Supporting Information

An Efficient Synthesis of a Novel, Twisted and Stable, Electroluminescent "Twistacene"

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Materials. Reagents were purchased from Aldrich. THF was distilled over sodium/benzophenone under nitrogen. Methylene chloride was distilled from calcium hydride under nitrogen. All experiments were carried out under argon or nitrogen. Other solvents and reagents were used as received.

Measurements. Solution NMR spectra were taken on a Bruker ARX 400 and 500 spectrometers. All chemical shifts (δ) are reported relative to tetramethylsilane (TMS) at 0.0 ppm.

X-ray data were collected on a Bruker Smart 1K X-ray diffractometer equipped with a large area CCD detector. Single crystals of each compound were mounted on a glass fiber and diffraction data were acquired using a MoK_α radiation source (λ = 0.71073 Å). Structure solution and refinement were conducted using a SHELXTL package from Bruker. The data was collected on a very small crystal (.08x.08x.13mm) that gave a weak set of reflection with a large number of very weak or unobserved data with aveI/sigI of 4.35 only. A relatively large R(int) is due to limited number of averaged data which was observed. Moreover there is a disordered THF molecule. Because of limited data (only 32% observed above 2-sigma) and disorder solvent the structure is of rather low quality with low bond precision on C-C bonds. The disordered solvent THF could not be refined anisotropically because of low data to parameter ratio, which further contributes to the resultant high weighted R-factor. FT-IR spectra were recorded on a Mattson

Infinity IIa spectrometer with a DRIFT accessory from PIKE Technologies, on a diamond frit. UV-visible spectra were obtained on a Hewlett Packard 8453 spectrophotometer. Fluorescence spectra were recorded on an ISA Fluorolog2 in a 1 cm quartz cuvette using toluene. The quantum efficiency was calculated using diphenylanthracene (Aldrich) as the reference. Melting points were measured using a capillary melting point apparatus and are uncorrected. Thermo-gravimetric analysis (TGA) was carried out on a Perkin Elmer TAC 7/DX Thermal Analyst system at a heating rate of 10 ^{o}C/min and at a nitrogen flow rate of 75 cm^3/min. Differential scanning calorimetry (DSC) was run on a Perkin Elmer DSC Pyris instrument.

Electrochemistry was carried out with a BAS 100B/W potentiostat, employing a platinum button (diameter: 1.6 mm; area 0.02 cm^2), a platinum wire and a 0.01 M $Ag/AgNO_3$ (Ag/Ag^+) as working, counter and reference electrode, respectively. 0.1 M of tetrabutylammonium perchlorate (TBAP) in ODCB was used as electrolyte. Since compound **3** was not very soluble in ODCB at room temperature, CV measurements were conducted at higher temperatures (*ca.* 150 °C).

EI-MS was taken using a VG Autospec EI/CI Magnetic Sector spectrometer. MALDI-TOF was taken using an Applied Biostems DE-STR MALDI-TOF spectrometer. Elemental analysis was performed by Desert Analytics Lab.

The GAUSSIAN 98[1] series of programs was used for all calculations. All molecules were fully optimized using the hybrid density functional[2] at B3LYP level[3] of theory with the 6-31G(d) basis set.

2,5-[Bis(trimethylsilyl)]phenyl-1,4-trifluoromethanesulfonate (5) – Triflic anhydride (2.77 g, 9.8 mmol) was added dropwise to a solution of the hydroquinone **4**[4] (1 g, 3.9 mmol) in 25 mL pyridine (freshly distilled over CaH_2) at 0 °C under N_2 atmosphere. The reaction was warmed to room temperature followed by heating at 50 °C for 1 hour. Pyridine was removed under reduced pressure to give a black residue. The residue was extracted using hexanes by successive heating and decanting. The combined organic fractions were concentrated to give a light yellow solid. Recrystallization using hexanes gave triflate **5** (1.8 g, 88%) as colorless solid: mp 123-125 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45 (s, 2H), 0.38 (s, 18H); ^{13}C NMR ($CDCl_3$, 400 MHz) δ 153.1, 137.4, 127.0, 118.5 (q, J = 318 Hz, CF_3), -1.3 ppm; IR (Drift) 3115, 2964, 2907, 1460, 1415,

1338, 1251, 1216, 1136, 1076, 883, 841, 766, 715, 629, 595, 509 cm^{-1}; EI-MS exact mass 518.0147, calcd mass 518.0144.

6,8,15,17-Tetraphenyl-1.18,4.5,9.10,13.14-tetrabenzoheptacene (3) – To a solution of cyclone **6**[5] (500 mg, 1.23 mmol) and triflate **5** (314 mg, 0.61 mmol) in 50 mL CH_2Cl_2 was added TBAF (1.23 mL of 1M THF solution, 1.23 mmol) dropwise. After stirring at rt for 48 h, the solution was refluxed for 24 h to give an orange solid precipitate collected by vacuum filtration. Successive washes (3X, 5mL) using THF followed by centrifugations gave **3** (110 mg, 22%) as an orange solid; IR (Drift) 3050, 1947, 1808, 1599, 1491, 1443, 1384, 1308, 1075, 957, 905, 860, 770, 730, 539 cm^{-1}; Elemental analysis, found C, 95.12, H, 4.76; calcd C, 95.39, H, 4.61; MALDI-TOF MS found 830.2940; calcd 830.2974; UV-vis (toluene) λ_{max} 306, 340, 381, 400, 464, 485, 528 nm.

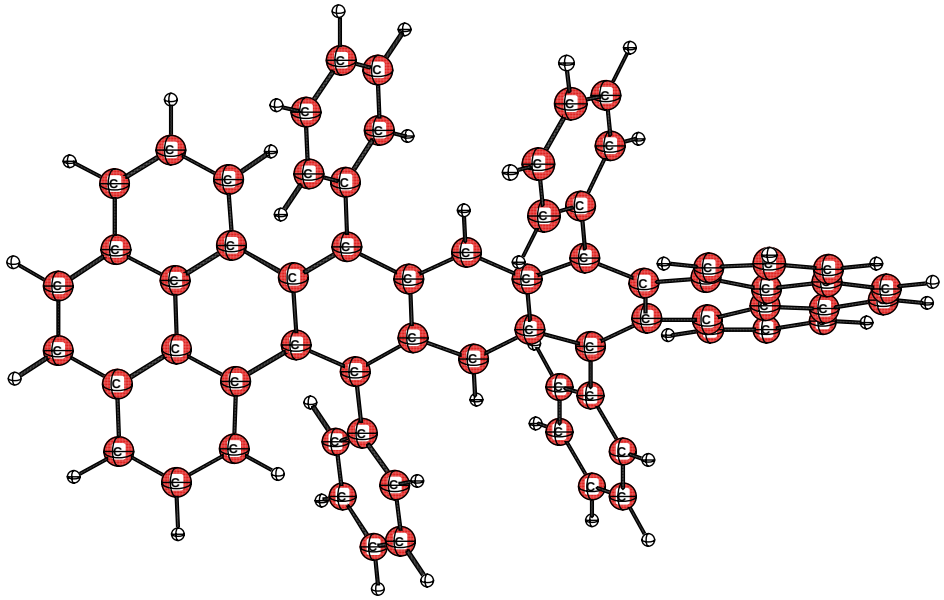


Figure S1 Calculated global minimum (**3a**) conformer for **3**.

