Supplementary Information

An attempt to consider cooperativity in helical-sense preferences induced in fused macrocycles

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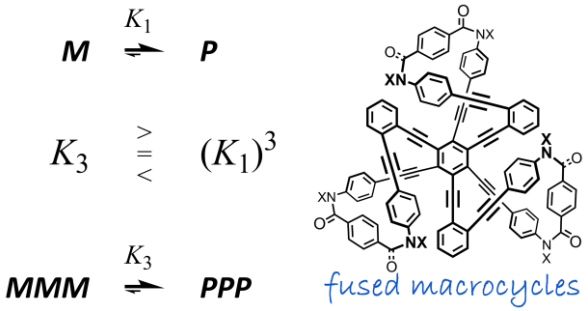

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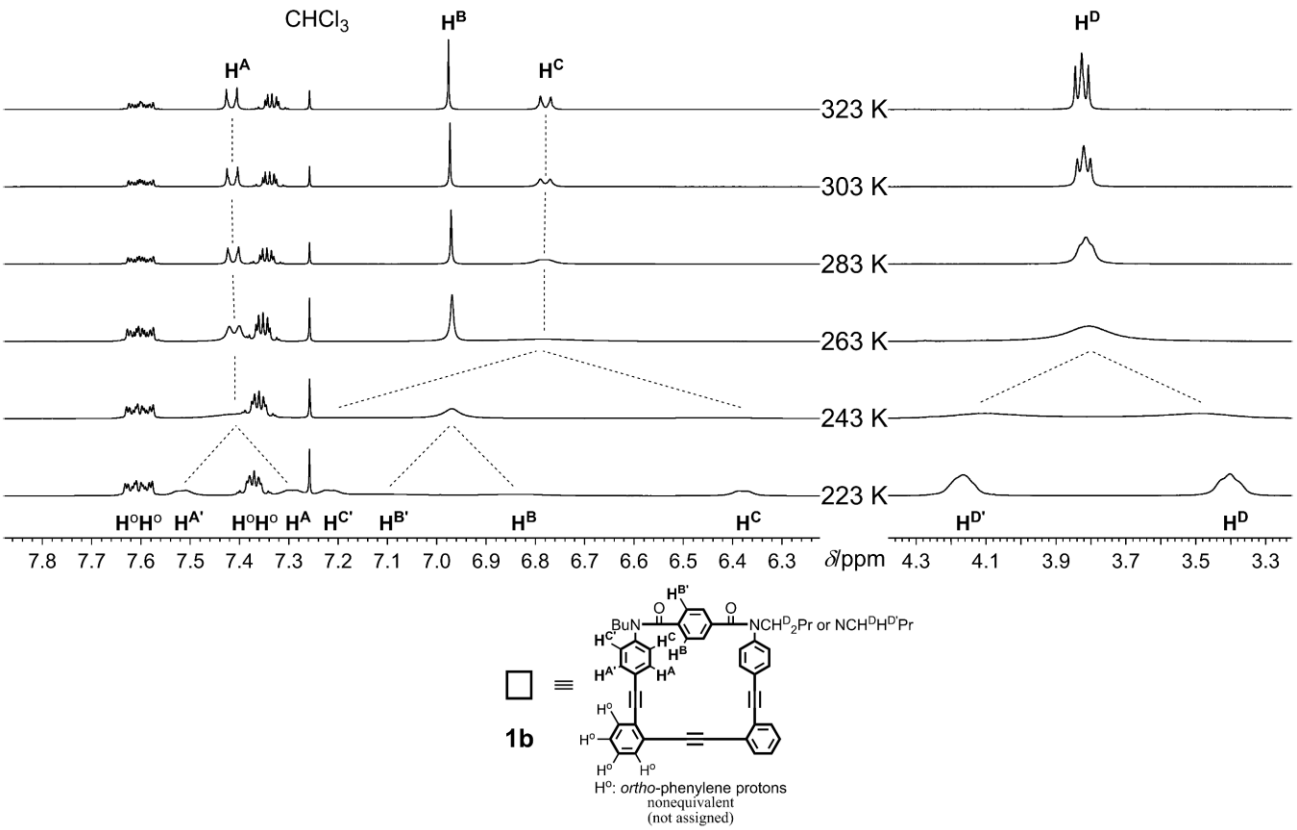


Fig. S1A Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methylene protons) of **1b**,[1] measured in chloroform-*d* at 223-323 K.

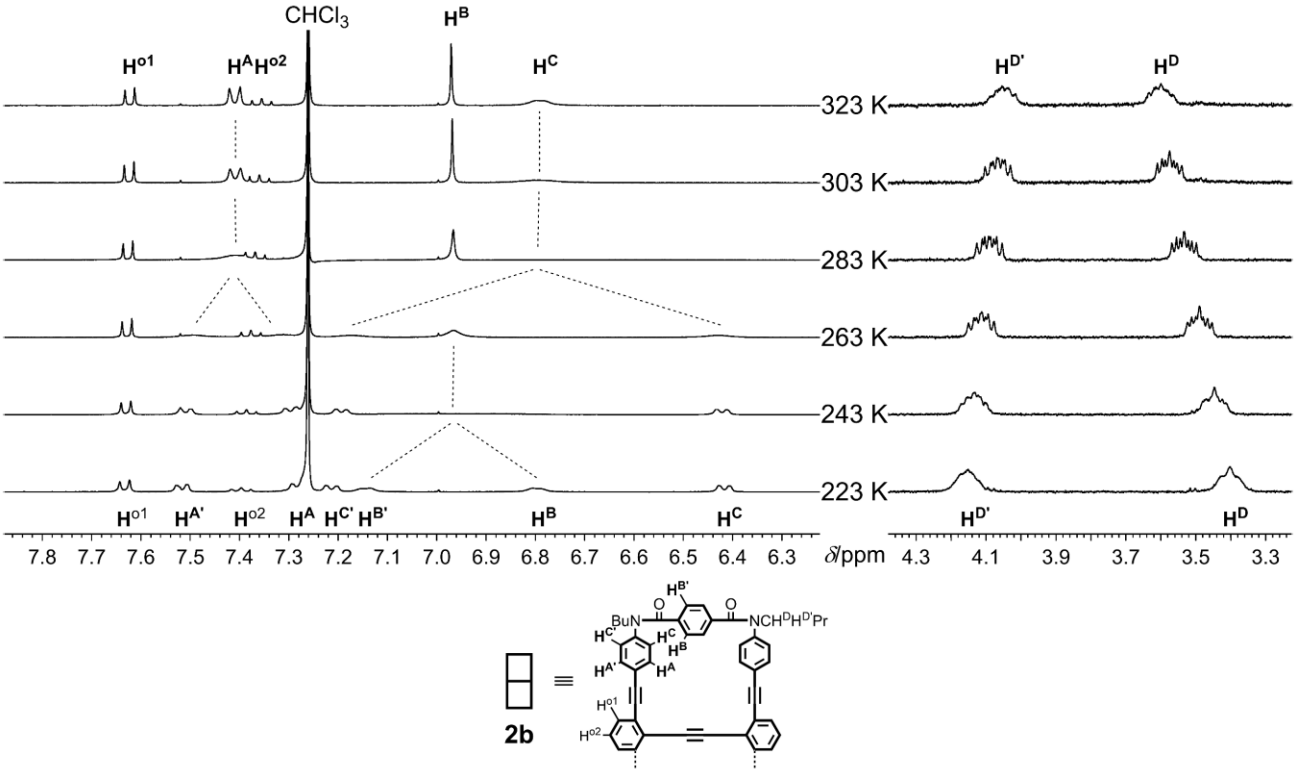


Fig. S1B Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methylene protons) of **2b**,[2] measured in chloroform-*d* at 213-313 K.

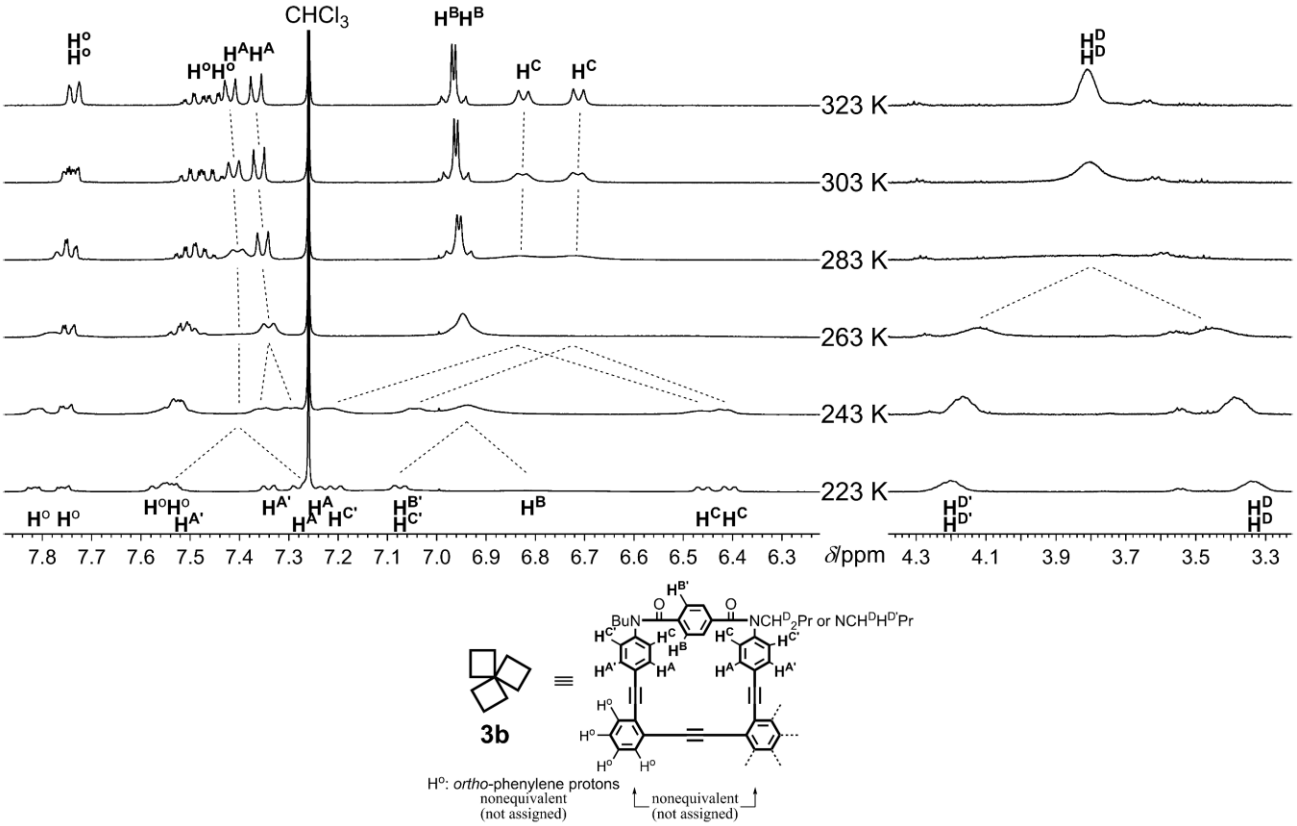


Fig. S1C Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methylene protons) of **3b**, measured in chloroform-*d* at 223-323 K.

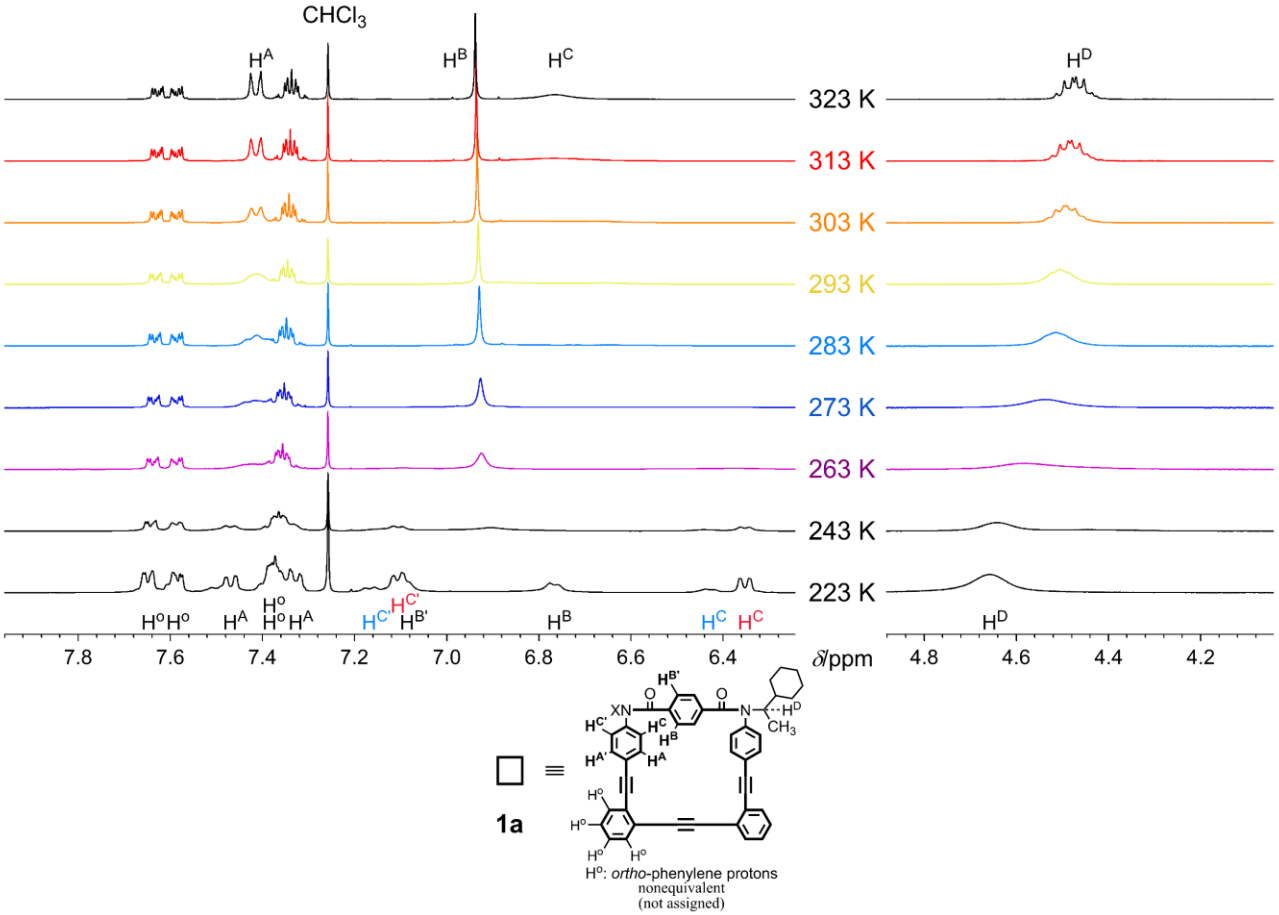


Fig. S2A Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methine protons) of **1a**,[1] measured in chloroform-*d* at 223-323 K.

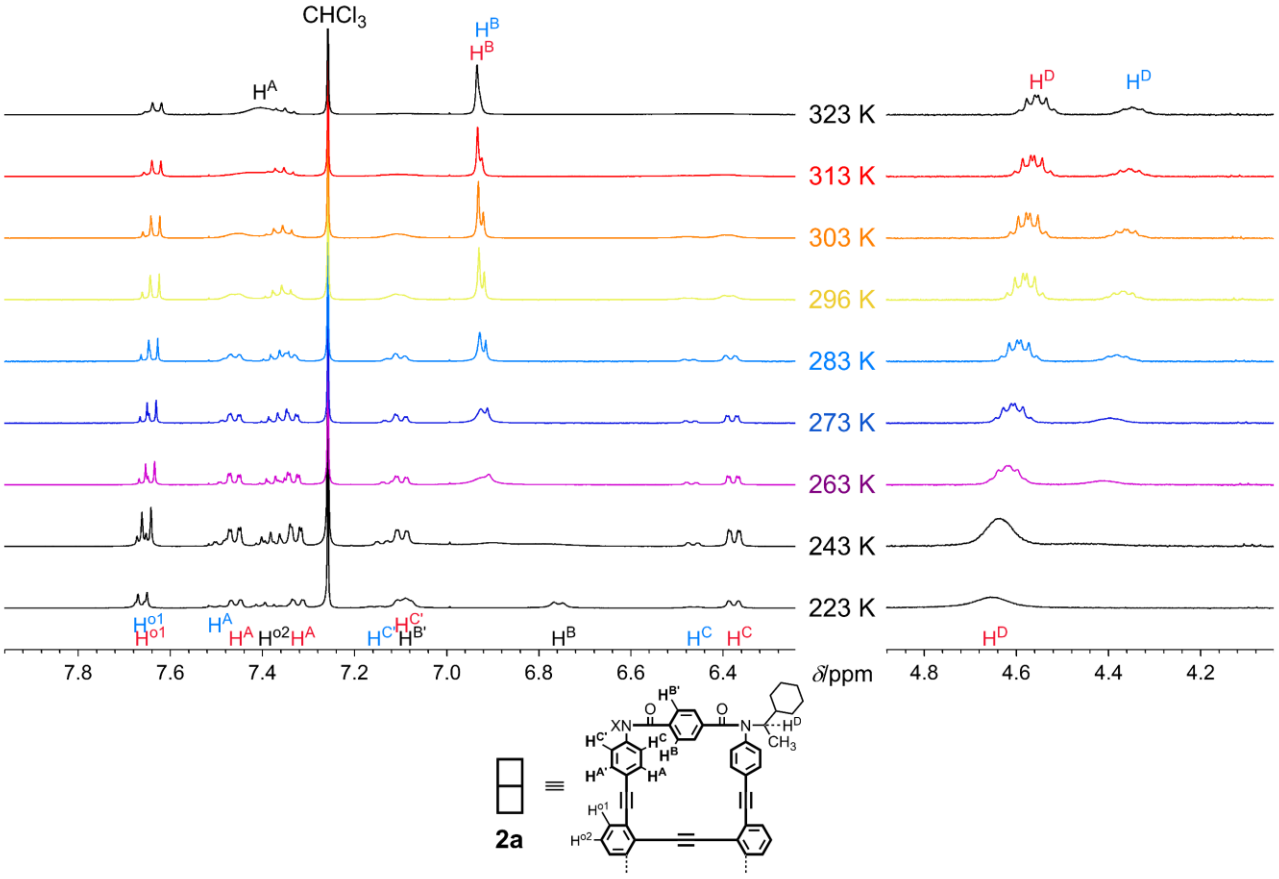


Fig. S2B Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methine protons) of **2a**,[2] measured in chloroform-*d* at 223-323 K.

