Electronic Supplementary Material (ESI) for Chemical Communications.

SUPPORTING INFORMATION

Palladium-catalyzed Cocyclotrimerization of Arynes with a Pyramidalized Alkene

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CONTENTS

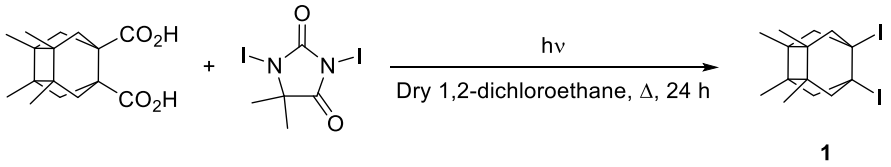
1) GENERAL METHODS

All reactions were carried out under argon atmosphere using oven-dried glassware. Solvents were dried by distillation from a drying agent. Commercial reagents were purchased from ABCR GmbH, Aldrich Chemical Co., or Stream Chemicals Inc, and were used without any further purification. *n*-BuLi was used from solution in hexanes (2.40 M). Flash chromatography was performed on Merck silica gel 60 F254. ^{1}H and ^{13}C NMR spectra were recorded at 300, 400 and 500 MHz (Varian Mercury instrument). Every experiment was performed at 298 K unless otherwise indicated. Low resolution electron impact mass spectra (EI-LRMS) were determined at 70 eV on a HP-5988A instrument. High resolution mass spectra (HRMS) were obtained on a Micromass Autoespec spectrometer.

2) EXPERIMENTAL PROCEDURES AND DATA

Synthesis of starting materials 1, 11, 15, 16, 19 and 20

Diiodoalkane **1** was prepared following a modification of a reported procedure.[1]



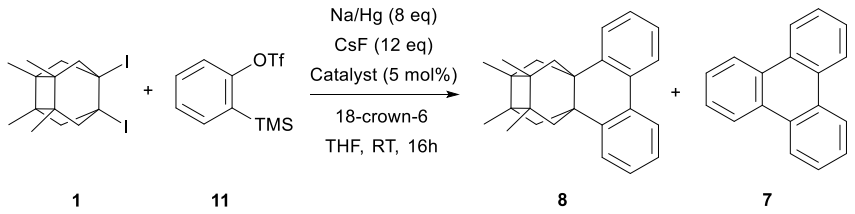
Scheme S1

To a solution of 3,4,8,9-tetramethyltetracyclo[4.4.0.0^{3,9}.0^{4,8}]decane-1,6-dicarboxylic acid (374 mg, 1.34 mmol) in dry 1,2-dichloroethane (20 mL) was added 1,3-diiodo-5,5-dimethylhydantoin (1.42, 3.74 mmol). The resulting orange solution was irradiated (2 x 60 W tungsten bulb) at reflux for 22 h. The suspension was cooled to room temperature and filtered. The solid was washed with dichloromethane (2 x 10 mL). The combined filtrate and washings were washed with 10% aqueous solution of $NaHSO_3$ (3 x 10 mL). The organic layer was washed with 2 N NaOH (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under vacuum to obtain 557 g of a yellow mixture of starting **1** and some anhydride derived from **1**. Purification by column chromatography (silica gel, *n*-hexane/ethyl acetate) gave **1** (eluted with *n*-hexane) as an off-white solid (235 mg, 40% yield). Further elution with *n*-hexane/ethyl acetate 95/5 furnished anhydride of the starting dicarboxylic acid (60 mg).

Aryne precursors **11, 15, 16, 19** and **20** were prepared following previously reported procedures.[2]

General procedure for cocyclotrimerization of arynes and a pyramidalized alkene

Synthesis of adduct 8

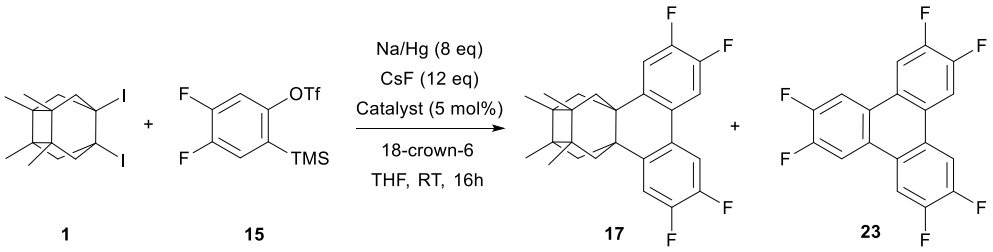


Scheme S2

Compound **1** (20 mg, 0.045 mmol), Na/Hg (5% Na, 168 mg, 0.361 mmol), CsF (84 mg, 0.552 mmol), 18-Crown-6 (73 mg, 0.276 mmol), and $Pd(PPh_3)_4$ (15 mg, 0.013 mmol) were placed in a oven-dried shlenck under Ar atmosphere. Then, solution of **11** (83 mg, 0.276 mmol) in THF (4 mL) was added. The mixture was stirred at room temperature for 16h. After that time, the reaction mixture was filtrated through a celite plug, and purified on column chromatography (hexanes) to yield **8** as a white solid (12 mg, 79%). Triphenylene (**7**)[3] was isolated as byproduct (42 mg, 67% from precursor **11**) Crystallization from slow diffusion of hexane in chloroform afforded **8** as colorless crystals.

Data for compound **8**: ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (12H, s, Me), 1.26 (4H, d, *J* = 11.8 Hz, CH_2), 2.24 (4H, d, *J* = 11.8 Hz, CH_2), 7.21 (4H, m, CH Ar), 7.22 (2H, m, CH Ar), 7.94 (2H, m, CH Ar). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 15.6 (4C, Me), 44.8 (2C, C), 45.8 (4C, C), 50.1 (4C, CH_2), 122.2 (2C, CH Ar), 125.9 (2C, CH Ar), 127.3 (2C, CH Ar), 127.6 (2C, CH Ar), 130.9 (2C, C Ar), 139.6 (2C, C Ar). EM (EI+), m/z (%): 340 (M+, 10). HRMS: for $C_{26}H_{28}$ calculated 340,2129, observed 340,2201. M. P. 249.2-252.0 ºC.

Synthesis of adduct 17



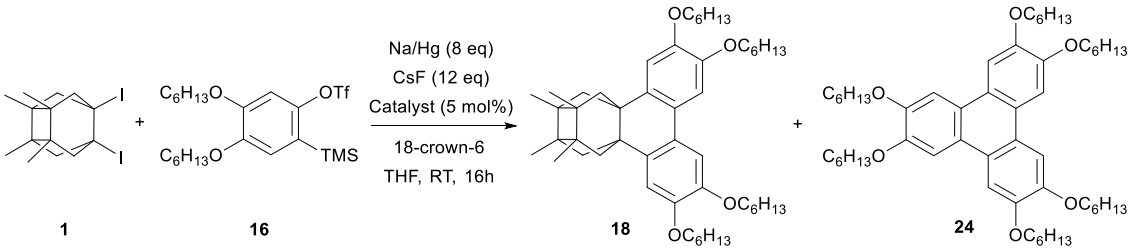
Scheme S3

Compound **1** (20 mg, 0.045 mmol), Na/Hg (5% Na, 164 mg, 0.361 mmol), CsF (84 mg, 0.552 mmol), 18-Crown-6 (73 mg, 0.276 mmol), and $Pd(PPh_3)_4$ (15 mg, 0.013 mmol) were placed in an oven-dried shlenck under Ar atmosphere. Then, solution of **15** (92 mg, 0.276 mmol) in THF (4.5 mL) was added. The mixture was stirred at room temperature for 16h. After that time, the reaction mixture was filtrated through a celite plug, and purified on column chromatography (hexane) to yield **17** as a white solid (10 mg, 53%). Hexafluorotriphenylene **23**[4] was isolated as byproduct (8 mg, 9% from precursor **15**).

Data for compound **17**: ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.04 (12H, s, Me), 1.12 (4H, d, *J* = 12.0 Hz, CH_2), 2.22 (4H, d, *J* = 11.8 Hz, CH_2), 7.09 (2H, dd, *J* = 12.4, 8.3 Hz, CH Ar), 7.45 (2H, dd, *J* = 12.3, 8.1 Hz, CH Ar). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 15.65 (4C, Me), 44.73 (2C, C), 45.94 (4C, C), 50.28 (4C, CH_2), 111.13 (2C, d, *J* = 18.3 Hz, CH Ar), 116.26 (2C, d, *J* = 17.5 Hz, CH Ar), 126.91 (2C, C Ar), 136.92 (2C, C Ar), 147.93 (2C, dd, *J* = 92.3, 13.0 Hz, CF), 151.20 (2C, dd, *J* = 94.7, 12.2 Hz, CF). ^{19}F NMR ($CDCl_3$, 282 MHz) δ -139.33 – -139.83 (m), -141.50 – -141.99 (m). EM (EI+), m/z (%): 412 (M^+, 100).

HRMS (APCI (M+1)): for $C_{26}H_{24}F_2$ calculated 413.1887, observed 413.1882. M. P. 268.8-270.2 ºC.

Synthesis of adduct 18

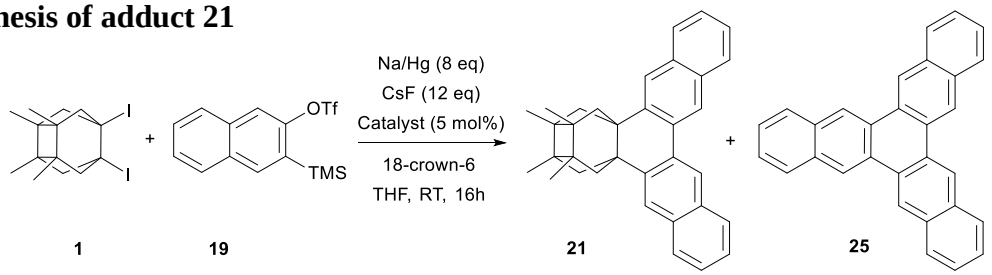


Scheme S4

Compound **1** (20 mg, 0.045 mmol), Na/Hg (5% Na, 164 mg, 0.361 mmol), CsF (84 mg, 0.552 mmol), 18-Crown-6 (73 mg, 0.276 mmol), and $Pd(PPh_3)_4$ (15 mg, 0.013 mmol) were placed in an oven-dried shlenck under Ar atmosphere. Then, solution of **16** (138 mg, 0.276 mmol) in THF (4.5 mL) was added. The mixture was stirred at room temperature for 16h. After that time, the reaction mixture was filtrated through a celite plug, and purified on column chromatography (two columns were necessary, first 5:9.5 Et_2O/hexane and second 1:1 CH_2Cl_2/hexane) to yield **18** as a white solid (14 mg, 42%). Hexyloxytriphenylene **24**[5] was detected as byproduct in the ^{1}H-NMR crude (3% from precursor **16**).

Data for compound **18**: ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.98 – 0.84 (12H, m, Me), 1.04 (12H, s, Me), 1.15 (4H, d, *J* = 12.1 Hz, CH_2), 1.58 – 1.23 (24H, m, CH_2), 1.92 – 1.73 (8H, m, CH_2), 2.18 (4H, d, *J* = 11.8 Hz, CH_2), 4.13 – 3.90 (8H, m, OCH_2), 6.79 (4H, s, CH Ar). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.21 (4C, Me), 15.81 (4C, Me), 22.81(4C, CH_2), 25.94 (4C, CH_2), 29.60 (4C, CH_2), 31.82 (4C, CH_2), 44.90 (2C, C), 45.71 (4C, C), 49.99 (4C, CH_2), 69.74 (2C, OCH_2), 70.19 (2C, OCH_2), 109.01 (2C, CH Ar), 113.63 (2C, CH Ar), 124.68 (2C, C Ar), 132.62 (2C, C Ar), 147.73 (2C, C Ar), 148.86 (2C, C Ar). EM (MALDI-TOF), m/z (%); 740 (100), 741 (55), 742 (12), 743 (9), 744 (3); calculated isotopic ratio: 740 (100), 741 (57), 742 (17), 743 (3), 744 (1). HRMS (MALDI-TOF) for $C_{50}H_{76}O_4$, calculated 740.574, observed 740.575. M. P. 72.5-73.8 ºC.

