

distortion can be approximately measured by the dihedral angle Ni-N-C'-C ($12 \pm 3^\circ$). Presumably, the lone pair of the nitrogen atom lies along the Ni-N line, and therefore the dihedral angle indicates, apart from a small distortion of the Ni-N bond, the rotation of the tetrahedral NH₂ group with respect to the C'-C line. The average O-C-C'-N torsional angle of $-4.9 (3)^\circ$ is also an indication of a systematic distortion of the chelate rings involving a slight twist about the C'-N line. Similar distortions of the chelate rings have been observed in the isostructural [Ni(Hyc)₃]⁻¹⁷ and [Cr(Gly)₃]⁻¹⁸ complexes.

The complexes in this structure are held together by a network of hydrogen bonds.¹⁶ All of the hydrogen atoms attached to the nitrogen atoms are involved as donors to oxygen atoms on the perchlorate anions with average N...O distances of 3.23 (6) Å and average H...O distances of 2.33 (5) Å. In addition, the hydrogen atoms of the water molecule form strong hydrogen bonds with O(2) and O(3) of adjacent [Ni(Gly)₃]⁻ anions with average O...O distances of 2.78 (5) Å and average H...O distances of 1.99 (6) Å. In all cases, the hydrogen atoms are slightly off the line joining the heavy atoms in accordance with the data quoted by Hamilton and Ibers and with the current view of bent hydrogen bonds.²⁰

One of the sodium cations, Na(1), is in an approximately octahedral environment, being surrounded by six oxygen atoms

of adjacent [Ni(Gly)₃]⁻ units with Na...O distances ranging from 2.288 (3) to 2.828 (3) Å and cis angles ranging from 65.40 (7) to 103.14 (9)°. The other sodium cation, Na(2), is in a much more irregular environment surrounded by six oxygen atoms from [Ni(Gly)₃]⁻ anions and water molecules with Na...O distances ranging from 2.329 (3) to 2.945 (5) Å. In neither case are the sodium cations adjacent to oxygen atoms from the perchlorate anions.

The perchlorate anions exhibit the expected tetrahedral geometry with an average Cl-O distance of 1.42 (2) Å and with O-Cl-O angles ranging from 106.3 (2) to 112.4 (3)°. The anisotropic thermal parameters for the perchlorate oxygen atoms are rather large compared to the thermal factors for other atoms in this structure. The apparent highly anisotropic thermal behavior of these atoms, coupled with the large errors in their thermal parameters, may be taken as an indication of a disorder in the positions of the perchlorate oxygen atoms.

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Registry No. Na₂[Ni(NH₂CH₂COO)₃]ClO₄·D₂O, 78919-05-8.

Supplementary Material Available: Listings of calculated and observed structure factor amplitudes, hydrogen bonds (Table VII), and Na...O contacts (Table VIII) (18 pages). Ordering information is given on any current masthead page.

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Synthesis and Crystal Structure of Barium Tetrakis(μ -squarato-01,02)-diplatinatate(II) Hexahydrate

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The new binuclear platinum(II) complex barium tetrakis(μ -squarato-01,02)-diplatinatate(II) hexahydrate, Ba₂[Pt₂(C₄O₄)₄·6H₂O], has been prepared and characterized. The crystal and molecular structure has been determined by single-crystal X-ray analysis using three-dimensional diffractometer data. The compound crystallizes in the monoclinic space group C₂/m, with two formula units in the unit cell. The lattice parameters are $a = 17.8726 (8) \text{ \AA}$, $b = 8.1526 (5) \text{ \AA}$, $c = 8.7679 (5) \text{ \AA}$, and $\beta = 91.423 (5)^\circ$. A final residual value $R = 0.073$ was obtained by full-matrix minimization of $\sum(|F_o| - |F_c|)^2$ using 1881 independent observed reflections. The structure contains discrete (squarato-01,02)-bridged binuclear platinum(II) anions with a Pt-Pt distance of 3.061 Å. The crystal structure is stabilized by the water molecules, which are weakly coordinated to the barium ions and form hydrogen bonds between the squarate ions.

Introduction

Current interest in synthesis of new platinum salts of the Krogmann salt type prompts an investigation of the coordination chemistry of the squarate ion (the dianion of 3,4-dihydroxycyclobutenedione) toward platinum(II).

