References

Canty, A. J., Skelton, B. W., Traill, P. R. & White, A. H. (1992). *Aust. J. Chem.* **45**, 417–422.
Constable, E. C. (1989). *Adv. Inorg. Chem. Radiochem.* **34**, 1–63.
Jones, P. L., Jeffery, J. C., McCleverty, J. A. & Ward, M. D. (1997). *Polyhedron*, **16**, 1567–1571.
Maekawa, M., Munakata, M., Kitagawa, S. & Nakamura, M. (1991). *Anal. Sci.* **7**, 521–522.
Sheldrick, G. M. (1996). *SADABS. Program for Empirical Absorption Correction.* University of Göttingen, Germany.
Siemens (1995*a*). *SHELXTL.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1995*b*). *SMART* and *SAINT. Area Detector Control and Integration Software.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1998). **C54**, 612-614

Bis[*N*-methylphenazinium(1+)] Tetranitropalladate(II)

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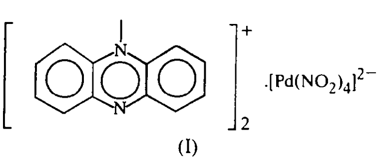
(*Received 11 November 1996; accepted 13 May 1997*)

Abstract

In the crystal structure of the title compound, $(C_{13}H_{11}N_2)_2[Pd(NO_2)_4]$, the anions are located between two very slightly bent cations, but the least-squares planes of the anions are nearly perpendicular to those of the cations. These triads are stacked along two different directions, [101] and [10$\bar{1}$], with a mean interplanar distance of 3.325 (4) Å between adjacent triads.

Comment

The title compound, (I), forms a mixed stack according to the terminology for structures of charge-transfer complexes (Soos & Keller, 1977). The donor–acceptor sequence is DAD–DAD, in which the planar ions are greatly overlapped either within a triad or between adjacent triads. The packing arrangement in the unit cell of compound (I) is shown in Fig. 1.



(I)

The triad stacks are arranged alternately along the [101] and [10$\bar{1}$] directions. Bond distances and angles of both ions are normal compared with those of corresponding NMP compounds (NMP is *N*-methyl-

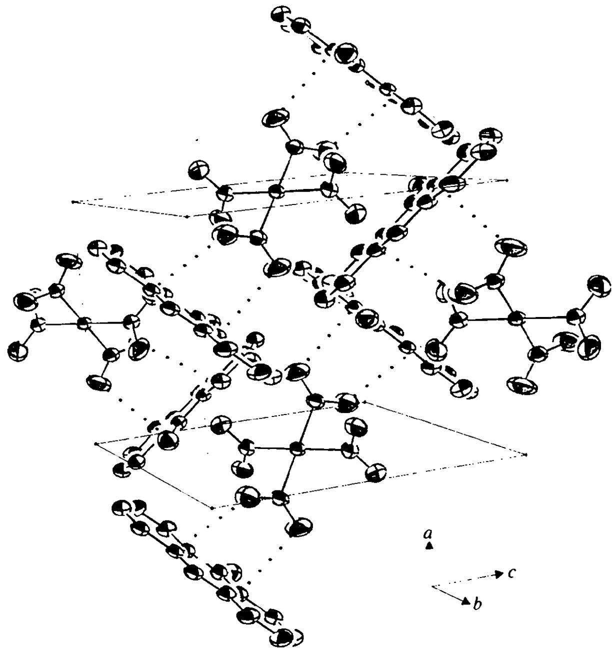


Fig. 1. View of the packing in the unit cell. Dotted lines represent O···C contacts of less than 3.10 Å. Displacement ellipsoids are drawn at the 50% probability level.

