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Manuscript Title:

Selective synthesis of dibenzo[*a*,*c*]cyclooctatetraenes via palladium-catalyzed [4+4] cyclic homocoupling of borylvinyl iodobenzene derivatives

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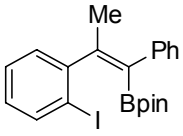
Contents:

1) General Information

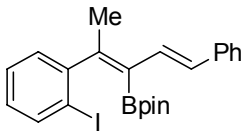
All reactions were carried out using standard Schlenk techniques under nitrogen. Et_2O and THF were distilled from sodium and benzophenone. All other commercial reagents were used without further purification. ^1H and ^{13}C NMR spectra were recorded at 300 and 75.4 MHz, in $CDCl_3$ solutions and with tetramethylsilane (0.00 ppm) as internal standard.

2) Procedures and Characterization Data

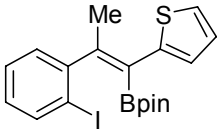
A typical procedure for the preparation of styrene derivatives 5. To a 4 mL solution of ArI (0.6 mmol) and $Pd(OAc)_2$ (0.025 mmol) in MeOH was added *gem*-diboryl reagent **4** (0.5 mmol) and K_2CO_3 (1.5 mmol), and the mixture was stirred at room temperature while the reaction was monitored by TLC. Then the MeOH was removed under reduced pressure and the residue was purified by column chromatography using silica gel (PE: Et_2O = 20:1) to afford the final products.



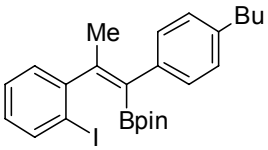
5a: colorless liquid, isolated yield 59% (132 mg). ^1H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.86 (s, 12H), 1.88 (s, 3H), 6.84-6.89 (m, 1H), 7.15-7.28 (m, 7H), 7.72-7.75 (d, J = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 21.9, 24.2, 24.3, 83.0, 98.8, 126.0, 127.6, 128.0, 128.1, 128.7, 128.9, 138.5, 140.8, 149.6, 151.4; HRMS calcd for $[C_{21}H_{24}BIO_2]H^+$ 447.0992, found 447.0985.



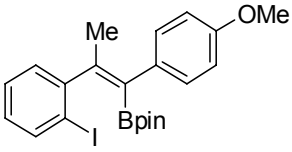
5b: colorless liquid, isolated yield 54% (127 mg). ^1H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.98-1.04 (s, 12H), 2.17 (s, 3H), 6.62-6.94 (m, 3H), 7.20-7.44 (m, 7H), 7.79-7.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 20.3, 24.3, 24.6, 83.3, 98.6, 126.4, 127.2, 127.7, 128.0, 128.3, 128.4, 129.4, 132.4, 138.1, 138.6, 149.9, 150.0; HRMS calcd for $[C_{23}H_{26}BIO_2]H^+$ 473.1149, found 473.1144.



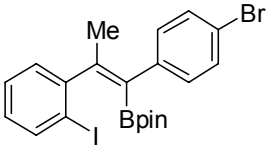
5c: colorless solid, isolated yield 55% (124 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.95 (s, 6H), 0.99 (s, 6H), 2.21 (s, 3H), 6.92-6.97 (m, 1H), 7.03-7.05 (m, 2H), 7.25-7.30 (m, 3H), 7.80-7.83 (d, J = 8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 22.6, 24.2, 24.3, 83.3, 98.6, 124.8, 126.6, 126.7, 127.6, 128.3, 129.1, 138.6, 142.2, 149.8, 150.5; HRMS calcd for $[C_{19}H_{22}BIO_2S]H^+$ 453.0557, found 453.0546. CCDC: 782988.[1]



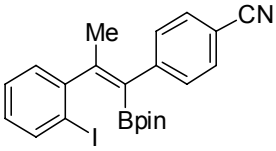
5d: colorless liquid, isolated yield 57% (143 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.94 (s, 15H), 1.36-1.42 (m, 2H), 1.58-1.66 (m, 2H), 1.97 (s, 3H), 2.58-2.64 (m, 2H), 6.90-6.96 (m, 1H), 7.14-7.30 (m, 6H), 7.78-7.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 14.0, 21.9, 22.5, 24.2, 24.3, 33.6, 35.4, 83.0, 98.9, 127.6, 128.0, 128.1, 128.7, 129.0, 137.9, 138.5, 140.5, 149.8, 150.8; HRMS calcd for $[C_{25}H_{32}BIO_2]H^+$ 503.1618, found 503.1602.



5e: colorless liquid, isolated yield 47% (112 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.94 (s, 12H), 1.97 (s, 3H), 3.82 (s, 3H), 6.89-6.92 (m, 3H), 7.22-7.29 (m, 4H), 7.79-7.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 21.9, 24.2, 24.3, 55.2, 83.0, 99.0, 113.4, 127.6, 128.1, 128.9, 129.9, 133.1, 138.5, 149.7, 150.8, 157.9; HRMS calcd for $[C_{22}H_{26}BIO_3]H^+$ 477.1098, found 477.1094.



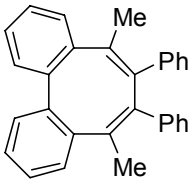
5f: colorless liquid, isolated yield 48% (126 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.94 (s, 12H), 1.93 (s, 3H), 6.92-6.97 (m, 1H), 7.16-7.20 (m, 2H), 7.25-7.32 (m, 2H), 7.46-7.49 (m, 2H), 7.79-7.82 (d, J = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 22.0, 24.2, 24.3, 83.1, 98.5, 120.0, 127.7, 128.2, 128.6, 130.5, 131.1, 138.5, 139.8, 149.2, 152.4; HRMS calcd for $[C_{21}H_{23}BBrIO_2]H^+$ 525.0097, found 525.0100.



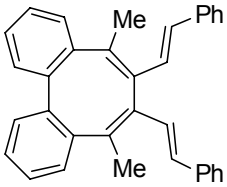
5g: colorless liquid, isolated yield 50% (118 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.95 (s, 12H), 1.92 (s, 3H), 6.94-6.99 (m, 1H), 7.25-7.34 (m, 2H), 7.40-7.42 (m, 2H),

7.64-7.68 (m, 2H), 7.79-7.82 (d, J = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 22.2, 24.2, 24.3, 83.3, 98.2, 109.7, 119.3, 127.7, 128.4, 129.6, 131.9, 138.5, 146.3, 148.8, 154.0; HRMS calcd for $[C_{22}H_{23}BINO_2]H^+$ 472.0945, found 472.0931.

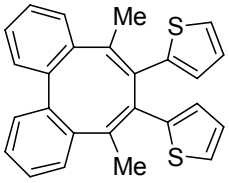
A typical procedure for the preparation of dibenzo[*a*,*c*]cyclooctatetraene 6. To a 3 mL solution of styrene derivatives **5** (0.3 mmol) and $Pd(PPh_3)_4$ (0.015 mmol) in DMF was added Cs_2CO_3 (0.9 mmol) and the mixture was stirred for 8 h at 120 ^{o}C. Then the resulting mixture was diluted with saturated NH_4Cl solution and extracted with Et_2O three times. The organic layers were combined, washed with brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by column chromatograph using silica gel (PE: Et_2O = 50:1) to afford the final products.



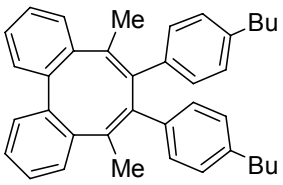
6a: colorless liquid, isolated yield 67% (39 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 1.28 (s, 6H), 6.42-6.48 (m, 4H), 6.56-6.78 (m, 6H), 6.80-6.82 (m, 2H), 6.84-6.90 (m, 2H), 6.92-7.02 (m, 4H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 21.6, 126.3, 126.4, 126.6, 127.0, 127.6, 128.6, 129.1, 133.9, 140.5, 140.6, 144.6; HRMS calcd for $[C_{30}H_{24}]H^+$ 385.1956, found 385.1948.



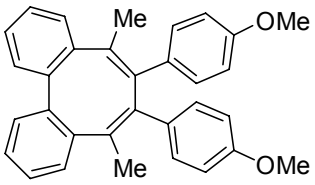
6b: colorless solid, isolated yield 66% (43 mg); mp: 190-192 ºC. ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 2.05 (s, 6H), 6.42-6.54 (m, 2H), 7.02-7.45 (m, 20H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 20.3, 125.0, 126.1, 126.3, 126.4, 127.0, 127.2, 128.4, 128.5, 130.5, 135.6, 137.2, 138.1, 140.2, 144.7; HRMS calcd for $[C_{34}H_{28}]H^+$ 437.2269, found 437.2258. CCDC: 775681.[1]



6c: colorless liquid, isolated yield 77% (46 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 1.98 (s, 6H), 6.78-6.88 (m, 4H), 7.10-7.14 (m, 2H), 7.22-7.34 (m, 8H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 22.0, 124.7, 126.4, 126.5, 126.6, 126.9, 127.1, 128.4, 133.2, 136.1, 140.0, 141.7, 144.1; HRMS calcd for $[C_{26}H_{20}S_2]H^+$ 397.1085, found 397.1083.