Synthesis of adduct 21

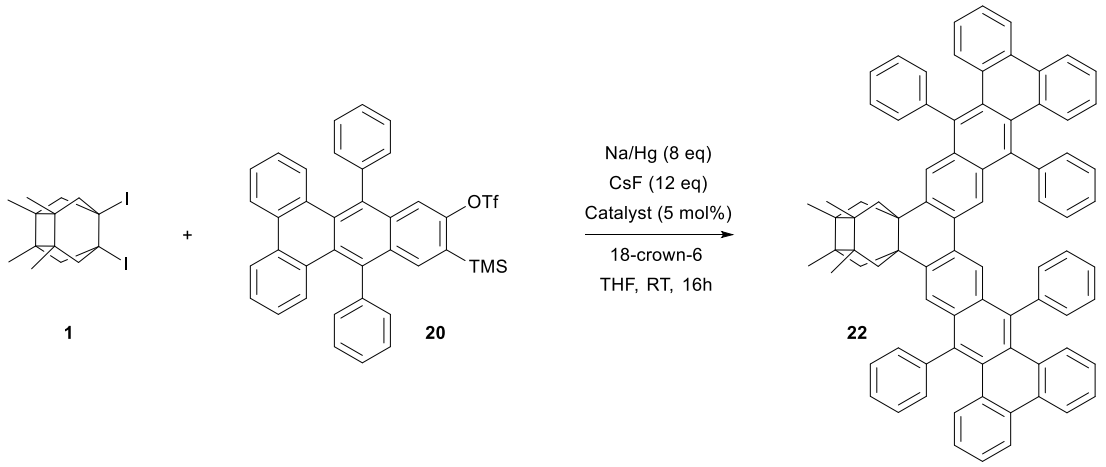


Scheme S5

Compound **1** (20 mg, 0.045 mmol), Na/Hg (5% Na, 168 mg, 0.361 mmol), CsF (84 mg, 0.552 mmol), 18-Crown-6 (73 mg, 0.276 mmol), and $Pd(PPh_3)_4$ (15 mg, 0.013 mmol) were placed in a oven-dried shlenck under Ar atmosphere. Then, solution of **19** (96 mg, 0.276 mmol) in THF (4 mL) was added. The mixture was stirred at room temperature for 16h. After that time, the reaction mixture was filtrated through a celite plug, and purified on column chromatography (hexanes) to yield **21** as a white solid (11 mg, 57%). Trinaphthylene (**25**)[6] was isolated as byproduct (28 mg, 26% from precursor **19**). Crystallization from slow diffusion of hexane in chloroform afforded **21** as colorless crystals.

Data for compound **21**: ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.12 (12H, s, Me), 1.48 (4H, d, *J* = 11.8 Hz, CH_2), 2.34 (4H, d, *J* = 11.8 Hz, CH_2), 7.39 (4H, m, CH Ar), 7.77 (2H, m, CH Ar), 7.89 (4H, m, CH Ar), 8.66 (2H, s, CH Ar). ^{13}C NMR ($CDCl_3$, 101 MHz): δ 15.7 (4C, Me), 45.1 (2C, C), 45.8 (4C, C), 51.5 (4C, CH_2), 121.5 (2C, CH Ar), 125.5 (2C, CH Ar), 125.8 (2C, CH Ar), 126.1 (2C, CH Ar), 127.1 (2C, CH Ar), 127.9 (2C, CH Ar), 129.9 (2C, C Ar), 132.2 (2C, C Ar), 133.4 (2C, C Ar), 138.9 (2C, C Ar). EM (EI+), m/z (%): 440 (M+, 26). HRMS: for $C_{34}H_{32}$ calculated 440,2504, observed 440,2518. M. P. 276.0-277.8 ºC.

Synthesis of adduct 22

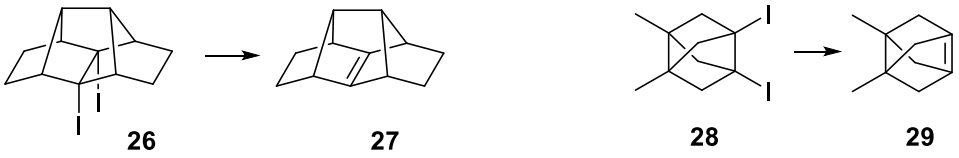


Scheme S6

Compound **1** (20 mg, 0.045 mmol), Na/Hg (5% Na, 164 mg, 0.361 mmol), CsF (84 mg, 0.552 mmol), 18-Crown-6 (73 mg, 0.276 mmol), and $Pd(PPh_3)_4$ (15 mg, 0.013 mmol) were placed in an oven-dried shlenck under Ar atmosphere. Then, solution of **20** (179 mg, 0.276 mmol) in THF (4.5 mL) was added. The mixture was stirred at room temperature for 16h. After that time, the reaction mixture was filtrated through a celite plug and purified on column chromatography (2.5:7.5 CH_2Cl_2/hexane) followed by a precipitation in Et_2O (1 mL) to yield **22** as a yellow solid (7 mg, 15%).

Data for compound **22**: ^{1}H NMR ($C_2D_2Cl_4$, 400 MHz, 353 K): δ 0.93 (12H, s, 6H), 1.10 (4H, d, *J* = 12.0 Hz, CH_2), 2.24 (4H, d, *J* = 11.7 Hz, CH_2), 6.96 – 6.86 (4H, m, CH Ar), 7.53 – 7.22 (28H, m, CH Ar), 7.86 (2H, s, CH Ar), 8.18 (4H, dd, *J* = 8.1, 3.5 Hz, CH Ar), 8.25 (2H, s, CH Ar). ^{13}C NMR ($C_2D_2Cl_4$, 101 MHz, 353 K): δ 15.72 (4C, Me), 45.63 (2C, C), 46.10 (4C, C), 51.35 (4C, CH_2), 121.17 (2C, CH Ar), 123.34 (4C, CH Ar), 125.41 (2C, CH Ar), 125.96 (4C, CH Ar), 126.75 (2C, CH Ar), 126.93 (2C, CH Ar), 127.80 (2C, CH Ar), 128.26 (2C, CH Ar), 128.94 (4C, CH Ar), 129.03 (4C, CH Ar), 129.24 (4C, C Ar), 130.48 (2C, C Ar), 130.76 (2C, CH Ar), 130.82 (2C, CH Ar), 131.14 (2C, C Ar), 131.84 (2C, C Ar), 131.94 (2C, C Ar), 132.16 (2C, C Ar), 132.29 (4C, C Ar), 132.56 (4C, CH Ar), 132.80 (4C, CH Ar), 134.64 (2C, C Ar), 135.69 (2C, C Ar), 139.04 (2C, C Ar), 141.15 (2C, C Ar), 141.92 (2C, C Ar). EM (MALDI-TOF), m/z (%); 1044 (100), 1045 (94), 1046 (42), 1047 (26), 1048 (13), 1049 (3); calculated isotopic ratio: 1044 (100), 1045 (92), 1046 (42), 1047 (13), 1048 (3), 1049 (3). HRMS (MALDI-TOF) for $C_{82}H_{60}$, calculated 1044.469, observed 1044.394.

3) ATTEMPTS WITH OTHER PYRAMIDALIZED ALKENES



Scheme S7

The palladium-catalyzed cocyclotrimerization of arynes with alkene **2** was tested with two additional highly reactive alkenes, **27** and **29**, generated *in situ* from compounds **26** and **28**, respectively. However, preliminary attempts using optimal reaction conditions developed for alkene **2** (see Scheme S2) did not lead to the isolation of the corresponding cocyclotrimerization adducts. This finding could be explain based on the high reactivity of alkenes **27** and **29** compare to alkene **2**. In fact, the pyramidalized angle for alkenes **27** and **29** is 62º,[7] while the corresponding pyramidalized angle for alkene **2** is 47º.[1] Therefore, it is expected that different reaction conditions would be needed depending on the reactivity of the pyramidalized alkene.

4) REFERENCES

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[6] A. J. Herbaut, E. Baranoff, *RSC Adv*. 2016, **6**, 10655.

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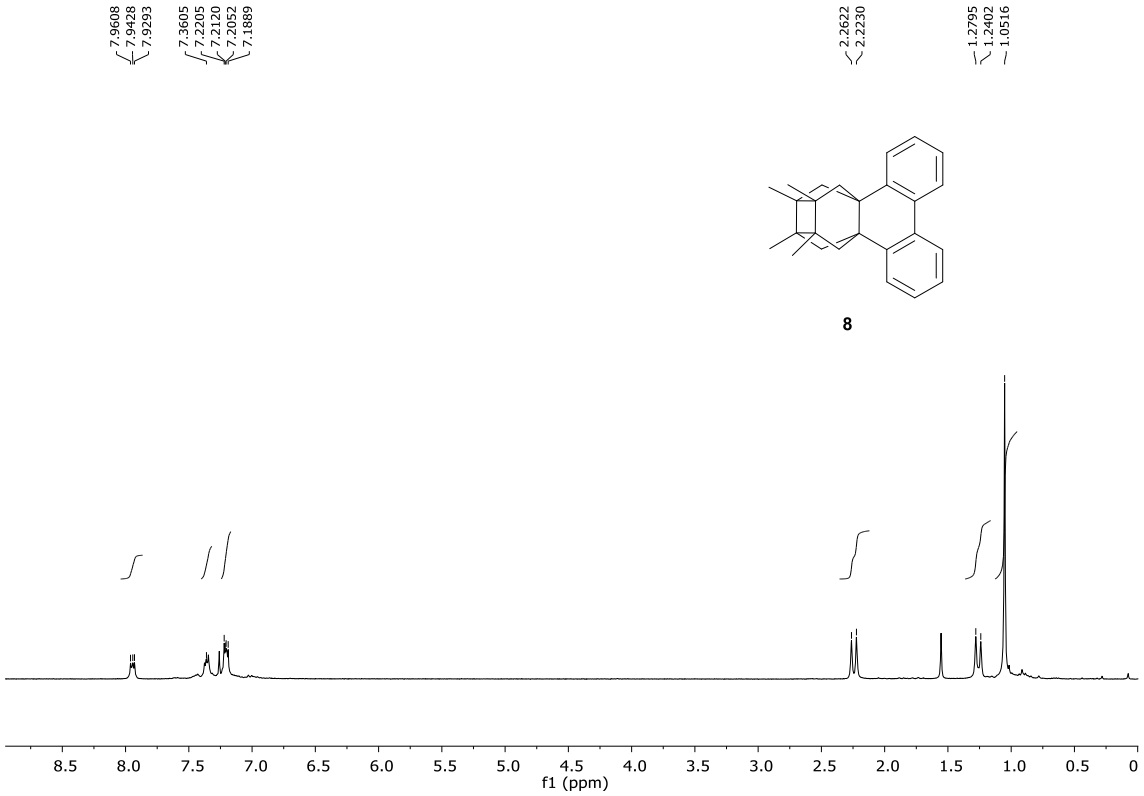
5) NMR Spectra



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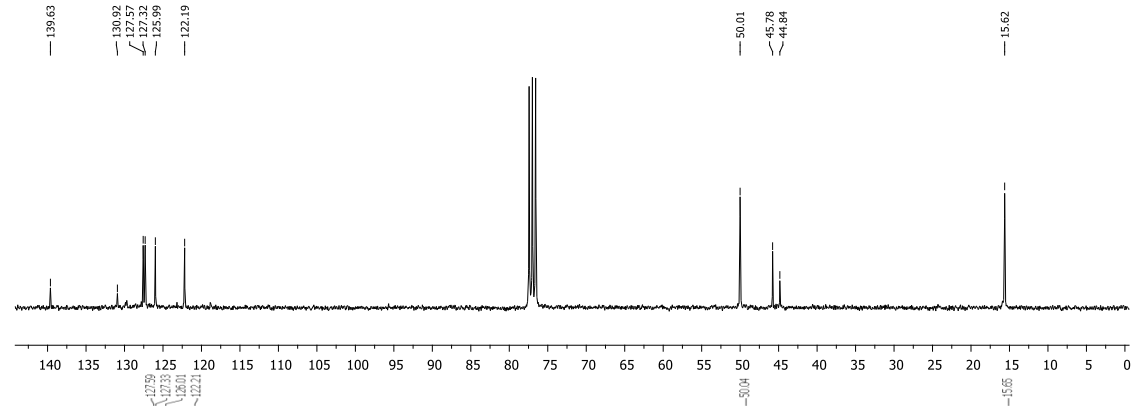

