C3	0.7920 (8)	1.4060 (8)	0.0839 (4)	0.056 (2)
C4	0.8034 (9)	1.5535 (9)	0.0868 (5)	0.063 (2)
C5	0.9009 (8)	1.6724 (8)	0.1531 (4)	0.054 (2)
C6	0.9881 (7)	1.6390 (7)	0.2139 (4)	0.044 (2)
N7	0.8989 (6)	1.2512 (6)	0.1628 (3)	0.0419 (13)
C8	0.8164 (7)	1.1053 (7)	0.1074 (4)	0.042 (2)
C9	0.8540 (11)	1.0891 (11)	0.0191 (5)	0.112 (4)
C10	0.7756 (12)	0.9451 (13)	−0.0308 (6)	0.125 (5)
C11	0.6556 (10)	0.8214 (9)	0.0021 (6)	0.069 (2)
C12	0.6202 (10)	0.8352 (9)	0.0903 (6)	0.080 (3)
C13	0.7014 (9)	0.9757 (8)	0.1436 (5)	0.064 (2)
N14	0.7558 (6)	0.5339 (5)	0.3904 (3)	0.0413 (12)
C15	0.6405 (7)	0.5464 (7)	0.3451 (4)	0.0419 (15)
C16	0.6564 (8)	0.6968 (8)	0.3370 (5)	0.054 (2)
C17	0.7888 (10)	0.8248 (8)	0.3756 (5)	0.064 (2)
C18	0.9079 (9)	0.8087 (8)	0.4212 (5)	0.057 (2)
C19	0.8885 (8)	0.6623 (8)	0.4287 (4)	0.050 (2)
N20	0.5147 (6)	0.4150 (6)	0.3071 (4)	0.0536 (15)
C21	0.4717 (7)	0.2561 (7)	0.3279 (5)	0.047 (2)
C22	0.4528 (8)	0.1442 (10)	0.2593 (6)	0.074 (2)
C23	0.3991 (11)	−0.0126 (12)	0.2799 (9)	0.103 (4)
C24	0.3711 (10)	−0.0456 (10)	0.3678 (10)	0.099 (4)
C25	0.3931 (9)	0.0658 (10)	0.4365 (7)	0.082 (3)
C26	0.4423 (8)	0.2192 (8)	0.4177 (6)	0.062 (2)

Table 2. *Selected geometric parameters (Å, °)*

Ti1—N7	1.966 (5)	N1—C2	1.348 (7)
Ti1—N1	2.189 (5)	C2—N7	1.356 (7)
Ti1—Cl1	2.238 (2)	N7—C8	1.426 (7)
Ti1—Cl4	2.326 (2)	N14—C15	1.344 (7)
Ti1—Cl3	2.373 (2)	N14—C19	1.363 (7)
Ti1—Cl2	2.384 (2)	C15—N20	1.343 (7)
N1—C6	1.324 (7)	N20—C21	1.443 (8)
N7—Ti1—N1	63.1 (2)	Cl4—Ti1—Cl2	87.19 (8)
N7—Ti1—Cl1	95.6 (2)	Cl3—Ti1—Cl2	173.91 (8)
N1—Ti1—Cl1	158.37 (14)	C6—N1—C2	121.0 (5)
N7—Ti1—Cl4	155.5 (2)	C6—N1—Ti1	149.1 (4)
N1—Ti1—Cl4	92.47 (14)	C2—N1—Ti1	89.8 (4)
Cl1—Ti1—Cl4	108.93 (8)	N1—C2—N7	107.5 (5)
N7—Ti1—Cl3	91.1 (2)	N1—C2—Ti1	58.5 (3)
N1—Ti1—Cl3	91.06 (14)	N7—C2—Ti1	49.0 (3)
Cl1—Ti1—Cl3	92.71 (8)	C2—N7—C8	122.8 (5)
Cl4—Ti1—Cl3	88.07 (7)	C2—N7—Ti1	99.6 (4)
N7—Ti1—Cl2	91.7 (2)	C8—N7—Ti1	137.6 (4)
N1—Ti1—Cl2	85.33 (14)	N20—C15—N14	119.4 (6)
Cl1—Ti1—Cl2	92.45 (8)		