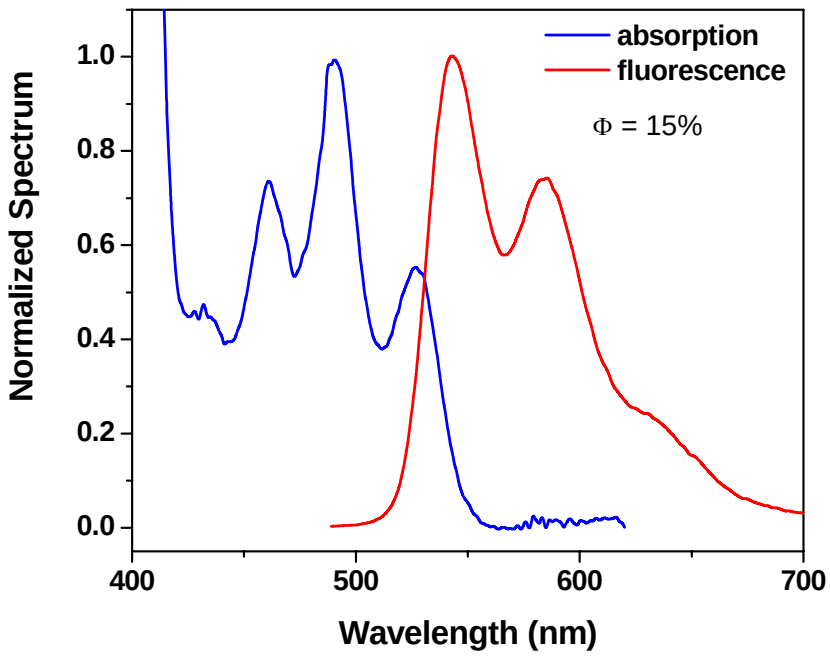


Figure S2 Absorption and fluorescence spectra of **3** in toluene.

Table S1. Calculated absolute energies at B3LYP/6-31G(d) for **3**, and **3a**.[a]

cpd.	B3LYP/6-31G(d)
3	-2538.014502
3a	-2538.018900

[a] in Hartrees.

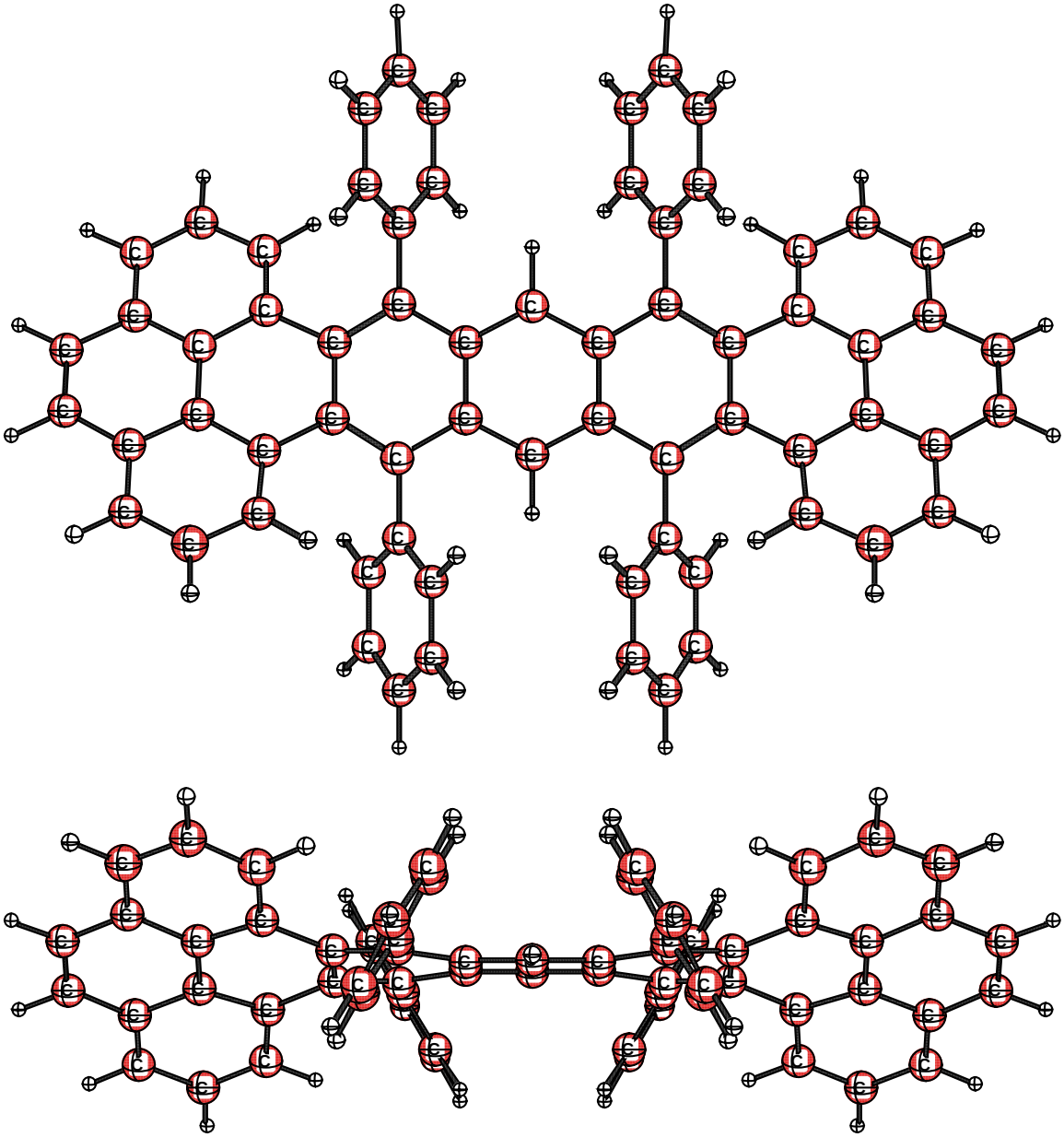
Table S2. Calculated HOMO and LUMO energies and the HOMO-LUMO gap at B3LYP/6-31G(d) for **3**, and **3a**.[a]

