Electronic Supplementary Material (ESI) for Dalton Transactions.

Luminescent Liquid Crystalline Hybrid Materials by Embedding Octahedral Molybdenum Cluster Anions with Soft Organic Shells Derived from Tribenzo[18]crown-6

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Supporting Information

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Experimental data of triphenylene derivatives [Tri(n)]

General procedure for the synthesis of the triphenylene crown ethers **Tri(n)** *by oxidative cyclization (GP S1)*

According to Wöhrle,[1] the respective *o*-terphenyl crown ether **Ter(n)** (41.6 µmol, 1 eq.) was dissolved in abs. CH_2Cl_2 (50 mL) under an N_2 atmosphere and cooled to 0°C. A solution of $FeCl_3$ (162 mg, 998 µmol, 24 eq.) in $MeNO_2$ (4 mL) was added dropwise while a stream of nitrogen was bubbled through the solution. After stirring for 15 min at 0°C, the reaction was quenched by the addition of methanol (50 mL). Water (50 mL) was added and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 × 15 mL), the combined organic layers washed with water (2 × 20 mL), dried ($MgSO_4$) and the solvent removed under reduced pressure. The crude product was purified by recrystallization from acetone and subsequent washing with CH_2Cl_2 (5–10 mL) to yield the desired triphenylene **Tri(n)** as brown solid.

Tris(5,6,9,10-tetrakis(octyloxy)triphenylene)[18]crown-6 [**Tri(8)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(8)** (100 mg, 41.6 µmol) and $FeCl_3$ (162 mg, 998 µmol). Purification by recrystallization from acetone (35 mL) and subsequent washing with cold CH_2Cl_2 (5 mL). Yield: 52 % (51.8 mg, 21.6 µmol), brown solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.86–0.93 (m, 36H, CH_3), 1.24–1.63 (m, 120H, CH_2), 1.84– 1.98 (m, 24H, OCH_2CH_2), 4.10 (t, J = 6.6 Hz, 12H, OCH_2), 4.21 (t, J = 6.6 Hz, 12H, OCH_2), 4.70 (s, 12H, a-H), 7.71 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.91 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.2, 29.4, 29.4, 29.5, 29.5, 31.9 (CH_2), 69.3 (C-a), 69.5, 69.7 (OCH_2), 106.9, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.6, 124.4 (C-4, C-1′, C-6′), 148.6, 148.9, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 2919 (s), 2850 (s), 1617 (w), 1514 (s), 1466 (m), 1432 (s), 1386 (m), 1259 (vs), 1169 (vs), 1067 (m), 1027 (m), 941 (w), 903 (w), 836 (m), 798 (w), 723 (w), 616 (w), 604 (w), 452 (w) cm^{-1}. MS (MALDI-TOF): *m/z* calcd. for [$C_{156}H_{234}O_{18}^+$ (M^+)]: 2396.7; found: 2391.2. CHN analysis calcd. (%) for $C_{156}H_{234}O_{18}$ (2397.57): C 78.15, H 9.84; found: C 77.93, H 9.91.

Tris(5,6,9,10-tetrakis(decyloxy)triphenylene)[18]crown-6 [**Tri(10)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(10)** (100 mg, 36.5 µmol) and $FeCl_3$ (142 mg, 876 µmol). Purification by recrystallization from acetone (35 mL) and subsequent washing with cold CH_2Cl_2 (5 mL). Yield: 41 % (40.7 mg, 14.9 µmol), brown solid. ^{1}H NMR (700 MHz, $CDCl_3$): δ = 0.83–0.92 (m, 36H, CH_3), 1.20–1.61 (m, 168H, CH_2), 1.84–1.98 (m, 24H, OCH_2CH_2), 4.09 (t, J = 6.6 Hz, 12H, OCH_2), 4.21 (t, J = 6.6 Hz, 12H, OCH_2), 4.70 (s,

12H, a-H), 7.70 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.90 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (176 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.3, 29.4, 29.5, 29.6, 29.6, 29.7, 29.7, 29.8, 32.0 (CH_2), 69.3 (C-a), 69.5, 69.7 (OCH_2), 107.0, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.6, 124.4 (C-4, C-1′, C-6′), 148.6, 149.0, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 2917 (s), 2849 (s), 1617 (w), 1512 (s), 1466 (m), 1432 (s), 1387 (m), 1259 (vs), 1169 (vs), 1069 (m), 1028 (m), 925 (w), 836 (m), 797 (w), 722 (w), 604 (w) cm^{-1}. MS (MALDI-TOF): *m/z* calcd. for [$C_{180}H_{282}O_{18}^+$ (M^+)]: 2733.1; found: 2730.6. HRMS (ESI): *m/z* calcd. for [$C_{180}H_{282}O_{18}Na]^+$: 2756.1077, found: 2756.0553. CHN analysis calcd. (%) for $C_{180}H_{282}O_{18}$ (2397.57): C 79.07, H 10.40; found: C 78.59, H 10.07.

Tris(5,6,9,10-tetrakis(undecyloxy)triphenylene)[18]crown-6 [**Tri(11)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(11)** (60.0 mg, 20.6 µmol) and $FeCl_3$ (80.3 mg, 495 µmol). Purification by washing with cold CH_2Cl_2 (5 mL). Yield: 67 % (40.0 mg, 13.8 µmol), brown solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.85–0.90 (m, 36H, CH_3), 1.21–1.45 (m, 172H, CH_2), 1.47–1.54 (m, 12H, $OCH_2CH_2CH_2$) 1.84–1.97 (m, 24H, OCH_2CH_2), 4.09 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.21 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.69 (s, 12H, a-H), 7.70 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.90 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.3, 29.4, 29.5, 29.6, 29.6, 29.7, 32.0 (CH_2), 69.3 (C-a), 69.5, 69.7 (OCH_2), 106.9, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.7, 124.4 (C-4, C-1′, C-6′), 148.6, 149.0, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 918 (s), 2849 (m), 1617 (w), 1516 (m), 1467 (w), 1433 (s), 1386 (w), 1260 (vs), 1170 (vs), 1070 (m), 1030 (m), 1064 (w), 934 (w), 836 (m), 721 (w) cm^{-1}. MS (MALDI-TOF): *m/z* calcd. for [$C_{192}H_{306}O_{18}^+$ (M^+)]: 2902.3; found: 2899.4. CHN analysis calcd. (%) for $C_{192}H_{306}O_{18}$ (2902.54): C 79.45, H 10.63; found: C 79.28, H 10.47.

Tris(5,6,9,10-tetrakis(dodecyloxy)triphenylene)[18]crown-6 [**Tri(12)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(12)** (60.0 mg, 19.5 µmol) and $FeCl_3$ (75.9 mg, 468 µmol). Purification by washing with cold CH_2Cl_2 (5 mL). Yield: 44 % (26.5 mg, 8.63 µmol), brown solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.85–0.89 (m, 36H, CH_3), 1.20–1.45 (m, 188H, CH_2), 1.47–1.54 (m, 12H, $OCH_2CH_2CH_2$) 1.84–1.97 (m, 24H, OCH_2CH_2), 4.09 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.21 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.69 (s, 12H, a-H), 7.70 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.90 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.2, 29.4, 29.5, 29.6, 29.6, 29.7, 29.8, 29.8, 32.0 (CH_2), 68.5 (C-a), 69.5, 69.7 (OCH_2), 106.9, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.6,

124.4 (C-4, C-1′, C-6′), 148.6, 149.0, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 2918 (vs), 2849 (s), 1617 (w), 1515 (s), 1466 (m), 1433 (s), 1387 (w), 1260 (vs), 1170 (vs), 1070 (m), 1044 (m), 1033 (m), 1022 (m), 940 (w), 909 (w), 861 (w), 835 (m), 720 (m) cm^{-1}. MS (MALDI-TOF): *m*/*z* calcd. for [$C_{204}H_{330}O_{18}^+$ (M^+)]: 3070.5; found: 3068.2. CHN analysis calcd. (%) for $C_{204}H_{330}O_{18}$ (3070.87): C 79.79, H 10.83; found: C 79.63, H 10.72.

Tris(5,6,9,10-tetrakis(tetradecyloxy)triphenylene)[18]crown-6 [**Tri(14)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(14)** (100 mg, 29.3 µmol) and $FeCl_3$ (114 mg, 703 µmol). Purification by washing with cold CH_2Cl_2 (15 mL). Yield: 44 % (44.0 mg, 12.9 µmol), brown solid. ^{1}H NMR (700 MHz, $CDCl_3$): δ = 0.85–0.91 (m, 36H, CH_3), 1.21–1.61 (m, 264H, CH_2), 1.85–1.97 (m, 24H, OCH_2CH_2), 4.09 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.21 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.70 (s, 12H, a-H), 7.70 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.90 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (176 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.3, 29.4, 29.5, 29.6, 29.6, 29.7, 29.7, 29.8, 29.8, 31.9 (CH_2), 69.3 (C-a), 69.5, 69.7 (OCH_2), 106.9, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.6, 124.4 (C-4, C-1′, C-6′), 148.6, 149.0, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 2916 (vs), 2848 (vs), 1617 (w), 1514 (s), 1467 (m), 1434 (s), 1387 (w), 1260 (vs), 1171 (vs), 1056 (m), 1039 (m), 1025 (m), 929 (w), 897 (w), 859 (w), 836 (m), 796 (w), 768 (w), 721 (w), 651 (w), 616 (w), 604 (w), 451 (w) cm^{-1}. MS (MALDI-TOF): *m/z* calcd. for [$C_{228}H_{378}O_{18}^+$ (M^+)]: 3406.9; found: 3405.4. HRMS (ESI): *m*/*z* calcd. for [$C_{228}H_{378}O_{18}Na]^+$: 3429.8623, found: 3429.7725. CHN analysis calcd. (%) for $C_{228}H_{378}O_{18}$ (3407.51): C 80.37, H 11.18; found: C 79.98, H 10.89.

Tris(5,6,9,10-tetrakis(hexadecyloxy)triphenylene)[18]crown-6 [**Tri(16)**]

Synthesis according to GP S1 with *o*-terphenyl **Ter(16)** (100 mg, 26.6 µmol) and $FeCl_3$ (103 mg, 638 µmol). Purification by washing with cold CH_2Cl_2 (15 mL). Yield: 10 % (9.92 mg, 2.65 µmol), brown solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.84–0.92 (m, 36H, CH_3), 1.19–1.62 (m, 312H, CH_2), 1.84–1.99 (m, 24H, OCH_2CH_2), 4.09 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.21 (t, *J* = 6.6 Hz, 12H, OCH_2), 4.70 (s, 12H, a-H), 7.70 (s, 6H, 3-H, 2′-H, 5′-H), 7.78 (s, 6H, 3-H, 2′-H, 5′-H), 7.90 (s, 6H, 3-H, 2′-H, 5′-H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.7, 26.2, 26.3, 29.4, 29.5, 29.6, 29.6, 29.6, 29.7, 29.8, 29.8, 29.8, 31.9 (CH_2), 69.3 (C-a), 69.5, 69.7 (OCH_2), 106.9, 107.2, 109.5 (C-3, C-2′, C-5′), 123.3, 123.6, 124.4 (C-4, C-1′, C-6′), 148.6, 148.9, 149.0 (C-1, C-2, C-3′, C-4′) ppm. FT-IR (ATR): $\tilde{\nu}$ = 2916 (vs), 2848 (s), 1617 (w), 1515 (m), 1467 (m), 1434 (m), 1387 (w), 1261 (s), 1171 (s), 1068 (w), 1039 (w), 1023 (w), 941 (w), 916 (w), 886 (w), 860 (w), 836 (w), 796 (w), 771 (w), 721 (w), 632 (w), 616 (w), 603 (w) cm^{-1}.

MS (MALDI-TOF): *m/z* calcd. for [$C_{252}H_{427}O_{18}^{+}$ (M^{+})]: 3744.3; found: 3737.5. CHN analysis calcd. (%) for $C_{252}H_{426}O_{18}$ (3744.16): C 80.84, H 11.47; found: C 80.46, H 11.47.

Mesomorphic properties of triphenylene derivatives [Tri(n)]

Table S1 DSC results of the triphenylene derivatives **Tri(n)** (2nd cooling). Phase transition temperatures *T* are given in °C and the transition enthalpies in kJ / mol.[a,b]

Compound	Cr	T_m (ΔH)	Col_h	T_c (ΔH)	I
Tri(8)	●	147 (–10.3)	●	214 (–28.7)	●
Tri(10)	G	72 (-)	●	176 (–8.5)	●
Tri(11)	●	121 (–90.2)	●	168 (–19.0)	●
Tri(12)	●	71 (–69.9)	●	157 (–14.4)	●
Tri(14) [c]	●	42 (–16.3)	●	142 (–17.0)	●
Tri(16) [d]	●	53 (–84.5)	●	134 (–15.6)	●

[a] Heating/cooling rate 5 K / min

[b] The following phases were observed: crystalline Cr, glass G, columnar hexagonal Col_h, columnar Col_x, isotropic I

[c] An additional crystal-to-crystal transition was detected at 85°C upon 2nd heating.

[d] An additional crystal-to-crystal transition was detected at 81°C upon 2nd heating.

Under the POM triphenylenes **Tri(n)** exhibited fan-shaped and conical fan textures typical for columnar mesophases upon cooling from the isotropic phase, as illustrated in Figure S1a and S1b for the compounds **Tri(8)** and **Tri(14)**.

DSC experiments revealed enantiotropic mesomorphism for all members of the series. Except for compound **Tri(10)**, which showed a glass transition at 72°C, all other derivatives showed clear crystallization (see Table S1). Both melting and clearing points decreased with increasing chain lengths from 147°C and 214°C for **Tri(8)** to 33°C and 134°C for **Tri(16)**, resulting in broad mesophases, particularly for **Tri(10)**, **Tri(14)** and **Tri(16)** (Figure S1c).

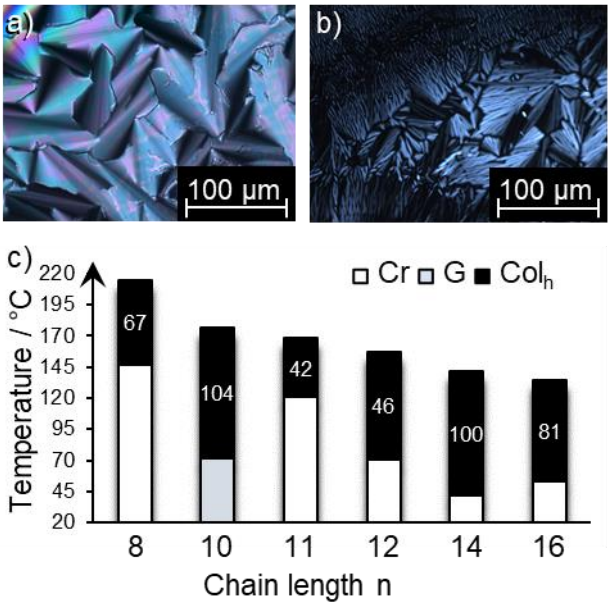


Figure S1 a) Fan-shaped POM textures of **Tri(8)** (207°C, 5 K / min) and b) **Tri(14)** (131°C, 5 K / min) upon cooling from the isotropic phase (magnification: ×200). c) Temperature ranges in K of the mesophases of triphenylenes **Tri(n)** for different chain lengths n. The phase transitions and phase widths were determined by DSC upon 2nd cooling. The DSC curves are shown in Fig. S9–S14.

XRD studies of the triphenylene **Tri(8)** revealed three distinct reflexes in the small-angle section in a ratio of $1 : 1/\sqrt{3} : 1/2$, which were indexed as (10), (11), (20) of a columnar hexagonal phase with *p6mm* symmetry (Table S2). In addition, a weak (001) reflex in the wide-angle region at 3.93 Å due to π–π interactions of the triphenylenes was observed. The (001) reflex together with the broad halo around 4.2 Å indicated the presence of an ordered Col$_{ho}$ phase with lattice parameters a = 32.34 Å and c = 3.93 Å. Similar results were obtained for **Tri(10)** and **Tri(11)**, albeit with increased lattice parameters due to the increased chain lengths. For **Tri(14)** a Col$_h$ phase with *p6mm* symmetry was found.

Table S2 Results of the XRD experiments for the triphenylenes **Tri(n)**.

Tri(n)	Mesophase	Lattice spacing / Å	*d* spacing / Å exp. (calcd.)	Miller indices
Tri(8)	Col$_{ho}$	a = 32.34	28.01	(10)
	p6mm	c = 3.93	16.12 (16.17)	(11)
	(180°C)		13.97 (14.00)	(20)
			3.9	(001) π–π
			4.2	Halo

Tri(10)	Col_{ho}	*a* = 35.62	30.85	(10)
	p6mm	*c* = 4.03	17.84 (17.81)	(11)
	(140°C)		15.51 (15.42)	(20)
			4.0	(001) π–π
			4.6	Halo
Tri(11)	Col_{h}	*a* = 36.87	31.93	(10)
	p6mm	*c* = 4.11	18.34 (18.43)	(11)
	(160°C)		15.85 (15.96)	(20)
			4.1	(001) π–π
			4.4	Halo
Tri(14)	Col_{h}	*a* = 41.46	35.90	(10)
	p6mm		4.0	Halo
	(120°C)			

POM textures

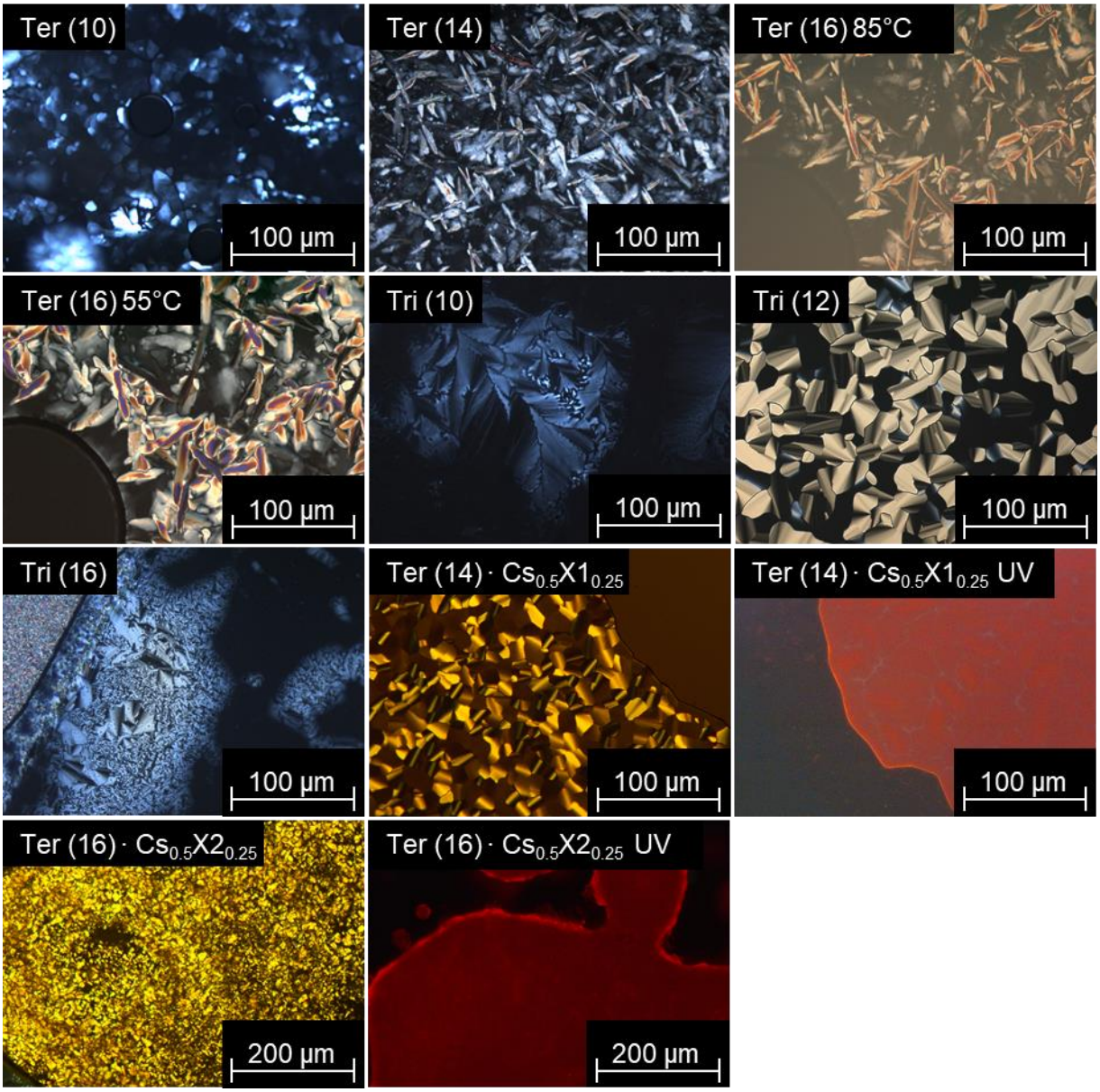


Figure S2 Observed POM textures of the synthesized compounds upon cooling from the isotropic phase (cooling rate: 5 K / min).

DSC traces

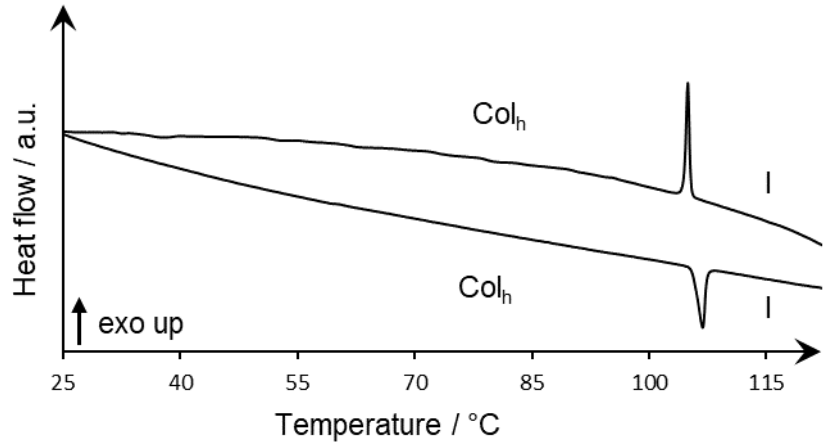


Figure S3 DSC traces of **Ter(8)** (2nd cooling, 2nd heating, 5 K / min).

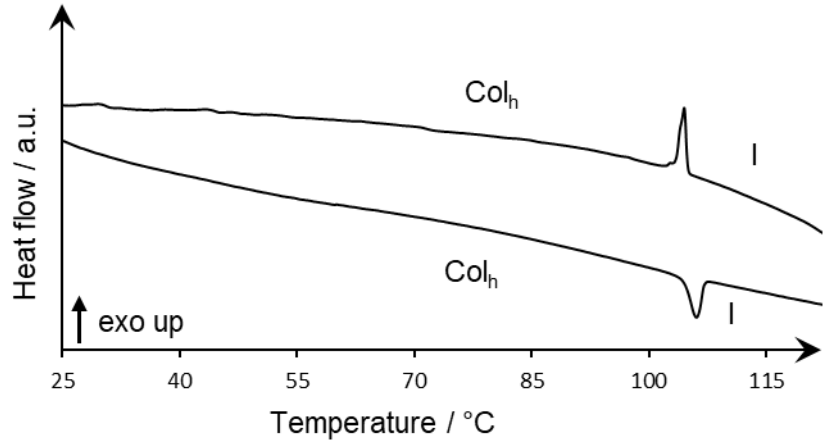


Figure S4 DSC traces of **Ter(10)** (2nd cooling, 2nd heating, 5 K / min).

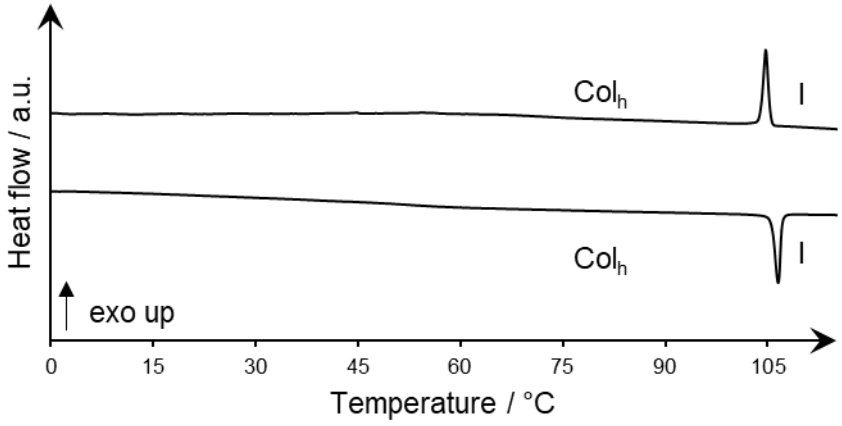


Figure S5 DSC traces of **Ter(11)** (2nd cooling, 3rd heating, 10 K / min).

