BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Direct borylation of terrylene and quaterrylene

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^{1}H and ^{13}C NMR as well as MS spectra for newly synthesized compounds

1. NMR Spectra

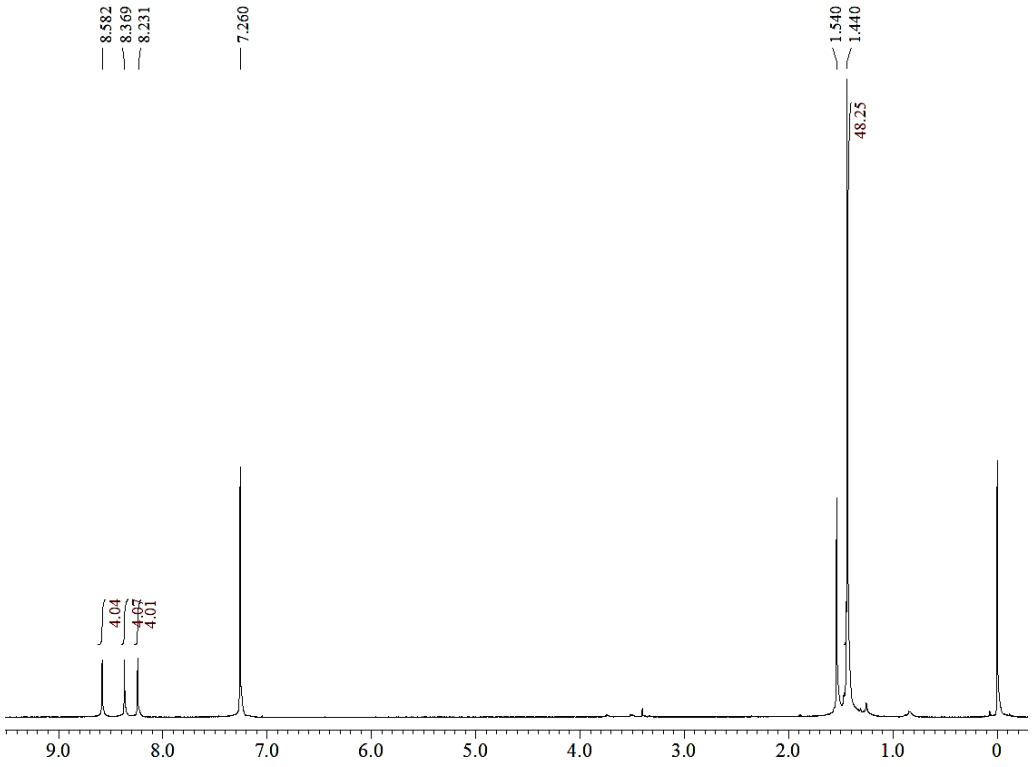

Figure S1. ^{1}H NMR spectrum of **TB4** in $CDCl_3$ at room temperature.

