CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 29 July 2005

Crystal Structure Analysis of:

TA17

(shown below)

For Investigator: Theo Agapie ext. 6576

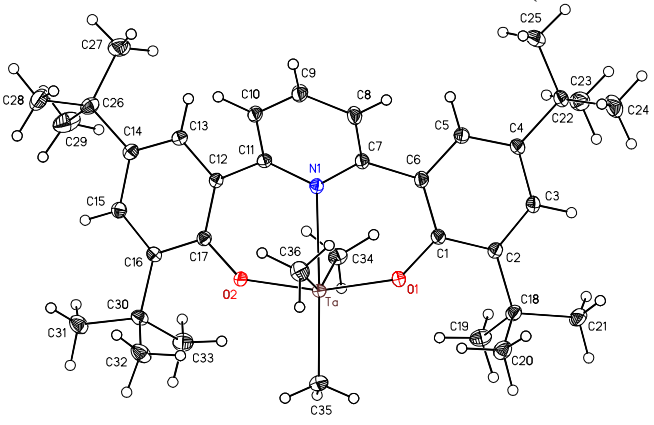
Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.65152-1-DOE.651520

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



TA17

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 279741. 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 279741."

Table 1. Crystal data and structure refinement for TA17 (CCDC 279741).

Empirical formula	$C_{36}H_{52}NO_2Ta$
Formula weight	711.74
Crystallization Solvent	Petroleum ether
Crystal Habit	Blade
Crystal size	0.23 x 0.21 x 0.08 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 24819 reflections used in lattice determination	2.25 to 46.45°	
Unit cell dimensions	a = 11.1983(3) Å b = 11.4259(3) Å c = 14.4511(4) Å	α= 88.7580(10)° β= 75.2440(10)° γ = 70.9590(10)°
Volume	1686.39(8) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.402 Mg/m^3	
F(000)	728	
Data collection program	Bruker SMART v5.630	
θ range for data collection	1.89 to 47.50°	
Completeness to θ = 47.50°	84.4 %	
Index ranges	$-22 \le h \le 23$, $-22 \le k \le 22$, $-25 \le l \le 29$	
Data collection scan type	ω scans at 7 ϕ settings	
Data reduction program	Bruker SAINT v6.45A	
Reflections collected	56267	
Independent reflections	26622 [R_{int}= 0.0468]	
Absorption coefficient	3.289 mm^{-1}	
Absorption correction	Face-indexed (SADABS)	
Max. and min. transmission	0.74943 and 0.42862 (0.885232)	

Table 1 (cont.)