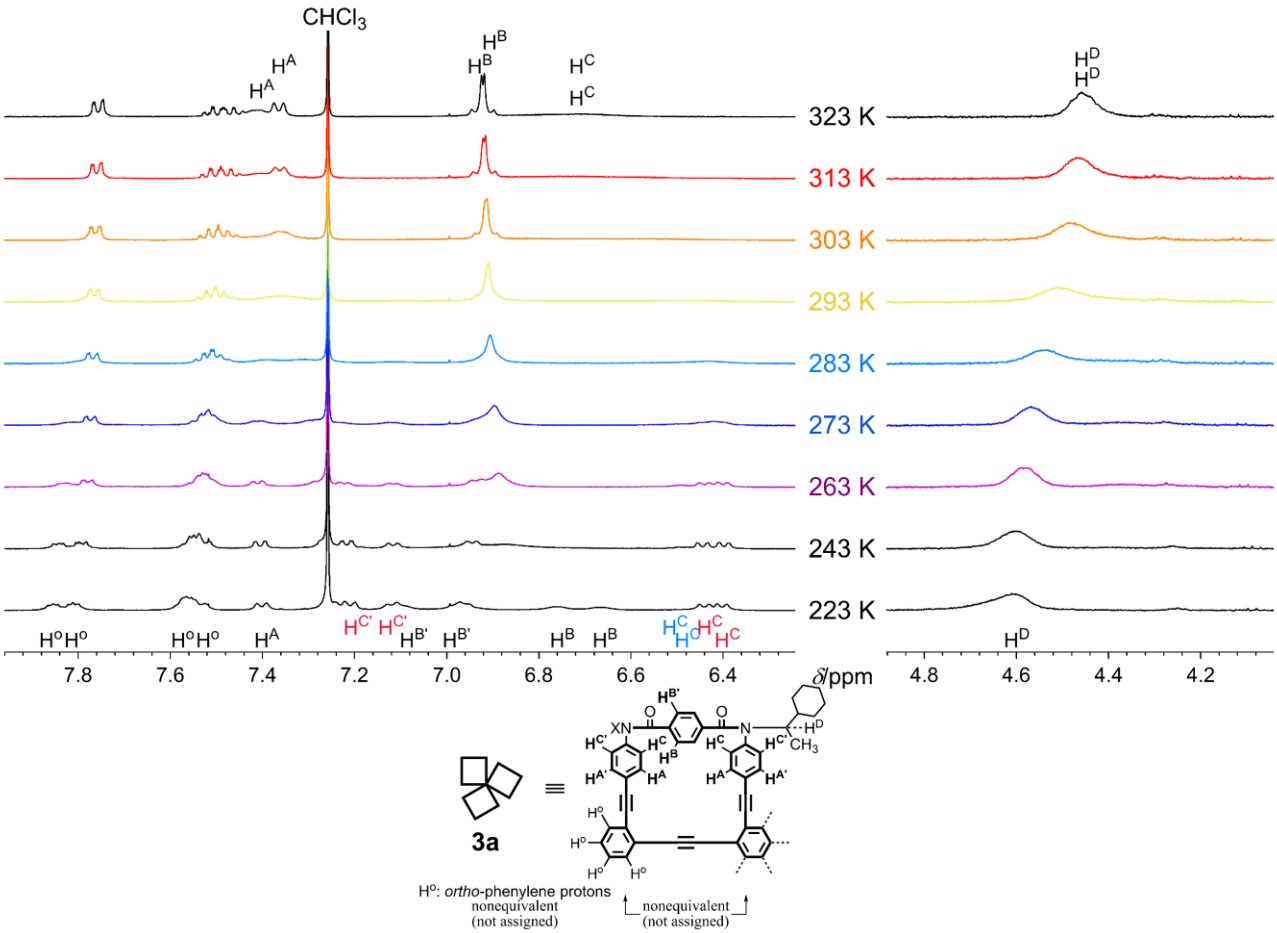


Fig. S2C Partial VT-^{1}H NMR spectra (400 MHz; left: aromatic protons and right: methine protons) of **3a**, measured in chloroform-*d* at 223-323 K.

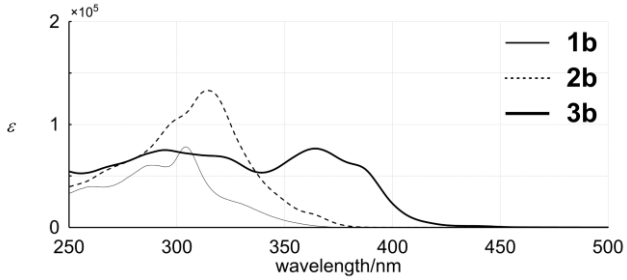


Fig. S3 UV spectra of **1b**,[1] **2b**[2] and **3b**, measured in dichloromethane at room temperature.

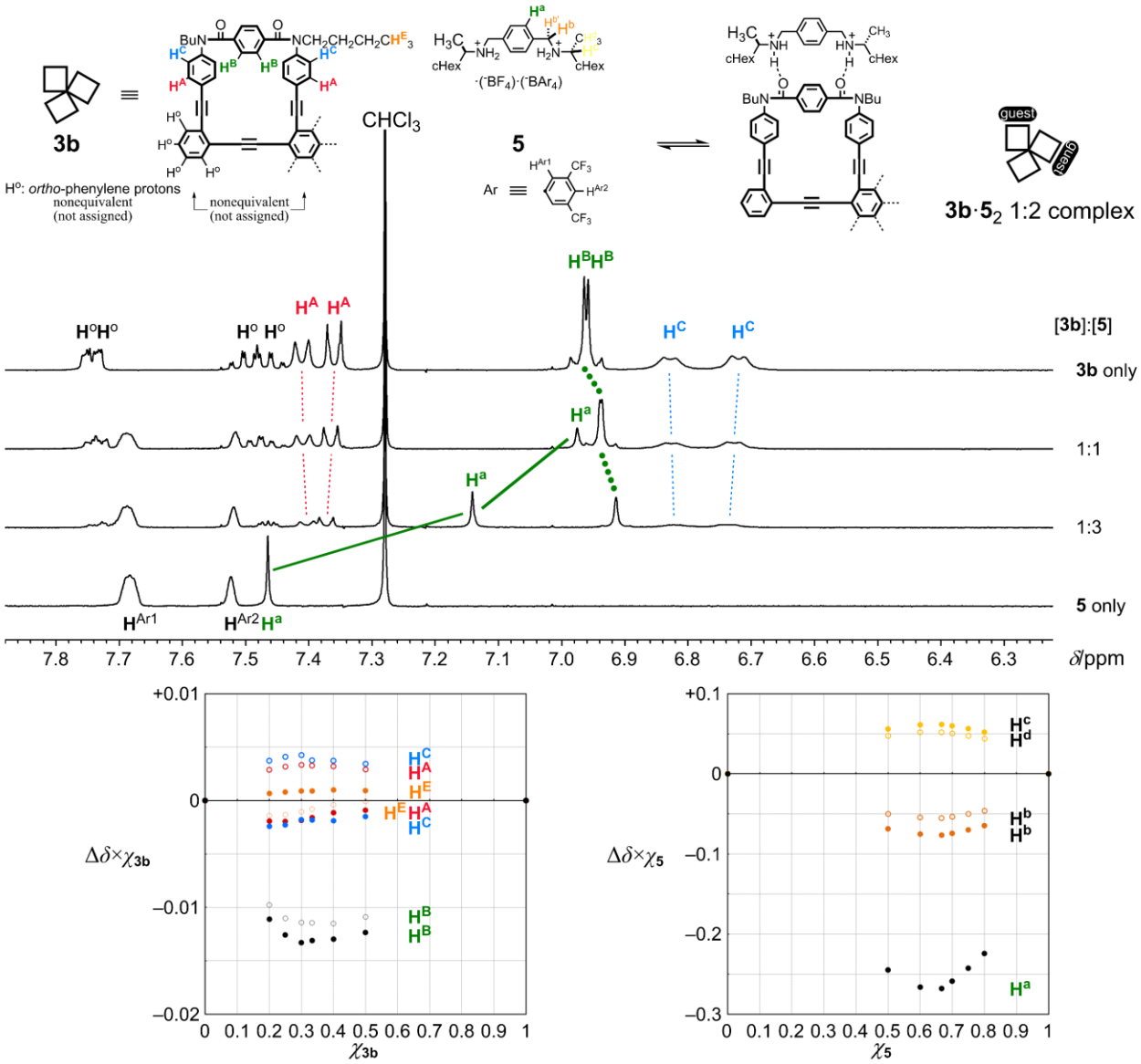


Fig. S4 Partial ^{1}H NMR spectra (aromatic region) of **3b** in the presence of (*R*)$_2$-**5** ([**3b**]:[**5**] = 10:0 (**3b** only), 1:1, 1:3 and 0:10 (**5** only)), and Job plots based on changes in the chemical shift ($\Delta\delta = \delta_{\mathbf{3b}\cdot\mathbf{5}} - \delta_{\mathbf{3b}\text{ or }\mathbf{5}}$) on complexation, measured in 3 vol% acetonitrile-d_3/chloroform-*d* at 303 K. χ denotes the molar fraction, [**3b**] or [**5**]/[**3b**]+[**5**], [**3b**]+[**5**] = 2 mM.

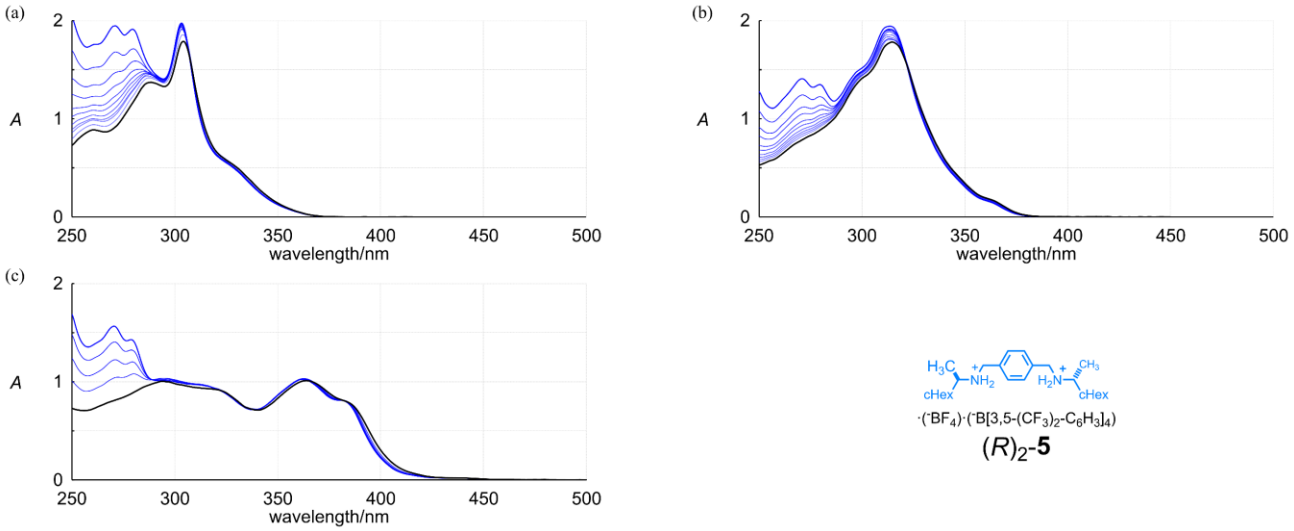


Fig. S5 UV spectra of (a) **1b** ([**1b**] = 2.28×10^{-4} M), (b) **2b** ([**2b**] = 1.34×10^{-4} M), and (c) **3b** ([**3b**] = 1.31 ×10^{-4} M) in the presence of (*R*)$_2$-**5** [(0 equiv., black line) and (a) 0.25, 0.50, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0 and 8.0 equiv., (b) 0.25, 0.50, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0 and 8.0 equiv., (c) 3.0, 6.0, 9.0 and 12 equiv. (blue lines)]. All spectra were measured in dichloromethane at room temperature. Cell length = 0.1 cm.

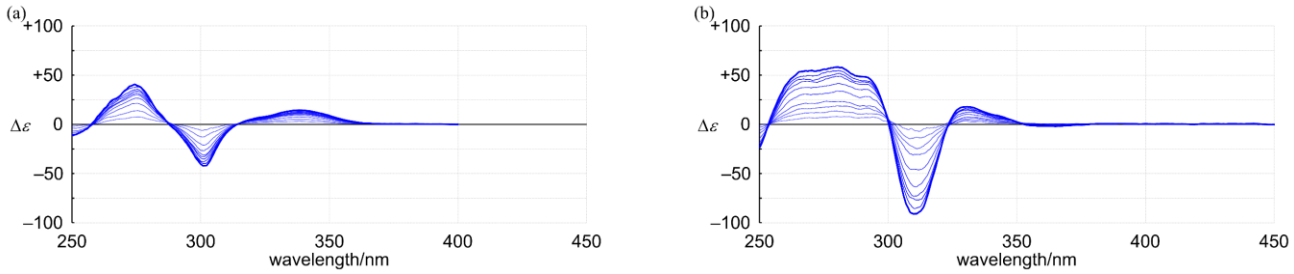


Fig. S6 CD spectra of (a) **1b** ([**1b**] = 2.28×10^{-4} M) and (b) **2b** ([**2b**] = 1.34×10^{-4} M) in the presence of (*R*)$_2$-**5** (0.25, 0.50, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0 and 8.0 equiv.). All spectra were measured in dichloromethane at 293 K.

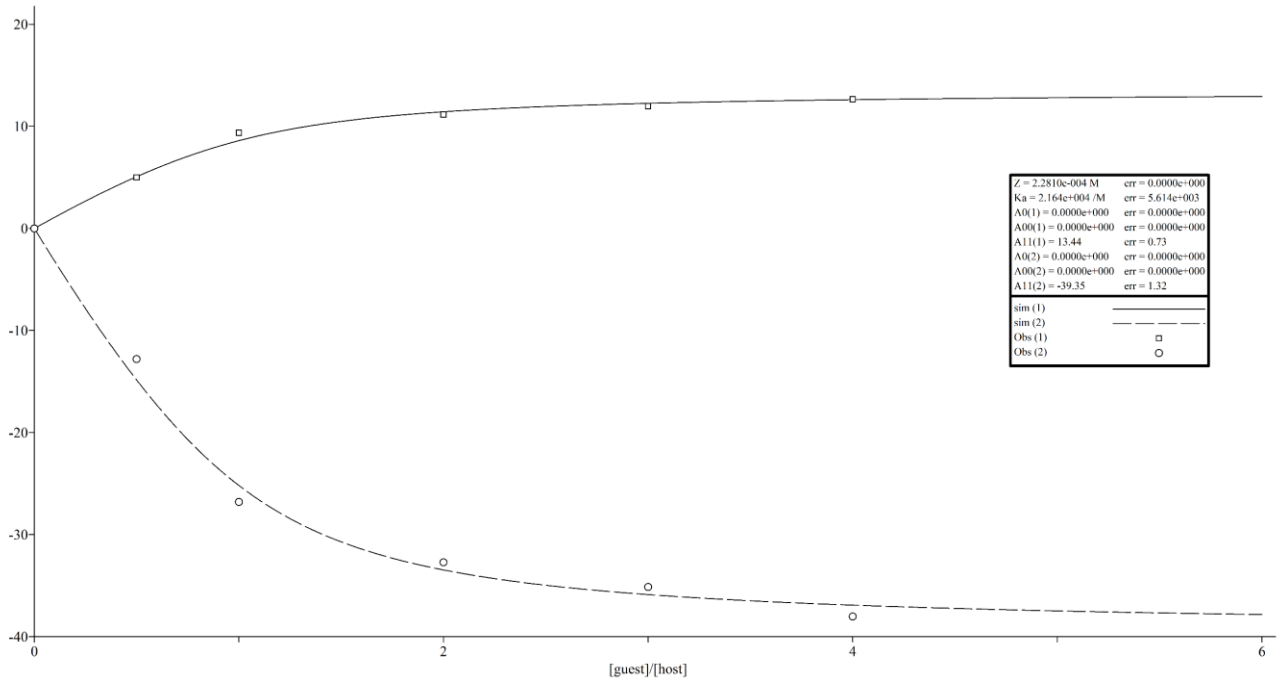


Fig. S7A Titration curves 1:1-fitted with a program of TitrationFit software[3], based on plots of complexation-induced molar CDs [$\Delta\varepsilon$ at 301 nm (circle) and 338 nm (square)] versus equivalents of $(R)_2$-**5** added to a solution of **1b** ([**1b**] = 2.28×10^{-4} M), measured in dichloromethane at 293 K.

