Synthesis of polymeric and macrocyclic Lewis acids: influence of backbone on degree of aggregation

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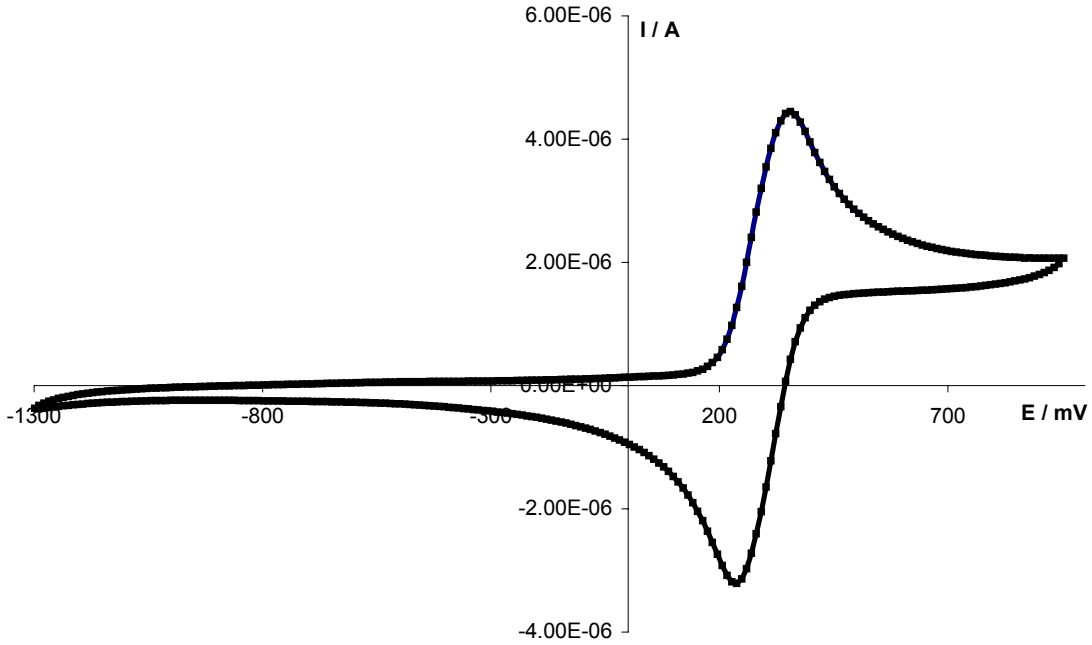
Supporting Information (28 pages)



CV trace for 2 in dichloromethane (referenced with respect to ferrocene/ferrocenium)

2. Crystal structure of [fcB(OCH$_2$)$_2$C(CH$_2$O)$_2$B]$_2$·CHCl$_3$ (2·CHCl$_3$)

Table 1. Crystal data and structure refinement

Empirical formula	C31 H33 B4 Cl3 Fe2 O8	
Formula weight	794.86	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P21/a	
Unit cell dimensions	a = 16.7826(3) Å	α= 90°.
	b = 9.9148(2) Å	β= 108.3390(10)°.
	c = 20.8325(4) Å	γ = 90°.
Volume	3290.39(11) Å^3	
Z	4	
Density (calculated)	1.605 Mg/m^3	
Absorption coefficient	1.176 mm^{-1}	
F(000)	1624	
Crystal size	0.20 x 0.15 x 0.05 mm^3	
Theta range for data collection	3.60 to 26.37°.	
Index ranges	-20<=h<=20, -12<=k<=12, -26<=l<=26	
Reflections collected	46133	
Independent reflections	6702 [R(int) = 0.1368]	
Completeness to theta = 26.37°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9435 and 0.7987	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6702 / 66 / 490	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2sigma(I)]	R1 = 0.0649, wR2 = 0.1297	
R indices (all data)	R1 = 0.0814, wR2 = 0.1358	
Largest diff. peak and hole	0.883 and -0.744 e.Å^{-3}	

Special details

Disorder in the chloroform molecule was modelled and refined based on 3 positions for each chlorine atom and summed to unity. The weightings respectively are Cl1A:Cl1B:Cl1C 0.45:0.35:0.20; Cl2A:Cl2B:Cl2C 0.4:0.3:0.3 and Cl3A:Cl3B:Cl3C 0.20:0.50:0.30. ISOR Restraints were also applied to all parts of the disordered chlorine atoms and the two Fe centres to approximate to isotropic behaviour due to large thermal parameters.

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

	x	y	z	U(eq)
C(1)	4594(2)	981(4)	5727(2)	28(1)
C(2)	4653(2)	-245(4)	6103(2)	29(1)
C(3)	4224(2)	-1269(5)	5654(2)	33(1)
C(4)	3898(3)	-722(4)	5001(2)	32(1)
C(5)	4121(3)	669(5)	5043(2)	33(1)
C(6)	2878(2)	719(4)	6391(2)	29(1)
C(7)	2524(2)	-492(5)	6031(2)	33(1)
C(8)	2160(2)	-155(5)	5337(2)	36(1)
C(9)	2296(3)	1240(5)	5261(2)	35(1)
C(10)	2731(2)	1771(5)	5902(2)	30(1)
C(11)	5892(2)	3647(4)	6942(2)	29(1)
C(12)	5166(3)	4746(4)	5844(2)	30(1)
C(13)	5366(3)	4869(4)	6609(2)	26(1)
C(14)	5855(3)	6180(5)	6835(2)	30(1)
C(15)	4553(2)	4935(4)	6796(2)	27(1)
C(16)	4078(3)	-379(4)	8204(2)	32(1)
C(17)	4038(3)	2110(4)	8167(2)	29(1)
C(18)	4600(3)	884(4)	8418(2)	26(1)
C(19)	5342(2)	905(4)	8142(2)	25(1)
C(20)	4947(3)	873(4)	9188(2)	30(1)
C(21)	5513(2)	6454(4)	8567(2)	25(1)
C(22)	5101(2)	5905(4)	9015(2)	27(1)
C(23)	5443(3)	6519(4)	9658(2)	32(1)
C(24)	6063(3)	7445(4)	9621(2)	30(1)
C(25)	6109(3)	7417(4)	8953(2)	30(1)
C(26)	6654(3)	3539(4)	9388(2)	29(1)
C(27)	7138(3)	4316(4)	9060(2)	30(1)
C(28)	7621(3)	5279(5)	9527(2)	34(1)
C(29)	7450(3)	5108(5)	10144(2)	35(1)
C(30)	6859(3)	4058(4)	10064(2)	31(1)
O(1)	4809(2)	3460(3)	5595(1)	31(1)
O(2)	5476(2)	2411(3)	6667(1)	30(1)
O(3)	3611(2)	2041(3)	7450(1)	30(1)
O(4)	3596(2)	-375(3)	7500(2)	34(1)
O(5)	4722(2)	5214(3)	7503(1)	29(1)
O(6)	5915(2)	6556(3)	7513(1)	33(1)
O(7)	5911(2)	1989(3)	8429(1)	28(1)
O(8)	5548(2)	1909(3)	9454(1)	30(1)
Fe(1)	3415(1)	268(1)	5657(1)	24(1)
Fe(2)	6374(1)	5566(1)	9384(1)	24(1)
B(1)	4975(3)	2363(5)	6012(2)	26(1)
B(2)	3392(3)	811(5)	7156(2)	28(1)
B(3)	5379(3)	6038(5)	7822(2)	26(1)
B(4)	6001(3)	2432(5)	9068(2)	28(1)
C(31)	2775(3)	5196(5)	7491(2)	36(1)
Cl(1A)	2327(3)	5528(5)	6641(2)	61(1)
Cl(2A)	2887(3)	6763(5)	7987(2)	54(1)
Cl(3A)	2683(5)	4891(8)	8291(4)	54(2)
Cl(1B)	2152(4)	5049(6)	6613(3)	60(1)
Cl(2B)	2953(4)	6805(6)	7772(4)	44(1)
Cl(3B)	2251(2)	4242(4)	7954(2)	43(1)
Cl(1C)	1865(6)	4599(10)	6900(5)	74(2)
Cl(2C)	2992(3)	6876(5)	7431(3)	56(1)
Cl(3C)	2124(4)	4117(6)	7745(3)	38(1)

Table 3. Bond lengths [Å] and angles [°].