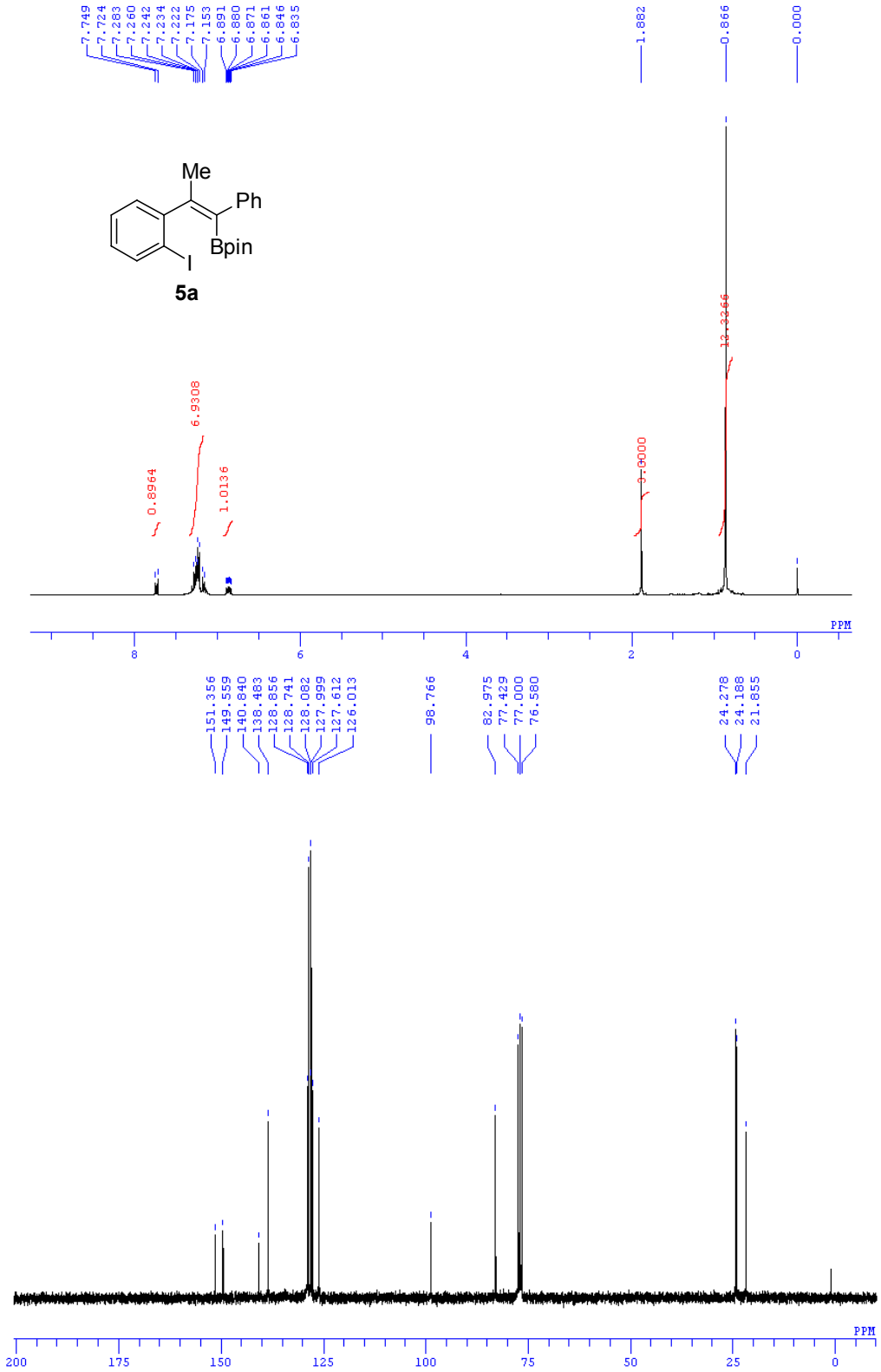


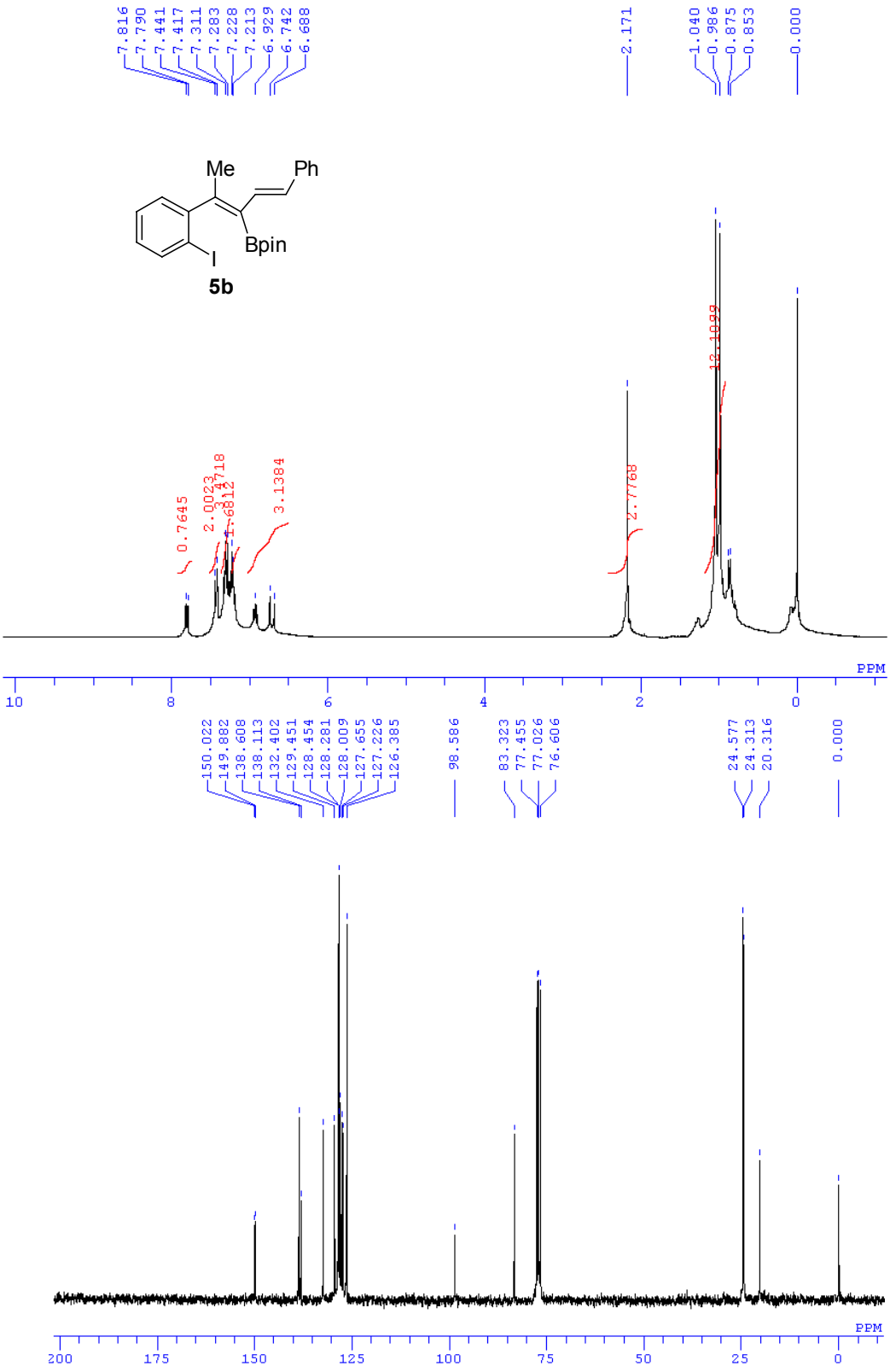
6d: colorless liquid, isolated yield 66% (49 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 0.85-0.90 (m, 6H), 1.26-1.34 (m, 4H), 1.48-1,56 (m, 4H), 1.70-1.74 (m, 6H), 2.46-2.54 (m, 4H), 6.80-6.96 (m, 8H), 7.26-7.40 (m, 8H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 14.0, 21.6, 22.3, 33.5, 35.3, 126.3, 126.6, 127.0, 128.5, 129.0, 133.4, 137.2, 140.5, 140.6, 140.7, 144.8; HRMS calcd for $[C_{38}H_{40}]H^+$ 497.3208, found 497.3205.



6e: colorless liquid, isolated yield 50% (33 mg). ^{1}H NMR ($CDCl_3$, 300 MHz, Me_4Si) δ 1.72 (s, 6H), 3.72 (s, 6H), 6.66-6.70 (m, 4H), 6.82-6.86 (m, 4H), 7.24-7.38 (m, 8H); ^{13}C NMR ($CDCl_3$, 75.4 MHz, Me_4Si) δ 21.6, 55.0, 113.0, 126.3, 126.6, 127.0, 128.5, 130.2, 132.4, 133.3, 140.2, 140.5, 144.8, 157.9; HRMS calcd for $[C_{32}H_{28}O_2]H^+$ 445.2168, found 445.2167.

