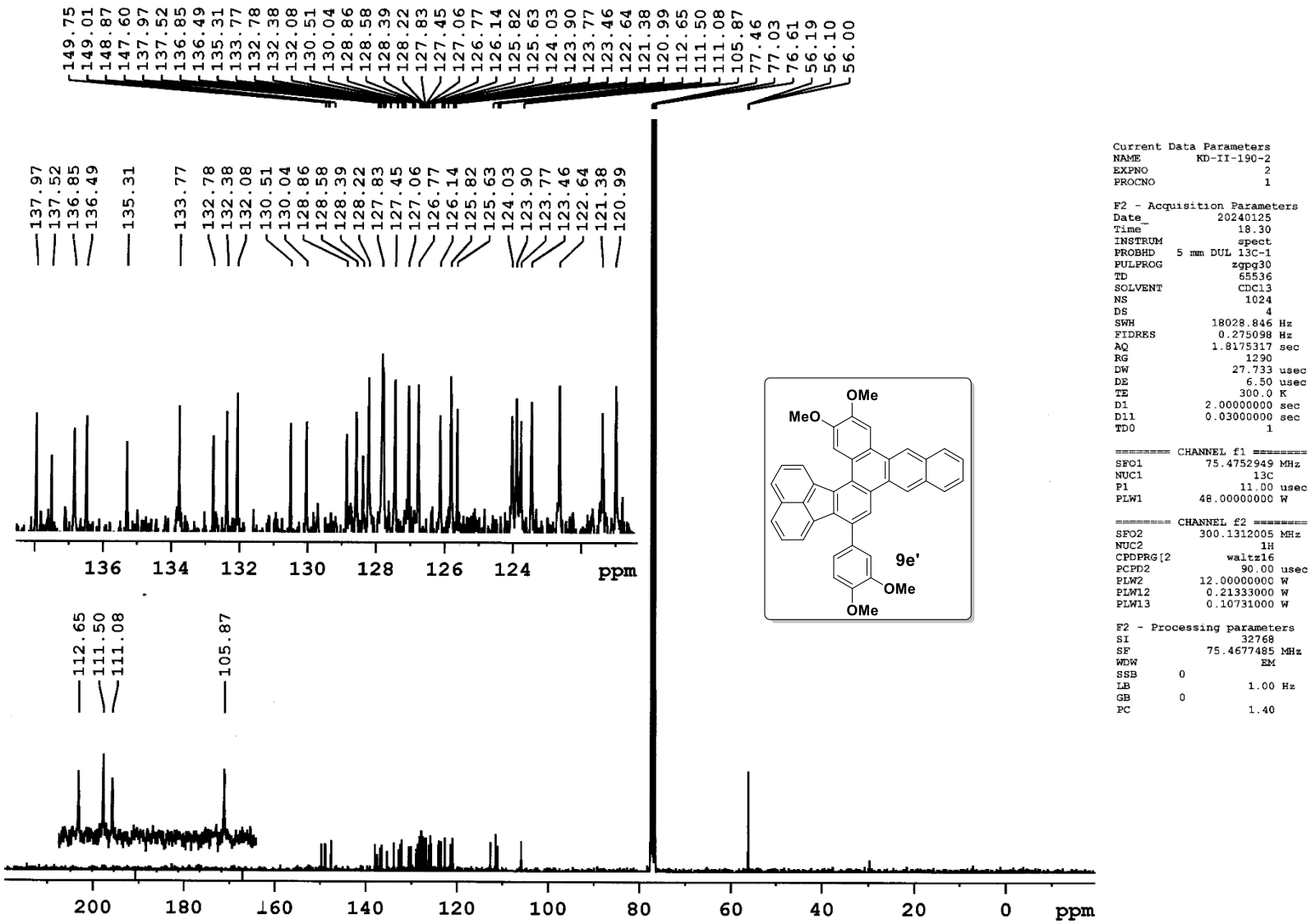
Current Data Parameters
NAME KD-II-190-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20240125
Time 18.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 287
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 12.00 usec
PLW1 12.00000000 W

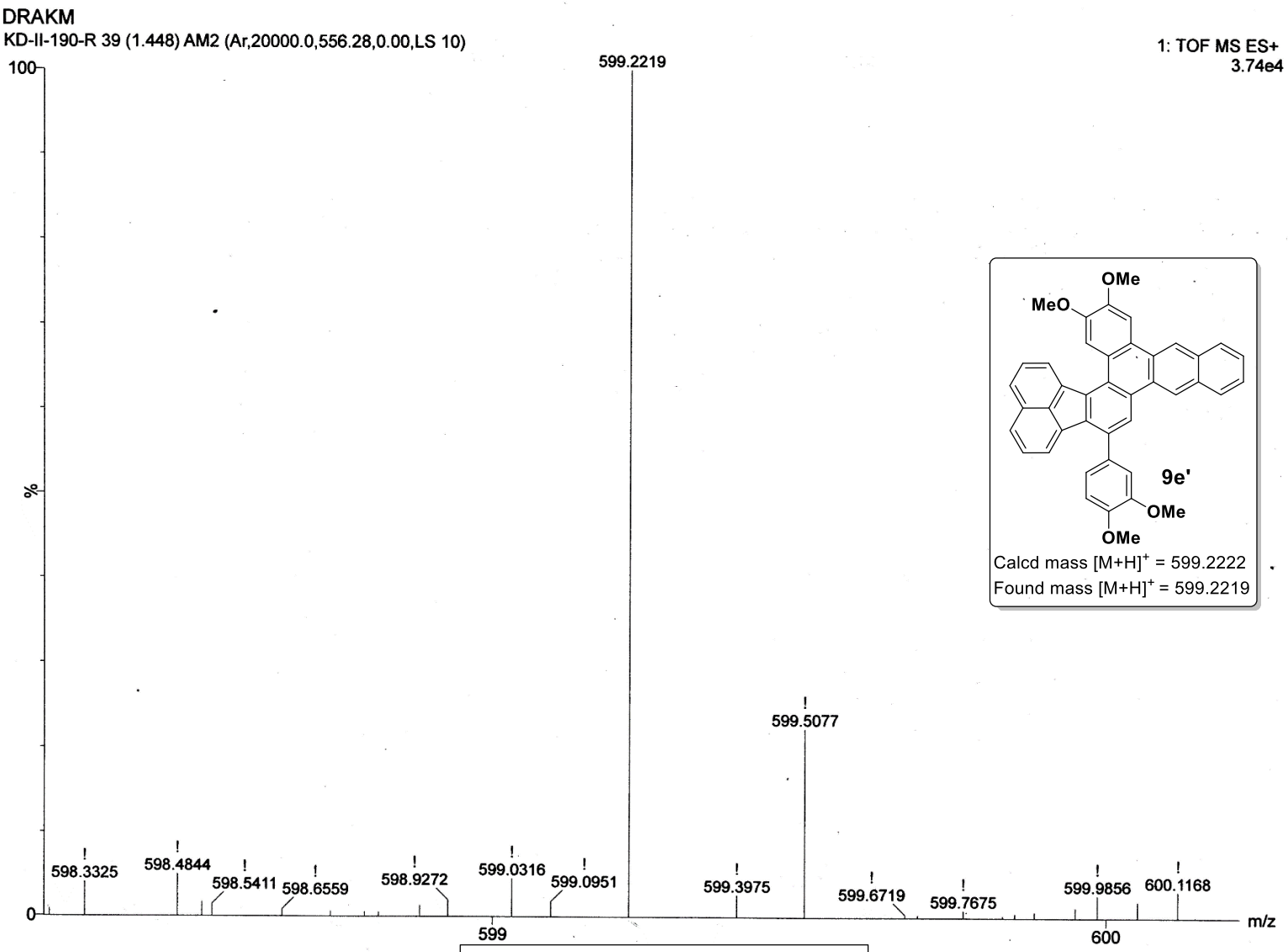
F2 - Processing parameters
SI 65536
SF 300.1300304 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9e'**

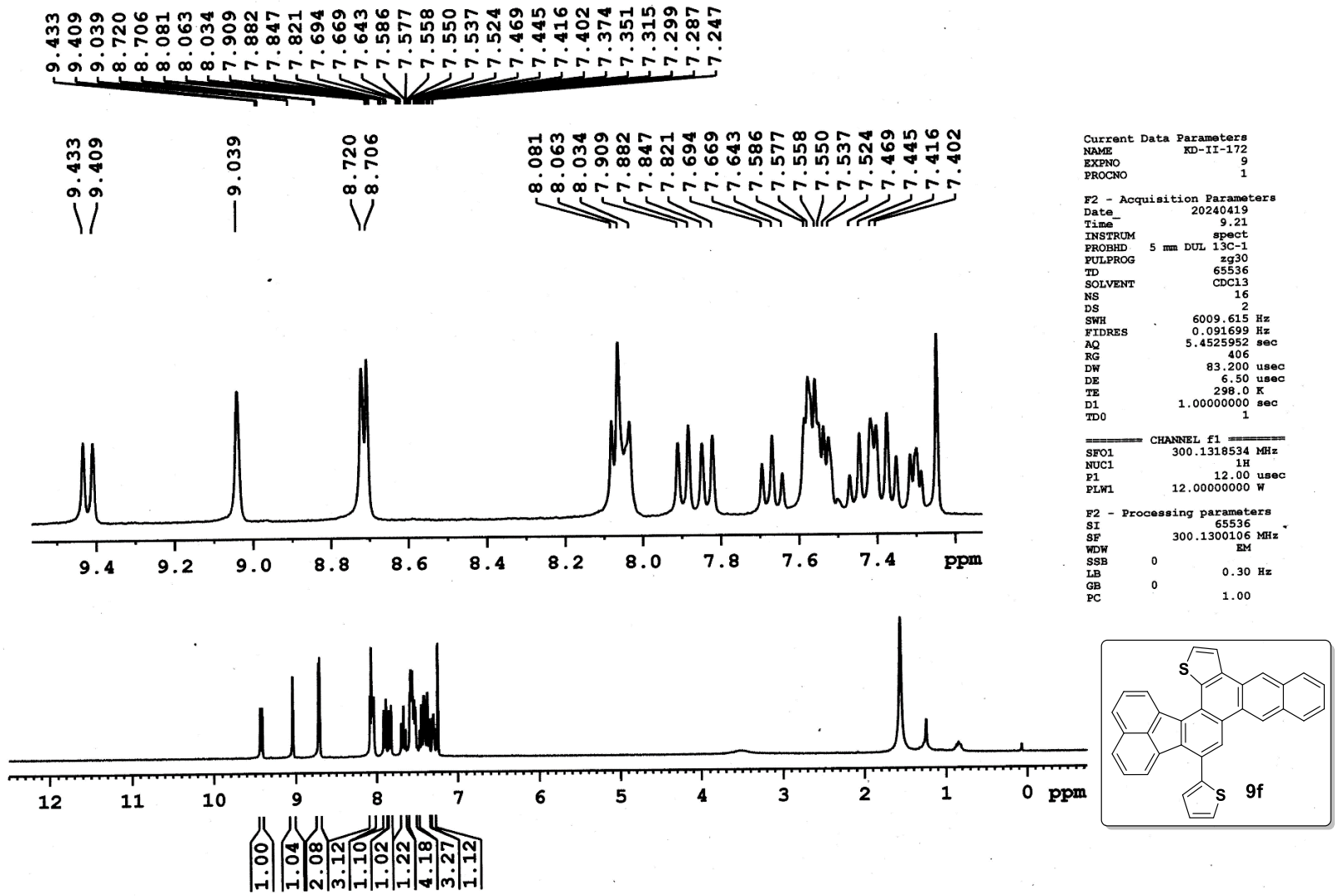



```
Current Data Parameters
NAME          KD-II-190-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_          20240125
Time              18.30
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175317 sec
RG                1290
DW              27.733 usec
DE               6.50 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1                13C
P1                11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI                32768
SF          75.4677485 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

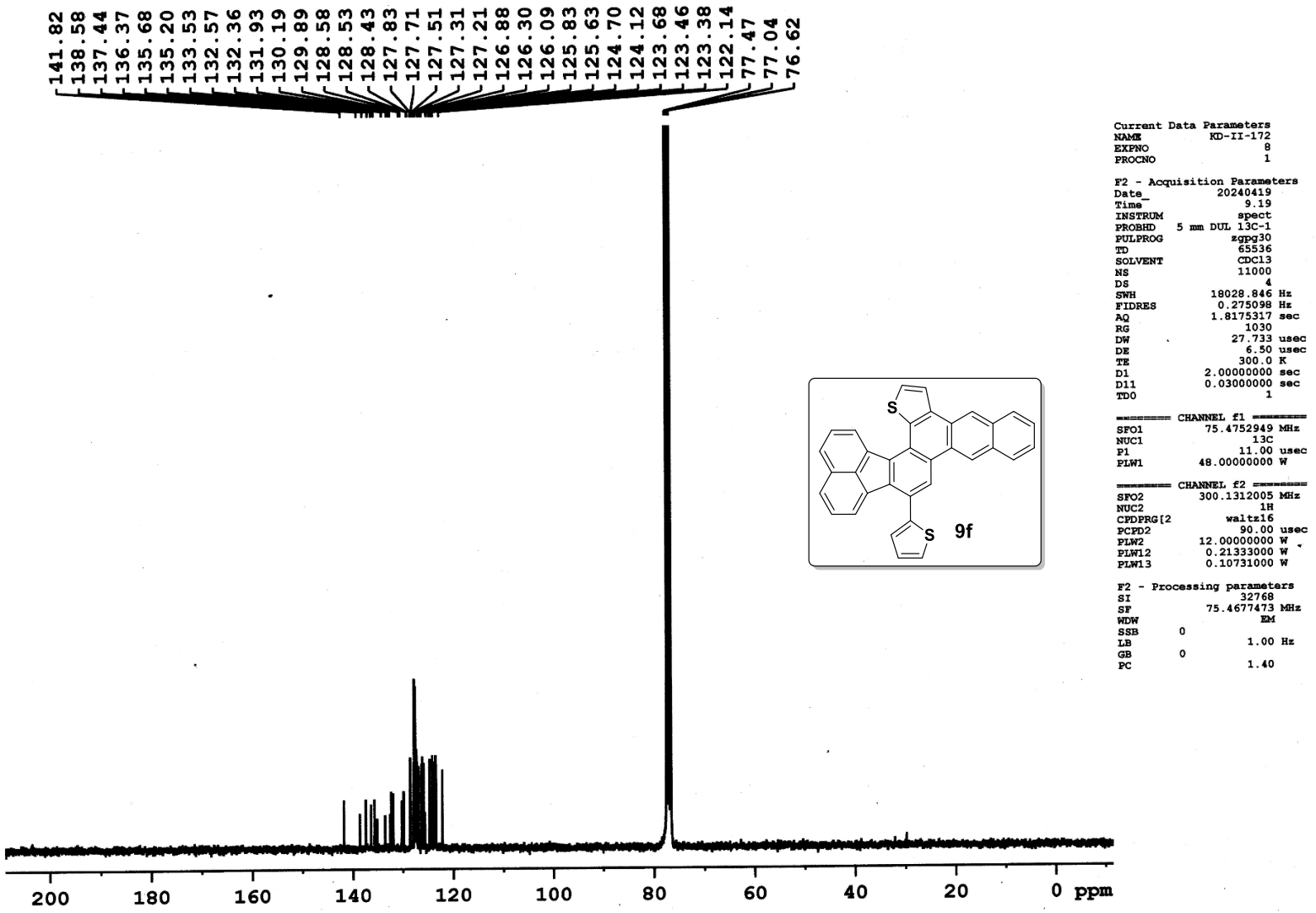
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9e'**



HRMS spectrum of compound **9e'**

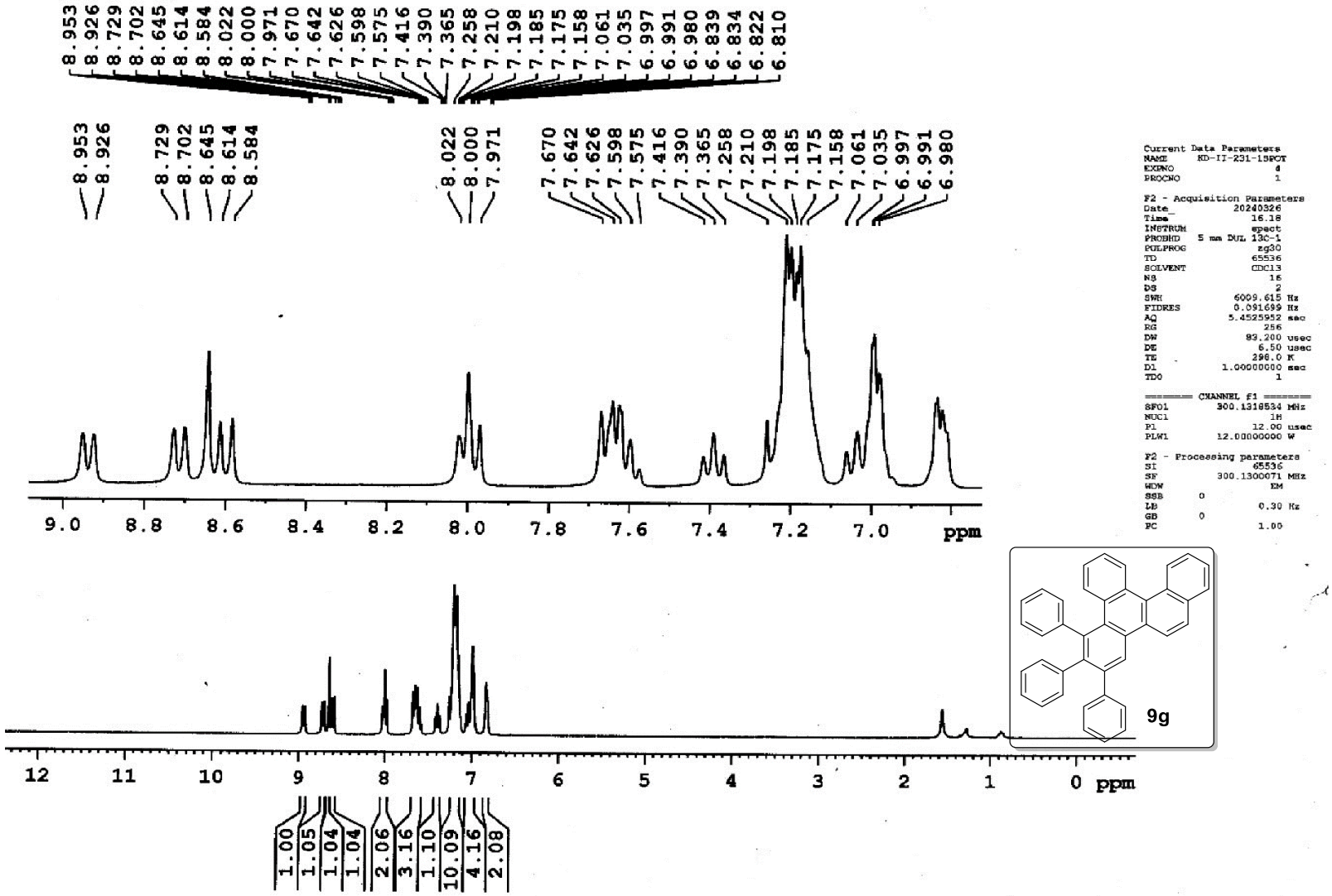


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9f**

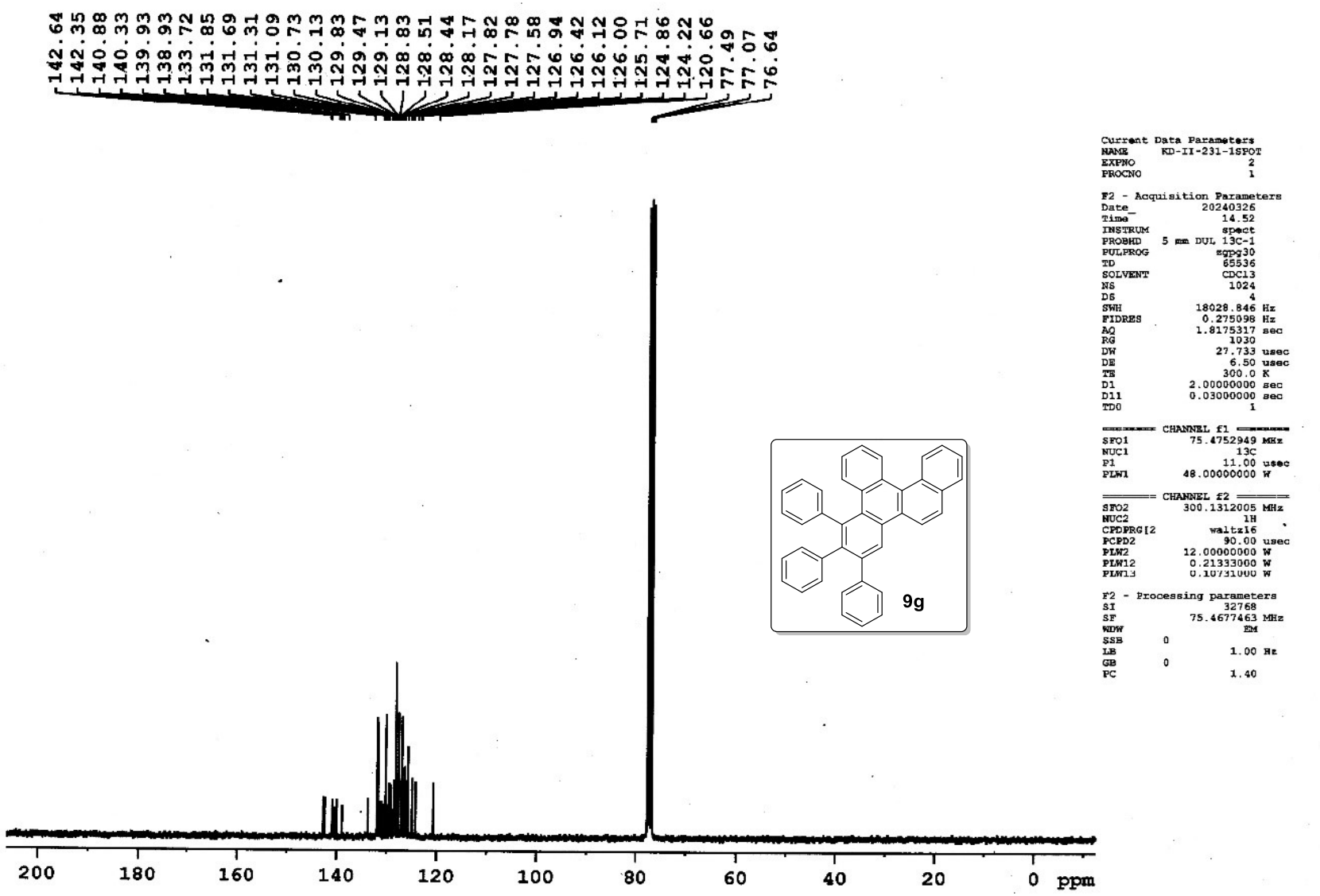



```
Current Data Parameters
NAME                  KD-II-172
EXPNO                         8
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240419
Time                       9.19
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                         11000
DS                             4
SWH                18028.846 Hz
FIDRES              0.275098 Hz
AQ                  1.8175317 sec
RG                          1030
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                       13C
P1                    11.00 usec
PLW1               48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                 90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

F2 - Processing parameters
SI                         32768
SF                 75.4677473 MHz
WDW                           EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9f**

