# Alkyne Hydroamination and Trimerization with Titanium *Bis*(phenolate)pyridine Complexes: Evidence for Low-Valent Titanium Intermediates and Synthesis of an Ethylene Adduct of Ti(II)

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## Supporting Information

### Contents

**Figure S1.** $^{1}$H NMR spectrum of **2** ($C_6D_6$).

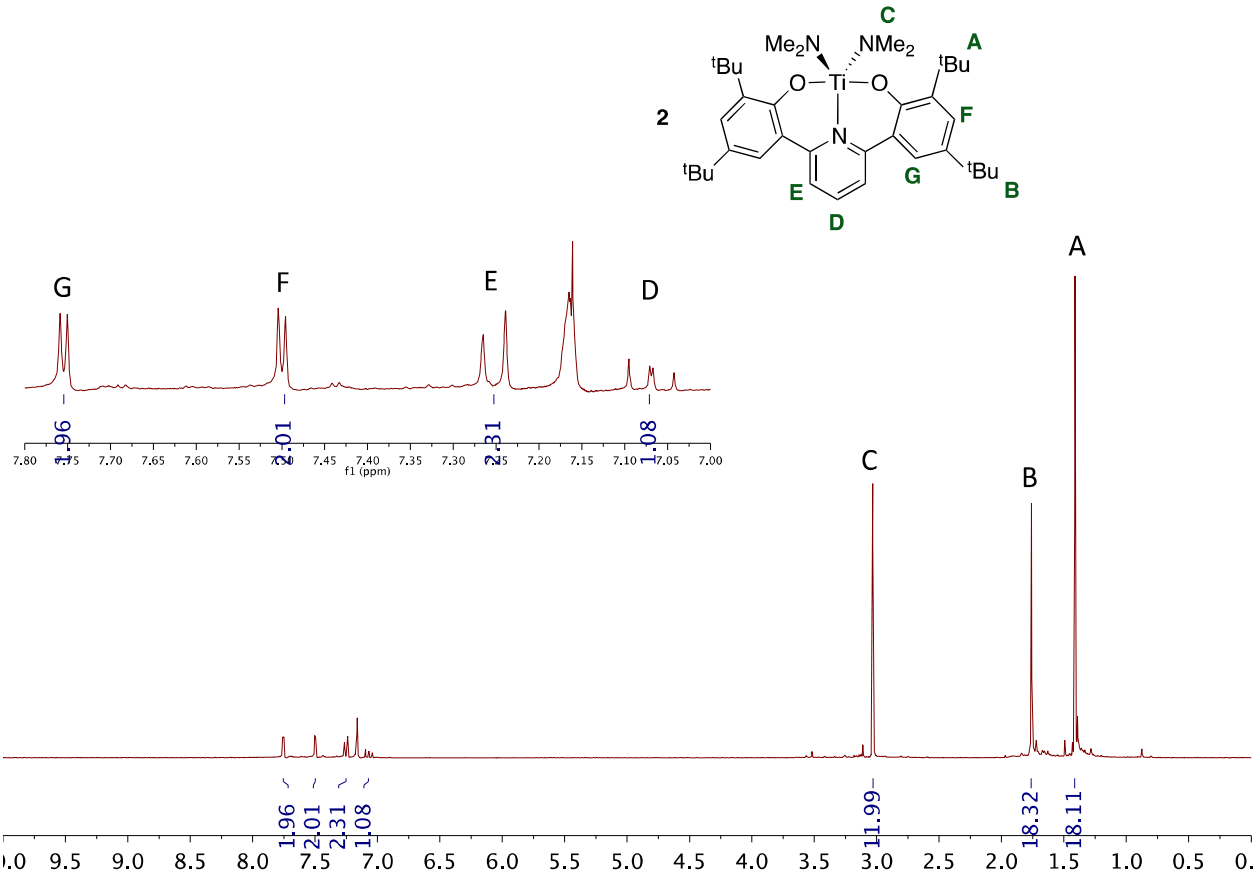

**Figure S2.** $^{1}$H NMR spectrum of **3** ($C_6D_6$).

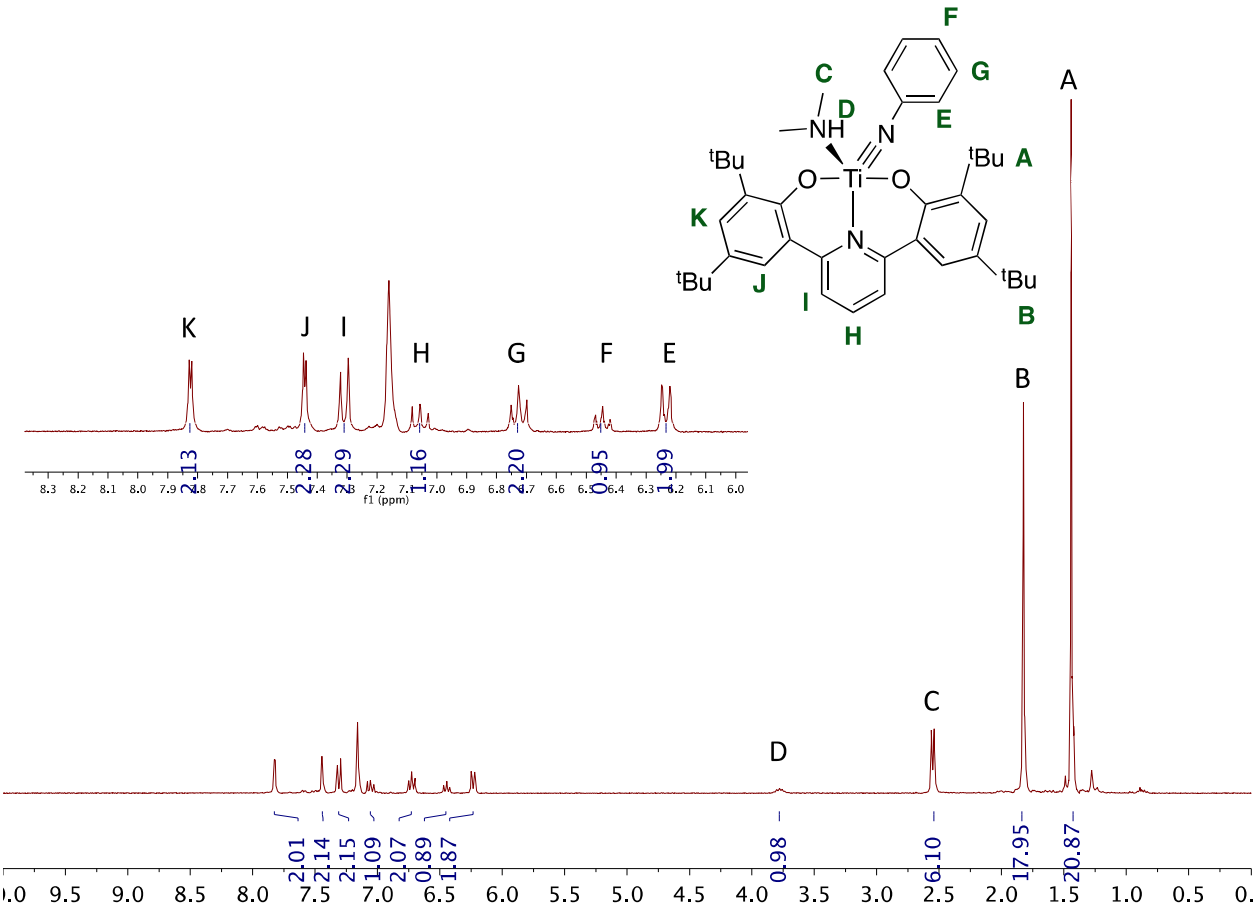

**Figure S3.** $^{1}$H NMR spectrum of **4** ($C_6D_6$).

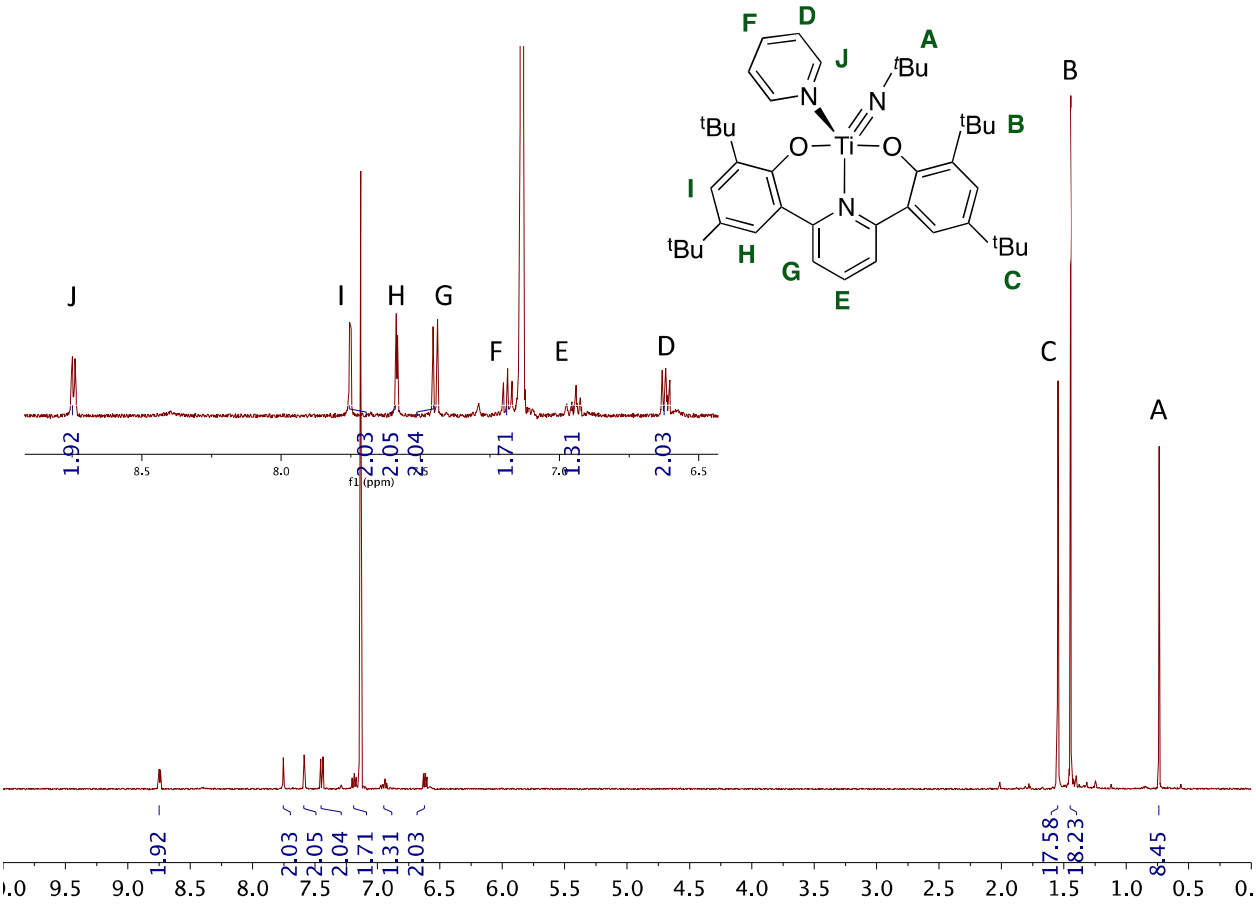

**Figure S4.** $^{1}$H NMR spectrum of **6** ($C_6D_6$).

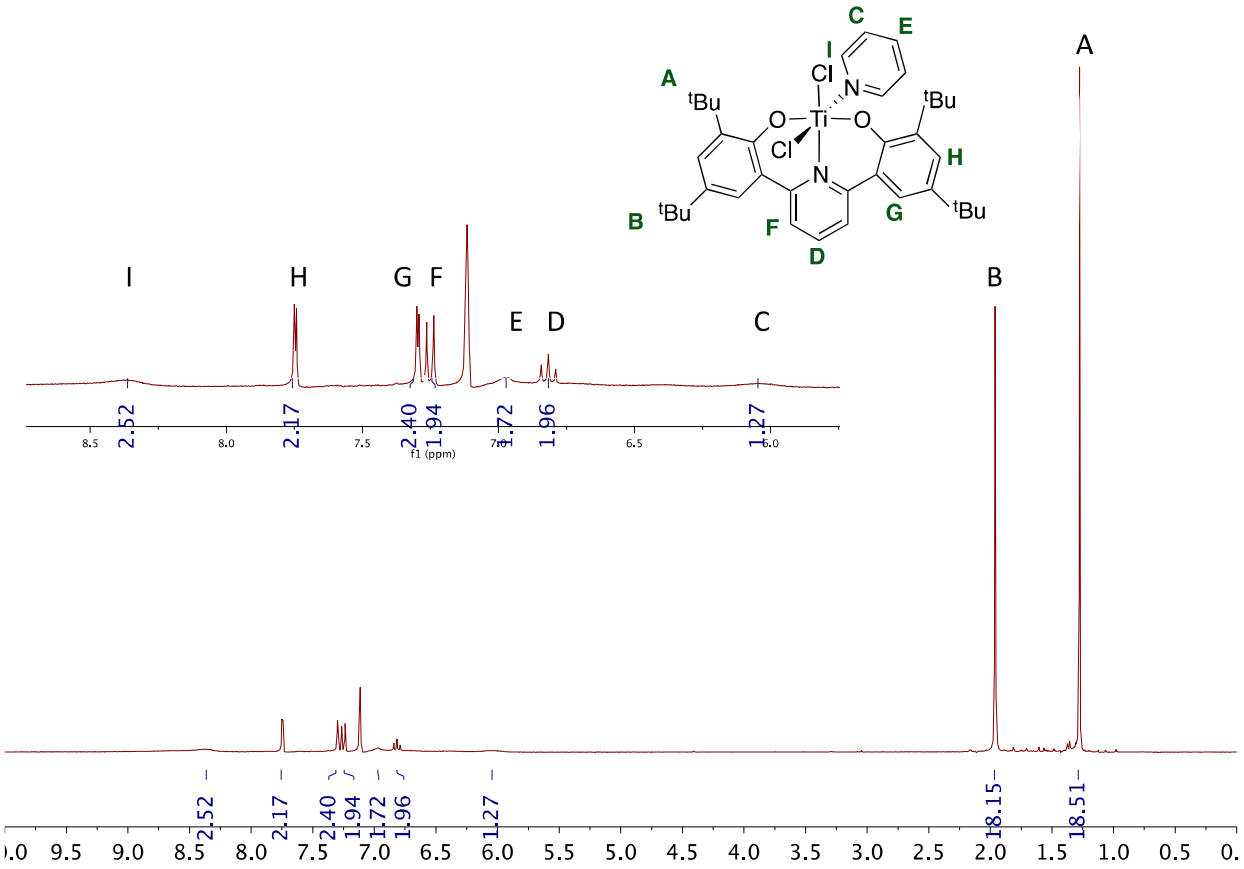

**Table S1.** Crystal and refinement data for complexes **2**, **4**, **6**, and **7**.

| | **2** | **4** | **6** | **7** |
|---|---|---|---|---|
| CCDC Number | 754732 | 847140 | 856446 | 822994 |
| Empirical formula | $C_{37}H_{55}N_3O_2Ti \bullet C_5H_{12}$ | $C_{42}H_{57}N_3O_2Ti \bullet 0.49(C_5H_{12}) \bullet 0.51(C_7H_8)$ | $2(C_{38}H_{48}N_2O_2Cl_2Ti) \bullet 5(C_6H_6)$ | $C_{37}H_{53}N_2O_2Ti \bullet 0.5(C_5H_{12})$ |
| Formula weight | 693.89 | 765.95 | 878.85 | 641.79 |
| T (K) | 100(2) | 100(2) | 100(2) | 100(2) |
| *a*, Å | 13.9103(6) | 14.6930(8) | 11.8721(6) | 17.7536(5) |
| *b*, Å | 10.2228(4) | 14.9636(8) | 28.5845(14) | 10.5239(3) |
| *c*, Å | 28.5988(12) | 20.1744(12) | 29.6698(15) | 20.7880(6) |
| α, deg | | | | |
| β, deg | 90.995(3) | 92.699(3) | 100.018(3) | 104.343(2) |
| γ, deg | | | | |
| Volume, Å$^3$ | 4066.2(3) | 4430.6(4) | 9915.2(9) | 3762.91(19) |
| Z | 4 | 4 | 8 | 4 |
| Crystal system | Monoclinic | Monoclinic | Monoclinic | Monoclinic |
| Space group | P $2_1/n$ | P $2_1/c$ | P $2_1/c$ | P $2_1/n$ |
| $d_{calc}$, g/cm$^3$ | 1.133 | 1.148 | 1.177 | 1.133 |
| θ range, deg | 1.62 to 29.10 | 1.70 to 30.51 | 1.59 to 35.13 | 2.02 to 29.99 |
| $\mu$, mm$^{-1}$ | 0.247 | 0.233 | 0.320 | 0.261 |
| Abs. Correction | None | None | None | None |
| GOF | 1.764 | 1.945 | 1.877 | 2.095 |
| $R_1$ ,[a] $wR_2$ [b] [I>2σ(I)] | R1 = 0.0650, *w*R2 = 0.0942 | R1 = 0.0584, *w*R2 = 0.0723 | R1 = 0.0415, *w*R2 = 0.0625 | R1 = 0.0612, *w*R2 = 0.0614 |

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR_2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$.

# CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 13 November 2009

**Crystal Structure Analysis of:**

**Complex 2 (ONO)Ti($NMe_2$)$_2$ (IAT26)**

(shown below)

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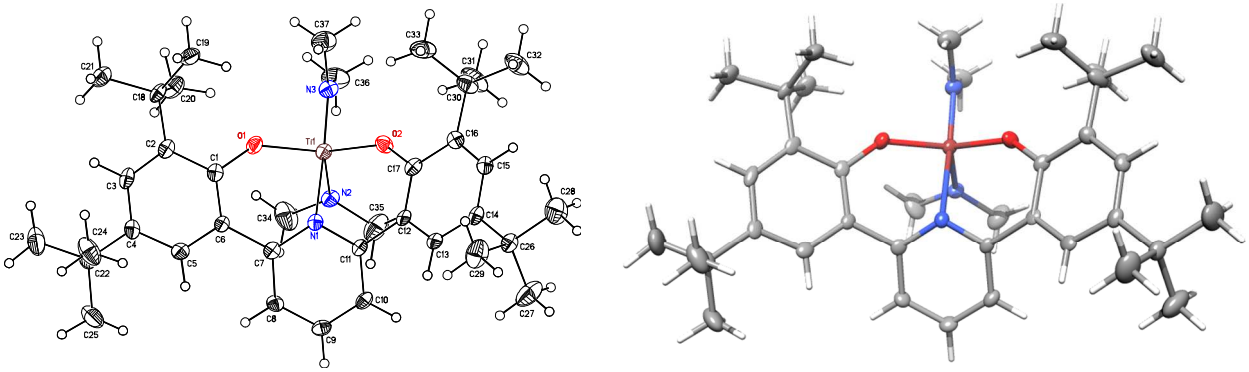
Contents






IAT26

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 754732.