The determination of the unit cell was not straightforward. All the crystals selected, including the one used for data collection, suffered from wide peak profiles with high, and sometimes asymmetric, backgrounds. The reliability of the unit-cell parameters is thus rather poor. Data were not corrected for decay (1.3%). All non-H atoms were refined anisotropically. H atoms were refined on calculated positions with displacement parameters 1.3 times those of their host atoms. Additional refinements were carried out in order to confirm that the correct locations had been chosen for the heterocyclic N atom and the site of ammonium protonation in the aminopyridine cation. In these refinements, an N atom was placed at the positions of the C16, C22 or C26 atoms and an additional proton was located on N20 instead of on N14. All these additional refinements resulted in less satisfying displacement factors and *R* values than the solution presented here.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software.* Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC.* Software used to prepare material for publication: *SHELXL93.*

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1287). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bradley, D. C. & Thomas, I. M. (1960). *J. Chem. Soc.* pp. 3857–3861.
Bürger, H. & Neese, H.-J. (1970). *Chimia*, **24**, 209–236.
Cowdell, R. T. & Fowles, W. A. (1960). *J. Am. Chem. Soc.* **82**, 2522–2526.
Dermer, O. C. & Fernelius, W. C. (1934). *Z. Anorg. Allg. Chem.* **221**, 83–97.
Kempe, R. & Arndt, P. (1996). *Inorg. Chem.* **35**, 2644–2649.
Kottke, T. & Stalke, D. (1993). *J. Appl. Cryst.* **26**, 615–619.
Molecular Structure Corporation (1993*a*). *MSC/AFC Diffractometer Control Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1993*b*). *TEXSAN. Single Crystal Structure Analysis Software.* Version 1.6c. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *SHELXTL/PC.* Version 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1996). C**52**, 2977–2980

Trichlorobis[2,6-di(phenylamino)-pyridinato-*N*,*N′*]tantalum(V) Toluene Solvate (1/1)

Mika Polamo

Inorganic Chemistry Laboratory, Department of Chemistry, University of Helsinki, PO Box 55 (A. I. Virtasen aukio 1), FIN-00014 University of Helsinki, Finland. E-mail: polamo@kumpu.helsinki.fi

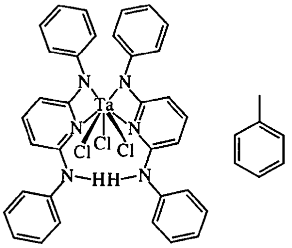
(*Received 5 June 1996; accepted 13 September 1996*)

Abstract

The title compound, $[TaCl_3(C_{17}H_{14}N_3)_2].C_7H_8$, is formed when 2,6-bis(phenylamino)pyridine and tantalum(V) chloride are refluxed in toluene. The organic ligand loses an amino proton and forms a σ bond with the metal atom, while its pyridine N atom acts as a donor. A small chelate ring is thus formed. The two bidentate ligands and three Cl atoms afford a seven-coordinate central atom.

Comment

Although many crystal structures of aminopyridines are known, few results of complexation studies have been reported. Many known complexes have *M*—*M* multiple bonds, as in tetrakis[2-(phenylamino)pyridinato]dimolybdenum(II) and tetrakis[2-(phenylamino)-pyridinato]ditungsten(II), where the ligand supports a short quadruple metal–metal interaction (Chakravarty, Cotton & Shamshoum, 1984). On the other hand, a bonding pattern similar to that observed in the title complex is found in bis[2-(methylamino)pyridinato]-(tetramethylethylenediamine)vanadium(II), which has bidentate aminopyridiato ligands (Edema, Gambarotta, Meetsma, Spek & Veldman, 1991). Most complexes of this type can be prepared from lithiated ligand precursors. The title complex, (I), with a toluene solvent molecule (C41–C47) included in the crystal, is a product of the direct reaction between the ligand precursor and metal halide in toluene.



(I)

Conformational changes in 2-phenylaminopyridines have been described as *E* and *Z* isomerism (Takasuka, Nakai & Shiro, 1986). We have recently reported the crystal structure of 2,6-bis(phenylamino)pyridine which displayed an *E*,*E* form in the solid state (Klinga, Polamo & Leskelä, 1994). In the *Z* conformation, the aromatic rings are in a coplanar orientation which is stabilized by hydrogen bonding, with the CH group in the β position of the phenyl ring as donor and the pyridine N atom as acceptor. In the present compound, pyridine is coordinated to tantalum with the uncoordinated phenylamino groups in *E* conformations. The aromatic rings are rotated in a form of screw in both ligands. In the ligand comprising atoms N1–C20, the dihedral angle between the phenyl and pyridine rings is 53.3 (3) for C8–C13 and 40.9 (4)° for C15–C20, while in the other ligand the corresponding angle is 55.4 (4) for C28–C33 and 50.0 (3)° for C35–C40. In 2,6-bis(phenylamino)-pyridine, which has two molecules in the asymmetric unit, dihedral angles of between 25.8 (2) and 53.4 (2)° were found.