C(1)-C(5)	1.429(6)
C(1)-C(2)	1.433(6)
C(1)-B(1)	1.550(6)
C(1)-Fe(1)	2.063(4)
C(2)-C(3)	1.414(6)
C(2)-Fe(1)	2.054(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.406(6)
C(3)-Fe(1)	2.042(4)
C(3)-H(3)	0.9500
C(4)-C(5)	1.425(6)
C(4)-Fe(1)	2.047(4)
C(4)-H(4)	0.9500
C(5)-Fe(1)	2.039(4)
C(5)-H(5)	0.9500
C(6)-C(10)	1.424(6)
C(6)-C(7)	1.441(6)
C(6)-B(2)	1.556(6)
C(6)-Fe(1)	2.053(4)
C(7)-C(8)	1.421(6)
C(7)-Fe(1)	2.036(4)
C(7)-H(7)	0.9500
C(8)-C(9)	1.419(7)
C(8)-Fe(1)	2.042(4)
C(8)-H(8)	0.9500
C(9)-C(10)	1.409(6)
C(9)-Fe(1)	2.040(4)
C(9)-H(9)	0.9500
C(10)-Fe(1)	2.042(4)
C(10)-H(10)	0.9500
C(11)-O(2)	1.438(5)
C(11)-C(13)	1.531(6)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-O(1)	1.434(5)
C(12)-C(13)	1.527(5)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-C(14)	1.531(6)
C(13)-C(15)	1.532(5)
C(14)-O(6)	1.434(5)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-O(5)	1.436(5)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-O(4)	1.436(5)
C(16)-C(18)	1.513(6)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-O(3)	1.441(5)
C(17)-C(18)	1.526(6)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-C(20)	1.525(6)
C(18)-C(19)	1.527(5)
C(19)-O(7)	1.437(5)
C(19)-H(19A)	0.9900
C(19)-H(19B)	0.9900
C(20)-O(8)	1.424(5)
C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900
C(21)-C(22)	1.432(6)
C(21)-C(25)	1.432(6)
C(21)-B(3)	1.553(6)

C(21)-Fe(2)	2.052(4)
C(22)-C(23)	1.418(6)
C(22)-Fe(2)	2.057(4)
C(22)-H(22)	0.9500
C(23)-C(24)	1.406(6)
C(23)-Fe(2)	2.053(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.419(6)
C(24)-Fe(2)	2.037(4)
C(24)-H(24)	0.9500
C(25)-Fe(2)	2.030(4)
C(25)-H(25)	0.9500
C(26)-C(30)	1.435(6)
C(26)-C(27)	1.438(6)
C(26)-B(4)	1.547(7)
C(26)-Fe(2)	2.063(4)
C(27)-C(28)	1.420(6)
C(27)-Fe(2)	2.044(4)
C(27)-H(27)	0.9500
C(28)-C(29)	1.414(6)
C(28)-Fe(2)	2.040(4)
C(28)-H(28)	0.9500
C(29)-C(30)	1.410(6)
C(29)-Fe(2)	2.043(4)
C(29)-H(29)	0.9500
C(30)-Fe(2)	2.045(4)
C(30)-H(30)	0.9500
O(1)-B(1)	1.365(6)
O(2)-B(1)	1.360(5)
O(3)-B(2)	1.363(6)
O(4)-B(2)	1.364(6)
O(5)-B(3)	1.365(6)
O(6)-B(3)	1.361(5)
O(7)-B(4)	1.364(5)
O(8)-B(4)	1.370(6)
C(31)-Cl(2B)	1.693(7)
C(31)-Cl(2C)	1.718(7)
C(31)-Cl(1A)	1.725(6)
C(31)-Cl(3C)	1.727(7)
C(31)-Cl(1C)	1.737(10)
C(31)-Cl(3A)	1.748(8)
C(31)-Cl(3B)	1.769(5)
C(31)-Cl(1B)	1.805(7)
C(31)-Cl(2A)	1.843(7)
C(31)-H(31)	1.0000
Cl(2A)-Cl(3A)	2.025(9)
Cl(1C)-Cl(3C)	1.743(11)

C(5)-C(1)-C(2)	106.4(4)
C(5)-C(1)-B(1)	127.3(4)
C(2)-C(1)-B(1)	126.2(4)
C(5)-C(1)-Fe(1)	68.7(2)
C(2)-C(1)-Fe(1)	69.3(2)
B(1)-C(1)-Fe(1)	126.9(3)
C(3)-C(2)-C(1)	108.3(4)
C(3)-C(2)-Fe(1)	69.3(2)
C(1)-C(2)-Fe(1)	70.0(2)
C(3)-C(2)-H(2)	125.9
C(1)-C(2)-H(2)	125.9
Fe(1)-C(2)-H(2)	126.4
C(4)-C(3)-C(2)	109.0(4)
C(4)-C(3)-Fe(1)	70.1(2)
C(2)-C(3)-Fe(1)	70.3(2)
C(4)-C(3)-H(3)	125.5
C(2)-C(3)-H(3)	125.5
Fe(1)-C(3)-H(3)	125.7
C(3)-C(4)-C(5)	107.4(4)

C(3)-C(4)-Fe(1)	69.7(2)
C(5)-C(4)-Fe(1)	69.3(2)
C(3)-C(4)-H(4)	126.3
C(5)-C(4)-H(4)	126.3
Fe(1)-C(4)-H(4)	126.3
C(4)-C(5)-C(1)	108.9(4)
C(4)-C(5)-Fe(1)	69.9(2)
C(1)-C(5)-Fe(1)	70.5(2)
C(4)-C(5)-H(5)	125.6
C(1)-C(5)-H(5)	125.6
Fe(1)-C(5)-H(5)	125.6
C(10)-C(6)-C(7)	106.7(4)
C(10)-C(6)-B(2)	127.2(4)
C(7)-C(6)-B(2)	125.9(4)
C(10)-C(6)-Fe(1)	69.2(2)
C(7)-C(6)-Fe(1)	68.7(2)
B(2)-C(6)-Fe(1)	122.9(3)
C(8)-C(7)-C(6)	108.0(4)
C(8)-C(7)-Fe(1)	69.9(2)
C(6)-C(7)-Fe(1)	70.0(2)
C(8)-C(7)-H(7)	126.0
C(6)-C(7)-H(7)	126.0
Fe(1)-C(7)-H(7)	125.7
C(9)-C(8)-C(7)	108.1(4)
C(9)-C(8)-Fe(1)	69.6(2)
C(7)-C(8)-Fe(1)	69.4(2)
C(9)-C(8)-H(8)	125.9
C(7)-C(8)-H(8)	125.9
Fe(1)-C(8)-H(8)	126.7
C(10)-C(9)-C(8)	108.1(4)
C(10)-C(9)-Fe(1)	69.9(2)
C(8)-C(9)-Fe(1)	69.7(2)
C(10)-C(9)-H(9)	125.9
C(8)-C(9)-H(9)	125.9
Fe(1)-C(9)-H(9)	126.1
C(9)-C(10)-C(6)	109.0(4)
C(9)-C(10)-Fe(1)	69.8(2)
C(6)-C(10)-Fe(1)	70.1(2)
C(9)-C(10)-H(10)	125.5
C(6)-C(10)-H(10)	125.5
Fe(1)-C(10)-H(10)	126.3
O(2)-C(11)-C(13)	110.8(3)
O(2)-C(11)-H(11A)	109.5
C(13)-C(11)-H(11A)	109.5
O(2)-C(11)-H(11B)	109.5
C(13)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	108.1
O(1)-C(12)-C(13)	112.3(3)
O(1)-C(12)-H(12A)	109.2
C(13)-C(12)-H(12A)	109.2
O(1)-C(12)-H(12B)	109.2
C(13)-C(12)-H(12B)	109.2
H(12A)-C(12)-H(12B)	107.9
C(12)-C(13)-C(11)	108.3(3)
C(12)-C(13)-C(14)	108.0(3)
C(11)-C(13)-C(14)	110.8(3)
C(12)-C(13)-C(15)	110.3(3)
C(11)-C(13)-C(15)	110.6(3)
C(14)-C(13)-C(15)	108.7(3)
O(6)-C(14)-C(13)	112.8(3)
O(6)-C(14)-H(14A)	109.0
C(13)-C(14)-H(14A)	109.0
O(6)-C(14)-H(14B)	109.0
C(13)-C(14)-H(14B)	109.0
H(14A)-C(14)-H(14B)	107.8
O(5)-C(15)-C(13)	111.3(3)
O(5)-C(15)-H(15A)	109.4