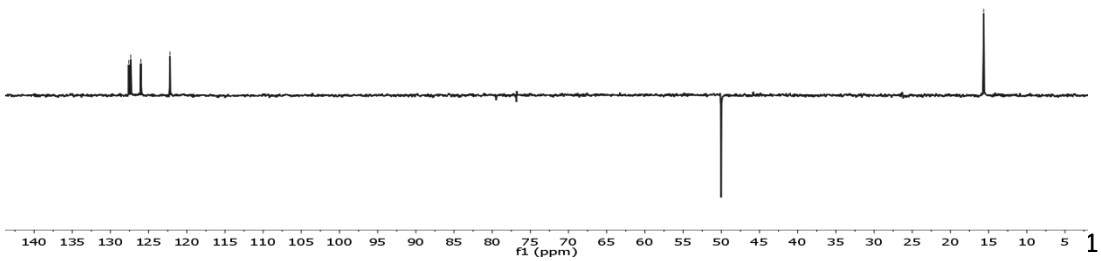

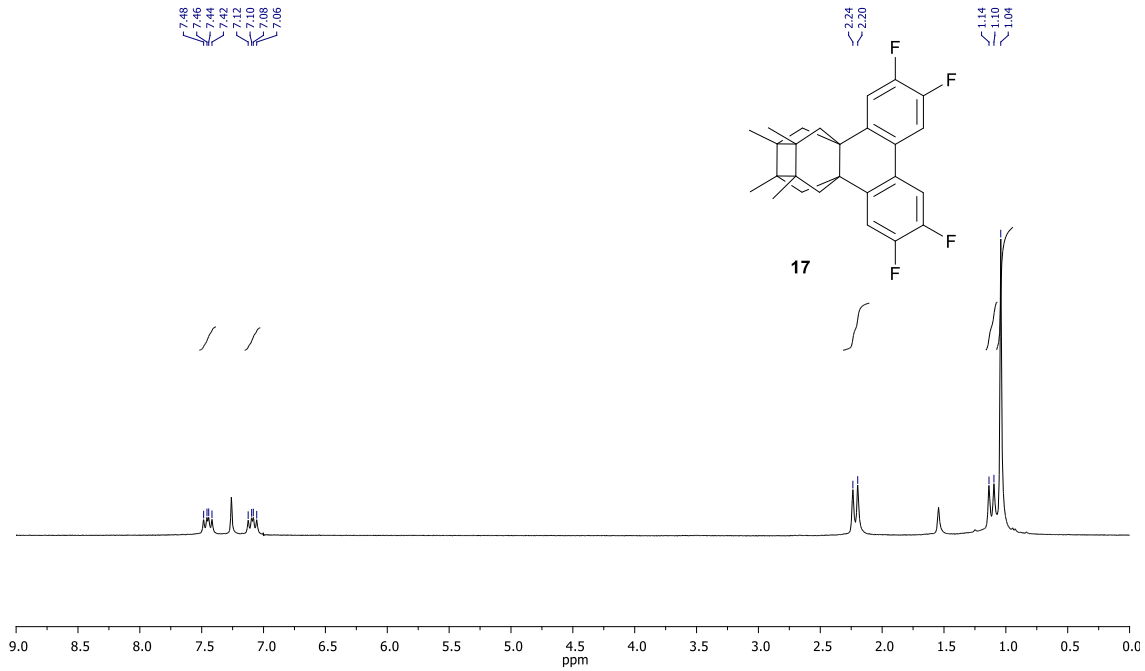
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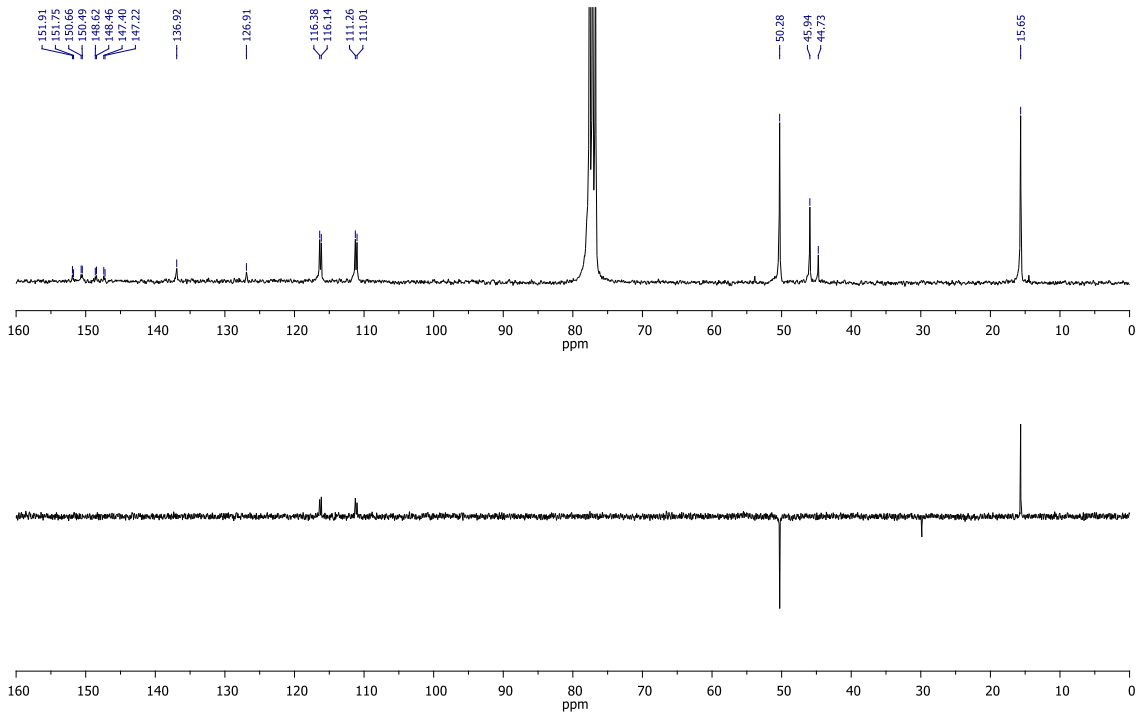
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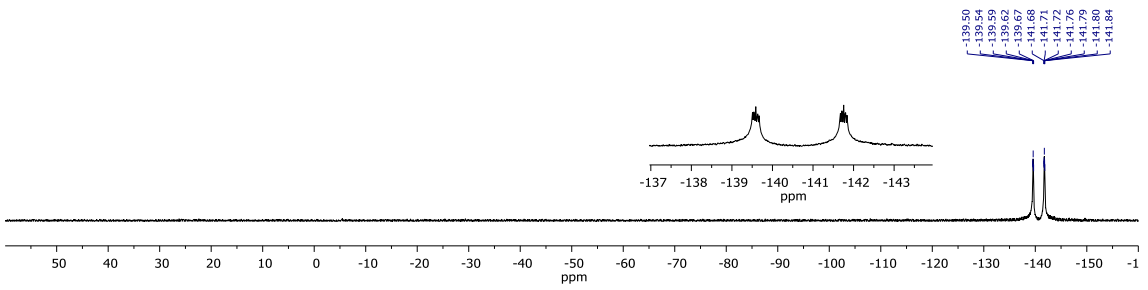
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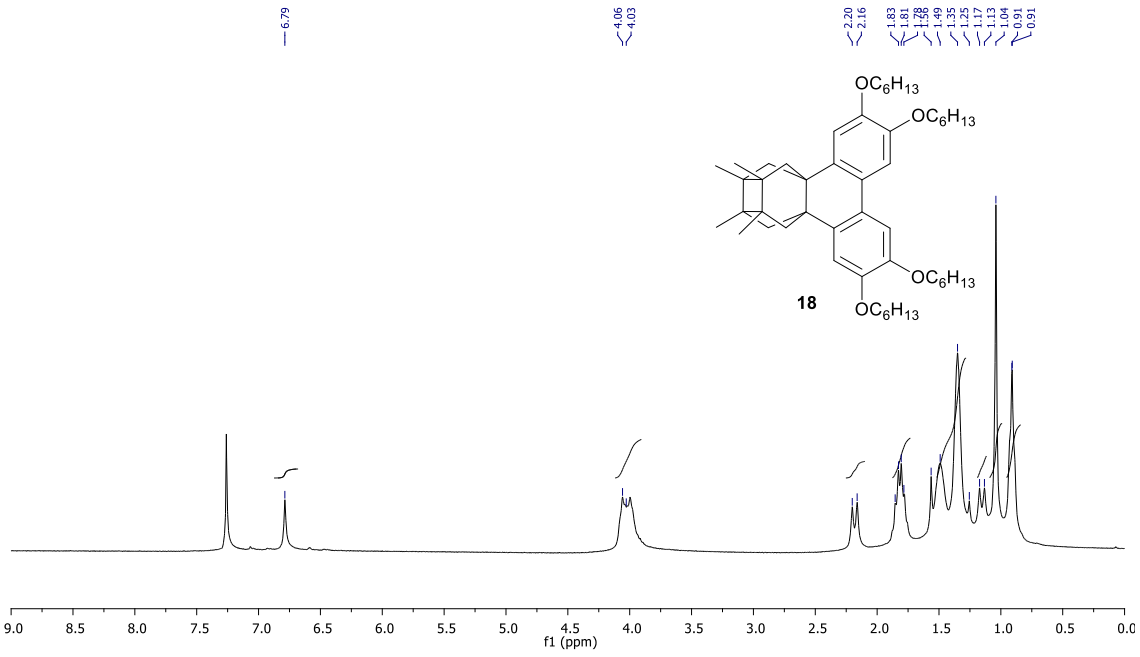
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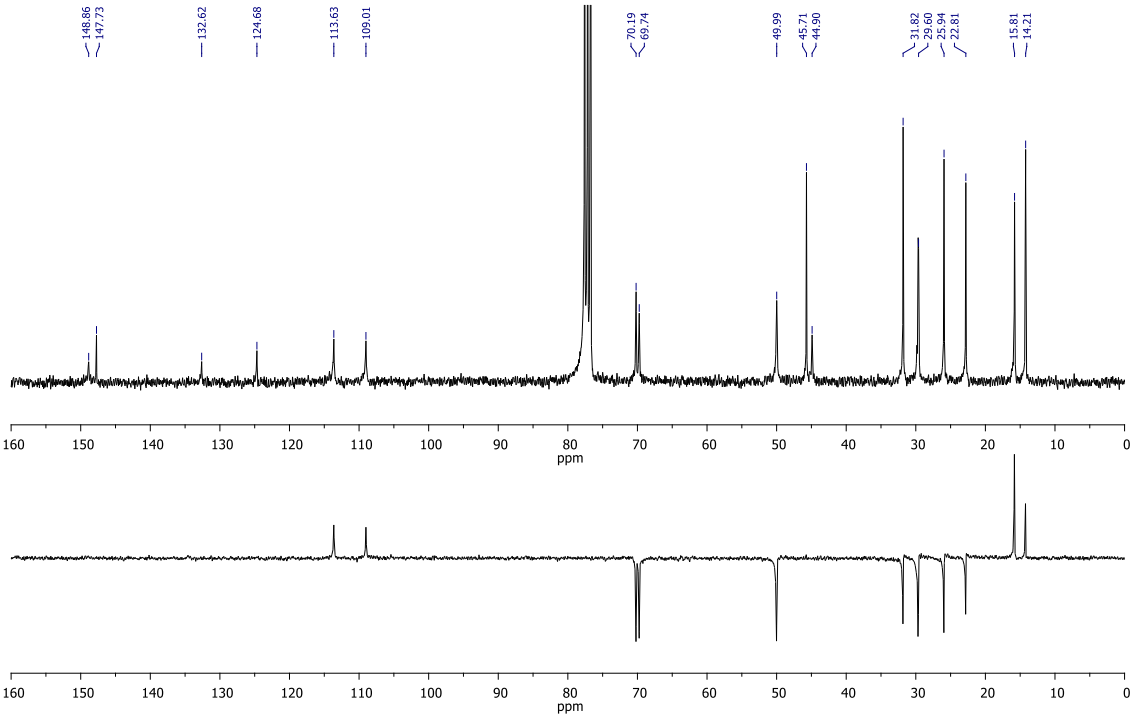
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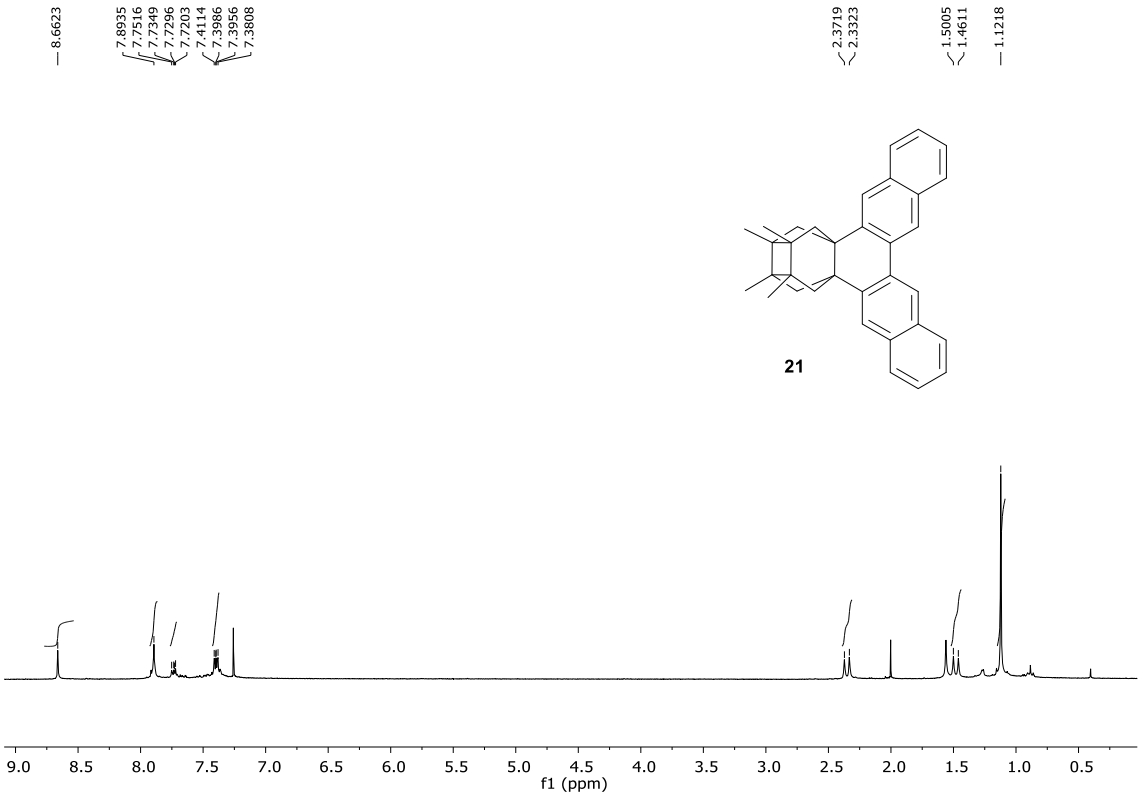