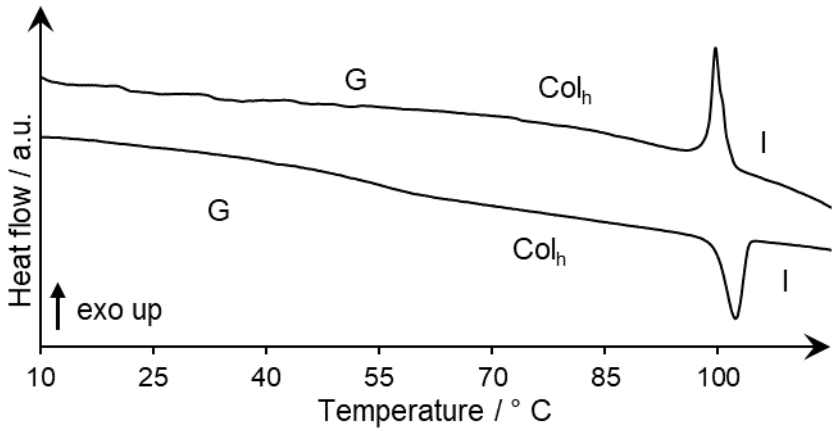


Figure S6 DSC traces of **Ter(12)** (2nd cooling, 2nd heating, cooling rate: 10 K / min).

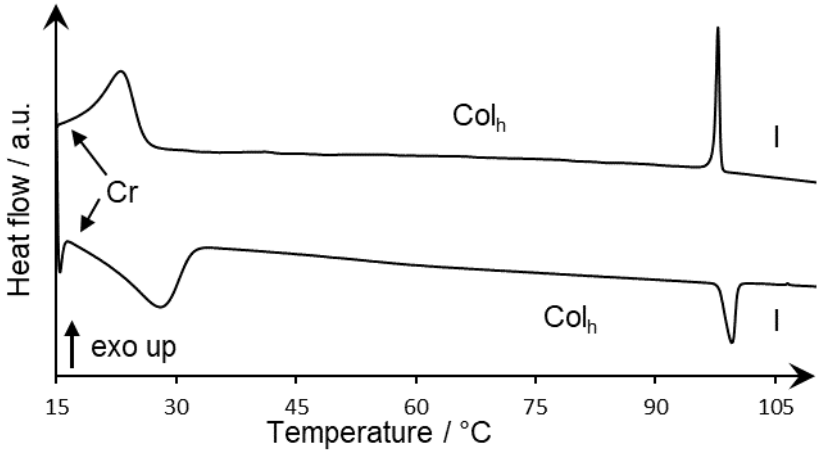


Figure S7 DSC traces of **Ter(14)** (2nd cooling, 3rd heating, cooling rate: 5 K / min).

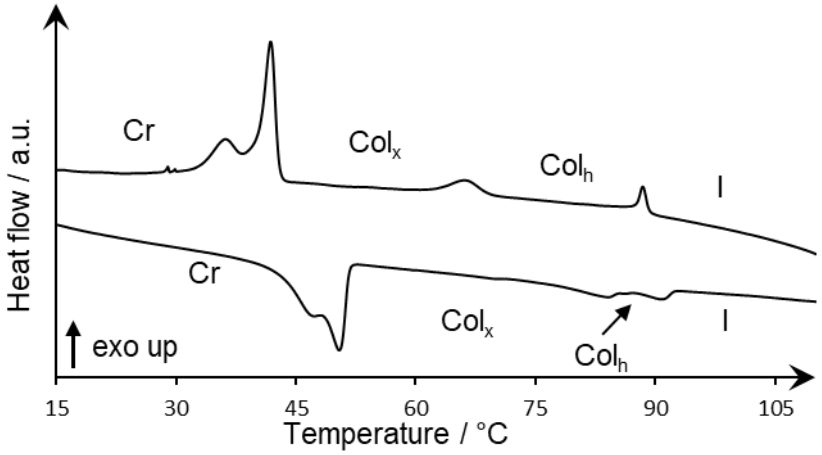


Figure S8 DSC traces of **Ter(16)** (2nd cooling, 2nd heating, 5 K / min).

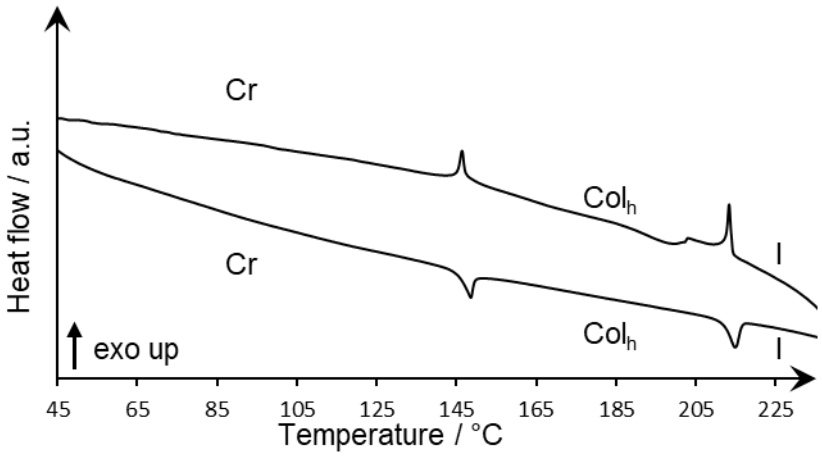


Figure S9 DSC traces of **Tri(8)** (2nd cooling, 2nd heating, 5 K / min).

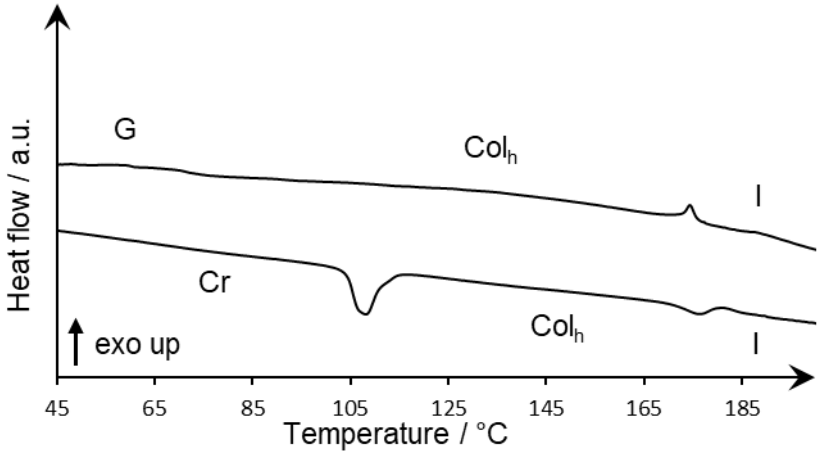


Figure S10 DSC traces of **Tri(10)** (2nd cooling, 2nd heating, 5 K / min).

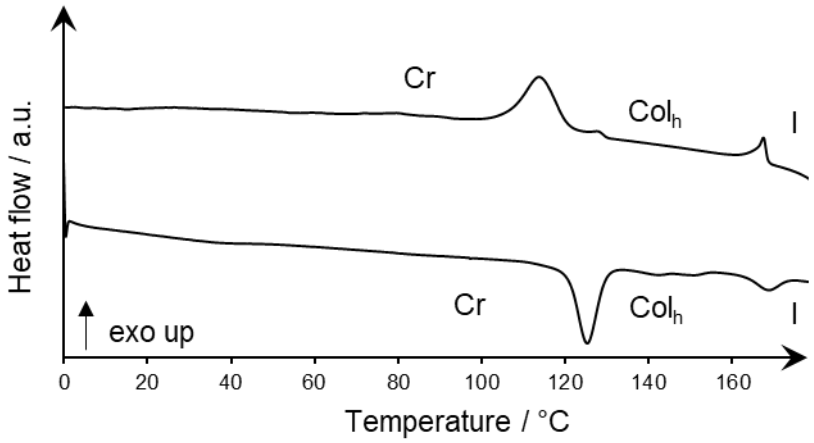


Figure S11 DSC traces of **Tri(11)** (2nd cooling, 2nd heating, 10 K / min).

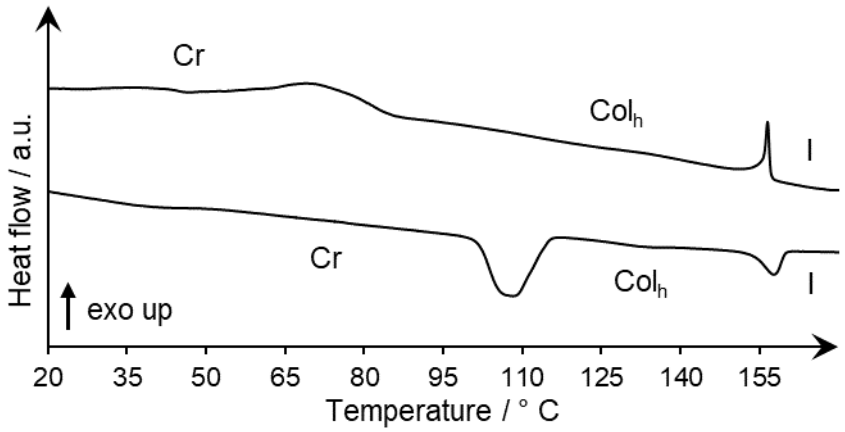


Figure S12 DSC traces of **Tri(12)** (2nd cooling, 2nd heating, 10 K / min).

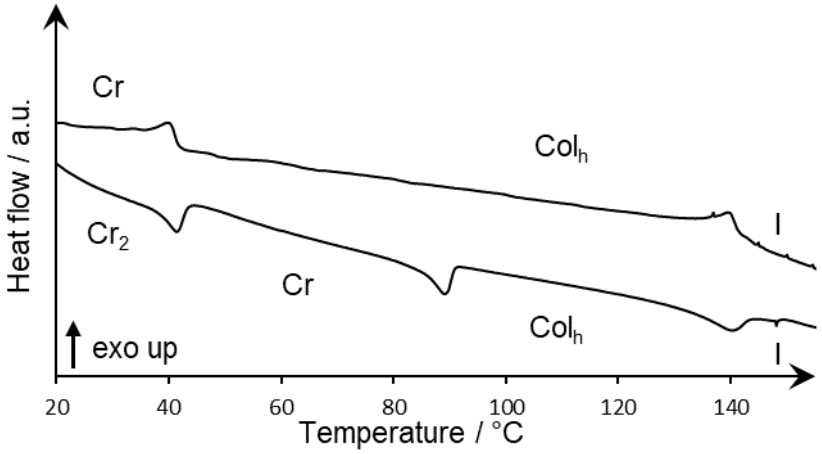


Figure S1 DSC traces of **Tri(14)** (2nd cooling, 2nd heating, 5 K / min).

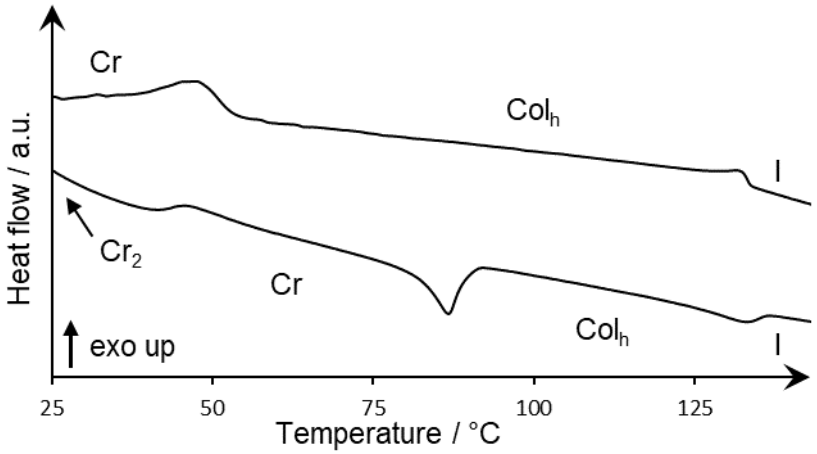


Figure S2 DSC traces of **Tri(16)** (2nd cooling, 2nd heating, 5 K / min).

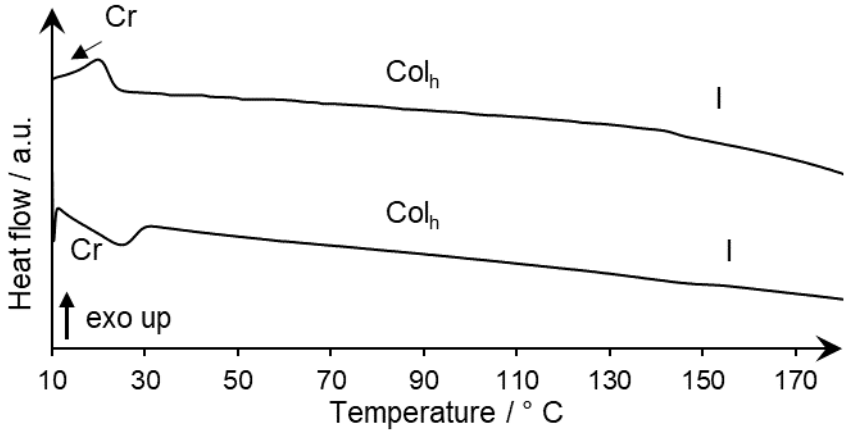


Figure S3 DSC traces of **Ter(14) · Cs$_{0.5}$X1$_{0.25}$** (2nd cooling, 2nd heating, 5 K / min).

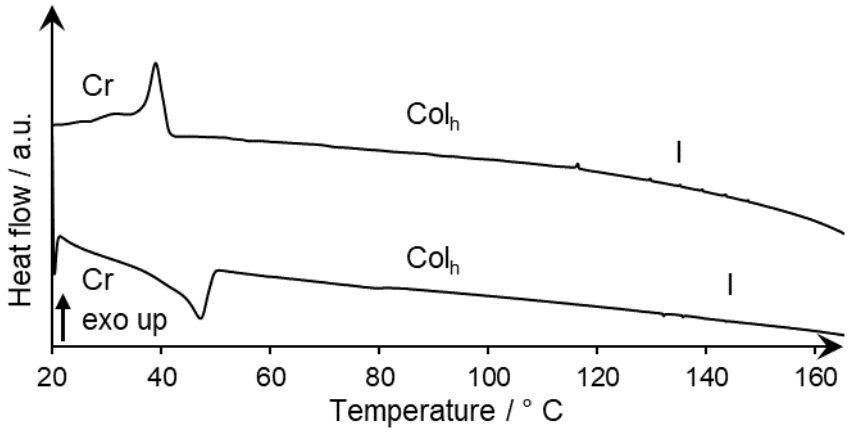


Figure S4 DSC traces of **Ter(16) · Cs$_{0.5}$X1$_{0.25}$** (2nd cooling, 2nd heating, 5 K / min).

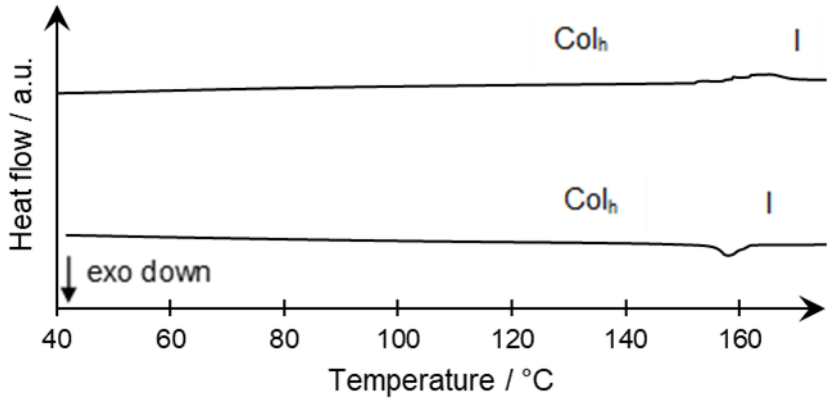


Figure S5 DSC traces of **Ter(14) · Cs$_{0.5}$X2$_{0.25}$** (3rd cooling, 2nd heating, 5 K / min).

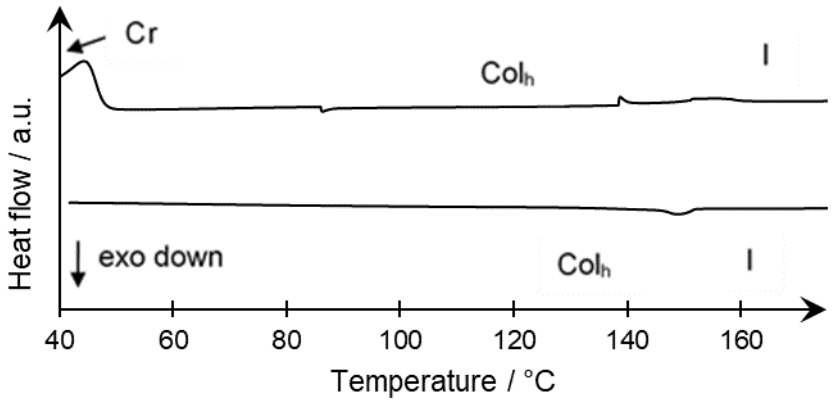


Figure S6 DSC traces of **Ter(16) · $Cs_{0.5}X2_{0.25}$** (3rd cooling, 3rd heating, 10 K / min).

XRD data and diffraction patterns

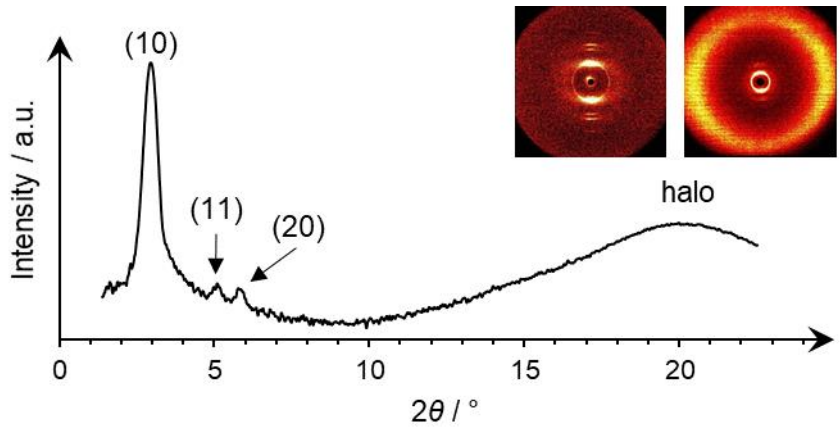


Figure S7 WAXS diffractogram of **Ter(8)** at 61°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

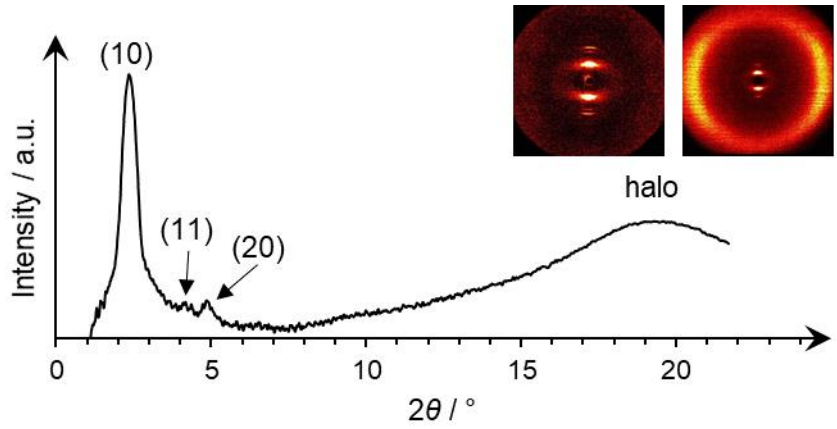


Figure S20 WAXS diffractogram of **Ter(10)** at 70°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

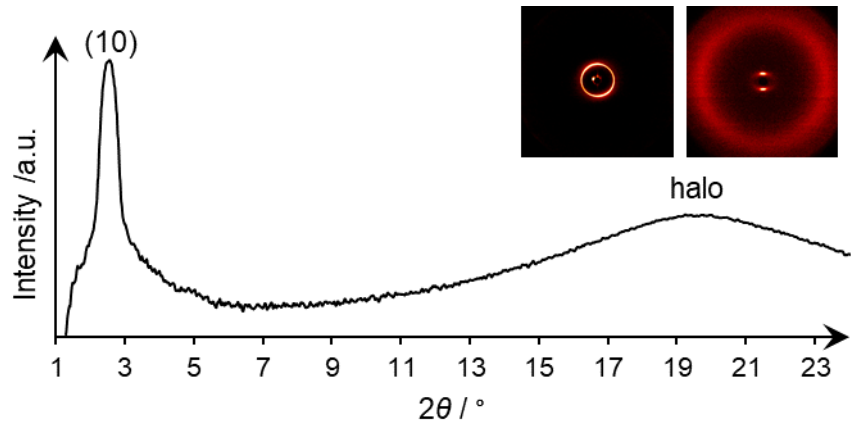


Figure S21 WAXS diffractogram of **Ter(12)** at 90°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

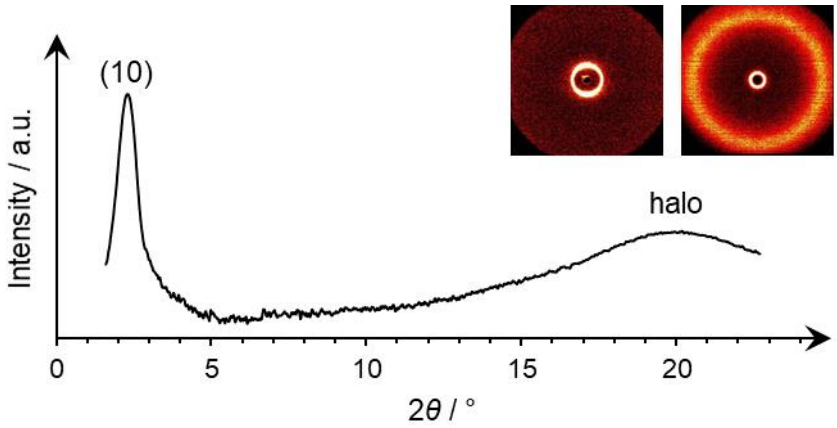


Figure S22 WAXS diffractogram of **Ter(14)** at 45°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

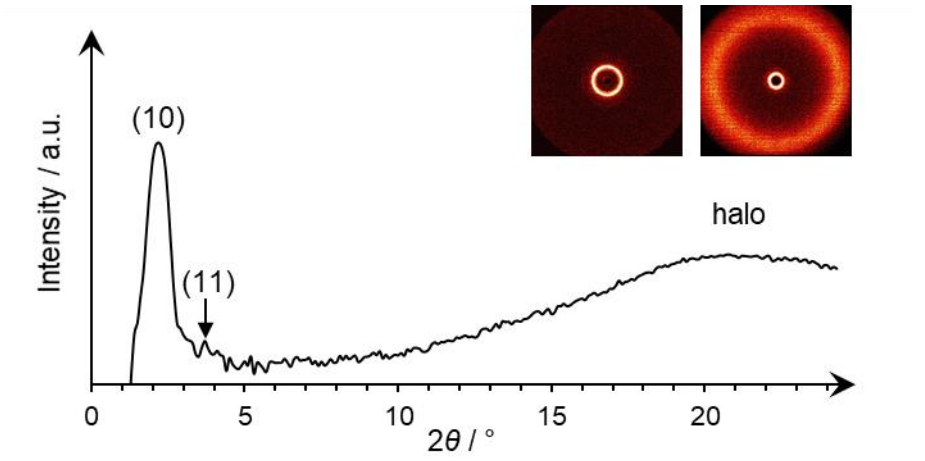


Figure S23 WAXS diffractogram of **Ter(16)** at 70°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