3) ^{1}H and ^{13}C NMR Spectra

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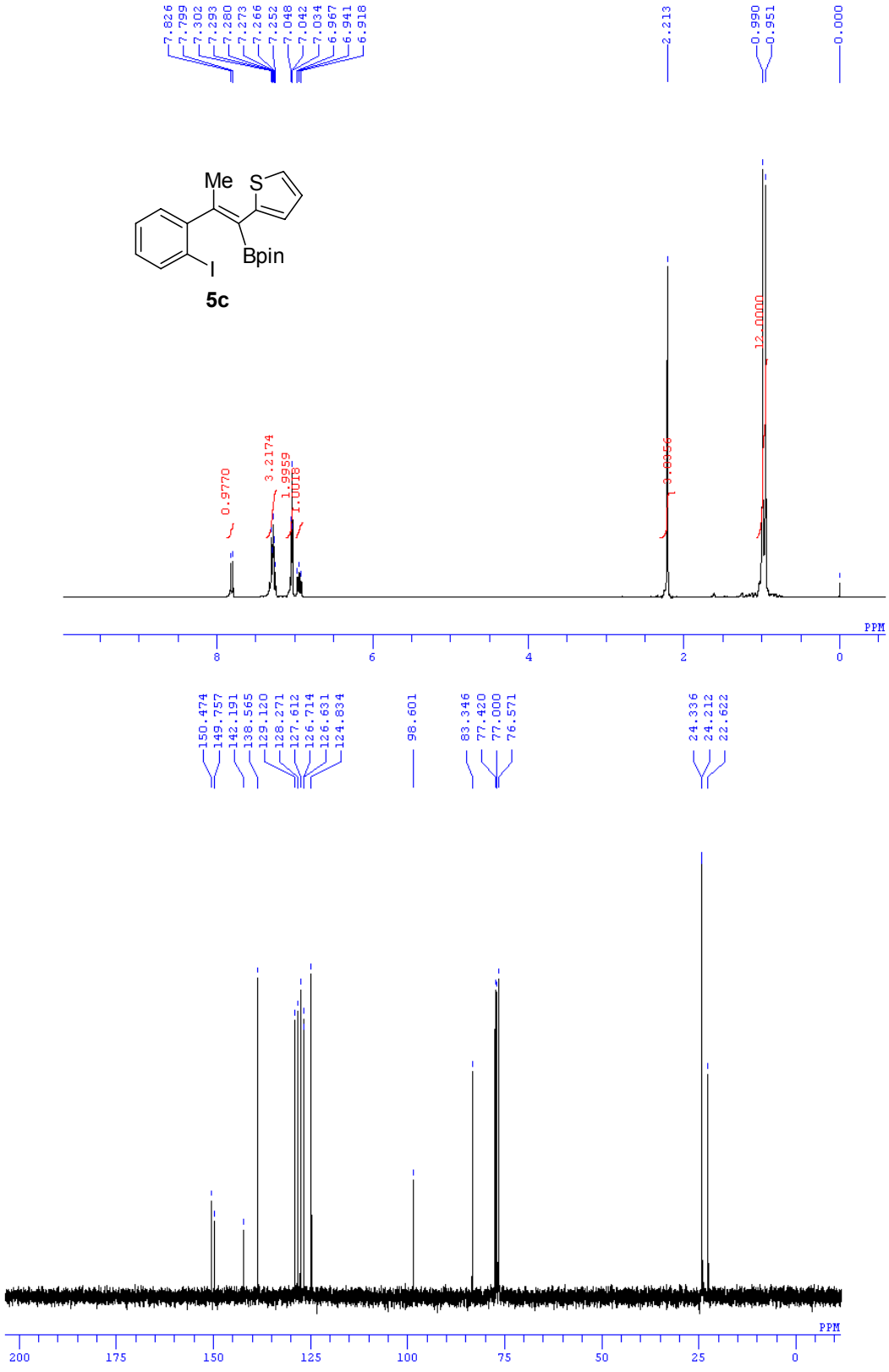
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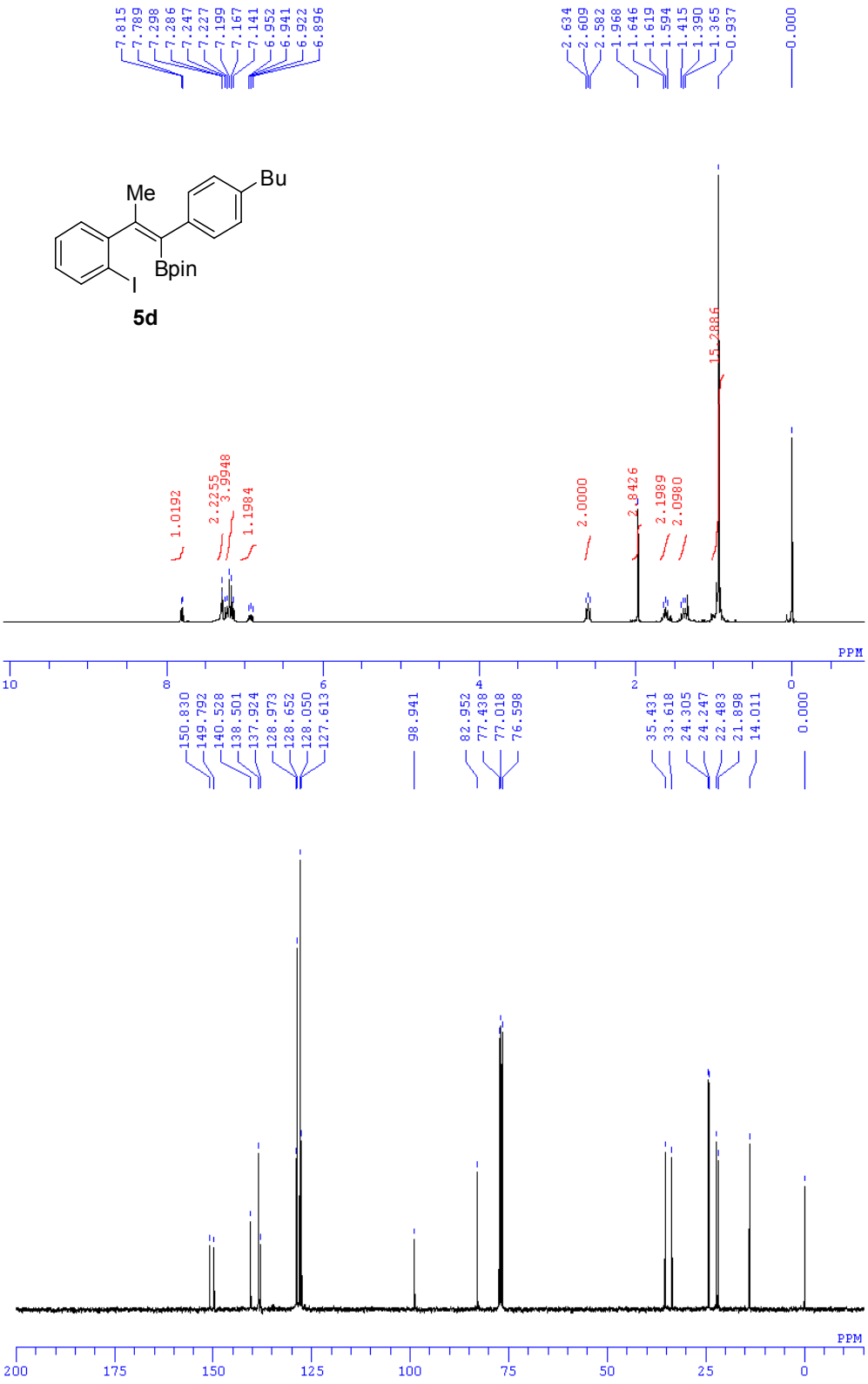
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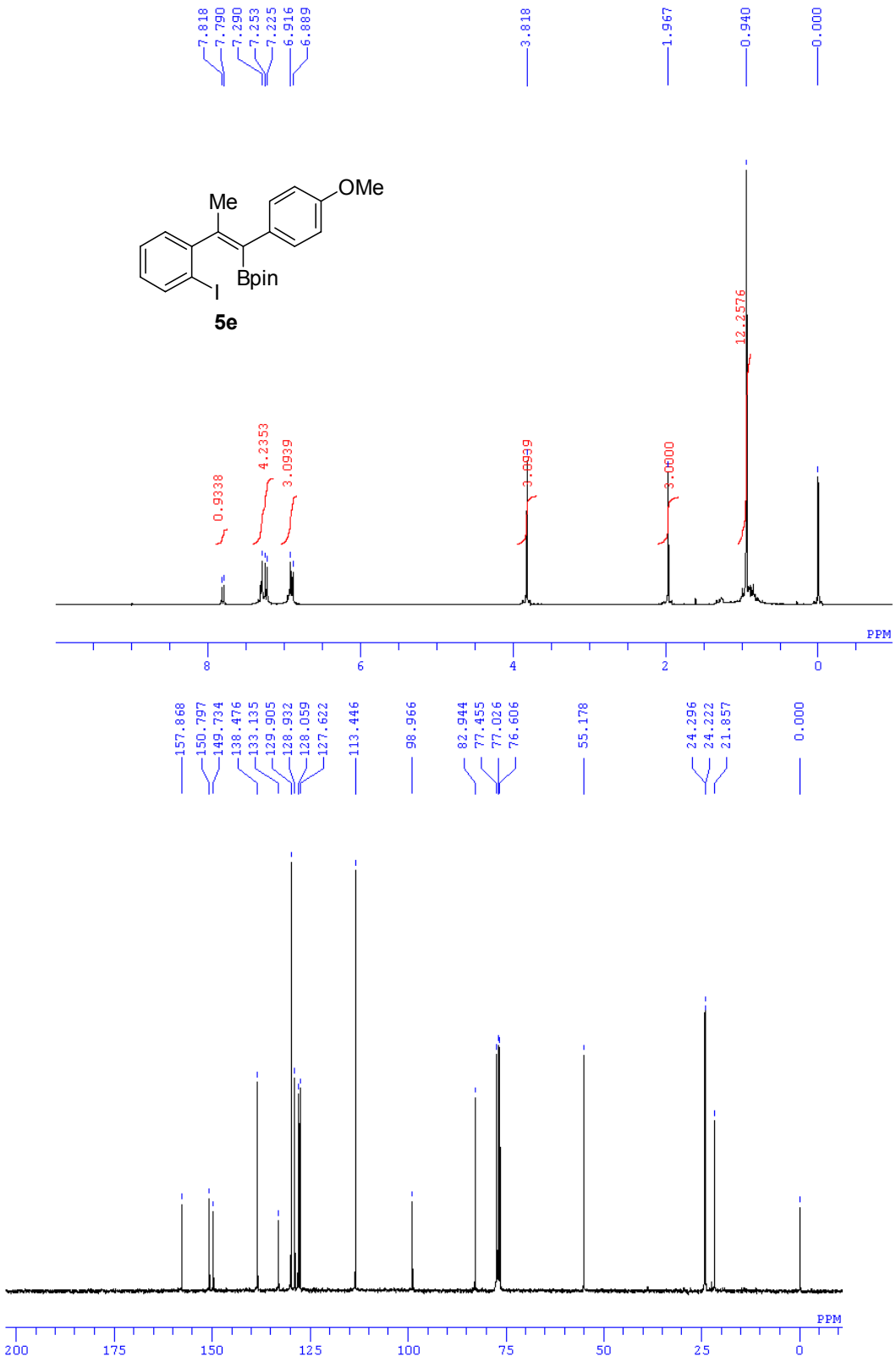
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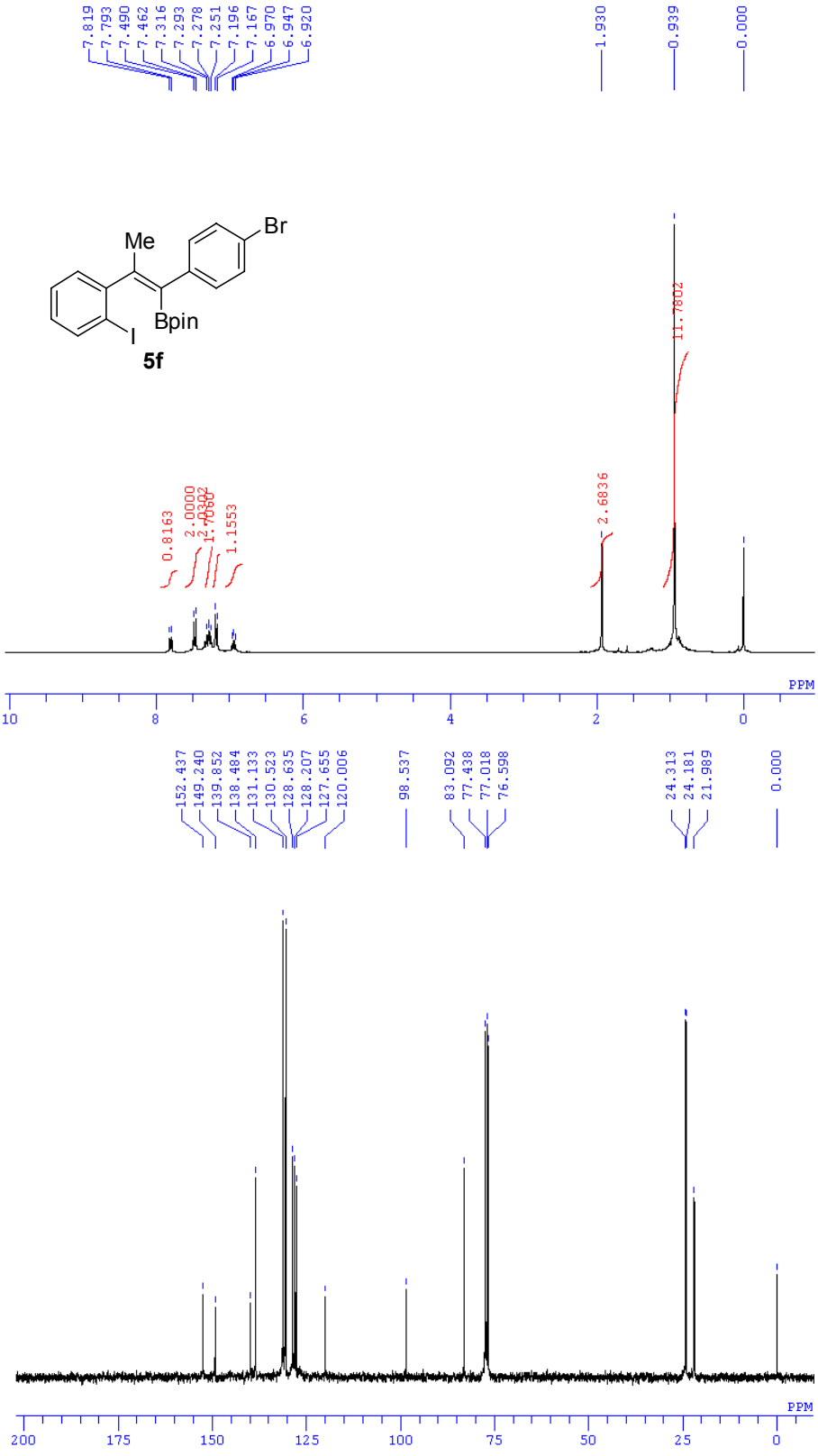
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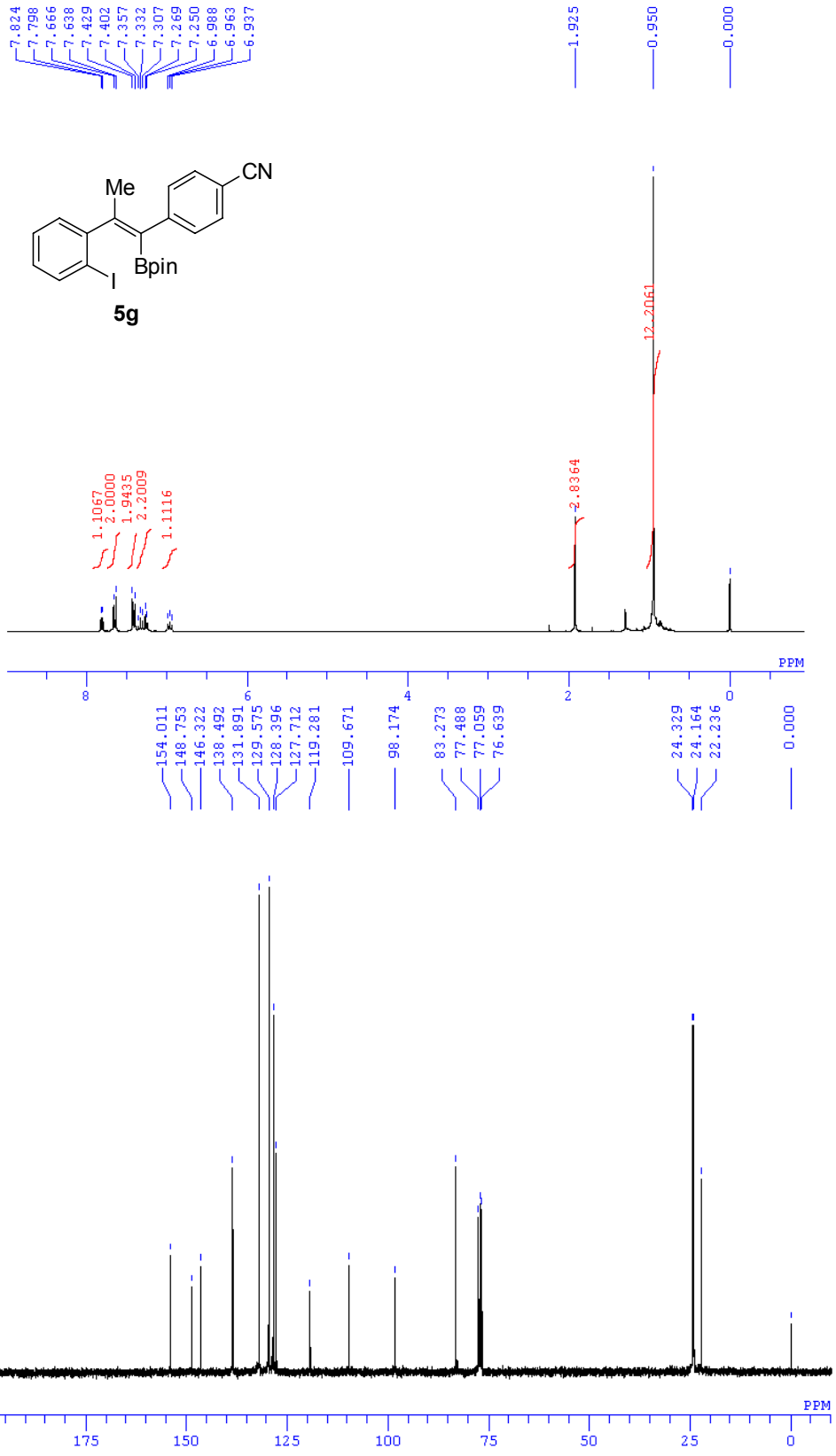
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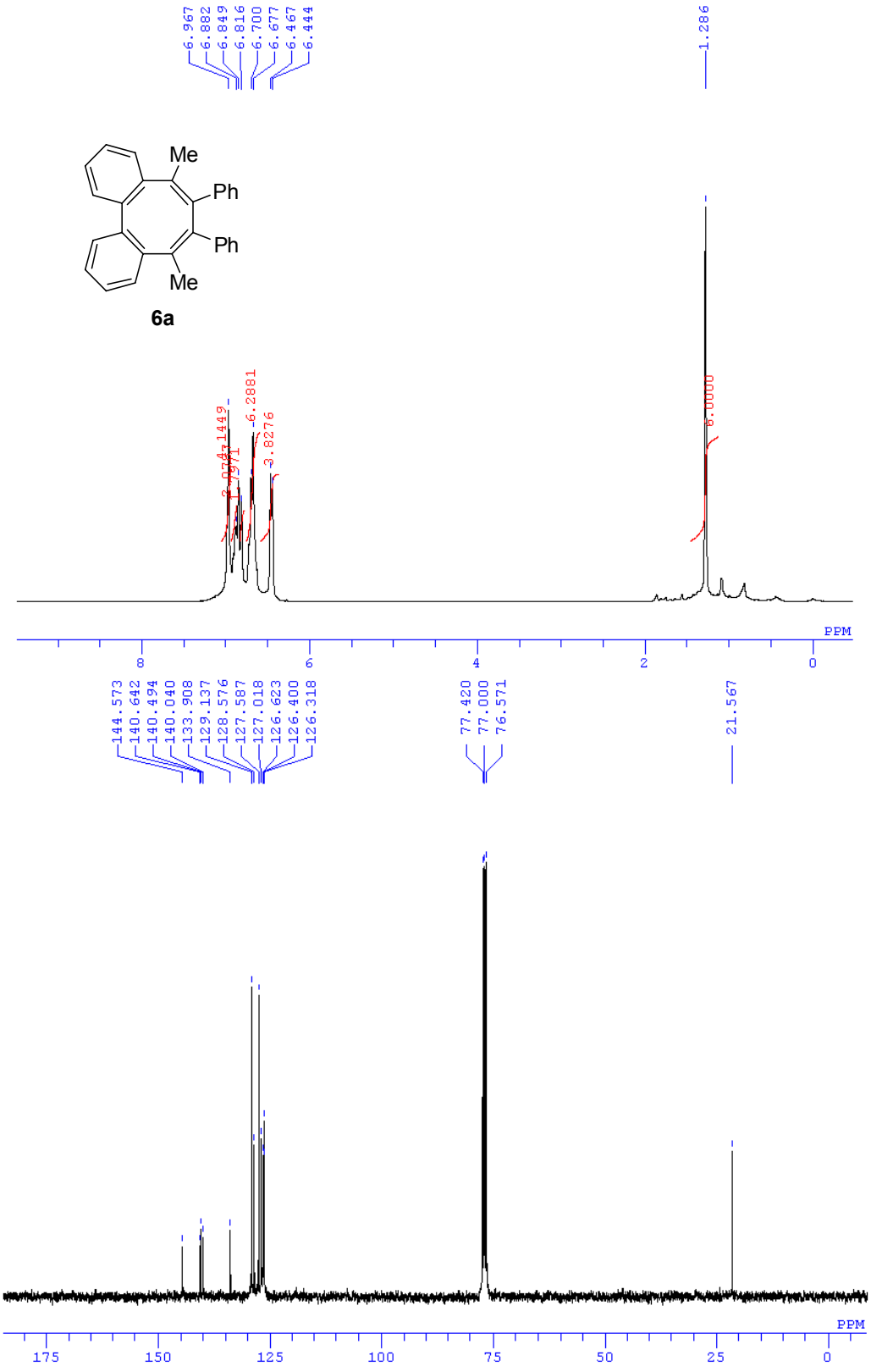
