$(\Delta/\sigma)_{\max} = 0{\cdot}05$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Data reduction carried out using Enraf–Nonius (1983) *SDP* program, a locally modified version of *ALLS* (Lapp & Jacobson, 1979) used for structure refinement and *ORFFE* (Busing, Martin & Levy, 1964) for structural parameters with e.s.d.'s. Table 1 gives atomic coordinates and Table 2 bond distances and angles.* Fig. 1 gives the atom numbering and perspective drawing of the molecule. Mn is in the plane with an average $Mn—N_p$ bond distance of 2·002 (4) Å and an axial Mn—O distance of 2·283 (5) Å. These distances are consistent with a high-spin six-coordinate manganese(III) state (Scheidt & Gouterman, 1983).

Related literature. The structures of a number of six-coordinate Mn^{III} porphyrinates have been reported (Kirner & Scheidt, 1975; Hatano, Anzaik & Iitaka, 1983; Hill & Williamson, 1985*a,b*). Most of these have axial oxygen donors. As expected for high-spin six-coordinate manganese(III) species, all have long axial bonds, Mn–O $\simeq$ 2·2–2·3 Å.

Support of this work by the National Institutes of Health (GM-38401) is gratefully acknowledged.

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE. A Fortran Crystallographic Function and Error Program.* Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

Enraf–Nonius (1983). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Hatano, K., Anzaik, K. & Iitaka, Y. (1983). *Bull. Chem. Soc. Jpn*, **56**, 422–427.

Hill, C. L. & Williamson, M. M. (1985*a*). *Inorg. Chem.* **24**, 2836–2841.

Hill, C. L. & Williamson, M. M. (1985*b*). *Inorg. Chem.* **24**, 3024–3030.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kirner, J. F. & Scheidt, W. R. (1975). *Inorg. Chem.* **14**, 2081–2086.

Lapp, R. L. & Jacobson, R. A. (1979). *ALLS. A Generalized Crystallographic Least-Squares Program.* National Technical Information Services IS-4708 UC-4, Springfield, Virginia, USA.

Scheidt, W. R. & Gouterman, M. (1983). *Iron Porphyrins, Part One,* edited by A. B. P. Lever & H. B. Gray, pp. 89–139. Reading, MA: Addison-Wesley.

---

* Lists of structure factors, anisotropic thermal parameters, and fixed hydrogen positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44681 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 929–931

Structure of Di-*μ*-acetato-(*O*,*O'*)-bis[*N*-(α-phenylbenzylidene)cyclohexylamine-*N*,*C*]-dipalladium(II)

By M. Gayoso, M. Alonso and J. M. Vila

Departamento de Quimica Inorganica, Facultad de Quimica, Universidad de Santiago, Santiago de Compostela, Spain

E. B. Rivero

Universidad Nacional de La Plata, Facultad de Ciencias Exactas, Departamento de Fisica, La Plata, Argentina

and W. Hiller and J. Strähle

Institut für anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, D-7400 Tübingen, Federal Republic of Germany

(*Received* 18 *October* 1987; *accepted* 14 *January* 1988)

Abstract. $[Pd_2(C_2H_3O_2)_2(C_{19}H_{20}N)_2]$, $M_r = 855{\cdot}65$, orthorhombic, *Pbcn*, $a = 19{\cdot}419\ (4)$, $b = 10{\cdot}739\ (4)$, $c = 18{\cdot}224\ (4)$ Å, $V = 3800{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}495$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 9{\cdot}750$ cm^{-1}, $F(000) = 1744$, $T = 293$ K. The final *R* value is 0·029 for 2494 significant $[I > 3\sigma(I)]$ reflections. In the dinuclear complex of site symmetry 2 each Pd atom is bonded to an N atom, an *ortho* C atom of the benzyl group and to two O atoms of the bridging acetato groups.

0108-2701/88/050929-03$03.00

Experimental. 0·34 g (1·51 mmol) of palladium(II) acetate and 0·4 g (1·52 mmol) of *N*-(α-phenylbenzylidene)cyclohexylamine were refluxed in glacial acetic acid for 2 h. Elimination of the solvent gave a residue which was column-chromatographed. The final product was recrystallized from CH_2Cl_2/*n*-hexane to give yellow prisms. The parameters for intensity-data

