CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 29 August 2006

Crystal Structure Analysis of:

TA22

(shown below)

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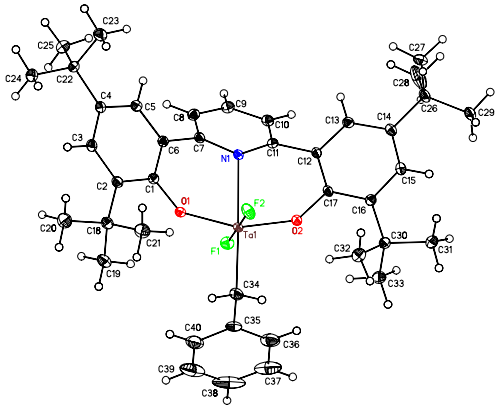


TA22

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 618859. 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 618859."

Table 1. Crystal data and structure refinement for TA22 (CCDC 618859).

Empirical formula	$C_{40}H_{50}NO_2F_2Ta \bullet C_6H_6$
Formula weight	873.87
Crystallization Solvent	Benzene
Crystal Habit	Blade
Crystal size	0.33 x 0.23 x 0.07 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 35940 reflections used in lattice determination	2.42 to 39.55°
Unit cell dimensions	a = 13.6879(4) Å b = 28.6266(7) Å β= 113.9350(10)° c = 11.3658(3) Å
Volume	4070.57(19) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.426 Mg/m^3
F(000)	1784
Data collection program	Bruker SMART v5.630
θ range for data collection	1.63 to 40.73°
Completeness to θ = 40.73°	95.4 %
Index ranges	$-25 \le h \le 24$, $-52 \le k \le 48$, $-20 \le l \le 20$
Data collection scan type	ω scans at 7 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	109242
Independent reflections	25167 [R_{int}= 0.0668]
Absorption coefficient	2.747 mm^{-1}
Absorption correction	SADABS
Max. and min. transmission	0.7294 and 0.0000

Table 1 (cont.)