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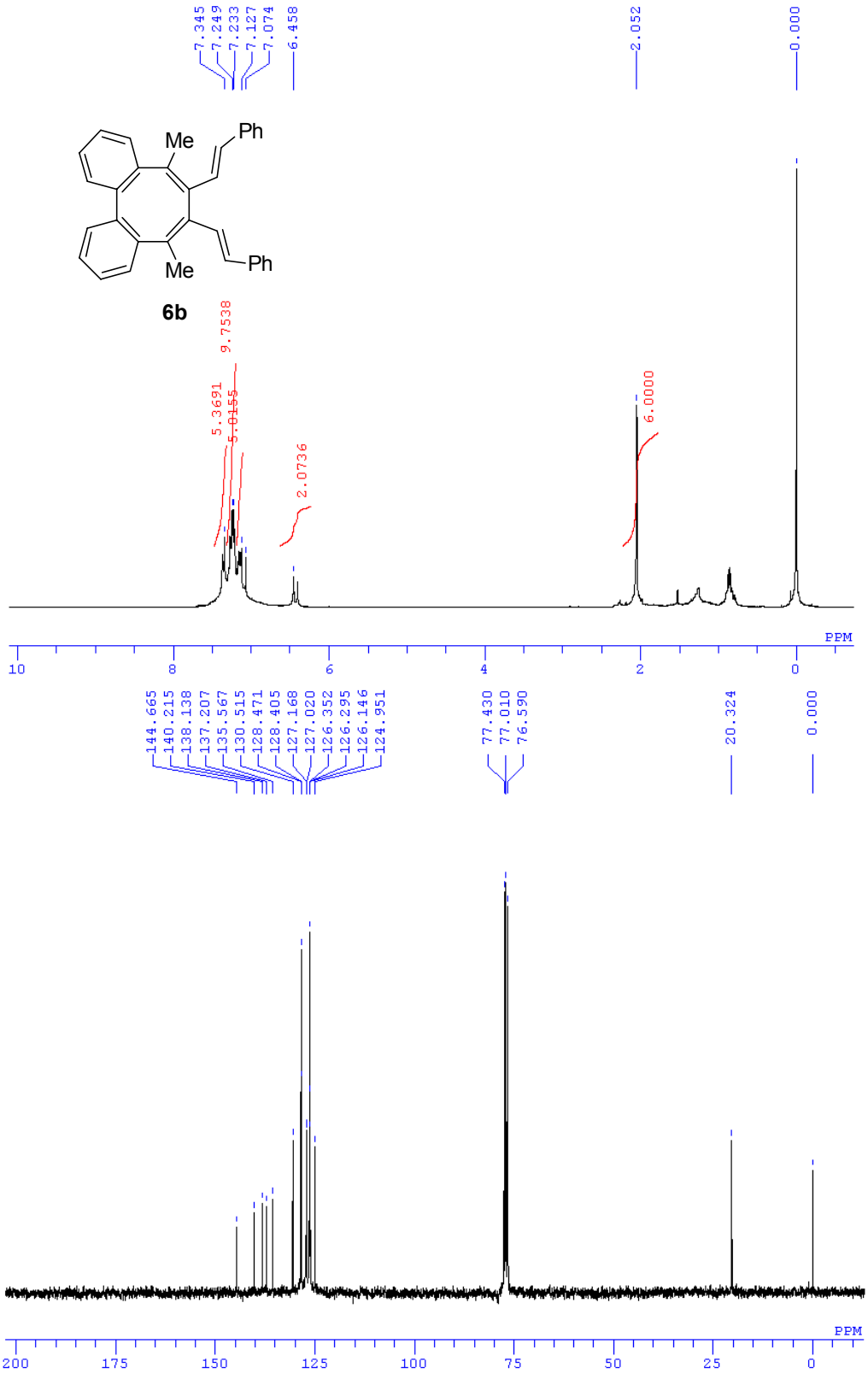
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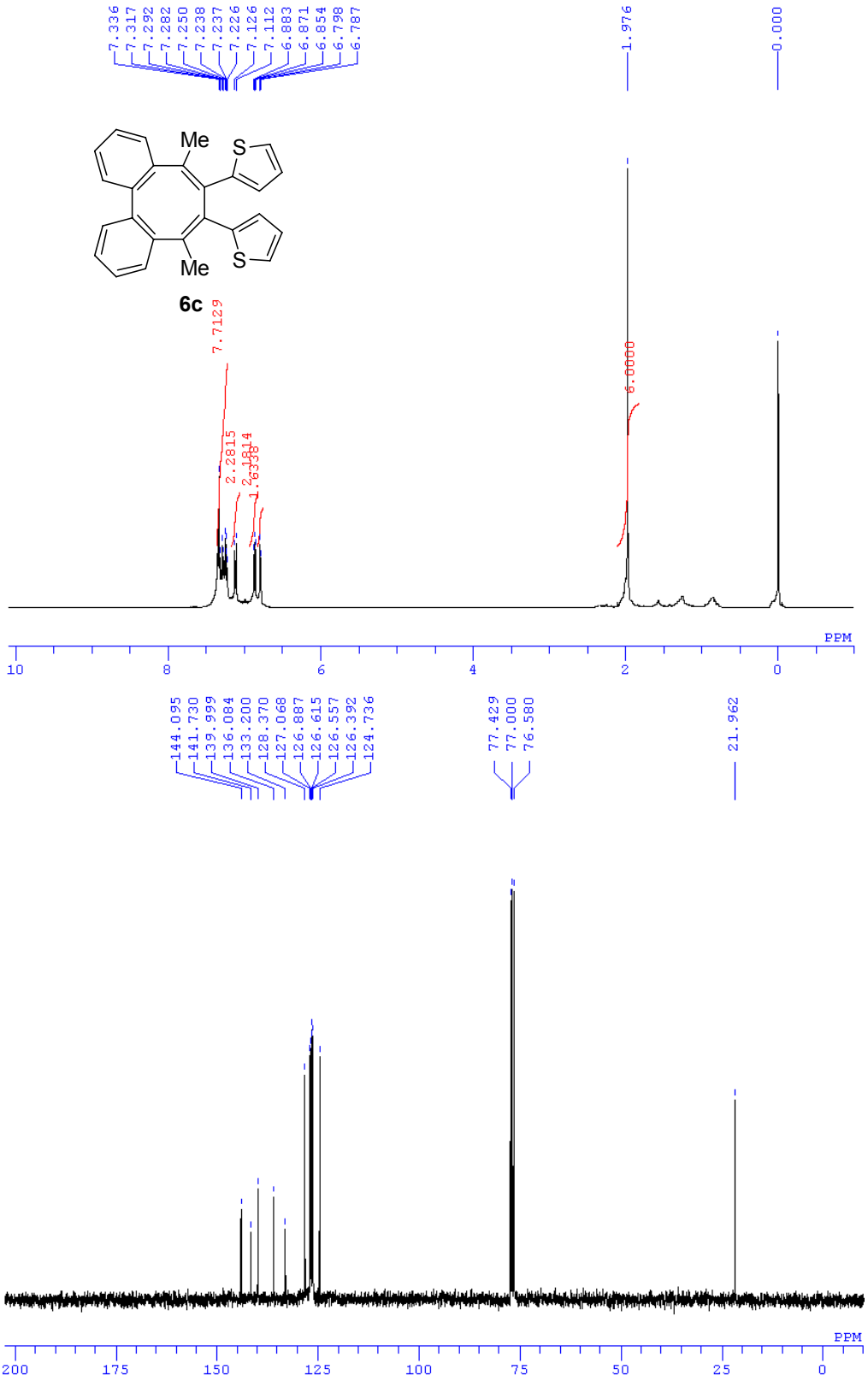
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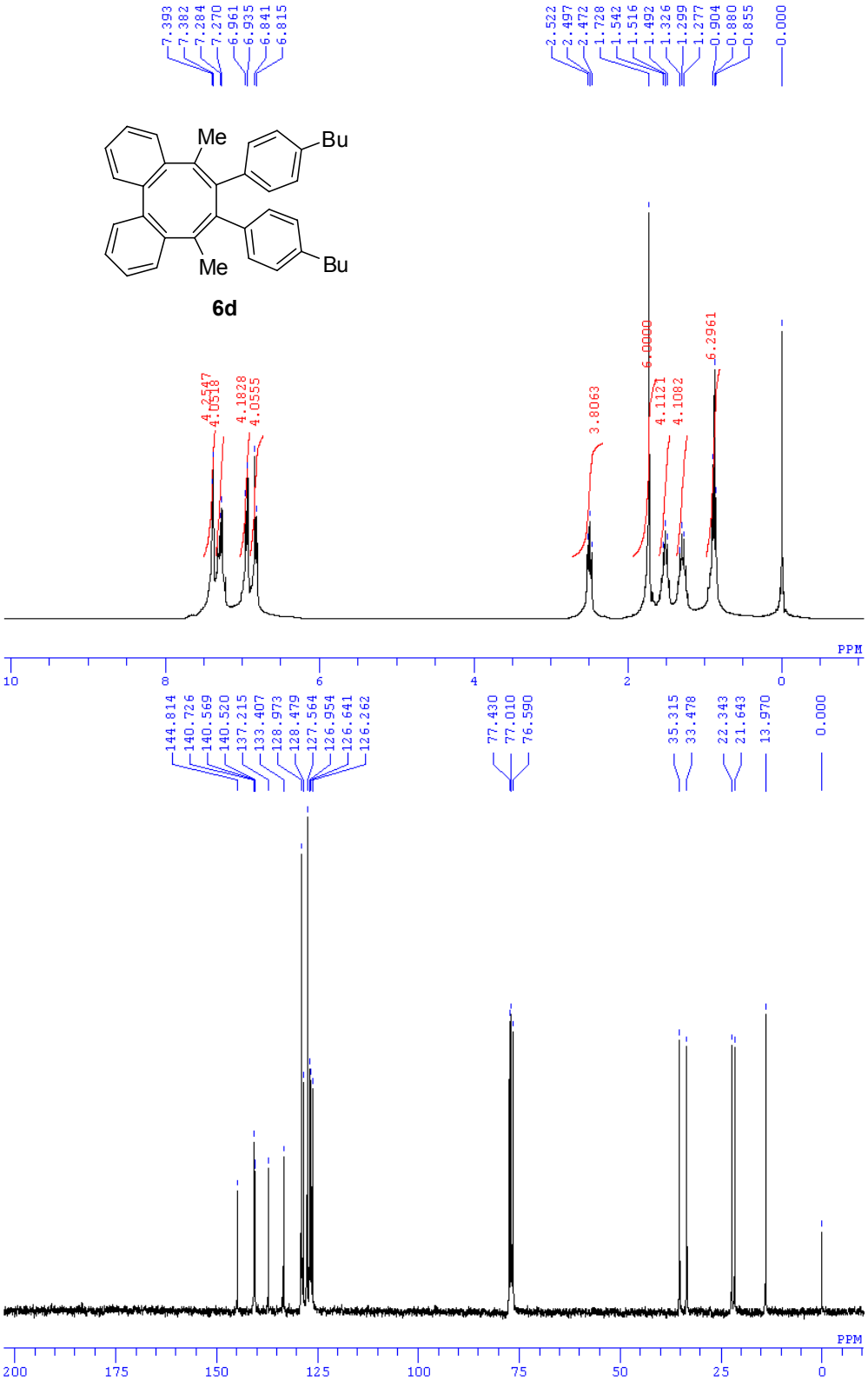
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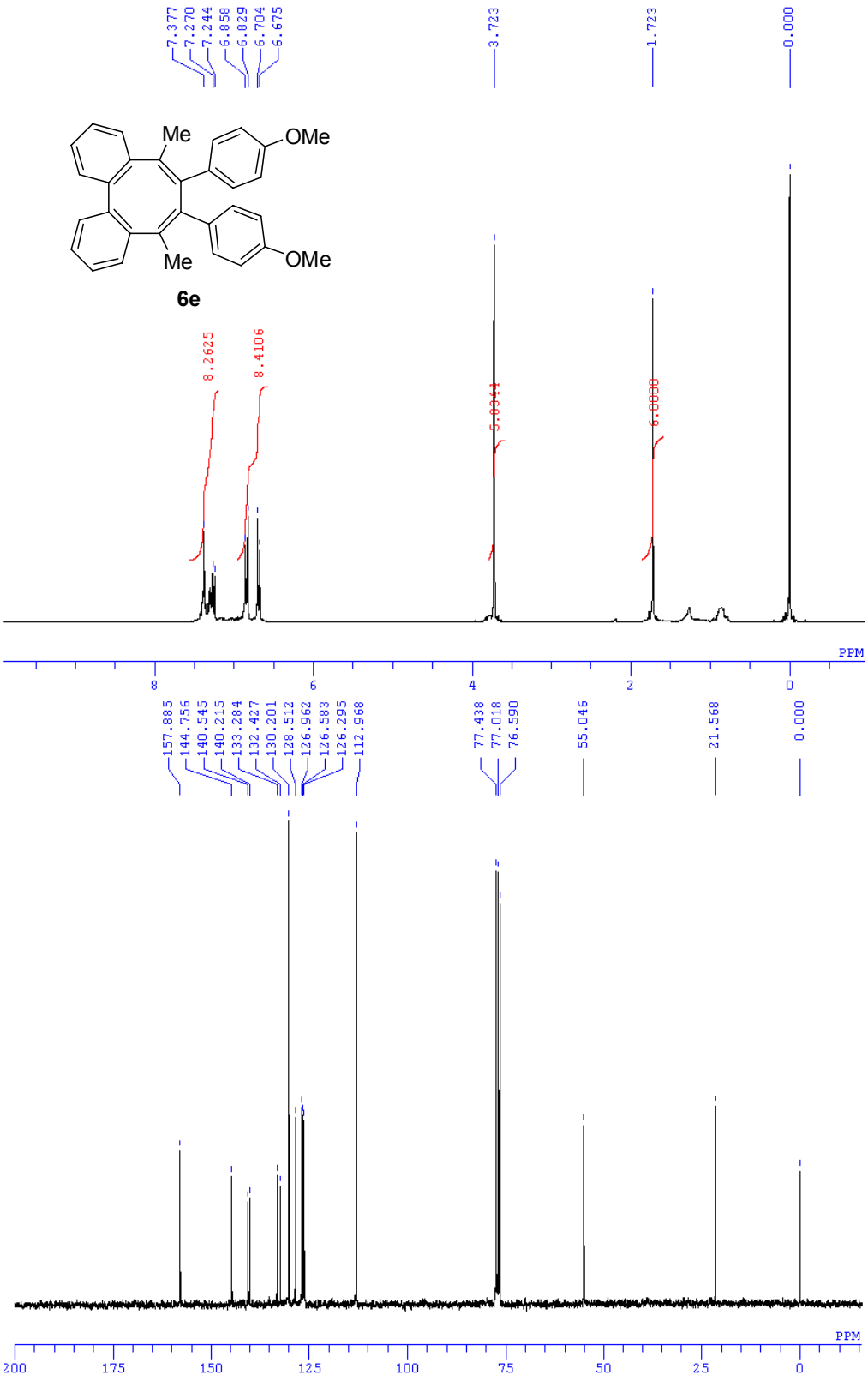
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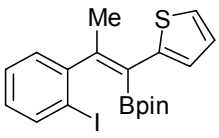
4) X-ray Single-crystal Structures of 5c and 6b

