CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 27 March 2007

Crystal Structure Analysis of:

TA32

(shown below)

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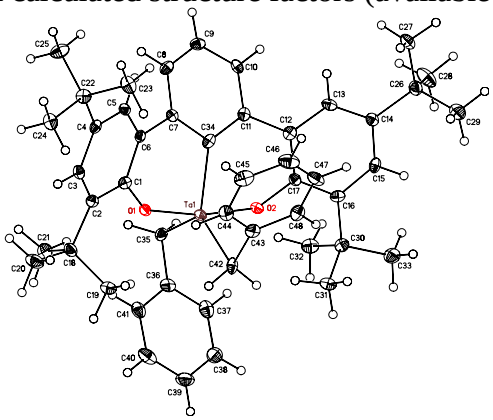

TA32

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 641994. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 641994."

Table 1. Crystal data and structure refinement for TA32 (CCDC 641994).

Empirical formula	$C_{48}H_{57}O_2Ta \cdot CH_2Cl_2$
Formula weight	931.81
Crystallization Solvent	Dichloromethane
Crystal Habit	Column
Crystal size	0.39 x 0.14 x 0.14 mm^3
Crystal color	Pale yellow

Data Collection

Type of diffractometer	Bruker SMART 1000	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 10124 reflections used in lattice determination	2.134 to 40.359°	
Unit cell dimensions	a = 10.7524(5) Å b = 11.2822(5) Å c = 18.5744(8) Å	α= 88.9940(10)° β= 82.324(2)° γ = 74.9580(10)°
Volume	2156.22(17) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.435 Mg/m^3	
F(000)	952	
Data collection program	Bruker SMART v5.630	
θ range for data collection	1.87 to 27.50°	
Completeness to θ = 27.50°	87.6 %	
Index ranges	$-13 \le h \le 13$, $-14 \le k \le 14$, $0 \le l \le 24$	
Data collection scan type	*w* scans at 9 ϕ settings	
Data reduction program	Bruker SAINT v6.45A	
Reflections collected	15033	
Independent reflections	15033 [R_{int}= 0.0000]	
Absorption coefficient	2.710 mm^{-1}	
Absorption correction	Semi-empirical from equivalents (TWINABS)	
Max. and min. transmission	0.7479 and 0.5247	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	15033 / 0 / 500
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.818
Final R indices [$I>2\sigma(I)$, 13977 reflections]	R1 = 0.0410, *w*R2 = 0.0908
R indices (all data)	R1 = 0.0449, *w*R2 = 0.0918
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Twin component scale factor	0.157
Largest diff. peak and hole	2.303 and -1.781 e.Å^{-3}

Special Refinement Details

The crystal is a twin. The orientation of two domains of the twin were defined using CELL_NOW from 999 reflections picked from four different runs. The domains are related to each other by a 180º rotation about the real *a* axis. Data were integrated (allowing for twinning) with SAINT and corrected for absorption with TWINABS producing a file suitable for twin refinement in SHELX.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