**Table 1. Crystal data and structure refinement for IAT26 (CCDC 754732).**

| | |
|---|---|
| Empirical formula | $C_{37}H_{55}N_3O_2Ti \bullet C_5H_{12}$ |
| Formula weight | 693.89 |
| Crystallization Solvent | Pentane |
| Crystal Habit | Column |
| Crystal size | 0.39 x 0.14 x 0.09 $mm^3$ |
| Crystal color | Yellow |

## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker KAPPA APEX II | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 9954 reflections used in lattice determination | 2.45 to 24.33° | |
| Unit cell dimensions | a = 13.9103(6) Å<br>b = 10.2228(4) Å<br>c = 28.5988(12) Å | α= 90°<br>β= 90.995(3)°<br>γ = 90° |
| Volume | 4066.2(3) $Å^3$ | |
| Z | 4 | |
| Crystal system | Monoclinic | |
| Space group | P $2_1/n$ | |
| Density (calculated) | 1.133 $Mg/m^3$ | |
| F(000) | 1512 | |
| Data collection program | Bruker APEX2 v2009.7-0 | |
| θ range for data collection | 1.62 to 29.10° | |
| Completeness to θ = 29.10° | 90.2 % | |
| Index ranges | $-17 \le h \le 18$, $-13 \le k \le 13$, $-38 \le l \le 36$ | |
| Data collection scan type | ω scans; 8 settings | |
| Data reduction program | Bruker SAINT-Plus v7.66A | |
| Reflections collected | 51468 | |
| Independent reflections | 9823 [$R_{int}$= 0.0899] | |
| Absorption coefficient | 0.247 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9781 and 0.9099 | |

**Table 1 (cont.)**

## Structure solution and Refinement

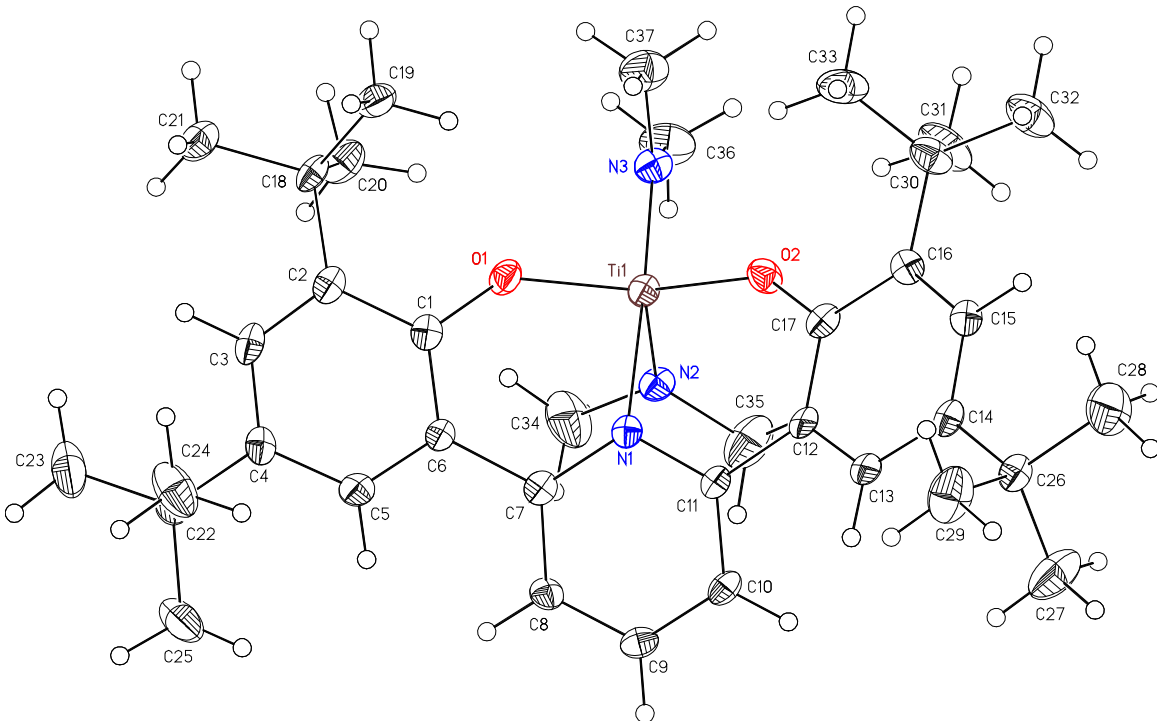
| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Geometric positions |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 9823 / 0 / 451 |
| Treatment of hydrogen atoms | Riding |
| Goodness-of-fit on $F^2$ | 1.764 |
| Final R indices [$I>2\sigma(I)$, 6111 reflections] | R1 = 0.0650, *w*R2 = 0.0942 |
| R indices (all data) | R1 = 0.1168, *w*R2 = 0.0977 |
| Type of weighting scheme used | Sigma |
| Weighting scheme used | $w=1/\sigma^2(Fo^2)$ |
| Max shift/error | 0.001 |
| Average shift/error | 0.000 |
| Largest diff. peak and hole | 0.895 and -0.736 e.Å$^{-3}$ |

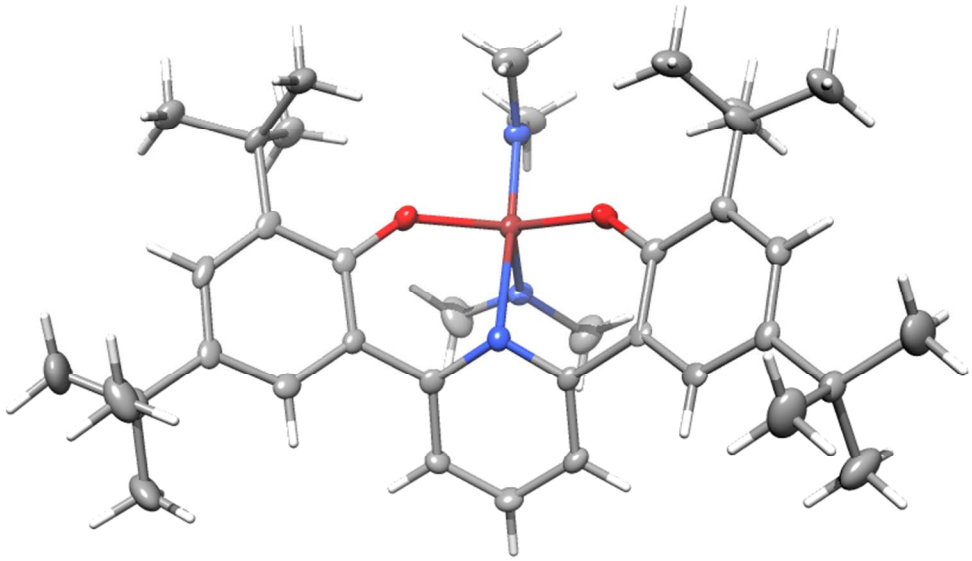
## Special Refinement Details

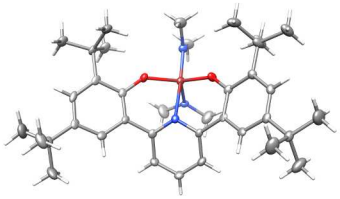
Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

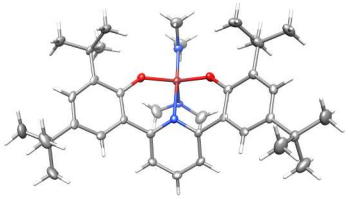
Refinement of $F^2$ against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on $F^2$, conventional R-factors (R) are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and 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.

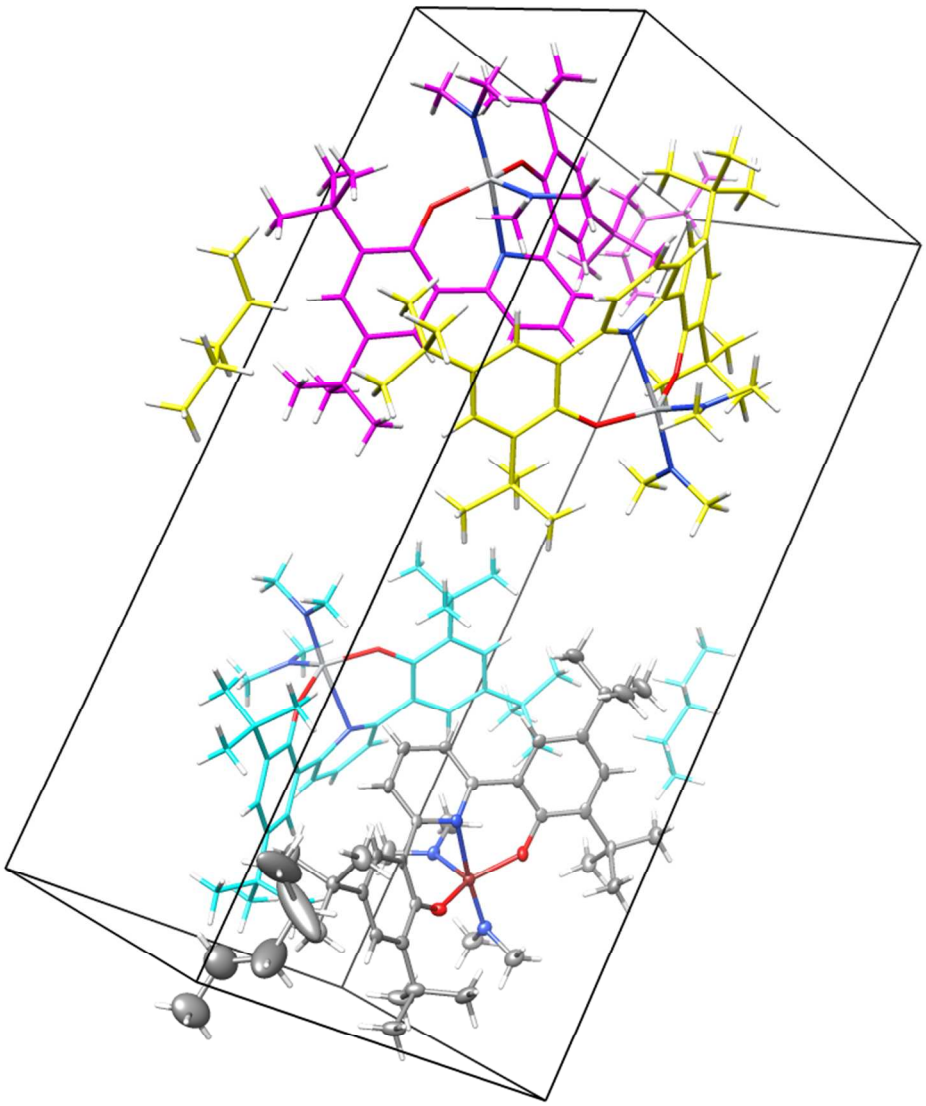
All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when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

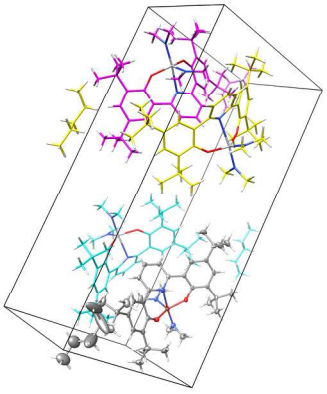

C37
C33
C19
C21
C18
C20
N3
C36
C31
C32
C30
O1
Ti1
O2
C16
C2
C1
C17
C15
C3
N2
C28
C35
C34
C14
C24
C23
C4
C6
N1
C12
C26
C5
C7
C11
C13
C29
C22
C10
C8
C27
C25
C9











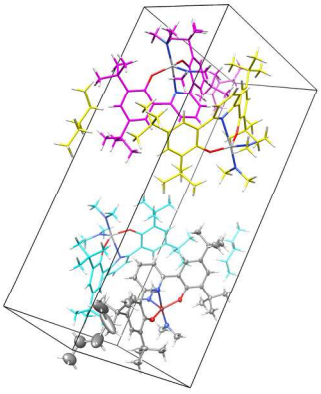


**Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for IAT26 (CCDC 754732). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.**

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| Ti(1) | 9254(1) | 4380(1) | 8263(1) | 17(1) |
| O(1) | 9890(1) | 5170(1) | 7765(1) | 22(1) |
| O(2) | 8338(1) | 5010(1) | 8677(1) | 22(1) |
| N(1) | 7928(1) | 4731(2) | 7734(1) | 16(1) |
| N(2) | 9142(1) | 2584(2) | 8154(1) | 19(1) |
| N(3) | 10320(1) | 4386(2) | 8687(1) | 22(1) |
| C(1) | 9782(2) | 5404(2) | 7303(1) | 16(1) |
| C(2) | 10533(2) | 6045(2) | 7065(1) | 18(1) |
| C(3) | 10394(2) | 6281(2) | 6589(1) | 20(1) |
| C(4) | 9577(2) | 5907(2) | 6335(1) | 19(1) |
| C(5) | 8873(2) | 5250(2) | 6581(1) | 19(1) |
| C(6) | 8959(2) | 4986(2) | 7060(1) | 15(1) |
| C(7) | 8137(2) | 4356(2) | 7290(1) | 16(1) |
| C(8) | 7549(2) | 3461(2) | 7048(1) | 19(1) |
| C(9) | 6724(2) | 3020(2) | 7250(1) | 21(1) |
| C(10) | 6451(2) | 3521(2) | 7677(1) | 19(1) |
| C(11) | 7064(2) | 4398(2) | 7908(1) | 14(1) |
| C(12) | 6754(2) | 5039(2) | 8349(1) | 15(1) |
| C(13) | 5790(2) | 5375(2) | 8392(1) | 16(1) |
| C(14) | 5445(2) | 5991(2) | 8789(1) | 17(1) |
| C(15) | 6112(2) | 6271(2) | 9141(1) | 18(1) |
| C(16) | 7081(2) | 5971(2) | 9121(1) | 18(1) |
| C(17) | 7408(2) | 5352(2) | 8710(1) | 16(1) |
| C(18) | 11466(2) | 6444(2) | 7326(1) | 20(1) |
| C(19) | 11243(2) | 7497(2) | 7692(1) | 28(1) |
| C(20) | 11927(2) | 5242(2) | 7561(1) | 32(1) |
| C(21) | 12213(2) | 7018(2) | 6992(1) | 29(1) |
| C(22) | 9441(2) | 6244(2) | 5816(1) | 22(1) |
| C(23) | 10396(2) | 6296(3) | 5569(1) | 46(1) |
| C(24) | 8957(2) | 7588(2) | 5771(1) | 40(1) |
| C(25) | 8806(2) | 5239(2) | 5567(1) | 40(1) |
| C(26) | 4373(2) | 6340(2) | 8820(1) | 21(1) |
| C(27) | 3761(2) | 5127(2) | 8731(1) | 45(1) |
| C(28) | 4112(2) | 6883(3) | 9300(1) | 45(1) |
| C(29) | 4118(2) | 7370(2) | 8448(1) | 42(1) |
| C(30) | 7780(2) | 6311(2) | 9526(1) | 26(1) |
| C(31) | 8329(2) | 5085(2) | 9697(1) | 40(1) |
| C(32) | 7261(2) | 6878(2) | 9949(1) | 35(1) |
| C(33) | 8490(2) | 7355(2) | 9357(1) | 37(1) |
| C(34) | 9678(2) | 1938(2) | 7789(1) | 44(1) |
| C(35) | 8424(2) | 1745(2) | 8354(1) | 42(1) |
| C(36) | 10909(2) | 3323(2) | 8885(1) | 38(1) |
| C(37) | 10666(2) | 5638(2) | 8854(1) | 36(1) |
| | | | | |
| C(41) | 1042(3) | 8522(3) | 10329(1) | 88(1) |
| C(42) | 1231(3) | 8503(3) | 9827(1) | 87(1) |
| C(43) | 1618(4) | 9583(5) | 9598(2) | 145(2) |
| C(44) | 1816(3) | 9515(6) | 9079(2) | 190(4) |
| C(45) | 981(4) | 9457(3) | 8790(1) | 135(2) |

**Table 3. Selected bond lengths [Å] and angles [°] for IAT26 (CCDC 754732).**

| | | | |
|---|---|---|---|
| Ti(1)-N(2) | 1.8683(17) | N(2)-Ti(1)-O(2) | 112.81(8) |
| Ti(1)-O(2) | 1.8688(15) | N(2)-Ti(1)-O(1) | 109.65(8) |
| Ti(1)-O(1) | 1.8727(15) | O(2)-Ti(1)-O(1) | 132.04(7) |
| Ti(1)-N(3) | 1.8986(19) | N(2)-Ti(1)-N(3) | 99.88(8) |
| Ti(1)-N(1) | 2.3917(18) | O(2)-Ti(1)-N(3) | 97.38(7) |
| | | O(1)-Ti(1)-N(3) | 96.33(8) |
| | | N(2)-Ti(1)-N(1) | 88.90(7) |
| | | O(2)-Ti(1)-N(1) | 79.71(7) |
| | | O(1)-Ti(1)-N(1) | 79.85(7) |
| | | N(3)-Ti(1)-N(1) | 171.19(7) |

**Table 4. Bond lengths [Å] and angles [°] for IAT26 (CCDC 754732).**

| | | | |
|---|---|---|---|
| Ti(1)-N(2) | 1.8683(17) | | |
| Ti(1)-O(2) | 1.8688(15) | N(2)-Ti(1)-O(2) | 112.81(8) |
| Ti(1)-O(1) | 1.8727(15) | N(2)-Ti(1)-O(1) | 109.65(8) |
| Ti(1)-N(3) | 1.8986(19) | O(2)-Ti(1)-O(1) | 132.04(7) |
| Ti(1)-N(1) | 2.3917(18) | N(2)-Ti(1)-N(3) | 99.88(8) |
| O(1)-C(1) | 1.350(3) | O(2)-Ti(1)-N(3) | 97.38(7) |
| O(2)-C(17) | 1.345(2) | O(1)-Ti(1)-N(3) | 96.33(8) |
| N(1)-C(11) | 1.353(3) | N(2)-Ti(1)-N(1) | 88.90(7) |
| N(1)-C(7) | 1.364(3) | O(2)-Ti(1)-N(1) | 79.71(7) |
| N(2)-C(35) | 1.442(3) | O(1)-Ti(1)-N(1) | 79.85(7) |
| N(2)-C(34) | 1.454(3) | N(3)-Ti(1)-N(1) | 171.19(7) |
| N(3)-C(37) | 1.446(3) | C(1)-O(1)-Ti(1) | 140.83(15) |
| N(3)-C(36) | 1.469(3) | C(17)-O(2)-Ti(1) | 143.37(15) |
| C(1)-C(6) | 1.396(3) | C(11)-N(1)-C(7) | 118.43(19) |
| C(1)-C(2) | 1.417(3) | C(11)-N(1)-Ti(1) | 114.26(14) |
| C(2)-C(3) | 1.394(3) | C(7)-N(1)-Ti(1) | 111.91(14) |
| C(2)-C(18) | 1.540(3) | C(35)-N(2)-C(34) | 112.50(19) |
| C(3)-C(4) | 1.391(3) | C(35)-N(2)-Ti(1) | 125.09(16) |
| C(4)-C(5) | 1.388(3) | C(34)-N(2)-Ti(1) | 121.59(16) |
| C(4)-C(22) | 1.533(3) | C(37)-N(3)-C(36) | 110.33(19) |
| C(5)-C(6) | 1.399(3) | C(37)-N(3)-Ti(1) | 117.76(15) |
| C(6)-C(7) | 1.478(3) | C(36)-N(3)-Ti(1) | 131.90(15) |
| C(7)-C(8) | 1.401(3) | O(1)-C(1)-C(6) | 120.7(2) |
| C(8)-C(9) | 1.369(3) | O(1)-C(1)-C(2) | 118.7(2) |
| C(9)-C(10) | 1.383(3) | C(6)-C(1)-C(2) | 120.5(2) |
| C(10)-C(11) | 1.396(3) | C(3)-C(2)-C(1) | 117.2(2) |
| C(11)-C(12) | 1.491(3) | C(3)-C(2)-C(18) | 122.0(2) |
| C(12)-C(13) | 1.391(3) | C(1)-C(2)-C(18) | 120.8(2) |
| C(12)-C(17) | 1.403(3) | C(4)-C(3)-C(2) | 124.1(2) |
| C(13)-C(14) | 1.391(3) | C(5)-C(4)-C(3) | 116.5(2) |
| C(14)-C(15) | 1.386(3) | C(5)-C(4)-C(22) | 121.5(2) |
| C(14)-C(26) | 1.537(3) | C(3)-C(4)-C(22) | 121.9(2) |
| C(15)-C(16) | 1.385(3) | C(4)-C(5)-C(6) | 122.7(2) |
| C(16)-C(17) | 1.416(3) | C(1)-C(6)-C(5) | 119.0(2) |
| C(16)-C(30) | 1.539(3) | C(1)-C(6)-C(7) | 123.1(2) |
| C(18)-C(20) | 1.536(3) | C(5)-C(6)-C(7) | 117.8(2) |
| C(18)-C(19) | 1.538(3) | N(1)-C(7)-C(8) | 120.8(2) |
| C(18)-C(21) | 1.541(3) | N(1)-C(7)-C(6) | 118.1(2) |
| C(22)-C(23) | 1.517(3) | C(8)-C(7)-C(6) | 121.0(2) |
| C(22)-C(25) | 1.524(3) | C(9)-C(8)-C(7) | 119.6(2) |
| C(22)-C(24) | 1.534(3) | C(8)-C(9)-C(10) | 119.6(2) |
| C(26)-C(27) | 1.522(3) | C(9)-C(10)-C(11) | 118.8(2) |
| C(26)-C(28) | 1.531(3) | N(1)-C(11)-C(10) | 121.8(2) |
| C(26)-C(29) | 1.533(3) | N(1)-C(11)-C(12) | 118.16(19) |
| C(30)-C(32) | 1.533(3) | C(10)-C(11)-C(12) | 120.0(2) |
| C(30)-C(33) | 1.538(3) | C(13)-C(12)-C(17) | 119.5(2) |
| C(30)-C(31) | 1.543(3) | C(13)-C(12)-C(11) | 118.5(2) |
| C(41)-C(42) | 1.464(4) | C(17)-C(12)-C(11) | 122.0(2) |
| C(42)-C(43) | 1.397(5) | C(14)-C(13)-C(12) | 122.0(2) |
| C(43)-C(44) | 1.515(6) | C(15)-C(14)-C(13) | 116.8(2) |
| C(44)-C(45) | 1.415(6) | C(15)-C(14)-C(26) | 123.2(2) |