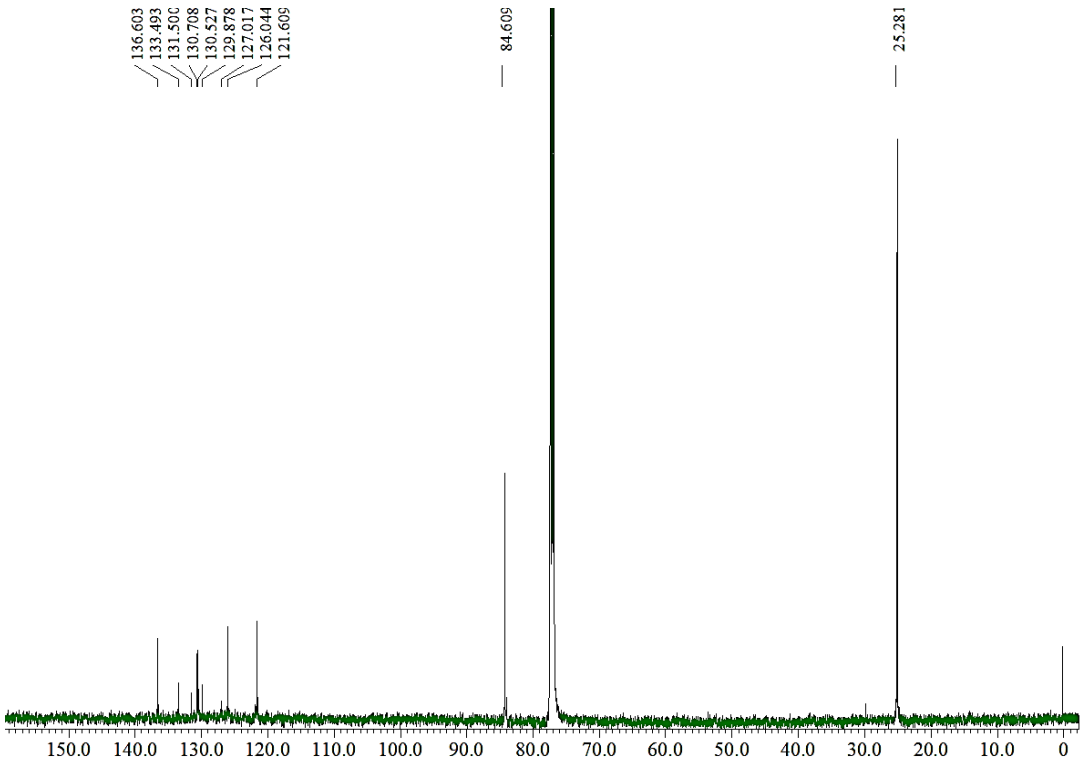

Figure S2. ^{13}C NMR spectrum of **TB4** in $CDCl_3$ at room temperature.

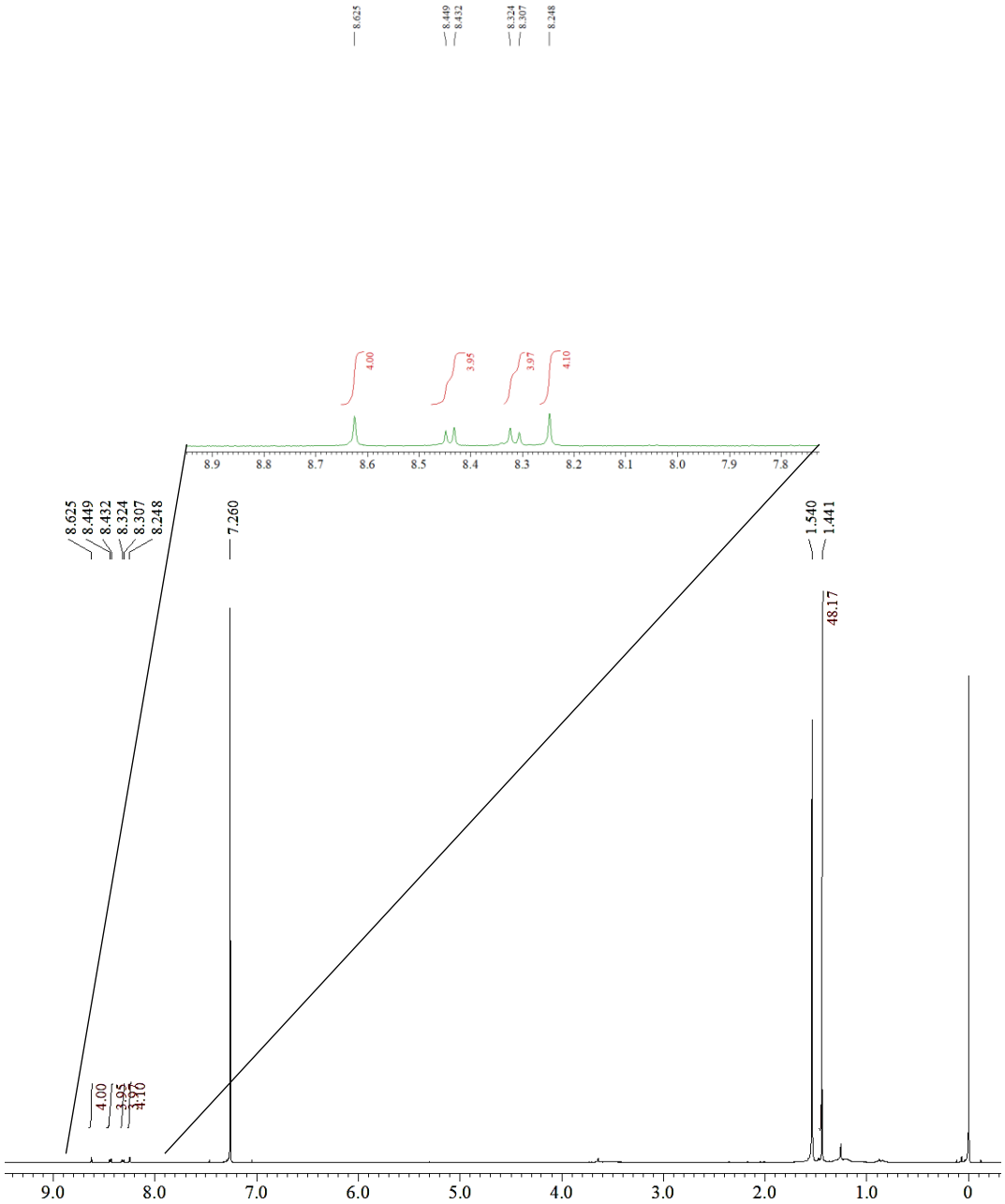

Figure S3. ^{1}H NMR spectrum of **QB4** in $CDCl_3$ at room temperature.

