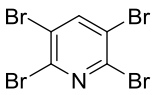
Supporting Information

Content

Experimental Data of Starting Materials

2,3,5,6-tetrabrompyridin 1

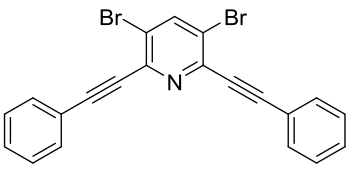


Product 1 could be obtained analogous to the synthesis procedure of Chen and Flowers[5] (27%); mp.: 85-87 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.01 (s, 1H, CH_{Pyr}) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 159.2 (C_{Pyr}), 146.7 (CH_{Pyr}), 134.8 (C_{Pyr}) ppm. IR (ATR): $\tilde{\nu}$ = 3056 (w), 1455 (w), 1284 (w), 1115 (w), 1053 (w), 1026 (w), 985 (w), 740 (m), 649 (w), 602 (w), 439 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 399 ([M]$^+$, $C_5HN^{81}Br_4$, 16), 397 ([M]$^+$, $C_5HN^{79}Br^{81}Br_3$, 66), 395 ([M]$^+$, $C_5HN^{79}Br_2{}^{81}Br_2$, 100), 393 ([M]$^+$, $C_5HN^{79}Br_3{}^{81}Br$, 70) 391 ([M]$^+$, $C_5HN^{79}Br_4$, 17), 318 (13), 316 (39), 314 (40), 312 (40), 237 (11), 233 (11), 156 (11), 154 (10), 75 (41), 74 (10). HRMS (ESI): calcd. for $C_5HN^{79}Br_4$ ([M]$^+$) 390.68370, found 390.68374; calcd. for $C_5HN^{79}Br_3{}^{81}Br$ ([M]$^+$) 392.68165, found 392.68187; calcd. for $C_5HN^{79}Br_2{}^{81}Br_2$ ([M]$^+$) 394.67961, found 394.67980; calcd. for $C_5HN^{79}Br^{81}Br_3$ ([M]$^+$) 396.67756, found 396.67773; calcd. for $C_5HN^{81}Br_4$ ([M]$^+$) 398.67551, found 398.67564.

General procedure A: Synthesis of 3,5-dibromo-2,6-bis(arylethynyl)pyridines **2a-g**

To an argon flushed pressure tube, 2,3,5,6-tetrabromopyridine (**1**) (0.51 mmol, 1.0 eq.), acetylene (1.12 mmol, 2.2 eq.), $Pd(PPh_3)_4$ (10 mol%), CuI (10 mol%) were suspended in 6 mL of diisopropylamine and then stirred at 30 °C for 45 min. After the reaction was completed, distilled water was added and extracted three times with dichloromethane. The organic phases were combined and removed on the rotary evaporator. This was followed by column chromatographic purification with a heptane/dichloromethane mixture. The products in several cases tend to be unstable and had to be used rapidly for the following step.

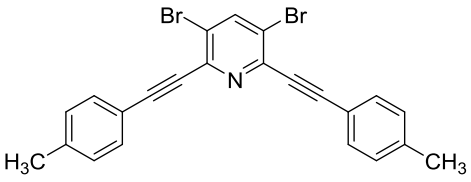
3,5-dibromo-2,6-bis(phenylethynyl)pyridine (2a)



Starting with **1** (200 mg, 0.51 mmol) and phenylacetylene (123 µL, 1.12 mmol), **2a** was isolated as a beige solid (176 mg, 79%); mp.: 128-130 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 8.15 (s, 1H, CH, Pyr), 7.69 - 7.61 (m, 4H, H_{Ph}), 7.42 - 7.33 (m, 6H, H_{Ph}). ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 142.8 ($CH_{Ph/\ Ar}$), 142.6 ($C_{Ph/Ar}$), 132.4, 129.8, 128.6 ($CH_{Ph/Pyr}$), 122.3, 121.7 ($C_{Ph/Pyr}$), 95.5, 86.7 (C_{Alkyne}). IR (ATR): $\tilde{\nu}$ = 2922 (w), 2210 (m), 1517 (m), 1490 (m), 1400 (m), 1208 (m), 1153 (m), 1039 (m), 887 (m), 755 (m), 684 (m), 529 (m) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 440 (11), 439 ([M]$^+$, $C_{21}H_{11}{}^{81}Br_2N$, 49), 438 (23), 437 ([M]$^+$, $C_{21}H_{11}{}^{81}Br\ ^{79}BrN$, 100), 436 (16), 435 ([M]$^+$, $C_{21}H_{11}{}^{79}Br_2N$, 50),

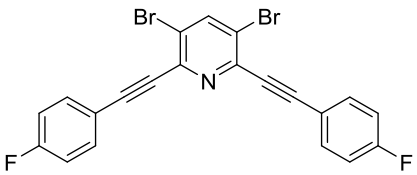
277 (22), 276 (11), 275 (21), 250 (7), 218 (8), 150 (28). HRMS (ESI-TOF): cald. for $C_{21}H_{11}Br_2N$ ($[M+H]^+$) 435.9336, found 435.9331.

3,5-dibromo-2,6-bis(*p*-tolylethynyl)pyridine (2b)



Starting with **1** (200 mg, 0.51 mmol) and 4-methylphenylacetylene (141.5 µL, 1.12 mmol), **2b** was isolated as a beige solid (210.3 mg, 89%); mp.: 173-175 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 8.15 (s, 1H, H_{Pyr}), 7.54 (d, ^3J =8 .1 Hz, 4H, H_{Ar}), 7.21 - 7.16 (m, 4H, H_{Ar}), 2.38 (s, 6H, CH_3) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 142.8 ($CH_{Ar/Pyr}$), 142.8 ($C_{Ar/Pyr}$), 140.2, 132.3, 129.4 ($CH_{Ar/Pyr}$), 122.0, 118.8 ($C_{Ar/Pyr}$), 95.9, 86.4 (C_{Alkyne}), 21.8 (CH_3) ppm. IR (ATR): $\tilde{\nu}$ = 3021 (w), 2950 (w), 2916 (w), 2853 (w), 2205 (m), 1777 (w), 1602 (w), 1518 (w), 1506 (w), 1398 (m), 1206 (w), 1154 (m), 1042 (m), 895 (w), 806 (m), 743 (w), 660 (w), 623 (w), 527 (m), 453 (m), 425 (m) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 468 (12), 467 ($[M]^+$, $C_{23}H_{15}{}^{81}Br_2N$, 51), 466 (27), 465 ($[M]^+$, $C_{23}H_{15}{}^{81}Br^{79}BrN$, 100), 464 (16), 463 ($[M]^+$, $C_{23}H_{15}{}^{79}Br_2N$, 50), 304 (7), 288 (9), 232 (6), 164 (6), 163 (11), 151 (7). HRMS (ESI): cald. for $C_{23}H_{15}{}^{79}Br^{81}BrN$ ($[M]^+$) 464.95453, found 464.95502.

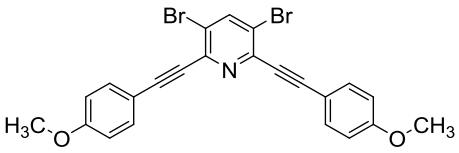
3,5-dibromo-2,6-bis((4-fluorophenyl)ethynyl)pyridine (2c)



Starting with **1** (200 mg, 0.51 mmol) and 4-fluorophenylacetylene (128.1 µL, 1.12 mmol), **2c** was isolated as a beige solid (173.0 mg, 72%); mp.: 169-171 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.19 - 8.17 (s, 1H, H_{Pyr}), 7.63 (dd, ^3J = 9.0 Hz, ^4J = 5.4 Hz, 4H, H_{Ar}), 7.11 - 7.06 (m, 4H, H_{Ar}) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 163.5 (d, $^1J_{C\text{-}F}$ = 252.0 Hz, C_{Ar}), 143.0 (CH_{Ar}), 142.5 (C_{Ar}), 143.5 (d, $^3J_{C\text{-}F}$ = 8.7 Hz, CH_{Ar}), 122.4 (C_{Ar}), 117.9 (d, $^4J_{C\text{-}F}$ = 3.5 Hz, C_{Ar}), 116.1 (d, $^2J_{C\text{-}F}$ = 22.3 Hz, CH_{Ar}), 94.5 (C_{Alkyne}), 86.5 (d, $^5J_{C\text{-}F}$ = 1.4 Hz, C_{Alkyne}) ppm. ^{19}F-NMR (471 MHz, $CDCl_3$): δ = -108.2 (s, 2F, CF) ppm. IR (ATR): $\tilde{\nu}$ = 3062 (w), 2961 (w), 2243 (w), 2213 (m), 1896 (w), 1597 (w), 1537 (w), 1519 (w), 1504 (m), 1402 (m), 1371 (w), 1227 (m), 1217 (m), 1151 (m), 1090 (w), 1044 (m), 832 (m), 727 (m), 526 (m), 532 (m) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 476 (12), 475 ($[M]^+$, $C_{23}H_{15}{}^{81}Br_2F_2N$, 51), 474 (24), 473 ($[M]^+$, $C_{23}H_{15}{}^{81}Br^{79}BrF_2N$, 100), 472 (13), 471 ($[M]^+$,

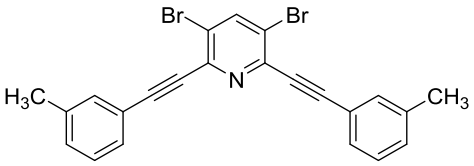
$C_{23}H_{15}{}^{79}Br_2F_2N$, 52), 313 (28), 312 (7), 311 (11), 236 (15), 168 (27). HRMS (ESI-TOF): cald. for $C_{21}H_9F_2{}^{79}Br_2N$ ($[M+H]^+$) 471.9148, found 471.9146.

3,5-dibromo-2,6-bis((4-methoxyphenyl)ethynyl)pyridine (2d)



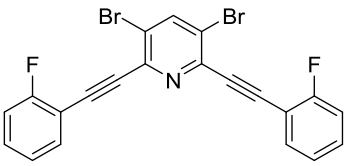
Starting with **1** (200 mg, 0.51 mmol) and 4-methoxyphenylacetylene (145.0 µL, 1.12 mmol), **2d** was isolated as a beige solid (212.2 mg, 84%); mp.: 160-162 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 8.13 (s, 1H, H_{Pyr}), 7.58 (d, ^3J = 8.9 Hz, 4H, H_{Ar}), 6.90 (d, ^3J = 8.9 Hz, 4H, H_{Ar}), 3.84 (s, 6H, OCH_3) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 160.9, 142.8 (C_{Ar}), 142.7, 134.1 (CH_{Ar}), 121.6 (C_{Ar}), 114.3 (CH_{Ar}), 113.8 (C_{Ar}), 96.0, 86.0 (C_{Alkyne}), 55.5 (OCH_3) ppm. IR (ATR): $\tilde{\nu}$ = 3076 (w), 2829 (w), 2245 (w), 2212 (m), 1604 (m), 1568 (w), 1523 (m), 1509 (m), 1417 (w), 1404 (w), 1370 (w), 1295 (m), 1245 (m), 1159 (m), 1106 (w), 1040 (m), 1019 (m), 826 (m), 793 (w), 531 (m). MS (EI, 70 eV): *m/z* (%) = 500 (12), 499 ($[M]^+$, $C_{23}H_{15}{}^{81}Br_2N$, 46), 498 (24), 497 ($[M]^+$, $C_{23}H_{15}{}^{81}Br^{79}BrN$, 100), 496 (19), 495 ($[M]^+$, $C_{23}H_{15}{}^{79}Br_2N$, 51), 484 (8), 482 (14), 480 (8), 251 (12), 250 (10), 249 (8), 248 (15). HRMS (ESI-TOF): calcd. for für $C_{23}H_{15}NO_2{}^{79}Br_2$ ($[M+H]^+$) 495.9548, found 495.9554.

3,5-dibromo-2,6-bis(*m*-tolylethynyl)pyridine (2e)



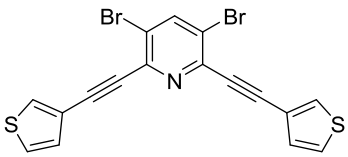
Starting with **1** (200 mg, 0.51 mmol) and 3-methylphenylacetylene (141.1 µL, 1.12 mmol), **2e** was isolated as a beige solid (168.6 mg, 72%); mp.: 124-125 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 8.16 (s, 1H, H_{Pyr}), 7.48 - 7.43 (m, 4H, H_{Ar}), 7.29 - 7.19 (m, 4H, H_{Ar}), 2.37 (s, 6H, CH_3) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 142.8 ($CH_{Ar/Pyr}$), 142.7, 138.4 ($C_{Ar/Pyr}$), 132.9, 130.7, 129.5, 128.5 ($CH_{Ar/Pyr}$), 122.3, 121.6 ($C_{Ar/Pyr}$), 95.8, 86.5 (C_{Alkyne}), 21.3 (CH_3) ppm. IR (ATR): $\tilde{\nu}$ = 2215 (m),1517 (m), 1484 (m), 1395 (s), 1212 (m), 1142 (m), 1039 (s), 892 (s), 872 (m), 775 (vs), 744 (s), 682 (vs) cm^{-1} MS (EI, 70 eV): *m/z* (%) = 468 (15), 476 ($[M]^+$, $C_{23}H_{15}{}^{81}Br_2N$, 51), 466 (27), 465 ($[M]^+$, $C_{23}H_{15}{}^{81}Br^{79}BrN$, 100), 464 (21), 463 ($[M]^+$, $C_{23}H_{15}{}^{79}Br_2N$, 49), 304 (9), 288 (8), 232 (8), 164 (7), 163 (14), 152 (11). HRMS (ESI-TOF): cald. for $C_{23}H_{15}{}^{79}Br_2N$ ($[M+H]^+$) 463.9649, found 463.9650.

3,5-dibromo-2,6-bis((2-fluorophenyl)ethynyl)pyridine (2f)



Starting with **1** (200 mg, 0.51 mmol) and 2-fluorophenylacetylene (126.7 µL, 1.12 mmol), **2e** was isolated as a beige solid (163.2 mg, 68%); mp.: 175-182 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.21 (s, 1H, H_{Pyr}), 7.63 (td, ^{3}J = 7.5 Hz, ^{4}J = 1.7 Hz, 2H, H_{Ar}), 7.43 - 7.38 (m, 2H, H_{Ar}), 7.19 - 7.11 (m, 4H, H_{Ar}) ppm. ^{13}C-NMR (126 MHz): δ = 163.3 (d, $^{1}J_{C-F}$ = 254.7 MHz, C_{Ar}), 143.1 ($CH_{Ar/Pyr}$), 142.4 (C_{Ar}), 134.1 ($CH_{Ar/Pyr}$), 131.7 (d, $^{3}J_{C-F}$ = 8.1 Hz, CH_{Ar}), 124.3 (d, $^{4}J_{C-F}$ = 3.8 Hz, CH_{Ar}), 122.8 (C_{Ar}), 115.9 (d, $^{2}J_{C-F}$ = 20.6 Hz, CH_{Ar}), 91.2 (d, $^{4}J_{C-F}$ = 3.3 Hz, C_{Alkin}), 88.9 (C_{Alkin}) ppm. ^{19}F-NMR (471 MHz, $CDCl_3$): δ = -107.6 (s, 2F, CF) ppm. **IR** (ATR): $\tilde{\nu}$ = 3087 (w), 2217 (w), 1574 (w), 1517 (w), 1492 (m), 1447 (w), 1401 (m), 1259 (m), 1220 (w), 1150 (w), 1100 (w), 1045 (m), 826 (w), 754 (s), 579 (w), 580 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 476 (11), 475 ([M]$^+$, $C_{21}H_9{}^{81}Br_2F_2N$, 48), 474 (23), 473 ([M]$^+$, $C_{21}H_9{}^{81}Br^{79}BrF_2N$, 100), 471 ([M]$^+$, $C_{21}H_9{}^{79}Br_2F_2N$, 50), 314 (6), 313 (25), 311 (10), 236 (9), 169 (6), 168 (33), 145 (6), 144 (8). HRMS (ESI-TOF): calcd. for $C_{21}H_9{}^{79}Br_2F_2N$ ([M+H]$^+$ 471.9148, found 471.9153.

3,5-dibromo-2,6-bis(thiophen-3-ylethynyl)pyridine (2g)



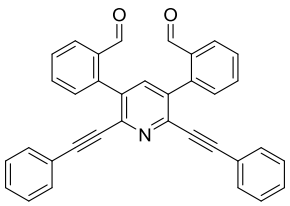
Starting with **1** (200 mg, 0.51 mmol) and 3-ethynylthiophene (110.7 µL, 1.12 mmol), **2g** was isolated as a beige solid (180.5 mg, 79%); mp.: 168-170 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.15 (s, 1H, H_{Pyr}), 7.71 (dd, ^{3}J = 2.9 Hz, ^{4}J = 1.2 Hz, 2H, H_{Thio}), 7.33 (dd, ^{3}J = 5.0 Hz, ^{4}J = 2.9 Hz, 2H, H_{Thio}), 7.28 (dd, ^{3}J = 5.0 Hz, ^{4}J = 1.2 Hz, 2H, H_{Thio}) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 142.9 (CH_{Hetar}), 142.6 (C_{Hetar}), 131.5, 130.1, 125.9 (CH_{Hetar}), 122.0, 120.9 (C_{Hetar}), 90.8, 86.4 (C_{Alkyne}) ppm. IR (ATR): $\tilde{\nu}$ = 2211 (m), 1529 (m), 1424 (m), 1381 (m), 1208 (m), 1142 (m), 1043 (s), 861 (m), 779 (vs), 622 (s) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 453 (6), 452 (12), 451 ([M]$^+$, $C_{17}H_7{}^{81}Br_2NS_2$, 59), 450 (21), 449 ([M]$^+$, $C_{17}H_7{}^{79}Br^{81}BrNS_2$, 100), 448 (11), 447 ([M]$^+$, $C_{17}H_7{}^{79}Br_2NS_2$, 50), 289 (17), 244 (11), 225 (6), 224 (7), 156 (21). HRMS (ESI-TOF): calcd. for $C_{17}H_7{}^{79}Br_2NS_2$ ([M+H]$^+$) 447.8469, found 447.8469.

General procedure B: Synthesis of 2,2'-(2,6-bis(arylethynyl)pyridine-3,5-diyl)arylaldehydes (**4a-l**)

3,5-dibromo-2,6-bis(arylethynyl)pyridines 2a-g (0.275 mmol, 1.0 eq.), boronic acids 3a-f (3.0 eq.), $Pd(OAc)_2$ (10 mol%), SPhos (20 mol%) and K_3PO_4 (4.0 eq.) were suspended in a THF/H_2O mixture (8:1) in an argon flushed pressure tube and stirred at 65 °C for 22 h. For products 4h-l the startingsmaterials 2a and 2g were used. (0.275 mmol, 1.0 eq.) and the corresponding boronic acids (3.0 eq.), $Pd(OAc)_2$ (10 mol%), SPhos (20 mol%), and K_3PO_4 (4.0 eq.) were suspended in a THF/H2O

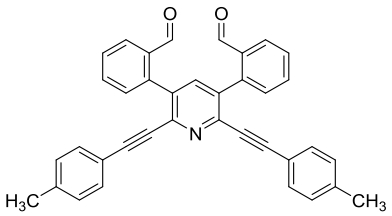
mixture (8:1) in an argon flushed pressure tube and stirred for 22 h at 65 °C After the reaction was completed, distilled water was added to the reaction mixtures and extracted with ethyl acetate. The organic phases were combined and the solvent was removed on the rotary evaporator. This was followed by column chromatographic purification with a heptane/ethyl acetate mixture. The products in several cases tend to be unstable and had to be used rapidly for the following step.

2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4a)



Starting with **2a** (120.2 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4a** was isolated as a beige solid (120.7 mg, 90%); mp.: 187-189 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 10.11 (s, 2H, C*H*O), 8.14 (dd, ^{3}J = 7.7 Hz, ^{4}J = 1.2 Hz, 2H, H_{Ar}), 7.77 (d, J = 1.5 Hz, 1H, H_{Pyr}), 7.73 (d, ^{3}J = 7.5 Hz, ^{4}J = 1.5 Hz, 2H, H_{Ar}), 7.71 - 7.60 (m, 2H, H_{Ar}), 7.54 (dd, ^{3}J = 7.6 Hz, ^{4}J = 1.4 Hz, 2H, H_{Ar}), 7.39 - 7.20 (m, 10H, H_{Ph}) ppm. ^{13}C-NMR (75 Hz, $CDCl_3$): δ = 190.8 (C=O), 143.0, 140.4 (C_{Ar}), 139.1 (CH_{Ar}), 135.9 (C_{Ar}), 134.5 (bs, C_{Ar}), 134.0 (bs, CH_{Ar}), 132.1 (CH_{Ar}), 131.7 (bs, CH_{Ar}), 129.6, 129.5 (CH_{Ar}), 128.7 (bs, CH_{Ar}), 128.5 (CH_{Ar}), 121.7 (C_{Ar}), 94.9, 87.4 (C_{Alkyne}) cm^{-1}. IR (ATR): $\tilde{\nu}$ = 3057 (w), 2923 (w), 2847 (w), 2746 (w), 2211 (w), 1691 (w), 1594 (w), 1570 (w), 1419 (m), 1393 (m), 1194 (m), 1093 (w), 1069 (w), 1000 (w), 825 (w), 754 (m), 687 (m), 634 (w), 532 (m), 443 (w). MS (EI, 70 eV): *m/z* (%) = 488 (12), 487 ($[M]^+$, $C_{35}H_{21}NO_2$, 35), 486 (20), 460 (8), 459 (34), 458 (72), 456 (9), 432 (14), 431 (57), 430 (100), 429 (10), 428 (23) 426 (9), 354 (10), 352 (14). HRMS (EI): calcd. for $C_{35}H_{21}NO_2$ ($[M]^+$) 487.15668, found 487.15624.

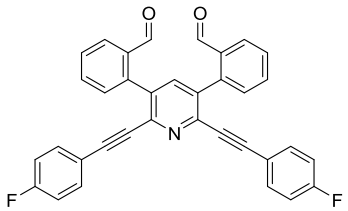
2,2'-(2,6-bis(*p*-tolylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4b)



Starting with **2b** (125.6 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4b** was isolated as a beige solid (94.7 mg, 68%); mp.: 201-203 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 10.09 (s, 2H, C*H*O), 8.11 (dd, ^{3}J = 7.6 Hz, ^{4}J = 1.1 Hz, 2H, H_{Ar}), 7.74 (d, J = 1.5, 1H, H_{Pyr}), 7.70 (dd, ^{3}J = 7.5 Hz, ^{3}J = 1.6 Hz, 2H, H_{Ar}), 7.65 - 7.58 (m, 2H, H_{Ar}), 7.51 (dd, ^{3}J = 7.4 Hz, ^{4}J = 0.8 Hz, 2H, H_{Ar}), 7.13 (d, ^{3}J = 8.2 Hz, 4H, H_{Ar}), 7.06 (d, ^{3}J = 8.0 Hz, 4H, H_{Ar}), 2.30 (s, 6H, CH_3) ppm. ^{13}C NMR (63 MHz, $CDCl_3$): δ = 190.6 (C=O), 143.0, 140.4, 139.8 (C_{Ar}), 138.9 (CH_{Ar}), 135.5, 134.4 (bs, C_{Ar}), 133.8 (bs, CH_{Ar}), 131.8, 131.5 (bs, CH_{Ar}), 129.3, 129.2 (CH_{Ar}), 128.4 (bs, CH_{Ar}), 118.5 (C_{Ar}), 95.2, 87.0 (C_{Alkyne}), 21.6 (CH_3) ppm. IR (ATR): $\tilde{\nu}$ = 2917 (w), 2844 (w), 2754 (w), 2203 (w), 1693 (m), 1594 (w), 1510 (w), 1410 (w), 1392 (w), 1271 (w), 1195 (w), 945 (w), 811 (m), 768 (m), 658 (w), 634 (w), 529 (m), 444 (w), 426 (w). MS

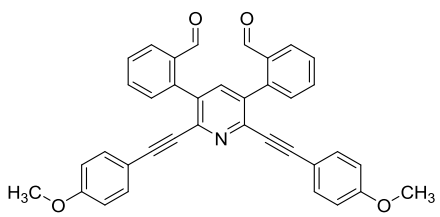
(EI, 70 eV): *m/z* (%) = 516 (27), 515 ([M]$^+$, $C_{37}H_{25}NO_2$, 82), 514 (36), 500 (10), 488 (16), 487 (56), 486 (94), 472 (17), 470 (10), 460 (21), 459 (83), 458 (100), 456 (11), 444 (10), 442 (12), 366 (10), 214 (10). HRMS (ESI-TOF): calcd. for $C_{37}H_{25}NO_2$ ([M+H]$^+$) 516.1964, found 516.1966.

2,2'-(2,6-bis((4-fluorophenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4c)



Starting with **2c** (130.1 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4c** was isolated as a beige solid (112.3 mg, 78%); mp.: 194-196 °C. ^{1}H-NMR (500 MHz, $CDCl_3$ δ = 10.10 (s, 2H, C*H*O), 8.13 (d, ^3J = 7.6 Hz, 2H, H_{Ar}), 7.79 (s, 1H, H_{Pyr}), 7.76 - 7.72 (m, 2H, H_{Ar}), 7.68 - 7.63 (m, 2H, H_{Ar}), 7.52 (d, ^3J = 7.9 Hz, 2H, H_{Ar}), 7.23 (dd, ^3J = 8.6 Hz, ^4J = 5.4 Hz, 4H H_{Ar}), 6.99 - 6.95 (m, 4H,CH_{Ar}) ppm. ^{13}C-NMR (126 Hz, $CDCl_3$): 190.7 (C=O), 163.3 (d, $^1J_{C\text{-}F}$ = 251.8 Hz, C_{Ar}), 142.9, 140.3 (C_{Ar}), 139.0 (CH_{Ar}), 135.9, 134.4 (C_{Ar}), 134.1 (d, $^3J_{C\text{-}F}$ = 8.5 Hz, CH_{Ar}), 133.9 (CH_{Ar}), 131.6 (bs, CH_{Ar}), 129.5 (CH_{Ar}), 128.7 (bs, CH_{Ar}), 117.7 (d, $^4J_{C\text{-}F}$ = 3.6 Hz, C_{Ar}), 116.0 (d, $^2J_{C\text{-}F}$ = 22.3 Hz, CH_{Ar}), 93.8, 87.1 (C_{Alkyne}) ppm. ^{19}F-NMR (471 MHz, $CDCl_3$): δ = -108.47 (bs, 2F, CF) ppm. **IR** (ATR): $\tilde{\nu}$ = 2919 (w), 2847 (w), 2751 (w), 2210 (w), 1692 (m), 1595 (m), 1506 (m), 1411 (w), 1390 (w), 1228 (m), 1196 (w), 1154 (m), 1001 (w), 944 (w), 833 (m), 763 (m), 657 (w), 635 (w), 530 (w), 448 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 524 (9), 523 ([M]$^+$, $C_{35}H_{19}O_2NF_2$, 26), 522 (11), 495 (27), 494 (51), 468 (13), 467 (55), 466 (100), 464 (15), 370 (10), HRMS (EI): calcd. for $C_{35}H_{19}O_2NF_2$ ([M]$^+$) 523.13784, found 523.13699.

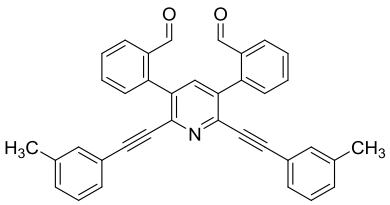
2,2'-(2,6-bis((4-methoxyphenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4d)



Starting with **2d** (136.7 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4d** was isolated as a beige solid (115.9 mg, 77%); mp: 98-100°C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 10.10 (s, 2H, CHO), 8.13 (dd, ^3J = 7.7 Hz, ^4J = 1.2 Hz, 2H, CH_{Ar}), 7.75 (d, J = 1.5 Hz, 1H, CH_{Pyr}), 7.73 - 7.70 (m, 2H, CH_{Ar}), 7.66 - 7.60 (m, 2H, CH_{Ar}), 7.54 - 7.50 (m, 2H, CH_{Ar}), 7.21 - 7.16 (m, 4H, CH_{Ar}), 6.82 - 6.77 (m, 4H, CH_{Ar}), 3.79 (s, 6H, OCH_3) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 190.8 (C=O), 160.7, 143.3, 140.7 (C_{Ar}), 139.0 (CH_{Ar}), 135.1, 134.5 (bs, C_{Ar}), 133.9 (bs, CH_{Ar}), 133.7 (CH_{Ar}), 131.7 (bs, CH_{Ar}), 129.3 (CH_{Ar}), 128.4 (bs, CH_{Ar}), 114.2 (CH_{Ar}), 113.7 (C_{Ar}), 95.4, 86.7 (C_{Alkyne}), 55.5 (OCH_3) ppm. IR (ATR):

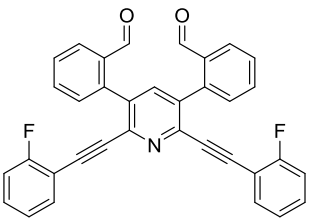
$\tilde{\nu}$ = 2957 (w), 2924 (w), 2747 (w), 2206 (m), 1692 (w), 1601 (m), 1508 (m), 1411 (w), 1247 (m), 1172 (w), 1107 (w), 1025 (m), 960 (w), 907 (w), 829 (m), 757 (w), 727 (m), 536 (m), 513 (w), 443 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 549 (8), 548 (14), 547 ($[M]^+$, $C_{37}H_{25}NO_4$, 71), 546 (31), 520 (13), 519 (47), 518 (100), 492 (8), 491 (25), 490 (33),404 (7), 403 (8), 402 (9), 201 (8). HRMS (EI): calcd. for $C_{37}H_{25}NO_4$ ($[M]^+$) 547.17781, found 547.17846.