| | |
|---|---|
| C(13)-C(14)-C(26) | 120.0(2) |
| C(14)-C(15)-C(16) | 124.3(2) |
| C(15)-C(16)-C(17) | 117.4(2) |
| C(15)-C(16)-C(30) | 121.5(2) |
| C(17)-C(16)-C(30) | 121.1(2) |
| O(2)-C(17)-C(12) | 120.0(2) |
| O(2)-C(17)-C(16) | 119.9(2) |
| C(12)-C(17)-C(16) | 120.0(2) |
| C(20)-C(18)-C(19) | 110.4(2) |
| C(20)-C(18)-C(2) | 110.03(18) |
| C(19)-C(18)-C(2) | 109.75(18) |
| C(20)-C(18)-C(21) | 107.20(19) |
| C(19)-C(18)-C(21) | 107.55(18) |
| C(2)-C(18)-C(21) | 111.8(2) |
| C(23)-C(22)-C(25) | 108.1(2) |
| C(23)-C(22)-C(4) | 111.4(2) |
| C(25)-C(22)-C(4) | 111.23(19) |
| C(23)-C(22)-C(24) | 108.5(2) |
| C(25)-C(22)-C(24) | 108.3(2) |
| C(4)-C(22)-C(24) | 109.20(18) |
| C(27)-C(26)-C(28) | 107.7(2) |
| C(27)-C(26)-C(29) | 108.8(2) |
| C(28)-C(26)-C(29) | 108.5(2) |
| C(27)-C(26)-C(14) | 110.00(18) |
| C(28)-C(26)-C(14) | 112.4(2) |
| C(29)-C(26)-C(14) | 109.44(19) |
| C(32)-C(30)-C(33) | 107.5(2) |
| C(32)-C(30)-C(16) | 112.2(2) |
| C(33)-C(30)-C(16) | 108.7(2) |
| C(32)-C(30)-C(31) | 107.1(2) |
| C(33)-C(30)-C(31) | 110.3(2) |
| C(16)-C(30)-C(31) | 110.99(18) |
| C(43)-C(42)-C(41) | 121.8(4) |
| C(42)-C(43)-C(44) | 120.1(5) |
| C(45)-C(44)-C(43) | 114.3(4) |

**Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for IAT26 (CCDC 754732). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12} ]$**

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Ti(1) | 175(2) | 140(2) | 207(2) | -11(2) | 26(2) | 5(2) |
| O(1) | 205(10) | 208(9) | 232(10) | 43(7) | 22(8) | -46(7) |
| O(2) | 202(10) | 253(9) | 197(10) | -48(7) | 8(8) | 40(7) |
| N(1) | 191(11) | 92(10) | 185(11) | 8(8) | 26(9) | 9(8) |
| N(2) | 207(12) | 120(10) | 256(13) | -22(9) | 21(10) | 0(9) |
| N(3) | 211(12) | 183(11) | 266(12) | -20(10) | 2(10) | 12(10) |
| C(1) | 203(14) | 74(12) | 214(14) | 1(10) | 42(11) | 44(10) |
| C(2) | 180(14) | 89(12) | 288(15) | -32(10) | 84(12) | 36(10) |
| C(3) | 245(15) | 74(12) | 294(16) | 3(11) | 132(13) | 5(10) |
| C(4) | 250(15) | 91(12) | 222(14) | -30(10) | 89(12) | 33(10) |
| C(5) | 213(14) | 117(13) | 246(15) | -52(10) | 25(12) | 11(10) |
| C(6) | 161(14) | 99(12) | 199(14) | -21(10) | 42(11) | 13(10) |
| C(7) | 154(13) | 113(12) | 219(14) | 19(11) | 5(11) | 50(10) |
| C(8) | 242(15) | 132(12) | 190(14) | -34(11) | 14(12) | -2(11) |
| C(9) | 235(15) | 114(12) | 264(16) | -15(11) | -12(12) | -30(10) |
| C(10) | 165(14) | 129(12) | 266(15) | 26(11) | 24(12) | -14(10) |
| C(11) | 130(12) | 97(11) | 193(13) | 47(10) | 5(10) | 25(10) |
| C(12) | 190(14) | 49(11) | 203(14) | 38(10) | 30(11) | -36(10) |
| C(13) | 215(14) | 85(13) | 192(14) | 48(10) | 5(11) | -39(10) |
| C(14) | 185(14) | 96(12) | 243(15) | 48(10) | 48(12) | 6(10) |
| C(15) | 231(15) | 102(12) | 220(14) | -7(10) | 52(12) | 16(10) |
| C(16) | 218(14) | 122(12) | 194(14) | 22(10) | 2(12) | 31(10) |
| C(17) | 161(13) | 82(12) | 247(14) | 43(10) | 6(12) | 25(10) |
| C(18) | 126(13) | 173(13) | 304(16) | 16(11) | 58(12) | -1(10) |
| C(19) | 236(15) | 268(15) | 330(17) | -44(12) | 14(13) | -78(12) |
| C(20) | 200(15) | 315(16) | 431(18) | 59(13) | -2(14) | 13(11) |
| C(21) | 197(15) | 296(15) | 382(18) | -16(13) | 47(14) | -25(11) |
| C(22) | 307(16) | 143(13) | 226(15) | -29(11) | 62(13) | 3(11) |
| C(23) | 490(20) | 620(20) | 282(18) | 40(15) | 152(16) | -25(16) |
| C(24) | 670(20) | 278(16) | 257(17) | 1(13) | 57(16) | 115(15) |
| C(25) | 690(20) | 317(16) | 203(16) | -31(12) | 8(15) | -111(14) |
| C(26) | 192(14) | 162(13) | 267(15) | -1(11) | 50(12) | 17(11) |
| C(27) | 244(17) | 363(17) | 740(20) | -57(15) | 106(16) | -36(13) |
| C(28) | 279(18) | 620(20) | 470(20) | -113(16) | 107(16) | 90(15) |
| C(29) | 274(17) | 471(18) | 510(20) | 111(16) | 61(16) | 107(14) |
| C(30) | 261(16) | 275(15) | 247(16) | -82(12) | -30(13) | 87(12) |
| C(31) | 432(19) | 485(17) | 266(17) | -82(14) | -106(15) | 239(15) |
| C(32) | 348(18) | 428(17) | 274(17) | -127(13) | -38(14) | 149(13) |
| C(33) | 265(17) | 416(17) | 430(20) | -229(14) | -34(15) | 15(13) |
| C(34) | 660(20) | 281(16) | 376(19) | -76(13) | 142(17) | 90(14) |
| C(35) | 374(19) | 237(16) | 650(20) | 58(14) | 107(17) | -28(13) |
| C(36) | 354(18) | 352(16) | 432(19) | -19(14) | -139(15) | 75(13) |
| C(37) | 277(16) | 360(16) | 433(18) | -71(14) | -29(14) | 28(13) |
| | | | | | | |
| C(41) | 1290(40) | 630(20) | 730(30) | 80(20) | 70(30) | 220(20) |
| C(42) | 1150(40) | 610(30) | 860(30) | 20(20) | 110(30) | 30(20) |
| C(43) | 1620(50) | 1520(50) | 1200(50) | 760(40) | -740(40) | -760(40) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(44) | 850(40) | 3320(80) | 1560(60) | 1700(60) | 630(40) | 1130(50) |
| C(45) | 3060(80) | 400(20) | 600(30) | -130(20) | 290(40) | -200(30) |

# CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 5 October 2011

**Crystal Structure Analysis of:**

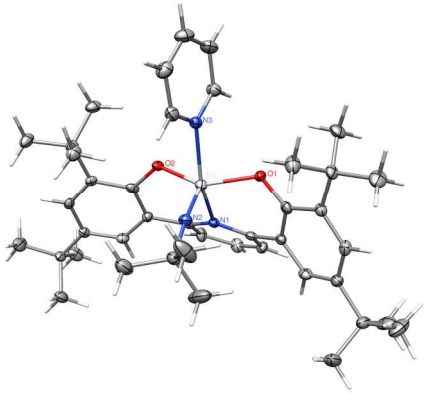
**Complex 4 (ONO)Ti(py)(N$^t$Bu) (IAT50)**

(shown below)

**By** Michael W. Day 116 Beckman ext. 2734
e-mail: mikeday@caltech.edu

Contents





IAT50

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 847140.

**Table 1. Crystal data and structure refinement for IAT50 (CCDC 847140).**

| | |
|---|---|
| Empirical formula | $C_{42}H_{57}N_3O_2Ti \bullet 0.49(C_5H_{12}) \bullet 0.51(C_7H_8)$ |
| Formula weight | 765.95 |
| Crystallization Solvent | Toluene/pentane |
| Crystal Habit | Block |
| Crystal size | 0.17 x 0.14 x 0.08 $mm^3$ |
| Crystal color | Yellow |

## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker KAPPA APEX II | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 9969 reflections used in lattice determination | 2.16 to 28.75° | |
| Unit cell dimensions | a = 14.6930(8) Å<br>b = 14.9636(8) Å<br>c = 20.1744(12) Å | α= 90°<br>β= 92.699(3)°<br>γ = 90° |
| Volume | 4430.6(4) $Å^3$ | |
| Z | 4 | |
| Crystal system | Monoclinic | |
| Space group | P $2_1/c$ | |
| Density (calculated) | 1.148 $Mg/m^3$ | |
| F(000) | 1656 | |
| θ range for data collection | 1.70 to 30.51° | |
| Completeness to θ = 27.50° | 100.0 % | |
| Index ranges | $-20 \le h \le 20$, $-21 \le k \le 20$, $-28 \le l \le 27$ | |
| Data collection scan type | ω scans; 8 settings | |
| Reflections collected | 72499 | |
| Independent reflections | 12470 [$R_{int}$= 0.0714] | |
| Absorption coefficient | 0.233 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9816 and 0.9615 | |

**Table 1 (cont.)**

## Structure solution and Refinement

| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Geometric positions |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 12470 / 8 / 485 |
| Treatment of hydrogen atoms | Riding |
| Goodness-of-fit on $F^2$ | 1.945 |
| Final R indices [$I>2\sigma(I)$, 7342 reflections] | R1 = 0.0584, *w*R2 = 0.0723 |
| R indices (all data) | R1 = 0.1107, *w*R2 = 0.0742 |
| Type of weighting scheme used | Sigma |
| Weighting scheme used | $w=1/\sigma^2(Fo^2)$ |
| Max shift/error | 0.001 |
| Average shift/error | 0.000 |
| Largest diff. peak and hole | 1.021 and -0.754 e.Å$^{-3}$ |

## Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The solvent region of this crystal is combination of toluene and pentane in nearly equal amounts, 51:49 respectively. The toluene was restrained to be flat with the six member ring constrained to a regular hexagon and the pentane was restrained to have similar carbon-carbon bond distances. Both were refined isotropically.

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.

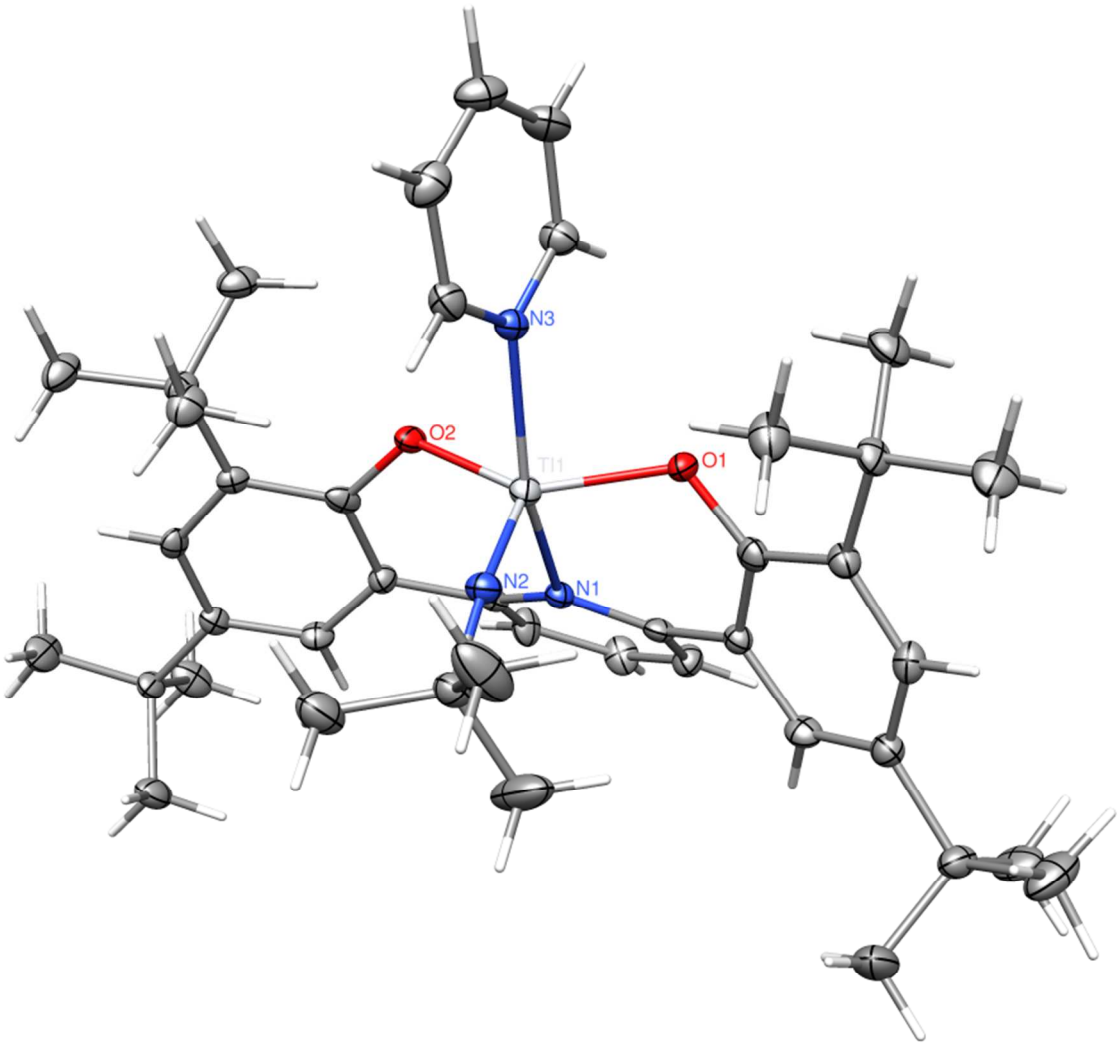

N3
O2
Tl1
O1
N2
N1

**Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for IAT50 (CCDC 847140). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.**

| | x | y | z | $U_{eq}$ | Occ |
|---|---|---|---|---|---|
| Ti(1) | 3121(1) | 4119(1) | 8795(1) | 17(1) | 1 |
| O(1) | 2405(1) | 5035(1) | 9187(1) | 17(1) | 1 |
| O(2) | 4090(1) | 3341(1) | 9121(1) | 17(1) | 1 |
| N(1) | 2226(1) | 3180(1) | 9314(1) | 15(1) | 1 |
| N(2) | 2779(1) | 3924(1) | 7997(1) | 18(1) | 1 |
| N(3) | 4190(1) | 5152(1) | 8722(1) | 17(1) | 1 |
| C(1) | 1492(1) | 5053(2) | 9058(1) | 18(1) | 1 |
| C(2) | 1043(1) | 5823(2) | 8798(1) | 17(1) | 1 |
| C(3) | 114(1) | 5751(2) | 8636(1) | 20(1) | 1 |
| C(4) | -397(1) | 4985(2) | 8735(1) | 18(1) | 1 |
| C(5) | 48(1) | 4266(1) | 9036(1) | 20(1) | 1 |
| C(6) | 982(1) | 4287(1) | 9194(1) | 15(1) | 1 |
| C(7) | 1408(1) | 3488(1) | 9518(1) | 17(1) | 1 |
| C(8) | 950(1) | 3052(2) | 10012(1) | 21(1) | 1 |
| C(9) | 1320(1) | 2299(1) | 10304(1) | 24(1) | 1 |
| C(10) | 2120(1) | 1959(1) | 10077(1) | 22(1) | 1 |
| C(11) | 2556(1) | 2391(1) | 9568(1) | 17(1) | 1 |
| C(12) | 3354(1) | 1954(1) | 9283(1) | 16(1) | 1 |
| C(13) | 3360(1) | 1026(1) | 9233(1) | 19(1) | 1 |
| C(14) | 4088(1) | 555(1) | 8994(1) | 16(1) | 1 |
| C(15) | 4826(1) | 1062(1) | 8800(1) | 16(1) | 1 |
| C(16) | 4868(1) | 1984(1) | 8832(1) | 15(1) | 1 |
| C(17) | 4104(1) | 2447(1) | 9071(1) | 17(1) | 1 |
| C(18) | 1568(1) | 6697(1) | 8687(1) | 19(1) | 1 |
| C(19) | 2250(1) | 6566(1) | 8140(1) | 27(1) | 1 |
| C(20) | 932(1) | 7471(1) | 8467(1) | 29(1) | 1 |
| C(21) | 2073(1) | 6983(1) | 9338(1) | 27(1) | 1 |
| C(22) | -1423(1) | 4911(2) | 8534(1) | 22(1) | 1 |
| C(23) | -1776(1) | 5746(2) | 8168(1) | 40(1) | 1 |
| C(24) | -1963(1) | 4801(2) | 9158(1) | 33(1) | 1 |
| C(25) | -1582(1) | 4099(2) | 8078(1) | 41(1) | 1 |
| C(26) | 4048(1) | -466(1) | 8941(1) | 18(1) | 1 |
| C(27) | 4942(1) | -869(2) | 8716(1) | 32(1) | 1 |
| C(28) | 3284(1) | -724(1) | 8429(1) | 27(1) | 1 |
| C(29) | 3856(1) | -875(2) | 9617(1) | 30(1) | 1 |
| C(30) | 5709(1) | 2498(1) | 8616(1) | 18(1) | 1 |
| C(31) | 6494(1) | 1867(1) | 8441(1) | 27(1) | 1 |
| C(32) | 6079(1) | 3119(1) | 9176(1) | 26(1) | 1 |
| C(33) | 5455(1) | 3048(1) | 7990(1) | 25(1) | 1 |
| C(34) | 2367(1) | 3674(2) | 7357(1) | 22(1) | 1 |
| C(35) | 2378(2) | 4477(2) | 6889(1) | 43(1) | 1 |
| C(36) | 2914(1) | 2914(2) | 7063(1) | 34(1) | 1 |
| C(37) | 1384(1) | 3380(2) | 7449(1) | 53(1) | 1 |
| C(38) | 4620(1) | 5336(1) | 8167(1) | 21(1) | 1 |
| C(39) | 5345(1) | 5919(2) | 8147(1) | 27(1) | 1 |
| C(40) | 5638(2) | 6333(2) | 8731(1) | 30(1) | 1 |
| C(41) | 5200(1) | 6166(2) | 9301(1) | 28(1) | 1 |
| C(42) | 4475(1) | 5576(1) | 9277(1) | 22(1) | 1 |

| | | | | | |
|---|---|---|---|---|---|
| C(51A) | 8507(3) | 709(3) | 9692(2) | 41(2) | 0.507(3) |
| C(52A) | 9238(3) | 807(4) | 9245(2) | 140(4) | 0.507(3) |
| C(53A) | 9886(4) | 144(3) | 9167(3) | 127(3) | 0.507(3) |
| C(54A) | 10615(4) | 296(3) | 8767(3) | 128(4) | 0.507(3) |
| C(55A) | 10695(3) | 1111(4) | 8444(2) | 223(7) | 0.507(3) |
| C(56A) | 10047(4) | 1775(3) | 8523(2) | 85(3) | 0.507(3) |
| C(57A) | 9318(3) | 1623(3) | 8923(3) | 95(3) | 0.507(3) |
| | | | | | |
| C(51B) | 10844(3) | 920(5) | 8703(3) | 65(2) | 0.493(3) |
| C(52B) | 9898(3) | 827(5) | 8353(3) | 96(3) | 0.493(3) |
| C(53B) | 9208(4) | 1477(4) | 8626(3) | 75(2) | 0.493(3) |
| C(54B) | 9091(6) | 1375(7) | 9389(4) | 145(4) | 0.493(3) |
| C(55B) | 8925(10) | 791(11) | 10026(7) | 485(14) | 0.493(3) |

