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Bis(acetylacetonato-κ^2O,O')[2,2′-methylenebis(4,6-xylenolato)-κ^2O,O']-titanium(IV)

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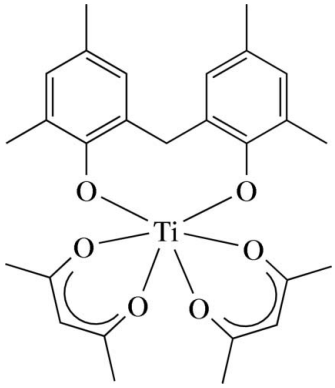
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.127; data-to-parameter ratio = 14.7.

The title compound, $[Ti(C_5H_7O_2)_2(C_{17}H_{18}O_2)]$, was synthesized by the reaction of $Ti(acac)_2Cl_2$ (acac = acetylacetonate) with one equivalent of the diol $\{[HOC_6H_2(Me)_2](\mu\text{-}CH_2)[(Me)_2C_6H_2OH]\}$ in the presence of sodium acetate. The coordination about the Ti atom is slightly distorted octahedral and the bisphenoxo ligand adopts an 'open book' conformation.

Related literature

For related literature, see: Chuck *et al.* (2006); Fokken *et al.* (1996); Groysman *et al.* (2003); Hanava *et al.* (2003); Priya *et al.* (2004).



Experimental

Crystal data

$[Ti(C_5H_7O_2)_2(C_{17}H_{18}O_2)]$
M_r = 500.43
Triclinic, $P\overline{1}$
a = 8.3605 (9) Å
b = 9.3419 (7) Å
c = 17.652 (1) Å
α = 92.472 (5)°
β = 95.599 (7)°
γ = 107.209 (7)°
V = 1306.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 298 (2) K
0.46 × 0.40 × 0.26 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.850, T_{max} = 0.911
4973 measured reflections
4628 independent reflections
3123 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
2 standard reflections
frequency: 2 min
intensity decay: <1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.127
S = 1.02
4628 reflections
314 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—O6	1.798 (2)	Ti1—O2	2.002 (2)
Ti1—O5	1.839 (2)	Ti1—O1	2.040 (2)
Ti1—O4	1.989 (2)	Ti1—O3	2.046 (2)
O6—Ti1—O5	95.80 (8)	O4—Ti1—O1	87.20 (8)
O6—Ti1—O4	96.15 (8)	O2—Ti1—O1	83.46 (8)
O5—Ti1—O4	90.98 (8)	O6—Ti1—O3	90.75 (8)
O6—Ti1—O2	92.84 (8)	O5—Ti1—O3	171.80 (8)
O5—Ti1—O2	95.41 (8)	O4—Ti1—O3	83.40 (8)
O4—Ti1—O2	168.39 (8)	O2—Ti1—O3	89.17 (8)
O6—Ti1—O1	175.50 (9)	O1—Ti1—O3	86.64 (8)
O5—Ti1—O1	87.13 (8)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2129).

References

Bruker (1997). *SHELXTL* Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Chuck, C. J., Davidson, M. G., Jones, M. D., Kociok-Kohn, G., Lunn, M. D. & Wu, S. (2006). *Inorg. Chem.* **45**, 6595–6597.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fokken, S., Spaniol, T. P., Kang, H.-C., Massa, W. & Okuda, J. (1996). *Organometallics*, **15**, 5069–5072.
Groysman, S., Goldberg, I., Kol, M., Genizi, E. & Goldschmidt, Z. (2003). *Inorg. Chim. Acta*, **345**, 137–144.
Hanava, H., Hashimoto, T. & Maruoka, K. (2003). *J. Am. Chem. Soc.* **125**, 1708–1709.
Harms, K. (1996). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Priya, S., Balakrishna, M. S. & Mague, J. T. (2004). *Chem. Lett.* **33**, 308–309.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

Acta Cryst. (2007). E**63**, m1815 [**doi:10.1107/S1600536807026761**]

Bis(acetylacetonato-κ^2O,O')[2,2'-methylenebis(4,6-xylenolato)-κ^2O,O']titanium(IV)