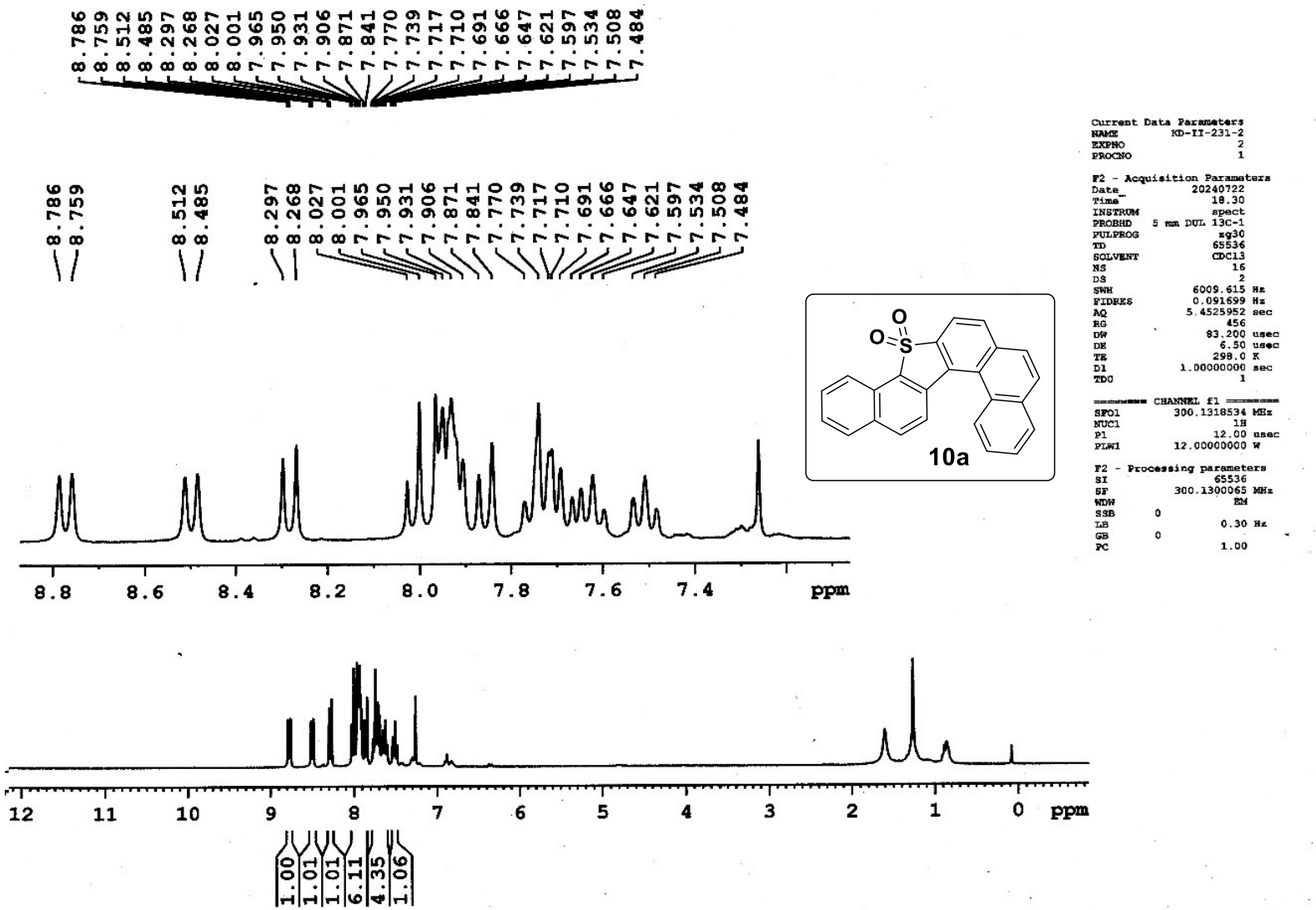


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9g**

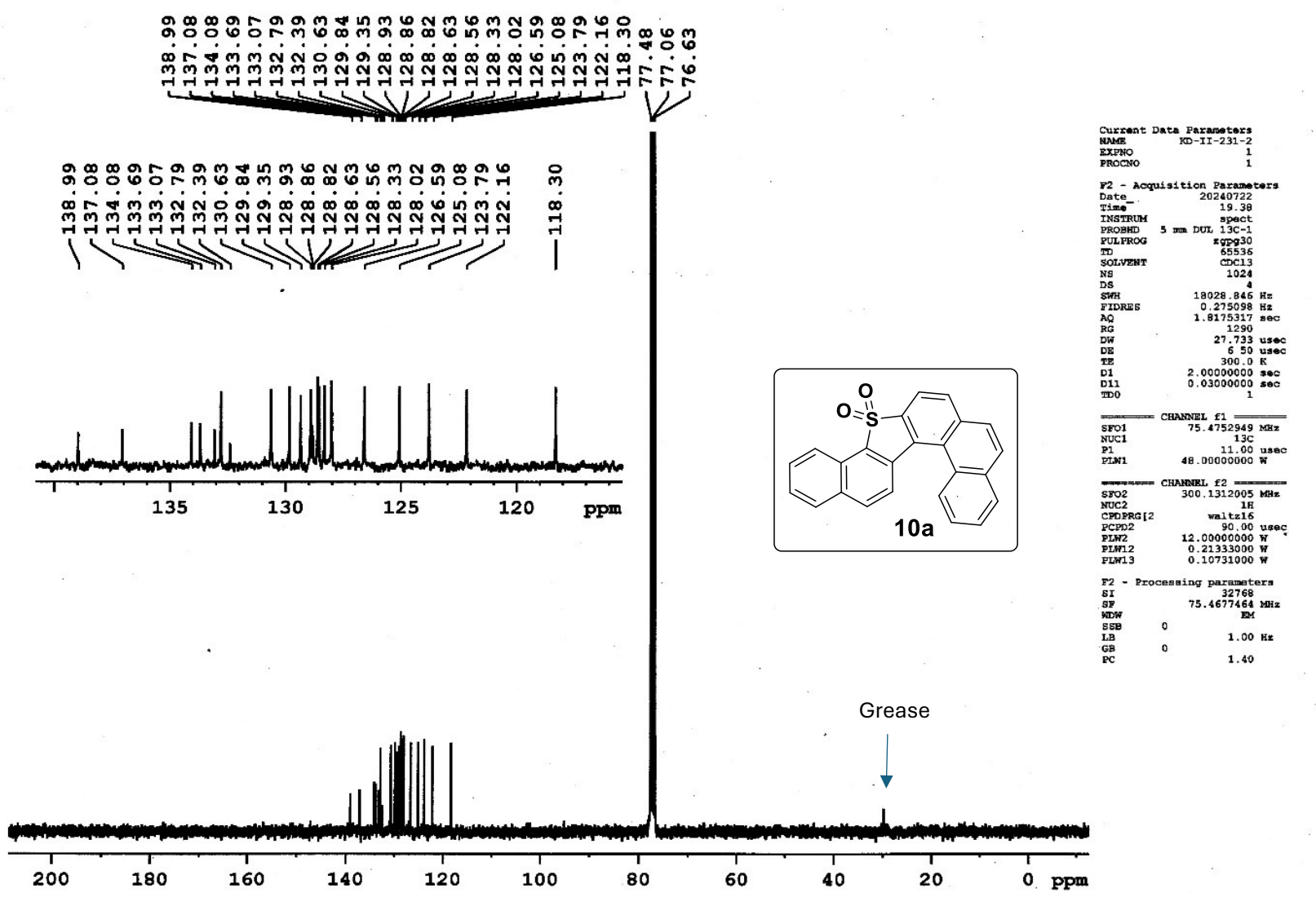



```
Current Data Parameters
NAME      KD-II-231-1SPOT
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240326
Time              14.52
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          18028.846 Hz
FIDRES        0.275098 Hz
AQ           1.8175317 sec
RG                1030
DW              27.733 usec
DE               6.50 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        75.4752949 MHz
NUC1               13C
P1               11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2       300.1312005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI               32768
SF          75.4677463 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9g**

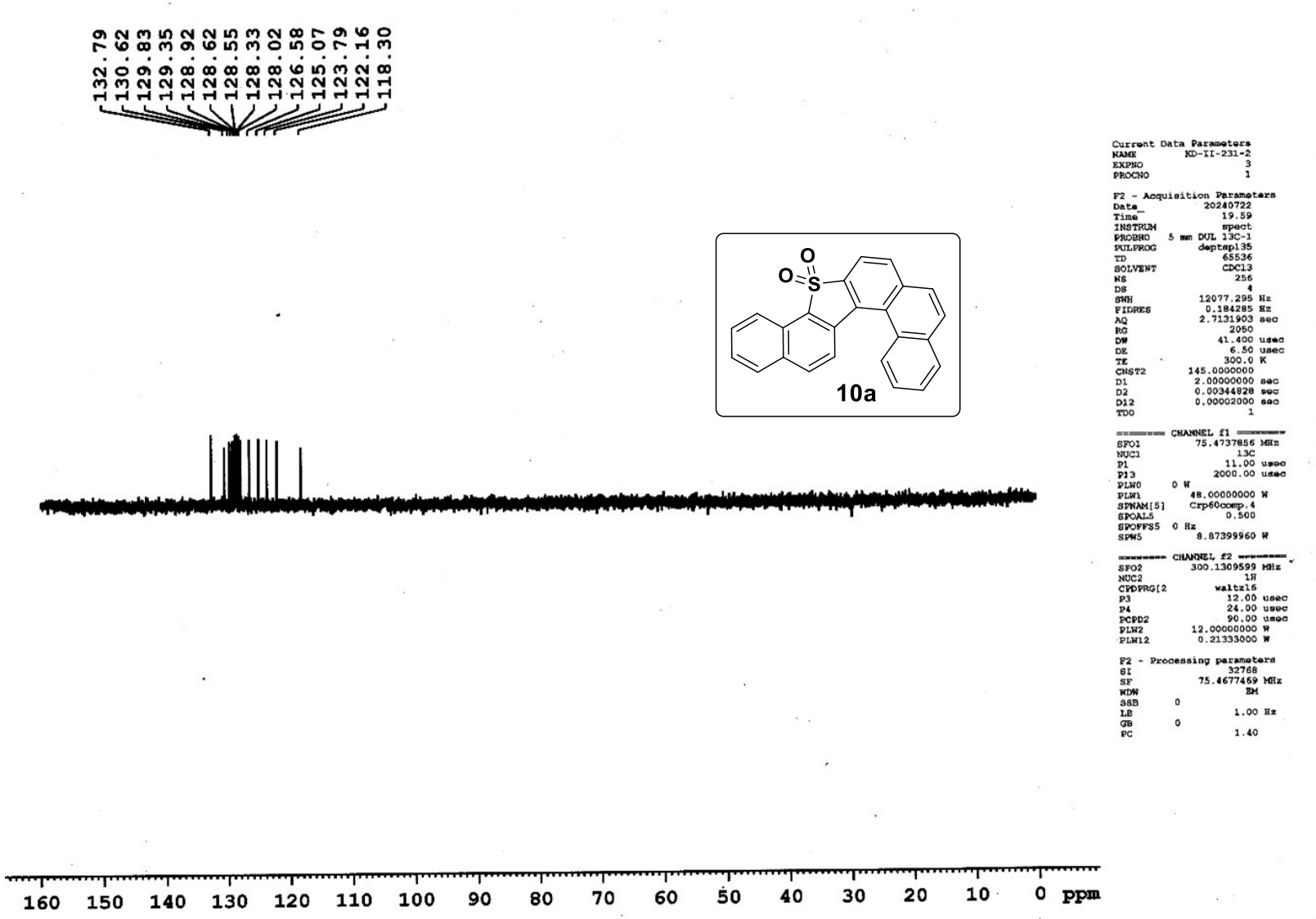


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **10a**

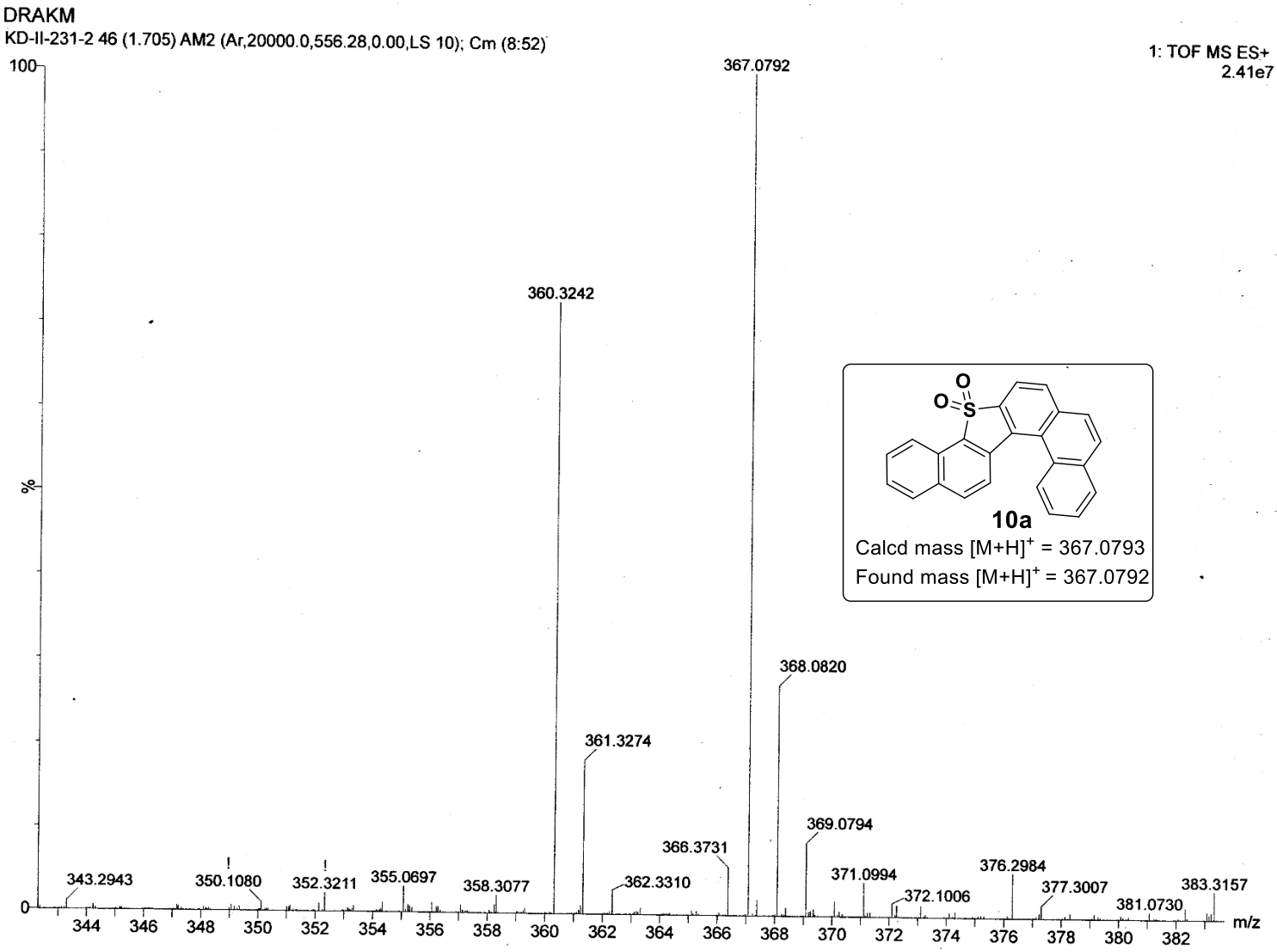



```
Current Data Parameters
NAME          KD-II-231-2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          20240722
Time_             19.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175317 sec
RG                 1290
DW               27.733 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1               13C
P1               11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI                32768
SF          75.4677464 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

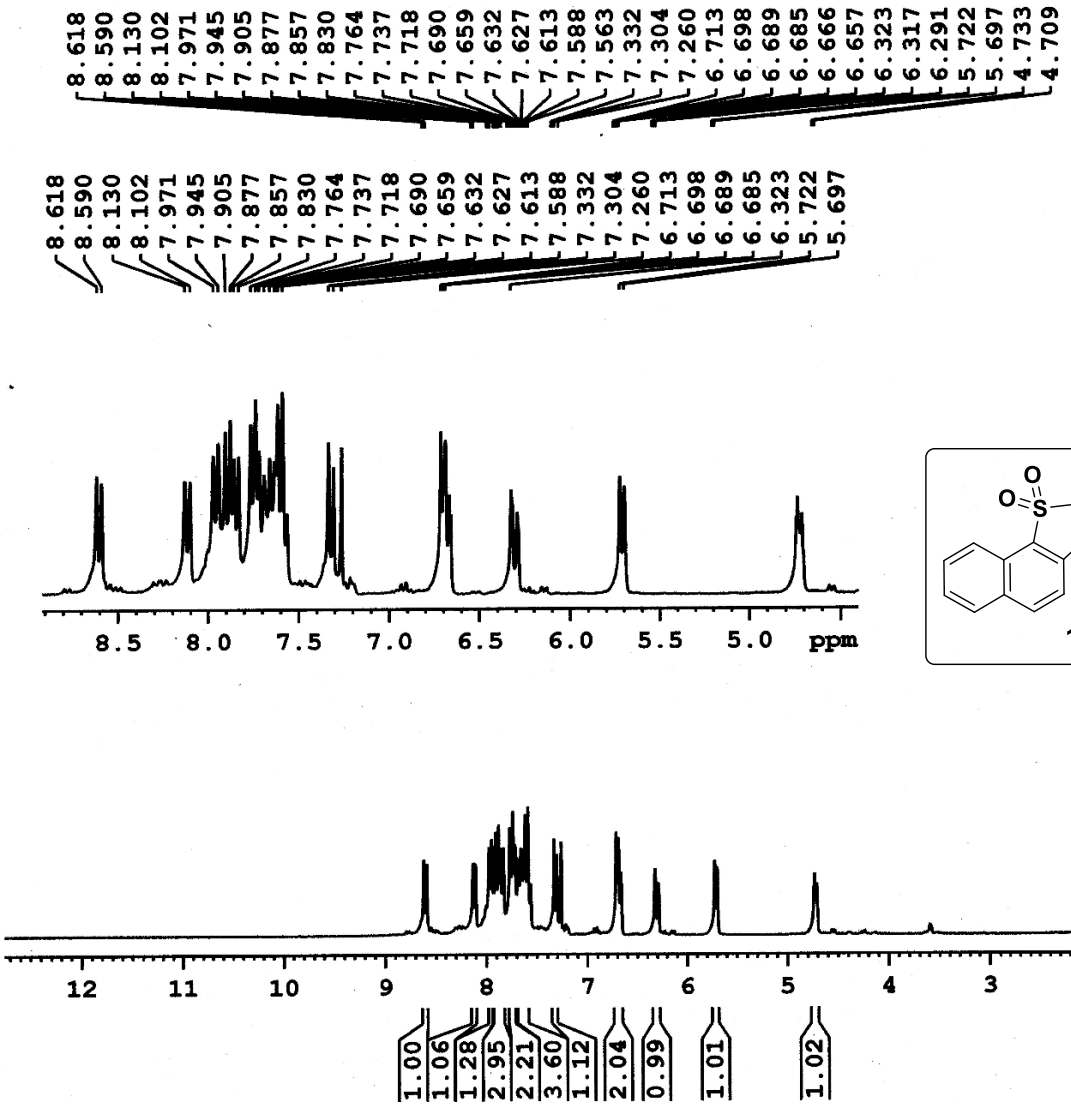
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **10a**

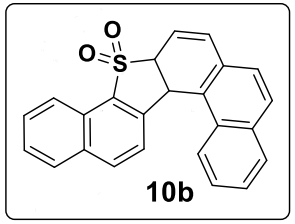


DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **10a**



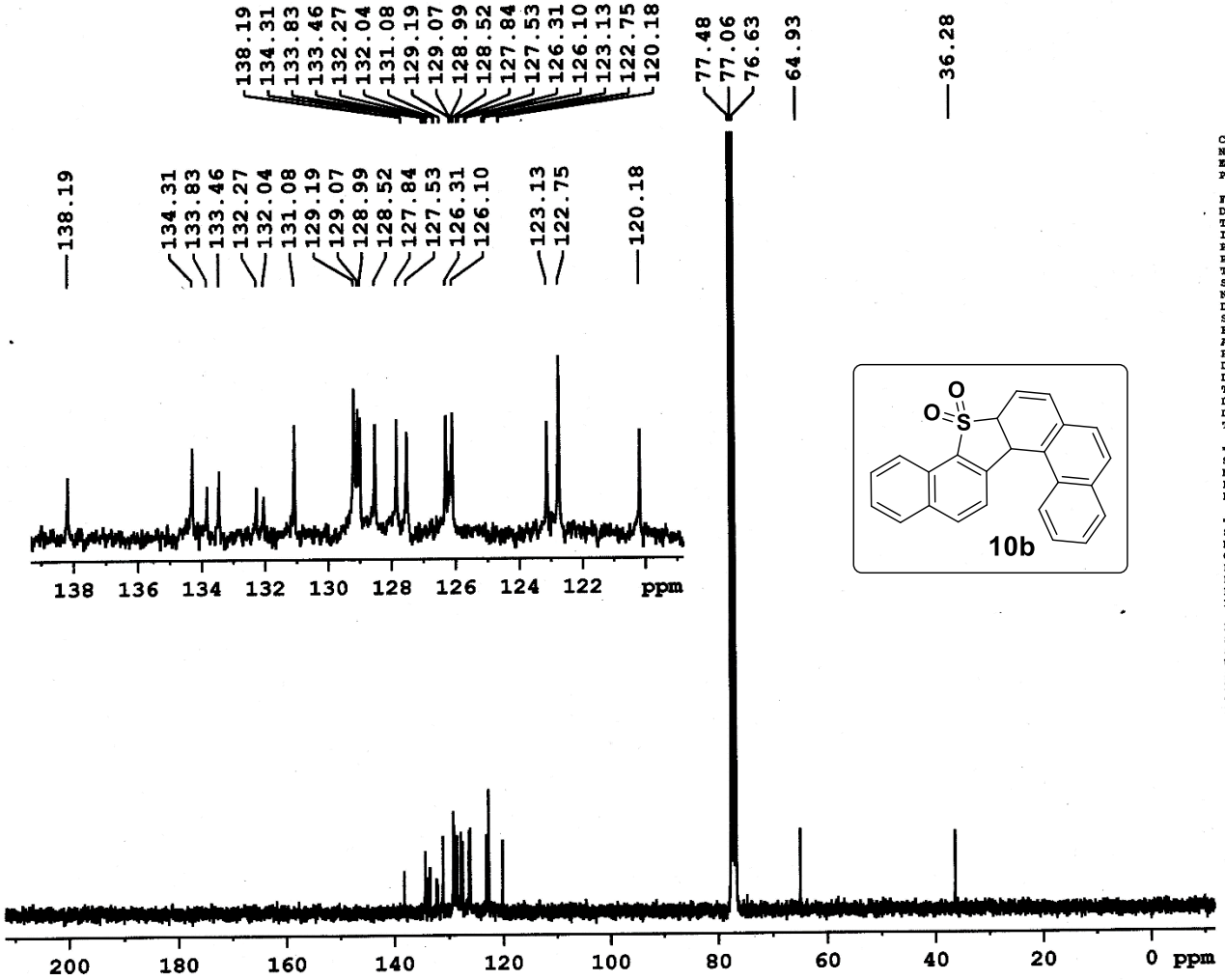
HRMS spectrum of compound **10a**





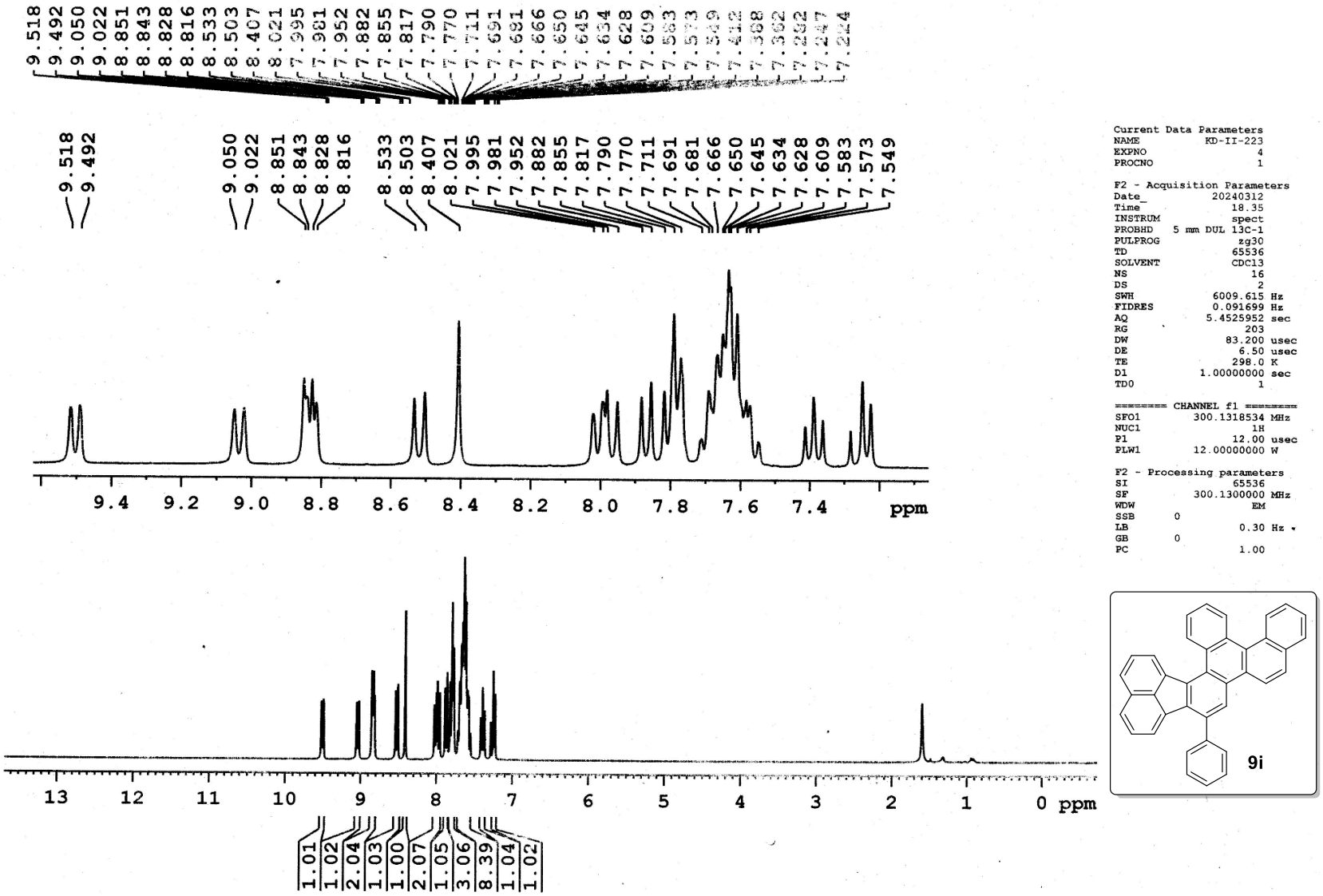

```
Current Data Parameters
NAME                  KD-II-285
EXPNO                         5
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240810
Time                       9.56
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 6009.615 Hz
FIDRES              0.091699 Hz
AQ                  5.4525952 sec
RG                         287
DW                    83.200 usec
DE                     6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               300.1318534 MHz
NUC1                       1H
P1                      12.00 usec
PLW1               12.00000000 W

F2 - Processing parameters
SI                       65536
SF                 300.1300062 MHz
WDW                         EM
SSB                  0
LB                      0.30 Hz
GB                   0
PC                      1.00
```

