

Monoclinic  
 $C2/c$   
 $a = 26.392 (6) \text{ \AA}$   
 $b = 8.041 (1) \text{ \AA}$   
 $c = 18.168 (4) \text{ \AA}$   
 $\beta = 132.39 (1)^\circ$   
 $V = 2847.6 (10) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.415 \text{ Mg m}^{-3}$   
 $D_m$  not measured

#### Data collection

Siemens P4-RA diffractometer  
 $\omega$  scans  
 Absorption correction:  
 $\psi$  scan (SHELXTL-Plus; Sheldrick, 1990)  
 $T_{\min} = 0.589$ ,  $T_{\max} = 0.806$   
 3828 measured reflections  
 3113 independent reflections

#### Refinement

Refinement on  $F^2$   
 $R(F) = 0.0245$   
 $wR(F^2) = 0.0646$   
 $S = 1.091$   
 3113 reflections  
 148 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 0.7817P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 18 reflections  
 $\theta = 10\text{--}20^\circ$   
 $\mu = 1.20 \text{ mm}^{-1}$   
 $T = 150 \text{ K}$   
 Column  
 $0.71 \times 0.26 \times 0.18 \text{ mm}$   
 Yellow

2861 reflections with  $F > 4\sigma(F)$   
 $R_{\text{int}} = 0.015$   
 $\theta_{\text{max}} = 27.01^\circ$   
 $h = -33 \rightarrow 5$   
 $k = -10 \rightarrow 0$   
 $l = -19 \rightarrow 23$   
 1 standard reflection every 99 reflections  
 intensity decay: none

$(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.301 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.165 \text{ e \AA}^{-3}$   
 Extinction correction: SHELXL93  
 Extinction coefficient: 0.0029 (5)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Mn—N2	2.251 (1)	Mn—S2'	2.493 (1)
Mn—N1	2.370 (1)	Mn—S2	2.613 (1)
Mn—S1	2.409 (1)		
N2—Mn—N1	91.13 (5)	N2—Mn—S2	81.16 (4)
N2—Mn—S1	124.26 (4)	N1—Mn—S2	170.38 (3)
N1—Mn—S1	82.51 (4)	S1—Mn—S2	97.02 (2)
N2—Mn—S2'	109.66 (4)	S2'—Mn—S2	91.94 (1)
N1—Mn—S2'	96.10 (3)	Mn'—S2—Mn	88.06 (1)
S1—Mn—S2'	126.06 (2)		

Symmetry code: (i)  $1 - x, -y, 1 - z$ .

A crystal of the title compound was mounted on a glass capillary with silicone grease and placed quickly into the cold nitrogen stream of the cooling device of the goniometer. Data were corrected for Lorentz, polarization and absorption effects. The structure was solved by direct methods and subsequent difference Fourier synthesis. After anisotropic refinement of this model, H atoms were taken from the difference Fourier synthesis and recalculated to occupy idealized positions. One common isotropic displacement parameter was refined for the otherwise riding H atoms.

Data collection: P3 *Diffractometer Control Program* (Siemens, 1991). Cell refinement: P3 *Diffractometer Control Program*. Data reduction: XDISK in P3 *Diffractometer Control Program*. Program(s) used to solve structure: SHELXTL-Plus (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics:

SHELXTL-Plus. Software used to prepare material for publication: SHELXL93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1322). Services for accessing these data are described at the back of the journal.

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### catena-Poly[[[diaqua(pyridine-2-carboxamide- $N^1,O$ )copper(II)]- $\mu$ -(sulfato- $O:O'$ )] monohydrate]

LESŁAW SIEROŃ AND MARIA BUKOWSKA-STRZYŻEWSKA

*Institute of General & Ecological Chemistry, Technical University of Łódź, Żwirki 36, 90-924 Łódź, Poland. E-mail: mbs@ck-sg.p.lodz.pl*

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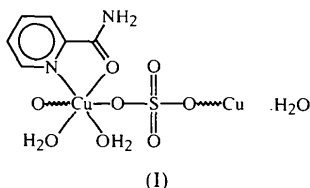
#### Abstract