Both ligands coordinate bidentately leaving one N atom uncoordinated (Table 2). The Ta—N σ-bond lengths are 2.052 (7) and 2.047 (6) Å to the N7 and N27 atoms, respectively, whereas the pyridine π-donor bonds Ta1—N1 and Ta1—N21 are 2.282 (7) and 2.263 (7) Å, respectively. The most separated chloro ligands, with Cl1—Ta1—Cl2 168.38 (8)°, have the shortest Ta—Cl bonds, namely, 2.392 (2) and 2.386 (2) Å to Cl1 and Cl3, respectively; Ta—Cl2 is 2.513 (2) Å.

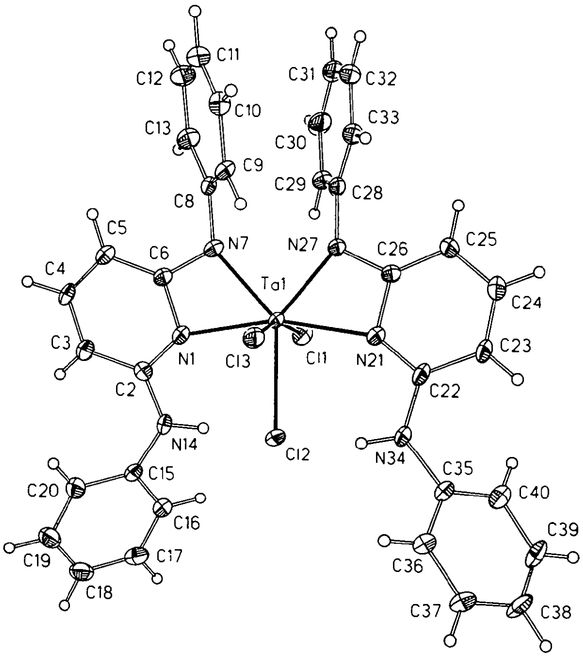


Fig. 1. Perspective view of the title compound showing the atomic labeling scheme. Displacement ellipsoids are shown at the 30% probability level. The toluene solvate molecule (C41–C47) has been omitted for clarity.

Experimental

2,6-Bis(phenylamino)pyridine (2.14 g, 8.19 mmol), which was prepared according to the method of Hamana & Yamazaki (1961), was dissolved in toluene (40 ml). The solution was cooled to 253 K. Tantalum(V) chloride (1.46 g, 4.08 mmol) was added and the temperature of the solution raised to 313 K over a period of 45 min and maintained for 1 h. The reaction mixture was then filtered. Dark-red prismatic crystals suitable for single-crystal measurements were obtained at room temperature after 15 h. All reaction steps were carried out under an argon atmosphere using standard Schlenk techniques. The crystal used for the X-ray measurements was mounted on a glass fiber using the oil-drop method (Kottke & Stalke, 1993).

Crystal data

$[TaCl_3(C_{17}H_{14}N_3)_2].C_7H_8$
$M_r = 900.06$
Triclinic
$P\overline{1}$
$a = 10.572\,(11)$ Å
$b = 11.070\,(10)$ Å
$c = 17.109\,(14)$ Å
$\alpha = 97.05\,(7)^\circ$
$\beta = 101.34\,(9)^\circ$
$\gamma = 108.38\,(9)^\circ$
$V = 1826\,(3)$ Å^3
$Z = 2$
$D_x = 1.637$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 3$–15°
$\mu = 3.269$ mm^{-1}
$T = 193\,(1)$ K
Block
0.50 × 0.50 × 0.35 mm
Dark red

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction:
ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.22$, $T_{\max} = 0.32$
6333 measured reflections
6333 independent reflections
6200 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 25°$
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 13$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 200 reflections intensity decay: 5.2%