Chemically, the squarate ion is related to the oxalate ion, although it forms less stable metal complexes, normally with a 1:1 stoichiometry.

Reaction between tetraaquaplatinum(II) and squaric acid results in formation of bis(squarato)platinatate(II) salts, which easily are partially oxidized.¹

Besides these salts, some blue ill-defined species could be isolated. Krogmann has reported similar species of unknown structure formed as byproducts during the synthesis of the bis(oxalato)platinatate(II) salts.² These species have several properties in common with the "platinum blues", and it is tempting to suggest a similar structure.

In the present paper we show that one of the platinum(II)-squarato complexes formed during the reaction between

squaric acid and tetraaquaplatinum(II) has a squarato-bridged binuclear structure with some resemblance to the binuclear species isolated together with some of the "platinum blues".^{3,4}

Experimental Section

Preparation. Squaric acid (Aldrich) (1 g, 8.77 mmol) was added to a tetraaquaplatinum(II) solution (100 mL, 0.02 M; 1 M HClO₄) prepared according to Elding.⁵ The mixture was heated to 55 °C for 1 h, during which time the solution became dark. If platinum black is observed during this procedure, the heating must at once be interrupted and the solution must be filtered through a micropore filter before the preparation is continued. A saturated solution of sodium perchlorate (5 mL) was added, and as the solution cooled to room temperature, a black precipitate of the partially oxidized bis(squarato)platinatate(II) was formed. This product was filtered off after 2 h; yield 0.25 g, 25%. The mother liquor was dark blue, and from this another black precipitate was formed by slow evaporation during several days; yield 0.1 g.

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Table I. Crystal Data

monoclinic <i>C2/m</i>	$V = 1277.15 \text{ \AA}^3$
$a = 17.8726 (8) \text{ \AA}$	$T = \text{room temperature}$
$b = 8.1526 (5) \text{ \AA}$	$Z = 4$
$c = 8.7679 (5) \text{ \AA}$	$\text{mol wt}^a = 610.6$
$\beta = 91.423 (5)^\circ$	$\rho(\text{obsd}) = 3.09 \text{ g cm}^{-3}$
$\mu(\text{Mo K}\alpha) = 154.6 \text{ cm}^{-1}$	$\rho(\text{calcd}) = 3.175 \text{ g cm}^{-3}$

^a Molecular weight refers to the asymmetric unit BaPtC₈O₈·3H₂O.

The last precipitate was dissolved in water (20 mL), giving a dark blue solution. To this solution was added a hydrazinium hydrogen sulfate solution (1 mL, 0.1 M), changing the color to green. To this solution was added a barium nitrate solution (2 mL, 0.1 M), which resulted in formation of a dark gel.

After the gel was allowed to stand for 2 weeks at room temperature, large orange crystals of the title compound formed. The crystals were removed from the gel and washed with water.

Anal. Calcd for Ba₂[Pt₂(C₄O₄)₄·6H₂O: C, 13.77; H, 0.99; Pt + BaCO₃, 64.27. Found: C, 12.99; H, 0.90; Pt + BaCO₃, 66.50.

The partially oxidized sodium bis(squarato)platinate(II) was reduced with hydrazinium hydrogen sulfate and precipitated with barium nitrate, giving a yellow compound, which had a powder diffraction diagram entirely different from the corresponding diagram of Ba₂-[Pt₂(C₄O₄)₄](aq).

Crystal Data and Intensity Measurements. Unit cell dimensions were determined by a least-squares refinement of data from a powder photograph taken with Cu K α radiation ($\lambda = 1.54049 \text{ \AA}$) and calibrated with silicon as an internal standard (camera: XDC 700 Hagg-Guinier). Fifty-three powder lines were used in the least-squares refinement of the monoclinic cell constants.

The results are $a = 17.8726 (8) \text{ \AA}$, $b = 8.1526 (5) \text{ \AA}$, $c = 8.7679 (5) \text{ \AA}$, and $\beta = 91.423 (5)^\circ$. From systematic absences the space group is either *C2*, *Cm*, or *C2/m*; after further investigation *C2/m* was considered the best choice.