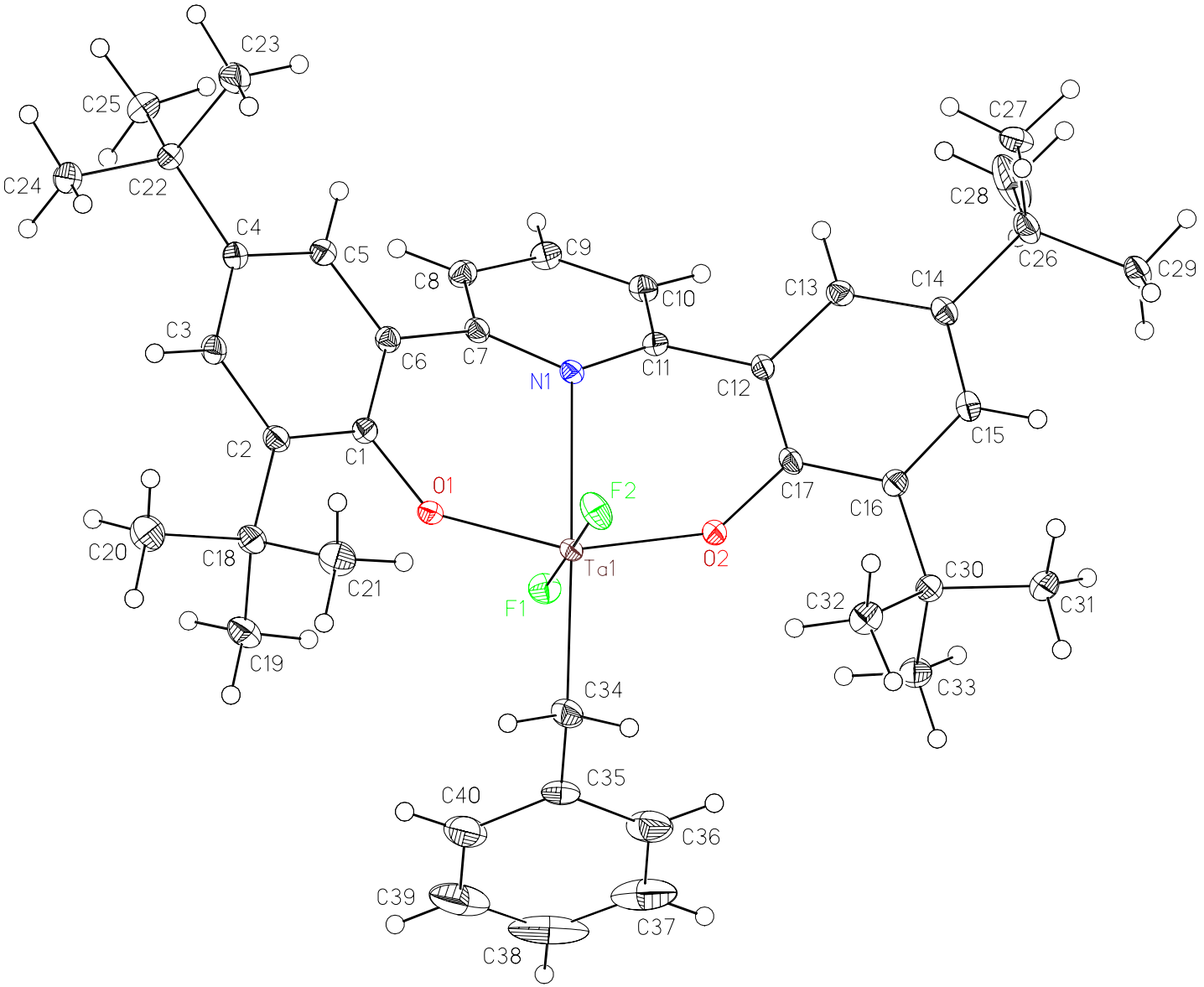
Structure solution and Refinement

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	25167 / 0 / 481
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.229
Final R indices [I>2σ(I), 17397 reflections]	R1 = 0.0398, *w*R2 = 0.0580
R indices (all data)	R1 = 0.0753, *w*R2 = 0.0632
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.004
Average shift/error	0.000
Largest diff. peak and hole	2.216 and -3.441 e.Å^{-3}

Special Refinement Details

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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C16
C20
C18
Ta1
O2
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F1
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C34
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C36
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C37
C38

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for TA22 (CCDC 618859). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ta(1)	1893(1)	1412(1)	1243(1)	10(1)
F(1)	1349(1)	1374(1)	2528(1)	16(1)
F(2)	2457(1)	1444(1)	-42(1)	19(1)
O(1)	3020(1)	1000(1)	2177(1)	13(1)
O(2)	1184(1)	1987(1)	640(1)	13(1)
N(1)	3187(1)	1957(1)	2732(1)	10(1)
C(1)	4093(1)	972(1)	2883(2)	12(1)
C(2)	4673(1)	574(1)	2838(2)	13(1)
C(3)	5778(1)	580(1)	3605(2)	14(1)
C(4)	6304(1)	958(1)	4385(2)	13(1)
C(5)	5688(1)	1341(1)	4408(2)	13(1)
C(6)	4578(1)	1353(1)	3680(2)	11(1)
C(7)	3979(1)	1767(1)	3800(2)	11(1)
C(8)	4283(1)	1966(1)	5016(2)	14(1)
C(9)	3806(2)	2376(1)	5161(2)	14(1)
C(10)	3086(1)	2594(1)	4073(2)	13(1)
C(11)	2808(1)	2386(1)	2863(2)	11(1)
C(12)	2123(1)	2659(1)	1714(2)	11(1)
C(13)	2262(1)	3144(1)	1750(2)	13(1)
C(14)	1613(2)	3432(1)	747(2)	13(1)
C(15)	817(1)	3214(1)	-305(2)	13(1)
C(16)	638(1)	2733(1)	-407(2)	12(1)
C(17)	1319(1)	2457(1)	630(2)	11(1)
C(18)	4131(2)	157(1)	1963(2)	17(1)
C(19)	3203(2)	-28(1)	2262(2)	22(1)
C(20)	4900(2)	-252(1)	2149(2)	27(1)
C(21)	3726(2)	313(1)	546(2)	22(1)
C(22)	7520(1)	975(1)	5155(2)	15(1)
C(23)	7990(2)	1333(1)	4519(2)	21(1)
C(24)	8054(2)	498(1)	5192(2)	21(1)
C(25)	7790(2)	1129(1)	6553(2)	20(1)
C(26)	1796(2)	3962(1)	814(2)	16(1)
C(27)	2795(2)	4066(1)	563(2)	24(1)
C(28)	1960(2)	4150(1)	2147(2)	36(1)
C(29)	853(2)	4223(1)	-205(2)	20(1)
C(30)	-298(1)	2524(1)	-1568(2)	13(1)
C(31)	-813(2)	2893(1)	-2621(2)	18(1)
C(32)	58(2)	2118(1)	-2195(2)	17(1)
C(33)	-1145(2)	2349(1)	-1103(2)	17(1)
C(34)	688(2)	916(1)	36(2)	17(1)
C(35)	-164(2)	782(1)	496(2)	19(1)
C(36)	-1064(2)	1059(1)	220(2)	29(1)
C(37)	-1822(2)	955(1)	708(3)	43(1)
C(38)	-1685(2)	568(1)	1482(3)	50(1)
C(39)	-810(2)	282(1)	1744(2)	41(1)
C(40)	-57(2)	387(1)	1256(2)	28(1)