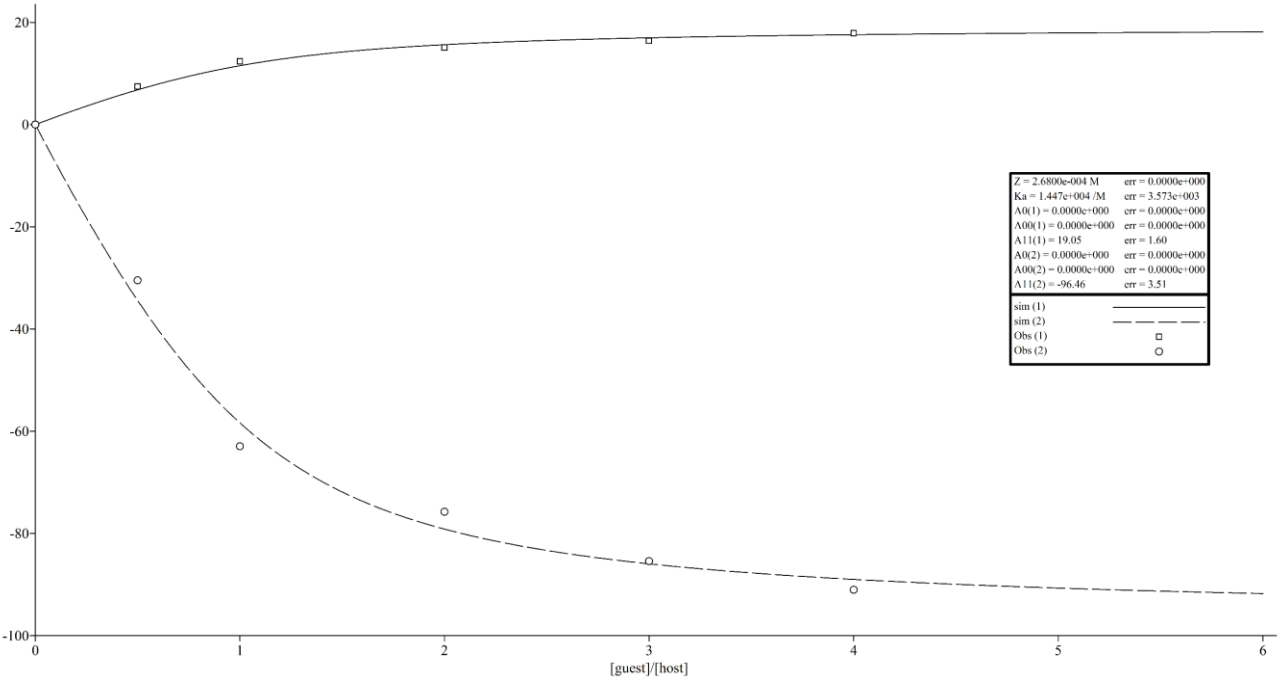


Fig. S7B Titration curves 1:1-fitted with a program of TitrationFit software[3], based on plots of complexation-induced molar CDs [$\Delta\varepsilon$ at 310 nm (circle) and 331 nm (square)] versus equivalents of $(R)_2$-**5** added to a solution of **2b** ([**2b**] = 1.34×10^{-4} M, [Terephthalamide] = 2.68×10^{-4} M), measured in dichloromethane at 293 K.

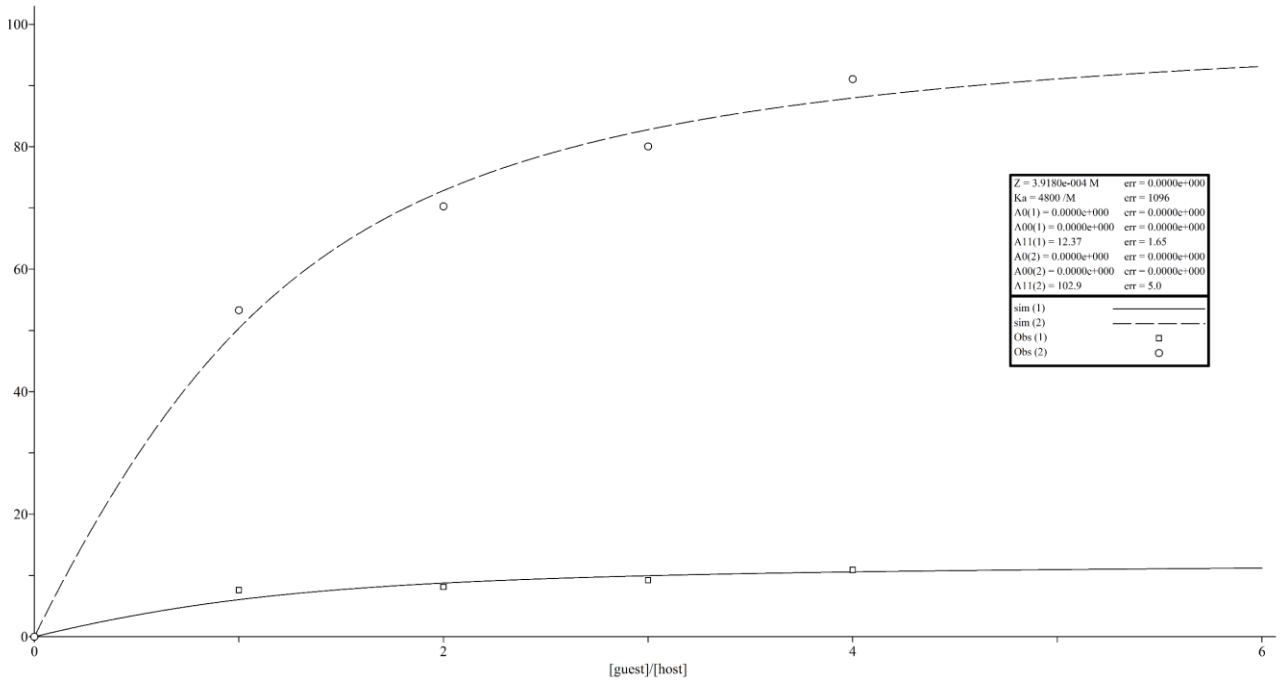
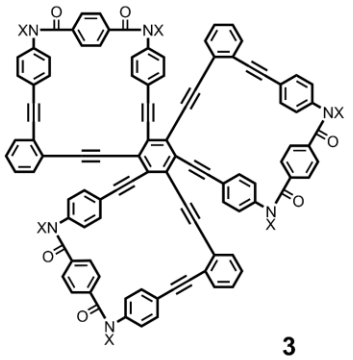


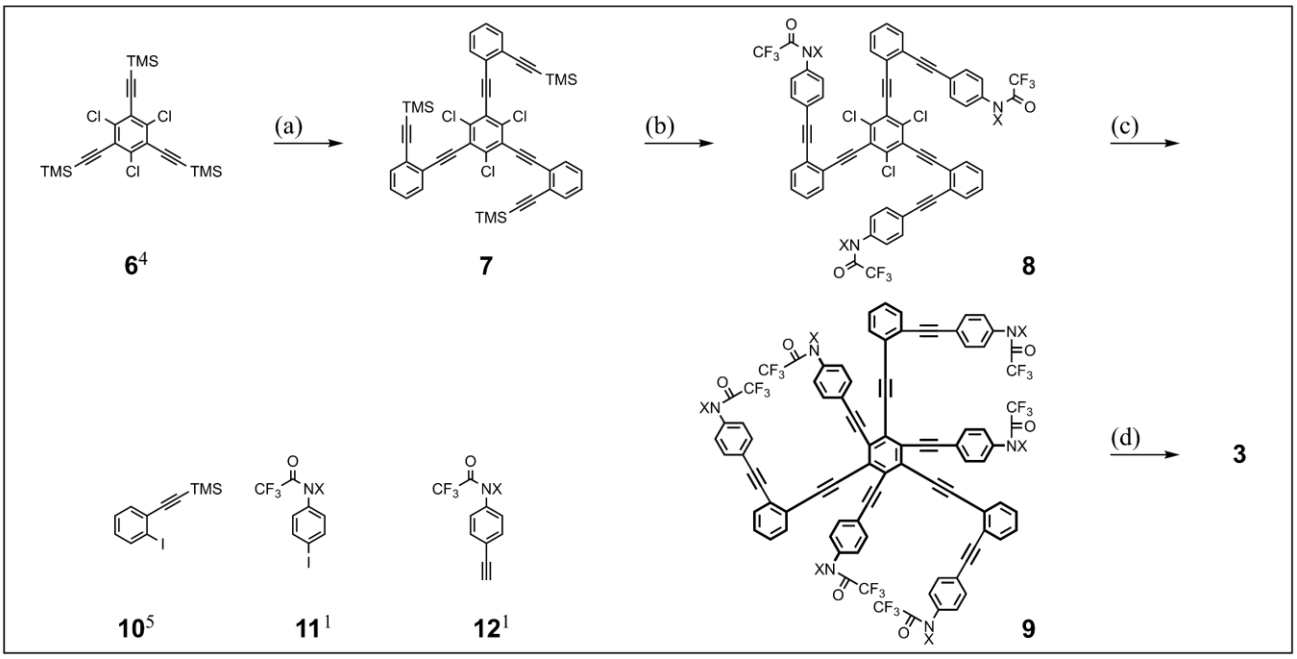
Fig. S7C Titration curves 1:1-fitted with a program of TitrationFit software[3], based on plots of complexation-induced molar CDs [$\Delta\varepsilon$ at 313.5 nm (circle) and 334 nm (square)] versus equivalents of $(R)_2$-**5** added to a solution of **3b** ([**3b**] = 1.31×10^{-4} M, [Terephthalamide] = 3.92×10^{-4} M), measured in dichloromethane at 293 K.

Experimental



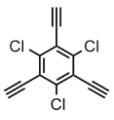
3

a: X = (*R*)-CH(CH$_3$)(cHex)
b: X = *n*Bu



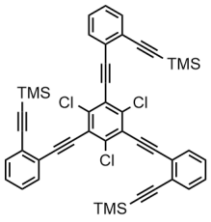
(a) Preparation of **7**

To a solution of **6** (250 mg, 0.532 mmol) in THF (3 mL) and MeOH (1 mL) was added K_2CO_3 (221 mg, 1.60 mmol) at room temperature. The mixture was stirred at room temperature for 30 min and diluted with water and dichloromethane. The organic layer was separated, dried over magnesium sulfate, and then purified by column chromatography on SiO_2 (dichloromethane) to give **6'** as a pale pinkish white solid (127 mg, 94%), which was immediately subjected to the next reaction.



6'

To a solution of **10** (5.72 g, 19.1 mmol), $Pd(PPh_3)_4$ (295 mg, 0.255 mmol) and CuI (98 mg, 0.51 mmol) in THF (85 mL) and ^{i}Pr$_2$NH (85 mL) was added a solution of **6'** (1.07 g, 4.23 mmol) in THF (15 mL) at 51 °C via a syringe pump over 2.5 h under an argon atmosphere and the mixture was further stirred at that temperature for 3 h. After removal of a solid by filtration through a Celite pad, the filtrate was concentrated and purified by column chromatography on SiO_2 (dichloromethane/hexane) to give **7** (1.75 g) as a white amorphous solid in 54% yield. An analytical sample was obtained as a white amorphous solid by further purification through GPC (chloroform; JAIGEL-1H & 2H, Japan Analytical Industry Co., Ltd., Japan).



7

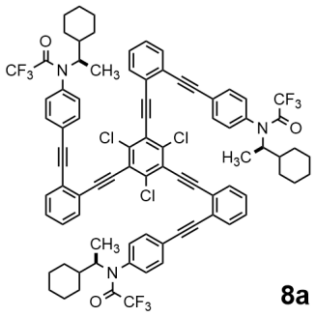
7: mp 128-129 °C; IR (KBr) ν_{max}/cm^{-1} 2958, 2216, 2159, 1479, 1441, 1387, 1371, 1249, 865, 841, 756, 644, 510; ^{1}H

NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.64-7.60 (3H, m), 7.57-7.52 (3H, m), 7.36-7.31 (6H, m), 0.25 (27H, s); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 138.4, 132.8, 132.7, 128.9, 128.2, 125.6, 124.9, 123.0, 102.9, 99.4, 99.4, 86.6, 0.0; FD-LRMS *m/z* 768.1 (M^+, 80%), 769.1 ([M+1]$^+$, 54), 770.1 ([M+2]$^+$, 100), 771.1 ([M+3]$^+$, 59), 772.1 ([M+4]$^+$, 52), 773.1 ([M+5]$^+$, 26); FD-HRMS Found: 768.14164, Calc. for $C_{45}H_{39}Cl_3Si_3$: 768.14251.

(b) Preparation of **8a** and **8b**

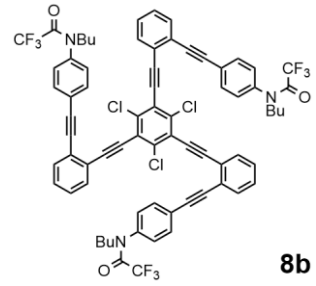
To a solution of **7**, **11a**/**b**, $Pd(PPh_3)_4$ and CuI in THF and Et_3N was added a solution of TBAF in THF under an argon atmosphere. The reaction mixture was diluted with ethyl acetate, which was passed through a Celite/SiO_2 pad. The filtrate was concentrated and purified by column chromatography on SiO_2 (dichloromethane/hexane) to give **8** (**a**: 482 mg, 85%) as a yellowish white amorphous solid. For **8b**, the product was dissolved in CH_2Cl_2 (90 mL) containing a small amount of Et_3N (2 mL), which was treated with trifluoroacetic anhydride (1.8 mL), washed with aq. satd. $NaHCO_3$, dried over magnesium sulfate, and then purified by column chromatography on SiO_2 (dichloromethane/hexane) to give **8b** as a yellow amorphous solid (2.07 g, 83%). Each analytical sample was obtained as a white amorphous solid by further purification through GPC (chloroform).

	7	**11a**/**b**	$Pd(PPh_3)_4$	CuI	1M TBAF	addition	THF/Et_3N
8a	301 mg (0.391 mmol)	995 mg (2.34 mmol)	41 mg (0.035 mmol)	12 mg (0.063 mmol)	1.2 mL (1.2 mmol)	61 ºC 20 min	18 mL/18 mL
8b	1.50 g (1.95 mmol)	4.34 g (11.7 mmol)	203 mg (0.176 mmol)	50 mg (0.26 mmol)	6.1 mL (6.1 mmol)	60 ºC 1 h	45 mL/45 mL



8a

8a: mp 125-127 °C; $[\alpha]_D^{26}$ = –29 (*c* = 0.10, chloroform); IR (KBr) ν_{max}/cm^{-1} 2930, 2852, 2213, 1695, 1509, 1447, 1419, 1372, 1207, 1188, 1150, 831, 756, 560; ^1H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.64-7.59 (6H, m), 7.57-7.48 (6H, br.m), 7.43-7.35 (6H, m), 7.18-7.07 (6H, br.m), 4.35 (3H, dq, *J* = 6.8, 10 Hz), 1.92 (3H, br.d), 1.79-1.49 (12H, m), 1.46-1.33 (3H, m), 1.33-0.97 (12H, m), 1.03 (9H, d, *J* = 6.8 Hz), 0.97-0.79 (3H, m); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 156.8 (C(=O)CF_3), 138.4, 136.0, 132.7, 132.3, 132.2, 132.1, 130.7, 129.3, 129.2, 128.5, 125.3, 124.6, 124.2, 123.0, 116.4 (CF_3), 99.5, 92.2, 89.6, 86.5, 59.8, 40.3, 30.6, 29.7, 26.1, 25.9, 25.7, 16.2; FD-LRMS *m/z* 1443.3 (M^+, 72%), 1444.3 ([M+1]$^+$, 68), 1445.3 ([M+2]$^+$, 100), 1446.3 ([M+3]$^+$, 75), 1447.3 ([M+4]$^+$, 56), 1448.3 ([M+5]$^+$, 32), 1449.3 ([M+6]$^+$, 15); FD-HRMS Found: 1443.42597, Calc. for $C_{84}H_{69}Cl_3F_9N_3O_3$: 1443.42608.