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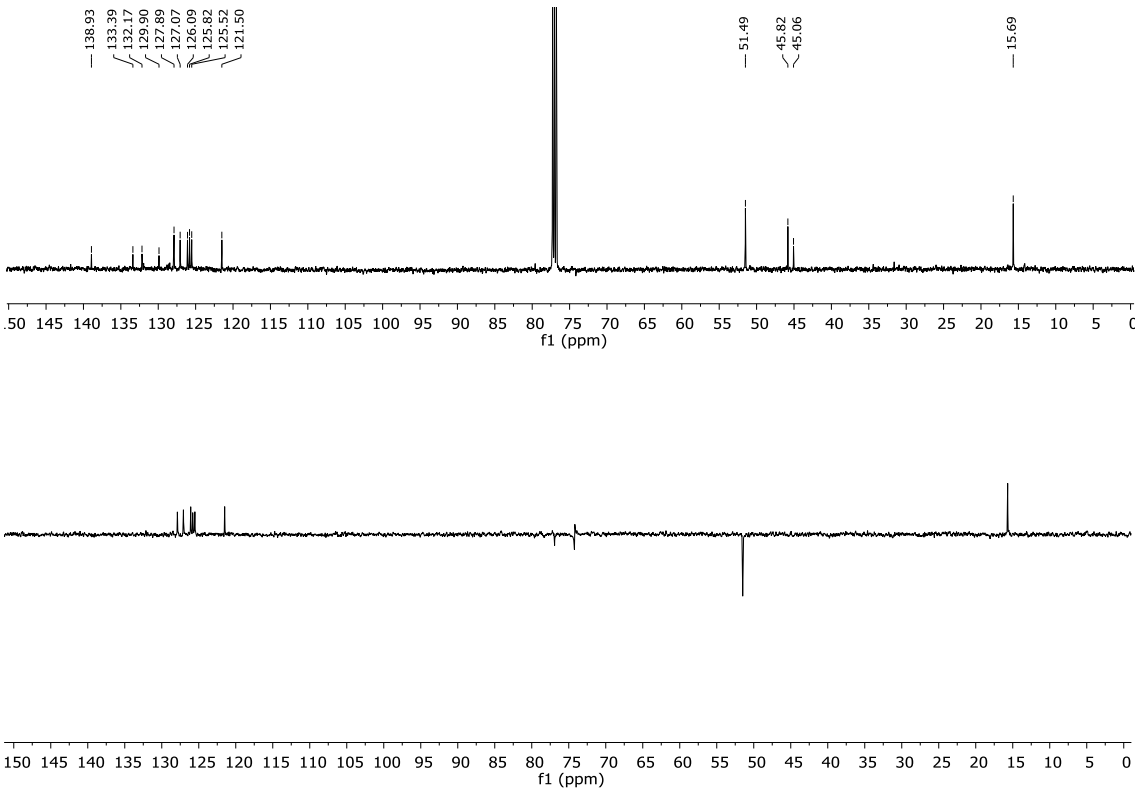
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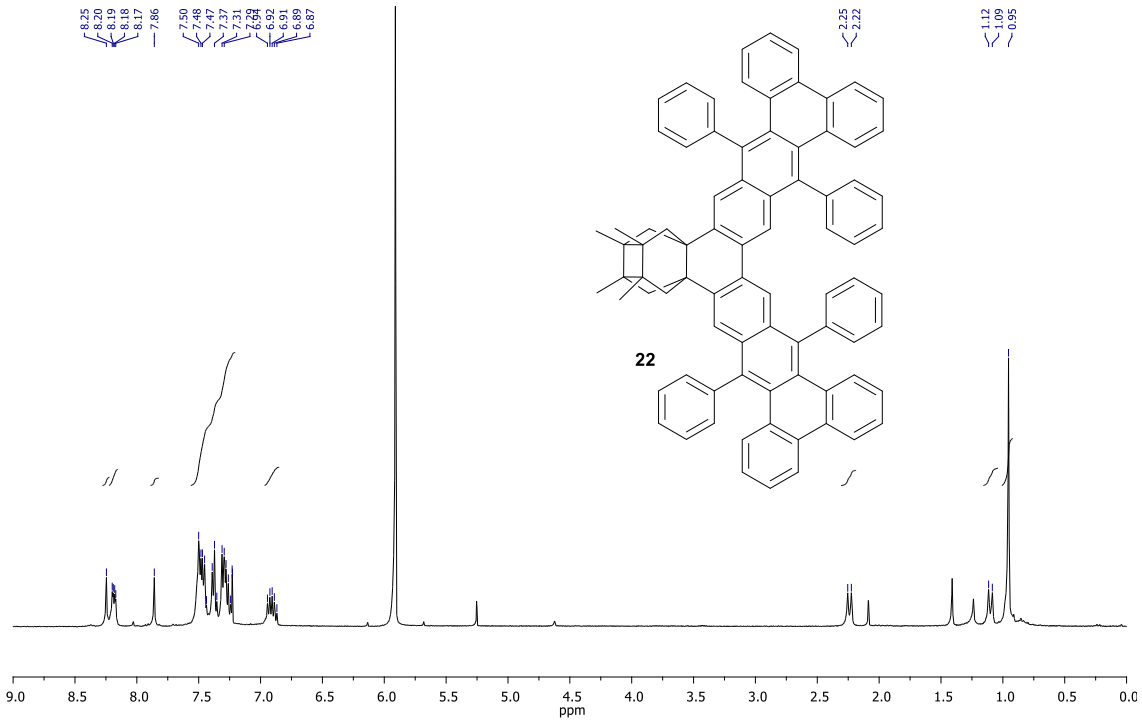
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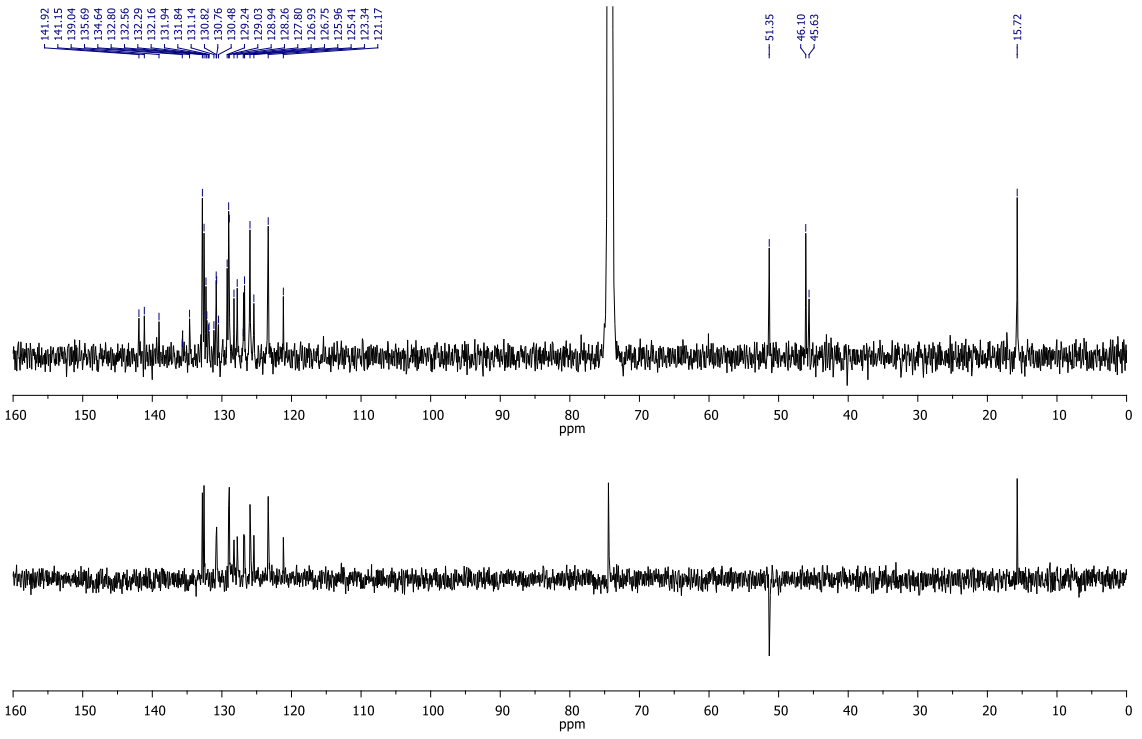
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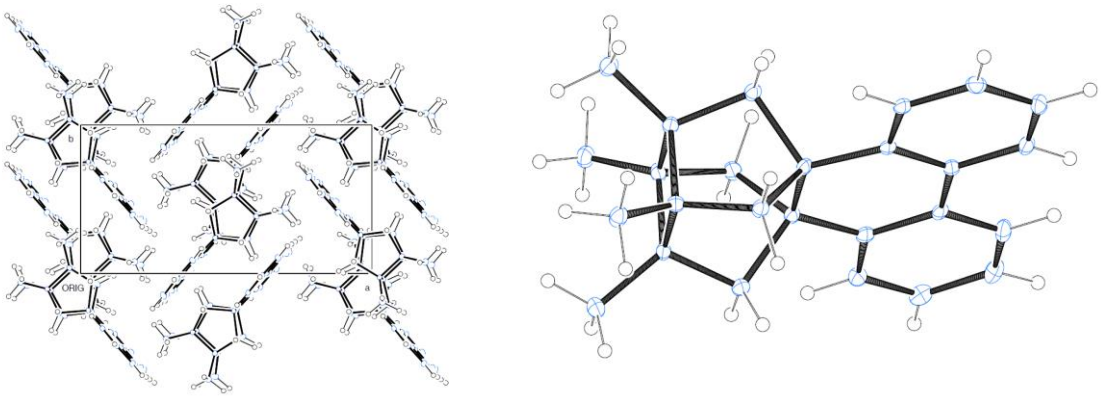
132.29
132.16
131.94
131.84
131.14
130.82
130.76
130.48
129.24
129.03
128.94
128.26
127.80
126.93
126.75
125.96
125.41
123.34
121.17
51.35
46.10
45.63
15.72
ppm