2,2'-(2,6-bis(*m*-tolylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4e)



Starting with **2e** (123.2 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4e** was isolated as a beige solid (91.9 mg, 66%); Smp: 189-194°C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 10.10 (s, 2H, C*H*O), 8.13 (d, ^3J = 7.7 Hz, 2H, H_{Ar}), 7.75 (d, J = 1.7 Hz, 1H, H_{Pyr}), 7.73 (dd, ^3J = 7.5 Hz, ^4J = 1.7 Hz, 2H, H_{Ar}), 7.66 - 7.62 (m, 2H, H_{Ar}), 7.53 (d, ^3J = 7.5 Hz, 2H, H_{Ar}), 7.17 - 7.11 (m, 4H, H_{Ar}), 7.07 - 7.03 (m, 4H, H_{Ar}), 2.28 (s, 6H, CH_3) ppm. ^{13}C-NMR (126 MHz, $CDCl_3$): δ = 190.7 (C=O), 143.0, 140.5 (C_{Ar}), 139.0 (CH_{Ar}), 138.1, 135.7 (C_{Ar}), 134.4 (bs, C_{Ar}), 133.9 (bs, CH_{Ar}), 132.5 (CH_{Ar}), 131.6 (bs, CH_{Ar}), 130.4, 129.4, 129.1 (CH_{Ar}), 128.6 (bs, CH_{Ar}), 128.4 (CH_{Ar}), 121.4 (C_{Ar}), 95.1, 87.1 (C_{Alkyne}), 21.3 (CH_3) ppm. IR (ATR): $\tilde{\nu}$ = 2833 (w), 2213 (m), 1605 (m), 1510 (s), 1412 (m), 1294 (m), 1245 (s), 1216 (m), 1156 (m), 1074 (m), 1018 (m), 822 (s), 746 (m), 560 (w), 530 (m) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 516 (26), 515 ($[M]^+$, $C_{37}H_{25}NO_2$, 76), 514 (32), 488 (15), 487 (51), 486 (88), 472 (12), 460 (23), 459 (87), 458 (100), 457 (11), 456 (12), 444 (10), 442 (12), 368 (11), 366 (11), 228 (10), 221 (14) 220 (15), 214 (18), 213 (18). HRMS (EI): calcd. for $C_{37}H_{25}NO_2$ ($[M]^+$) 515.18798, found 515.18807.

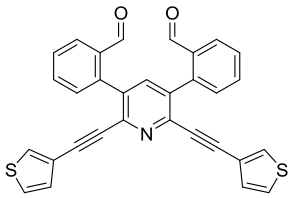
2,2'-(2,6-bis((2-fluorophenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4f)



Starting with **2f** (130.1 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4f** was isolated as a beige solid (105.1 mg, 73%); mp: 223-225°C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 10.09 (s, 2H, C*H*O), 8.10 (dd, ^3J = 7.7 Hz, ^4J = 1.3 Hz, 2H, H_{Ar}), 7.76 - 7.72 (m, 2H, H_{Ar}), 7.70 (d, J = 1.5 Hz, 1H, H_{Pyr}), 7.67 - 7.60 (m, 2H, H_{Ar}), 7.56 - 7.51 (m, 2H, H_{Ar}), 7.38 - 7.27 (m, 4H, H_{Ar}), 7.06 (ddd, ^3J = 7.6 Hz, ^3J = 6.3 Hz, ^4J = 1.2 Hz, 2H, H_{Ar}), 6.99 (ddd, ^3J = 9.4 Hz, ^4J = 6.0 Hz, ^5J = 1.3 Hz, 2H, H_{Ar}) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 191.0 (C=O), 163.1 (d, $^1J_{C-F}$ = 254.6 Hz, C_{Ar}), 142.4, 139.9 (C_{Ar}), 139.3 (CH_{Ar}), 136.5, 134.4, 134.0 (C_{Ar}), 133.9 (CH_{Ar}), 131.3 (d, $^3J_{C-F}$ = 7.9 Hz, CH_{Ar}), 129.4 (CH_{Ar}) 129.3 (C_{Ar}), 129.3 (CH_{Ar}), 124.1 (d, $^4J_{C-F}$ = 3.7 Hz, CH_{Ar}), 115.7 (d, $^2J_{C-F}$ = 20.3 Hz, CH_{Ar}), 110.6, 110.4 (C_{Ar}), 91.9 (d, $^4J_{C-F}$ = 3.1 Hz, C_{Alkyne}), 88.0 (C_{Alkyne}) ppm. ^{19}F-NMR

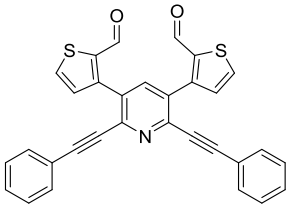
(282 MHz, $CDCl_3$): δ = -108.03 (s, 2F, CF) ppm. IR (ATR): $\tilde{\nu}$ = 2922 (w), 2848 (w), 2228 (w), 1527 (w), 1412 (m), 1348 (w), 1234 (w), 1078 (w), 991 (w), 950 (w), 888 (w), 779 (w), 750 (w), 515 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 524 (14), 523 ($[M]^+$, $C_{35}H_{19}NO_2$, 42), 522 (15), 496 (11), 495 (38), 494 (73), 468 (25), 467 (100), 465 (14), 464 (23), 372 (13), 370 (15), 232 (20), 231 (12), 223 (12), 222 (15). HRMS (EI): calcd. for $C_{35}H_{19}NO_2$ ($[M]^+$) 523.13784, found 523.13763.

2,2'-(2,6-bis(thiophen-3-ylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4g)



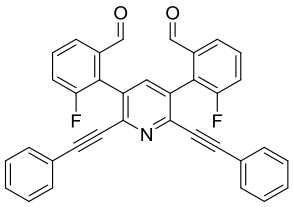
Starting with **2g** (123.5 mg, 0.275 mmol) and 2-formylphenylboronic acid **3a** (123.7 mg, 0.825 mmol), **4g** was isolated as a beige solid (108.5 mg, 73%); mp: 120-122°C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 10.09 (s, 2H, C*H*O), 8.12 (dd, ^3J = 7.7 Hz, ^4J = 1.3 Hz, 2H, H_{Ar}), 7.76 (d, J = 1.5 Hz, 1H, H_{Pyr}), 7.72 (dd, ^3J = 7.5 Hz, ^4J = 1.6 , 2H, H_{Ar}), 7.67 - 7.59 (m, 2H, H_{Ar}), 7.51 (dd, ^3J = 7.61 Hz, ^4J = 0.9 Hz, 2H, H_{Ar}), 7.37 (dd, ^3J = 3.0 Hz, ^4J = 1.2 Hz, 2H, H_{Thio}), 7.22 (dd, ^3J = 5.0 Hz, ^4J = 3.0 Hz, 2H, H_{Thio}), 6.93 (dd, ^3J = 5.0 Hz, ^4J = 1.2 Hz, 2H, H_{Thio}) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 190.7 (C=O), 143.0, 140.4 ($C_{Ar/Hetar}$), 139.0 ($CH_{Ar/Hetar}$), 135.6, 134.5 ($C_{Ar/Hetar}$), 133.9 (bs, $CH_{Ar/Hetar}$), 131.7 (bs, $CH_{Ar/Hetar}$), 131.0, 129.7, 129.4 ($CH_{Ar/Hetar}$), 128.5 (bs, $CH_{Ar/Hetar}$), 125.7 ($CH_{Ar/Hetar}$), 120.8 ($C_{Ar/Hetar}$), 90.3, 87.1 (C_{Alkyne}) ppm. **IR** (ATR): $\tilde{\nu}$ = 2922 (w), 1599.1 (w), 1451 (w), 1284 (w), 1082 (w), 1016 (w), 966 (w), 890 (w), 863 (w), 806 (m), 752 (m), 744 (m), 723 (w), 439 (m) cm^{-1}. HRMS (ESI-TOF): calcd. for $C_{31}H_{17}NO_2S_2$ ($[M+H]^+$) 500.0779, found 500.0781.

3,3'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(thiophene-2-carbaldehyde) (4h)



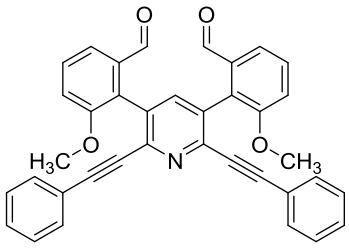
Starting with **2a** (120.2 mg, 0.275 mmol) and (2-formylthiophen-3-yl)boronic acid **3f** (123.7 mg, 0.825 mmol), **4h** was isolated as a beige solid (129.1 mg, 94%); mp: 170-173°C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 9.96 (d, J = 1.2 Hz, 2H, C*H*O), 7.86 (dd, ^3J = 5.0 Hz, ^4J = 1.2 Hz, 2H, H_{Ar}), 7.81 (s, 1H, H_{Pyr}), 7.42 - 7.31 (m, 12H, H_{Ar}) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 182.7 (C=O), 144.8, 143.0, 140.7 ($C_{Ph/Hetar}$), 139.3, 134.2, 132.3 ($CH_{Ph/Hetar}$), 131.5 ($C_{Ph/Hetar}$), 131.3, 129.9, 128.6 ($CH_{Ph/Hetar}$), 121.5 ($C_{Ph/Hetar}$), 95.1, 87.2 (C_{Alkyne}) ppm. IR (ATR): $\tilde{\nu}$ = 2990 (w), 2850 (w), 2209 (w), 1661 (w), 1490 (w), 1418 (w), 1368 (w), 1197 (w), 1030 (w), 882 (w), 785 (w), 754 (w), 686 (w), 666 (w), 532 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 500 (12), 499 ($[M]^+$, $C_{31}H_{17}NO_2S_2$, 29), 498 (58), 472 (13), 471 (29), 470 (70), 468 (12), 444 (29), 443 (76), 442 (100), 441 (18), 440 (31), 408 (12), 366 (12), 364 (13), 220 (20), 219 (12), 105 (13). HRMS (ESI-TOF): calcd. For $C_{31}H_{17}NO_2S_2$ ($[M]^+$) 500.0779, found 500.0779.

2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-fluorobenzaldehyde) (4i)



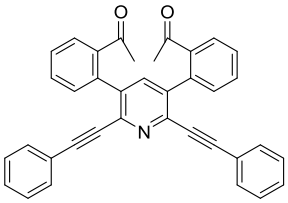
Starting with **2a** (120.2 mg, 0.275 mmol) and (2-fluoro-6-formylphenyl)boronic acid **3c** (138.5 mg, 0.825 mmol), **4i** was isolated as a beige solid (106.4 mg, 80%); mp: 162-164°C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 9.94 (s, 2H, C*H*O), 8.09 (dd, ^{3}J = 8.7 Hz, ^{3}J = 5.8 Hz, 2H, H_{Ar}), 7.68 (s, 1H, H_{Pyr}), 7.30 - 7.15 (m, 14H, H_{Ar}) ppm. ^{13}C -NMR (75 MHz, $CDCl_3$): δ = 189.0 (C=O), 165.7 (d, $^{1}J_{C-F}$ = 258.3 Hz, C_{Ar}), 143.1 (C_{Ar}), 142.8 (d, $^{3}J_{C-F}$ = 9.2 Hz, C_{Ar}), 138.7 (CH_{Ar}), 134.5 (C_{Ar}), 132.0 (CH_{Ar}), 131.8 (d, $^{3}J_{C-F}$ = 8.6 Hz, CH_{Ar}), 131.1 (bs, C_{Ar}), 129.8, 128.6 (CH_{Ar}), 121.3 (C_{Ar}), 118.6 (bs, CH_{Ar}), 116.9 (d, $^{2}J_{C-F}$ = 21.7 Hz, CH_{Ar}), 95.5, 86.9 (C_{Alkyne}) ppm. ^{19}F-NMR (282 MHz, $CDCl_3$): δ = -102.69 (bs, 2F, CF) ppm.IR (ATR): $\tilde{\nu}$ = 2954 (w), 2922 (w), 2866 (w), 2852 (w), 2206 (w), 1689 (m), 1600 (m), 1577 (m), 1521 (w), 1489 (w), 1418 (m), 1240 (m), 1189 (m), 1092 (w), 883 (m), 827 (m), 757 (m), 688 (m), 533 (m), 469 (m). MS (EI, 70 eV): *m/z* (%) = 523 ($[M]^+$, $C_{35}H_{19}F_2NO_2$, 26), 522 (15), 495 (29), 494 (62), 468 (14), 467 (55), 466 (100), 464 (17), 390 (10), 388 (11). HRMS (EI): calcd. for $C_{35}H_{19}F_2NO_2$ ($[M]^+$) 523.13784, found 523.13632.

2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-methoxybenzaldehyde) (4j)



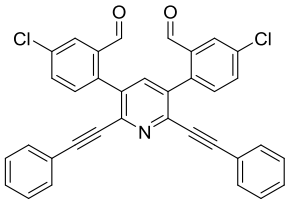
Starting with **2a** (120.2 mg, 0.275 mmol) and (2-formyl-6-methoxyphenyl)boronic acid **3b** (148.5 mg, 0.825 mmol), **4i** was isolated as a beige solid (140.0 mg, 93%); mp: 100-102 °C. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 9.95 (s, 2H, C*H*O), 8.10 (d, ^{3}J = 8.7 Hz, 2H, H_{Ar}), 7.75 (s, 1H, H_{Pyr}), 7.35 - 7.25 (m, 10H, H_{Ar}), 7.12 (ddd, ^{3}J = 8.8 Hz, ^{4}J = 2.5 Hz, ^{5}J = 0.7 Hz, 2H, H_{Ar}), 6.98 (m, 2H, H_{Ar}), 3.91 (s, 6H, OCH_3) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 189.5 (C=O), 163.9 (bs, C_{Ar}), 163.8 (bs, C_{Ar}), 142.8 (bs, C_{Ar}), 138.8 (CH_{Ar}), 135.8 (C_{Ar}), 132.1 (CH_{Ar}), 131.1 (bs, CH_{Ar}), 129.5 (CH_{Ar}), 128.5 (bs, CH_{Ar}), 127.9, 121.7 (C_{Ar}), 116.4 (bs, CH_{Ar}), 115.1 (bs, CH_{Ar}), 94.8, 87.3 (C_{Alkyne}), 55.9 (OCH_3) ppm. IR (ATR): $\tilde{\nu}$ = 2931 (w), 2838 (w), 2209 (w), 1682 (m), 1591 (m), 1565 (m), 1490 (m), 1394 (w), 1306 (w), 1284 (w), 1240 (m), 1205 (m), 1175 (w), 1092 (w), 1017 (m), 965 (m), 754 (m), 687 (m), 558 (w), 466 (w). MS (EI, 70 eV): *m/z* (%) = 547 ($[M]^+$, $C_{37}H_{25}NO_4$, 38), 546 (41), 518 (22), 435 (13), 331 (35), 197 (16), 196 (100), 195 (26), 181 (14), 177 (22), 165 (21). HRMS (EI): calcd. for $C_{37}H_{25}NO_4$ ($[M]^+$) 547.17781, found 547.17797.

1,1'-((2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(2,1-phenylene))bis(ethan-1-one) (4k)



Starting with **2a** (120.2 mg, 0.275 mmol) and (2-acetylphenyl)boronic acid **3b** (135.3 mg, 0.825 mmol), **4k** was isolated as a beige solid (89.3 mg, 63%); mp: 212-214 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 7.82 - 7.76 (m, 2H, H_{Ar}), 7.65 - 7.43 (m, 7H, H_{Ar}), 7.33 - 7.18 (m, 10H, H_{Ph}), 2.38 (s, 6H, CH_3) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 201.0 (C=O), 141.0, 139.9, 139.4 (C_{Ar}), 137.6 (CH_{Ar}), 137.2 (C_{Ar}), 132.0 (CH_{Ar}), 131.6 (bs, CH_{Ar}), 131.4, 129.2, 128.7, 128.6, 128.5 (CH_{Ar}), 122.2 (C_{Ar}), 93.3, 87.9 (C_{Alkyne}), 29.2(CH_3) ppm. IR (ATR): $\tilde{\nu}$ = 3058 (w), 2922 (w), 2852 (w), 2215 (w), 1681 (m), 1594 (w), 1489 (m), 1415 (m), 1353 (m), 1284 (m), 1246 (m), 1223 (m), 1071 (w), 953 (m), 919 (m), 758 (m), 691 (m), 597 (m), 531 (m), 478 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 516 (13), 515 ([M]$^+$, $C_{37}H_{25}NO_2$, 39), 514 (39), 486 (7), 474 (13), 473 (50), 472 (100), 444 (11), 431 (10), 430 (29), 429 (11), 428 (22), 426 (9). HRMS (ESI-TOF): calcd. for $C_{37}H_{25}NO_2$ ([M+H]$^+$) 516.1964, found 516.1965.

6,6'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-chlorobenzaldehyde) (4l)



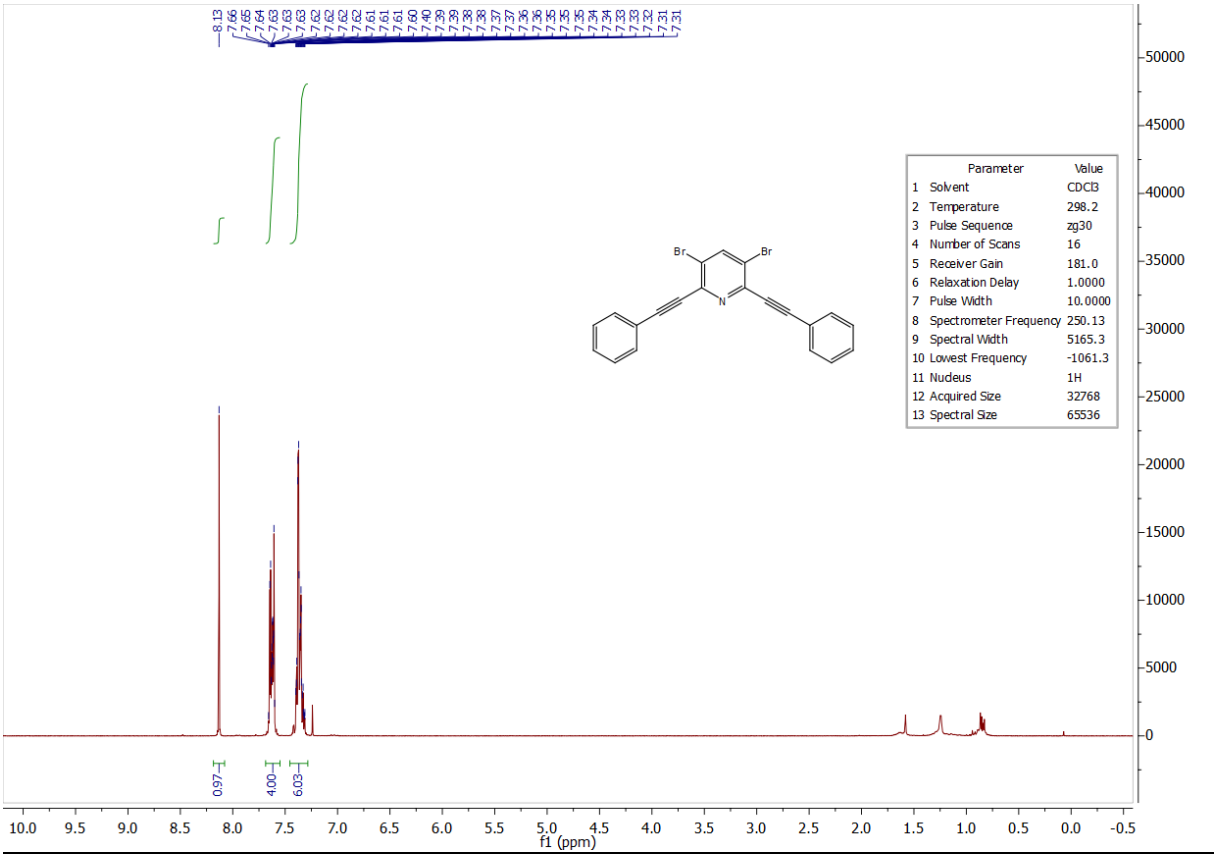
Starting with **2a** (120.2 mg, 0.275 mmol) and (4-cloro-2-formylphenyl)boronic acid **3d** (152.1 mg, 0.825 mmol), **4l** was isolated as a beige solid (105.6 mg, 69%); mp: 158-160 °C. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 9.96 (s, 2H, C*H*O), 8.03 (d, ^{4}J = 2.3 Hz, 2H, H_{Ar}), 7.66 - 7.64 (m, 2H, H_{Ar}), 7.62 (d, ^{4}J = 2.3 Hz, 1H, H_{Ar}), 7.41 (d, ^{3}J = 8.2 Hz, 2H, H_{Ar}), 7.34 - 7.10 (m, 10H, H_{Ph}) ppm. ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 189.2 (C=O), 143.2 (C_{Ar}), 139.0 (CH_{Ar}), 138.4, 136.2 (C_{Ar}), 135.6 (bs, C_{Ar}), 134.7 (C_{Ar}), 133.9 (bs, CH_{Ar}), 133.1, 133.0, 132.0, 129.8, 128.6 (CH_{Ar}), 121.3 (C_{Ar}), 95.5, 87.0 (C_{Alkyne}) ppm. IR (ATR): $\tilde{\nu}$ = 3058 (w), 2850 (w), 2746 (w), 2207 (w), 1694 (m), 1586 (m), 1491 (w), 1422 (m), 1382 (m), 1182 (m), 1112 (w), 999 (w), 892 (m), 832 (m), 754 (m), 686 (m), 542 (m), 464 (w), 447 (w) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 555 ([M]$^+$, $C_{37}H_{19}Cl_2NO_2$, 15), 528 (15), 526 (19), 500 (13), 498 (18), 356 (19), 355 (70), 354 (21), 353 (100), 226 (19), 73 (15). HRMS (ESI-TOF): calcd. for $C_{37}H_{19}Cl_2NO_2$ ([M+H]$^+$) 556.0871, found 556.0870.

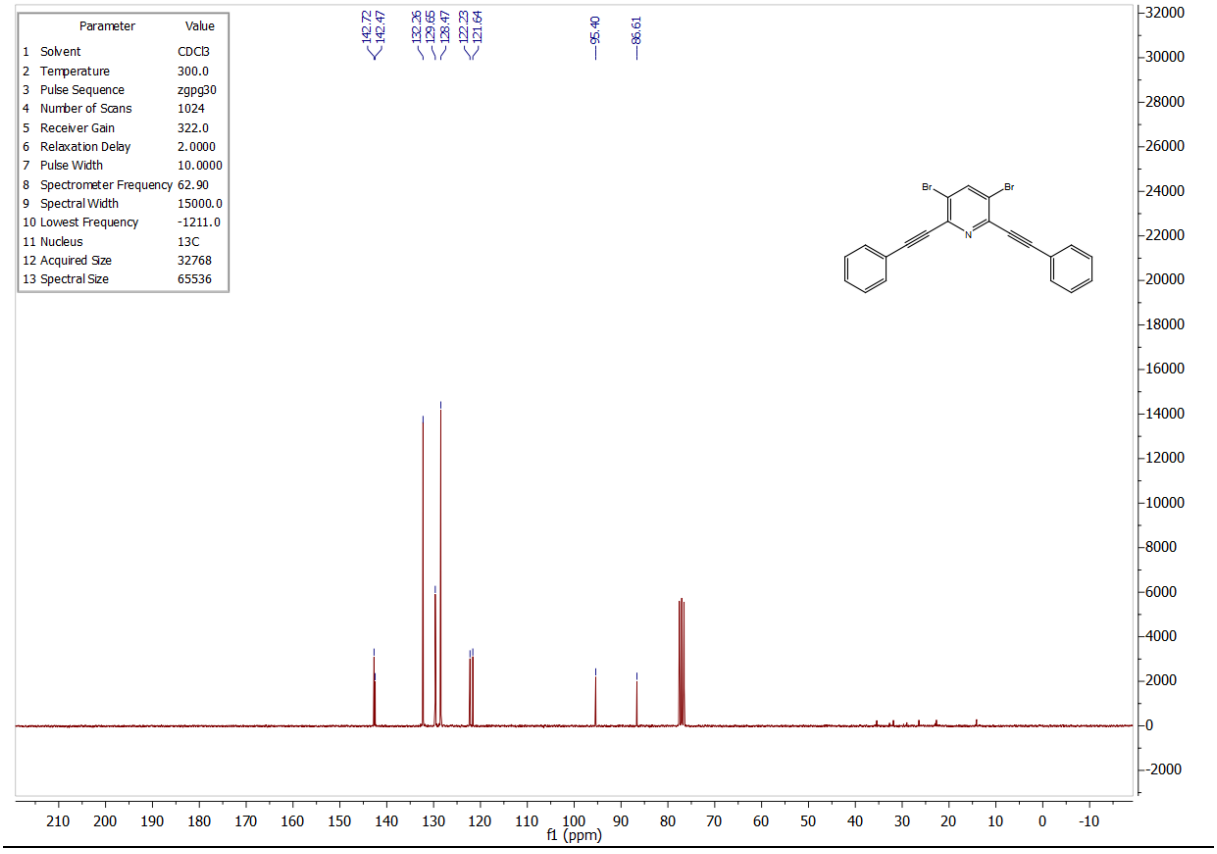
Single-crystal Xray diffraction data

	5a	**5h**
Chem. Formula	$C_{35}H_{21}NO_2$	$C_{31}H_{17}NO_2S_2$
Form. Wght [g mol^{-1}]	487.53	499.57
Cryst. system	monoclinic	monoclinic
Space group (Hall group)	P 21/c (-P 2ybc)	C 2/c (-C 2yc)
Color	beige	beige
a [Å]	7.2154 (11)	15.245 (4)
b [Å]	12.0896 (17)	18.740 (5)
c [Å]	27.443 (4)	8.084 (2)
α [°]	90	90
β [°]	96.196 (4)	100.440 (7)
γ [°]	90	90
V [Å3]	2379.9	2271.3 (11)
Z	4	4
N_{ref}	6308	3939
θ [°]	28.999 (6)	31.998
h,k,l_{max}	9,16,37	22,27,12
D_x [g cm^{-3}]	1.361	1.461
μ [mm^{-1}]	0.084	0.267
$\lambda_{MoK\backslash\alpha}$ [Å]	0.71073	0.71073
T [K]	123 K	123 K
F(000)	1016.0	1032.0
N_{par}	343	164
R	0.0518 (4437)	0.0460 (2964)
wR2	0.1449 (6308)	0.1265 (3939)
S	1.015	1.025