M. S. Balakrishna, R. Panda and J. T. Mague

Comment

Titanium complexes containing aryloxides with bulky substituents are of interest in homogeneous catalysis, particularly in relation to polymerization processes (Priya *et al.*, 2004; Fokken *et al.*,1996; Groysman *et al.*, 2003). When bulky phenolate ligands are employed instead of metallocenes better results were obtained in polymerization of olefins (Hanava *et al.*, 2003). The utility of titanium aryloxides in catalytic reactions such as olefin polymerization, oxidation, epoxidation and carbon-carbon coupling reactions are well documented (Chuck *et al.*, 2006). Here, we report the mixed ligand complex $[(–OC(CH_3)CHC(CH_3)O–)_2Ti\{(–OC_6H_2(Me)_2)(\mu\text{-}CH_2)\ ((Me)_2C_6H_2O–)\}]$.

The coordination about the titanium(IV) center is distorted octahedral (Table 1). The acetylacetonate ligands are planar within 0.01 Å but are not coplanar with the metal. Instead, the O1, O2, C1, C2, C3 plane makes a dihedral angle of 16.1 (2)° with the Ti1 O1 O2 plane while the corresponding angle for the other acetylacetonate ligand is 14.0 (1)°. The bisphenoxo ligand adopts a open book conformation with the dihedral angle between the two aromatic rings being 106.79 (8)°. The two Ti—O—C angles associated with this ligand differ significantly (Table 1). This is attributed to packing considerations since the rings containing C20 through C26 in two adjacent molecules are parallel, partially interleaved and separated by 3.74 Å while C12 and C13 in the other ring make contacts, respectively, with H8 (2.88 Å) and H10B (2.89 Å) in the molecule at *x*,-1 + *y*,*z*. The former interaction tends to open up the Ti—O6—C20 angle while the latter acts to decrease the Ti—O5—C11 angle.

Experimental

To a solution of $Ti(acac)_2Cl_2$ (0.225 g, 0.71 mmol) in CH_3CN (15 ml), a mixture of diol (0.181 g, 0.71 mmol) and sodium acetate (0.116 g, 1.42 mmol) in the same solvent (15 ml) was added dropwise at 300 K and the mixture was refluxed for 8 h. The reaction mixture was cooled to room temperature and filtered through celite. The filtrate was concentrated to a small volume (3 ml) and hexane was added (2 ml). Storage of the solution at 263 K yielded dark red blocks of the title compound (0.237 g, 67%; m.p.: 419–421 K). Analysis, calculated for $C_{27}H_{32}O_6Ti$: C 64.77, H 6.45; found: C 64.62, H 6.46%. Spectroscopic analysis: ^{1}H NMR (300 MHz, 298 K, $CDCl_3$, δ, p.p.m.): 7.12–7.26 (m, 4 H, phenyl), 5.95 (s, 1 H, CH, acac), 4.41 (s, 2 H, CH_2), 2.08 (m, 6 H, CH_3), 1.42 (s, 18 H, *tert*-butyl).

Refinement

H atoms were placed in calculated positions (C—H = 0.95–0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2–1.5 times those of the attached carbon atoms.

Figures

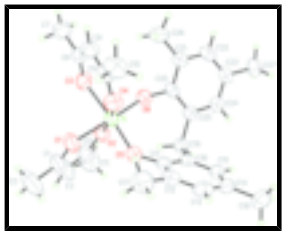


Fig. 1. Perspective view of the title compound. Displacement ellipsoids are drawn at the 50% level and H-atoms are drawn as spheres of arbitrary radius.

Bis(acetylacetonato-κ^2*O*,*O*')[2,2'-methylenebis(4,6-xylenolato)-κ^2*O*,*O*'] titanium(IV)