C(13)-C(15)-H(15A)	109.4
O(5)-C(15)-H(15B)	109.4
C(13)-C(15)-H(15B)	109.4
H(15A)-C(15)-H(15B)	108.0
O(4)-C(16)-C(18)	112.7(3)
O(4)-C(16)-H(16A)	109.0
C(18)-C(16)-H(16A)	109.0
O(4)-C(16)-H(16B)	109.0
C(18)-C(16)-H(16B)	109.0
H(16A)-C(16)-H(16B)	107.8
O(3)-C(17)-C(18)	111.7(3)
O(3)-C(17)-H(17A)	109.3
C(18)-C(17)-H(17A)	109.3
O(3)-C(17)-H(17B)	109.3
C(18)-C(17)-H(17B)	109.3
H(17A)-C(17)-H(17B)	107.9
C(16)-C(18)-C(20)	107.3(3)
C(16)-C(18)-C(17)	108.7(3)
C(20)-C(18)-C(17)	110.9(3)
C(16)-C(18)-C(19)	111.4(3)
C(20)-C(18)-C(19)	108.0(3)
C(17)-C(18)-C(19)	110.4(3)
O(7)-C(19)-C(18)	111.6(3)
O(7)-C(19)-H(19A)	109.3
C(18)-C(19)-H(19A)	109.3
O(7)-C(19)-H(19B)	109.3
C(18)-C(19)-H(19B)	109.3
H(19A)-C(19)-H(19B)	108.0
O(8)-C(20)-C(18)	113.1(3)
O(8)-C(20)-H(20A)	109.0
C(18)-C(20)-H(20A)	109.0
O(8)-C(20)-H(20B)	109.0
C(18)-C(20)-H(20B)	109.0
H(20A)-C(20)-H(20B)	107.8
C(22)-C(21)-C(25)	106.5(4)
C(22)-C(21)-B(3)	127.0(4)
C(25)-C(21)-B(3)	126.4(4)
C(22)-C(21)-Fe(2)	69.8(2)
C(25)-C(21)-Fe(2)	68.7(2)
B(3)-C(21)-Fe(2)	123.9(3)
C(23)-C(22)-C(21)	108.4(4)
C(23)-C(22)-Fe(2)	69.7(2)
C(21)-C(22)-Fe(2)	69.4(2)
C(23)-C(22)-H(22)	125.8
C(21)-C(22)-H(22)	125.8
Fe(2)-C(22)-H(22)	126.7
C(24)-C(23)-C(22)	108.4(4)
C(24)-C(23)-Fe(2)	69.3(2)
C(22)-C(23)-Fe(2)	70.0(2)
C(24)-C(23)-H(23)	125.8
C(22)-C(23)-H(23)	125.8
Fe(2)-C(23)-H(23)	126.5
C(23)-C(24)-C(25)	108.1(4)
C(23)-C(24)-Fe(2)	70.5(2)
C(25)-C(24)-Fe(2)	69.3(2)
C(23)-C(24)-H(24)	126.0
C(25)-C(24)-H(24)	126.0
Fe(2)-C(24)-H(24)	125.8
C(24)-C(25)-C(21)	108.6(4)
C(24)-C(25)-Fe(2)	69.8(2)
C(21)-C(25)-Fe(2)	70.3(2)
C(24)-C(25)-H(25)	125.7
C(21)-C(25)-H(25)	125.7
Fe(2)-C(25)-H(25)	125.8
C(30)-C(26)-C(27)	106.1(4)
C(30)-C(26)-B(4)	126.6(4)
C(27)-C(26)-B(4)	127.2(4)

C(30)-C(26)-Fe(2)	68.9(2)
C(27)-C(26)-Fe(2)	68.8(2)
B(4)-C(26)-Fe(2)	124.1(3)
C(28)-C(27)-C(26)	108.8(4)
C(28)-C(27)-Fe(2)	69.5(2)
C(26)-C(27)-Fe(2)	70.2(2)
C(28)-C(27)-H(27)	125.6
C(26)-C(27)-H(27)	125.6
Fe(2)-C(27)-H(27)	126.3
C(29)-C(28)-C(27)	107.8(4)
C(29)-C(28)-Fe(2)	69.9(3)
C(27)-C(28)-Fe(2)	69.8(2)
C(29)-C(28)-H(28)	126.1
C(27)-C(28)-H(28)	126.1
Fe(2)-C(28)-H(28)	125.8
C(30)-C(29)-C(28)	108.6(4)
C(30)-C(29)-Fe(2)	69.9(2)
C(28)-C(29)-Fe(2)	69.6(2)
C(30)-C(29)-H(29)	125.7
C(28)-C(29)-H(29)	125.7
Fe(2)-C(29)-H(29)	126.4
C(29)-C(30)-C(26)	108.8(4)
C(29)-C(30)-Fe(2)	69.8(2)
C(26)-C(30)-Fe(2)	70.2(2)
C(29)-C(30)-H(30)	125.6
C(26)-C(30)-H(30)	125.6
Fe(2)-C(30)-H(30)	126.0
B(1)-O(1)-C(12)	119.9(3)
B(1)-O(2)-C(11)	119.9(3)
B(2)-O(3)-C(17)	119.0(3)
B(2)-O(4)-C(16)	120.5(3)
B(3)-O(5)-C(15)	118.9(3)
B(3)-O(6)-C(14)	121.1(3)
B(4)-O(7)-C(19)	120.1(3)
B(4)-O(8)-C(20)	120.1(3)
C(7)-Fe(1)-C(5)	162.69(18)
C(7)-Fe(1)-C(9)	68.69(19)
C(5)-Fe(1)-C(9)	107.25(19)
C(7)-Fe(1)-C(10)	68.67(18)
C(5)-Fe(1)-C(10)	119.88(18)
C(9)-Fe(1)-C(10)	40.37(17)
C(7)-Fe(1)-C(3)	107.11(18)
C(5)-Fe(1)-C(3)	67.97(19)
C(9)-Fe(1)-C(3)	151.56(18)
C(10)-Fe(1)-C(3)	166.45(18)
C(7)-Fe(1)-C(8)	40.79(18)
C(5)-Fe(1)-C(8)	125.34(18)
C(9)-Fe(1)-C(8)	40.68(19)
C(10)-Fe(1)-C(8)	68.20(18)
C(3)-Fe(1)-C(8)	117.64(19)
C(7)-Fe(1)-C(4)	124.83(18)
C(5)-Fe(1)-C(4)	40.82(17)
C(9)-Fe(1)-C(4)	117.61(18)
C(10)-Fe(1)-C(4)	152.78(18)
C(3)-Fe(1)-C(4)	40.22(18)
C(8)-Fe(1)-C(4)	105.53(17)
C(7)-Fe(1)-C(6)	41.28(17)
C(5)-Fe(1)-C(6)	154.56(18)
C(9)-Fe(1)-C(6)	68.60(17)
C(10)-Fe(1)-C(6)	40.71(17)
C(3)-Fe(1)-C(6)	127.80(18)
C(8)-Fe(1)-C(6)	68.84(17)
C(4)-Fe(1)-C(6)	163.92(18)
C(7)-Fe(1)-C(2)	119.70(18)
C(5)-Fe(1)-C(2)	68.10(18)
C(9)-Fe(1)-C(2)	166.13(19)
C(10)-Fe(1)-C(2)	129.46(17)

C(3)-Fe(1)-C(2)	40.39(17)
C(8)-Fe(1)-C(2)	152.71(19)
C(4)-Fe(1)-C(2)	68.08(17)
C(6)-Fe(1)-C(2)	109.60(17)
C(7)-Fe(1)-C(1)	154.68(17)
C(5)-Fe(1)-C(1)	40.78(17)
C(9)-Fe(1)-C(1)	127.36(19)
C(10)-Fe(1)-C(1)	109.54(17)
C(3)-Fe(1)-C(1)	68.41(17)
C(8)-Fe(1)-C(1)	163.92(18)
C(4)-Fe(1)-C(1)	68.80(16)
C(6)-Fe(1)-C(1)	120.60(17)
C(2)-Fe(1)-C(1)	40.73(17)
C(25)-Fe(2)-C(24)	40.85(17)
C(25)-Fe(2)-C(28)	105.21(18)
C(24)-Fe(2)-C(28)	114.95(18)
C(25)-Fe(2)-C(29)	124.11(18)
C(24)-Fe(2)-C(29)	104.27(18)
C(28)-Fe(2)-C(29)	40.51(18)
C(25)-Fe(2)-C(27)	118.37(17)
C(24)-Fe(2)-C(27)	150.44(18)
C(28)-Fe(2)-C(27)	40.71(17)
C(29)-Fe(2)-C(27)	68.13(18)
C(25)-Fe(2)-C(30)	162.10(17)
C(24)-Fe(2)-C(30)	125.23(17)
C(28)-Fe(2)-C(30)	68.29(18)
C(29)-Fe(2)-C(30)	40.36(18)
C(27)-Fe(2)-C(30)	68.33(18)
C(25)-Fe(2)-C(21)	41.07(16)
C(24)-Fe(2)-C(21)	68.98(17)
C(28)-Fe(2)-C(21)	127.11(17)
C(29)-Fe(2)-C(21)	163.31(18)
C(27)-Fe(2)-C(21)	109.76(17)
C(30)-Fe(2)-C(21)	155.74(17)
C(25)-Fe(2)-C(23)	68.11(17)
C(24)-Fe(2)-C(23)	40.21(17)
C(28)-Fe(2)-C(23)	149.19(18)
C(29)-Fe(2)-C(23)	116.79(18)
C(27)-Fe(2)-C(23)	168.91(18)
C(30)-Fe(2)-C(23)	108.40(18)
C(21)-Fe(2)-C(23)	68.55(16)
C(25)-Fe(2)-C(22)	68.28(17)
C(24)-Fe(2)-C(22)	68.03(17)
C(28)-Fe(2)-C(22)	167.06(17)
C(29)-Fe(2)-C(22)	152.40(18)
C(27)-Fe(2)-C(22)	131.40(17)
C(30)-Fe(2)-C(22)	121.25(17)
C(21)-Fe(2)-C(22)	40.79(16)
C(23)-Fe(2)-C(22)	40.34(16)
C(25)-Fe(2)-C(26)	154.23(17)
C(24)-Fe(2)-C(26)	164.79(17)
C(28)-Fe(2)-C(26)	68.99(18)
C(29)-Fe(2)-C(26)	68.61(17)
C(27)-Fe(2)-C(26)	41.00(17)
C(30)-Fe(2)-C(26)	40.90(17)
C(21)-Fe(2)-C(26)	121.47(17)
C(23)-Fe(2)-C(26)	129.72(18)
C(22)-Fe(2)-C(26)	111.67(17)
O(2)-B(1)-O(1)	123.3(4)
O(2)-B(1)-C(1)	117.8(4)
O(1)-B(1)-C(1)	118.9(4)
O(3)-B(2)-O(4)	123.2(4)
O(3)-B(2)-C(6)	119.8(4)
O(4)-B(2)-C(6)	117.0(4)
O(6)-B(3)-O(5)	123.2(4)
O(6)-B(3)-C(21)	117.6(4)
O(5)-B(3)-C(21)	119.1(4)