The new title  $\text{Cu}^{\text{II}}$  complex of pyridine-2-carboxamide (pca,  $\text{C}_6\text{H}_6\text{N}_2\text{O}$ ),  $\{[\text{Cu}(\text{SO}_4)(\text{pca})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}\}_n$ , has been synthesized and its crystal structure determined. It forms two crystallographically independent polymeric  $[\text{Cu}(\text{H}_2\text{O})_2(\text{pca})(\text{SO}_4)]_\infty$  chains in which the sulfate ion adopts a bridging position between two Cu atoms, coordinating *via* two of its O atoms, with Cu—O bond lengths in the range 2.318 (1)–2.455 (1) Å. The pca

ligand is coordinated through the ring N and amide O atoms, forming a five-membered chelate ring with Cu—N distances of 2.010(1) and 1.985(1) Å, and Cu—O distances of 1.964(4) and 1.985(1) Å. The base of the elongated square-bipyramidal Cu<sup>II</sup> coordination is completed by two Cu—O(H<sub>2</sub>O) bonds with lengths in the range 1.961(1)–1.975(1) Å. The polymeric chains are connected by two types of hydrogen bonds, namely, N—H $\cdots$ O bonds between amino N and sulfate O atoms, and O—H $\cdots$ O bonds between the uncoordinated H<sub>2</sub>O molecule and sulfate ions. The influence of the observed hydrogen bonds on the hard structural parameters, such as bond lengths and angles, is analysed.

### Comment

This work is a continuation of our study of the preparation, properties and structure of copper(II) complexes with pyridine-2-carboxamide (Sieroń & Bukowska-Strzyżewska, 1997, 1998). Fig. 1 shows the repeat fragment of the polymeric chains formed in the crystals of the title compound, (I). In both chains, the Cu atoms



have a similar elongated octahedral environment with four short and two long bonds. The short bonds formed by the chelating pca ligand through the amide O and ring N atoms are Cu—O1 1.964(1), Cu—O1' 1.985(1), Cu—N1 2.010(1) and Cu—N1' 1.985(1) Å, and by the two equatorial *cis*-H<sub>2</sub>O molecules are Cu—O2 1.965(1), Cu—O3 1.966(1), Cu—O2' 1.961(1) and Cu—O3' 1.975(1) Å. The observed Cu—N bonds are distinctly longer than similar bonds in more symmetrical complexes, *e.g.* [Cu(H<sub>2</sub>O)<sub>2</sub>(pca)<sub>2</sub>]Cl<sub>2</sub> and [Cu(H<sub>2</sub>O)<sub>2</sub>(pca)<sub>2</sub>]Br<sub>2</sub> (Sieroń & Bukowska-Strzyżewska, 1997), [Cu(ClO<sub>4</sub>)<sub>2</sub>(pca)<sub>2</sub>] (Sieroń & Bukowska-Strzyżewska, 1998) and [Cu(H<sub>2</sub>O)(NO<sub>3</sub>)(pca)<sub>2</sub>]NO<sub>3</sub> $\cdot$ H<sub>2</sub>O (Castro *et al.*, 1990), where their average value is 1.967(1) Å. Observed Cu—O(pca) bonds are also longer than the corresponding bonds in the above-cited perchlorate and nitrate complexes, where they are 1.937(2) and 1.955(2) Å, respectively. In both [Cu(H<sub>2</sub>O)<sub>2</sub>(pca)(SO<sub>4</sub>)<sub>∞</sub>] chains, the sulfate (SO<sub>4</sub><sup>2-</sup>) ions occupy the bridge positions between the CuO<sub>3</sub>N basal planes, forming two long Cu—O bonds ranging from 2.318(1) to 2.455(1) Å. The geometrical shape of both crystallographically independent chains is similar, but not identical. The deformation of the square arrangement of four short Cu<sup>II</sup> bonds around the Cu and Cu' atoms is dif-