Crystal data

$[Ti(C_5H_7O_2)_2(C_{17}H_{18}O_2)]$	$Z = 2$
$M_r = 500.43$	$F_{000} = 528$
Triclinic, $P\overline{1}$	$D_x = 1.272$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.3605$ (9) Å	Cell parameters from 25 reflections
$b = 9.3419$ (7) Å	$\theta = 18.2$–23.1º
$c = 17.652$ (1) Å	$\mu = 0.37$ mm^{-1}
$\alpha = 92.472$ (5)º	$T = 298$ (2) K
$\beta = 95.599$ (7)º	Block, red
$\gamma = 107.209$ (7)º	0.46 × 0.40 × 0.26 mm
$V = 1306.9$ (2) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.021$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.1$º
Monochromator: graphite	$\theta_{min} = 2.3$º
$T = 298(2)$ K	$h = 0 \rightarrow 9$
θ/2θ scans	$k = -11 \rightarrow 10$
Absorption correction: empirical (using intensity measurements) ψ scans (North *et al.*, 1968)	$l = -21 \rightarrow 20$
$T_{min} = 0.850$, $T_{max} = 0.911$	2 standard reflections
4973 measured reflections	every 2 min
4628 independent reflections	intensity decay: <1%
3123 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.127$	$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.4456P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4628 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
314 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* 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 $F^2 > \sigma(F^2)$ 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. H-atoms were placed in calculated positions (C—H = 0.95 − 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 − 1.5 times those of the attached carbon atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ti1	0.83024 (6)	0.95224 (5)	0.68559 (3)	0.04474 (16)
O1	0.7004 (3)	0.8836 (2)	0.57985 (11)	0.0600 (5)
O2	1.0249 (2)	0.9282 (2)	0.63419 (10)	0.0541 (5)
O3	0.8816 (2)	1.1664 (2)	0.65189 (10)	0.0516 (5)
O4	0.6234 (2)	0.9907 (2)	0.71662 (11)	0.0531 (5)
O5	0.7537 (2)	0.7554 (2)	0.70880 (10)	0.0508 (5)
O6	0.9587 (2)	1.02024 (19)	0.77511 (10)	0.0476 (4)
C1	0.7354 (5)	0.8132 (4)	0.52428 (17)	0.0732 (10)
C2	0.5923 (6)	0.7500 (6)	0.4607 (2)	0.1285 (19)
H2A	0.5136	0.6618	0.4762	0.193*
H2B	0.6367	0.7243	0.4156	0.193*
H2C	0.5361	0.8241	0.4499	0.193*
C3	0.8900 (5)	0.7935 (5)	0.5200 (2)	0.0895 (12)
H3	0.9025	0.7357	0.4780	0.107*
C4	1.0287 (4)	0.8529 (4)	0.57311 (17)	0.0637 (8)
C5	1.1956 (5)	0.8313 (5)	0.5618 (2)	0.0929 (13)
H5A	1.2845	0.9137	0.5879	0.111*
H5B	1.2076	0.8273	0.5083	0.111*
H5C	1.2015	0.7390	0.5820	0.111*
C6	0.8209 (3)	1.2722 (3)	0.67020 (15)	0.0481 (6)
C7	0.9100 (4)	1.4253 (3)	0.64689 (19)	0.0692 (9)
H7A	1.0217	1.4601	0.6736	0.083*