5c:



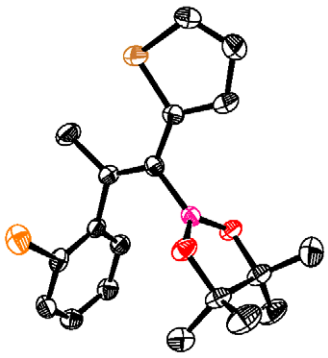


Table 1. Crystal data and structure refinement for **5c**.

Identification code	**5c**
Empirical formula	C19 H22 B I O2 S
Formula weight	452.14
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 7.7475(4) A alpha = 90 deg.
	b = 14.4249(8) A beta = 90 deg.
	c = 34.686(2) A gamma = 90 deg.
Volume	3876.4(4) A^3
Z, Calculated density	8, 1.549 Mg/m^3
Absorption coefficient	1.768 mm^-1
F(000)	1808
Crystal size	0.18 x 0.16 x 0.11 mm
Theta range for data collection	2.82 to 24.99 deg.
Limiting indices	-8<=h<=8, -17<=k<=12, -41<=l<=31
Reflections collected / unique	10497 / 3370 [R(int) = 0.0374]
Completeness to theta = 24.99	98.7 %
Absorption correction	Numerical
Max. and min. transmission	0.8293 and 0.7414
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3370 / 216 / 241
Goodness-of-fit on F^2	1.151
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.0988

R indices (all data)	R1 = 0.0536, wR2 = 0.1024
Largest diff. peak and hole	0.468 and -0.650 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **5c**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
I(1)	1804(1)	7540(1)	2927(1)	44(1)
S(1)	-2686(2)	6938(1)	4090(1)	37(1)
C(13)	-966(14)	7948(6)	4550(2)	40(2)
S(1')	-780(30)	8177(10)	4611(3)	37(3)
C(13')	-2390(60)	7080(20)	4196(7)	36(3)
O(1)	571(4)	10029(2)	4125(1)	31(1)
O(2)	2493(4)	8940(2)	3918(1)	33(1)
C(1)	-410(5)	9165(3)	3202(1)	27(1)
C(2)	-937(5)	10082(3)	3205(1)	30(1)
C(3)	-312(6)	10714(3)	2937(1)	32(1)
C(4)	862(6)	10427(3)	2662(1)	33(1)
C(5)	1448(6)	9523(3)	2657(1)	33(1)
C(6)	807(6)	8901(3)	2929(1)	29(1)
C(7)	-1195(5)	8487(3)	3482(1)	29(1)
C(8)	-2562(8)	7853(4)	3307(1)	49(1)
C(9)	-702(5)	8488(3)	3852(1)	26(1)
C(10)	-1343(5)	7881(3)	4156(1)	27(1)
C(11)	-2670(7)	6671(3)	4564(1)	39(1)
C(12)	-1729(7)	7255(3)	4779(1)	45(1)
C(14)	2288(6)	10385(3)	4229(1)	34(1)
C(15)	2473(8)	10231(4)	4662(2)	55(1)
C(16)	2353(7)	11408(3)	4137(2)	50(1)
C(17)	3535(6)	9778(3)	3983(1)	34(1)
C(18)	5189(6)	9489(4)	4186(2)	51(1)
C(19)	3917(7)	10182(4)	3588(2)	48(1)

B(1)	819(6)	9169(3)	3968(1)	25(1)

Table 3. Bond lengths [A] and angles [deg] for **5c**.

I(1)-C(6)	2.110(4)	C(9)-C(10)	1.459(6)
S(1)-C(11)	1.689(5)	C(9)-B(1)	1.587(6)
S(1)-C(10)	1.728(4)	C(11)-C(12)	1.341(7)
C(13)-C(10)	1.399(8)	C(11)-H(11)	0.9500
C(13)-C(12)	1.408(8)	C(12)-H(12)	0.9500
C(13)-H(13)	0.9500	C(14)-C(16)	1.511(6)
S(1')-C(12)	1.628(12)	C(14)-C(15)	1.525(7)
S(1')-C(10)	1.691(12)	C(14)-C(17)	1.558(6)
C(13')-C(10)	1.421(16)	C(15)-H(15A)	0.9800
C(13')-C(11)	1.424(16)	C(15)-H(15B)	0.9800
C(13')-H(13')	0.9500	C(15)-H(15C)	0.9800
O(1)-B(1)	1.368(5)	C(16)-H(16A)	0.9800
O(1)-C(14)	1.471(5)	C(16)-H(16B)	0.9800
O(2)-B(1)	1.349(6)	C(16)-H(16C)	0.9800
O(2)-C(17)	1.471(5)	C(17)-C(19)	1.517(7)
C(1)-C(2)	1.385(6)	C(17)-C(18)	1.521(6)
C(1)-C(6)	1.390(6)	C(18)-H(18A)	0.9800
C(1)-C(7)	1.506(6)	C(18)-H(18B)	0.9800
C(2)-C(3)	1.391(6)	C(18)-H(18C)	0.9800
C(2)-H(2A)	0.9500	C(19)-H(19A)	0.9800
C(3)-C(4)	1.381(6)	C(19)-H(19B)	0.9800
C(3)-H(3A)	0.9500	C(19)-H(19C)	0.9800
C(4)-C(5)	1.382(6)	C(11)-S(1)-C(10)	92.6(2)
C(4)-H(4A)	0.9500	C(10)-C(13)-C(12)	114.6(6)
C(5)-C(6)	1.394(6)	C(10)-C(13)-H(13)	122.7
C(5)-H(5A)	0.9500	C(12)-C(13)-H(13)	122.7
C(7)-C(9)	1.339(6)	C(12)-S(1')-C(10)	90.7(6)
C(7)-C(8)	1.525(6)	C(10)-C(13')-C(11)	120.6(19)
C(8)-H(8A)	0.9800	C(10)-C(13')-H(13')	119.7
C(8)-H(8B)	0.9800	C(11)-C(13')-H(13')	119.7
C(8)-H(8C)	0.9800	B(1)-O(1)-C(14)	106.7(3)