6) X-RAY DIFFRACTION DATA

Analysis of compound 8 (CCDC 1554197)



8

*Crystal data and structure refinement for compound **8**.*

Identification code	shelx	
Empirical formula	$C_{26}H_{28}$	
Formula weight	340.48	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 15.0392(10) Å	α= 90°
	b = 7.6824(6) Å	β= 91.082(5)°
	c = 15.9139(11) Å	γ = 90°
Volume	1838.3(2) $Å^3$	
Z	4	
Density (calculated)	1.230 Mg/m^3	
Absorption coefficient	0.069 mm^{-1}	
F(000)	736	
Crystal size	0.600 x 0.140 x 0.060 mm^3	

Theta range for data collection	1.354 to 26.413°
Index ranges	-18<=h<=18, -9<=k<=9, -19<=l<=18
Reflections collected	26454
Independent reflections	3755 [R(int) = 0.0731]
Completeness to theta = 25.242°	100.0 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3755 / 0 / 264
Goodness-of-fit on F^2	1.074
Final R indices [I>2sigma(I)]	R1 = 0.0684, wR2 = 0.1912
R indices (all data)	R1 = 0.1059, wR2 = 0.2170
Largest diff. peak and hole	0.329 and -0.317 e.$Å^{-3}$

*Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) compound **8**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.*

	x	y	z	U(eq)
C(1)	6031(2)	8954(4)	1663(2)	17(1)
C(2)	6157(2)	9106(4)	798(2)	21(1)
C(3)	6711(2)	10348(4)	463(2)	24(1)
C(4)	7156(2)	11489(4)	995(2)	24(1)
C(5)	7054(2)	11357(4)	1849(2)	22(1)
C(6)	6497(2)	10101(4)	2207(2)	16(1)
C(7)	5381(2)	7595(4)	1968(2)	17(1)
C(8)	4446(2)	7830(4)	1533(2)	18(1)
C(9)	3967(2)	6142(4)	1764(2)	18(1)
C(10)	4731(2)	4735(4)	1821(2)	17(1)
C(11)	5581(2)	5742(4)	1625(2)	18(1)
C(12)	3159(2)	5775(4)	1199(2)	23(1)
C(13)	4627(2)	3068(4)	1313(2)	24(1)
C(21)	5823(2)	8758(3)	3485(2)	15(1)
C(22)	5758(2)	8712(4)	4356(2)	18(1)
C(23)	6226(2)	9833(4)	4879(2)	21(1)
C(24)	6786(2)	11066(4)	4526(2)	22(1)
C(25)	6866(2)	11133(4)	3671(2)	20(1)
C(26)	6395(2)	9994(4)	3129(2)	16(1)
C(27)	5264(2)	7487(4)	2962(2)	16(1)
C(28)	5384(2)	5566(4)	3252(2)	18(1)
C(29)	4615(2)	4627(4)	2803(2)	18(1)
C(30)	3854(2)	6042(4)	2742(2)	17(1)
C(31)	4255(2)	7651(4)	3166(2)	17(1)
C(32)	4397(2)	2873(4)	3182(2)	22(1)
C(33)	2938(2)	5582(4)	3069(2)	22(1)

*Bond lengths [Å] and angles [°] for compound **8**.*

C(1)-C(2)	1.397(4)
C(1)-C(6)	1.412(4)
C(1)-C(7)	1.518(4)
C(2)-C(3)	1.381(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.382(4)
C(3)-H(3)	0.9500
C(4)-C(5)	1.375(4)
C(4)-H(4)	0.9500
C(5)-C(6)	1.406(4)
C(5)-H(5)	0.9500
C(6)-C(26)	1.480(4)
C(7)-C(11)	1.556(4)
C(7)-C(8)	1.565(4)
C(7)-C(27)	1.597(4)
C(8)-C(9)	1.532(4)
C(8)-H(8A)	1.04(3)
C(8)-H(8B)	0.96(3)
C(9)-C(12)	1.524(4)
C(9)-C(30)	1.570(4)
C(9)-C(10)	1.579(4)
C(10)-C(13)	1.521(4)
C(10)-C(11)	1.532(4)
C(10)-C(29)	1.577(4)
C(11)-H(11A)	0.97(3)
C(11)-H(11B)	1.02(3)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(21)-C(22)	1.392(4)
C(21)-C(26)	1.408(4)
C(21)-C(27)	1.525(4)
C(22)-C(23)	1.381(4)

C(22)-H(22)	0.9500
C(23)-C(24)	1.393(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.369(4)
C(24)-H(24)	0.9500
C(25)-C(26)	1.411(4)
C(25)-H(25)	0.9500
C(27)-C(28)	1.556(4)
C(27)-C(31)	1.563(4)
C(28)-C(29)	1.529(4)
C(28)-H(28A)	1.00(3)
C(28)-H(28B)	1.02(3)
C(29)-C(32)	1.515(4)
C(29)-C(30)	1.581(4)
C(30)-C(33)	1.524(4)
C(30)-C(31)	1.526(4)
C(31)-H(31A)	1.00(3)
C(31)-H(31B)	1.04(3)
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800
C(33)-H(33A)	0.9800
C(33)-H(33B)	0.9800
C(33)-H(33C)	0.9800

C(2)-C(1)-C(6)	118.5(3)
C(2)-C(1)-C(7)	118.2(2)
C(6)-C(1)-C(7)	123.2(3)
C(3)-C(2)-C(1)	122.1(3)
C(3)-C(2)-H(2)	118.9
C(1)-C(2)-H(2)	118.9
C(2)-C(3)-C(4)	119.3(3)
C(2)-C(3)-H(3)	120.3
C(4)-C(3)-H(3)	120.3
C(5)-C(4)-C(3)	119.8(3)
C(5)-C(4)-H(4)	120.1
C(3)-C(4)-H(4)	120.1
C(4)-C(5)-C(6)	122.1(3)

C(4)-C(5)-H(5)	119.0
C(6)-C(5)-H(5)	119.0
C(5)-C(6)-C(1)	118.1(3)
C(5)-C(6)-C(26)	120.9(2)
C(1)-C(6)-C(26)	120.9(3)
C(1)-C(7)-C(11)	112.8(2)
C(1)-C(7)-C(8)	111.0(2)
C(11)-C(7)-C(8)	97.3(2)
C(1)-C(7)-C(27)	115.9(2)
C(11)-C(7)-C(27)	109.0(2)
C(8)-C(7)-C(27)	109.3(2)
C(9)-C(8)-C(7)	102.6(2)
C(9)-C(8)-H(8A)	116.5(17)
C(7)-C(8)-H(8A)	113.9(17)
C(9)-C(8)-H(8B)	109.4(19)
C(7)-C(8)-H(8B)	112.3(19)
H(8A)-C(8)-H(8B)	102(2)
C(12)-C(9)-C(8)	112.9(2)
C(12)-C(9)-C(30)	118.4(2)
C(8)-C(9)-C(30)	109.8(2)
C(12)-C(9)-C(10)	118.6(2)
C(8)-C(9)-C(10)	104.3(2)
C(30)-C(9)-C(10)	90.1(2)
C(13)-C(10)-C(11)	113.2(2)
C(13)-C(10)-C(29)	118.0(2)
C(11)-C(10)-C(29)	109.6(2)
C(13)-C(10)-C(9)	118.6(2)
C(11)-C(10)-C(9)	104.6(2)
C(29)-C(10)-C(9)	90.0(2)
C(10)-C(11)-C(7)	102.9(2)
C(10)-C(11)-H(11A)	107.0(19)
C(7)-C(11)-H(11A)	108.6(19)
C(10)-C(11)-H(11B)	112.6(18)
C(7)-C(11)-H(11B)	115.3(18)
H(11A)-C(11)-H(11B)	110(3)
C(9)-C(12)-H(12A)	109.5
C(9)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5

C(9)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(10)-C(13)-H(13A)	109.5
C(10)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(10)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(22)-C(21)-C(26)	118.2(2)
C(22)-C(21)-C(27)	118.6(2)
C(26)-C(21)-C(27)	123.2(2)
C(23)-C(22)-C(21)	122.7(3)
C(23)-C(22)-H(22)	118.7
C(21)-C(22)-H(22)	118.7
C(22)-C(23)-C(24)	119.1(3)
C(22)-C(23)-H(23)	120.4
C(24)-C(23)-H(23)	120.4
C(25)-C(24)-C(23)	119.4(3)
C(25)-C(24)-H(24)	120.3
C(23)-C(24)-H(24)	120.3
C(24)-C(25)-C(26)	122.2(3)
C(24)-C(25)-H(25)	118.9
C(26)-C(25)-H(25)	118.9
C(21)-C(26)-C(25)	118.4(3)
C(21)-C(26)-C(6)	120.8(2)
C(25)-C(26)-C(6)	120.8(3)
C(21)-C(27)-C(28)	112.7(2)
C(21)-C(27)-C(31)	111.3(2)
C(28)-C(27)-C(31)	97.1(2)
C(21)-C(27)-C(7)	115.9(2)
C(28)-C(27)-C(7)	109.2(2)
C(31)-C(27)-C(7)	109.1(2)
C(29)-C(28)-C(27)	103.0(2)
C(29)-C(28)-H(28A)	112.3(18)
C(27)-C(28)-H(28A)	114.9(18)
C(29)-C(28)-H(28B)	112.1(18)
C(27)-C(28)-H(28B)	108.4(18)

H(28A)-C(28)-H(28B)	106(2)
C(32)-C(29)-C(28)	113.6(2)
C(32)-C(29)-C(10)	118.0(2)
C(28)-C(29)-C(10)	110.0(2)
C(32)-C(29)-C(30)	118.3(2)
C(28)-C(29)-C(30)	104.2(2)
C(10)-C(29)-C(30)	89.8(2)
C(33)-C(30)-C(31)	112.9(2)
C(33)-C(30)-C(9)	117.7(2)
C(31)-C(30)-C(9)	110.5(2)
C(33)-C(30)-C(29)	118.5(2)
C(31)-C(30)-C(29)	104.5(2)
C(9)-C(30)-C(29)	90.1(2)
C(30)-C(31)-C(27)	102.7(2)
C(30)-C(31)-H(31A)	110.7(18)
C(27)-C(31)-H(31A)	107.0(17)
C(30)-C(31)-H(31B)	115.1(16)
C(27)-C(31)-H(31B)	111.5(17)
H(31A)-C(31)-H(31B)	109(2)
C(29)-C(32)-H(32A)	109.5
C(29)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(29)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(30)-C(33)-H(33A)	109.5
C(30)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(30)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5

*Anisotropic displacement parameters ($Å^2 x\ 10^3$)for compound **8**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$*

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	12(1)	16(2)	25(2)	0(1)	-1(1)	5(1)
C(2)	17(2)	20(2)	26(2)	-2(1)	-1(1)	0(1)
C(3)	19(2)	29(2)	23(2)	3(1)	2(1)	2(1)
C(4)	17(2)	25(2)	30(2)	8(1)	-1(1)	-5(1)
C(5)	17(2)	20(2)	29(2)	1(1)	-6(1)	-4(1)
C(6)	9(1)	14(1)	27(2)	1(1)	-1(1)	3(1)
C(7)	12(1)	13(1)	26(2)	-1(1)	0(1)	0(1)
C(8)	17(2)	16(2)	20(2)	0(1)	-3(1)	1(1)
C(9)	17(2)	14(2)	24(2)	-3(1)	-3(1)	-1(1)
C(10)	12(1)	13(1)	27(2)	-2(1)	-2(1)	-1(1)
C(11)	14(2)	17(2)	23(2)	-1(1)	0(1)	2(1)
C(12)	18(2)	22(2)	29(2)	-2(1)	-5(1)	-1(1)
C(13)	21(2)	18(2)	33(2)	-6(1)	-1(1)	-1(1)
C(21)	10(1)	12(1)	24(2)	-1(1)	-1(1)	4(1)
C(22)	12(1)	16(2)	26(2)	-1(1)	1(1)	3(1)
C(23)	18(2)	23(2)	21(2)	-5(1)	-1(1)	5(1)
C(24)	15(2)	20(2)	29(2)	-5(1)	-5(1)	-1(1)
C(25)	12(1)	16(2)	31(2)	0(1)	-2(1)	-1(1)
C(26)	11(1)	13(1)	25(2)	-1(1)	-3(1)	4(1)
C(27)	10(1)	14(1)	24(2)	0(1)	-1(1)	1(1)
C(28)	14(2)	14(2)	26(2)	1(1)	0(1)	0(1)
C(29)	14(1)	14(2)	25(2)	-2(1)	-1(1)	3(1)
C(30)	13(1)	12(1)	26(2)	-1(1)	-2(1)	1(1)
C(31)	13(1)	15(2)	21(2)	1(1)	0(1)	1(1)
C(32)	16(2)	18(2)	32(2)	3(1)	-2(1)	0(1)
C(33)	16(2)	18(2)	33(2)	0(1)	2(1)	0(1)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)

*for compound **8***

	x	y	z	U(eq)
H(2)	5851	8329	429	25
H(3)	6786	10417	-128	29
H(4)	7531	12362	770	29
H(5)	7369	12139	2208	26
H(8A)	4130(20)	8980(40)	1687(19)	21
H(8B)	4480(20)	7910(40)	930(20)	21
H(11A)	5620(20)	5810(40)	1020(20)	22
H(11B)	6140(20)	5160(40)	1870(20)	22
H(12A)	3342	5741	611	34
H(12B)	2717	6698	1270	34
H(12C)	2899	4652	1351	34
H(13A)	4639	3343	712	36
H(13B)	4060	2513	1444	36
H(13C)	5117	2272	1456	36
H(22)	5377	7875	4601	21
H(23)	6166	9764	5471	25
H(24)	7110	11853	4876	26
H(25)	7252	11974	3435	23
H(28A)	5980(20)	5040(40)	3120(20)	21
H(28B)	5330(20)	5520(40)	3890(20)	21
H(31A)	4210(20)	7570(40)	3793(19)	20
H(31B)	3990(20)	8830(40)	2960(18)	20
H(32A)	3888	2361	2878	33
H(32B)	4249	3024	3775	33
H(32C)	4912	2100	3139	33
H(33A)	2526	6547	2956	33
H(33B)	2979	5371	3676	33
H(33C)	2718	4530	2785	33