H7B	0.8488	1.4937	0.6593	0.083*
H7C	0.9164	1.4201	0.5929	0.083*
C8	0.6801 (3)	1.2504 (3)	0.70863 (16)	0.0529 (7)
H8	0.6478	1.3342	0.7224	0.063*
C9	0.5848 (3)	1.1124 (3)	0.72778 (14)	0.0477 (6)
C10	0.4241 (4)	1.0942 (4)	0.76311 (19)	0.0669 (9)
H10A	0.3294	1.0519	0.7251	0.080*
H10B	0.4213	1.1906	0.7830	0.080*
H10C	0.4191	1.0286	0.8038	0.080*
C11	0.7272 (3)	0.6725 (3)	0.77042 (14)	0.0434 (6)
C12	0.5619 (3)	0.5910 (3)	0.78049 (16)	0.0507 (7)
C13	0.5362 (4)	0.5020 (3)	0.84165 (17)	0.0552 (7)
H13	0.4264	0.4485	0.8488	0.066*
C14	0.6676 (4)	0.4898 (3)	0.89250 (16)	0.0499 (6)
C15	0.8289 (3)	0.5727 (3)	0.88113 (15)	0.0466 (6)
H15	0.9186	0.5661	0.9149	0.056*
C16	0.8635 (3)	0.6658 (3)	0.82145 (14)	0.0414 (6)
C17	0.4165 (4)	0.6007 (4)	0.7242 (2)	0.0755 (10)
H17A	0.3135	0.5307	0.7356	0.113*
H17B	0.4368	0.5769	0.6733	0.113*
H17C	0.4074	0.7008	0.7282	0.113*
C18	0.6375 (5)	0.3883 (4)	0.9572 (2)	0.0732 (9)
H18A	0.7436	0.3836	0.9814	0.088*
H18B	0.5694	0.2893	0.9376	0.088*
H18C	0.5804	0.4274	0.9938	0.088*
C19	1.0431 (3)	0.7572 (3)	0.81441 (15)	0.0432 (6)
H19A	1.0531	0.7811	0.7618	0.052*
H19B	1.1177	0.6977	0.8278	0.052*
C20	1.0499 (3)	1.0264 (3)	0.84343 (13)	0.0397 (5)
C21	1.0956 (3)	1.1597 (3)	0.89035 (15)	0.0439 (6)
C22	1.1872 (3)	1.1633 (3)	0.96104 (15)	0.0480 (6)
H22	1.2159	1.2506	0.9933	0.058*
C23	1.2369 (3)	1.0433 (3)	0.98512 (14)	0.0470 (6)
C24	1.1904 (3)	0.9132 (3)	0.93643 (15)	0.0448 (6)
H24	1.2227	0.8309	0.9519	0.054*
C25	1.0974 (3)	0.9015 (3)	0.86565 (14)	0.0399 (5)
C26	1.0431 (4)	1.2922 (3)	0.86477 (19)	0.0668 (9)
H26A	1.0880	1.3752	0.9026	0.080*
H26B	0.9223	1.2659	0.8582	0.080*
H26C	1.0855	1.3202	0.8172	0.080*
C27	1.3356 (4)	1.0516 (4)	1.06257 (16)	0.0612 (8)
H27A	1.3620	1.1512	1.0867	0.073*
H27B	1.4381	1.0282	1.0565	0.073*
H27C	1.2693	0.9807	1.0937	0.073*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Ti1	0.0526 (3)	0.0502 (3)	0.0340 (3)	0.0202 (2)	0.00336 (19)	0.00056 (19)
O1	0.0672 (13)	0.0742 (14)	0.0399 (11)	0.0279 (11)	−0.0059 (9)	0.0002 (10)
O2	0.0589 (12)	0.0664 (12)	0.0435 (11)	0.0275 (10)	0.0117 (9)	0.0018 (9)
O3	0.0589 (12)	0.0553 (11)	0.0485 (11)	0.0248 (9)	0.0173 (9)	0.0113 (9)
O4	0.0501 (11)	0.0556 (11)	0.0554 (12)	0.0167 (9)	0.0127 (9)	0.0044 (9)
O5	0.0581 (11)	0.0503 (11)	0.0426 (10)	0.0164 (9)	−0.0002 (8)	0.0008 (8)
O6	0.0576 (11)	0.0481 (10)	0.0382 (10)	0.0195 (9)	0.0016 (8)	−0.0011 (8)
C1	0.097 (3)	0.085 (2)	0.0383 (17)	0.036 (2)	−0.0102 (16)	−0.0087 (16)
C2	0.140 (4)	0.160 (5)	0.075 (3)	0.055 (4)	−0.043 (3)	−0.050 (3)
C3	0.111 (3)	0.123 (3)	0.0485 (19)	0.062 (3)	0.0014 (19)	−0.024 (2)
C4	0.083 (2)	0.074 (2)	0.0461 (17)	0.0388 (18)	0.0187 (16)	0.0100 (15)
C5	0.102 (3)	0.126 (3)	0.075 (2)	0.065 (3)	0.031 (2)	0.004 (2)
C6	0.0546 (16)	0.0529 (16)	0.0370 (14)	0.0190 (13)	−0.0016 (11)	0.0021 (11)
C7	0.082 (2)	0.0534 (18)	0.072 (2)	0.0177 (16)	0.0174 (18)	0.0075 (15)
C8	0.0530 (16)	0.0559 (17)	0.0537 (17)	0.0238 (14)	0.0055 (13)	−0.0030 (13)
C9	0.0443 (15)	0.0617 (17)	0.0366 (14)	0.0184 (13)	−0.0010 (11)	−0.0065 (12)
C10	0.0517 (18)	0.082 (2)	0.069 (2)	0.0227 (16)	0.0139 (15)	−0.0076 (17)
C11	0.0513 (15)	0.0360 (13)	0.0438 (14)	0.0165 (12)	0.0031 (12)	−0.0059 (11)
C12	0.0463 (15)	0.0465 (15)	0.0566 (17)	0.0128 (12)	0.0025 (12)	−0.0093 (13)
C13	0.0477 (16)	0.0434 (15)	0.070 (2)	0.0061 (12)	0.0136 (14)	−0.0020 (14)
C14	0.0574 (17)	0.0361 (13)	0.0533 (16)	0.0089 (12)	0.0107 (13)	−0.0006 (12)
C15	0.0558 (16)	0.0381 (13)	0.0475 (15)	0.0187 (12)	0.0019 (12)	−0.0026 (11)
C16	0.0461 (14)	0.0336 (12)	0.0456 (14)	0.0146 (11)	0.0073 (11)	−0.0060 (10)
C17	0.0539 (19)	0.081 (2)	0.086 (3)	0.0190 (17)	−0.0121 (17)	0.0027 (19)
C18	0.084 (2)	0.0562 (18)	0.077 (2)	0.0113 (17)	0.0199 (19)	0.0170 (16)
C19	0.0445 (14)	0.0425 (14)	0.0461 (14)	0.0189 (11)	0.0053 (11)	0.0003 (11)
C20	0.0351 (13)	0.0477 (14)	0.0355 (13)	0.0114 (11)	0.0056 (10)	−0.0013 (11)
C21	0.0365 (13)	0.0457 (14)	0.0484 (15)	0.0122 (11)	0.0043 (11)	−0.0039 (12)
C22	0.0387 (14)	0.0526 (16)	0.0483 (15)	0.0096 (12)	0.0030 (11)	−0.0118 (12)
C23	0.0361 (13)	0.0606 (17)	0.0400 (14)	0.0088 (12)	0.0048 (11)	−0.0011 (12)
C24	0.0378 (13)	0.0491 (15)	0.0480 (15)	0.0127 (11)	0.0056 (11)	0.0090 (12)
C25	0.0342 (13)	0.0448 (14)	0.0414 (13)	0.0112 (11)	0.0100 (10)	0.0027 (11)
C26	0.0651 (19)	0.0509 (17)	0.081 (2)	0.0219 (15)	−0.0137 (16)	−0.0161 (16)
C27	0.0547 (18)	0.075 (2)	0.0468 (16)	0.0123 (15)	−0.0026 (13)	0.0025 (15)