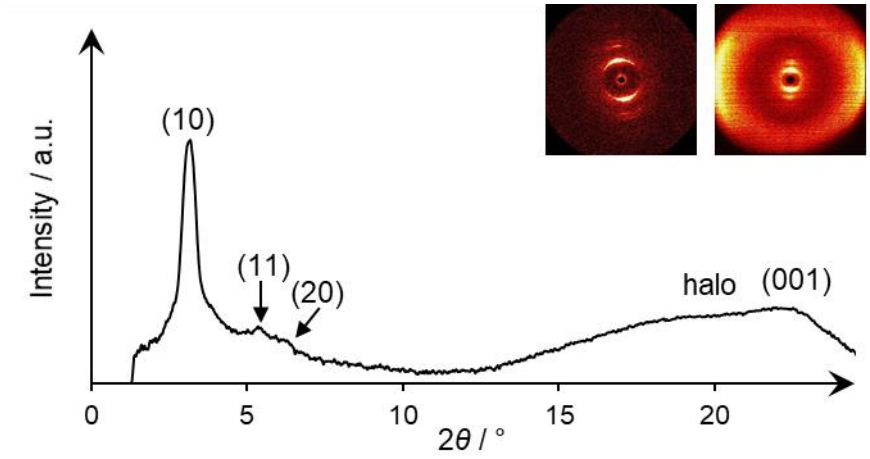


Figure S24 WAXS diffractogram of **Tri(8)** at 180°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

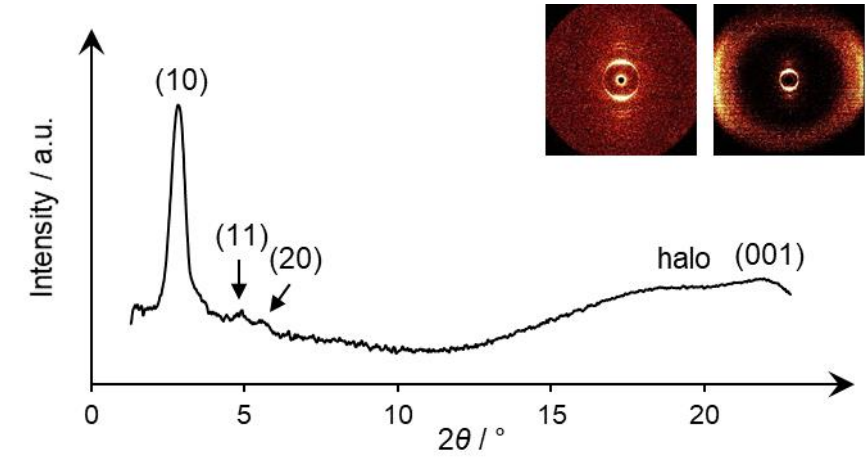


Figure S25 WAXS diffractogram of **Tri(10)** at 140°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

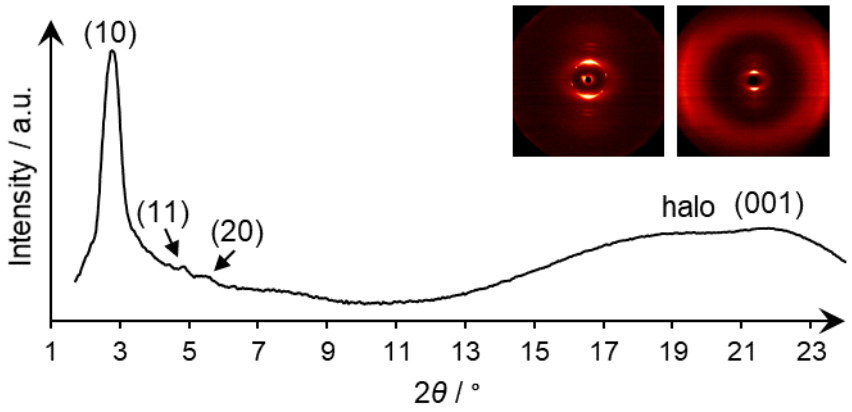


Figure S86 WAXS diffractogram of **Tri(11)** at 160°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

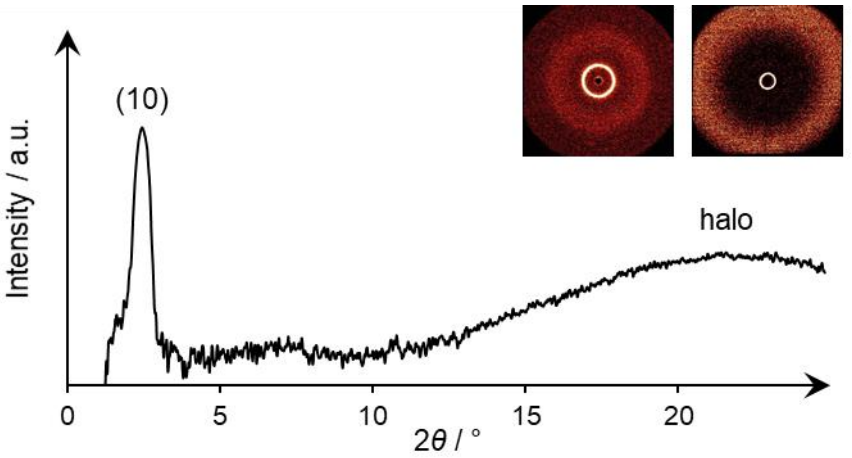


Figure S9 WAXS diffractogram of **Tri(14)** at 120°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

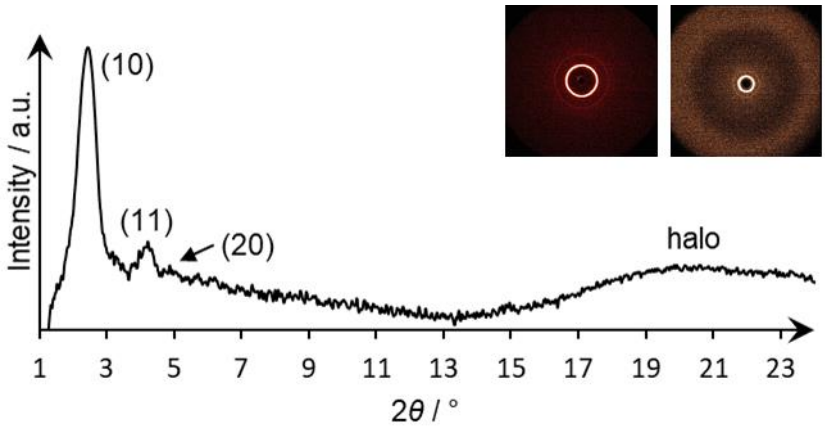


Figure S10 WAXS diffractogram of **Ter(14) · $Cs_{0.5}X1_{0.25}$** at 93°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

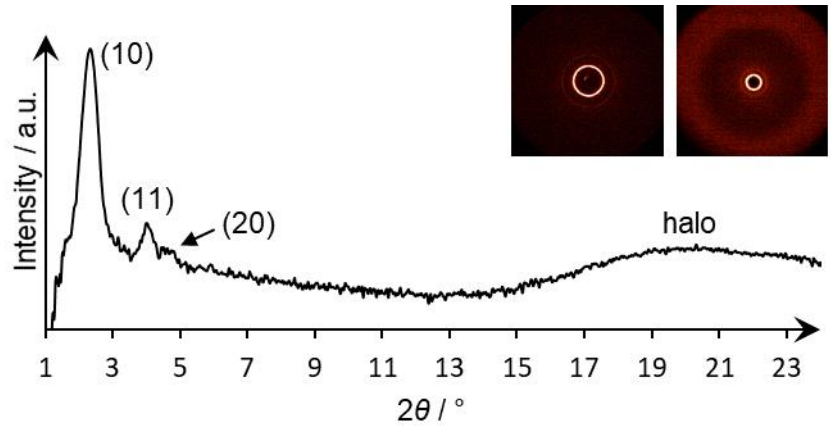


Figure S11 WAXS diffractogram of **Ter(16) · $Cs_{0.5}X1_{0.25}$** at 112°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

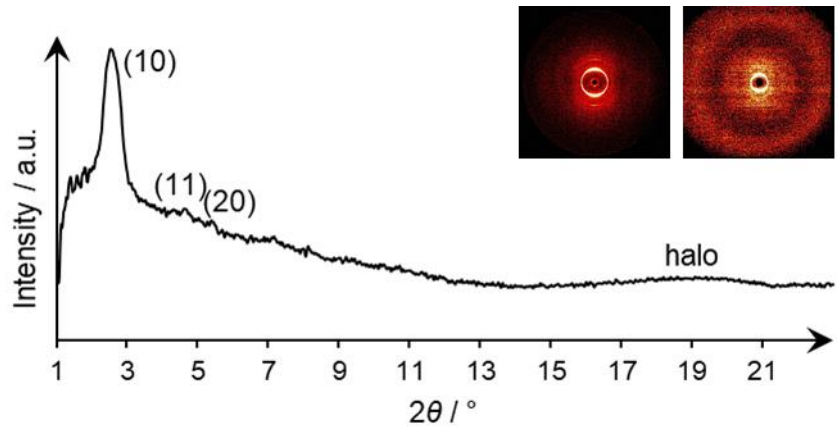


Figure S30 WAXS diffractogram of **Ter(14) · $Cs_{0.5}X2_{0.25}$** at 140°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

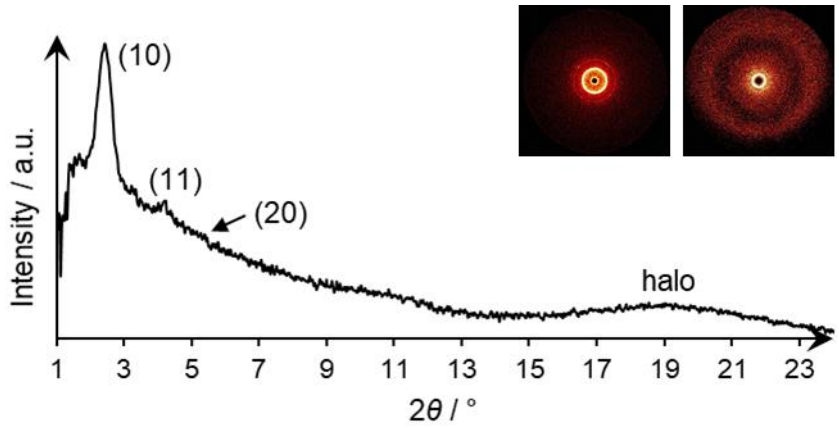


Figure S31 WAXS diffractogram of **Ter(16) · $Cs_{0.5}X2_{0.25}$** at 131°C (logarithmic scale) with the corresponding diffraction patterns (SAXS left and WAXS right).

Luminescence spectra

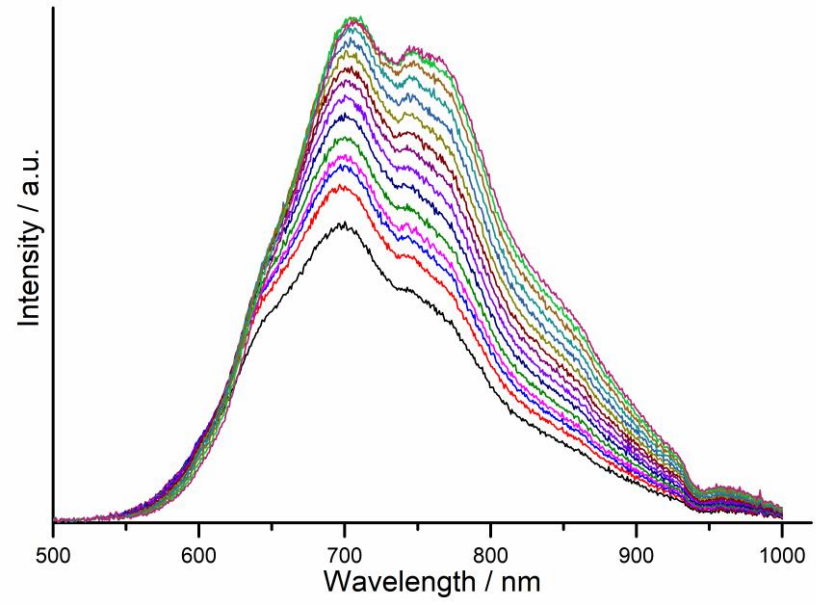


Figure S32 Temperature dependent solid state emission spectra of **Ter(14) · $Cs_{0.5}X1_{0.25}$** from 30°C (top, brown) up to 170°C (bottom, black) in steps of 10 K.

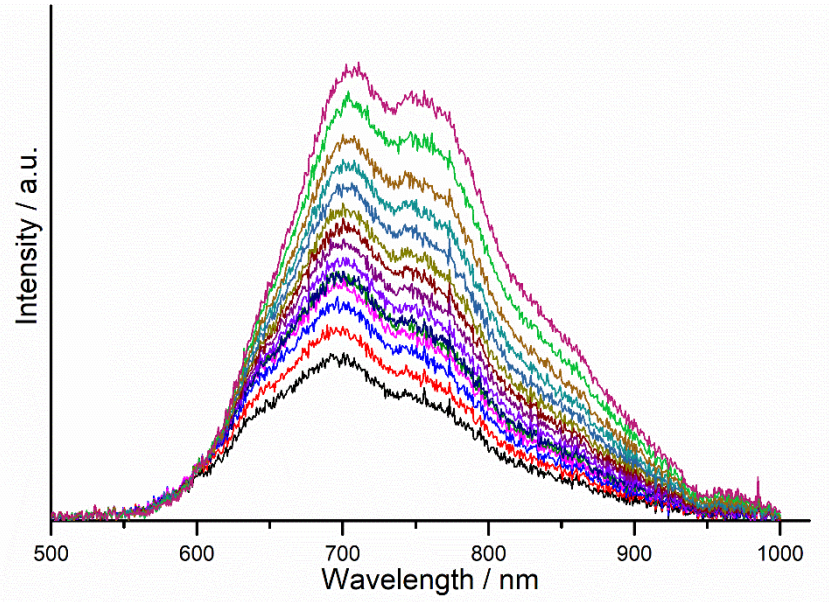


Figure S12 Temperature dependent solid state emission spectra of **Ter(16) · $Cs_{0.5}X1_{0.25}$** from 30°C (top, brown) up to 170°C (bottom, black) in steps of 10 K.

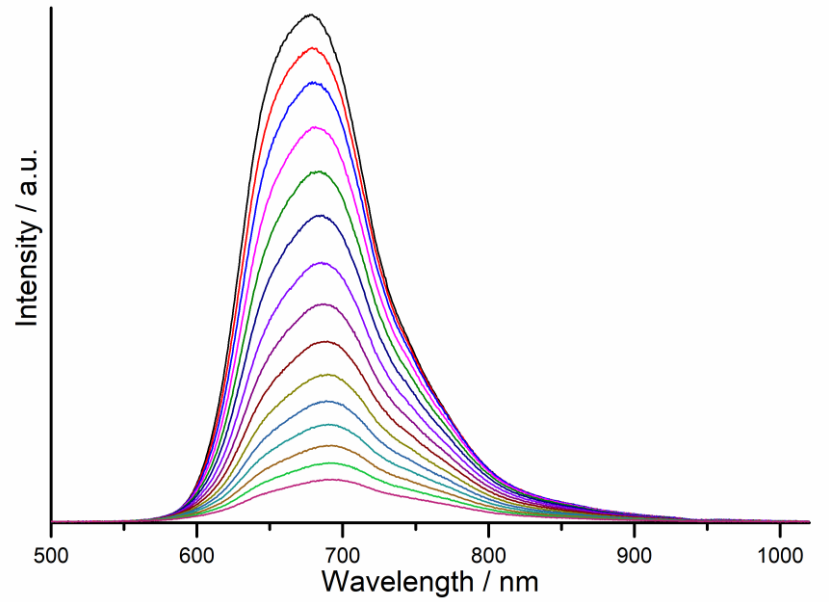


Figure S13 Temperature dependent solid state emission spectra of **Ter(14) · $Cs_{0.5}X2_{0.25}$** from 30°C (top, black) up to 170°C (bottom, brown) in steps of 10 K.

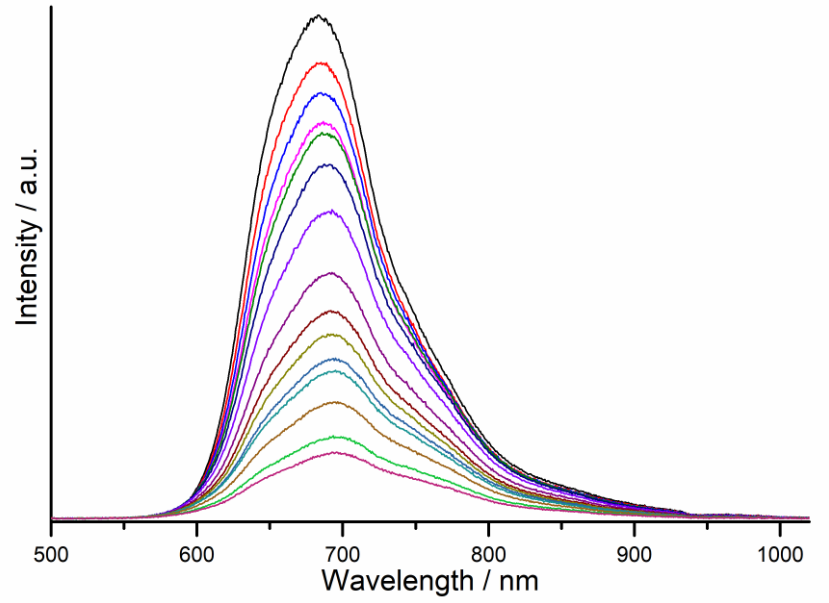


Figure S14 Temperature dependent solid state emission spectra of **Ter(16) · $Cs_{0.5}X2_{0.25}$** from 30°C (top, black) up to 170°C (bottom, brown) in steps of 10 K.

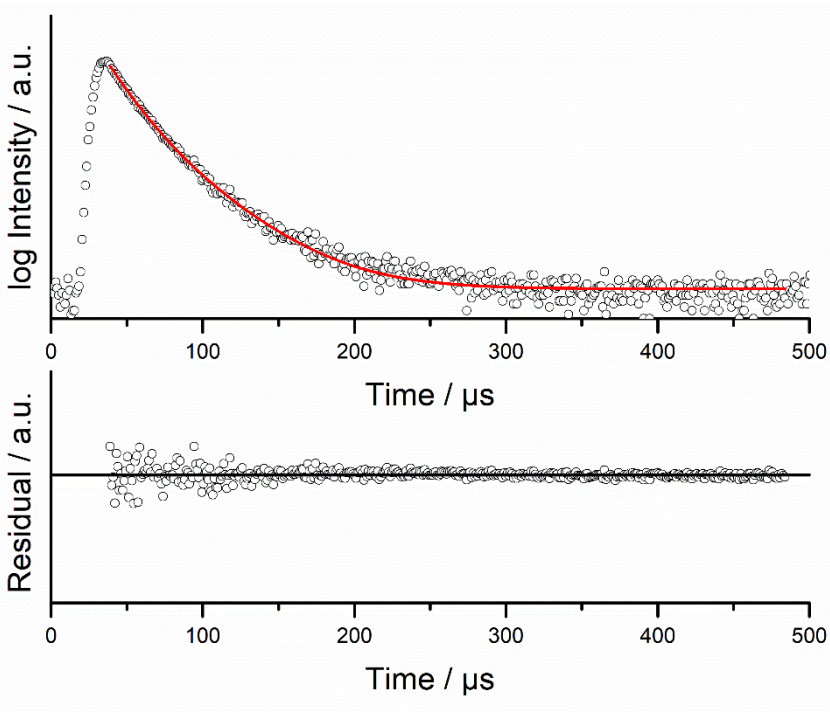


Figure S15 Solid state emission lifetime decay (2nd order) of **Ter(14) · $Cs_{0.5}X1_{0.25}$** at ambient temperature (top). Excitation wavelength: λ = 375 nm; signal detection at λ = 735–745 nm. The respective residual (bottom) represents the goodness of fit.

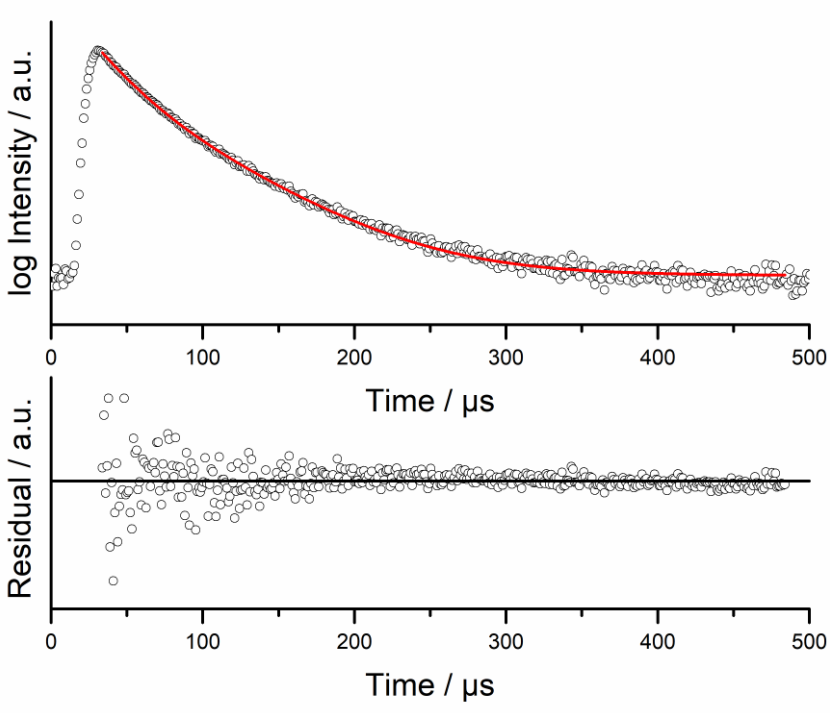


Figure S16 Solid state emission lifetime decay (2nd order) of **Ter(16) · $Cs_{0.5}X1_{0.25}$** at ambient temperature (top). Excitation wavelength: λ = 375 nm; signal detection at λ = 735–745 nm. The respective residual (bottom) represents the goodness of fit.

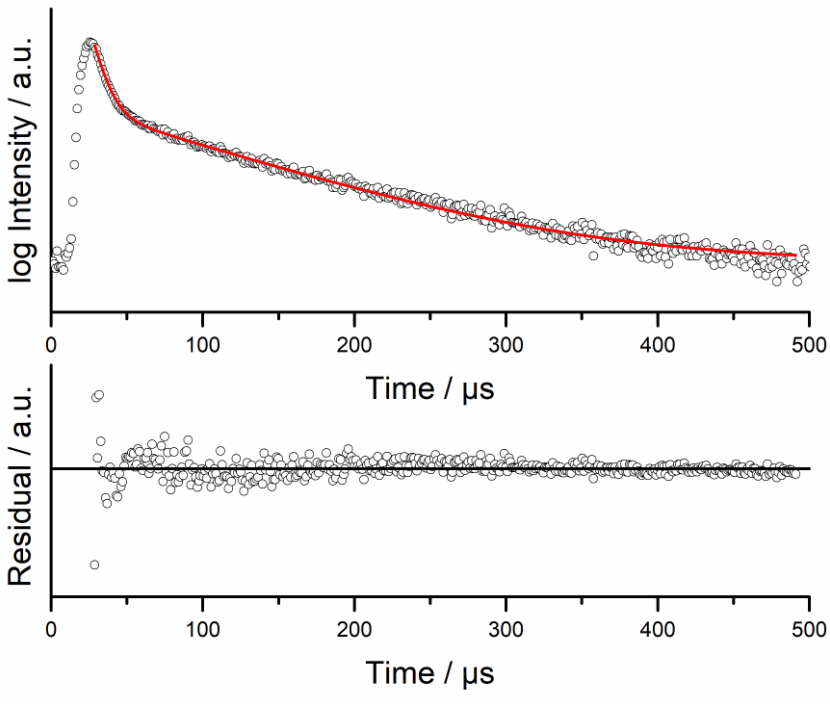


Figure S17 Solid state emission lifetime decay (2nd order) of **Ter(14) · $Cs_{0.5}X2_{0.25}$** at ambient temperature (top). Excitation wavelength: λ = 375 nm; signal detection at λ = 690–700 nm. The respective residual (bottom) represents the goodness of fit.