8b

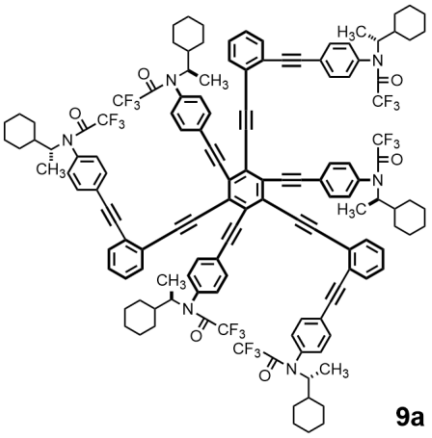
8b: mp 95-97 °C; IR (KBr) ν_{max}/cm^{-1} 2931, 2213, 1698, 1510, 1384, 1208, 1151, 846, 756; ^1H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.64-7.59 (6H, m), 7.55 (6H, d, *J* = 8.4 Hz), 7.43-7.35 (6H, m), 7.15 (6H, d, *J* = 8.4 Hz), 3.68 (6H, t, *J* = 7.6 Hz), 1.52-1.44 (6H, m), 1.31-1.21 (6H, m), 0.84 (9H, t, *J* = 7.6 Hz); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 156.4 (C(=O)CF_3), 138.9, 138.4, 132.7, 132.7, 132.3, 129.2, 128.5, 128.3, 125.2, 124.6, 124.0, 123.0, 116.3 (CF_3), 99.5, 92.2, 89.5, 86.5, 51.5, 28.8, 19.7, 13.6; FD-LRMS *m/z* 1281.2 (M^+, 80%), 1282.2 ([M+1]$^+$, 65), 1283.2 ([M+2]$^+$, 100), 1284.2 ([M+3]$^+$, 70), 1285.2 ([M+4]$^+$, 52), 1286.2 ([M+5]$^+$, 28), 1287.1 ([M+6]$^+$, 13); FD-HRMS Found: 1281.28474, Calc.

for $C_{72}H_{51}Cl_3F_9N_3O_3$: 1281.28523.

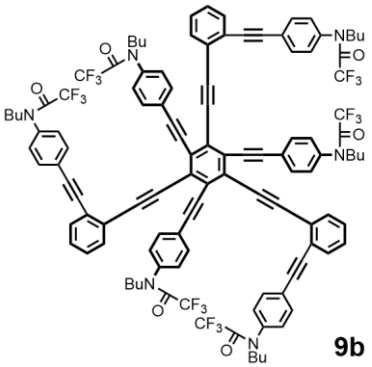
(c) Preparation of **9a** and **9b**

To a solution of **8a**/**b** and **12a**/**b** in 1,4-dioxane and $^{i}Pr_2NH$ were added $PdCl_2(CH_3CN)_2$, X-Phos[6] and CuI at room temperature under an argon atmosphere. The mixture was stirred at 90 ºC for 23 h for **9a** or 28 h for **9b**. After removal of a solid by filtration through a Celite pad, the filtrate was concentrated and purified by column chromatography on SiO_2 (dichloromethane-ethyl acetate/dichloromethane for **9a** or dichloromethane/hexane-ethyl acetate/dichloromethane for **9b**), followed by GPC (chloroform) to give **9** (**a**: 479 mg, 63% and **b**: 320 mg, 69%) as a brown solid. Each analytical sample was obtained as a yellow amorphous solid by further purification through HPLC with a standard normal-phase column (0.5vol% for **9a** or 1vol% for **9b** tetrahydrofuran/dichloromethane; YMC-Pack SIL, SIL-06, YMC Co., Ltd., Japan).

	8a/**b**	**12a**/**b**	$PdCl_2(CH_3CN)_2$	X-Phos	CuI	dioxane/$^{i}Pr_2NH$
9a	480 mg (0.332 mmol)	3.22 g (9.96 mmol)	13 mg (0.050 mmol)	47 mg (0.099 mmol)	16 mg (0.084 mmol)	6.4 mL/3.7 mL
9b	300 mg (0.234 mmol)	1.89 g (7.00 mmol)	13 mg (0.050 mmol)	45 mg (0.093 mmol)	18 mg (0.095 mmol)	12 mL/7.3 mL



9a: mp 136-137 °C; $[\alpha]_D^{26}$ = –27 (*c* = 0.12, chloroform); IR (KBr) ν_{max}/cm^{-1} 2931, 2853, 2204, 1698, 1509, 1449, 1415, 1207, 831, 756; ^{1}H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.71-7.66 (6H, m), 7.48-7.37 (18H, m), 6.99 (6H, d, *J* = 8.8 Hz), 6.97 (6H, d, *J* = 8.8 Hz), 4.36-4.24 (6H, m), 1.91 (3H, br.d), 1.86 (3H, br.d), 1.78-1.50 (24H, m), 1.43-0.80 (36H, m), 1.03 (9H, d, *J* = 6.8 Hz), 0.97 (9H, d, *J* = 7.2 Hz); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 156.7 (C(=O)CF_3), 136.5, 135.8, 132.6, 132.3, 132.2, 132.1, 130.4, 129.1, 129.1, 128.9, 128.5, 127.8, 127.6, 125.4, 125.3, 123.9 123.6, 116.4 (CF_3), 116.3 (CF_3), 98.9, 98.3, 92.9, 90.2, 89.7, 88.5, 60.0, 59.8, 40.2, 40.1, 30.6, 30.5, 29.8, 29.7, 26.1, 26.0, 25.9, 25.8, 25.8, 25.6, 16.2, 16.1; FD-LRMS *m*/*z* 2304.9 (M^+, 65%), 2305.9 ([M+1]$^+$, 100), 2306.9 ([M+2]$^+$, 79), 2307.9 ([M+3]$^+$, 43), 2308.9 ([M+4]$^+$, 19); FD-HRMS Found: 2304.94488, Calc. for $C_{138}H_{126}F_{18}N_6O_6$: 2304.94514.

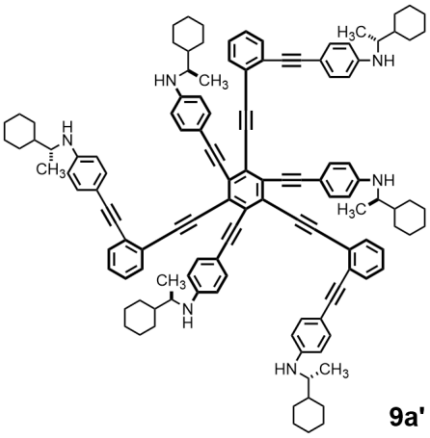


9b: mp 105-107 °C; IR (KBr) ν_{max}/cm^{-1} 2960, 2934, 2874, 2205, 1698, 1510, 1427, 1209, 1149, 845, 756; ^{1}H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.71 (3H, br.d), 7.67 (3H, br.d), 7.48-7.40 (6H, m), 7.46 (6H, d, *J* = 8.4 Hz), 7.40 (6H, d, *J* = 8.4 Hz), 7.01 (6H, d, *J* = 8.4 Hz), 6.99 (6H, d, *J* = 8.4 Hz), 3.65 (6H, t, *J* = 7.2 Hz), 3.59 (6H, t, *J* = 7.2 Hz), 1.50-1.37 (12H, m), 1.34-1.17 (12H, m), 0.89 (9H, t, *J* = 7.2 Hz), 0.81 (9H, t, *J* = 7.2 Hz); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 156.3 (C(=O)CF_3), 156.3 (C(=O)CF_3), 139.3, 138.7, 133.0, 132.7, 132.2, 132.1, 129.2, 128.5, 128.1, 128.0, 127.8, 127.6, 125.3, 125.3, 123.7 123.4, 116.3 (CF_3), 116.2 (CF_3), 98.9, 98.3, 92.8, 90.2, 89.6, 88.4, 51.5, 51.5, 28.8, 28.8, 19.7, 19.7, 13.6, 13.5; FD-LRMS *m*/*z* 1980.6 (M^+,

78%), 1981.6 ([M+1]$^+$, 100), 1982.6 ([M+2]$^+$, 65), 1983.6 ([M+3]$^+$, 31), 1984.6 ([M+4]$^+$, 11); FD-HRMS Found: 1980.66542, Calc. for $C_{114}H_{90}F_{18}N_6O_6$: 1980.66344.

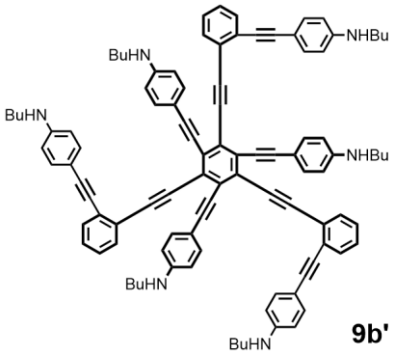
(d) Preparation of **3a** and **3b**

i) To an ice-cooled solution of **9a**/**b** and 60% NaH in THF was added MeOH. The mixture was stirred at room temperature for 25 min and quenched with water. After extraction with ethyl acetate, the organic layer was washed with water and brine, dried over magnesium sulfate, and then purified by column chromatography on Al_2O_3 (dichloromethane/hexane) to give **9'** (**a**: 128 mg, 85% and **b**: 105 mg, 99%) as a yellow amorphous solid, which was immediately subjected to the next reaction.



9a'

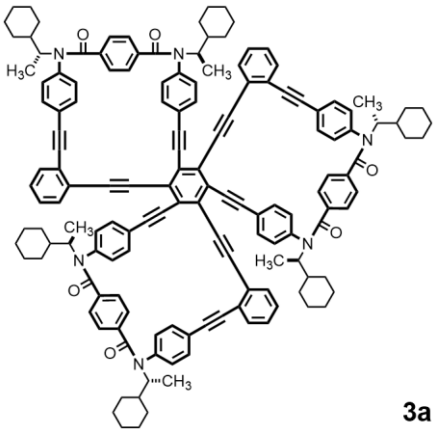
ii) To a solution of **9a'**/**9b'** in toluene containing a small amount of Et_3N was added terephthaloyl chloride, the mixture was stirred (**a**) at 100 ºC for several hours while adding several portions of terephthaloyl chloride with an interval of 30 min or (**b**) at 85 ºC for 45 min. After extraction with (**a**) ethyl acetate or (**b**) dichloromethane, the organic layer was washed with aq. 0.1M NaOH and brine, dried over magnesium sulfate, and then purified by column chromatography on Al_2O_3/SiO_2 (ethyl acetate/dichloromethane), followed by GPC (chloroform) to give **3** (**a**: 42 mg, 27% and **b**: 30 mg, 42%) as a yellow solid. Each analytical sample was obtained as a yellow solid by further purification through HPLC with a standard normal-phase column (ethyl acetate/dichloromethane for **3a** and tetrahydrofuran/chloroform for **3b**).



9b'

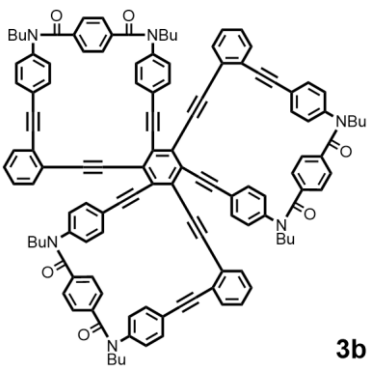
	9a/**b**	60% NaH in oil	MeOH	THF	terephthaloyl chloride	Et_3N	Toluene
3a	200 mg (0.0867 mmol)	420 mg (10.5 mmol)	0.5 mL	5 mL	50 mg×7 (1.7 mmol)	0.3 mL	74 mL
3b	150 mg (0.0757 mmol)	61 mg (1.5 mmol)	0.5 mL	5 mL	27 mg (0.13 mmol)	0.2 mL	40 mL

3a: mp >300 °C; $[\alpha]_D^{23}$ = –596 (*c* = 0.181, chloroform); IR (KBr) ν_{max}/cm^{-1} 2929, 2853, 2202, 1655, 1600, 1510, 1384, 1318, 834, 757; ^{1}H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.78-7.76 (6H, m), 7.6-7.3 (18H, br.m), 7.2-6.9 (6H, br.), 6.94-6.89 (12H, br.d×2), 6.5 (6H, br.), 4.6-4.3 (6H, br.m), 2.2-2.0 (6H, br.m), 1.9-1.4 (30H, br.m), 1.4-0.8 (48H, br.m); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 170.4, 141.6, 138.1, 137.8, 132.9, 132.6, 132.3, 129.2, 128.4, 128.0, 127.5, 127.2, 127.1, 125.2, 125.1, 121.3, 121.2, 99.1, 97.9, 93.1, 90.5, 89.5, 88.4, 30.8, 30.2, 29.7, 26.2, 26.2, 26.1, 26.0, 26.0, 16.7; FD-LRMS *m*/*z* 2119.0 (M$^+$, 49%), 2120.0 ([M+1]$^+$, 93), 2121.0 ([M+2]$^+$, 100), 2122.0 ([M+3]$^+$, 67), 2123.0 ([M+4]$^+$, 34); FD-HRMS



3a

Found: 2119.06902, Calc. for $C_{150}H_{138}N_6O_6$: 2119.06779; UV $\lambda_{max}(CH_2Cl_2)$/nm (log ε) 387 (shoulder 4.71), 364 (4.83), 316 (sh. 4.80), 294 (4.84), 270 (s. 4.76); CD $\lambda(CH_2Cl_2)$/nm ($\Delta\varepsilon$ at 293 K) 394 (–6.4), 387 (–5.0), 377 (–6.2), 355 (–0.8), 336 (–12), 314 (–100), 292 (+35), 269 (–39).



3b

3b: mp >300 °C; IR (KBr) ν_{max}/cm^{-1} 2929, 2870, 2201, 1656, 1600, 1510, 1383, 1295, 1122, 836, 756; ^{1}H NMR δ_H(400 MHz; $CDCl_3$; Me_4Si)/ppm 7.76-7.73 (6H, m), 7.52-7.44 (6H, m), 7.41 (6H, d, J = 8.4 Hz), 7.36 (6H, d, J = 8.8 Hz), 6.98 (6H, d, J = 8.4 Hz), 6.95 (6H, d, J = 8.4 Hz), 6.83 (6H, br.d), 6.72 (6H, br.d), 3.80 (12H, br.s), 1.53-1.43 (12H, m), 1.36-1.22 (12H, m), 0.87 (9H, t, J = 7.2 Hz), 0.78 (9H, t, J = 7.2 Hz); ^{13}C NMR δ_C(100 MHz; $CDCl_3$)/ppm 169.9, 169.9, 143.5, 143.0, 137.5, 137.2, 133.0, 132.9, 132.6, 132.6, 129.2, 128.6, 128.4, 127.9, 127.5, 127.3, 127.3, 125.1, 125.0, 121.1, 99.0, 97.9, 92.9, 90.5, 89.3, 88.2, 49.3, 49.1, 29.7, 20.1, 20.0, 13.8, 13.7; FD-LRMS m/z 1794.7 (M^+, 69%), 1795.7 ($[M+1]^+$, 100), 1796.7 ($[M+2]^+$, 75), 1797.7 ($[M+3]^+$, 38), 1798.7 ($[M+4]^+$, 15); FD-HRMS Found: 1794.78409, Calc. for $C_{126}H_{102}N_6O_6$: 1794.78609; UV $\lambda_{max}(CH_2Cl_2)$/nm (log ε) 385 (sh. 4.78), 364 (4.88), 320 (sh. 4.84), 295 (4.88), 265 (sh. 4.76).

