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## Dihydrogen ( $\pm$ )-*trans*-1,2-Cyclohexanediaminetetraacetic Acid\* Tetrachloropalladate(II) and Tetrachloroplatinate(II) Pentahydrates, [C<sub>14</sub>H<sub>24</sub>N<sub>2</sub>O<sub>8</sub>][PdCl<sub>4</sub>].5H<sub>2</sub>O and [C<sub>14</sub>H<sub>24</sub>N<sub>2</sub>O<sub>8</sub>][PtCl<sub>4</sub>].5H<sub>2</sub>O

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**Abstract.** [PdCl<sub>4</sub>]<sup>2-</sup> salt:  $M_r = 686.7$ ,  $P2_1/c$ ,  $a = 12.457$  (3),  $b = 11.427$  (3),  $c = 18.833$  (4) Å,  $\beta = 106.11$  (2)°,  $V = 2575.5$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.77$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 1.18$  mm<sup>-1</sup>,  $F(000) = 1400$ ,  $T = 292$  (2) K,  $R = 0.040$  for 4672 observed unique reflections. Isomorphous [PtCl<sub>4</sub>]<sup>2-</sup> salt:  $M_r = 775.4$ ,  $P2_1/c$ ,  $a = 12.499$  (3),  $b = 11.371$  (3),  $c = 18.885$  (5) Å,  $\beta = 106.09$  (2)°,  $V = 2579.0$  (11) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.00$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 5.98$  mm<sup>-1</sup>,  $F(000) = 1528$ ,  $T = 293$  (2) K,  $R = 0.048$  for 5223 observed unique reflections. The cations are *trans*-1,2-cyclohexanediaminetetraacetic acid molecules with two additional protons residing on amine nitrogen atoms. The crystals contain alternating layers of ions and water molecules parallel to the *ab* plane, with an extensive hydrogen-bonding network. The conformation of the molecule is close to that required for a six- or seven-coordinate ligand attached to a metal atom, as is usual for coordinated ethylenediaminetetraacetate-like ligands.

**Introduction.** The stereochemistry of complexes of ethylenediaminetetraacetic acid and related compounds has been well investigated; however, to date there have been few structures reported for uncoordinated ligands and none on a diprotonated derivative. Information on the uncoordinated *trans*-1,2-cyclohexanediamine

derivative, which exhibits marked stereoselectivity upon coordination, would be of particular interest. Here we report the structure of diprotonated racemic *trans*-1,2-cyclohexanediaminetetraacetic acid as the pentahydrated tetrachloropalladate(II) and tetrachloroplatinate(II) salts.

**Experimental.** Compounds prepared by mixing a solution of PdCl<sub>2</sub> or PtCl<sub>2</sub> in 2 M HCl with aqueous solution of *trans*-1,2-cyclohexanediaminetetraacetic acid. Crystals grown from hydrochloric acid, rectangular prisms, 0.15 × 0.23 × 0.25 mm for [PdCl<sub>4</sub>]<sup>2-</sup> salt, 0.21 × 0.35 × 0.46 mm for [PtCl<sub>4</sub>]<sup>2-</sup> salt.  $D_m$  not determined. Syntex P3/F diffractometer, graphite monochromator. Lattice parameters from 25 reflections for each structure ( $12 < 2\theta < 25^\circ$ ). Empirical absorption corrections based on  $\psi$  scans; min. and max. transmission coefficient 0.546 and 0.604 ([PdCl<sub>4</sub>]<sup>2-</sup> salt), 0.453 and 0.942 ([PtCl<sub>4</sub>]<sup>2-</sup> salt).  $\theta$ - $2\theta$  scans,  $\sin\theta/\lambda = 0.01$  to  $0.70$  Å<sup>-1</sup> ( $1 \leq 2\theta \leq 60^\circ$ ;  $\pm h, -k, -l$  for [PdCl<sub>4</sub>]<sup>2-</sup> compound;  $\pm h, +k, -l$  for [PtCl<sub>4</sub>]<sup>2-</sup> compound). Three standard reflections (Pd: 800, 080, 008; Pt: 505, 060, 008) every 141 reflections (5% variation). For Pd compound, 8461 reflections measured, 7491 unique, 2819 unobserved [ $I \leq 5.0\sigma(I)$ ]. For Pt compound, 8191 reflections measured, 7506 unique, 2283 unobserved [ $I \leq 5.0\sigma(I)$ ]. Space group for both structures, as determined from systematic absences ( $h0l, h+1 = 2n+1$  and  $0k0, k = 2n+1$ ), unambiguous. All computer programs from SHELXTL (Sheldrick, 1981) and

\* ( $\pm$ )-*trans*-N,N'-(1,2-Cyclohexylene)bis[(carboxymethyl)-ammonium].