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **10b**

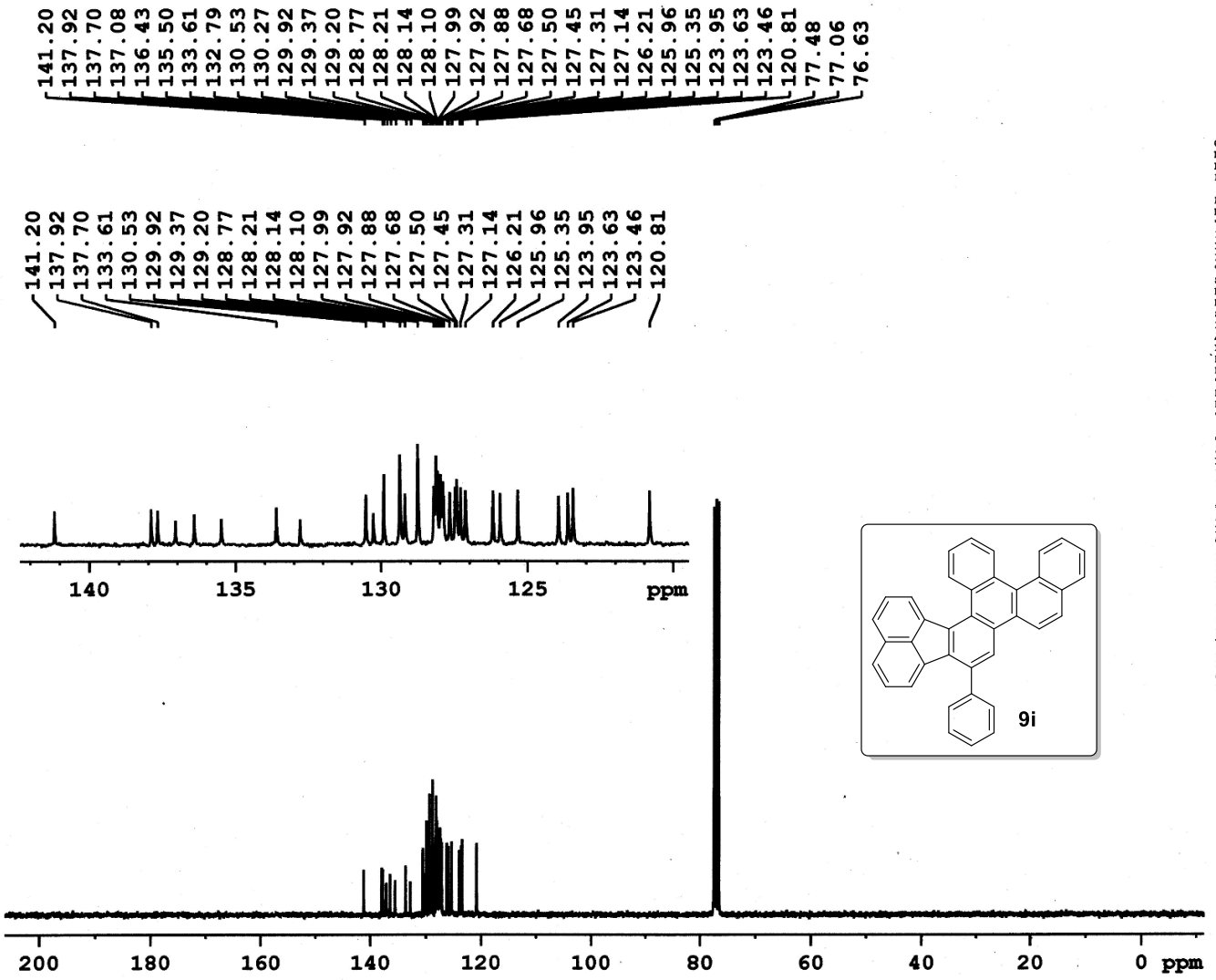



```
Current Data Parameters
NAME                  KD-II-285
EXPNO                         6
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240812
Time                      14.30
INSTRUM                   spect
PROBHD    5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                   1.8175317 sec
RG                        1030
DW                      27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

========== CHANNEL f1 ==========
SFO1                75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                48.00000000 W

========== CHANNEL f2 ==========
SFO2               300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W
PLW13                0.10731000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677479 MHz
WDW                           EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **10b**

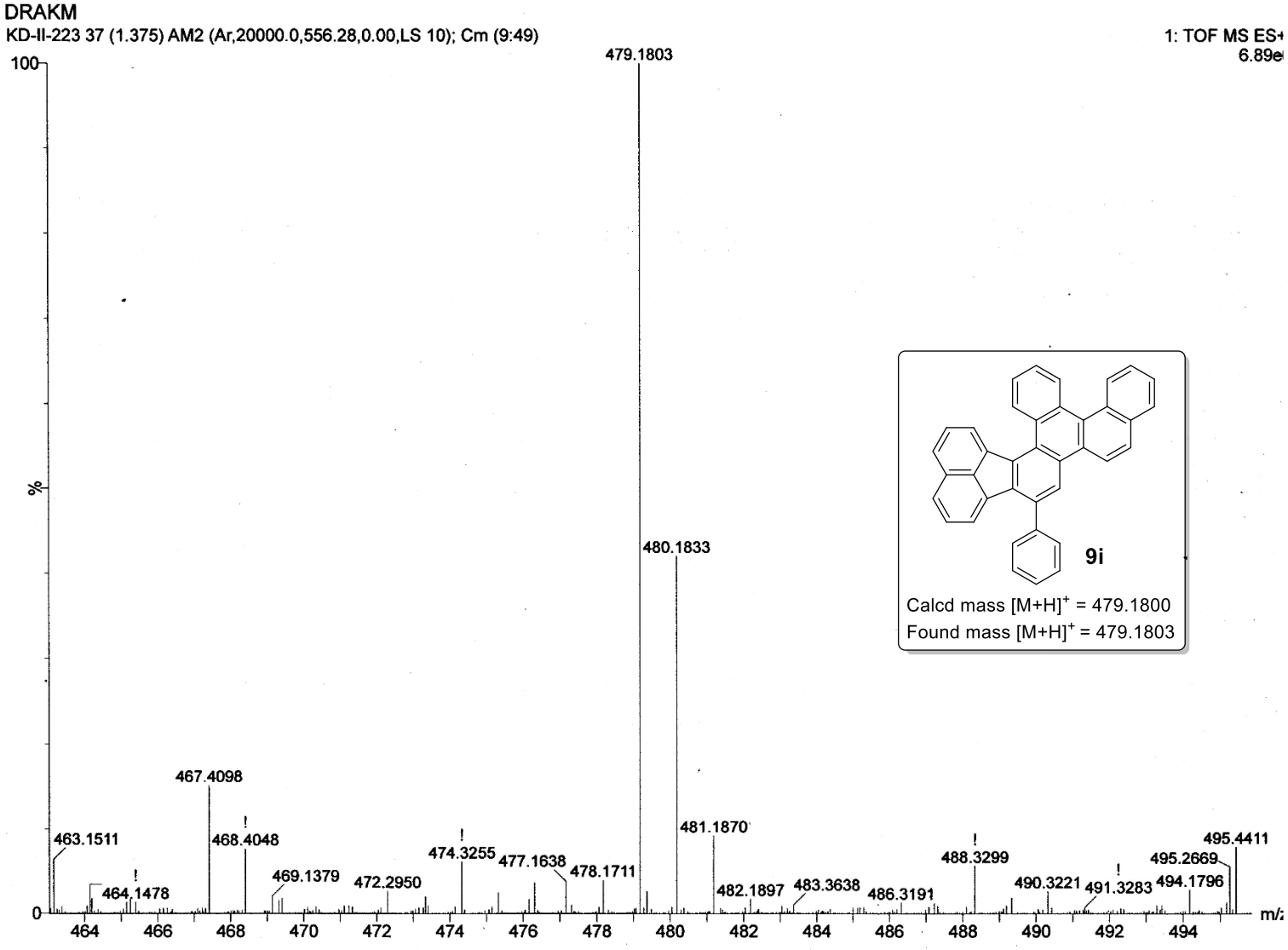


^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9i**

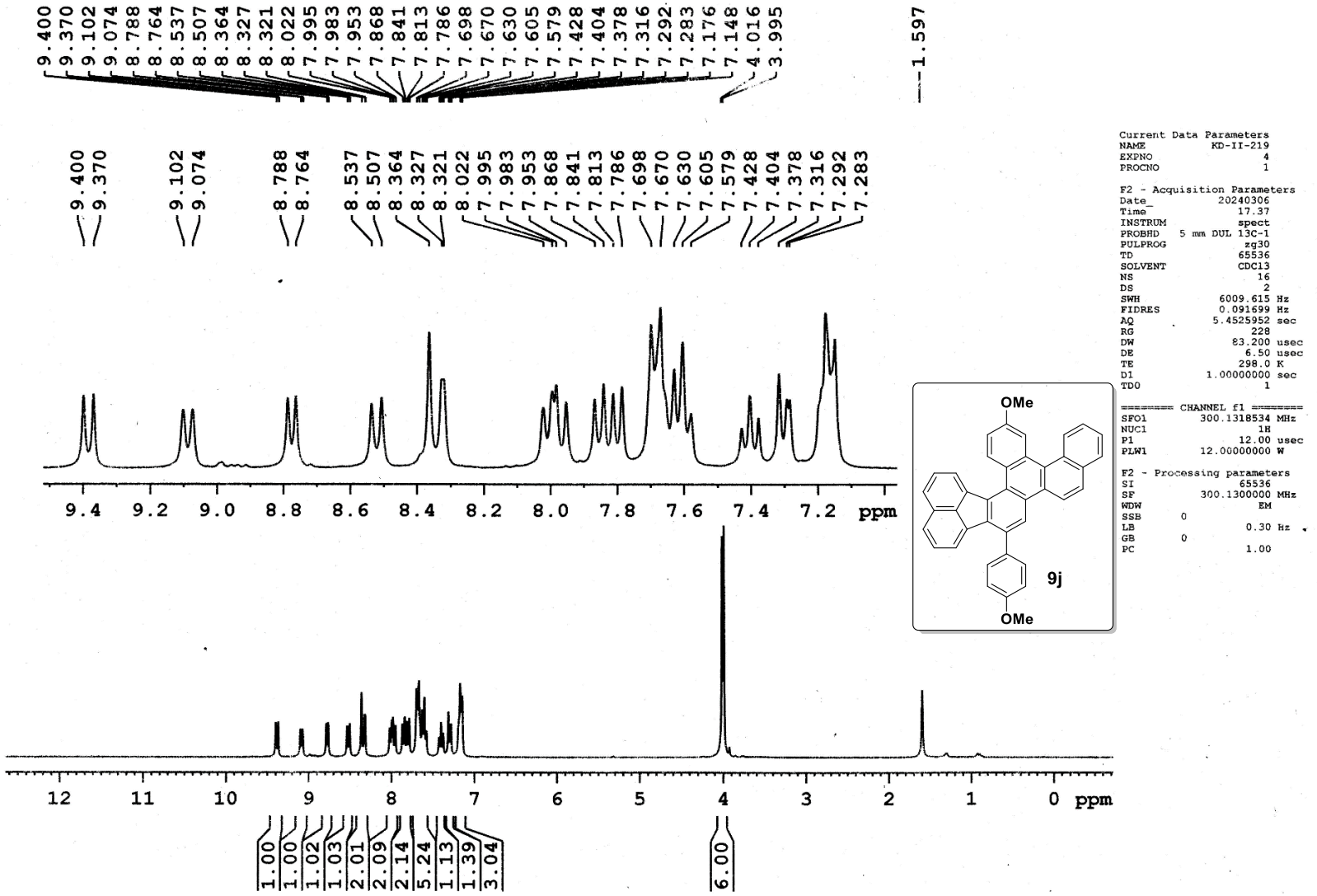



```
Current Data Parameters
NAME                  KD-II-223
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240312
Time                      17.27
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                18028.846 Hz
FIDRES             0.275098 Hz
AQ                 1.8175317 sec
RG                          645
DW                    27.733 usec
DE                     6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                      13C
P1                     11.00 usec
PLW1              48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                  90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W
PLW13              0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677485 MHz
WDW                          EM
SSB               0
LB                     1.00 Hz
GB                0
PC                     1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9i**



HRMS spectrum of compound **9i**



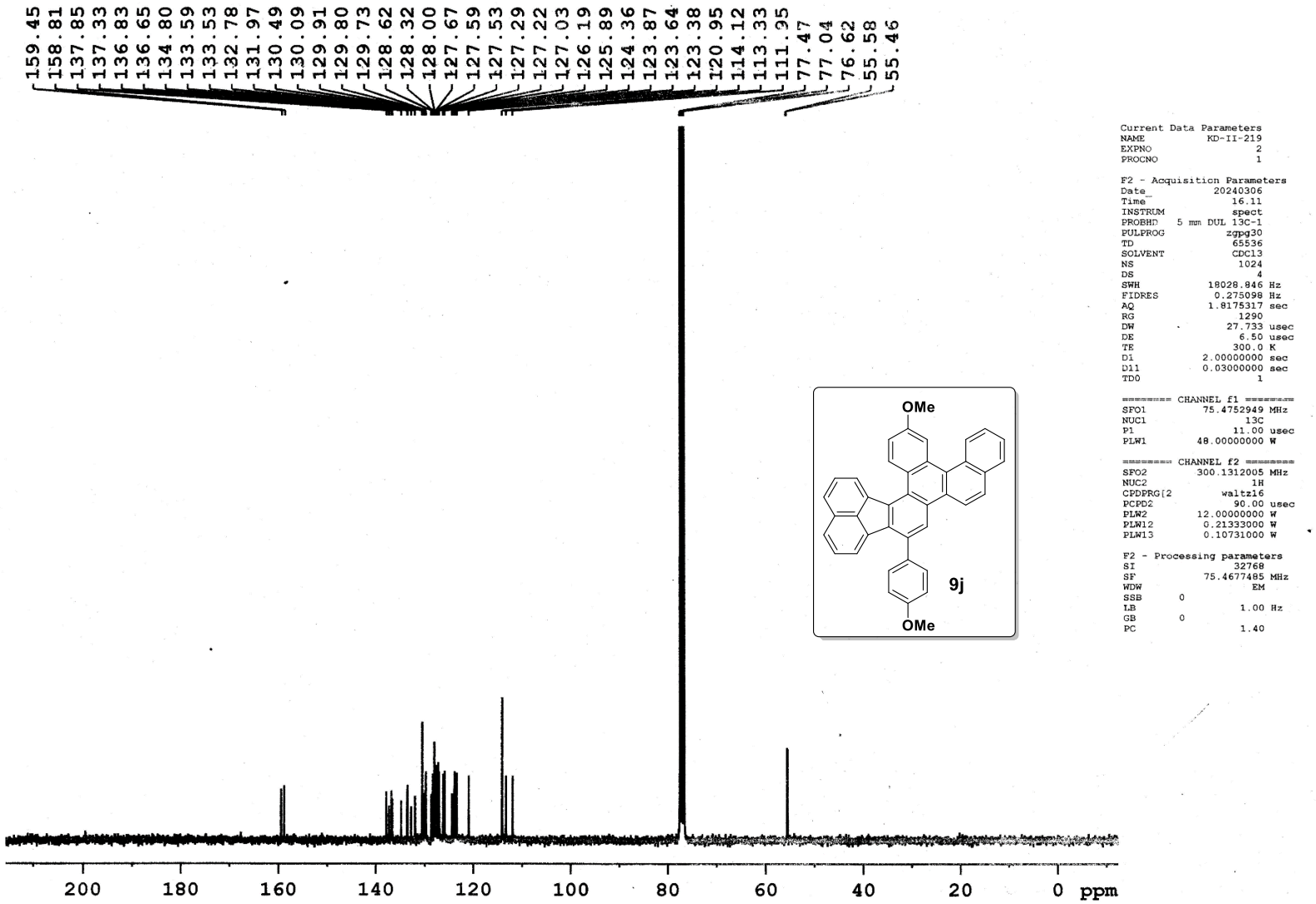
Current Data Parameters
NAME KD-II-219
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240306
Time 17.37
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 228
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 12.00 usec
PLW1 12.00000000 W

F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **9j**



Current Data Parameters
NAME KD-II-219
EXPNO 2
PROCNO 1

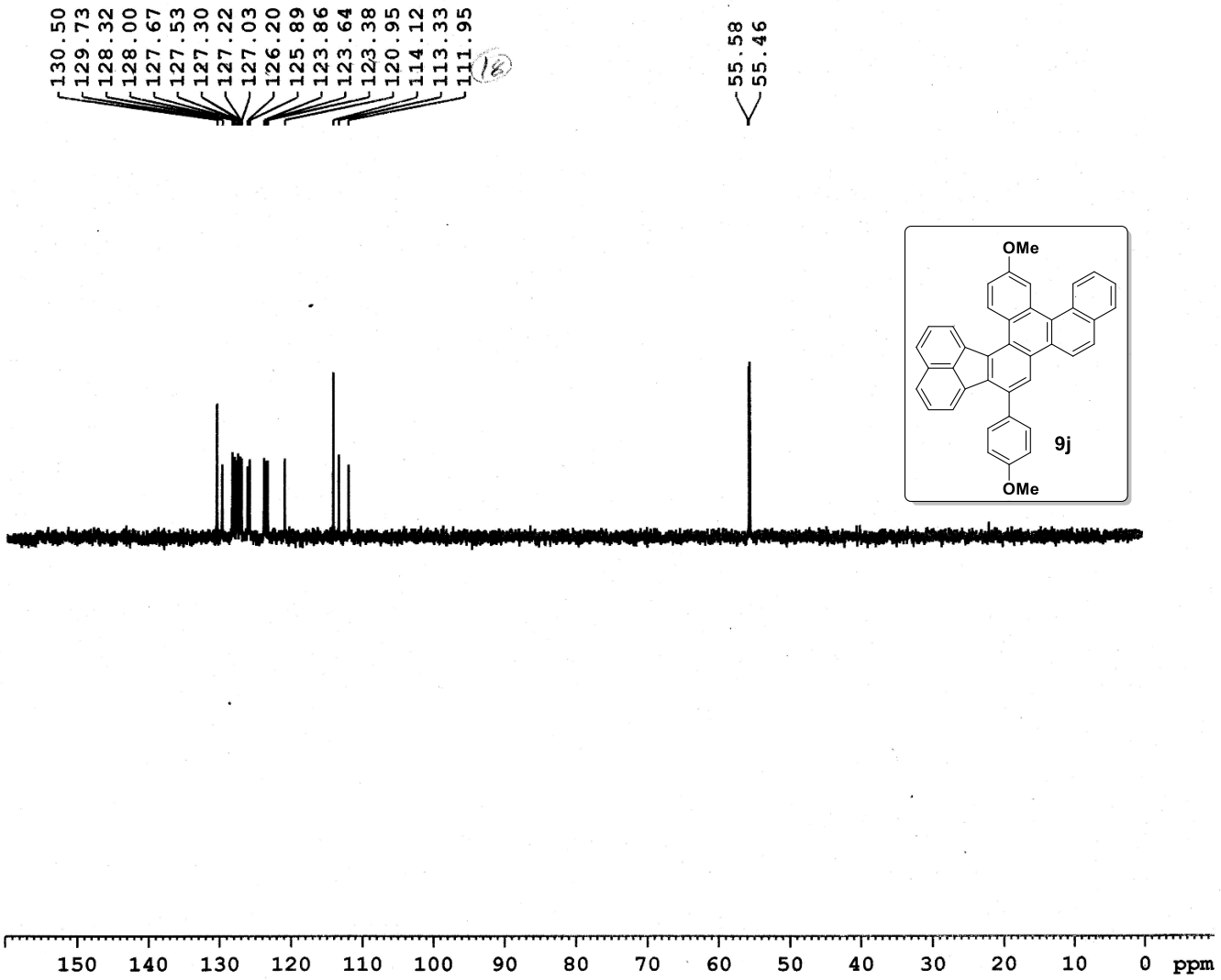
F2 - Acquisition Parameters
Date_ 20240306
Time 16.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 1290
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13C
P1 11.00 usec
PLW1 48.00000000 W

======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.21333000 W
PLW13 0.10731000 W

F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

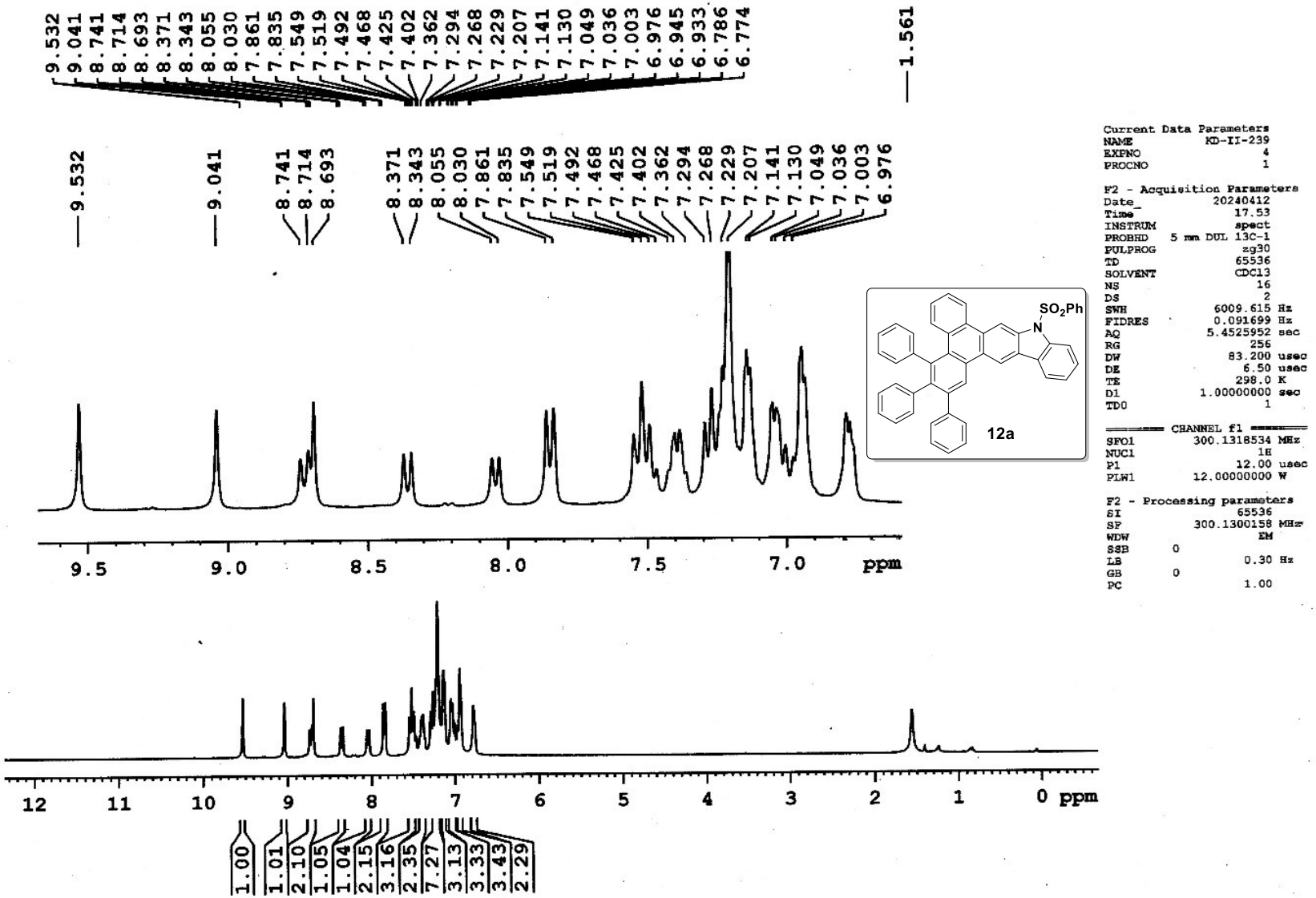
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **9j**




```
Current Data Parameters
NAME                  KD-II-219
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_              20240306
Time                   17.35
INSTRUM                spect
PROBHD   5 mm DUL 13C-1
PULPROG            deptsp135
TD                     65536
SOLVENT                CDCl3
NS                       256
DS                         4
SWH                12077.295 Hz
FIDRES             0.184285 Hz
AQ                 2.7131903 sec
RG                      2050
DW                    41.400 usec
DE                     6.50 usec
TE                    300.0 K
CNST2             145.0000000
D1                  2.00000000 sec
D2                  0.00344828 sec
D12                 0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               75.4737856 MHz
NUC1                       13C
P1                       11.00 usec
P13                    2000.00 usec
PLW0        0 W
PLW1              48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                  0.500
SPOFFS5     0 Hz
SPW5               8.87399960 W

======== CHANNEL f2 ========
SFO2              300.1309599 MHz
NUC2                       1H
CPDPRG[2               waltz16
P3                       12.00 usec
P4                       24.00 usec
PCPD2                    90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W

F2 - Processing parameters
SI                         32768
SF                 75.4677485 MHz
WDW                           EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **9j**

9. ^{1}H, ^{13}C {^{1}H}, DEPT 135 (selected) NMR & HRMS (selected) spectra of phenanthrene fused carbazoles **12a-h**



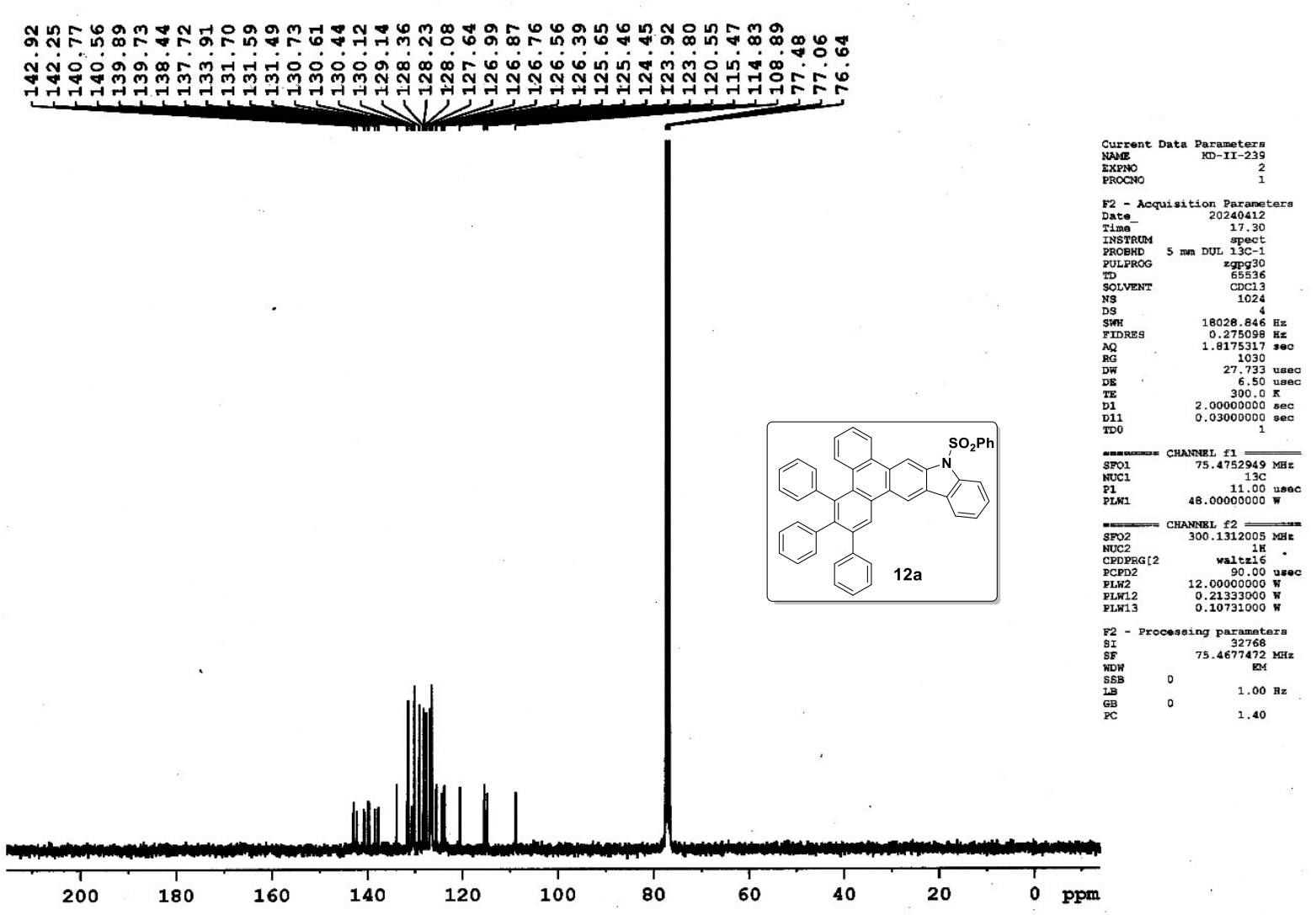
Current Data Parameters
NAME KD-II-239
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240412
Time 17.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 256
DW 83.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
SFO1 300.1318534 MHz
NUC1 1H
P1 12.00 usec
PLW1 12.00000000 W

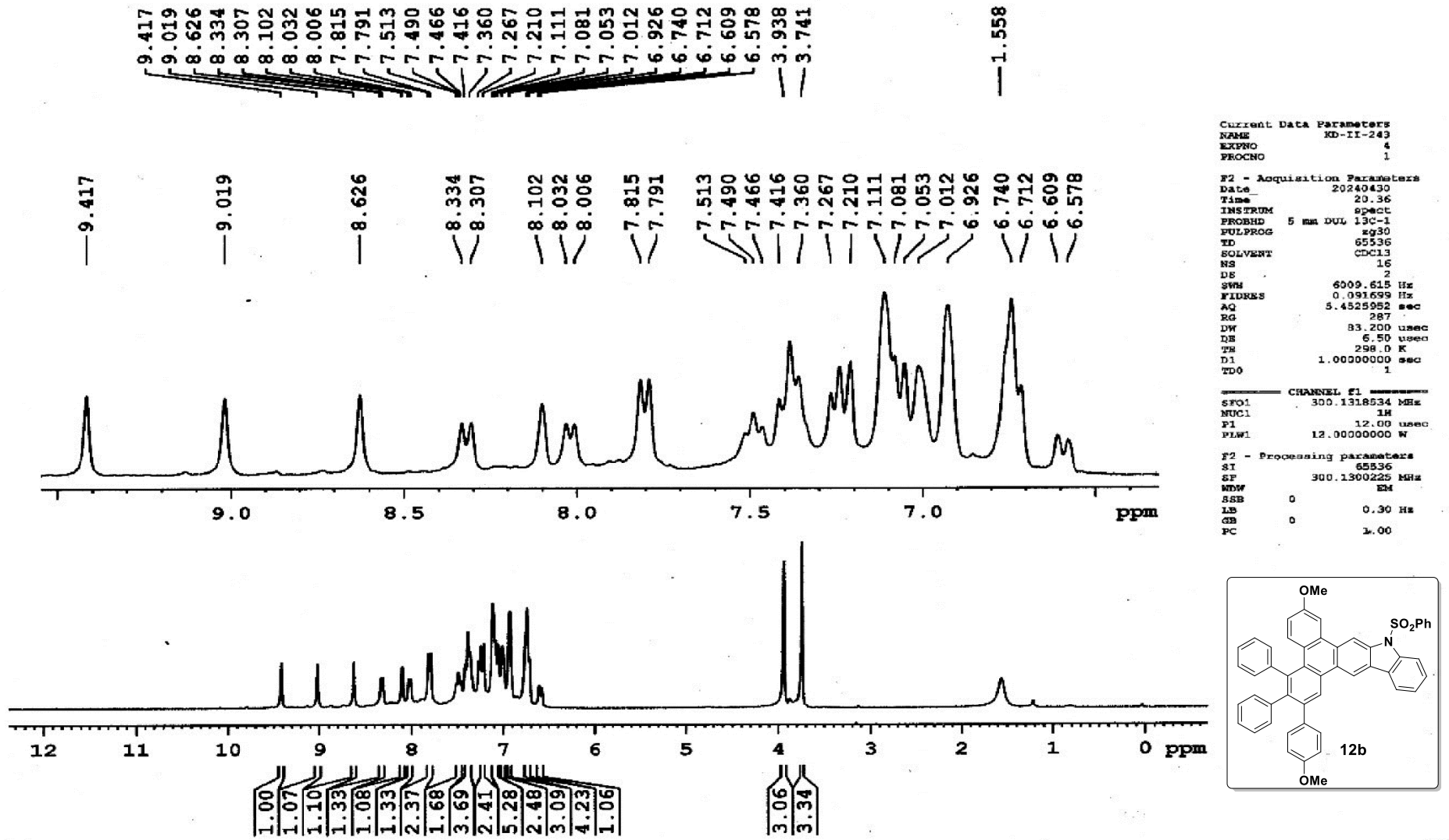
F2 - Processing parameters
SI 65536
SF 300.1300158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12a**

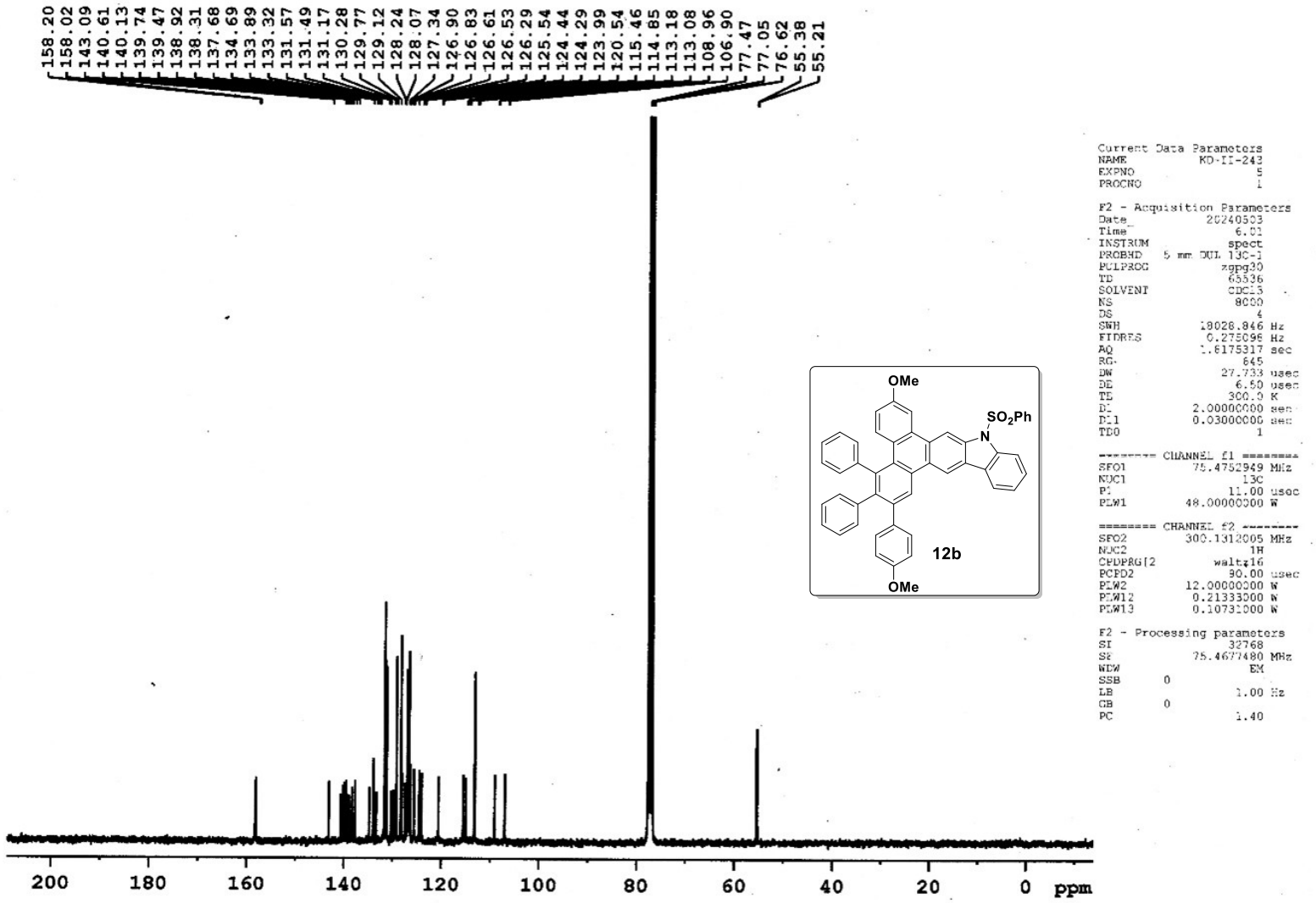