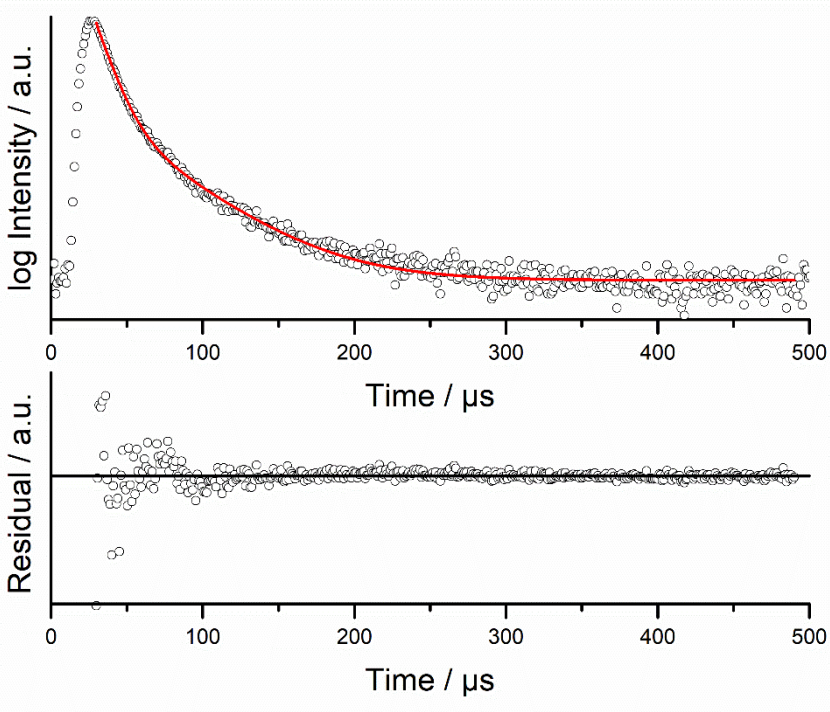


Figure S18 Solid state emission lifetime decay (2nd order) of **Ter(16) · $Cs_{0.5}X2_{0.25}$** at ambient temperature (top). Excitation wavelength: λ = 375 nm; signal detection at λ = 680–690 nm. The respective residual (bottom) represents the goodness of fit.

NMR spectra

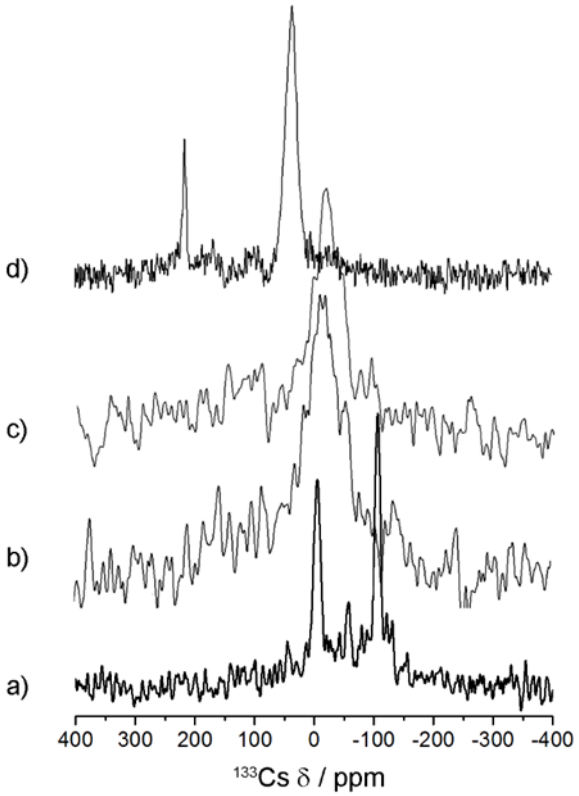


Figure S40 Solid state MAS ^{133}Cs NMR spectra of a) **Cs_2X2**, b) **Ter(14) · $Cs_{0.5}X2_{0.25}$,** c) **Ter(14) · $Cs_{0.5}X1_{0.25}$** and d) **Cs_2X1**. **X1** = $[Mo_6Br_{14}]^{2-}$; **X2** = $[Mo_6I_8(C_2F_5COO)_6]^{2-}$.

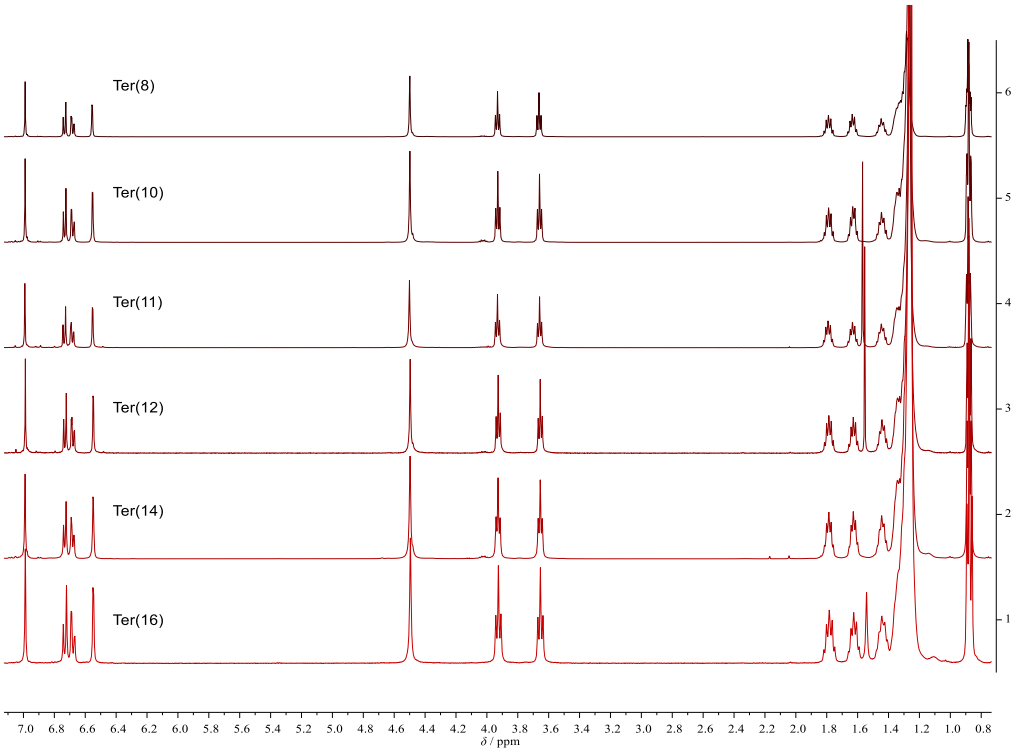


Figure S41 Stacked ^{1}H-NMR spectra of the *o*-terphenyls **Ter(n)**.

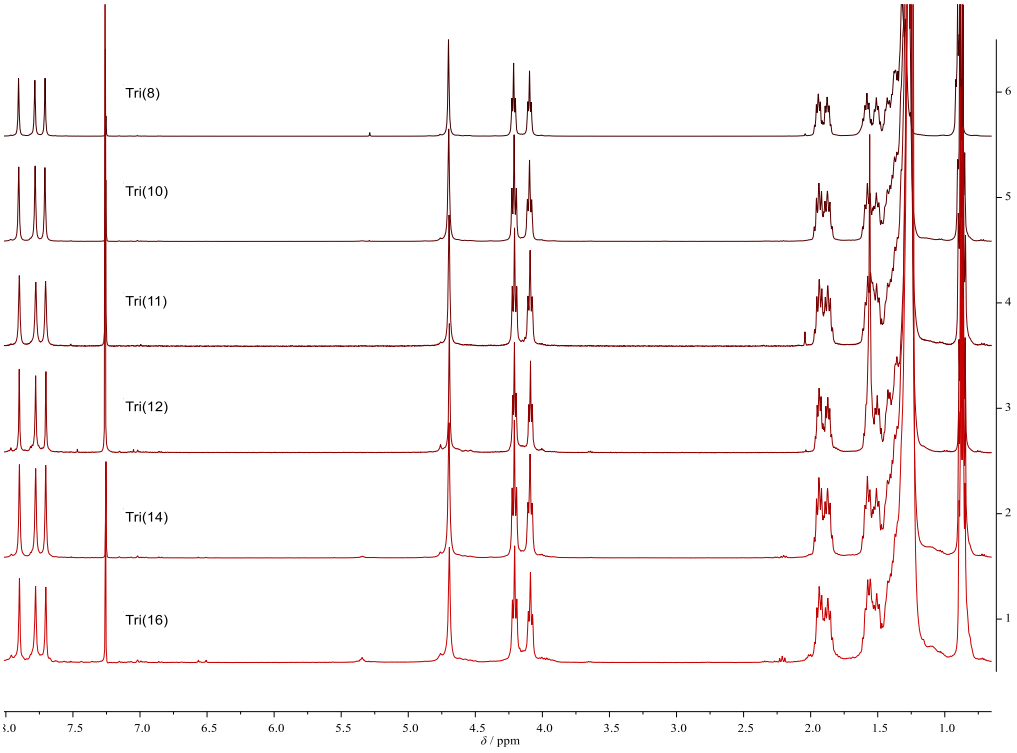
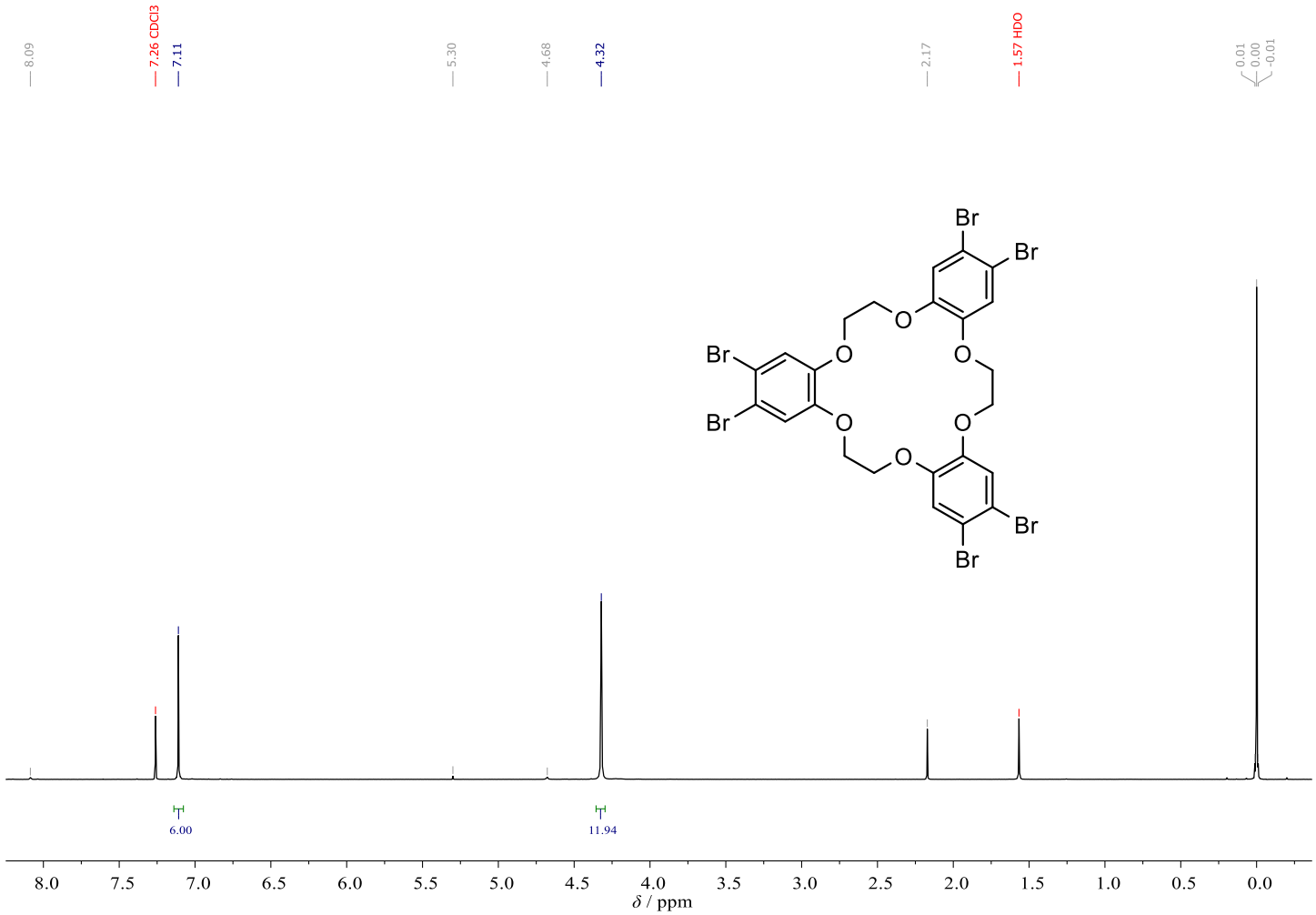


Figure S42 Stacked ^{1}H-NMR spectra of the triphenylenes **Tri(n)**.



Parameter	Value
Title	Jun10-2016.140.fid
Comment	2 Ehni EHN-92
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	297.5
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	456.0
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
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Modification Date	2016-06-10T11:45:19
Class	
Purity	89.54 %
Spectrum Quality	-0.686
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Lowest Frequency	-1247.1
Nucleus	1H
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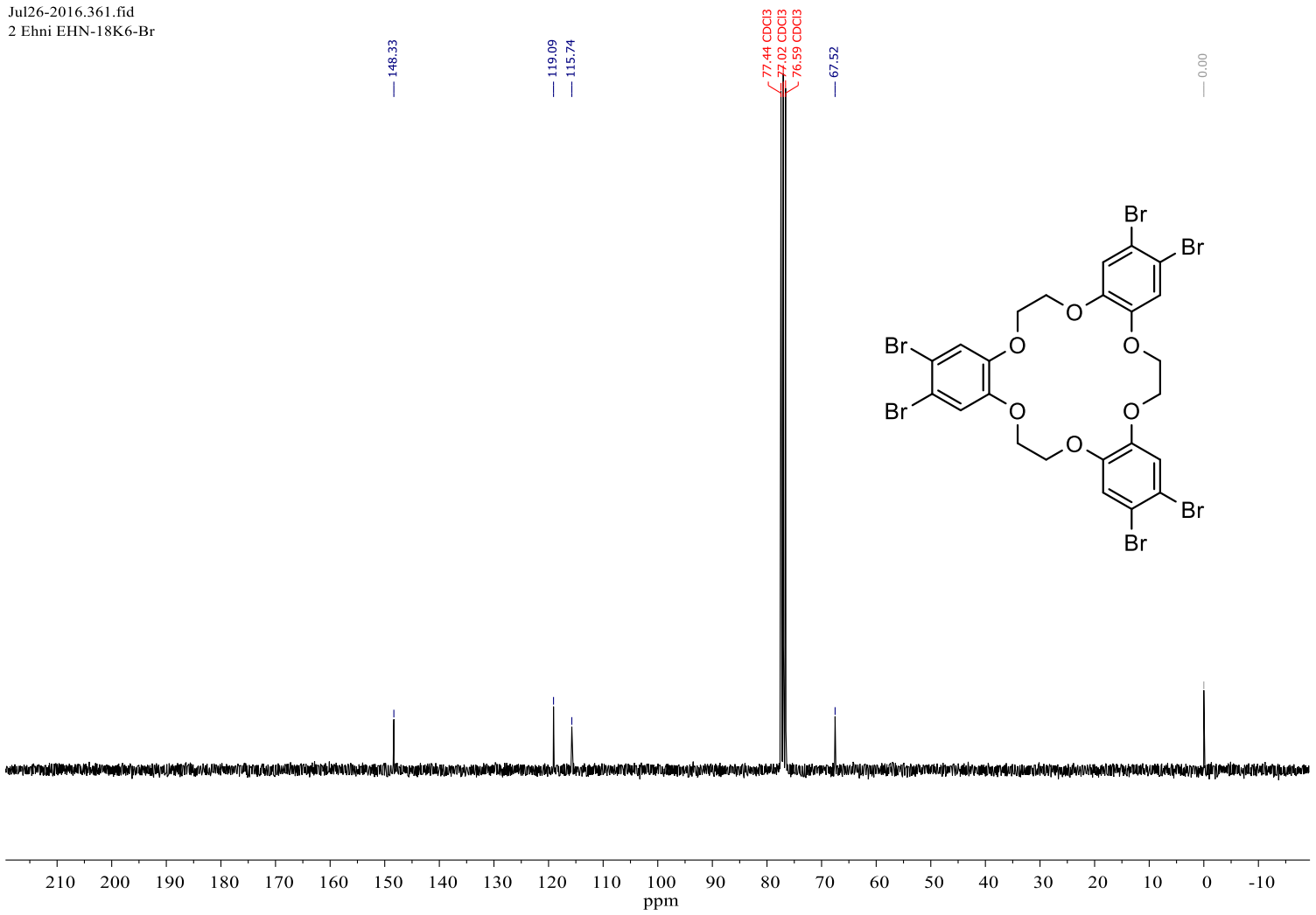
Spectral Size	65536



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Origin	Bruker BioSpin GmbH
Owner	guest
Instrument	spect
Solvent	CDCl3
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Number of Scans	1024
Receiver Gain	2580.0
Relaxation Delay	2.0000
Pulse Width	11.7500
Presaturation Frequency	
Acquisition Time	1.8176
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Modification Date	2016-07-27T00:43:19
Purity	100.00 %
Spectrum Quality	0.251
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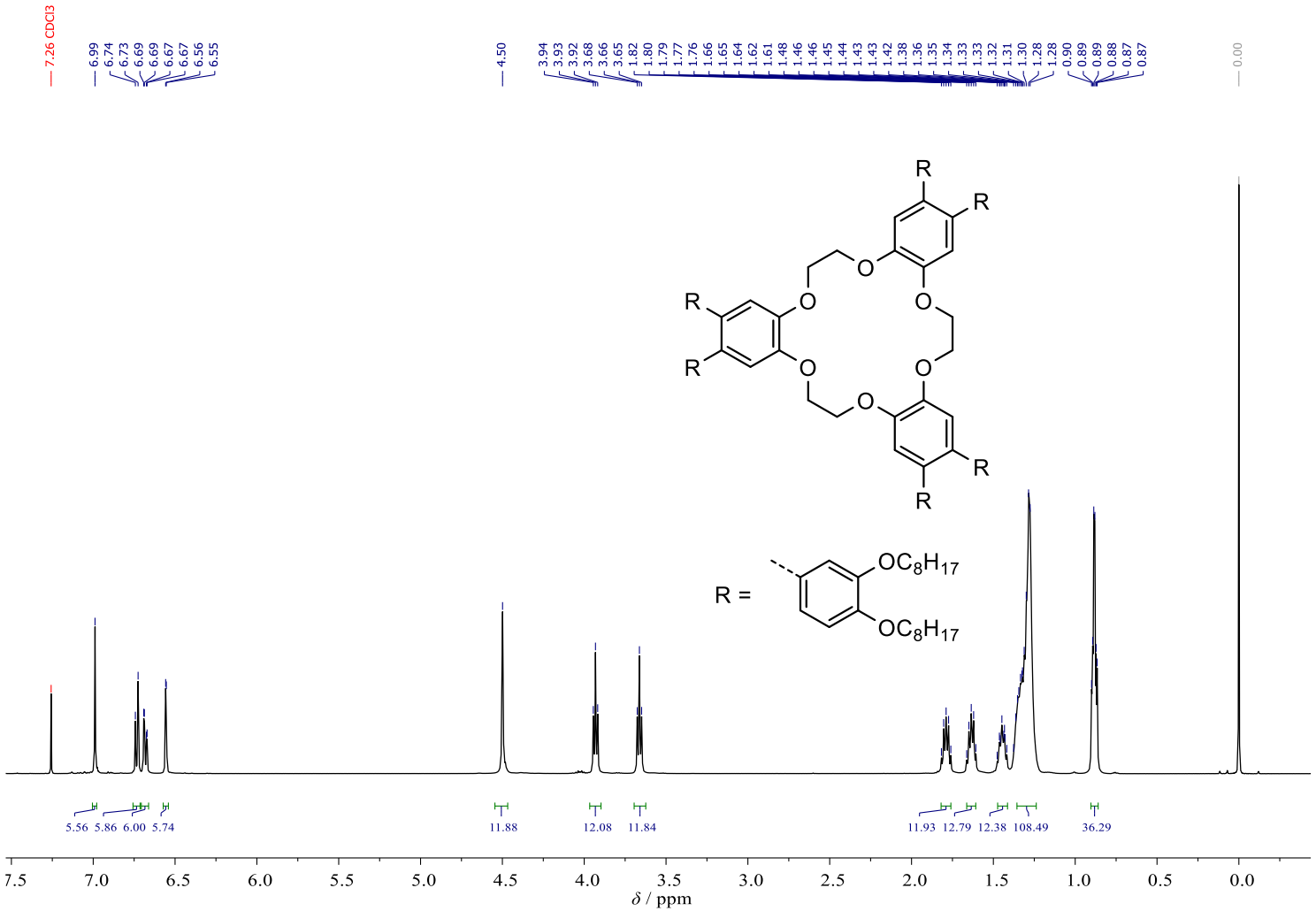
Acquired Size	32768
Spectral Size	65536



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Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	128
Receiver Gain	161.0
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
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Modification Date	2016-07-14T19:46:47
Class	
Purity	100.00 %
Spectrum Quality	0.000
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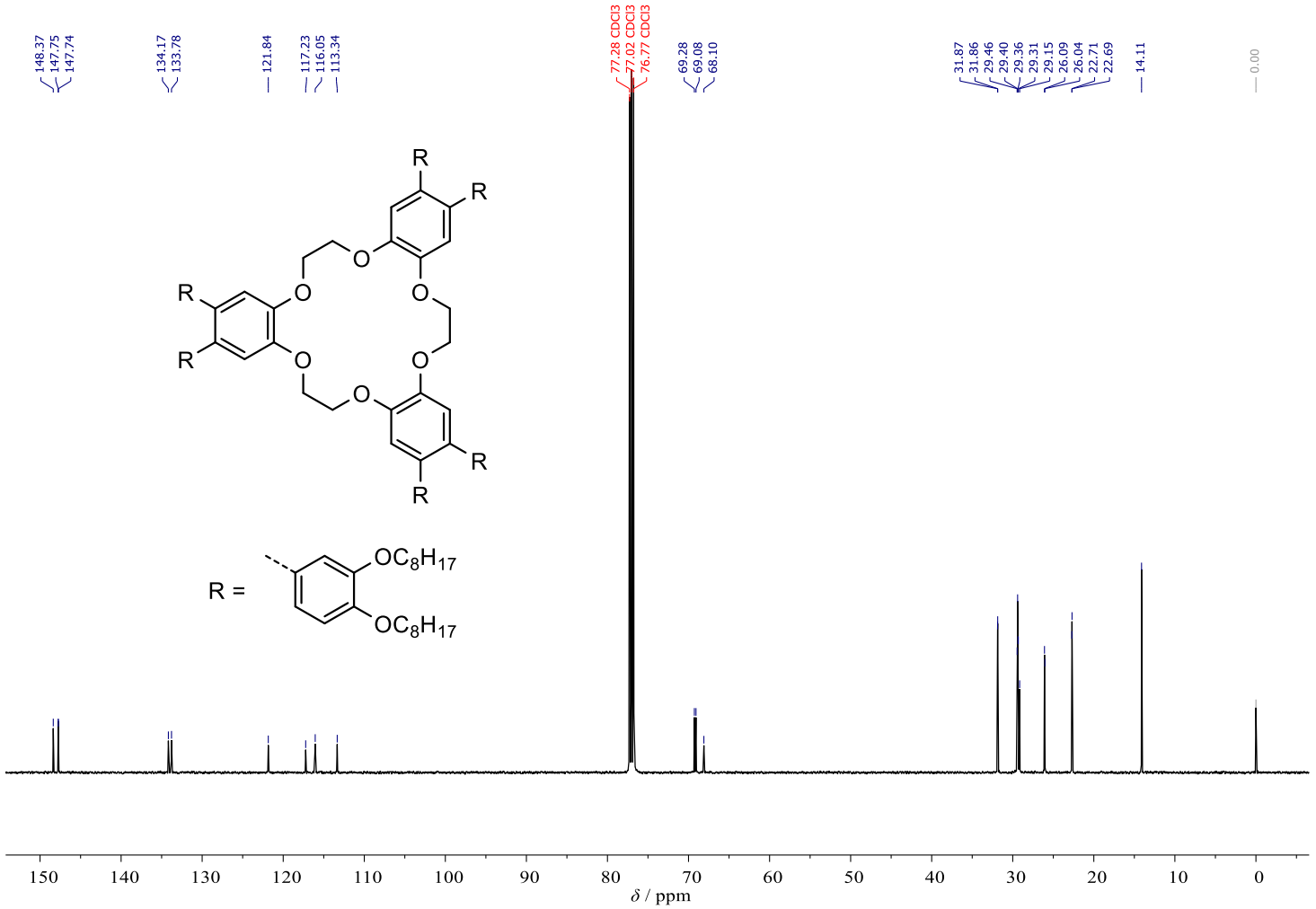
Spectral Width	10330.6
Lowest Frequency	-2094.0
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