Table 1. *Data-collection and structure-refinement parameters*

Crystal shape, dimensions	Prismatic, 0·10 × 0·10 × 0·30 mm
Diffractometer used	CAD-4, Enraf–Nonius
Method of intensity measurement	$\theta/2\theta$
No. and θ range of reflections for lattice parameters	25; 11–19°
Method used for absorption correction	*DIFABS* (Walker & Stuart, 1983)
Minimum absorption correction	0·871
Maximum absorption correction	1·114
Average absorption correction	0·996
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement	0·595 Å^{-1}
Range of h, k, l	−23→23, 0→12, 0→21
Standard reflections	12,0,12, $\bar{14}$,2,0
Interval, standard reflections measured	2h, no intensity variation
Total No. of reflections measured; θ range	7317; 25° (741 systematic absences included)
No. of unique reflections; $R_{\rm int}$	3344; 0·018
No. of observed reflections	2494
Criterion for observed reflections	$I > 3\sigma(I)$
Methods used to solve structure	Patterson and subsequent difference Fourier map
Use of F or F^2 in LS refinement	F
Method of locating H atoms	ΔF map (H atoms of acetato group not found)
Method of refining H atoms	Positional parameters with fixed isotropic temperature factors
Weighting scheme	$1/\sigma^2$
Parameters refined	287
Value of R	0·029
Value of wR	0·032
Ratio of max. LS shift to e.s.d. (Δ/σ)	0·05
Max. height in final ΔF map	0·39 e Å^{-3}
Error in an observation of unit weight	2·322
Secondary-extinction coefficient	1·8770 (1) × 10^{-9} (Zachariasen, 1963)
Source of atomic scattering factors	*International Tables for X-ray Crystallography* (1974)
Computer used	DEC Micro VAXII
Programs used	*VAXSDP* (B. A. Frenz & Associates Inc., 1985)

Table 2. *Positional and equivalent isotropic thermal parameters* U_{eq}(Å^2)

$U_{\rm eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

	x	y	z	$U_{\rm eq}$
Pd	0·46243 (2)	0·55536 (3)	0·32963 (2)	0·0313 (1)
O(1)	0·4259 (2)	0·7140 (3)	0·2818 (2)	0·038 (2)
O(2)	0·4391 (2)	0·6498 (3)	0·1658 (2)	0·044 (2)
N	0·4843 (2)	0·3878 (3)	0·3780 (2)	0·033 (2)
C(1)	0·4174 (2)	0·7218 (5)	0·2134 (3)	0·040 (2)
C(2)	0·3756 (3)	0·8355 (5)	0·1888 (3)	0·054 (3)
C(3)	0·4313 (2)	0·3162 (4)	0·3873 (2)	0·033 (2)
C(11)	0·3691 (2)	0·4898 (4)	0·3378 (2)	0·034 (2)
C(12)	0·3649 (2)	0·3708 (4)	0·3691 (2)	0·035 (2)
C(13)	0·3017 (3)	0·3126 (5)	0·3802 (3)	0·045 (3)
C(14)	0·2418 (3)	0·3734 (5)	0·3613 (3)	0·052 (3)
C(15)	0·2453 (2)	0·4902 (5)	0·3311 (3)	0·053 (3)
C(16)	0·3081 (2)	0·5494 (5)	0·3188 (3)	0·044 (3)
C(21)	0·5543 (2)	0·3440 (4)	0·3964 (2)	0·038 (2)
C(22)	0·5857 (3)	0·4290 (5)	0·4551 (2)	0·047 (3)
C(23)	0·6598 (3)	0·3918 (6)	0·4716 (3)	0·064 (4)
C(24)	0·7029 (3)	0·3873 (8)	0·4028 (4)	0·082 (6)
C(25)	0·6726 (3)	0·3045 (7)	0·3458 (3)	0·057 (5)
C(26)	0·5983 (2)	0·3402 (5)	0·3274 (3)	0·048 (3)
C(31)	0·4343 (2)	0·1829 (4)	0·4105 (3)	0·038 (2)
C(32)	0·4106 (3)	0·1454 (5)	0·4786 (3)	0·055 (3)
C(33)	0·4074 (3)	0·0190 (5)	0·4948 (3)	0·070 (4)
C(34)	0·4264 (3)	−0·0682 (5)	0·4442 (4)	0·067 (4)
C(35)	0·4501 (3)	−0·0313 (5)	0·3769 (3)	0·060 (3)
C(36)	0·4535 (3)	0·0940 (5)	0·3594 (3)	0·051 (3)

Table 3. *Selected distances* (Å) *and angles* (°) *with e.s.d.'s*

Symmetry operator: (i) 1−*x*, *y*, 0·5−*z*.