**Table 3. Selected bond lengths [Å] and angles [°] for IAT50 (CCDC 847140).**

| | | | |
|---|---|---|---|
| Ti(1)-N(2) | 1.6903(16) | N(2)-Ti(1)-O(1) | 111.81(7) |
| Ti(1)-O(1) | 1.9204(14) | N(2)-Ti(1)-O(2) | 113.70(7) |
| Ti(1)-O(2) | 1.9309(13) | O(1)-Ti(1)-O(2) | 134.39(6) |
| Ti(1)-N(3) | 2.2132(17) | N(2)-Ti(1)-N(3) | 103.72(7) |
| Ti(1)-N(1) | 2.2205(17) | O(1)-Ti(1)-N(3) | 86.21(6) |
| | | O(2)-Ti(1)-N(3) | 85.98(6) |
| | | N(2)-Ti(1)-N(1) | 100.56(7) |
| | | O(1)-Ti(1)-N(1) | 84.80(6) |
| | | O(2)-Ti(1)-N(1) | 84.33(6) |
| | | N(3)-Ti(1)-N(1) | 155.72(6) |

**Table 4. Bond lengths [Å] and angles [°] for IAT50 (CCDC 847140).**

| | | | |
|---|---|---|---|
| Ti(1)-N(2) | 1.6903(16) | C(40)-C(41) | 1.368(3) |
| Ti(1)-O(1) | 1.9204(14) | C(41)-C(42) | 1.382(3) |
| Ti(1)-O(2) | 1.9309(13) | C(51A)-C(52A) | 1.441(6) |
| Ti(1)-N(3) | 2.2132(17) | C(52A)-C(53A) | 1.3900 |
| Ti(1)-N(1) | 2.2205(17) | C(52A)-C(57A) | 1.3900 |
| O(1)-C(1) | 1.354(2) | C(53A)-C(54A) | 1.3900 |
| O(2)-C(17) | 1.342(2) | C(54A)-C(55A) | 1.3900 |
| N(1)-C(11) | 1.366(2) | C(55A)-C(56A) | 1.3900 |
| N(1)-C(7) | 1.368(2) | C(56A)-C(57A) | 1.3900 |
| N(2)-C(34) | 1.448(2) | C(51B)-C(52B) | 1.535(4) |
| N(3)-C(42) | 1.338(2) | C(52B)-C(53B) | 1.525(4) |
| N(3)-C(38) | 1.339(2) | C(53B)-C(54B) | 1.565(5) |
| C(1)-C(6) | 1.402(3) | C(54B)-C(55B) | 1.582(5) |
| C(1)-C(2) | 1.417(3) | | |
| C(2)-C(3) | 1.392(2) | N(2)-Ti(1)-O(1) | 111.81(7) |
| C(2)-C(18) | 1.541(3) | N(2)-Ti(1)-O(2) | 113.70(7) |
| C(3)-C(4) | 1.390(3) | O(1)-Ti(1)-O(2) | 134.39(6) |
| C(4)-C(5) | 1.384(3) | N(2)-Ti(1)-N(3) | 103.72(7) |
| C(4)-C(22) | 1.546(3) | O(1)-Ti(1)-N(3) | 86.21(6) |
| C(5)-C(6) | 1.395(2) | O(2)-Ti(1)-N(3) | 85.98(6) |
| C(6)-C(7) | 1.487(3) | N(2)-Ti(1)-N(1) | 100.56(7) |
| C(7)-C(8) | 1.391(3) | O(1)-Ti(1)-N(1) | 84.80(6) |
| C(8)-C(9) | 1.371(3) | O(2)-Ti(1)-N(1) | 84.33(6) |
| C(9)-C(10) | 1.380(3) | N(3)-Ti(1)-N(1) | 155.72(6) |
| C(10)-C(11) | 1.395(3) | C(1)-O(1)-Ti(1) | 119.47(12) |
| C(11)-C(12) | 1.482(3) | C(17)-O(2)-Ti(1) | 126.09(12) |
| C(12)-C(13) | 1.393(3) | C(11)-N(1)-C(7) | 118.63(18) |
| C(12)-C(17) | 1.409(3) | C(11)-N(1)-Ti(1) | 121.08(14) |
| C(13)-C(14) | 1.386(3) | C(7)-N(1)-Ti(1) | 118.47(14) |
| C(14)-C(15) | 1.394(3) | C(34)-N(2)-Ti(1) | 170.76(15) |
| C(14)-C(26) | 1.533(3) | C(42)-N(3)-C(38) | 117.45(19) |
| C(15)-C(16) | 1.383(3) | C(42)-N(3)-Ti(1) | 118.05(15) |
| C(16)-C(17) | 1.422(3) | C(38)-N(3)-Ti(1) | 124.31(15) |
| C(16)-C(30) | 1.535(3) | O(1)-C(1)-C(6) | 118.66(19) |
| C(18)-C(19) | 1.537(3) | O(1)-C(1)-C(2) | 121.80(19) |
| C(18)-C(21) | 1.538(3) | C(6)-C(1)-C(2) | 119.53(19) |
| C(18)-C(20) | 1.540(3) | C(3)-C(2)-C(1) | 117.3(2) |
| C(22)-C(23) | 1.529(3) | C(3)-C(2)-C(18) | 121.59(19) |
| C(22)-C(24) | 1.530(3) | C(1)-C(2)-C(18) | 121.13(17) |
| C(22)-C(25) | 1.535(3) | C(4)-C(3)-C(2) | 124.1(2) |
| C(26)-C(29) | 1.533(3) | C(5)-C(4)-C(3) | 117.19(19) |
| C(26)-C(27) | 1.533(3) | C(5)-C(4)-C(22) | 119.6(2) |
| C(26)-C(28) | 1.538(2) | C(3)-C(4)-C(22) | 123.21(19) |
| C(30)-C(33) | 1.538(3) | C(4)-C(5)-C(6) | 121.4(2) |
| C(30)-C(32) | 1.542(3) | C(5)-C(6)-C(1) | 120.2(2) |
| C(30)-C(31) | 1.545(3) | C(5)-C(6)-C(7) | 118.09(19) |
| C(34)-C(35) | 1.528(3) | C(1)-C(6)-C(7) | 121.68(18) |
| C(34)-C(36) | 1.528(3) | N(1)-C(7)-C(8) | 121.3(2) |
| C(34)-C(37) | 1.529(3) | N(1)-C(7)-C(6) | 119.70(19) |
| C(38)-C(39) | 1.380(3) | C(8)-C(7)-C(6) | 118.98(19) |
| C(39)-C(40) | 1.380(3) | C(9)-C(8)-C(7) | 119.8(2) |

| | | | |
|---|---|---|---|
| C(8)-C(9)-C(10) | 119.2(2) | C(53A)-C(52A)-C(57A) | 120.0 |
| C(9)-C(10)-C(11) | 120.0(2) | C(53A)-C(52A)-C(51A) | 122.2(5) |
| N(1)-C(11)-C(10) | 120.7(2) | C(57A)-C(52A)-C(51A) | 117.7(5) |
| N(1)-C(11)-C(12) | 120.6(2) | C(54A)-C(53A)-C(52A) | 120.0 |
| C(10)-C(11)-C(12) | 118.6(2) | C(53A)-C(54A)-C(55A) | 120.0 |
| C(13)-C(12)-C(17) | 119.5(2) | C(56A)-C(55A)-C(54A) | 120.0 |
| C(13)-C(12)-C(11) | 118.42(19) | C(57A)-C(56A)-C(55A) | 120.0 |
| C(17)-C(12)-C(11) | 122.1(2) | C(56A)-C(57A)-C(52A) | 120.0 |
| C(14)-C(13)-C(12) | 122.6(2) | C(53B)-C(52B)-C(51B) | 112.2(5) |
| C(13)-C(14)-C(15) | 116.4(2) | C(52B)-C(53B)-C(54B) | 113.5(6) |
| C(13)-C(14)-C(26) | 120.20(19) | C(53B)-C(54B)-C(55B) | 151.8(11) |
| C(15)-C(14)-C(26) | 123.39(19) | | |
| C(16)-C(15)-C(14) | 124.3(2) | | |
| C(15)-C(16)-C(17) | 117.8(2) | | |
| C(15)-C(16)-C(30) | 121.43(19) | | |
| C(17)-C(16)-C(30) | 120.75(19) | | |
| O(2)-C(17)-C(12) | 118.9(2) | | |
| O(2)-C(17)-C(16) | 121.7(2) | | |
| C(12)-C(17)-C(16) | 119.3(2) | | |
| C(19)-C(18)-C(21) | 110.17(17) | | |
| C(19)-C(18)-C(2) | 110.10(17) | | |
| C(21)-C(18)-C(2) | 109.62(17) | | |
| C(19)-C(18)-C(20) | 107.26(18) | | |
| C(21)-C(18)-C(20) | 107.37(18) | | |
| C(2)-C(18)-C(20) | 112.26(17) | | |
| C(23)-C(22)-C(24) | 107.92(19) | | |
| C(23)-C(22)-C(25) | 108.62(19) | | |
| C(24)-C(22)-C(25) | 109.73(19) | | |
| C(23)-C(22)-C(4) | 111.90(18) | | |
| C(24)-C(22)-C(4) | 109.24(17) | | |
| C(25)-C(22)-C(4) | 109.40(18) | | |
| C(29)-C(26)-C(27) | 107.64(18) | | |
| C(29)-C(26)-C(14) | 110.08(18) | | |
| C(27)-C(26)-C(14) | 112.44(18) | | |
| C(29)-C(26)-C(28) | 109.78(18) | | |
| C(27)-C(26)-C(28) | 108.15(18) | | |
| C(14)-C(26)-C(28) | 108.72(17) | | |
| C(16)-C(30)-C(33) | 109.52(16) | | |
| C(16)-C(30)-C(32) | 110.76(18) | | |
| C(33)-C(30)-C(32) | 109.99(18) | | |
| C(16)-C(30)-C(31) | 112.27(18) | | |
| C(33)-C(30)-C(31) | 107.10(18) | | |
| C(32)-C(30)-C(31) | 107.11(17) | | |
| N(2)-C(34)-C(35) | 109.40(18) | | |
| N(2)-C(34)-C(36) | 109.47(17) | | |
| C(35)-C(34)-C(36) | 108.9(2) | | |
| N(2)-C(34)-C(37) | 108.85(18) | | |
| C(35)-C(34)-C(37) | 109.79(19) | | |
| C(36)-C(34)-C(37) | 110.44(19) | | |
| N(3)-C(38)-C(39) | 123.3(2) | | |
| C(40)-C(39)-C(38) | 118.0(2) | | |
| C(41)-C(40)-C(39) | 119.7(2) | | |
| C(40)-C(41)-C(42) | 118.6(2) | | |
| N(3)-C(42)-C(41) | 122.9(2) | | |

**Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for IAT50 (CCDC 847140). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12} ]$**

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Ti(1) | 166(2) | 146(2) | 196(2) | -5(2) | 20(2) | -16(2) |
| O(1) | 131(8) | 152(9) | 216(9) | -11(7) | 9(7) | -8(7) |
| O(2) | 157(8) | 124(9) | 229(10) | -11(7) | -6(7) | -20(7) |
| N(1) | 152(10) | 135(11) | 168(11) | -13(9) | 15(8) | -20(8) |
| N(2) | 176(10) | 190(12) | 163(11) | -12(9) | 8(8) | -33(8) |
| N(3) | 165(10) | 160(11) | 176(11) | -2(9) | 1(9) | -13(8) |
| C(1) | 199(13) | 178(14) | 155(13) | -32(11) | 42(10) | -1(11) |
| C(2) | 182(12) | 148(13) | 185(13) | 1(11) | 45(10) | -9(11) |
| C(3) | 204(13) | 189(14) | 204(14) | 30(11) | 47(10) | 39(11) |
| C(4) | 176(13) | 184(14) | 187(14) | -9(11) | 15(10) | -5(11) |
| C(5) | 195(13) | 182(14) | 213(14) | 0(11) | 41(10) | -63(11) |
| C(6) | 156(12) | 148(14) | 148(13) | -10(10) | 30(10) | -8(10) |
| C(7) | 174(13) | 149(13) | 193(14) | -40(11) | -7(10) | -47(10) |
| C(8) | 178(13) | 205(14) | 242(15) | -3(11) | 50(11) | -11(11) |
| C(9) | 302(15) | 213(15) | 208(15) | 53(11) | 102(11) | -21(12) |
| C(10) | 268(14) | 150(13) | 245(15) | 39(11) | 72(11) | 37(11) |
| C(11) | 189(13) | 137(13) | 179(14) | -30(10) | -11(10) | -33(10) |
| C(12) | 151(12) | 162(13) | 167(13) | 2(11) | 19(10) | -6(10) |
| C(13) | 188(13) | 174(14) | 198(14) | 28(11) | 17(10) | -52(11) |
| C(14) | 185(13) | 151(13) | 139(13) | 16(10) | -13(10) | 0(10) |
| C(15) | 136(12) | 188(14) | 148(13) | 8(10) | -13(9) | 32(10) |
| C(16) | 146(12) | 164(13) | 140(13) | 24(10) | -11(10) | -19(10) |
| C(17) | 205(13) | 150(13) | 146(13) | 11(10) | -59(10) | -12(11) |
| C(18) | 210(13) | 109(13) | 258(15) | 24(11) | 31(11) | 2(10) |
| C(19) | 285(14) | 200(15) | 333(17) | 41(12) | 122(12) | 0(11) |
| C(20) | 283(15) | 169(14) | 428(18) | 85(12) | 62(12) | -40(11) |
| C(21) | 310(15) | 143(14) | 355(17) | -23(12) | 34(12) | -40(11) |
| C(22) | 185(13) | 201(14) | 258(15) | 17(12) | -45(11) | -12(11) |
| C(23) | 193(14) | 387(19) | 600(20) | 149(15) | -47(13) | -34(13) |
| C(24) | 164(13) | 447(18) | 390(18) | -16(14) | 26(12) | -104(12) |
| C(25) | 335(15) | 405(18) | 473(19) | -189(16) | -176(13) | 51(14) |
| C(26) | 178(13) | 127(13) | 238(15) | -21(11) | 12(11) | -7(10) |
| C(27) | 303(14) | 169(14) | 494(18) | -23(14) | 33(12) | 1(12) |
| C(28) | 285(14) | 224(16) | 305(16) | -70(12) | -34(12) | -16(11) |
| C(29) | 429(15) | 199(14) | 279(15) | 33(13) | -23(12) | -16(13) |
| C(30) | 166(13) | 170(13) | 204(14) | 20(11) | 20(11) | -22(10) |
| C(31) | 195(13) | 227(15) | 389(17) | 42(12) | 70(12) | -19(11) |
| C(32) | 216(13) | 232(15) | 336(16) | 26(12) | 8(12) | -74(11) |
| C(33) | 242(14) | 234(15) | 289(16) | 53(12) | 55(11) | -6(11) |
| C(34) | 220(14) | 229(15) | 197(15) | -47(11) | 10(11) | -24(11) |
| C(35) | 730(20) | 283(17) | 267(17) | -23(13) | -125(15) | 87(14) |
| C(36) | 391(16) | 291(17) | 323(17) | -83(13) | -41(13) | -7(13) |
| C(37) | 271(16) | 940(30) | 390(20) | -261(17) | 15(13) | -187(16) |
| C(38) | 236(14) | 192(14) | 209(15) | -4(11) | 21(11) | 2(11) |
| C(39) | 243(13) | 285(15) | 297(16) | 64(13) | 76(11) | -34(13) |
| C(40) | 247(14) | 277(16) | 371(18) | 31(13) | -4(13) | -104(12) |
| C(41) | 291(15) | 266(16) | 290(16) | -34(12) | -38(12) | -69(12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(42) | 233(14) | 223(14) | 200(15) | 1(11) | 36(11) | -40(11) |

# CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 2 December 2011

**Crystal Structure Analysis of:**

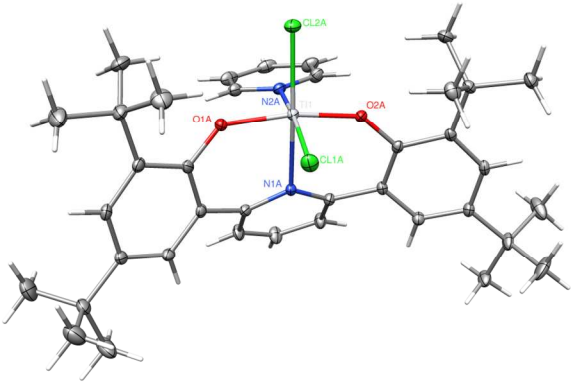
**Complex 6 (ONO)$TiCl_2$(py) (IAT98)**

(shown below)

**By** Michael W. Day 116 Beckman ext. 2734
e-mail: mikeday@caltech.edu

Contents



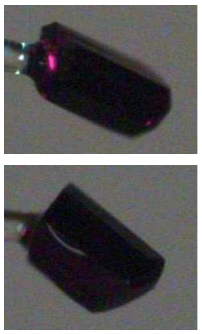



IAT98

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 856446.

