Crystal structure of trichloro(phenyl-2-pyridylamido)phenylimido-tungsten(VI), $WCl_3(C_6H_5N)(C_{11}H_9N_2)$

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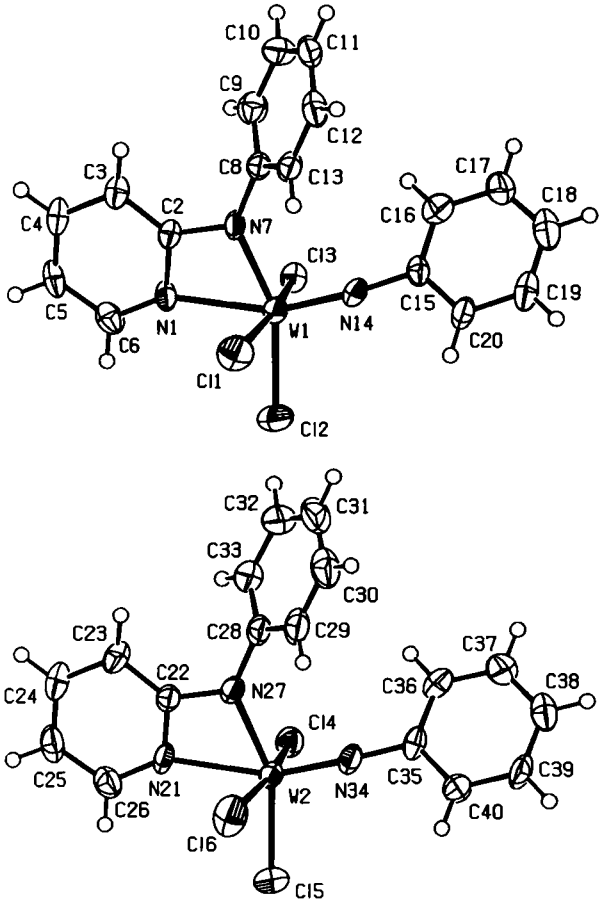

Abstract

$C_{17}H_{14}Cl_3N_3W$, triclinic, $P\bar{1}$ (no. 2), $a = 8.855(2)$ Å, $b = 14.313(3)$ Å, $c = 16.214(3)$ Å, $\alpha = 106.73(3)°$, $\beta = 102.93(3)°$, $\gamma = 97.82(3)°$, $V = 1873.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.113$, $T = 193$ K.

Source of material

The title compound was obtained in a two-step procedure. Phenylimidotetrachlorotungsten(IV) was synthesized by previously described method [1]. $WOCl_4$ (5.70 g) and phenylisocyanate (2.00 g) were refluxed in toluene for 6 hours to give phenylimidotetrachlorotungsten(IV). After isolation and recrystallization, phenylimidotetrachlorotungsten(IV) (4.20 g) was refluxed with 2-phenylamidopyridine (1.70 g) in toluene for 2 hours. The mixture was filtered through silica and crystals were obtained from toluene. All reaction steps were carried out under argon atmosphere using standard Schlenk technique.

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Discussion

The title compound crystallizes with two highly similar complex molecules in the asymmetric unit. The W atoms have pseudo-octahedral coordination spheres. The main reason for distortions are the narrow N–W–N angles (61.4(2)° and 60.9(3)°) in the four membered chelate rings. These *cis*-angles involving imino nitrogens are the widest, N14–W1–Cl2 is 107.4(2)° and N34–W2–Cl5 105.7(2)°. The effect of imido-group can also be seen in the bent *trans*-chlorine angles that are 167.63(8)° for Cl1–W1–Cl3 and 167.11(9)° in Cl6–W2–Cl4, respectively. The complex units are also highly similar in their bond lengths, and comparable to trichloro-2-(phenylamido)pyridineoxotungsten(VI) [2].

Table 1. Data collection and handling.