Table 1. Atomic coordinates and equivalent isotropic thermal parameters for non-H atoms of dihydrogen ( $\pm$ )-*trans*-1,2-cyclohexanediaminetetraacetic acid tetrachloropalladate(II) and tetrachloroplatinate(II) pentahydrate

Coordinates in fractions of the unit-cell edge. Equivalent isotropic thermal parameters in  $\text{\AA}^2$  are calculated as  $U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$  with the  $U_{ii}$ 's in an orthogonal coordinate system (Willis & Pryor, 1975).

Pd/Pt	[C <sub>14</sub> H <sub>24</sub> N <sub>2</sub> O <sub>8</sub> ][PdCl <sub>4</sub> ].5H <sub>2</sub> O			$U_{\text{eq}}$	[C <sub>14</sub> H <sub>24</sub> N <sub>2</sub> O <sub>8</sub> ][PtCl <sub>4</sub> ].5H <sub>2</sub> O			$U_{\text{eq}}$
	<i>x</i>	<i>y</i>	<i>z</i>		<i>x</i>	<i>y</i>	<i>z</i>	
Pd/Pt	0.00615 (2)	0.75937 (2)	0.77298 (2)	0.0295 (1)	0.00592 (2)	0.75977 (2)	0.77279 (2)	0.0273 (1)
Cl(1)	-0.10751 (8)	0.62928 (9)	0.69323 (6)	0.0496 (4)	-0.1071 (2)	0.6283 (2)	0.6935 (1)	0.049 (1)
Cl(2)	-0.01570 (9)	0.64659 (10)	0.86971 (6)	0.0569 (4)	-0.0177 (2)	0.6489 (2)	0.8698 (1)	0.057 (1)
Cl(3)	0.10969 (9)	0.89857 (9)	0.85122 (6)	0.0518 (4)	0.1111 (2)	0.8985 (2)	0.8506 (1)	0.051 (1)
Cl(4)	0.03543 (9)	0.86392 (9)	0.67523 (6)	0.0459 (4)	0.0350 (2)	0.8642 (2)	0.6754 (1)	0.047 (1)
O(1)	0.1316 (2)	0.6430 (2)	0.3798 (1)	0.031 (1)	0.1321 (4)	0.6417 (4)	0.3801 (3)	0.033 (2)
O(2)	0.3015 (2)	0.5880 (2)	0.3735 (1)	0.041 (1)	0.3002 (4)	0.5858 (5)	0.3738 (3)	0.039 (2)
O(3)	0.2110 (2)	0.9145 (2)	0.4094 (1)	0.033 (1)	0.2109 (4)	0.9136 (4)	0.4093 (3)	0.032 (2)
O(4)	0.2661 (2)	1.0760 (2)	0.3610 (1)	0.037 (1)	0.2678 (4)	1.0757 (4)	0.3624 (3)	0.036 (2)
O(5)	-0.0241 (2)	0.8410 (2)	0.3752 (1)	0.031 (1)	-0.0225 (4)	0.8411 (4)	0.3755 (3)	0.031 (2)
O(6)	-0.1996 (2)	0.8983 (2)	0.3637 (1)	0.040 (1)	-0.1976 (4)	0.8998 (5)	0.3651 (3)	0.038 (2)
O(7)	-0.0841 (2)	0.5627 (2)	0.4082 (1)	0.039 (1)	-0.0840 (5)	0.5623 (5)	0.4078 (3)	0.040 (2)
O(8)	-0.1739 (2)	0.4008 (2)	0.3563 (1)	0.040 (1)	-0.1747 (4)	0.3994 (4)	0.3578 (3)	0.038 (2)