Refinement

Refinement on F^2
$R(F) = 0.0680$
$wR(F^2) = 0.1908$
$S = 1.152$
6333 reflections
461 parameters
$w = 1/[\sigma^2(F_o^2) + (0.147P)^2 + 4.756P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.019$
$\Delta\rho_{\max} = 4.054$ e Å^{-3}
$\Delta\rho_{\min} = -6.696$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.007 (1)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	U_{eq}
Ta1	0.21660 (3)	0.76102 (2)	0.24462 (2)	0.0319 (2)
Cl1	−0.0055 (2)	0.6131 (2)	0.24236 (14)	0.0452 (5)
Cl2	0.0926 (2)	0.9207 (2)	0.24819 (12)	0.0444 (5)
Cl3	0.4081 (2)	0.9536 (2)	0.25648 (13)	0.0423 (5)
N1	0.1210 (7)	0.7285 (7)	0.1086 (4)	0.0362 (15)
C2	0.0212 (9)	0.7365 (8)	0.0483 (5)	0.038 (2)
C3	0.0187 (10)	0.6935 (9)	−0.0335 (5)	0.044 (2)
C4	0.1187 (12)	0.6468 (11)	−0.0498 (6)	0.051 (2)
C5	0.2194 (11)	0.6359 (9)	0.0117 (5)	0.047 (2)
C6	0.2133 (9)	0.6741 (8)	0.0898 (5)	0.037 (2)
N7	0.2932 (7)	0.6704 (7)	0.1633 (4)	0.0373 (15)
C8	0.4068 (9)	0.6280 (8)	0.1649 (5)	0.035 (2)
C9	0.5377 (8)	0.7081 (8)	0.2083 (5)	0.037 (2)
C10	0.6498 (10)	0.6650 (9)	0.2089 (5)	0.045 (2)
C11	0.6285 (10)	0.5431 (10)	0.1668 (6)	0.051 (2)
C12	0.4976 (11)	0.4629 (9)	0.1232 (6)	0.053 (2)
C13	0.3874 (10)	0.5055 (9)	0.1225 (6)	0.046 (2)
N14	−0.0753 (9)	0.7803 (8)	0.0698 (4)	0.046 (2)
C15	−0.1696 (10)	0.8254 (9)	0.0227 (5)	0.039 (2)
C16	−0.2893 (12)	0.8173 (10)	0.0459 (6)	0.052 (2)
C17	−0.3821 (11)	0.8668 (10)	0.0053 (7)	0.054 (2)
C18	−0.3576 (11)	0.9241 (9)	−0.0592 (6)	0.052 (2)
C19	−0.2392 (11)	0.9313 (9)	−0.0842 (6)	0.052 (2)
C20	−0.1447 (10)	0.8837 (9)	−0.0430 (5)	0.045 (2)
N21	0.2713 (8)	0.8276 (7)	0.3813 (4)	0.039 (2)
C22	0.2908 (10)	0.9212 (8)	0.4466 (5)	0.041 (2)
C23	0.3487 (11)	0.9102 (9)	0.5257 (5)	0.046 (2)
C24	0.3888 (12)	0.8063 (11)	0.5360 (6)	0.050 (2)
C25	0.3731 (10)	0.7097 (9)	0.4698 (5)	0.046 (2)
C26	0.3189 (9)	0.7280 (8)	0.3950 (5)	0.037 (2)
N27	0.2961 (8)	0.6561 (6)	0.3180 (4)	0.0373 (14)
C28	0.3142 (10)	0.5329 (8)	0.3081 (5)	0.041 (2)
C29	0.2047 (10)	0.4217 (9)	0.2687 (5)	0.045 (2)
C30	0.2236 (12)	0.3027 (10)	0.2552 (6)	0.054 (2)
C31	0.3536 (12)	0.2954 (9)	0.2834 (6)	0.053 (2)
C32	0.4647 (12)	0.4076 (10)	0.3253 (6)	0.055 (2)
C33	0.4444 (11)	0.5265 (9)	0.3381 (5)	0.046 (2)
N34	0.2546 (9)	1.0242 (7)	0.4304 (4)	0.046 (2)
C35	0.2272 (9)	1.1138 (9)	0.4858 (5)	0.042 (2)
C36	0.2567 (11)	1.2415 (9)	0.4744 (6)	0.049 (2)
C37	0.2239 (14)	1.3287 (11)	0.5243 (7)	0.058 (3)
C38	0.1599 (12)	1.2893 (11)	0.5851 (6)	0.059 (3)
C39	0.1304 (12)	1.1651 (11)	0.5958 (6)	0.059 (3)
C40	0.1637 (10)	1.0754 (10)	0.5467 (6)	0.050 (2)
C41	0.0592 (19)	0.6099 (17)	0.5911 (8)	0.102 (5)
C42	0.1359 (12)	0.7089 (11)	0.6693 (7)	0.058 (3)
C43	0.1054 (15)	0.8194 (13)	0.6889 (9)	0.075 (3)
C44	0.1686 (16)	0.9034 (13)	0.7614 (12)	0.097 (5)
C45	0.2700 (18)	0.8793 (15)	0.8154 (11)	0.101 (5)
C46	0.3021 (16)	0.7706 (14)	0.7959 (10)	0.079 (4)
C47	0.2362 (12)	0.6888 (12)	0.7240 (8)	0.059 (3)