```
Current Data Parameters
NAME                  KD-II-239
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20240412
Time                      17.30
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  18028.846 Hz
FIDRES                0.275098 Hz
AQ                   1.8175317 sec
RG                        1030
DW                      27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 75.4752949 MHz
NUC1                        13C
P1                        11.00 usec
PLW1                48.00000000 W

======== CHANNEL f2 ========
SFO2                300.1312005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                12.00000000 W
PLW12                0.21333000 W
PLW13                0.10731000 W

F2 - Processing parameters
SI                         32768
SF                  75.4677472 MHz
WDW                           EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12a**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12b**

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Current Data Parameters
NAME                  KD-II-243
EXPNO                         5
PROCNO                        1

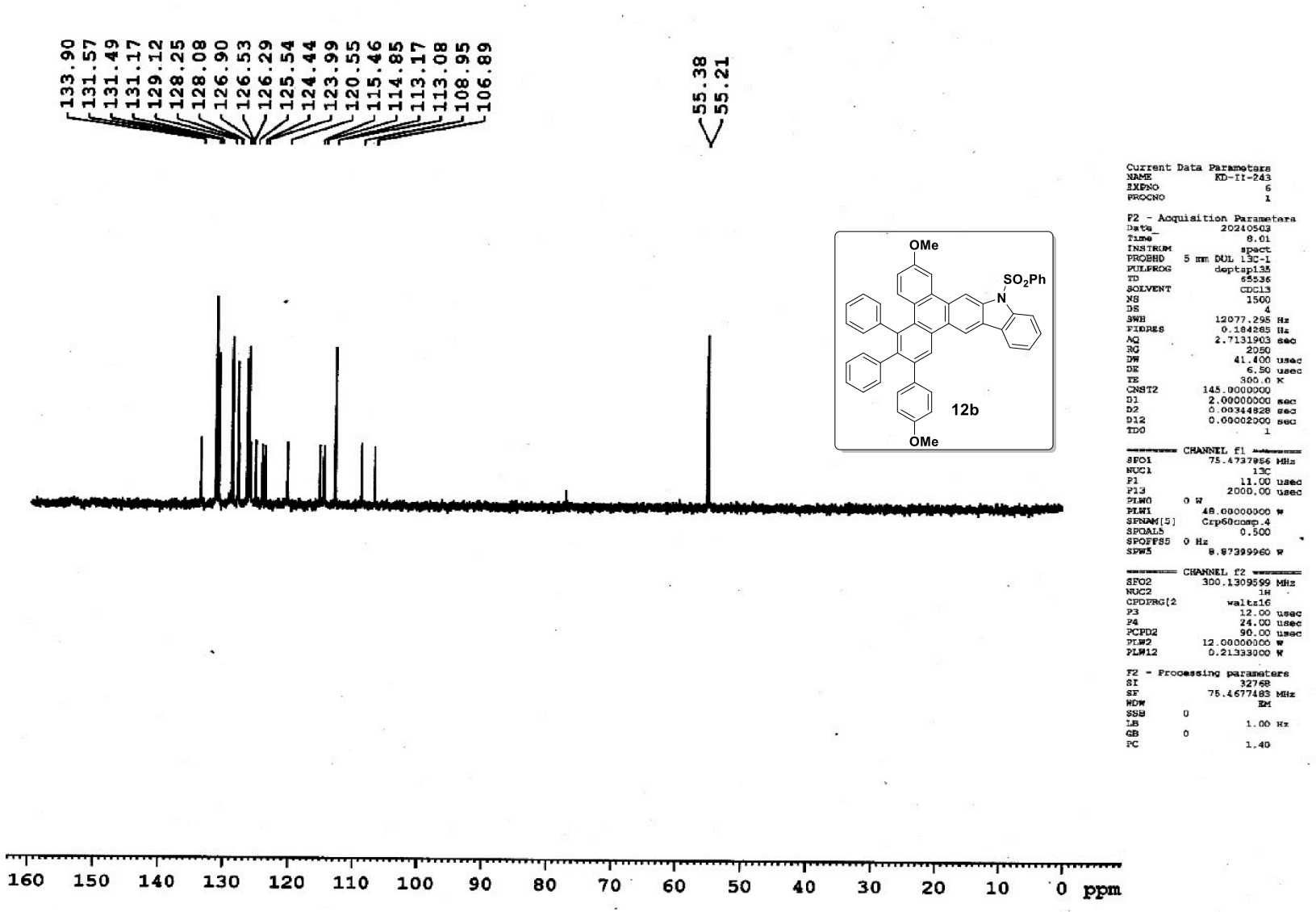
F2 - Acquisition Parameters
Date_                  20240503
Time                       6.01
INSTRUM                    spect
PROBHD    5 mm DUL 13C-1
PULPROG                zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          8000
DS                             4
SWH                    18028.846 Hz
FIDRES                 0.275096 Hz
AQ                     1.8175317 sec
RG                           645
DW                        27.733 usec
DE                         6.50 usec
TE                        300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                  75.4752949 MHz
NUC1                          13C
P1                          11.00 usec
PLW1                 48.00000000 W

======== CHANNEL f2 ========
SFO2                 300.1312005 MHz
NUC2                          1H
CPDPRG[2                  waltz16
PCPD2                       90.00 usec
PLW2                 12.00000000 W
PLW12                 0.21333000 W
PLW13                 0.10731000 W

F2 - Processing parameters
SI                            32768
SF                    75.4677480 MHz
WDW                              EM
SSB                   0
LB                            1.00 Hz
GB                    0
PC                            1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12b**




```
Current Data Parameters
NAME            KD-II-243
EXPNO                   6
PROCNO                  1

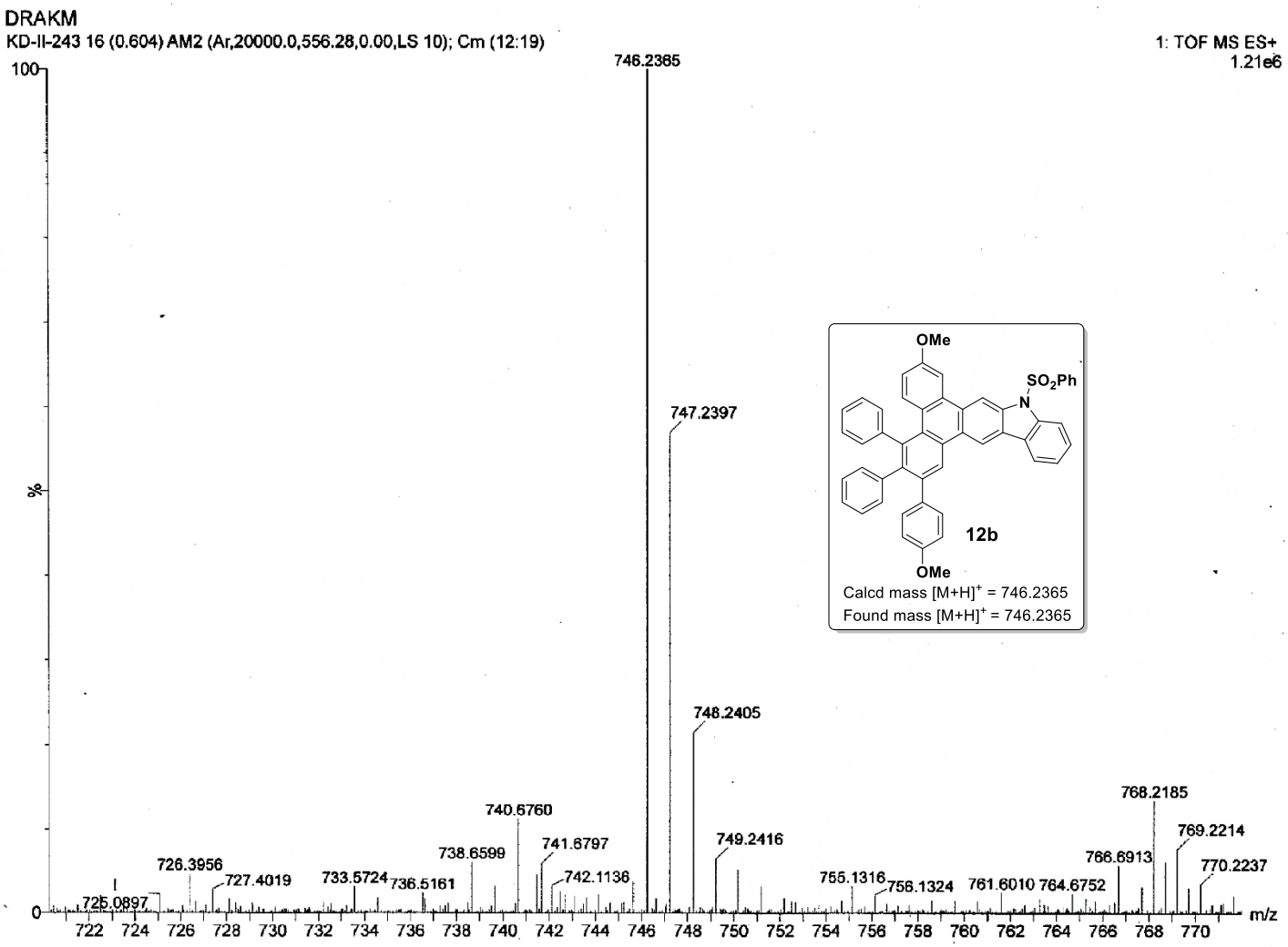
F2 - Acquisition Parameters
Date_          20240503
Time                8.01
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG         deptsp135
TD                  65536
SOLVENT             CDCl3
NS                   1500
DS                      4
SWH           12077.295 Hz
FIDRES        0.184285 Hz
AQ            2.7131903 sec
RG                   2050
DW                41.400 usec
DE                 6.50 usec
TE                300.0 K
CNST2         145.0000000
D1            2.00000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
TD0                     1

====== CHANNEL f1 ======
SFO1          75.4737956 MHz
NUC1                  13C
P1                 11.00 usec
P13              2000.00 usec
PLW0     0 W
PLW1        48.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5  0 Hz
SPW5           8.87399960 W

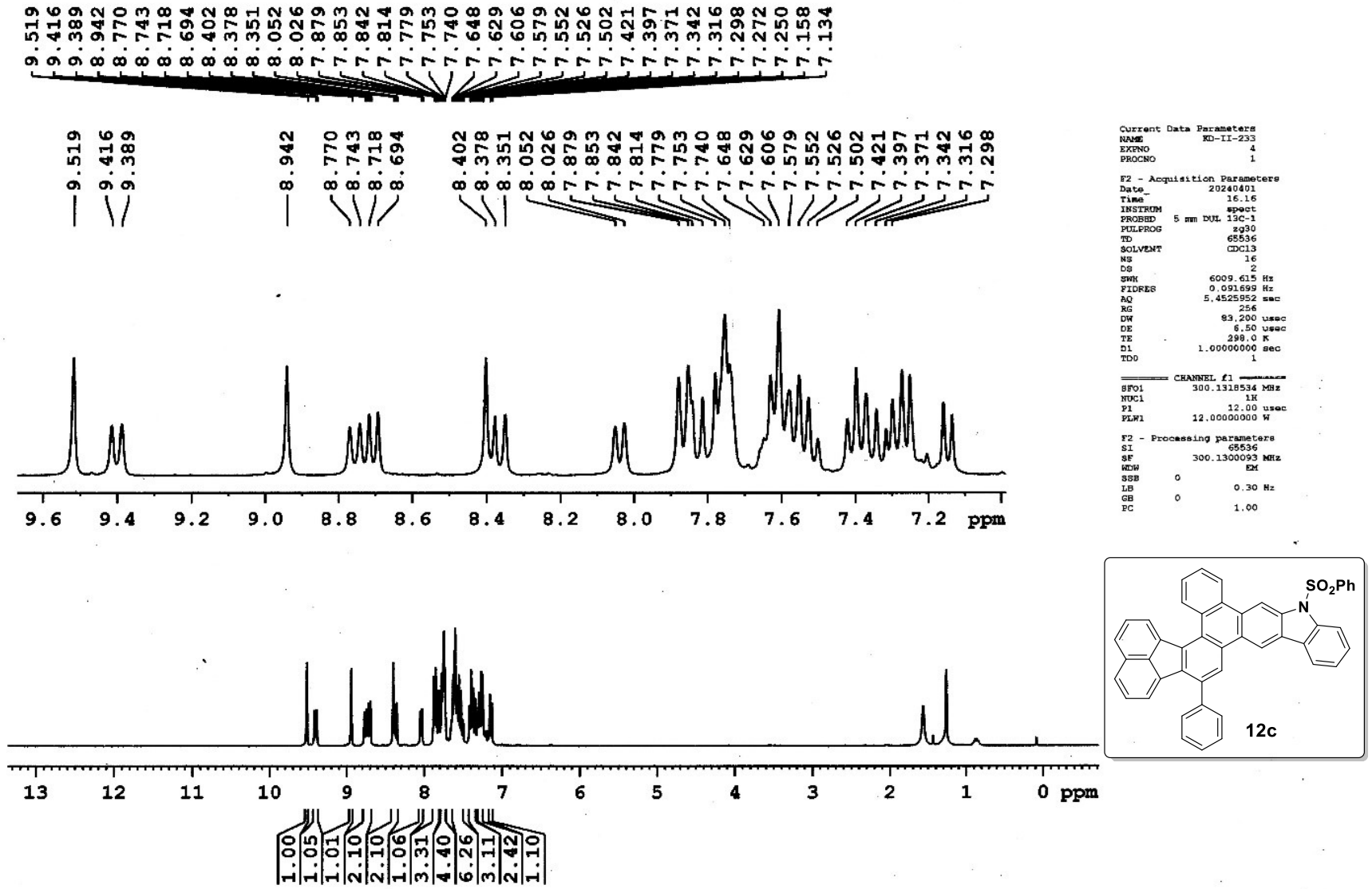
====== CHANNEL f2 ======
SFO2        300.1309599 MHz
NUC2                  1H
CPDPRG[2          waltz16
P3                 12.00 usec
P4                 24.00 usec
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W

F2 - Processing parameters
SI                  32768
SF            75.4677483 MHz
WDW                    EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

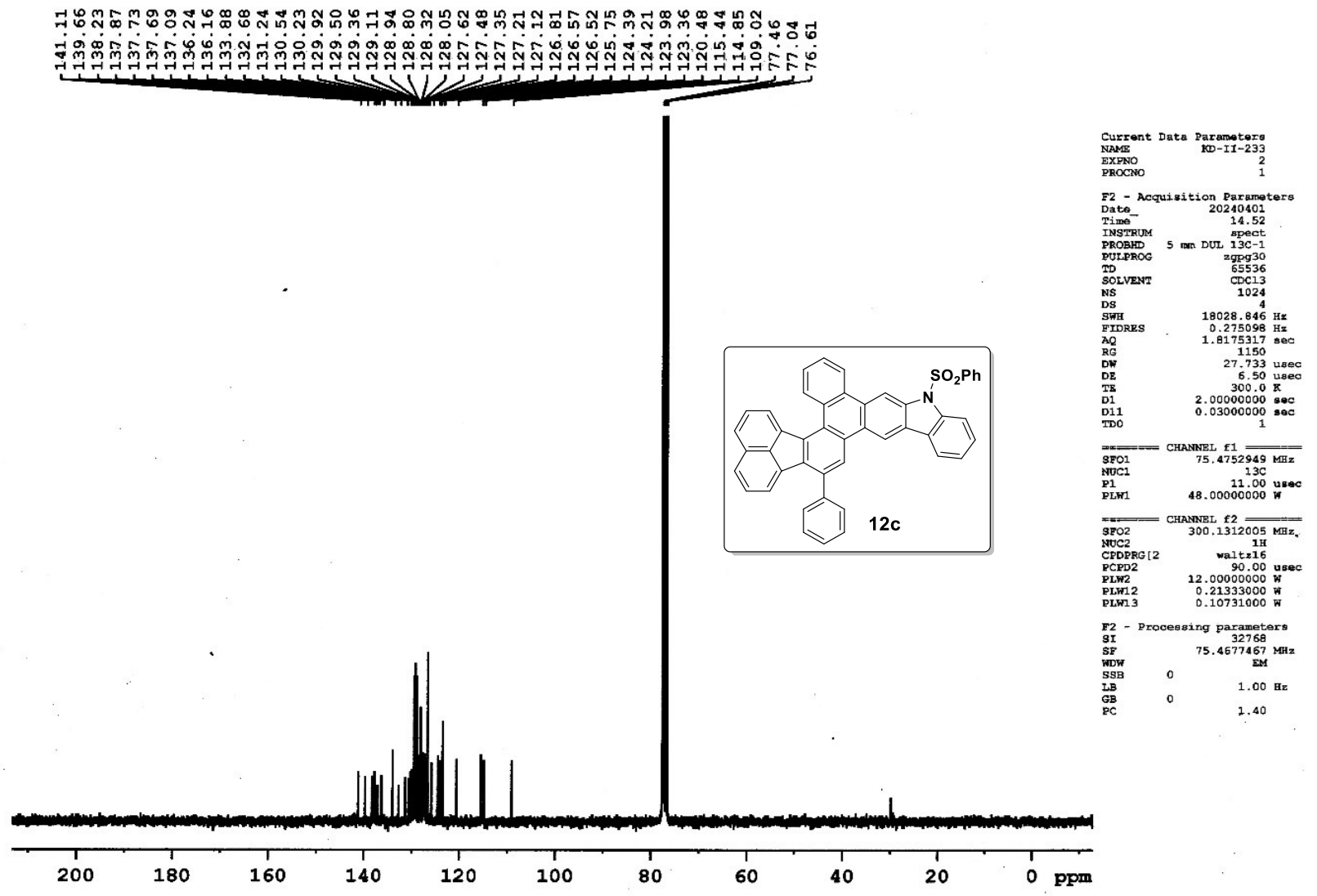
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **12b**



HRMS spectrum of compound **12b**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12c**




```
Current Data Parameters
NAME                  KD-II-233
EXPNO                         2
PROCNO                        1