References and note

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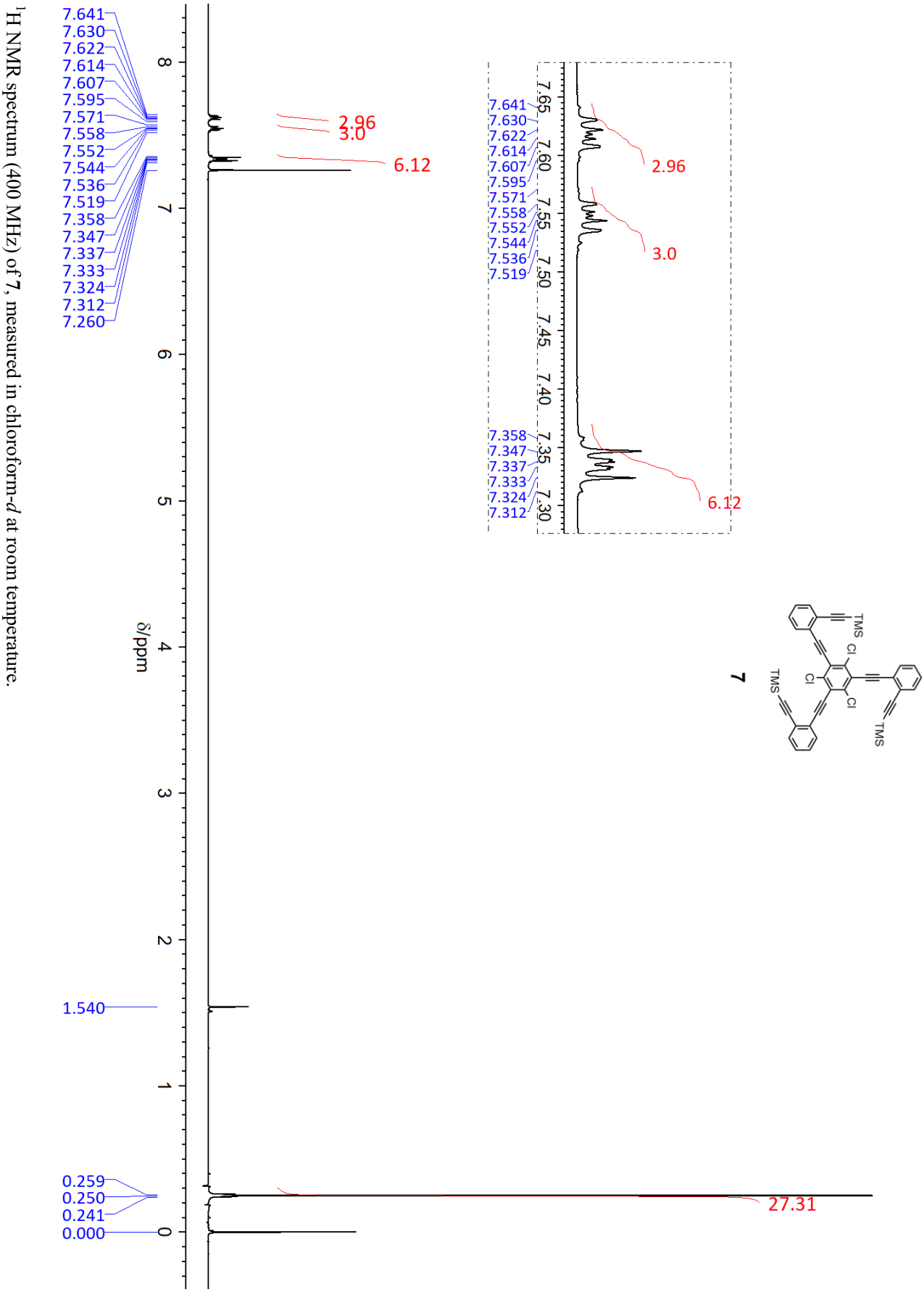
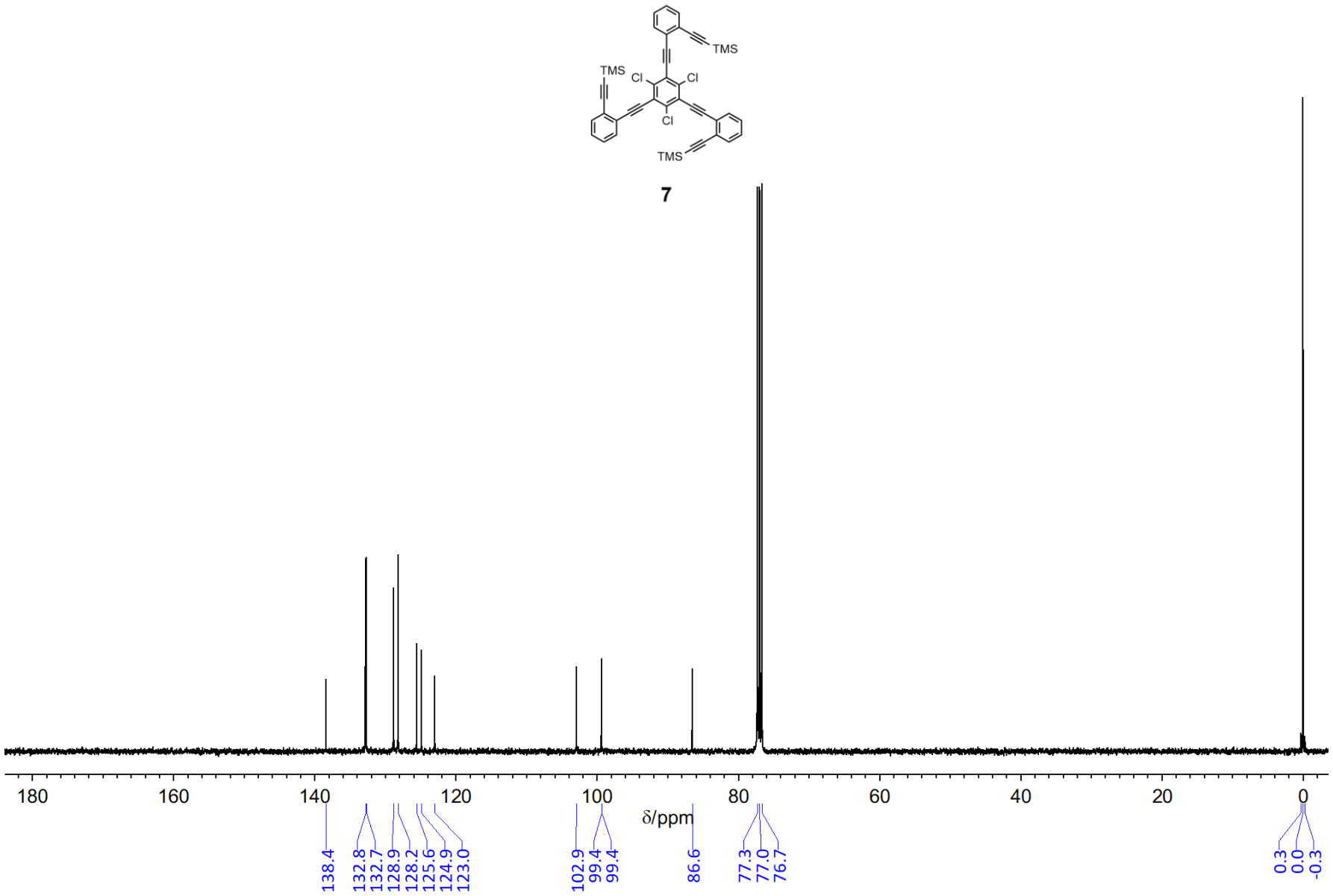
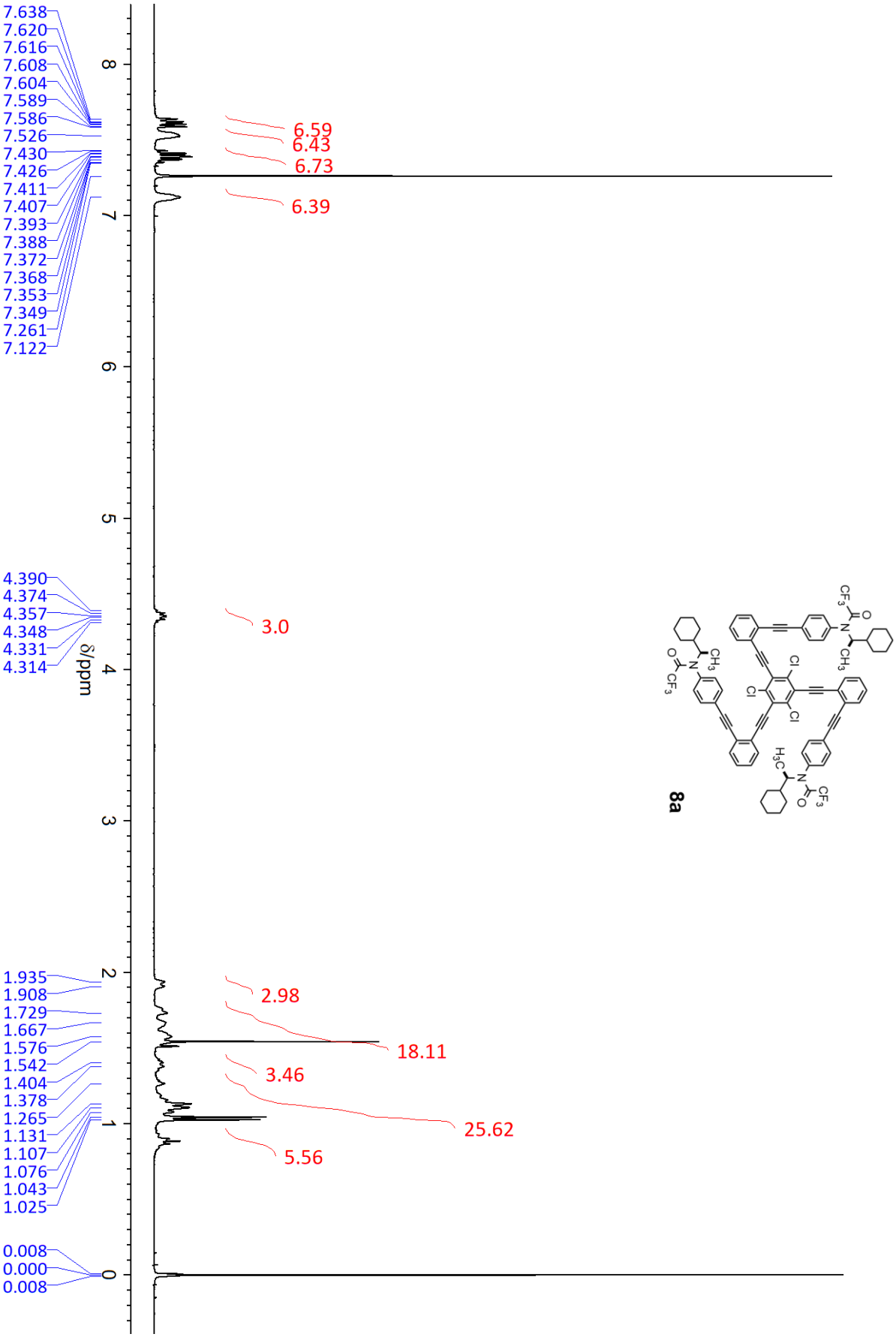


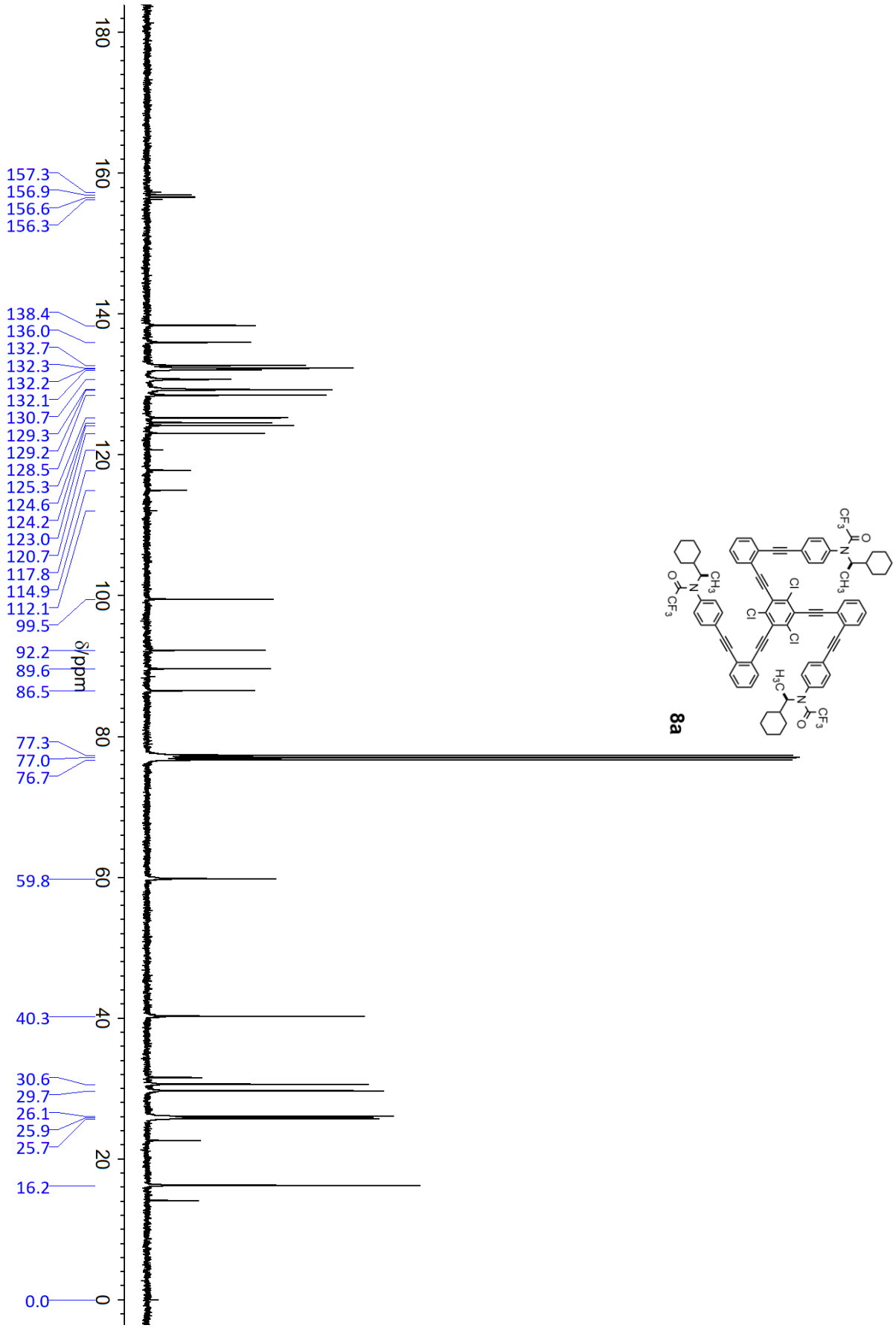
^{1}H NMR spectrum (400 MHz) of **7**, measured in chloroform-*d* at room temperature.



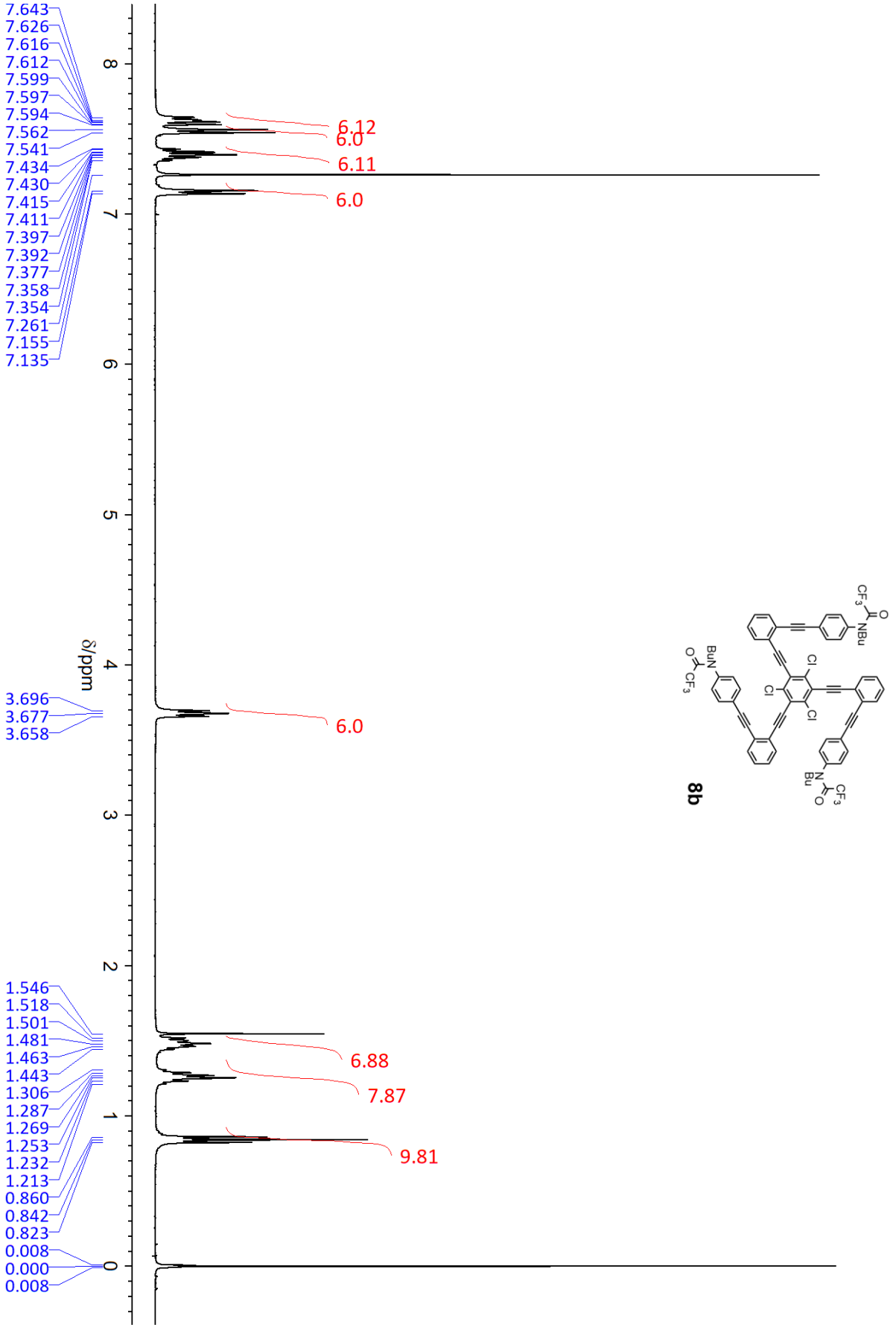
^{13}C NMR spectrum (100 MHz) of **7**, measured in chloroform-*d* at room temperature.