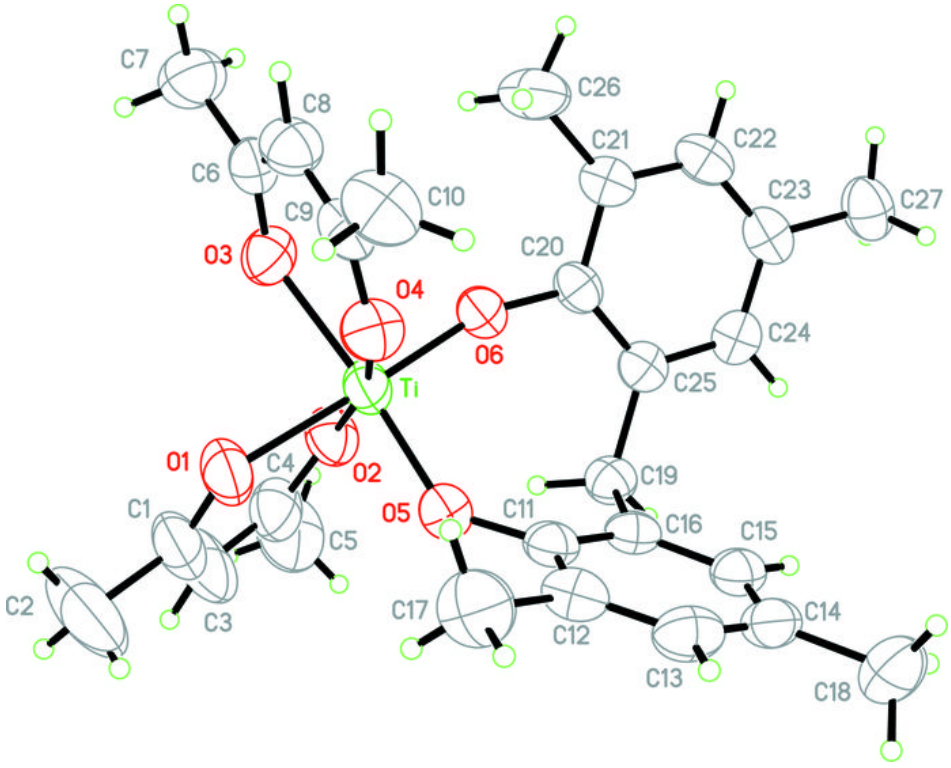
Geometric parameters (Å, °)