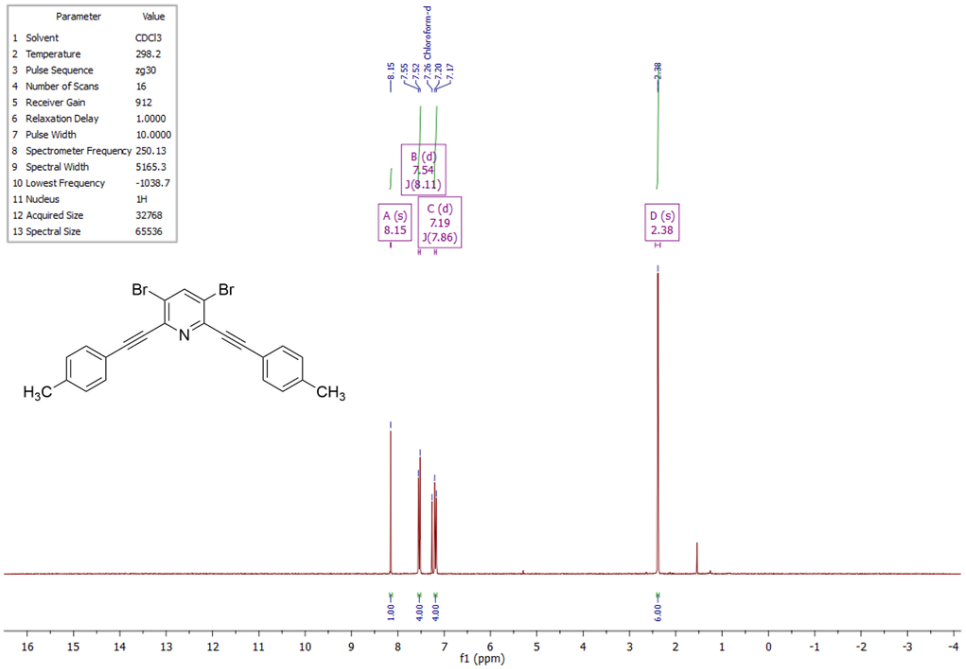
^{1}H, ^{19}F, ^{13}C Spectra

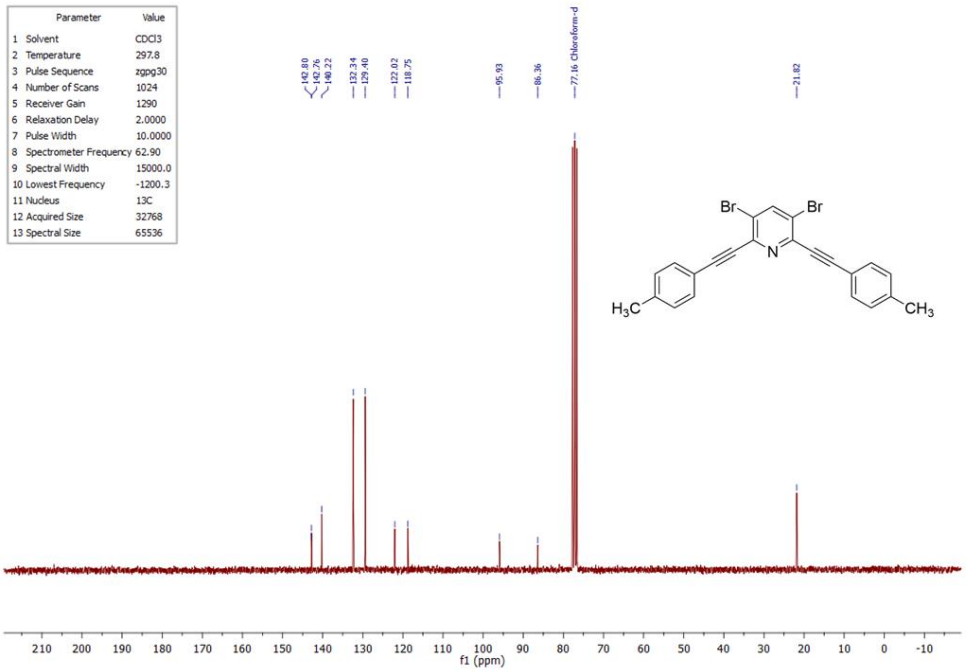
3,5-dibromo-2,6-bis(phenylethynyl)pyridine (2a)



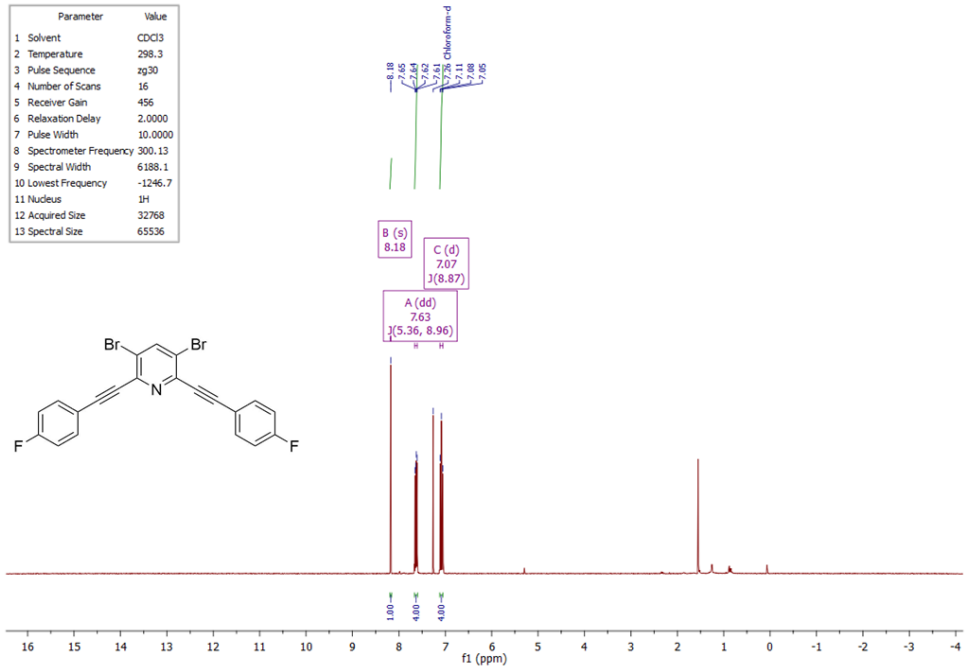


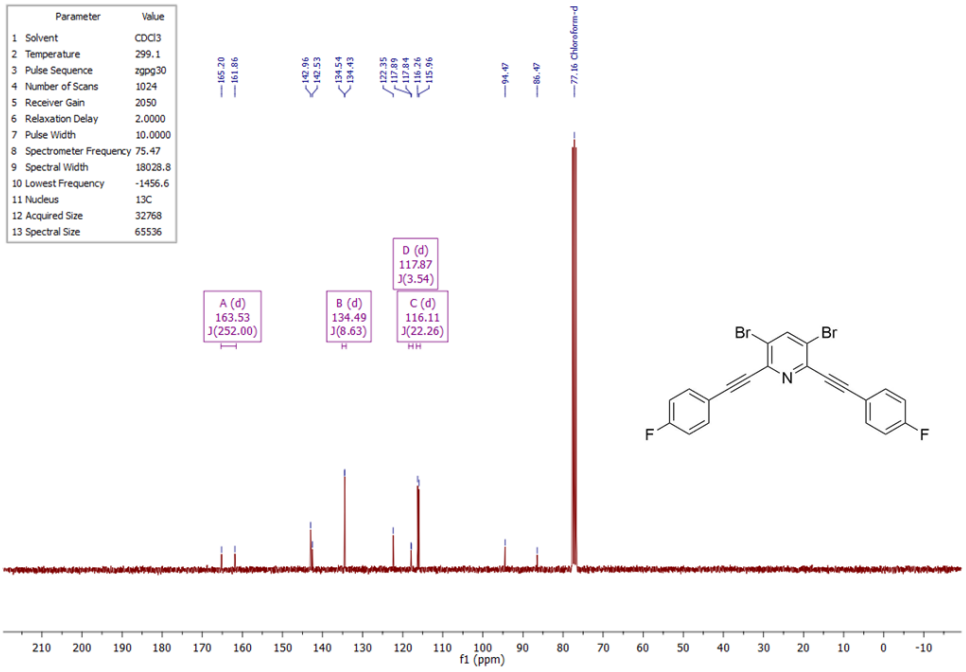
3,5-dibromo-2,6-bis(*p*-tolylethynyl)pyridine (2b)



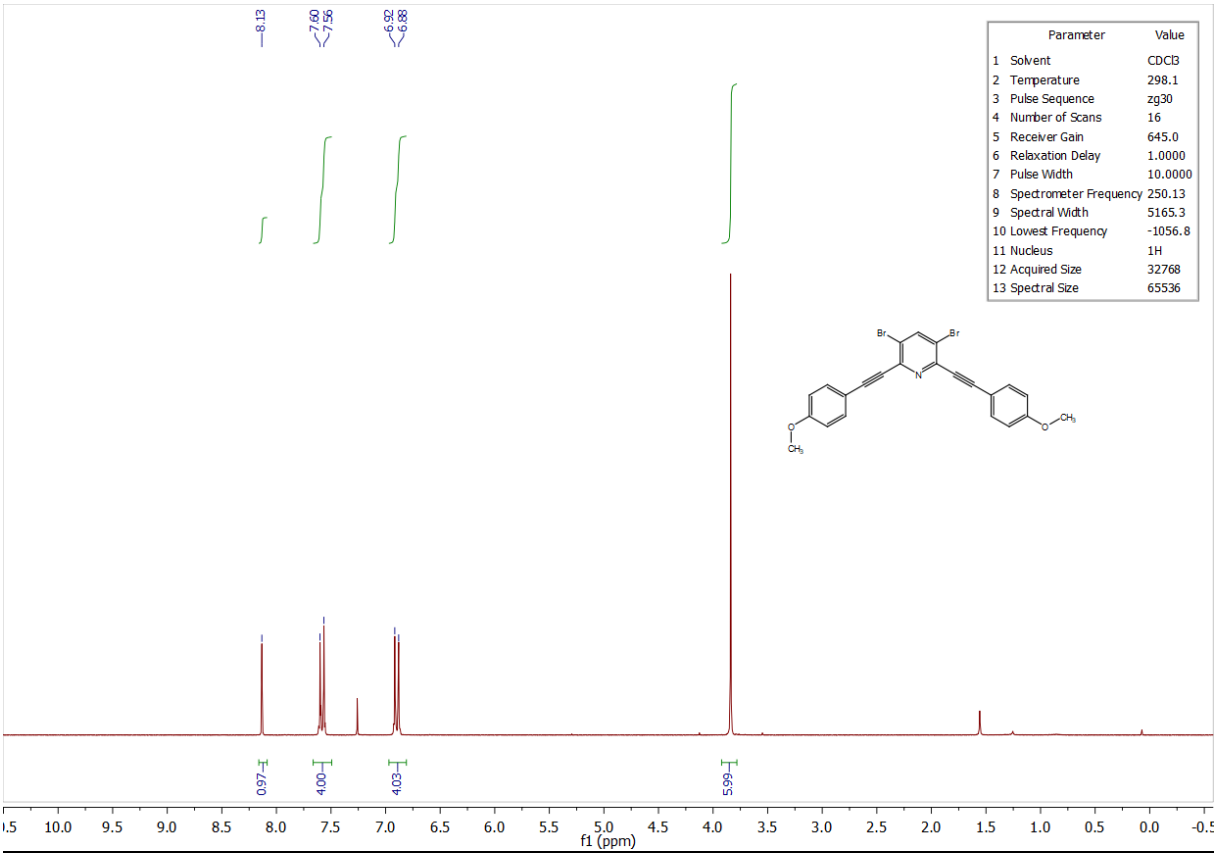


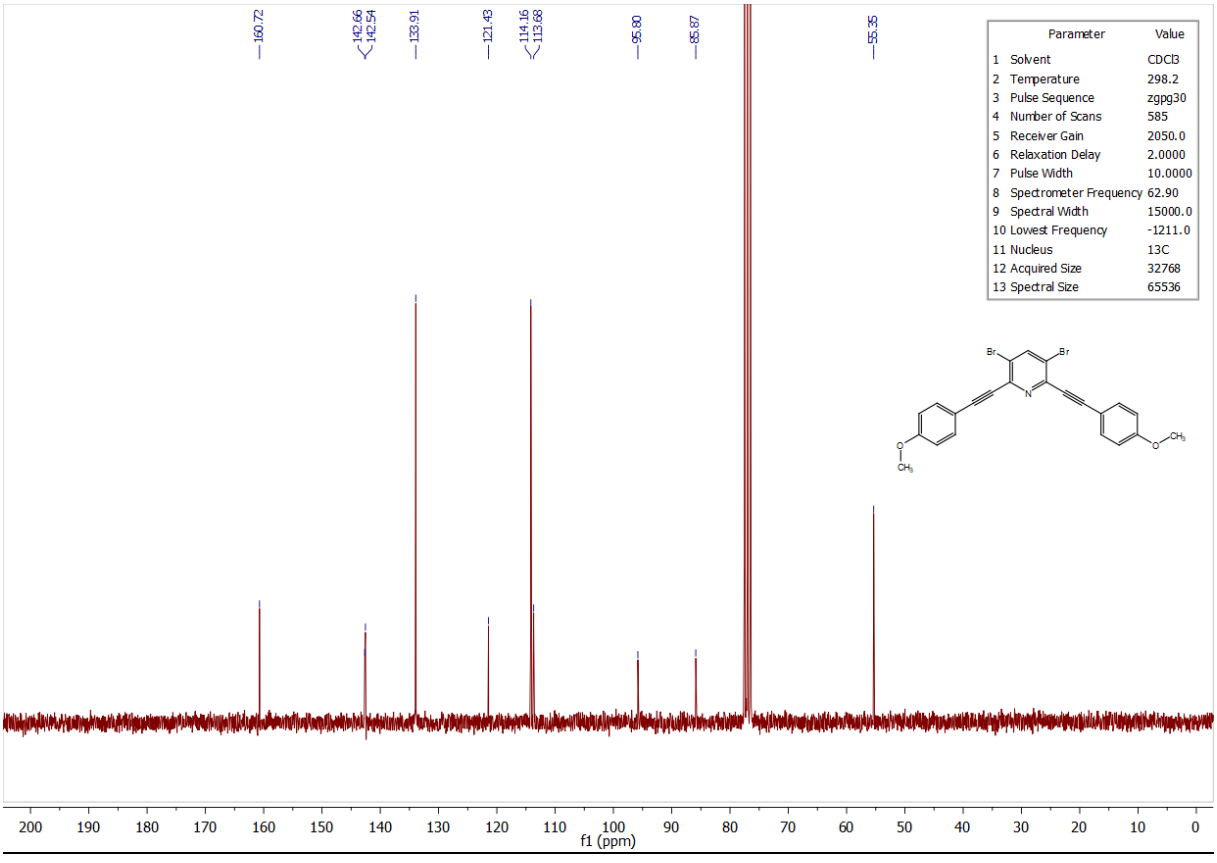
3,5-dibromo-2,6-bis((4-fluorophenyl)ethynyl)pyridine (2c)



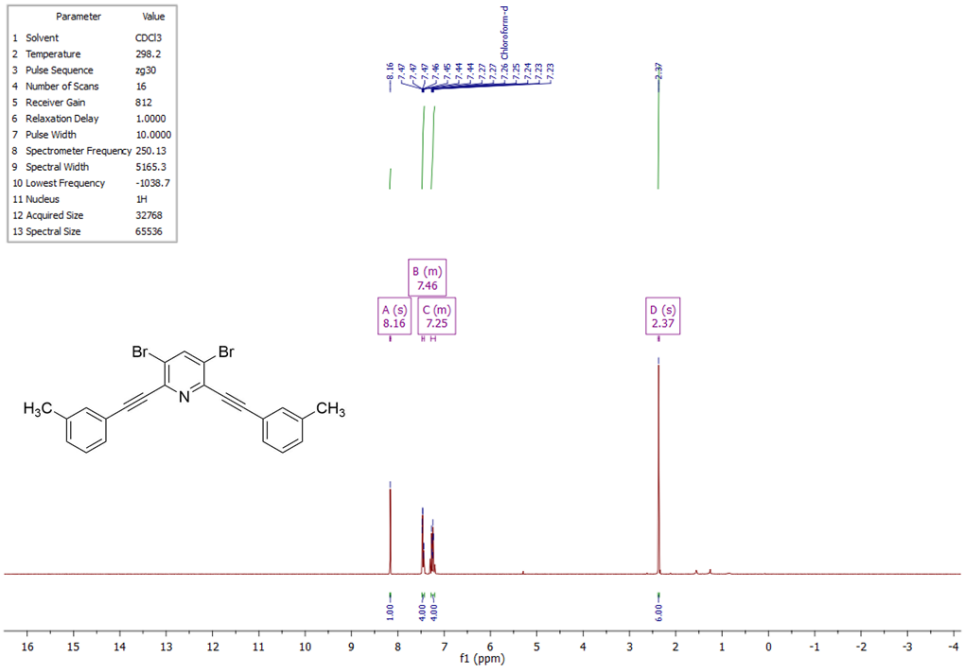


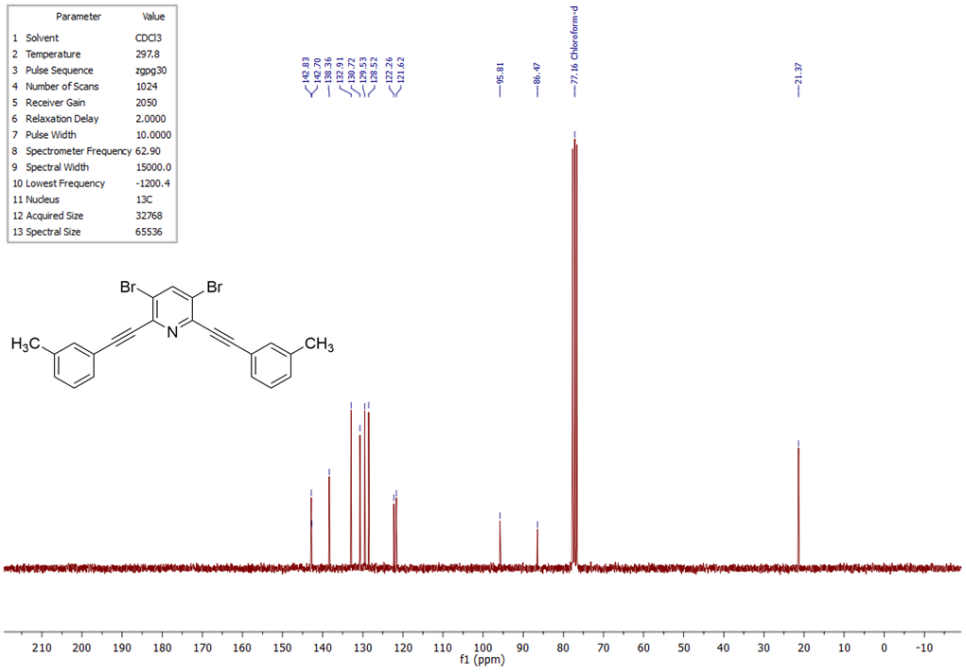
3,5-dibromo-2,6-bis((4-methoxyphenyl)ethynyl)pyridine (2d)



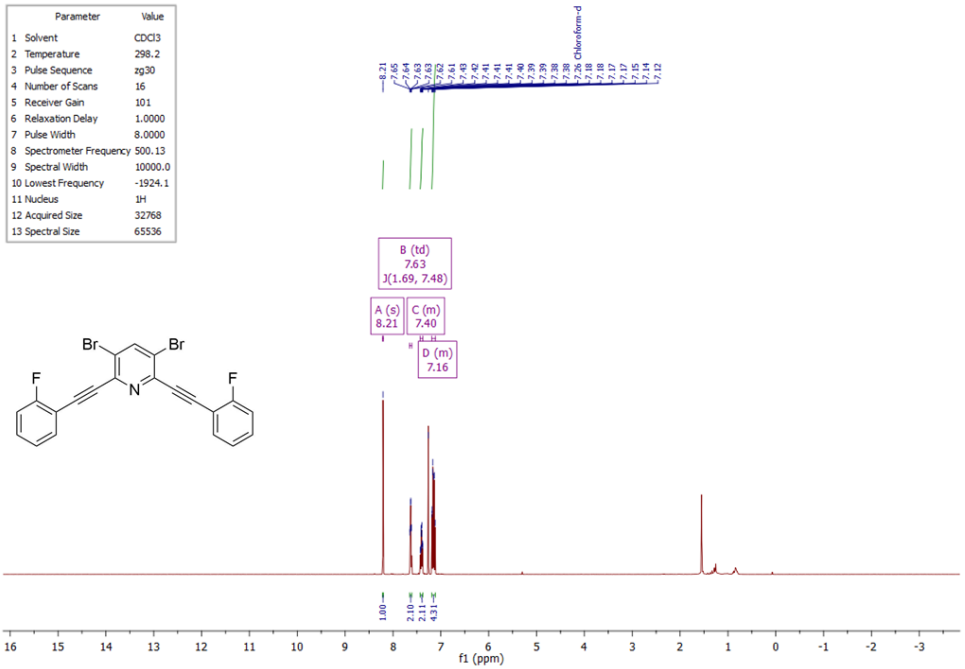


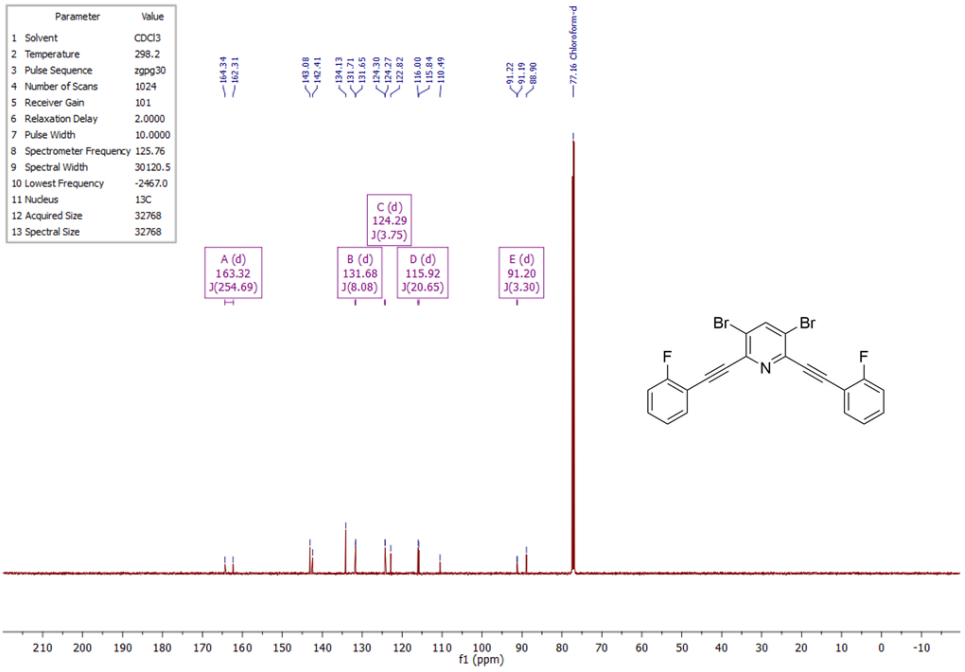
3,5-dibromo-2,6-bis(*m*-tolylethynyl)pyridine (2e)



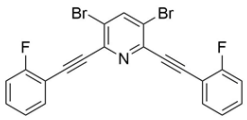


3,5-dibromo-2,6-bis((2-fluorophenyl)ethynyl)pyridine (2f)



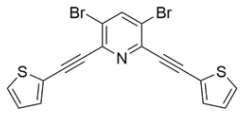


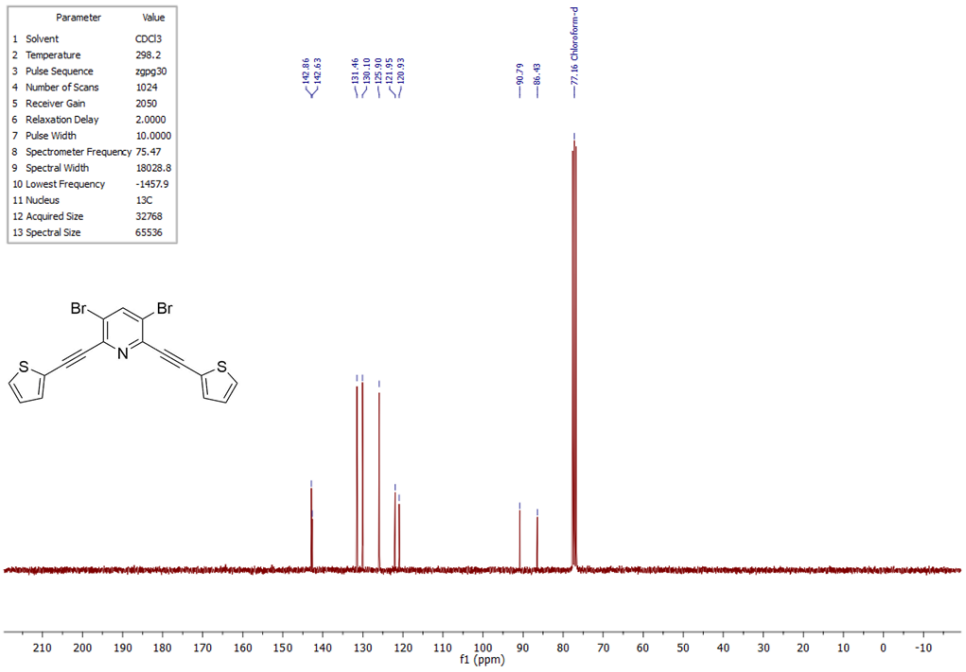
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.2
3	Pulse Sequence	zgig
4	Number of Scans	16
5	Receiver Gain	101
6	Relaxation Delay	1.0000
7	Pulse Width	12.0000
8	Spectrometer Frequency	470.59
9	Spectral Width	113636.4
10	Lowest Frequency	-103877.4
11	Nucleus	19F
12	Acquired Size	65536
13	Spectral Size	65536



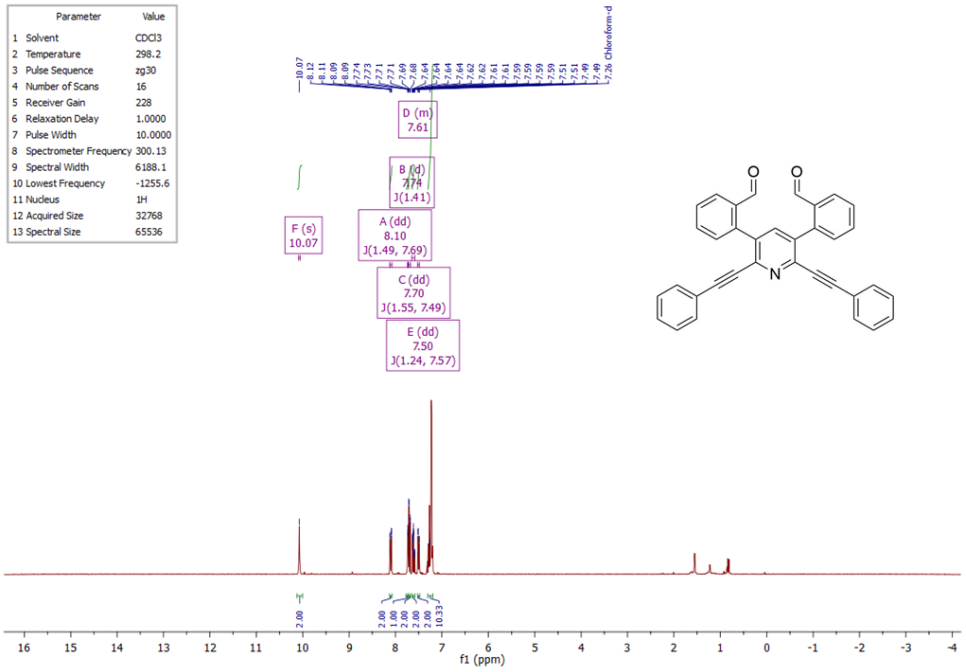
3,5-dibromo-2,6-bis(thiophen-3-ylethynyl)pyridine (2g)

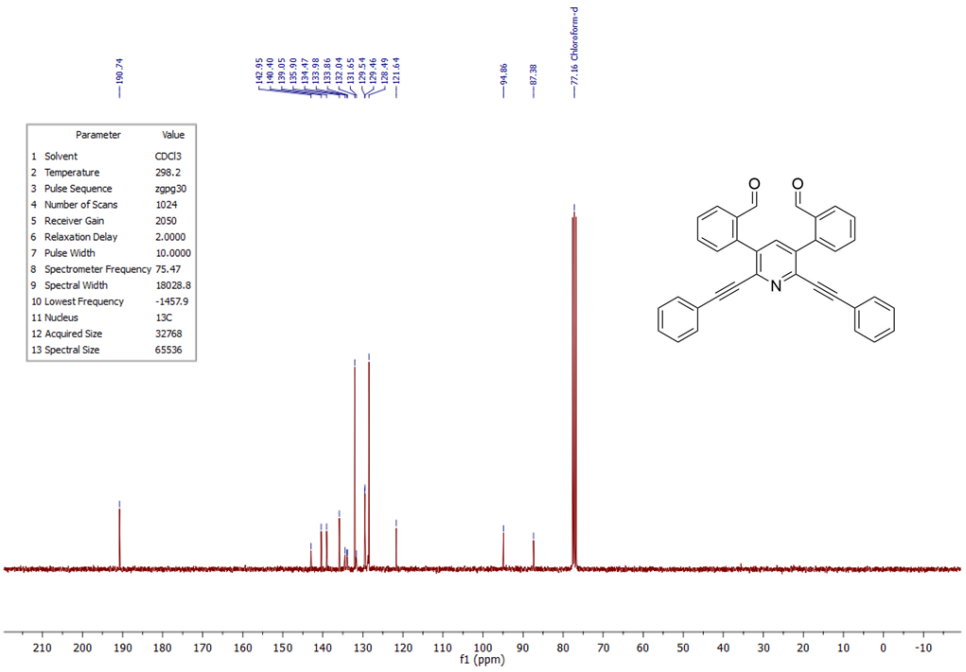
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.2
3	Pulse Sequence	zg30
4	Number of Scans	16
5	Receiver Gain	362
6	Relaxation Delay	2.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	300.13
9	Spectral Width	6188.1
10	Lowest Frequency	-1246.5
11	Nucleus	1H
12	Acquired Size	32768
13	Spectral Size	65536