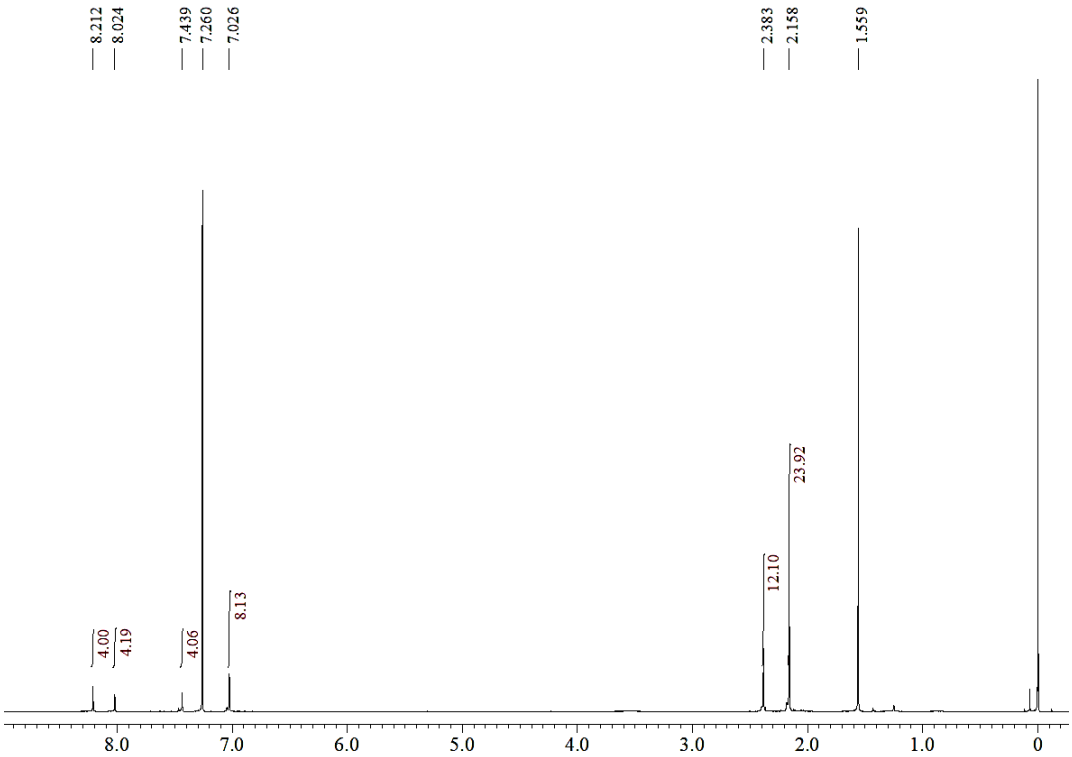

Figure S4. ^{1}H NMR spectrum of **TM4** in $CDCl_3$ at room temperature.