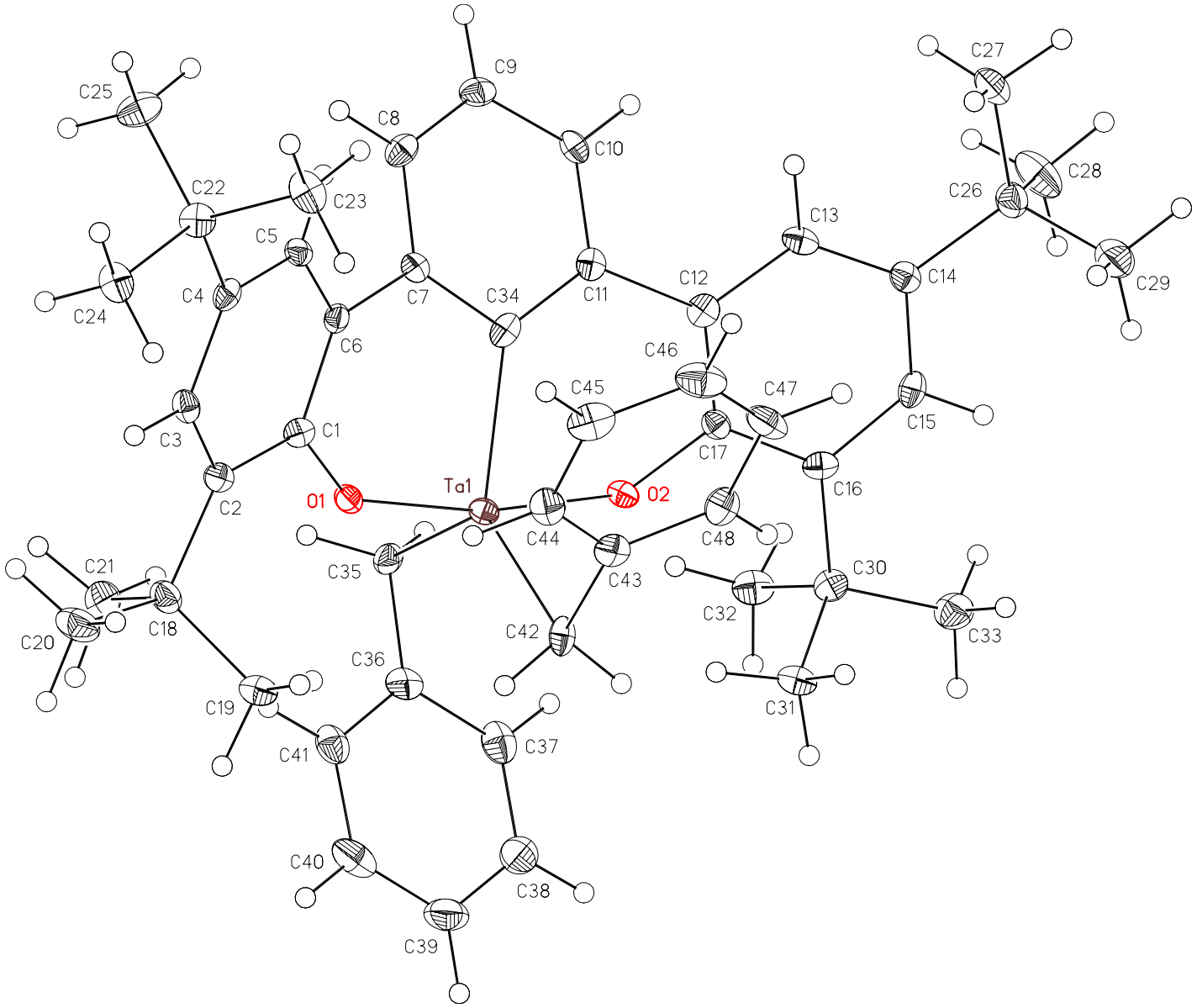
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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for TA32 (CCDC 641994). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ta(1)	7216(1)	8093(1)	6985(1)	11(1)
O(1)	7092(2)	9577(2)	6463(1)	13(1)
O(2)	7684(2)	6579(2)	7476(1)	12(1)
C(1)	7777(3)	10428(3)	6257(2)	12(1)
C(2)	7151(3)	11653(3)	6130(2)	12(1)
C(3)	7940(3)	12481(4)	6023(2)	13(1)
C(4)	9289(3)	12111(3)	6013(2)	12(1)
C(5)	9860(3)	10881(3)	6082(2)	11(1)
C(6)	9139(3)	10005(3)	6203(2)	11(1)
C(7)	9818(3)	8690(3)	6284(2)	10(1)
C(8)	11056(3)	8282(4)	5885(2)	14(1)
C(9)	11828(3)	7124(4)	5978(2)	15(1)
C(10)	11430(3)	6383(3)	6517(2)	12(1)
C(11)	10197(3)	6759(3)	6930(2)	12(1)
C(12)	9937(3)	5936(3)	7537(2)	12(1)
C(13)	10954(3)	5249(3)	7886(2)	13(1)
C(14)	10784(3)	4427(3)	8428(2)	13(1)
C(15)	9523(3)	4286(4)	8620(2)	14(1)
C(16)	8449(3)	4955(3)	8312(2)	13(1)
C(17)	8677(3)	5814(3)	7777(2)	12(1)
C(18)	5683(3)	12088(4)	6090(2)	15(1)
C(19)	4893(3)	11893(4)	6817(2)	18(1)
C(20)	5240(4)	13466(4)	5925(3)	23(1)
C(21)	5382(4)	11377(4)	5469(2)	22(1)
C(22)	10135(3)	13034(4)	5947(2)	14(1)
C(23)	10790(4)	12976(4)	6630(2)	19(1)
C(24)	9320(4)	14360(4)	5870(2)	18(1)
C(25)	11167(4)	12708(4)	5278(2)	21(1)
C(26)	11904(4)	3635(4)	8799(2)	17(1)
C(27)	13168(4)	4026(4)	8588(3)	28(1)
C(28)	12132(4)	2293(4)	8562(3)	30(1)
C(29)	11574(4)	3750(5)	9624(2)	28(1)
C(30)	7101(3)	4700(4)	8508(2)	15(1)
C(31)	6048(3)	5880(4)	8767(2)	16(1)
C(32)	6777(4)	4144(4)	7830(2)	19(1)
C(33)	7095(4)	3777(4)	9126(2)	21(1)
C(34)	9304(3)	7870(3)	6769(2)	12(1)
C(35)	6299(3)	7310(4)	6195(2)	14(1)
C(36)	4982(3)	7712(4)	6625(2)	15(1)
C(37)	4531(4)	6944(4)	7147(2)	20(1)
C(38)	3366(4)	7352(4)	7583(2)	24(1)
C(39)	2592(4)	8523(5)	7517(2)	28(1)
C(40)	3028(4)	9305(4)	7018(3)	24(1)
C(41)	4200(4)	8912(4)	6572(2)	19(1)
C(42)	6156(4)	9064(4)	7992(2)	14(1)
C(43)	7305(4)	9324(4)	8263(2)	15(1)
C(44)	7712(4)	10392(4)	8050(2)	18(1)