**Table 1. Crystal data and structure refinement for IAT98 (CCDC 856446).**

| | |
|---|---|
| Empirical formula | $2(C_{38}H_{48}N_2O_2Cl_2Ti) \bullet 5(C_6H_6)$ |
| Formula weight | 878.85 |
| Crystallization Solvent | Dichloromethane |
| Crystal Habit | Block |
| Crystal size | 0.36 x 0.25 x 0.20 $mm^3$ |
| Crystal color | Orange-red |



## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker KAPPA APEX II | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 9190 reflections used in lattice determination | 2.55 to 34.09° | |
| Unit cell dimensions | a = 11.8721(6) Å<br>b = 28.5845(14) Å<br>c = 29.6698(15) Å | α= 90°<br>β= 100.018(3)°<br>γ = 90° |
| Volume | 9915.2(9) $Å^3$ | |
| Z | 8 | |
| Crystal system | Monoclinic | |
| Space group | P $2_1/c$ | |
| Density (calculated) | 1.177 $Mg/m^3$ | |
| F(000) | 3736 | |
| Data collection program | Bruker APEX2 v2009.7-0 | |
| θ range for data collection | 1.59 to 35.13° | |
| Completeness to θ = 35.13° | 99.7 % | |
| Index ranges | $-13 \le h \le 19$, $-46 \le k \le 46$, $-48 \le l \le 48$ | |
| Data collection scan type | ω scans; 12 settings | |
| Data reduction program | Bruker SAINT-Plus v7.68A | |
| Reflections collected | 325048 | |
| Independent reflections | 43933 [$R_{int}$= 0.0602] | |
| Absorption coefficient | 0.320 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9387 and 0.8934 | |

**Table 1 (cont.)**

## Structure solution and Refinement

| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Geometric positions |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 43933 / 0 / 1105 |
| Treatment of hydrogen atoms | Riding |
| Goodness-of-fit on $F^2$ | 1.877 |
| Final R indices [$I>2\sigma(I)$, 30745 reflections] | R1 = 0.0415, *w*R2 = 0.0625 |
| R indices (all data) | R1 = 0.0689, *w*R2 = 0.0636 |
| 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 | 0.756 and -0.589 e.Å$^{-3}$ |

## Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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.

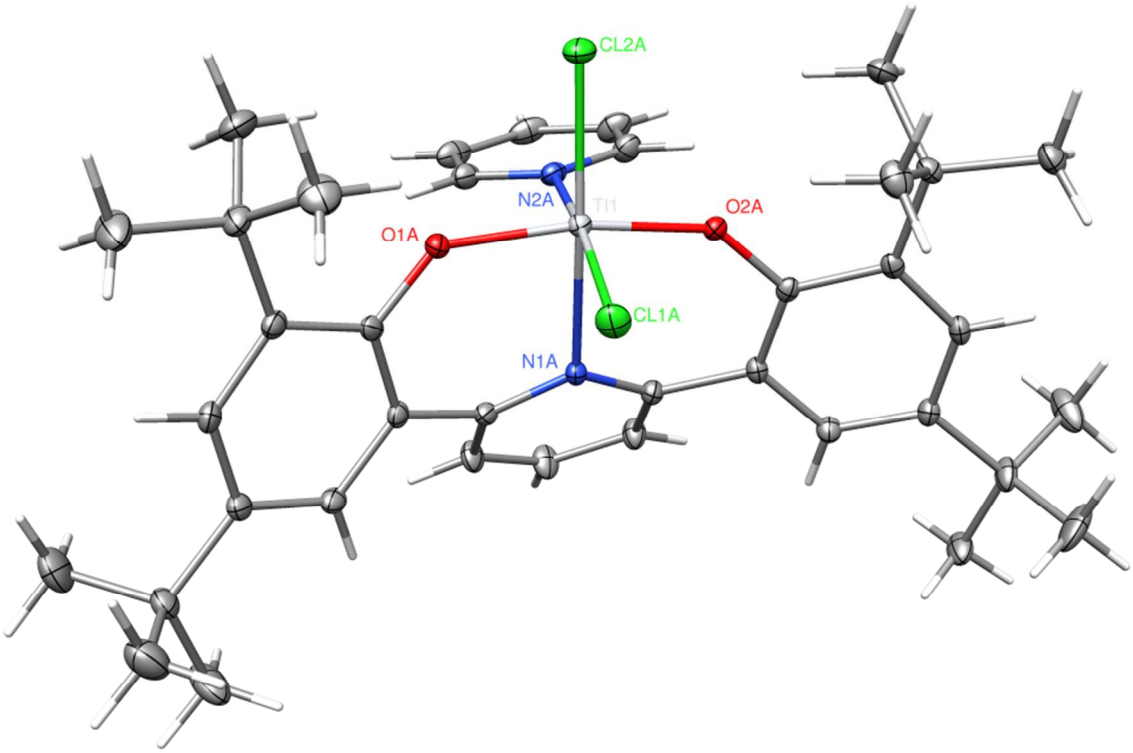

CL2A
N2A
Ti1
O2A
O1A
CL1A
N1A

**Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for IAT98 (CCDC 856446). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.**

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| Ti(1) | 5235(1) | 1029(1) | 6098(1) | 11(1) |
| Cl(1A) | 5402(1) | 1419(1) | 6783(1) | 19(1) |
| Cl(2A) | 3740(1) | 536(1) | 6133(1) | 18(1) |
| O(1A) | 6375(1) | 616(1) | 6336(1) | 13(1) |
| O(2A) | 4383(1) | 1498(1) | 5803(1) | 12(1) |
| N(1A) | 6724(1) | 1463(1) | 5921(1) | 11(1) |
| N(2A) | 5375(1) | 755(1) | 5398(1) | 14(1) |
| C(1A) | 7462(1) | 641(1) | 6559(1) | 12(1) |
| C(2A) | 7874(1) | 333(1) | 6922(1) | 14(1) |
| C(3A) | 9007(1) | 388(1) | 7129(1) | 15(1) |
| C(4A) | 9748(1) | 725(1) | 6999(1) | 14(1) |
| C(5A) | 9310(1) | 1012(1) | 6636(1) | 14(1) |
| C(6A) | 8178(1) | 973(1) | 6409(1) | 12(1) |
| C(7A) | 7794(1) | 1272(1) | 6002(1) | 13(1) |
| C(8A) | 8578(1) | 1359(1) | 5712(1) | 17(1) |
| C(9A) | 8298(1) | 1657(1) | 5346(1) | 19(1) |
| C(10A) | 7269(1) | 1892(1) | 5294(1) | 17(1) |
| C(11A) | 6504(1) | 1803(1) | 5591(1) | 13(1) |
| C(12A) | 5461(1) | 2101(1) | 5550(1) | 12(1) |
| C(13A) | 5513(1) | 2563(1) | 5396(1) | 14(1) |
| C(14A) | 4567(1) | 2854(1) | 5341(1) | 14(1) |
| C(15A) | 3548(1) | 2672(1) | 5446(1) | 13(1) |
| C(16A) | 3442(1) | 2223(1) | 5607(1) | 11(1) |
| C(17A) | 4431(1) | 1941(1) | 5658(1) | 11(1) |
| C(18A) | 7099(1) | -44(1) | 7078(1) | 20(1) |
| C(19A) | 6126(1) | 186(1) | 7278(1) | 27(1) |
| C(20A) | 6599(1) | -356(1) | 6673(1) | 28(1) |
| C(21A) | 7774(1) | -360(1) | 7448(1) | 35(1) |
| C(22A) | 10974(1) | 764(1) | 7260(1) | 19(1) |
| C(23A) | 11590(1) | 295(1) | 7247(1) | 36(1) |
| C(24A) | 10950(1) | 888(1) | 7766(1) | 33(1) |
| C(25A) | 11650(1) | 1141(1) | 7064(1) | 39(1) |
| C(26A) | 4590(1) | 3361(1) | 5176(1) | 19(1) |
| C(27A) | 5749(1) | 3494(1) | 5052(1) | 24(1) |
| C(28A) | 4357(1) | 3691(1) | 5557(1) | 35(1) |
| C(29A) | 3661(1) | 3428(1) | 4754(1) | 32(1) |
| C(30A) | 2320(1) | 2036(1) | 5732(1) | 13(1) |
| C(31A) | 1913(1) | 1598(1) | 5450(1) | 20(1) |
| C(32A) | 1368(1) | 2402(1) | 5634(1) | 19(1) |
| C(33A) | 2492(1) | 1921(1) | 6244(1) | 19(1) |
| C(34A) | 6160(1) | 432(1) | 5336(1) | 17(1) |
| C(35A) | 6303(1) | 278(1) | 4909(1) | 22(1) |
| C(36A) | 5632(1) | 463(1) | 4526(1) | 23(1) |
| C(37A) | 4821(1) | 796(1) | 4584(1) | 22(1) |
| C(38A) | 4718(1) | 929(1) | 5020(1) | 18(1) |

| | | | | |
|---|---|---|---|---|
| Ti(2) | 8749(1) | 2409(1) | 8959(1) | 11(1) |
| Cl(1B) | 7058(1) | 2437(1) | 9247(1) | 17(1) |
| Cl(2B) | 9902(1) | 2874(1) | 9468(1) | 17(1) |
| O(1B) | 8272(1) | 2876(1) | 8549(1) | 13(1) |
| O(2B) | 9136(1) | 1854(1) | 9261(1) | 14(1) |
| N(1B) | 7883(1) | 1938(1) | 8394(1) | 12(1) |
| N(2B) | 10215(1) | 2290(1) | 8589(1) | 15(1) |
| C(1B) | 7429(1) | 2966(1) | 8194(1) | 12(1) |
| C(2B) | 6963(1) | 3418(1) | 8119(1) | 14(1) |
| C(3B) | 6067(1) | 3467(1) | 7751(1) | 18(1) |
| C(4B) | 5631(1) | 3101(1) | 7461(1) | 19(1) |
| C(5B) | 6153(1) | 2667(1) | 7540(1) | 17(1) |
| C(6B) | 7053(1) | 2594(1) | 7901(1) | 13(1) |
| C(7B) | 7562(1) | 2119(1) | 7964(1) | 13(1) |
| C(8B) | 7614(1) | 1854(1) | 7576(1) | 16(1) |
| C(9B) | 7921(1) | 1389(1) | 7620(1) | 19(1) |
| C(10B) | 8092(1) | 1191(1) | 8047(1) | 18(1) |
| C(11B) | 8043(1) | 1465(1) | 8431(1) | 14(1) |
| C(12B) | 8102(1) | 1231(1) | 8880(1) | 14(1) |
| C(13B) | 7564(1) | 794(1) | 8891(1) | 17(1) |
| C(14B) | 7595(1) | 556(1) | 9298(1) | 18(1) |
| C(15B) | 8177(1) | 764(1) | 9697(1) | 18(1) |
| C(16B) | 8719(1) | 1197(1) | 9709(1) | 15(1) |
| C(17B) | 8647(1) | 1430(1) | 9285(1) | 14(1) |
| C(18B) | 7430(1) | 3830(1) | 8427(1) | 18(1) |
| C(19B) | 6823(1) | 4290(1) | 8268(1) | 29(1) |
| C(20B) | 8712(1) | 3898(1) | 8418(1) | 24(1) |
| C(21B) | 7249(1) | 3741(1) | 8920(1) | 23(1) |
| C(22B) | 4594(1) | 3151(1) | 7078(1) | 31(1) |
| C(23B) | 4220(2) | 3657(1) | 6999(1) | 64(1) |
| C(24B) | 4862(1) | 2952(1) | 6633(1) | 34(1) |
| C(25B) | 3589(1) | 2869(1) | 7212(1) | 48(1) |
| C(26B) | 7014(1) | 80(1) | 9330(1) | 21(1) |
| C(27B) | 6443(1) | -103(1) | 8862(1) | 29(1) |
| C(28B) | 6108(1) | 125(1) | 9637(1) | 28(1) |
| C(29B) | 7928(1) | -279(1) | 9542(1) | 28(1) |
| C(30B) | 9389(1) | 1399(1) | 10153(1) | 17(1) |
| C(31B) | 10653(1) | 1444(1) | 10102(1) | 21(1) |
| C(32B) | 8926(1) | 1880(1) | 10263(1) | 20(1) |
| C(33B) | 9332(1) | 1079(1) | 10564(1) | 26(1) |
| C(34B) | 10306(1) | 2515(1) | 8202(1) | 23(1) |
| C(35B) | 11205(1) | 2447(1) | 7970(1) | 33(1) |
| C(36B) | 12058(1) | 2140(1) | 8145(1) | 32(1) |
| C(37B) | 11980(1) | 1908(1) | 8541(1) | 32(1) |
| C(38B) | 11055(1) | 1989(1) | 8753(1) | 25(1) |
| | | | | |
| C(41) | 8180(2) | 4688(1) | 6414(1) | 64(1) |
| C(42) | 9339(2) | 4707(1) | 6516(1) | 52(1) |
| C(43) | 9924(2) | 5037(1) | 6319(1) | 46(1) |
| C(44) | 9370(2) | 5354(1) | 6018(1) | 49(1) |
| C(45) | 8210(2) | 5344(1) | 5908(1) | 63(1) |
| C(46) | 7588(2) | 5009(1) | 6104(1) | 75(1) |

| | | | | |
|---|---|---|---|---|
| C(51) | 8347(2) | 2859(1) | 6771(1) | 111(1) |
| C(52) | 9269(2) | 2716(1) | 6576(1) | 111(1) |
| C(53) | 10277(2) | 2944(1) | 6678(1) | 117(1) |
| C(54) | 10416(2) | 3321(1) | 6978(1) | 106(1) |
| C(55) | 9501(2) | 3470(1) | 7175(1) | 107(1) |
| C(56) | 8486(3) | 3234(1) | 7072(1) | 125(1) |
| | | | | |
| C(61) | 4416(1) | 3057(1) | 8772(1) | 33(1) |
| C(62) | 3689(1) | 3337(1) | 8483(1) | 32(1) |
| C(63) | 2625(1) | 3445(1) | 8580(1) | 35(1) |
| C(64) | 2294(1) | 3272(1) | 8967(1) | 37(1) |
| C(65) | 3031(1) | 2992(1) | 9258(1) | 39(1) |
| C(66) | 4093(1) | 2886(1) | 9160(1) | 37(1) |
| | | | | |
| C(71) | 5116(1) | 1743(1) | 8330(1) | 32(1) |
| C(72) | 4951(1) | 1501(1) | 8710(1) | 34(1) |
| C(73) | 4545(1) | 1049(1) | 8670(1) | 38(1) |
| C(74) | 4326(1) | 836(1) | 8245(1) | 34(1) |
| C(75) | 4518(1) | 1073(1) | 7864(1) | 28(1) |
| C(76) | 4898(1) | 1530(1) | 7904(1) | 32(1) |
| | | | | |
| C(81) | 420(2) | 4310(1) | 9620(1) | 49(1) |
| C(82) | 1120(1) | 4507(1) | 9324(1) | 51(1) |
| C(83) | 737(1) | 4898(1) | 9077(1) | 42(1) |
| C(84) | -278(1) | 5090(1) | 9115(1) | 36(1) |
| C(85) | -937(1) | 4906(1) | 9394(1) | 34(1) |
| C(86) | -605(1) | 4522(1) | 9646(1) | 35(1) |

**Table 3. Selected bond lengths [Å] and angles [°] for IAT98 (CCDC 856446).**

| | | | |
|---|---|---|---|
| Ti(1)-O(2A) | 1.8119(7) | Ti(2)-O(1B) | 1.8294(7) |
| Ti(1)-O(1A) | 1.8426(7) | Ti(2)-O(2B) | 1.8391(7) |
| Ti(1)-N(2A) | 2.2533(9) | Ti(2)-N(2B) | 2.2400(9) |
| Ti(1)-Cl(2A) | 2.2832(3) | Ti(2)-N(1B) | 2.2536(9) |
| Ti(1)-N(1A) | 2.2950(8) | Ti(2)-Cl(2B) | 2.2798(3) |
| Ti(1)-Cl(1A) | 2.2961(3) | Ti(2)-Cl(1B) | 2.3149(3) |
| | | | |
| O(2A)-Ti(1)-O(1A) | 166.32(3) | O(1B)-Ti(2)-O(2B) | 167.35(3) |
| O(2A)-Ti(1)-N(2A) | 86.38(3) | O(1B)-Ti(2)-N(2B) | 87.79(3) |
| O(1A)-Ti(1)-N(2A) | 87.98(3) | O(2B)-Ti(2)-N(2B) | 87.65(3) |
| O(2A)-Ti(1)-Cl(2A) | 96.07(2) | O(1B)-Ti(2)-N(1B) | 83.83(3) |
| O(1A)-Ti(1)-Cl(2A) | 96.43(2) | O(2B)-Ti(2)-N(1B) | 83.80(3) |
| N(2A)-Ti(1)-Cl(2A) | 90.59(2) | N(2B)-Ti(2)-N(1B) | 80.64(3) |
| O(2A)-Ti(1)-N(1A) | 82.89(3) | O(1B)-Ti(2)-Cl(2B) | 95.44(2) |
| O(1A)-Ti(1)-N(1A) | 83.75(3) | O(2B)-Ti(2)-Cl(2B) | 96.27(2) |
| N(2A)-Ti(1)-N(1A) | 78.09(3) | N(2B)-Ti(2)-Cl(2B) | 89.09(2) |
| Cl(2A)-Ti(1)-N(1A) | 168.67(2) | N(1B)-Ti(2)-Cl(2B) | 169.72(2) |
| O(2A)-Ti(1)-Cl(1A) | 91.31(2) | O(1B)-Ti(2)-Cl(1B) | 91.83(2) |
| O(1A)-Ti(1)-Cl(1A) | 91.50(2) | O(2B)-Ti(2)-Cl(1B) | 90.55(2) |
| N(2A)-Ti(1)-Cl(1A) | 167.43(2) | N(2B)-Ti(2)-Cl(1B) | 169.63(2) |
| Cl(2A)-Ti(1)-Cl(1A) | 101.947(12) | N(1B)-Ti(2)-Cl(1B) | 89.02(2) |
| N(1A)-Ti(1)-Cl(1A) | 89.36(2) | Cl(2B)-Ti(2)-Cl(1B) | 101.259(12) |