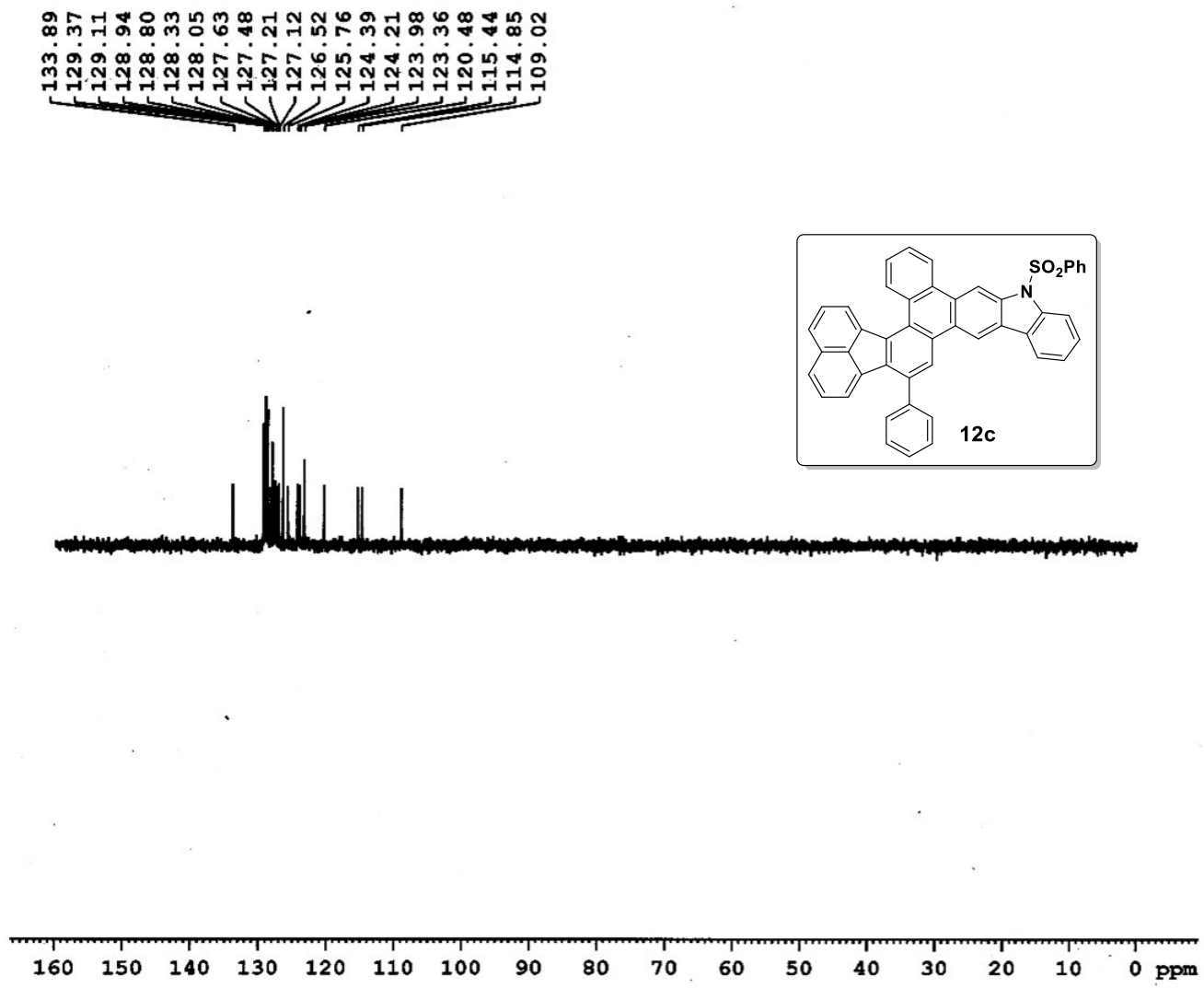
F2 - Acquisition Parameters
Date_              20240401
Time                    14.52
INSTRUM                 spect
PROBHD     5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                 18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175317 sec
RG                         1150
DW                       27.733 usec
DE                        6.50 usec
TE                       300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               75.4752949 MHz
NUC1                       13C
P1                       11.00 usec
PLW1               48.00000000 W

======== CHANNEL f2 ========
SFO2              300.1312005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

F2 - Processing parameters
SI                        32768
SF                 75.4677467 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12c**




```
Current Data Parameters
NAME                  KD-II-233
EXPNO                         3
PROCNO                        1

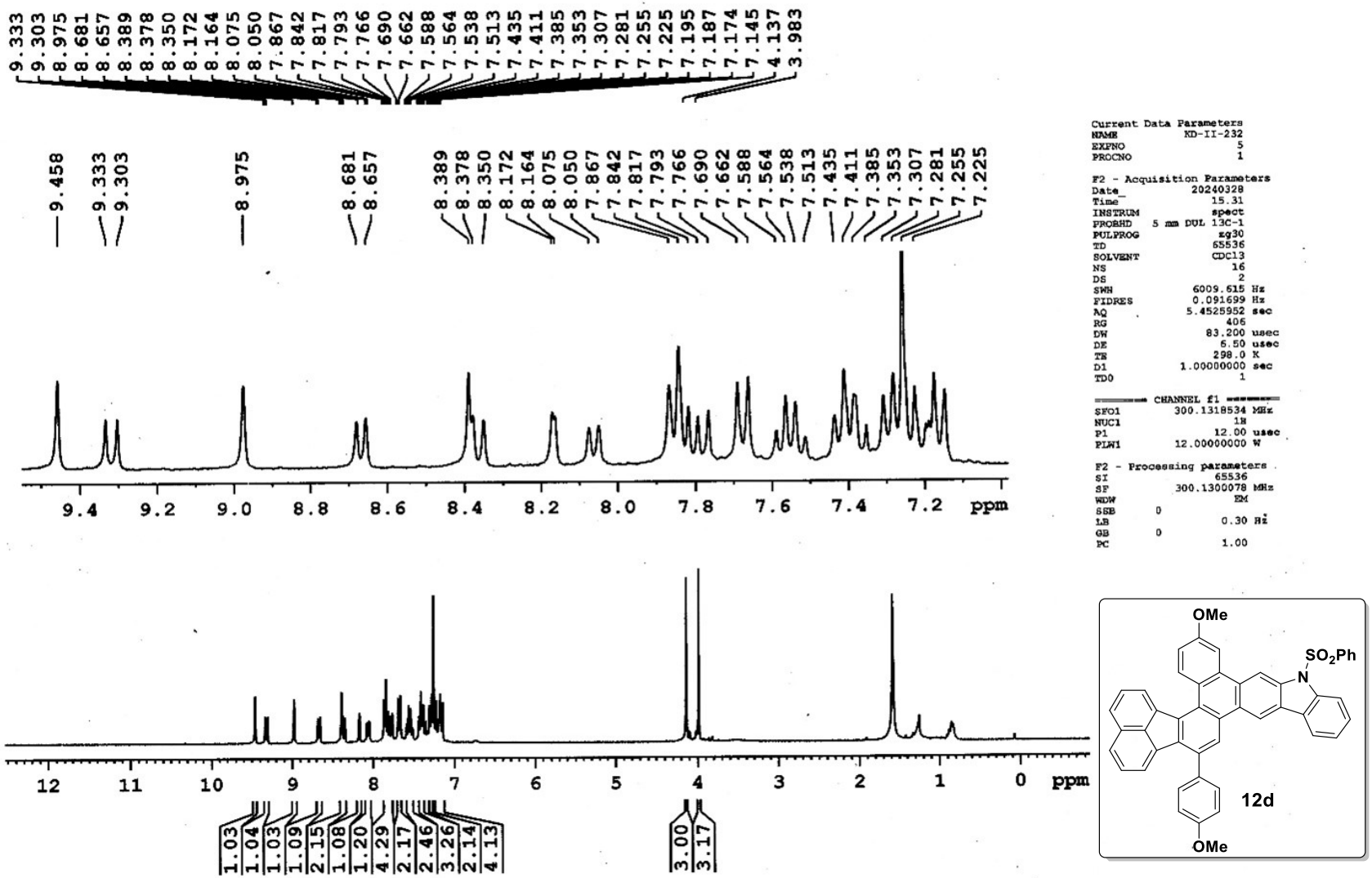
F2 - Acquisition Parameters
Date_                  20240401
Time                      16.13
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG              deptsp135
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                12077.295 Hz
FIDRES              0.184285 Hz
AQ                  2.7131903 sec
RG                        2050
DW                      41.400 usec
DE                       6.50 usec
TE                      300.0 K
CNST2               145.0000000
D1                    2.00000000 sec
D2                    0.00344828 sec
D12                   0.00002000 sec
TD0                           1

========== CHANNEL f1 ==========
SFO1                75.4737856 MHz
NUC1                        13C
P1                        11.00 usec
P13                     2000.00 usec
PLW0        0 W
PLW1              48.00000000 W
SPNAM[5]          Crp60comp.4
SPOAL5                  0.500
SPOFFS5     0 Hz
SPW5               8.87399960 W

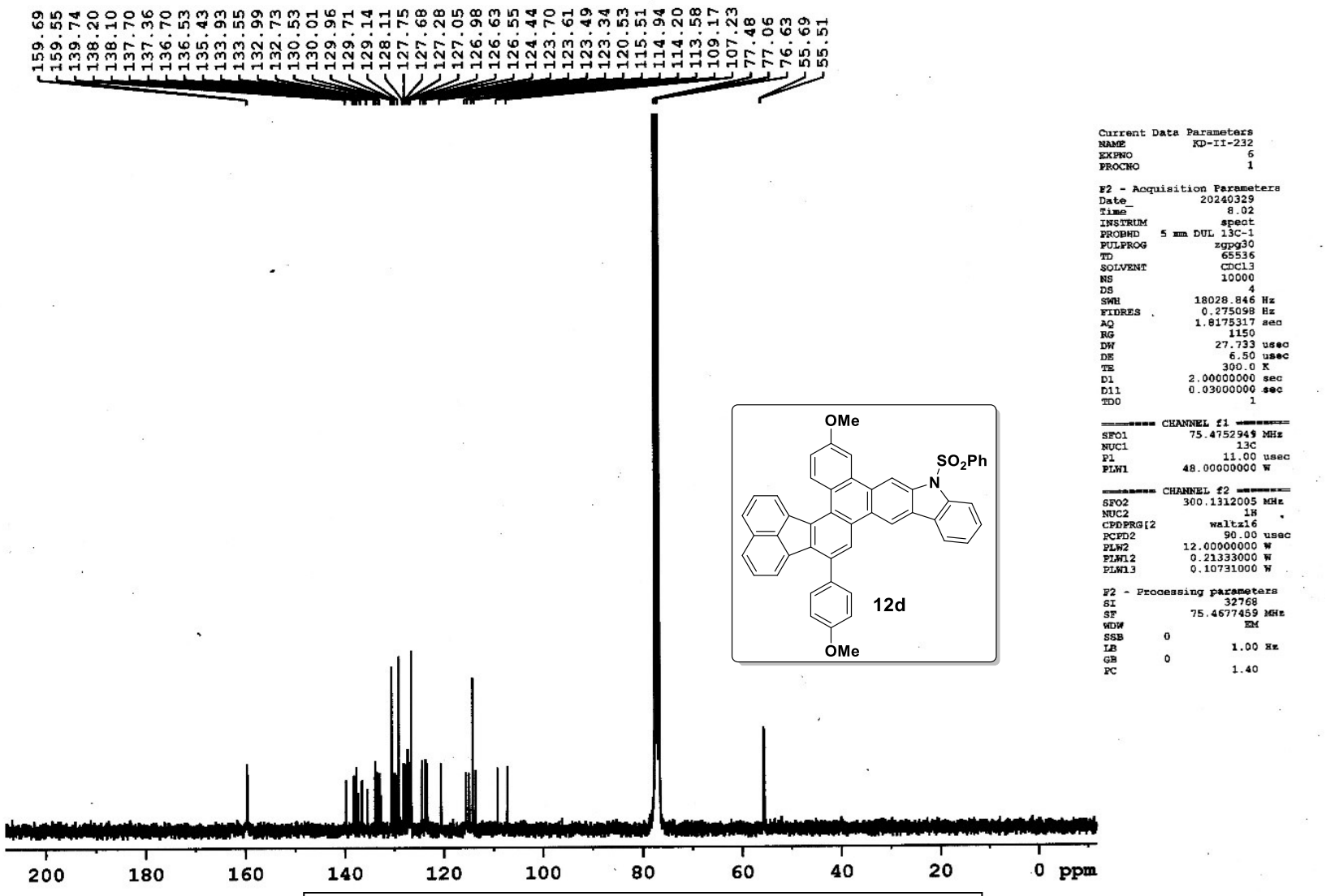
========== CHANNEL f2 ==========
SFO2              300.1309599 MHz
NUC2                        1H
CPDPRG[2                waltz16
P3                        12.00 usec
P4                        24.00 usec
PCPD2                     90.00 usec
PLW2              12.00000000 W
PLW12              0.21333000 W

F2 - Processing parameters
SI                        32768
SF                  75.4677485 MHz
WDW                         EM
SSB         0
LB                        1.00 Hz
GB          0
PC                        1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **12c**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12d**



Current Data Parameters

NAME	KD-II-232
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20240329
Time	8.02
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	10000
DS	4
SWH	18028.846 Hz
FIDRES	0.275098 Hz
AQ	1.8175317 sec
RG	1150
DW	27.733 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

CHANNEL f1

SFO1	75.4752949 MHz
NUC1	13C
P1	11.00 usec
PLW1	48.00000000 W

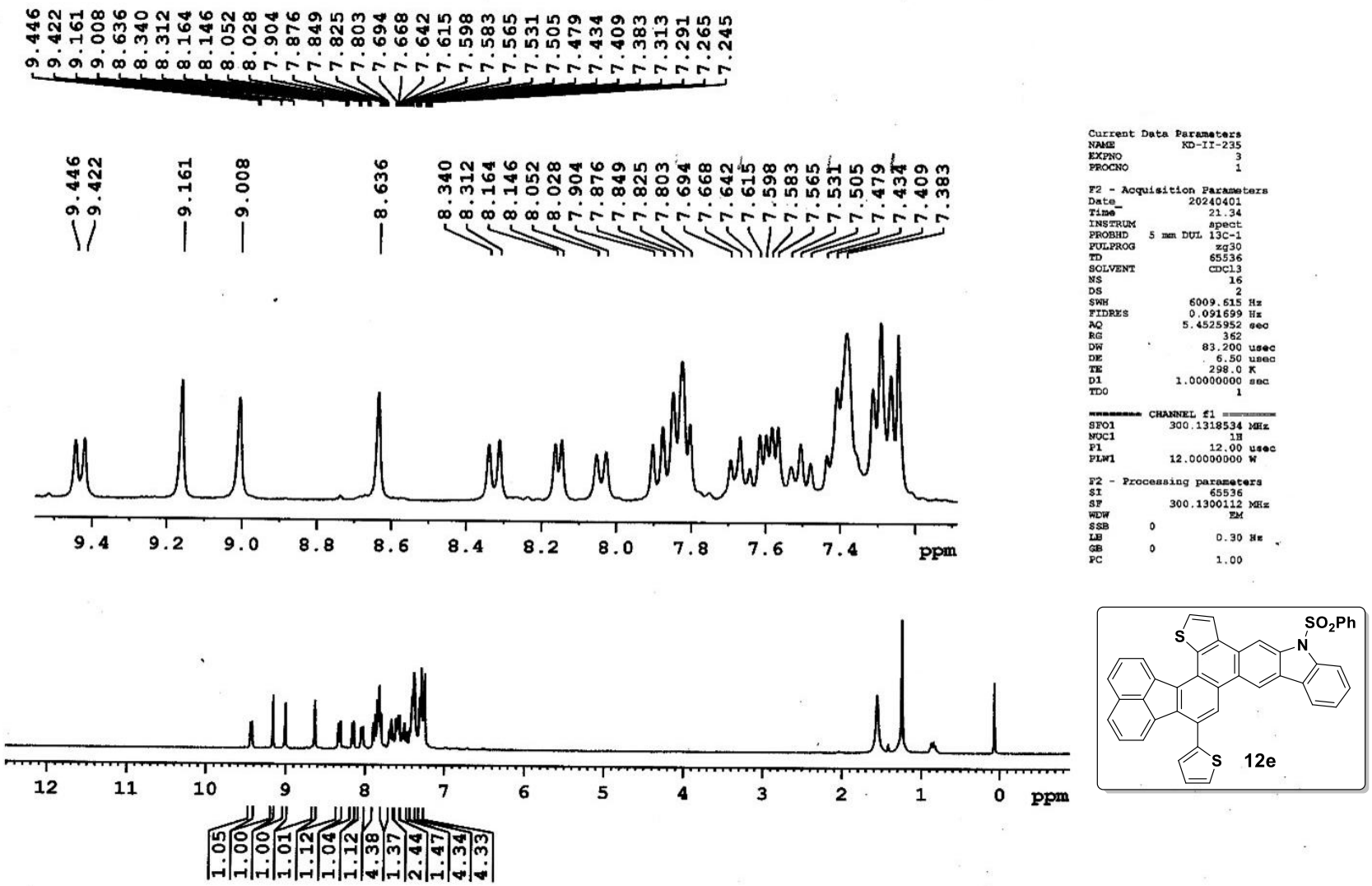
CHANNEL f2

SFO2	300.1312005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.21333000 W
PLW13	0.10731000 W

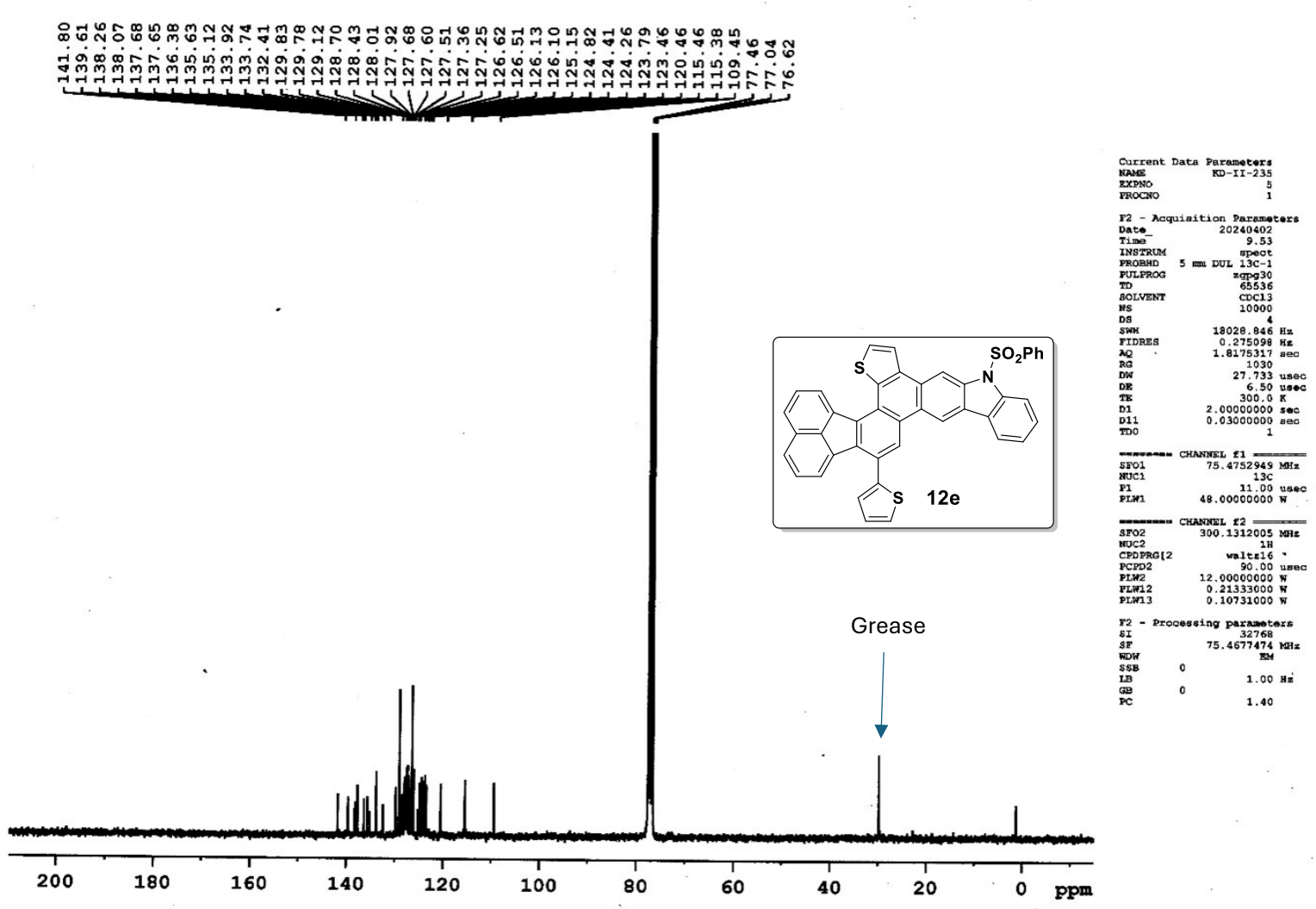
F2 - Processing parameters

SI		32768
SF		75.4677459 MHz
WDW		EM
SSB	0	
LB		1.00 Hz
GB	0	
PC		1.40

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12d**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12e**




```
Current Data Parameters
NAME                  KD-II-235
EXPNO                         5
PROCNO                        1

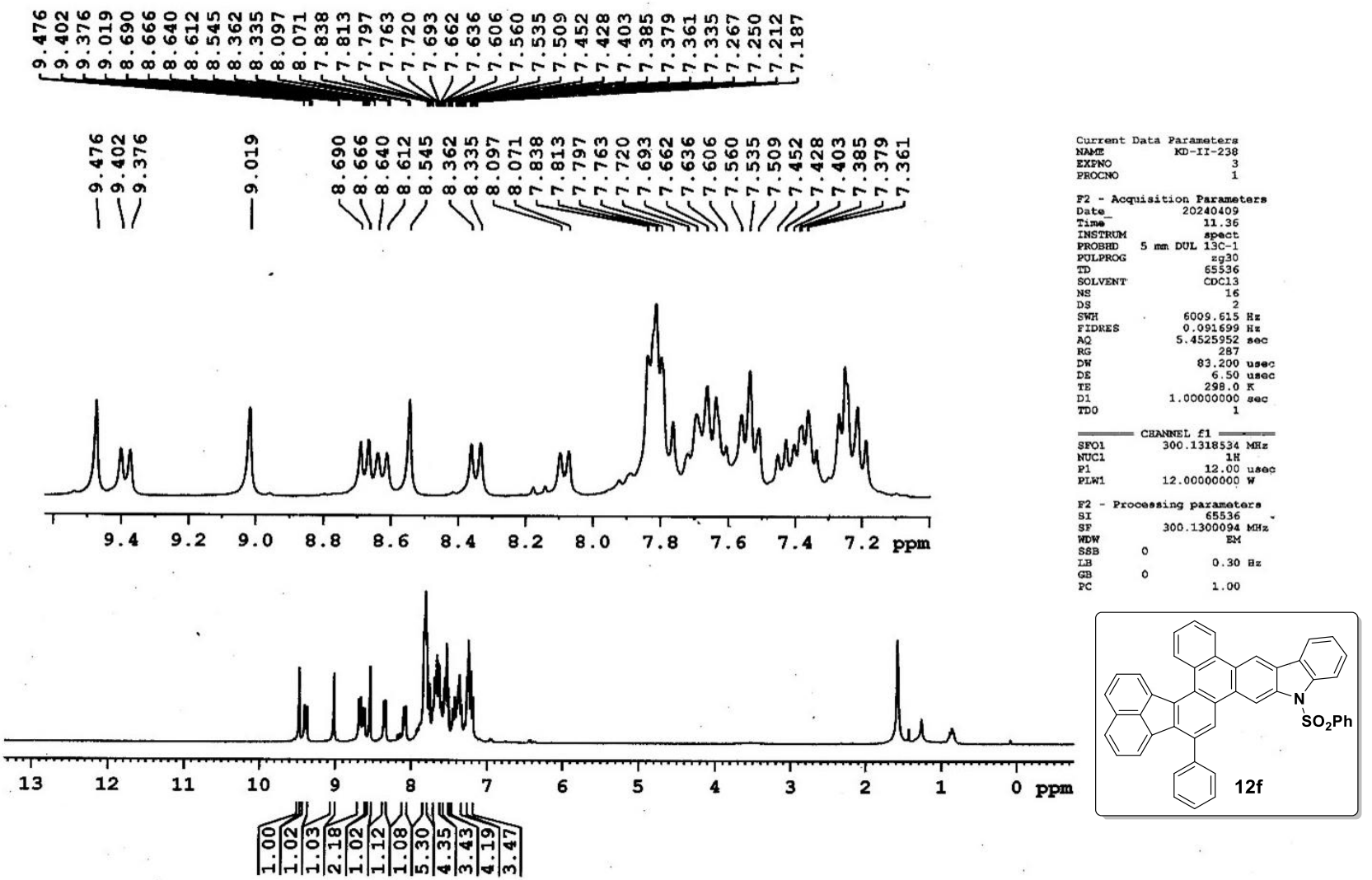
F2 - Acquisition Parameters
Date_                  20240402
Time                       9.53
INSTRUM                   spect
PROBHD     5 mm DUL 13C-1
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                        10000
DS                            4
SWH                18028.846 Hz
FIDRES                 0.275098 Hz
AQ                     1.8175317 sec
RG                         1030
DW                        27.733 usec
DE                         6.50 usec
TE                        300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                  75.4752949 MHz
NUC1                         13C
P1                         11.00 usec
PLW1                  48.00000000 W

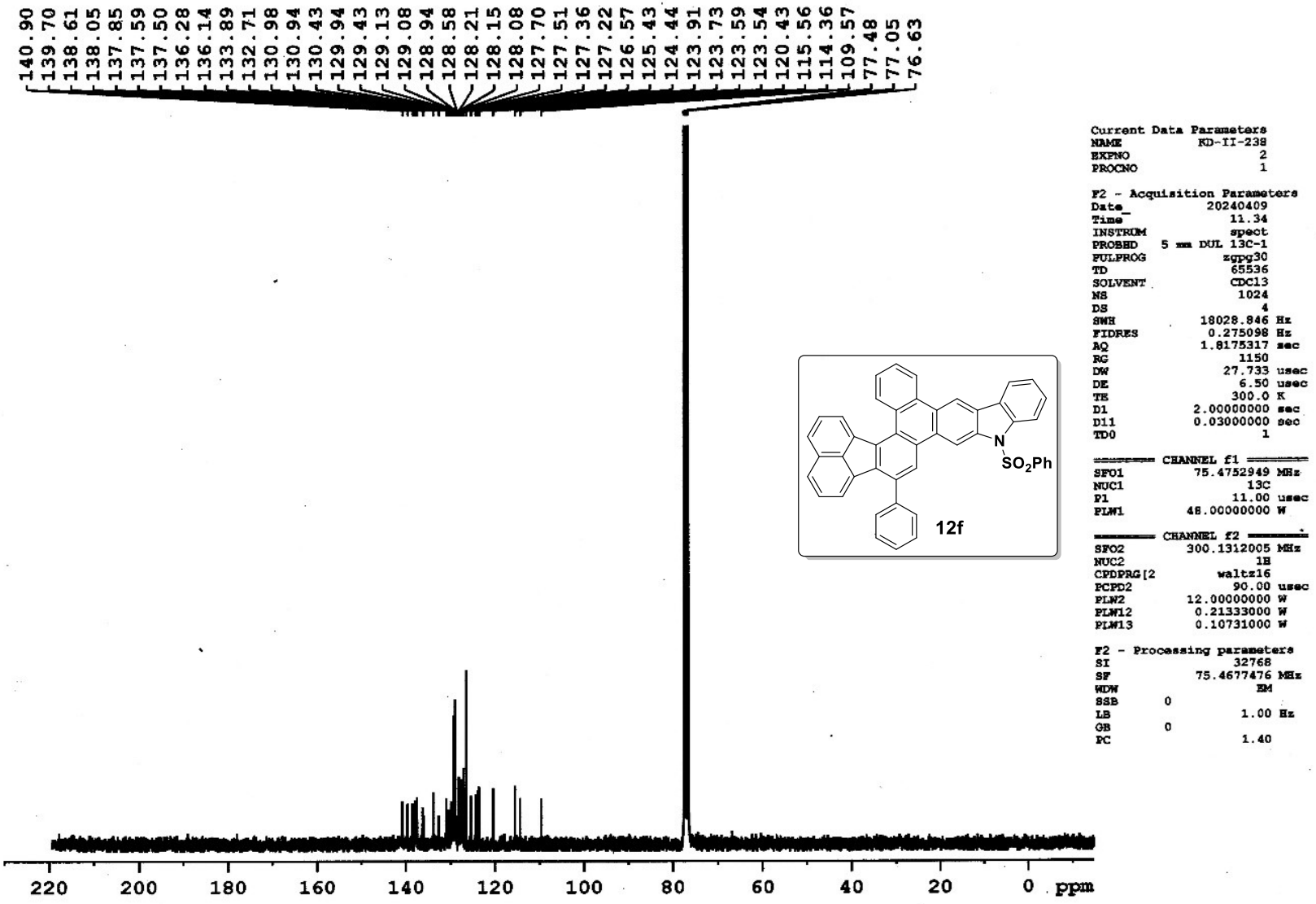
======== CHANNEL f2 ========
SFO2                 300.1312005 MHz
NUC2                          1H
CPDPRG[2                 waltz16
PCPD2                      90.00 usec
PLW2                  12.00000000 W
PLW12                  0.21333000 W
PLW13                  0.10731000 W