N(1)	0.1158 (2)	0.8167 (2)	0.2788 (1)	0.021 (1)	0.1159 (4)	0.8161 (4)	0.2787 (3)	0.020 (1)
N(2)	-0.0891 (2)	0.6616 (2)	0.2775 (1)	0.023 (1)	-0.0883 (4)	0.6610 (4)	0.2776 (3)	0.021 (2)
C(1)	0.0147 (3)	0.7977 (3)	0.2113 (2)	0.024 (1)	0.0161 (5)	0.7982 (5)	0.2120 (4)	0.022 (2)
C(2)	-0.0402 (3)	0.6771 (3)	0.2117 (2)	0.024 (1)	-0.0393 (5)	0.6765 (5)	0.2126 (4)	0.024 (2)
C(3)	-0.1308 (3)	0.6533 (3)	0.1394 (2)	0.035 (1)	-0.1307 (6)	0.6522 (7)	0.1394 (4)	0.031 (2)
C(4)	-0.0873 (3)	0.6714 (3)	0.0725 (2)	0.041 (1)	-0.0867 (7)	0.6700 (7)	0.0728 (4)	0.040 (3)
C(5)	-0.0451 (3)	0.7944 (3)	0.0716 (2)	0.040 (1)	-0.0428 (7)	0.7958 (7)	0.0721 (4)	0.040 (3)
C(6)	0.0501 (3)	0.8181 (3)	0.1405 (2)	0.033 (1)	0.0515 (6)	0.8175 (7)	0.1416 (4)	0.031 (2)
C(1A)	0.2087 (3)	0.6483 (3)	0.3529 (2)	0.026 (1)	0.2095 (5)	0.6473 (5)	0.3525 (4)	0.026 (2)
C(1B)	0.2087 (3)	0.7287 (3)	0.2896 (2)	0.025 (1)	0.2081 (5)	0.7269 (5)	0.2891 (4)	0.025 (2)
C(1C)	0.2163 (3)	0.9739 (3)	0.3576 (2)	0.026 (1)	0.2159 (5)	0.9731 (6)	0.3575 (4)	0.026 (2)
C(1D)	0.1609 (3)	0.9386 (3)	0.2789 (2)	0.026 (1)	0.1624 (6)	0.9391 (5)	0.2791 (4)	0.024 (2)
C(2A)	-0.1235 (3)	0.8355 (3)	0.3463 (2)	0.026 (1)	-0.1223 (5)	0.8354 (5)	0.3468 (4)	0.025 (2)
C(2B)	-0.1742 (2)	0.7501 (3)	0.2854 (2)	0.028 (1)	-0.1737 (5)	0.7518 (5)	0.2854 (4)	0.029 (2)
C(2C)	-0.1281 (3)	0.5045 (3)	0.3555 (2)	0.028 (1)	-0.1290 (5)	0.5042 (6)	0.3554 (4)	0.028 (2)
C(2D)	-0.1365 (3)	0.5402 (3)	0.2775 (2)	0.028 (1)	-0.1368 (6)	0.5388 (5)	0.2774 (4)	0.028 (2)
O(9)	-0.1157 (3)	0.7106 (3)	0.5257 (2)	0.084 (2)	-0.1148 (7)	0.7107 (9)	0.5260 (5)	0.081 (3)
O(10)	0.1189 (3)	0.7114 (3)	0.5224 (2)	0.055 (1)	0.1170 (6)	0.7098 (6)	0.5204 (3)	0.051 (2)
O(11)	0.1138 (2)	0.9652 (2)	0.5253 (1)	0.047 (1)	0.1136 (5)	0.9629 (5)	0.5241 (3)	0.044 (2)
O(12)	0.1922 (3)	0.6624 (4)	1.0095 (2)	0.086 (2)	0.1914 (6)	0.6619 (8)	0.0081 (4)	0.079 (3)
O(13)	0.1895 (5)	0.9416 (4)	1.0250 (2)	0.088 (2)	0.1893 (8)	0.9415 (7)	0.0232 (4)	0.088 (4)