^{1}H NMR spectrum (400 MHz) of **8a**, cont. residual hexane, measured in chloroform-*d* at room temperature.

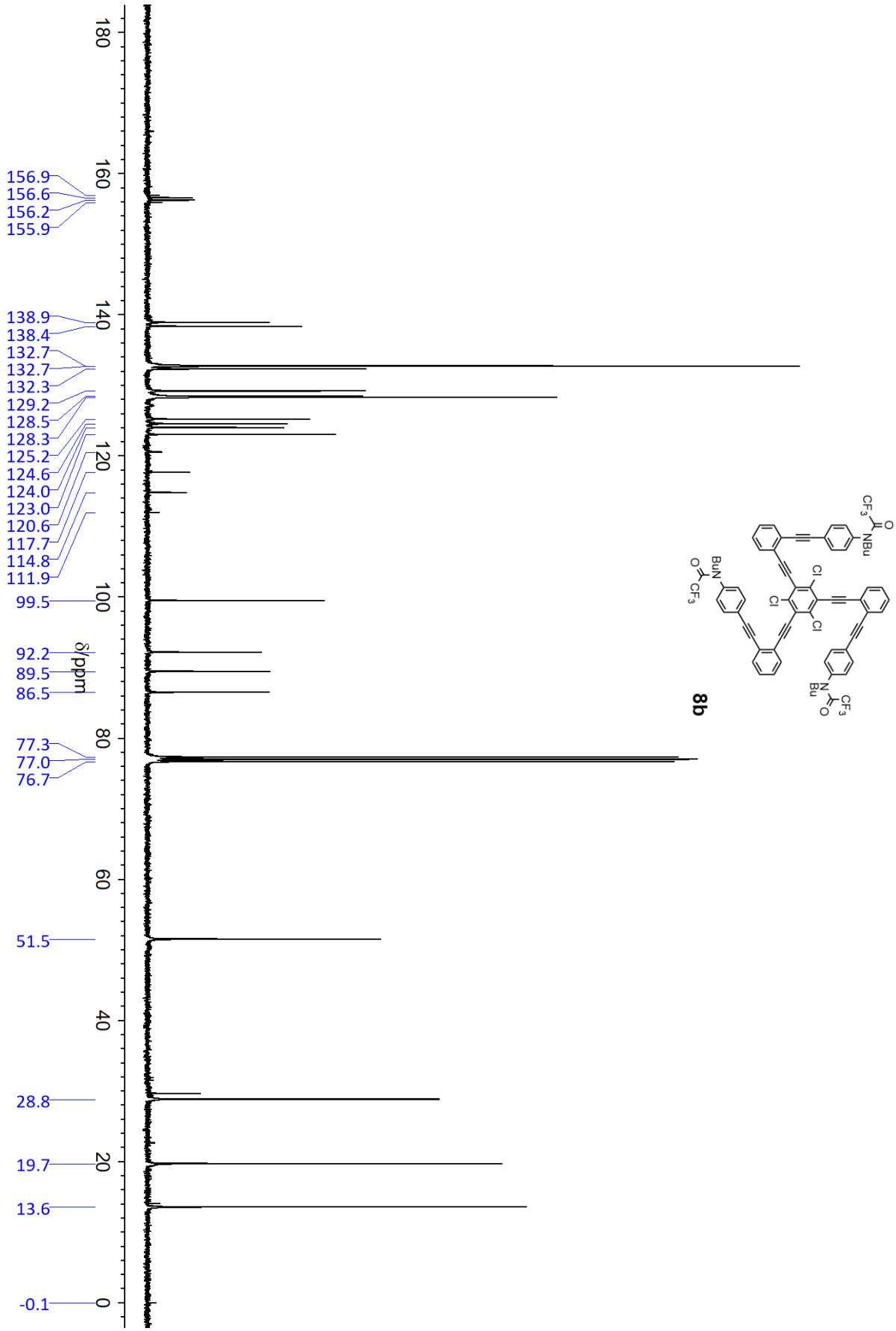




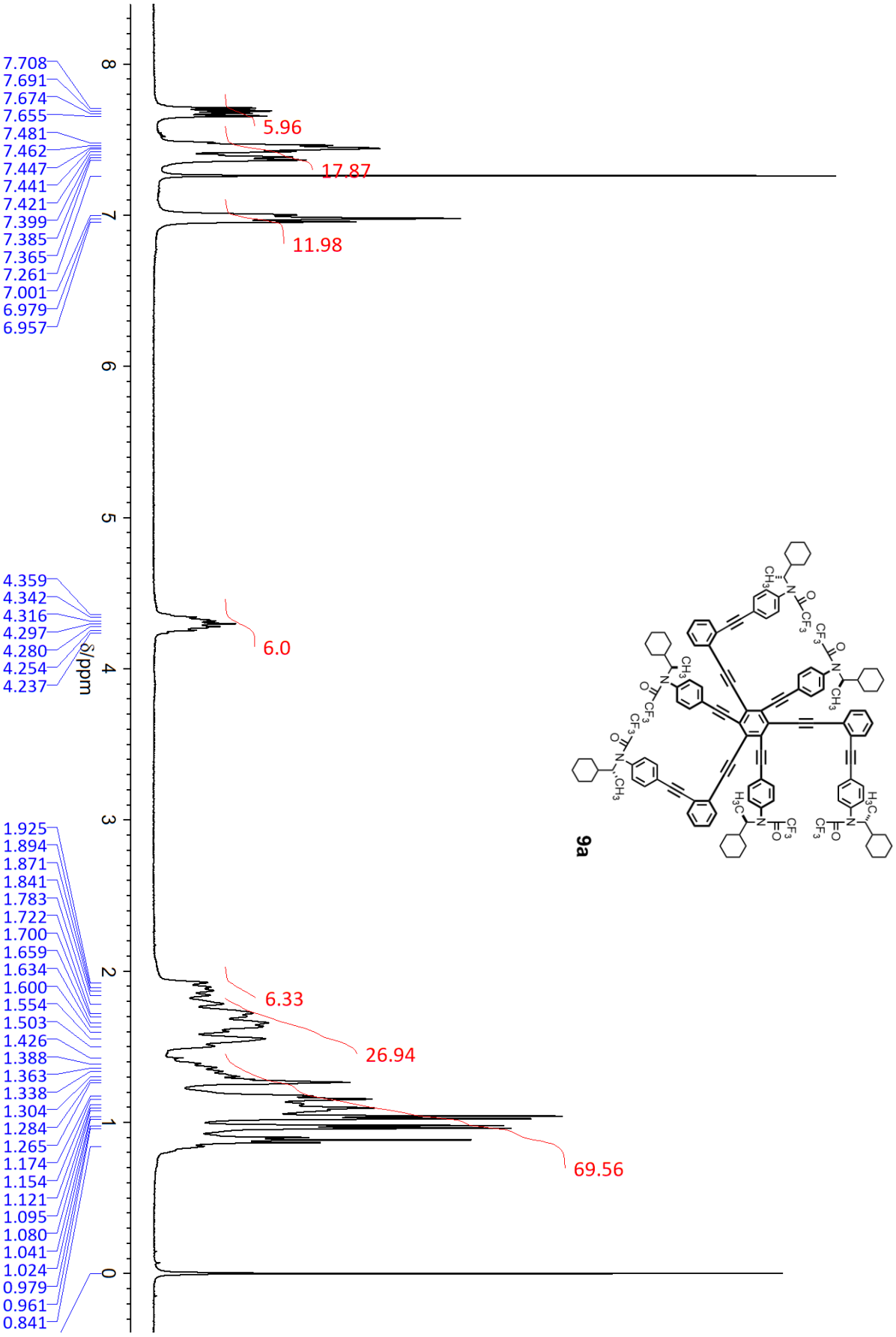
^{13}C NMR spectrum (100 MHz) of **8a**, cont. residual hexane, measured in chloroform-*d* at room temperature.



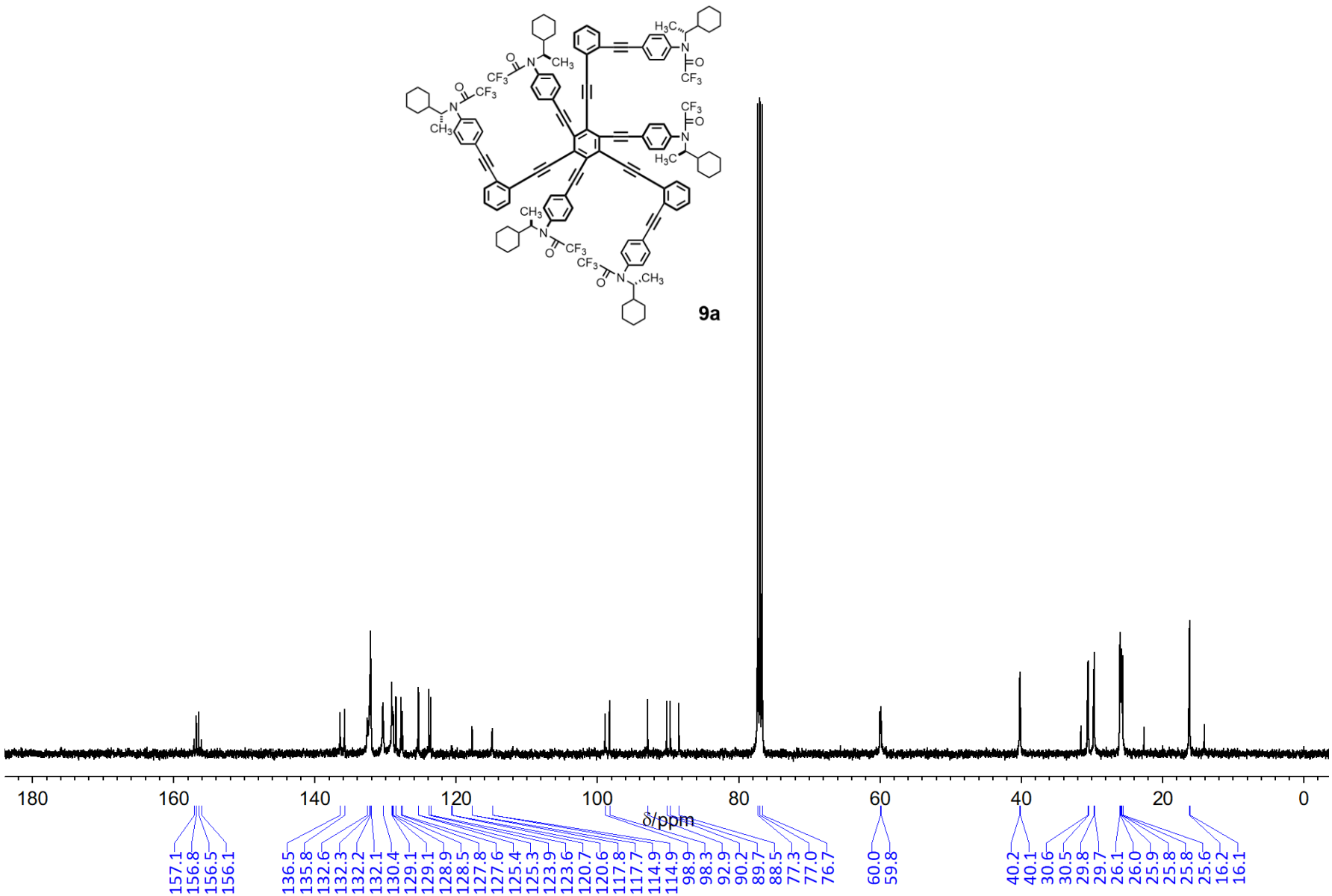
^{1}H NMR spectrum (400 MHz) of **8b**, cont. residual hexane, measured in chloroform-*d* at room temperature.



^{13}C NMR spectrum (100 MHz) of **8b**, cont. residual hexane, measured in chloroform-*d* at room temperature.

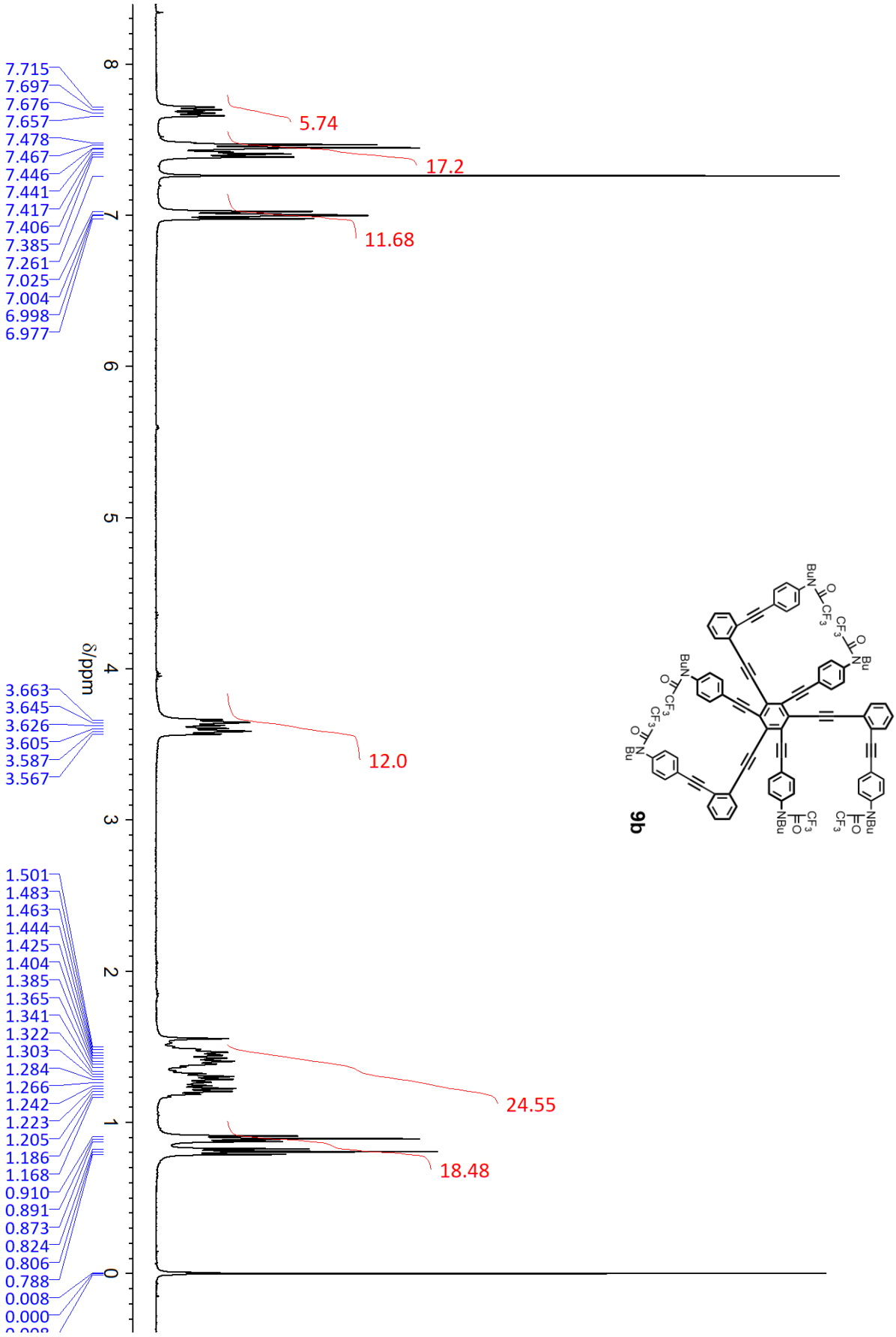


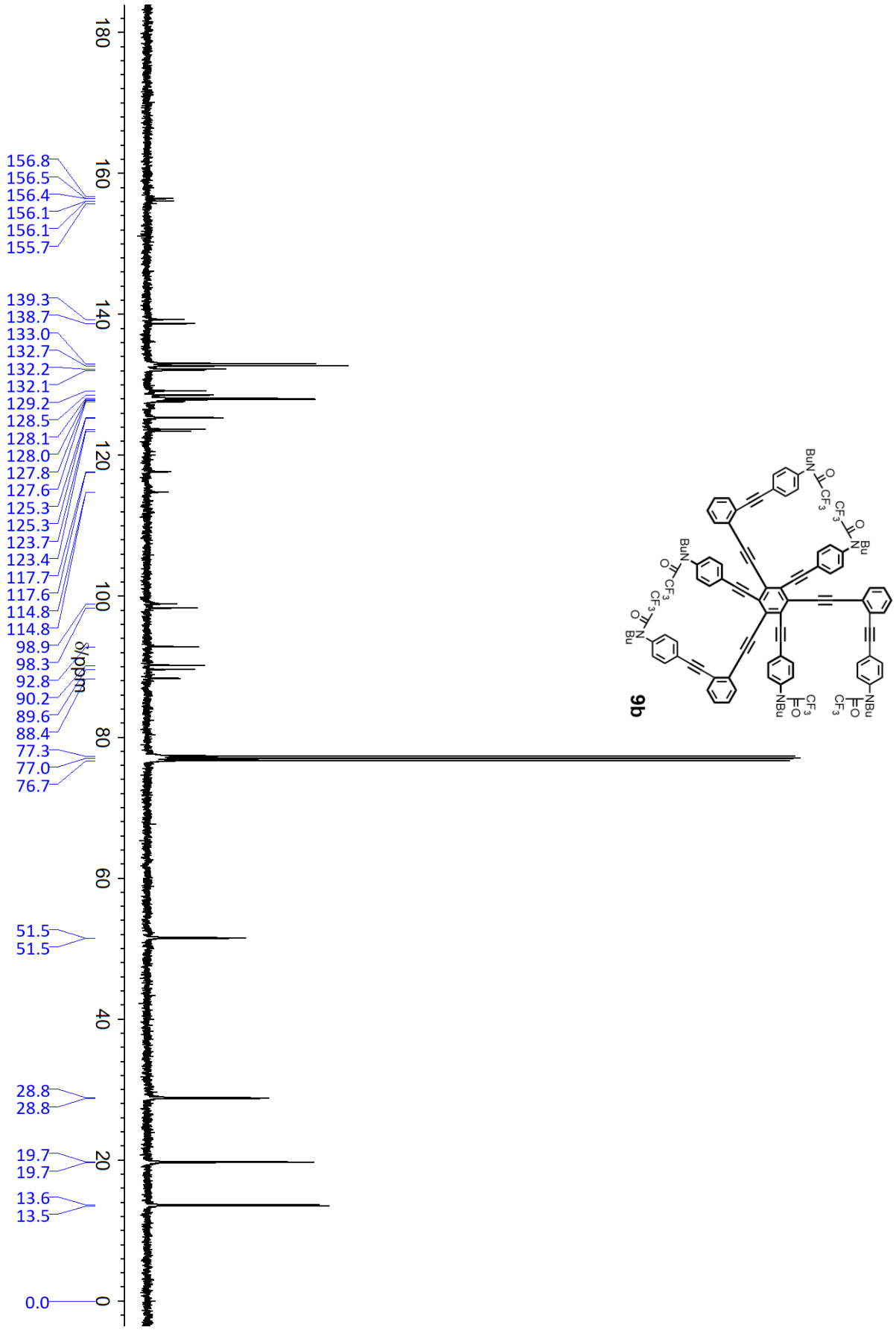
^{1}H NMR spectrum (400 MHz) of **9a**, cont. residual hexane, measured in chloroform-*d* at room temperature.