**Table 4. Bond lengths [Å] and angles [°] for IAT98 (CCDC 856446).**

| | | | |
|---|---|---|---|
| Ti(1)-O(2A) | 1.8119(7) | Ti(2)-O(2B) | 1.8391(7) |
| Ti(1)-O(1A) | 1.8426(7) | Ti(2)-N(2B) | 2.2400(9) |
| Ti(1)-N(2A) | 2.2533(9) | Ti(2)-N(1B) | 2.2536(9) |
| Ti(1)-Cl(2A) | 2.2832(3) | Ti(2)-Cl(2B) | 2.2798(3) |
| Ti(1)-N(1A) | 2.2950(8) | Ti(2)-Cl(1B) | 2.3149(3) |
| Ti(1)-Cl(1A) | 2.2961(3) | O(1B)-C(1B) | 1.3453(12) |
| O(1A)-C(1A) | 1.3445(12) | O(2B)-C(17B) | 1.3520(12) |
| O(2A)-C(17A) | 1.3406(11) | N(1B)-C(7B) | 1.3670(13) |
| N(1A)-C(7A) | 1.3661(13) | N(1B)-C(11B) | 1.3685(12) |
| N(1A)-C(11A) | 1.3698(13) | N(2B)-C(34B) | 1.3356(14) |
| N(2A)-C(38A) | 1.3445(14) | N(2B)-C(38B) | 1.3434(14) |
| N(2A)-C(34A) | 1.3474(13) | C(1B)-C(6B) | 1.3970(14) |
| C(1A)-C(6A) | 1.3981(14) | C(1B)-C(2B) | 1.4071(14) |
| C(1A)-C(2A) | 1.4112(14) | C(2B)-C(3B) | 1.3913(15) |
| C(2A)-C(3A) | 1.3868(15) | C(2B)-C(18B) | 1.5355(15) |
| C(2A)-C(18A) | 1.5412(15) | C(3B)-C(4B) | 1.3956(15) |
| C(3A)-C(4A) | 1.4032(14) | C(4B)-C(5B) | 1.3888(15) |
| C(4A)-C(5A) | 1.3818(14) | C(4B)-C(22B) | 1.5301(16) |
| C(4A)-C(22A) | 1.5289(15) | C(5B)-C(6B) | 1.3904(15) |
| C(5A)-C(6A) | 1.4003(15) | C(6B)-C(7B) | 1.4859(14) |
| C(6A)-C(7A) | 1.4833(14) | C(7B)-C(8B) | 1.3887(14) |
| C(7A)-C(8A) | 1.3939(14) | C(8B)-C(9B) | 1.3775(14) |
| C(8A)-C(9A) | 1.3760(15) | C(9B)-C(10B) | 1.3715(15) |
| C(9A)-C(10A) | 1.3786(15) | C(10B)-C(11B) | 1.3905(14) |
| C(10A)-C(11A) | 1.3939(14) | C(11B)-C(12B) | 1.4823(15) |
| C(11A)-C(12A) | 1.4910(14) | C(12B)-C(17B) | 1.3853(15) |
| C(12A)-C(17A) | 1.3940(14) | C(12B)-C(13B) | 1.4053(14) |
| C(12A)-C(13A) | 1.4028(14) | C(13B)-C(14B) | 1.3806(15) |
| C(13A)-C(14A) | 1.3829(14) | C(14B)-C(15B) | 1.3952(16) |
| C(14A)-C(15A) | 1.4008(14) | C(14B)-C(26B) | 1.5372(15) |
| C(14A)-C(26A) | 1.5336(14) | C(15B)-C(16B) | 1.3918(14) |
| C(15A)-C(16A) | 1.3827(13) | C(16B)-C(17B) | 1.4122(15) |
| C(16A)-C(17A) | 1.4099(14) | C(16B)-C(30B) | 1.5298(15) |
| C(16A)-C(30A) | 1.5382(14) | C(18B)-C(19B) | 1.5328(16) |
| C(18A)-C(20A) | 1.5306(17) | C(18B)-C(21B) | 1.5377(15) |
| C(18A)-C(21A) | 1.5340(16) | C(18B)-C(20B) | 1.5382(16) |
| C(18A)-C(19A) | 1.5365(16) | C(22B)-C(23B) | 1.5194(18) |
| C(22A)-C(25A) | 1.5193(16) | C(22B)-C(24B) | 1.5214(17) |
| C(22A)-C(23A) | 1.5292(16) | C(22B)-C(25B) | 1.547(2) |
| C(22A)-C(24A) | 1.5466(17) | C(26B)-C(27B) | 1.5284(17) |
| C(26A)-C(29A) | 1.5297(17) | C(26B)-C(28B) | 1.5301(16) |
| C(26A)-C(27A) | 1.5324(15) | C(26B)-C(29B) | 1.5434(16) |
| C(26A)-C(28A) | 1.5352(16) | C(30B)-C(33B) | 1.5333(15) |
| C(30A)-C(32A) | 1.5286(14) | C(30B)-C(32B) | 1.5395(15) |
| C(30A)-C(33A) | 1.5326(15) | C(30B)-C(31B) | 1.5395(15) |
| C(30A)-C(31A) | 1.5381(15) | C(34B)-C(35B) | 1.3806(16) |
| C(34A)-C(35A) | 1.3808(15) | C(35B)-C(36B) | 1.3711(18) |
| C(35A)-C(36A) | 1.3741(17) | C(36B)-C(37B) | 1.3676(18) |
| C(36A)-C(37A) | 1.3860(16) | C(37B)-C(38B) | 1.3762(16) |
| C(37A)-C(38A) | 1.3744(15) | C(41)-C(42) | 1.358(3) |
| Ti(2)-O(1B) | 1.8294(7) | C(41)-C(46) | 1.398(3) |

| | | | |
|---|---|---|---|
| C(42)-C(43) | 1.361(2) | C(6A)-C(1A)-C(2A) | 121.16(10) |
| C(43)-C(44) | 1.361(2) | C(3A)-C(2A)-C(1A) | 116.71(9) |
| C(44)-C(45) | 1.359(2) | C(3A)-C(2A)-C(18A) | 121.92(9) |
| C(45)-C(46) | 1.396(3) | C(1A)-C(2A)-C(18A) | 121.36(10) |
| C(51)-C(56) | 1.387(4) | C(2A)-C(3A)-C(4A) | 124.16(10) |
| C(51)-C(52) | 1.388(3) | C(5A)-C(4A)-C(3A) | 116.92(10) |
| C(52)-C(53) | 1.350(4) | C(5A)-C(4A)-C(22A) | 123.17(9) |
| C(53)-C(54) | 1.388(4) | C(3A)-C(4A)-C(22A) | 119.91(9) |
| C(54)-C(55) | 1.388(3) | C(4A)-C(5A)-C(6A) | 121.94(9) |
| C(55)-C(56) | 1.367(4) | C(1A)-C(6A)-C(5A) | 119.05(9) |
| C(61)-C(62) | 1.3647(19) | C(1A)-C(6A)-C(7A) | 122.17(9) |
| C(61)-C(66) | 1.3661(19) | C(5A)-C(6A)-C(7A) | 118.71(9) |
| C(62)-C(63) | 1.3783(19) | N(1A)-C(7A)-C(8A) | 121.27(9) |
| C(63)-C(64) | 1.3705(19) | N(1A)-C(7A)-C(6A) | 121.22(9) |
| C(64)-C(65) | 1.374(2) | C(8A)-C(7A)-C(6A) | 117.48(9) |
| C(65)-C(66) | 1.375(2) | C(9A)-C(8A)-C(7A) | 120.19(10) |
| C(71)-C(72) | 1.3648(18) | C(8A)-C(9A)-C(10A) | 118.44(10) |
| C(71)-C(76) | 1.3843(18) | C(9A)-C(10A)-C(11A) | 120.20(10) |
| C(72)-C(73) | 1.3777(19) | N(1A)-C(11A)-C(10A) | 121.06(9) |
| C(73)-C(74) | 1.3844(19) | N(1A)-C(11A)-C(12A) | 121.07(9) |
| C(74)-C(75) | 1.3714(18) | C(10A)-C(11A)-C(12A) | 117.83(9) |
| C(75)-C(76) | 1.3794(18) | C(17A)-C(12A)-C(13A) | 118.33(9) |
| C(81)-C(86) | 1.374(2) | C(17A)-C(12A)-C(11A) | 122.85(9) |
| C(81)-C(82) | 1.426(2) | C(13A)-C(12A)-C(11A) | 118.83(9) |
| C(82)-C(83) | 1.371(2) | C(14A)-C(13A)-C(12A) | 121.49(9) |
| C(83)-C(84) | 1.345(2) | C(13A)-C(14A)-C(15A) | 117.80(9) |
| C(84)-C(85) | 1.3426(18) | C(13A)-C(14A)-C(26A) | 123.07(9) |
| C(85)-C(86) | 1.3461(18) | C(15A)-C(14A)-C(26A) | 119.13(9) |
| | | C(16A)-C(15A)-C(14A) | 123.71(9) |
| O(2A)-Ti(1)-O(1A) | 166.32(3) | C(15A)-C(16A)-C(17A) | 116.33(9) |
| O(2A)-Ti(1)-N(2A) | 86.38(3) | C(15A)-C(16A)-C(30A) | 122.58(9) |
| O(1A)-Ti(1)-N(2A) | 87.98(3) | C(17A)-C(16A)-C(30A) | 121.08(9) |
| O(2A)-Ti(1)-Cl(2A) | 96.07(2) | O(2A)-C(17A)-C(12A) | 118.18(9) |
| O(1A)-Ti(1)-Cl(2A) | 96.43(2) | O(2A)-C(17A)-C(16A) | 119.46(9) |
| N(2A)-Ti(1)-Cl(2A) | 90.59(2) | C(12A)-C(17A)-C(16A) | 122.33(9) |
| O(2A)-Ti(1)-N(1A) | 82.89(3) | C(20A)-C(18A)-C(21A) | 107.71(9) |
| O(1A)-Ti(1)-N(1A) | 83.75(3) | C(20A)-C(18A)-C(19A) | 109.76(10) |
| N(2A)-Ti(1)-N(1A) | 78.09(3) | C(21A)-C(18A)-C(19A) | 108.04(10) |
| Cl(2A)-Ti(1)-N(1A) | 168.67(2) | C(20A)-C(18A)-C(2A) | 109.97(9) |
| O(2A)-Ti(1)-Cl(1A) | 91.31(2) | C(21A)-C(18A)-C(2A) | 111.22(9) |
| O(1A)-Ti(1)-Cl(1A) | 91.50(2) | C(19A)-C(18A)-C(2A) | 110.09(9) |
| N(2A)-Ti(1)-Cl(1A) | 167.43(2) | C(25A)-C(22A)-C(23A) | 108.97(10) |
| Cl(2A)-Ti(1)-Cl(1A) | 101.947(12) | C(25A)-C(22A)-C(4A) | 112.20(9) |
| N(1A)-Ti(1)-Cl(1A) | 89.36(2) | C(23A)-C(22A)-C(4A) | 109.90(9) |
| C(1A)-O(1A)-Ti(1) | 137.07(6) | C(25A)-C(22A)-C(24A) | 108.01(10) |
| C(17A)-O(2A)-Ti(1) | 143.37(6) | C(23A)-C(22A)-C(24A) | 108.37(10) |
| C(7A)-N(1A)-C(11A) | 117.77(8) | C(4A)-C(22A)-C(24A) | 109.31(9) |
| C(7A)-N(1A)-Ti(1) | 118.19(6) | C(29A)-C(26A)-C(27A) | 108.85(9) |
| C(11A)-N(1A)-Ti(1) | 119.23(7) | C(29A)-C(26A)-C(14A) | 109.19(10) |
| C(38A)-N(2A)-C(34A) | 117.16(9) | C(27A)-C(26A)-C(14A) | 112.19(9) |
| C(38A)-N(2A)-Ti(1) | 121.00(7) | C(29A)-C(26A)-C(28A) | 109.25(10) |
| C(34A)-N(2A)-Ti(1) | 121.73(7) | C(27A)-C(26A)-C(28A) | 108.02(10) |
| O(1A)-C(1A)-C(6A) | 117.68(9) | C(14A)-C(26A)-C(28A) | 109.29(9) |
| O(1A)-C(1A)-C(2A) | 121.13(9) | C(32A)-C(30A)-C(33A) | 107.49(8) |

| | | | |
|---|---|---|---|
| C(32A)-C(30A)-C(31A) | 107.54(9) | N(1B)-C(11B)-C(12B) | 120.20(9) |
| C(33A)-C(30A)-C(31A) | 109.80(9) | C(10B)-C(11B)-C(12B) | 118.70(9) |
| C(32A)-C(30A)-C(16A) | 111.17(8) | C(17B)-C(12B)-C(13B) | 119.38(10) |
| C(33A)-C(30A)-C(16A) | 110.01(9) | C(17B)-C(12B)-C(11B) | 122.47(9) |
| C(31A)-C(30A)-C(16A) | 110.75(8) | C(13B)-C(12B)-C(11B) | 118.14(10) |
| N(2A)-C(34A)-C(35A) | 122.65(11) | C(14B)-C(13B)-C(12B) | 121.00(10) |
| C(36A)-C(35A)-C(34A) | 119.43(11) | C(13B)-C(14B)-C(15B) | 117.79(10) |
| C(35A)-C(36A)-C(37A) | 118.55(11) | C(13B)-C(14B)-C(26B) | 123.17(10) |
| C(38A)-C(37A)-C(36A) | 118.88(11) | C(15B)-C(14B)-C(26B) | 119.03(10) |
| N(2A)-C(38A)-C(37A) | 123.31(10) | C(16B)-C(15B)-C(14B) | 123.93(10) |
| O(1B)-Ti(2)-O(2B) | 167.35(3) | C(15B)-C(16B)-C(17B) | 116.20(10) |
| O(1B)-Ti(2)-N(2B) | 87.79(3) | C(15B)-C(16B)-C(30B) | 121.76(10) |
| O(2B)-Ti(2)-N(2B) | 87.65(3) | C(17B)-C(16B)-C(30B) | 122.01(9) |
| O(1B)-Ti(2)-N(1B) | 83.83(3) | O(2B)-C(17B)-C(12B) | 117.57(9) |
| O(2B)-Ti(2)-N(1B) | 83.80(3) | O(2B)-C(17B)-C(16B) | 120.75(9) |
| N(2B)-Ti(2)-N(1B) | 80.64(3) | C(12B)-C(17B)-C(16B) | 121.66(9) |
| O(1B)-Ti(2)-Cl(2B) | 95.44(2) | C(19B)-C(18B)-C(2B) | 111.91(10) |
| O(2B)-Ti(2)-Cl(2B) | 96.27(2) | C(19B)-C(18B)-C(21B) | 107.34(9) |
| N(2B)-Ti(2)-Cl(2B) | 89.09(2) | C(2B)-C(18B)-C(21B) | 110.13(8) |
| N(1B)-Ti(2)-Cl(2B) | 169.72(2) | C(19B)-C(18B)-C(20B) | 107.46(9) |
| O(1B)-Ti(2)-Cl(1B) | 91.83(2) | C(2B)-C(18B)-C(20B) | 110.03(9) |
| O(2B)-Ti(2)-Cl(1B) | 90.55(2) | C(21B)-C(18B)-C(20B) | 109.90(10) |
| N(2B)-Ti(2)-Cl(1B) | 169.63(2) | C(23B)-C(22B)-C(24B) | 108.82(11) |
| N(1B)-Ti(2)-Cl(1B) | 89.02(2) | C(23B)-C(22B)-C(4B) | 112.22(11) |
| Cl(2B)-Ti(2)-Cl(1B) | 101.259(12) | C(24B)-C(22B)-C(4B) | 110.33(10) |
| C(1B)-O(1B)-Ti(2) | 139.65(6) | C(23B)-C(22B)-C(25B) | 108.42(12) |
| C(17B)-O(2B)-Ti(2) | 136.50(7) | C(24B)-C(22B)-C(25B) | 108.41(11) |
| C(7B)-N(1B)-C(11B) | 117.44(8) | C(4B)-C(22B)-C(25B) | 108.55(11) |
| C(7B)-N(1B)-Ti(2) | 119.11(6) | C(27B)-C(26B)-C(28B) | 108.86(10) |
| C(11B)-N(1B)-Ti(2) | 119.40(7) | C(27B)-C(26B)-C(14B) | 112.26(9) |
| C(34B)-N(2B)-C(38B) | 117.01(9) | C(28B)-C(26B)-C(14B) | 109.38(9) |
| C(34B)-N(2B)-Ti(2) | 122.29(7) | C(27B)-C(26B)-C(29B) | 108.14(9) |
| C(38B)-N(2B)-Ti(2) | 120.70(7) | C(28B)-C(26B)-C(29B) | 109.36(10) |
| O(1B)-C(1B)-C(6B) | 117.12(9) | C(14B)-C(26B)-C(29B) | 108.79(9) |
| O(1B)-C(1B)-C(2B) | 121.21(9) | C(16B)-C(30B)-C(33B) | 111.82(9) |
| C(6B)-C(1B)-C(2B) | 121.66(10) | C(16B)-C(30B)-C(32B) | 111.64(9) |
| C(3B)-C(2B)-C(1B) | 116.27(10) | C(33B)-C(30B)-C(32B) | 107.29(9) |
| C(3B)-C(2B)-C(18B) | 122.50(9) | C(16B)-C(30B)-C(31B) | 108.49(9) |
| C(1B)-C(2B)-C(18B) | 121.23(10) | C(33B)-C(30B)-C(31B) | 107.87(9) |
| C(2B)-C(3B)-C(4B) | 124.03(10) | C(32B)-C(30B)-C(31B) | 109.66(9) |
| C(5B)-C(4B)-C(3B) | 117.27(10) | N(2B)-C(34B)-C(35B) | 123.03(11) |
| C(5B)-C(4B)-C(22B) | 119.09(10) | C(36B)-C(35B)-C(34B) | 119.10(12) |
| C(3B)-C(4B)-C(22B) | 123.60(10) | C(37B)-C(36B)-C(35B) | 118.62(11) |
| C(4B)-C(5B)-C(6B) | 121.60(10) | C(36B)-C(37B)-C(38B) | 119.34(12) |
| C(5B)-C(6B)-C(1B) | 119.06(9) | N(2B)-C(38B)-C(37B) | 122.90(11) |
| C(5B)-C(6B)-C(7B) | 118.33(9) | C(42)-C(41)-C(46) | 119.66(17) |
| C(1B)-C(6B)-C(7B) | 122.60(9) | C(41)-C(42)-C(43) | 120.35(17) |
| N(1B)-C(7B)-C(8B) | 121.50(9) | C(44)-C(43)-C(42) | 121.23(17) |
| N(1B)-C(7B)-C(6B) | 120.24(9) | C(45)-C(44)-C(43) | 119.78(16) |
| C(8B)-C(7B)-C(6B) | 118.06(9) | C(44)-C(45)-C(46) | 120.22(18) |
| C(9B)-C(8B)-C(7B) | 119.77(10) | C(45)-C(46)-C(41) | 118.77(18) |
| C(10B)-C(9B)-C(8B) | 118.64(10) | C(56)-C(51)-C(52) | 118.9(3) |
| C(9B)-C(10B)-C(11B) | 120.32(10) | C(53)-C(52)-C(51) | 119.7(3) |
| N(1B)-C(11B)-C(10B) | 121.00(10) | C(52)-C(53)-C(54) | 121.4(2) |

| | |
|---|---|
| C(55)-C(54)-C(53) | 119.6(3) |
| C(56)-C(55)-C(54) | 118.6(3) |
| C(55)-C(56)-C(51) | 121.8(2) |
| C(62)-C(61)-C(66) | 120.00(12) |
| C(61)-C(62)-C(63) | 120.17(12) |
| C(64)-C(63)-C(62) | 120.08(13) |
| C(63)-C(64)-C(65) | 119.44(12) |
| C(64)-C(65)-C(66) | 120.25(13) |
| C(61)-C(66)-C(65) | 120.06(13) |
| C(72)-C(71)-C(76) | 119.99(13) |
| C(71)-C(72)-C(73) | 120.19(12) |
| C(72)-C(73)-C(74) | 119.88(13) |
| C(75)-C(74)-C(73) | 120.08(13) |
| C(74)-C(75)-C(76) | 119.72(12) |
| C(75)-C(76)-C(71) | 120.10(13) |
| C(86)-C(81)-C(82) | 118.51(13) |
| C(83)-C(82)-C(81) | 118.44(13) |
| C(84)-C(83)-C(82) | 120.46(13) |
| C(85)-C(84)-C(83) | 121.16(14) |
| C(84)-C(85)-C(86) | 121.11(14) |
| C(85)-C(86)-C(81) | 120.32(13) |

**Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for IAT98 (CCDC 856446). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12} ]$**