Crystal:	black block, size 0.22 × 0.22 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	65.97 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7S, $\omega/2\theta$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6886, 6596
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5505
$N(param)_{refined}$:	433
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	–0.1620	0.3194	–0.3246	0.041
H(4)	2*i*	–0.3378	0.3987	–0.3963	0.047
H(5)	2*i*	–0.2936	0.4410	–0.5167	0.053
H(6)	2*i*	–0.0805	0.3995	–0.5691	0.045
H(9)	2*i*	0.1198	0.3678	–0.2144	0.049
H(10)	2*i*	0.1633	0.3116	–0.0928	0.057
H(11)	2*i*	0.1765	0.1458	–0.1132	0.055
H(12)	2*i*	0.1620	0.0404	–0.2533	0.051
H(13)	2*i*	0.1248	0.0971	–0.3751	0.042
H(16)	2*i*	0.4932	0.2622	–0.2560	0.055
H(17)	2*i*	0.6993	0.2098	–0.1823	0.061
H(18)	2*i*	0.8278	0.0986	–0.2628	0.064
H(19)	2*i*	0.7460	0.0437	–0.4173	0.058
H(20)	2*i*	0.5429	0.1002	–0.4924	0.046
H(23)	2*i*	0.3130	0.3078	0.2200	0.047
H(24)	2*i*	0.1804	0.4354	0.2082	0.057
H(25)	2*i*	0.2742	0.5513	0.1475	0.057
H(26)	2*i*	0.5055	0.5418	0.1025	0.049
H(29)	2*i*	0.5832	0.0985	0.0890	0.059
H(30)	2*i*	0.5831	–0.0266	0.1539	0.070
H(31)	2*i*	0.5676	0.0064	0.2978	0.088
H(32)	2*i*	0.5700	0.1669	0.3832	0.082

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(33)	2*i*	0.5608	0.2925	0.3197	0.058
H(36)	2*i*	0.9440	0.1856	0.2354	0.049
H(37)	2*i*	1.1247	0.0881	0.2532	0.060

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(38)	2*i*	1.2461	0.0277	0.1422	0.058
H(39)	2*i*	1.1976	0.0752	0.0157	0.058
H(40)	2*i*	1.0187	0.1746	−0.0046	0.051