Ti1—O6	1.798 (2)	C11—C16	1.400 (4)
Ti1—O5	1.839 (2)	C12—C13	1.386 (4)
Ti1—O4	1.989 (2)	C12—C17	1.520 (4)
Ti1—O2	2.002 (2)	C13—C14	1.385 (4)
Ti1—O1	2.040 (2)	C13—H13	0.9300
Ti1—O3	2.046 (2)	C14—C15	1.381 (4)
O1—C1	1.263 (4)	C14—C18	1.508 (4)
O2—C4	1.270 (3)	C15—C16	1.393 (4)
O3—C6	1.280 (3)	C15—H15	0.9300
O4—C9	1.283 (3)	C16—C19	1.512 (4)
O5—C11	1.360 (3)	C17—H17A	0.9600
O6—C20	1.352 (3)	C17—H17B	0.9600

C1—C3	1.367 (5)	C17—H17C	0.9600
C1—C2	1.518 (5)	C18—H18A	0.9600
C2—H2A	0.9600	C18—H18B	0.9600
C2—H2B	0.9600	C18—H18C	0.9600
C2—H2C	0.9600	C19—C25	1.515 (3)
C3—C4	1.376 (5)	C19—H19A	0.9700
C3—H3	0.9300	C19—H19B	0.9700
C4—C5	1.499 (5)	C20—C21	1.396 (3)
C5—H5A	0.9600	C20—C25	1.398 (3)
C5—H5B	0.9600	C21—C22	1.392 (4)
C5—H5C	0.9600	C21—C26	1.504 (4)
C6—C8	1.383 (4)	C22—C23	1.376 (4)
C6—C7	1.500 (4)	C22—H22	0.9300
C7—H7A	0.9600	C23—C24	1.390 (4)
C7—H7B	0.9600	C23—C27	1.513 (4)
C7—H7C	0.9600	C24—C25	1.388 (3)
C8—C9	1.376 (4)	C24—H24	0.9300
C8—H8	0.9300	C26—H26A	0.9600
C9—C10	1.503 (4)	C26—H26B	0.9600
C10—H10A	0.9600	C26—H26C	0.9600
C10—H10B	0.9600	C27—H27A	0.9600
C10—H10C	0.9600	C27—H27B	0.9600
C11—C12	1.399 (4)	C27—H27C	0.9600
O6—Ti1—O5	95.80 (8)	C12—C11—C16	120.8 (2)
O6—Ti1—O4	96.15 (8)	C13—C12—C11	118.5 (3)
O5—Ti1—O4	90.98 (8)	C13—C12—C17	121.9 (3)
O6—Ti1—O2	92.84 (8)	C11—C12—C17	119.6 (3)
O5—Ti1—O2	95.41 (8)	C14—C13—C12	122.5 (3)
O4—Ti1—O2	168.39 (8)	C14—C13—H13	118.7
O6—Ti1—O1	175.50 (9)	C12—C13—H13	118.7
O5—Ti1—O1	87.13 (8)	C15—C14—C13	117.4 (3)
O4—Ti1—O1	87.20 (8)	C15—C14—C18	120.9 (3)
O2—Ti1—O1	83.46 (8)	C13—C14—C18	121.7 (3)
O6—Ti1—O3	90.75 (8)	C14—C15—C16	123.0 (3)
O5—Ti1—O3	171.80 (8)	C14—C15—H15	118.5
O4—Ti1—O3	83.40 (8)	C16—C15—H15	118.5
O2—Ti1—O3	89.17 (8)	C15—C16—C11	117.7 (2)
O1—Ti1—O3	86.64 (8)	C15—C16—C19	119.9 (2)
C1—O1—Ti1	129.8 (2)	C11—C16—C19	122.4 (2)
C4—O2—Ti1	130.9 (2)	C12—C17—H17A	109.5
C6—O3—Ti1	130.3 (2)	C12—C17—H17B	109.5
C9—O4—Ti1	132.1 (2)	H17A—C17—H17B	109.5
C11—O5—Ti1	140.2 (2)	C12—C17—H17C	109.5
C20—O6—Ti1	162.5 (2)	H17A—C17—H17C	109.5
O1—C1—C3	123.7 (3)	H17B—C17—H17C	109.5
O1—C1—C2	115.3 (3)	C14—C18—H18A	109.5
C3—C1—C2	121.1 (3)	C14—C18—H18B	109.5
C1—C2—H2A	109.5	H18A—C18—H18B	109.5
C1—C2—H2B	109.5	C14—C18—H18C	109.5