The measured density (flotation method) is 3.09 g cm^{-3} . The rather poor agreement between the measured density and the calculated density is probably caused by the experimental difficulties with the flotation method applied to a very small amount of "micro" crystals. The calculated density for two units of Ba₂[Pt₂(C₄O₄)₄· x H₂O in the cell is 3.08 g cm^{-3} when $x = 4$ and 3.17 g cm^{-3} when $x = 6$. The electron density map confirmed the value $x = 6$ in agreement with the analysis. The linear absorption coefficient $\mu(\text{Mo K}\alpha)$ has a value of 154.6 cm^{-1} . The crystal data are summarized in Table I. A single crystal with dimensions $0.25 \times 0.31 \times 0.09 \text{ mm}$ was used for data collection on an Enraf-Nonius AD-3 diffractometer employing graphite-monochromatized Mo K α radiation. Reflections hkl and $\bar{h}k\bar{l}$ in the range $2.5 < \theta < 30^\circ$ were measured applying the ω -scan technique. Systematically absent reflections ($h + k = 2n + 1$) were not measured. A reflection was designated "not observed" if $I < 2.5\sigma(I)$. With this criterion 1881 out of 1982 measured reflections were regarded as observed. Each reflection was measured with a scan width of 1.6° , a scan speed of $0.01^\circ \text{ s}^{-1}$, and two background measurements for 80 s on each side of the reflection. One of the three reflections (800), (040), and (008) was monitored every 15 reflections. Lorentz, polarization, and absorption corrections were applied, but no extinction correction was made.

Structure Solution and Refinement. An analysis of the Patterson map showed that the Pt and Ba atoms lie in special positions on 2-fold axes and mirror planes, respectively. The Patterson analysis yielded the coordinates of the Pt and Ba atoms. A Fourier synthesis with phases from the Pt and Ba positions revealed all the other nonhydrogen atoms. The refinement was accomplished by a full-matrix least-squares technique. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1$. Contributions from unobserved reflections were included if $F_c > F_o$. Isotropic thermal parameters were used for C and O and anisotropic were used for the heavier atoms. The final agreement index, $R = \sum ||F_o| - |F_c|| / \sum |F_o|$, was 0.073. All calculations were performed on a Univac 1110 computer by using the X-RAY 76 system.⁶ The atomic scattering factors for Ba²⁺ and the neutral atoms C, O, and Pt were taken from ref 7. Tables of observed and calculated structure

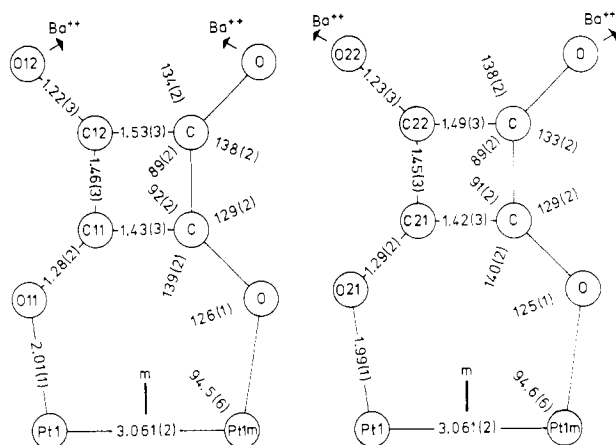


Figure 1. Bond distances (\AA) angles (deg), and the atom numbering scheme for the two crystallographic independent squarate ligands, each of which is divided by a crystallographic mirror plane as indicated with *m*.

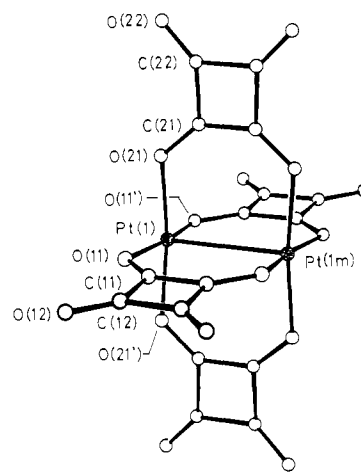


Figure 2. Perspective view of the tetrakis(μ -squarato-*O1,O2*)-diplatinate(II) anion. The atoms O(11), O(11') and O(21), O(21') are interrelated by a crystallographic 2-fold axis along Pt(1)-Pt(1m). The anion is divided by a crystallographic mirror plane perpendicular to the line Pt(1)-Pt(1m).

factors are available as supplementary material. The final positional and thermal parameters are given in Table II. Interatomic bond lengths and angles are quoted in Figure 1.