^{13}C NMR spectrum (100 MHz) of **9a**, cont. residual hexane, measured in chloroform-*d* at room temperature.

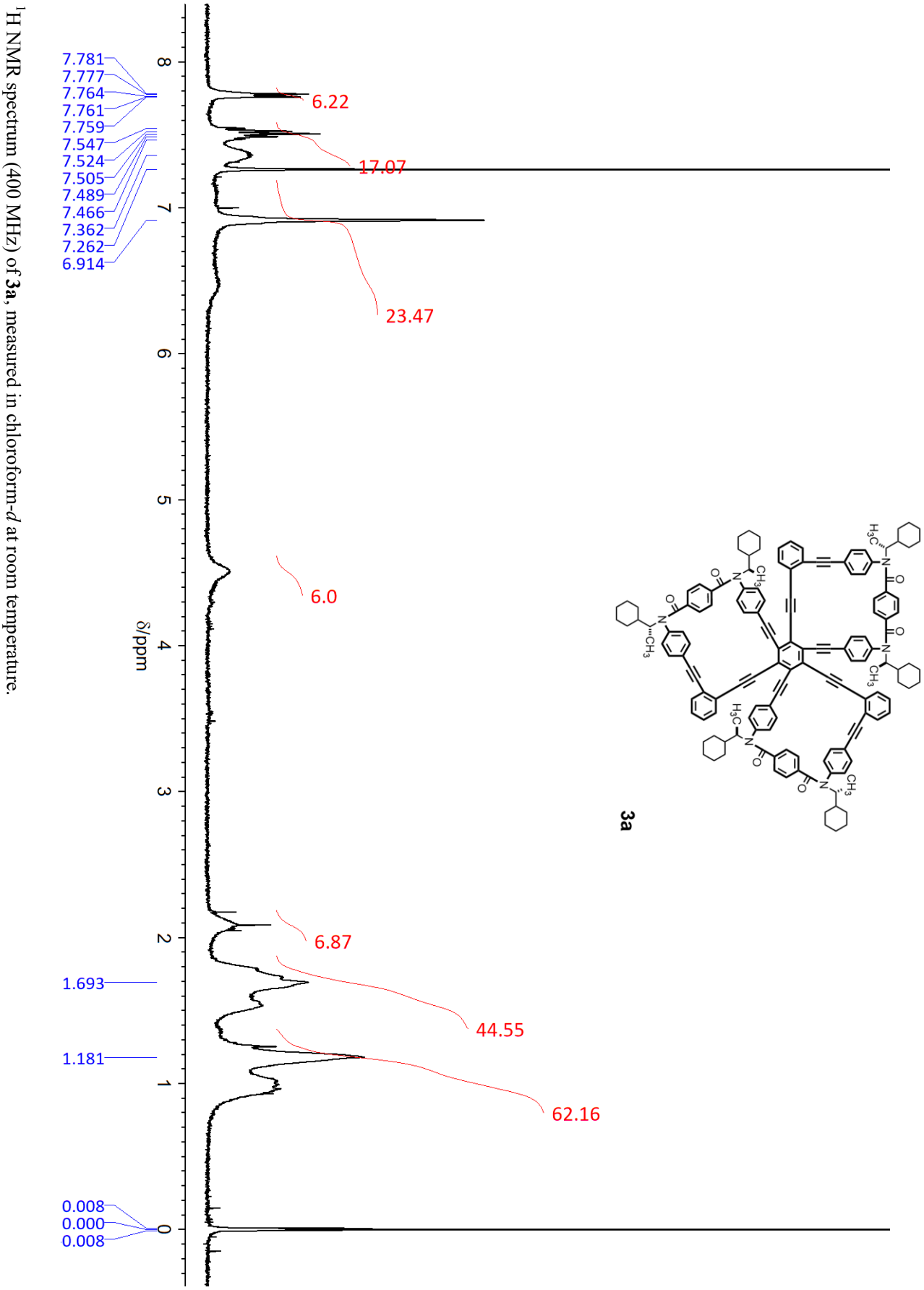
^{1}H NMR spectrum (400 MHz) of **9b**, measured in chloroform-*d* at room temperature.

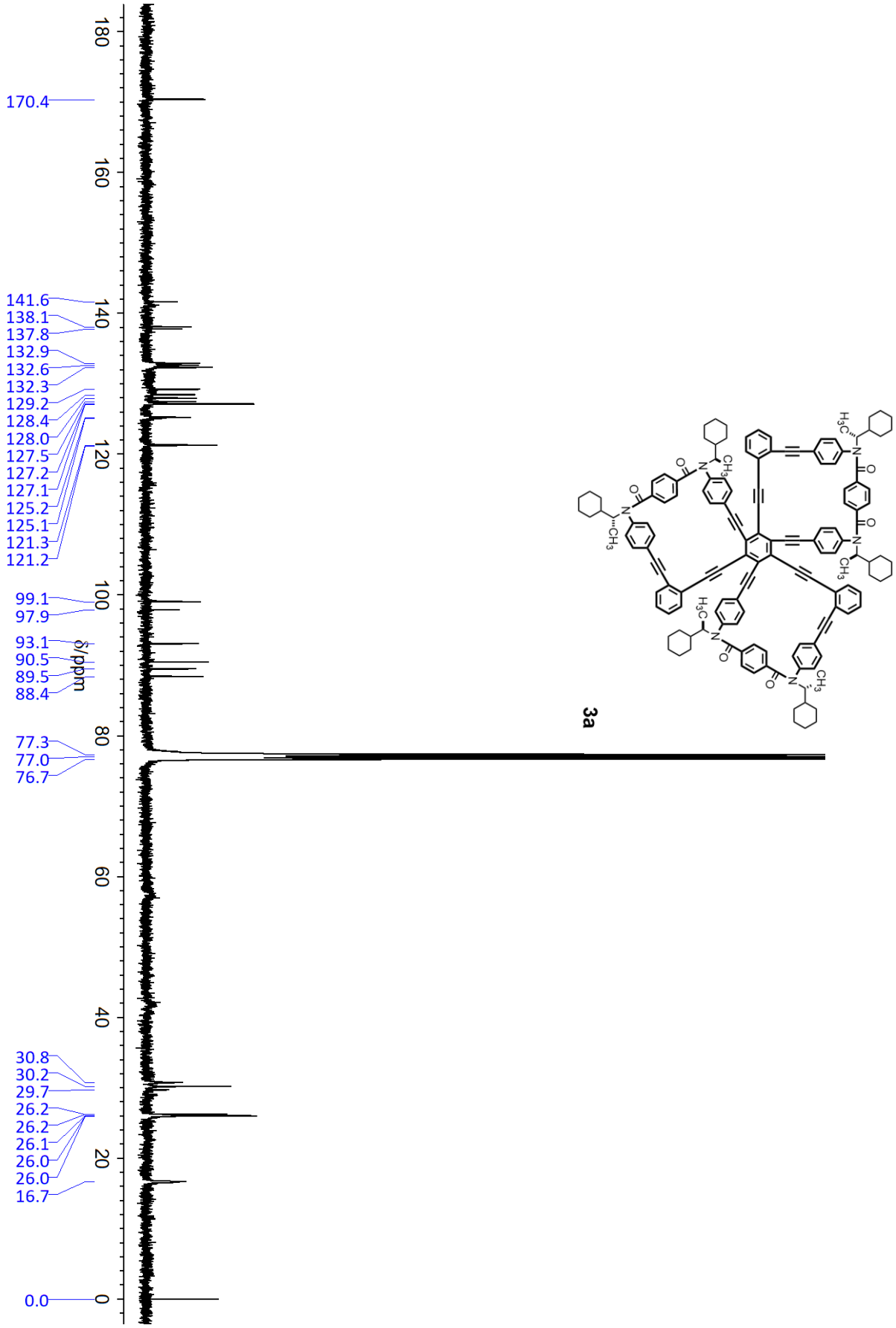




^{13}C NMR spectrum (100 MHz) of **9b**, measured in chloroform-*d* at room temperature.

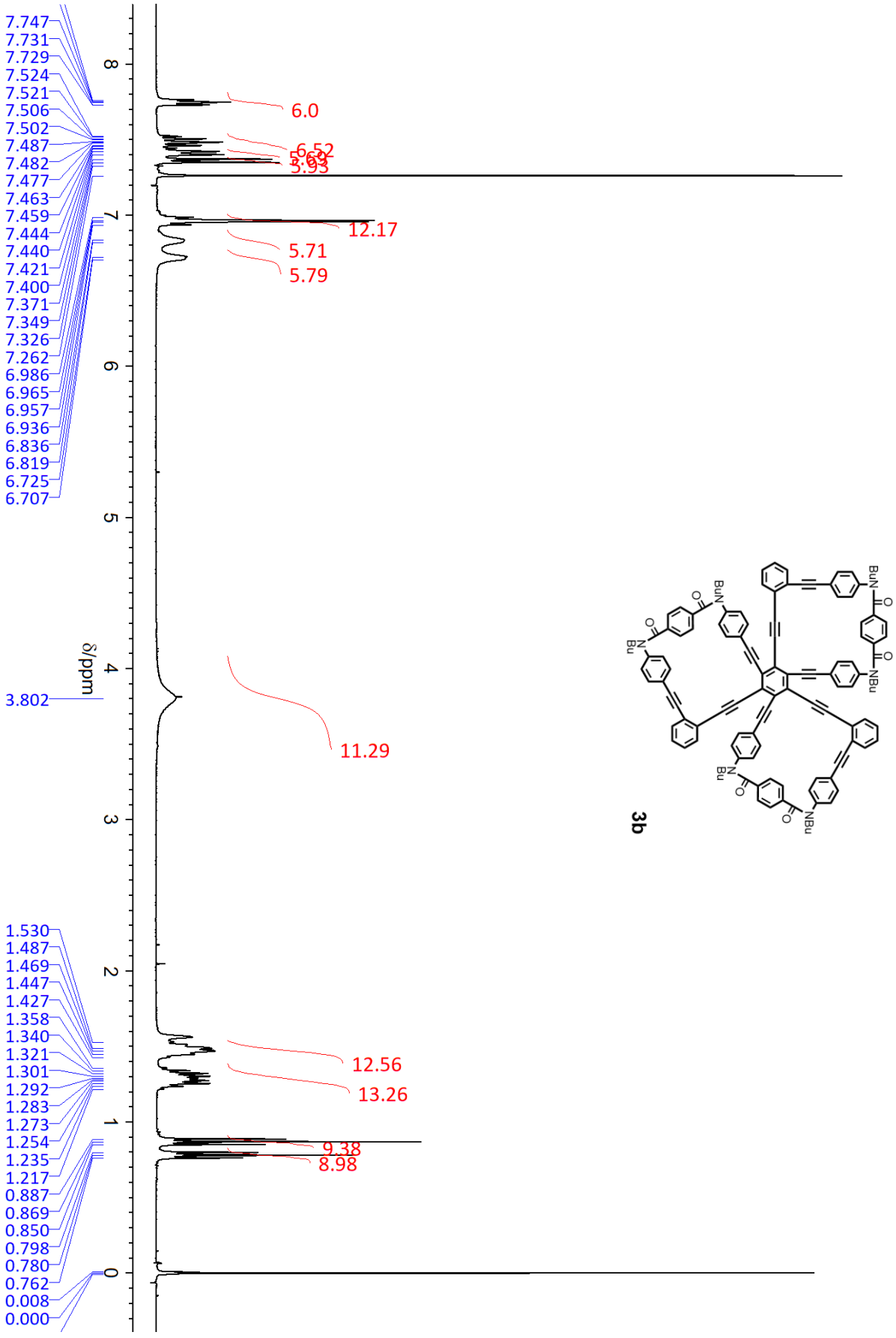
^{1}H NMR spectrum (400 MHz) of **3a**, measured in chloroform-*d* at room temperature.