F2 - Processing parameters
SI                        32768
SF                    75.4677474 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12e**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12f**



Current Data Parameters
NAME KD-II-238
EXPNO 2
PROCNO 1

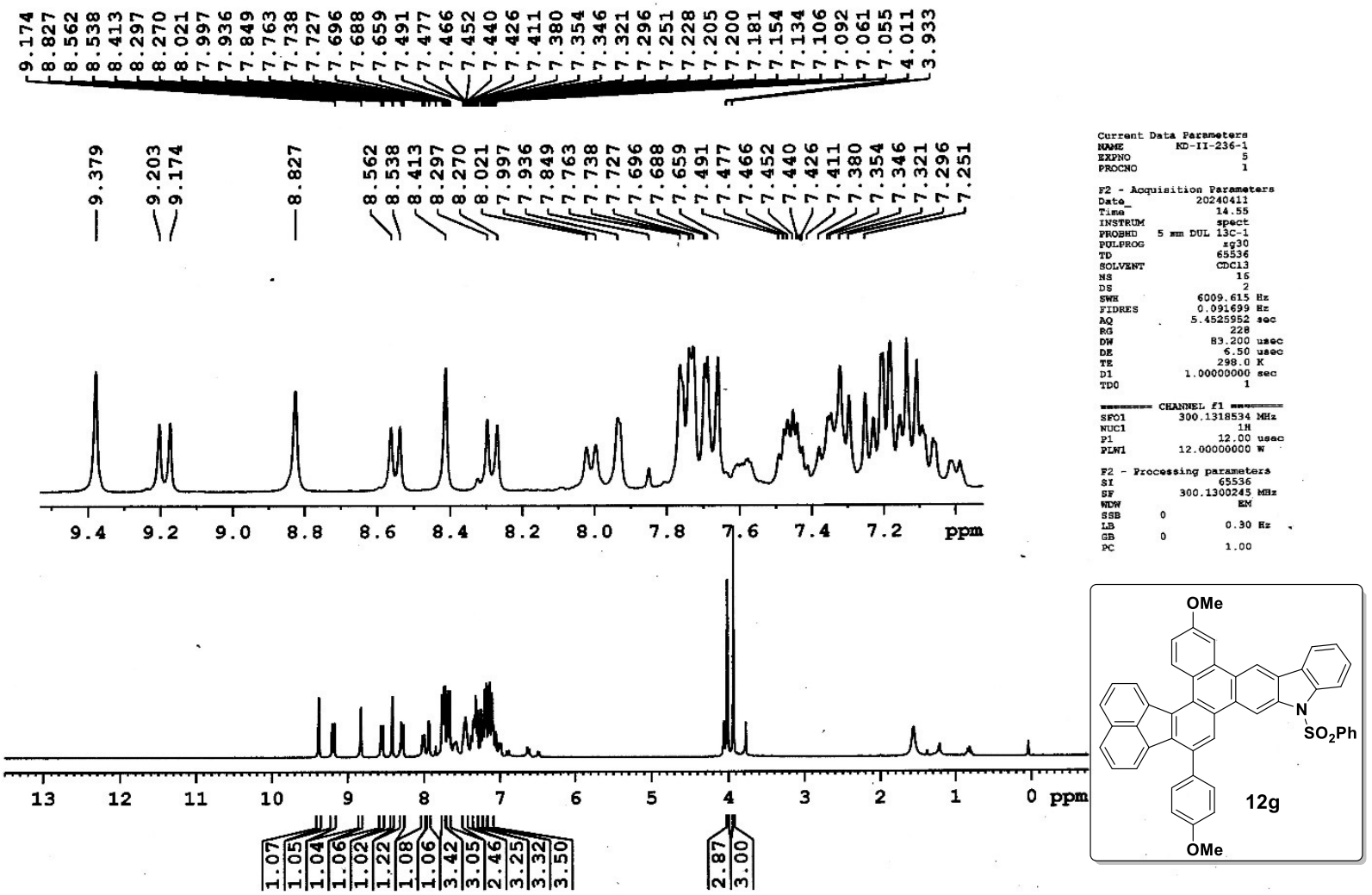
F2 - Acquisition Parameters
Date_ 20240409
Time 11.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 1150
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

CHANNEL f1
SFO1 75.4752949 MHz
NUC1 13C
P1 11.00 usec
PLW1 48.00000000 W

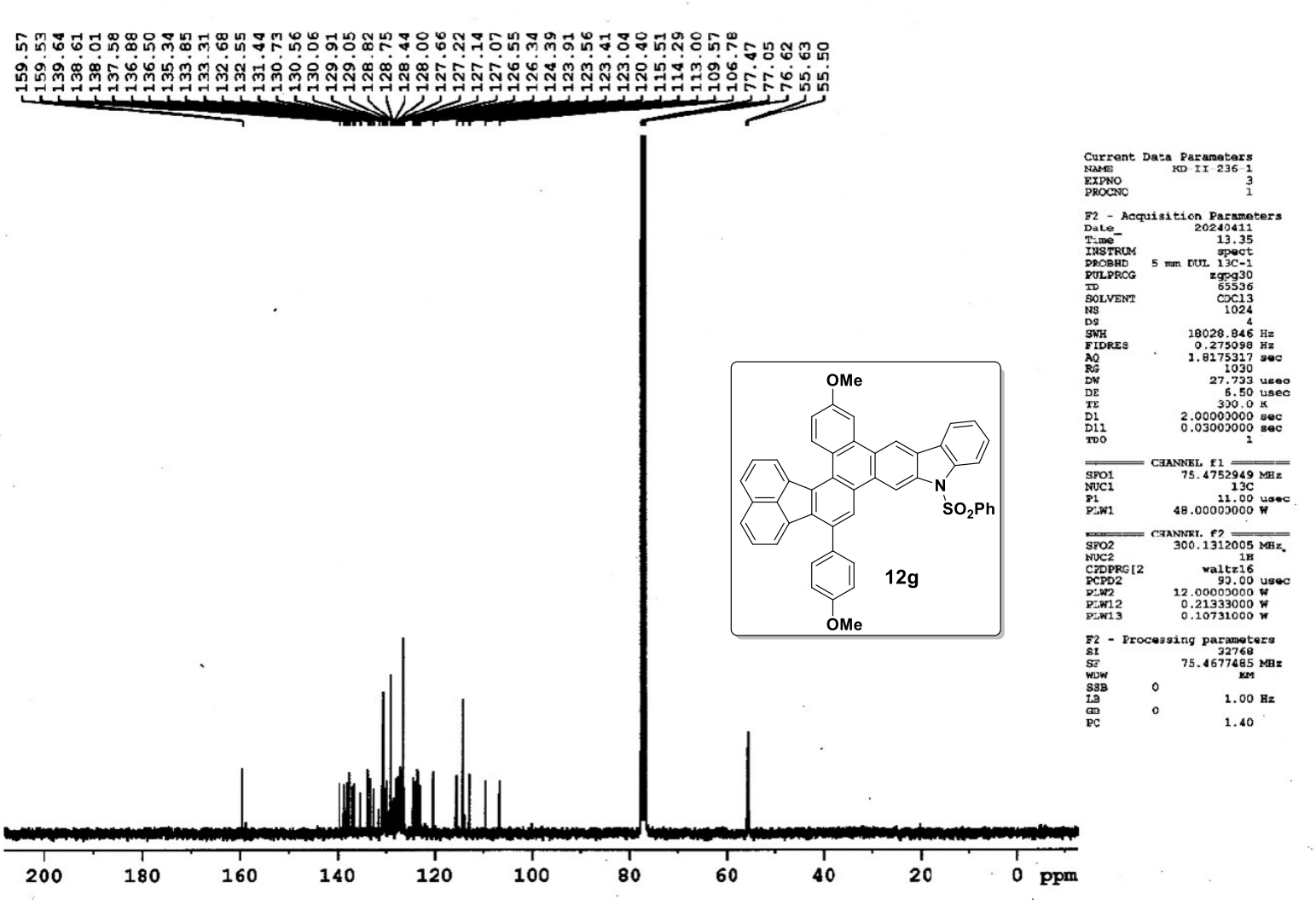
CHANNEL f2
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.21333000 W
PLW13 0.10731000 W

F2 - Processing parameters
SI 32768
SF 75.4677476 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

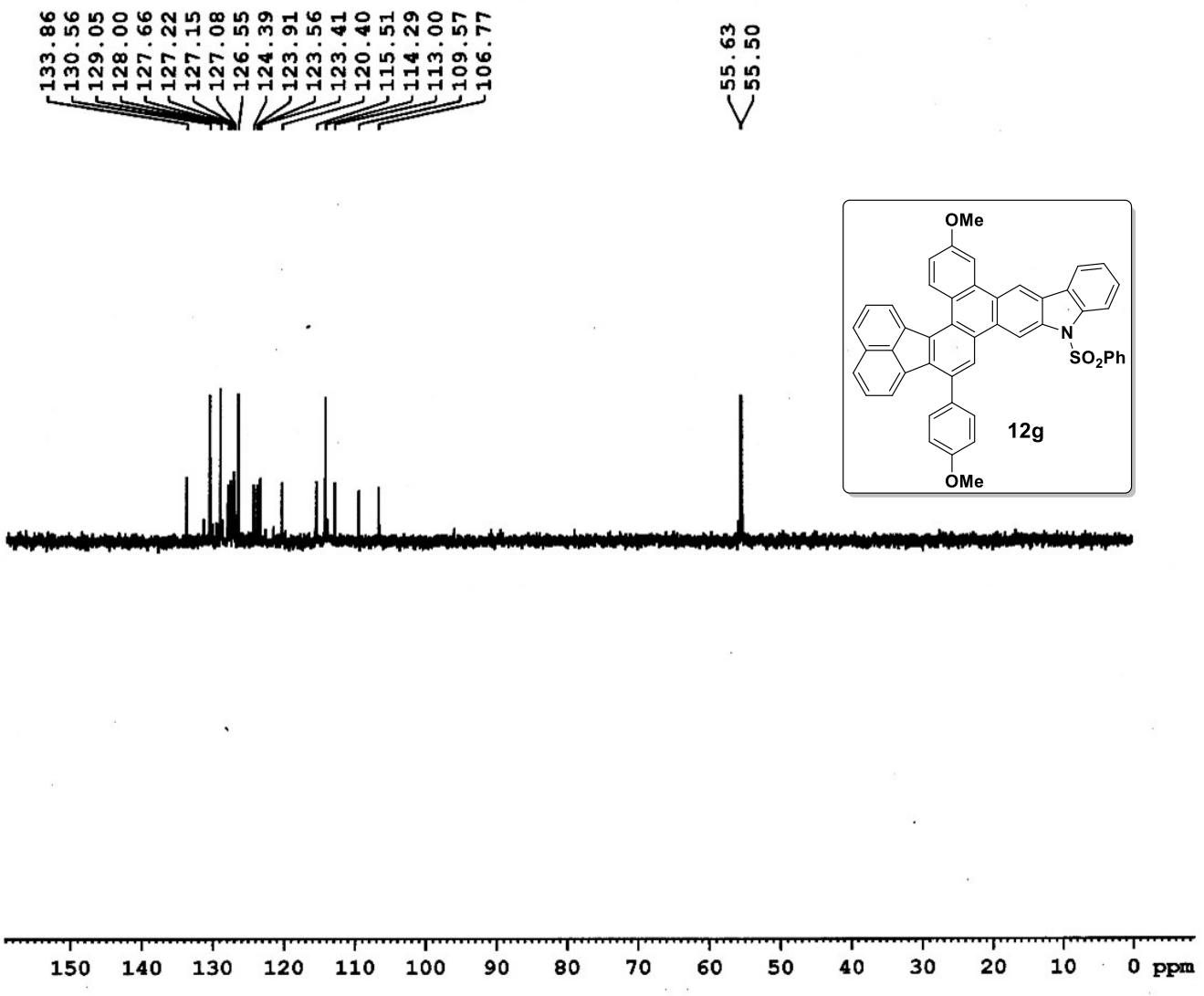
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12f**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12g**



