Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Tris(8-hydroxyquinolinato-κ^2O,N)-oxoniobium(V) dichloromethane disolvate

Mostafa M. Amini,[a] Mehdi Mirzaee,[a] Farnaz Yeganeh[a] and Seik Weng Ng[b]*

[a]Department of Chemistry, Shahid Beheshti University, Tehran, Iran, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 18 December 2003
Accepted 22 December 2003
Online 10 January 2004

Key indicators

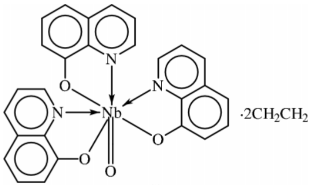
Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.097
Data-to-parameter ratio = 14.9

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

The Nb^V atom in $[NbO(C_9H_6NO)_3]\cdot 2CH_2Cl_2$ is chelated by the 8-hydroxyquinolinato ligand and it exists in a pentagonal bipyramidal geometry. The Nb and oxo O atoms, and one of the 8-hydroxyquinolinate groups lie on a crystallographic mirror plane. The structural features of the molecule in the dichloromethane solvate are almost identical to those in the chloroform solvate reported by García-Granda *et al.* [*Acta Cryst*. (1990). C**46**, 753–755].

Comment

8-Hydroxyquinoline (oxine) is a widely used reagent in analytical chemistry and it is a reagent for the gravimetric analysis of Nb^V. The structure of $(C_9H_6NO)_3NbO$ is known as the chloroform solvate; the Nb atom exists in a seven-coordinate pentagonal bipyramidal environment in which the apical positions are occupied by the oxo O atom and the N atom of one of the chelating oxinate groups. All atoms lies in general positions in the unit cell (García-Granda *et al.*, 1990). In the dichloromethane disolvate, (I), the Nb and oxo O atoms, and one of the 8-hydroxyquinolinate groups lie on a crystallographic mirror plane; the symmetry element relates one of the three 8-hydroxyquinolinate groups to the other (Fig. 1). The bond dimensions of the molecule in the dichloromethane solvate are almost identical to those of the chloroform solvate. Both solvates are unstable with respect to loss of the solvent.



(I)

Experimental

Manipulations were carried out under nitrogen, using standard Schlenk techniques. Niobium(V) pentaethoxide was prepared according to a previously reported procedure and it was purified by vacuum distillation (Amini & Sacks, 1990). 8-Hydroxyquinoline (2.03 g, 14.0 mmol) was reacted with this reagent (2.23 g, 7.0 mmol) in benzene (20 ml) for 24 h with stirring. The solvent was removed under reduced pressure to furnish an orange solid. The solid was recrystallized from dichloromethane; crystals separated when the solution was left in a refrigerator; m.p. > 573 K. UV (CH_2Cl_2, nm): 254 (π to π*) and 359 (*n* to π*). IR (cm^{-1}): 1556 (C═C, C═N), 1266 (C—O), 632 ($Nb—O—C_{sym}$), 525 ($Nb—O—C_{asym}$), 503 (Nb═O) (de Oliviera *et al.*, 2001). ^1H NMR ($CDCl_3$, p.p.m.): 7–9 (aromatic), 5.3 (CH_2Cl_2).

Crystal data

$[NbO(C_9H_6NO)_3]\cdot 2CH_2Cl_2$
$M_r = 711.21$
Orthorhombic, *Pnma*
$a = 12.599$ (1) Å
$b = 17.659$ (1) Å
$c = 13.024$ (1) Å
$V = 2897.5$ (4) Å^3
$Z = 4$
$D_x = 1.630$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 5268 reflections
$\theta = 2.4$–26.0°
$\mu = 0.83$ mm^{-1}
$T = 163$ (2) K
Plate, orange
0.35 × 0.13 × 0.07 mm

Data collection

Siemens *P*4/CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.711$, $T_{max} = 0.945$
35 672 measured reflections

3058 independent reflections
2422 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 26.5°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 21$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.097$
$S = 1.03$
3058 reflections
205 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 2.9577P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Nb1—O1	1.732 (3)	Nb1—N1	2.366 (2)
Nb1—O2	2.052 (2)	Nb1—N2	2.386 (3)
Nb1—O3	2.054 (3)		
O1—Nb1—O2	103.6 (1)	O2—Nb1—N1^i	143.5 (1)
O1—Nb1—O3	97.6 (1)	O2—Nb1—N2	84.3 (1)
O1—Nb1—N1	87.7 (1)	O3—Nb1—N1	73.3 (1)
O1—Nb1—N2	170.1 (1)	N1^i—Nb1—N1	145.2 (1)
O2—Nb1—O2^i	72.8 (1)	O3—Nb1—N2	72.5 (1)
O2—Nb1—O3	137.0 (1)	N1—Nb1—N2	89.4 (1)
O2—Nb1—N1	70.8 (1)		

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

A $\mu \times 2r$ value of 0.16 was used in the absorption correction. The H atoms were placed at calculated positions (C—H = 0.95 Å for the aromatic H atoms and 0.99 Å for the methylene H atoms), and they were included in the refinement in the riding-model approximation; $U_{iso}(H) = 1.2U_{eq}(C)$.

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.

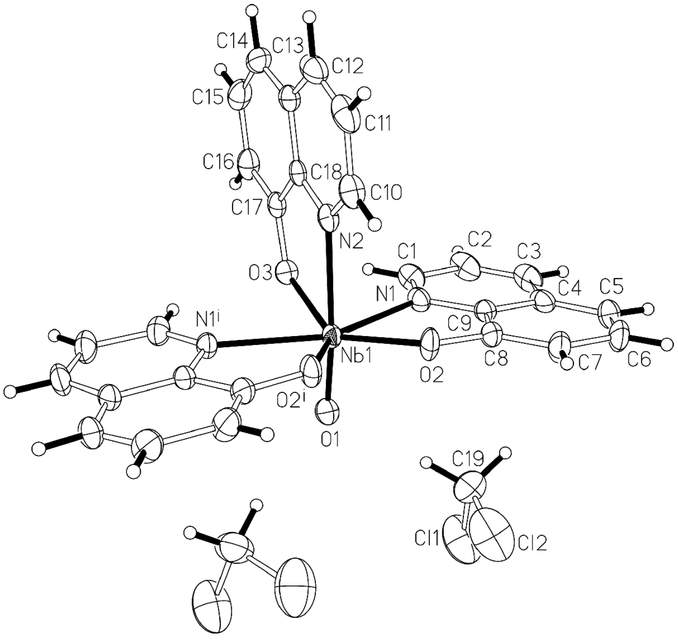


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

The authors thank Dr Jan Wikaira of the University of Canterbury, New Zealand, for the diffraction measurements, and the Vice-President's Office for Research Affairs of Shahid Beheshti University and the University of Malaya for supporting this work.

References

Amini, M. M. & Sacks, D. (1990). *Mater. Res. Soc. Symp. Proc.* **180**, 675–683.
Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
García-Granda, S., Díaz, M. R., Serra, L., Sanz-Medel, A. & Gómez-Beltrán, F. (1990). *Acta Cryst.* C**46**, 753–755.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Oliveira, V. de, da Sliva San Gil, R. A. & Lachter, E. R. (2001). *Polyhedron*, **20**, 2647 2649.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

m148 Mostafa M. Amini *et al.* • $[NbO(C_9H_6NO)_3]\cdot 2CH_2Cl_2$ *Acta Cryst.* (2004). E**60**, m147–m148