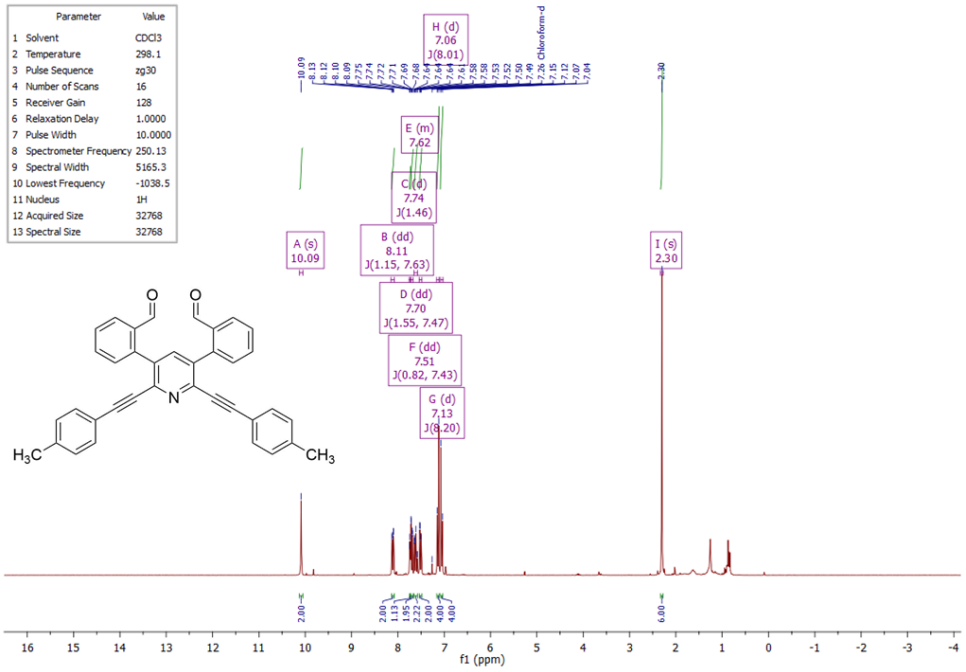


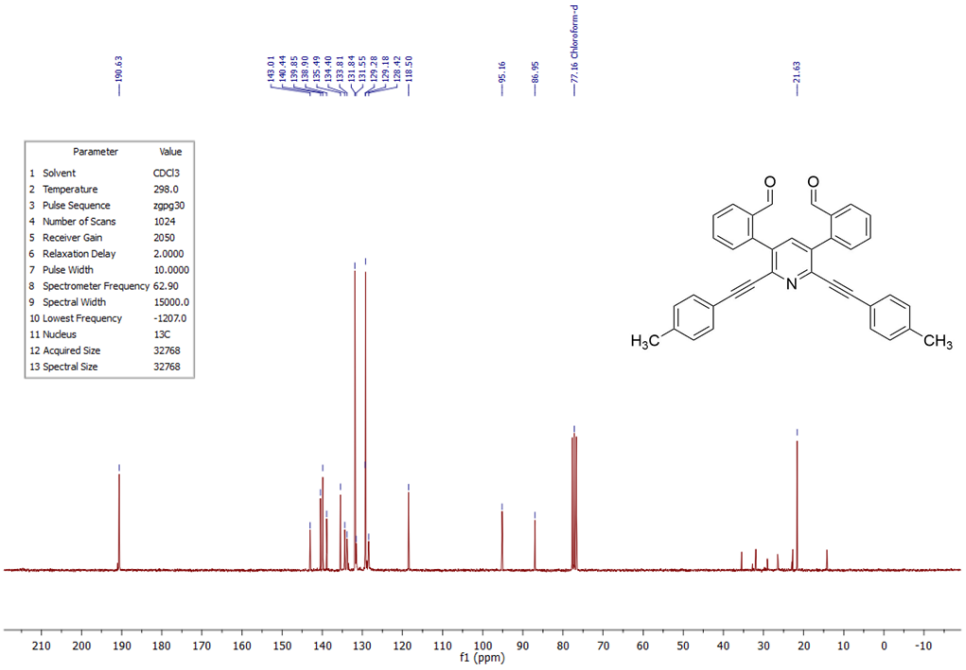
2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4a)



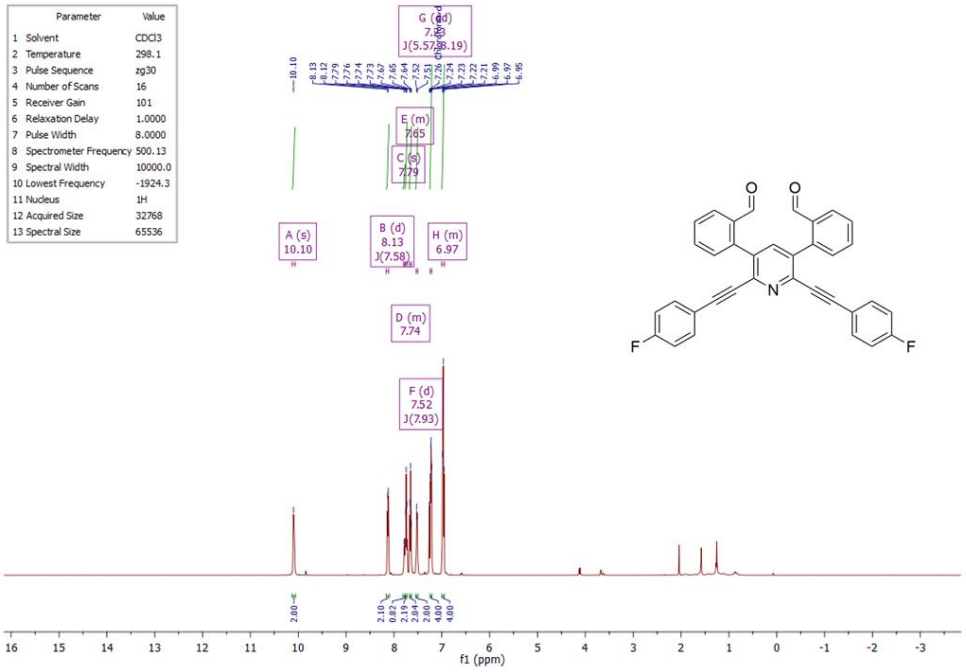


2,2'-(2,6-bis(*p*-tolylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4b)

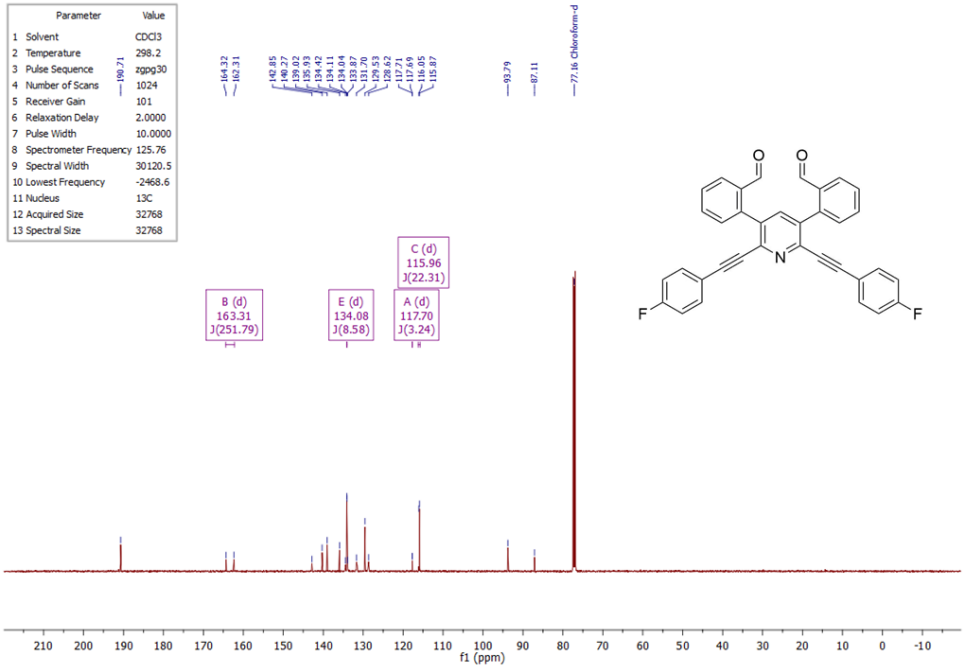




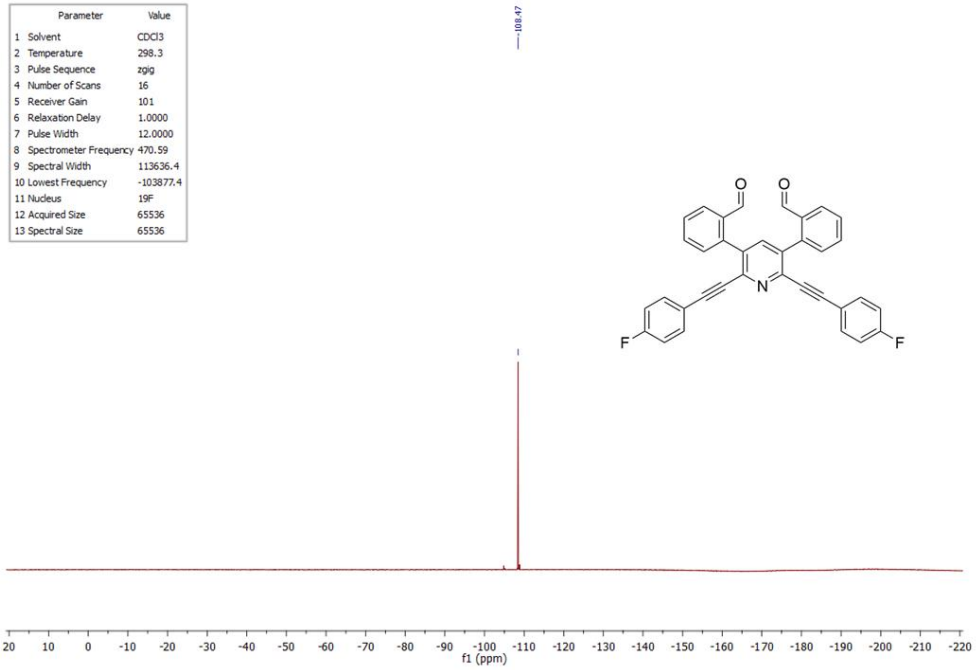
2,2'-(2,6-bis((4-fluorophenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4c)



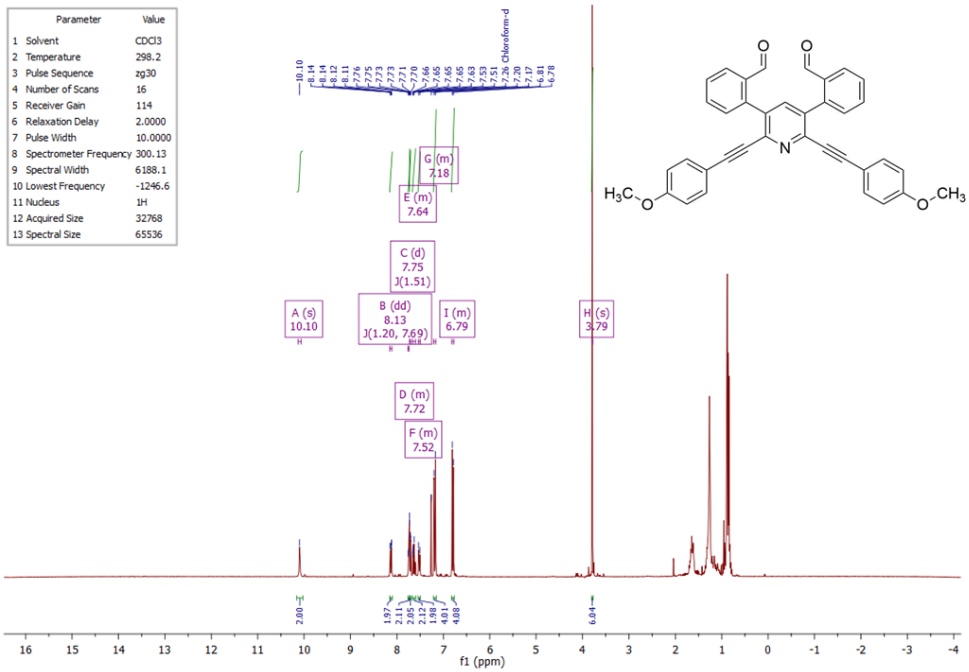
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.2
3	Pulse Sequence	zgpg30
4	Number of Scans	1024
5	Receiver Gain	101
6	Relaxation Delay	2.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	125.76
9	Spectral Width	30120.5
10	Lowest Frequency	-2468.6
11	Nucleus	13C
12	Acquired Size	32768
13	Spectral Size	32768

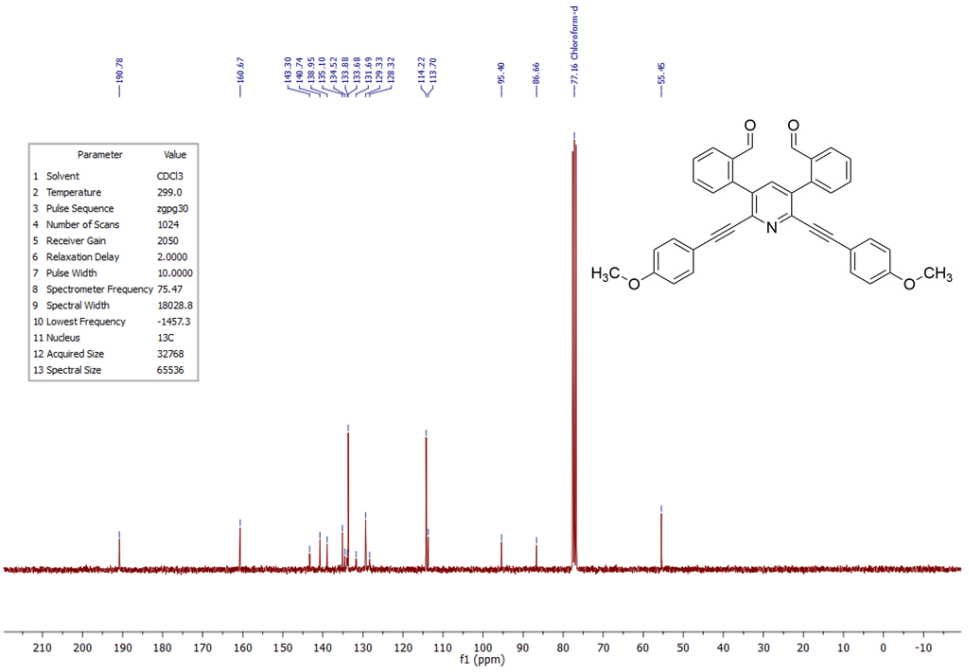


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.3
3	Pulse Sequence	zgig
4	Number of Scans	16
5	Receiver Gain	101
6	Relaxation Delay	1.0000
7	Pulse Width	12.0000
8	Spectrometer Frequency	470.59
9	Spectral Width	113636.4
10	Lowest Frequency	-103877.4
11	Nucleus	19F
12	Acquired Size	65536
13	Spectral Size	65536

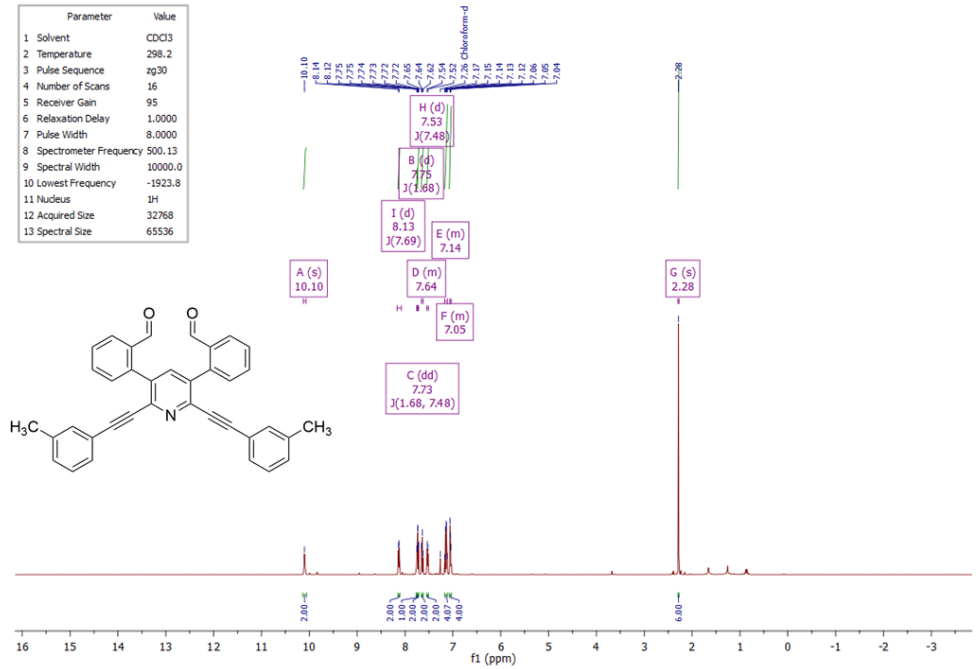


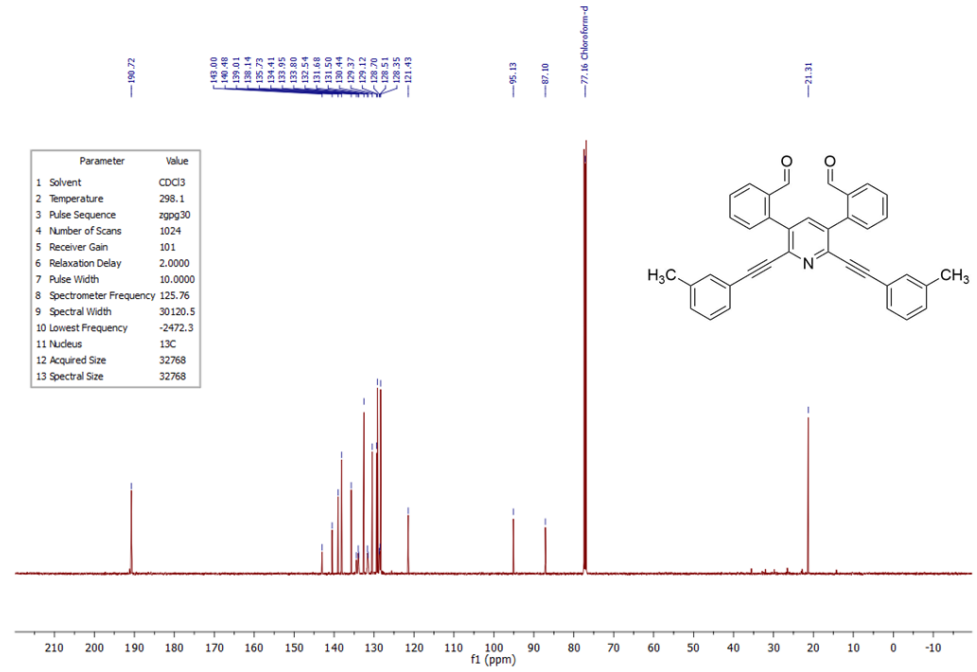
2,2'-(2,6-bis((4-methoxyphenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4d)



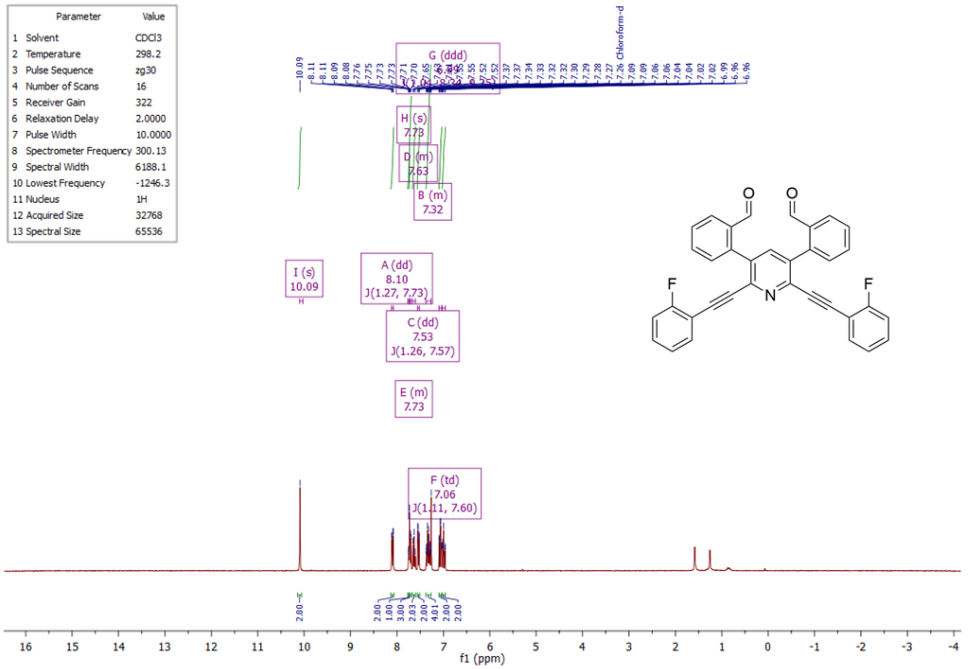


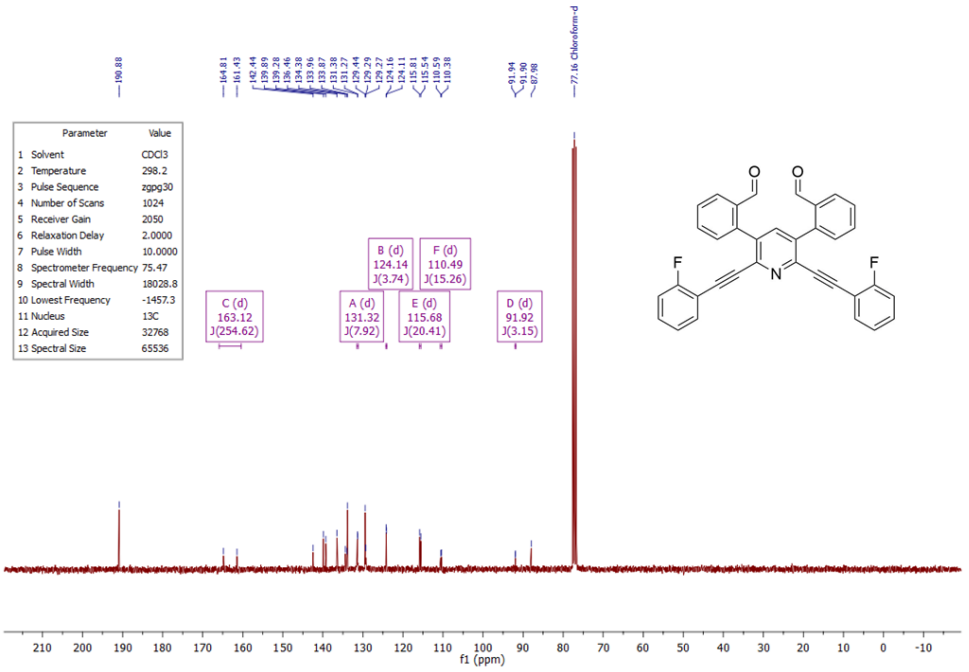
2,2'-(2,6-bis(*m*-tolylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4e)



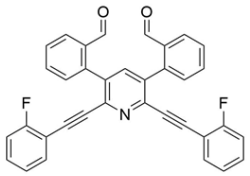


2,2'-(2,6-bis((2-fluorophenyl)ethynyl)pyridine-3,5-diyl)dibenzaldehyde (4f)



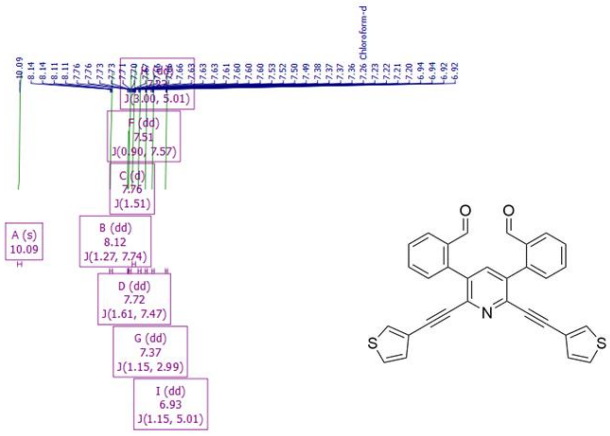


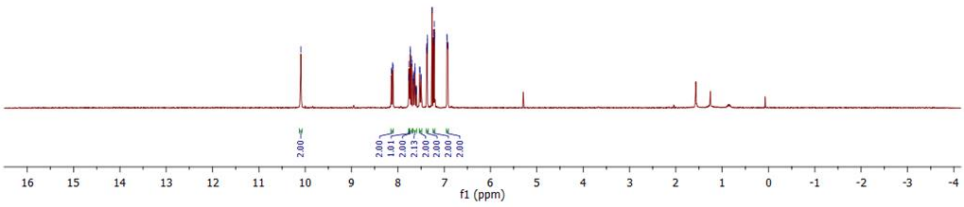
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.2
3	Pulse Sequence	zgfhigqn
4	Number of Scans	64
5	Receiver Gain	2050
6	Relaxation Delay	1.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	282.40
9	Spectral Width	66964.3
10	Lowest Frequency	-61722.4
11	Nucleus	19F
12	Acquired Size	65536
13	Spectral Size	131072

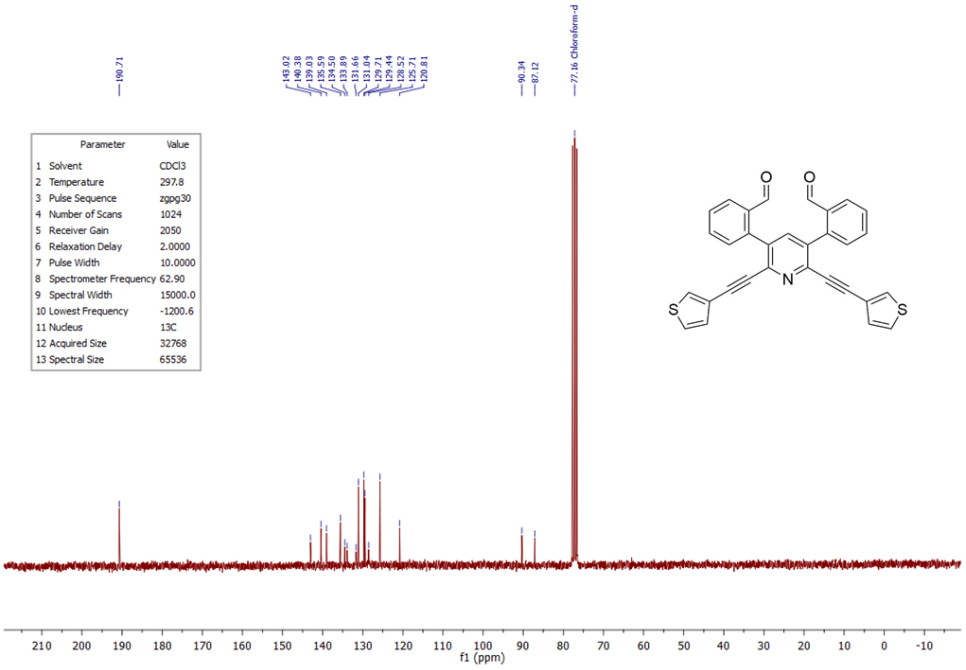


2,2'-(2,6-bis(thiophen-3-ylethynyl)pyridine-3,5-diyl)dibenzaldehyde (4g)

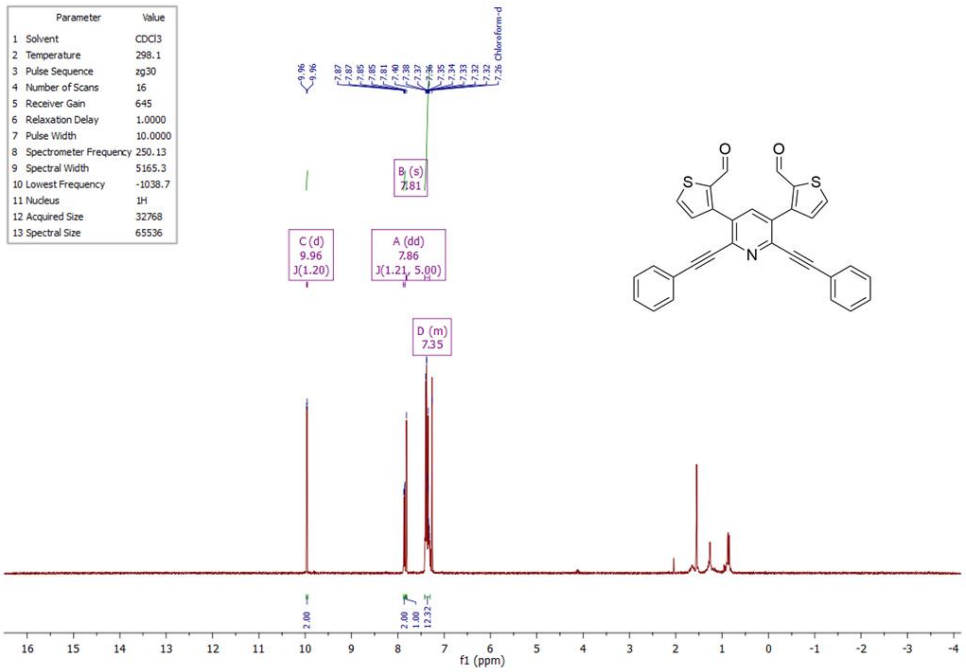
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.1
3	Pulse Sequence	zg30
4	Number of Scans	16
5	Receiver Gain	645
6	Relaxation Delay	1.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	250.13
9	Spectral Width	5165.3
10	Lowest Frequency	-1038.6
11	Nucleus	1H
12	Acquired Size	32768
13	Spectral Size	65536