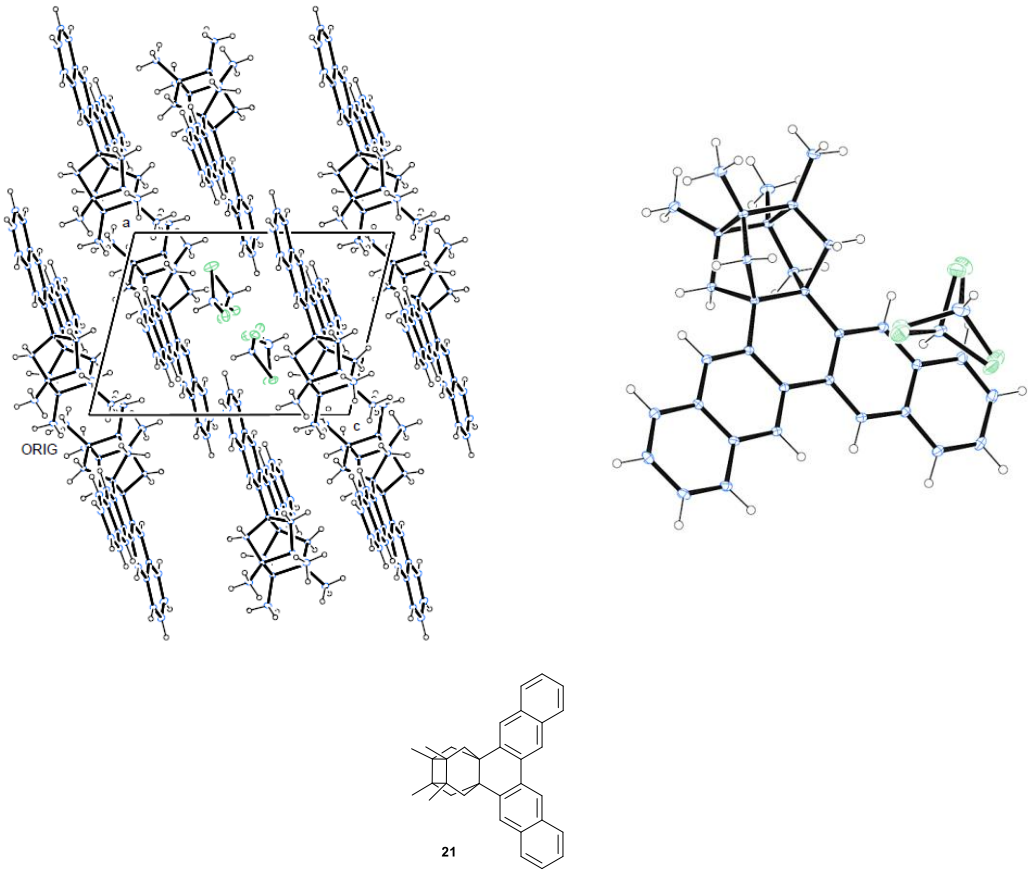
*Torsion angles [°] for compound **8***

C(6)-C(1)-C(2)-C(3)	-0.7(4)
C(7)-C(1)-C(2)-C(3)	177.6(3)
C(1)-C(2)-C(3)-C(4)	-0.3(5)
C(2)-C(3)-C(4)-C(5)	1.1(5)
C(3)-C(4)-C(5)-C(6)	-0.8(5)
C(4)-C(5)-C(6)-C(1)	-0.2(4)
C(4)-C(5)-C(6)-C(26)	-179.6(3)
C(2)-C(1)-C(6)-C(5)	1.0(4)
C(7)-C(1)-C(6)-C(5)	-177.3(3)
C(2)-C(1)-C(6)-C(26)	-179.6(2)
C(7)-C(1)-C(6)-C(26)	2.1(4)
C(2)-C(1)-C(7)-C(11)	53.5(3)
C(6)-C(1)-C(7)-C(11)	-128.2(3)
C(2)-C(1)-C(7)-C(8)	-54.5(3)
C(6)-C(1)-C(7)-C(8)	123.8(3)
C(2)-C(1)-C(7)-C(27)	-179.9(2)
C(6)-C(1)-C(7)-C(27)	-1.6(4)
C(1)-C(7)-C(8)-C(9)	167.8(2)
C(11)-C(7)-C(8)-C(9)	50.0(3)
C(27)-C(7)-C(8)-C(9)	-63.2(3)
C(7)-C(8)-C(9)-C(12)	-161.7(2)
C(7)-C(8)-C(9)-C(30)	63.8(3)
C(7)-C(8)-C(9)-C(10)	-31.7(3)
C(12)-C(9)-C(10)-C(13)	-0.3(4)
C(8)-C(9)-C(10)-C(13)	-126.9(3)
C(30)-C(9)-C(10)-C(13)	122.6(3)
C(12)-C(9)-C(10)-C(11)	127.0(3)
C(8)-C(9)-C(10)-C(11)	0.5(3)
C(30)-C(9)-C(10)-C(11)	-110.1(2)
C(12)-C(9)-C(10)-C(29)	-122.7(3)
C(8)-C(9)-C(10)-C(29)	110.8(2)
C(30)-C(9)-C(10)-C(29)	0.2(2)
C(13)-C(10)-C(11)-C(7)	161.7(2)
C(29)-C(10)-C(11)-C(7)	-64.1(3)
C(9)-C(10)-C(11)-C(7)	31.1(3)
C(1)-C(7)-C(11)-C(10)	-166.1(2)

C(8)-C(7)-C(11)-C(10)	-49.7(3)
C(27)-C(7)-C(11)-C(10)	63.7(3)
C(26)-C(21)-C(22)-C(23)	0.5(4)
C(27)-C(21)-C(22)-C(23)	-178.8(3)
C(21)-C(22)-C(23)-C(24)	0.0(4)
C(22)-C(23)-C(24)-C(25)	-0.4(4)
C(23)-C(24)-C(25)-C(26)	0.3(4)
C(22)-C(21)-C(26)-C(25)	-0.6(4)
C(27)-C(21)-C(26)-C(25)	178.6(2)
C(22)-C(21)-C(26)-C(6)	179.6(2)
C(27)-C(21)-C(26)-C(6)	-1.2(4)
C(24)-C(25)-C(26)-C(21)	0.3(4)
C(24)-C(25)-C(26)-C(6)	-180.0(3)
C(5)-C(6)-C(26)-C(21)	178.7(3)
C(1)-C(6)-C(26)-C(21)	-0.7(4)
C(5)-C(6)-C(26)-C(25)	-1.1(4)
C(1)-C(6)-C(26)-C(25)	179.5(3)
C(22)-C(21)-C(27)-C(28)	-52.4(3)
C(26)-C(21)-C(27)-C(28)	128.4(3)
C(22)-C(21)-C(27)-C(31)	55.4(3)
C(26)-C(21)-C(27)-C(31)	-123.8(3)
C(22)-C(21)-C(27)-C(7)	-179.2(2)
C(26)-C(21)-C(27)-C(7)	1.6(4)
C(1)-C(7)-C(27)-C(21)	-0.2(3)
C(11)-C(7)-C(27)-C(21)	128.3(2)
C(8)-C(7)-C(27)-C(21)	-126.4(2)
C(1)-C(7)-C(27)-C(28)	-128.8(2)
C(11)-C(7)-C(27)-C(28)	-0.3(3)
C(8)-C(7)-C(27)-C(28)	105.0(2)
C(1)-C(7)-C(27)-C(31)	126.3(2)
C(11)-C(7)-C(27)-C(31)	-105.2(3)
C(8)-C(7)-C(27)-C(31)	0.0(3)
C(21)-C(27)-C(28)-C(29)	166.6(2)
C(31)-C(27)-C(28)-C(29)	50.0(3)
C(7)-C(27)-C(28)-C(29)	-63.1(3)
C(27)-C(28)-C(29)-C(32)	-161.5(2)
C(27)-C(28)-C(29)-C(10)	63.6(3)
C(27)-C(28)-C(29)-C(30)	-31.4(3)

C(13)-C(10)-C(29)-C(32)	-0.8(4)
C(11)-C(10)-C(29)-C(32)	-132.4(3)
C(9)-C(10)-C(29)-C(32)	122.1(3)
C(13)-C(10)-C(29)-C(28)	131.9(3)
C(11)-C(10)-C(29)-C(28)	0.2(3)
C(9)-C(10)-C(29)-C(28)	-105.3(2)
C(13)-C(10)-C(29)-C(30)	-123.1(3)
C(11)-C(10)-C(29)-C(30)	105.2(2)
C(9)-C(10)-C(29)-C(30)	-0.2(2)
C(12)-C(9)-C(30)-C(33)	0.0(4)
C(8)-C(9)-C(30)-C(33)	131.7(3)
C(10)-C(9)-C(30)-C(33)	-123.0(3)
C(12)-C(9)-C(30)-C(31)	-131.7(3)
C(8)-C(9)-C(30)-C(31)	0.0(3)
C(10)-C(9)-C(30)-C(31)	105.3(2)
C(12)-C(9)-C(30)-C(29)	122.8(3)
C(8)-C(9)-C(30)-C(29)	-105.5(2)
C(10)-C(9)-C(30)-C(29)	-0.2(2)
C(32)-C(29)-C(30)-C(33)	0.2(4)
C(28)-C(29)-C(30)-C(33)	-127.1(3)
C(10)-C(29)-C(30)-C(33)	122.3(3)
C(32)-C(29)-C(30)-C(31)	126.9(3)
C(28)-C(29)-C(30)-C(31)	-0.4(3)
C(10)-C(29)-C(30)-C(31)	-111.0(2)
C(32)-C(29)-C(30)-C(9)	-121.9(3)
C(28)-C(29)-C(30)-C(9)	110.8(2)
C(10)-C(29)-C(30)-C(9)	0.2(2)
C(33)-C(30)-C(31)-C(27)	162.0(2)
C(9)-C(30)-C(31)-C(27)	-63.9(3)
C(29)-C(30)-C(31)-C(27)	31.8(3)
C(21)-C(27)-C(31)-C(30)	-167.8(2)
C(28)-C(27)-C(31)-C(30)	-50.1(3)
C(7)-C(27)-C(31)-C(30)	63.0(3)

X-Ray analysis of compound 21 (CCDC1554198):



*Crystal data and structure refinement for compound **21**.*

Identification code	shelx	
Empirical formula	$C_{34}H_{32}$, $CHCl_3$	
Formula weight	559.96	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 9.5957(2) Å	α= 73.4440(10)°
	b = 12.2865(3) Å	β= 72.2900(10)°
	c = 13.2430(3) Å	γ = 73.1600(10)°
Volume	1389.98(6) $Å^3$	
Z	2	
Density (calculated)	1.338 Mg/m^3	

Absorption coefficient	0.354 mm^{-1}
F(000)	588
Crystal size	0.420 x 0.400 x 0.200 mm^3
Theta range for data collection	1.653 to 30.546°
Index ranges	-13<=h<=13, -17<=k<=17, -18<=l<=18
Reflections collected	63677
Independent reflections	8507 [R(int) = 0.0514]
Completeness to theta = 25.242°	100.0 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8507 / 0 / 482
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0472, wR2 = 0.1146
R indices (all data)	R1 = 0.0743, wR2 = 0.1279
Largest diff. peak and hole	0.443 and -0.384 e.$Å^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2) for compound **21***

	x	*y*	*z*	*U*iso*/*U*eq	Occ. (<1)
C1	0.40274 (13)	0.33423 (11)	0.27405 (10)	0.0124 (2)	
C2	0.31290 (13)	0.44681 (11)	0.28672 (10)	0.0127 (2)	
C3	0.16243 (13)	0.45746 (12)	0.33915 (11)	0.0149 (3)	
H3	0.1023 (16)	0.5330 (13)	0.3433 (12)	0.018*	
C4	0.09465 (13)	0.36117 (11)	0.38401 (10)	0.0140 (2)	
C5	−0.06095 (14)	0.37329 (13)	0.43617 (11)	0.0180 (3)	
H5	−0.1229 (17)	0.4524 (14)	0.4373 (13)	0.022*	
C6	−0.12152 (15)	0.27737 (13)	0.47864 (12)	0.0207 (3)	
H6	−0.2269 (18)	0.2846 (14)	0.5156 (13)	0.025*	
C7	−0.03025 (15)	0.16505 (13)	0.47316 (12)	0.0228 (3)	
H7	−0.0725 (18)	0.0961 (15)	0.5035 (14)	0.027*	
C8	0.11949 (15)	0.15029 (13)	0.42426 (12)	0.0203 (3)	
H8	0.1875 (18)	0.0726 (14)	0.4195 (13)	0.024*	
C9	0.18544 (14)	0.24864 (11)	0.37663 (10)	0.0147 (3)	
C10	0.33756 (14)	0.23933 (12)	0.32014 (11)	0.0156 (3)	
H10	0.3964 (16)	0.1600 (14)	0.3136 (12)	0.019*	
C11	0.56535 (13)	0.31389 (10)	0.21008 (10)	0.0120 (2)	
C12	0.67071 (13)	0.21984 (11)	0.27667 (11)	0.0139 (2)	
H12A	0.6837 (15)	0.2446 (13)	0.3378 (13)	0.017*	
H12B	0.6266 (16)	0.1504 (13)	0.3112 (12)	0.017*	
C13	0.81461 (13)	0.19249 (11)	0.18949 (10)	0.0139 (2)	
C14	0.91893 (15)	0.07755 (12)	0.22462 (13)	0.0199 (3)	
H14A	1.0042 (19)	0.0544 (15)	0.1697 (14)	0.03*	
H14B	0.8679 (19)	0.0106 (15)	0.2498 (14)	0.03*	

