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Di-μ-ethoxy-bis[diethoxy(8-quinolinato)-titanium(IV)]

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Key indicators

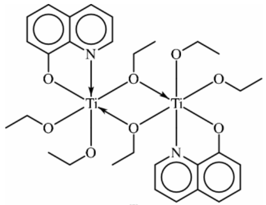
Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.091
Data-to-parameter ratio = 17.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The hydroxyquinolinate group chelates to the Ti atom in the centrosymmetric title compound, $[Ti_2(C_9H_6NO)_2(C_2H_5O)_6]$. Two of the four ethoxy groups function as bridges, and the geometry of the six-coordinate Ti atom is that of an octahedron.

Comment

8-Hydroxyquinoline is a reagent for the gravimetric analysis of metal ions and the crystal structures of a large number of metal 8-hydroxyquinolinates have been documented. However, for Ti^{IV}, only two 8-hydroxyquinolinates are known, these being the bis(2,6-diisopropylphenoxide)titanium (Bird *et al.*, 1973; Matthews *et al.*, 1970) and the dichlorotitanium (Studd & Swallow, 1968) derivative. A bis(chloro-substituted 8-hydroxyquinolinate) has also been reported (Demakopoulos *et al.*, 1995).



(I)

Molecules of the title compound, (I), lie across crystallographic inversion centres and the asymmetric unit therefore contains one-half of a molecule. The 8-hydroxyquinolinate

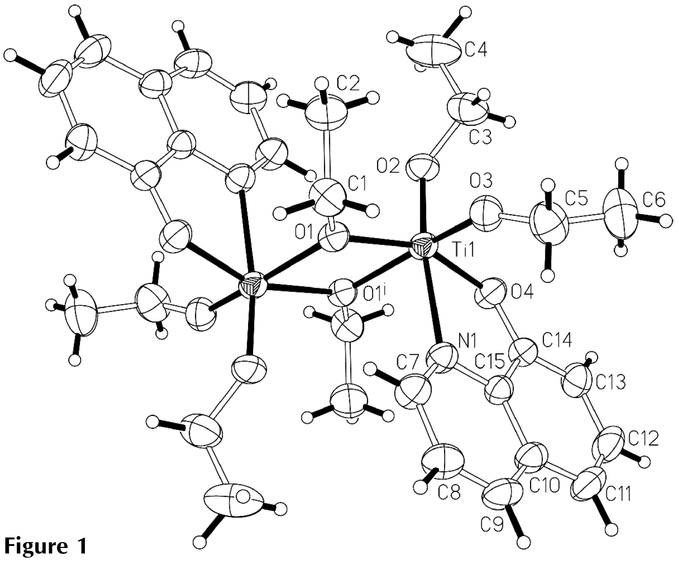


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code (i): $1 - x$, $1 - y$, $1 - z$.]

anion chelates to Ti in the triethoxy derivative; however, the coordination number is raised to six as one of the three ethoxy groups is bridging [Ti1—O1 = 2.013 (1) Å and Ti1—O1^{1} = 2.065 (1) Å] (Fig. 1). The two briding Ti—O distances are nearly identical, but much longer than the other two Ti—O$_{\text{ethoxy}}$ distances. The bridging and covalent distances are similar to those found in the three centrosymmetric dimeric triethoxy–(chelate)–titanium complexes, the acetyacetonate [1.796 (1), 1.805 (1) and 2.064 (1) Å; Errington *et al.*, 1998], the glycinate [1.765 (6), 1.813 (6) and 2.087 (5) Å; Schubert *et al.*, 1995] and the maltolate [1.778 (3), 1.809 (3) and 1.969 (3) Å; Subota *et al.*, 2001].

Experimental

Manipulations were carried out under nitrogen, using standard Schlenk techniques. Titanium(IV) tetraethoxide was prepared from titanium(IV) tetrapropoxide (Fluka) and dry ethanol by the alkoxide exchange method and it was purified by vacuum distillation. 8-Hydroxyquinoline (2.18 g, 15 mmol) was added to the reagent (3.42 g, 15 mmol) in benzene (20 ml). The mixture was stirred for a day and the solvent then removed under reduced pressure to furnish a yellow solid. The solid was crystallized from chloroform–hexane (3:4) to give pale-yellow crystals, m.p. 412–413 K. UV (CH_2Cl_2, nm): 255 (π to π^*), 382 (n to π^*). IR (cm^{-1}): 1570 (C=C, C=N), 1268 (C—O). ^{1}H NMR ($CDCl_3$, p.p.m.): 1.0 (CH_3, triplet), 1.3 (CH_3, triplet), 3.8 (CH_2, quartet), 4.4 (CH_2, quartet), 6.8–9.2 (aromatic H atoms).

Crystal data

$[Ti_2(C_9H_6NO)_2(C_2H_5O)_6]$
M_r = 654.46
Monoclinic, $P2_1/n$
a = 8.7135 (6) Å
b = 13.5629 (9) Å
c = 13.930 (1) Å
β = 91.986 (1)°
V = 1645.3 (2) Å^3
Z = 2
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5733 reflections
θ = 2.7–26.3°
μ = 0.53 mm^{-1}
T = 163 (2) K
Block, yellow
0.50 × 0.30 × 0.17 mm

Data collection

Bruker *P*4/CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.693, $T_{\max}$ = 0.915
20052 measured reflections
3344 independent reflections
2793 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
$\theta_{\max}$ = 26.4°
$h = -10 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.091
S = 1.03
3344 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.6250P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.32 e Å^{-3}
$\Delta\rho_{\min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—O1	2.013 (1)	Ti1—O3	1.799 (1)
Ti1—O1^{i}	2.065 (1)	Ti1—O4	1.972 (1)
Ti1—O2	1.819 (1)	Ti1—N1	2.294 (1)
O1—Ti1—O1^{i}	72.7 (1)	O1^{i}—Ti1—N1	86.1 (1)
O1—Ti1—O2	104.3 (1)	O2—Ti1—O3	97.0 (1)
O1—Ti1—O3	94.4 (1)	O2—Ti1—O4	93.6 (1)
O1—Ti1—O4	156.5 (1)	O2—Ti1—N1	168.8 (1)
O1—Ti1—N1	86.7 (1)	O3—Ti1—O4	98.5 (1)
O1^{i}—Ti1—O2	95.1 (1)	O3—Ti1—N1	83.9 (1)
O1^{i}—Ti1—O3	164.1 (1)	O4—Ti1—N1	75.3 (1)
O1^{i}—Ti1—O4	90.9 (1)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

A $\mu \times 2r$ value of 0.20 was used in the multi-scan absorption correction. H atoms were placed at calculated positions (C—H = 0.95 Å for the aromatic, 0.99 Å for the methylene and 0.98 Å for the methyl H atoms), and they were included in the refinement in the riding-model approximation with U_{iso}(H) set to $1.2U_{\text{eq}}$(C) for the aromatic and methylene H atoms, and to $1.5U_{\text{eq}}$(C) for the methyl H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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