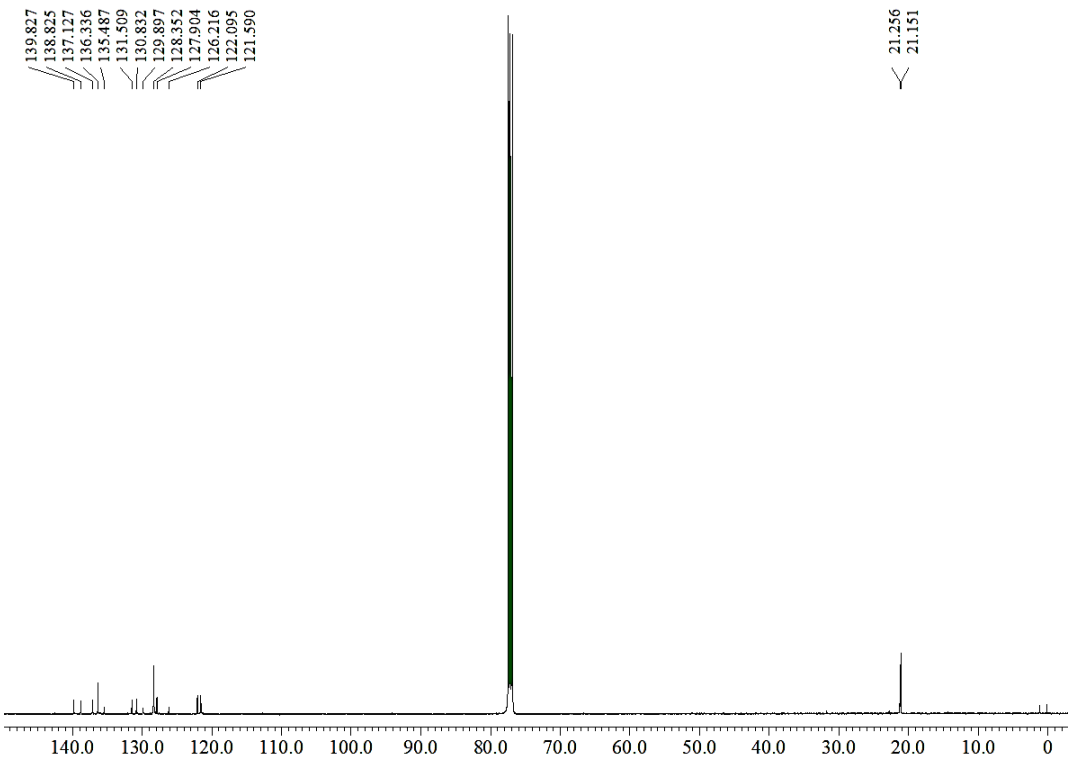

Figure S5. ^{13}C NMR spectrum of **TM4** in $CDCl_3$ at room temperature.

2. HR-MS

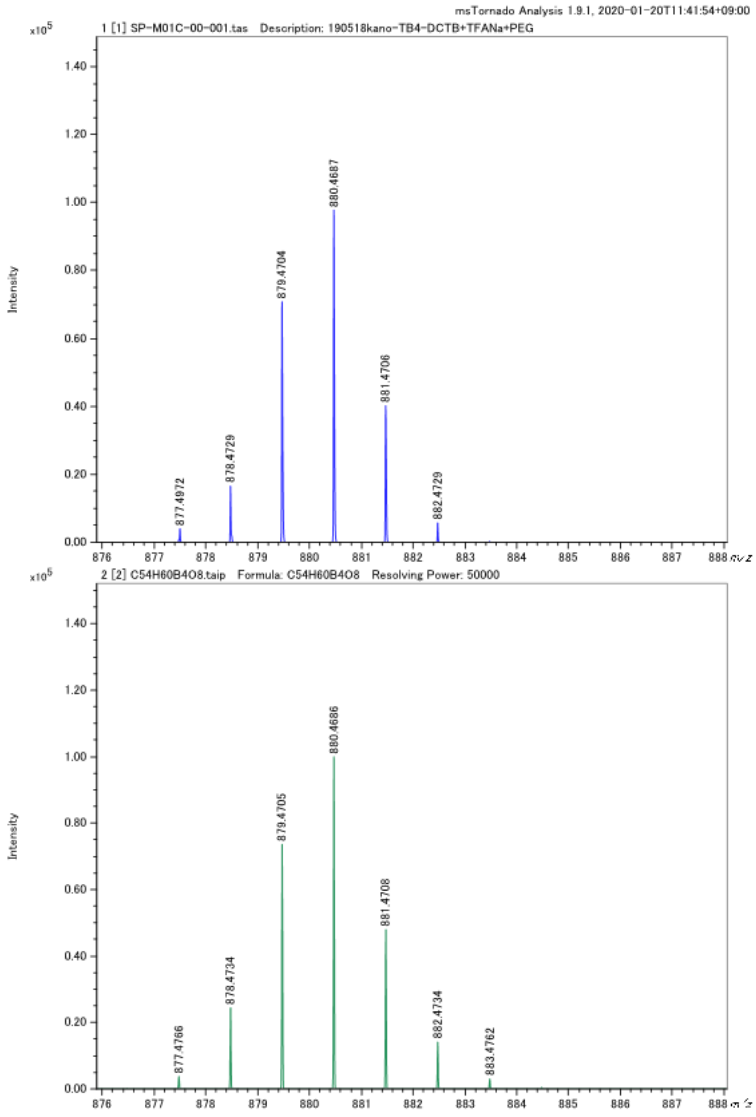

Figure S6. HR-Spiral-MALDI-TOF mass spectrum of **TB4**.

