

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

PRINCE, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.

WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, P. W. (1985). *CRYSTALS User Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.

Acta Cryst. (1989). **C45**, 815–816

Bis(4,6-dimethyl-2-nitrosophenylamido)palladium(II) Monohydrate

By R. G. PRITCHARD, G. S. HEATON AND I. M. EL-NAHAL

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

(Received 30 September 1988; accepted 10 November 1988)

Abstract. $[\text{Pd}(\text{C}_8\text{H}_9\text{N}_2\text{O})_2]\cdot\text{H}_2\text{O}$, $M_r = 422.76$, monoclinic, $C2/c$, $a = 16.156(5)$, $b = 13.883(4)$, $c = 7.609(4)$ Å, $\beta = 105.15(5)^\circ$, $V = 1647.3$ Å³, $Z = 4$, $D_x = 1.70$ Mg m⁻³, $F(000) = 856$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.089$ mm⁻¹, $T = 293$ K, $R = 0.043$ for 1153 unique reflexions [$F \geq 3\sigma(F)$]. The water molecule lies on a crystallographic twofold axis and associates with adjacent centrosymmetric bis(4,6-dimethyl-2-nitrosophenylamido)palladium(II) molecules [$\text{Pd}-\text{N}$ 1.984(4), 2.023(4) Å] via two pairs of hydrogen bonds [$\text{H}_2\text{O}\cdots\text{HN}$ 2.38(8), $\text{O}\cdots\text{N}$ 3.02(1) Å, $\text{N}-\text{H}\cdots\text{O}$ 161(4)° and $\text{HOH}\cdots\text{O}=\text{N}$ 1.99(9), $\text{O}\cdots\text{O}$ 2.73(1) Å, $\text{O}-\text{H}\cdots\text{O}$ 165(4)°] to form infinite diagonal chains. An amine C–N bond order of approximately 2 [1.292(6) Å], considerable phenyl 3,5-diene character, and a shortened nitrosyl C–N [1.333(6) Å] indicate that excess amine negative charge, due to replacement of hydrogen by palladium, has been redistributed.

Experimental. The sample was prepared by adding 4,6-dimethyl-2-nitrosoaniline (1 mol) and sodium hydroxide (2 mol) to a methanolic solution of (dichlorodibenzonitrile)palladium(II) (1 mol) and stirring at room temperature for 5 h. The black precipitate thus formed was recrystallized from chloroform.

Crystal dimensions 0.30 × 0.15 × 0.08 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($11.1 \leq \theta \leq 16.8^\circ$), $\omega-2\theta$ scan mode, ω scan width of $(0.90 + 0.35\tan\theta)^\circ$ and scan speed ranging from 0.5 to 5° min⁻¹ according to the intensity gathered in a pre-scan, $-18 \leq h \leq 18$, $0 \leq k \leq 16$, $0 \leq l \leq 9$, $0 \leq \theta \leq 25^\circ$, 3047 reflexions measured, 1315 unique, $R_{\text{int}} = 0.029$, 1153 observed [$F \geq 3\sigma(F)$], no drift in intensity standards (913, 751, 660) measured every 2.5 h, Lp and absorption corrections (transmission

factors max., min. 0.92, 0.80). Structure solved by normal heavy-atom techniques followed by full-matrix least squares based on F using *SHELX76* (Sheldrick, 1976), final $R = 0.043$, $wR = 0.050$, $w = 0.2607/[\sigma^2(F_o) + 0.0008F_o^2]$, anisotropic thermal parameters for heavier atoms, H from ΔF subjected to isotropic refinement. Maximum fluctuation in final ΔF map ± 1.42 e Å⁻³ near to Pd, ± 0.32 e Å⁻³ elsewhere, maximum Δ/σ 0.002. Scattering factors from *International Tables for X-ray Crystallography* (1974), computation carried out on the joint CDC7600/Amdahl 470 system of the University of Manchester Regional Computing Centre. Literature survey from the Cambridge Structural Database was performed using the Crystal Structural Search and Retrieval interactive system (CSSR, 1984). The molecule including labelling scheme is illustrated in Fig. 1. Final atomic coordinates and selected molecular geometry are presented in Tables 1* and 2 respectively.

* Lists of structure factors, H-atom coordinates, anisotropic vibrational parameters and complete molecular geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51592 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

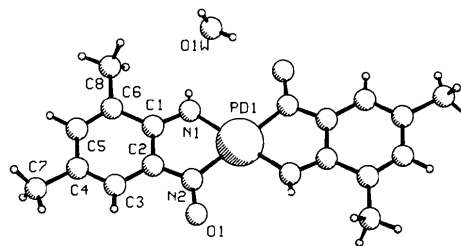


Fig. 1. The centrosymmetric title molecule and associated water drawn using *PLUTO* (Motherwell & Clegg, 1978).