C(45)	8826(4)	10587(4)	8263(2)	22(1)
C(46)	9548(4)	9762(4)	8706(2)	25(1)
C(47)	9168(4)	8704(4)	8925(2)	21(1)
C(48)	8057(4)	8495(4)	8702(2)	18(1)
C(51)	6613(4)	818(4)	-17(3)	36(1)
Cl(1)	5047(1)	1561(1)	-219(1)	35(1)
Cl(2)	7055(2)	1540(2)	670(1)	84(1)

Table 3. Selected bond lengths [Å] and angles [°] for TA32 (CCDC 641994).

Ta(1)-O(1)	1.904(2)	O(1)-Ta(1)-O(2)	169.17(9)
Ta(1)-O(2)	1.903(2)	O(1)-Ta(1)-C(34)	84.98(12)
Ta(1)-C(34)	2.176(3)	O(2)-Ta(1)-C(34)	84.19(12)
Ta(1)-C(35)	2.190(4)	O(1)-Ta(1)-C(35)	92.67(13)
Ta(1)-C(42)	2.203(4)	O(2)-Ta(1)-C(35)	92.61(13)
		C(34)-Ta(1)-C(35)	117.86(13)
		O(1)-Ta(1)-C(42)	92.91(13)
		O(2)-Ta(1)-C(42)	91.98(13)
		C(34)-Ta(1)-C(42)	118.24(14)
		C(35)-Ta(1)-C(42)	123.89(13)

Table 4. Bond lengths [Å] and angles [°] for TA32 (CCDC 641994).