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Ti(1) | 102(1) | 100(1) | 127(1) | 4(1) | 20(1) | -2(1) |
| Cl(1A) | 209(1) | 210(1) | 139(1) | -34(1) | 14(1) | 8(1) |
| Cl(2A) | 149(1) | 154(1) | 254(1) | 9(1) | 47(1) | -40(1) |
| O(1A) | 113(4) | 119(4) | 168(4) | 22(3) | 13(3) | -2(3) |
| O(2A) | 116(4) | 97(3) | 146(4) | 4(3) | 22(3) | -2(3) |
| N(1A) | 104(4) | 101(4) | 133(4) | 7(3) | 17(3) | 11(3) |
| N(2A) | 142(5) | 111(4) | 159(4) | -11(3) | 33(4) | -15(4) |
| C(1A) | 110(5) | 108(5) | 151(5) | -22(4) | 21(4) | 19(4) |
| C(2A) | 147(6) | 104(5) | 172(5) | 7(4) | 35(4) | 16(4) |
| C(3A) | 167(6) | 117(5) | 169(5) | 28(4) | 22(4) | 34(4) |
| C(4A) | 124(5) | 133(5) | 172(5) | 6(4) | 21(4) | 15(4) |
| C(5A) | 121(5) | 122(5) | 182(5) | 11(4) | 43(4) | -1(4) |
| C(6A) | 122(5) | 99(5) | 150(5) | 4(4) | 29(4) | 26(4) |
| C(7A) | 114(5) | 107(5) | 155(5) | -2(4) | 23(4) | -1(4) |
| C(8A) | 122(5) | 190(6) | 220(6) | 46(5) | 58(5) | 44(4) |
| C(9A) | 154(6) | 228(6) | 216(6) | 65(5) | 94(5) | 40(5) |
| C(10A) | 167(6) | 165(5) | 193(6) | 70(4) | 57(5) | 34(4) |
| C(11A) | 124(5) | 110(5) | 145(5) | 10(4) | 19(4) | 7(4) |
| C(12A) | 114(5) | 125(5) | 113(5) | 7(4) | 18(4) | 22(4) |
| C(13A) | 132(5) | 136(5) | 160(5) | 21(4) | 44(4) | 2(4) |
| C(14A) | 148(5) | 123(5) | 139(5) | 12(4) | 27(4) | 28(4) |
| C(15A) | 127(5) | 144(5) | 125(5) | 2(4) | 16(4) | 45(4) |
| C(16A) | 109(5) | 131(5) | 90(5) | -18(4) | 3(4) | 12(4) |
| C(17A) | 133(5) | 101(5) | 92(5) | -7(4) | 11(4) | 7(4) |
| C(18A) | 170(6) | 147(5) | 267(6) | 85(5) | 29(5) | -14(5) |
| C(19A) | 236(7) | 299(7) | 285(7) | 94(5) | 100(6) | -37(5) |
| C(20A) | 264(7) | 150(6) | 419(8) | 28(5) | 34(6) | -57(5) |
| C(21A) | 254(7) | 303(7) | 480(9) | 246(6) | 8(6) | -48(6) |
| C(22A) | 130(6) | 199(6) | 236(6) | 44(5) | -7(5) | 3(5) |
| C(23A) | 221(7) | 388(8) | 424(8) | 41(6) | -38(6) | 82(6) |
| C(24A) | 258(7) | 402(8) | 289(7) | -9(6) | -51(6) | -25(6) |
| C(25A) | 196(7) | 485(9) | 456(9) | 202(7) | -62(6) | -70(6) |
| C(26A) | 176(6) | 143(5) | 279(6) | 90(5) | 83(5) | 52(4) |
| C(27A) | 193(6) | 151(6) | 369(7) | 95(5) | 74(5) | 16(5) |
| C(28A) | 500(9) | 109(6) | 492(9) | 32(6) | 257(7) | 57(6) |
| C(29A) | 194(7) | 383(8) | 404(8) | 253(6) | 84(6) | 68(6) |
| C(30A) | 94(5) | 150(5) | 158(5) | 3(4) | 20(4) | 4(4) |
| C(31A) | 127(6) | 186(6) | 261(6) | -19(5) | 5(5) | -22(4) |
| C(32A) | 126(5) | 201(6) | 239(6) | 12(5) | 43(5) | 29(5) |
| C(33A) | 172(6) | 236(6) | 189(6) | 29(5) | 70(5) | 25(5) |
| C(34A) | 157(6) | 149(5) | 216(6) | -17(4) | 46(5) | -12(4) |
| C(35A) | 216(6) | 186(6) | 294(7) | -70(5) | 137(5) | -28(5) |
| C(36A) | 317(7) | 197(6) | 208(6) | -66(5) | 132(5) | -93(5) |
| C(37A) | 318(7) | 181(6) | 164(6) | -9(4) | 29(5) | -40(5) |
| C(38A) | 222(6) | 146(5) | 176(6) | -11(4) | 27(5) | -3(5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Ti(2) | 106(1) | 105(1) | 127(1) | -10(1) | 9(1) | -12(1) |
| Cl(1B) | 138(1) | 199(1) | 189(1) | -35(1) | 47(1) | -16(1) |
| Cl(2B) | 165(1) | 173(1) | 155(1) | -31(1) | 6(1) | -52(1) |
| O(1B) | 121(4) | 114(3) | 150(4) | -11(3) | 0(3) | -9(3) |
| O(2B) | 134(4) | 105(3) | 171(4) | 9(3) | 1(3) | -28(3) |
| N(1B) | 92(4) | 109(4) | 153(4) | -15(3) | 12(3) | -3(3) |
| N(2B) | 128(5) | 152(4) | 163(5) | -28(4) | 10(4) | -21(4) |
| C(1B) | 107(5) | 145(5) | 124(5) | 7(4) | 31(4) | -9(4) |
| C(2B) | 141(5) | 125(5) | 161(5) | -4(4) | 51(4) | 8(4) |
| C(3B) | 192(6) | 158(5) | 184(6) | 12(4) | 34(5) | 58(5) |
| C(4B) | 198(6) | 206(6) | 151(5) | -8(4) | -1(5) | 63(5) |
| C(5B) | 172(6) | 179(6) | 147(5) | -38(4) | 18(4) | 12(4) |
| C(6B) | 125(5) | 136(5) | 126(5) | -3(4) | 34(4) | 14(4) |
| C(7B) | 92(5) | 138(5) | 154(5) | -26(4) | 6(4) | -8(4) |
| C(8B) | 156(6) | 179(6) | 151(5) | -33(4) | 2(4) | 6(4) |
| C(9B) | 191(6) | 191(6) | 191(6) | -77(5) | 18(5) | 31(5) |
| C(10B) | 172(6) | 126(5) | 226(6) | -40(4) | -1(5) | 26(4) |
| C(11B) | 90(5) | 125(5) | 194(5) | -25(4) | -4(4) | -4(4) |
| C(12B) | 109(5) | 110(5) | 200(6) | -7(4) | 15(4) | 10(4) |
| C(13B) | 140(6) | 124(5) | 243(6) | -39(4) | -4(5) | 3(4) |
| C(14B) | 130(5) | 120(5) | 275(6) | 7(5) | 31(5) | 3(4) |
| C(15B) | 148(6) | 152(5) | 231(6) | 48(4) | 28(5) | 5(4) |
| C(16B) | 112(5) | 131(5) | 205(6) | 25(4) | 20(4) | 7(4) |
| C(17B) | 102(5) | 106(5) | 210(6) | 3(4) | 23(4) | 0(4) |
| C(18B) | 180(6) | 115(5) | 228(6) | -27(4) | 30(5) | -9(4) |
| C(19B) | 327(8) | 142(6) | 383(8) | -34(5) | -13(6) | 28(5) |
| C(20B) | 216(6) | 182(6) | 335(7) | -45(5) | 70(5) | -58(5) |
| C(21B) | 272(7) | 183(6) | 244(6) | -84(5) | 71(5) | -31(5) |
| C(22B) | 329(8) | 296(7) | 233(7) | -72(5) | -112(6) | 142(6) |
| C(23B) | 779(13) | 421(9) | 536(10) | -160(8) | -409(9) | 347(9) |
| C(24B) | 450(9) | 334(7) | 192(6) | -5(5) | -98(6) | 75(6) |
| C(25B) | 215(8) | 760(11) | 431(9) | -217(8) | -78(7) | 56(8) |
| C(26B) | 183(6) | 102(5) | 328(7) | 15(5) | 30(5) | -22(4) |
| C(27B) | 277(7) | 156(6) | 415(8) | -7(5) | 28(6) | -62(5) |
| C(28B) | 238(7) | 169(6) | 452(8) | 15(5) | 99(6) | -51(5) |
| C(29B) | 252(7) | 137(6) | 451(8) | 47(5) | 55(6) | 2(5) |
| C(30B) | 149(6) | 178(6) | 185(6) | 37(4) | -5(4) | -26(4) |
| C(31B) | 143(6) | 214(6) | 243(6) | 32(5) | -21(5) | -3(5) |
| C(32B) | 172(6) | 225(6) | 185(6) | -5(5) | 21(5) | -36(5) |
| C(33B) | 289(7) | 254(7) | 221(6) | 69(5) | -12(5) | -61(5) |
| C(34B) | 215(6) | 236(6) | 267(6) | 38(5) | 82(5) | 25(5) |
| C(35B) | 305(8) | 410(8) | 312(7) | 40(6) | 177(6) | 2(6) |
| C(36B) | 180(7) | 454(8) | 345(8) | -106(6) | 121(6) | -16(6) |
| C(37B) | 175(7) | 444(8) | 329(7) | -40(6) | 33(6) | 108(6) |
| C(38B) | 176(6) | 333(7) | 224(6) | 10(5) | 14(5) | 70(5) |
| | | | | | | |
| C(41) | 768(15) | 614(12) | 625(13) | -185(10) | 356(12) | -219(11) |
| C(42) | 716(13) | 438(10) | 418(10) | -107(8) | 155(9) | 34(9) |
| C(43) | 505(10) | 516(10) | 371(9) | -149(8) | 105(8) | 7(8) |
| C(44) | 567(12) | 518(10) | 417(9) | -49(8) | 154(9) | -7(9) |
| C(45) | 687(14) | 705(13) | 507(11) | 53(9) | 95(10) | 264(11) |
| C(46) | 373(11) | 1098(17) | 792(15) | -337(14) | 171(11) | 24(12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(51) | 1110(20) | 1210(20) | 1250(20) | 732(18) | 854(18) | 582(17) |
| C(52) | 980(20) | 1950(30) | 462(13) | 287(15) | 292(14) | 810(20) |
| C(53) | 691(16) | 2530(30) | 315(11) | 26(16) | 150(11) | 920(20) |
| C(54) | 830(16) | 1990(30) | 444(12) | 359(15) | 310(12) | 828(17) |
| C(55) | 1270(20) | 1187(19) | 1015(18) | 650(15) | 905(18) | 750(17) |
| C(56) | 1420(30) | 934(18) | 1790(30) | 770(19) | 1400(20) | 700(18) |
| | | | | | | |
| C(61) | 243(7) | 224(7) | 547(9) | -59(6) | 154(7) | -40(6) |
| C(62) | 428(9) | 255(7) | 301(7) | -20(6) | 159(7) | -116(6) |
| C(63) | 289(8) | 238(7) | 471(9) | 85(6) | -45(7) | -39(6) |
| C(64) | 259(7) | 240(7) | 663(11) | -33(7) | 241(7) | -30(6) |
| C(65) | 552(10) | 348(8) | 310(8) | 1(6) | 211(7) | -89(7) |
| C(66) | 353(8) | 303(7) | 419(8) | 47(6) | -40(7) | -2(6) |
| | | | | | | |
| C(71) | 195(7) | 443(8) | 358(8) | -146(6) | 110(6) | -71(6) |
| C(72) | 178(7) | 618(10) | 234(7) | -112(7) | 20(5) | 59(6) |
| C(73) | 332(8) | 544(10) | 273(7) | 92(7) | 48(6) | 158(7) |
| C(74) | 249(7) | 335(8) | 401(8) | 0(6) | 7(6) | 82(6) |
| C(75) | 154(6) | 413(8) | 275(7) | -103(6) | 4(5) | 24(6) |
| C(76) | 254(7) | 476(9) | 267(7) | -53(6) | 119(6) | -52(6) |
| | | | | | | |
| C(81) | 675(12) | 243(8) | 429(9) | 55(6) | -201(8) | 21(8) |
| C(82) | 226(8) | 585(11) | 689(12) | -289(9) | -4(8) | 80(8) |
| C(83) | 452(10) | 418(9) | 434(9) | -77(7) | 199(8) | -214(8) |
| C(84) | 484(10) | 256(7) | 316(8) | -10(6) | 43(7) | -62(7) |
| C(85) | 323(8) | 368(8) | 306(7) | -57(6) | 33(6) | -13(6) |
| C(86) | 399(9) | 419(8) | 232(7) | 33(6) | 48(6) | -130(7) |

# CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 27 April 2011

**Crystal Structure Analysis of:**

**Complex 7 (ONO)Ti(HNMe$_2$)(C$_2$H$_4$) (IAT87)**

(shown below)

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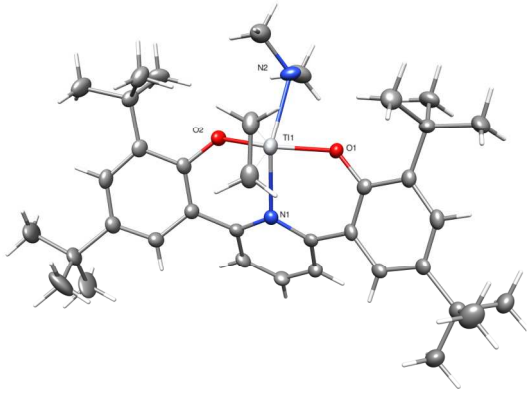
Contents






IAT87

**Note:** The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 822994.

**Table 1. Crystal data and structure refinement for IAT87 (CCDC 822994).**

| | |
|---|---|
| Empirical formula | $C_{37}H_{53}N_2O_2Ti \bullet 0.5(C_5H_{12})$ |
| Formula weight | 641.79 |
| Crystallization Solvent | Pentane |
| Crystal Habit | Blade |
| Crystal size | 0.21 x 0.13 x 0.08 $mm^3$ |
| Crystal color | Dark red |

## Data Collection

| | | |
|---|---|---|
| Type of diffractometer | Bruker KAPPA APEX II | |
| Wavelength | 0.71073 Å MoKα | |
| Data Collection Temperature | 100(2) K | |
| θ range for 9906 reflections used in lattice determination | 2.18 to 25.01° | |
| Unit cell dimensions | a = 17.7536(5) Å<br>b = 10.5239(3) Å<br>c = 20.7880(6) Å | α= 90°<br>β= 104.343(2)°<br>γ = 90° |
| Volume | 3762.91(19) $Å^3$ | |
| Z | 4 | |
| Crystal system | Monoclinic | |
| Space group | P $2_1/n$ | |
| Density (calculated) | 1.133 $Mg/m^3$ | |
| F(000) | 1392 | |
| θ range for data collection | 2.02 to 29.99° | |
| Completeness to θ = 29.99° | 93.2 % | |
| Index ranges | $-23 \le h \le 24$, $-14 \le k \le 14$, $-28 \le l \le 29$ | |
| Data collection scan type | ω scans; 14 settings | |
| Reflections collected | 109497 | |
| Independent reflections | 10215 [$R_{int}$= 0.0878] | |
| Absorption coefficient | 0.261 $mm^{-1}$ | |
| Absorption correction | None | |
| Max. and min. transmission | 0.9794 and 0.9472 | |

**Table 1 (cont.)**

## Structure solution and Refinement

| | |
|---|---|
| Structure solution program | SHELXS-97 (Sheldrick, 2008) |
| Primary solution method | Direct methods |
| Secondary solution method | Difference Fourier map |
| Hydrogen placement | Geometric positions |
| Structure refinement program | SHELXL-97 (Sheldrick, 2008) |
| Refinement method | Full matrix least-squares on $F^2$ |
| Data / restraints / parameters | 10215 / 7 / 464 |
| Treatment of hydrogen atoms | Riding |
| Goodness-of-fit on $F^2$ | 2.095 |
| Final R indices [$I>2\sigma(I)$, 5835 reflections] | R1 = 0.0612, *w*R2 = 0.0614 |
| R indices (all data) | R1 = 0.1167, *w*R2 = 0.0627 |
| 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 |
| Largest diff. peak and hole | 1.294 and -0.596 e.Å$^{-3}$ |

## Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

One t-Bu is disordered by the common rotation around the central carbon and was included in refinement as both positions with a total occupancy of one. Pentane is included as a solvent of crystallization and sits near a center of symmetry and bridges that center. It was included as a restrained pentane at half occupancy.

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.

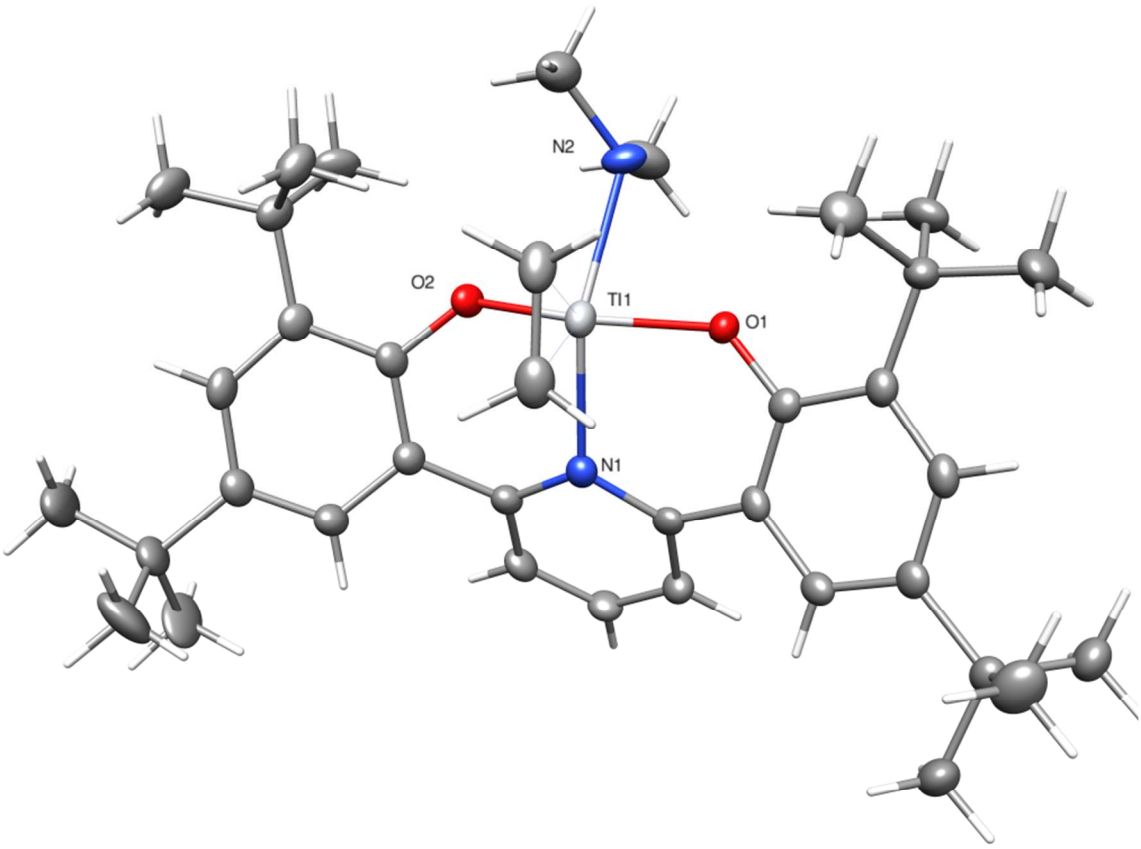

N2
O2
Tl1
O1
N1

**Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for IAT87 (CCDC 822994). U(eq) is defined as the trace of the orthogonalized $U^{ij}$ tensor.**