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(1)	2*i*	0.22534(4)	0.27424(2)	−0.46783(2)	0.0274(2)	0.0384(2)	0.0327(2)	0.0131(1)	0.0112(1)	0.0152(2)
Cl(1)	2*i*	0.0510(3)	0.1273(2)	−0.5733(2)	0.054(1)	0.042(1)	0.049(1)	0.007(1)	0.012(1)	0.005(1)
Cl(2)	2*i*	0.2841(3)	0.3148(2)	−0.5894(2)	0.055(1)	0.059(1)	0.039(1)	0.015(1)	0.023(1)	0.021(1)
Cl(3)	2*i*	0.3638(2)	0.4391(2)	−0.3786(1)	0.030(1)	0.043(1)	0.038(1)	0.0104(8)	0.0092(8)	0.0133(9)
N(1)	2*i*	0.0040(7)	0.3419(5)	−0.4787(4)	0.020(3)	0.038(4)	0.040(4)	0.009(3)	0.005(3)	0.019(3)
C(2)	2*i*	−0.0199(9)	0.3195(6)	−0.4070(5)	0.021(4)	0.033(4)	0.037(4)	0.007(3)	0.009(3)	0.012(4)
C(3)	2*i*	−0.1468(9)	0.3375(6)	−0.3733(6)	0.026(4)	0.032(4)	0.051(5)	0.010(3)	0.012(4)	0.018(4)
C(4)	2*i*	−0.2506(9)	0.3841(6)	−0.4165(6)	0.022(4)	0.036(5)	0.060(6)	0.010(3)	0.009(4)	0.016(4)
C(5)	2*i*	−0.225(1)	0.4087(7)	−0.4885(6)	0.031(5)	0.048(5)	0.054(6)	0.018(4)	0.000(4)	0.025(5)
C(6)	2*i*	−0.096(1)	0.3847(6)	−0.5190(6)	0.034(4)	0.037(5)	0.042(5)	0.004(4)	0.003(4)	0.021(4)
N(7)	2*i*	0.1028(7)	0.2721(5)	−0.3804(4)	0.021(3)	0.041(4)	0.041(4)	0.015(3)	0.010(3)	0.022(3)
C(8)	2*i*	0.1208(9)	0.2380(6)	−0.3063(5)	0.025(4)	0.037(4)	0.037(4)	0.010(3)	0.011(3)	0.019(4)
C(9)	2*i*	0.130(1)	0.3022(7)	−0.2220(6)	0.037(5)	0.040(5)	0.048(5)	0.008(4)	0.013(4)	0.017(4)
C(10)	2*i*	0.154(1)	0.2683(8)	−0.1497(6)	0.047(5)	0.063(6)	0.032(5)	0.011(5)	0.011(4)	0.014(4)
C(11)	2*i*	0.164(1)	0.1693(8)	−0.1617(6)	0.029(4)	0.073(7)	0.050(6)	0.018(4)	0.010(4)	0.039(5)
C(12)	2*i*	0.154(1)	0.1063(7)	−0.2455(7)	0.029(4)	0.048(5)	0.059(6)	0.011(4)	0.007(4)	0.032(5)
C(13)	2*i*	0.1319(9)	0.1401(6)	−0.3184(6)	0.026(4)	0.040(5)	0.044(5)	0.011(3)	0.014(4)	0.019(4)
N(14)	2*i*	0.3746(8)	0.2196(5)	−0.4256(5)	0.031(4)	0.041(4)	0.039(4)	0.018(3)	0.017(3)	0.013(3)
C(15)	2*i*	0.4948(9)	0.1850(6)	−0.3815(6)	0.020(4)	0.035(4)	0.042(5)	0.006(3)	0.007(3)	0.021(4)
C(16)	2*i*	0.544(1)	0.2184(7)	−0.2884(6)	0.043(5)	0.057(6)	0.048(5)	0.015(4)	0.022(4)	0.021(5)
C(17)	2*i*	0.667(1)	0.1870(8)	−0.2446(7)	0.031(5)	0.077(7)	0.044(5)	0.010(5)	0.005(4)	0.023(5)
C(18)	2*i*	0.745(1)	0.1204(8)	−0.2928(8)	0.031(5)	0.060(6)	0.077(7)	0.011(4)	0.008(5)	0.041(6)
C(19)	2*i*	0.696(1)	0.0881(7)	−0.3849(7)	0.030(5)	0.051(6)	0.073(7)	0.020(4)	0.017(5)	0.025(5)
C(20)	2*i*	0.5730(9)	0.1208(6)	−0.4301(6)	0.025(4)	0.045(5)	0.047(5)	0.015(4)	0.013(4)	0.013(4)
W(2)	2*i*	0.73691(4)	0.33784(2)	0.10452(2)	0.0259(2)	0.0405(2)	0.0313(2)	0.0117(1)	0.0119(1)	0.0131(2)
Cl(4)	2*i*	0.8883(2)	0.4450(2)	0.2498(1)	0.032(1)	0.044(1)	0.037(1)	0.0095(9)	0.0078(8)	0.0120(9)