O(7)-B(4)-O(8)	122.6(4)
O(7)-B(4)-C(26)	119.7(4)
O(8)-B(4)-C(26)	117.7(4)
Cl(2B)-C(31)-Cl(2C)	25.0(2)
Cl(2B)-C(31)-Cl(1A)	98.5(4)
Cl(2C)-C(31)-Cl(1A)	76.9(3)
Cl(2B)-C(31)-Cl(3C)	122.5(4)
Cl(2C)-C(31)-Cl(3C)	142.4(4)
Cl(1A)-C(31)-Cl(3C)	108.6(3)
Cl(2B)-C(31)-Cl(1C)	125.9(5)
Cl(2C)-C(31)-Cl(1C)	115.9(5)
Cl(1A)-C(31)-Cl(1C)	48.3(4)
Cl(3C)-C(31)-Cl(1C)	60.4(4)
Cl(2B)-C(31)-Cl(3A)	83.8(4)
Cl(2C)-C(31)-Cl(3A)	108.8(4)
Cl(1A)-C(31)-Cl(3A)	150.6(4)
Cl(3C)-C(31)-Cl(3A)	49.2(3)
Cl(1C)-C(31)-Cl(3A)	107.2(5)
Cl(2B)-C(31)-Cl(3B)	112.3(4)
Cl(2C)-C(31)-Cl(3B)	135.3(4)
Cl(1A)-C(31)-Cl(3B)	122.0(3)
Cl(3C)-C(31)-Cl(3B)	14.3(2)
Cl(1C)-C(31)-Cl(3B)	74.1(4)
Cl(3A)-C(31)-Cl(3B)	34.9(3)
Cl(2B)-C(31)-Cl(1B)	114.1(4)
Cl(2C)-C(31)-Cl(1B)	94.1(4)
Cl(1A)-C(31)-Cl(1B)	17.8(2)
Cl(3C)-C(31)-Cl(1B)	92.3(4)
Cl(1C)-C(31)-Cl(1B)	32.2(3)
Cl(3A)-C(31)-Cl(1B)	139.1(4)
Cl(3B)-C(31)-Cl(1B)	106.3(3)
Cl(2B)-C(31)-Cl(2A)	15.4(2)
Cl(2C)-C(31)-Cl(2A)	40.2(3)
Cl(1A)-C(31)-Cl(2A)	110.4(3)
Cl(3C)-C(31)-Cl(2A)	108.3(3)
Cl(1C)-C(31)-Cl(2A)	126.6(4)
Cl(3A)-C(31)-Cl(2A)	68.6(3)
Cl(3B)-C(31)-Cl(2A)	97.3(3)
Cl(1B)-C(31)-Cl(2A)	124.1(3)
Cl(2B)-C(31)-H(31)	98.9
Cl(2C)-C(31)-H(31)	92.1
Cl(1A)-C(31)-H(31)	104.3
Cl(3C)-C(31)-H(31)	120.7
Cl(1C)-C(31)-H(31)	126.9
Cl(3A)-C(31)-H(31)	104.3
Cl(3B)-C(31)-H(31)	117.1
Cl(1B)-C(31)-H(31)	108.4
Cl(2A)-C(31)-H(31)	104.3
C(31)-Cl(2A)-Cl(3A)	53.5(3)
C(31)-Cl(3A)-Cl(2A)	57.9(3)
C(31)-Cl(1C)-Cl(3C)	59.5(4)
C(31)-Cl(3C)-Cl(1C)	60.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$).
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)	22(2)	31(2)	33(2)	-4(2)	14(2)	-3(2)
C(2)	18(2)	31(2)	38(2)	-1(2)	9(2)	6(2)
C(3)	22(2)	29(2)	51(3)	-5(2)	14(2)	0(2)
C(4)	24(2)	34(2)	41(3)	-15(2)	15(2)	-6(2)
C(5)	30(2)	39(3)	35(2)	-3(2)	19(2)	-5(2)
C(6)	22(2)	33(2)	34(2)	-1(2)	13(2)	3(2)
C(7)	21(2)	36(3)	45(3)	-2(2)	17(2)	-7(2)
C(8)	12(2)	55(3)	38(3)	-11(2)	6(2)	-1(2)
C(9)	26(2)	44(3)	35(2)	1(2)	7(2)	13(2)
C(10)	23(2)	32(2)	36(2)	1(2)	9(2)	9(2)
C(11)	24(2)	32(2)	28(2)	-4(2)	7(2)	-5(2)
C(12)	37(2)	27(2)	27(2)	4(2)	14(2)	-4(2)
C(13)	29(2)	25(2)	26(2)	0(2)	11(2)	-5(2)
C(14)	30(2)	36(2)	29(2)	0(2)	15(2)	-7(2)
C(15)	25(2)	32(2)	23(2)	-1(2)	7(2)	-1(2)
C(16)	37(2)	28(2)	30(2)	4(2)	12(2)	-2(2)
C(17)	34(2)	26(2)	28(2)	-2(2)	9(2)	3(2)
C(18)	31(2)	21(2)	25(2)	5(2)	11(2)	4(2)
C(19)	30(2)	19(2)	26(2)	0(2)	9(2)	3(2)
C(20)	40(2)	24(2)	30(2)	3(2)	15(2)	4(2)
C(21)	24(2)	21(2)	32(2)	3(2)	9(2)	6(2)
C(22)	24(2)	25(2)	34(2)	-4(2)	12(2)	2(2)
C(23)	36(2)	31(2)	32(2)	-2(2)	16(2)	6(2)
C(24)	35(2)	20(2)	34(2)	-8(2)	10(2)	3(2)
C(25)	34(2)	18(2)	38(2)	0(2)	12(2)	2(2)
C(26)	28(2)	21(2)	35(2)	2(2)	7(2)	8(2)
C(27)	27(2)	27(2)	36(2)	-2(2)	11(2)	7(2)
C(28)	26(2)	31(2)	43(3)	-4(2)	5(2)	1(2)
C(29)	33(2)	32(3)	31(2)	-3(2)	-4(2)	4(2)
C(30)	32(2)	28(2)	28(2)	2(2)	2(2)	5(2)
O(1)	36(2)	32(2)	25(2)	-2(1)	12(1)	-7(1)
O(2)	30(2)	26(2)	31(2)	1(1)	6(1)	-2(1)
O(3)	33(2)	24(2)	31(2)	0(1)	7(1)	4(1)
O(4)	42(2)	27(2)	31(2)	0(1)	8(1)	-8(1)
O(5)	28(1)	34(2)	28(2)	-4(1)	14(1)	-5(1)
O(6)	39(2)	36(2)	28(2)	-7(1)	14(1)	-13(1)
O(7)	30(2)	23(2)	31(2)	-3(1)	11(1)	-2(1)
O(8)	37(2)	30(2)	23(2)	-4(1)	10(1)	1(1)
Fe(1)	19(1)	25(1)	27(1)	-1(1)	9(1)	1(1)
Fe(2)	25(1)	21(1)	25(1)	-3(1)	7(1)	1(1)
B(1)	19(2)	34(3)	30(3)	-3(2)	14(2)	0(2)
B(2)	23(2)	31(3)	34(3)	0(2)	15(2)	-3(2)
B(3)	22(2)	22(2)	33(3)	4(2)	10(2)	3(2)
B(4)	27(2)	21(2)	32(3)	2(2)	6(2)	10(2)
C(31)	28(2)	33(3)	47(3)	7(2)	13(2)	1(2)
Cl(1A)	62(1)	62(1)	57(1)	4(1)	15(1)	-2(1)
Cl(2A)	57(2)	52(2)	54(2)	-2(1)	19(1)	-6(1)
Cl(3A)	55(2)	56(2)	54(2)	0(1)	20(1)	2(1)
Cl(1B)	62(2)	60(2)	56(2)	2(1)	15(1)	-2(1)
Cl(2B)	47(2)	40(2)	44(2)	-2(1)	14(1)	-4(1)
Cl(3B)	42(1)	48(1)	42(1)	3(1)	20(1)	-1(1)
Cl(1C)	73(2)	74(2)	75(2)	-2(1)	22(1)	-1(1)
Cl(2C)	56(2)	53(2)	59(2)	1(1)	16(1)	-1(1)
Cl(3C)	36(2)	39(2)	40(2)	-1(1)	13(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3).

