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Oxotris(quinolin-8-olato-κ^2O,N)tantalum(V) dichloromethane disolvate

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The Ta^V atom in the title compound, $[Ta(C_9H_6NO)_3O]\cdot 2CH_2Cl_2$, is chelated by three quinolin-8-olate ligands and exists in a pentagonal–bipramidal geometry. The Ta and oxo O atoms and one of the quinolin-8-olate ligands lie on a mirror plane that relates the other two quinolin-8-olate ligands to one another. The compound is isostructural with the Nb^V dichloromethane disolvate reported by Amini, Mirzaee, Yeganeh & Ng [*Acta Cryst.* (2004), E**60**, m147–m148].

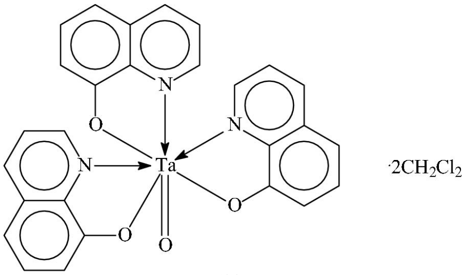
Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.005 Å
R factor = 0.024
wR factor = 0.064
Data-to-parameter ratio = 14.5

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

Comment

Tris(8-hydroxyquinolinato)oxotantalum(V) crystallizes from dichloromethane as a disolvate, (I) (Fig. 1); the compound is isostructural with the niobium(V) derivative (Amini *et al.*, 2004) but it does not lose the solvent molecules when exposed to air. The Ta atom exists in a seven-coordinate pentagonal–bipyramidal environment in which the axial positions are occupied by the oxo O atom and the N atom of one of the chelating quinolin-8-olate ligands. The Ta and oxo O atoms and one of the quinolin-8-olate ligands lie on a mirror plane that relates the other two quinolin-8-olate ligands to one another. Although quinolin-8-olate furnishes a large number of metal derivatives and is, in fact, a reagent in analytical chemistry, the oxotantalum compound is rarely mentioned, even in the analytical chemistry literature (Magee & Martin, 1963). There are only nine examples of oxotantalum structures in the Cambridge Structural Database (Version 5.26; Allen, 2002). This report is an addition to the list.



(I)

Experimental

Manipulations were carried out under nitrogen using standard Schlenk techniques. Tantalum(V) pentaethoxide (0.93 g, 2.3 mmol) and quinolin-8-ol (0.67 g, 4.6 mmol) were stirred in toluene (10 ml) for 24 h. The solvent was removed under reduced pressure to furnish a yellow solid that was recrystallized from dichloromethane to give orange crystals (m.p. > 573 K). MS (m/e): 629 M^+, 485 $[(C_9H_6NO)_2TaO]^+$. UV (in CH_2Cl_2, nm): 255 (π to π^*) and 377 (n to π^*).

Crystal data

$[Ta(C_9H_6NO)_3O]\cdot 2CH_2Cl_2$
$M_r = 799.25$
Orthorhombic, *Pnma*
$a = 12.4473\ (5)$ Å
$b = 17.4957\ (7)$ Å
$c = 12.8805\ (5)$ Å
$V = 2805.0\ (2)$ Å^3
$Z = 4$
$D_x = 1.893$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8025 reflections
$\theta = 2.6$–$26.5°$
$\mu = 4.34$ mm^{-1}
$T = 93\ (2)$ K
Block, yellow
$0.32 \times 0.30 \times 0.28$ mm

Data collection

Siemens P4/SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.189$, $T_{\max} = 0.376$
21 960 measured reflections

2975 independent reflections
2718 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 26.5°$
$h = -15 \rightarrow 15$
$k = -21 \rightarrow 21$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.064$
$S = 1.05$
2975 reflections
205 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 4.9159P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.78$ e Å^{-3}
$\Delta\rho_{\min} = -2.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ta1—O1	1.747 (3)	Ta1—N1$^{\rm i}$	2.342 (3)
Ta1—O2	2.036 (2)	Ta1—N1	2.342 (3)
Ta1—O2$^{\rm i}$	2.036 (2)	Ta1—N2	2.356 (3)
Ta1—O3	2.049 (3)		
O1—Ta1—O2	103.7 (1)	O2—Ta1—N1	71.2 (1)
O1—Ta1—O3	97.8 (1)	O2—Ta1—N2	84.0 (1)
O1—Ta1—N2	170.4 (1)	O3—Ta1—N1	73.1 (1)
O2—Ta1—O2$^{\rm i}$	72.2 (1)	O3—Ta1—N2	72.7 (1)
O2—Ta1—O3	137.2 (1)	N1$^{\rm i}$—Ta1—N1	144.9 (1)
O2—Ta1—N1$^{\rm i}$	143.4 (1)	N1—Ta1—N2	89.3 (1)

Symmetry code: (i) $x, \frac{1}{2} - y, z$.

H atoms were placed in calculated positions (C—H = 0.95 Å for the aromatic H atoms and 0.99 Å for the methylene H atoms), and were included in the refinement in the riding-model approximation; $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The largest peak and deepest hole in the final difference Fourier map lie about 1 Å from the Ta1 atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from the isostructural Nb analogue; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

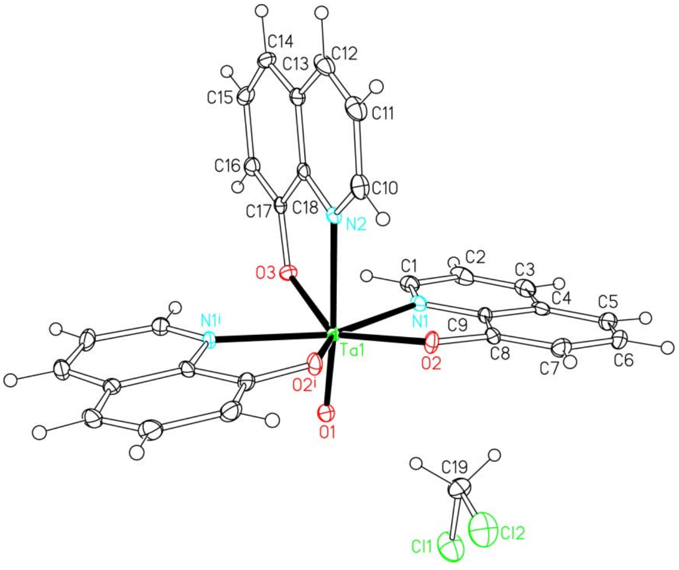


Figure 1
*ORTEP*II plot (Johnson, 1976) of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code (i): $x, \frac{1}{2} - y, z$.]

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m1054 M. M. Amini *et al.* • $[Ta(C_9H_6NO)_3O]\cdot 2CH_2Cl_2$ *Acta Cryst.* (2005). E**61**, m1053–m1054