Structure solution and Refinement

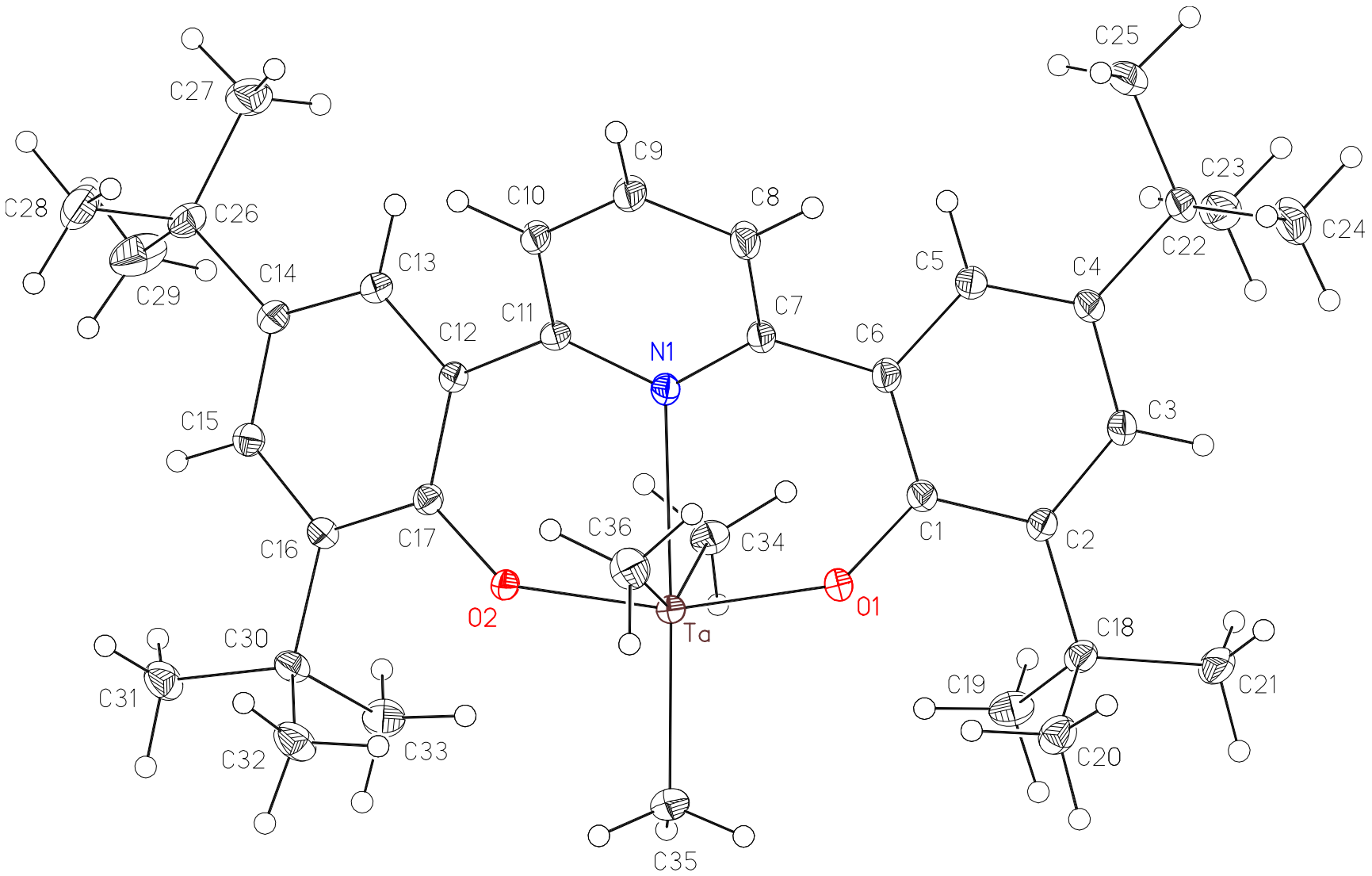
Structure solution program	Bruker XS v6.12
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	Bruker XL v6.12
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	26622 / 0 / 376
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.206
Final R indices [$I>2\sigma(I)$, 21140 reflections]	R1 = 0.0392, *w*R2 = 0.0703
R indices (all data)	R1 = 0.0594, *w*R2 = 0.0759
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.006
Average shift/error	0.000
Largest diff. peak and hole	3.346 and -2.410 e.Å^{-3}