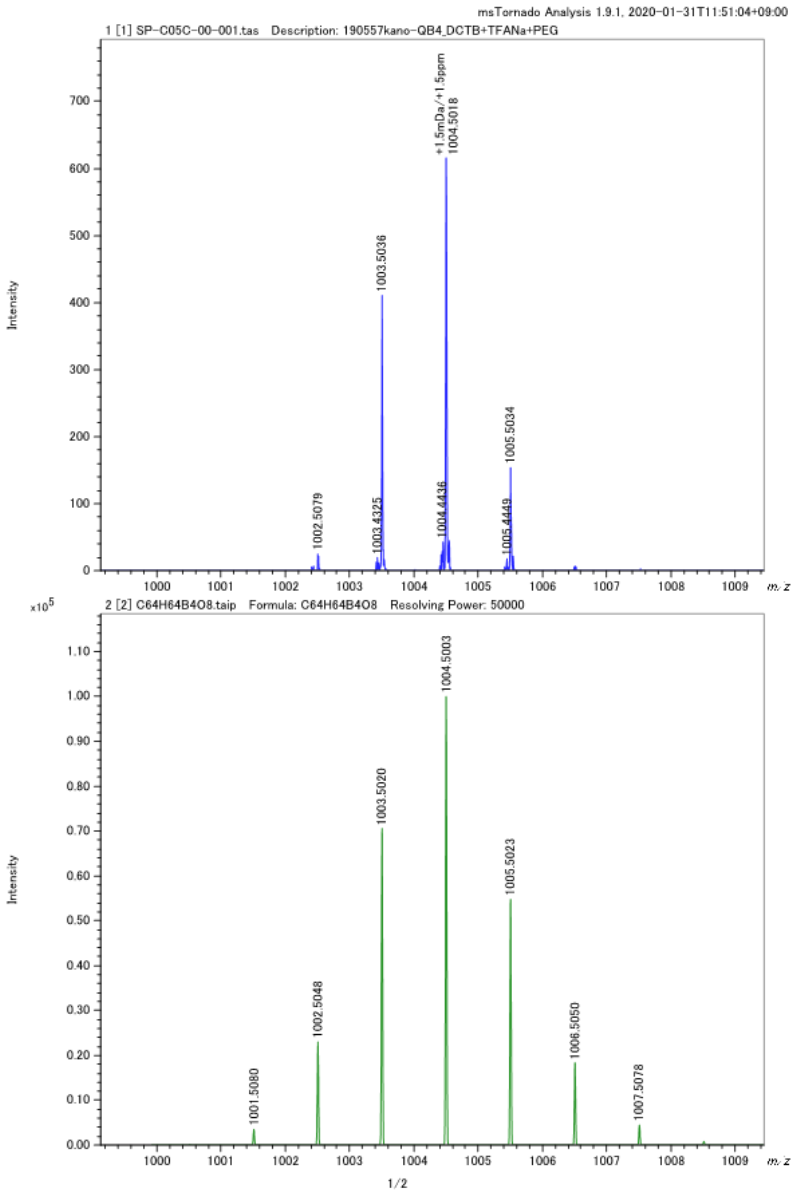

Figure S7. HR-Spiral-MALDI-TOF mass spectrum of **QB4**.

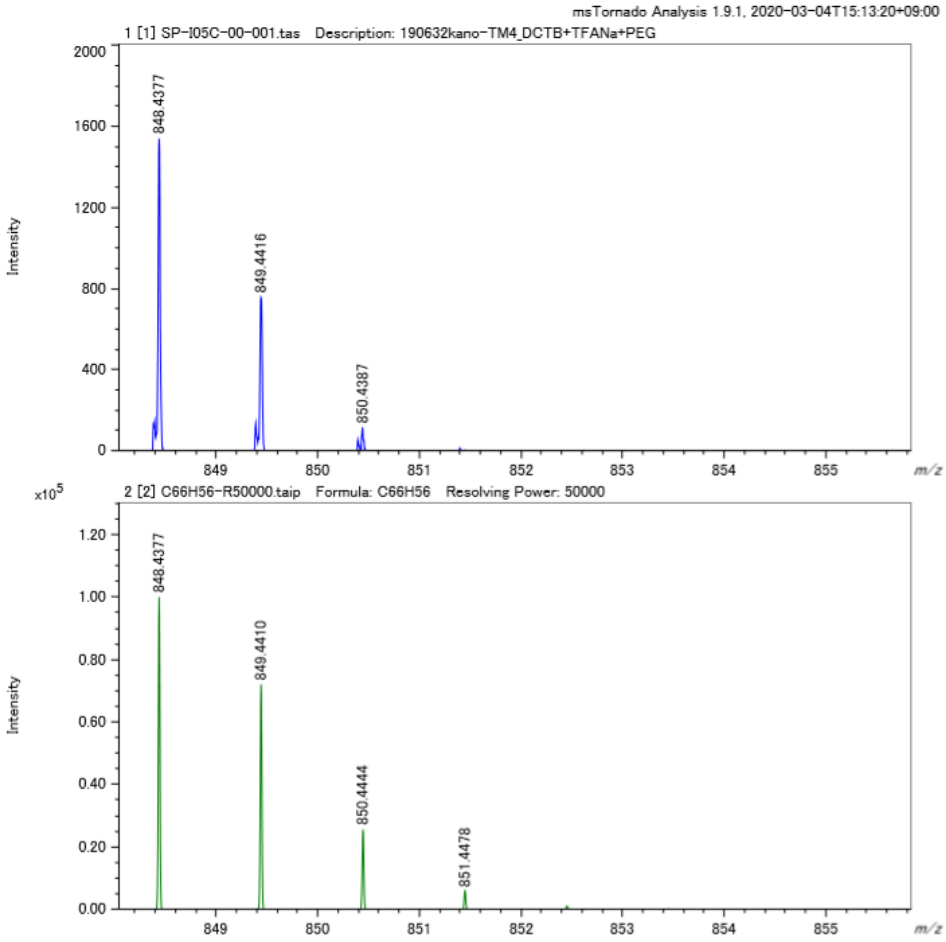

Figure S8. HR-Spiral-MALDI-TOF mass spectrum of **TM4**.

