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References

BEKAROGLU, Ö., BREER, H., ENDRES, H., KELLER, H. J. & NAM GUNG, H. (1977). *Inorg. Chim. Acta*, **21**, 183–186.

BERDESINSKI, W. & NUBER, B. (1966). *Neues Jahrb. Mineral. Abh.* **104**, 113–146.

BREER, H., ENDRES, H., KELLER, H. J. & MARTIN, R. (1978). *Acta Cryst.* B**34**, 2295–2297.

ENDRES, H., KELLER, H. J., MARTIN, R. & NÖTZEL, S. (1979). To be published.

ENDRES, H., KELLER, H. J., MARTIN, R. & TRAEGER, U. (1979). *Acta Cryst.* Submitted.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KELLER, H. J., MARTIN, R. & TRAEGER, U. (1978). *Z. Naturforsch. Teil. B*, **33**, 1263–1266.

MATSUMOTO, N., YAMASHITA, M., KIDA, S. & UEDA, I. (1979). *Acta Cryst.* B**35**, 1458–1460.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1979). B**35**, 1887–1888

Structure of Dibromobis(1,2-diaminoethane)platinum(IV) 3,4-Dihydroxy-3-cyclobutene-1,2-dionate Dihydrate

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Abstract. $[PtBr_2(C_2H_8N_2)_2][C_4O_4].2H_2O$, $C_4H_{16}Br_2N_4Pt^{2+}.C_4O_4^{2-}.2H_2O$, monoclinic, $P2_1/n$, a = 11·038 (19), b = 14·289 (4), c = 6·983 (12) Å, β = 132·24 (10)°, V = 815·6 Å^3, d = 2·57 Mg m^{-3}, Z = 2. The structure was refined to R = 0·079 for 1553 counter data and consists of six-coordinate $[PtBr_2en_2]^{2+}$ units (en = 1,2-diaminoethane) with planar $[C_4O_4]^{2-}$ counterions which are strongly hydrogen bonded to each other and to the water molecules in the lattice. The reduced cell has a = 6·984, b = 14·290, c = 8·184 Å, β = 93·07°.

Introduction. In recent papers we have reported the structure of mixed-valence Wolffram's salt analogues (Endres, Keller, Martin, Nam Gung & Traeger, 1979). In some cases *three*-dimensional order is observed in the *linear* chain compounds (Endres, Keller, Martin & Traeger, 1979), and it was suggested that this ordering is caused by hydrogen bridges between the complex ligands and the perchlorate counterions. We therefore investigated the system $[PtBr_2en_2]^{2+}$–$[Pten_2]^{2+}$–$C_4H_2O_4$ (squaric acid) – the latter compound being well known for its ability to form hydrogen bridges – in the hope of strengthening the interchain coupling in Wolffram's salt analogues. After hot aqueous solutions containing 0·5 mmol $[PtBr_2en_2]^{2+}$ and 0·5 mmol $[Pten_2]^{2+}$ are mixed with a hot aqueous solution of 3,4-dihydroxy-3-cyclobutene-1,2-dione (squaric acid), the title compound crystallizes in red platelets. {Analysis for $[PtBr_2en_2][C_4O_4].2H_2O$, M_r = 622·9: calculated: C 15·40, N 8·98, H 3·46, Pt 31·31%; found: C 15·40, N 8·26, H 3·41, Pt (residue) 32·44%.}

Rotating-crystal and Weissenberg photographs (Cu $K\alpha$ radiation) showed the symmetry and systematic extinctions and established approximate lattice constants. Exact lattice constants (see *Abstract*) were calculated by least squares (Berdesinski & Nuber, 1966) from the θ values of 33 reflections, measured on a diffractometer. Intensity measurements on an automatic single-crystal diffractometer (AED, Siemens, Mo $K\alpha$ radiation, θ–2θ scans, 'five-value method', 4° < 2θ < 60°) furnished 1553 observed independent intensities. Reflections with $I < 3{\cdot}0\sigma(I)$ were classified as unobserved. Owing to the small crystal size (0·04 × 0·05 × 0·03 mm), no absorption correction was applied (μ = 14·5 mm^{-1}). Observed reflections were corrected for polarization and Lorentz factors. The systematic absences ($h0l$ with $h + l = 2n + 1$ and $0k0$ with $k = 2n + 1$) correspond to the space group $P2_1/n$ (C_{2h}^5). A Patterson synthesis showed the positions of Pt and Br. The positions of N, O and C atoms could be located by distance and angle calculations from the