| | x | y | z | $U_{eq}$ | Occ |
|---|---|---|---|---|---|
| Ti(1) | 6825(1) | 1750(1) | -437(1) | 26(1) | 1 |
| O(1) | 7089(1) | 1327(1) | -1243(1) | 24(1) | 1 |
| O(2) | 6142(1) | 1328(1) | 100(1) | 26(1) | 1 |
| N(1) | 7463(1) | -17(2) | -36(1) | 22(1) | 1 |
| N(2) | 5779(1) | 2486(2) | -1209(1) | 31(1) | 1 |
| C(1) | 7798(1) | 986(2) | -1315(1) | 23(1) | 1 |
| C(2) | 8099(1) | 1520(2) | -1820(1) | 25(1) | 1 |
| C(3) | 8832(1) | 1134(2) | -1852(1) | 26(1) | 1 |
| C(4) | 9288(1) | 244(2) | -1425(1) | 24(1) | 1 |
| C(5) | 8966(1) | -283(2) | -950(1) | 24(1) | 1 |
| C(6) | 8219(1) | 58(2) | -890(1) | 22(1) | 1 |
| C(7) | 7892(1) | -637(2) | -403(1) | 22(1) | 1 |
| C(8) | 8037(1) | -1944(2) | -320(1) | 27(1) | 1 |
| C(9) | 7782(1) | -2619(2) | 146(1) | 27(1) | 1 |
| C(10) | 7404(1) | -1973(2) | 547(1) | 27(1) | 1 |
| C(11) | 7258(1) | -676(2) | 463(1) | 24(1) | 1 |
| C(12) | 6881(1) | -27(2) | 931(1) | 24(1) | 1 |
| C(13) | 7087(1) | -415(2) | 1597(1) | 29(1) | 1 |
| C(14) | 6726(1) | 59(2) | 2064(1) | 31(1) | 1 |
| C(15) | 6130(1) | 940(2) | 1839(1) | 29(1) | 1 |
| C(16) | 5897(1) | 1375(2) | 1189(1) | 26(1) | 1 |
| C(17) | 6311(1) | 907(2) | 731(1) | 24(1) | 1 |
| C(18) | 7630(1) | 2503(2) | -2314(1) | 28(1) | 1 |
| C(19) | 6862(1) | 1940(2) | -2699(1) | 38(1) | 1 |
| C(20) | 7471(1) | 3687(2) | -1939(1) | 36(1) | 1 |
| C(21) | 8077(1) | 2950(2) | -2816(1) | 45(1) | 1 |
| C(22) | 10098(1) | -94(2) | -1503(1) | 28(1) | 1 |
| C(23) | 10600(1) | 1106(2) | -1443(1) | 48(1) | 1 |
| C(24) | 10029(1) | -691(2) | -2188(1) | 38(1) | 1 |
| C(25) | 10512(1) | -1037(2) | -981(1) | 47(1) | 1 |
| C(26) | 6981(2) | -340(3) | 2800(1) | 39(1) | 1 |
| C(27A) | 7772(3) | 382(5) | 3108(2) | 63(2) | 0.701(8) |
| C(28A) | 7116(4) | -1713(7) | 2865(3) | 60(2) | 0.701(8) |
| C(29A) | 6441(3) | 139(6) | 3206(2) | 64(2) | 0.701(8) |
| C(27B) | 7619(9) | -1360(20) | 2940(7) | 97(9) | 0.299(8) |
| C(28B) | 6257(6) | -1090(17) | 2975(5) | 84(7) | 0.299(8) |
| C(29B) | 7171(9) | 674(11) | 3258(5) | 71(5) | 0.299(8) |
| C(30) | 5222(1) | 2315(2) | 968(1) | 28(1) | 1 |
| C(31) | 4594(1) | 1748(2) | 400(1) | 39(1) | 1 |
| C(32) | 4834(1) | 2631(2) | 1530(1) | 44(1) | 1 |
| C(33) | 5522(1) | 3574(2) | 747(1) | 39(1) | 1 |
| C(34) | 7756(1) | 2804(2) | 128(1) | 35(1) | 1 |
| C(35) | 7164(1) | 3668(2) | -203(1) | 34(1) | 1 |
| C(36) | 5306(1) | 3518(2) | -1054(1) | 50(1) | 1 |
| C(37) | 5267(1) | 1399(2) | -1468(1) | 49(1) | 1 |

| | | | | | |
|---|---|---|---|---|---|
| C(41) | 9722(8) | 5208(8) | 8889(4) | 371(16) | 0.50 |
| C(42) | 10005(6) | 4068(7) | 9349(4) | 213(7) | 0.50 |
| C(43) | 9933(6) | 4262(8) | 10064(3) | 178(6) | 0.50 |
| C(44) | 10476(5) | 5294(9) | 10450(4) | 136(5) | 0.50 |
| C(45) | 10312(5) | 5626(8) | 11124(3) | 128(5) | 0.50 |

**Table 3. Selected bond lengths [Å] and angles [°] for IAT87 (CCDC 822994).**

| | | | |
|---|---|---|---|
| Ti(1)-O(2) | 1.8924(14) | O(2)-Ti(1)-O(1) | 143.01(6) |
| Ti(1)-O(1) | 1.9014(14) | O(2)-Ti(1)-C(34) | 108.97(8) |
| Ti(1)-C(34) | 2.093(2) | O(1)-Ti(1)-C(34) | 106.60(8) |
| Ti(1)-C(35) | 2.128(2) | O(2)-Ti(1)-C(35) | 105.92(8) |
| Ti(1)-N(1) | 2.2288(17) | O(1)-Ti(1)-C(35) | 107.80(8) |
| Ti(1)-N(2) | 2.2677(17) | C(34)-Ti(1)-C(35) | 39.59(7) |
| | | O(2)-Ti(1)-N(1) | 85.62(6) |
| | | O(1)-Ti(1)-N(1) | 85.47(6) |
| | | C(34)-Ti(1)-N(1) | 88.55(8) |
| | | C(35)-Ti(1)-N(1) | 128.08(7) |
| | | O(2)-Ti(1)-N(2) | 88.05(6) |
| | | O(1)-Ti(1)-N(2) | 78.12(6) |
| | | C(34)-Ti(1)-N(2) | 127.60(8) |
| | | C(35)-Ti(1)-N(2) | 88.49(8) |
| | | N(1)-Ti(1)-N(2) | 143.18(7) |

**Table 4. Bond lengths [Å] and angles [°] for IAT87 (CCDC 822994).**

| | | | |
|---|---|---|---|
| Ti(1)-O(2) | 1.8924(14) | C(42)-C(43) | 1.5373(10) |
| Ti(1)-O(1) | 1.9014(14) | C(43)-C(44) | 1.5397(10) |
| Ti(1)-C(34) | 2.093(2) | C(44)-C(45) | 1.5409(10) |
| Ti(1)-C(35) | 2.128(2) | | |
| Ti(1)-N(1) | 2.2288(17) | O(2)-Ti(1)-O(1) | 143.01(6) |
| Ti(1)-N(2) | 2.2677(17) | O(2)-Ti(1)-C(34) | 108.97(8) |
| O(1)-C(1) | 1.351(2) | O(1)-Ti(1)-C(34) | 106.60(8) |
| O(2)-C(17) | 1.347(2) | O(2)-Ti(1)-C(35) | 105.92(8) |
| N(1)-C(11) | 1.369(3) | O(1)-Ti(1)-C(35) | 107.80(8) |
| N(1)-C(7) | 1.370(3) | C(34)-Ti(1)-C(35) | 39.59(7) |
| N(2)-C(36) | 1.458(2) | O(2)-Ti(1)-N(1) | 85.62(6) |
| N(2)-C(37) | 1.478(2) | O(1)-Ti(1)-N(1) | 85.47(6) |
| C(1)-C(6) | 1.401(3) | C(34)-Ti(1)-N(1) | 88.55(8) |
| C(1)-C(2) | 1.410(3) | C(35)-Ti(1)-N(1) | 128.08(7) |
| C(2)-C(3) | 1.380(3) | O(2)-Ti(1)-N(2) | 88.05(6) |
| C(2)-C(18) | 1.547(3) | O(1)-Ti(1)-N(2) | 78.12(6) |
| C(3)-C(4) | 1.401(3) | C(34)-Ti(1)-N(2) | 127.60(8) |
| C(4)-C(5) | 1.375(3) | C(35)-Ti(1)-N(2) | 88.49(8) |
| C(4)-C(22) | 1.529(3) | N(1)-Ti(1)-N(2) | 143.18(7) |
| C(5)-C(6) | 1.407(3) | C(1)-O(1)-Ti(1) | 126.81(13) |
| C(6)-C(7) | 1.481(3) | C(17)-O(2)-Ti(1) | 129.15(13) |
| C(7)-C(8) | 1.403(3) | C(11)-N(1)-C(7) | 117.98(19) |
| C(8)-C(9) | 1.365(3) | C(11)-N(1)-Ti(1) | 120.03(15) |
| C(9)-C(10) | 1.373(3) | C(7)-N(1)-Ti(1) | 119.33(15) |
| C(10)-C(11) | 1.393(3) | C(36)-N(2)-C(37) | 108.71(18) |
| C(11)-C(12) | 1.478(3) | C(36)-N(2)-Ti(1) | 120.91(14) |
| C(12)-C(17) | 1.397(3) | C(37)-N(2)-Ti(1) | 108.43(13) |
| C(12)-C(13) | 1.402(3) | O(1)-C(1)-C(6) | 118.8(2) |
| C(13)-C(14) | 1.384(3) | O(1)-C(1)-C(2) | 120.8(2) |
| C(14)-C(15) | 1.398(3) | C(6)-C(1)-C(2) | 120.3(2) |
| C(14)-C(26) | 1.542(3) | C(3)-C(2)-C(1) | 117.0(2) |
| C(15)-C(16) | 1.389(3) | C(3)-C(2)-C(18) | 121.6(2) |
| C(16)-C(17) | 1.426(3) | C(1)-C(2)-C(18) | 121.3(2) |
| C(16)-C(30) | 1.534(3) | C(2)-C(3)-C(4) | 124.7(2) |
| C(18)-C(19) | 1.520(3) | C(5)-C(4)-C(3) | 116.6(2) |
| C(18)-C(21) | 1.533(3) | C(5)-C(4)-C(22) | 123.5(2) |
| C(18)-C(20) | 1.533(3) | C(3)-C(4)-C(22) | 119.9(2) |
| C(22)-C(25) | 1.518(3) | C(4)-C(5)-C(6) | 121.8(2) |
| C(22)-C(24) | 1.533(3) | C(1)-C(6)-C(5) | 119.4(2) |
| C(22)-C(23) | 1.533(3) | C(1)-C(6)-C(7) | 122.4(2) |
| C(26)-C(29B) | 1.414(11) | C(5)-C(6)-C(7) | 118.2(2) |
| C(26)-C(28A) | 1.466(7) | N(1)-C(7)-C(8) | 120.7(2) |
| C(26)-C(29A) | 1.512(4) | N(1)-C(7)-C(6) | 120.9(2) |
| C(26)-C(27B) | 1.535(17) | C(8)-C(7)-C(6) | 118.5(2) |
| C(26)-C(27A) | 1.584(5) | C(9)-C(8)-C(7) | 120.9(2) |
| C(26)-C(28B) | 1.625(10) | C(8)-C(9)-C(10) | 118.1(2) |
| C(30)-C(31) | 1.530(3) | C(9)-C(10)-C(11) | 120.8(2) |
| C(30)-C(32) | 1.532(3) | N(1)-C(11)-C(10) | 121.0(2) |
| C(30)-C(33) | 1.540(3) | N(1)-C(11)-C(12) | 120.9(2) |
| C(34)-C(35) | 1.430(2) | C(10)-C(11)-C(12) | 118.1(2) |
| C(41)-C(42) | 1.5389(10) | C(17)-C(12)-C(13) | 119.4(2) |

| | | | |
|---|---|---|---|
| C(17)-C(12)-C(11) | 122.8(2) | C(43)-C(42)-C(41) | 113.81(11) |
| C(13)-C(12)-C(11) | 117.7(2) | C(42)-C(43)-C(44) | 113.90(11) |
| C(14)-C(13)-C(12) | 122.4(2) | C(43)-C(44)-C(45) | 113.48(11) |
| C(13)-C(14)-C(15) | 116.6(2) | | |
| C(13)-C(14)-C(26) | 121.7(2) | | |
| C(15)-C(14)-C(26) | 121.7(2) | | |
| C(16)-C(15)-C(14) | 124.2(2) | | |
| C(15)-C(16)-C(17) | 117.2(2) | | |
| C(15)-C(16)-C(30) | 121.7(2) | | |
| C(17)-C(16)-C(30) | 121.2(2) | | |
| O(2)-C(17)-C(12) | 119.4(2) | | |
| O(2)-C(17)-C(16) | 120.6(2) | | |
| C(12)-C(17)-C(16) | 120.0(2) | | |
| C(19)-C(18)-C(21) | 108.06(19) | | |
| C(19)-C(18)-C(20) | 109.22(19) | | |
| C(21)-C(18)-C(20) | 106.75(19) | | |
| C(19)-C(18)-C(2) | 110.73(19) | | |
| C(21)-C(18)-C(2) | 111.86(19) | | |
| C(20)-C(18)-C(2) | 110.09(18) | | |
| C(25)-C(22)-C(4) | 112.25(19) | | |
| C(25)-C(22)-C(24) | 107.99(19) | | |
| C(4)-C(22)-C(24) | 109.44(17) | | |
| C(25)-C(22)-C(23) | 108.15(19) | | |
| C(4)-C(22)-C(23) | 110.00(19) | | |
| C(24)-C(22)-C(23) | 108.93(19) | | |
| C(29B)-C(26)-C(28A) | 132.5(5) | | |
| C(29B)-C(26)-C(29A) | 56.7(5) | | |
| C(28A)-C(26)-C(29A) | 112.9(4) | | |
| C(29B)-C(26)-C(27B) | 111.0(8) | | |
| C(28A)-C(26)-C(27B) | 36.6(7) | | |
| C(29A)-C(26)-C(27B) | 131.9(6) | | |
| C(29B)-C(26)-C(14) | 115.1(5) | | |
| C(28A)-C(26)-C(14) | 111.2(3) | | |
| C(29A)-C(26)-C(14) | 113.0(2) | | |
| C(27B)-C(26)-C(14) | 113.6(6) | | |
| C(29B)-C(26)-C(27A) | 47.7(6) | | |
| C(28A)-C(26)-C(27A) | 109.1(3) | | |
| C(29A)-C(26)-C(27A) | 103.7(3) | | |
| C(27B)-C(26)-C(27A) | 73.8(7) | | |
| C(14)-C(26)-C(27A) | 106.4(2) | | |
| C(29B)-C(26)-C(28B) | 106.6(7) | | |
| C(28A)-C(26)-C(28B) | 67.7(6) | | |
| C(29A)-C(26)-C(28B) | 52.4(5) | | |
| C(27B)-C(26)-C(28B) | 101.9(7) | | |
| C(14)-C(26)-C(28B) | 107.5(4) | | |
| C(27A)-C(26)-C(28B) | 144.4(4) | | |
| C(31)-C(30)-C(32) | 107.17(19) | | |
| C(31)-C(30)-C(16) | 110.21(19) | | |
| C(32)-C(30)-C(16) | 112.19(19) | | |
| C(31)-C(30)-C(33) | 110.10(19) | | |
| C(32)-C(30)-C(33) | 107.12(18) | | |
| C(16)-C(30)-C(33) | 109.96(18) | | |
| C(35)-C(34)-Ti(1) | 71.52(12) | | |
| C(34)-C(35)-Ti(1) | 68.89(12) | | |

**Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for IAT87 (CCDC 822994). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12} ]$**

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Ti(1) | 310(3) | 256(2) | 226(2) | 3(2) | 113(2) | 44(2) |
| O(1) | 249(10) | 283(10) | 201(8) | 7(7) | 80(7) | 71(8) |
| O(2) | 265(10) | 290(10) | 235(9) | 47(8) | 95(8) | 50(8) |
| N(1) | 226(12) | 251(12) | 189(10) | -21(9) | 34(9) | 25(10) |
| N(2) | 292(14) | 224(12) | 378(13) | -12(10) | 32(10) | 91(10) |
| C(1) | 237(15) | 239(15) | 199(13) | -53(11) | 46(12) | 27(12) |
| C(2) | 281(16) | 273(16) | 193(13) | -25(11) | 77(11) | 18(12) |
| C(3) | 316(16) | 294(15) | 189(13) | -24(11) | 97(12) | -14(13) |
| C(4) | 257(16) | 288(15) | 162(13) | -58(11) | 34(11) | 14(12) |
| C(5) | 275(16) | 246(15) | 170(13) | -29(11) | 21(11) | 51(12) |
| C(6) | 282(16) | 223(14) | 148(12) | -31(11) | 52(11) | 6(12) |
| C(7) | 197(15) | 273(15) | 161(12) | -42(11) | -10(11) | 46(12) |
| C(8) | 313(16) | 291(16) | 217(13) | -22(12) | 65(11) | 85(13) |
| C(9) | 327(16) | 213(14) | 259(14) | 24(12) | 49(12) | 62(12) |
| C(10) | 309(16) | 296(16) | 207(13) | 54(12) | 48(11) | 20(13) |
| C(11) | 214(15) | 290(15) | 196(13) | 13(12) | 28(11) | 52(12) |
| C(12) | 243(15) | 272(15) | 224(13) | 9(12) | 68(12) | 28(12) |
| C(13) | 284(16) | 343(16) | 241(14) | 27(12) | 56(12) | 74(12) |
| C(14) | 310(17) | 372(17) | 237(14) | 4(13) | 75(12) | 31(13) |
| C(15) | 327(17) | 326(16) | 261(14) | -44(12) | 141(12) | -15(13) |
| C(16) | 258(15) | 259(15) | 282(14) | -8(12) | 109(12) | -25(12) |
| C(17) | 238(15) | 277(15) | 212(13) | 14(12) | 68(12) | -19(12) |
| C(18) | 302(17) | 315(16) | 236(14) | 61(12) | 88(12) | 130(13) |
| C(19) | 408(17) | 444(18) | 254(14) | 41(13) | 5(12) | 157(15) |
| C(20) | 457(18) | 284(16) | 366(15) | 57(13) | 133(13) | 51(13) |
| C(21) | 530(19) | 550(20) | 327(15) | 215(14) | 198(14) | 251(15) |
| C(22) | 217(15) | 392(17) | 215(13) | -33(13) | 39(12) | 31(13) |
| C(23) | 299(18) | 660(20) | 471(18) | -130(16) | 65(14) | -36(15) |
| C(24) | 323(17) | 517(19) | 339(16) | -3(14) | 143(13) | 85(14) |
| C(25) | 298(17) | 790(20) | 329(16) | 18(15) | 81(13) | 194(15) |
| C(26) | 480(20) | 510(20) | 195(15) | 64(14) | 98(14) | 148(17) |
| C(27A) | 660(40) | 830(40) | 260(30) | 80(30) | -130(30) | 10(30) |
| C(28A) | 1040(60) | 510(30) | 330(30) | 220(30) | 330(40) | 180(50) |
| C(29A) | 710(40) | 990(60) | 270(20) | 130(30) | 200(30) | 380(40) |
| C(27B) | 1090(150) | 1700(200) | 70(50) | 230(90) | 10(100) | 740(160) |
| C(28B) | 680(90) | 1290(160) | 430(70) | 520(90) | -110(60) | -400(90) |
| C(29B) | 850(140) | 960(110) | 260(60) | 150(70) | 0(70) | -240(90) |
| C(30) | 287(16) | 274(15) | 323(15) | 11(12) | 131(13) | 39(13) |
| C(31) | 268(16) | 423(17) | 467(16) | 92(15) | 87(13) | 78(14) |
| C(32) | 451(19) | 449(18) | 495(18) | 75(15) | 280(15) | 149(14) |
| C(33) | 473(18) | 334(17) | 431(16) | -27(13) | 227(14) | 91(14) |
| C(34) | 363(17) | 394(17) | 302(15) | -95(13) | 103(13) | -66(13) |
| C(35) | 499(19) | 251(15) | 317(15) | -34(12) | 196(13) | -22(13) |
| C(36) | 570(20) | 540(20) | 407(16) | 130(15) | 174(14) | 229(16) |
| C(37) | 425(19) | 520(20) | 444(18) | 111(15) | -25(14) | 60(16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(41) | 940(100) | 580(80) | 8700(400) | 1150(170) | -530(180) | -70(80) |
| C(42) | 2070(150) | 2720(170) | 1530(110) | -240(110) | 280(110) | 1720(110) |
| C(43) | 1430(100) | 2360(140) | 1190(90) | 990(100) | -370(80) | -890(100) |
| C(44) | 1160(90) | 2040(140) | 1090(80) | 960(90) | 700(80) | -10(90) |
| C(45) | 1050(80) | 620(70) | 1650(110) | 410(60) | -690(70) | -10(60) |