	x	y	z	U(eq)
H(2)	4932	-352	6573	35
H(3)	4165	-2179	5774	40
H(4)	3586	-1194	4604	38
H(5)	3979	1288	4676	39
H(7)	2532	-1362	6224	39
H(8)	1876	-759	4986	43
H(9)	2123	1731	4848	42
H(10)	2900	2685	5994	36
H(11A)	6445	3687	6866	34
H(11B)	5989	3671	7435	34
H(12A)	4767	5467	5620	35
H(12B)	5688	4877	5726	35
H(14A)	6428	6071	6803	36
H(14B)	5576	6915	6523	36
H(15A)	4185	5649	6526	32
H(15B)	4252	4064	6684	32
H(16A)	4454	-1174	8300	38
H(16B)	3692	-467	8476	38
H(17A)	3617	2163	8410	35
H(17B)	4383	2939	8270	35
H(19A)	5645	36	8246	30
H(19B)	5131	1008	7644	30
H(20A)	4477	979	9376	36
H(20B)	5213	-13	9339	36
H(22)	4671	5241	8900	33
H(23)	5282	6336	10047	38
H(24)	6392	7993	9981	35
H(25)	6474	7949	8788	35
H(27)	7135	4204	8607	36
H(28)	7994	5923	9439	41
H(29)	7691	5615	10545	42
H(30)	6634	3746	10402	37
H(31)	3365	4929	7537	43

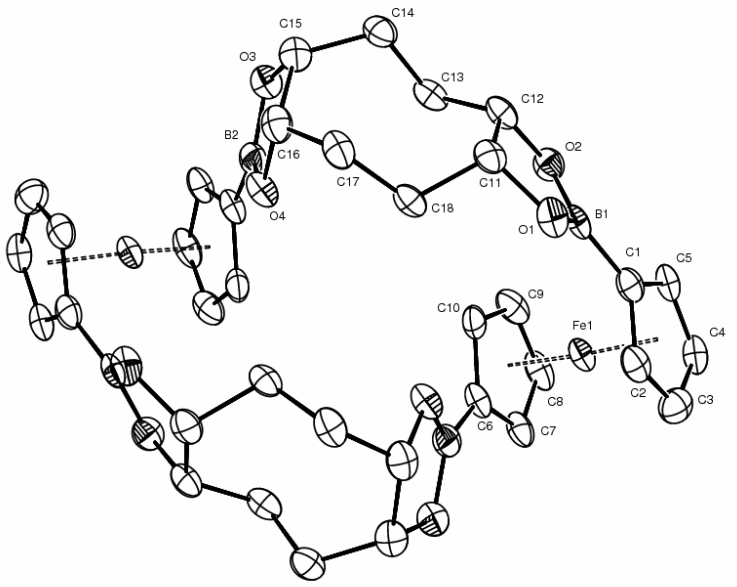
3. Crystal structure of $[fcBO_2C_8H_{12}O_2B]_2$ (4)

Table 1. Crystal data and structure refinement details.

Empirical formula	$C_{36}H_{40}B_4Fe_2O_8$	
Formula weight	755.62	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pbcn*	
Unit cell dimensions	a = 19.6222(11) Å	α = 90°
	b = 7.8609(3) Å	β = 90°
	c = 21.3982(11) Å	γ = 90°
Volume	3300.6(3) Å^3	
Z	4	
Density (calculated)	1.521 Mg / m^3	
Absorption coefficient	0.934 mm^{-1}	
F(000)	1568	
Crystal	Plate; Brown	
Crystal size	0.19 × 0.04 × 0.01 mm^3	
θ range for data collection	2.95 – 26.37°	
Index ranges	$-24 \le h \le 13$, $-8 \le k \le 9$, $-26 \le l \le 26$	
Reflections collected	25134	
Independent reflections	3366 [R_{int} = 0.1290]	
Completeness to θ = 26.37°	99.5 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	0.9907 and 0.8425	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3366 / 0 / 226	
Goodness-of-fit on F^2	1.219	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0905, *wR2* = 0.1459	
R indices (all data)	*R1* = 0.1451, *wR2* = 0.1676	
Largest diff. peak and hole	0.546 and –0.476 e Å^{-3}	

Special details:

Hydrogen atoms were fixed as riding models.
The molecule lies on a two-fold crystallographic rotation axis.



Structure of 4: ORTEP ellipsoids at the 50 % probability level

Table 2. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
C1	5953(3)	2405(8)	4634(3)	30(2)	1
C2	6610(3)	1926(8)	4393(3)	31(2)	1
C3	7095(4)	3159(8)	4566(3)	39(2)	1
C4	6756(4)	4399(8)	4928(3)	34(2)	1
C5	6055(4)	3954(8)	4966(3)	32(2)	1
C6	6293(3)	4174(8)	3101(2)	30(1)	1
C7	6692(4)	5590(8)	3318(3)	34(2)	1
C8	6256(4)	6638(8)	3688(3)	37(2)	1
C9	5609(4)	5916(8)	3708(3)	38(2)	1
C10	5621(3)	4419(8)	3350(3)	33(2)	1
C11	4505(3)	–236(7)	4009(3)	30(1)	1
C12	4122(3)	1345(8)	4264(3)	30(2)	1
C13	3660(3)	2235(8)	3813(3)	32(1)	1
C14	3024(3)	1176(8)	3651(3)	33(2)	1
C15	2919(3)	850(8)	2955(3)	31(1)	1
C16	3476(3)	–168(8)	2604(3)	33(2)	1
C17	3968(4)	–1314(8)	2968(3)	34(2)	1
C18	4567(3)	–464(7)	3298(3)	27(1)	1
O1	5194(2)	–51(5)	4253(2)	34(1)	1
O2	4665(2)	2486(5)	4452(2)	32(1)	1
O3	7091(2)	2472(5)	2372(2)	33(1)	1
O4	6143(2)	1106(5)	2743(2)	33(1)	1
Fe1	6316(1)	4248(1)	4058(1)	28(1)	1
B1	5271(4)	1592(9)	4453(3)	29(2)	1
B2	6514(4)	2561(9)	2740(3)	30(2)	1

Table 3. Bond lengths [Å] and angles [°].

C1–C5	1.424(8)
C1–C2	1.439(9)
C1–B1	1.533(10)
C1–Fe1	2.031(6)
C2–C3	1.408(9)
C2–Fe1	2.044(6)
C2–H2	0.9500
C3–C4	1.412(9)
C3–Fe1	2.063(7)
C3–H3	0.9500
C4–C5	1.421(9)
C4–Fe1	2.056(6)
C4–H4	0.9500
C5–Fe1	2.023(6)
C5–H5	0.9500
C6–C10	1.434(9)
C6–C7	1.438(9)
C6–B2	1.547(9)
C6–Fe1	2.049(5)
C7–C8	1.427(9)
C7–Fe1	2.042(6)
C7–H7	0.9500
C8–C9	1.391(9)
C8–Fe1	2.043(6)
C8–H8	0.9500
C9–C10	1.404(9)
C9–Fe1	2.050(7)
C9–H9	0.9500
C10–Fe1	2.043(6)
C10–H10	0.9500
C11–O1	1.457(7)
C11–C18	1.538(7)
C11–C12	1.551(8)
C11–H11	1.0000
C12–O2	1.450(7)
C12–C13	1.498(8)
C12–H12	1.0000
C13–C14	1.539(9)
C13–H13A	0.9900
C13–H13B	0.9900
C14–C15	1.525(8)
C14–H14A	0.9900
C14–H14B	0.9900
C15–O3^{i}	1.455(7)
C15–C16	1.550(8)
C15–H15	1.0000
C16–O4^{i}	1.454(7)
C16–C17	1.532(9)
C16–H16	1.0000
C17–C18	1.526(8)
C17–H17A	0.9900
C17–H17B	0.9900
C18–H18A	0.9900
C18–H18B	0.9900
O1–B1	1.369(8)
O2–B1	1.382(9)
O3–B2	1.380(8)
O3–C15^{i}	1.455(7)
O4–B2	1.356(8)
O4–C16^{i}	1.454(7)