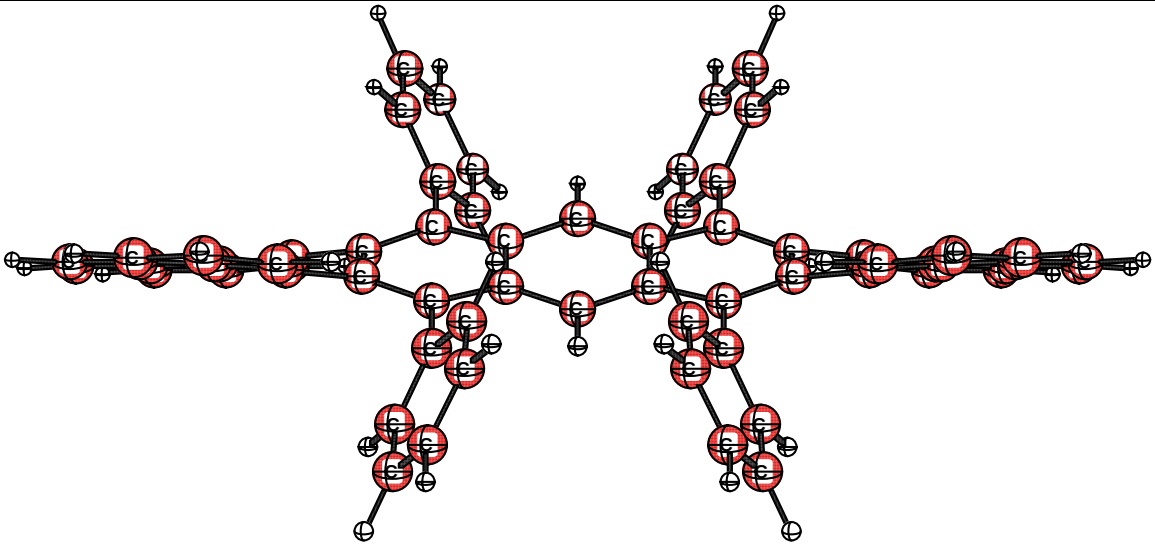
	HOMO	LUMO	HOMO-LUMO gap
3	-4.74	-2.11	2.63
3a	-4.71	-2.10	2.61
Tetracene	-4.86	-2.08	2.78
Pentacene	-4.60	-2.39	2.21

[a] in eV.

Table S3. Optimized geometries (at B3LYP/6-31G(d)).

3





1	6	0	1.428796	0.071986	2.485748
2	6	0	1.428796	0.071986	-2.485748
3	6	0	-1.428796	-0.071986	2.485748
4	6	0	-1.428796	-0.071986	-2.485748
5	1	0	-2.787122	-2.133624	3.542534
6	1	0	-2.787122	-2.133624	-3.542534
7	1	0	2.787122	2.133624	-3.542534
8	1	0	2.787122	2.133624	3.542534
9	1	0	-5.230844	-2.534336	3.550289
10	1	0	-5.230844	-2.534336	-3.550289
11	1	0	5.230844	2.534336	3.550289
12	1	0	5.230844	2.534336	-3.550289
13	1	0	-5.830498	1.139214	1.396111
14	1	0	-5.830498	1.139214	-1.396111
15	1	0	5.830498	-1.139214	-1.396111
16	1	0	5.830498	-1.139214	1.396111
17	6	0	-3.455117	-1.420107	3.068771
18	6	0	-3.455117	-1.420107	-3.068771
19	6	0	3.455117	1.420107	-3.068771
20	6	0	3.455117	1.420107	3.068771
21	1	0	-6.767789	-0.890134	2.487504
22	1	0	-6.767789	-0.890134	-2.487504
23	1	0	6.767789	0.890134	2.487504
24	1	0	6.767789	0.890134	-2.487504
25	6	0	-4.832003	-1.641866	3.075267
26	6	0	-4.832003	-1.641866	-3.075267
27	6	0	4.832003	1.641866	-3.075267
28	6	0	4.832003	1.641866	3.075267
29	6	0	0.713677	0.104873	1.235253
30	6	0	0.713677	0.104873	-1.235253
31	6	0	-0.713677	-0.104873	1.235253
32	6	0	-0.713677	-0.104873	-1.235253
33	6	0	1.367176	0.246849	0.000000
34	6	0	-1.367176	-0.246849	0.000000
35	1	0	-2.429500	-0.441603	0.000000
36	1	0	2.429500	0.441603	0.000000
37	6	0	-5.694339	-0.721426	2.477736
38	6	0	-5.694339	-0.721426	-2.477736

39	6	0	5.694339	0.721426	-2.477736
40	6	0	5.694339	0.721426	2.477736
41	6	0	-5.167358	0.415938	1.863214
42	6	0	-5.167358	0.415938	-1.863214
43	6	0	5.167358	-0.415938	-1.863214
44	6	0	5.167358	-0.415938	1.863214
45	1	0	-3.386057	1.524809	1.378551
46	1	0	-3.386057	1.524809	-1.378551
47	1	0	3.386057	-1.524809	-1.378551
48	1	0	3.386057	-1.524809	1.378551
49	6	0	-3.789295	0.631526	1.847995
50	6	0	-3.789295	0.631526	-1.847995
51	6	0	3.789295	-0.631526	-1.847995
52	6	0	3.789295	-0.631526	1.847995
53	6	0	0.724021	-0.082219	3.682510
54	6	0	0.724021	-0.082219	-3.682510
55	6	0	-0.724021	0.082219	3.682510
56	6	0	-0.724021	0.082219	-3.682510
57	6	0	2.912010	0.276237	2.463421
58	6	0	2.912010	0.276237	-2.463421
59	6	0	-2.912010	-0.276237	2.463421
60	6	0	-2.912010	-0.276237	-2.463421
61	6	0	1.357364	-0.483476	4.963277
62	6	0	1.357364	-0.483476	-4.963277
63	6	0	-1.357364	0.483476	4.963277
64	6	0	-1.357364	0.483476	-4.963277
65	6	0	-0.661387	0.279498	6.190103
66	6	0	-0.661387	0.279498	-6.190103
67	6	0	0.661387	-0.279498	6.190103
68	6	0	0.661387	-0.279498	-6.190103
69	6	0	-2.584029	1.157330	5.021222
70	6	0	-2.584029	1.157330	-5.021222
71	6	0	2.584029	-1.157330	5.021222
72	6	0	2.584029	-1.157330	-5.021222
73	6	0	-1.278856	0.617621	7.432097
74	6	0	-1.278856	0.617621	-7.432097
75	6	0	1.278856	-0.617621	7.432097
76	6	0	1.278856	-0.617621	-7.432097
77	6	0	-2.538782	1.244022	7.434376
78	6	0	-2.538782	1.244022	-7.434376
79	6	0	2.538782	-1.244022	7.434376
80	6	0	2.538782	-1.244022	-7.434376
81	6	0	0.606201	-0.306934	8.660637
82	6	0	0.606201	-0.306934	-8.660637
83	6	0	-0.606201	0.306934	-8.660637
84	6	0	-0.606201	0.306934	8.660637
85	1	0	1.099779	-0.560708	9.595677
86	1	0	1.099779	-0.560708	-9.595677
87	1	0	-1.099779	0.560708	9.595677
88	1	0	-1.099779	0.560708	-9.595677
89	6	0	3.164880	-1.535732	-6.236483
90	6	0	3.164880	-1.535732	6.236483
91	6	0	-3.164880	1.535732	-6.236483
92	6	0	-3.164880	1.535732	6.236483
93	1	0	3.000297	-1.505436	-8.383516
94	1	0	3.000297	-1.505436	8.383516
95	1	0	-3.000297	1.505436	-8.383516

96	1	0	-3.000297	1.505436	8.383516
97	1	0	3.104873	-1.411431	-4.110901
98	1	0	3.104873	-1.411431	4.110901
99	1	0	-3.104873	1.411431	-4.110901
100	1	0	-3.104873	1.411431	4.110901
101	1	0	4.118846	-2.055963	-6.228076
102	1	0	4.118846	-2.055963	6.228076
103	1	0	-4.118846	2.055963	-6.228076
104	1	0	-4.118846	2.055963	6.228076