Results and Discussion

Preparation. Squarate cannot replace chloride in tetrachloroplatinate(II); however, reacting tetraaquaplatinum(II) with squaric acid at 55°C results in formation of a mixture of several (squarato)platinate(II) salts. Salts of partially oxidized bis(squarato)platinate(II) are easy to precipitate due to their low solubility.¹ The title compound was isolated from a blue fraction of (squarato)platinate(II) remaining in solution after all bis(squarato)platinate(II) was precipitated as the partially oxidized sodium salt. Large crystals of Ba₂[Pt₂(squa)₄·6H₂O were grown by slow diffusion in a gel of reduced barium (squarato)platinate(II).

The crystal exhibits a clear pleochroism, being red with the electric light vector along the crystallographic *b* axis and yellow with the light vector perpendicular to this axis.

Description of the Structure. The crystal structure consists of separate barium cations and binuclear squarato-bridged platinate anions with the platinum atoms placed on the crystallographic 2-fold axes.

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Table II. Fractional Atomic Coordinates and Thermal Parameters^{a,b}

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> , Å ²		<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> , Å ²
Pt(1)	0.0000	0.1877 (1)	0.0000		O(21)	0.5500 (8)	0.292 (2)	0.800 (2)	1.5 (2)
Ba	0.3842 (1)	0.0000	0.3638 (2)		O(22)	0.6053 (9)	0.296 (2)	0.476 (2)	2.2 (3)
O(1)	0.358 (1)	0.000	0.049 (3)	3.2 (6)	C(11)	0.358 (1)	0.412 (2)	0.849 (2)	1.3 (3)
O(2)	0.459 (1)	0.0000	0.674 (3)	2.7 (6)	C(12)	0.283 (1)	0.406 (3)	0.776 (2)	1.5 (3)
O(3)	0.285 (2)	0.000	0.600 (4)	4.0 (8)	C(21)	0.566 (1)	0.413 (2)	0.711 (2)	1.3 (3)
O(11)	0.4000 (8)	0.293 (2)	0.893 (2)	1.6 (2)	C(22)	0.592 (1)	0.409 (3)	0.557 (2)	1.8 (3)
O(12)	0.2386 (8)	0.302 (2)	0.732 (2)	2.0 (2)					
	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃			<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃	
Pt(1)	0.0124 (4)	0.0100 (4)	0.0144 (4)			0.0000	0.0043 (3)	0.0000	
Ba	0.0136 (7)	0.0149 (7)	0.0258 (8)			0.0000	0.0013 (5)	0.0000	

^a Estimated standard deviations in parentheses refer to the last digit. ^b The anisotropic thermal parameters are in the form $\exp[-2\pi^2(h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12} + \dots)]$.

Table III. Distances of Atoms (Å) from Least-Squares Planes^{a,b}

<i>P</i> = -0.436 47	<i>P</i> = 0.936 36	<i>P</i> = 0.000 00
<i>Q</i> = 0.000 00	<i>Q</i> = 0.000 00	<i>Q</i> = 1.000 00
<i>R</i> = 0.899 72	<i>R</i> = 0.351 04	<i>R</i> = 0.000 00
<i>S</i> = 3.985 62	<i>S</i> = 11.506 97	<i>S</i> = 2.386 67
C(11)* 0.000	C(21)* 0.000	O(11)* 0.002
C(11m)* 0.000	C(21m)* 0.000	O(11')* 0.002
C(12)* 0.000	C(22)* 0.000	O(21)* -0.002
C(12m)* 0.000	C(22m)* 0.000	O(21')* -0.002
O(11) 0.020	O(21) -0.003	Pt(1) 0.159
O(11m) 0.020	O(21m) -0.003	
O(12) -0.001	O(22) -0.034	
O(12m) -0.001	O(22m) -0.034	

^a The planes are in the form $Px + Qy + Rz = S$. The coordinates *x*, *y*, and *z* are expressed parallel to the orthogonal axial directions *a*, *b*, and *c**. ^b The least-squares planes are defined by atoms marked with an asterisk.

