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Crystal and Molecular Structure of Bis(2,2'-bipyridyl)mercury(II) Nitrate Dihydrate

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The crystal structure of bis(2,2'-bipyridyl)mercury(II) nitrate dihydrate has been determined from diffractometer X-ray intensity data by means of Patterson and Fourier methods and refined by the full-matrix least-squares technique based on 3589 independent reflexions to the index R of 0.069. Crystals are monoclinic holohedral, space group $P2_1/c$ with four formula units in the unit cell of dimensions $a = 10.091(2)$, $b = 7.378(3)$, $c = 32.990(6)$ Å, $\beta = 105.49(3)^\circ$, $D_{\text{obs}} = 1.87 \text{ g cm}^{-3}$, $D_{\text{calc}} = 1.888 \text{ g cm}^{-3}$. The nitrogen atoms of the bipyridyl ligands, which are both in their chelate function, form a flattened tetrahedron around the mercury atom at a mean Hg-N distance of 2.29 Å. Three nitrate oxygen atoms at a mean Hg...O distance of 2.72 Å, complete the coordination of mercury to a distorted capped octahedron, whereas the water molecules are out of the mercury coordination sphere.

INTRODUCTION

The existing data about the molecular structure of metal chelate complexes are very numerous, but little is known about the mercury chelate complexes. The structure of certain complexes with the mercury atom chelated by classical bidentate ligands, such as ethylenediamine,^{1,2} 1,10-phenanthroline,^{3,4} and 2,2'-bipyridyl^{5,6} has been known only recently. Also the structure of the mercury complex with 1,8-naphthyridine,⁷ a less usual chelate ligand, was solved in order to examine the chelation of the mercury atom by a ligand with the small »bite«.

Unexpectedly, the data obtained are not sufficient for the understanding of the chelate-complex chemistry of mercury. In distinction from most transition metals, whose coordination numbers and polyhedra are characteristic and well defined for a given metal, mercury in complexes appears in different coordinations, mostly in deformed tetrahedra or octahedra.⁸ Since complexes with 2,2'-bipyridyl are typical for other metals, we have continued our study of the mercury complexes also with this ligand. In addition to the structure of the one-to-one complex of mercury(II) nitrate with 2,2'-bipyridyl⁶, we have also solved the structure of the one-to-two complex, which occurs as dihydrate, and which is the subject of the present paper. This complex has not been reported yet in the chemical literature and a detailed description of its preparation and properties will be published separately.

EXPERIMENTAL

Preparation

White, fine, needle-shaped crystals of the complex separate from a warm aqueous solution of mercury(II) nitrate dihydrate (1.71 g in 10 ml of water slightly acidified with diluted nitric acid) to which an ethanolic solution of 2,2'-bipyridyl (1.56 g in 4 ml) was added. When exposed to free atmosphere, the complex loses water of crystallization, so that the crystal specimen for X-ray analysis, obtained by crystallization from a warm saturated aqueous solution, was sealed in a Lindemann glass capillary tube together with a small amount of mother liquor.

Crystal Data

From rotation and Weissenberg photographs and from single crystal diffractometry, $\text{CuK}\alpha$, $\lambda = 1.5418 \text{ \AA}$: $a = 10.091(2)$, $b = 7.378(3)$, $c = 32.990(6) \text{ \AA}$, $\beta = 105.49(3)$, $V = 2366.94 \text{ \AA}^3$, $z = 4 (\text{C}_{10}\text{H}_8\text{N}_2)_2\text{Hg}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, $D_{\text{obs}} = 1.87 \text{ g cm}^{-3}$, $D_{\text{calc}} = 1.888 \text{ g cm}^{-3}$, $\mu(\text{CuK}\alpha) = 129.8 \text{ cm}^{-1}$, space group $P2_1/c$ (No. 14).

*Intensity Data, Structure Determination and Refinement**

The intensities of 3734 independent reflexions from an approximately prismatic specimen ($0.39 \times 0.24 \times 0.23 \text{ mm}^3$) were collected up to $\sin\theta/\lambda = 0.65$ on a computer controlled automatic diffractometer Philips PW 1100 (graphite monochromatized $\text{CuK}\alpha$ radiation, $\theta - 2\theta$ scan technique, scan range 1.2° , scan rate $1.8^\circ/\text{min}$), from which 3589 reflexions with $I > 2\sigma(I)$ were used. Neither absorption nor extinction corrections were applied. The preliminary atom coordinates, obtained from the three-dimensional Patterson and electron density syntheses and refined by the full-matrix least-squares method with the isotropic temperature factors, led to a discrepancy factor R of 0.145. The final R value of 0.069 was obtained by a further full-matrix least-squares refinement assuming anisotropic thermal parameters for the mercury, nitrogen and oxygen atoms from the nitrate ions and water molecules, and isotropic thermal parameters for the atoms from bipyridyl ligands. Unit weights were allotted to all reflexions. The anomalous dispersion for all non-hydrogen atoms was included in the F_c calculation⁹ with the atomic scattering factors by Cromer and Mann.¹⁰ Final atomic coordinates and thermal parameters are given in Table I.