C(51)	5821(2)	2926(1)	3957(3)	45(1)
C(52)	6342(2)	2764(1)	5204(3)	45(1)
C(53)	6228(2)	2999(1)	6204(3)	39(1)
C(54)	5580(2)	3390(1)	5954(2)	32(1)
C(55)	5064(2)	3546(1)	4708(2)	31(1)
C(56)	5190(2)	3315(1)	3720(2)	40(1)

Table 3. Selected bond lengths [Å] and angles [°] for TA22 (CCDC 618859).

Ta(1)-O(1)	1.8906(13)	O(1)-Ta(1)-F(1)	91.00(5)
Ta(1)-F(1)	1.8927(10)	O(1)-Ta(1)-O(2)	158.28(5)
Ta(1)-O(2)	1.8908(13)	F(1)-Ta(1)-O(2)	91.47(5)
Ta(1)-F(2)	1.9110(11)	O(1)-Ta(1)-F(2)	88.16(5)
Ta(1)-C(34)	2.1872(19)	F(1)-Ta(1)-F(2)	179.13(5)
Ta(1)-N(1)	2.4486(14)	O(2)-Ta(1)-F(2)	89.39(5)
		O(1)-Ta(1)-C(34)	100.50(6)
		F(1)-Ta(1)-C(34)	90.74(6)
		O(2)-Ta(1)-C(34)	101.05(7)
		F(2)-Ta(1)-C(34)	89.21(6)
		O(1)-Ta(1)-N(1)	79.26(5)
		F(1)-Ta(1)-N(1)	84.55(5)
		O(2)-Ta(1)-N(1)	79.50(5)
		F(2)-Ta(1)-N(1)	95.49(5)
		C(34)-Ta(1)-N(1)	175.28(6)

Table 4. Bond lengths [Å] and angles [°] for TA22 (CCDC 618859).