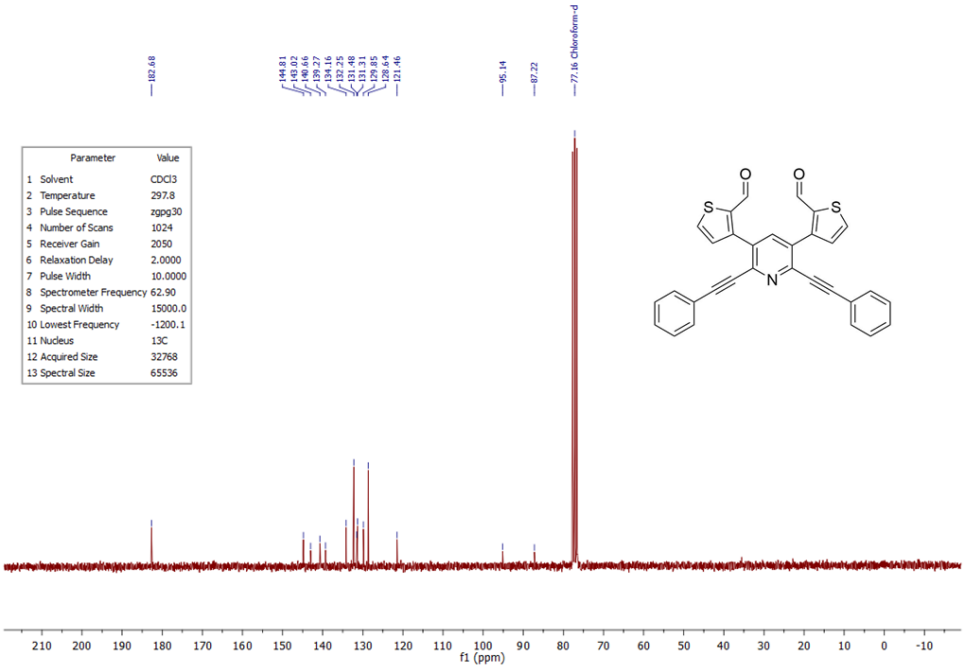




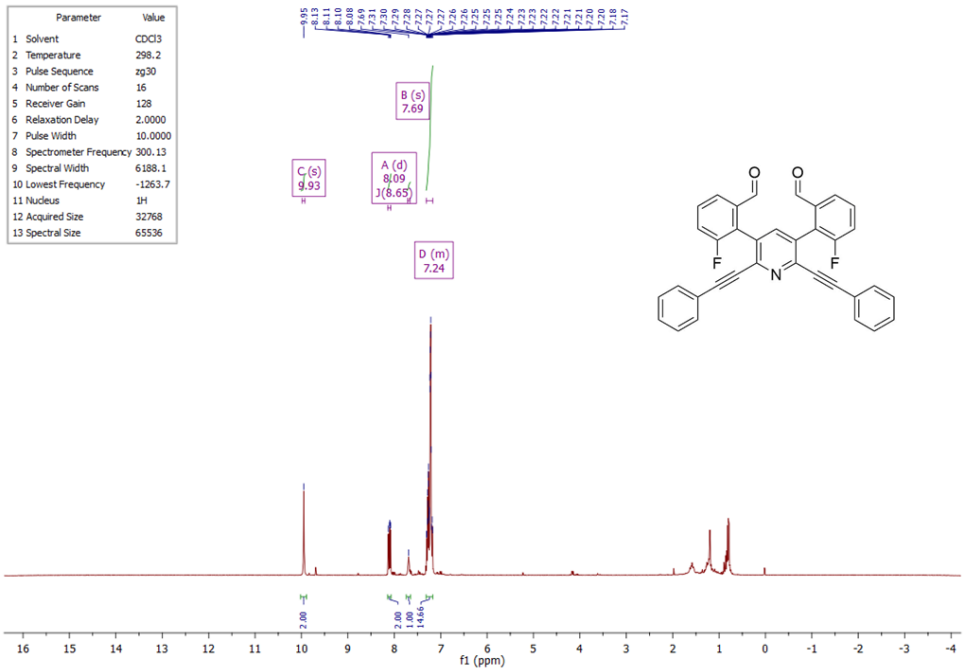


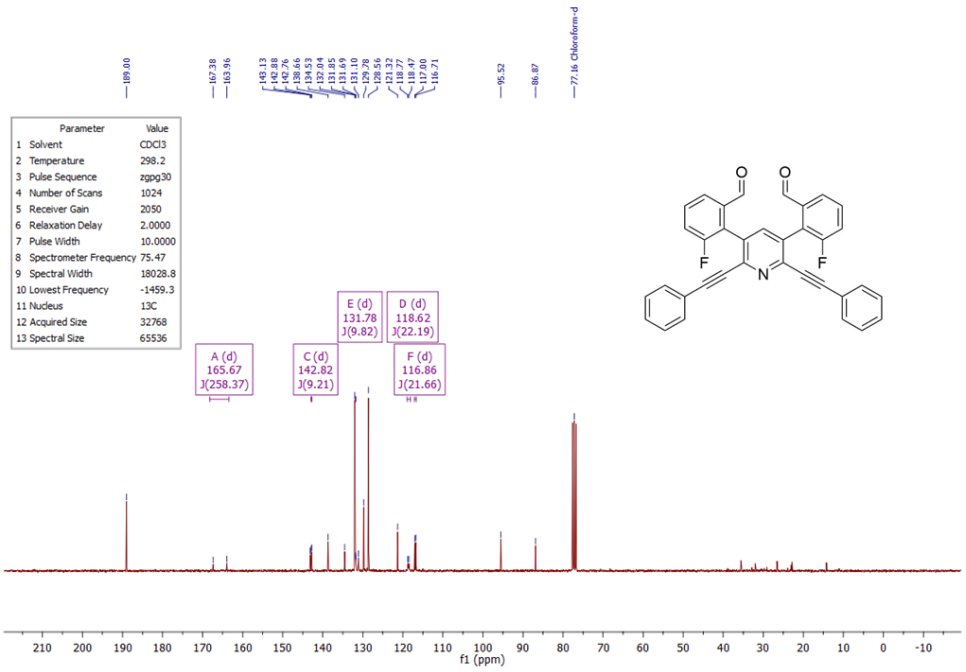
3,3'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(thiophene-2-carbaldehyde) (4h)

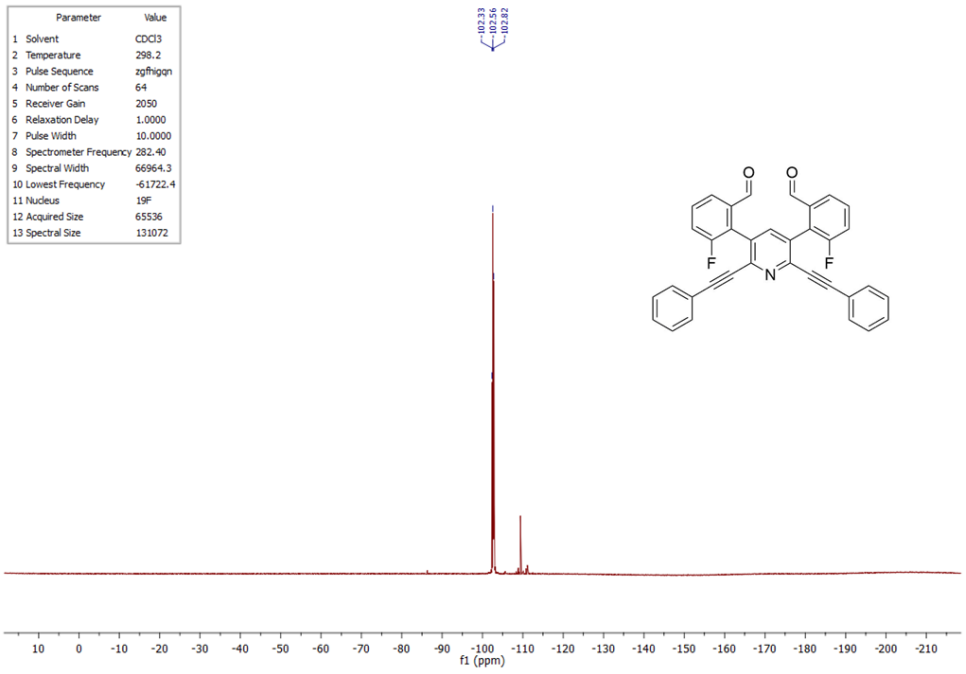




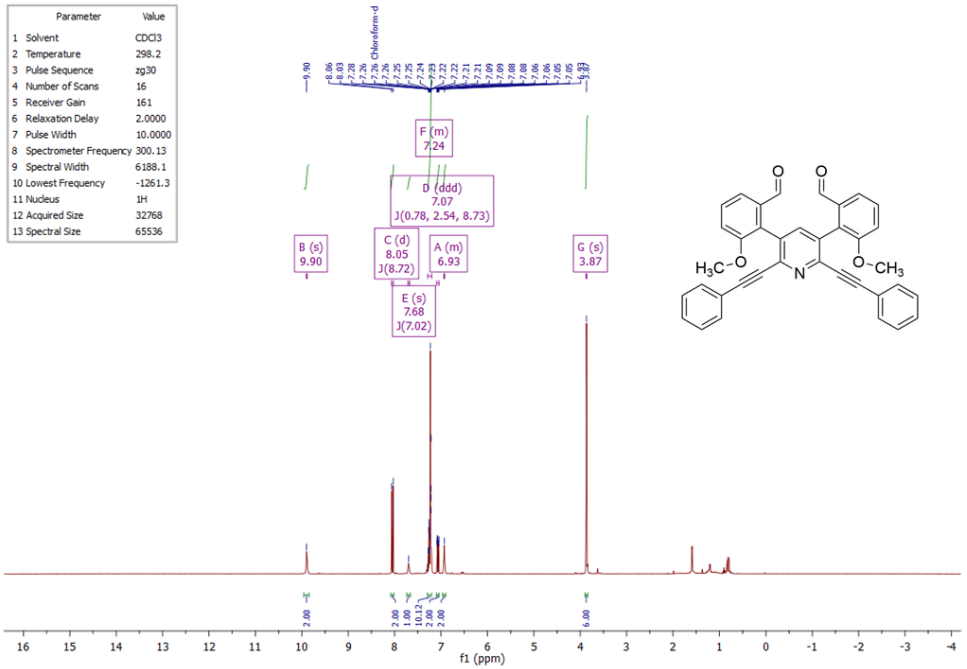
2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-fluorobenzaldehyde) (4i)

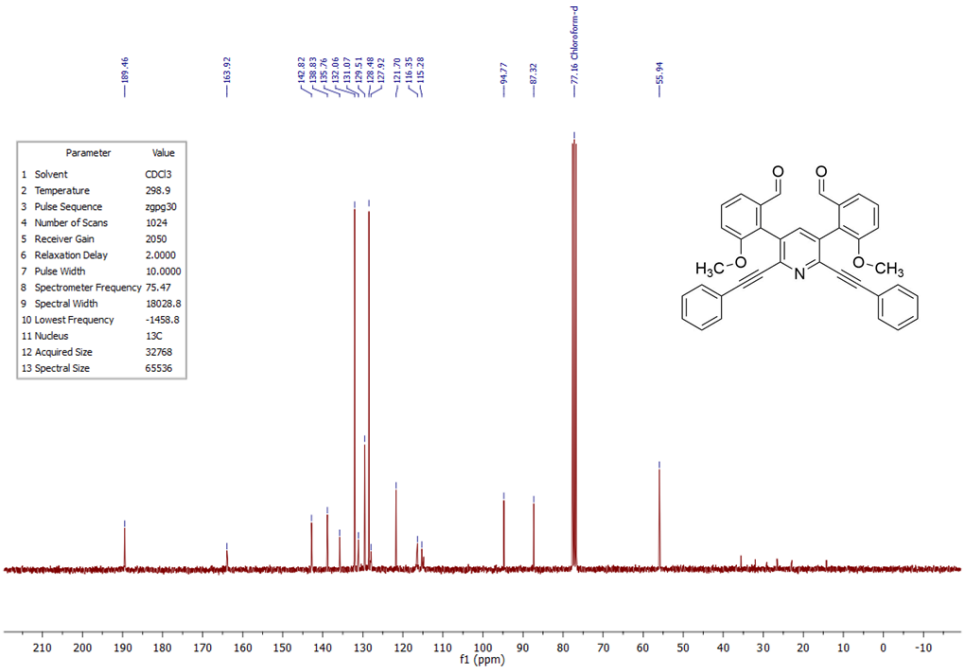



189.00
167.38
163.96
145.13
142.88
142.76
138.66
134.53
132.04
131.85
131.69
131.10
129.78
128.56
121.33
118.77
118.47
117.00
116.71
95.52
86.87
77.16 Chloroform-d
Parameter Value
1 Solvent CDCl3
2 Temperature 298.2
3 Pulse Sequence zgpg30
4 Number of Scans 1024
5 Receiver Gain 2050
6 Relaxation Delay 2.0000
7 Pulse Width 10.0000
8 Spectrometer Frequency 75.47
9 Spectral Width 18028.8
10 Lowest Frequency -1459.3
11 Nucleus 13C
12 Acquired Size 32768
13 Spectral Size 65536
A (d) 165.67 J(258.37)
C (d) 142.82 J(9.21)
E (d) 131.78 J(9.82)
D (d) 118.62 J(22.19)
F (d) 116.86 J(21.66)
O
O
F
F
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

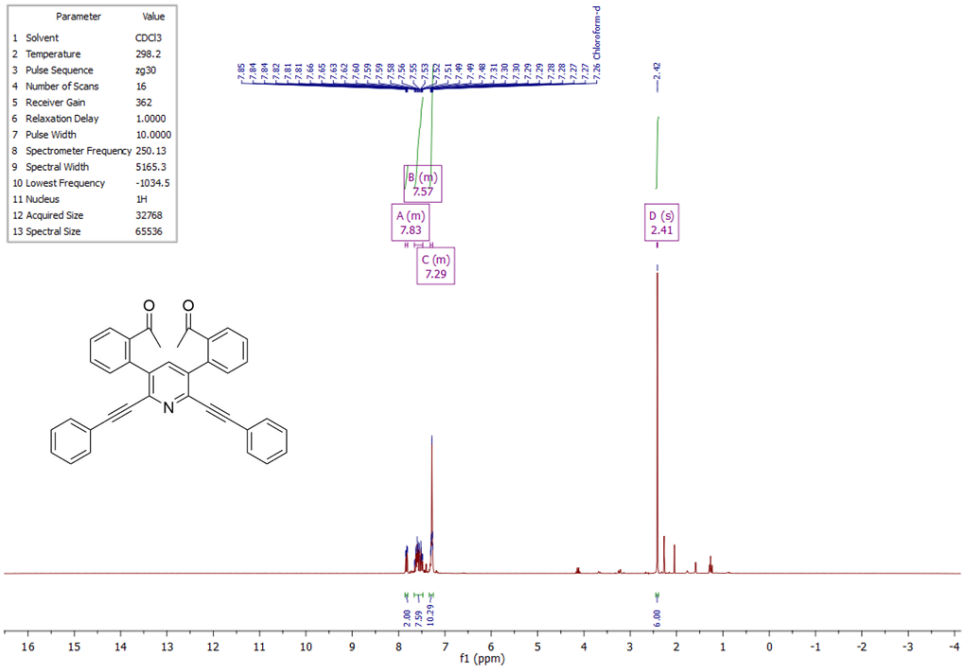

102.33
102.56
102.82
Parameter Value
1 Solvent CDCl3
2 Temperature 298.2
3 Pulse Sequence zgfhigqn
4 Number of Scans 64
5 Receiver Gain 2050
6 Relaxation Delay 1.0000
7 Pulse Width 10.0000
8 Spectrometer Frequency 282.40
9 Spectral Width 66964.3
10 Lowest Frequency -61722.4
11 Nucleus 19F
12 Acquired Size 65536
13 Spectral Size 131072
O
O
F
F
N
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

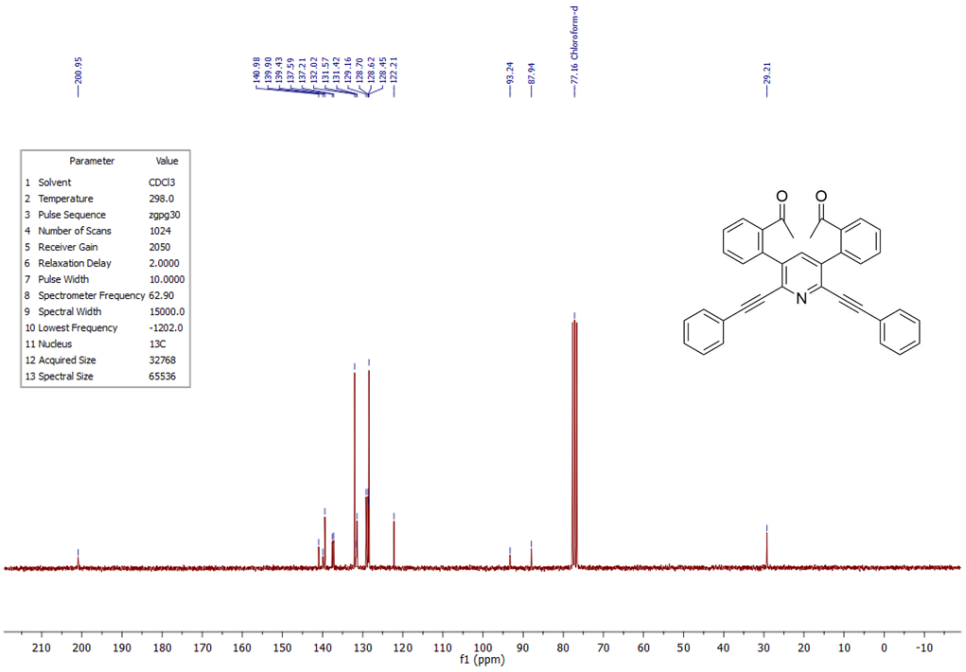
2,2'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-methoxybenzaldehyde) (4j)



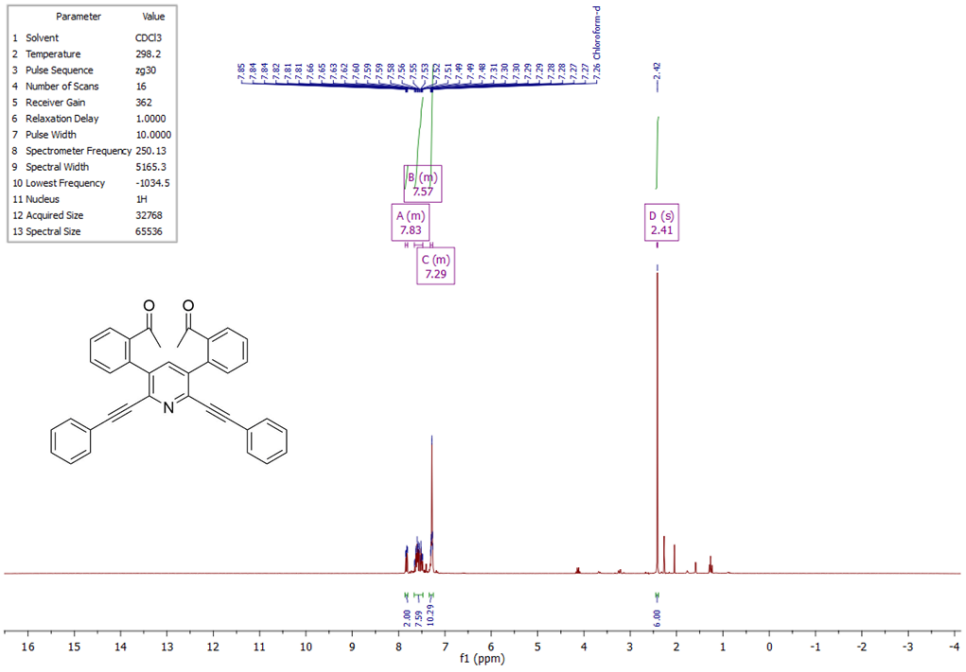


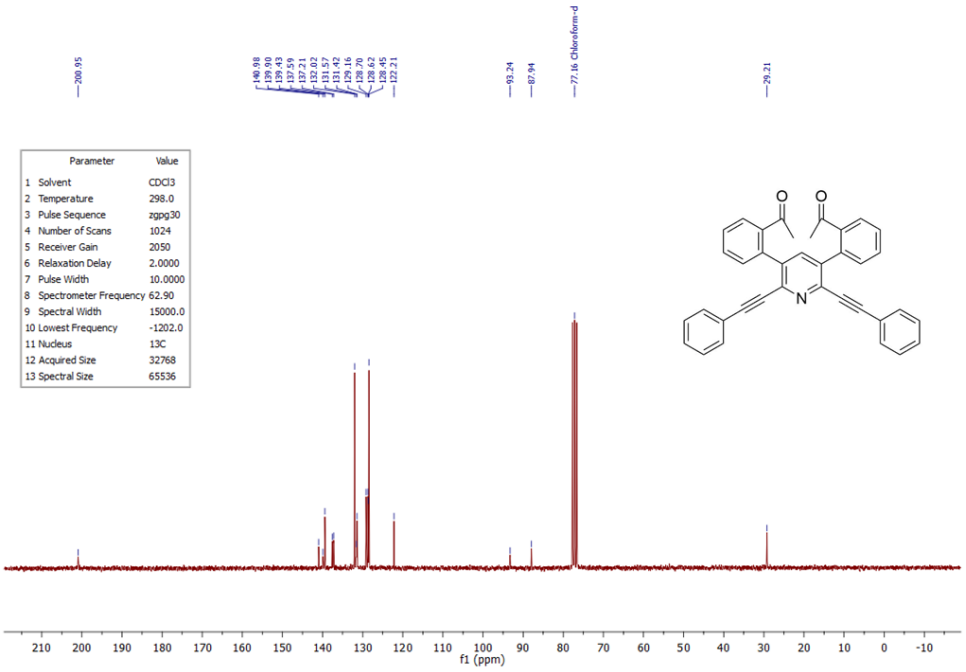
1,1'-((2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(2,1-phenylene))bis(ethan-1-one) (4k)



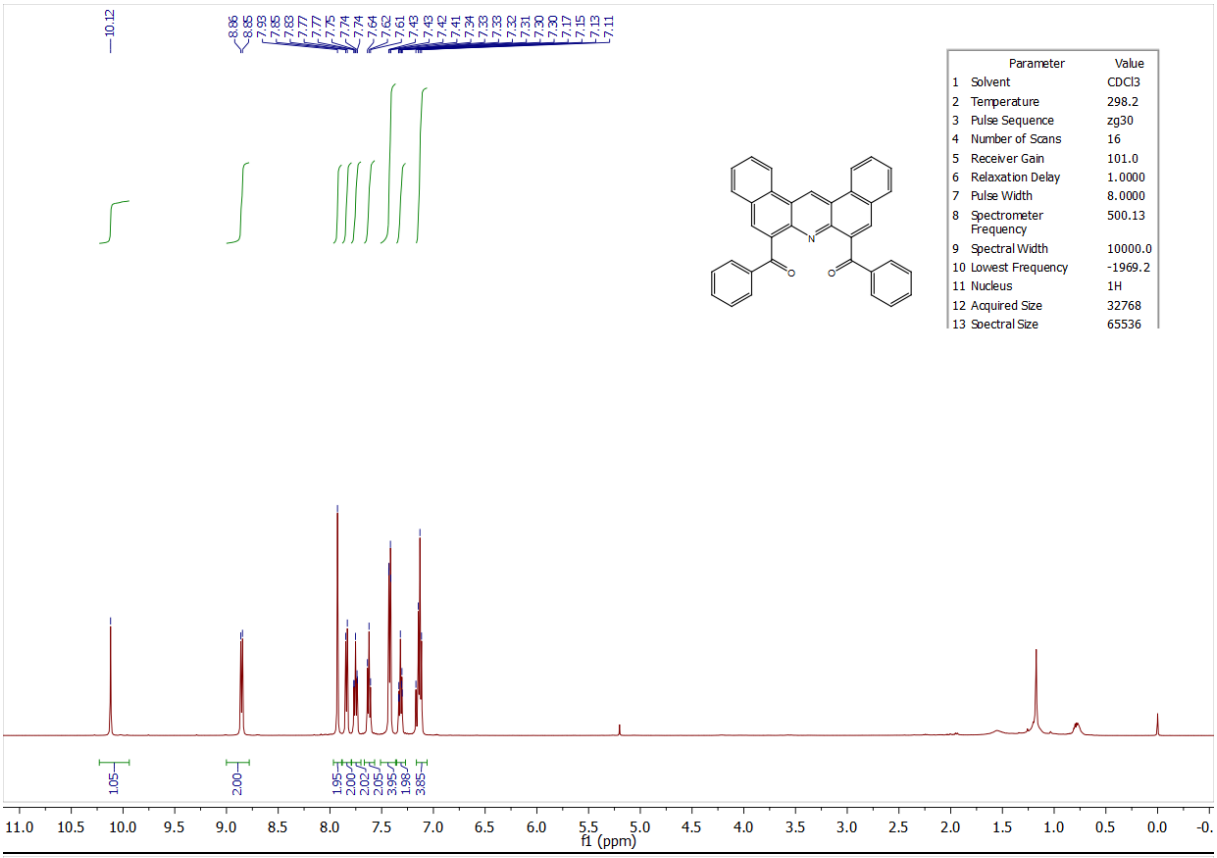


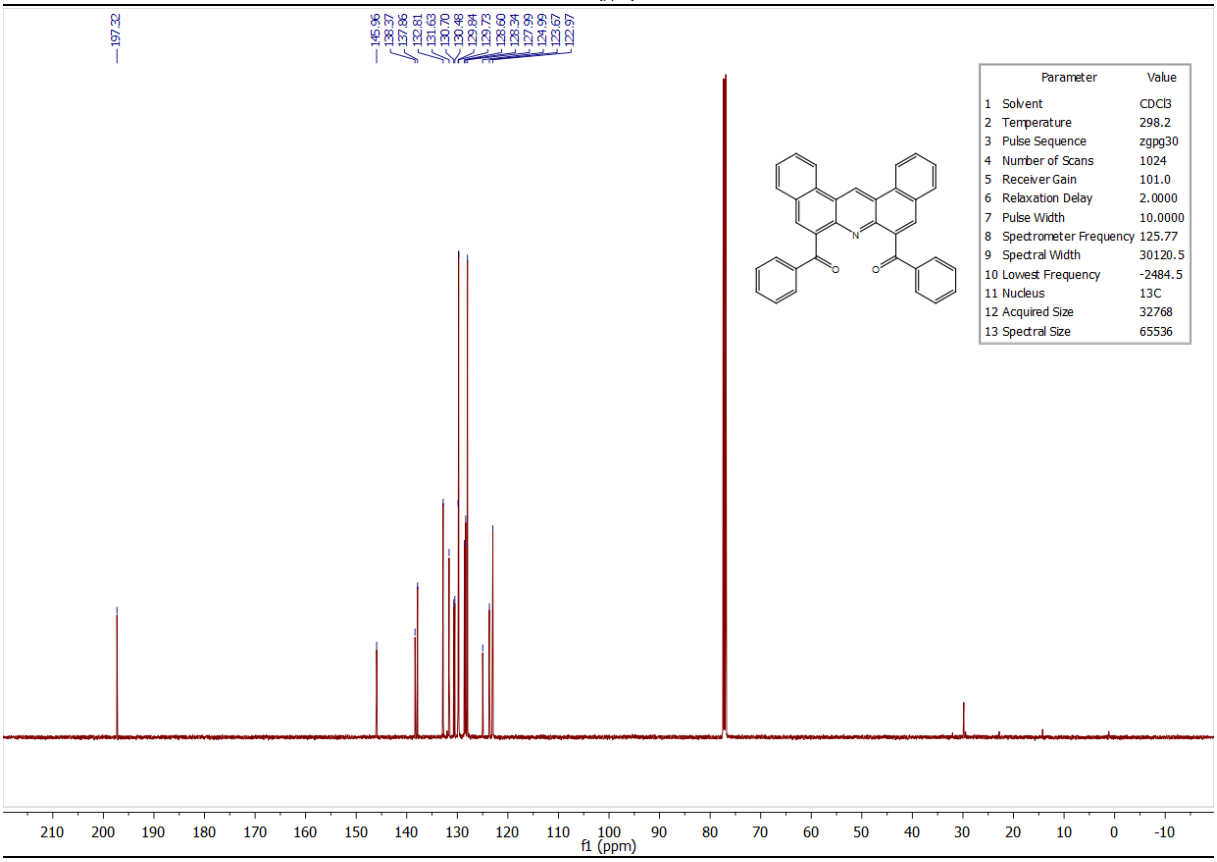
6,6'-(2,6-bis(phenylethynyl)pyridine-3,5-diyl)bis(3-chlorobenzaldehyde) (4l)