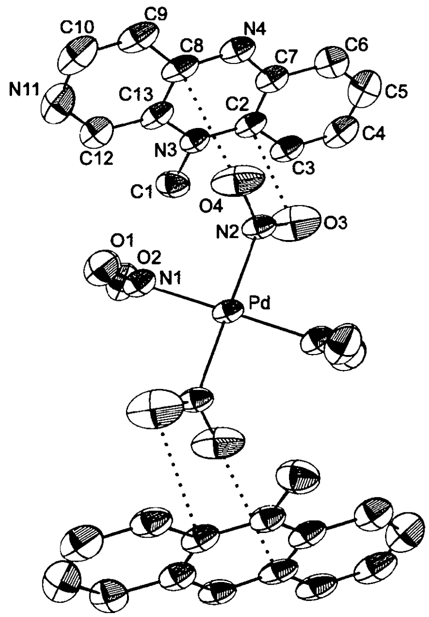


Fig. 2. The formula unit of the title compound showing O···C contacts of less than 3.10 Å as dotted lines and the atomic numbering. H atoms have been omitted. Displacement ellipsoids are drawn at the 50% probability level.

phenazinium), Ni(mnt)$_2^{2-}$ (mnt is dicyanoethenedithiolate) (Endres *et al.*, 1979), Cu(mnt)$_2^{2-}$ (Kuppusamy *et al.*, 1984), V(dmit)$_3^{2-}$ (dmit is 2-thioxo-1,3-dithio-4,5-dithiolate) (Matsubayashi *et al.*, 1988) and $K_2Pd(NO_2)_4$ (Blake *et al.*, 1992). The two shortest contacts [O3···C2 3.026 (4) and O4···C8 3.053 (4) Å] between the different ions in a triad are shown in Fig. 2.

The Pd and four N atoms of the anion are coplanar (plane *A*). The non-H atoms of the cations are planar, with a maximum deviation from the least-squares plane (plane *B*) of 0.042 (7) Å, although least-squares planes through each half of the cation, with C1, N3 and N4 in common, make a dihedral angle of 1.75 (8)°. The unique NO_2 ligand planes make dihedral angles of 82.8 (5) with each other, and 79.3 (1) (N1) and 53.2 (2)° (N2) with plane *A*, and 83.7 (2) (N1) and 90.6 (2)° (N2) with plane *B*. The dihedral angle between planes *A* and *B* is 141.49 (8)°. The mean interplanar distance between adjacent NMP^+ cations is 3.325 (4) Å.

Experimental

Preparation of the starting compounds, *N*-methylphenazinium methylsulfate ($NMP.CH_3SO_4$; Vivian, 1956) and $K_2Pd(NO_2)_4.H_2O$ (James & Nolan, 1973), were carried out according to literature procedures. The title compound was prepared by addition of a solution of *N*-methylphenazinium methylsulfate (0.159 g, 0.528 mmol) to a solution of $K_2Pd(NO_2)_4.H_2O$ (0.1 g, 0.259 mmol), both in 10 ml 1:1 aqueous ethanol. The powdery product was dissolved in a warm mixture of acetonitrile, ethanol and water. The filtrate was allowed to cool to room temperature and was kept for two weeks in a refrigerator, resulting in brown tabular crystals.

Crystal data

$(C_{13}H_{11}N_2)_2[Pd(NO_2)_4]$
$M_r = 680.92$
Monoclinic
$P2_1/n$
$a = 10.898\ (4)$ Å
$b = 8.807\ (2)$ Å
$c = 14.189\ (2)$ Å
$\beta = 94.14\ (2)°$
$V = 1358.3\ (6)$ Å^3
$Z = 2$
$D_x = 1.665$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.91–13.46°$
$\mu = 0.749$ mm^{-1}
$T = 293\ (2)$ K
Tablet
0.30 × 0.24 × 0.06 mm
Brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration (B. A. Frenz & Associates Inc, 1983)
$T_{min} = 0.886$, $T_{max} = 0.984$
2383 measured reflections
2383 independent reflections