3a

1	6	0	2.474722	1.395536	0.321508
2	1	0	1.201396	-2.774223	-2.325774
3	1	0	1.128432	-5.223130	-2.664727
4	1	0	3.554149	-5.775411	0.844816
5	6	0	1.717659	-3.431069	-1.631034
6	1	0	2.309979	-6.739026	-1.083350
7	6	0	1.679164	-4.812200	-1.822661
8	6	0	1.229971	0.717189	0.072437
9	6	0	-0.000016	1.389324	-0.000035
10	6	0	2.474770	-1.395478	-0.321554
11	1	0	0.000020	-2.469807	-0.000030
12	6	0	1.229990	-0.717171	-0.072512
13	6	0	-1.229993	0.717171	-0.072511
14	1	0	-0.000022	2.469806	-0.000020
15	1	0	1.128570	5.222972	2.665131
16	6	0	-1.229975	-0.717189	0.072438
17	6	0	2.342010	-5.662843	-0.935889
18	6	0	0.000013	-1.389325	-0.000039
19	6	0	3.041095	-5.121414	0.144453
20	1	0	2.309554	6.739040	1.083498
21	6	0	-2.474775	1.395478	-0.321550
22	1	0	3.631344	-3.322613	1.172343
23	6	0	-2.474726	-1.395537	0.321512
24	6	0	3.084152	-3.740301	0.332082
25	6	0	3.669480	0.672328	0.282109
26	6	0	2.430382	2.872165	0.556587
27	6	0	3.669496	-0.672211	-0.282163
28	6	0	2.430530	-2.872117	-0.556585
29	6	0	4.947477	1.164707	0.849042
30	6	0	4.947498	-1.164559	-0.849101
31	6	0	-3.669500	0.672210	-0.282158
32	6	0	-2.430539	2.872118	-0.556581
33	6	0	-3.669485	-0.672330	0.282110
34	6	0	-2.430383	-2.872164	0.556601
35	6	0	-4.947484	-1.164710	0.849039
36	6	0	-4.947500	1.164558	-0.849101
37	6	0	6.173905	-0.554369	-0.456604
38	6	0	5.001310	-2.151312	-1.841660
39	6	0	6.173892	0.554480	0.456611
40	6	0	5.001285	2.151536	1.841527
41	6	0	8.643468	-0.493284	-0.467399

42	6	0	7.415082	-1.050135	-0.957534
43	6	0	7.414895	-2.080361	-1.916165
44	6	0	7.415062	1.050243	0.957563
45	6	0	8.643457	0.493358	0.467487
46	6	0	7.414865	2.080517	1.916142
47	1	0	9.578865	0.893690	0.851072
48	1	0	9.578885	-0.893630	-0.850948
49	6	0	-6.173908	0.554369	-0.456607
50	6	0	-5.001309	2.151313	-1.841658
51	6	0	-6.173898	-0.554481	0.456607
52	6	0	-5.001297	-2.151546	1.841516
53	6	0	-7.415084	1.050138	-0.957539
54	6	0	-7.415070	-1.050245	0.957553
55	6	0	-8.643472	0.493287	-0.467407
56	6	0	-7.414894	2.080365	-1.916168
57	6	0	-8.643464	-0.493357	0.467476
58	6	0	-7.414877	-2.080523	1.916127
59	1	0	-9.578887	0.893635	-0.850958
60	1	0	-9.578872	-0.893689	0.851057
61	6	0	-6.216230	2.599519	-2.371546
62	1	0	-8.363299	2.448599	-2.299867
63	1	0	-4.087757	2.579243	-2.227174
64	1	0	-6.208037	3.367201	-3.140528
65	6	0	-6.216213	-2.599749	2.371419
66	1	0	-8.363280	-2.448753	2.299836
67	1	0	-4.087752	-2.579538	2.226974
68	1	0	-6.208014	-3.367485	3.140346
69	6	0	6.216199	2.599738	2.371434
70	1	0	4.087738	2.579523	2.226984
71	1	0	8.363266	2.448746	2.299855
72	1	0	6.207997	3.367470	3.140366
73	6	0	6.216233	-2.599516	-2.371546
74	1	0	4.087760	-2.579243	-2.227177
75	1	0	8.363301	-2.448593	-2.299862
76	1	0	6.208043	-3.367197	-3.140529
77	6	0	-2.341995	5.662847	-0.935863
78	6	0	-3.084200	3.740298	0.332061
79	6	0	-1.717622	3.431076	-1.630997
80	6	0	-1.679114	4.812208	-1.822612
81	6	0	-3.041131	5.121411	0.144444
82	1	0	-3.631427	3.322606	1.172298
83	1	0	-1.201329	2.774235	-2.325719
84	1	0	-1.128342	5.223142	-2.664650
85	1	0	-3.554212	5.775405	0.844789
86	1	0	-2.309953	6.739031	-1.083313
87	6	0	-2.341634	-5.662864	0.936016
88	6	0	-1.717703	-3.430996	1.631238
89	6	0	-3.083693	-3.740447	-0.332195
90	6	0	-3.040525	-5.121550	-0.144510
91	6	0	-1.679093	-4.812116	1.822916
92	1	0	-1.201671	-2.774066	2.326071
93	1	0	-3.630735	-3.322845	-1.172598
94	1	0	-3.553339	-5.775629	-0.844971
95	1	0	-1.128508	-5.222957	2.665123
96	1	0	-2.309512	-6.739038	1.083517
97	6	0	2.341663	5.662866	0.935998
98	6	0	1.717726	3.431004	1.631236

99	6	0	3.083679	3.740441	-0.332226
100	6	0	3.040525	5.121544	-0.144542
101	6	0	1.679131	4.812125	1.822913
102	1	0	1.201705	2.774080	2.326082
103	1	0	3.630701	3.322832	-1.172638
104	1	0	3.553332	5.775618	-0.845015

Table S4. Crystal data and structure refinement for **3**.

Identification code	wd500ms	
Empirical formula	C74 H54 O2	
Formula weight	975.17	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 13.153(2) Å	α= 90°.
	b = 15.005(3) Å	β= 110.845(4)°.
	c = 14.196(3) Å	γ = 90°.
Volume	2618.3(9) $Å^3$	
Z	2	
Density (calculated)	1.237 Mg/m^3	
Absorption coefficient	0.073 mm^{-1}	
F(000)	1028	
Crystal size	0.15 x 0.08 x 0.08 mm^3	
Theta range for data collection	1.81 to 23.28°.	
Index ranges	-14<=h<=14, -8<=k<=16, -15<=l<=15	
Reflections collected	11706	
Independent reflections	3761 [R(int) = 0.1525]	
Completeness to theta = 23.28°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.9492	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3761 / 17 / 328	
Goodness-of-fit on F^2	0.992	
Final R indices [I>2sigma(I)]	R1 = 0.1020, wR2 = 0.2564	
R indices (all data)	R1 = 0.2848, wR2 = 0.3759	
Extinction coefficient	0.016(3)	
Largest diff. peak and hole	0.402 and -0.423 e.$Å^{-3}$	