Ta(1)-O(1)	1.904(2)	C(43)-C(48)	1.397(6)
Ta(1)-O(2)	1.903(2)	C(43)-C(44)	1.417(5)
Ta(1)-C(34)	2.176(3)	C(44)-C(45)	1.382(5)
Ta(1)-C(35)	2.190(4)	C(45)-C(46)	1.384(6)
Ta(1)-C(42)	2.203(4)	C(46)-C(47)	1.397(6)
O(1)-C(1)	1.375(4)	C(47)-C(48)	1.391(5)
O(2)-C(17)	1.365(5)	C(51)-Cl(2)	1.708(5)
C(1)-C(2)	1.402(5)	C(51)-Cl(1)	1.761(5)
C(1)-C(6)	1.408(5)		
C(2)-C(3)	1.411(5)	O(1)-Ta(1)-O(2)	169.17(9)
C(2)-C(18)	1.539(5)	O(1)-Ta(1)-C(34)	84.98(12)
C(3)-C(4)	1.399(5)	O(2)-Ta(1)-C(34)	84.19(12)
C(4)-C(5)	1.375(5)	O(1)-Ta(1)-C(35)	92.67(13)
C(4)-C(22)	1.544(5)	O(2)-Ta(1)-C(35)	92.61(13)
C(5)-C(6)	1.404(5)	C(34)-Ta(1)-C(35)	117.86(13)
C(6)-C(7)	1.488(5)	O(1)-Ta(1)-C(42)	92.91(13)
C(7)-C(8)	1.402(5)	O(2)-Ta(1)-C(42)	91.98(13)
C(7)-C(34)	1.440(5)	C(34)-Ta(1)-C(42)	118.24(14)
C(8)-C(9)	1.376(5)	C(35)-Ta(1)-C(42)	123.89(13)
C(9)-C(10)	1.387(5)	C(1)-O(1)-Ta(1)	141.6(2)
C(10)-C(11)	1.405(5)	C(17)-O(2)-Ta(1)	142.9(2)
C(11)-C(34)	1.422(5)	O(1)-C(1)-C(2)	121.8(3)
C(11)-C(12)	1.490(5)	O(1)-C(1)-C(6)	116.0(3)
C(12)-C(17)	1.410(4)	C(2)-C(1)-C(6)	122.2(3)
C(12)-C(13)	1.397(5)	C(1)-C(2)-C(3)	116.8(3)
C(13)-C(14)	1.382(5)	C(1)-C(2)-C(18)	122.5(3)
C(14)-C(15)	1.404(4)	C(3)-C(2)-C(18)	120.7(3)
C(14)-C(26)	1.538(6)	C(4)-C(3)-C(2)	122.6(4)
C(15)-C(16)	1.392(6)	C(5)-C(4)-C(3)	118.0(3)
C(16)-C(17)	1.419(5)	C(5)-C(4)-C(22)	119.7(3)
C(16)-C(30)	1.545(4)	C(3)-C(4)-C(22)	122.3(3)
C(18)-C(19)	1.541(5)	C(4)-C(5)-C(6)	122.7(3)
C(18)-C(21)	1.531(6)	C(5)-C(6)-C(1)	117.4(3)
C(18)-C(20)	1.541(5)	C(5)-C(6)-C(7)	120.0(3)
C(22)-C(24)	1.540(5)	C(1)-C(6)-C(7)	122.6(3)
C(22)-C(25)	1.531(5)	C(8)-C(7)-C(34)	120.1(4)
C(22)-C(23)	1.525(6)	C(8)-C(7)-C(6)	116.0(3)
C(26)-C(29)	1.527(6)	C(34)-C(7)-C(6)	123.8(3)
C(26)-C(28)	1.532(6)	C(9)-C(8)-C(7)	120.9(3)
C(26)-C(27)	1.535(5)	C(8)-C(9)-C(10)	119.9(3)
C(30)-C(33)	1.537(5)	C(9)-C(10)-C(11)	120.7(4)
C(30)-C(31)	1.542(5)	C(10)-C(11)-C(34)	120.3(3)
C(30)-C(32)	1.537(6)	C(10)-C(11)-C(12)	115.4(3)
C(35)-C(36)	1.492(5)	C(34)-C(11)-C(12)	124.4(3)
C(36)-C(41)	1.405(6)	C(17)-C(12)-C(13)	117.5(3)
C(36)-C(37)	1.410(5)	C(17)-C(12)-C(11)	122.0(3)
C(37)-C(38)	1.372(6)	C(13)-C(12)-C(11)	120.4(3)
C(38)-C(39)	1.380(6)	C(14)-C(13)-C(12)	123.0(3)
C(39)-C(40)	1.389(6)	C(13)-C(14)-C(15)	117.3(3)
C(40)-C(41)	1.383(6)	C(13)-C(14)-C(26)	123.4(3)
C(42)-C(43)	1.491(5)	C(15)-C(14)-C(26)	119.3(3)