Pd—O(1)	2·040 (3)	O(1)—Pd—O(2$^{\rm i}$)	86·2 (1)
Pd—O(2$^{\rm i}$)	2·165 (4)	O(1)—Pd—N	171·6 (1)
Pd—N	2·048 (4)	O(1)—Pd—C(11)	90·6 (2)
Pd—C(11)	1·949 (4)	O(2$^{\rm i}$)—Pd—N	102·2 (1)
O(1)—C(1)	1·261 (5)	O(2$^{\rm i}$)—Pd—C(11)	170·7 (2)
O(2)—C(1)	1·236 (6)	N—Pd—C(11)	80·9 (2)
C(1)—C(2)	1·534 (7)	Pd—O(1)—C(1)	121·5 (3)
N—C(3)	1·297 (6)	Pd—N—C(3)	114·4 (3)
N—C(21)	1·478 (6)	Pd—N—C(21)	124·7 (3)
C(3)—C(12)	1·454 (6)	C(3)—N—C(21)	120·8 (4)
C(3)—C(31)	1·493 (6)	Pd—C(11)—C(12)	114·5 (4)
		Pd—C(11)—C(16)	127·3 (3)
C(21)—C(22)	1·532 (7)	O(1)—C(1)—O(2)	127·4 (4)
C(21)—C(26)	1·520 (6)	O(1)—C(1)—C(2)	114·4 (5)
C(22)—C(23)	1·523 (7)	O(2)—C(1)—C(2)	118·3 (4)
C(23)—C(24)	1·507 (8)	N—C(3)—C(12)	115·7 (4)
C(24)—C(25)	1·490 (9)	N—C(3)—C(31)	125·1 (4)
C(25)—C(26)	1·530 (7)	C(12)—C(3)—C(31)	119·0 (4)
		C(3)—C(12)—C(11)	114·2 (4)
Pd···Pd$^{\rm i}$	3·2484 (4)		

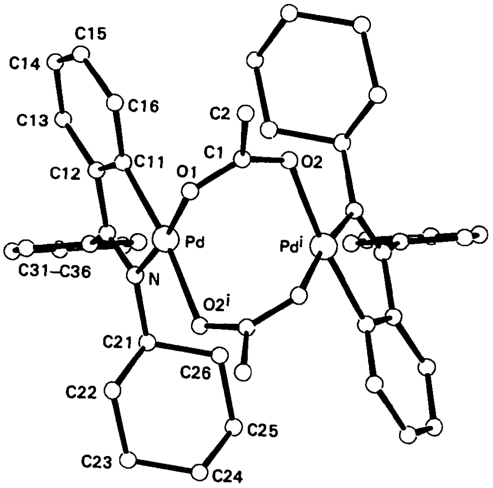

Fig. 1. Plot of the molecule showing the numbering scheme.

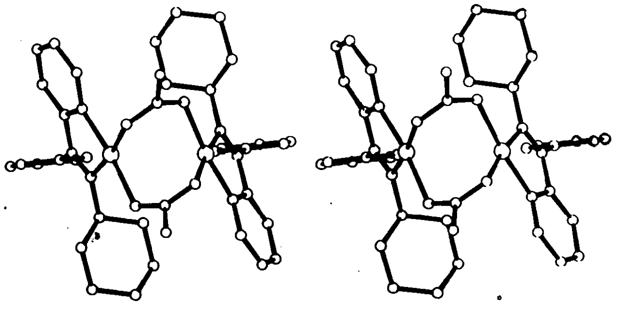

Fig. 2. Stereoscopic view.

collection, structure solution and refinement are listed in Table 1. The final atomic coordinates are given in Table 2, selected bond distances and angles in Table 3.* Figs. 1 and 2 show the numbering scheme and a stereoview, respectively.

Related literature. This structure is one of a series of Pd compounds with Schiff-base ligands. A previous structure is described in Hiller, Castineiras, Vila, Suarez, Pereira & Gayoso (1986).

We thank the DAAD for financial support.