scattering-factor data from *International Tables for X-ray Crystallography* (1974). Structures solved by heavy-atom techniques with metal-atom positions determined and positions of other non-H atoms obtained from subsequent Fourier maps. All H atoms for PdCl<sub>4</sub><sup>2-</sup> salt located in difference maps; no H atoms for PtCl<sub>4</sub><sup>2-</sup> salt could be unambiguously located. For both structures, H atoms attached to carbon atoms were calculated in idealized positions, their positions being fixed using a riding model, with  $U_{\text{iso}}$  values set at 1.2 times last value of  $U_{\text{eq}}$  for the atom to which they were attached. For PdCl<sub>4</sub><sup>2-</sup> structure, positions of amine, carboxylic and water H atoms as initially determined in a difference map were allowed to vary and  $U_{\text{iso}}$  values also allowed to vary except for water H atoms, which were fixed at 1.2 times  $U_{\text{eq}}$  of the O atom. For PtCl<sub>4</sub><sup>2-</sup> structure, amine H atoms were fixed in idealized positions with  $U_{\text{iso}}$  values fixed at 1.2 times  $U_{\text{eq}}$  values of attached N atoms. Carboxylic and water H atoms not included in refinement. Refinement on  $F$  converged to  $R = 0.040$ ,  $wR = 0.035$  [ $w^{-1} = \sigma(F)^2 + |g|F_{\text{p}}^2$ ,  $g = 0.00011$ ],  $S = 1.34$  for 4672 observations and 361 refined variables (PdCl<sub>4</sub><sup>2-</sup> salt) and to  $R = 0.048$ ,  $wR = 0.050$  [ $g = 0.0017$ ],  $S = 1.20$  for 5223 observations and 307 refined variables (PtCl<sub>4</sub><sup>2-</sup> salt). Max. and min. peak heights in final difference map 0.58 and

$-0.66 \text{ e \AA}^{-3}$  (PdCl<sub>4</sub><sup>2-</sup>) and  $2.44$  [near Pt] and  $-4.01 \text{ e \AA}^{-3}$  (PtCl<sub>4</sub><sup>2-</sup>). Max. and mean  $\Delta/\sigma$  during final cycle of refinement 0.13 and 0.03 (PdCl<sub>4</sub><sup>2-</sup>) and 0.37 and 0.02 (PtCl<sub>4</sub><sup>2-</sup>).

**Discussion.** The final fractional coordinates and  $U_{\text{eq}}$  values for all non-H atoms are given in Table 1.\*

The isostructural crystals contain dipositive *trans*-1,2-cyclohexanediaminetetraacetic acid molecular cations (protonated at all four carboxylic acid positions and at the two nitrogen atoms) and PdCl<sub>4</sub><sup>2-</sup> or PtCl<sub>4</sub><sup>2-</sup> anions. Though coordination of Pd<sup>II</sup> by ethylenediaminetetraacetic acid is known (Robinson & Kennard, 1970), no metal-ion coordination by the *trans*-1,2-cyclohexanediaminetetraacetic acid molecules occurs in either of these two structures. The isostructural crystals contain alternating layers of ions and water molecules parallel to the *ab* plane (Fig. 1). All molecules and ions are located at general positions.

\* Lists of structure factors, anisotropic thermal parameters and H-atom positional and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39935 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

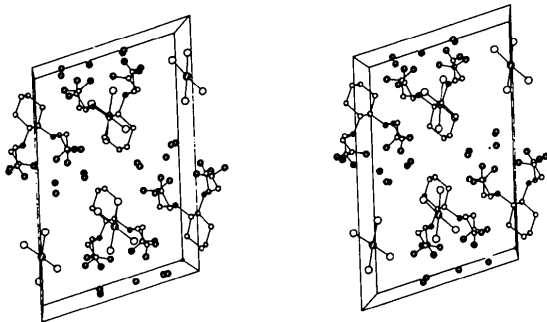


Fig. 1. Stereoview of the crystal packing for [C<sub>14</sub>H<sub>24</sub>N<sub>2</sub>O<sub>8</sub>][PdCl<sub>4</sub>].5H<sub>2</sub>O as viewed down the *b* axis. The origin lies at the back bottom right corner with the *a* axis pointing to the left, the *c* axis pointing up, and the *b* axis projecting toward the viewer. The atoms are pictured as spheres of arbitrary size (O atoms, small, cross hatched; Cl atoms, large, cross hatched; C atoms, small, open; Pd atoms, large, open). H atoms have been omitted for clarity.