3. X-ray Crystal Structure

Table S1. Crystal data and structure refinement for **TB4**.

Empirical formula	$C_{56}H_{64}B_4Cl_4O_8$
Formula weight	1050.11
Temperature	103(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 6.691(5) Å
	b = 29.13(2) Å β = 97.183(17)°
	c = 14.044(12) Å
Volume	2716(4) Å^3
Z	2
Density (calculated)	1.284 g/cm^3
Absorption coefficient	0.271 mm^{-1}
F(000)	1104
Crystal size	0.150 x 0.020 x 0.010 mm^3
Theta range for data collection	1.620 to 23.619°
Index ranges	$-7 \le h \le 7$, $-30 \le k \le 31$, $-15 \le l \le 5$
Reflections collected	6825
Independent reflections	3943 [R(int) = 0.1642]
Completeness to theta = 23.619°	96.5%
Max. and min. transmission	0.997 and 0.817
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3943 / 18 / 333
Goodness-of-fit on F^2	0.925
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0847, wR_2 = 0.1476
R indices (all data)	R_1 = 0.2590, wR_2 = 0.2254
Largest diff. peak and hole	0.435 and –0.323 e.Å^{-3}

4. Cartesian Coordinates

The cartesian coordinates (in Å) optimized at the B3LYP/6-31G(d) level are shown.

Terrylene

C	2.48025	-4.64431	-0.01703
C	1.25785	-3.92981	-0.01863
C	1.20705	-2.54161	-0.02883
C	2.44825	-1.82651	-0.02873
C	3.66675	-2.54041	-0.02613
C	3.67265	-3.96481	-0.02193
C	2.46425	-0.39421	-0.02813
C	3.70015	0.24039	-0.02813
C	4.92115	-0.47611	-0.02803
C	4.91165	-1.84851	-0.02643
C	-0.05835	-1.78991	-0.02773
C	-0.06265	-0.35691	-0.01563
C	1.18895	0.34099	-0.02433
C	-1.28685	-2.43111	-0.05703
C	-2.50265	-1.71941	-0.02223
C	-2.53945	-0.33601	0.04097
C	-1.28845	0.36039	-0.00193
C	-1.29715	1.79279	-0.03783
C	-0.07075	2.43739	-0.03243
C	1.14625	1.72619	-0.02413
C	-3.81155	0.39869	0.11957
C	-3.80195	1.82739	0.02567
C	-2.56445	2.54089	-0.07463
C	-5.03495	-0.23401	0.29887
C	-6.25725	0.48029	0.32997
C	-6.25915	1.84589	0.18827
C	-5.02225	2.53739	0.04107
C	-5.03405	3.95609	-0.08633
C	-3.84655	4.63259	-0.21433
C	-2.62135	3.92259	-0.20473
H	2.47421	-5.74428	-0.01186
H	0.31393	-4.49458	-0.01153
H	4.62487	-4.51552	-0.02261
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H	5.8776	0.06721	-0.02923
H	5.85782	-2.40954	-0.02539

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H	-0.04602	3.53711	-0.03468
H	2.09175	2.28832	-0.01728
H	-5.05503	-1.32704	0.42085
H	-7.20585	-0.05932	0.46775
H	-7.20833	2.40181	0.18839
H	-5.98721	4.50516	-0.08217
H	-3.84665	5.72707	-0.32444
H	-1.68136	4.48526	-0.30382

TB4

C	8.54731	0.2419	-0.06966
C	9.76831	-0.4746	-0.06956
C	9.75881	-1.847	-0.06796
C	8.51391	-2.5389	-0.06766
C	7.29541	-1.825	-0.07026
C	7.31141	-0.3927	-0.06966
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C	6.10501	-3.9283	-0.06016
C	6.05421	-2.5401	-0.07036
C	6.03611	0.3425	-0.06586
C	4.78881	-1.7884	-0.06926
C	4.78451	-0.3554	-0.05716
C	3.55871	0.3619	-0.04346
C	3.55001	1.7943	-0.07936
C	4.77641	2.4389	-0.07396
C	5.99341	1.7277	-0.06566
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C	2.34451	-1.7179	-0.06376
C	2.30771	-0.3345	-0.00056
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C	1.00061	4.6341	-0.25586
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C	-0.18779	-0.2325	0.25734