ferent. Atoms N1', O1' O2' and O3' form a strongly flattened tetrahedral coordination around the Cu' atom. The average deviations of the *trans*-situated O1'/O3' and N1'/O2' atoms of the N1'/O1'/O2'/O3' least-squares plane are 0.1161(5) and -0.1161(5) Å, while the deviation of the central Cu' atom is only 0.0087(7) Å. In the second chain, the N1, O1, O2 and O3 atoms are nearly coplanar. Their deviations from the least-squares plane range from -0.0151(6) to 0.0185(8) Å. The Cu atom [deviation from this plane: 0.0943(7) Å] is in the apical position of the flattened square pyramid. Also, the flatness of the chelate rings is different in both chains. The deviation of the Cu atom from the plane of the ideally coplanar N1, O1, C1 and C2 atoms is only -0.15(3) Å, and of the Cu' atom from the plane of the coplanar N1', O1', C1' and C2' atoms is 0.096(3) Å.

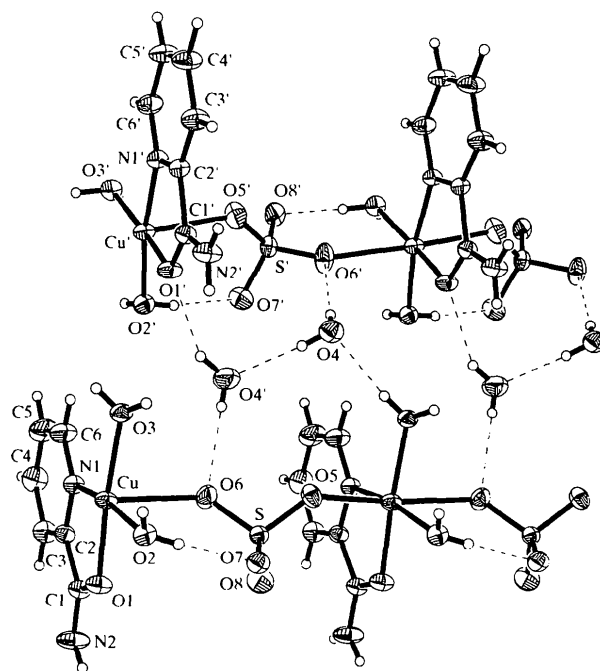


Fig. 1. A fragment of the polymeric [Cu(H<sub>2</sub>O)<sub>2</sub>(pca)(SO<sub>4</sub>)<sub>∞</sub>] chains. Displacement ellipsoids are drawn at the 50% probability level.

We suppose the influence of hydrogen bonds on the observed geometrical shape of both chains. The hydrogen bonds may be responsible for the elongation of the short Cu bonds, for the differentiation of S—O bonds and for the different deformation of the Cu<sup>II</sup> coordination polyhedra of both polymeric chains. The geometry of the observed hydrogen bonds is given in Table 2. They may be divided into intra- and inter-chain bonds and into eight types, in agreement with the different types of connected groups: N—H(NH<sub>2</sub>) $\cdots$ O(pca), N—H(NH<sub>2</sub>) $\cdots$ O(H<sub>2</sub>O),

$N-H(NH_2) \cdots O(SO_4)$ ,  $O-H(H_2O) \cdots O(pca)$ ,  $O-H(H_2O_{coord}) \cdots O(SO_4)$ ,  $O-H(H_2O_{coord}) \cdots O(H_2O)$ ,  $O-H(H_2O) \cdots O(H_2O)$  and  $O-H(H_2O) \cdots O(SO_4)$ . All H atoms of the  $NH_2$  group and  $H_2O$  molecules are involved in these bonds, but the donor groups of each chain form hydrogen bonds of different types. That is why their influence on the deformation of the bond lengths and angles is different in the individual chains. The observed S—O bonds of the sulfate groups are distinctly differentiated [1.451 (1)–1.497 (1) Å], which may be explained by the different number of hydrogen bonds formed by individual sulfate O atoms. The longest S—O bond is formed by the O7 atom, which is involved in three hydrogen bonds. The shortest S—O bond is formed by the O8 atom, which is involved in only one hydrogen bond; the copper-semi-coordinated O5' atom is not involved in the hydrogen-bond system. Fig. 2 shows the molecular packing viewed along the  $x$  axis perpendicular to the  $[Cu(H_2O)_2(pca)(SO_4)]_\infty$  polymeric chains. The uncoordinated  $H_2O$  molecules coupled in pairs by hydrogen bonds are located between the two crystallographically independent chains, connecting these chains through hydrogen bonding.