C(16)-C(15)-C(14)	123.5(3)	C(16)-C(30)-C(31)	111.6(3)
C(15)-C(16)-C(17)	116.6(3)	C(33)-C(30)-C(32)	108.2(3)
C(15)-C(16)-C(30)	121.1(3)	C(16)-C(30)-C(32)	107.6(3)
C(17)-C(16)-C(30)	122.1(3)	C(31)-C(30)-C(32)	110.9(3)
O(2)-C(17)-C(12)	116.5(3)	C(11)-C(34)-C(7)	116.6(3)
O(2)-C(17)-C(16)	121.6(3)	C(11)-C(34)-Ta(1)	121.2(2)
C(12)-C(17)-C(16)	121.9(3)	C(7)-C(34)-Ta(1)	120.0(3)
C(19)-C(18)-C(21)	111.1(3)	C(36)-C(35)-Ta(1)	93.2(2)
C(19)-C(18)-C(2)	111.0(3)	C(41)-C(36)-C(37)	117.7(3)
C(21)-C(18)-C(2)	108.0(3)	C(41)-C(36)-C(35)	120.9(3)
C(19)-C(18)-C(20)	106.6(3)	C(37)-C(36)-C(35)	121.1(4)
C(21)-C(18)-C(20)	107.6(4)	C(38)-C(37)-C(36)	121.1(4)
C(2)-C(18)-C(20)	112.5(3)	C(37)-C(38)-C(39)	120.9(4)
C(24)-C(22)-C(25)	108.3(3)	C(40)-C(39)-C(38)	119.1(4)
C(24)-C(22)-C(4)	111.9(3)	C(39)-C(40)-C(41)	120.9(4)
C(25)-C(22)-C(4)	109.8(3)	C(40)-C(41)-C(36)	120.4(4)
C(24)-C(22)-C(23)	108.2(3)	C(43)-C(42)-Ta(1)	96.4(2)
C(25)-C(22)-C(23)	109.8(3)	C(48)-C(43)-C(44)	117.7(4)
C(4)-C(22)-C(23)	108.8(3)	C(48)-C(43)-C(42)	121.3(3)
C(29)-C(26)-C(28)	109.5(4)	C(44)-C(43)-C(42)	120.9(4)
C(29)-C(26)-C(14)	110.7(3)	C(45)-C(44)-C(43)	120.7(4)
C(28)-C(26)-C(14)	108.7(3)	C(44)-C(45)-C(46)	120.7(4)
C(29)-C(26)-C(27)	107.7(4)	C(47)-C(46)-C(45)	119.8(4)
C(28)-C(26)-C(27)	108.7(3)	C(46)-C(47)-C(48)	119.7(4)
C(14)-C(26)-C(27)	111.6(3)	C(47)-C(48)-C(43)	121.4(4)
C(33)-C(30)-C(16)	111.9(3)	Cl(2)-C(51)-Cl(1)	112.4(3)
C(33)-C(30)-C(31)	106.7(3)		