Table 2. *Selected geometric parameters (Å, °)*

Ta1—N27	2.047 (6)	C6—N7	1.383 (11)
Ta1—N7	2.052 (7)	N7—C8	1.417 (11)
Ta1—N21	2.263 (7)	N14—C15	1.411 (12)
Ta1—N1	2.282 (7)	N21—C22	1.361 (11)
Ta1—Cl3	2.386 (2)	N21—C26	1.375 (11)
Ta1—Cl1	2.392 (2)	C22—N34	1.354 (11)
Ta1—Cl2	2.513 (2)	C26—N27	1.387 (10)
N1—C2	1.356 (11)	N27—C28	1.431 (10)
N1—C6	1.366 (11)	N34—C35	1.423 (10)
C2—N14	1.351 (11)		
N27—Ta1—N7	81.1 (3)	Cl1—Ta1—Cl2	81.86 (8)
N27—Ta1—N21	60.6 (3)	C2—N1—C6	119.8 (7)
N7—Ta1—N21	135.8 (3)	C2—N1—Ta1	148.4 (5)
N27—Ta1—N1	134.4 (3)	C6—N1—Ta1	91.8 (5)
N7—Ta1—N1	60.6 (3)	N14—C2—N1	117.9 (7)
N21—Ta1—N1	163.4 (3)	N1—C6—N7	106.0 (7)
N27—Ta1—Cl3	103.9 (2)	C6—N7—C8	119.9 (7)
N7—Ta1—Cl3	87.4 (2)	C6—N7—Ta1	101.6 (5)
N21—Ta1—Cl3	81.8 (2)	C8—N7—Ta1	137.8 (6)
N1—Ta1—Cl3	98.4 (2)	C2—N14—C15	129.5 (7)
N27—Ta1—Cl1	86.7 (2)	C22—N21—C26	117.7 (7)
N7—Ta1—Cl1	107.2 (2)	C22—N21—Ta1	148.9 (6)
N21—Ta1—Cl1	92.7 (2)	C26—N21—Ta1	92.7 (5)
N1—Ta1—Cl1	82.5 (2)	N34—C22—N21	116.4 (7)
Cl3—Ta1—Cl1	163.38 (8)	N21—C26—N27	104.5 (7)
N27—Ta1—Cl2	140.7 (2)	C26—N27—C28	120.6 (6)
N7—Ta1—Cl2	138.2 (2)	C26—N27—Ta1	102.1 (5)
N21—Ta1—Cl2	82.4 (2)	C28—N27—Ta1	136.7 (5)
N1—Ta1—Cl2	81.2 (2)	C22—N34—C35	127.0 (7)
Cl3—Ta1—Cl2	81.87 (8)		

The intensity data were corrected for decay. All non-H atoms, except those of the solvate molecule, were refined anisotropically. H atoms were refined on calculated positions with isotropic displacement parameters $1.5U_{eq}$ of the host atom (riding model).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1399). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chakravarty, A. R., Cotton, F. A. & Shamshoum, E. S. (1984). *Inorg. Chem.* **23**, 4216–4221.
Edema, J. J. H., Gambarotta, S., Meetsma, A., Spek, A. L. & Veldman, N. (1991). *Inorg. Chem.* **30**, 2062–2066.
Hamana, M. & Yamazaki, M. (1961). *Yakugaku Zasshi*, **81**, 574–578.

Klinga, M., Polamo, M. & Leskelä, M. (1994). *Acta Cryst.* C**50**, 2051–2054.
Kottke, T. & Stalke, D. (1993). *J. Appl. Cryst.* **26**, 615–619.
Molecular Structure Corporation (1993*a*). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1993*b*). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.6c. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Version 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Takasuka, M., Nakai, H. & Shiro, M. (1986). *J. Chem. Soc. Perkin Trans.* 2, pp. 1969–1978.