Cl(5)	2*i*	0.8543(3)	0.4550(2)	0.0495(2)	0.057(1)	0.070(2)	0.055(1)	0.019(1)	0.030(1)	0.038(1)
Cl(6)	2*i*	0.5592(3)	0.2620(2)	−0.0397(2)	0.046(1)	0.079(2)	0.037(1)	0.016(1)	0.002(1)	0.003(1)
N(21)	2*i*	0.5407(7)	0.4221(5)	0.1340(4)	0.022(3)	0.047(4)	0.033(4)	0.016(3)	0.011(3)	0.009(3)
C(22)	2*i*	0.4850(9)	0.3544(6)	0.1675(5)	0.026(4)	0.040(5)	0.028(4)	0.011(3)	0.008(3)	0.011(3)
C(23)	2*i*	0.3498(9)	0.3557(6)	0.1965(6)	0.026(4)	0.039(5)	0.051(5)	0.005(4)	0.016(4)	0.010(4)
C(24)	2*i*	0.272(1)	0.4314(7)	0.1890(7)	0.035(5)	0.050(6)	0.060(6)	0.020(4)	0.019(4)	0.010(5)
C(25)	2*i*	0.328(1)	0.5009(7)	0.1533(6)	0.038(5)	0.060(6)	0.046(5)	0.027(5)	0.005(4)	0.017(5)
C(26)	2*i*	0.466(1)	0.4949(7)	0.1261(6)	0.035(5)	0.045(5)	0.044(5)	0.012(4)	0.004(4)	0.019(4)
N(27)	2*i*	0.5843(7)	0.2854(5)	0.1606(4)	0.027(3)	0.033(4)	0.039(4)	0.010(3)	0.015(3)	0.013(3)
C(28)	2*i*	0.575(1)	0.2086(6)	0.1988(6)	0.024(4)	0.041(5)	0.056(5)	0.011(3)	0.014(4)	0.021(4)
C(29)	2*i*	0.580(1)	0.1123(7)	0.1481(7)	0.030(5)	0.052(6)	0.072(7)	0.017(4)	0.018(5)	0.023(5)
C(30)	2*i*	0.579(1)	0.0375(8)	0.1870(9)	0.034(5)	0.047(6)	0.097(9)	0.010(4)	0.010(6)	0.031(6)
C(31)	2*i*	0.572(1)	0.0573(9)	0.273(1)	0.052(7)	0.071(8)	0.14(1)	0.029(6)	0.037(7)	0.074(9)
C(32)	2*i*	0.570(1)	0.153(1)	0.3235(9)	0.054(7)	0.10(1)	0.086(8)	0.028(6)	0.034(6)	0.065(8)
C(33)	2*i*	0.568(1)	0.2291(8)	0.2864(7)	0.038(5)	0.064(6)	0.056(6)	0.018(5)	0.018(4)	0.030(5)
N(34)	2*i*	0.8577(8)	0.2523(5)	0.1038(5)	0.024(3)	0.046(4)	0.038(4)	0.012(3)	0.012(3)	0.012(3)
C(35)	2*i*	0.9625(9)	0.1908(6)	0.1142(5)	0.019(4)	0.036(4)	0.036(4)	0.006(3)	0.005(3)	0.001(4)
C(36)	2*i*	0.995(1)	0.1637(7)	0.1913(6)	0.036(5)	0.048(5)	0.038(5)	0.009(4)	0.019(4)	0.009(4)
C(37)	2*i*	1.101(1)	0.1050(8)	0.2012(7)	0.043(5)	0.063(6)	0.051(6)	0.015(5)	0.011(5)	0.029(5)
C(38)	2*i*	1.176(1)	0.0696(7)	0.1352(7)	0.033(5)	0.043(5)	0.066(6)	0.013(4)	0.005(4)	0.018(5)
C(39)	2*i*	1.146(1)	0.0972(8)	0.0592(6)	0.038(5)	0.073(7)	0.046(5)	0.032(5)	0.025(4)	0.018(5)
C(40)	2*i*	1.040(1)	0.1567(7)	0.0470(6)	0.052(5)	0.056(6)	0.033(4)	0.028(5)	0.023(4)	0.018(4)

References

1. Nielson, A. J.: Phenylimido complexes of tungsten and rhenium. Inorg. Syn. **24** (1986) 194-200.
2. Polamo, M.: *mer*-Trichloro[2-(phenylamino)pyridinato]oxotungsten(VI). Acta Crystallogr. C**52** 1996 2980-2982.
3. Molecular Structure Corporation: teXsan. Single Crystal Structure Analysis Software. Version 1.6c. MSC, The Woodlands, Texas, USA 1993.
4. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXTL. Version 5.1. Structure Determination Software Suite. Bruker AXS, Madison, Wisconsin, USA 1997.