Table S5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for wd500ms. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5832(7)	1742(6)	5640(7)	49(2)
C(2)	6084(7)	1985(6)	6636(7)	50(3)
C(3)	6257(7)	2924(7)	6998(8)	53(3)
C(4)	5861(8)	3656(7)	6353(8)	63(3)
C(5)	6054(9)	4518(7)	6720(9)	70(3)
C(6)	6610(9)	4690(7)	7699(10)	75(3)
C(7)	6963(8)	4006(7)	8379(9)	64(3)
C(8)	6752(7)	3097(7)	8037(8)	53(3)
C(9)	7536(9)	4150(9)	9444(10)	87(4)
C(10)	7882(9)	3468(9)	10105(9)	77(4)
C(11)	7671(8)	2566(8)	9792(9)	66(3)
C(12)	8065(8)	1865(9)	10449(8)	72(3)
C(13)	7865(8)	1006(8)	10140(8)	73(3)
C(14)	7231(7)	821(7)	9136(7)	62(3)
C(15)	6787(8)	1501(7)	8439(7)	56(3)
C(16)	7062(7)	2391(7)	8754(8)	55(3)
C(17)	6130(7)	1305(6)	7367(7)	45(2)
C(18)	5598(7)	505(6)	7066(6)	48(2)
C(19)	5245(7)	265(6)	6018(7)	46(2)
C(20)	5470(7)	853(6)	5318(7)	44(2)
C(21)	5276(7)	-73(6)	7757(7)	47(2)
C(22)	4556(8)	245(7)	8175(7)	63(3)
C(23)	4205(10)	-274(9)	8800(9)	80(3)
C(24)	4529(10)	-1141(9)	8996(9)	83(4)
C(25)	5234(9)	-1472(7)	8595(7)	71(3)
C(26)	5634(8)	-957(6)	7969(7)	58(3)
C(27)	5926(9)	2370(6)	4862(8)	56(3)
C(28)	5026(9)	2610(6)	4022(7)	61(3)
C(29)	5134(13)	3211(8)	3318(9)	90(4)
C(30)	6141(19)	3543(9)	3433(13)	114(6)
C(31)	7038(15)	3311(9)	4238(13)	113(5)

C(32)	6914(9)	2715(7)	4924(9)	72(3)
C(33)	4747(7)	-563(6)	5670(7)	50(3)
O(1A)	7015(16)	6650(20)	1840(16)	250(30)
C(2A)	7063(19)	5920(20)	1179(17)	108(16)
O(1B)	6320(20)	5957(15)	1945(10)	248(15)
C(2B)	6480(30)	5465(17)	1140(17)	260(20)
C(3A)	6139(15)	6051(12)	255(10)	221(10)
C(4A)	5427(12)	6678(11)	457(11)	214(9)
C(5A)	5839(16)	6819(12)	1526(12)	242(11)

Table S6. Bond lengths [Å] and angles [°] for wd500ms.

C(1)-C(2)	1.381(11)
C(1)-C(20)	1.435(11)
C(1)-C(27)	1.491(12)
C(2)-C(17)	1.441(12)
C(2)-C(3)	1.489(12)
C(3)-C(4)	1.406(12)
C(3)-C(8)	1.409(12)
C(4)-C(5)	1.384(13)
C(4)-H(4)	0.9300
C(5)-C(6)	1.346(13)
C(5)-H(5)	0.9300
C(6)-C(7)	1.371(14)
C(6)-H(6)	0.9300
C(7)-C(8)	1.441(13)
C(7)-C(9)	1.446(14)
C(8)-C(16)	1.425(13)
C(9)-C(10)	1.353(15)
C(9)-H(9)	0.9300
C(10)-C(11)	1.421(14)
C(10)-H(10)	0.9300
C(11)-C(12)	1.378(14)
C(11)-C(16)	1.429(13)
C(12)-C(13)	1.357(14)
C(12)-H(12)	0.9300
C(13)-C(14)	1.399(13)
C(13)-H(13)	0.9300
C(14)-C(15)	1.397(12)
C(14)-H(14)	0.9300
C(15)-C(16)	1.413(13)
C(15)-C(17)	1.487(12)
C(17)-C(18)	1.379(12)
C(18)-C(19)	1.439(11)
C(18)-C(21)	1.480(12)
C(19)-C(33)	1.410(11)

C(19)-C(20)	1.435(11)
C(20)-C(33)#1	1.397(11)
C(21)-C(22)	1.370(12)
C(21)-C(26)	1.403(12)
C(22)-C(23)	1.377(13)
C(22)-H(22)	0.9300
C(23)-C(24)	1.367(14)
C(23)-H(23)	0.9300
C(24)-C(25)	1.345(14)
C(24)-H(24)	0.9300
C(25)-C(26)	1.413(12)
C(25)-H(25)	0.9300
C(26)-H(26)	0.9300
C(27)-C(32)	1.373(13)
C(27)-C(28)	1.396(12)
C(28)-C(29)	1.389(14)
C(28)-H(28)	0.9300
C(29)-C(30)	1.369(18)
C(29)-H(29)	0.9300
C(30)-C(31)	1.364(19)
C(30)-H(30)	0.9300
C(31)-C(32)	1.373(15)
C(31)-H(31)	0.9300
C(32)-H(32)	0.9300
C(33)-C(20)#1	1.397(11)
C(33)-H(33)	0.9300
O(1A)-C(2A)	1.462(10)
O(1A)-C(5A)	1.472(10)
O(1A)-H(5A4)	1.2091
C(2A)-C(3A)	1.451(9)
C(2A)-H(2A1)	0.9700
C(2A)-H(2A2)	0.9700
O(1B)-C(2B)	1.439(9)
O(1B)-C(5A)	1.470(9)
C(2B)-C(3A)	1.467(9)
C(2B)-H(2A3)	0.9700

C(2B)-H(2A4)	0.9700
C(3A)-C(4A)	1.43(3)
C(3A)-H(3A1)	0.9700
C(3A)-H(3A2)	0.9700
C(3A)-H(3A3)	0.9700
C(3A)-H(3A4)	0.9700
C(4A)-C(5A)	1.43(2)
C(4A)-H(4A1)	0.9700
C(4A)-H(4A2)	0.9700
C(5A)-H(5A1)	0.9700
C(5A)-H(5A2)	0.9700
C(5A)-H(5A3)	0.9700
C(5A)-H(5A4)	0.9700

C(2)-C(1)-C(20)	120.2(9)
C(2)-C(1)-C(27)	122.6(9)
C(20)-C(1)-C(27)	117.2(8)
C(1)-C(2)-C(17)	118.9(8)
C(1)-C(2)-C(3)	123.7(9)
C(17)-C(2)-C(3)	117.3(8)
C(4)-C(3)-C(8)	117.6(9)
C(4)-C(3)-C(2)	122.6(9)
C(8)-C(3)-C(2)	119.5(9)
C(5)-C(4)-C(3)	120.7(10)
C(5)-C(4)-H(4)	119.7
C(3)-C(4)-H(4)	119.7
C(6)-C(5)-C(4)	121.8(11)
C(6)-C(5)-H(5)	119.1
C(4)-C(5)-H(5)	119.1
C(5)-C(6)-C(7)	120.5(10)
C(5)-C(6)-H(6)	119.8
C(7)-C(6)-H(6)	119.8
C(6)-C(7)-C(8)	119.7(10)
C(6)-C(7)-C(9)	123.0(11)
C(8)-C(7)-C(9)	117.3(11)
C(3)-C(8)-C(16)	121.2(9)