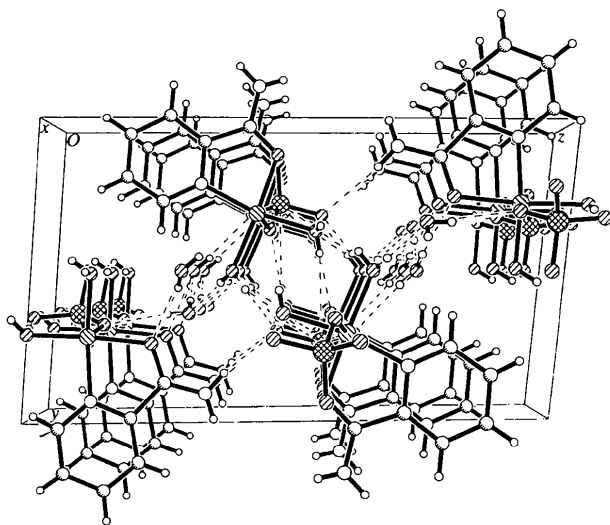


Fig. 2. The molecular packing viewed along the  $x$  axis.

## Experimental

The title crystals were grown from an aqueous solution of 2-pyridinecarbonitrile and copper(II) sulfate, indicating that the copper-assisted hydrolysis of 2-pyridinecarbonitrile to pyridine-2-carboxamide (Watanabe *et al.*, 1973) had occurred. A pale-blue solution formed when  $CuSO_4 \cdot 5H_2O$  (1 mmol) was added to an aqueous solution (50 ml) containing 2-pyridinecarbonitrile (1 mmol). After heating to boiling point, the solution became dark blue. After a few days, blue prismatic crystals of the title compound were obtained.

## Crystal data

$[Cu(SO_4)(C_6H_6N_2O) \cdot (H_2O)_2] \cdot H_2O$   
 $M_r = 335.78$   
 Triclinic  
 $P\bar{1}$   
 $a = 6.9446$  (6) Å  
 $b = 9.8415$  (7) Å  
 $c = 17.0921$  (13) Å  
 $\alpha = 94.102$  (6)°  
 $\beta = 94.906$  (6)°  
 $\gamma = 100.050$  (6)°  
 $V = 1141.57$  (15) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.954$  Mg m<sup>-3</sup>  
 $D_m$  not measured

## Mo $K\alpha$ radiation

$\lambda = 0.71073$  Å  
 Cell parameters from 48 reflections  
 $\theta = 4.22$ – $17.53$ °  
 $\mu = 2.133$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism  
 $0.63 \times 0.15 \times 0.14$  mm  
 Blue

## Data collection

Siemens P3 diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (North *et al.*, 1968)  
 $T_{min} = 0.349$ ,  $T_{max} = 0.754$   
 7546 measured reflections  
 6115 independent reflections  
 5524 reflections with  
 $I > 2\sigma(I)$