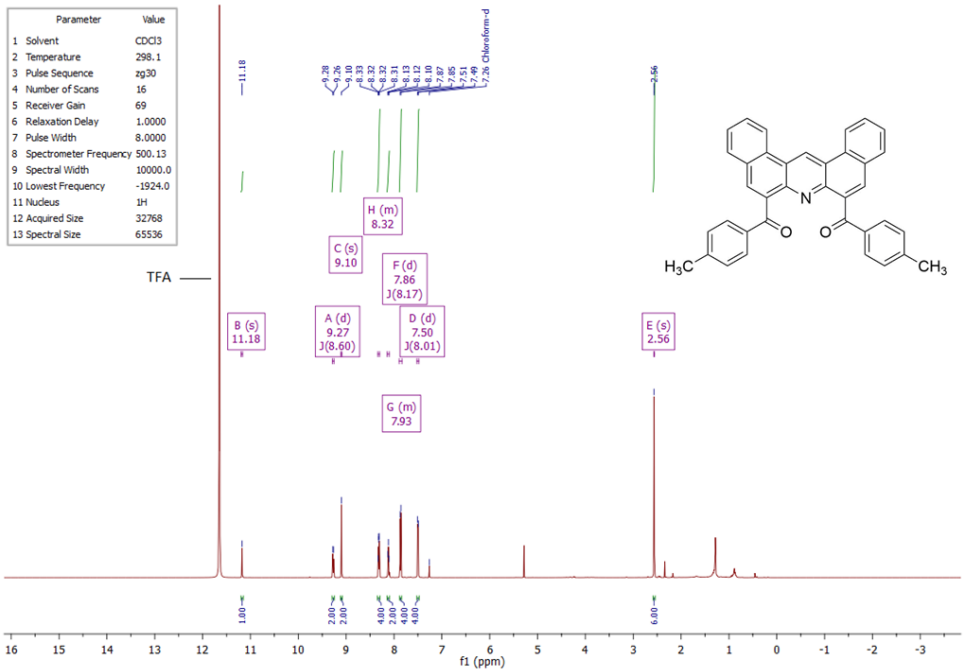


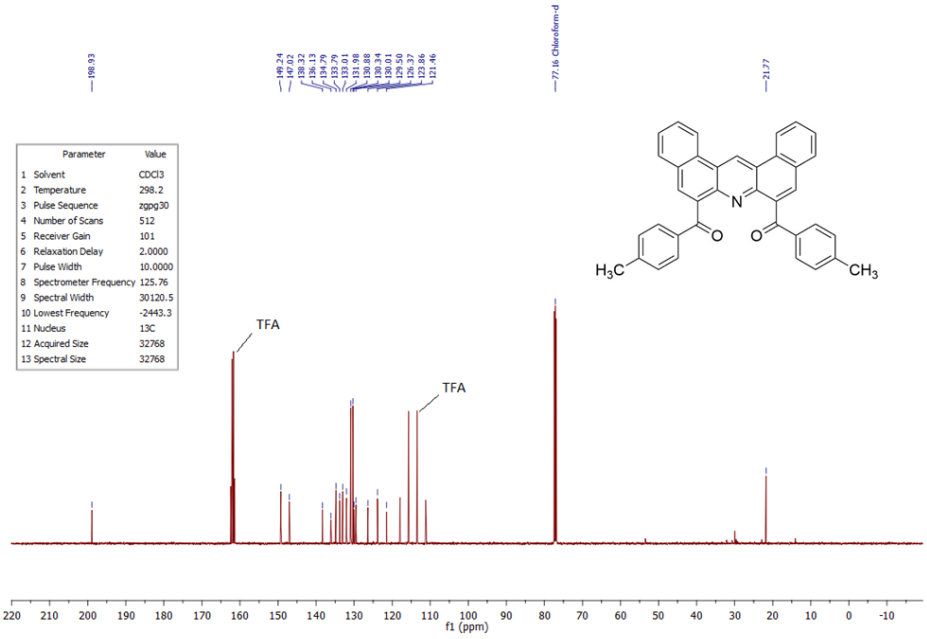
dibenzo[*a*,*j*]acridine-6,8-diylbis(phenylmethanone) (5a)



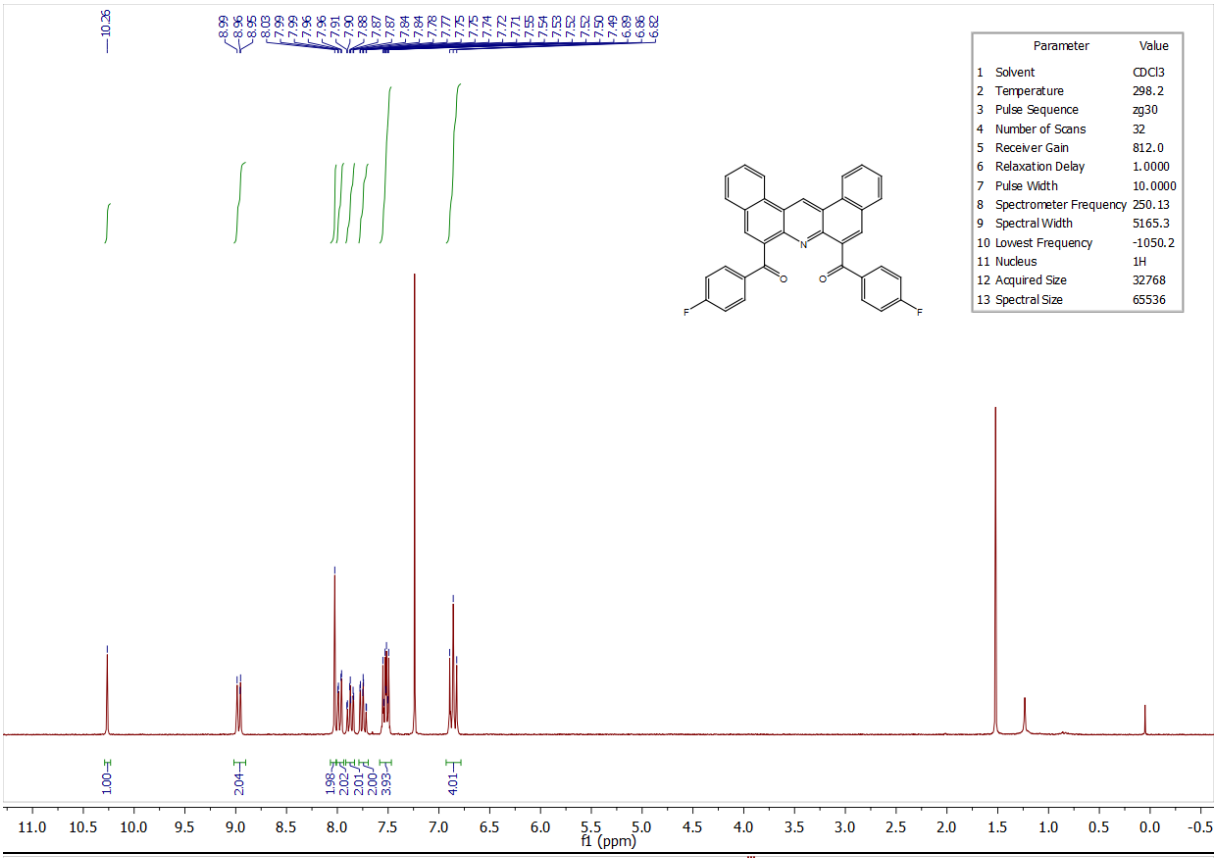


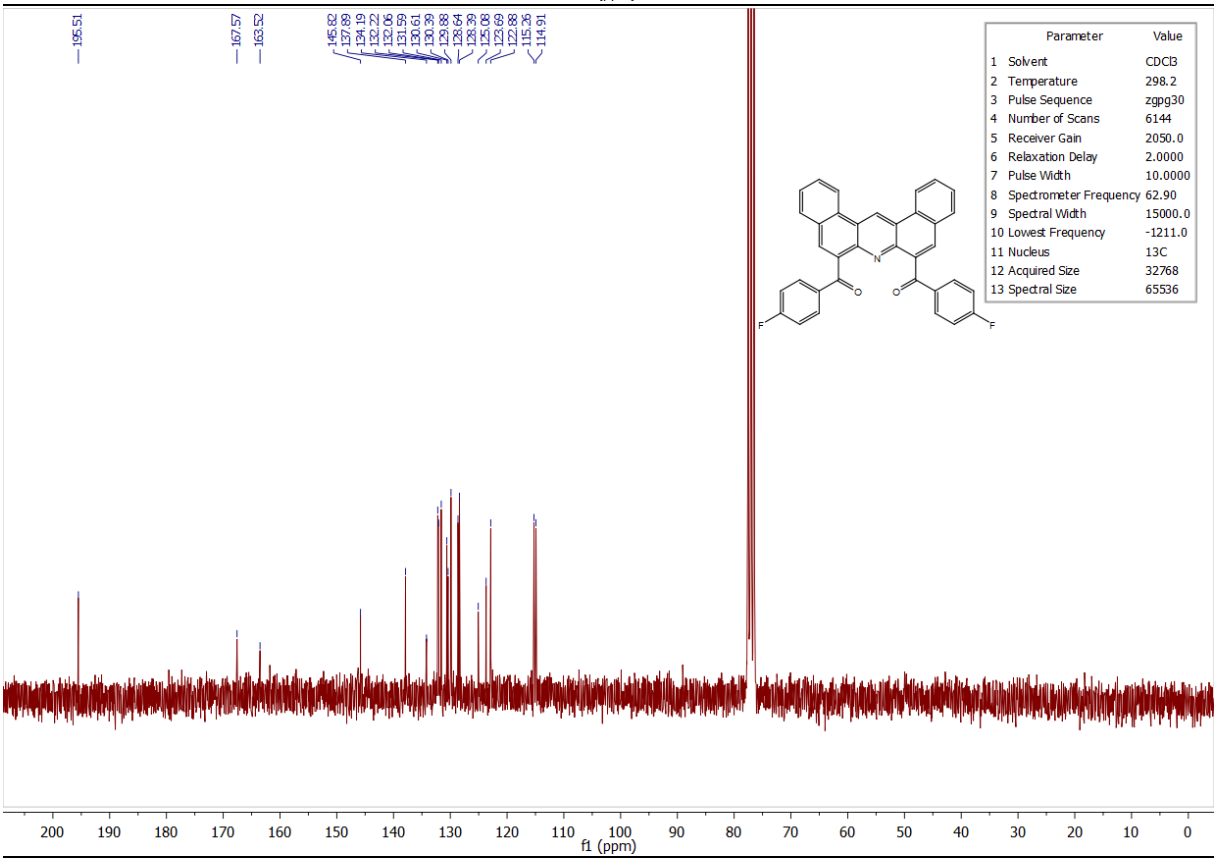
dibenzo[*a*,*j*]acridine-6,8-diylbis(*p*-tolylmethanone) (5b)



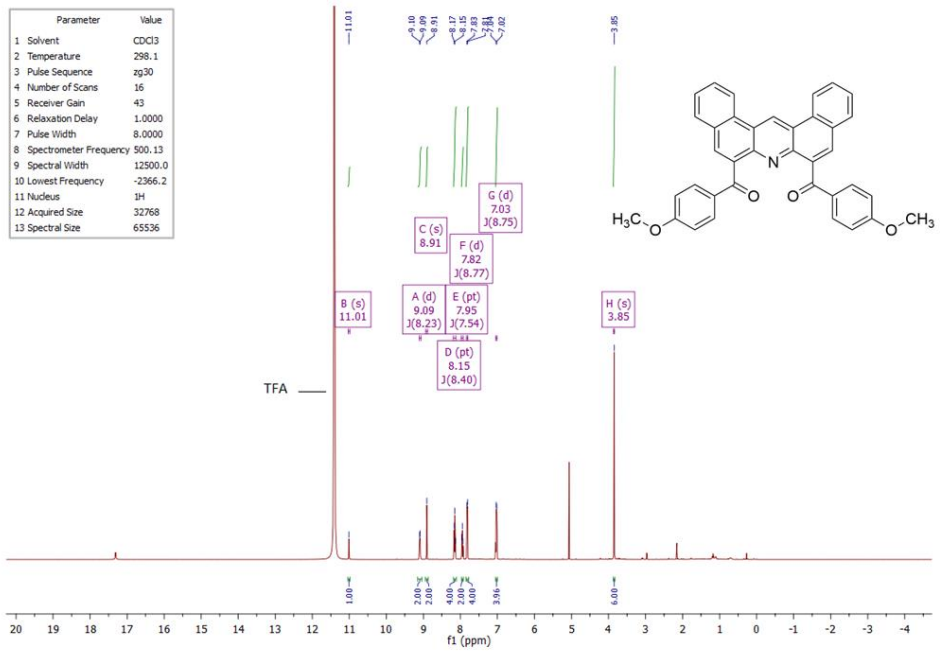


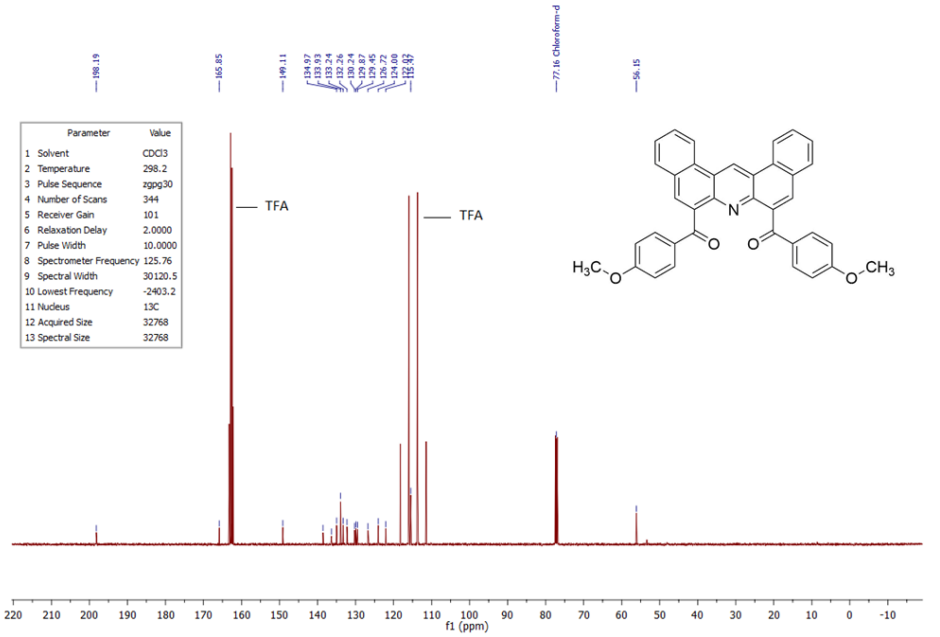
dibenzo[*a*,*j*]acridine-6,8-diylbis((4-fluorophenyl)methanone) (5c)



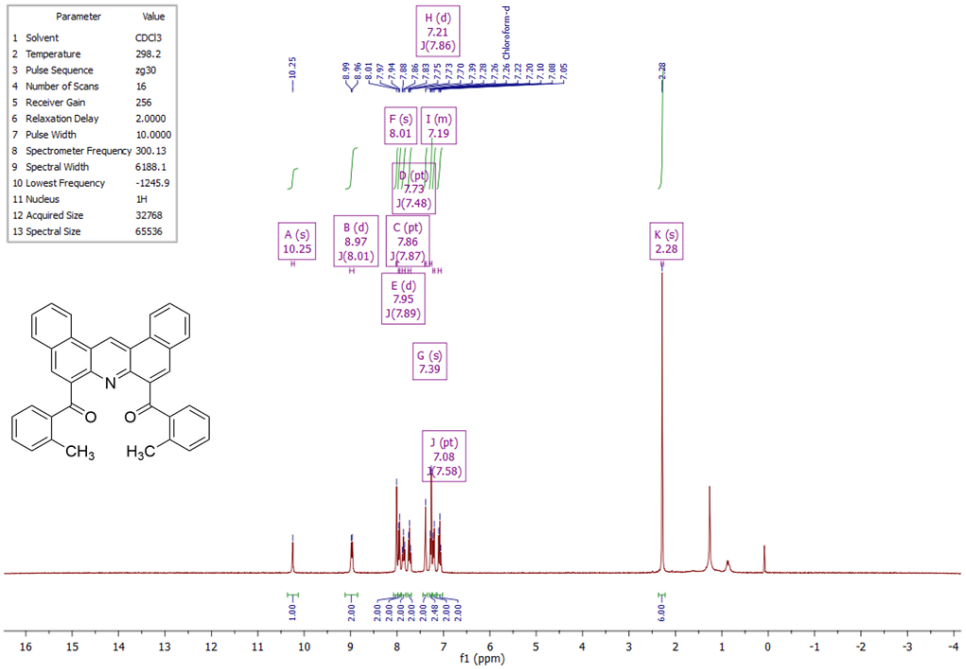


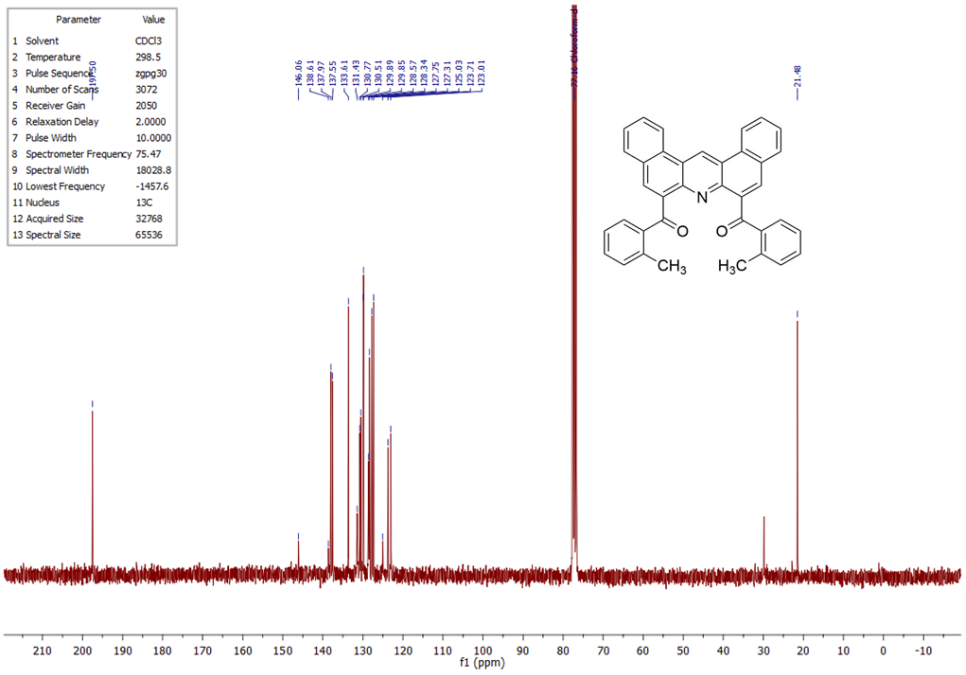
dibenzo[*a*,*j*]acridine-6,8-diylbis((4-methoxyphenyl)methanone) (5d)



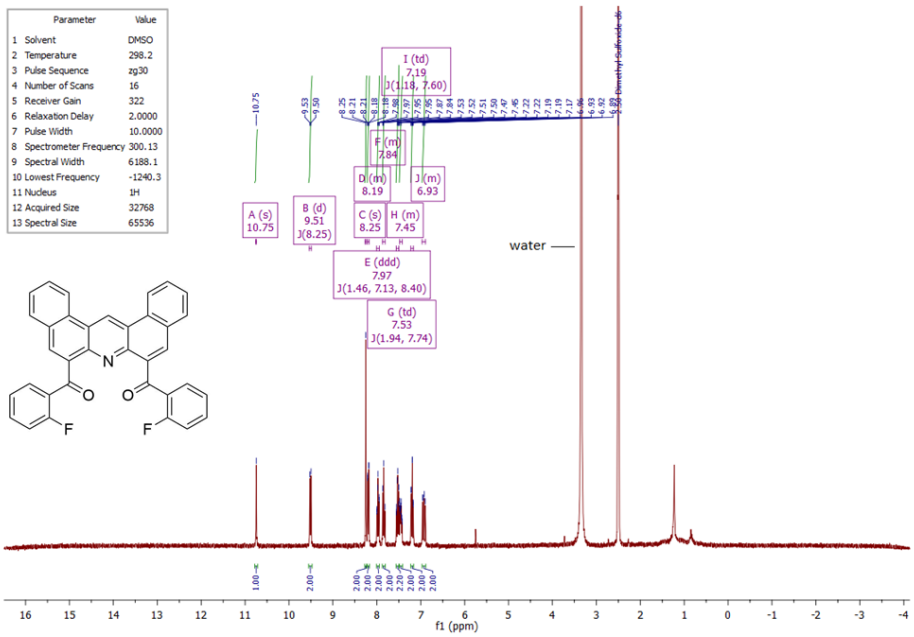


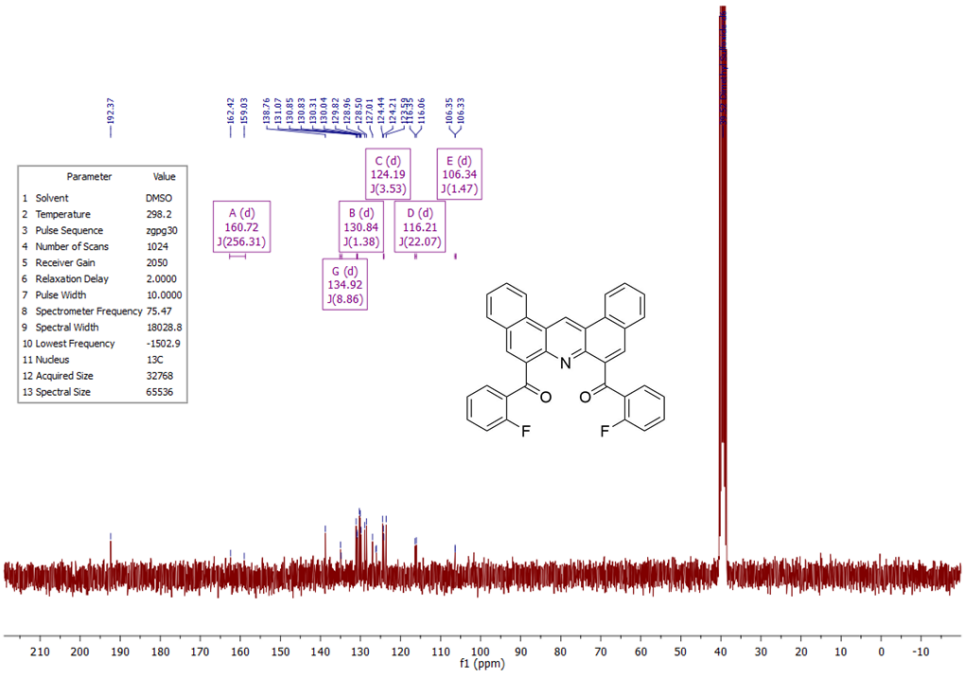
dibenzo[*a*,*j*]acridine-6,8-diylbis(*m*-tolylmethanone) (5e)



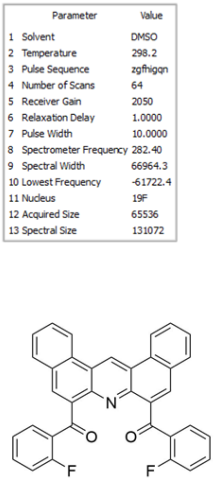


dibenzo[*a*,*j*]acridine-6,8-diylbis((2-fluorophenyl)methanone) (5f)





	Parameter	Value
1	Solvent	DMSO
2	Temperature	298.2
3	Pulse Sequence	zgfhigqn
4	Number of Scans	64
5	Receiver Gain	2050
6	Relaxation Delay	1.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	282.40
9	Spectral Width	66964.3
10	Lowest Frequency	-61722.4
11	Nucleus	19F
12	Acquired Size	65536
13	Spectral Size	131072



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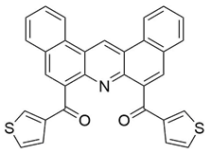
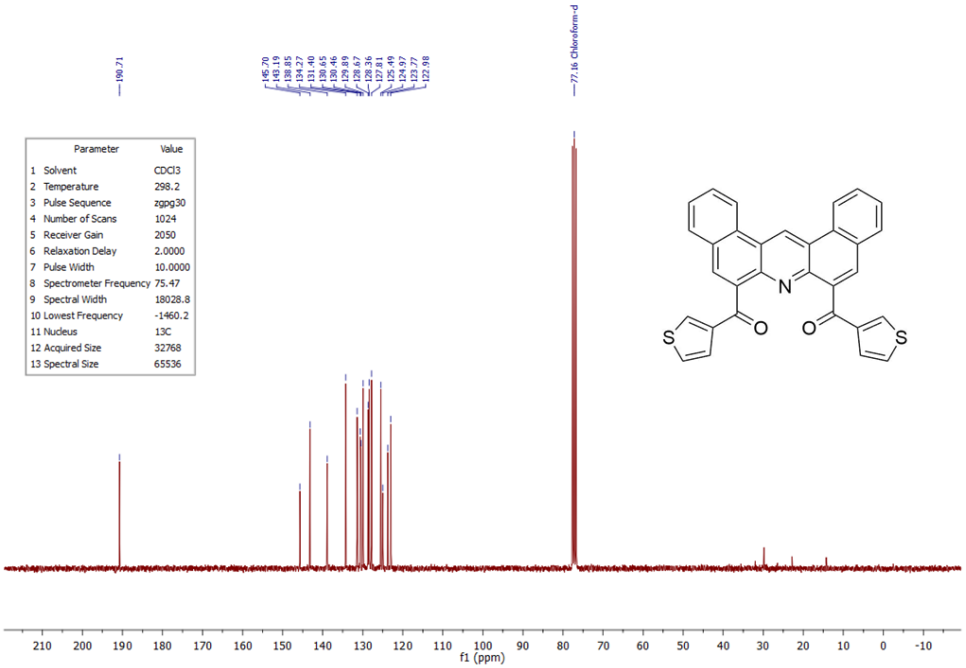
dibenzo[*a*,*j*]acridine-6,8-diylbis(thiophen-3-ylmethanone) (5g)

	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.2
3	Pulse Sequence	zg30
4	Number of Scans	16
5	Receiver Gain	161
6	Relaxation Delay	2.0000
7	Pulse Width	10.0000
8	Spectrometer Frequency	300.13
9	Spectral Width	6188.1
10	Lowest Frequency	-1246.4
11	Nucleus	1H
12	Acquired Size	32768
13	Spectral Size	65536