B(1)-O(2)-C(17)	107.8(3)	C(9)-C(10)-S(1')	115.8(5)
C(2)-C(1)-C(6)	117.8(4)	C(13)-C(10)-S(1)	108.0(4)
C(2)-C(1)-C(7)	119.8(4)	C(13')-C(10)-S(1)	13.3(10)
C(6)-C(1)-C(7)	122.4(4)	C(9)-C(10)-S(1)	125.6(3)
C(1)-C(2)-C(3)	121.3(4)	S(1')-C(10)-S(1)	118.6(5)
C(1)-C(2)-H(2A)	119.4	C(12)-C(11)-C(13')	99.1(11)
C(3)-C(2)-H(2A)	119.4	C(12)-C(11)-S(1)	113.7(4)
C(4)-C(3)-C(2)	119.7(4)	C(13')-C(11)-S(1)	14.6(10)
C(4)-C(3)-H(3A)	120.2	C(12)-C(11)-H(11)	123.2
C(2)-C(3)-H(3A)	120.2	C(13')-C(11)-H(11)	137.8
C(3)-C(4)-C(5)	120.6(4)	S(1)-C(11)-H(11)	123.2
C(3)-C(4)-H(4A)	119.7	C(11)-C(12)-C(13)	111.1(5)
C(5)-C(4)-H(4A)	119.7	C(11)-C(12)-S(1')	124.0(6)
C(4)-C(5)-C(6)	118.8(4)	C(13)-C(12)-S(1')	13.5(6)
C(4)-C(5)-H(5A)	120.6	C(11)-C(12)-H(12)	124.5
C(6)-C(5)-H(5A)	120.6	C(13)-C(12)-H(12)	124.5
C(1)-C(6)-C(5)	121.8(4)	S(1')-C(12)-H(12)	111.4
C(1)-C(6)-I(1)	120.4(3)	O(1)-C(14)-C(16)	108.6(4)
C(5)-C(6)-I(1)	117.7(3)	O(1)-C(14)-C(15)	106.0(4)
C(9)-C(7)-C(1)	120.1(4)	C(16)-C(14)-C(15)	110.4(4)
C(9)-C(7)-C(8)	125.5(4)	O(1)-C(14)-C(17)	103.3(3)
C(1)-C(7)-C(8)	114.4(4)	C(16)-C(14)-C(17)	114.4(4)
C(7)-C(8)-H(8A)	109.5	C(15)-C(14)-C(17)	113.5(4)
C(7)-C(8)-H(8B)	109.5	C(14)-C(15)-H(15A)	109.5
H(8A)-C(8)-H(8B)	109.5	C(14)-C(15)-H(15B)	109.5
C(7)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15B)	109.5
H(8A)-C(8)-H(8C)	109.5	C(14)-C(15)-H(15C)	109.5
H(8B)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15C)	109.5
C(7)-C(9)-C(10)	126.5(4)	H(15B)-C(15)-H(15C)	109.5
C(7)-C(9)-B(1)	117.1(4)	C(14)-C(16)-H(16A)	109.5
C(10)-C(9)-B(1)	116.2(3)	C(14)-C(16)-H(16B)	109.5
C(13)-C(10)-C(13')	94.7(11)	H(16A)-C(16)-H(16B)	109.5
C(13)-C(10)-C(9)	126.4(5)	C(14)-C(16)-H(16C)	109.5
C(13')-C(10)-C(9)	138.9(10)	H(16A)-C(16)-H(16C)	109.5
C(13)-C(10)-S(1')	11.2(6)	H(16B)-C(16)-H(16C)	109.5
C(13')-C(10)-S(1')	105.3(10)	O(2)-C(17)-C(19)	106.5(4)

O(2)-C(17)-C(18)	107.9(4)	H(18B)-C(18)-H(18C)	109.5
C(19)-C(17)-C(18)	111.1(4)	C(17)-C(19)-H(19A)	109.5
O(2)-C(17)-C(14)	101.9(3)	C(17)-C(19)-H(19B)	109.5
C(19)-C(17)-C(14)	113.5(4)	H(19A)-C(19)-H(19B)	109.5
C(18)-C(17)-C(14)	115.0(4)	C(17)-C(19)-H(19C)	109.5
C(17)-C(18)-H(18A)	109.5	H(19A)-C(19)-H(19C)	109.5
C(17)-C(18)-H(18B)	109.5	H(19B)-C(19)-H(19C)	109.5
H(18A)-C(18)-H(18B)	109.5	O(2)-B(1)-O(1)	114.1(4)
C(17)-C(18)-H(18C)	109.5	O(2)-B(1)-C(9)	122.0(4)
H(18A)-C(18)-H(18C)	109.5	O(1)-B(1)-C(9)	123.9(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **5c**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
I(1)	64(1)	25(1)	41(1)	0(1)	5(1)	11(1)
S(1)	41(1)	33(1)	36(1)	5(1)	1(1)	-10(1)
C(13)	45(4)	30(4)	44(3)	0(3)	1(3)	-14(3)
S(1')	48(4)	30(5)	35(4)	5(4)	-3(4)	-15(4)
C(13')	39(5)	33(5)	34(5)	1(5)	0(5)	-3(5)
O(1)	24(2)	26(2)	41(2)	-8(1)	2(1)	-3(1)
O(2)	25(2)	26(2)	49(2)	-7(1)	0(2)	0(1)
C(1)	29(2)	25(2)	26(2)	-3(2)	-4(2)	-4(2)
C(2)	28(2)	30(2)	31(2)	-3(2)	0(2)	-1(2)
C(3)	38(2)	24(2)	36(2)	0(2)	-1(2)	3(2)
C(4)	40(3)	26(2)	33(2)	6(2)	1(2)	-3(2)
C(5)	40(2)	31(2)	27(2)	-1(2)	5(2)	2(2)
C(6)	35(2)	22(2)	31(2)	-5(2)	-5(2)	3(2)
C(7)	28(2)	27(2)	31(2)	-3(2)	1(2)	-5(2)
C(8)	60(3)	51(3)	36(3)	4(2)	-11(3)	-28(3)
C(9)	24(2)	26(2)	27(2)	-4(2)	4(2)	3(2)

C(10)	27(2)	24(2)	29(2)	1(2)	3(2)	2(2)
C(11)	45(3)	35(2)	37(2)	8(2)	5(2)	-7(2)
C(12)	58(3)	44(3)	32(2)	2(2)	3(2)	-6(2)
C(14)	27(2)	27(2)	48(3)	-6(2)	-1(2)	-4(2)
C(15)	55(3)	70(4)	40(3)	-10(3)	-4(3)	-10(3)
C(16)	46(3)	26(2)	78(4)	-12(2)	-1(3)	-6(2)
C(17)	27(2)	27(2)	48(3)	-2(2)	0(2)	-5(2)
C(18)	30(3)	43(3)	80(4)	-2(3)	-13(3)	2(2)
C(19)	42(3)	51(3)	50(3)	-2(2)	6(3)	-9(2)
B(1)	27(2)	25(2)	23(2)	0(2)	4(2)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **5c**.

	x	y	z	U(eq)
H(13)	-253	8422	4654	48
H(13')	-2910	6810	3974	43
H(2A)	-1741	10283	3394	36
H(3A)	-691	11341	2942	39
H(4A)	1270	10855	2475	40
H(5A)	2273	9328	2471	39
H(8A)	-3607	7872	3466	74
H(8B)	-2122	7217	3297	74
H(8C)	-2837	8064	3046	74
H(11)	-3271	6154	4669	47
H(12)	-1595	7208	5051	53
H(15A)	1507	10528	4796	82
H(15B)	3562	10501	4751	82
H(15C)	2467	9564	4717	82
H(16A)	1580	11745	4311	75
H(16B)	1984	11507	3869	75
H(16C)	3535	11636	4169	75

H(18A)	5869	9090	4015	76
H(18B)	4902	9148	4422	76
H(18C)	5862	10042	4252	76
H(19A)	2836	10382	3467	72
H(19B)	4469	9709	3427	72
H(19C)	4692	10715	3616	72

Table 6. Torsion angles [deg] for **5c**.

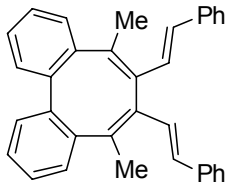
C(6)-C(1)-C(2)-C(3)	2.0(6)
C(7)-C(1)-C(2)-C(3)	-176.3(4)
C(1)-C(2)-C(3)-C(4)	-0.3(7)
C(2)-C(3)-C(4)-C(5)	-1.4(7)
C(3)-C(4)-C(5)-C(6)	1.3(7)
C(2)-C(1)-C(6)-C(5)	-2.1(6)
C(7)-C(1)-C(6)-C(5)	176.1(4)
C(2)-C(1)-C(6)-I(1)	176.0(3)
C(7)-C(1)-C(6)-I(1)	-5.8(5)
C(4)-C(5)-C(6)-C(1)	0.5(7)
C(4)-C(5)-C(6)-I(1)	-177.6(3)
C(2)-C(1)-C(7)-C(9)	-76.6(5)
C(6)-C(1)-C(7)-C(9)	105.2(5)
C(2)-C(1)-C(7)-C(8)	102.1(5)
C(6)-C(1)-C(7)-C(8)	-76.1(5)
C(1)-C(7)-C(9)-C(10)	179.7(4)
C(8)-C(7)-C(9)-C(10)	1.2(7)
C(1)-C(7)-C(9)-B(1)	-4.7(6)
C(8)-C(7)-C(9)-B(1)	176.8(4)
C(12)-C(13)-C(10)-C(13')	0(3)
C(12)-C(13)-C(10)-C(9)	-178.5(5)
C(12)-C(13)-C(10)-S(1')	162(6)
C(12)-C(13)-C(10)-S(1)	0.6(10)
C(11)-C(13')-C(10)-C(13)	0(4)

C(11)-C(13')-C(10)-C(9)	178.2(15)
C(11)-C(13')-C(10)-S(1')	-4(5)
C(11)-C(13')-C(10)-S(1)	-177(14)
C(7)-C(9)-C(10)-C(13)	-172.7(7)
B(1)-C(9)-C(10)-C(13)	11.7(8)
C(7)-C(9)-C(10)-C(13')	10(4)
B(1)-C(9)-C(10)-C(13')	-166(4)
C(7)-C(9)-C(10)-S(1')	-168.5(9)
B(1)-C(9)-C(10)-S(1')	15.9(9)
C(7)-C(9)-C(10)-S(1)	8.3(6)
B(1)-C(9)-C(10)-S(1)	-167.3(3)
C(12)-S(1')-C(10)-C(13)	-14(5)
C(12)-S(1')-C(10)-C(13')	5(3)
C(12)-S(1')-C(10)-C(9)	-176.7(5)
C(12)-S(1')-C(10)-S(1)	6.2(12)
C(11)-S(1)-C(10)-C(13)	-0.8(6)
C(11)-S(1)-C(10)-C(13')	2(10)
C(11)-S(1)-C(10)-C(9)	178.3(4)
C(11)-S(1)-C(10)-S(1')	-4.9(9)
C(10)-C(13')-C(11)-C(12)	0(5)
C(10)-C(13')-C(11)-S(1)	177(13)
C(10)-S(1)-C(11)-C(12)	0.9(4)
C(10)-S(1)-C(11)-C(13')	-2(10)
C(13')-C(11)-C(12)-C(13)	0(2)
S(1)-C(11)-C(12)-C(13)	-0.7(7)
C(13')-C(11)-C(12)-S(1')	4(3)
S(1)-C(11)-C(12)-S(1')	3.6(11)
C(10)-C(13)-C(12)-C(11)	0.0(10)
C(10)-C(13)-C(12)-S(1')	-164(6)
C(10)-S(1')-C(12)-C(11)	-5.9(13)
C(10)-S(1')-C(12)-C(13)	12(4)
B(1)-O(1)-C(14)-C(16)	141.0(4)
B(1)-O(1)-C(14)-C(15)	-100.4(4)
B(1)-O(1)-C(14)-C(17)	19.2(4)
B(1)-O(2)-C(17)-C(19)	-97.7(4)
B(1)-O(2)-C(17)-C(18)	143.0(4)

B(1)-O(2)-C(17)-C(14)	21.5(4)
O(1)-C(14)-C(17)-O(2)	-24.3(4)
C(16)-C(14)-C(17)-O(2)	-142.1(4)
C(15)-C(14)-C(17)-O(2)	90.0(4)
O(1)-C(14)-C(17)-C(19)	89.8(4)
C(16)-C(14)-C(17)-C(19)	-28.0(6)
C(15)-C(14)-C(17)-C(19)	-155.9(4)
O(1)-C(14)-C(17)-C(18)	-140.7(4)
C(16)-C(14)-C(17)-C(18)	101.5(5)
C(15)-C(14)-C(17)-C(18)	-26.4(5)
C(17)-O(2)-B(1)-O(1)	-10.6(5)
C(17)-O(2)-B(1)-C(9)	169.3(4)
C(14)-O(1)-B(1)-O(2)	-6.3(5)
C(14)-O(1)-B(1)-C(9)	173.8(4)
C(7)-C(9)-B(1)-O(2)	-83.1(5)
C(10)-C(9)-B(1)-O(2)	93.0(5)
C(7)-C(9)-B(1)-O(1)	96.8(5)
C(10)-C(9)-B(1)-O(1)	-87.1(5)

Symmetry transformations used to generate equivalent atoms:

6b:



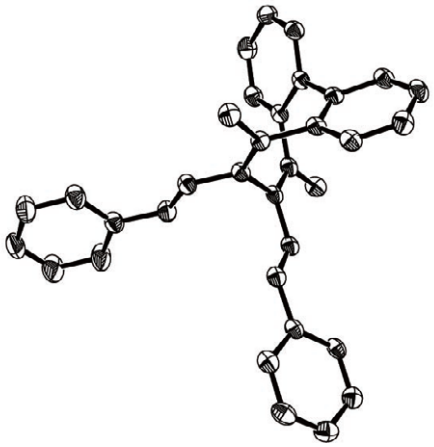


Table 1. Crystal data and structure refinement for **6b**.