H2A—C2—H2B	109.5	H18A—C18—H18C	109.5
C1—C2—H2C	109.5	H18B—C18—H18C	109.5
H2A—C2—H2C	109.5	C16—C19—C25	111.9 (2)
H2B—C2—H2C	109.5	C16—C19—H19A	109.2
C1—C3—C4	124.9 (3)	C25—C19—H19A	109.2
C1—C3—H3	117.6	C16—C19—H19B	109.2
C4—C3—H3	117.6	C25—C19—H19B	109.2
O2—C4—C3	123.2 (3)	H19A—C19—H19B	107.9
O2—C4—C5	115.7 (3)	O6—C20—C21	118.5 (2)
C3—C4—C5	121.1 (3)	O6—C20—C25	120.1 (2)
C4—C5—H5A	109.5	C21—C20—C25	121.4 (2)
C4—C5—H5B	109.5	C22—C21—C20	117.8 (2)
H5A—C5—H5B	109.5	C22—C21—C26	122.1 (2)
C4—C5—H5C	109.5	C20—C21—C26	120.1 (2)
H5A—C5—H5C	109.5	C23—C22—C21	122.8 (2)
H5B—C5—H5C	109.5	C23—C22—H22	118.6
O3—C6—C8	123.4 (3)	C21—C22—H22	118.6
O3—C6—C7	116.4 (3)	C22—C23—C24	117.7 (2)
C8—C6—C7	120.3 (3)	C22—C23—C27	121.1 (3)
C6—C7—H7A	109.5	C24—C23—C27	121.2 (3)
C6—C7—H7B	109.5	C25—C24—C23	122.4 (2)
H7A—C7—H7B	109.5	C25—C24—H24	118.8
C6—C7—H7C	109.5	C23—C24—H24	118.8
H7A—C7—H7C	109.5	C24—C25—C20	118.0 (2)
H7B—C7—H7C	109.5	C24—C25—C19	121.7 (2)
C9—C8—C6	124.0 (3)	C20—C25—C19	120.3 (2)
C9—C8—H8	118.0	C21—C26—H26A	109.5
C6—C8—H8	118.0	C21—C26—H26B	109.5
O4—C9—C8	123.6 (2)	H26A—C26—H26B	109.5
O4—C9—C10	115.0 (3)	C21—C26—H26C	109.5
C8—C9—C10	121.4 (3)	H26A—C26—H26C	109.5
C9—C10—H10A	109.5	H26B—C26—H26C	109.5
C9—C10—H10B	109.5	C23—C27—H27A	109.5
H10A—C10—H10B	109.5	C23—C27—H27B	109.5
C9—C10—H10C	109.5	H27A—C27—H27B	109.5
H10A—C10—H10C	109.5	C23—C27—H27C	109.5
H10B—C10—H10C	109.5	H27A—C27—H27C	109.5
O5—C11—C12	118.7 (2)	H27B—C27—H27C	109.5
O5—C11—C16	120.4 (2)		

Fig. 1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