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C	-5.49333	-1.03036	-0.96663
C	0.19375	8.37538	-0.64439
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O	7.76103	-7.09745	1.13602
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C	7.3247	-8.40907	0.75106
C	6.38102	-9.4789	-1.37941
C	8.70532	-8.71126	-1.41314
C	8.28484	-9.46559	1.28155
C	5.94831	-8.7347	1.3159
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H	10.70499	-2.40803	-0.06692

H	9.47203	-4.51401	-0.06414
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H	1.39745	-2.27692	-0.08746
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H	3.03256	7.70663	-2.22311
H	1.5929	8.69939	-2.7203
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H	13.99766	3.01096	-1.17589
H	12.35905	3.78154	-1.01301
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H	13.56843	1.40525	2.09385

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H	13.20571	0.05684	-2.38067
H	13.37693	1.8567	-2.18941
H	6.94739	-10.18356	-2.0286
H	5.93587	-10.03776	-0.52602
H	5.57091	-8.99818	-1.97221
H	8.65316	-9.62021	-2.05335
H	9.05344	-7.8479	-2.02321
H	9.4183	-8.88718	-0.57678
H	7.72973	-10.18028	1.92951
H	8.73895	-10.01587	0.4273
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H	5.23847	-8.92043	0.479

Quaterrylene

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C	2.56445	-1.23089	-0.00931
C	5.44585	1.23271	-0.03051
C	-1.05125	2.40531	0.00549
C	3.29925	2.40271	-0.02791
C	2.56435	1.22691	-0.01811
C	6.91865	-1.24389	-0.02951
C	7.62825	0.00031	-0.03171
C	6.91745	1.24381	-0.03151
C	7.66425	-2.41609	-0.03081
C	9.08015	-2.41399	-0.03281
C	9.76545	-1.22469	-0.03361
C	9.04055	0.00151	-0.03311
C	9.76385	1.22881	-0.03291
C	9.07705	2.41721	-0.03091
C	7.66125	2.41711	-0.03011

C	1.09305	-1.23089	0.00269
C	1.09315	1.22701	-0.00571
C	0.35985	-0.00179	0.00839
C	-1.06075	-0.00109	0.02269
C	-1.78985	-1.23389	0.03699
C	-1.05265	-2.40739	0.02369
C	0.35635	-2.40589	0.00549
C	0.35775	2.40271	-0.00791
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C	-3.26145	-1.24339	0.05569
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H	7.13554	-3.3807	-0.03027
H	9.63126	-3.36598	-0.03373
H	10.86543	-1.21856	-0.03463
H	10.86384	1.22412	-0.03435
H	9.62681	3.36997	-0.02993
H	7.13105	3.3809	-0.02832
H	-1.58424	-3.37041	0.02746
H	0.88932	-3.36807	-0.00691
H	0.89164	3.36438	-0.02053
H	5.23911	3.36919	-0.04311
H	-7.20833	-1.21902	0.07081
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H	-3.47785	-3.37973	0.10132
H	-3.47412	3.3804	-0.01565
H	-5.96955	3.3693	-0.02159
H	-7.20694	1.22395	0.02141

QB4

C	6.01306	-2.41144	-0.01976
C	7.42226	-2.41354	-0.02796
C	8.15956	-1.24014	-0.03586
C	7.43106	-0.00674	-0.03626
C	6.01046	-0.00724	-0.02956
C	5.27706	-1.23624	-0.02076
C	8.15846	1.22736	-0.04196
C	1.66136	2.39996	-0.00596
C	6.01186	2.39736	-0.03936
C	5.27696	1.22156	-0.02956
C	9.63126	-1.24924	-0.04096
C	10.34086	-0.00504	-0.04316
C	9.63006	1.23846	-0.04296
C	10.37686	-2.42144	-0.04226
C	11.79276	-2.41934	-0.04426
C	12.47806	-1.23004	-0.04506
C	11.75316	-0.00384	-0.04456
C	12.47646	1.22346	-0.04436
C	11.78966	2.41186	-0.04236
C	10.37386	2.41176	-0.04156
C	3.80566	-1.23624	-0.00876
C	3.80576	1.22166	-0.01716
C	3.07246	-0.00714	-0.00306
C	1.65186	-0.00644	0.01125
C	0.92276	-1.23924	0.02555
C	1.65996	-2.41274	0.01225
C	3.06896	-2.41124	-0.00596
C	3.07036	2.39736	-0.01936
C	7.42086	2.40046	-0.04596
C	0.92376	1.22686	0.00725
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C	-0.54774	1.23836	0.01065
C	-1.25884	-0.00484	0.03095
C	-2.67124	-0.00374	0.03435
C	-3.39574	-1.23004	0.05775
C	-2.70984	-2.41874	0.07825
C	-1.29414	-2.42074	0.07205
C	-1.29164	2.41136	-0.01096