C5–C1–C2	106.1(6)
C5–C1–B1	127.3(6)
C2–C1–B1	125.6(6)
C5–C1–Fe1	69.1(3)
C2–C1–Fe1	69.8(3)
B1–C1–Fe1	116.7(4)
C3–C2–C1	109.3(6)
C3–C2–Fe1	70.7(4)
C1–C2–Fe1	68.8(3)
C3–C2–H2	125.3
C1–C2–H2	125.3
Fe1–C2–H2	126.8
C2–C3–C4	107.5(6)
C2–C3–Fe1	69.2(4)
C4–C3–Fe1	69.7(4)
C2–C3–H3	126.3
C4–C3–H3	126.3
Fe1–C3–H3	126.4
C3–C4–C5	108.5(6)
C3–C4–Fe1	70.2(4)
C5–C4–Fe1	68.4(3)
C3–C4–H4	125.7
C5–C4–H4	125.7
Fe1–C4–H4	127.3
C4–C5–C1	108.5(6)
C4–C5–Fe1	70.9(4)
C1–C5–Fe1	69.7(3)
C4–C5–H5	125.7
C1–C5–H5	125.7
Fe1–C5–H5	125.3
C10–C6–C7	106.1(5)
C10–C6–B2	123.6(6)
C7–C6–B2	129.9(6)
C10–C6–Fe1	69.3(3)
C7–C6–Fe1	69.1(3)
B2–C6–Fe1	121.0(4)
C8–C7–C6	107.3(6)
C8–C7–Fe1	69.6(3)
C6–C7–Fe1	69.7(3)
C8–C7–H7	126.3
C6–C7–H7	126.3
Fe1–C7–H7	126.0
C9–C8–C7	109.2(6)
C9–C8–Fe1	70.4(4)
C7–C8–Fe1	69.5(3)
C9–C8–H8	125.4
C7–C8–H8	125.4
Fe1–C8–H8	126.3
C8–C9–C10	108.1(6)
C8–C9–Fe1	69.8(4)
C10–C9–Fe1	69.7(4)
C8–C9–H9	126.0
C10–C9–H9	126.0
Fe1–C9–H9	126.1
C9–C10–C6	109.2(6)
C9–C10–Fe1	70.2(4)
C6–C10–Fe1	69.7(3)
C9–C10–H10	125.4
C6–C10–H10	125.4
Fe1–C10–H10	126.3
O1–C11–C18	107.0(5)
O1–C11–C12	104.1(5)
C18–C11–C12	118.7(5)

O1–C11–H11	108.9
C18–C11–H11	108.9
C12–C11–H11	108.9
O2–C12–C13	109.6(5)
O2–C12–C11	103.8(5)
C13–C12–C11	116.2(5)
O2–C12–H12	109.0
C13–C12–H12	109.0
C11–C12–H12	109.0
C12–C13–C14	112.5(5)
C12–C13–H13A	109.1
C14–C13–H13A	109.1
C12–C13–H13B	109.1
C14–C13–H13B	109.1
H13A–C13–H13B	107.8
C15–C14–C13	114.9(5)
C15–C14–H14A	108.5
C13–C14–H14A	108.5
C15–C14–H14B	108.5
C13–C14–H14B	108.5
H14A–C14–H14B	107.5
$O3^{i}$–C15–C14	109.0(5)
$O3^{i}$–C15–C16	103.1(4)
C14–C15–C16	117.7(5)
$O3^{i}$–C15–H15	108.9
C14–C15–H15	108.9
C16–C15–H15	108.9
$O4^{i}$–C16–C17	109.9(5)
$O4^{i}$–C16–C15	104.8(4)
C17–C16–C15	120.1(5)
$O4^{i}$–C16–H16	107.1
C17–C16–H16	107.1
C15–C16–H16	107.1
C18–C17–C16	117.6(5)
C18–C17–H17A	107.9
C16–C17–H17A	107.9
C18–C17–H17B	107.9
C16–C17–H17B	107.9
H17A–C17–H17B	107.2
C17–C18–C11	116.6(5)
C17–C18–H18A	108.1
C11–C18–H18A	108.1
C17–C18–H18B	108.1
C11–C18–H18B	108.1
H18A–C18–H18B	107.3
B1–O1–C11	108.0(5)
B1–O2–C12	108.6(5)
B2–O3–$C15^{i}$	108.0(5)
B2–O4–$C16^{i}$	107.6(5)
C5–Fe1–C1	41.1(2)
C5–Fe1–C7	153.6(2)
C1–Fe1–C7	165.1(2)
C5–Fe1–C8	117.6(2)
C1–Fe1–C8	150.7(3)
C7–Fe1–C8	40.9(2)
C5–Fe1–C10	123.5(3)
C1–Fe1–C10	105.3(3)
C7–Fe1–C10	68.4(3)
C8–Fe1–C10	67.2(3)
C5–Fe1–C2	68.4(2)
C1–Fe1–C2	41.3(2)
C7–Fe1–C2	129.1(3)
C8–Fe1–C2	166.8(3)

C10–Fe1–C2	120.5(3)
C5–Fe1–C6	162.1(3)
C1–Fe1–C6	125.4(3)
C7–Fe1–C6	41.2(2)
C8–Fe1–C6	68.7(2)
C10–Fe1–C6	41.0(2)
C2–Fe1–C6	109.4(2)
C5–Fe1–C9	104.7(3)
C1–Fe1–C9	116.2(3)
C7–Fe1–C9	68.3(3)
C8–Fe1–C9	39.7(3)
C10–Fe1–C9	40.1(3)
C2–Fe1–C9	152.9(3)
C6–Fe1–C9	68.8(3)
C5–Fe1–C4	40.8(2)
C1–Fe1–C4	68.8(3)
C7–Fe1–C4	121.4(3)
C8–Fe1–C4	108.8(3)
C10–Fe1–C4	161.5(3)
C2–Fe1–C4	67.4(2)
C6–Fe1–C4	156.3(3)
C9–Fe1–C4	125.3(3)
C5–Fe1–C3	68.5(3)
C1–Fe1–C3	69.1(3)
C7–Fe1–C3	110.8(3)
C8–Fe1–C3	129.0(3)
C10–Fe1–C3	155.9(3)
C2–Fe1–C3	40.1(3)
C6–Fe1–C3	122.1(3)
C9–Fe1–C3	163.7(3)
C4–Fe1–C3	40.1(3)
O1–B1–O2	112.6(6)
O1–B1–C1	124.7(6)
O2–B1–C1	122.7(6)
O4–B2–O3	113.6(6)
O4–B2–C6	122.5(6)
O3–B2–C6	123.9(6)

Symmetry transformations used to generate equivalent atoms:
(i) –x+1,y,–z+1/2

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	41(4)	28(3)	23(3)	9(3)	−1(3)	2(3)
C2	46(4)	24(3)	24(3)	4(3)	0(3)	12(3)
C3	37(4)	36(4)	43(4)	8(3)	−4(3)	2(3)
C4	49(4)	31(4)	23(3)	2(3)	−9(3)	−2(3)
C5	47(4)	31(4)	18(3)	1(3)	−4(3)	2(3)
C6	46(4)	27(3)	17(2)	2(2)	1(3)	1(3)
C7	49(4)	31(3)	21(3)	6(3)	1(3)	−7(3)
C8	67(5)	20(3)	23(3)	3(2)	1(3)	−3(3)
C9	54(5)	31(4)	29(3)	10(3)	9(3)	15(4)
C10	37(4)	40(4)	20(3)	5(3)	−6(3)	0(3)
C11	38(4)	24(3)	27(3)	7(3)	2(3)	−5(3)
C12	42(4)	26(3)	22(3)	5(2)	7(3)	2(3)
C13	41(4)	28(3)	27(3)	0(3)	10(3)	2(3)
C14	37(4)	32(4)	30(3)	2(3)	6(3)	−5(3)
C15	38(4)	23(3)	32(3)	3(3)	−2(3)	−4(3)
C16	43(4)	25(3)	29(3)	−2(3)	−4(3)	−5(3)
C17	47(4)	30(3)	25(3)	−1(3)	1(3)	−4(3)
C18	37(4)	21(3)	23(3)	−1(2)	7(3)	9(3)
O1	47(3)	26(2)	27(2)	−2(2)	−3(2)	7(2)
O2	37(3)	29(2)	29(2)	−5(2)	1(2)	−1(2)
O3	38(3)	32(2)	30(2)	0(2)	1(2)	−2(2)
O4	45(3)	30(2)	22(2)	−1(2)	5(2)	−2(2)
Fe1	42(1)	23(1)	18(1)	1(1)	2(1)	4(1)
B1	41(5)	31(4)	14(3)	5(3)	1(3)	3(4)
B2	34(4)	31(4)	24(3)	8(3)	−1(3)	0(3)

Table 5. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
H2	6702	933	4155	38	1
H3	7565	3157	4459	46	1
H4	6962	5368	5115	41	1
H5	5713	4584	5178	38	1
H7	7160	5791	3231	40	1
H8	6386	7666	3888	44	1
H9	5226	6358	3926	46	1
H10	5243	3684	3283	39	1
H11	4295	–1284	4192	36	1
H12	3854	1008	4642	36	1
H13A	3915	2484	3425	39	1
H13B	3514	3334	3996	39	1
H14A	3058	66	3868	40	1
H14B	2618	1769	3816	40	1
H15	2470	272	2894	37	1
H16	3239	–903	2290	39	1
H17A	3701	–1939	3286	41	1
H17B	4154	–2169	2673	41	1
H18A	4637	671	3108	32	1
H18B	4982	–1144	3212	32	1

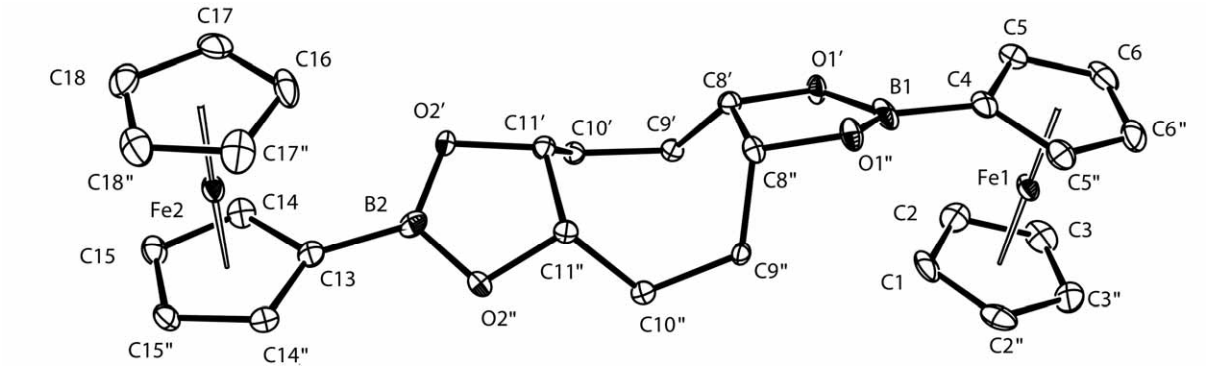
4. Crystal structure of $FcBO_2C_8H_{12}O_2BFc$ (5)

Table 1. Crystal data and structure refinement.