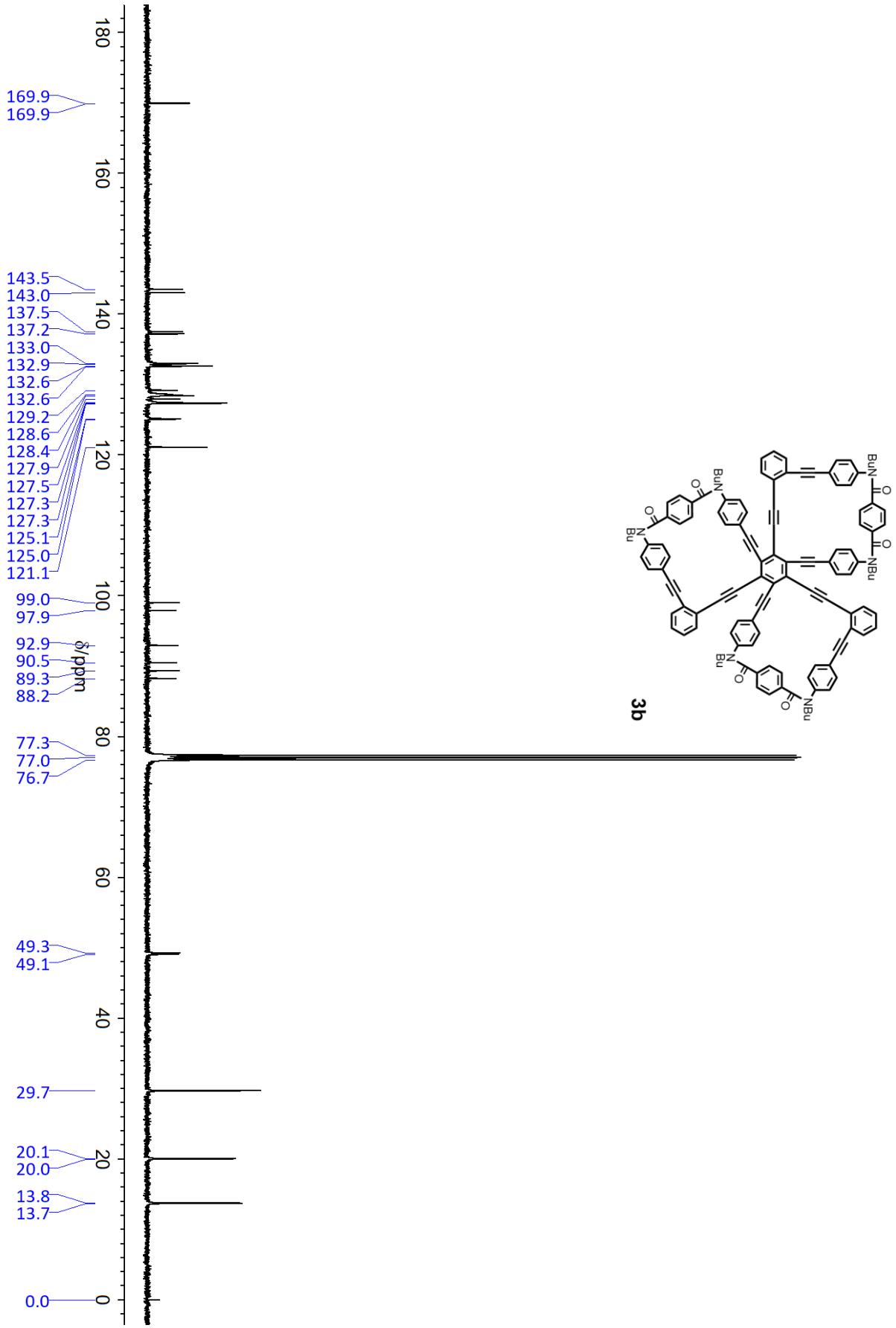




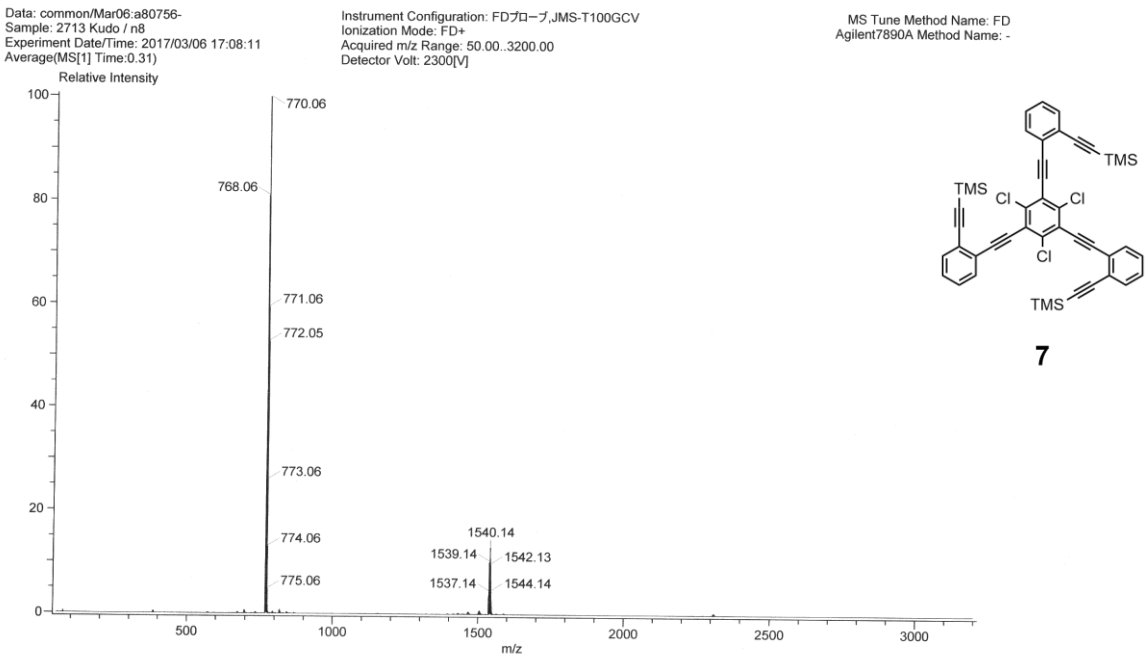
^{13}C NMR spectrum (100 MHz) of **3a**, measured in chloroform-*d* at room temperature.

^{1}H NMR spectrum (400 MHz) of **3b**, measured in chloroform-*d* at room temperature.

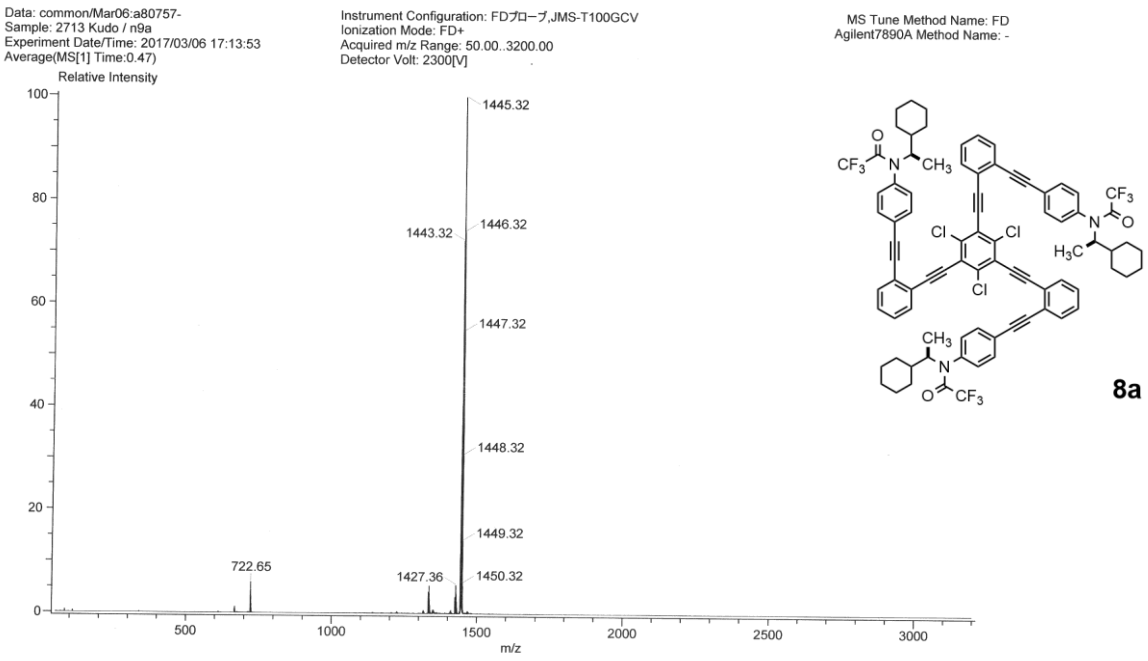




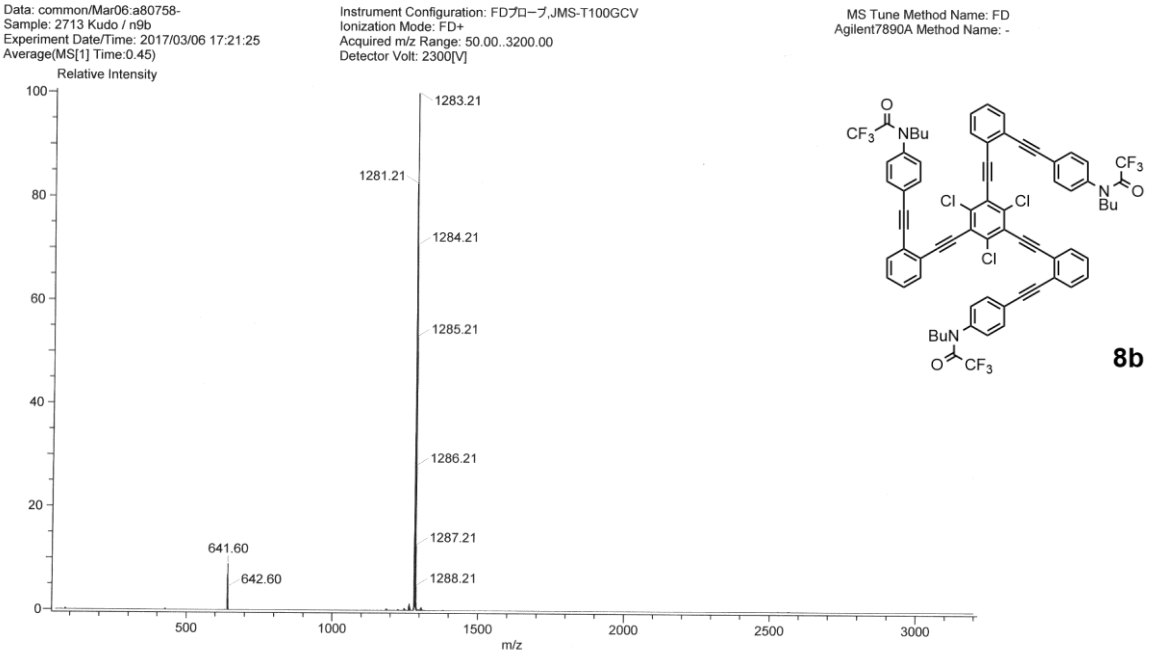
^{13}C NMR spectrum (100 MHz) of **3b**, measured in chloroform-*d* at room temperature.



LR MS spectrum (FD) of **7**.



LR MS spectrum (FD) of **8a**.



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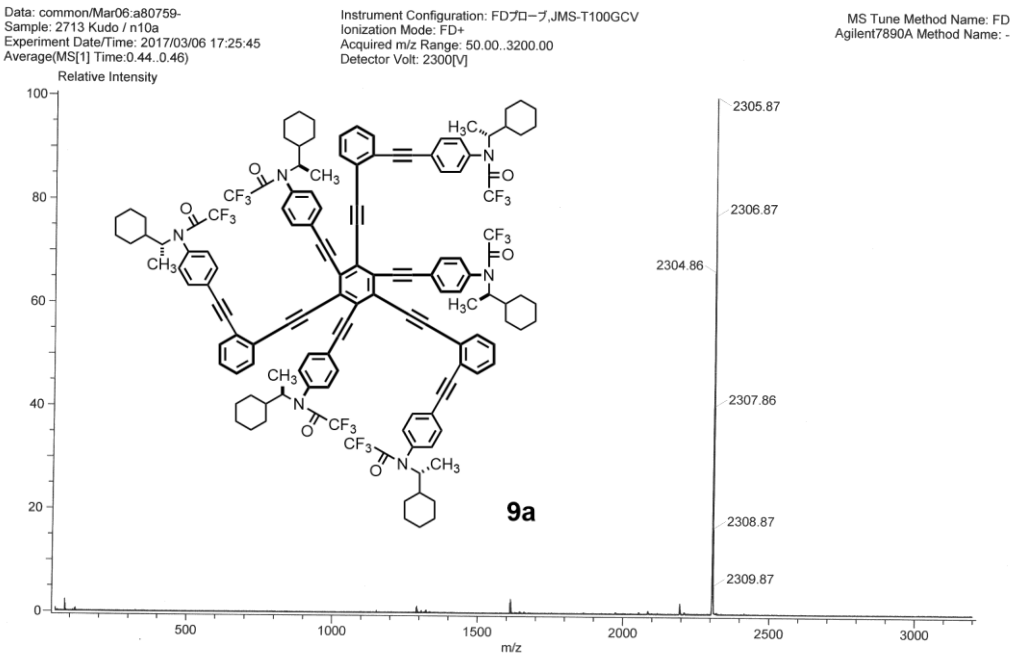
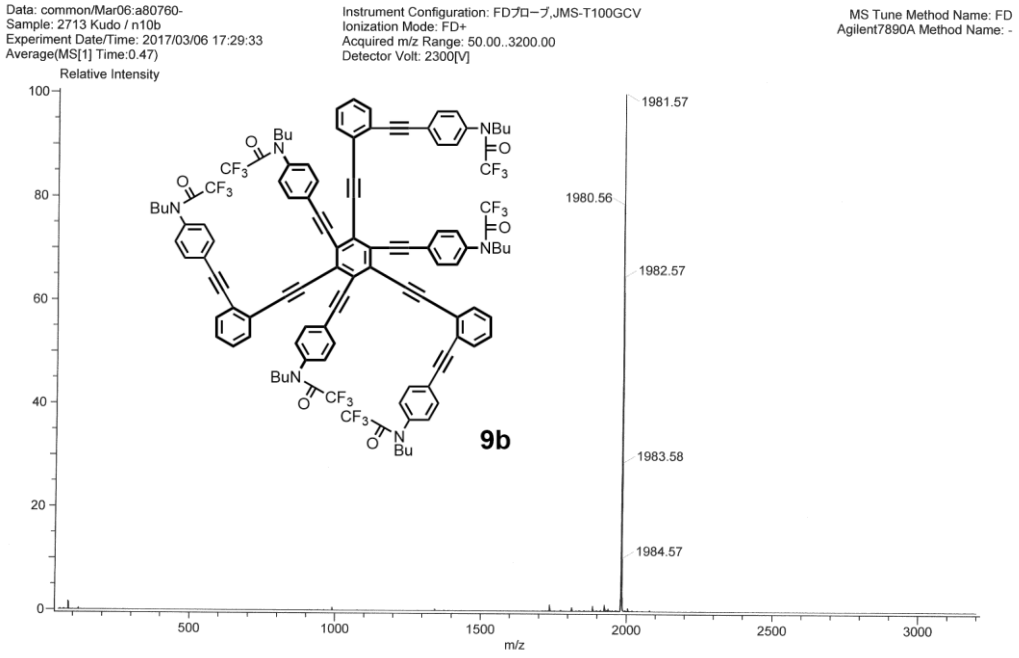


LR MS spectrum (FD) of **9a**.



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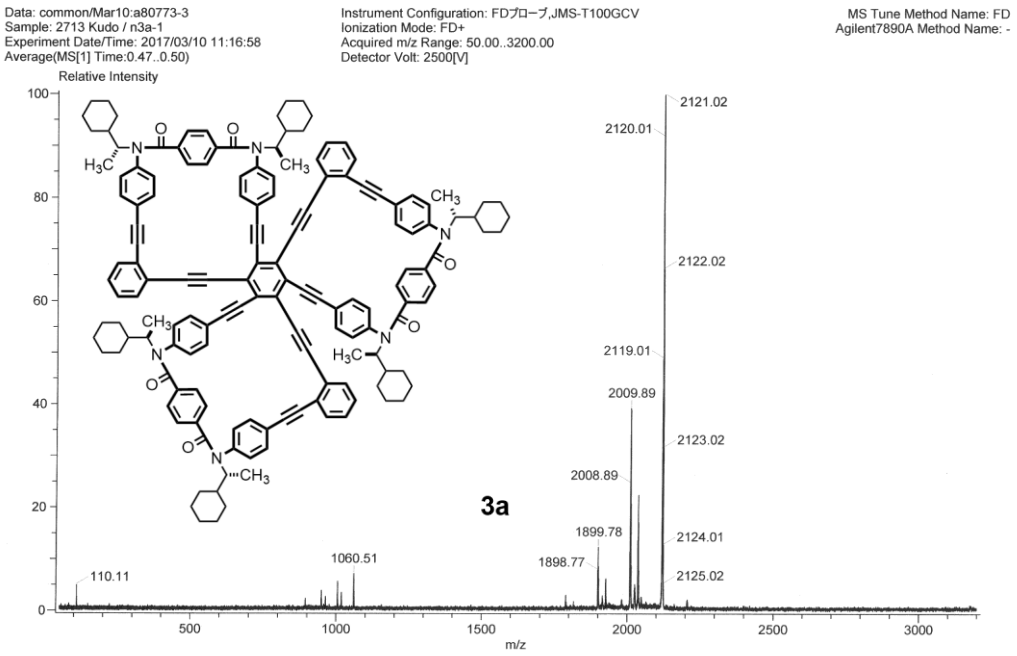
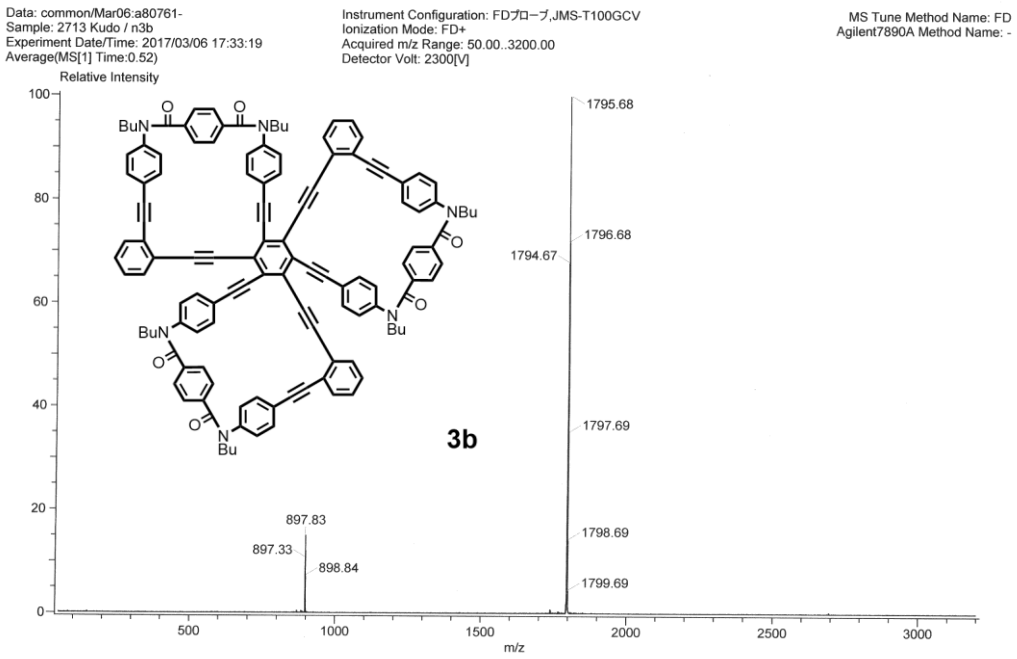


LR MS spectrum (FD) of **3a**.



LR MS spectrum (FD) of **3b**.