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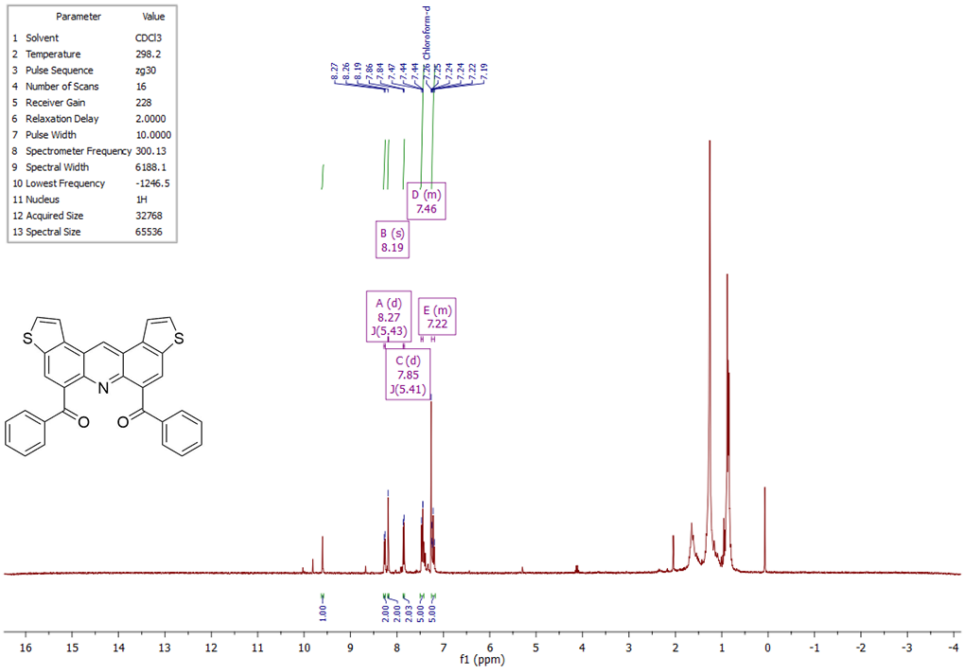
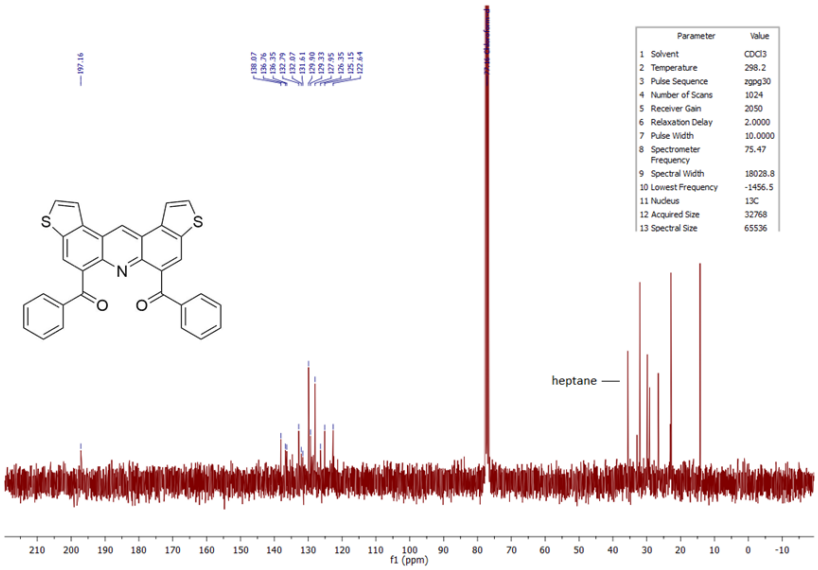
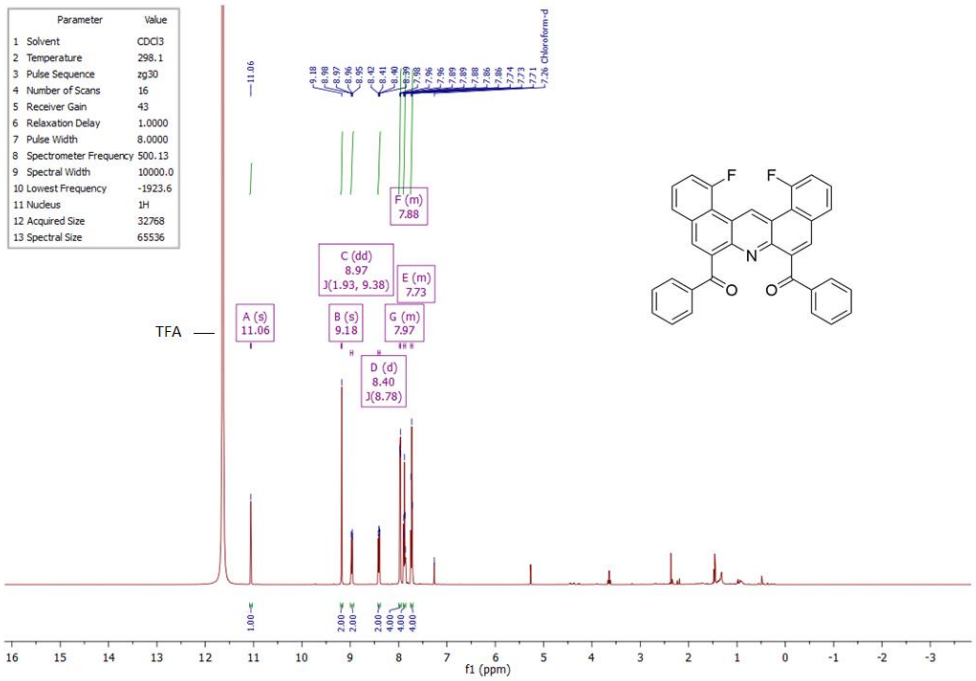
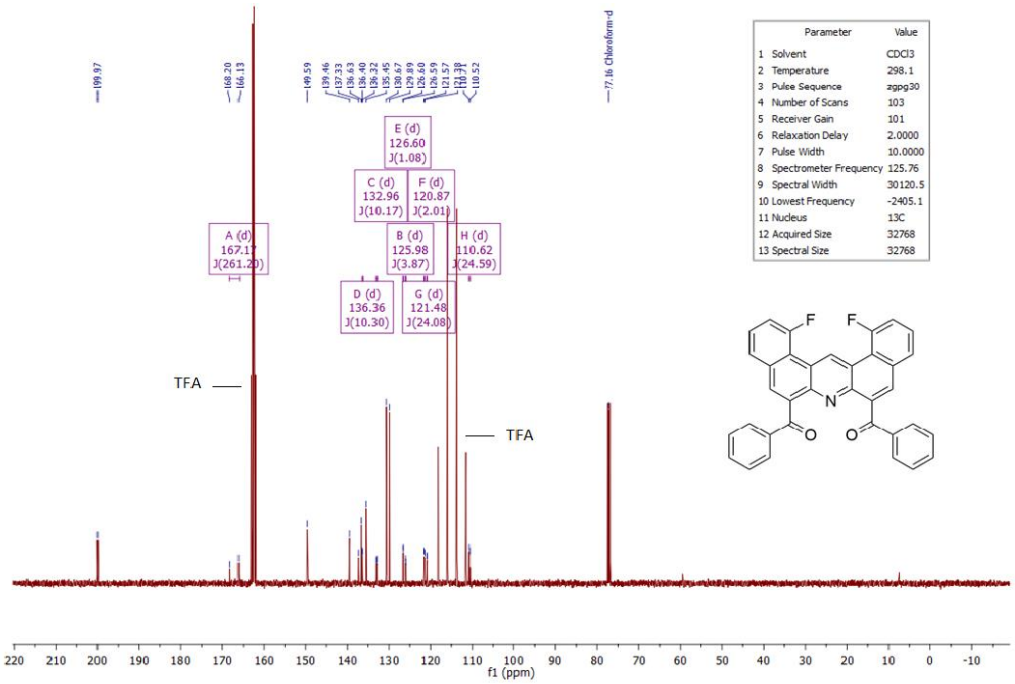
dithieno[3,2-*a*:2',3'-*j*]acridine-5,7-diylbis(phenylmethanone) (5h)

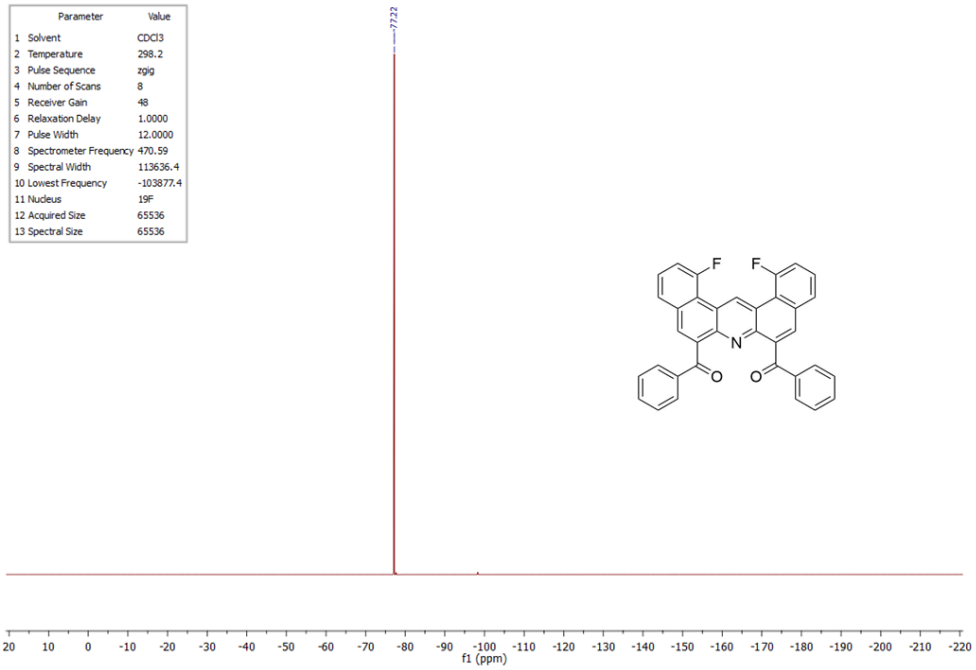




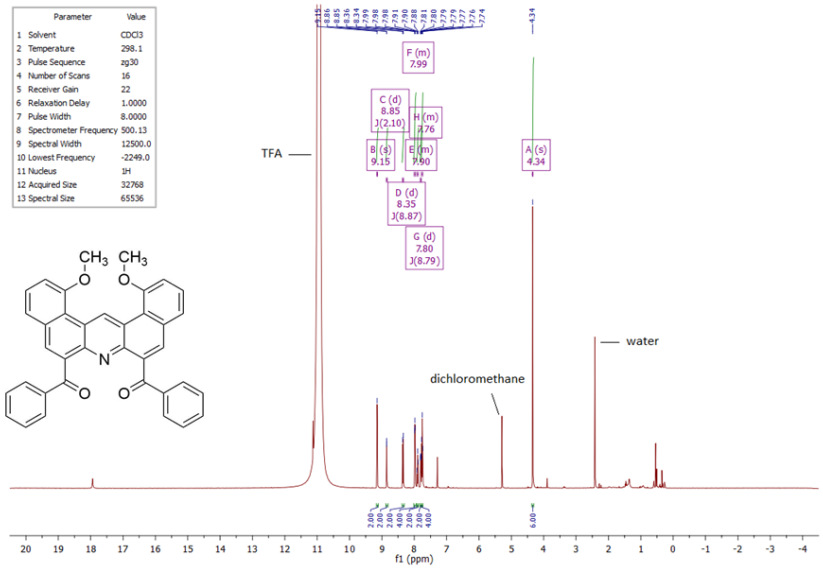
(1,13-difluorodibenzo[*a*,*j*]acridine-6,8-diyl)bis(phenylmethanone) (5i)

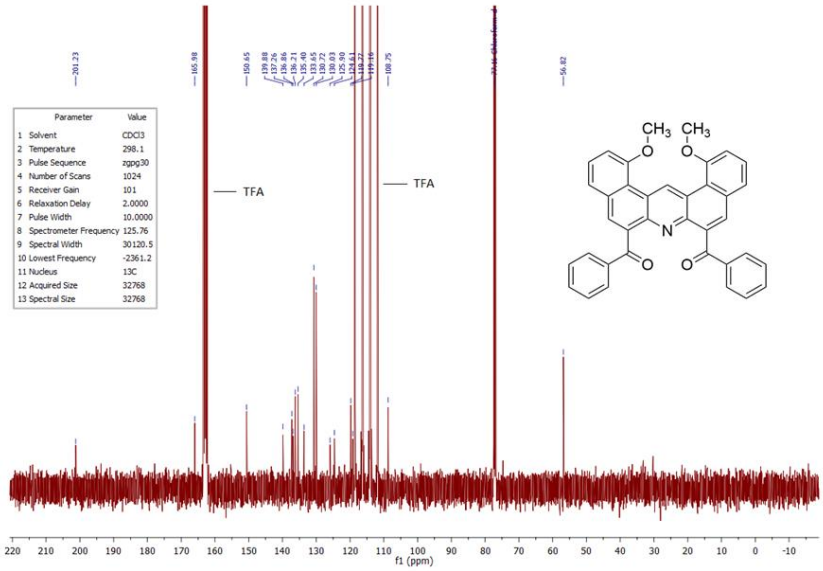



199.97
168.20
166.13
149.59
139.46
137.33
136.63
136.40
136.32
135.45
135.17
129.89
126.60
126.59
121.57
121.38
110.52
77.16 Chloroform-d
E (d)
126.60
J(1.08)
C (d)
132.96
J(10.17)
F (d)
120.87
J(2.01)
A (d)
167.17
J(261.20)
B (d)
125.98
J(3.87)
H (d)
110.62
J(24.59)
D (d)
136.36
J(10.30)
G (d)
121.48
J(24.08)
TFA
TFA
Parameter Value
1 Solvent CDCl3
2 Temperature 298.1
3 Pulse Sequence zgpg30
4 Number of Scans 103
5 Receiver Gain 101
6 Relaxation Delay 2.0000
7 Pulse Width 10.0000
8 Spectrometer Frequency 125.76
9 Spectral Width 30120.5
10 Lowest Frequency -2405.1
11 Nucleus 13C
12 Acquired Size 32768
13 Spectral Size 32768
F
F
N
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

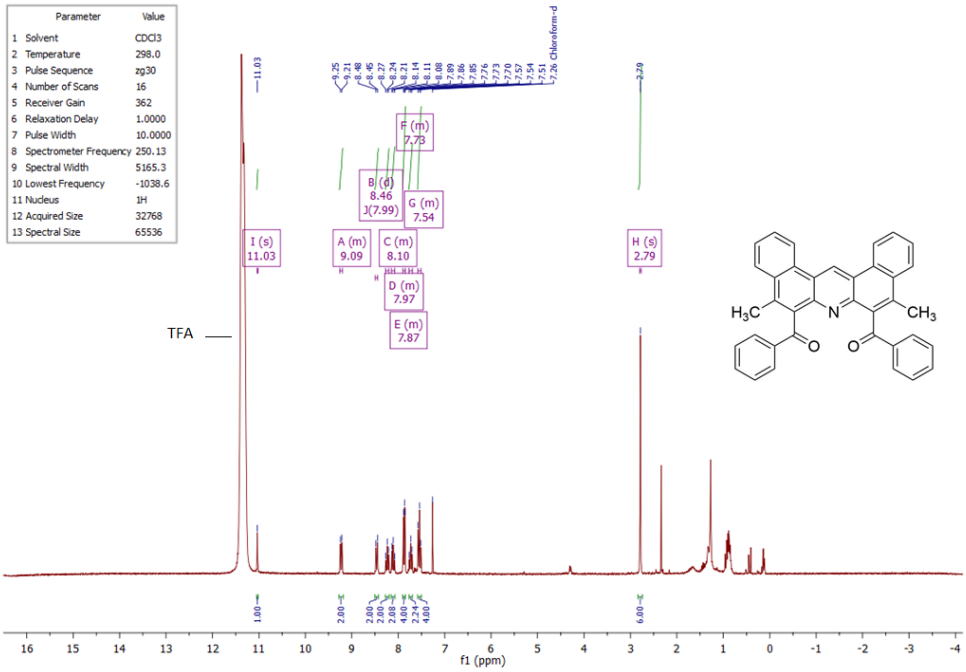

Parameter Value
1 Solvent CDCl3
2 Temperature 298.2
3 Pulse Sequence zgig
4 Number of Scans 8
5 Receiver Gain 48
6 Relaxation Delay 1.0000
7 Pulse Width 12.0000
8 Spectrometer Frequency 470.59
9 Spectral Width 113636.4
10 Lowest Frequency -103877.4
11 Nucleus 19F
12 Acquired Size 65536
13 Spectral Size 65536
-77.22
F
F
N
O
O
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

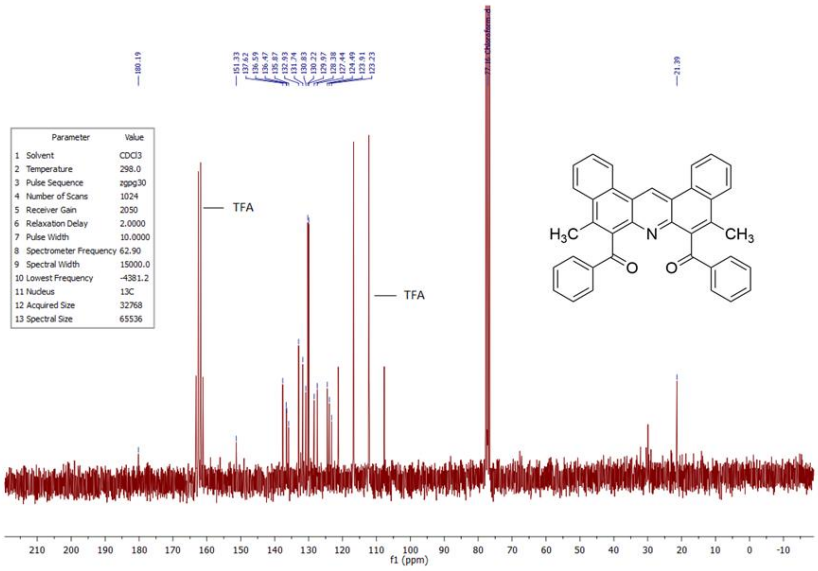
(1,13-dimethoxydibenzo[a,j]acridine-6,8-diyl)bis(phenylmethanone) (5j)



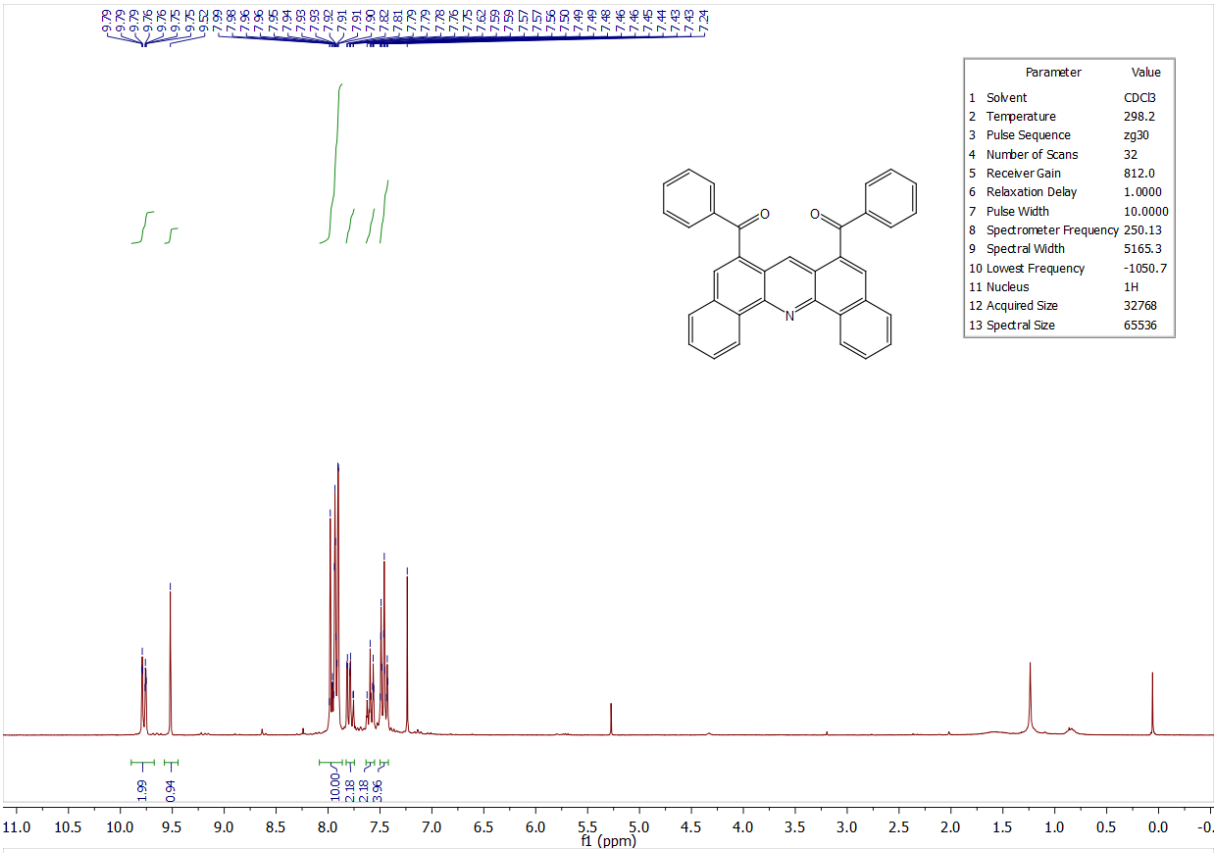


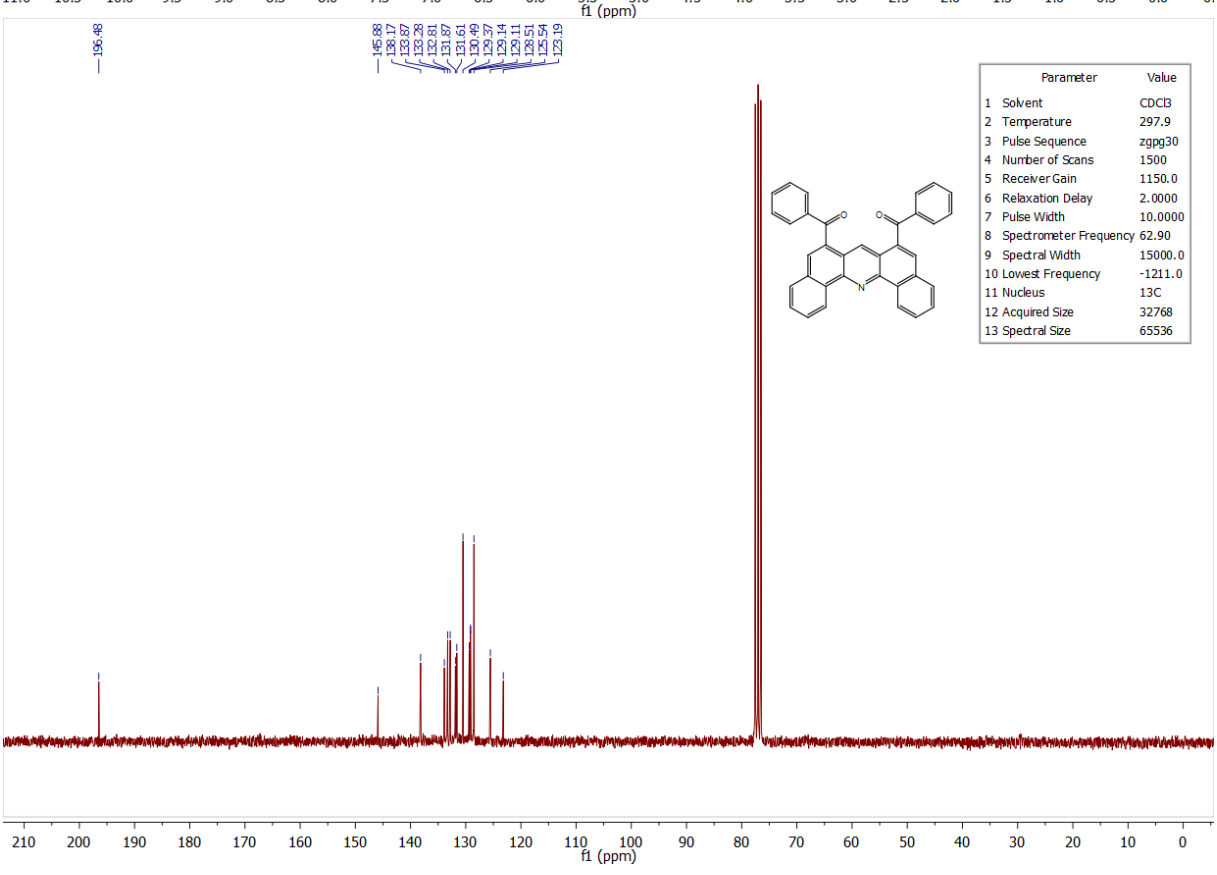
(5,9-dimethyldibenzo[*a*,*j*]acridine-6,8-diyl)bis(phenylmethanone) (5k)



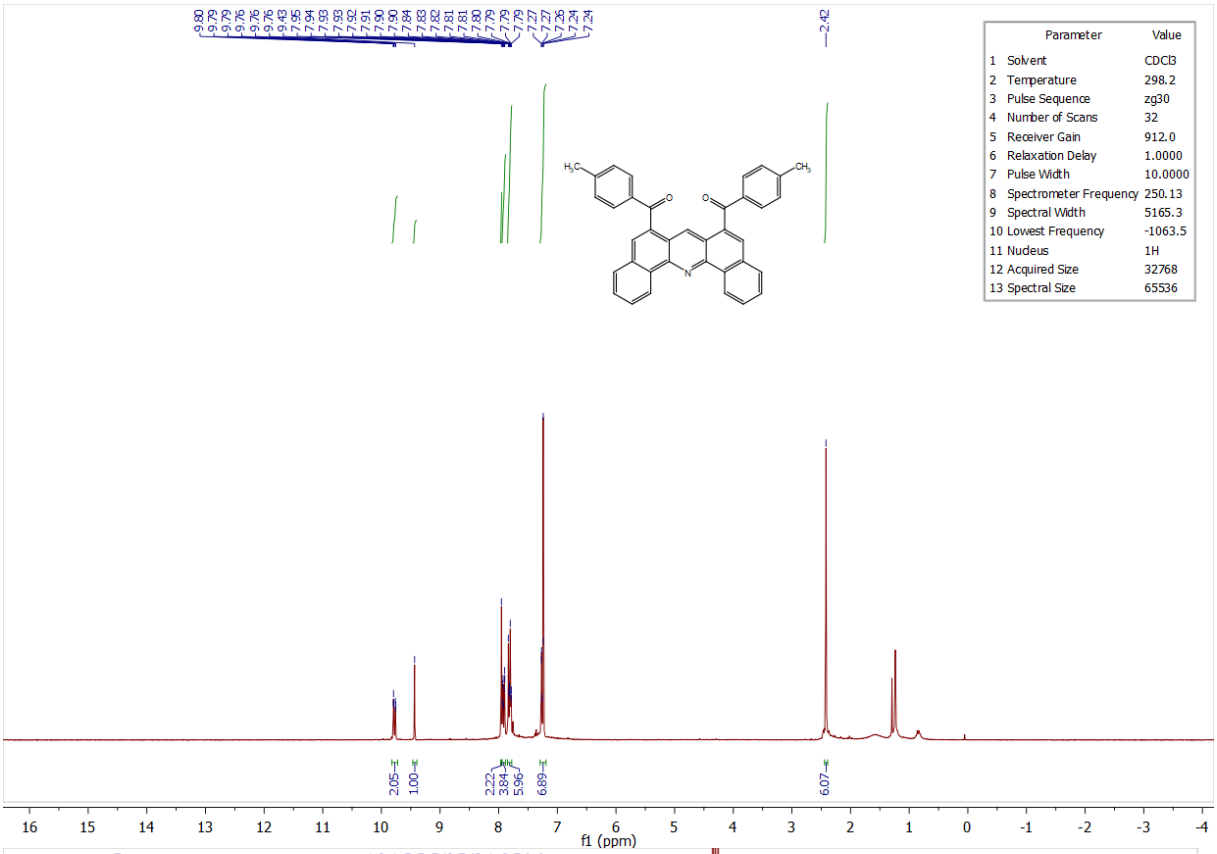


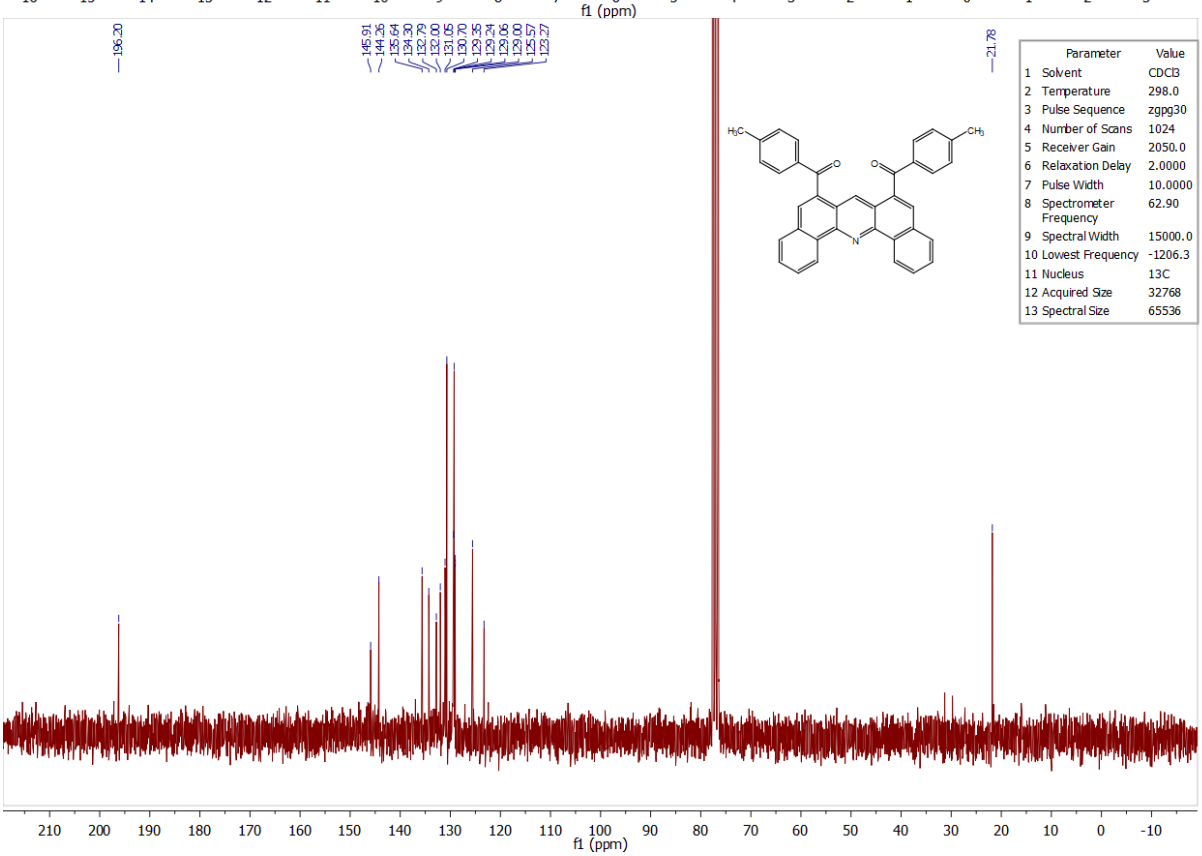
Dibenzo[*c*,*h*]acridin-6,8-diylbis(phenylmethanone) (8a)



