

(OC-6-22)-Bis{2-[1-(2-oxidoethylimino)-ethyl]phenolato- κ^3O,N,O' }titanium(IV)

Uwe Böhme* and Betty Günther

Institut für Anorganische Chemie, Technische
Universität Bergakademie Freiberg, Leipziger
Strasse 29, 09596 Freiberg, GermanyCorrespondence e-mail:
uwe.boehme@chemie.tu-freiberg.de**Key indicators**Single-crystal X-ray study
 $T = 93\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.026
 wR factor = 0.087
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $[\text{Ti}(\text{C}_{10}\text{H}_{11}\text{NO}_2)_2]$, the Ti atom is octahedrally coordinated by two tridentate chelate ligands in a meridional arrangement.

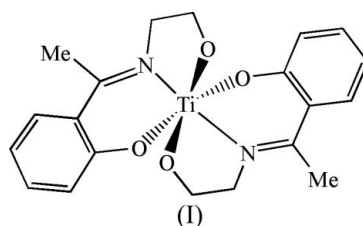
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Comment

Over the past few decades, much attention has been focused on the chemistry of metal complexes with multidentate Schiff bases due to their ability to coordinate the majority of metal ions (Wilkinson *et al.*, 1987; McCleverty & Meyer, 2004). Schiff base complexes of titanium have been investigated with respect to different aspects, for instance as chiral dopants for converting nematic into cholesteric phases (Braun *et al.* 2005), as catalysts for the polymerization of ethylene (Owiny *et al.*, 2003), or for enantioselective catalytic transformations (Braun *et al.*, 2004; Yaozhong *et al.*, 1995; Hayashi *et al.*, 1994; Flores-Lopez *et al.*, 2000). A small number of six-coordinate titanium complexes containing two O,N,O' -tridentate ligands of Schiff base type have already been structurally characterized (Fleischer, *et al.* 1998; Rao *et al.*, 1999, 2002; Hu *et al.*, 2001; Owiny *et al.*, 2003; Flores-Lopez *et al.*, 2000).

In the title compound, (I), the Ti atom is octahedrally coordinated by two tridentate 2-oxyacetophenone- N -[2-oxyethyl)iminato ligands (Fig. 1). The *cis* bond angles range from $76.80(4)$ ($\text{O}3-\text{Ti}1-\text{N}2$) to $102.17(4)^\circ$ ($\text{O}2-\text{Ti}1-\text{N}2$), thus indicating a distorted octahedral geometry around the metal atom. The O,N,O' -tridentate ligands are coordinated in meridional geometry. Four O atoms are arranged in one plane. The Ti—O bond lengths to the phenolate atoms O2 and O4 are somewhat longer than the bond lengths to the aliphatic atoms O1 and O3. This is due to the stronger C—O bond in the phenoxy groups and was also observed in related structures (Rao *et al.*, 1999). The N atoms are situated in *trans* positions to each other and have substantially longer distances to the Ti atom than the O atoms.

**Experimental**

o-Hydroxyacetophenone- N -(2-hydroxyethyl)imine (6 g, 0.033 mol) in THF (80 ml) was slowly added to a cooled solution (273 K) of

TiCl₄-tetrahydrofuran adduct (5.5 g, 0.0165 mol) and triethylamine (8.3 g, 0.0825 mol) in THF (120 ml). The cooling bath was removed and the solution was stirred for 70 h at room temperature. The solvent was removed *in vacuo* and the residue taken up in 120 ml of 1,2-dimethoxyethane. The suspension was filtered with a Schlenk filter and the remaining solid was dried *in vacuo*. NMR investigation of the amorphous solid showed that it was a mixture of (I) and NEt₃HCl. Extraction with hot 1,2-dimethoxyethane gave a pale-yellow solution. Yellow crystals of (I) (1.43 g, 21%) were obtained by cooling this solution for some weeks to 278 K. These crystals were used for the X-ray study without recrystallization. Further product was obtained from the filtered solution in 1,2-dimethoxyethane by partial removal of the solvent *in vacuo* and cooling over several days (yield 2.1 g, 31.6%; m.p. 516 K). Analysis calculated for C₂₀H₂₂N₂O₄Ti: C 59.71, H 5.51, N 6.96%; found: C 59.52, H 5.62, N 6.95%.

Crystal data

[Ti(C ₁₀ H ₁₁ NO ₂) ₂]	Z = 4
M _r = 402.30	D _x = 1.514 Mg m ⁻³
Monoclinic, P2 ₁ /c	Mo Kα radiation
a = 7.1006 (2) Å	μ = 0.52 mm ⁻¹
b = 26.4054 (7) Å	T = 93 (2) K
c = 9.5367 (2) Å	Prism, pale yellow
β = 99.1180 (10)°	0.40 × 0.38 × 0.21 mm
V = 1765.48 (8) Å ³	

Data collection

Bruker SMART CCD area-detector diffractometer	63605 measured reflections
φ and ω scans	4060 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick 1996)	3952 reflections with I > 2σ(I)
T _{min} = 0.821, T _{max} = 0.900	R _{int} = 0.020
	θ _{max} = 27.5°

Refinement

Refinement on F ²	w = 1/[σ ² (F _o ²) + (0.0487P) ² + 0.9328P]
R[F ² > 2σ(F ²)] = 0.026	where P = (F _o ² + 2F _c ²)/3
wR(F ²) = 0.088	(Δ/σ) _{max} = 0.001
S = 1.13	Δρ _{max} = 0.42 e Å ⁻³
4060 reflections	Δρ _{min} = -0.37 e Å ⁻³
246 parameters	
H-atom parameters constrained	

All H atoms were treated as riding on their parent C atoms, with C–H distances of 0.93 (C_{aromatic}), 0.97 (CH₂) and 0.96 Å (CH₃), and with U_{iso}(H) values of 1.2U_{eq}(C) (C_{aromatic}, CH₂) and 1.5U_{eq}(C) (CH₃).

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

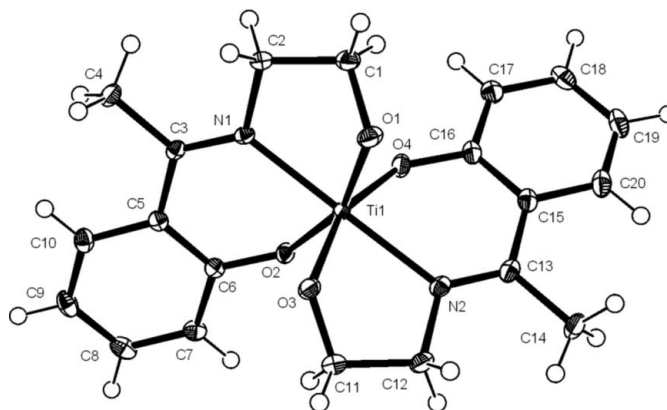


Figure 1 The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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