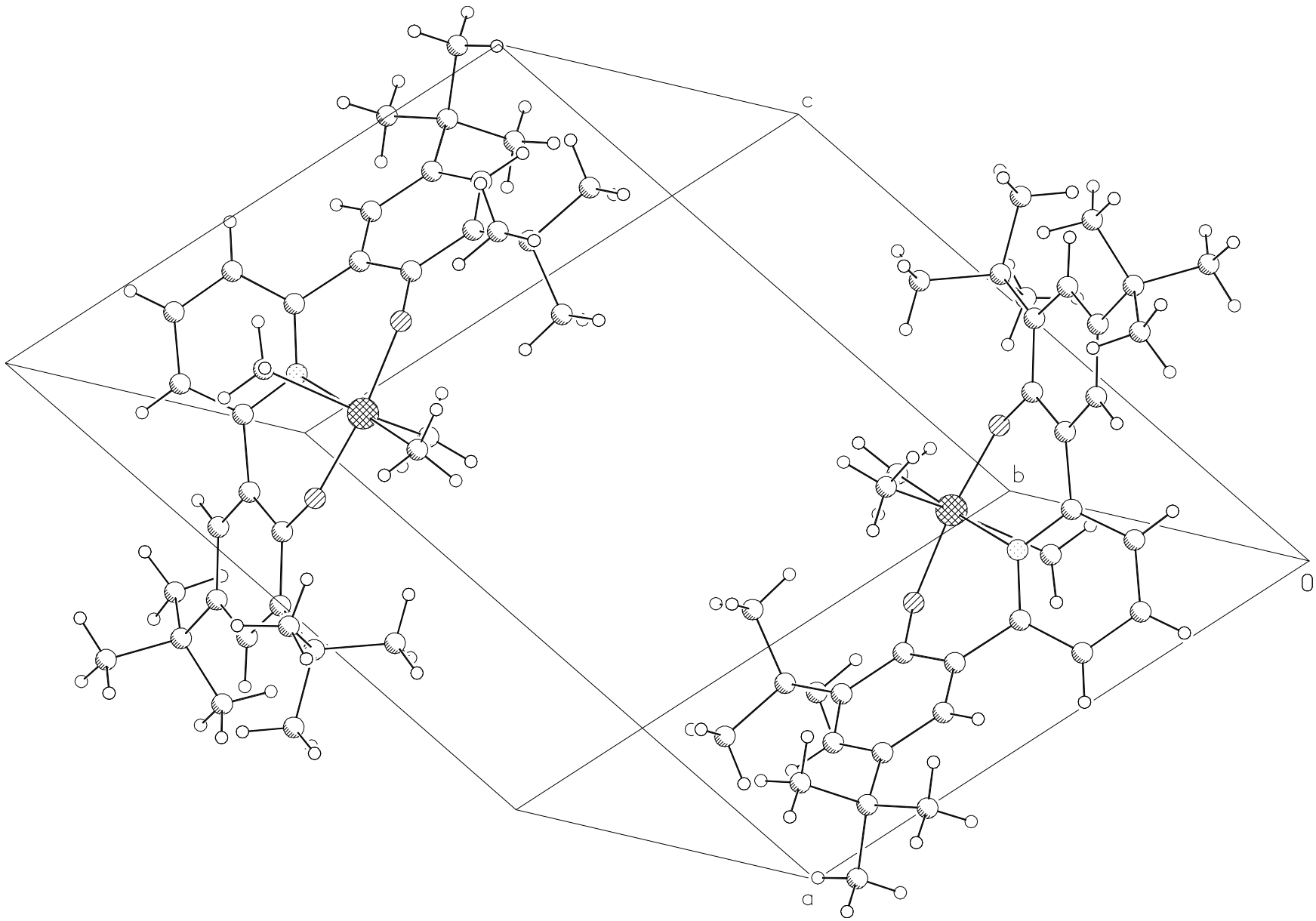
Special Refinement Details

All residual electron density peaks lie within 1Å of Ta and may be accounted for by an incomplete absorption correction model. Absorption corrections were based on measured faces and then finished with SADABS.

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.


C25
C27
C23
C28
C26
C9
C10
C8
C24
C22
C29
C14
C13
C5
C4
C7
C11
C12
N1
C6
C15
C3
C36
C34
C16
C17
C1
C2
O2
O1
Ta
C30
C18
C31
C19
C21
C32
C33
C20
C35