$R_{int} = 0.011$   
 $\theta_{max} = 29.06$ °  
 $h = -1 \rightarrow 9$   
 $k = -13 \rightarrow 13$   
 $l = -23 \rightarrow 23$   
 3 standard reflections  
 every 100 reflections  
 intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.066$   
 $S = 1.034$   
 6115 reflections  
 422 parameters  
 All H-atom parameters refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.7550P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.427$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.372$  e Å<sup>-3</sup>  
 Extinction correction:  
 SHELXL97 (Sheldrick, 1997a)  
 Extinction coefficient:  
 0.0052 (4)  
 Scattering factors from  
 International Tables for  
 Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Cu—O1	1.9639 (12)	Cu'—O1'	1.9847 (12)
Cu—O2	1.9649 (12)	Cu'—O2'	1.9612 (13)
Cu—O3	1.9655 (12)	Cu'—O3'	1.9750 (12)
Cu—O5'	2.3179 (12)	Cu'—O5'	2.3928 (14)
Cu—O6	2.4551 (13)	Cu'—O6''	2.4299 (14)
Cu—N1	2.0090 (14)	Cu'—N1'	1.9845 (14)
S—O5	1.4848 (13)	S'—O5'	1.4615 (14)
S—O6	1.4775 (13)	S'—O6'	1.4740 (13)
S—O7	1.4970 (12)	S'—O7'	1.4818 (14)
S—O8	1.4513 (13)	S'—O8'	1.4891 (13)
O1—Cu—O2	87.10 (5)	O1'—Cu'—O2'	91.41 (5)
O1—Cu—O3	172.96 (6)	O1'—Cu'—O3'	170.87 (6)
O1—Cu—O5'	98.51 (5)	O1'—Cu'—O5'	96.22 (6)
O1—Cu—O6	88.86 (5)	O1'—Cu'—O6''	88.05 (5)
O1—Cu—N1	81.17 (5)	O1'—Cu'—N1'	81.49 (5)
O2—Cu—O3	95.38 (5)	O2'—Cu'—O3'	94.54 (5)
O2—Cu—O5'	89.01 (5)	O2'—Cu'—O5'	88.67 (5)
O2—Cu—O6	84.88 (5)	O2'—Cu'—O6''	92.17 (6)
O2—Cu—N1	167.67 (6)	O2'—Cu'—N1'	170.21 (6)

O3—Cu—O5'	88.13 (5)	O3'—Cu'—O5'	90.84 (6)
O3—Cu—O6	84.81 (5)	O3'—Cu'—O6''	84.81 (5)
O3—Cu—N1	95.84 (6)	O3'—Cu'—N1'	93.36 (5)
O5'—Cu—O6	170.18 (4)	O5'—Cu'—O6''	175.63 (5)
O5'—Cu—N1	96.46 (5)	O5'—Cu'—N1'	85.42 (5)
O6—Cu—N1	91.05 (5)	O6''—Cu'—N1'	94.31 (6)

Symmetry code: (i)  $x - 1, y, z$ .

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N2—H21...O1 <sup>i</sup>	0.78 (3)	2.18 (3)	2.882 (2)	151 (3)
N2—H22...O4 <sup>ii</sup>	0.88 (3)	2.04 (3)	2.883 (2)	161 (2)
O2—H23...O7	0.79 (3)	1.97 (3)	2.755 (2)	172 (3)
O2—H24...O5 <sup>iii</sup>	0.73 (3)	1.94 (3)	2.666 (2)	172 (3)
O3—H31...O4 <sup>iv</sup>	0.76 (3)	1.93 (3)	2.693 (2)	174 (3)
O3—H32...O7 <sup>iii</sup>	0.80 (3)	1.89 (3)	2.687 (2)	175 (2)
O4—H42...O4 <sup>v</sup>	0.77 (3)	1.90 (3)	2.669 (2)	177 (3)
O4—H41...O6 <sup>v</sup>	0.72 (3)	2.04 (3)	2.754 (2)	170 (3)
N2'—H21'...O7 <sup>iii</sup>	0.83 (3)	2.06 (3)	2.887 (2)	170 (3)
N2'—H22'...O8 <sup>v</sup>	0.82 (3)	1.99 (3)	2.763 (2)	158 (3)
O2'—H24'...O7 <sup>v</sup>	0.79 (3)	1.91 (3)	2.695 (2)	172 (3)
O2'—H23'...O8 <sup>vi</sup>	0.78 (3)	1.91 (3)	2.683 (2)	173 (3)
O3'—H31'...O7 <sup>vi</sup>	0.73 (3)	1.95 (3)	2.670 (2)	168 (3)
O3'—H32'...O8 <sup>vi</sup>	0.86 (3)	1.77 (3)	2.627 (2)	178 (3)
O4'—H41'...O1 <sup>v</sup>	0.88 (3)	2.11 (3)	2.903 (2)	149 (3)
O4'—H42'...O6	0.67 (3)	2.09 (4)	2.762 (2)	174 (4)