C(3)-C(8)-C(7)	119.3(10)
C(16)-C(8)-C(7)	119.5(10)
C(10)-C(9)-C(7)	122.3(11)
C(10)-C(9)-H(9)	118.9
C(7)-C(9)-H(9)	118.9
C(9)-C(10)-C(11)	121.6(11)
C(9)-C(10)-H(10)	119.2
C(11)-C(10)-H(10)	119.2
C(12)-C(11)-C(10)	122.1(11)
C(12)-C(11)-C(16)	119.7(10)
C(10)-C(11)-C(16)	118.2(12)
C(13)-C(12)-C(11)	121.6(10)
C(13)-C(12)-H(12)	119.2
C(11)-C(12)-H(12)	119.2
C(12)-C(13)-C(14)	119.6(11)
C(12)-C(13)-H(13)	120.2
C(14)-C(13)-H(13)	120.2
C(13)-C(14)-C(15)	121.6(10)
C(13)-C(14)-H(14)	119.2
C(15)-C(14)-H(14)	119.2
C(14)-C(15)-C(16)	118.0(9)
C(14)-C(15)-C(17)	121.6(9)
C(16)-C(15)-C(17)	120.0(9)
C(15)-C(16)-C(8)	120.0(9)
C(15)-C(16)-C(11)	119.1(10)
C(8)-C(16)-C(11)	120.8(10)
C(18)-C(17)-C(2)	120.4(8)
C(18)-C(17)-C(15)	122.3(9)
C(2)-C(17)-C(15)	117.2(8)
C(17)-C(18)-C(19)	118.6(8)
C(17)-C(18)-C(21)	122.6(8)
C(19)-C(18)-C(21)	118.5(8)
C(33)-C(19)-C(20)	118.9(8)
C(33)-C(19)-C(18)	121.4(9)
C(20)-C(19)-C(18)	119.5(8)
C(33)#1-C(20)-C(19)	119.0(8)

C(33)#1-C(20)-C(1)	122.4(8)
C(19)-C(20)-C(1)	118.4(8)
C(22)-C(21)-C(26)	117.9(9)
C(22)-C(21)-C(18)	119.2(9)
C(26)-C(21)-C(18)	122.8(9)
C(21)-C(22)-C(23)	121.6(10)
C(21)-C(22)-H(22)	119.2
C(23)-C(22)-H(22)	119.2
C(24)-C(23)-C(22)	121.2(12)
C(24)-C(23)-H(23)	119.4
C(22)-C(23)-H(23)	119.4
C(25)-C(24)-C(23)	118.5(12)
C(25)-C(24)-H(24)	120.8
C(23)-C(24)-H(24)	120.7
C(24)-C(25)-C(26)	122.1(11)
C(24)-C(25)-H(25)	119.0
C(26)-C(25)-H(25)	119.0
C(21)-C(26)-C(25)	118.7(10)
C(21)-C(26)-H(26)	120.7
C(25)-C(26)-H(26)	120.7
C(32)-C(27)-C(28)	116.9(10)
C(32)-C(27)-C(1)	121.1(10)
C(28)-C(27)-C(1)	121.9(10)
C(29)-C(28)-C(27)	120.6(11)
C(29)-C(28)-H(28)	119.7
C(27)-C(28)-H(28)	119.7
C(30)-C(29)-C(28)	119.5(13)
C(30)-C(29)-H(29)	120.2
C(28)-C(29)-H(29)	120.2
C(31)-C(30)-C(29)	121.2(14)
C(31)-C(30)-H(30)	119.4
C(29)-C(30)-H(30)	119.4
C(30)-C(31)-C(32)	118.3(14)
C(30)-C(31)-H(31)	120.8
C(32)-C(31)-H(31)	120.8
C(27)-C(32)-C(31)	123.3(13)

C(27)-C(32)-H(32)	118.4
C(31)-C(32)-H(32)	118.4
C(20)#1-C(33)-C(19)	121.9(9)
C(20)#1-C(33)-H(33)	119.0
C(19)-C(33)-H(33)	119.0
C(2A)-O(1A)-C(5A)	102.5(8)
C(2A)-O(1A)-H(5A4)	129.3
C(3A)-C(2A)-O(1A)	105.2(7)
C(3A)-C(2A)-H(2A1)	110.4
O(1A)-C(2A)-H(2A1)	110.5
C(3A)-C(2A)-H(2A2)	110.9
O(1A)-C(2A)-H(2A2)	111.0
H(2A1)-C(2A)-H(2A2)	108.8
C(2B)-O(1B)-C(5A)	107.1(6)
O(1B)-C(2B)-C(3A)	106.6(6)
O(1B)-C(2B)-H(2A3)	111.2
C(3A)-C(2B)-H(2A3)	111.0
O(1B)-C(2B)-H(2A4)	109.6
C(3A)-C(2B)-H(2A4)	109.8
H(2A3)-C(2B)-H(2A4)	108.6
C(4A)-C(3A)-C(2A)	108.0(5)
C(4A)-C(3A)-C(2B)	104.3(7)
C(4A)-C(3A)-H(3A1)	110.1
C(2A)-C(3A)-H(3A1)	110.4
C(2B)-C(3A)-H(3A1)	141.6
C(4A)-C(3A)-H(3A2)	110.1
C(2A)-C(3A)-H(3A2)	109.9
H(3A1)-C(3A)-H(3A2)	108.4
C(4A)-C(3A)-H(3A3)	109.0
C(2A)-C(3A)-H(3A3)	93.0
C(2B)-C(3A)-H(3A3)	129.7
H(3A2)-C(3A)-H(3A3)	124.8
C(4A)-C(3A)-H(3A4)	108.1
C(2A)-C(3A)-H(3A4)	128.2
C(2B)-C(3A)-H(3A4)	95.1
H(3A3)-C(3A)-H(3A4)	108.6

C(3A)-C(4A)-C(5A)	106.4(14)
C(3A)-C(4A)-H(4A1)	110.5
C(5A)-C(4A)-H(4A1)	110.3
C(3A)-C(4A)-H(4A2)	110.5
C(5A)-C(4A)-H(4A2)	110.5
H(4A1)-C(4A)-H(4A2)	108.6
C(4A)-C(5A)-O(1A)	104.8(7)
C(4A)-C(5A)-O(1B)	104.1(7)
C(4A)-C(5A)-H(5A1)	110.9
O(1A)-C(5A)-H(5A1)	110.9
C(4A)-C(5A)-H(5A2)	110.8
O(1A)-C(5A)-H(5A2)	110.6
H(5A1)-C(5A)-H(5A2)	108.8
C(4A)-C(5A)-H(5A3)	116.0
O(1B)-C(5A)-H(5A3)	109.5
C(4A)-C(5A)-H(5A4)	107.2
O(1B)-C(5A)-H(5A4)	110.6
H(5A3)-C(5A)-H(5A4)	109.2