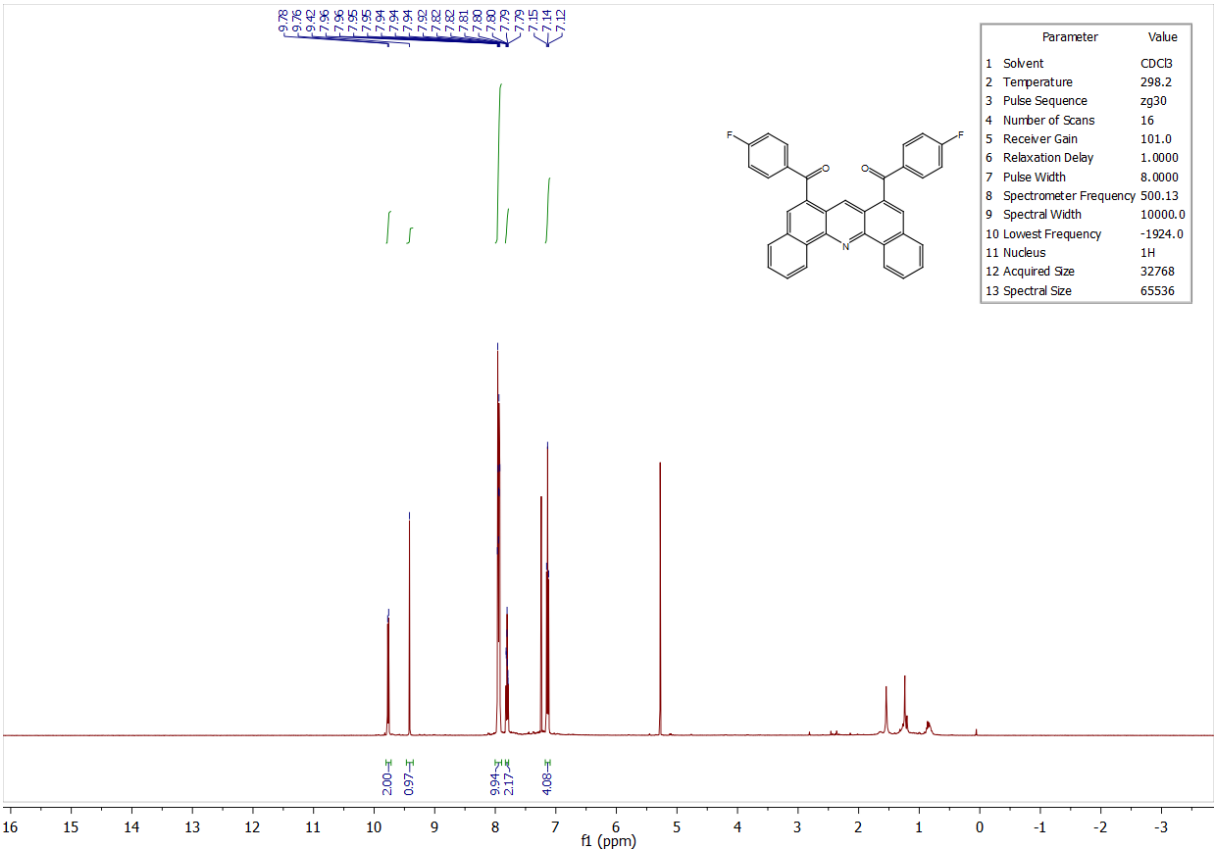


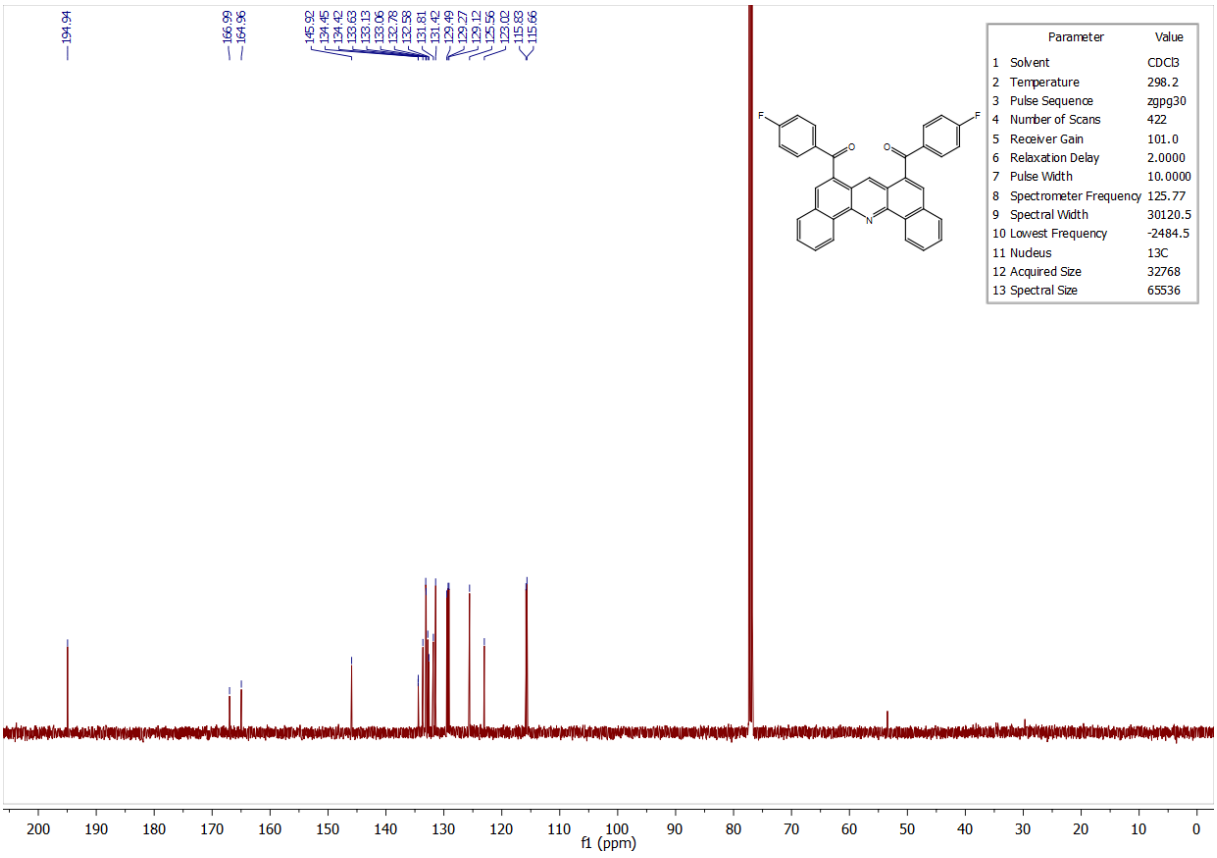
Dibenzo[*c*,*h*]acridin-6,8-diylbis(*p*-tolylmethanone) (8b)

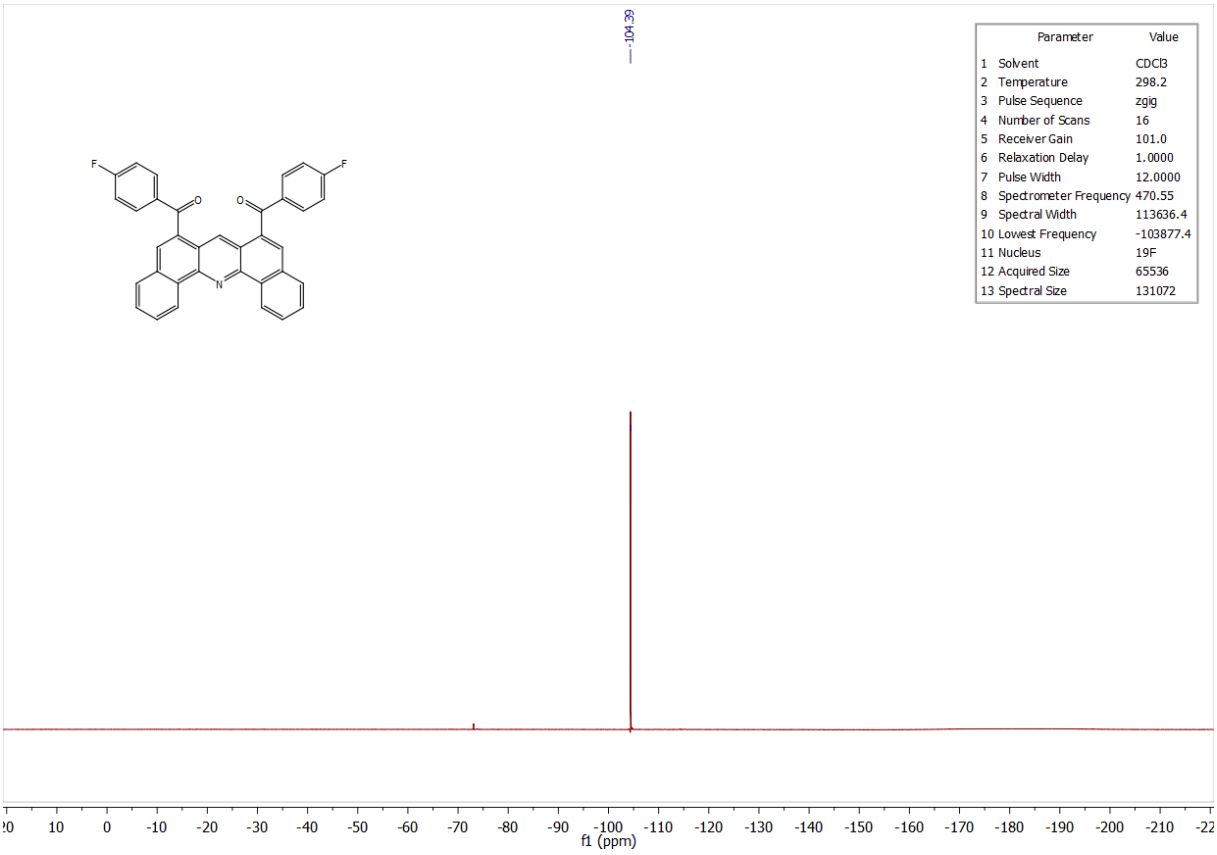




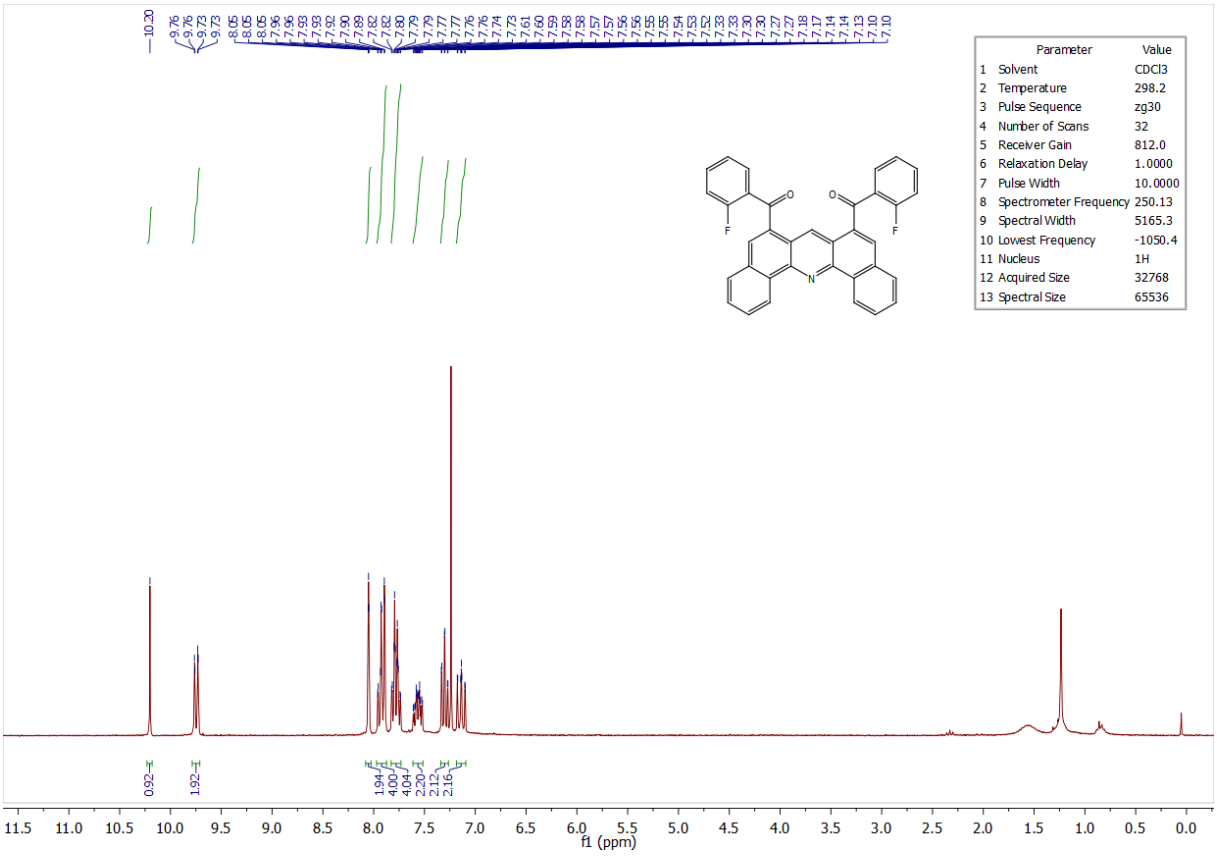
Dibenzo[*c*,*h*]acridin-6,8-diylbis((4-fluorophenyl)methanon) (8c)

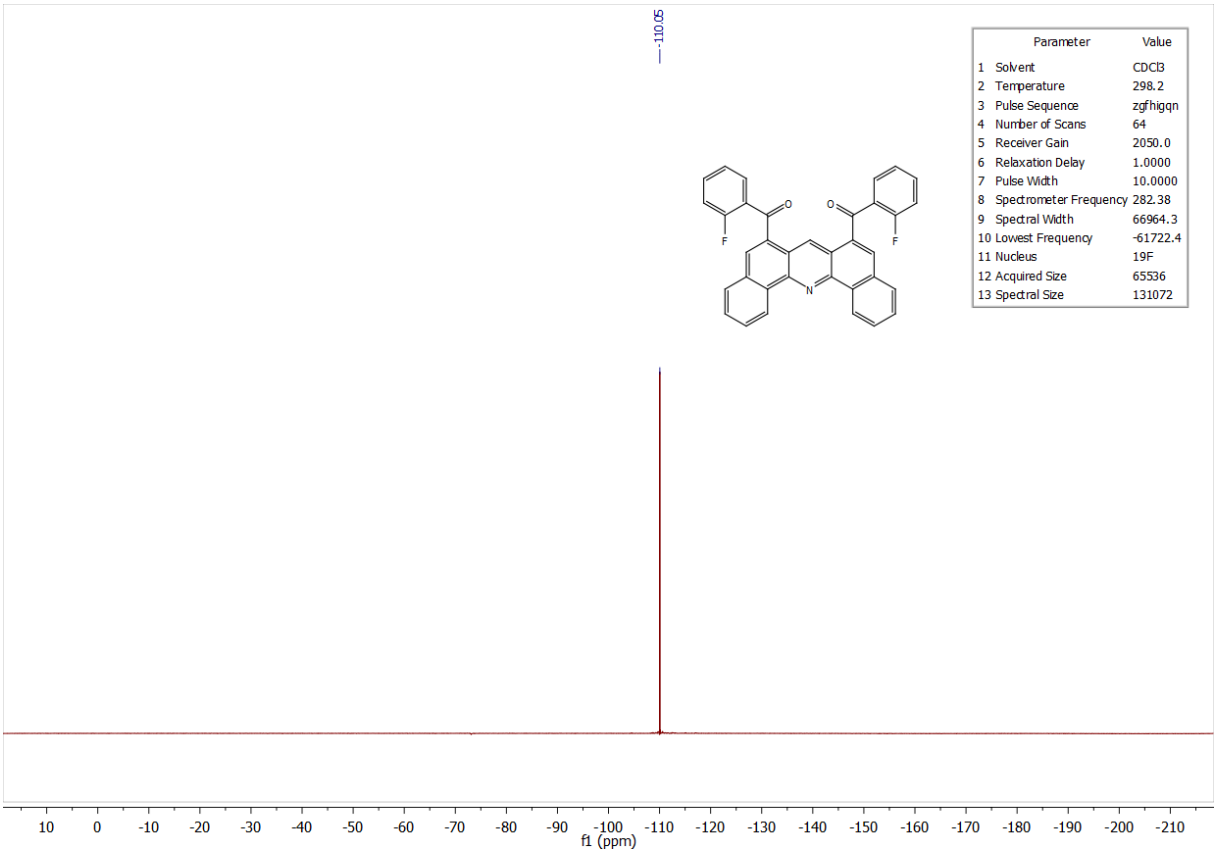


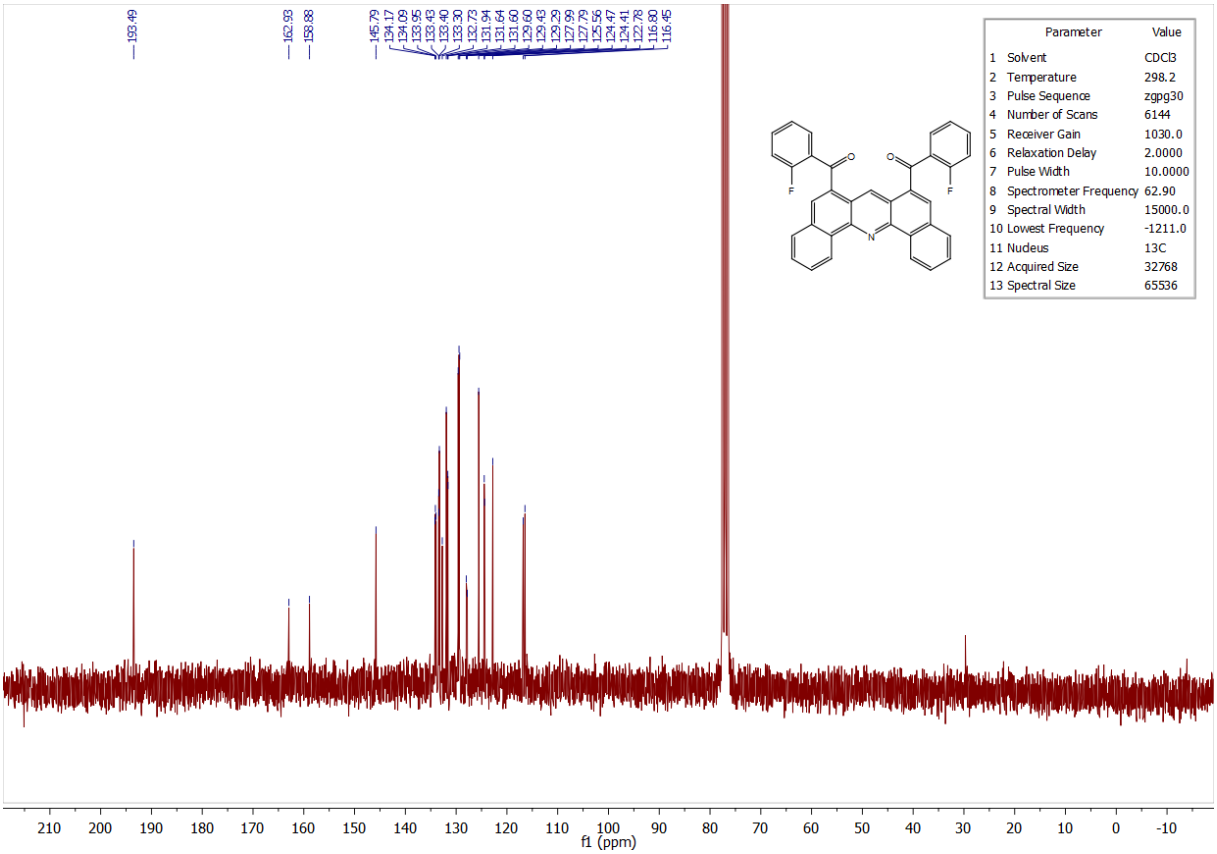




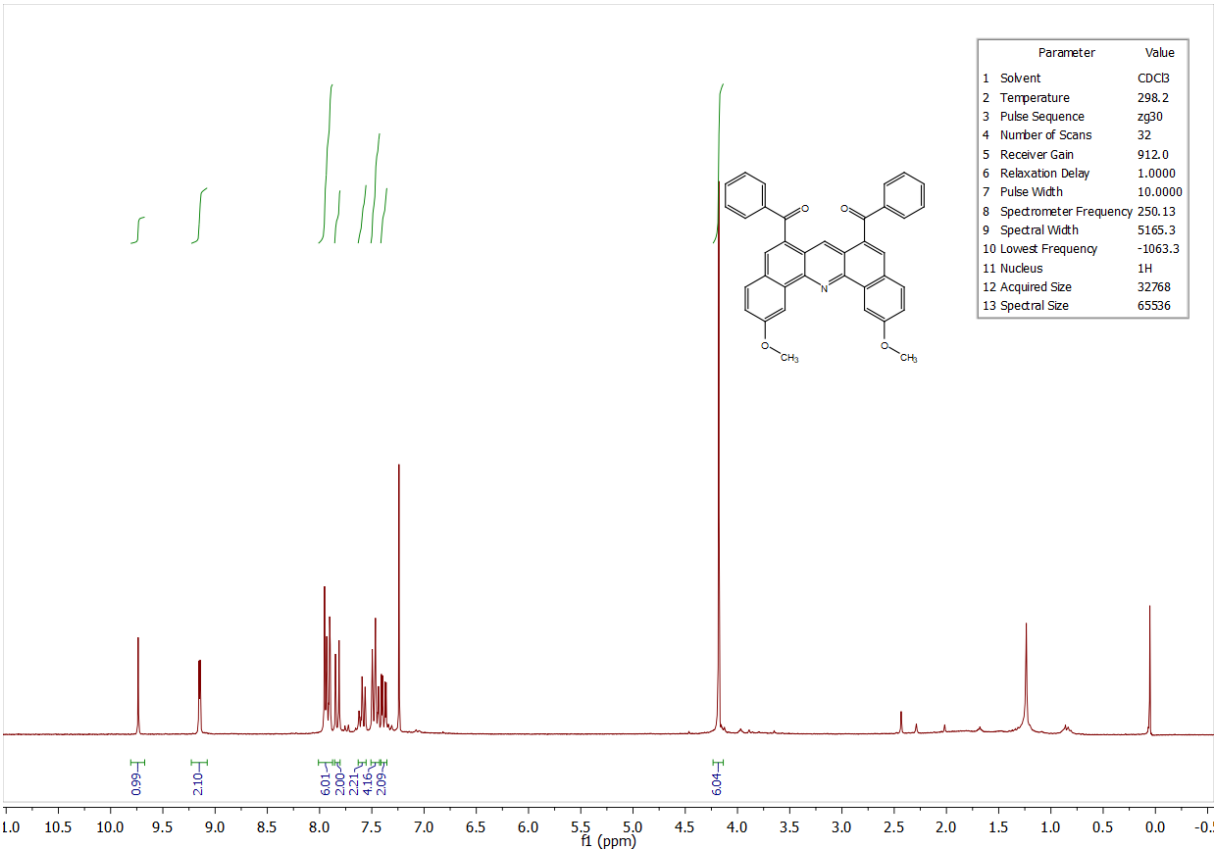
Dibenzo[*c*,*h*]acridin-6,8-diylbis((2-fluorophenyl)methanone) (8d)

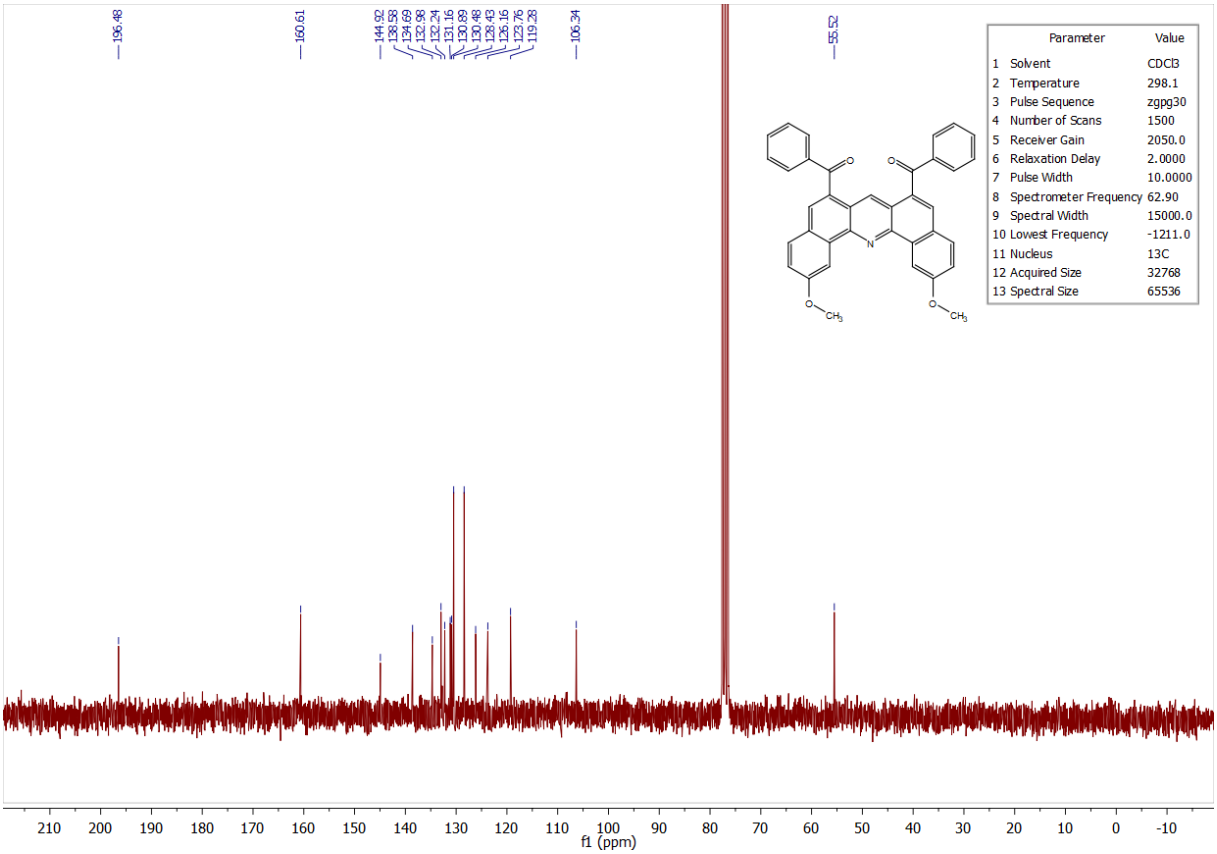






(2,12-Dimethoxydibenzo[*c*,*h*]acridin-6,8-diyl)bis(phenylmethanone) (8e)


Parameter Value
1 Solvent CDCl3
2 Temperature 298.2
3 Pulse Sequence zg30
4 Number of Scans 32
5 Receiver Gain 912.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.0000
8 Spectrometer Frequency 250.13
9 Spectral Width 5165.3
10 Lowest Frequency -1063.3
11 Nucleus 1H
12 Acquired Size 32768
13 Spectral Size 65536
0.99
2.10
6.01
2.00
2.21
4.16
2.09
6.04
f1 (ppm)


196.48
160.61
144.92
138.58
134.69
132.98
132.24
131.16
130.89
130.48
128.43
126.16
123.76
119.28
106.34
55.52
Parameter Value
1 Solvent CDCl3
2 Temperature 298.1
3 Pulse Sequence zgpg30
4 Number of Scans 1500
5 Receiver Gain 2050.0
6 Relaxation Delay 2.0000
7 Pulse Width 10.0000
8 Spectrometer Frequency 62.90
9 Spectral Width 15000.0
10 Lowest Frequency -1211.0
11 Nucleus 13C
12 Acquired Size 32768
13 Spectral Size 65536
f1 (ppm)