The bond lengths and selected bond and torsion angles of both the cations and anions of the two structures are summarized in Table 2. The atom nomenclature is the same for both salts and is shown in Fig. 2.

With the exception of O(5), which is not involved in hydrogen bonding, each oxygen atom in the cation is associated with exactly one water molecule. The hydroxyl groups of the organic ion act as hydrogen-atom donors and the carbonyl groups act as hydrogen-atom acceptors in these associations. Owing to hydrogen-bonding interactions, water-molecule oxygen atoms O(9), O(10) and O(11) are in a pseudo-tetrahedral environment of four hydrogen atoms, while O(12) and O(13) are coordinated to only three hydrogen atoms. All chlorine atoms except Cl(1) are in close contact with water molecules with Cl...HOH distances of 2.42 (5) to 2.56 (4) Å in the PdCl<sub>4</sub><sup>2-</sup> salt, whose H atoms could be located unambiguously.

Attached to each nitrogen atom are two prochiral acetate groups, one of which may be designated *pro-R* and the other *pro-S* (Hirschmann & Hanson, 1971). Owing to the presence of chiral centers on the cyclohexane ring, the two acetate groups on each nitrogen atom are intrinsically nonequivalent irrespective of any crystal-symmetry considerations. In the present case, there are significant differences between bond angles in the two types of acetate groups (Table 2). Thus, in enantiomers containing cyclohexane rings with two carbon atoms of *S* chirality (pictured in Fig. 2), the nitrogen–aliphatic acetate carbon–carboxyl carbon and the cyclohexane carbon–nitrogen–aliphatic acetate carbon angles are larger for the *pro-R* acetate groups than for the *pro-S* groups (the former angles by ~1.5° and the latter by ~6°).

Table 2. Bond lengths (Å) and selected bond angles and torsion angles (°) involving non-H atoms