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1

Table S7. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for wd500ms. 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)	56(6)	39(6)	50(6)	1(5)	17(5)	-1(5)
C(2)	53(6)	41(6)	53(6)	-12(5)	17(5)	-5(5)
C(3)	47(6)	52(7)	62(7)	-8(6)	20(5)	0(5)
C(4)	62(7)	53(7)	73(8)	-6(6)	24(6)	-4(6)
C(5)	80(8)	47(7)	82(9)	-17(6)	27(7)	-6(6)
C(6)	87(9)	40(7)	105(11)	-16(8)	41(8)	-12(6)
C(7)	60(7)	59(8)	78(8)	-27(7)	30(7)	-18(6)
C(8)	47(6)	49(7)	66(7)	-13(6)	23(6)	-8(5)
C(9)	79(9)	92(11)	96(10)	-49(9)	37(8)	-38(8)
C(10)	68(8)	91(10)	64(8)	-27(8)	15(7)	-29(7)
C(11)	47(7)	82(9)	66(8)	-31(7)	16(6)	-23(6)
C(12)	57(7)	93(10)	55(7)	-9(8)	8(6)	-13(7)
C(13)	70(8)	87(9)	57(8)	-4(7)	16(6)	-7(7)
C(14)	54(7)	76(8)	52(7)	-9(6)	14(6)	2(6)
C(15)	54(7)	67(8)	48(7)	-3(6)	19(5)	-6(6)
C(16)	41(6)	65(8)	61(7)	-13(6)	20(5)	-11(6)
C(17)	55(6)	40(6)	37(6)	-2(5)	11(5)	-1(5)
C(18)	53(6)	48(6)	38(6)	-8(5)	11(5)	-1(5)
C(19)	44(6)	43(6)	48(6)	2(5)	11(5)	-2(5)
C(20)	53(6)	33(5)	44(6)	0(5)	16(5)	-2(5)
C(21)	49(6)	46(6)	45(6)	-2(5)	16(5)	-6(5)
C(22)	76(8)	48(7)	68(7)	3(6)	30(6)	-4(6)
C(23)	97(9)	77(9)	76(8)	-2(7)	42(7)	-5(8)
C(24)	94(10)	86(10)	79(9)	16(8)	46(8)	4(8)
C(25)	96(9)	53(7)	62(7)	15(6)	25(7)	-7(7)
C(26)	68(7)	52(7)	56(6)	7(6)	22(6)	2(6)
C(27)	68(7)	40(6)	64(7)	1(5)	28(6)	5(6)
C(28)	75(8)	43(6)	58(7)	0(6)	16(6)	0(6)
C(29)	133(13)	53(8)	81(9)	18(7)	35(9)	21(8)
C(30)	210(20)	61(10)	103(13)	21(9)	89(14)	2(12)
C(31)	154(15)	72(10)	134(13)	6(10)	77(13)	-43(10)

C(32)	75(8)	57(7)	92(9)	0(7)	40(7)	-10(6)
C(33)	57(6)	38(6)	55(7)	-2(5)	20(5)	-6(5)

Table S8. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for wd500ms.

	x	y	z	U(eq)
H(4)	5465	3560	5674	75
H(5)	5791	4992	6277	84
H(6)	6756	5277	7917	90
H(9)	7672	4731	9683	105
H(10)	8267	3593	10780	92
H(12)	8478	1984	11119	86
H(13)	8147	542	10592	88
H(14)	7102	231	8928	74
H(22)	4297	825	8034	76
H(23)	3739	-29	9093	96
H(24)	4267	-1494	9398	99
H(25)	5466	-2060	8733	85
H(26)	6126	-1198	7703	70
H(28)	4348	2367	3932	73
H(29)	4527	3386	2774	107
H(30)	6214	3933	2953	137
H(31)	7717	3550	4321	136
H(32)	7531	2536	5457	86
H(33)	4556	-927	6111	60
H(2A1)	7738	5932	1049	50
H(2A2)	7009	5345	1481	50
H(2A3)	6049	4924	1001	50
H(2A4)	7241	5304	1320	50
H(3A1)	6382	6277	-270	50
H(3A2)	5762	5491	27	50
H(3A3)	6573	6356	-73	50
H(3A4)	5697	5604	-204	50
H(4A1)	4691	6444	239	50
H(4A2)	5419	7233	104	50
H(5A1)	5503	6411	1858	50

H(5A2)	5702	7426	1686	50
H(5A3)	5308	6993	1822	50
H(5A4)	6397	7275	1667	50

Table S9. Torsion angles [°] for wd500ms.

C(20)-C(1)-C(2)-C(17)	8.9(13)
C(27)-C(1)-C(2)-C(17)	-170.7(9)
C(20)-C(1)-C(2)-C(3)	-167.8(8)
C(27)-C(1)-C(2)-C(3)	12.7(14)
C(1)-C(2)-C(3)-C(4)	20.4(14)
C(17)-C(2)-C(3)-C(4)	-156.3(9)
C(1)-C(2)-C(3)-C(8)	-165.4(9)
C(17)-C(2)-C(3)-C(8)	17.9(13)
C(8)-C(3)-C(4)-C(5)	6.5(14)
C(2)-C(3)-C(4)-C(5)	-179.2(9)
C(3)-C(4)-C(5)-C(6)	-0.9(16)
C(4)-C(5)-C(6)-C(7)	-3.1(17)
C(5)-C(6)-C(7)-C(8)	1.1(16)
C(5)-C(6)-C(7)-C(9)	-178.2(10)
C(4)-C(3)-C(8)-C(16)	172.7(8)
C(2)-C(3)-C(8)-C(16)	-1.8(13)
C(4)-C(3)-C(8)-C(7)	-8.3(13)
C(2)-C(3)-C(8)-C(7)	177.2(8)
C(6)-C(7)-C(8)-C(3)	4.7(14)
C(9)-C(7)-C(8)-C(3)	-176.0(9)
C(6)-C(7)-C(8)-C(16)	-176.3(9)
C(9)-C(7)-C(8)-C(16)	3.0(13)
C(6)-C(7)-C(9)-C(10)	-179.8(11)
C(8)-C(7)-C(9)-C(10)	0.9(16)
C(7)-C(9)-C(10)-C(11)	-2.0(18)
C(9)-C(10)-C(11)-C(12)	176.7(11)
C(9)-C(10)-C(11)-C(16)	-1.0(16)
C(10)-C(11)-C(12)-C(13)	-179.5(10)
C(16)-C(11)-C(12)-C(13)	-1.8(16)
C(11)-C(12)-C(13)-C(14)	-0.9(17)
C(12)-C(13)-C(14)-C(15)	-0.6(16)
C(13)-C(14)-C(15)-C(16)	4.7(14)
C(13)-C(14)-C(15)-C(17)	178.8(9)
C(14)-C(15)-C(16)-C(8)	171.5(8)