Empirical formula	$C_{28}H_{30}B_2Fe_2O_4$	
Formula weight	563.84	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pnma*	
Unit cell dimensions	a = 29.8942(10) Å	α = 90°
	b = 10.7482(3) Å	β = 90°
	c = 7.4301(3) Å	γ = 90°
Volume	2387.36(14) Å^3	
Z	4	
Density (calculated)	1.569 Mg / m^3	
Absorption coefficient	1.251 mm^{-1}	
F(000)	1168	
Crystal	Plate; pale yellow	
Crystal size	0.14 × 0.10 × 0.02 mm^3	
θ range for data collection	2.33 – 27.50°	
Index ranges	$-38 \le h \le 33$, $-13 \le k \le 13$, $-9 \le l \le 9$	
Reflections collected	20380	
Independent reflections	2868 [R_{int} = 0.1209]	
Completeness to θ = 27.50°	99.5 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	0.9754 and 0.8444	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2868 / 36 / 229	
Goodness-of-fit on F^2	1.138	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0724, *wR2* = 0.1183	
R indices (all data)	*R1* = 0.1096, *wR2* = 0.1277	
Largest diff. peak and hole	0.573 and –0.624 e Å^{-3}	

Special details:

The molecular structure of **5** lies about a crystallographic mirror plane and contains some disorder in the central region (8 membered ring and both 5-membered BO_2C_2 rings). These fused rings occupy two distinct orientations in a 50:50 ratio. A number of restraints in the refinement were applied to maintain a sensible geometry and thermal motion for these orientations.



Structure of 5: ORTEP ellipsoids at the 50 % probability level; atoms with the " character in the atom label are at equivalent position (x, ½-y, z).

Table 2. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
C1	2894(2)	2500	7744(6)	28(1)	1
C2	2674(1)	1430(4)	8439(5)	28(1)	1
C3	2321(1)	1845(4)	9548(5)	31(1)	1
C4	3579(2)	2500	11228(6)	21(1)	1
C5	3354(1)	1432(4)	11943(4)	23(1)	1
C6	3002(1)	1848(4)	13065(4)	28(1)	1
C13	5521(2)	2500	−129(6)	19(1)	1
C14	5695(1)	1433(4)	−1052(5)	21(1)	1
C15	5963(1)	1839(4)	−2520(4)	21(1)	1
C16	6388(2)	2500	2514(7)	33(2)	1
C17	6562(1)	1436(5)	1626(5)	32(1)	1
C18	6842(1)	1839(4)	218(5)	31(1)	1
Fe1	2919(1)	2500	10496(1)	19(1)	1
Fe2	6206(1)	2500	−126(1)	17(1)	1
B1	3936(2)	2500	9728(7)	28(2)	1
B2	5270(2)	2500	1695(8)	19(1)	1
O1	4228(2)	1713(5)	9333(6)	21(1)	0.50
C8	4441(3)	2016(8)	7646(10)	15(2)	0.50
C9	4247(3)	1105(7)	6273(10)	14(2)	0.50
C10	4413(3)	1174(8)	4302(10)	16(2)	0.50
C11	4890(3)	1643(9)	4080(11)	15(2)	0.50
O2	5031(2)	1428(5)	2223(6)	20(1)	0.50
O1'	3972(2)	1337(5)	8623(6)	19(1)	0.50
C8'	4340(3)	1589(9)	7354(12)	15(2)	0.50
C9'	4189(3)	1161(8)	5517(11)	15(2)	0.50
C10'	4563(3)	1102(8)	4107(10)	14(2)	0.50
C11'	4965(3)	1944(8)	4338(11)	14(2)	0.50
O2'	5278(2)	1604(5)	2927(6)	18(1)	0.50

Table 3. Bond lengths [Å] and angles [°].

C1–C2	1.421(5)
C1–C2^{i}	1.421(5)
C1–Fe1	2.046(5)
C2–C3	1.413(5)
C2–Fe1	2.048(4)
C3–C3^{i}	1.408(9)
C3–Fe1	2.048(4)
C4–C5	1.431(5)
C4–C5^{i}	1.431(5)
C4–B1	1.543(8)
C4–Fe1	2.046(5)
C5–C6	1.417(5)
C5–Fe1	2.042(4)
C6–C6^{i}	1.402(9)
C6–Fe1	2.049(3)
C13–C14	1.433(5)
C13–C14^{i}	1.433(5)
C13–B2	1.551(8)
C13–Fe2	2.046(5)
C14–C15	1.423(5)
C14–Fe2	2.031(4)
C15–C15^{i}	1.421(8)
C15–Fe2	2.048(3)
C16–C17^{i}	1.419(6)
C16–C17	1.419(6)
C16–Fe2	2.035(5)
C17–C18	1.409(5)
C17–Fe2	2.033(4)
C18–C18^{i}	1.421(9)
C18–Fe2	2.047(4)
Fe1–C5^{i}	2.042(4)
Fe1–C2^{i}	2.048(4)
Fe1–C6^{i}	2.049(3)
Fe1–C3^{i}	2.048(4)
Fe2–C14^{i}	2.031(4)
Fe2–C17^{i}	2.033(4)
Fe2–C18^{i}	2.047(4)
Fe2–C15^{i}	2.048(3)
B1–O1^{i}	1.251(7)
B1–O1	1.251(7)
B1–O1'	1.500(6)
B1–O1'^{i}	1.500(6)
B2–O2'^{i}	1.329(6)
B2–O2'	1.329(6)
B2–O2	1.411(6)
B2–O2^{i}	1.411(6)
O1–C8	1.443(9)
C8–C8'^{i}	1.546(13)
C8–C9	1.528(11)
C9–C10	1.548(10)
C10–C11	1.522(12)
C11–O2	1.460(9)
C11–C11'^{i}	1.547(12)
O1'–C8'	1.473(10)
C8'–C9'	1.509(11)
C8'–C8^{i}	1.546(13)
C9'–C10'	1.534(10)
C10'–C11'	1.513(12)
C11'–O2'	1.452(9)
C11'–C11^{i}	1.547(12)

C2–C1–C2^{i}	108.0(5)
C2–C1–Fe1	69.8(2)
C2^{i}–C1–Fe1	69.8(2)
C3–C2–C1	107.6(4)
C3–C2–Fe1	69.8(2)
C1–C2–Fe1	69.6(2)
C3^{i}–C3–C2	108.4(3)
C3^{i}–C3–Fe1	69.89(12)
C2–C3–Fe1	69.8(2)
C5–C4–C5^{i}	106.6(5)
C5–C4–B1	126.3(2)
C5^{i}–C4–B1	126.3(2)
C5–C4–Fe1	69.4(3)
C5^{i}–C4–Fe1	69.4(3)
B1–C4–Fe1	118.4(3)
C6–C5–C4	108.3(4)
C6–C5–Fe1	70.0(2)
C4–C5–Fe1	69.6(3)
C6^{i}–C6–C5	108.4(2)
C6^{i}–C6–Fe1	69.99(12)
C5–C6–Fe1	69.5(2)
C14–C13–C14^{i}	106.4(4)
C14–C13–B2	126.4(2)
C14^{i}–C13–B2	126.4(2)
C14–C13–Fe2	68.9(2)
C14^{i}–C13–Fe2	68.9(2)
B2–C13–Fe2	119.0(3)
C15–C14–C13	108.9(3)
C15–C14–Fe2	70.2(2)
C13–C14–Fe2	70.0(2)
C14–C15–C15^{i}	107.9(2)
C14–C15–Fe2	68.9(2)
C15^{i}–C15–Fe2	69.71(11)
C17^{i}–C16–C17	107.4(5)
C17^{i}–C16–Fe2	69.5(2)
C17–C16–Fe2	69.5(2)
C18–C17–C16	108.4(4)
C18–C17–Fe2	70.3(2)
C16–C17–Fe2	69.7(3)
C17–C18–C18^{i}	107.9(3)
C17–C18–Fe2	69.3(2)
C18^{i}–C18–Fe2	69.70(13)
C5^{i}–Fe1–C5	68.4(2)
C5^{i}–Fe1–C1	123.35(16)
C5–Fe1–C1	123.35(16)
C5^{i}–Fe1–C4	40.99(13)
C5–Fe1–C4	40.99(13)
C1–Fe1–C4	107.5(2)
C5^{i}–Fe1–C2	159.48(16)
C5–Fe1–C2	107.77(16)
C1–Fe1–C2	40.63(14)
C4–Fe1–C2	122.88(16)
C5^{i}–Fe1–C2^{i}	107.77(16)
C5–Fe1–C2^{i}	159.48(16)
C1–Fe1–C2^{i}	40.63(14)
C4–Fe1–C2^{i}	122.88(16)
C2–Fe1–C2^{i}	68.3(2)
C5^{i}–Fe1–C6^{i}	40.53(15)
C5–Fe1–C6^{i}	67.96(16)
C1–Fe1–C6^{i}	159.33(13)
C4–Fe1–C6^{i}	68.65(17)
C2–Fe1–C6^{i}	158.55(17)