c
b
a
O

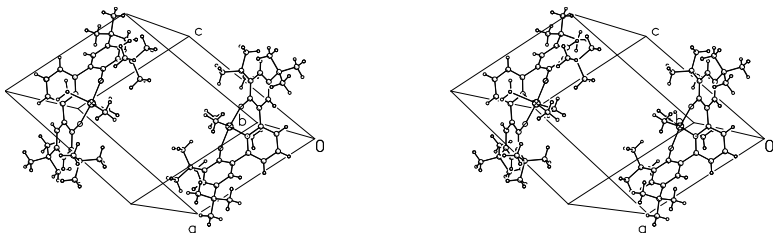

c
b
0
a
c
0
a

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for TA17 (CCDC 279741). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ta	6849(1)	8658(1)	6829(1)	11(1)
O(1)	5091(1)	8995(1)	7589(1)	14(1)
O(2)	8702(1)	8008(1)	6355(1)	13(1)
N(1)	7358(1)	7615(1)	8244(1)	13(1)
C(1)	4430(2)	8496(2)	8319(1)	13(1)
C(2)	3164(2)	8486(2)	8337(1)	14(1)
C(3)	2565(2)	7911(2)	9091(1)	15(1)
C(4)	3148(2)	7359(2)	9818(1)	14(1)
C(5)	4375(2)	7424(2)	9783(1)	15(1)
C(6)	5031(2)	7984(2)	9046(1)	14(1)
C(7)	6376(2)	7915(2)	9067(1)	14(1)
C(8)	6623(2)	8075(2)	9948(1)	18(1)
C(9)	7908(2)	7845(2)	9985(1)	19(1)
C(10)	8912(2)	7381(2)	9164(1)	16(1)
C(11)	8617(2)	7257(2)	8292(1)	13(1)
C(12)	9719(2)	6700(2)	7427(1)	13(1)
C(13)	10799(2)	5723(2)	7567(1)	14(1)
C(14)	11921(2)	5193(2)	6823(1)	14(1)
C(15)	11940(2)	5682(2)	5925(1)	14(1)
C(16)	10892(2)	6628(2)	5732(1)	13(1)
C(17)	9759(2)	7117(2)	6501(1)	12(1)
C(18)	2506(2)	9047(2)	7540(1)	14(1)
C(19)	3297(2)	8310(2)	6572(1)	20(1)
C(20)	2400(2)	10418(2)	7457(2)	19(1)
C(21)	1112(2)	8989(2)	7742(2)	22(1)
C(22)	2447(2)	6687(2)	10590(1)	16(1)
C(23)	2236(2)	5611(2)	10114(2)	22(1)
C(24)	1114(2)	7606(2)	11142(2)	26(1)
C(25)	3244(2)	6148(2)	11316(1)	23(1)
C(26)	13115(2)	4158(2)	6975(1)	17(1)
C(27)	12753(2)	3458(2)	7859(2)	24(1)
C(28)	14125(2)	4744(2)	7124(2)	26(1)
C(29)	13755(3)	3207(2)	6108(2)	30(1)
C(30)	10965(2)	7102(2)	4721(1)	14(1)
C(31)	12363(2)	6600(2)	4063(1)	20(1)
C(32)	10582(2)	8524(2)	4752(2)	21(1)
C(33)	10041(2)	6682(2)	4277(1)	21(1)
C(34)	6888(2)	6747(2)	6633(1)	17(1)
C(35)	6451(2)	9623(2)	5584(2)	21(1)
C(36)	7058(2)	10045(2)	7756(2)	20(1)

Table 3. Selected bond lengths [Å] and angles [°] for TA17 (CCDC 279741).

Ta-O(2)	1.9020(13)	O(2)-Ta-O(1)	163.45(5)
Ta-O(1)	1.9113(13)	O(2)-Ta-C(35)	97.26(7)
Ta-C(35)	2.1573(18)	O(1)-Ta-C(35)	99.06(7)
Ta-C(34)	2.1931(18)	O(2)-Ta-C(34)	85.92(7)
Ta-C(36)	2.2032(19)	O(1)-Ta-C(34)	86.31(7)
Ta-N(1)	2.4428(14)	C(35)-Ta-C(34)	108.42(7)
		O(2)-Ta-C(36)	89.60(7)
		O(1)-Ta-C(36)	88.97(7)
		C(35)-Ta-C(36)	104.62(8)
		C(34)-Ta-C(36)	146.95(7)
		O(2)-Ta-N(1)	81.48(5)
		O(1)-Ta-N(1)	82.32(5)
		C(35)-Ta-N(1)	177.61(7)
		C(34)-Ta-N(1)	73.56(6)
		C(36)-Ta-N(1)	73.40(6)

Table 4. Bond lengths [Å] and angles [°] for TA17 (CCDC 279741).