Parameter	Value
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Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
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Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	4096
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Relaxation Delay	2.0000
Pulse Width	10.2000
Presaturation Frequency	
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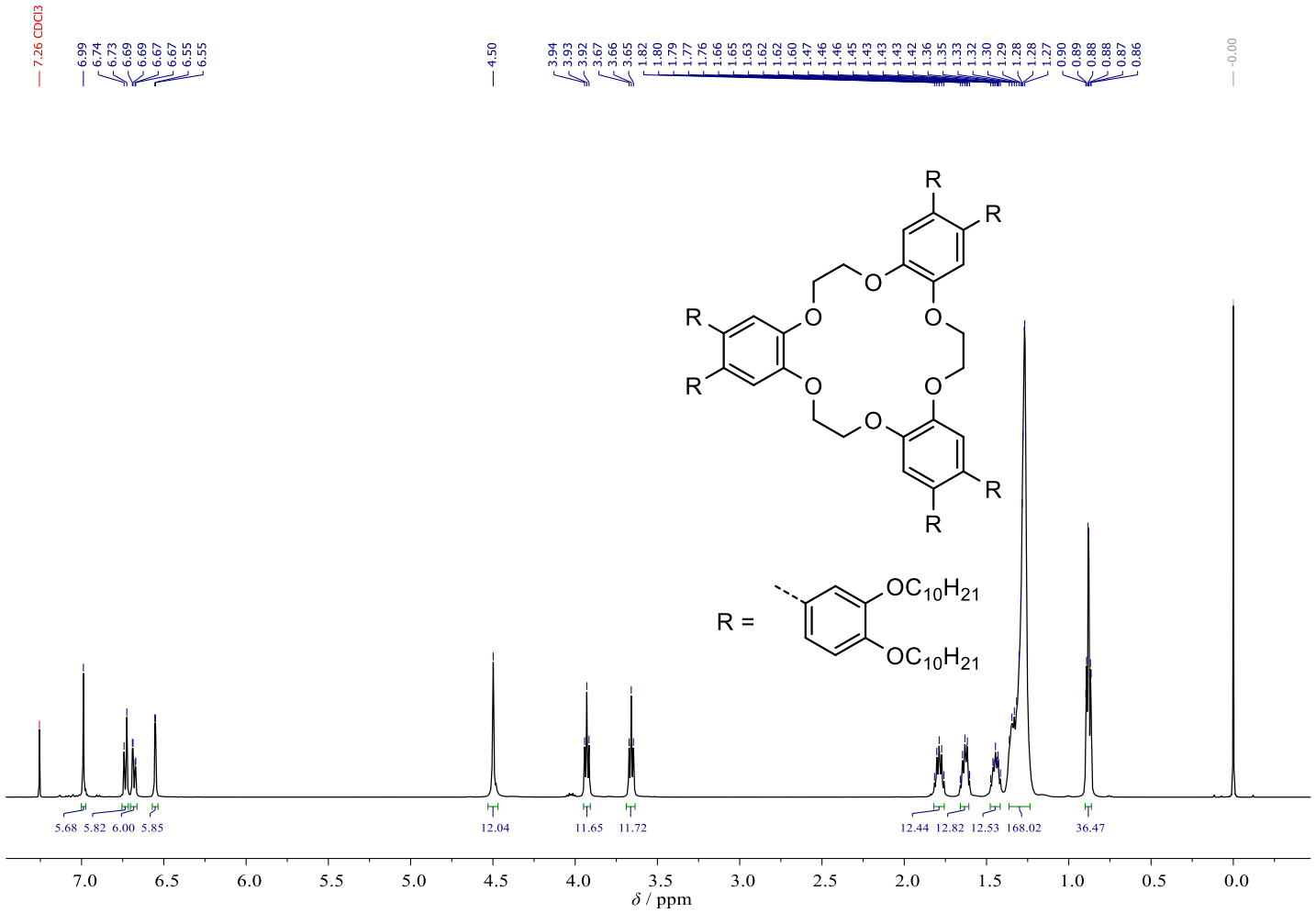
Modification Date	2016-07-14T23:16:42
Class	
Purity	100.00 %
Spectrum Quality	0.251
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3873.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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Site	
Instrument	spect
Author	
Solvent	CDCl3
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Pulse Sequence	zg30
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Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2016-07-14T23:29:00
Modification Date	2016-07-14T23:30:02
Class	
Purity	99.99 %
Spectrum Quality	-0.687
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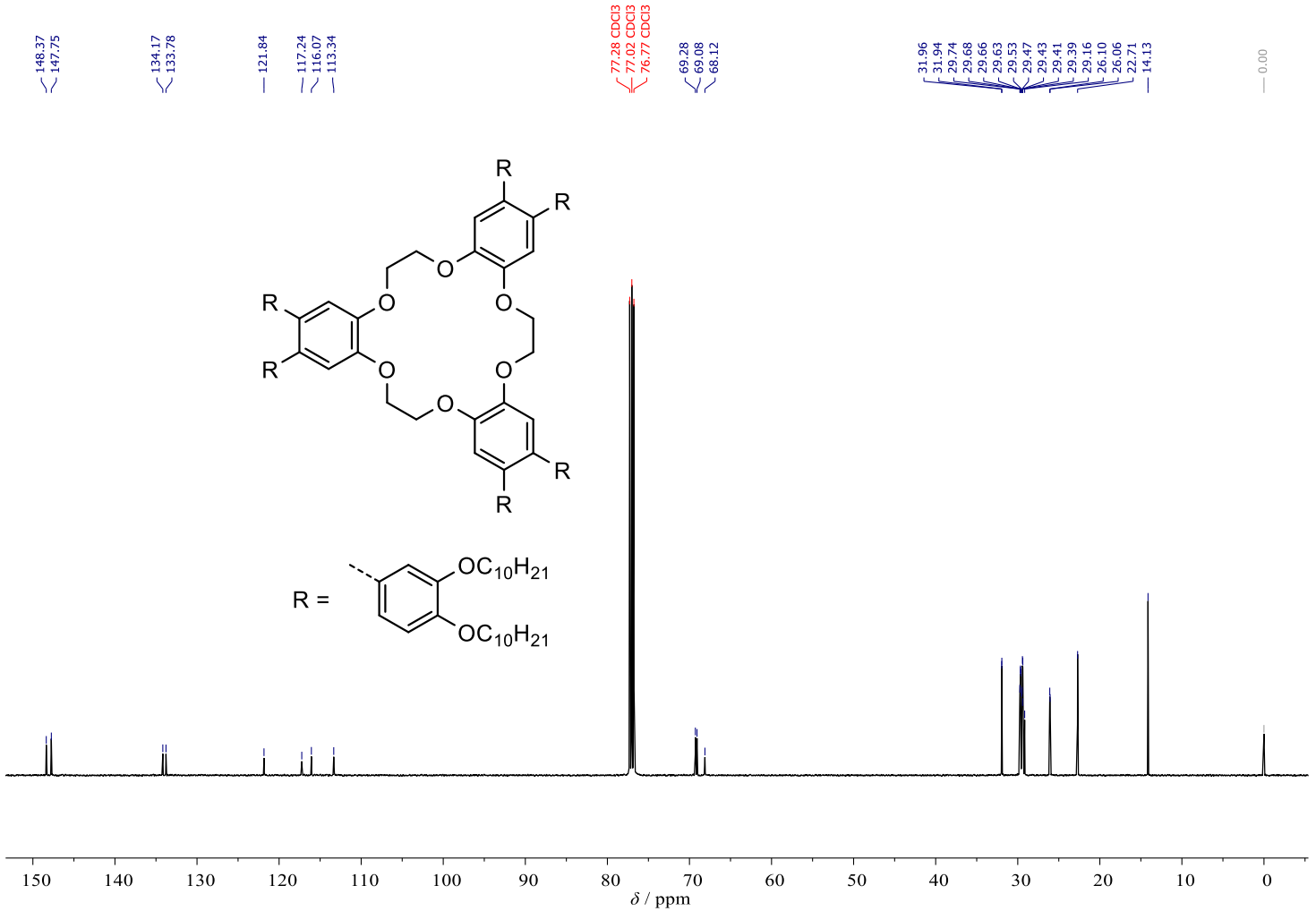
Spectral Width	10330.6
Lowest Frequency	-2094.5
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



Parameter	Value
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Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zgpg30
Experiment	1D
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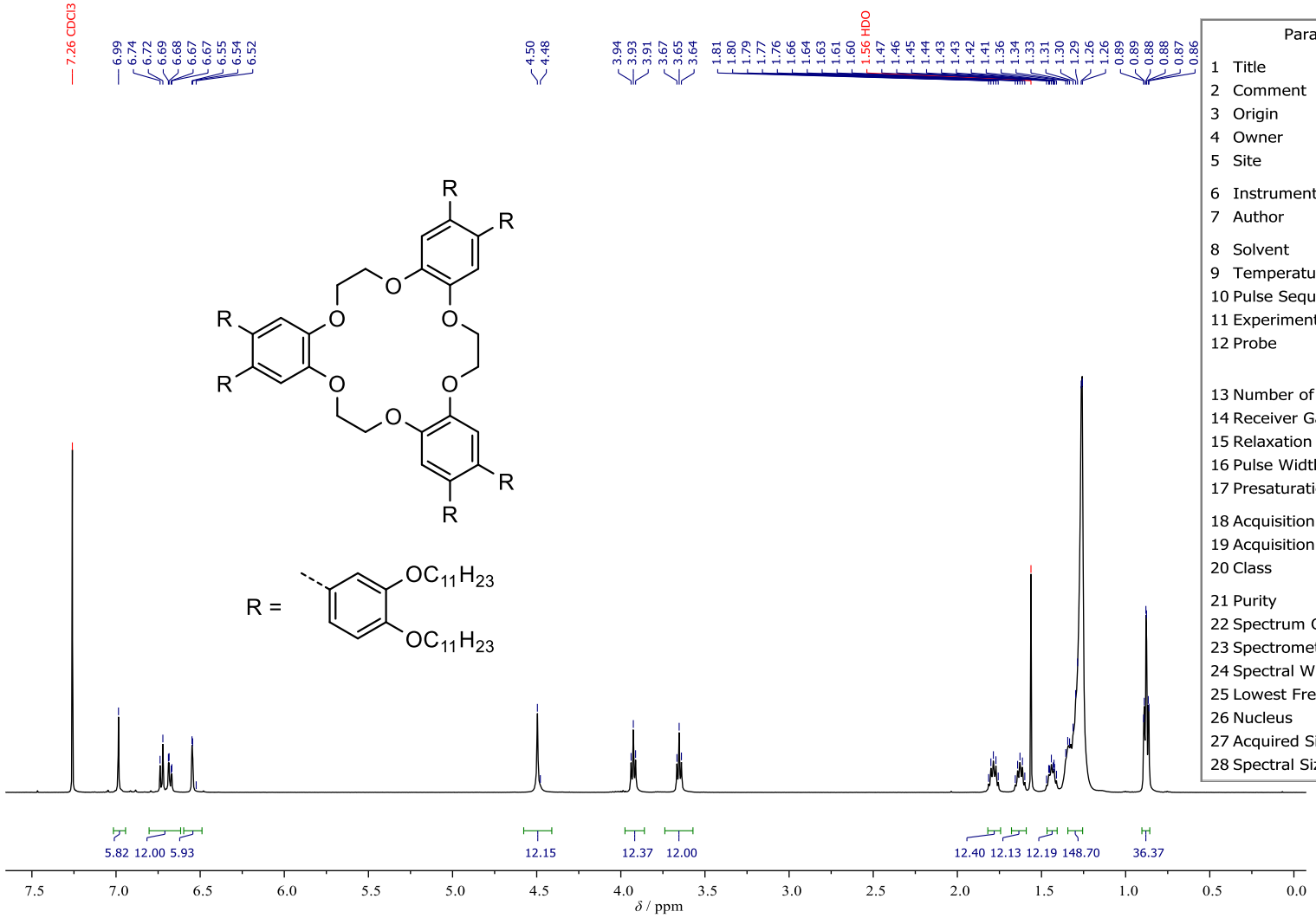
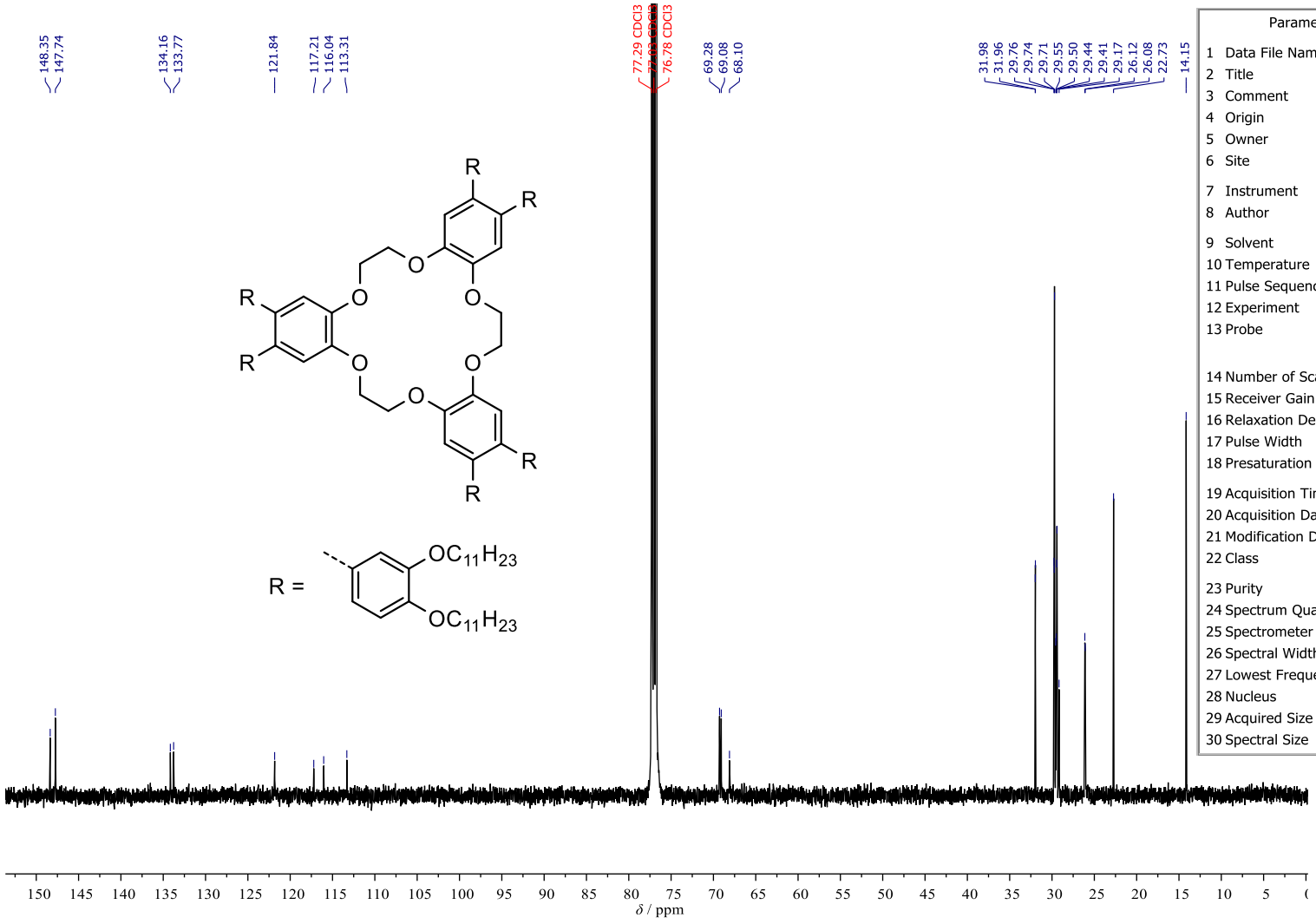
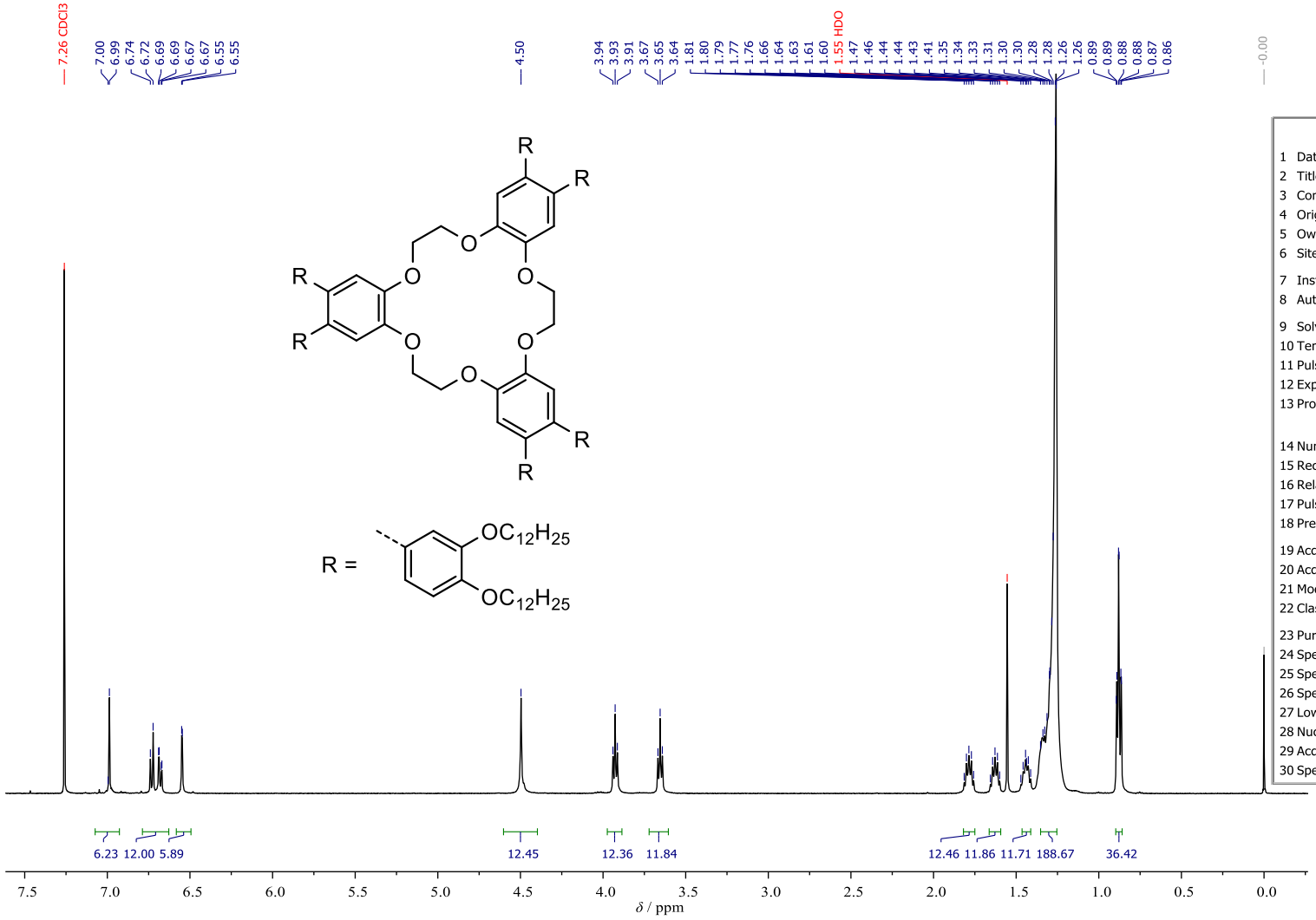
Number of Scans	4096
Receiver Gain	2890.0
Relaxation Delay	2.0000
Pulse Width	10.2000
Presaturation Frequency	
Acquisition Time	0.9962
Acquisition Date	2016-07-15T02:59:00
Modification Date	2016-07-15T02:59:58
Class	
Purity	99.91 %
Spectrum Quality	0.251
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3873.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



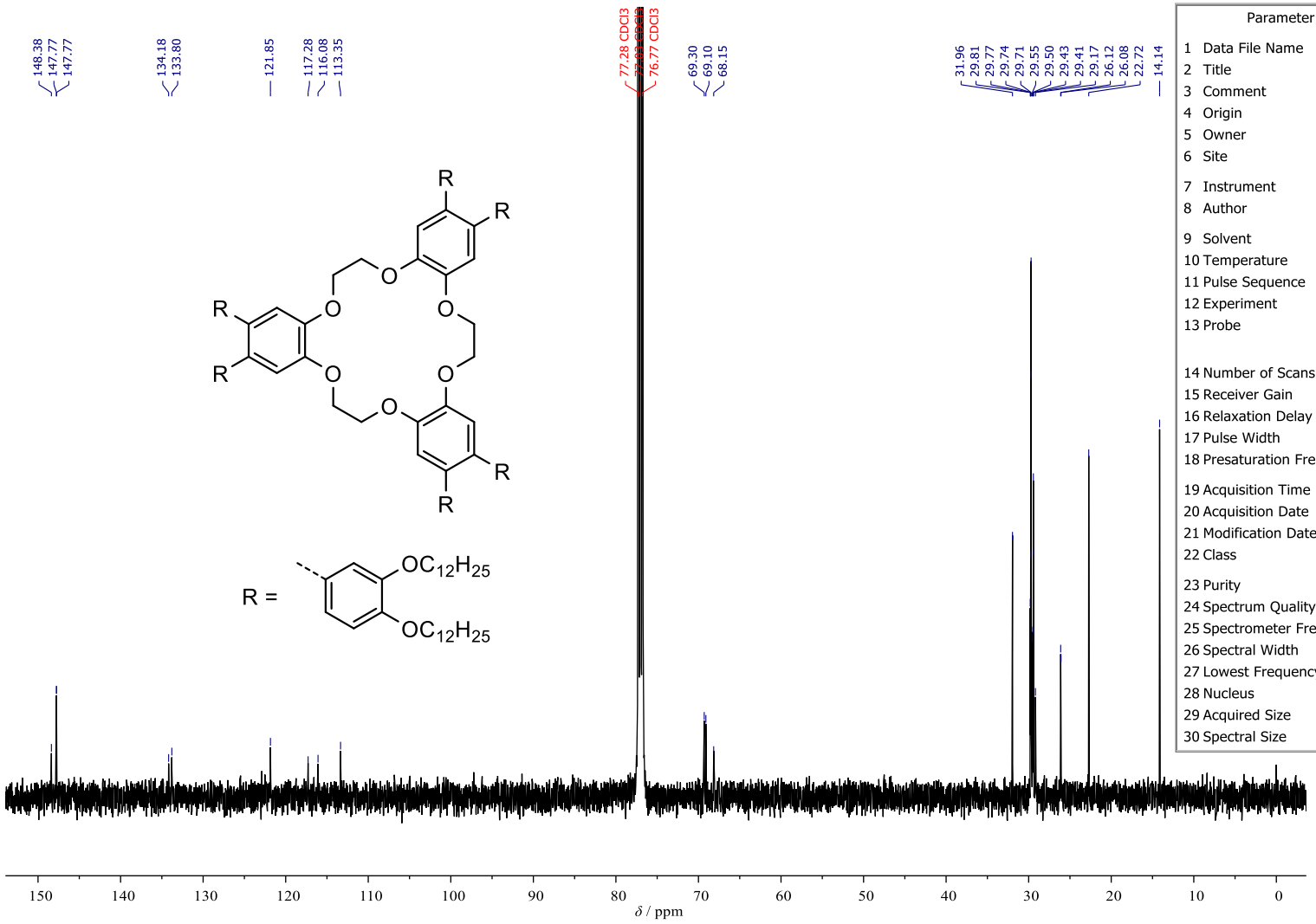
Parameter	Value
1 Title	Jun01-2011.160.fid
2 Comment	2 Beardsworth BEA059
3 Origin	Bruker BioSpin GmbH
4 Owner	guest
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	CDCl3
9 Temperature	294.7
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
13 Number of Scans	128
14 Receiver Gain	512.0
15 Relaxation Delay	2.0000
16 Pulse Width	11.0500
17 Presaturation Frequency	
18 Acquisition Time	1.5860
19 Acquisition Date	2011-06-02T21:25:00
20 Class	
21 Purity	100.00 %
22 Spectrum Quality	-0.687
23 Spectrometer Frequency	500.16
24 Spectral Width	10330.6
25 Lowest Frequency	-2092.8
26 Nucleus	1H
27 Acquired Size	16384
28 Spectral Size	65536