	PdCl <sub>4</sub> <sup>2-</sup> salt	PtCl <sub>4</sub> <sup>2-</sup> salt	PdCl <sub>4</sub> <sup>2-</sup> salt	PtCl <sub>4</sub> <sup>2-</sup> salt	
M—Cl(1)	2.300 (1)	2.302 (2)	O(1)—C(1A)—O(2)	126.2 (3)	124.4 (7)
M—Cl(2)	2.308 (1)	2.309 (3)	O(3)—C(1C)—O(4)	126.2 (3)	124.9 (6)
M—Cl(3)	2.304 (1)	2.303 (2)	O(5)—C(2A)—O(6)	125.9 (3)	125.0 (6)
M—Cl(4)	2.307 (1)	2.302 (3)	O(7)—C(2C)—O(8)	126.4 (3)	125.3 (7)
C(1A)—O(1)	1.205 (5)	1.221 (10)	C(1A)—C(1B)—N(1)	110.3 (3)	110.5 (6)
C(1C)—O(3)	1.205 (4)	1.205 (9)	C(1C)—C(1D)—N(1)	108.6 (3)	108.0 (5)
C(2A)—O(5)	1.208 (4)	1.217 (8)	C(2A)—C(2B)—N(2)	110.7 (2)	110.1 (5)
C(2C)—O(7)	1.194 (4)	1.192 (8)	C(2C)—C(2D)—N(2)	109.3 (2)	108.7 (5)
C(1A)—O(2)	1.308 (4)	1.298 (8)	C(1B)—N(1)—C(1)	116.2 (2)	116.1 (5)
C(1C)—O(4)	1.314 (4)	1.325 (8)	C(1D)—N(1)—C(1)	110.7 (2)	110.7 (4)
C(2A)—O(6)	1.302 (4)	1.313 (9)	C(2B)—N(2)—C(2)	116.8 (2)	116.7 (5)
C(2C)—O(8)	1.316 (4)	1.328 (8)	C(2D)—N(2)—C(2)	110.5 (2)	110.7 (5)
C(1A)—C(1B)	1.506 (5)	1.496 (10)	N(1)—C(1)—C(2)	112.7 (2)	112.3 (5)
C(1C)—C(1D)	1.506 (4)	1.496 (9)	N(1)—C(1)—C(6)	109.3 (3)	109.4 (5)
C(2A)—C(2B)	1.506 (4)	1.499 (9)	N(2)—C(2)—C(1)	112.6 (3)	112.9 (5)
C(2C)—C(2D)	1.501 (5)	1.502 (11)	N(2)—C(2)—C(3)	109.7 (3)	109.6 (5)
N(1)—C(1B)	1.503 (4)	1.507 (8)	C(1)—C(2)—C(3)	111.9 (3)	111.8 (5)
N(1)—C(1D)	1.502 (4)	1.514 (8)	C(2)—C(3)—C(4)	111.6 (3)	111.5 (6)
N(2)—C(2B)	1.502 (4)	1.521 (9)	C(3)—C(4)—C(5)	110.1 (3)	110.2 (7)
N(2)—C(2D)	1.508 (4)	1.516 (8)	C(4)—C(5)—C(6)	110.5 (3)	109.7 (6)
C(1)—N(1)	1.536 (3)	1.522 (7)	C(5)—C(6)—C(1)	111.8 (3)	112.7 (6)
C(2)—N(2)	1.534 (5)	1.526 (10)	C(6)—C(1)—C(2)	112.0 (3)	111.9 (6)
C(1)—C(2)	1.539 (4)	1.548 (9)			
C(2)—C(3)	1.533 (4)	1.555 (9)			
C(3)—C(4)	1.518 (6)	1.518 (12)			
C(4)—C(5)	1.503 (5)	1.533 (12)			
C(5)—C(6)	1.520 (4)	1.523 (9)			
C(6)—C(1)	1.534 (5)	1.528 (11)			
			PdCl <sub>4</sub> <sup>2-</sup> salt	PtCl <sub>4</sub> <sup>2-</sup> salt	
O(1)—C(1A)—C(1B)—N(1)			13.4 (4)	14.2 (8)	
O(3)—C(1C)—C(1D)—N(1)			-7.3 (4)	-6.3 (9)	
O(5)—C(2A)—C(2B)—N(2)			11.1 (5)	12.6 (10)	
O(7)—C(2C)—C(2D)—N(2)			-4.1 (5)	-4.9 (9)	
C(1A)—C(1B)—N(1)—C(1)			-106.3 (3)	-107.4 (6)	
C(1C)—C(1D)—N(1)—C(1)			152.4 (3)	151.8 (6)	
C(2A)—C(2B)—N(2)—C(2)			-104.2 (3)	-104.7 (6)	
C(2C)—C(2D)—N(2)—C(2)			150.7 (2)	150.9 (5)	
C(1B)—N(1)—C(1)—C(6)			-67.6 (3)	-67.0 (7)	
C(1D)—N(1)—C(1)—C(6)			59.2 (3)	59.4 (7)	
C(2B)—N(2)—C(2)—C(3)			-68.3 (3)	-68.7 (6)	
C(2D)—N(2)—C(2)—C(3)			57.6 (3)	57.1 (6)	
N(1)—C(1)—C(2)—N(2)			63.4 (3)	63.7 (7)	
C(1)—C(2)—C(3)—C(4)			52.5 (4)	52.4 (8)	
C(2)—C(3)—C(4)—C(5)			-58.5 (4)	-58.1 (8)	
C(3)—C(4)—C(5)—C(6)			60.6 (4)	60.3 (9)	
C(4)—C(5)—C(6)—C(1)			-57.5 (4)	-57.8 (9)	
C(5)—C(6)—C(1)—C(2)			51.5 (4)	52.4 (8)	
C(6)—C(1)—C(2)—C(3)			-48.8 (4)	-48.8 (7)	

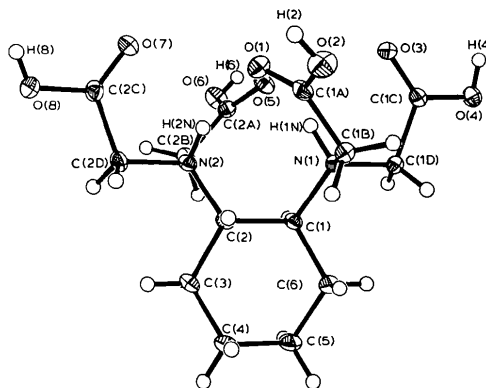


Fig. 2. View of the cation in the PdCl<sub>4</sub><sup>2-</sup> salt. The thermal ellipsoids (except for the H atoms, which are depicted as arbitrarily sized spheres) are shown at the 50% probability level.