	x	*y*	*z*	*U*iso*/*U*eq	Occ. (<1)
H14C	0.9551 (18)	0.0803 (14)	0.2861 (14)	0.03*	
C15	0.58841 (13)	0.24829 (11)	0.12016 (11)	0.0135 (2)	
H15A	0.5431 (16)	0.1787 (13)	0.1531 (12)	0.016*	
H15B	0.5375 (16)	0.2915 (13)	0.0625 (12)	0.016*	
C16	0.75934 (13)	0.21178 (11)	0.08364 (11)	0.0142 (2)	
C17	0.81243 (15)	0.11437 (13)	0.02211 (13)	0.0202 (3)	
H17A	0.7691 (18)	0.1357 (14)	−0.0407 (14)	0.03*	
H17B	0.7832 (18)	0.0439 (16)	0.0662 (14)	0.03*	
H17C	0.9169 (19)	0.0958 (15)	−0.0026 (14)	0.03*	
C21	0.53137 (13)	0.54121 (11)	0.18107 (10)	0.0126 (2)	
C22	0.38029 (13)	0.55049 (11)	0.24398 (10)	0.0123 (2)	
C23	0.29694 (13)	0.65784 (11)	0.26493 (11)	0.0144 (3)	
H23	0.1947 (17)	0.6648 (13)	0.3105 (12)	0.017*	
C24	0.35420 (13)	0.75942 (11)	0.22431 (10)	0.0141 (3)	
C25	0.26714 (15)	0.86982 (11)	0.24489 (11)	0.0171 (3)	
H25	0.1664 (17)	0.8724 (13)	0.2888 (13)	0.02*	
C26	0.32594 (15)	0.96654 (12)	0.20174 (11)	0.0184 (3)	
H26	0.2651 (17)	1.0407 (14)	0.2157 (13)	0.022*	
C27	0.47428 (15)	0.95818 (12)	0.13660 (12)	0.0192 (3)	
H27	0.5170 (17)	1.0274 (14)	0.1036 (13)	0.023*	
C28	0.56050 (15)	0.85325 (12)	0.11619 (12)	0.0185 (3)	
H28	0.6596 (18)	0.8457 (13)	0.0706 (13)	0.022*	
C29	0.50296 (14)	0.75102 (11)	0.15923 (11)	0.0142 (2)	
C30	0.58719 (14)	0.64085 (11)	0.14029 (11)	0.0150 (3)	
H30	0.6902 (17)	0.6382 (13)	0.0983 (12)	0.018*	

	x	*y*	*z*	*U*iso*/*U*eq	Occ. (<1)
C31	0.63492 (13)	0.42672 (10)	0.15748 (10)	0.0120 (2)	
C32	0.70195 (13)	0.43296 (11)	0.03392 (10)	0.0136 (2)	
H32A	0.6301 (16)	0.4380 (12)	−0.0062 (12)	0.016*	
H32B	0.7453 (16)	0.5032 (13)	−0.0016 (12)	0.016*	
C33	0.82798 (13)	0.32241 (11)	0.03197 (10)	0.0136 (2)	
C34	0.94274 (15)	0.32537 (13)	−0.07616 (11)	0.0192 (3)	
H34A	0.9001 (18)	0.3266 (14)	−0.1357 (14)	0.029*	
H34B	1.0270 (18)	0.2570 (15)	−0.0788 (13)	0.029*	
H34C	0.9843 (18)	0.3939 (15)	−0.0985 (14)	0.029*	
C35	0.78461 (13)	0.40429 (11)	0.19039 (11)	0.0137 (2)	
H35A	0.8275 (16)	0.4744 (13)	0.1577 (12)	0.016*	
H35B	0.7742 (16)	0.3900 (13)	0.2682 (13)	0.016*	
C36	0.88330 (13)	0.30325 (11)	0.13779 (11)	0.0139 (2)	
C37	1.04975 (14)	0.28896 (13)	0.12704 (13)	0.0200 (3)	
H37A	1.1136 (18)	0.2254 (15)	0.0878 (14)	0.03*	
H37B	1.0762 (18)	0.2703 (14)	0.1943 (14)	0.03*	
H37C	1.0831 (18)	0.3645 (15)	0.0862 (14)	0.03*	
C40	0.3441 (2)	0.27165 (19)	0.57125 (18)	0.0248 (6)	0.758 (5)
H40	0.309 (2)	0.2901 (19)	0.5062 (19)	0.03*	0.758 (5)
Cl41	0.46921 (15)	0.13392 (15)	0.58104 (11)	0.0452 (3)	0.758 (5)
Cl42	0.18552 (19)	0.27006 (17)	0.68161 (14)	0.0411 (4)	0.758 (5)
Cl43	0.43489 (9)	0.37886 (13)	0.56581 (10)	0.0400 (3)	0.758 (5)
C50	0.3820 (8)	0.2198 (6)	0.6275 (7)	0.034 (2)	0.242 (5)
H50	0.427 (8)	0.205 (6)	0.688 (6)	0.041*	0.242 (5)
Cl51	0.4555 (5)	0.0991 (4)	0.5688 (3)	0.0396 (7)	0.242 (5)

Cl52	0.1873 (8)	0.2395 (6)	0.6702 (6)	0.0596 (16)	0.242 (5)
Cl53	0.4299 (3)	0.3473 (3)	0.5356 (4)	0.0395 (9)	0.242 (5)

Atomic displacement parameters (Å2)

	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
C1	0.0118 (5)	0.0140 (6)	0.0117 (6)	−0.0023 (4)	−0.0029 (4)	−0.0038 (5)
C2	0.0123 (5)	0.0138 (6)	0.0126 (6)	−0.0030 (4)	−0.0030 (5)	−0.0036 (5)
C3	0.0127 (5)	0.0147 (6)	0.0163 (7)	−0.0002 (5)	−0.0034 (5)	−0.0049 (5)
C4	0.0130 (5)	0.0177 (6)	0.0119 (6)	−0.0037 (5)	−0.0025 (5)	−0.0043 (5)
C5	0.0134 (6)	0.0222 (7)	0.0178 (7)	−0.0031 (5)	−0.0028 (5)	−0.0055 (6)
C6	0.0149 (6)	0.0294 (8)	0.0177 (7)	−0.0085 (5)	0.0011 (5)	−0.0067 (6)
C7	0.0223 (7)	0.0236 (8)	0.0231 (8)	−0.0118 (6)	0.0023 (6)	−0.0073 (6)
C8	0.0207 (6)	0.0179 (7)	0.0216 (7)	−0.0070 (5)	0.0003 (5)	−0.0062 (6)
C9	0.0157 (6)	0.0161 (6)	0.0132 (6)	−0.0051 (5)	−0.0025 (5)	−0.0039 (5)
C10	0.0156 (6)	0.0142 (6)	0.0165 (7)	−0.0023 (5)	−0.0023 (5)	−0.0055 (5)
C11	0.0112 (5)	0.0109 (6)	0.0133 (6)	−0.0017 (4)	−0.0024 (4)	−0.0032 (5)
C12	0.0136 (5)	0.0127 (6)	0.0136 (6)	−0.0011 (5)	−0.0036 (5)	−0.0017 (5)
C13	0.0127 (5)	0.0126 (6)	0.0155 (6)	0.0007 (4)	−0.0047 (5)	−0.0035 (5)
C14	0.0169 (6)	0.0150 (7)	0.0250 (8)	0.0015 (5)	−0.0072 (6)	−0.0033 (6)
C15	0.0112 (5)	0.0147 (6)	0.0156 (6)	−0.0021 (5)	−0.0023 (5)	−0.0066 (5)
C16	0.0123 (5)	0.0138 (6)	0.0161 (6)	0.0009 (4)	−0.0033 (5)	−0.0064 (5)
C17	0.0178 (6)	0.0192 (7)	0.0243 (8)	0.0013 (5)	−0.0047 (6)	−0.0117 (6)
C21	0.0121 (5)	0.0129 (6)	0.0127 (6)	−0.0008 (4)	−0.0041 (5)	−0.0036 (5)
C22	0.0118 (5)	0.0131 (6)	0.0125 (6)	−0.0020 (4)	−0.0036 (4)	−0.0035 (5)
C23	0.0118 (5)	0.0150 (6)	0.0156 (6)	−0.0006 (5)	−0.0025 (5)	−0.0050 (5)
C24	0.0148 (6)	0.0129 (6)	0.0143 (6)	0.0003 (5)	−0.0052 (5)	−0.0043 (5)
C25	0.0174 (6)	0.0156 (6)	0.0173 (7)	0.0008 (5)	−0.0048 (5)	−0.0061 (5)

	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
C26	0.0235 (6)	0.0126 (6)	0.0196 (7)	0.0004 (5)	−0.0081 (5)	−0.0057 (5)
C27	0.0258 (7)	0.0126 (6)	0.0212 (7)	−0.0050 (5)	−0.0083 (6)	−0.0031 (5)
C28	0.0186 (6)	0.0155 (7)	0.0207 (7)	−0.0044 (5)	−0.0034 (5)	−0.0037 (5)
C29	0.0159 (6)	0.0119 (6)	0.0150 (6)	−0.0024 (5)	−0.0054 (5)	−0.0021 (5)
C30	0.0128 (5)	0.0139 (6)	0.0170 (6)	−0.0021 (5)	−0.0012 (5)	−0.0046 (5)
C31	0.0103 (5)	0.0116 (6)	0.0132 (6)	−0.0013 (4)	−0.0023 (4)	−0.0030 (5)
C32	0.0111 (5)	0.0135 (6)	0.0133 (6)	−0.0005 (4)	−0.0017 (5)	−0.0023 (5)
C33	0.0102 (5)	0.0160 (6)	0.0131 (6)	−0.0008 (4)	−0.0010 (4)	−0.0048 (5)
C34	0.0146 (6)	0.0229 (7)	0.0161 (7)	−0.0013 (5)	0.0000 (5)	−0.0054 (6)
C35	0.0122 (5)	0.0135 (6)	0.0160 (6)	−0.0025 (4)	−0.0035 (5)	−0.0043 (5)
C36	0.0101 (5)	0.0144 (6)	0.0162 (6)	−0.0003 (4)	−0.0033 (5)	−0.0039 (5)
C37	0.0126 (6)	0.0219 (7)	0.0254 (8)	−0.0013 (5)	−0.0062 (5)	−0.0060 (6)
C40	0.0194 (9)	0.0339 (13)	0.0175 (11)	0.0027 (8)	−0.0056 (8)	−0.0074 (9)
Cl41	0.0427 (5)	0.0413 (7)	0.0326 (5)	0.0137 (4)	−0.0067 (3)	−0.0049 (4)
Cl42	0.0221 (3)	0.0531 (9)	0.0315 (4)	0.0013 (5)	0.0032 (3)	−0.0041 (5)
Cl43	0.0283 (3)	0.0533 (6)	0.0443 (5)	−0.0096 (3)	−0.0133 (3)	−0.0142 (4)
C50	0.035 (4)	0.029 (4)	0.036 (5)	0.005 (3)	−0.013 (3)	−0.012 (3)
Cl51	0.0550 (14)	0.0305 (17)	0.0339 (14)	−0.0037 (12)	−0.0189 (10)	−0.0052 (11)
Cl52	0.0403 (16)	0.054 (3)	0.072 (3)	−0.0155 (16)	0.0152 (16)	−0.0206 (18)
Cl53	0.0345 (10)	0.0386 (14)	0.0407 (16)	−0.0073 (9)	−0.0034 (10)	−0.0086 (11)

Geometric parameters (Å, º)

C1—C10 1.3813 (17)	C21—C31 1.5217 (17)
C1—C2 1.4276 (17)	C22—C23 1.3852 (17)
C1—C11 1.5184 (16)	C23—C24 1.4084 (18)
C2—C3 1.3844 (17)	C23—H23 0.977 (15)

C2—C22 1.4848 (17)
C3—C4 1.4093 (18)
C3—H3 0.947 (15)
C4—C9 1.4168 (18)
C4—C5 1.4251 (17)
C5—C6 1.3650 (19)
C5—H5 0.983 (16)
C6—C7 1.412 (2)
C6—H6 0.971 (16)
C7—C8 1.3663 (19)
C7—H7 0.974 (17)
C8—C9 1.4228 (18)
C8—H8 0.995 (16)
C9—C10 1.4111 (17)
C10—H10 0.988 (15)
C11—C15 1.5492 (17)
C11—C12 1.5556 (17)
C11—C31 1.5986 (17)
C12—C13 1.5280 (18)
C12—H12A 0.994 (15)
C12—H12B 0.993 (15)
C13—C14 1.5175 (18)
C13—C36 1.5714 (17)
C13—C16 1.5788 (17)
C14—H14A 0.949 (18)
C14—H14B 1.003 (17)
C24—C29 1.4170 (17)
C24—C25 1.4239 (18)
C25—C26 1.3647 (19)
C25—H25 0.961 (15)
C26—C27 1.4140 (19)
C26—H26 0.963 (16)
C27—C28 1.3654 (19)
C27—H27 0.982 (16)
C28—C29 1.4176 (18)
C28—H28 0.953 (16)
C29—C30 1.4088 (18)
C30—H30 0.972 (15)
C31—C35 1.5531 (16)
C31—C32 1.5536 (18)
C32—C33 1.5353 (17)
C32—H32A 0.970 (15)
C32—H32B 0.998 (15)
C33—C34 1.5161 (18)
C33—C36 1.5790 (17)
C34—H34A 0.988 (17)
C34—H34B 0.983 (17)
C34—H34C 0.967 (17)
C35—C36 1.5275 (18)
C35—H35A 0.991 (15)
C35—H35B 0.973 (15)
C36—C37 1.5216 (16)