Ta-O(2)	1.9020(13)	O(2)-Ta-C(36)	89.60(7)
Ta-O(1)	1.9113(13)	O(1)-Ta-C(36)	88.97(7)
Ta-C(35)	2.1573(18)	C(35)-Ta-C(36)	104.62(8)
Ta-C(34)	2.1931(18)	C(34)-Ta-C(36)	146.95(7)
Ta-C(36)	2.2032(19)	O(2)-Ta-N(1)	81.48(5)
Ta-N(1)	2.4428(14)	O(1)-Ta-N(1)	82.32(5)
O(1)-C(1)	1.356(2)	C(35)-Ta-N(1)	177.61(7)
O(2)-C(17)	1.3469(19)	C(34)-Ta-N(1)	73.56(6)
N(1)-C(11)	1.354(2)	C(36)-Ta-N(1)	73.40(6)
N(1)-C(7)	1.360(2)	C(1)-O(1)-Ta	138.09(11)
C(1)-C(6)	1.406(2)	C(17)-O(2)-Ta	141.66(11)
C(1)-C(2)	1.414(2)	C(11)-N(1)-C(7)	119.43(14)
C(2)-C(3)	1.398(2)	C(11)-N(1)-Ta	117.63(11)
C(2)-C(18)	1.540(2)	C(7)-N(1)-Ta	116.39(11)
C(3)-C(4)	1.407(2)	O(1)-C(1)-C(6)	118.73(15)
C(4)-C(5)	1.389(2)	O(1)-C(1)-C(2)	120.13(14)
C(4)-C(22)	1.536(2)	C(6)-C(1)-C(2)	121.14(16)
C(5)-C(6)	1.401(3)	C(3)-C(2)-C(1)	116.89(14)
C(6)-C(7)	1.490(2)	C(3)-C(2)-C(18)	121.94(15)
C(7)-C(8)	1.397(2)	C(1)-C(2)-C(18)	121.13(15)
C(8)-C(9)	1.390(3)	C(2)-C(3)-C(4)	123.68(16)
C(9)-C(10)	1.382(3)	C(5)-C(4)-C(3)	117.18(16)
C(10)-C(11)	1.403(2)	C(5)-C(4)-C(22)	122.70(15)
C(11)-C(12)	1.493(2)	C(3)-C(4)-C(22)	120.10(16)
C(12)-C(17)	1.405(2)	C(4)-C(5)-C(6)	122.00(15)
C(12)-C(13)	1.408(2)	C(5)-C(6)-C(1)	119.06(16)
C(13)-C(14)	1.395(2)	C(5)-C(6)-C(7)	116.07(15)
C(14)-C(15)	1.400(2)	C(1)-C(6)-C(7)	124.75(16)
C(14)-C(26)	1.530(2)	N(1)-C(7)-C(8)	121.07(16)
C(15)-C(16)	1.396(2)	N(1)-C(7)-C(6)	119.67(14)
C(16)-C(17)	1.415(2)	C(8)-C(7)-C(6)	119.10(16)
C(16)-C(30)	1.538(2)	C(9)-C(8)-C(7)	119.05(17)
C(18)-C(20)	1.536(2)	C(10)-C(9)-C(8)	119.11(16)
C(18)-C(21)	1.537(3)	C(9)-C(10)-C(11)	119.70(16)
C(18)-C(19)	1.544(3)	N(1)-C(11)-C(10)	120.52(16)
C(22)-C(23)	1.532(3)	N(1)-C(11)-C(12)	120.92(14)
C(22)-C(25)	1.538(3)	C(10)-C(11)-C(12)	118.54(15)
C(22)-C(24)	1.541(3)	C(17)-C(12)-C(13)	118.76(15)
C(26)-C(27)	1.530(3)	C(17)-C(12)-C(11)	124.15(14)
C(26)-C(29)	1.535(3)	C(13)-C(12)-C(11)	117.04(14)
C(26)-C(28)	1.549(3)	C(14)-C(13)-C(12)	121.91(15)
C(30)-C(31)	1.538(3)	C(13)-C(14)-C(15)	117.17(15)
C(30)-C(32)	1.538(2)	C(13)-C(14)-C(26)	122.32(15)
C(30)-C(33)	1.545(3)	C(15)-C(14)-C(26)	120.46(16)
		C(16)-C(15)-C(14)	123.78(16)
O(2)-Ta-O(1)	163.45(5)	C(15)-C(16)-C(17)	117.17(15)
O(2)-Ta-C(35)	97.26(7)	C(15)-C(16)-C(30)	121.16(15)
O(1)-Ta-C(35)	99.06(7)	C(17)-C(16)-C(30)	121.66(14)
O(2)-Ta-C(34)	85.92(7)	O(2)-C(17)-C(12)	118.87(15)
O(1)-Ta-C(34)	86.31(7)	O(2)-C(17)-C(16)	120.06(14)
C(35)-Ta-C(34)	108.42(7)	C(12)-C(17)-C(16)	121.07(14)