The conformation of the *trans*-1,2-cyclohexanediaminetetraacetic acid cation is very similar to that required to give a six-coordinate octahedral or a seven-coordinate pentagonal bipyramidal coordination with a metal ion. Such geometries are very common with hexadentate ethylenediaminetetraacetate-like ligands, seven coordination being found for larger metal ions (with a monodentate ligand, usually a water molecule, occupying the seventh site) and six coordination being found for smaller metal ions (Cohen & Hoard, 1966). The two nitrogen atoms and two oxygen atoms [O(7) and O(3)] from the *pro-S* acetate groups (for the *S,S* enantiomer) are positioned to function as equatorial ligands while two oxygen atoms [O(1) and O(5)] from the *pro-R* acetate group would function as axial ligands. The overall absolute configuration for this enantiomer would be  $\Delta\Delta\Delta$  (IUPAC Commission on the Nomenclature of Inorganic Chemistry, 1970) if complexation were to occur without a large change in conformation. Others have predicted stereospecific formation of octahedral  $\Delta\Delta\Delta$  diastereomers from hexadentate (*S,S*)-1,2-cyclohexanediaminetetraacetate (Reinbold & Pearson, 1970; Erickson, Young, Ho, Watkins, Terrill & Reilly, 1971). This diastereomer is found for the six-coordinate  $\text{Mn}^{\text{III}}$  complex (Rettig & Trotter, 1973). On the other hand, the other diastereomeric pair, *R,R*( $\Delta\Delta\Delta$ ) and *S,S*( $\Delta\Delta\Delta$ ), is found for the pentagonal bipyramidal complex of  $\text{Fe}^{\text{III}}$  with an equatorial water molecule as the seventh ligand (Cohen & Hoard, 1966). Furthermore, an  $\text{Rh}^{\text{III}}$  complex, where the *trans*-1,2-cyclohexanediaminetetraacetate ligand is only tetradentate, exhibits a geometry where the ligand

is wrapping in a direction to give the unexpected isomer (Filippova, Polynova, Il'inskii, Porai-Koshits & Ezerskaya, 1981). Thus the stereoselectivity does not seem to be as great as expected.

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### Structure of *trans*-Diaquabis(oxalato)vanadate(III) Complexes: $A[\text{V}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot x\text{H}_2\text{O}$ , $A = \text{Cs}$ ( $x = 4$ ) and $A = \text{CH}_3\text{NH}_3$ ( $x = 4.5$ )

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**Abstract.**  $\text{Cs}[\text{V}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot 4\text{H}_2\text{O}$  (I):  $M_r = 467.98$ , monoclinic,  $C2/m$ ,  $a = 9.256$  (4),  $b = 7.510$  (3),  $c = 10.191$  (3) Å,  $\beta = 101.79$  (7)°,  $V = 693.5$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.241$  Mg m<sup>-3</sup>,  $\text{Mo K}\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 3.40$  mm<sup>-1</sup>,  $F(000) = 452$ , room temperature, final  $R = 0.029$  for 1077 independent reflections.

$[\text{CH}_3\text{NH}_3][\text{V}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot 4.5\text{H}_2\text{O}$  (II):  $M_r = 376.1$ , orthorhombic,  $Cmcm$ ,  $a = 7.572$  (3),  $b = 9.251$  (4),  $c = 23.334$  (6) Å,  $V = 1634.5$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.528$  Mg m<sup>-3</sup>,  $\text{Mo K}\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 0.66$  mm<sup>-1</sup>,  $F(000) = 780$ , room temperature, final  $R = 0.087$  for 774 independent reflections. The V atom