Ta(1)-O(1)	1.8906(13)	C(51)-C(56)	1.366(4)
Ta(1)-F(1)	1.8927(10)	C(51)-C(52)	1.383(4)
Ta(1)-O(2)	1.8908(13)	C(52)-C(53)	1.381(4)
Ta(1)-F(2)	1.9110(11)	C(53)-C(54)	1.384(4)
Ta(1)-C(34)	2.1872(19)	C(54)-C(55)	1.375(4)
Ta(1)-N(1)	2.4486(14)	C(55)-C(56)	1.374(4)
O(1)-C(1)	1.360(2)		
O(2)-C(17)	1.360(2)	O(1)-Ta(1)-F(1)	91.00(5)
N(1)-C(11)	1.366(2)	O(1)-Ta(1)-O(2)	158.28(5)
N(1)-C(7)	1.370(2)	F(1)-Ta(1)-O(2)	91.47(5)
C(1)-C(6)	1.399(2)	O(1)-Ta(1)-F(2)	88.16(5)
C(1)-C(2)	1.403(3)	F(1)-Ta(1)-F(2)	179.13(5)
C(2)-C(3)	1.406(3)	O(2)-Ta(1)-F(2)	89.39(5)
C(2)-C(18)	1.537(3)	O(1)-Ta(1)-C(34)	100.50(6)
C(3)-C(4)	1.399(3)	F(1)-Ta(1)-C(34)	90.74(6)
C(4)-C(5)	1.391(2)	O(2)-Ta(1)-C(34)	101.05(7)
C(4)-C(22)	1.535(2)	F(2)-Ta(1)-C(34)	89.21(6)
C(5)-C(6)	1.405(2)	O(1)-Ta(1)-N(1)	79.26(5)
C(6)-C(7)	1.478(2)	F(1)-Ta(1)-N(1)	84.55(5)
C(7)-C(8)	1.393(2)	O(2)-Ta(1)-N(1)	79.50(5)
C(8)-C(9)	1.385(3)	F(2)-Ta(1)-N(1)	95.49(5)
C(9)-C(10)	1.378(3)	C(34)-Ta(1)-N(1)	175.28(6)
C(10)-C(11)	1.401(2)	C(1)-O(1)-Ta(1)	143.78(12)
C(11)-C(12)	1.483(2)	C(17)-O(2)-Ta(1)	144.38(11)
C(12)-C(13)	1.401(3)	C(11)-N(1)-C(7)	117.39(15)
C(12)-C(17)	1.401(2)	C(11)-N(1)-Ta(1)	116.64(11)
C(13)-C(14)	1.395(3)	C(7)-N(1)-Ta(1)	116.70(11)
C(14)-C(15)	1.396(3)	O(1)-C(1)-C(6)	116.91(16)
C(14)-C(26)	1.536(3)	O(1)-C(1)-C(2)	121.11(16)
C(15)-C(16)	1.395(3)	C(6)-C(1)-C(2)	121.96(16)
C(16)-C(17)	1.410(2)	C(3)-C(2)-C(1)	116.74(16)
C(16)-C(30)	1.539(2)	C(3)-C(2)-C(18)	121.76(16)
C(18)-C(20)	1.532(3)	C(1)-C(2)-C(18)	121.49(16)
C(18)-C(19)	1.536(3)	C(4)-C(3)-C(2)	123.38(17)
C(18)-C(21)	1.542(3)	C(5)-C(4)-C(3)	117.48(16)
C(22)-C(24)	1.540(3)	C(5)-C(4)-C(22)	119.31(16)
C(22)-C(23)	1.539(3)	C(3)-C(4)-C(22)	123.15(16)
C(22)-C(25)	1.542(3)	C(4)-C(5)-C(6)	121.81(16)
C(26)-C(27)	1.535(3)	C(5)-C(6)-C(1)	118.58(16)
C(26)-C(28)	1.536(3)	C(5)-C(6)-C(7)	118.20(15)
C(26)-C(29)	1.534(3)	C(1)-C(6)-C(7)	123.23(15)
C(30)-C(31)	1.536(3)	N(1)-C(7)-C(8)	121.77(16)
C(30)-C(32)	1.542(3)	N(1)-C(7)-C(6)	120.41(15)
C(30)-C(33)	1.541(2)	C(8)-C(7)-C(6)	117.75(16)
C(34)-C(35)	1.509(3)	C(9)-C(8)-C(7)	119.93(17)
C(35)-C(40)	1.393(3)	C(10)-C(9)-C(8)	118.48(16)
C(35)-C(36)	1.391(3)	C(9)-C(10)-C(11)	119.96(17)
C(36)-C(37)	1.394(3)	N(1)-C(11)-C(10)	121.63(16)
C(37)-C(38)	1.379(5)	N(1)-C(11)-C(12)	120.57(15)
C(38)-C(39)	1.381(5)	C(10)-C(11)-C(12)	117.74(16)
C(39)-C(40)	1.388(3)	C(13)-C(12)-C(17)	118.88(16)