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Table 1. *Atomic coordinates* ($\times 10^3$)

	x	y	z
Pt	0	0	0
Br	205·2 (3)	471·2 (2)	225·0 (4)
O(1)	227 (2)	40 (1)	699 (3)
O(2)	66 (2)	346 (1)	561 (4)
O(3)	324 (3)	266 (2)	637 (4)
N(1)	552 (2)	371 (1)	664 (4)
N(2)	498 (3)	428 (1)	240 (3)
C(1)	594 (6)	306 (2)	564 (6)
C(2)	485 (4)	325 (2)	275 (6)
C(3)	882 (3)	481 (2)	367 (4)
C(4)	33 (2)	430 (2)	531 (4)

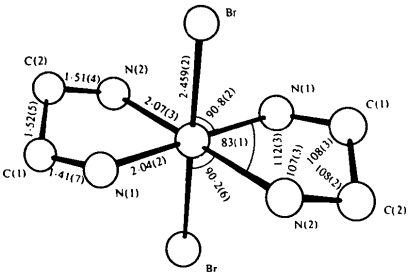

Fig. 1. View of the Pt complex unit with the numbering scheme, bond distances (Å) and bond angles (°).

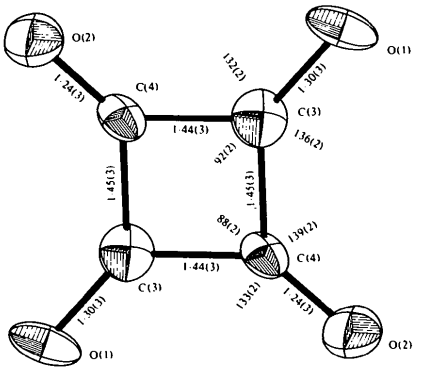

Fig. 2. The 3,4-dihydroxy-3-cyclobutene-1,2-dionate dianion with the numbering scheme, bond distances (Å) and angles (°).

maxima of a Fourier synthesis. The refinements resulted in $R = 0{\cdot}120$ with isotropic temperature factors. In the final cycles with anisotropic temperature factors all atoms were refined simultaneously. Refinement converged with $R = 0{\cdot}079$. Atomic coordinates are listed in Table 1.* Calculations were performed with programs of the XRAY system (Stewart, Kundell & Baldwin, 1970). Scattering factors were derived from *International Tables for X-ray Crystallography* (1974). The reduced lattice constants were calculated by the procedure from *International Tables for X-ray Crystallography* (1969).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34425 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

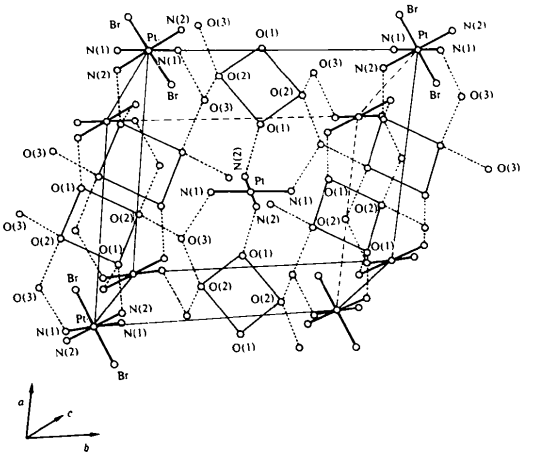

Fig. 3. Unit cell of the title compound. All the C atoms (of the diamine ligand *and* of the squaric acid) and some Br atoms are omitted for clarity. The hydrogen-bond network between O(3)–N(1), O(3)–O(2) and O(1)–N(2) is indicated by broken lines.

Discussion. The numbering scheme, bond distances and angles of the Pt complex dication and of the 3,4-dihydroxy-3-cyclobutene-1,2-dione dianion are shown in Figs. 1 and 2. The Pt–Br distance in the complex unit is 2·459 (2) Å, which is much shorter than in the Wolffram's salt analogues, but is normal for a Pt–Br bond in Pt^{IV} complexes. As expected the squaric acid dianion forms strong hydrogen bonds with the N atoms of the ligands, which leads to a structure very different from those of Wolffram's salts. Only Pt^{IV} complex units are built into the lattice and no chains are formed. The hydrogen-bond network is shown in Fig. 3. Characteristic distances in this network are: O(1)–N(2) = 2·81 (2), O(1)–N(2) = 2·77 (4), O(3)–N(1) = 2·83 (4), O(3)–O(2) = 2·80 (3), O(3)–O(2) = 2·76 (4) Å. O(1) and O(2) are from the squaric acid dianion; O(3) is the water molecule.

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References

BERDESINSKI, W. & NUBER, B. (1966). *Neues Jahrb. Mineral. Abh.* **104**, 113–146.

ENDRES, H., KELLER, H. J., MARTIN, R., NAM GUNG, H. & TRAEGER, U. (1979). *Acta Cryst.* B**35**, 1885–1887.

ENDRES, H., KELLER, H. J., MARTIN, R. & TRAEGER, U. (1979). *Acta Cryst.* Submitted.

International Tables for X-ray Crystallography (1969). Vol. I, 3rd ed. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.