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic vibrational parameters for non-hydrogen atoms ($\text{\AA}^2 \times 10^3$ except Pd, $\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}^*
Pd(1)	0	5000	5000	381 (1)
N(1)	611 (3)	5753 (3)	7168 (5)	44 (1)
N(2)	-950 (3)	5229 (3)	6233 (6)	43 (1)
O(1)	-1721 (3)	4944 (2)	5643 (6)	63 (1)
C(1)	170 (3)	5982 (3)	8297 (6)	38 (1)
C(2)	-720 (3)	5703 (3)	7810 (6)	39 (1)
C(3)	-1262 (3)	5916 (4)	8933 (6)	43 (1)
C(4)	-964 (3)	6408 (3)	10480 (6)	46 (1)
C(5)	-76 (3)	6683 (3)	10995 (6)	48 (1)
C(6)	481 (3)	6496 (3)	9993 (6)	43 (1)
C(7)	-1520 (5)	6673 (5)	11713 (9)	67 (1)
C(8)	1412 (3)	6773 (5)	10597 (8)	60 (1)
O(1W)	2500	6005 (5)	7500	57 (1)

$$* U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i a_j^*$$

Related literature. The title molecule shares some structural features with the following: α -bis(1,2-benzoquinone dioximato)palladium(II) (Kistenmacher & Destro, 1983), β -bis(1,2-benzoquinone dioximato)palladium(II), (Endres, Mégnamisi-Bélombé, Little & Wolfe, 1979), 1:1 pyridine adduct of (4-methyl-1-quinone 2-oximato)copper(II) (McPartlin, 1973).

The authors thank the SERC for financial support via an equipment grant.

Acta Cryst. (1989). **C45**, 816–817

Structure of [ZnCl(C₄H₈O)(μ -Cl)]_∞

BY FRANK BOTTOMLEY, EDITH C. FERRIS AND PETER S. WHITE

Department of Chemistry, University of New Brunswick, Fredericton, New Brunswick, Canada E3B 5A3

(Received 6 September 1988; accepted 8 November 1988)

Abstract. *catena*-Poly[[chloro(tetrahydrofuran)zinc(μ -chloro)], $M_r = 208.39$, orthorhombic, $Pc2_1b$, $a = 6.928$ (1), $b = 7.306$ (1), $c = 15.197$ (3) \AA , $V = 769.2$ (4) \AA^3 , $Z = 4$, $D_x = 1.80$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ \AA , $\mu = 38.8$ cm⁻¹, $F(000) = 416$, $T = 295$ K, $R = 0.037$, $wR = 0.044$ for 1065 unique observed reflections [$I_o \geq 2.5\sigma(I)$]. Zn is tetrahedrally coordinated by a C₄H₈O (THF) ligand [Zn–O = 1.981 (3) \AA], a terminal Cl [Zn–Cl = 2.169 (1) \AA] and two bridging Cl [Zn–Cl = 2.289 (4) and 2.323 (4) \AA]. Infinite chains of [ZnCl(THF)(μ -Cl)] units are formed along the 2₁ axis of the crystal.

Experimental. Colourless needles of [ZnCl(THF)(μ -Cl)]_∞ were obtained as a coproduct (with [(η -C₅H₅)MoCl(O)]₂(μ -O)) of the oxidation of [(η -C₅H₅)MoCl(O)]₂(μ -Cl)₂Zn(THF)] by O₂ (Bottomley,

Table 2. Selected bond lengths (\AA) and angles ($^\circ$)

Pd(1)–N(1)	1.984 (4)	Pd(1)–N(2)	2.023 (4)
N(1)–C(1)	1.292 (6)	N(2)–O(1)	1.272 (6)
N(2)–C(2)	1.333 (6)	C(1)–C(2)	1.441 (6)
C(1)–C(6)	1.446 (6)	C(2)–C(3)	1.405 (6)
C(3)–C(4)	1.336 (7)	C(4)–C(5)	1.436 (7)
C(4)–C(7)	1.505 (7)	C(5)–C(6)	1.348 (7)
C(6)–C(8)	1.503 (7)		
N(2)–Pd(1)–N(1)	78.7 (2)	C(3)–C(2)–C(1)	121.7 (4)
C(1)–N(1)–Pd(1)	116.1 (3)	C(4)–C(3)–C(2)	120.3 (5)
C(2)–N(2)–Pd(1)	115.4 (3)	C(5)–C(4)–C(3)	118.7 (4)
C(2)–C(1)–N(1)	116.8 (4)	C(6)–C(5)–C(4)	124.1 (4)
C(6)–C(1)–C(2)	116.9 (4)	C(5)–C(6)–C(1)	118.2 (4)
C(1)–C(2)–N(2)	112.8 (4)		

References

- CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual*. SERC Daresbury Laboratory, Warrington, England.
- ENDRES, H., MÉGNAMISI-BÉLOMBÉ, M., LITTLE, W. A. & WOLFE, C. R. (1979). *Acta Cryst.* **B35**, 169–171.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- KISTENMACHER, T. J. & DESTRO, R. (1983). *Inorg. Chem.* **22**, 2104–2110.
- MCPARTLIN, M. (1973). *Inorg. Nucl. Chem. Lett.* **9**, 1207–1210.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Ferris & White, 1988). The crystals were extremely hygroscopic. One, of dimensions 0.15 \times 0.15 \times 0.40 mm, was sealed in a capillary in a dry box and then mounted on an Enraf–Nonius CAD-4 diffractometer. Lattice constants were obtained by accurate centring of 16 reflections in the range $30 < 2\theta < 40^\circ$. Intensities were measured using the $\omega/2\theta$ scan mode to a $2\theta_{\text{max}}$ of 50° (h_{max} 7, k_{max} 8, l_{max} 17). Three standard reflections were monitored every hour. There was no significant change in their intensity. The intensities of 1883 reflections were measured and averaged to yield 1337 unique reflections (including Friedel pairs), of which 1065 were judged as being significant by the criterion that $I > 2.5\sigma(I)$. No absorption correction was made. The structure was solved using direct methods (*MULTAN*80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and all other