C(13)-C(12)-C(11)	117.75(16)	C(27)-C(26)-C(29)	107.87(16)
C(17)-C(12)-C(11)	123.30(16)	C(28)-C(26)-C(29)	108.44(18)
C(14)-C(13)-C(12)	121.84(17)	C(27)-C(26)-C(14)	108.81(16)
C(13)-C(14)-C(15)	116.97(17)	C(28)-C(26)-C(14)	110.48(16)
C(13)-C(14)-C(26)	120.39(16)	C(29)-C(26)-C(14)	111.85(16)
C(15)-C(14)-C(26)	122.64(16)	C(31)-C(30)-C(32)	106.88(15)
C(16)-C(15)-C(14)	124.17(17)	C(31)-C(30)-C(16)	111.12(15)
C(15)-C(16)-C(17)	116.66(16)	C(32)-C(30)-C(16)	112.31(15)
C(15)-C(16)-C(30)	120.75(16)	C(31)-C(30)-C(33)	108.62(15)
C(17)-C(16)-C(30)	122.51(16)	C(32)-C(30)-C(33)	109.65(16)
O(2)-C(17)-C(12)	117.43(15)	C(16)-C(30)-C(33)	108.20(14)
O(2)-C(17)-C(16)	121.03(16)	C(35)-C(34)-Ta(1)	116.14(13)
C(12)-C(17)-C(16)	121.46(16)	C(40)-C(35)-C(36)	117.7(2)
C(2)-C(18)-C(20)	112.30(16)	C(40)-C(35)-C(34)	121.4(2)
C(2)-C(18)-C(19)	110.52(16)	C(36)-C(35)-C(34)	120.9(2)
C(20)-C(18)-C(19)	106.79(17)	C(35)-C(36)-C(37)	121.5(3)
C(2)-C(18)-C(21)	108.99(16)	C(38)-C(37)-C(36)	119.7(3)
C(20)-C(18)-C(21)	107.57(17)	C(39)-C(38)-C(37)	119.7(2)
C(19)-C(18)-C(21)	110.63(17)	C(38)-C(39)-C(40)	120.4(3)
C(24)-C(22)-C(23)	108.62(16)	C(35)-C(40)-C(39)	121.0(3)
C(24)-C(22)-C(4)	112.34(16)	C(56)-C(51)-C(52)	119.8(2)
C(23)-C(22)-C(4)	108.43(15)	C(53)-C(52)-C(51)	119.8(3)
C(24)-C(22)-C(25)	108.19(16)	C(52)-C(53)-C(54)	120.0(2)
C(23)-C(22)-C(25)	108.81(16)	C(55)-C(54)-C(53)	119.5(2)
C(4)-C(22)-C(25)	110.38(16)	C(56)-C(55)-C(54)	120.2(3)
C(27)-C(26)-C(28)	109.32(19)	C(55)-C(56)-C(51)	120.6(3)