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for TA32 (CCDC 641994). 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}
Ta(1)	95(1)	110(1)	129(1)	-7(1)	-20(1)	-29(1)
O(1)	106(11)	132(13)	148(14)	31(11)	-34(11)	-29(11)
O(2)	94(11)	109(12)	154(14)	1(11)	-21(11)	-19(10)
C(1)	127(16)	120(18)	115(19)	-22(16)	-10(14)	-44(15)
C(2)	124(16)	138(18)	102(18)	-29(15)	-19(15)	-28(16)
C(3)	156(17)	97(18)	115(19)	-9(16)	-35(15)	-4(15)
C(4)	160(17)	144(18)	81(18)	-9(15)	-15(15)	-67(16)
C(5)	103(16)	141(18)	99(18)	-13(16)	-29(14)	-25(15)
C(6)	134(16)	98(18)	95(19)	13(15)	-1(15)	-10(15)
C(7)	116(16)	100(18)	109(19)	-18(16)	-38(14)	-42(14)
C(8)	170(17)	160(20)	120(20)	-9(17)	-1(16)	-82(16)
C(9)	117(16)	190(20)	170(20)	-23(18)	-14(15)	-68(16)
C(10)	136(17)	83(18)	150(20)	-5(16)	-56(15)	-15(15)
C(11)	147(17)	109(18)	118(19)	-9(16)	-41(15)	-73(15)
C(12)	164(17)	58(16)	131(19)	-38(15)	-15(15)	-43(15)
C(13)	101(16)	125(18)	170(20)	-49(16)	-32(15)	-43(15)
C(14)	133(16)	139(18)	122(19)	-22(16)	-36(15)	-36(15)
C(15)	197(18)	119(19)	110(20)	15(16)	-28(16)	-68(16)
C(16)	119(16)	122(18)	140(20)	-50(16)	7(15)	-38(15)
C(17)	119(16)	112(18)	123(19)	-7(16)	-27(15)	-20(14)
C(18)	143(17)	130(19)	170(20)	14(17)	-35(16)	-2(16)
C(19)	125(17)	180(20)	220(20)	-8(18)	-13(16)	-16(16)
C(20)	151(18)	160(20)	370(30)	30(20)	-29(18)	2(17)
C(21)	190(19)	290(20)	190(20)	20(20)	-44(17)	-67(18)
C(22)	166(17)	97(19)	180(20)	-29(17)	-21(16)	-57(16)
C(23)	231(19)	142(19)	230(20)	-11(18)	-96(18)	-54(17)
C(24)	216(19)	133(19)	200(20)	10(17)	-74(17)	-65(16)
C(25)	205(19)	200(20)	220(20)	-51(18)	33(17)	-109(17)
C(26)	173(18)	160(20)	190(20)	40(18)	-40(16)	-42(16)
C(27)	153(19)	340(30)	330(30)	150(20)	-85(19)	-37(19)
C(28)	250(20)	190(20)	430(30)	10(20)	-140(20)	21(19)
C(29)	200(20)	380(30)	210(20)	10(20)	-51(18)	-10(20)
C(30)	155(17)	136(19)	160(20)	0(17)	-14(16)	-62(16)
C(31)	115(17)	145(19)	220(20)	-29(18)	-17(16)	-24(15)
C(32)	174(18)	180(20)	210(20)	-46(19)	-18(17)	-80(17)
C(33)	191(19)	200(20)	250(20)	28(19)	-18(17)	-81(17)
C(34)	181(17)	105(19)	96(19)	-25(16)	-8(15)	-78(15)
C(35)	143(17)	190(20)	96(19)	-2(17)	-16(15)	-57(16)
C(36)	129(17)	190(20)	160(20)	-53(18)	-50(15)	-82(16)
C(37)	230(20)	180(20)	230(20)	29(19)	-104(18)	-92(18)
C(38)	185(19)	370(30)	200(20)	30(20)	-69(17)	-131(19)
C(39)	147(19)	430(30)	210(20)	-50(20)	-2(18)	-10(20)
C(40)	176(19)	250(20)	280(30)	-70(20)	-91(18)	12(18)
C(41)	187(19)	180(20)	220(20)	36(19)	-95(17)	-81(17)
C(42)	218(18)	133(19)	91(19)	-1(16)	-63(16)	-82(16)
C(43)	160(17)	180(20)	101(19)	-82(17)	-7(16)	-32(16)
C(44)	200(19)	150(20)	170(20)	-14(18)	-31(17)	-25(16)

C(45)	228(19)	210(20)	240(20)	-80(19)	20(18)	-99(18)
C(46)	170(19)	290(20)	290(20)	-120(20)	-20(18)	-63(18)
C(47)	135(17)	260(20)	220(20)	-60(20)	-59(16)	14(17)
C(48)	222(19)	180(20)	130(20)	-34(17)	-15(17)	-57(17)
C(51)	200(20)	260(20)	590(40)	-50(30)	-70(20)	-10(20)
Cl(1)	276(5)	243(6)	531(8)	70(6)	-76(5)	-45(5)
Cl(2)	909(13)	769(13)	690(12)	-404(11)	-595(11)	310(10)