Symmetry codes: (i)  $-x, -y, 1 - z$ ; (ii)  $x - 1, y - 1, z$ ; (iii)  $1 - x, 1 - y, 1 - z$ ; (iv)  $x - 1, y, z$ ; (v)  $x, 1 + y, z$ ; (vi)  $1 - x, 1 - y, -z$ .

The title structure was solved by direct methods and refined by full-matrix least-squares calculations. All non-H atoms were refined anisotropically. All H atoms were located from a difference synthesis and refined isotropically.

Data collection: Siemens P3 software. Cell refinement: Siemens P3 software. Data reduction: XDISK in SHELXTL/PC (Sheldrick, 1990). Program(s) used to solve structure: SHELS97 (Sheldrick, 1997b). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a). Molecular graphics: XP in SHELXTL/PC. Software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1250). Services for accessing these data are described at the back of the journal.

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## [Bis(diphenylphosphino)methane]dinitratopalladium(II) tris(deuteriochloroform) solvate

NIGAM P. RATH,\* ROBERT A. STOCKLAND JR AND GORDON K. ANDERSON\*

Department of Chemistry, University of Missouri–St Louis, 8001 Natural Bridge Road, St Louis, MO 63121, USA.  
E-mail: nigam\_rath@umsl.edu

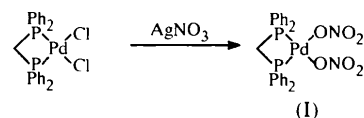
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### Abstract

In the title compound, [methylenebis(diphenylphosphine-P)]bis(nitrate-O)palladium(II) tris(deuteriochloroform) solvate, [Pd(NO<sub>3</sub>)<sub>2</sub>(C<sub>25</sub>H<sub>22</sub>P<sub>2</sub>)]·3CDCl<sub>3</sub> or [Pd(NO<sub>3</sub>)<sub>2</sub>(dppm)]·3CDCl<sub>3</sub> [where dppm is bis(diphenylphosphino)methane], the PdO<sub>2</sub>P<sub>2</sub> unit is planar, but the molecule is significantly distorted from ideal square-planar geometry. Principal dimensions include: Pd—P 2.2240(8) and 2.2345(7), Pd—O 2.103(2) and 2.130(2) Å, P—Pd—P 72.52(3), O—Pd—O 85.23(9), and P—Pd—O 97.55(7) and 104.73(6)°. The CDCl<sub>3</sub> molecules associate with the complex via C—D...O hydrogen bonds.

### Comment

In the course of our investigations of palladium and platinum complexes containing weakly coordinating anionic ligands (Fallis *et al.*, 1991; Oliver & Anderson, 1992), we have investigated the role of nitrate ligands. Reactions of palladium or platinum complexes of the type [MCl<sub>2</sub>(P—P)] with AgNO<sub>3</sub> (2 mole equivalents) produce the corresponding dinitrate derivatives in good yields. The nitrate groups are displaced readily, so these complexes serve as good sources of the M(P—P)<sup>2+</sup> fragment. During our studies, we obtained crystals of the palladium species [Pd(NO<sub>3</sub>)<sub>2</sub>(dppm)] [where dppm is bis(diphenylphosphino)methane], (I), as its tris(deuteriochloroform) solvate.



The structure analysis of [Pd(NO<sub>3</sub>)<sub>2</sub>(dppm)] reveals that the nitrate anions are coordinated through oxygen, and the PdO<sub>2</sub>P<sub>2</sub> unit is perfectly planar (the sum of the angles around palladium is 360.03°), although the molecule is distorted significantly from square-planar geometry (Fig. 1). The P—Pd—P angle of 72.52(3)°