1683 reflections with $I > 2\sigma(I)$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = -16 \rightarrow 16$
3 standard reflections frequency: 60 min intensity decay: −0.046%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.062$
$S = 1.102$
2383 reflections
229 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.274$ e Å^{-3}
$\Delta\rho_{min} = -0.218$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Pd—N1	2.021 (2)	C4—C5	1.407 (5)
Pd—N2	2.044 (3)	C5—C6	1.340 (6)
N1—O1	1.221 (4)	C6—C7	1.413 (5)
N1—O2	1.224 (4)	C7—N4	1.331 (4)
N2—O3	1.195 (4)	N4—C8	1.337 (4)
N2—O4	1.208 (3)	C8—C13	1.424 (4)
C1—N3	1.484 (4)	C8—C9	1.426 (4)
N3—C2	1.360 (4)	C9—C10	1.342 (5)
N3—C13	1.371 (4)	C10—C11	1.401 (6)
C2—C3	1.413 (5)	C11—C12	1.350 (5)
C2—C7	1.431 (4)	C12—C13	1.405 (5)
C3—C4	1.356 (5)		
N1—Pd—N2	89.80 (11)	C5—C6—C7	121.1 (4)
O1—N1—O2	119.1 (2)	N4—C7—C6	118.7 (3)
O1—N1—Pd	121.8 (2)	N4—C7—C2	122.7 (3)
O2—N1—Pd	119.0 (2)	C6—C7—C2	118.6 (3)
O3—N2—O4	121.2 (3)	C7—N4—C8	117.9 (3)
O3—N2—Pd	120.8 (2)	N4—C8—C13	123.0 (3)
O4—N2—Pd	118.0 (2)	N4—C8—C9	118.5 (3)
C2—N3—C13	121.1 (3)	C13—C8—C9	118.4 (3)
C2—N3—C1	119.8 (3)	C10—C9—C8	119.8 (4)
C13—N3—C1	119.1 (3)	C9—C10—C11	120.7 (4)
N3—C2—C3	123.0 (3)	C12—C11—C10	122.4 (4)
N3—C2—C7	117.8 (3)	C11—C12—C13	118.5 (4)
C3—C2—C7	119.1 (3)	N3—C13—C12	122.4 (3)
C4—C3—C2	119.2 (3)	N3—C13—C8	117.4 (3)
C3—C4—C5	122.0 (4)	C12—C13—C8	120.2 (3)
C6—C5—C4	119.9 (4)		

The structure was solved by direct methods and refined using full-matrix least-squares techniques, with anisotropic displacement parameters for all non-H atoms and fixed isotropic parameters for H atoms.

Data collection: *SDP-Plus* (B. A. Frenz & Associates Inc, 1983). Cell refinement: *SDP-Plus*. Data reduction: *SDP-Plus*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

This work is supported by the Research Fund administered by the Kookmin University.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1144). Services for accessing these data are described at the back of the journal.

References

B. A. Frenz & Associates Inc. (1983). *SDP-Plus Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Blake, A. J., Gould, R. O., Johnson, B. F. G. & Parisini, E. (1992). *Acta Cryst.* C**48**, 1749–1751.

Endres, H., Keller, H. J., Moroni, W. & Noethe, D. (1979). *Acta Cryst.* B**35**, 353–357.
James, D. W. & Nolan, M. J. (1973). *Aust. J. Chem.* **26**, 1433–1441.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kuppusamy, P., Ramakrishna, B. L. & Manoharan, P. T. (1984). *Inorg. Chem.* **23**, 3886–3892.
Matsubayashi, G. E., Akiba, K. & Tanaka, T. (1988). *Inorg. Chem.* **27**, 4744–4749.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Soos, Z. G. & Keller, H. J. (1977). In *Chemistry and Physics of One-Dimensional Metals*, edited by H. J. Keller. NATO ASI Series No. 25. New York: NATO.
Vivian, D. L. (1956). *J. Org. Chem.* **21**, 822–824.