C	-2.70724	2.41136	-0.01296
C	-3.39434	1.22316	0.01015
B	12.58936	-3.7954	-0.04557
B	12.58432	3.78903	-0.04094
B	-3.5018	3.78829	-0.04197
B	-3.50596	-3.79474	0.10898
O	13.37124	-4.34385	-1.24476
O	12.67666	-4.74876	1.15278
O	13.41094	4.31469	-1.22015
O	12.62472	4.76643	1.14043
O	-5.03015	3.90785	-0.04655
O	-2.84001	5.17171	-0.06906
O	-2.84608	-5.17826	0.13345
O	-5.03521	-3.91254	0.11806
C	13.26032	-5.92651	0.5773
C	14.13022	-5.41187	-0.66089
C	14.33345	-6.53366	-1.67075
C	15.51454	-4.94941	-0.22578
C	14.14839	-6.63093	1.59448
C	12.19867	-6.92874	0.14375
C	13.65423	5.68774	0.7528
C	13.65858	5.67702	-0.8455
C	13.34153	7.08419	1.27404
C	15.00974	5.29272	1.32382
C	15.00971	6.13733	-1.37663
C	12.61029	6.62073	-1.41996
C	-3.94369	6.04411	-0.3515
C	-5.23541	5.30104	0.22643
C	-6.49316	5.80014	-0.4725
C	-5.42013	5.56412	1.71512
C	-3.74967	7.3874	0.33948
C	-4.07392	6.32533	-1.84263
C	-3.94426	-6.04256	0.45689
C	-5.24535	-5.31245	-0.11652
C	-3.76301	-7.40265	-0.20403
C	-4.04819	-6.28746	1.95648
C	-6.49091	-5.79303	0.6164
C	-5.45552	-5.61194	-1.59492
H	5.47957	-3.37338	-0.01232
H	7.95363	-3.37669	-0.02809

H	1.1303	3.36327	-0.00597
H	5.47755	3.35887	-0.04206
H	9.84815	-3.38605	-0.04171
H	13.57804	-1.22391	-0.04607
H	13.57645	1.21877	-0.04579
H	9.84366	3.37555	-0.03976
H	1.12837	-3.37576	0.01602
H	3.60193	-3.37342	-0.01835
H	3.60425	3.35903	-0.03197
H	7.95172	3.36384	-0.05455
H	-4.49572	-1.22437	0.05937
H	-0.76524	-3.38508	0.08988
H	-0.76151	3.37505	-0.02709
H	-4.49433	1.2186	0.00997
H	15.42276	-6.71	-1.81596
H	13.85476	-7.46433	-1.29197
H	13.87158	-6.24658	-2.64185
H	16.28955	-5.54735	-0.75551
H	15.64143	-3.87263	-0.47719
H	15.62426	-5.09023	0.87282
H	13.78724	-7.67297	1.74444
H	15.19614	-6.65232	1.21959
H	14.11082	-6.08254	2.56228
H	12.3559	-7.8916	0.67942
H	11.18943	-6.52878	0.38917
H	12.27479	-7.09818	-0.95364
H	14.17584	7.43426	1.92224
H	13.22176	7.78196	0.41525
H	12.39895	7.05687	1.86528
H	15.40051	6.1191	1.95878
H	14.89819	4.37281	1.94033
H	15.72078	5.09768	0.49006
H	14.86826	7.02605	-2.03157
H	15.67076	6.40767	-0.523
H	15.47726	5.31507	-1.9632
H	13.10736	7.38081	-2.06337
H	11.88213	6.03926	-2.02859
H	12.07556	7.13154	-0.58815
H	-7.19735	6.21626	0.2823
H	-6.22069	6.59475	-1.20264

H	-6.98035	4.95455	-1.00766
H	-6.40987	6.04305	1.88777
H	-5.37294	4.6006	2.27025
H	-4.61296	6.24063	2.07509
H	-3.75531	8.20043	-0.42061
H	-4.57669	7.55336	1.06561
H	-2.7757	7.39008	0.87813
H	-3.99335	7.42074	-2.02255
H	-3.26105	5.79902	-2.39127
H	-5.06206	5.96194	-2.20362
H	-3.75555	-8.19667	0.57587
H	-4.60279	-7.58579	-0.91114
H	-2.7987	-7.41944	-0.75955
H	-3.96484	-7.37824	2.16144
H	-3.22555	-5.74865	2.47773
H	-5.02965	-5.91465	2.32596
H	-7.20833	-6.22715	-0.11546
H	-6.20605	-6.56952	1.36116
H	-6.96835	-4.93403	1.13883
H	-6.44822	-6.09422	-1.73884
H	-5.41746	-4.66241	-2.17432
H	-4.65484	-6.29773	-1.95183