C14—H14C 0.990 (17)
C15—C16 1.5297 (16)
C15—H15A 0.999 (15)
C15—H15B 0.974 (15)
C16—C17 1.5186 (18)
C16—C33 1.5697 (18)
C17—H17A 0.977 (17)
C17—H17B 0.960 (18)
C17—H17C 0.936 (17)
C21—C30 1.3800 (18)
C21—C22 1.4256 (16)
C10—C1—C2 118.40 (11)
C10—C1—C11 118.31 (11)
C2—C1—C11 123.27 (11)
C3—C2—C1 118.90 (11)
C3—C2—C22 120.79 (11)
C1—C2—C22 120.31 (10)
C2—C3—C4 122.80 (12)
C2—C3—H3 118.3 (9)
C4—C3—H3 118.8 (9)
C3—C4—C9 118.37 (11)
C3—C4—C5 122.36 (12)
C9—C4—C5 119.28 (12)
C6—C5—C4 120.24 (13)
C6—C5—H5 121.7 (9)
C4—C5—H5 118.0 (9)
C37—H37A 1.008 (17)
C37—H37B 0.948 (17)
C37—H37C 1.019 (17)
C40—Cl43 1.753 (3)
C40—Cl42 1.760 (3)
C40—Cl41 1.765 (2)
C40—H40 0.96 (2)
C50—Cl51 1.737 (8)
C50—Cl52 1.744 (10)
C50—Cl53 1.771 (8)
C50—H50 0.97 (8)
C22—C23—C24 122.63 (11)
C22—C23—H23 119.8 (9)
C24—C23—H23 117.5 (9)
C23—C24—C29 118.44 (11)
C23—C24—C25 122.33 (12)
C29—C24—C25 119.23 (12)
C26—C25—C24 120.37 (12)
C26—C25—H25 122.6 (9)
C24—C25—H25 117.0 (9)
C25—C26—C27 120.40 (12)
C25—C26—H26 119.3 (9)
C27—C26—H26 120.3 (9)
C28—C27—C26 120.42 (13)
C28—C27—H27 118.4 (9)
C26—C27—H27 121.2 (9)

C5—C6—C7 120.40 (12)
C5—C6—H6 120.9 (10)
C7—C6—H6 118.7 (10)
C8—C7—C6 120.84 (13)
C8—C7—H7 118.2 (10)
C6—C7—H7 120.9 (10)
C7—C8—C9 120.19 (13)
C7—C8—H8 123.1 (9)
C9—C8—H8 116.7 (9)
C10—C9—C4 118.24 (11)
C10—C9—C8 122.75 (12)
C4—C9—C8 119.00 (11)
C1—C10—C9 123.17 (12)
C1—C10—H10 120.7 (9)
C9—C10—H10 116.1 (9)
C1—C11—C15 110.72 (9)
C1—C11—C12 112.34 (10)
C15—C11—C12 97.69 (10)
C1—C11—C31 116.32 (10)
C15—C11—C31 109.00 (10)
C12—C11—C31 109.16 (9)
C13—C12—C11 102.55 (10)
C13—C12—H12A 114.8 (8)
C11—C12—H12A 113.9 (8)
C13—C12—H12B 111.2 (8)
C11—C12—H12B 109.5 (8)
C27—C28—C29 120.76 (13)
C27—C28—H28 122.0 (9)
C29—C28—H28 117.2 (9)
C30—C29—C24 118.24 (11)
C30—C29—C28 122.92 (12)
C24—C29—C28 118.83 (11)
C21—C30—C29 123.21 (11)
C21—C30—H30 121.1 (9)
C29—C30—H30 115.6 (9)
C21—C31—C35 111.31 (10)
C21—C31—C32 112.27 (10)
C35—C31—C32 97.66 (9)
C21—C31—C11 115.84 (9)
C35—C31—C11 108.98 (9)
C32—C31—C11 109.24 (10)
C33—C32—C31 102.48 (10)
C33—C32—H32A 113.6 (9)
C31—C32—H32A 114.5 (9)
C33—C32—H32B 109.8 (8)
C31—C32—H32B 111.2 (8)
H32A—C32—H32B 105.3 (12)
C34—C33—C32 113.21 (11)
C34—C33—C16 118.00 (11)
C32—C33—C16 109.81 (9)
C34—C33—C36 118.75 (10)
C32—C33—C36 104.45 (10)

H12A—C12—H12B 104.9 (12)
C14—C13—C12 113.41 (11)
C14—C13—C36 117.88 (10)
C12—C13—C36 110.03 (10)
C14—C13—C16 118.37 (11)
C12—C13—C16 104.56 (9)
C36—C13—C16 89.80 (9)
C13—C14—H14A 115.2 (10)
C13—C14—H14B 112.3 (9)
H14A—C14—H14B 103.9 (14)
C13—C14—H14C 110.2 (10)
H14A—C14—H14C 107.7 (13)
H14B—C14—H14C 106.9 (13)
C16—C15—C11 102.81 (9)
C16—C15—H15A 111.0 (8)
C11—C15—H15A 108.8 (8)
C16—C15—H15B 115.4 (8)
C11—C15—H15B 115.2 (8)
H15A—C15—H15B 103.8 (11)
C17—C16—C15 113.31 (10)
C17—C16—C33 117.83 (11)
C15—C16—C33 110.06 (10)
C17—C16—C13 118.51 (11)
C15—C16—C13 104.18 (10)
C33—C16—C13 90.20 (9)
C16—C17—H17A 111.0 (10)
C16—C33—C36 89.86 (9)
C33—C34—H34A 112.8 (10)
C33—C34—H34B 115.0 (10)
H34A—C34—H34B 102.7 (13)
C33—C34—H34C 111.9 (10)
H34A—C34—H34C 106.0 (13)
H34B—C34—H34C 107.7 (13)
C36—C35—C31 102.87 (10)
C36—C35—H35A 110.1 (9)
C31—C35—H35A 108.8 (8)
C36—C35—H35B 114.6 (9)
C31—C35—H35B 114.3 (8)
H35A—C35—H35B 106.1 (12)
C37—C36—C35 113.13 (11)
C37—C36—C13 118.07 (10)
C35—C36—C13 109.89 (10)
C37—C36—C33 118.63 (11)
C35—C36—C33 104.27 (9)
C13—C36—C33 90.13 (9)
C36—C37—H37A 112.1 (9)
C36—C37—H37B 114.0 (10)
H37A—C37—H37B 106.1 (13)
C36—C37—H37C 110.8 (9)
H37A—C37—H37C 108.4 (13)
H37B—C37—H37C 105.1 (13)
Cl43—C40—Cl42 110.34 (15)

C16—C17—H17B 112.1 (10)
H17A—C17—H17B 106.5 (13)
C16—C17—H17C 112.1 (10)
H17A—C17—H17C 108.0 (14)
H17B—C17—H17C 106.7 (14)
C30—C21—C22 118.43 (11)
C30—C21—C31 118.24 (10)
C22—C21—C31 123.33 (11)
C23—C22—C21 119.01 (11)
C23—C22—C2 120.32 (11)
C21—C22—C2 120.67 (11)
C10—C1—C2—C3 −3.58 (18)
C11—C1—C2—C3 174.86 (11)
C10—C1—C2—C22 176.55 (11)
C11—C1—C2—C22 −5.01 (18)
C1—C2—C3—C4 2.18 (19)
C22—C2—C3—C4 −177.95 (11)
C2—C3—C4—C9 1.16 (19)
C2—C3—C4—C5 −178.89 (12)
C3—C4—C5—C6 −179.85 (12)
C9—C4—C5—C6 0.10 (19)
C4—C5—C6—C7 1.2 (2)
C5—C6—C7—C8 −0.8 (2)
C6—C7—C8—C9 −0.9 (2)
C3—C4—C9—C10 −2.99 (18)
C5—C4—C9—C10 177.06 (12)
Cl43—C40—Cl41 110.22 (13)
Cl42—C40—Cl41 110.67 (14)
Cl43—C40—H40 109.0 (13)
Cl42—C40—H40 107.4 (13)
Cl41—C40—H40 109.2 (13)
Cl51—C50—Cl52 109.2 (5)
Cl51—C50—Cl53 111.6 (5)
Cl52—C50—Cl53 109.7 (5)
Cl51—C50—H50 107 (4)
Cl52—C50—H50 112 (4)
Cl53—C50—H50 107 (4)
C26—C27—C28—C29 0.4 (2)
C23—C24—C29—C30 −1.28 (18)
C25—C24—C29—C30 179.76 (11)
C23—C24—C29—C28 178.75 (11)
C25—C24—C29—C28 −0.21 (18)
C27—C28—C29—C30 179.82 (13)
C27—C28—C29—C24 −0.22 (19)
C22—C21—C30—C29 1.05 (19)
C31—C21—C30—C29 −178.35 (11)
C24—C29—C30—C21 0.68 (19)
C28—C29—C30—C21 −179.35 (12)
C30—C21—C31—C35 52.89 (15)
C22—C21—C31—C35 −126.49 (12)
C30—C21—C31—C32 −55.43 (14)
C22—C21—C31—C32 125.19 (12)

C3—C4—C9—C8 178.23 (12)
C5—C4—C9—C8 −1.72 (18)
C7—C8—C9—C10 −176.61 (13)
C7—C8—C9—C4 2.1 (2)
C2—C1—C10—C9 1.76 (19)
C11—C1—C10—C9 −176.76 (11)
C4—C9—C10—C1 1.57 (19)
C8—C9—C10—C1 −179.70 (12)
C10—C1—C11—C15 54.31 (15)
C2—C1—C11—C15 −124.14 (12)
C10—C1—C11—C12 −53.76 (15)
C2—C1—C11—C12 127.80 (12)
C10—C1—C11—C31 179.43 (10)
C2—C1—C11—C31 0.99 (17)
C1—C11—C12—C13 166.02 (10)
C15—C11—C12—C13 49.82 (11)
C31—C11—C12—C13 −63.42 (12)
C11—C12—C13—C14 −161.50 (10)
C11—C12—C13—C36 64.07 (12)
C11—C12—C13—C16 −31.15 (12)
C1—C11—C15—C16 −167.47 (10)
C12—C11—C15—C16 −50.01 (11)
C31—C11—C15—C16 63.37 (12)
C11—C15—C16—C17 161.65 (11)
C11—C15—C16—C33 −64.03 (12)
C11—C15—C16—C13 31.48 (12)
C30—C21—C31—C11 178.12 (10)
C22—C21—C31—C11 −1.25 (17)
C1—C11—C31—C21 2.09 (15)
C15—C11—C31—C21 128.08 (10)
C12—C11—C31—C21 −126.29 (11)
C1—C11—C31—C35 128.51 (11)
C15—C11—C31—C35 −105.50 (11)
C12—C11—C31—C35 0.13 (13)
C1—C11—C31—C32 −125.87 (10)
C15—C11—C31—C32 0.13 (12)
C12—C11—C31—C32 105.76 (11)
C21—C31—C32—C33 166.63 (9)
C35—C31—C32—C33 49.79 (11)
C11—C31—C32—C33 −63.45 (11)
C31—C32—C33—C34 −161.83 (10)
C31—C32—C33—C16 63.96 (12)
C31—C32—C33—C36 −31.21 (11)
C17—C16—C33—C34 0.10 (16)
C15—C16—C33—C34 −131.90 (11)
C13—C16—C33—C34 122.90 (11)
C17—C16—C33—C32 131.86 (11)
C15—C16—C33—C32 −0.15 (14)
C13—C16—C33—C32 −105.34 (10)
C17—C16—C33—C36 −122.81 (11)
C15—C16—C33—C36 105.18 (10)
C13—C16—C33—C36 −0.01 (8)

C14—C13—C16—C17 0.27 (17)	C21—C31—C35—C36 −167.71 (10)
C12—C13—C16—C17 −127.09 (12)	C32—C31—C35—C36 −50.13 (11)
C36—C13—C16—C17 122.24 (12)	C11—C31—C35—C36 63.31 (12)
C14—C13—C16—C15 127.26 (12)	C31—C35—C36—C37 161.74 (11)
C12—C13—C16—C15 −0.10 (13)	C31—C35—C36—C13 −63.94 (12)
C36—C13—C16—C15 −110.76 (10)	C31—C35—C36—C33 31.49 (12)