Acta Cryst. (1998). C**54**, 614–616

Bis(1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol-κ^3N)copper(II) Dichloride Tetrahydrate†

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(*Received 28 July 1997; accepted 28 November 1997*)

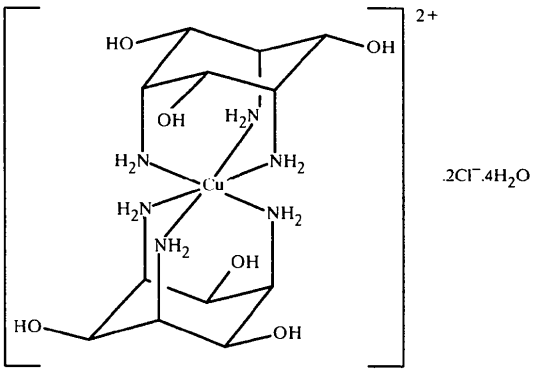
Abstract

Crystals of the title compound, $[Cu(C_6H_{15}N_3O_3)_2]Cl_2.4H_2O$, are isotypic with those of the analogous Cu^{II} bromide and the corresponding Ni^{II} and Zn^{II} bromides [Hegetschweiler *et al.* (1992). *Inorg. Chem.* **31**, 2341–2346]. The coordination polyhedron of the Cu atom shows a strong Jahn–Teller distortion from octahedral symmetry. The location of all H-atom positions gives a more detailed understanding of the three-dimensional hydrogen-bonding framework in (I) and its related compounds.

Comment

A remarkable property of the ligand 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol (taci) is its rigidity. Its large number of OH and NH_2 groups gives rise to a variety of different coordination modes and to extended inter- and intramolecular hydrogen bonding. In particular, it has been found for some metal complexes of taci that these substituents are arranged so as to form characteristic cavities that accommodate the counteranions (Hegetschweiler *et al.*, 1992).

Crystals of the title compound, (I), are composed of the cationic 2:1 complex of 1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol with a copper(II) ion (Fig. 1), two chloride anions and four water molecules, all connected by hydrogen bonds. The coordination polyhedron of the Cu atom (Cu1, site symmetry 2/*m*) shows a strong Jahn–Teller distortion from octahedral symmetry. The N atoms of four crystallographically equivalent NH_2 groups (N2) form a square [N—Cu—N 88.91 (5)–91.09 (5)°], with short N—Cu distances [2.0712 (9) Å]. Two more weakly bonded NH_2 groups, with their N atoms on the mirror plane 0*yz* [N4—Cu1 2.3472 (15) Å], occupy the axial positions, additionally displaced by 4.54 (4)° from the normal of the equatorial square plane, reducing the point-group symmetry to $C_{2h}(2/m)$ (Fig. 1).



(I)

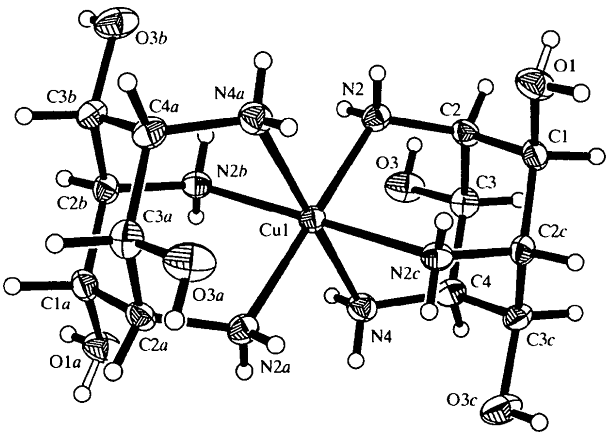


Fig. 1. Diagram of the bis(1,3,5-triamino-1,3,5-trideoxy-*cis*-inositol)-copper(II) cation. Displacement ellipsoids are drawn at the 50% probability level. The two H atoms at O1 represent the two possible positions of this disordered H atom. Symmetry codes: (*a*) −*x*, 1 − *y*, −*z*; (*b*) *x*, 1 − *y*, −*z*; (*c*) −*x*, *y*, *z*.

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† Dedicated to Professor C. G. Kreiter on the occasion of his 60th birthday.

Acta Crystallographica Section C
ISSN 0108-2701 © 1998