Acta Cryst. (1996). C**52**, 2980-2982

mer-Trichloro[2-(phenylamino)pyridinato]-oxotungsten(VI)

Mika Polamo

Inorganic Chemistry Laboratory, Department of Chemistry, University of Helsinki, PO Box 55 (A. I. Virtasen aukio 1), FIN-00014 University of Helsinki, Finland. E-mail: polamo@kumpu.helsinki.fi

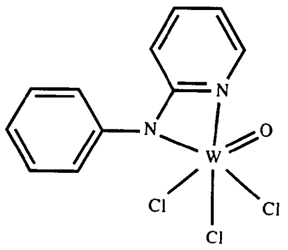
(*Received 6 June 1996; accepted 1 August 1996*)

Abstract

mer-Trichloro(oxo)[2-(phenylamino)pyridinato-*N*,*N*′]-tungsten, [$WCl_3(O)(C_{11}H_9N_2)$], is formed when tungsten(VI) oxotetrachloride and 2-(phenylamino)pyridine are heated in toluene under reflux. The ligand precursor loses an amino proton and forms a four-membered chelate ring with an N—W—N angle of 61.0(4)°. The monomeric complex displays a distorted octahedral coordination at tungsten.

Comment

Only a few aminopyridine complexes have been structurally characterized. Most closely related to the present compound, (I), is bis[2-(methylamino)pyridinato](tetramethylethylenediamine)vanadium, (II), which has a very similar aminopyridinato bonding pattern to that found here (Edema, Gambarotta, Meetsma, Spek & Veldman, 1991). The known aminopyridinato derivatives of tungsten, however, are quite different to the title compound. Tungsten(IV) chloride and 2-(phenylamino)-pyridine have been found to form tetrakis(phenylaminopyridinato)ditungsten(II) when sodium amalgam is used as a reducing agent with the lithium salt of the ligand precursor (Chakravarty, Cotton & Shamshoum, 1984). 2-Amino-6-methylpyridine has also been used as a ligand precursor for quadruply bonded dichromium, dimolybdenum and ditungsten complexes (Cotton, Niswander & Sekutowski, 1978). The only monomeric aminopyridine tungsten complex known previously is bis(2-aminopyridine)tris(3,5-dimethylpyrazoyl)-boratonitrosyltungsten, where 2-aminopyridine is monodentate and the heterocyclic N atom is uncoordinated (Obaidi, Hamor, Jones, McCleverty & Paxton, 1987).



(I)

The monomeric title complex has a symmetrically bidentate 2-(phenylamino)pyridinato group, with W—N distances of 1.983 (8) Å for the amido bond (W1—N7) and 2.298 (11) Å for the pyridine (py) donor bond (W1—N1). These bond lengths seem to be typical for tungsten(VI) complexes; in trichlorotris(diethylamido)-tungsten, the W—N(amido) distances are 1.913 (5)–1.938 (5) Å (Dietz, Allured & Dubois, 1993) and in dichlorobis(phenylimido)bipyridinetungsten, W—N(py) bond lengths of 2.315 (9) and 2.319 (8) Å occur (Clark, Nielson & Richard, 1988).

In the title complex, the W=O distance is 1.683 (10) Å and the W—Cl distances are between

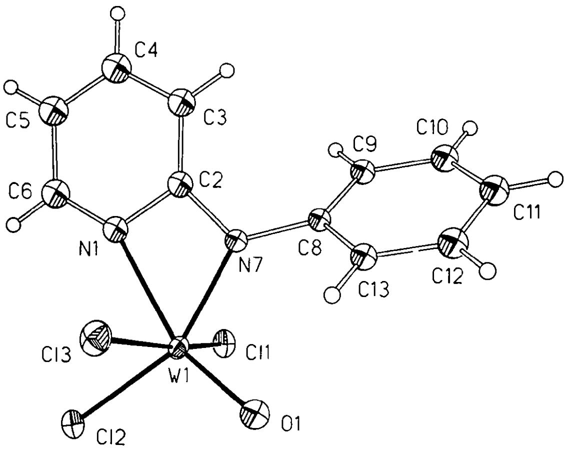


Fig. 1. The molecular structure of the title molecule with ellipsoids drawn at the 30% probability level.