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for TA22 (CCDC 618859). The anisotropic displacement factorexponent 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)	103(1)	76(1)	106(1)	-4(1)	38(1)	-8(1)
F(1)	161(5)	178(6)	157(5)	3(4)	92(4)	-12(5)
F(2)	266(6)	137(5)	215(5)	21(5)	159(5)	9(5)
O(1)	104(5)	92(6)	166(6)	-11(4)	39(5)	-12(5)
O(2)	125(6)	92(6)	140(6)	-1(4)	21(5)	1(5)
N(1)	105(6)	81(6)	123(6)	-8(5)	44(5)	-2(5)
C(1)	114(7)	115(8)	126(7)	11(6)	49(6)	0(6)
C(2)	131(7)	105(8)	159(8)	-2(6)	64(6)	-4(6)
C(3)	127(7)	108(8)	192(8)	6(6)	68(7)	25(6)
C(4)	120(7)	108(8)	156(8)	6(6)	49(6)	10(6)
C(5)	125(7)	119(9)	144(7)	-6(6)	47(6)	-3(6)
C(6)	115(7)	103(8)	123(7)	-2(5)	52(6)	7(6)
C(7)	114(7)	90(7)	137(7)	-2(6)	57(6)	1(6)
C(8)	126(7)	154(8)	120(7)	2(6)	42(6)	-5(6)
C(9)	144(8)	153(8)	116(7)	-26(6)	58(6)	-7(7)
C(10)	139(8)	112(8)	143(7)	-36(6)	68(6)	0(6)
C(11)	97(7)	115(8)	122(7)	1(6)	40(6)	-8(6)
C(12)	118(7)	91(7)	117(7)	7(5)	45(6)	3(6)
C(13)	125(7)	108(8)	140(7)	-21(6)	48(6)	-8(6)
C(14)	153(8)	100(7)	155(8)	-2(6)	81(6)	4(6)
C(15)	149(8)	116(8)	137(7)	21(6)	66(6)	19(6)
C(16)	110(7)	121(8)	136(7)	-1(6)	61(6)	9(6)
C(17)	128(7)	80(7)	145(7)	1(6)	68(6)	7(6)
C(18)	166(8)	98(8)	211(9)	-38(6)	54(7)	2(7)
C(19)	226(10)	125(9)	292(10)	-18(7)	90(8)	-31(8)
C(20)	252(11)	133(9)	351(12)	-77(8)	54(9)	30(8)
C(21)	256(10)	183(10)	200(9)	-66(7)	72(8)	-10(8)
C(22)	103(7)	130(8)	190(8)	13(6)	37(6)	2(6)
C(23)	158(8)	165(10)	310(10)	35(7)	104(8)	-6(7)
C(24)	140(8)	141(9)	311(11)	16(8)	51(8)	23(7)
C(25)	153(9)	208(10)	204(9)	9(7)	23(7)	-12(8)
C(26)	213(9)	87(8)	168(8)	-4(6)	74(7)	8(7)
C(27)	161(9)	136(9)	377(12)	-5(8)	57(9)	-46(8)
C(28)	726(19)	142(10)	219(10)	-29(8)	197(12)	43(11)
C(29)	175(9)	99(8)	302(10)	27(7)	80(8)	14(7)
C(30)	117(7)	131(8)	135(7)	0(6)	53(6)	1(6)
C(31)	154(8)	182(9)	156(8)	24(7)	15(7)	-10(7)
C(32)	174(8)	191(10)	142(8)	-31(7)	65(7)	7(7)
C(33)	143(8)	185(9)	195(9)	10(7)	81(7)	-10(7)
C(34)	192(9)	126(9)	187(9)	-30(6)	62(7)	-27(7)
C(35)	172(8)	183(9)	185(8)	-68(7)	57(7)	-75(7)
C(36)	224(10)	237(12)	403(13)	-131(10)	132(10)	-61(9)
C(37)	254(12)	469(17)	613(18)	-344(15)	236(13)	-166(12)
C(38)	467(16)	710(20)	447(16)	-374(15)	321(14)	-436(16)
C(39)	489(16)	471(17)	262(12)	-54(11)	154(12)	-319(14)
C(40)	292(12)	259(12)	258(11)	-3(9)	69(9)	-119(9)

C(51)	252(12)	600(20)	455(16)	-294(14)	118(12)	11(13)
C(52)	187(11)	237(13)	770(20)	-77(13)	33(13)	4(10)
C(53)	263(12)	425(16)	331(13)	102(11)	-30(10)	-139(11)
C(54)	316(12)	386(14)	292(12)	-112(10)	162(10)	-124(11)
C(55)	243(10)	314(13)	381(12)	-1(10)	127(9)	42(10)
C(56)	270(12)	670(20)	227(11)	-9(12)	63(10)	59(13)