Identification code	**6b**	
Empirical formula	C38 H38 O	
Formula weight	510.68	
Temperature	123(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.7703(16) Å	α= 96.96(3) °
	b = 11.284(2) Å	β= 96.69(3) °
	c = 17.614(4) Å	γ = 99.11(3) °
Volume	1499.2(5) $Å^3$	
Z	2	
Density (calculated)	1.131 Mg/m^3	
Absorption coefficient	0.066 mm^{-1}	
F(000)	548	
Crystal size	0.40 x 0.40 x 0.30 mm^3	
Theta range for data collection	2.33 to 27.48°.	
Index ranges	-10<=h<=10, -14<=k<=14, -22<=l<=22	
Reflections collected	10828	
Independent reflections	6783 [R(int) = 0.0393]	
Completeness to theta = 27.48°	98.7 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9805 and 0.9742	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6783 / 0 / 357
Goodness-of-fit on F^2	1.092
Final R indices [I>2sigma(I)]	R1 = 0.0528, wR2 = 0.1238
R indices (all data)	R1 = 0.0886, wR2 = 0.1333
Extinction coefficient	0.019(2)
Largest diff. peak and hole	0.627 and -0.230 e. $Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **6b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	320(2)	7653(2)	3543(1)	24(1)
C(2)	141(2)	8367(2)	4232(1)	29(1)
C(3)	-517(2)	7850(2)	4833(1)	33(1)
C(4)	-1010(2)	6608(2)	4765(1)	34(1)
C(5)	-881(2)	5885(2)	4082(1)	29(1)
C(6)	-229(2)	6395(2)	3465(1)	25(1)
C(7)	-263(2)	5611(1)	2710(1)	24(1)
C(8)	1256(2)	5463(1)	2431(1)	24(1)
C(9)	2999(2)	6004(2)	2909(1)	23(1)
C(10)	3642(2)	7198(2)	2944(1)	24(1)
C(11)	2551(2)	8033(1)	2608(1)	24(1)
C(12)	999(2)	8263(1)	2901(1)	24(1)
C(13)	117(2)	9135(2)	2615(1)	29(1)
C(14)	705(2)	9770(2)	2043(1)	33(1)
C(15)	2232(2)	9541(2)	1751(1)	35(1)
C(16)	3141(2)	8691(2)	2034(1)	30(1)
C(17)	-2091(2)	5091(2)	2301(1)	33(1)
C(18)	5459(2)	7803(2)	3334(1)	31(1)
C(19)	1284(2)	4783(2)	1669(1)	27(1)
C(20)	2726(2)	4639(2)	1344(1)	31(1)
C(21)	2763(2)	3972(2)	574(1)	31(1)
C(22)	1290(2)	3646(2)	12(1)	40(1)
C(23)	1377(3)	3010(2)	-705(1)	47(1)

C(24)	2955(3)	2706(2)	-877(1)	46(1)
C(25)	4434(3)	3057(2)	-347(1)	57(1)
C(26)	4347(3)	3687(2)	372(1)	51(1)
C(27)	3905(2)	5195(2)	3334(1)	25(1)
C(28)	3328(2)	4013(2)	3319(1)	26(1)
C(29)	4120(2)	3184(2)	3778(1)	25(1)
C(30)	3554(2)	1938(2)	3580(1)	31(1)
C(31)	4261(2)	1114(2)	3994(1)	36(1)
C(32)	5548(2)	1527(2)	4620(1)	35(1)
C(33)	6121(2)	2771(2)	4840(1)	31(1)
C(34)	5416(2)	3586(2)	4421(1)	26(1)
C(35)	8370(3)	2249(2)	3052(1)	49(1)
C(36)	6979(3)	1702(2)	2391(1)	49(1)
C(37)	6666(3)	1029(3)	1065(1)	68(1)
C(38)	7627(4)	932(3)	372(1)	100(1)
O(1)	7804(2)	1641(1)	1708(1)	53(1)
H(2)	481	9223	4284	35
H(3)	-631	8348	5293	40
H(4)	-1435	6247	5182	41
H(5)	-1242	5032	4035	35
H(13)	-914	9298	2819	35
H(14)	77	10353	1852	39
H(15)	2649	9970	1358	41
H(16)	4186	8550	1834	36
H(17A)	-2626	5737	2096	49
H(17B)	-2809	4738	2667	49
H(17C)	-2025	4461	1876	49
H(18A)	6130	7184	3487	46
H(18B)	5356	8357	3794	46
H(18C)	6067	8260	2975	46
H(19)	181	4413	1380	33
H(20)	3829	5000	1637	37
H(22)	203	3862	122	48
H(23)	352	2786	-1077	56
H(24)	3012	2253	-1362	56
H(25)	5528	2870	-470	68

H(26)	5388	3929	734	61
H(27)	4992	5538	3646	30
H(28)	2288	3669	2974	32
H(30)	2663	1644	3151	37
H(31)	3860	269	3847	43
H(32)	6043	966	4901	42
H(33)	6994	3059	5275	37
H(34)	5816	4431	4572	32
H(35A)	9244	1717	3108	74
H(35B)	7835	2349	3527	74
H(35C)	8947	3043	2953	74
H(36A)	6433	879	2470	58
H(36B)	6050	2207	2348	58
H(37A)	5676	1468	962	81
H(37B)	6176	207	1165	81
H(38A)	8105	1747	273	150
H(38B)	6810	504	-79	150
H(38C)	8591	483	472	150

Table 3. Bond lengths [Å] and angles [°] for **6b**.

C(1)-C(6)	1.401(2)	C(8)-C(19)	1.468(2)
C(1)-C(2)	1.406(2)	C(8)-C(9)	1.505(2)
C(1)-C(12)	1.498(2)	C(9)-C(10)	1.352(2)
C(2)-C(3)	1.380(2)	C(9)-C(27)	1.462(2)
C(2)-H(2)	0.9500	C(10)-C(11)	1.493(2)
C(3)-C(4)	1.380(3)	C(10)-C(18)	1.512(2)
C(3)-H(3)	0.9500	C(11)-C(16)	1.402(2)
C(4)-C(5)	1.391(2)	C(11)-C(12)	1.414(2)
C(4)-H(4)	0.9500	C(12)-C(13)	1.395(2)
C(5)-C(6)	1.398(2)	C(13)-C(14)	1.384(2)
C(5)-H(5)	0.9500	C(13)-H(13)	0.9500
C(6)-C(7)	1.501(2)	C(14)-C(15)	1.395(2)
C(7)-C(8)	1.357(2)	C(14)-H(14)	0.9500
C(7)-C(17)	1.508(2)	C(15)-C(16)	1.383(2)

C(15)-H(15)	0.9500	C(33)-H(33)	0.9500
C(16)-H(16)	0.9500	C(34)-H(34)	0.9500
C(17)-H(17A)	0.9800	C(35)-C(36)	1.492(3)
C(17)-H(17B)	0.9800	C(35)-H(35A)	0.9800
C(17)-H(17C)	0.9800	C(35)-H(35B)	0.9800
C(18)-H(18A)	0.9800	C(35)-H(35C)	0.9800
C(18)-H(18B)	0.9800	C(36)-O(1)	1.428(2)
C(18)-H(18C)	0.9800	C(36)-H(36A)	0.9900
C(19)-C(20)	1.339(2)	C(36)-H(36B)	0.9900
C(19)-H(19)	0.9500	C(37)-O(1)	1.388(3)
C(20)-C(21)	1.475(2)	C(37)-C(38)	1.504(3)
C(20)-H(20)	0.9500	C(37)-H(37A)	0.9900
C(21)-C(22)	1.392(2)	C(37)-H(37B)	0.9900
C(21)-C(26)	1.394(2)	C(38)-H(38A)	0.9800
C(22)-C(23)	1.391(2)	C(38)-H(38B)	0.9800
C(22)-H(22)	0.9500	C(38)-H(38C)	0.9800
C(23)-C(24)	1.381(3)	C(6)-C(1)-C(2)	118.81(15)
C(23)-H(23)	0.9500	C(6)-C(1)-C(12)	122.01(13)
C(24)-C(25)	1.367(3)	C(2)-C(1)-C(12)	119.10(15)
C(24)-H(24)	0.9500	C(3)-C(2)-C(1)	121.30(16)
C(25)-C(26)	1.389(3)	C(3)-C(2)-H(2)	119.4
C(25)-H(25)	0.9500	C(1)-C(2)-H(2)	119.4
C(26)-H(26)	0.9500	C(4)-C(3)-C(2)	119.88(15)
C(27)-C(28)	1.335(2)	C(4)-C(3)-H(3)	120.1
C(27)-H(27)	0.9500	C(2)-C(3)-H(3)	120.1
C(28)-C(29)	1.468(2)	C(3)-C(4)-C(5)	119.78(16)
C(28)-H(28)	0.9500	C(3)-C(4)-H(4)	120.1
C(29)-C(30)	1.394(2)	C(5)-C(4)-H(4)	120.1
C(29)-C(34)	1.403(2)	C(4)-C(5)-C(6)	121.09(16)
C(30)-C(31)	1.390(2)	C(4)-C(5)-H(5)	119.5
C(30)-H(30)	0.9500	C(6)-C(5)-H(5)	119.5
C(31)-C(32)	1.379(3)	C(5)-C(6)-C(1)	119.10(14)
C(31)-H(31)	0.9500	C(5)-C(6)-C(7)	119.98(15)
C(32)-C(33)	1.396(3)	C(1)-C(6)-C(7)	120.73(14)
C(32)-H(32)	0.9500	C(8)-C(7)-C(6)	120.60(14)
C(33)-C(34)	1.385(2)	C(8)-C(7)-C(17)	125.39(14)