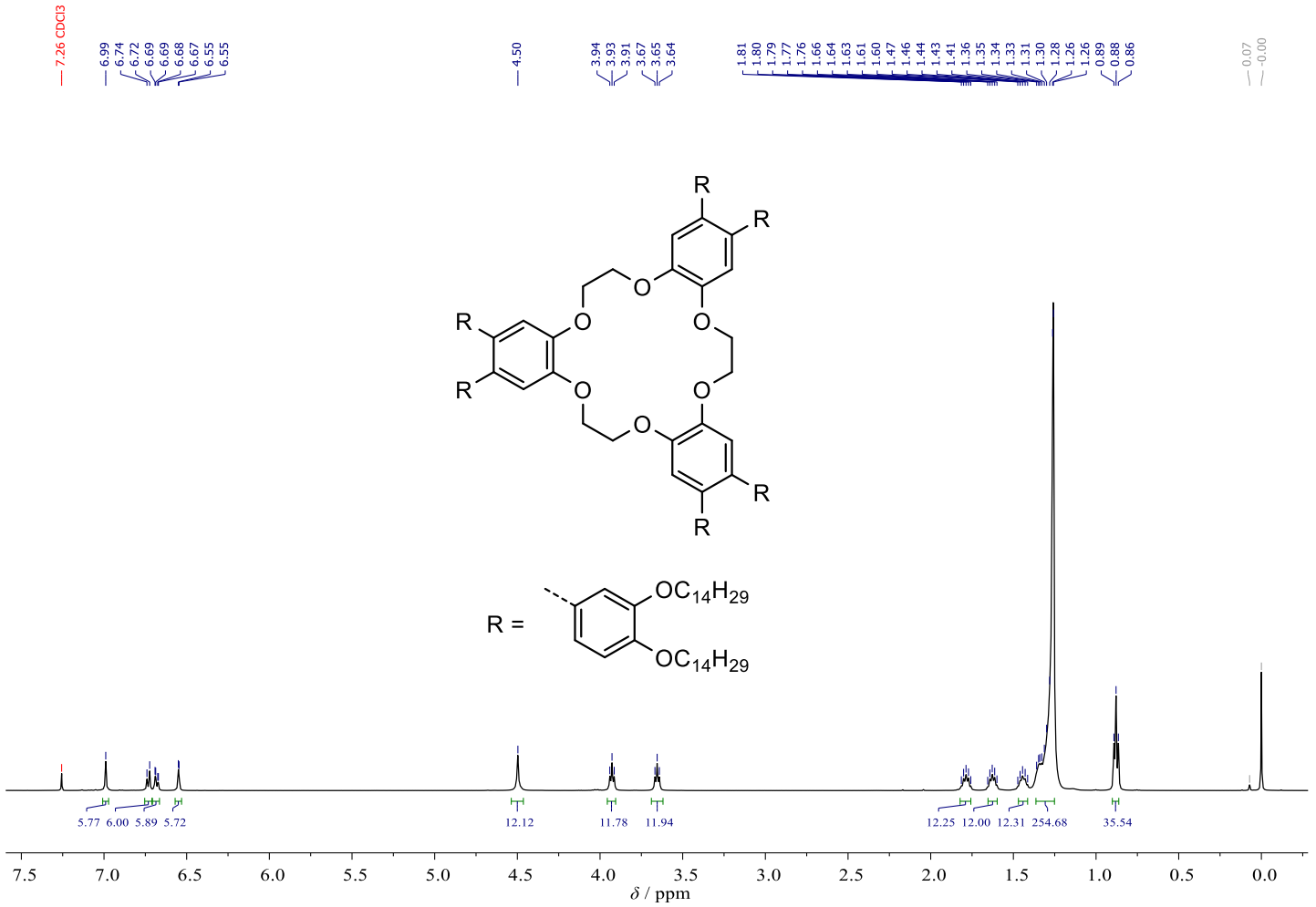
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ Jun01-2011/ 161/ fic
2 Title	Jun01-2011.161.fid
3 Comment	2 Beardsworth BEA059
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	295.6
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	5120
15 Receiver Gain	2890.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.4000
18 Presaturation Frequency	
19 Acquisition Time	0.9962
20 Acquisition Date	2011-06-03T01:47:00
21 Modification Date	2011-06-03T01:47:26
22 Class	
23 Purity	100.00 %
24 Spectrum Quality	0.251
25 Spectrometer Frequency	125.78
26 Spectral Width	32894.7
27 Lowest Frequency	-3871.1
28 Nucleus	13C
29 Acquired Size	32768
30 Spectral Size	65536



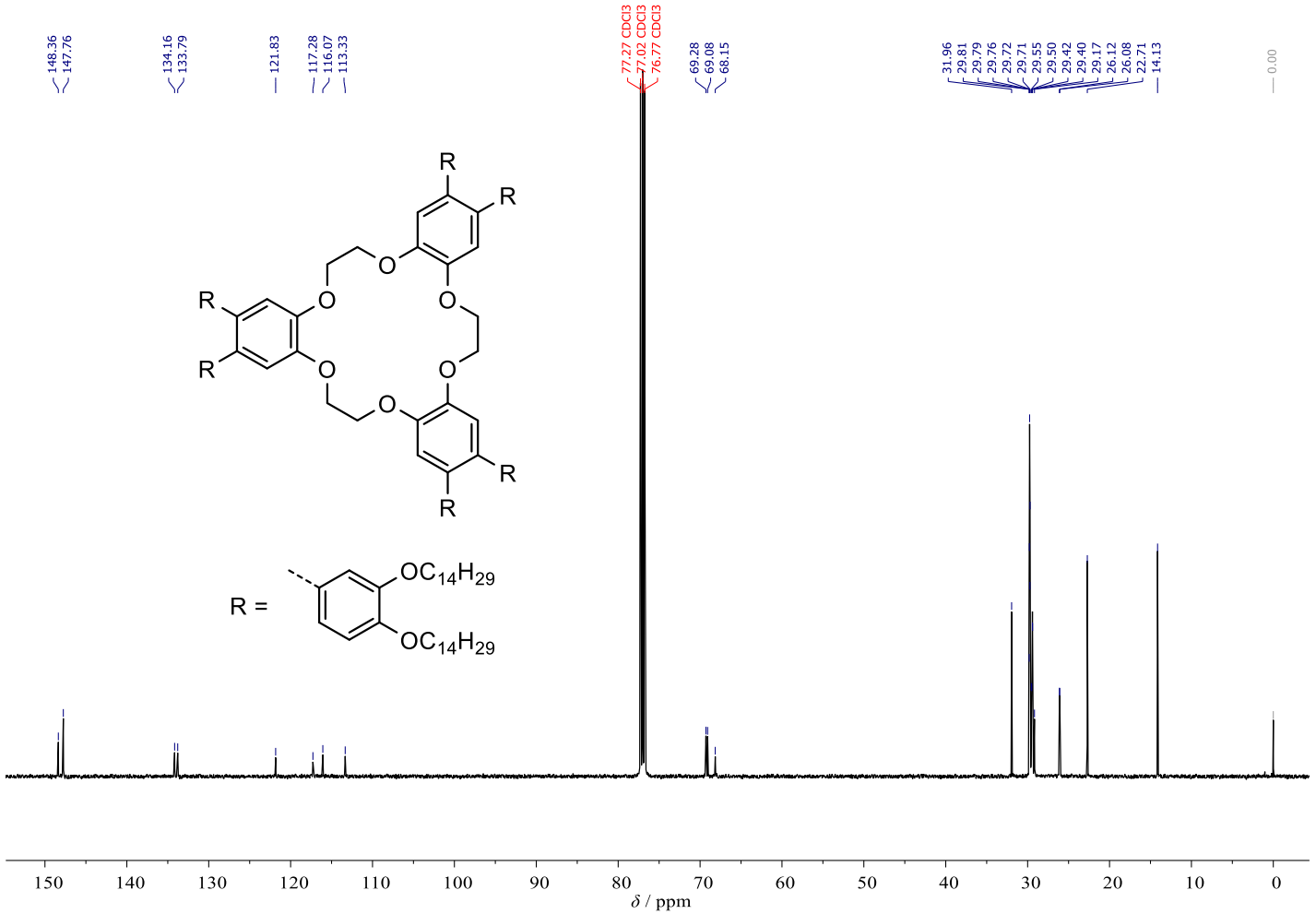
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ May18-2011/ 200/ fic
2 Title	May18-2011.200.fid
3 Comment	2 Beardsworth BEA058
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	296.5
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	64
15 Receiver Gain	456.0
16 Relaxation Delay	2.0000
17 Pulse Width	11.0500
18 Presaturation Frequency	
19 Acquisition Time	1.5860
20 Acquisition Date	2011-05-19T06:30:00
21 Modification Date	2011-05-19T06:30:13
22 Class	
23 Purity	99.98 %
24 Spectrum Quality	-0.687
25 Spectrometer Frequency	500.16
26 Spectral Width	10330.6
27 Lowest Frequency	-2092.4
28 Nucleus	1H
29 Acquired Size	16384
30 Spectral Size	65536



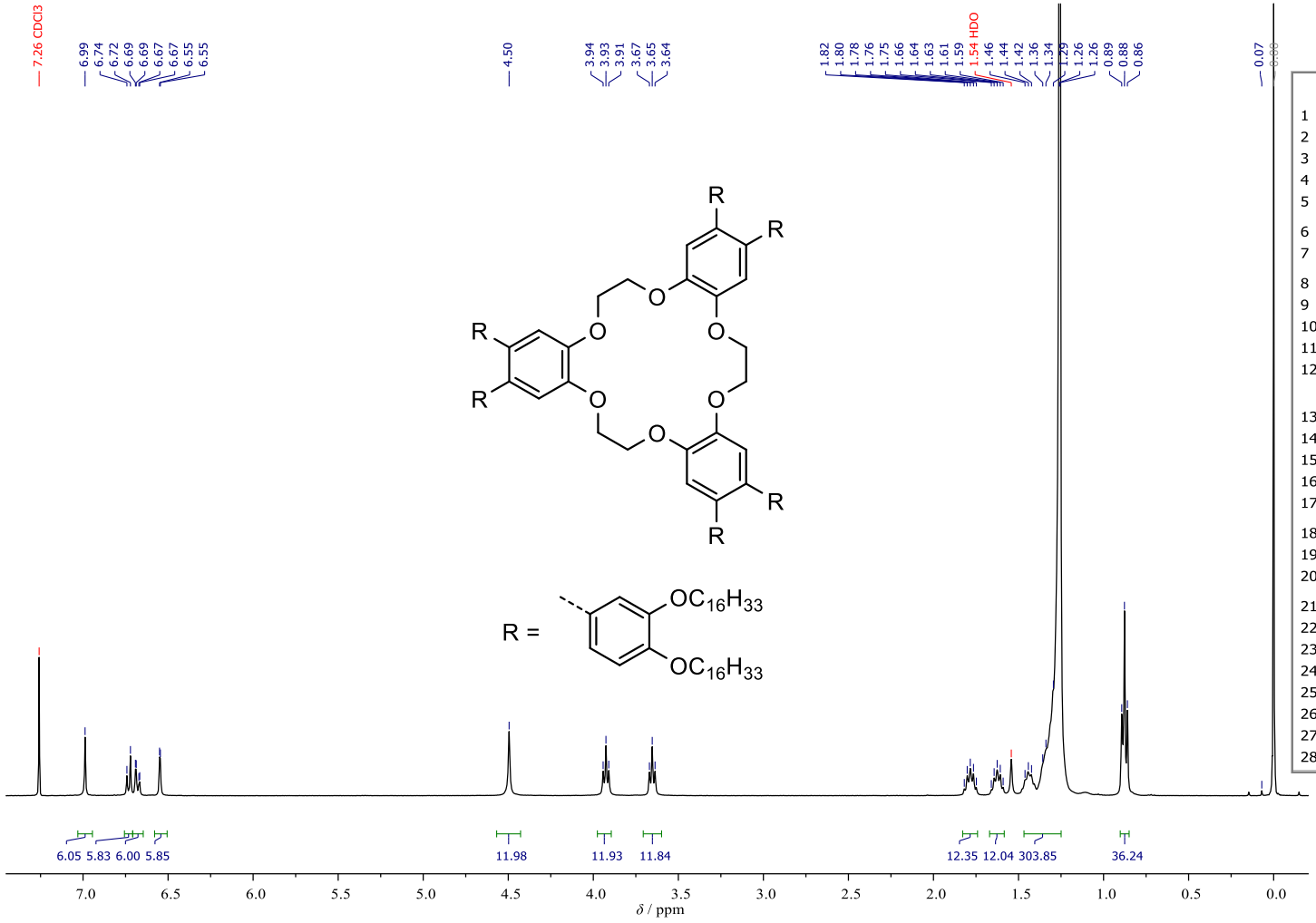
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ May18-2011/ 201/ fic
2 Title	May18-2011.201.fid
3 Comment	2 Beardsworth BEA058
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	298.0
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	2048
15 Receiver Gain	3640.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.4000
18 Presaturation Frequency	
19 Acquisition Time	0.9962
20 Acquisition Date	2011-05-19T07:51:00
21 Modification Date	2011-05-19T08:16:32
22 Class	
23 Purity	100.00 %
24 Spectrum Quality	0.000
25 Spectrometer Frequency	125.78
26 Spectral Width	32894.7
27 Lowest Frequency	-3871.1
28 Nucleus	13C
29 Acquired Size	32768
30 Spectral Size	65536



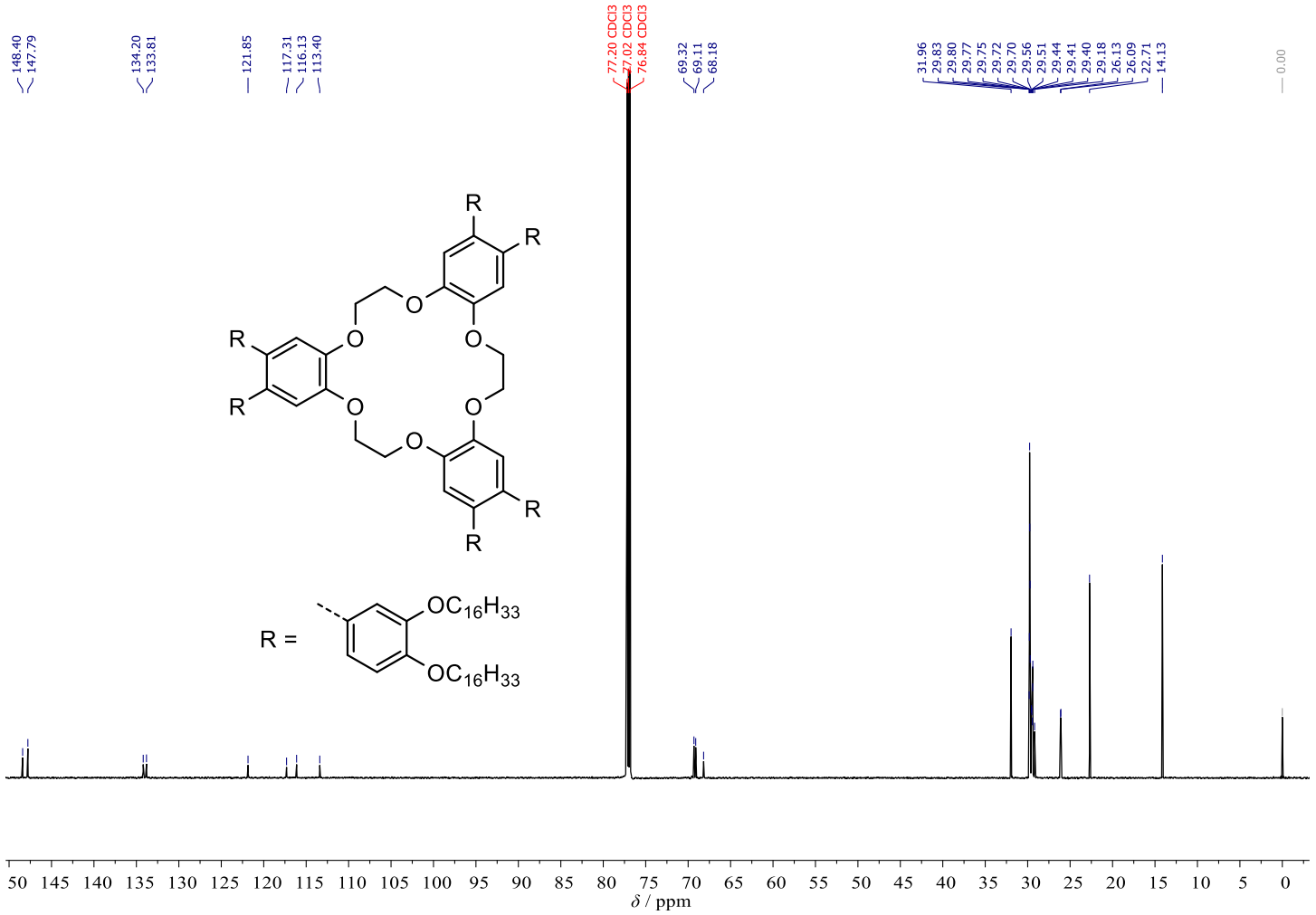
Parameter	Value
Title	Jul20-2016.140.fid
Comment	2 Ehni EHN-C14-ter
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	64
Receiver Gain	114.0
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2016-07-20T14:56:00
Modification Date	2016-07-20T14:57:06
Class	
Purity	99.57 %
Spectrum Quality	-0.687
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2094.5
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



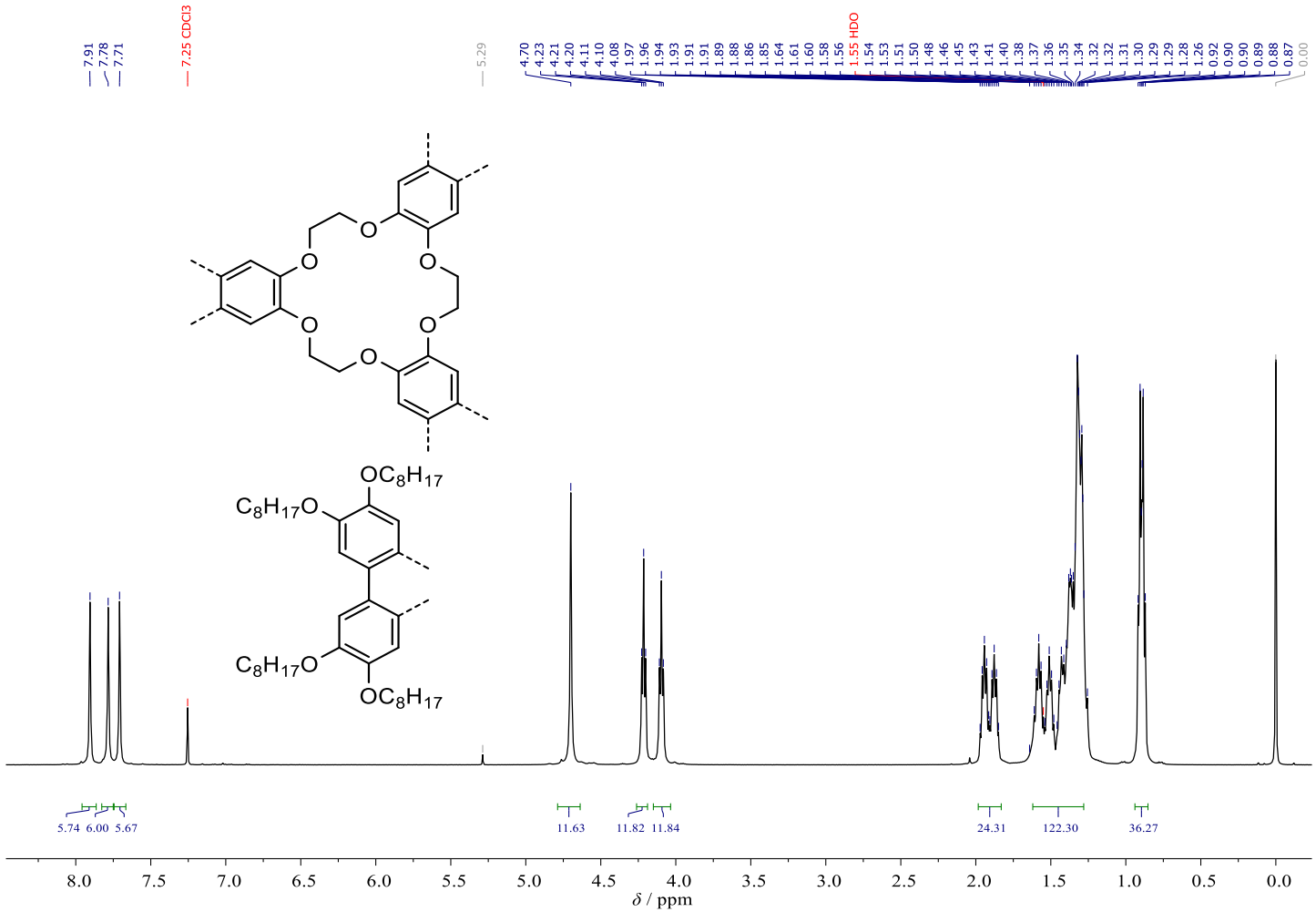
Parameter	Value
Title	Jul20-2016.141.fid
Comment	2 Ehni EHN-C14-ter
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	2048
Receiver Gain	2580.0
Relaxation Delay	2.0000
Pulse Width	10.2000
Presaturation Frequency	
Acquisition Time	0.9962
Acquisition Date	2016-07-20T16:42:00
Modification Date	2016-07-20T16:42:40
Class	
Purity	100.00 %
Spectrum Quality	0.251
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3873.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