$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12g**




```
Current Data Parameters
NAME          KD-II-236-1
EXPNO                   4
PROCNO                  1

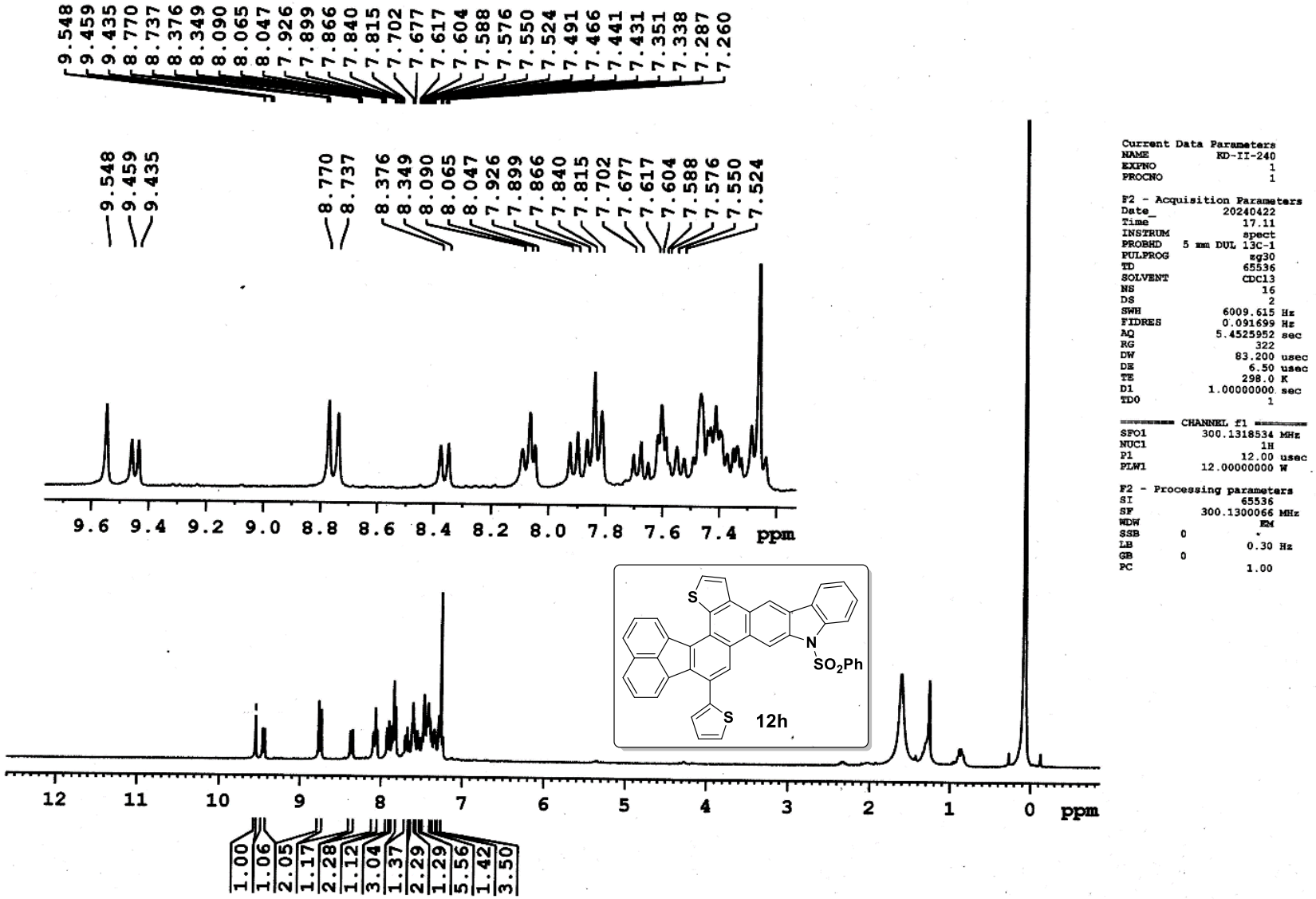
F2 - Acquisition Parameters
Date_            20240411
Time                14.52
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG         deptsp135
TD                  65536
SOLVENT             CDCl3
NS                    256
DS                      4
SWH           12077.295 Hz
FIDRES         0.184285 Hz
AQ             2.7131903 sec
RG                   2050
DW                 41.400 usec
DE                  6.50 usec
TE                 300.0 K
CNST2         145.0000000
D1              2.00000000 sec
D2              0.00344828 sec
D12             0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          75.4737856 MHz
NUC1                  13C
P1                  11.00 usec
P13               2000.00 usec
PLW0      0 W
PLW1        48.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5   0 Hz
SPW5          8.87399960 W

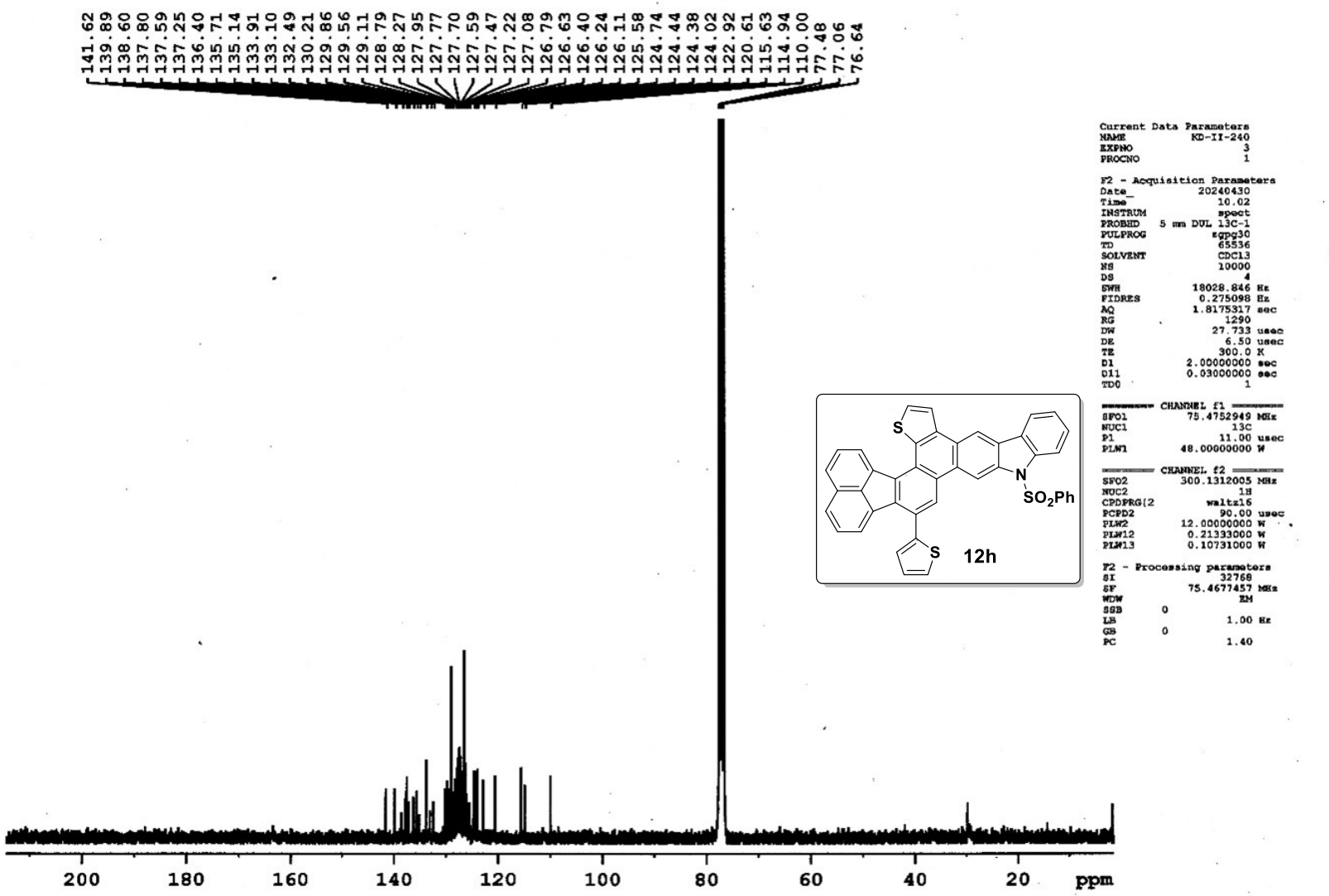
======== CHANNEL f2 ========
SFO2         300.1309599 MHz
NUC2                  1H
CPDPRG[2          waltz16
P3                  12.00 usec
P4                  24.00 usec
PCPD2               90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W

F2 - Processing parameters
SI                  32768
SF           75.4677485 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

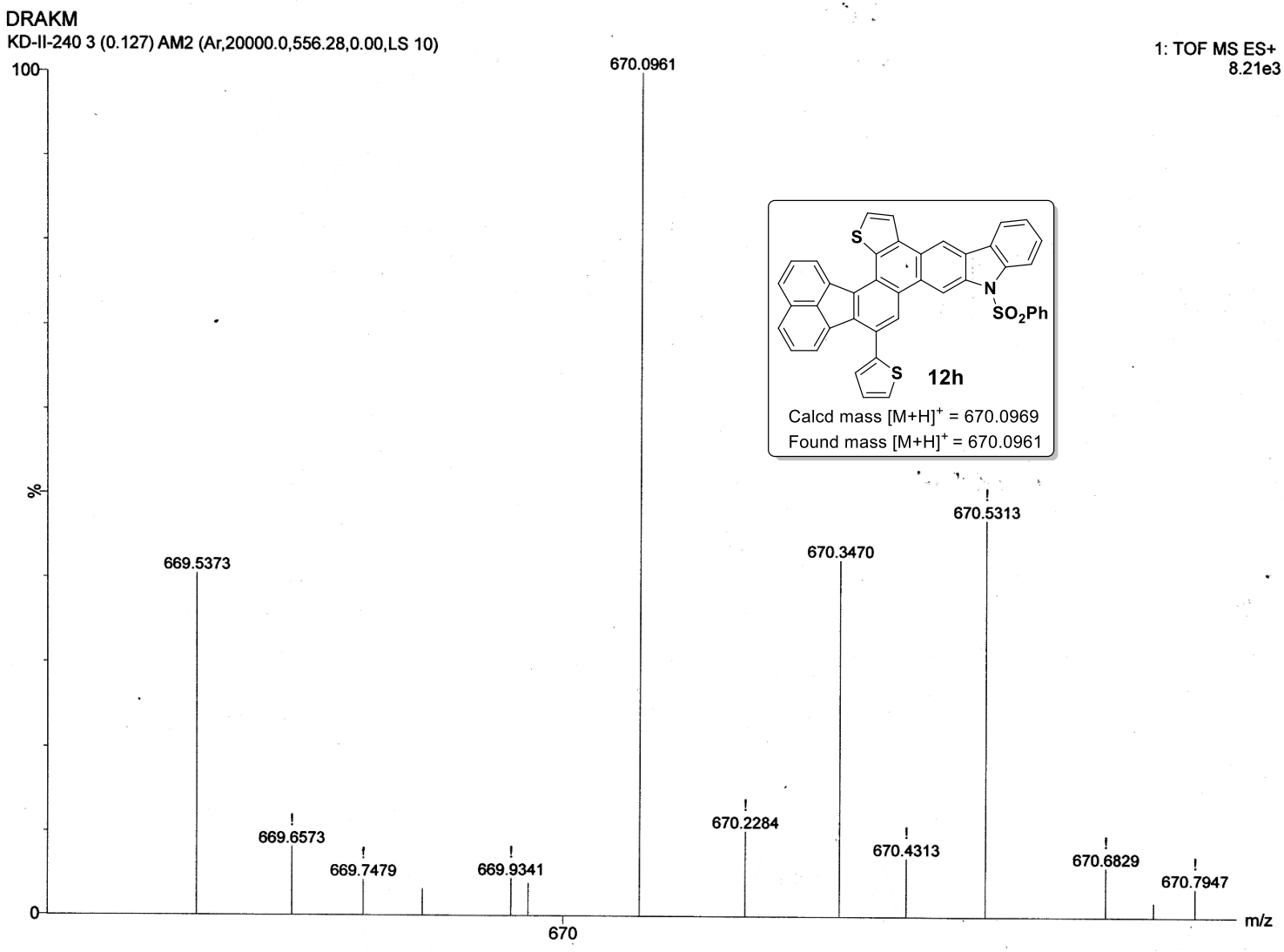
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **12g**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **12h**

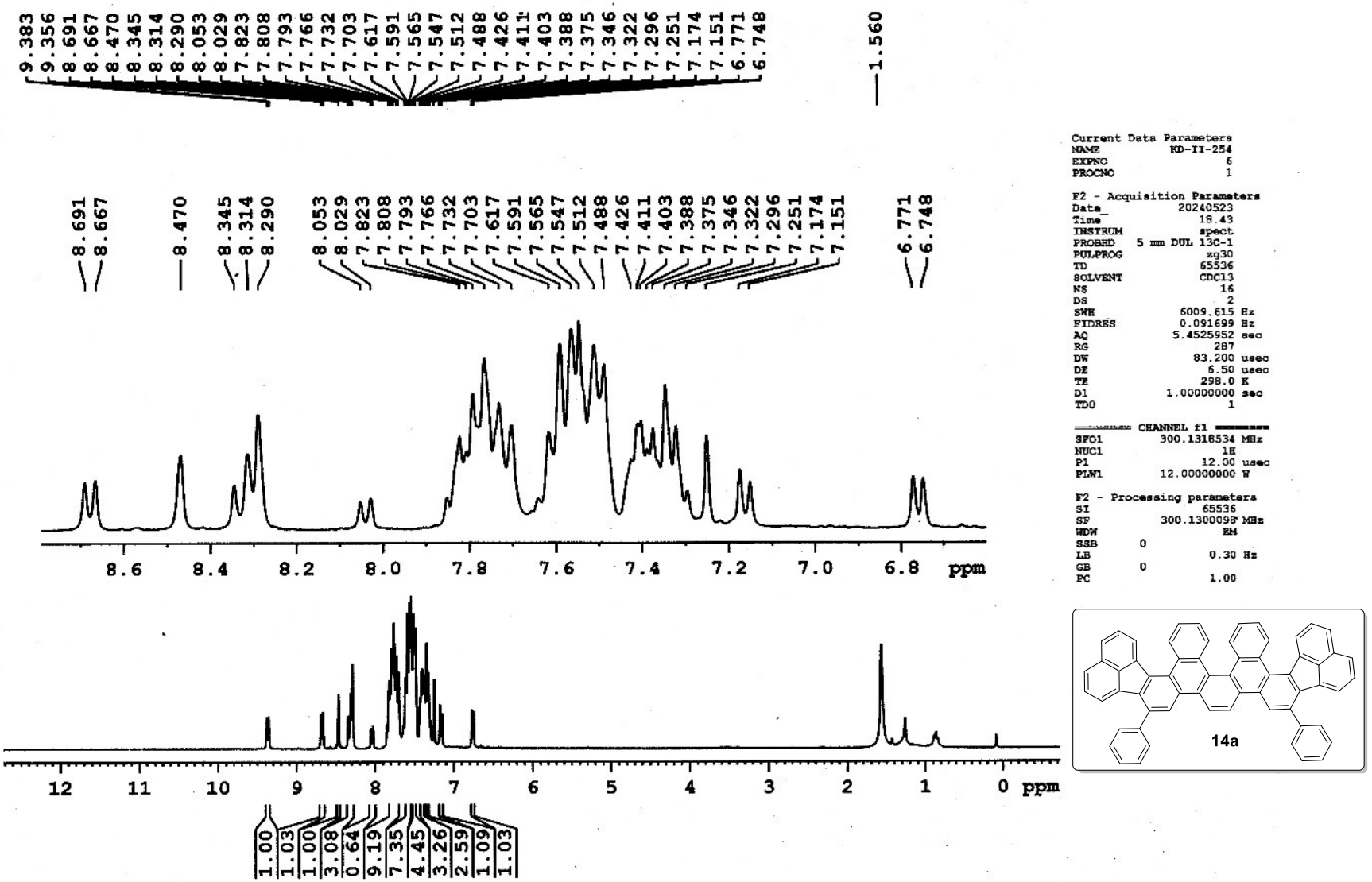


$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **12h**

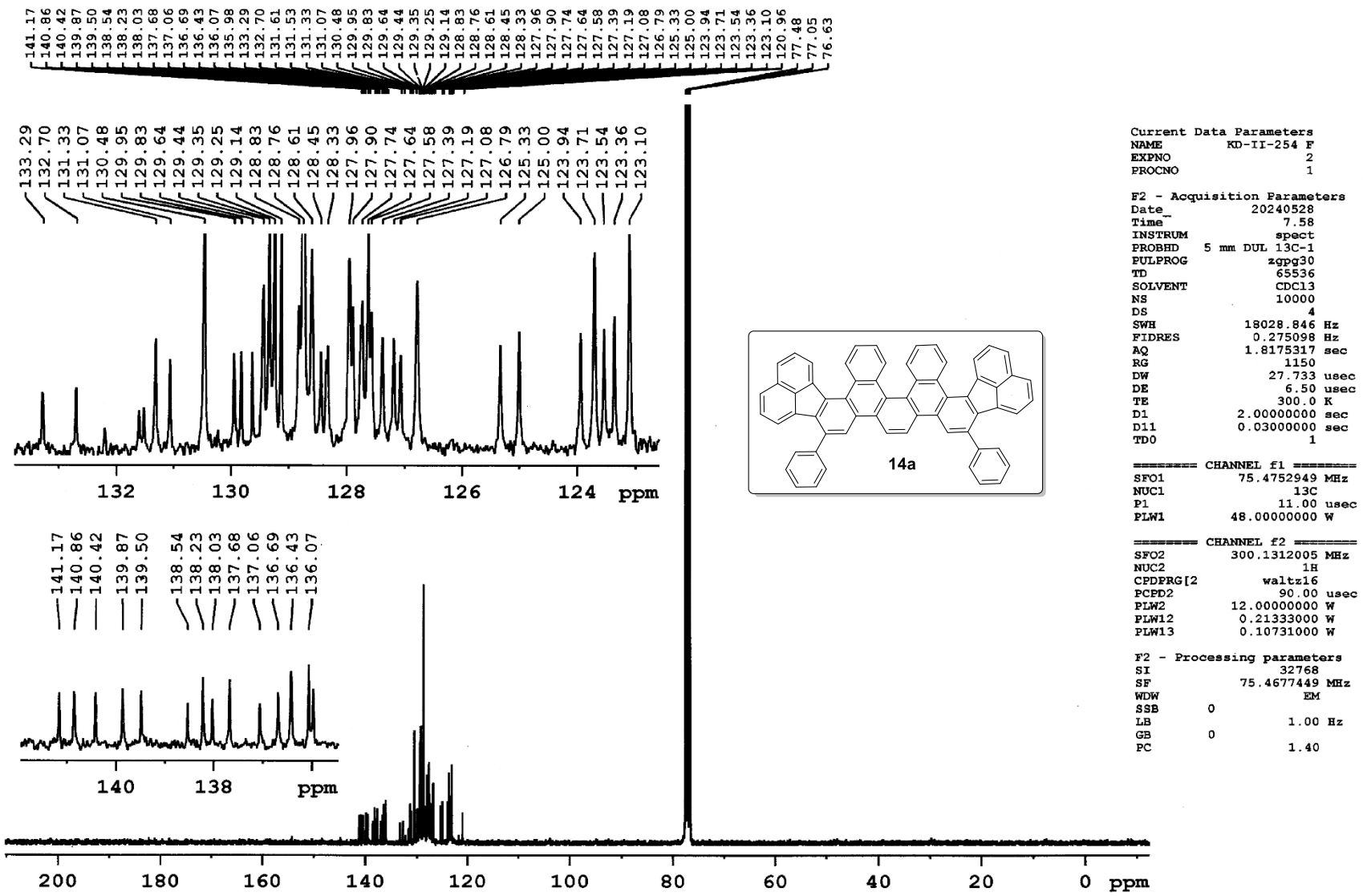


HRMS spectrum of compound **12h**

10. ^{1}H, ^{13}C {^{1}H}, DEPT 135 NMR & HRMS (selected) Spectra of dibenzo picenes **14a** and **14b**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **14a**

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Current Data Parameters
NAME          KD-II-254 F
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240528
Time                7.58
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 10000
DS                     4
SWH           18028.846 Hz
FIDRES         0.275098 Hz
AQ             1.8175317 sec
RG                  1150
DW                27.733 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                  13C
P1                 11.00 usec
PLW1         48.00000000 W

======== CHANNEL f2 ========
SFO2          300.1312005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.21333000 W
PLW13         0.10731000 W

F2 - Processing parameters
SI                 32768
SF            75.4677449 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```
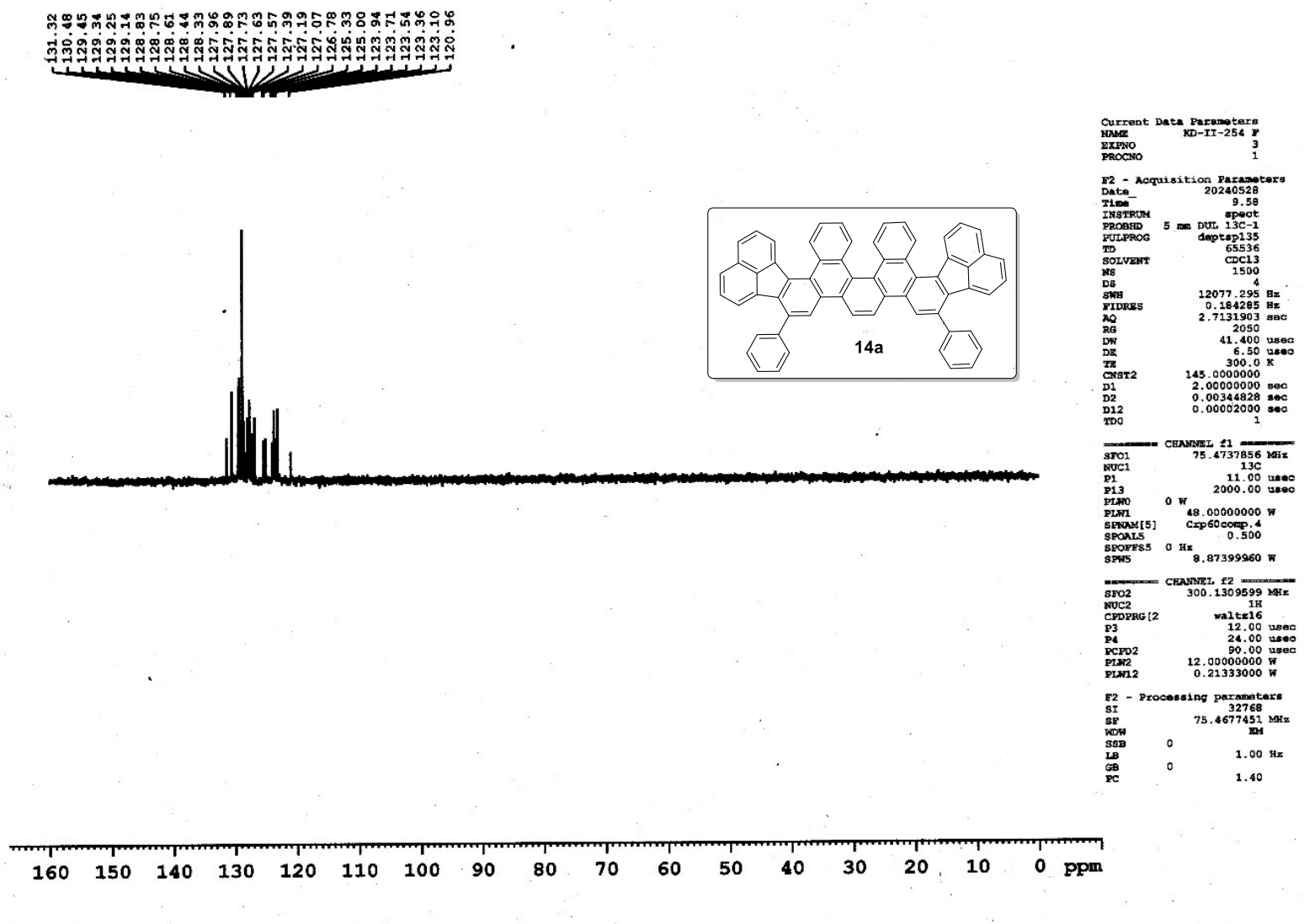
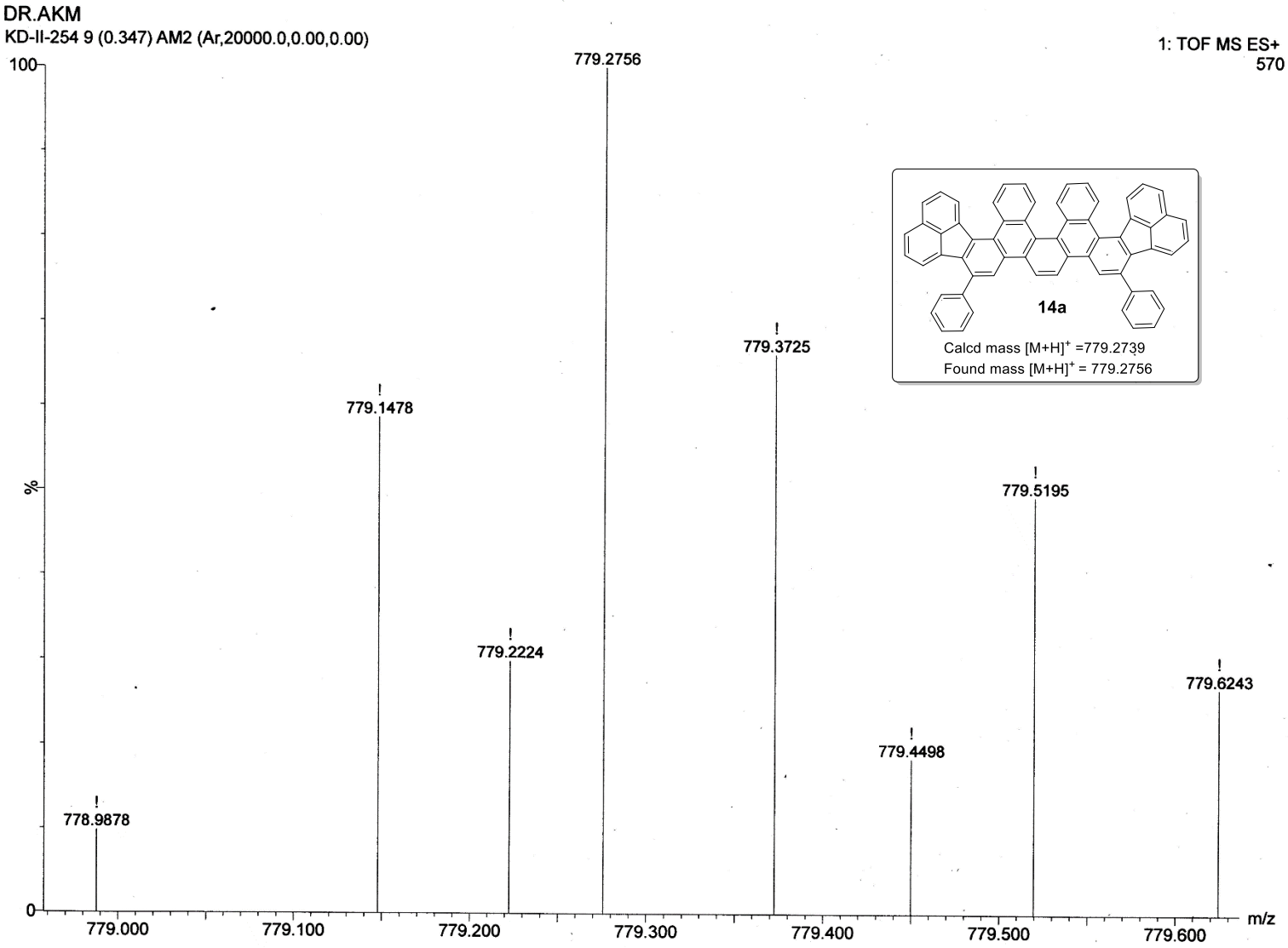
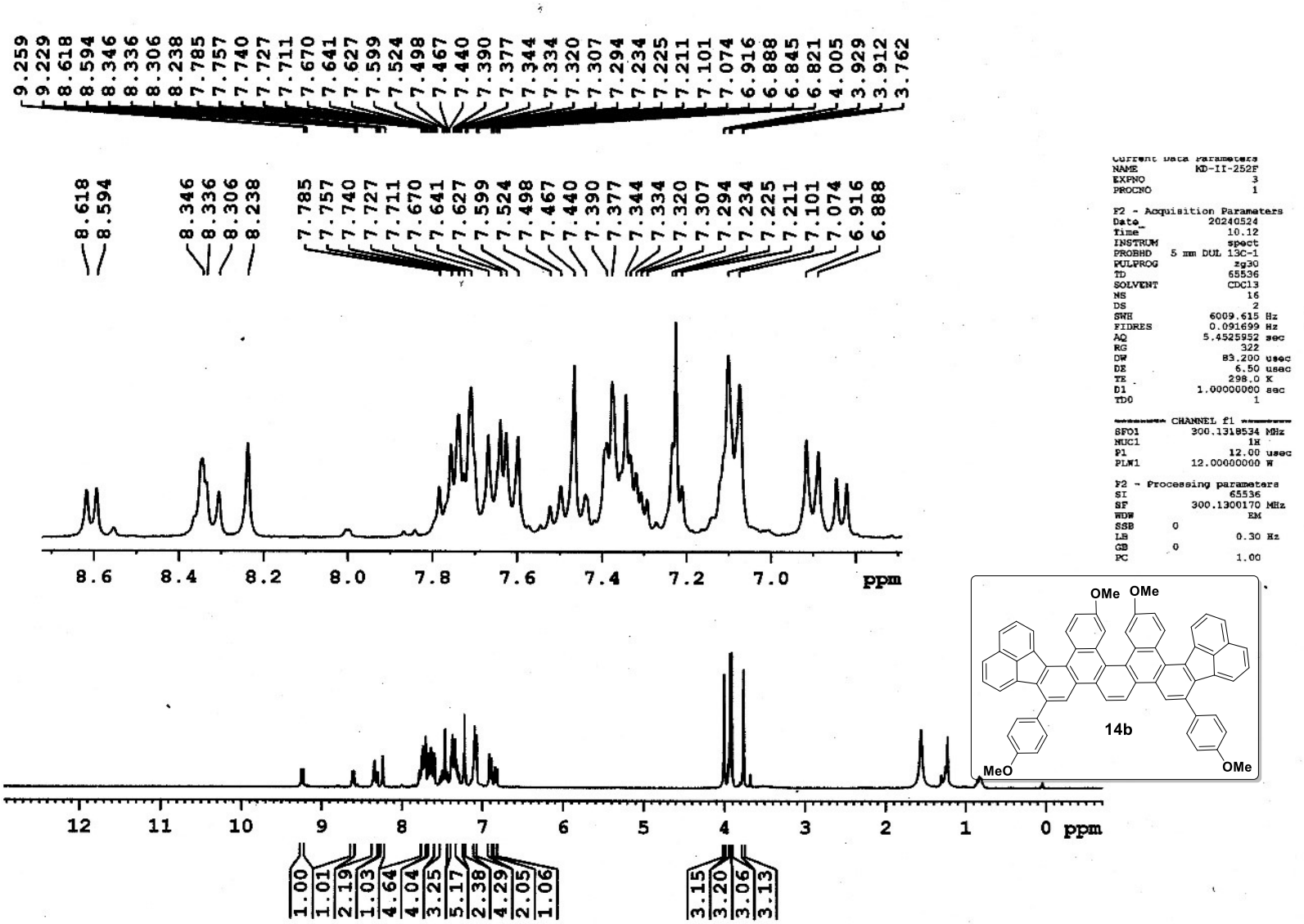

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **14a**



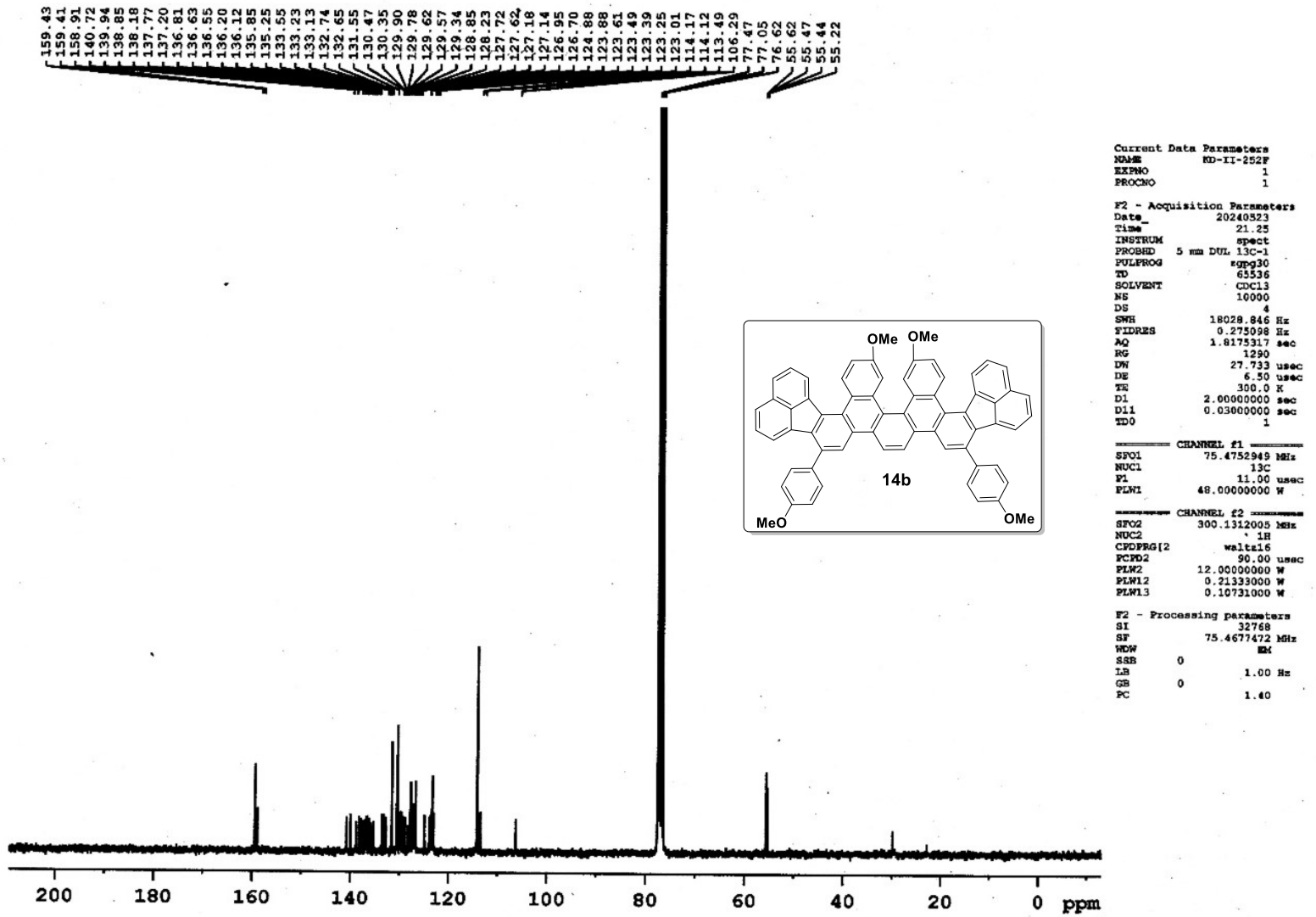
DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **14a**



HRMS spectrum of compound **14a**



^{1}H-NMR (300 MHz, $CDCl_3$) spectrum of compound **14b**




```
Current Data Parameters
NAME          KD-II-252F
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240523
Time              21.25
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS               10000
DS                   4
SWH          18028.846 Hz
FIDRES         0.275098 Hz
AQ           1.8175317 sec
RG                1290
DW              27.733 usec
DE               6.50 usec
TE              300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         75.4752949 MHz
NUC1               13C
P1              11.00 usec
PLW1        48.00000000 W

======== CHANNEL f2 ========
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NUC2               1H
CPDPRG[2        waltz16
PCPD2           90.00 usec
PLW2        12.00000000 W
PLW12        0.21333000 W
PLW13        0.10731000 W

F2 - Processing parameters
SI                32768
SF           75.4677472 MHz
WDW                  EM
SSB          0
LB                1.00 Hz
GB           0
PC                1.40
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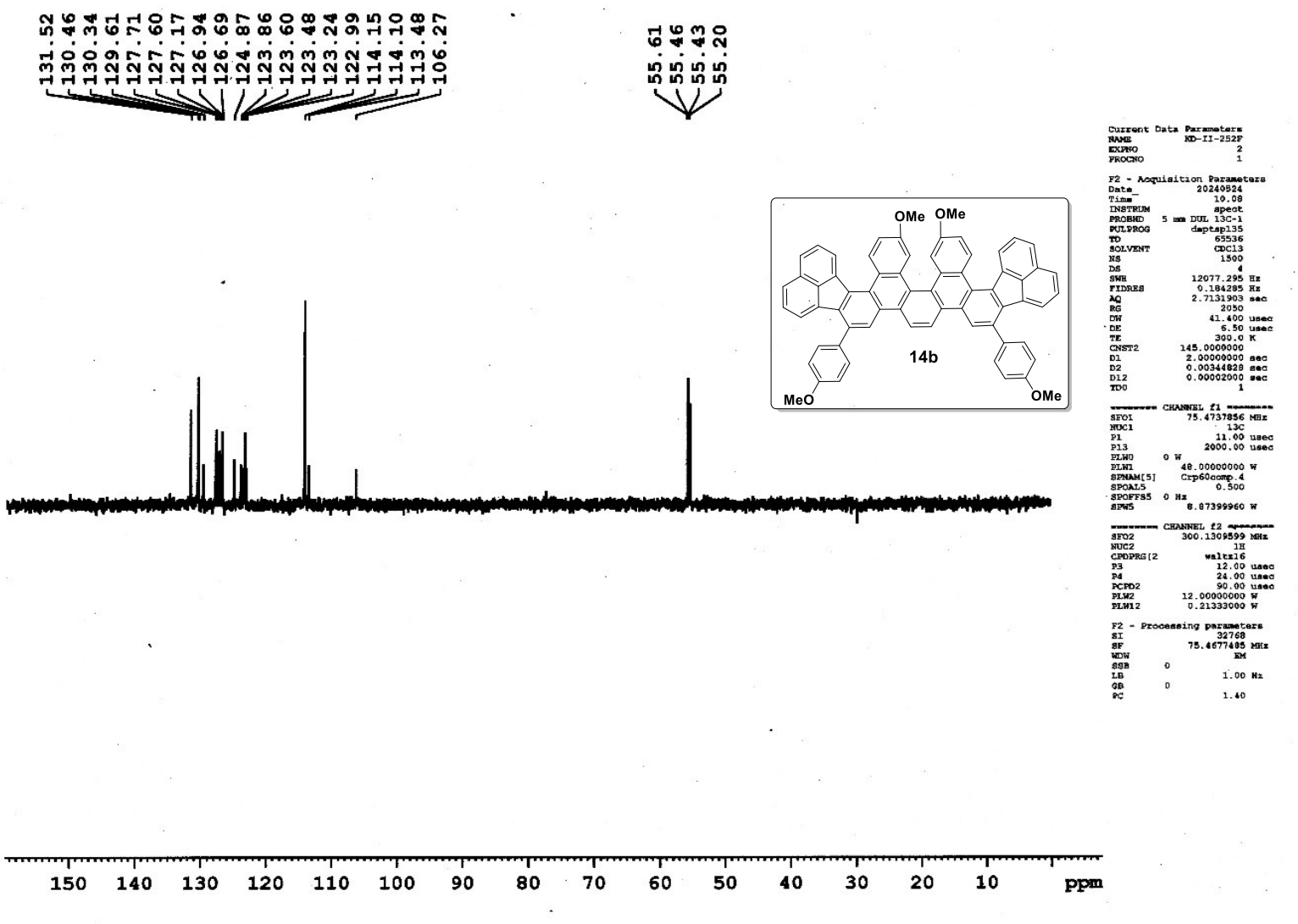
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound **14b**

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D12               0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
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NUC1                    13C
P1                   11.00 usec
P13                2000.00 usec
PLW0     0 W
PLW1          48.00000000 W
SPNAM[5]     Crp60comp.4
SPOAL5             0.500
SPOFFS5  0 Hz
SPW5           8.87399960 W

======== CHANNEL f2 ========
SFO2           300.1309599 MHz
NUC2                    1H
CPDPRG[2           waltz16
P3                   12.00 usec
P4                   24.00 usec
PCPD2                90.00 usec
PLW2          12.00000000 W
PLW12          0.21333000 W

F2 - Processing parameters
SI                   32768
SF              75.4677485 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```

DEPT-135 (75 MHz, $CDCl_3$) NMR spectrum of compound **14b**

11. X-ray structure and crystallographic data of phenanthrenes **3a, 3f, 6a, 6e** and hetero helicene **10**

11.1 X-Ray Structure & Crystallographic data of 3a

Crystallographic data of 1,2,3-Triphenyltriphenylene **3a** (ellipsoid contour % probability is 50%). The crystals of suitable quality were obtained from chloroform by slow evaporation under air conditions and were analysed by single crystal diffractometer.