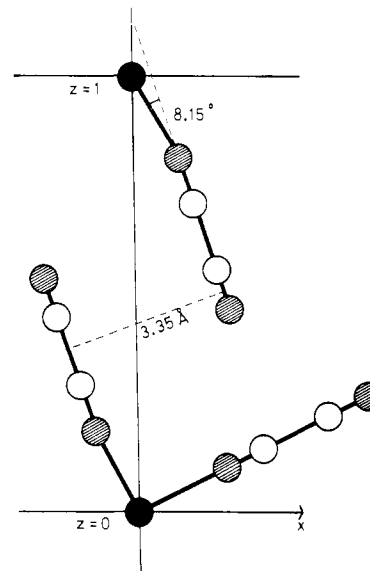
The binuclear anion is shown in Figure 2. Both of the platinum atoms are coordinated to four oxygens from four squarate bridges in two equivalent pairs. Viewed down the Pt-Pt axis, the complex ion has approximately 4-fold symmetry with square-planar arrangement of O atoms around each platinum (Table III).

The unusual tetrakis(μ -squarate)-diplatinum arrangement bears some resemblance to the tetrakis(μ -carboxylato)-dimetal ions known for many divalent transition-metal ions such as Cr(II), Rh(II), Re(II), and Mo(II).⁸

Each of the two platinum atoms are displaced by approximately 0.16 Å from the plane made by the four coordinating atoms in such a way that the platinum atoms get closer to each other.

This and the fact that the Pt-Pt distance within the dimer is 3.061 Å indicate a small degree of metal-metal bond, which might be the explanation of the observed extra optical absorption along the Pt-Pt axis. The Pt-Pt distance, however, is clearly longer than in the two bis(μ -(1-methylthyminato-*N3,O4*))-bis(*cis*-diammineplatinum(II)) complexes^{3,4} (2.909 and 2.974 Å), bis(μ -(α -pyridonato-*N,O*))-bis(*cis*-diammineplatinum(II)) nitrate (2.90 Å),¹⁷ bis(μ -(1-methyluracilato-*N3,O4*))-bis(*cis*-diammineplatinum(II)) dinitrate (2.954 Å),¹⁸ and the octakis(phosphito)diplatinate(II) (2.925 Å).⁹ Compared to the very short Pt-Pt distance observed in platinum(III) dimers as $K_2[Pt_2(SO_4)_4(H_2O)_2]$, (2.466 Å),¹⁰ it is obvious that the platinum in $Ba_2[Pt_2(squa)_4] \cdot 6H_2O$ has the oxidation state +2.

The Pt-Pt distance between adjacent dimers is much longer (5.092 Å) and is within the range known for structures without

**Figure 3.** *y*-Axis projection of crystal packing along the *z* axis showing the π - π interaction distance of 3.35 Å.**Table IV.** Dimensions of the Ba²⁺...O Ionic Bonds and the O...O Distances in Potential O-H...O Hydrogen Bonds

atoms defining the distances		<i>a</i>	symmetry operations for O atoms	dist, ^b Å
Ba ²⁺ , O	O			
Ba ²⁺	O(1)		<i>x</i> , <i>y</i> , <i>z</i>	2.79 (3)
Ba ²⁺	O(2)		<i>x</i> , <i>y</i> , <i>z</i>	3.00 (2)
Ba ²⁺	O(3)		<i>x</i> , <i>y</i> , <i>z</i>	2.76 (3)
Ba ²⁺	O(12)	G	$1/2 - x$, $y - 1/2$, $1 - z$	2.83 (2)
Ba ²⁺	O(21)	E	$1 - x$, <i>y</i> , $1 - z$	3.04 (1)
Ba ²⁺	O(22)	D	$1 - x$, <i>y</i> , $1 - z$	2.84 (2)
O(1)	O(11)	F	<i>x</i> , <i>y</i> , $z - 1$	2.87 (2)
O(1)	O(12)	G	$1/2 - x$, $y - 1/2$, $1 - z$	3.06 (3)
O(2)	O(3)			3.16 (4)
O(2)	O(21)	B	<i>x</i> , <i>y</i> , <i>z</i>	3.08 (2)
O(2)	O(22)	D	$1 - x$, <i>y</i> , $1 - z$	2.94 (2)
O(3)	O(12)	A	<i>x</i> , <i>y</i> , <i>z</i>	2.86 (2)
O(3)	O(22)	D	$1 - x$, <i>y</i> , $1 - z$	3.18 (3)