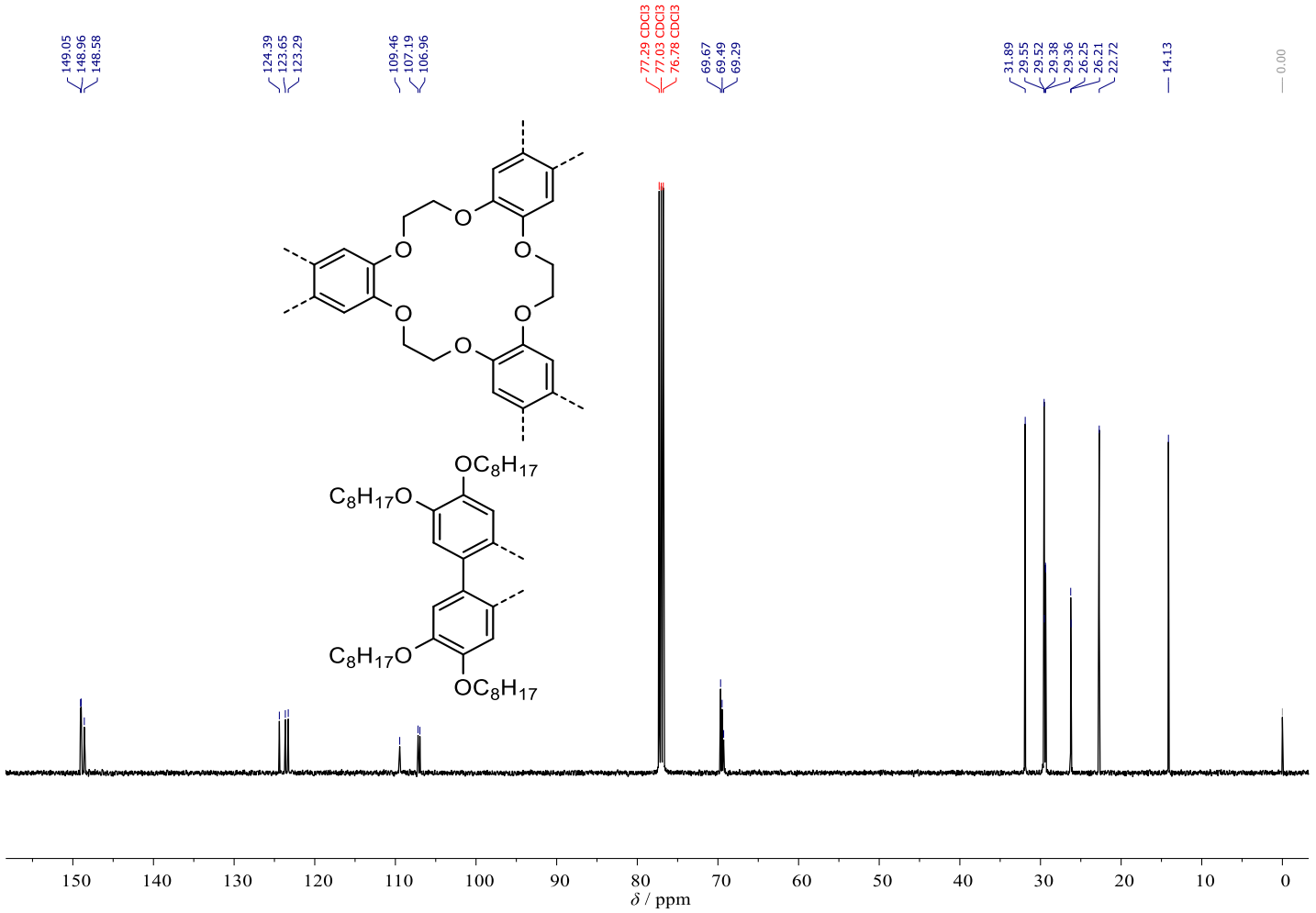
Parameter		Value
1	Title	Jul17-2018.740.fid
2	Comment	02 Ebert Ter-C16-18C6
3	Origin	Bruker BioSpin GmbH
4	Owner	guest
5	Site	
6	Instrument	spect
7	Author	
8	Solvent	CDCl3
9	Temperature	298.0
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 080
13	Number of Scans	32
14	Receiver Gain	182.6
15	Relaxation Delay	1.5000
16	Pulse Width	13.7000
17	Presaturation Frequency	
18	Acquisition Time	4.0894
19	Acquisition Date	2018-07-17T16:51:00
20	Class	
21	Purity	100.00 %
22	Spectrum Quality	0.001
23	Spectrometer Frequency	400.10
24	Spectral Width	8012.8
25	Lowest Frequency	-1545.6
26	Nucleus	1H
27	Acquired Size	32768
28	Spectral Size	65536



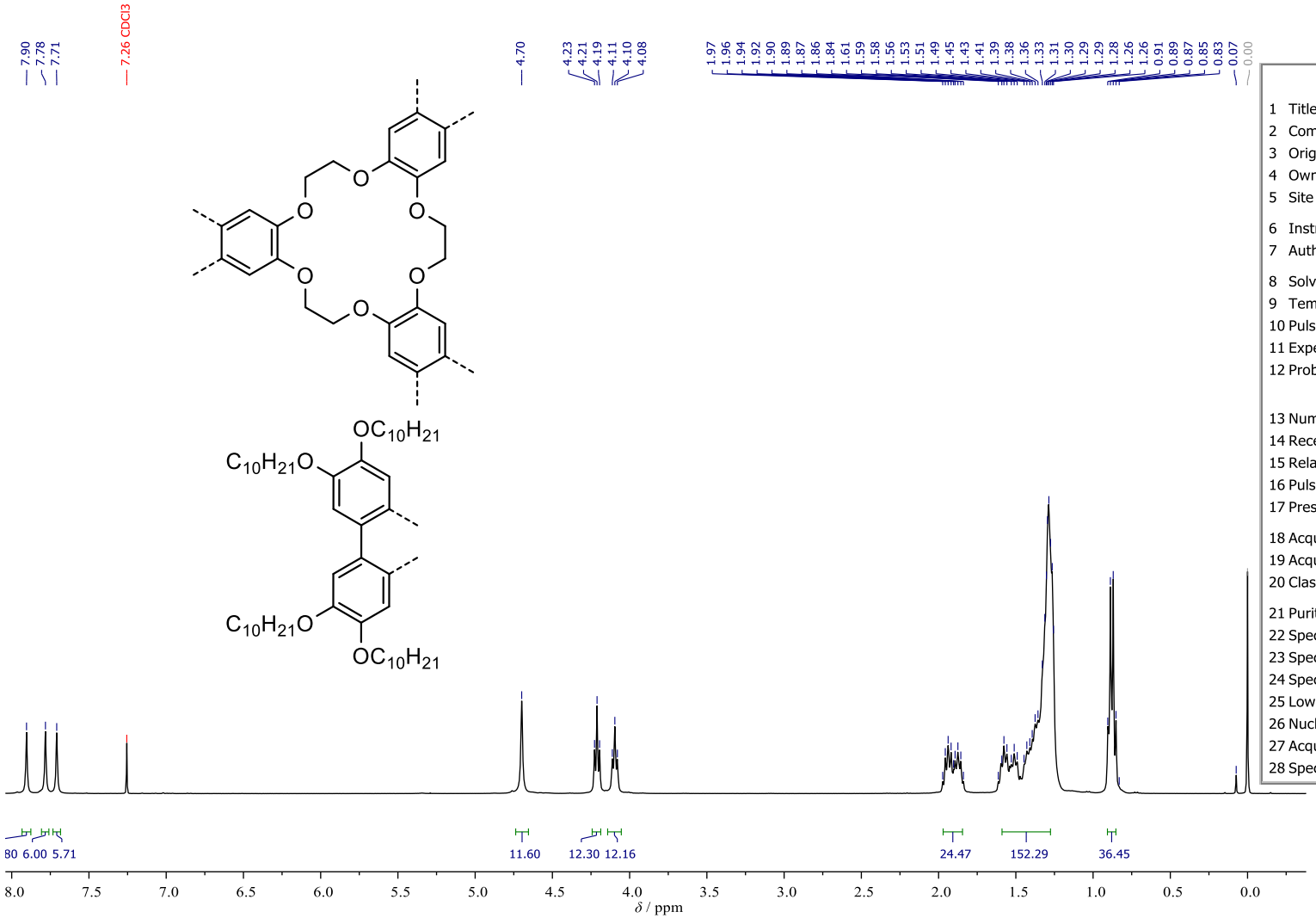
Parameter	Value
Title	Jul26-2016.61.fid
Comment	02 Ehni EHN-C16-Ter
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z123726/ 0024
Number of Scans	2048
Receiver Gain	182.5
Relaxation Delay	4.0000
Pulse Width	12.0000
Presaturation Frequency	
Acquisition Time	0.7864
Acquisition Date	2016-07-26T17:13:00
Modification Date	2016-07-26T17:11:20
Class	
Purity	100.00 %
Spectrum Quality	0.251
Spectrometer Frequency	176.12
Spectral Width	41666.7
Lowest Frequency	-3223.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



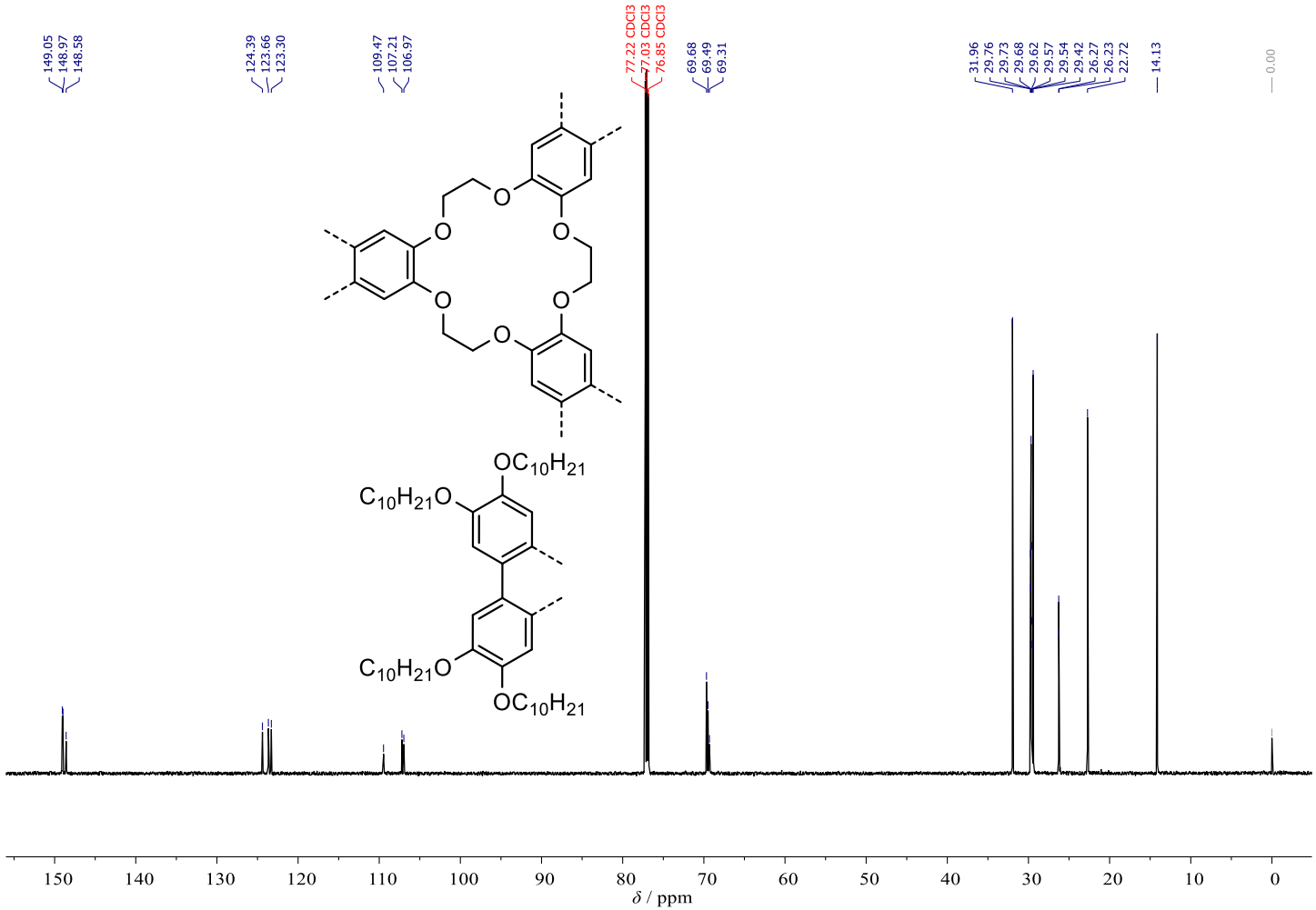
Parameter	Value
Title	Aug31-2016.40.fid
Comment	2 Ehni EHN-C8-tri
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/D Z-GRD Z800701/0072
Number of Scans	32
Receiver Gain	101.0
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2016-08-31T08:59:00
Modification Date	2016-08-31T08:59:31
Class	
Purity	99.92 %
Spectrum Quality	-0.687
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2095.5
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



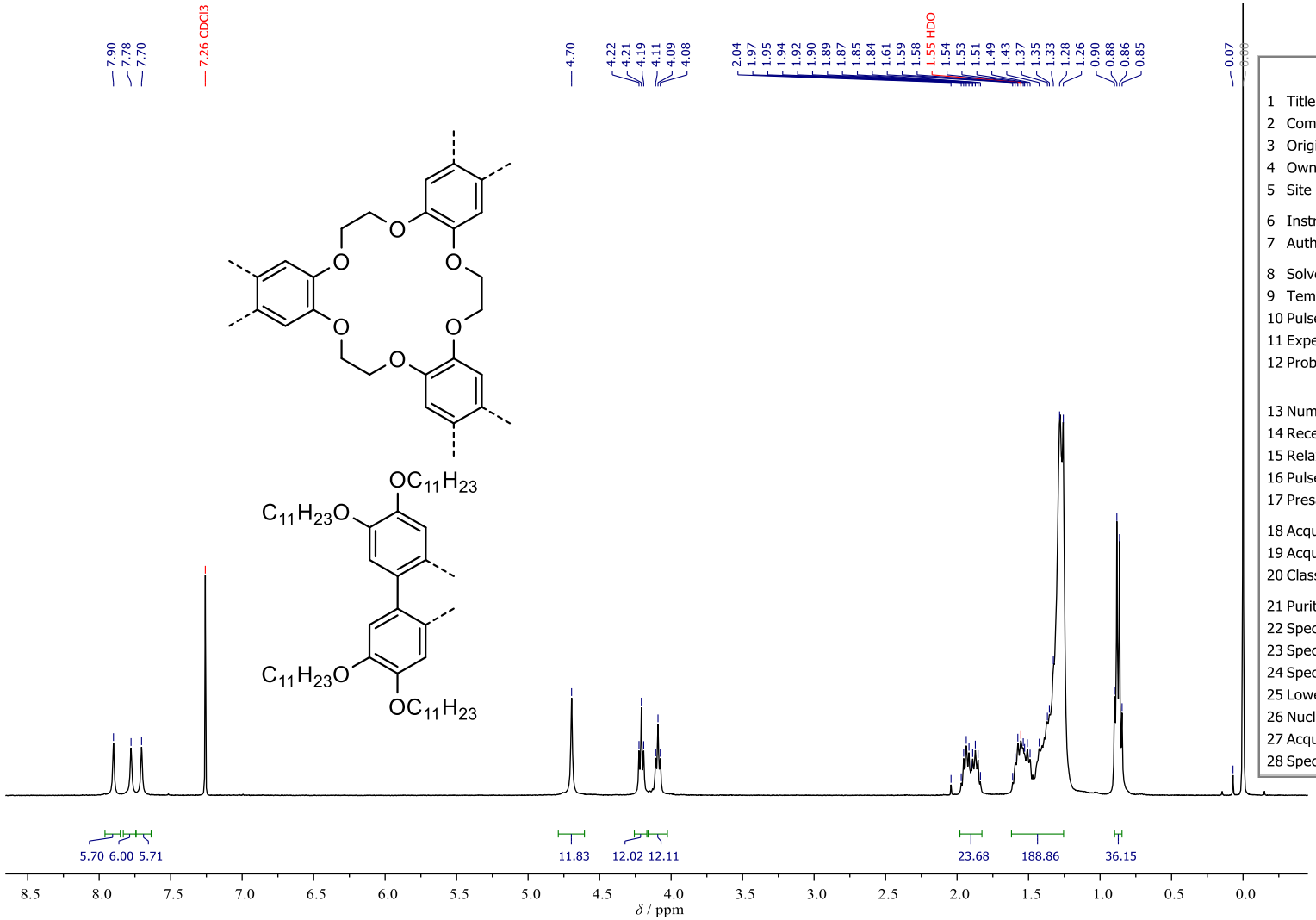
Parameter	Value
Title	Aug31-2016.41.fid
Comment	2 Ehni EHN-C8-tri
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	1024
Receiver Gain	2890.0
Relaxation Delay	2.0000
Pulse Width	10.2000
Presaturation Frequency	
Acquisition Time	0.9962
Acquisition Date	2016-08-31T09:53:00
Modification Date	2016-08-31T09:53:35
Class	
Purity	100.00 %
Spectrum Quality	0.251
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3873.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



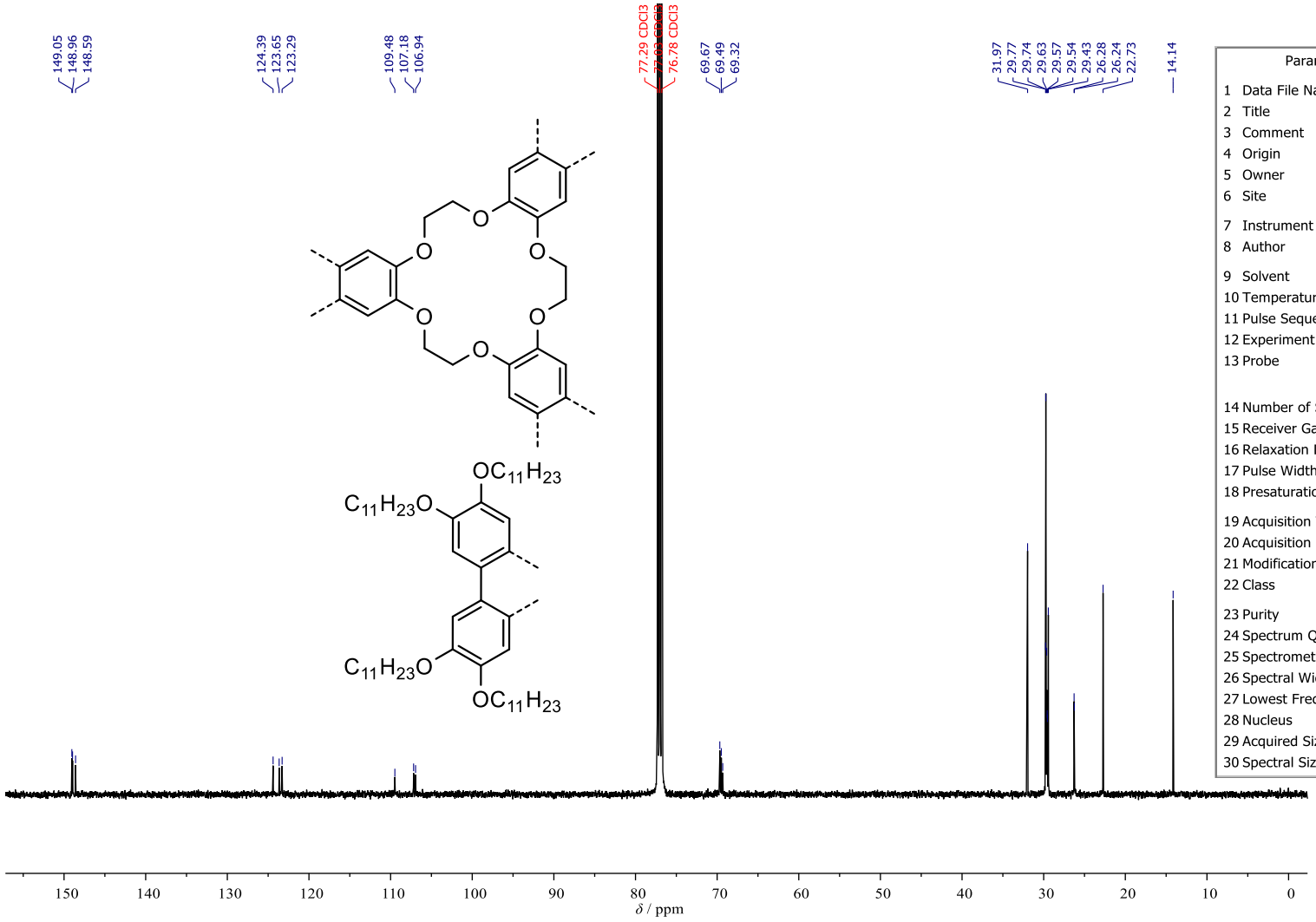
Parameter	Value
1 Title	Jul17-2018.780.fid
2 Comment	02 Ehni EHN-TRI10
3 Origin	Bruker BioSpin GmbH
4 Owner	guest
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	CDCl3
9 Temperature	298.0
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0806
13 Number of Scans	32
14 Receiver Gain	100.2
15 Relaxation Delay	1.0000
16 Pulse Width	13.7000
17 Presaturation Frequency	
18 Acquisition Time	4.0894
19 Acquisition Date	2018-07-17T17:31:00
20 Class	
21 Purity	100.00 %
22 Spectrum Quality	-0.190
23 Spectrometer Frequency	400.10
24 Spectral Width	8012.8
25 Lowest Frequency	-1546.7
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



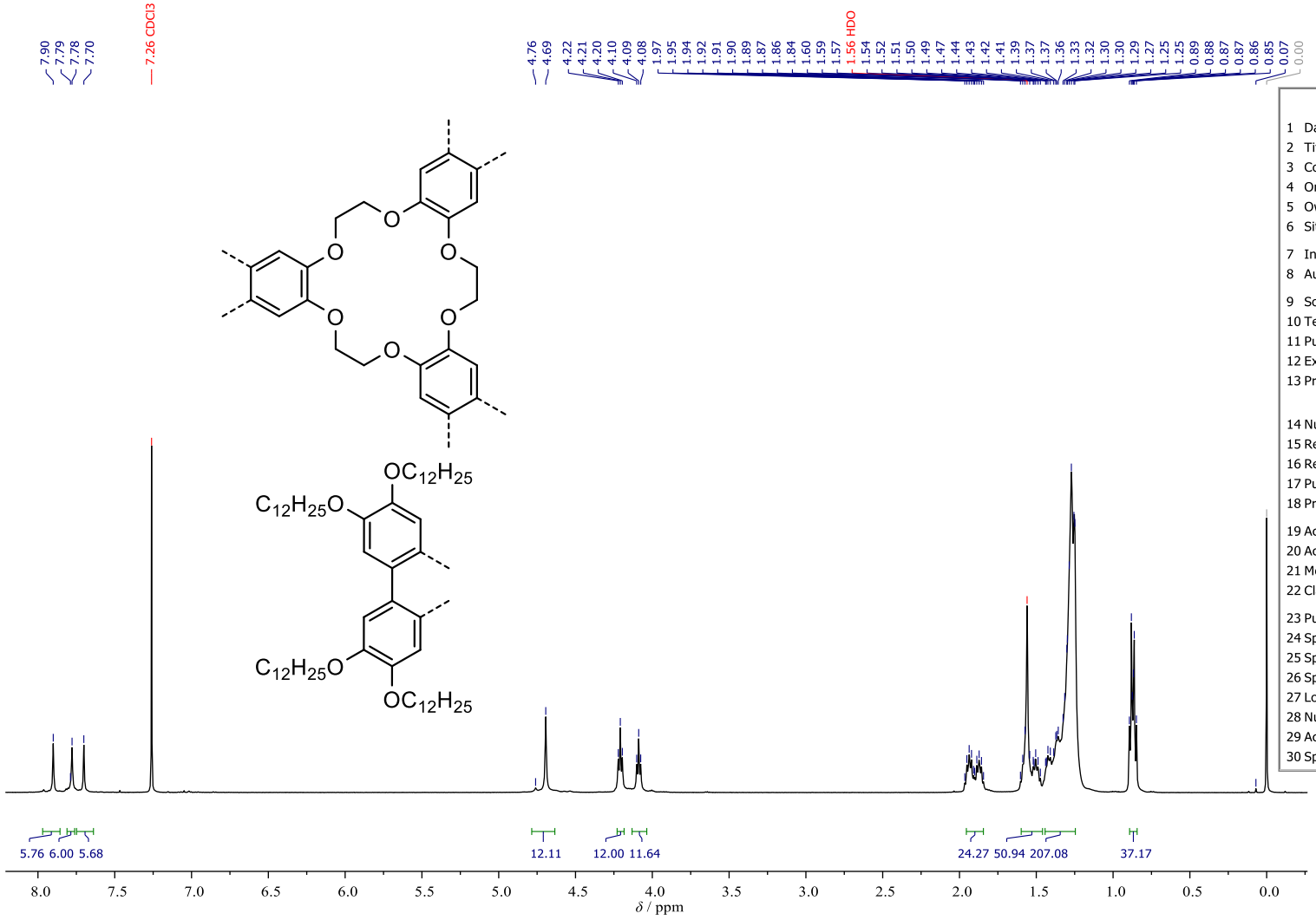
Parameter	Value
Title	Sep05-2016.51.fid
Comment	02 Ehni EHN-C10tri
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z123726/ 0024
Number of Scans	1024
Receiver Gain	182.5
Relaxation Delay	2.0000
Pulse Width	12.0000
Presaturation Frequency	
Acquisition Time	0.7864
Acquisition Date	2016-09-05T19:23:00
Modification Date	2016-09-05T19:20:32
Class	
Purity	99.95 %
Spectrum Quality	0.251
Spectrometer Frequency	176.12
Spectral Width	41666.7
Lowest Frequency	-3224.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