C2^{i}–Fe1–C6^{i}	123.12(17)
C5^{i}–Fe1–C6	67.96(16)
C5–Fe1–C6	40.53(15)
C1–Fe1–C6	159.33(13)
C4–Fe1–C6	68.65(17)
C2–Fe1–C6	123.12(17)
C2^{i}–Fe1–C6	158.55(17)
C6^{i}–Fe1–C6	40.0(2)
C5^{i}–Fe1–C3	158.68(17)
C5–Fe1–C3	123.03(17)
C1–Fe1–C3	67.93(19)
C4–Fe1–C3	159.04(13)
C2–Fe1–C3	40.37(16)
C2^{i}–Fe1–C3	67.93(17)
C6^{i}–Fe1–C3	122.92(16)
C6–Fe1–C3	107.95(16)
C5^{i}–Fe1–C3^{i}	123.03(17)
C5–Fe1–C3^{i}	158.68(17)
C1–Fe1–C3^{i}	67.93(19)
C4–Fe1–C3^{i}	159.04(13)
C2–Fe1–C3^{i}	67.93(17)
C2^{i}–Fe1–C3^{i}	40.37(16)
C6^{i}–Fe1–C3^{i}	107.95(16)
C6–Fe1–C3^{i}	122.92(16)
C3–Fe1–C3^{i}	40.2(3)
C14–Fe2–C14^{i}	68.8(2)
C14–Fe2–C17^{i}	158.17(16)
C14^{i}–Fe2–C17^{i}	107.04(18)
C14–Fe2–C17	107.04(18)
C14^{i}–Fe2–C17	158.17(16)
C17^{i}–Fe2–C17	68.4(3)
C14–Fe2–C16	121.84(16)
C14^{i}–Fe2–C16	121.84(16)
C17^{i}–Fe2–C16	40.81(15)
C17–Fe2–C16	40.81(15)
C14–Fe2–C13	41.16(13)
C14^{i}–Fe2–C13	41.16(13)
C17^{i}–Fe2–C13	121.61(16)
C17–Fe2–C13	121.61(16)
C16–Fe2–C13	105.6(2)
C14–Fe2–C18^{i}	159.67(16)
C14^{i}–Fe2–C18^{i}	123.08(17)
C17^{i}–Fe2–C18^{i}	40.40(16)
C17–Fe2–C18^{i}	68.21(18)
C16–Fe2–C18^{i}	68.36(18)
C13–Fe2–C18^{i}	158.38(14)
C14–Fe2–C18	123.08(17)
C14^{i}–Fe2–C18	159.67(16)
C17^{i}–Fe2–C18	68.21(18)
C17–Fe2–C18	40.40(16)
C16–Fe2–C18	68.36(18)
C13–Fe2–C18	158.38(14)
C18^{i}–Fe2–C18	40.6(3)
C14–Fe2–C15	40.84(14)
C14^{i}–Fe2–C15	68.61(15)
C17^{i}–Fe2–C15	159.46(17)
C17–Fe2–C15	123.11(17)
C16–Fe2–C15	158.68(12)
C13–Fe2–C15	69.20(16)
C18^{i}–Fe2–C15	123.91(15)
C18–Fe2–C15	108.50(15)
C14–Fe2–C15^{i}	68.61(15)
C14^{i}–Fe2–C15^{i}	40.84(14)

C17^{i}–Fe2–C15^{i}	123.11(17)
C17–Fe2–C15^{i}	159.46(17)
C16–Fe2–C15^{i}	158.68(12)
C13–Fe2–C15^{i}	69.20(16)
C18^{i}–Fe2–C15^{i}	108.50(15)
C18–Fe2–C15^{i}	123.91(15)
C15–Fe2–C15^{i}	40.6(2)
O1^{i}–B1–O1	85.1(7)
O1^{i}–B1–O1'	112.6(5)
O1–B1–O1'	42.0(3)
O1^{i}–B1–O1''^{i}	42.0(3)
O1–B1–O1''^{i}	112.6(5)
O1'–B1–O1''^{i}	112.9(5)
O1^{i}–B1–C4	130.7(3)
O1–B1–C4	130.7(3)
O1'–B1–C4	116.5(3)
O1''^{i}–B1–C4	116.5(3)
O2''^{i}–B2–O2'	92.9(6)
O2''^{i}–B2–O2	114.2(5)
O2'–B2–O2	39.3(3)
O2''^{i}–B2–O2^{i}	39.3(3)
O2'–B2–O2^{i}	114.2(5)
O2–B2–O2^{i}	109.4(6)
O2''^{i}–B2–C13	126.4(4)
O2'–B2–C13	126.4(4)
O2–B2–C13	119.3(3)
O2^{i}–B2–C13	119.3(3)
B1–O1–C8	111.1(5)
O1–C8–C8''^{i}	104.8(7)
O1–C8–C9	105.6(7)
C8''^{i}–C8–C9	117.0(7)
C8–C9–C10	118.6(6)
C11–C10–C9	114.8(6)
O2–C11–C11''^{i}	103.3(8)
O2–C11–C10	108.6(7)
C11''^{i}–C11–C10	116.5(8)
B2–O2–C11	106.2(6)
C8'–O1'–B1	104.6(5)
O1'–C8'–C9'	107.5(7)
O1'–C8'–C8^{i}	103.5(8)
C9'–C8'–C8^{i}	118.8(8)
C8'–C9'–C10'	114.4(7)
C11'–C10'–C9'	118.4(7)
O2'–C11'–C10'	106.2(7)
O2'–C11'–C11^{i}	104.5(7)
C10'–C11'–C11^{i}	117.3(8)
B2–O2'–C11'	107.6(5)

Symmetry transformations used to generate equivalent atoms:
(i) x,–y+1/2,z

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	24(3)	52(4)	9(2)	0	−3(2)	0
C2	24(2)	37(3)	23(2)	−10(2)	−12(2)	5(2)
C3	14(2)	52(3)	26(2)	2(2)	−7(2)	−9(2)
C4	15(3)	36(4)	13(2)	0	−5(2)	0
C5	26(2)	27(2)	17(2)	3(2)	−4(2)	3(2)
C6	25(2)	44(2)	14(2)	6(2)	−2(2)	−5(2)
C13	10(3)	28(3)	18(2)	0	−4(2)	0
C14	20(2)	22(2)	21(2)	−3(2)	−1(2)	−4(2)
C15	22(2)	27(2)	13(2)	−4(2)	0(1)	−1(2)
C16	16(3)	72(5)	12(2)	0	0(2)	0
C17	22(2)	50(3)	25(2)	15(2)	−6(2)	6(2)
C18	18(2)	51(3)	24(2)	−2(2)	−2(2)	9(2)
Fe1	15(1)	30(1)	12(1)	0	0(1)	0
Fe2	13(1)	28(1)	12(1)	0	0(1)	0
B1	18(3)	55(5)	9(2)	0	−4(2)	0
B2	8(3)	24(4)	26(3)	0	−3(2)	0
O1	24(3)	26(3)	12(2)	8(2)	1(2)	4(3)
C8	14(4)	19(5)	11(3)	3(3)	−1(3)	5(3)
C9	13(4)	17(4)	11(4)	3(3)	0(3)	−4(3)
C10	15(2)	15(2)	16(2)	0(1)	0(1)	0(1)
C11	15(2)	14(2)	15(2)	0(1)	−1(1)	1(1)
O2	22(3)	22(3)	15(2)	−5(2)	4(2)	−2(3)
O1'	18(3)	19(3)	18(2)	0(2)	9(2)	−2(2)
C8'	15(2)	15(2)	15(2)	0(1)	−1(1)	0(1)
C9'	15(2)	14(2)	15(2)	0(1)	0(1)	−1(1)
C10'	15(2)	14(2)	14(2)	0(1)	1(1)	0(1)
C11'	14(2)	15(2)	15(2)	0(1)	0(1)	0(1)
O2'	16(3)	21(3)	17(2)	2(2)	6(2)	5(3)