^a Identification of squarate O atoms in Figure 4. ^b Estimated standard deviations in parentheses refer to the last digit.

any dimer-dimer interaction (5.063⁹-5.92 Å^{3,4}) but is much longer than the distance observed for head-to-head bis(μ -(α -pyridonato-*N,O*))-bis(*cis*-diammineplatinum(II)) nitrate (3.13 Å),¹⁷ which is to be compared with the distances observed for the *cis*-diammineplatinum α -pyridone blue¹¹ (2.885 Å), described as a tetramer with the formal oxidation state +2.25.

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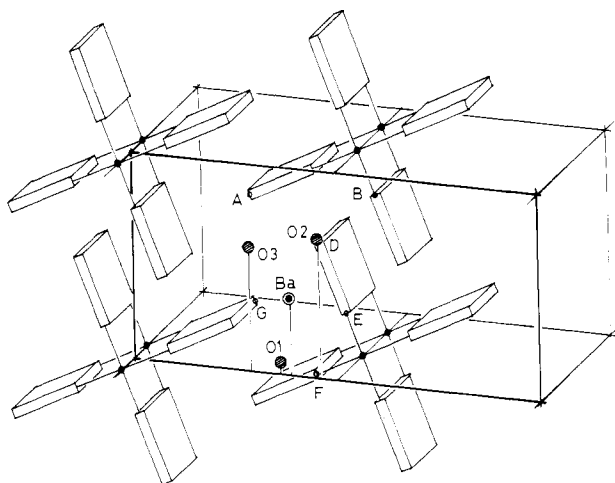


Figure 4. Parallel projection of the crystal structure. The squarate ligands are symbolized by square-shaped plates. O1, O2, and O3 are the oxygen atoms in the water molecules, and A-G are squarate oxygen atoms. Table IV gives a list of the ionic bonds involving these atoms.

The Pt-O and C-O distances in the bridges (Figure 1) are very near the distances found in the bis(oxalato)platinate(II)¹² and bis(croconato)platinate(II)¹³ salts (croconate = the dianion of 4,5-dihydroxycyclopentenetrione). Where oxalate and croconate in these complexes are bidentate chelate ligands with O-O distances shorter than in the free ligands, the O-O distance in the bridged squarate of 3.37 Å is significantly longer than for the free ligand (3.24 Å).¹⁴

When the accuracy of the bond lengths and bond angles in the squarate units is considered, there might be some doubt that the observed differences in C-O and C-C bond lengths

are real. However, the differences in bond lengths are chemically reasonable, and the values for chemically equivalent bonds in the two independent squarate units agree very well (Figure 1). The bond lengths in the two squarate ligands are comparable to the bond lengths in squaric acid¹⁵ and the hydrogen squarate anion,¹⁶ an indication of a similar π -electron distribution.

The O atoms in the squarate ligands deviate less than 0.04 Å from the best plane through the C atoms (Table III), as is the case for the free squarate ion.¹⁴

The two crystallographically independent squarate ligands with the numbers 1 and 2 differ in their Ba...O interactions. The atoms O(12) and O(12m) have ionic bonds to the same Ba²⁺ ion, whereas O(22) and O(22m) have ionic bonds to two different Ba²⁺ ions (Figure 1).

Squarate ligands 2 in adjacent dimeric units are bent 8.15° toward each other as shown in Figure 3. The close intermolecular contact between these planes (3.35 Å) might suggest some stabilization through π - π interaction between the aromatic systems. This point of view is supported by the similar π - π interaction found in the crystal structure of potassium squarate monohydrate.¹⁴

The barium ions and the water molecules lie on the crystallographic mirror planes. Distances from the barium ion to the surrounding O atoms are quoted in Table IV. The crystal structure is stabilized by the water molecules partly by O...HOH...O hydrogen bonds between the squarate units (Figure 4) and partly by H₂O...Ba²⁺ interactions.

Registry No. Ba₂[Pt₂(C₄O₄)₄]-6H₂O, 78891-43-7.

Supplementary Material Available: A listing of observed and calculated structure factors (15 pages). Ordering information is given on any current masthead page.

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