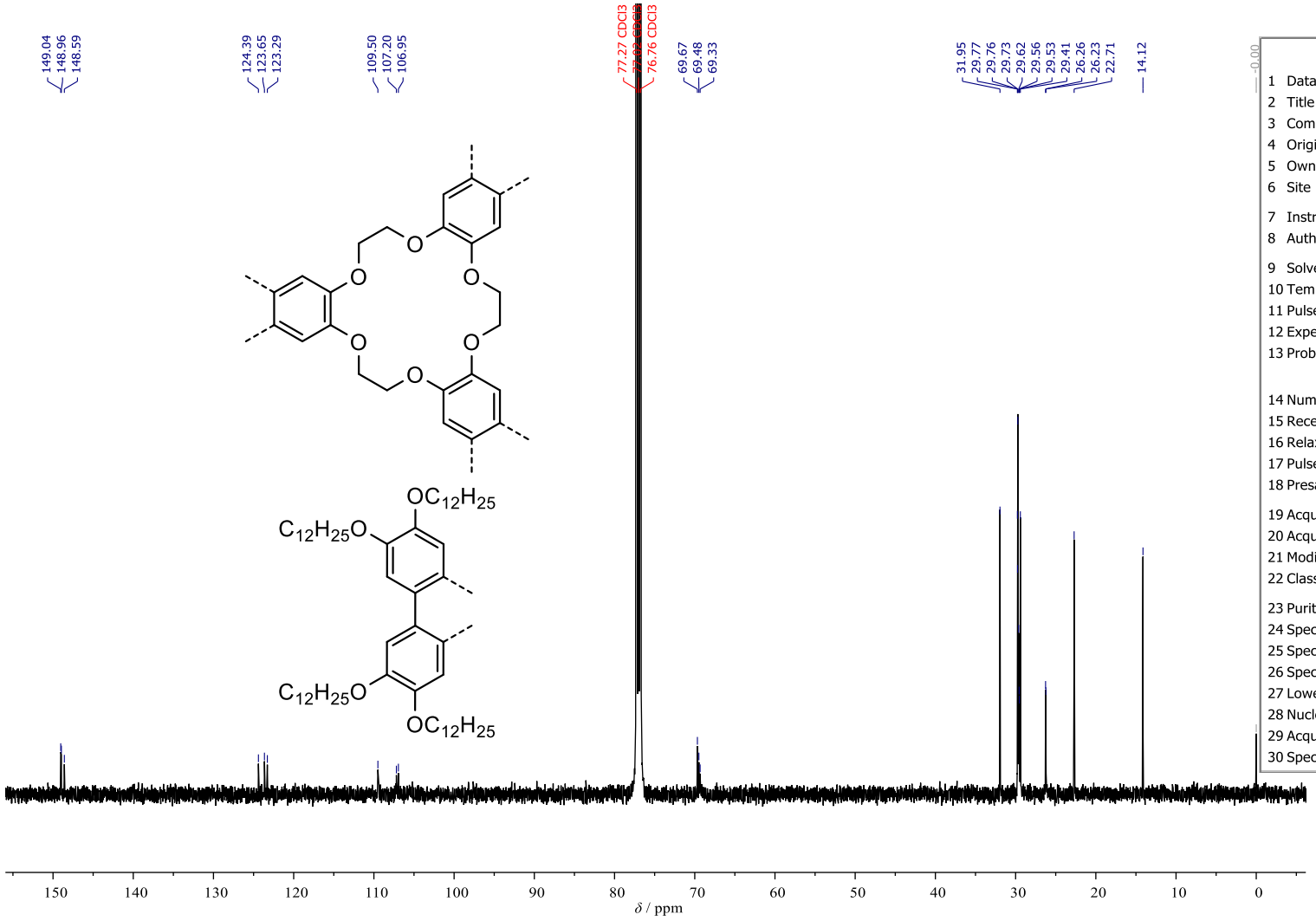
Parameter	Value
1 Title	Jul17-2018.750.fid
2 Comment	02 Ebert Tri-C11-18C6
3 Origin	Bruker BioSpin GmbH
4 Owner	guest
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	CDCl3
9 Temperature	298.0
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0806
13 Number of Scans	16
14 Receiver Gain	205.3
15 Relaxation Delay	1.0000
16 Pulse Width	13.7000
17 Presaturation Frequency	
18 Acquisition Time	4.0894
19 Acquisition Date	2018-07-17T16:56:00
20 Class	
21 Purity	100.00 %
22 Spectrum Quality	-0.190
23 Spectrometer Frequency	400.10
24 Spectral Width	8012.8
25 Lowest Frequency	-1545.7
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



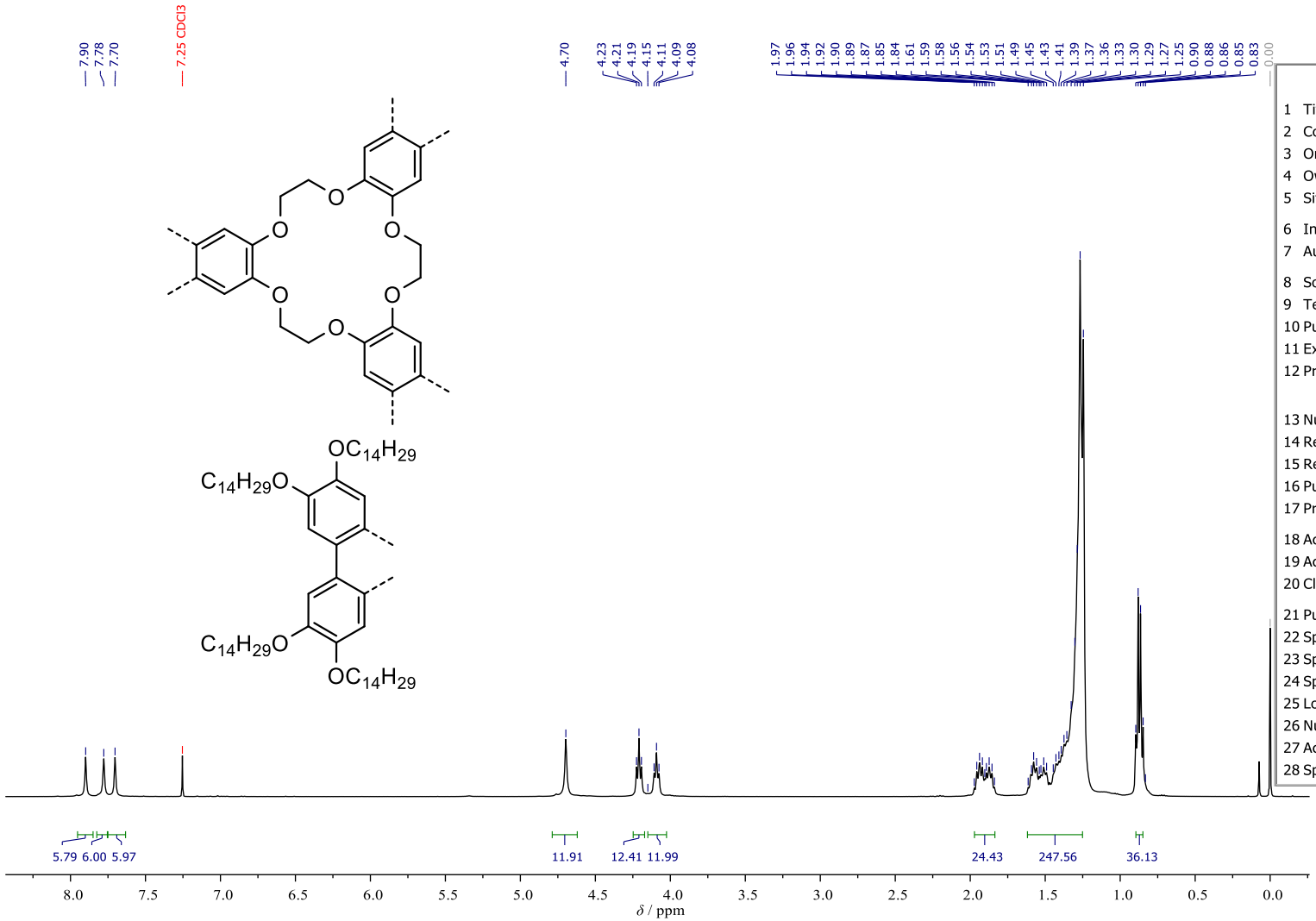
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ Jun09-2011/ 141/ fic
2 Title	Jun09-2011.141.fid
3 Comment	2 Beardsworth BEA066
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	296.8
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	4096
15 Receiver Gain	2890.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.4000
18 Presaturation Frequency	
19 Acquisition Time	0.9962
20 Acquisition Date	2011-06-09T23:29:00
21 Modification Date	2011-06-09T23:29:31
22 Class	
23 Purity	99.69 %
24 Spectrum Quality	0.251
25 Spectrometer Frequency	125.78
26 Spectral Width	32894.7
27 Lowest Frequency	-3871.1
28 Nucleus	13C
29 Acquired Size	32768
30 Spectral Size	65536



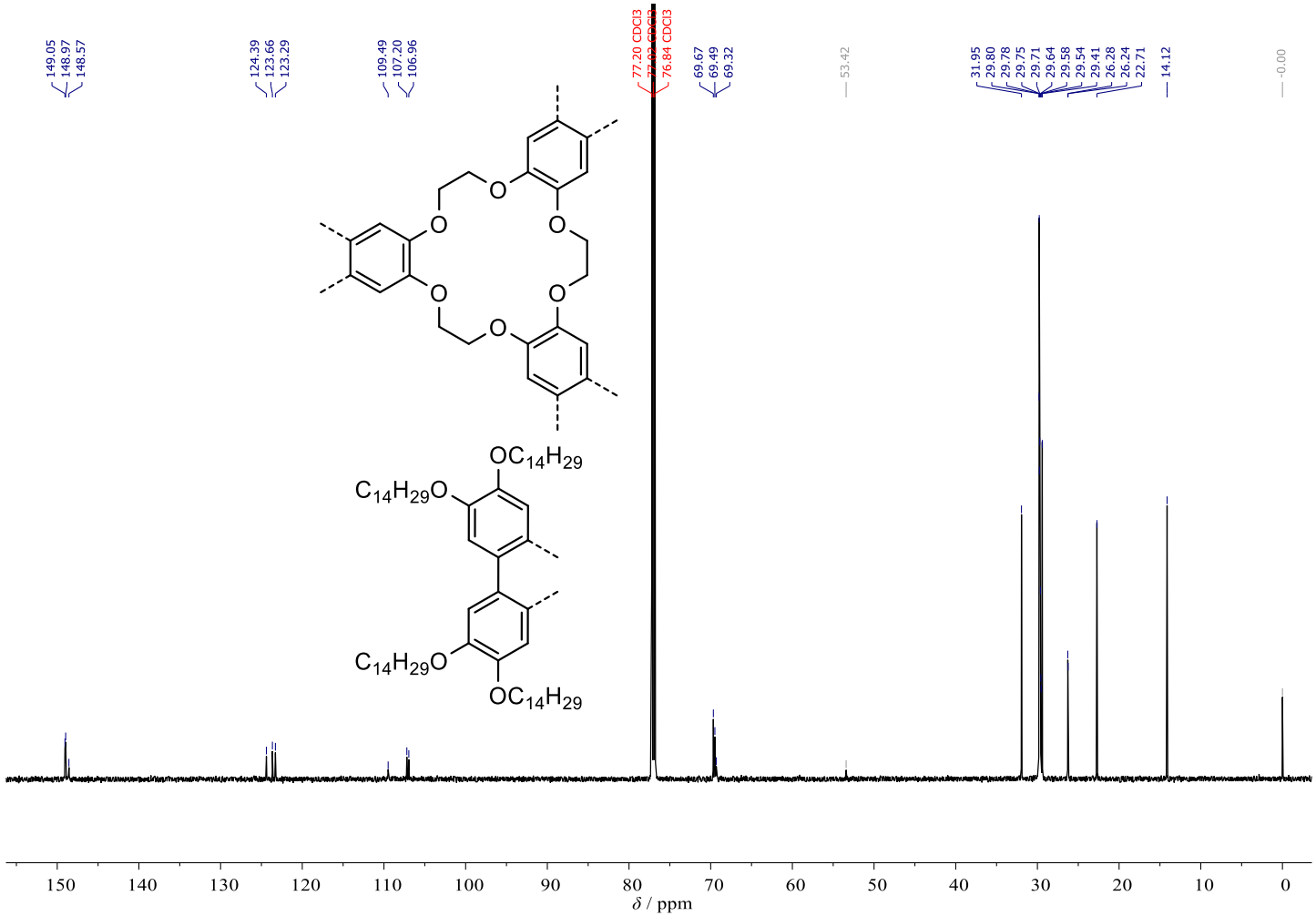
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ May23-2011/ 60/ fic
2 Title	May23-2011.60.fid
3 Comment	2 Beardsworth BEA 060
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	297.1
11 Pulse Sequence	zg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	96
15 Receiver Gain	456.0
16 Relaxation Delay	2.0000
17 Pulse Width	11.0500
18 Presaturation Frequency	
19 Acquisition Time	1.5860
20 Acquisition Date	2011-05-23T18:44:00
21 Modification Date	2011-05-23T18:44:26
22 Class	
23 Purity	99.99 %
24 Spectrum Quality	-0.687
25 Spectrometer Frequency	500.16
26 Spectral Width	10330.6
27 Lowest Frequency	-2092.5
28 Nucleus	1H
29 Acquired Size	16384
30 Spectral Size	65536



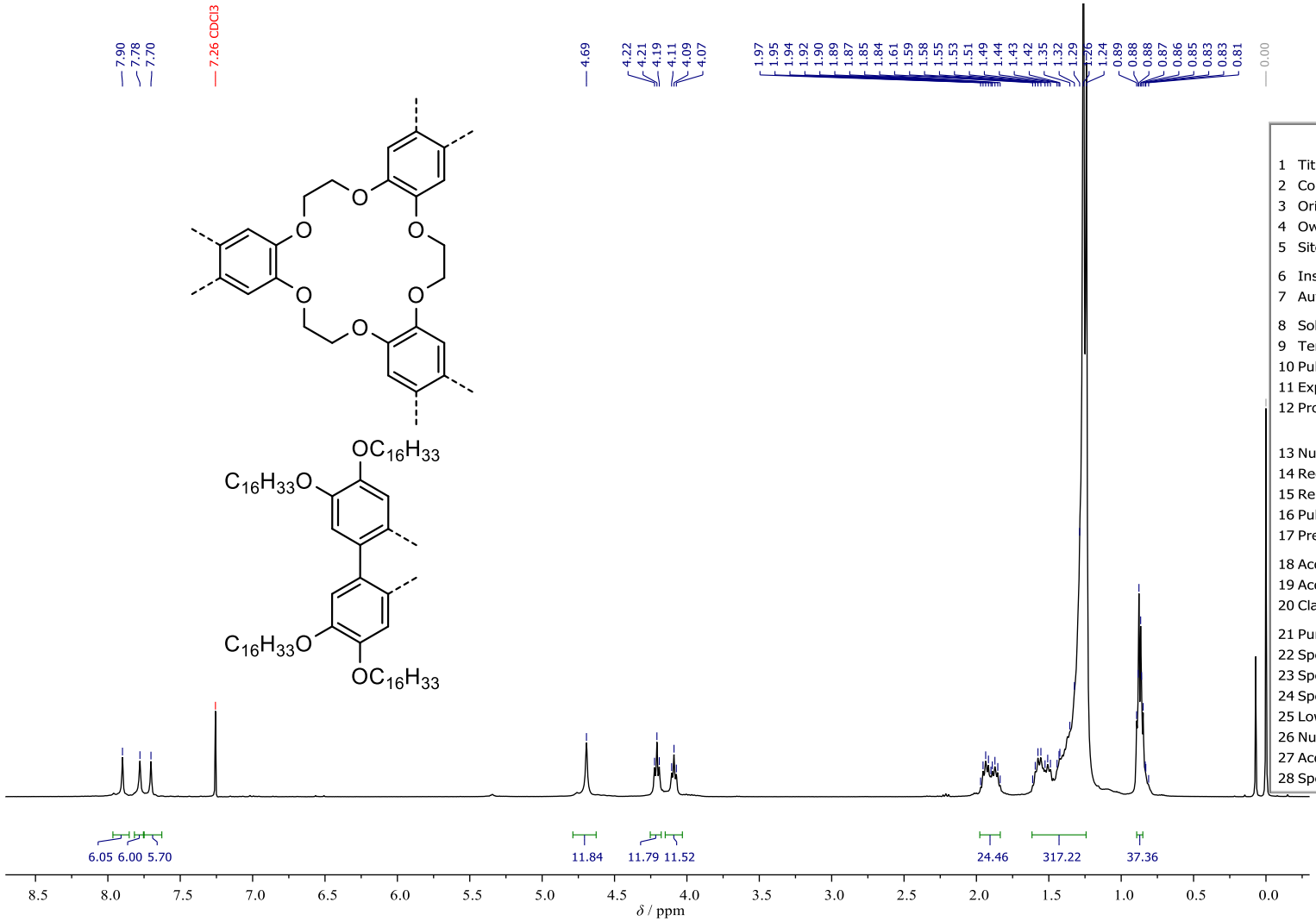
Parameter	Value
1 Data File Name	N:/ data/ BEARDSWORTH_500/ nmr/ May23-2011/ 61/ fic
2 Title	May23-2011.61.fid
3 Comment	2 Beardsworth BEA 060
4 Origin	Bruker BioSpin GmbH
5 Owner	guest
6 Site	
7 Instrument	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	297.7
11 Pulse Sequence	zgpg30
12 Experiment	1D
13 Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
14 Number of Scans	4096
15 Receiver Gain	2890.0
16 Relaxation Delay	2.0000
17 Pulse Width	10.4000
18 Presaturation Frequency	
19 Acquisition Time	0.9962
20 Acquisition Date	2011-05-23T22:14:00
21 Modification Date	2011-05-23T22:14:18
22 Class	
23 Purity	98.94 %
24 Spectrum Quality	0.251
25 Spectrometer Frequency	125.78
26 Spectral Width	32894.7
27 Lowest Frequency	-3872.8
28 Nucleus	13C
29 Acquired Size	32768
30 Spectral Size	65536



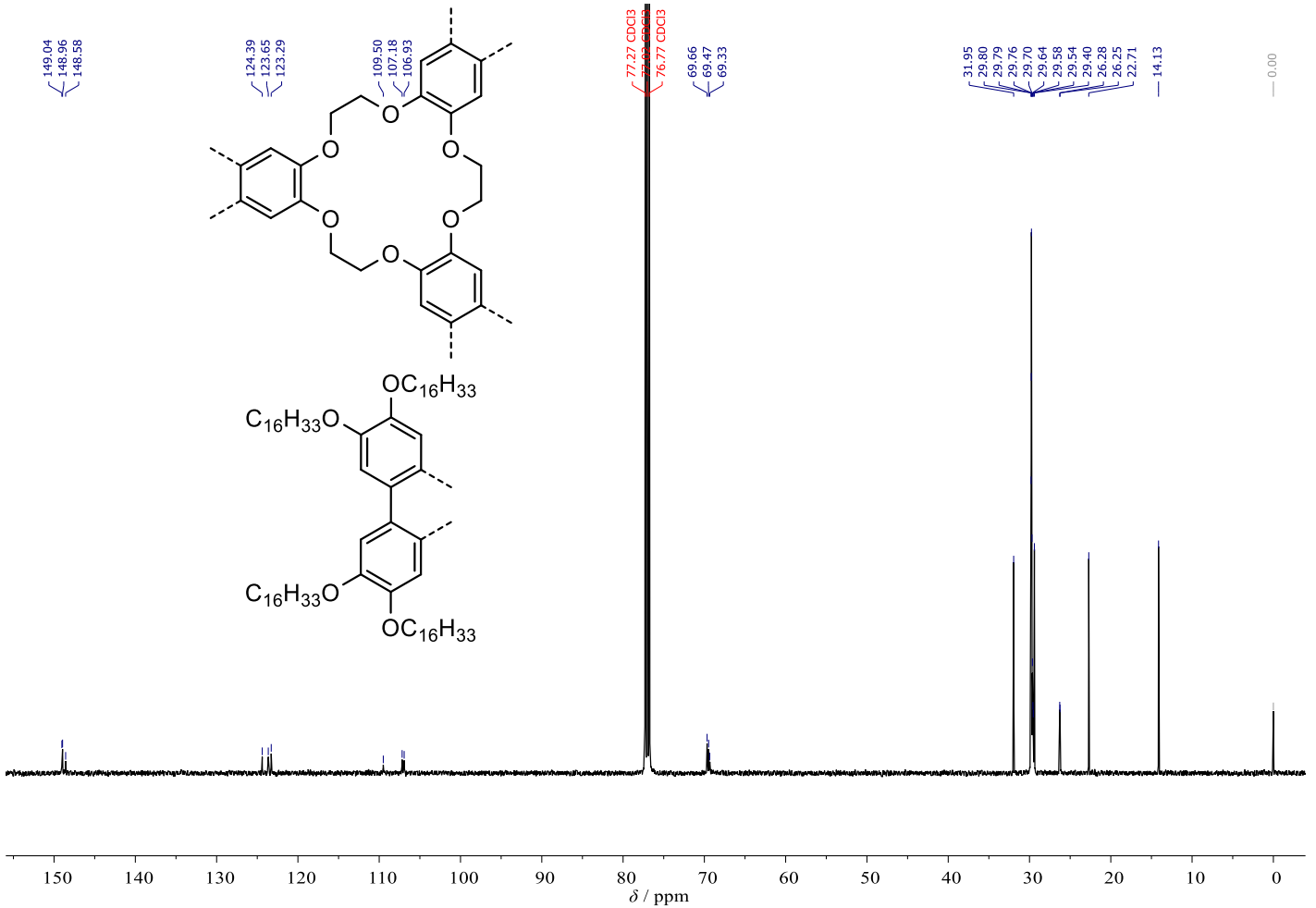
Parameter	Value
1 Title	Jul17-2018.770.fid
2 Comment	02 Ehni EHN-TRI14
3 Origin	Bruker BioSpin GmbH
4 Owner	guest
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	CDCl3
9 Temperature	298.0
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0806
13 Number of Scans	32
14 Receiver Gain	68.9
15 Relaxation Delay	1.0000
16 Pulse Width	13.7000
17 Presaturation Frequency	
18 Acquisition Time	4.0894
19 Acquisition Date	2018-07-17T17:25:00
20 Class	
21 Purity	100.00 %
22 Spectrum Quality	0.000
23 Spectrometer Frequency	400.10
24 Spectral Width	8012.8
25 Lowest Frequency	-1547.3
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



Parameter	Value
Title	Sep15-2016.12.fid
Comment	02 Ehni EHN-C14tri CDCl3 13C{1H}
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z123726/ 0024
Number of Scans	722
Receiver Gain	182.5
Relaxation Delay	5.0000
Pulse Width	12.0000
Presaturation Frequency	
Acquisition Time	0.7864
Acquisition Date	2016-09-16T12:05:00
Modification Date	2016-09-16T13:07:11
Class	
Purity	99.68 %
Spectrum Quality	0.000
Spectrometer Frequency	176.12
Spectral Width	41666.7
Lowest Frequency	-3224.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
1 Title	Jul17-2018.760.fid
2 Comment	02 Ehni EHN-TRI16
3 Origin	Bruker BioSpin GmbH
4 Owner	guest
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	CDCl3
9 Temperature	298.0
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0806
13 Number of Scans	64
14 Receiver Gain	114.4
15 Relaxation Delay	1.0000
16 Pulse Width	13.7000
17 Presaturation Frequency	
18 Acquisition Time	4.0894
19 Acquisition Date	2018-07-17T17:19:00
20 Class	
21 Purity	100.00 %
22 Spectrum Quality	0.000
23 Spectrometer Frequency	400.10
24 Spectral Width	8012.8
25 Lowest Frequency	-1546.1
26 Nucleus	1H
27 Acquired Size	32768
28 Spectral Size	65536



Parameter	Value
Title	Sept19-2016.51.fid
Comment	2 Ehni EHN-C16tri CDCl3 13C{1H}
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Instrument	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	1280
Receiver Gain	2580.0
Relaxation Delay	5.0000
Pulse Width	10.2000
Presaturation Frequency	
Acquisition Time	0.9962
Acquisition Date	2016-09-19T13:30:00
Modification Date	2016-09-19T14:46:07
Class	
Purity	100.00 %
Spectrum Quality	0.251
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3872.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

References

1 T. Wöhrle, J. Kirres, M. Kaller, M. Mansueto, S. Tussetschläger and S. Laschat, *J. Org. Chem.*, 2014, **79**, 10143–10152.