C(17)-C(15)-C(16)-C(8)	-2.7(13)
C(14)-C(15)-C(16)-C(11)	-7.3(13)
C(17)-C(15)-C(16)-C(11)	178.5(8)
C(3)-C(8)-C(16)-C(15)	-5.8(14)
C(7)-C(8)-C(16)-C(15)	175.2(9)
C(3)-C(8)-C(16)-C(11)	173.0(8)
C(7)-C(8)-C(16)-C(11)	-6.0(13)
C(12)-C(11)-C(16)-C(15)	6.0(14)
C(10)-C(11)-C(16)-C(15)	-176.2(10)
C(12)-C(11)-C(16)-C(8)	-172.8(9)
C(10)-C(11)-C(16)-C(8)	5.0(14)
C(1)-C(2)-C(17)-C(18)	-21.4(13)
C(3)-C(2)-C(17)-C(18)	155.5(8)
C(1)-C(2)-C(17)-C(15)	157.4(8)
C(3)-C(2)-C(17)-C(15)	-25.7(12)
C(14)-C(15)-C(17)-C(18)	23.6(14)
C(16)-C(15)-C(17)-C(18)	-162.4(9)
C(14)-C(15)-C(17)-C(2)	-155.2(9)
C(16)-C(15)-C(17)-C(2)	18.7(13)
C(2)-C(17)-C(18)-C(19)	16.2(13)
C(15)-C(17)-C(18)-C(19)	-162.7(8)
C(2)-C(17)-C(18)-C(21)	-157.4(9)
C(15)-C(17)-C(18)-C(21)	23.8(14)
C(17)-C(18)-C(19)-C(33)	176.7(8)
C(21)-C(18)-C(19)-C(33)	-9.4(13)
C(17)-C(18)-C(19)-C(20)	1.0(12)
C(21)-C(18)-C(19)-C(20)	174.9(8)
C(33)-C(19)-C(20)-C(33)#1	-4.2(14)
C(18)-C(19)-C(20)-C(33)#1	171.7(8)
C(33)-C(19)-C(20)-C(1)	171.2(8)
C(18)-C(19)-C(20)-C(1)	-13.0(12)
C(2)-C(1)-C(20)-C(33)#1	-177.0(9)
C(27)-C(1)-C(20)-C(33)#1	2.6(13)
C(2)-C(1)-C(20)-C(19)	7.8(13)
C(27)-C(1)-C(20)-C(19)	-172.6(8)
C(17)-C(18)-C(21)-C(22)	61.8(13)

C(19)-C(18)-C(21)-C(22)	-111.8(10)
C(17)-C(18)-C(21)-C(26)	-121.4(10)
C(19)-C(18)-C(21)-C(26)	65.0(12)
C(26)-C(21)-C(22)-C(23)	1.2(15)
C(18)-C(21)-C(22)-C(23)	178.1(9)
C(21)-C(22)-C(23)-C(24)	-2.7(18)
C(22)-C(23)-C(24)-C(25)	2.4(19)
C(23)-C(24)-C(25)-C(26)	-0.9(18)
C(22)-C(21)-C(26)-C(25)	0.3(14)
C(18)-C(21)-C(26)-C(25)	-176.5(8)
C(24)-C(25)-C(26)-C(21)	-0.4(16)
C(2)-C(1)-C(27)-C(32)	63.6(13)
C(20)-C(1)-C(27)-C(32)	-116.0(10)
C(2)-C(1)-C(27)-C(28)	-118.7(11)
C(20)-C(1)-C(27)-C(28)	61.7(12)
C(32)-C(27)-C(28)-C(29)	-3.4(14)
C(1)-C(27)-C(28)-C(29)	178.8(9)
C(27)-C(28)-C(29)-C(30)	2.4(17)
C(28)-C(29)-C(30)-C(31)	-1(2)
C(29)-C(30)-C(31)-C(32)	2(2)
C(28)-C(27)-C(32)-C(31)	3.7(16)
C(1)-C(27)-C(32)-C(31)	-178.5(10)
C(30)-C(31)-C(32)-C(27)	-3(2)
C(20)-C(19)-C(33)-C(20)#1	4.3(14)
C(18)-C(19)-C(33)-C(20)#1	-171.4(8)
C(5A)-O(1A)-C(2A)-C(3A)	31(3)
C(5A)-O(1B)-C(2B)-C(3A)	2(3)
O(1A)-C(2A)-C(3A)-C(4A)	-15(3)
O(1A)-C(2A)-C(3A)-C(2B)	-106(2)
O(1B)-C(2B)-C(3A)-C(4A)	-22(3)
O(1B)-C(2B)-C(3A)-C(2A)	79(3)
C(2A)-C(3A)-C(4A)-C(5A)	-8(2)
C(2B)-C(3A)-C(4A)-C(5A)	33.8(19)
C(3A)-C(4A)-C(5A)-O(1A)	27.8(18)
C(3A)-C(4A)-C(5A)-O(1B)	-32.3(16)
C(2A)-O(1A)-C(5A)-C(4A)	-36(2)

C(2A)-O(1A)-C(5A)-O(1B)	61.2(18)
C(2B)-O(1B)-C(5A)-C(4A)	18(3)
C(2B)-O(1B)-C(5A)-O(1A)	-81(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1

[1] All calculations used the GAUSSIAN 98 series of programs. Gaussian 98, Revision A.11.4, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Zakrzewski, V. G.; Montgomery, Jr., J. A.; Stratmann, R. E.; Burant, J. C.; Dapprich, S.; Millam, J. M.; Daniels, A. D.; Kudin, K. N.; Strain, M. C.; Farkas, O.; Tomasi, J.; Barone, V.; Cossi, M.; Cammi, R.; Mennucci, B.; Pomelli, C.; Adamo, C.; Clifford, S.; Ochterski, J.; Petersson, G. A.; Ayala, P. Y.; Cui, Q.; Morokuma, K.; Rega, N.; Salvador, P.; Dannenberg, J. J.; Malick, D. K.; Rabuck, A. D.; Raghavachari, K.; Foresman, J. B.; Cioslowski, J.; Ortiz, J. V.; Baboul, A. G.; Stefanov, B. B.; Liu, G. Liashenko, A. Piskorz, P.; Komaromi, I.; Gomperts, R.; Martin, R. L.; Fox, D. J.; Keith, T.; Al-Laham, M. A.; Peng, C. Y.; Nanayakkara, A.; Challacombe, M.; Gill, P. M. W.; Johnson, B.; Chen, W.; Wong, M. W.; Andres, J. L.; Gonzalez, C.; Head-Gordon, M.; Replogle, E. S.; and Pople, J. A. Gaussian, Inc., Pittsburgh PA, 2002.

[2] (a) Parr, R. G.; Yang, W. *Density-functional theory of atoms and molecules*, Oxford University Press: New York, 1989. (b) Koch, W; Holthausen M. C. *A Chemist's guide to density functional theory*, Wiley-VCH: 2000.

[3] (a) Lee, C.; Yang, W.; Parr, R. G. *Phys. Rev. B* **1988**, *37*, 785. (b) Becke, A. D. *J. Chem. Phys.* **1993**, *98*, 5648.

[4] Bock, H.; Nick, S.; Ruppert, K. *Z. Naturforsch* **1995**, *50*, 595.

[5] Pascal, Jr. R. A.; McMillan, W. D.; Engen, V. D.; Eason, R. G. *J. Am. Chem. Soc.* **1987**, *109*, 4660-4665.