C(20)-C(18)-C(21)	107.22(15)	C(14)-C(26)-C(27)	111.75(17)
C(20)-C(18)-C(2)	110.94(14)	C(14)-C(26)-C(29)	111.47(15)
C(21)-C(18)-C(2)	111.62(15)	C(27)-C(26)-C(29)	107.64(17)
C(20)-C(18)-C(19)	109.70(16)	C(14)-C(26)-C(28)	108.63(15)
C(21)-C(18)-C(19)	107.32(15)	C(27)-C(26)-C(28)	108.20(16)
C(2)-C(18)-C(19)	109.94(14)	C(29)-C(26)-C(28)	109.06(19)
C(23)-C(22)-C(4)	109.59(15)	C(31)-C(30)-C(32)	105.87(15)
C(23)-C(22)-C(25)	107.87(16)	C(31)-C(30)-C(16)	111.66(14)
C(4)-C(22)-C(25)	112.06(15)	C(32)-C(30)-C(16)	111.21(14)
C(23)-C(22)-C(24)	109.62(17)	C(31)-C(30)-C(33)	108.96(15)
C(4)-C(22)-C(24)	109.45(16)	C(32)-C(30)-C(33)	109.96(16)
C(25)-C(22)-C(24)	108.21(16)	C(16)-C(30)-C(33)	109.11(15)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for TA17 (CCDC 279741). 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	104(1)	125(1)	105(1)	19(1)	-29(1)	-32(1)
O(1)	107(5)	173(5)	138(5)	32(4)	-22(4)	-52(4)
O(2)	92(5)	167(5)	117(5)	25(4)	-17(4)	-16(4)
N(1)	109(5)	176(6)	116(5)	16(4)	-31(4)	-56(5)
C(1)	121(6)	148(6)	116(6)	8(4)	-25(5)	-46(5)
C(2)	118(6)	154(6)	142(6)	12(5)	-41(5)	-44(5)
C(3)	117(6)	190(7)	154(7)	20(5)	-34(5)	-66(5)
C(4)	128(6)	176(6)	111(6)	11(5)	-15(5)	-53(5)
C(5)	133(7)	212(7)	114(6)	12(5)	-34(5)	-57(6)
C(6)	110(6)	194(7)	120(6)	0(5)	-20(5)	-47(5)
C(7)	111(6)	192(7)	116(6)	14(5)	-24(5)	-56(5)
C(8)	132(7)	290(9)	116(7)	-5(6)	-15(5)	-85(6)
C(9)	151(7)	318(9)	124(7)	16(6)	-57(6)	-95(7)
C(10)	130(7)	277(8)	115(6)	25(5)	-54(5)	-92(6)
C(11)	124(6)	178(6)	108(6)	22(5)	-44(5)	-58(5)
C(12)	109(6)	157(6)	120(6)	20(5)	-35(5)	-48(5)
C(13)	133(6)	174(6)	133(6)	30(5)	-47(5)	-48(5)
C(14)	132(6)	129(6)	154(7)	10(5)	-47(5)	-32(5)
C(15)	129(6)	140(6)	138(6)	10(5)	-25(5)	-33(5)
C(16)	130(6)	123(5)	118(6)	11(4)	-22(5)	-46(5)
C(17)	113(6)	133(5)	113(6)	2(4)	-27(5)	-33(5)
C(18)	140(7)	141(6)	171(7)	27(5)	-68(5)	-55(5)
C(19)	252(9)	201(7)	161(7)	6(6)	-96(7)	-64(7)
C(20)	178(8)	152(6)	256(9)	31(6)	-83(7)	-49(6)
C(21)	183(8)	275(9)	281(9)	110(7)	-132(7)	-123(7)
C(22)	132(7)	211(7)	128(7)	18(5)	-13(5)	-63(6)
C(23)	235(9)	213(8)	233(9)	43(7)	-84(7)	-96(7)
C(24)	193(9)	286(10)	226(10)	10(7)	44(7)	-57(8)
C(25)	232(9)	329(10)	156(8)	84(7)	-57(7)	-131(8)
C(26)	161(7)	147(6)	183(7)	4(5)	-64(6)	-6(5)
C(27)	256(10)	176(7)	260(10)	66(7)	-78(8)	-26(7)
C(28)	169(8)	249(9)	365(11)	63(8)	-123(8)	-55(7)
C(29)	331(12)	220(8)	229(10)	-53(7)	-100(9)	75(8)
C(30)	164(7)	135(6)	117(6)	15(5)	-15(5)	-42(5)
C(31)	192(8)	190(7)	156(8)	4(6)	19(6)	-35(6)
C(32)	234(9)	146(7)	200(8)	34(6)	13(7)	-44(6)
C(33)	234(9)	270(9)	148(8)	1(6)	-64(7)	-85(7)
C(34)	189(8)	167(6)	160(7)	8(5)	-65(6)	-46(6)
C(35)	180(8)	237(8)	200(8)	84(6)	-67(7)	-38(7)
C(36)	214(9)	193(7)	216(8)	6(6)	-53(7)	-87(7)