CCDC: 2375301

https://www.ccdc.cam.ac.uk/structures/Search?access=referee&ccdc=2375301&Author=Divya+bharathi

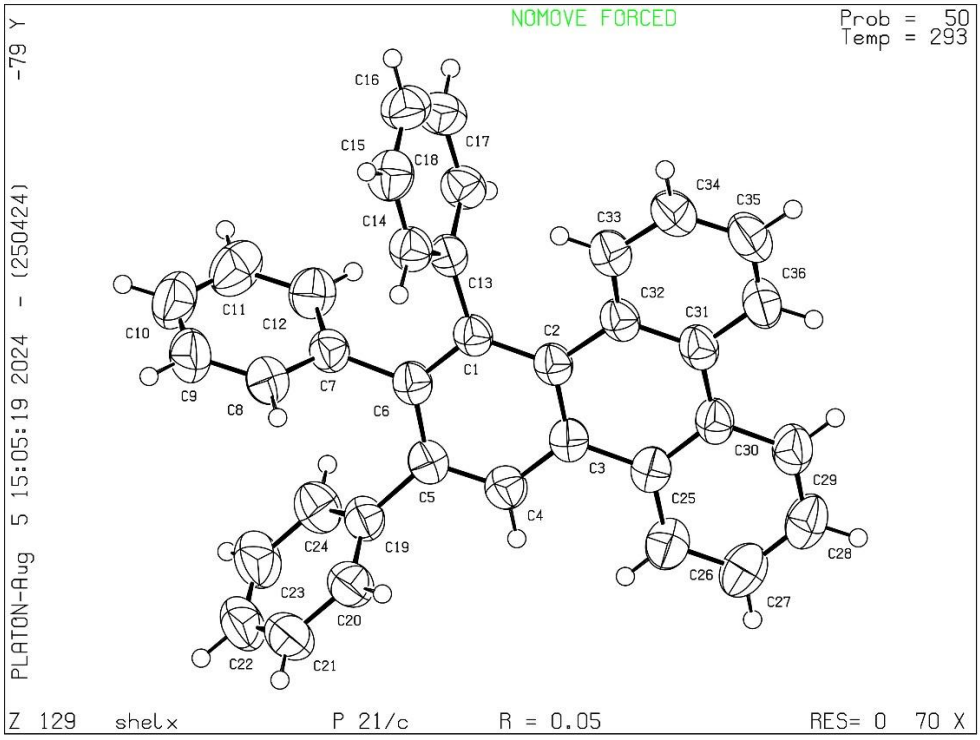

Table S1. Crystal data and structure refinement for 1-c2_red1_abs1_sx.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision:	C-C = 0.0033 A		Wavelength = 0.71073
Cell:	a = 11.434(8)	b = 22.093(18)	c = 9.934(9)
	alpha = 90	beta = 96.97(3)	gamma = 90

Temperature: 293 K

	Calculated	Reported
Volume	2491(4)	2491(3)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C36 H24	C36 H24
Sum formula	C36 H24	C36 H24
Mr	456.55	456.55
Dx,g cm-3	1.217	1.217
Z	4	4
Mu (mm-1)	0.069	0.069
F000	960.0	960.0
F000'	960.36	
h,k,lmax	13,26,11	13,26,11
Nref	4394	4370
Tmin,Tmax	0.983,0.987	0.970,0.987
Tmin'	0.970	

Correction method= # Reported T Limits: Tmin = 0.970 Tmax = 0.987 AbsCorr = MULTI-SCAN

Data completeness = 0.995 Theta(max) = 25.026

R(reflections) = 0.0475(3260) wR2(reflections) = 0.2211(4370)

S = 0.907 Npar = 325

Computing Details and Refinement

The **3a** single-crystal dimensions were 0.445 x 0.205 x 0.195 mm^3 chosen for an X-ray diffraction study. The X-ray diffraction intensity data was collected at a temperature of 303 K on a Bruker Kappa Apex II CCD Diffractometer using graphite monochromate Mo-Kα (λ = 0.71073 Å) radiation. The crystal structure **3a** was solved by the direct methods SHELXS. The crystal structure of **3a** was refined by the full-matrix least-squares method on *F*2 using *SHELXL* programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned geometrically and refined using a riding model. The geometrical calculations were carried out using the program *PLATON*. The molecular and packing diagrams were generated using the software *MERCURY*. The details of the crystal structure and data refinement was listed in **Table S1**. The

synthesized title compound was crystallized in the Monoclinic, crystal system in the P 21/c space group with unit cell parameters a = 11.434(8) Å, b = 22.093(18) Å, c = 9.934(9) Å, α = 90º, β = 96.97(3)°, γ = 90º, Z = 4 and V = 2491(4) Å^3.

11.2 X-Ray Structure & Crystallographic data of 3f

Crystallographic data of 6-phenylacenaphtho[1,2-*a*]triphenylene **3f** (ellipsoid contour % probability is 50%). The crystals of suitable quality were obtained from chloroform by slow evaporation under air conditions and were analysed by single crystal diffractometer.

CCDC: 2375299

https://www.ccdc.cam.ac.uk/structures/Search?access=referee&ccdc=2375299&Author=Divya+bharathi

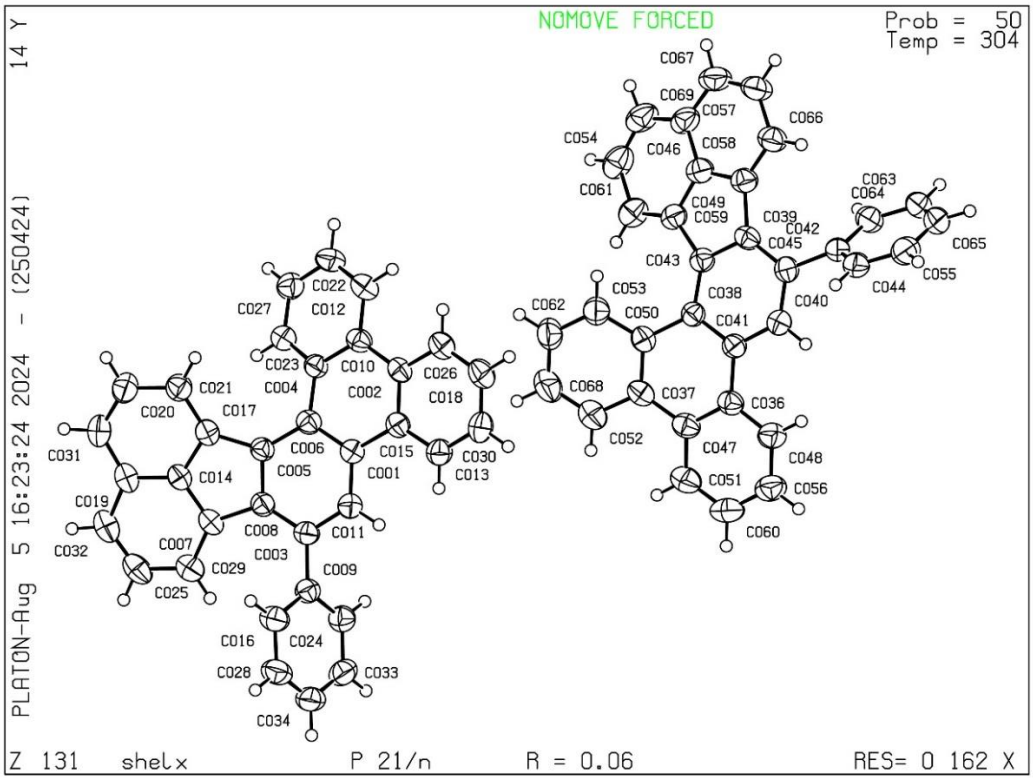

Table S2. Crystal data and structure refinement for 1-c2_red1_abs1_sx.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0053 A Wavelength = 0.71073

Cell: a = 9.8987(7) b = 21.3915(15) c = 20.2646(15)

alpha = 90 beta = 96.076(3) gamma = 90

Temperature: 304 K

	Calculated	Reported
Volume	4266.9(5)	4266.9(5)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C34 H20	C34 H20
Sum formula	C34 H20	C34 H20
Mr	428.50	428.50
Dx,g cm-3	1.334	1.334
Z	8	8
Mu (mm-1)	0.076	0.076
F000	1792.0	1792.0
F000'	1792.67	
h,k,lmax	11,25,24	11,25,24
Nref	7553	7523
Tmin,Tmax	0.983,0.995	0.983,0.995
Tmin'	0.983	

Correction method= # Reported T Limits: Tmin = 0.983 Tmax = 0.995 AbsCorr = MULTI-SCAN

Data completeness = 0.996 Theta(max) = 25.054

R(reflections) = 0.0593(4324) wR2(reflections) = 0.2582 (7523)

S = 0.768 Npar = 613

Computing Details and Refinement

The **3f** single-crystal dimensions were 0.225 x 0.193 x 0.069 mm^3 chosen for an X-ray diffraction study. The X-ray diffraction intensity data was collected at a temperature of 304 K on a Bruker Kappa Apex II CCD Diffractometer using graphite monochromate Mo-Kα (λ = 0.71073 Å) radiation. The crystal structure **3f** was solved by the direct methods SHELXS. The crystal structure of **3f** was refined by the full-matrix least-squares method on *F*2 using *SHELXL* programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned

geometrically and refined using a riding model. The geometrical calculations were carried out using the program *PLATON*. The molecular and packing diagrams were generated using the software *MERCURY*. The details of the crystal structure and data refinement was listed **in Table S2**. The synthesized title compound was crystallized in the Monoclinic, crystal system in the P 21/n space group with unit cell parameters a = 9.8987(7) Å, b = 21.3915(15) Å, c = 20.2646(15) Å, α = 90º, β = 96.076(3)°, γ = 90º, Z = 8 and V = 4266.9(5) $Å^3$.

11.3 X-Ray Structure & Crystallographic data of 6a

Crystallographic data of 5,8-dimethoxy-1,2,3-triphenyltriphenylene **6a** (ellipsoid contour % probability is 50%). The crystals of suitable quality were obtained from chloroform by slow evaporation under air conditions and were analysed by single crystal diffractometer.

CCDC: 2375302

https://www.ccdc.cam.ac.uk/structures/Search?access=referee&ccdc=2375302&Author=Divya+bharathi

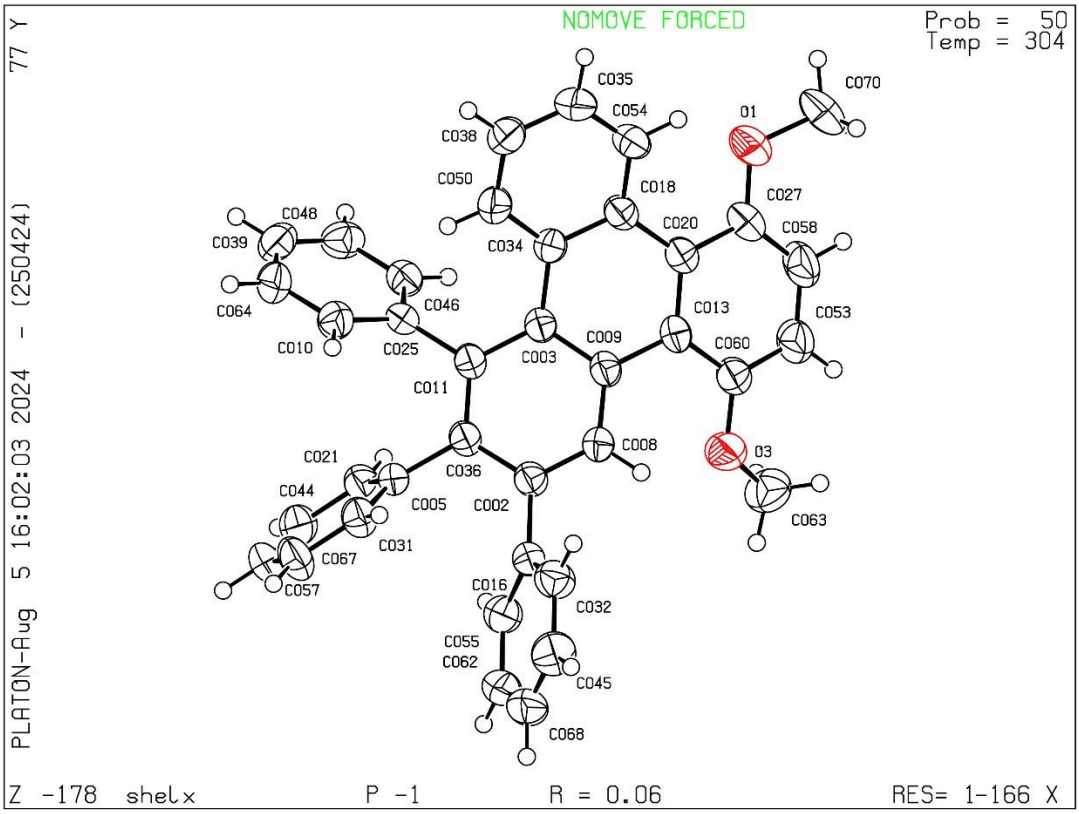

Table S3. Crystal data and structure refinement for 1-c2_red1_abs1_sx.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report
Datablock: shelx

Bond precision:	C-C = 0.0042 A		Wavelength = 0.71073
Cell:	a = 12.1839(9)	b = 13.885(1)	c = 16.4844(12)
	alpha = 91.638(2)	beta = 102.362(2)	gamma = 91.987(2)
Temperature:	304 K		

	Calculated	Reported
Volume	2720.6(3)	2720.6(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C38 H28 O2	C76 H56 O4
Sum formula	C38 H28 O2	C76 H56 O4
Mr	516.60	1033.20
Dx,g cm-3	1.261	1.261
Z	4	2
Mu (mm-1)	0.076	0.076
F000	1088.0	1088.0
F000'	1088.45	
h,k,lmax	14,16,19	14,16,19
Nref	9861	9752
Tmin,Tmax	0.980,0.996	0.979,0.996
Tmin'	0.979	

Correction method= # Reported T Limits: Tmin = 0.979 Tmax = 0.996 AbsCorr = MULTI-SCAN

Data completeness = 0.990 Theta(max) = 25.248

R(reflections) = 0.0572(5578) wR2(reflections) = 0.1757(9752)

S = 0.985 Npar = 721

Computing Details and Refinement

The **6a** single-crystal dimensions were 0.284 x 0.221 x 0.058 mm^3 chosen for an X-ray diffraction study. The X-ray diffraction intensity data was collected at a temperature of

304 K on a Bruker Kappa Apex II CCD Diffractometer using graphite monochromate Mo-Kα (λ = 0.71073 Å) radiation. The crystal structure **6a** was solved by the direct methods SHELXS. The crystal structure of **6a** was refined by the full-matrix least-squares method on *F*2 using *SHELXL* programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned geometrically and refined using a riding model. The geometrical calculations were carried out using the program *PLATON*. The molecular and packing diagrams were generated using the software *MERCURY*. The details of the crystal structure and data refinement was listed in **Table S3**. The synthesized title compound was crystallized in the Triclinic, crystal system in the P -1 space group with unit cell parameters a = 12.1839(9) Å, b = 13.885(1) Å, c = 16.4844(12) Å, α = 91.638(2)º, β = 102.362(2)°, γ = 91.987(2)º, Z = 4 and V = 2720.6(3) $Å^3$.

11.4 X-Ray Structure & Crystallographic data of 6e

Crystallographic data of 4,7-dimethoxy-9-(thiophen-2-yl)acenaphtho[1’2’:7,8]phenanthro [9,10-*b*]thiophene **6e** (ellipsoid contour % probability is 50%). The crystals of suitable quality were obtained from chloroform by slow evaporation under air conditions and were analysed by single crystal diffractometer.

CCDC: 2375531

https://www.ccdc.cam.ac.uk/structures/Search?access=referee&ccdc=2375531&Author=PAVUNKUMAR+pavunkumar2002

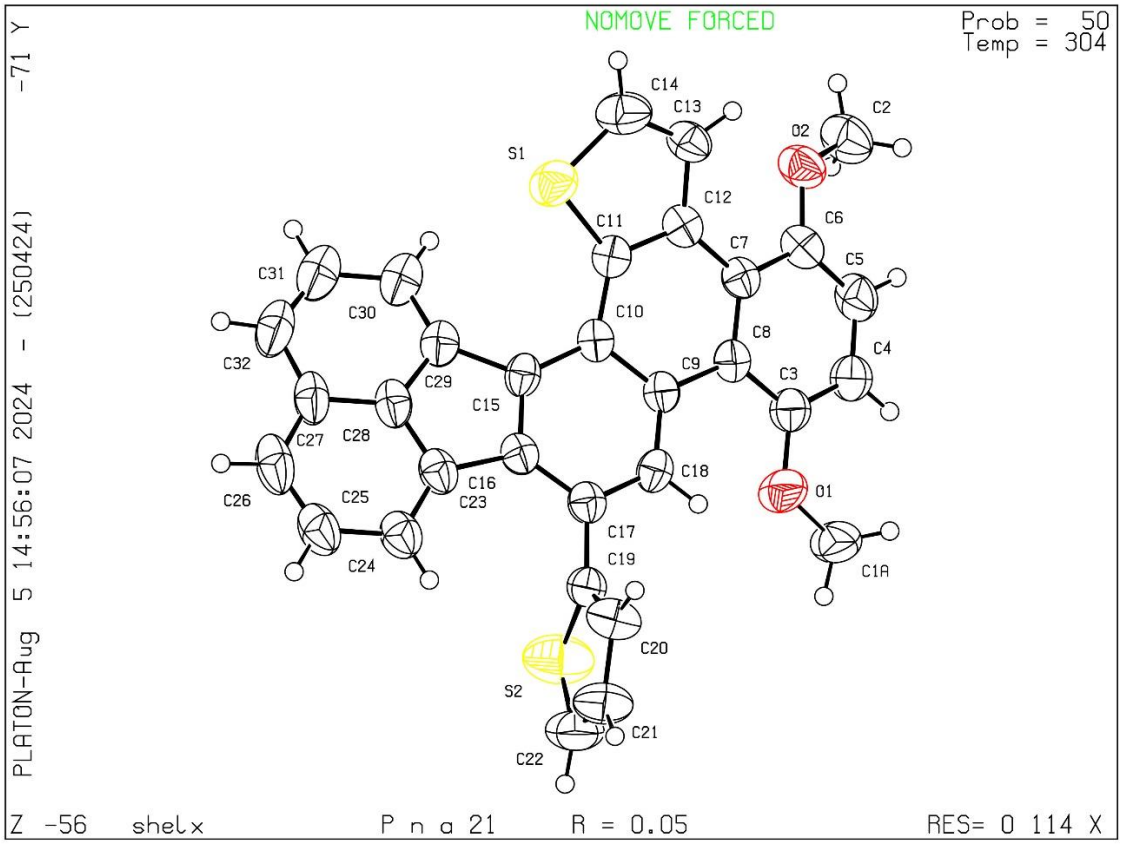

Table S4. Crystal data and structure refinement for 1-c2_red1_abs1_sx.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0089 A Wavelength = 0.71073

Cell: a = 12.8414(7) b = 18.5591(11) c = 10.0685(6)

alpha = 90 beta = 90 gamma = 90

Temperature: 304 K

	Calculated	Reported
Volume	2399.6(2)	2399.6(2)
Space group	P n a 21	P n a 21
Hall group	P 2c -2n	P 2c -2n
Moiety formula	C32 H20 O2 S2	C32 H20 O2 S2
Sum formula	C32 H20 O2 S2	C32 H20 O2 S2
Mr	500.60	500.60
Dx,g cm-3	1.386	1.386
Z	4	4
Mu (mm-1)	0.252	0.252
F000	1040.0	1040.0
F000'	1041.39	
h,k,lmax	15,22,11	15,22,11
Nref	4250[2257]	4233
Tmin,Tmax	0.944,0.984	0.659,1.000
Tmin'	0.922	

Correction method= # Reported T Limits: Tmin = 0.659 Tmax = 1.000 AbsCorr = MULTI-SCAN

Data completeness = 1.88/1.00 Theta(max) = 25.050

R(reflections) = 0.0506(3072) wR2(reflections) = 0.1384(4233)

S = 1.057 Npar = 333

Computing Details and Refinement

The **6e** single-crystal dimensions were 0.322 x 0.192 x 0.063 mm^3 chosen for an X-ray diffraction study. The X-ray diffraction intensity data was collected at a temperature of 304 K on a Bruker Kappa Apex II CCD Diffractometer using graphite monochromate Mo-Kα (λ = 0.71073 Å) radiation. The crystal structure **6e** was solved by the direct methods SHELXS. The crystal structure of **6e** was refined by the full-matrix least-squares method on *F*2 using *SHELXL* programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned geometrically and refined using a riding model. The geometrical calculations were carried out using the program *PLATON*. The molecular and packing diagrams were generated using the software *MERCURY*. The details of the crystal structure and data refinement was listed in **Table S4**. The synthesized title compound was crystallized in the Orthorhombic, crystal system in the P n a 21 space group with unit cell parameters a = 12.8414(7) Å, b = 18.5591(11) Å, c = 10.0685(6) Å, α = 90º, β = 90°, γ = 90º, Z = 4 and V = 2399.6(2) $Å^3$.

11.5 X-Ray Structure & Crystallographic data of 10

Crystallographic data of naphtho[1,2-*b*]phenanthro[4,3-*d*]thiophene *S, S*-dioxide **10** (ellipsoid contour % probability is 50%). The crystals of suitable quality were obtained from chloroform by slow evaporation under air conditions and were analysed by single crystal diffractometer.

CCDC: 2376199

https://www.ccdc.cam.ac.uk/structures/Search?access=referee&ccdc=2376199&Author=Divya+bharathi

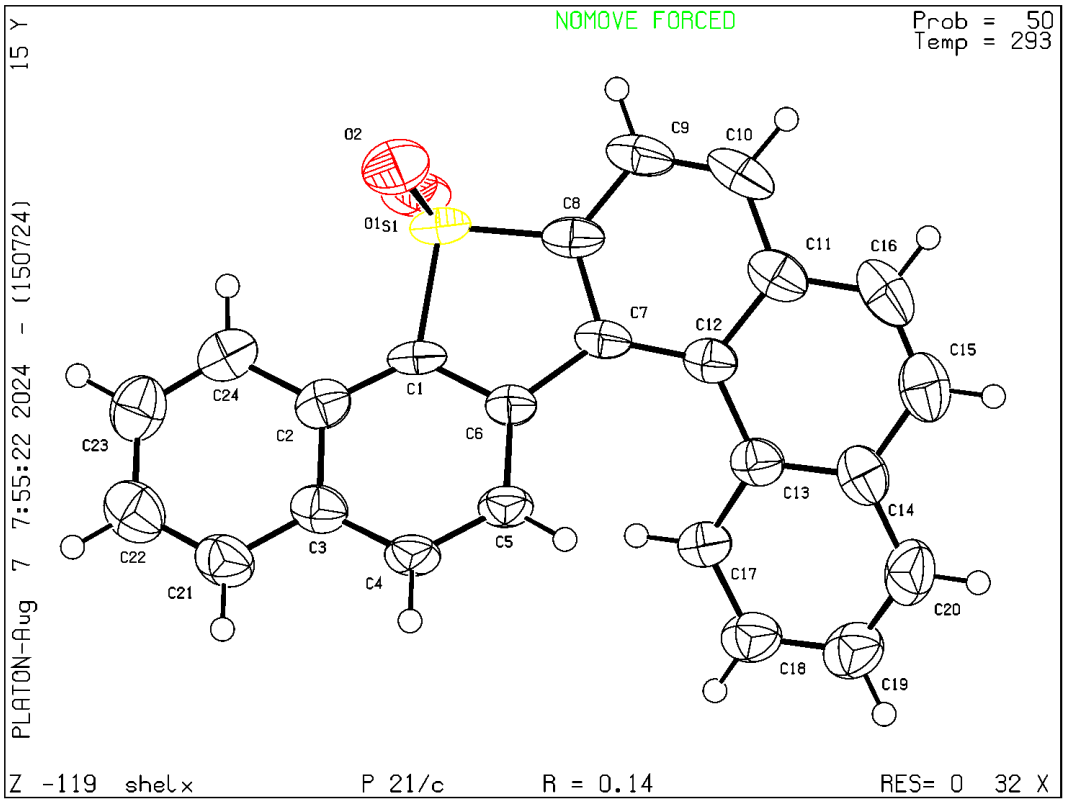

Table S5. Crystal data and structure refinement for 1-c2_red1_abs1_sx.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0106 A Wavelength = 0.71073

Cell: a = 10.7358(17) b = 7.2630(11) c = 22.198(4)

alpha = 90 beta = 95.021(5) gamma = 90

Temperature: 293 K

	Calculated	Reported
Volume	1724.2(5)	1724.2(5)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C24 H14 O2 S	C24 H14 O2 S

Sum formula	C24 H14 O2 S	C24 H14 O2 S
Mr	366.41	366.41
Dx,g cm-3	1.411	1.411
Z	4	4
Mu (mm-1)	0.205	0.203
F000	760.0	760.0
F000'	760.82	
h,k,lmax	12,8,26	12,8,26
Nref	3176	3169
Tmin,Tmax	0.970, 0.983	0.983, 0.995
Tmin'	0.909	

Correction method= # Reported T Limits: Tmin = 0.983 Tmax = 0.995 AbsCorr = MULTI-SCAN

Data completeness = 0.998 Theta(max) = 25.403

R(reflections) = 0.1406(2537) wR2(reflections) = 0.3752(3169)

S = 1.133 Npar = 244

Computing Details and Refinement

The **10** single-crystal dimensions were 0.465 x 0.125 x 0.085mm^3 chosen for an X-ray diffraction study. The X-ray diffraction intensity data was collected at a temperature of 293 K on a Bruker Kappa Apex II CCD Diffractometer using graphite monochromate Mo-Kα (λ = 0.71073 Å) radiation. The crystal structure **10** was solved by the direct methods SHELXS. The crystal structure of **10** was refined by the full-matrix least-squares method on *F*2 using *SHELXL* programs. All the non-hydrogen atoms were revealed in the first difference Fourier map itself. All the hydrogen atoms were positioned geometrically and refined using a riding model. The geometrical calculations were carried out using the program *PLATON*. The molecular and packing diagrams were generated using the software *MERCURY*. The details of the crystal structure and data refinement was listed in **Table S5**. The synthesized title compound was crystallized in the Orthorhombic, crystal system in the P 21/c space group with unit cell parameters a = 10.7358(17) Å, b = 7.2630(11) Å, c = 22.198(4) Å, α = 90º, β = 95.021 (5)°, γ = 90º, Z = 4 and V = 1724.2(5) Å^3.