References

B. A. Frenz & Associates Inc. (1985). *VAXSDP Structure Determination Package,* version 2.2. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.
Hiller, W., Castineiras, A., Vila, J. M., Suarez, A., Pereira, M. T. & Gayoso, M. (1986). *Acta Cryst.* C**42**, 1136–1138.
International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

---

* Lists of structure factors, anisotropic thermal parameters, further distances and angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44684 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 931–933

The Structure of Two Gel-Grown Oxamate Complexes: Bis(oxamato-*O,O'*)calcium(II) Tetrahydrate and Bis(oxamato-*O,O'*)cadmium(II) Tetrahydrate

By S. Skoulika,* A. Michaelides* and A. Aubry

Laboratoire de Minéralogie-Cristallographie, UA 809, *Université de Nancy* I, *Faculté des Sciences, BP* 239, 54506 *Vandoeuvre les Nancy CEDEX, France*

(*Received* 4 *December* 1987; *accepted* 12 *January* 1988)

Abstract. The two compounds of general formula $M(C_2O_3NH_2)_2.4H_2O$, where $M = Ca^{II}$ (A), Cd^{II} (B), are isostructural, space group $C2/c$, $Z = 4$, Ag $K\alpha$, $\lambda = 0{\cdot}56087$ Å, 295 K. Compound (A): $[Ca(C_2H_2NO_3)_2].4H_2O$, $M_r = 288{\cdot}2$, $a = 14{\cdot}466\,(3)$, $b = 5{\cdot}993\,(1)$, $c = 12{\cdot}256\,(2)$ Å, $\beta = 99{\cdot}74\,(1)^\circ$, $V = 1047{\cdot}3$ Å^3, $D_x = 1{\cdot}828$ g cm^{-3}, $\mu = 3{\cdot}5$ cm^{-1}, $F(000) = 595$, $R = 0{\cdot}021$ and $wR = 0{\cdot}027$ for 895 reflections with $I > 3\sigma(I)$. Compound (B): $[Cd(C_2H_2NO_3)_2].4H_2O$, $M_r = 360{\cdot}5$, $a = 14{\cdot}347\,(6)$, $b = 5{\cdot}975\,(1)$, $c = 12{\cdot}184\,(2)$ Å, $\beta = 100{\cdot}09\,(2)^\circ$, $V = 1028{\cdot}4$ Å^3, $D_x = 2{\cdot}328$ g cm^{-3}, $\mu = 10{\cdot}3$ cm^{-1}, $F(000) = 704$, $R = 0{\cdot}020$ and $wR = 0{\cdot}022$ for 919 reflections with $I > 1{\cdot}5\sigma(I)$. M, on a crystallographic twofold axis, is coordinated by two chelating ligands and four water molecules. The coordination number of the metal is eight and the resulting coordination polyhedron is a distorted square antiprism. The observed coordination number which is frequently encountered in the case of Ca compounds is, in contrast, very rarely adopted by Cd^{II} ions. The distances Cd—O are generally slightly shorter than Ca—O in accordance with small differences in the ionic radii of the metal ions. The crystal structure is stabilized by a three-dimensional network of hydrogen bonds of the types O—H···O and N—H···O. Four of these bonds hold the molecules together in chains parallel to (010). The chains are joined to one another by the four remaining hydrogen bonds.

Experimental. Crystals prepared by slow diffusion of the corresponding hydrated metal(II) salt (chloride or nitrate) into a silica-gel mixture containing oxamic acid at neutral pH (Michaelides & Skoulika, 1988). The Cd compound was stored in the presence of its aqueous solution to prevent partial dehydration. Crystals $0{\cdot}25 \times 0{\cdot}30 \times 0{\cdot}20$ mm for (A) and $0{\cdot}25 \times 0{\cdot}20 \times 0{\cdot}20$ mm for (B) were used. Automated CAD-4 Enraf–Nonius diffractometer with incident-beam (Ag $K\alpha$) graphite monochromator. 25 centered reflections within $8{\cdot}4 < \theta < 11{\cdot}7^\circ$ for (A) and $5{\cdot}4 < \theta < 11{\cdot}4^\circ$ for (B) used for determining lattice parameters. ω, 2θ scan mode, max. 2θ 20°. Two standard reflections, no intensity variation. 895 reflections used $[I > 3\sigma(I)]$ from 953 unique reflections measured for (A) and 919 reflections used $[I > 1{\cdot}5\sigma(I)]$ from 952 unique reflections for (B). $-17 < h < 17$, $0 < k < 7$, $0 < l < 14$ for (A) and (B). Data corrected for Lorentz and polarization effects, empirical isotropic correction for (B), $x = 2{\cdot}1\,(1) \times 10^{-3}$, $[F_c' = F_c(1 - 0{\cdot}0001xF_c^2/\sin\theta)]$. No absorption correction made. Data reduction with *SDP* package (Enraf–Nonius, 1979). Structure solved by Patterson and Fourier methods. Least-squares program *SHELX*76 (Sheldrick, 1976). Refine-

---

* Permanent address: Department of Chemistry, University of Ioannina, Ioannina, Greece.

0108-2701/88/050931-03$03.00