C(6)-C(7)-C(17)	113.93(13)	H(18A)-C(18)-H(18B)	109.5
C(7)-C(8)-C(19)	122.53(15)	C(10)-C(18)-H(18C)	109.5
C(7)-C(8)-C(9)	119.97(14)	H(18A)-C(18)-H(18C)	109.5
C(19)-C(8)-C(9)	117.50(13)	H(18B)-C(18)-H(18C)	109.5
C(10)-C(9)-C(27)	122.92(15)	C(20)-C(19)-C(8)	125.83(16)
C(10)-C(9)-C(8)	119.89(14)	C(20)-C(19)-H(19)	117.1
C(27)-C(9)-C(8)	117.18(14)	C(8)-C(19)-H(19)	117.1
C(9)-C(10)-C(11)	121.22(14)	C(19)-C(20)-C(21)	126.12(16)
C(9)-C(10)-C(18)	124.06(15)	C(19)-C(20)-H(20)	116.9
C(11)-C(10)-C(18)	114.69(14)	C(21)-C(20)-H(20)	116.9
C(16)-C(11)-C(12)	118.44(15)	C(22)-C(21)-C(26)	117.09(16)
C(16)-C(11)-C(10)	119.84(14)	C(22)-C(21)-C(20)	123.16(15)
C(12)-C(11)-C(10)	121.49(14)	C(26)-C(21)-C(20)	119.69(16)
C(13)-C(12)-C(11)	119.17(14)	C(23)-C(22)-C(21)	121.39(17)
C(13)-C(12)-C(1)	118.47(14)	C(23)-C(22)-H(22)	119.3
C(11)-C(12)-C(1)	122.26(14)	C(21)-C(22)-H(22)	119.3
C(14)-C(13)-C(12)	121.77(15)	C(24)-C(23)-C(22)	119.98(18)
C(14)-C(13)-H(13)	119.1	C(24)-C(23)-H(23)	120.0
C(12)-C(13)-H(13)	119.1	C(22)-C(23)-H(23)	120.0
C(13)-C(14)-C(15)	119.10(16)	C(25)-C(24)-C(23)	119.70(17)
C(13)-C(14)-H(14)	120.5	C(25)-C(24)-H(24)	120.2
C(15)-C(14)-H(14)	120.5	C(23)-C(24)-H(24)	120.1
C(16)-C(15)-C(14)	120.12(16)	C(24)-C(25)-C(26)	120.34(19)
C(16)-C(15)-H(15)	119.9	C(24)-C(25)-H(25)	119.8
C(14)-C(15)-H(15)	119.9	C(26)-C(25)-H(25)	119.8
C(15)-C(16)-C(11)	121.39(15)	C(25)-C(26)-C(21)	121.41(19)
C(15)-C(16)-H(16)	119.3	C(25)-C(26)-H(26)	119.3
C(11)-C(16)-H(16)	119.3	C(21)-C(26)-H(26)	119.3
C(7)-C(17)-H(17A)	109.5	C(28)-C(27)-C(9)	125.24(15)
C(7)-C(17)-H(17B)	109.5	C(28)-C(27)-H(27)	117.4
H(17A)-C(17)-H(17B)	109.5	C(9)-C(27)-H(27)	117.4
C(7)-C(17)-H(17C)	109.5	C(27)-C(28)-C(29)	127.23(15)
H(17A)-C(17)-H(17C)	109.5	C(27)-C(28)-H(28)	116.4
H(17B)-C(17)-H(17C)	109.5	C(29)-C(28)-H(28)	116.4
C(10)-C(18)-H(18A)	109.5	C(30)-C(29)-C(34)	117.73(15)
C(10)-C(18)-H(18B)	109.5		

C(30)-C(29)-C(28)	119.23(15)	C(37)-C(38)-H(38B)	109.5
C(34)-C(29)-C(28)	123.04(15)	H(38A)-C(38)-H(38B)	109.5
C(31)-C(30)-C(29)	121.50(16)	C(37)-C(38)-H(38C)	109.5
C(31)-C(30)-H(30)	119.2	H(38A)-C(38)-H(38C)	109.5
C(29)-C(30)-H(30)	119.2	H(38B)-C(38)-H(38C)	109.5
C(32)-C(31)-C(30)	119.91(17)	C(37)-O(1)-C(36)	112.37(17)
C(32)-C(31)-H(31)	120.0		
C(30)-C(31)-H(31)	120.0		
C(31)-C(32)-C(33)	119.88(17)		
C(31)-C(32)-H(32)	120.1		
C(33)-C(32)-H(32)	120.1		
C(34)-C(33)-C(32)	119.86(16)		
C(34)-C(33)-H(33)	120.1		
C(32)-C(33)-H(33)	120.1		
C(33)-C(34)-C(29)	121.11(16)		
C(33)-C(34)-H(34)	119.4		
C(29)-C(34)-H(34)	119.4		
C(36)-C(35)-H(35A)	109.5		
C(36)-C(35)-H(35B)	109.5		
H(35A)-C(35)-H(35B)	109.5		
C(36)-C(35)-H(35C)	109.5		
H(35A)-C(35)-H(35C)	109.5		
H(35B)-C(35)-H(35C)	109.5		
O(1)-C(36)-C(35)	107.35(17)		
O(1)-C(36)-H(36A)	110.2		
C(35)-C(36)-H(36A)	110.2		
O(1)-C(36)-H(36B)	110.2		
C(35)-C(36)-H(36B)	110.2		
H(36A)-C(36)-H(36B)	108.5		
O(1)-C(37)-C(38)	110.1(2)		
O(1)-C(37)-H(37A)	109.6		
C(38)-C(37)-H(37A)	109.6		
O(1)-C(37)-H(37B)	109.6		
C(38)-C(37)-H(37B)	109.6		
H(37A)-C(37)-H(37B)	108.1		
C(37)-C(38)-H(38A)	109.5		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **6b**. 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)	14(1)	30(1)	29(1)	1(1)	4(1)	6(1)
C(2)	21(1)	30(1)	35(1)	-2(1)	5(1)	5(1)
C(3)	27(1)	43(1)	31(1)	-3(1)	10(1)	9(1)
C(4)	24(1)	49(1)	32(1)	6(1)	12(1)	6(1)
C(5)	20(1)	31(1)	35(1)	3(1)	8(1)	3(1)
C(6)	13(1)	31(1)	31(1)	2(1)	5(1)	5(1)
C(7)	18(1)	26(1)	27(1)	1(1)	3(1)	1(1)
C(8)	19(1)	24(1)	28(1)	2(1)	4(1)	4(1)
C(9)	16(1)	28(1)	25(1)	-3(1)	7(1)	5(1)
C(10)	17(1)	30(1)	26(1)	-1(1)	7(1)	4(1)
C(11)	18(1)	24(1)	27(1)	-3(1)	4(1)	1(1)
C(12)	19(1)	23(1)	27(1)	-3(1)	2(1)	1(1)
C(13)	22(1)	31(1)	34(1)	0(1)	3(1)	6(1)
C(14)	34(1)	30(1)	34(1)	3(1)	-1(1)	7(1)
C(15)	38(1)	35(1)	30(1)	5(1)	8(1)	1(1)
C(16)	28(1)	34(1)	30(1)	2(1)	10(1)	5(1)
C(17)	18(1)	40(1)	36(1)	-1(1)	4(1)	2(1)
C(18)	18(1)	32(1)	40(1)	1(1)	4(1)	1(1)
C(19)	22(1)	28(1)	29(1)	-2(1)	2(1)	2(1)
C(20)	24(1)	37(1)	29(1)	-3(1)	3(1)	5(1)
C(21)	29(1)	34(1)	31(1)	-1(1)	7(1)	6(1)
C(22)	30(1)	49(1)	37(1)	-8(1)	4(1)	7(1)
C(23)	41(1)	57(1)	36(1)	-12(1)	2(1)	7(1)
C(24)	53(1)	50(1)	34(1)	-11(1)	8(1)	16(1)
C(25)	45(1)	86(2)	41(1)	-11(1)	11(1)	28(1)
C(26)	32(1)	82(2)	36(1)	-14(1)	2(1)	19(1)
C(27)	17(1)	32(1)	27(1)	-2(1)	6(1)	6(1)

C(28)	18(1)	31(1)	29(1)	-1(1)	5(1)	4(1)
C(29)	19(1)	29(1)	29(1)	0(1)	10(1)	6(1)
C(30)	25(1)	31(1)	36(1)	-1(1)	5(1)	3(1)
C(31)	35(1)	24(1)	47(1)	2(1)	10(1)	3(1)
C(32)	32(1)	35(1)	42(1)	11(1)	9(1)	9(1)
C(33)	25(1)	38(1)	31(1)	3(1)	6(1)	6(1)
C(34)	21(1)	26(1)	31(1)	-3(1)	8(1)	3(1)
C(35)	45(1)	49(1)	54(1)	1(1)	20(1)	9(1)
C(36)	46(1)	51(1)	53(1)	7(1)	26(1)	11(1)
C(37)	66(2)	89(2)	49(1)	13(1)	10(1)	10(2)
C(38)	88(2)	165(3)	42(1)	10(2)	16(1)	5(2)
O(1)	44(1)	74(1)	49(1)	11(1)	16(1)	19(1)

Table 5. Torsion angles [°] for **6b**.

C(6)-C(1)-C(2)-C(3)	1.7(2)
C(12)-C(1)-C(2)-C(3)	178.46(15)
C(1)-C(2)-C(3)-C(4)	0.2(2)
C(2)-C(3)-C(4)-C(5)	-1.6(3)
C(3)-C(4)-C(5)-C(6)	1.2(3)
C(4)-C(5)-C(6)-C(1)	0.7(2)
C(4)-C(5)-C(6)-C(7)	-174.21(15)
C(2)-C(1)-C(6)-C(5)	-2.1(2)
C(12)-C(1)-C(6)-C(5)	-178.78(14)
C(2)-C(1)-C(6)-C(7)	172.78(14)
C(12)-C(1)-C(6)-C(7)	-3.9(2)
C(5)-C(6)-C(7)-C(8)	-117.38(18)
C(1)-C(6)-C(7)-C(8)	67.8(2)
C(5)-C(6)-C(7)-C(17)	65.57(19)
C(1)-C(6)-C(7)-C(17)	-109.29(17)
C(6)-C(7)-C(8)-C(19)	-175.16(15)
C(17)-C(7)-C(8)-C(19)	1.5(3)
C(6)-C(7)-C(8)-C(9)	5.0(2)
C(17)-C(7)-C(8)-C(9)	-178.34(15)

C(7)-C(8)-C(9)-C(10)	-79.2(2)
C(19)-C(8)-C(9)-C(10)	100.96(17)
C(7)-C(8)-C(9)-C(27)	99.41(17)
C(19)-C(8)-C(9)-C(27)	-80.47(18)
C(27)-C(9)-C(10)-C(11)	-169.85(13)
C(8)-C(9)-C(10)-C(11)	8.6(2)
C(27)-C(9)-C(10)-C(18)	8.4(2)
C(8)-C(9)-C(10)-C(18)	-173.12(14)
C(9)-C(10)-C(11)-C(16)	-121.54(17)
C(18)-C(10)-C(11)-C(16)	60.06(19)
C(9)-C(10)-C(11)-C(12)	64.0(2)
C(18)-C(10)-C(11)-C(12)	-114.38(17)
C(16)-C(11)-C(12)-C(13)	-0.4(2)
C(10)-C(11)-C(12)-C(13)	174.10(14)
C(16)-C(11)-C(12)-C(1)	-176.77(15)
C(10)-C(11)-C(12)-C(1)	-2.3(2)
C(6)-C(1)-C(12)-C(13)	120.33(17)
C(2)-C(1)-C(12)-C(13)	-56.3(2)
C(6)-C(1)-C(12)-C(11)	-63.3(2)
C(2)-C(1)-C(12)-C(11)	120.05(17)
C(11)-C(12)-C(13)-C(14)	1.0(2)
C(1)-C(12)-C(13)-C(14)	177.53(15)
C(12)-C(13)-C(14)-C(15)	-0.8(2)
C(13)-C(14)-C(15)-C(16)	-0.1(3)
C(14)-C(15)-C(16)-C(11)	0.7(3)
C(12)-C(11)-C(16)-C(15)	-0.4(2)
C(10)-C(11)-C(16)-C(15)	-175.04(14)
C(7)-C(8)-C(19)-C(20)	177.31(17)
C(9)-C(8)-C(19)-C(20)	-2.8(3)
C(8)-C(19)-C(20)-C(21)	-179.07(16)
C(19)-C(20)-C(21)-C(22)	15.6(3)
C(19)-C(20)-C(21)-C(26)	-167.13(19)
C(26)-C(21)-C(22)-C(23)	3.1(3)
C(20)-C(21)-C(22)-C(23)	-179.60(19)
C(21)-C(22)-C(23)-C(24)	-1.0(3)
C(22)-C(23)-C(24)-C(25)	-1.6(3)

C(23)-C(24)-C(25)-C(26)	1.9(4)
C(24)-C(25)-C(26)-C(21)	0.3(4)
C(22)-C(21)-C(26)-C(25)	-2.8(3)
C(20)-C(21)-C(26)-C(25)	179.8(2)
C(10)-C(9)-C(27)-C(28)	179.99(15)
C(8)-C(9)-C(27)-C(28)	1.5(2)
C(9)-C(27)-C(28)-C(29)	-175.18(14)
C(27)-C(28)-C(29)-C(30)	-167.14(15)
C(27)-C(28)-C(29)-C(34)	13.9(2)
C(34)-C(29)-C(30)-C(31)	-1.1(2)
C(28)-C(29)-C(30)-C(31)	179.98(15)
C(29)-C(30)-C(31)-C(32)	0.4(3)
C(30)-C(31)-C(32)-C(33)	0.7(3)
C(31)-C(32)-C(33)-C(34)	-1.0(2)
C(32)-C(33)-C(34)-C(29)	0.3(2)
C(30)-C(29)-C(34)-C(33)	0.7(2)
C(28)-C(29)-C(34)-C(33)	179.63(14)
C(38)-C(37)-O(1)-C(36)	-176.9(2)
C(35)-C(36)-O(1)-C(37)	173.70(18)

Symmetry transformations used to generate equivalent atoms:

Reference:

1. Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).