DESCRIPTION OF THE STRUCTURE AND DISCUSSION

The crystal structure consists of discrete $[(\text{bipy})_2\text{Hg}]^{2+}$ cations, with bipyridyl as a bidentate ligand, nitrate anions and water molecules. Both nitrates are closer to the mercury atom than one would expect for usual intermolecular contacts, while there are no such contacts between the mercury atom and water molecules (Figure 1 and Table II). The most interesting feature of the structure is the mutual spatial orientation of the chelate ligands: the planes defined by the coordinates of the Hg, N(1), N(2) and Hg, N(3), N(4) atoms form an angle of 40.5° . Thus, in terms of the coordination polyhedron, the bipyridyl nitrogen atoms form a flattened tetrahedron or, more exactly, a disphenoid around the mercury atom. By analogy with simple complexes, in which mercury is tetra-coordinated, one would expect an essentially tetrahedral coordination like that found in the crystal structure of bis(ethylenediamine)mercury(II) perchlorate and dithiocyanato(ethylenediamine)mercury(II),¹ where the angle between the ligand planes is 63° and 89.8° , respectively. In the former structure, the tetrahedron flattening is caused by the $\text{NH} \dots \text{O}$ hydrogen bond, i. e. it is due to intermolecular forces and packing conditions in the crystal. Consequently, the mercury sp^3 hybrid orbitals, dictating the coordination polyhedron in the free complex, are not solely decisive for the mutual orientation of the ligands. It

* A list of observed and calculated structure factors is obtainable from the authors on request.

TABLE I

Atomic Coordinates ($\times 10^4$) and Thermal Parameters, with Estimated Standard Deviations in Parantheses

	x/a	y/b	z/c	B (\AA^2)
Hg	2015(3)	1493(1)	3961(0)	*
N(6)	807(11)	—1543(23)	4433(4)	*
O(4)	1921(8)	—1688(19)	4340(3)	*
O(5)	492(15)	—2687(25)	4661(5)	*
O(6)	122(12)	— 125(23)	4294(5)	*
O(7)	4359(19)	1766(27)	2715(5)	*
N(5)	3355(18)	4396(36)	3430(8)	*
O(1)	3232(15)	4971(26)	3784(6)	*
O(2)	2801(15)	2985(27)	3288(5)	*
O(3)	3917(32)	5271(40)	3213(15)	*
O(8)	4995(26)	3235(27)	2056(8)	*
N(2)	2861(10)	2470(14)	4639(3)	1.80(17)
C(1)	2050(13)	3081(19)	4884(4)	2.48(23)
C(2)	2596(15)	3636(24)	5295(5)	3.37(27)
C(3)	3991(15)	3571(25)	5464(5)	3.45(27)
C(4)	4814(13)	2925(19)	5219(4)	2.38(22)
C(5)	4202(11)	2406(16)	4799(3)	1.43(18)
N(1)	4347(10)	1122(14)	4141(3)	1.91(17)
C(6)	5038(11)	1724(16)	4523(3)	1.55(18)
C(7)	6457(14)	1702(22)	4647(4)	3.02(25)
C(8)	7150(17)	1041(25)	4367(5)	4.05(33)
C(9)	6449(17)	406(26)	3983(5)	4.12(32)
C(10)	5025(15)	464(23)	3871(5)	3.30(27)
N(4)	31(12)	3067(17)	3658(4)	3.10(22)
C(11)	— 220(21)	4695(31)	3822(6)	5.40(42)
C(12)	—1524(26)	5523(38)	3660(8)	7.04(55)
C(13)	—2399(28)	4628(42)	3343(9)	7.82(63)
C(14)	—2173(22)	2935(32)	3158(7)	5.86(46)
C(15)	— 878(14)	2214(21)	3338(4)	2.81(24)
N(3)	767(10)	— 148(15)	3406(3)	2.25(18)
C(16)	— 463(13)	482(20)	3199(4)	2.44(22)
C(17)	—1306(20)	— 507(30)	2859(6)	5.13(39)
C(18)	— 795(24)	—2109(34)	2751(7)	6.45(51)
C(19)	475(25)	—2764(35)	2957(8)	6.60(52)
C(20)	1252(18)	—1760(27)	3299(6)	4.42(34)

* Anisotropic thermal parameters ($\times 10^4$) in the form $\exp[-B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + 2B_{12}hk + 2B_{13}hl + 2B_{23}kl]$ with co-efficients:

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Hg	20(0.5)	204(2)	5(0.1)	39(1)	—9(0.2)	—7(0.5)
N(6)	39(10)	281(36)	9(1)	0.3(37)	21(6)	—17(13)
O(4)	26(8)	378(36)	7(1)	117(29)	8(5)	15(11)
O(5)	145(19)	430(51)	17(2)	—120(51)	46(10)	47(17)
O(6)	88(14)	404(47)	21(2)	239(44)	55(9)	29(16)
O(7)	294(31)	415(53)	16(2)	183(69)	84(13)	35(18)
N(5)	100(21)	378(62)	28(4)	221(62)	80(16)	108(28)
O(1)	116(18)	402(54)	25(3)	96(50)	25(12)	—32(20)
O(2)	130(19)	390(52)	12(2)	—2(50)	0.3(9)	—7(15)
O(3)	485(64)	532(88)	122(14)	361(126)	422(57)	274(61)
O(8)	471(50)	298(50)	36(4)	—274(85)	183(25)	36(4)

is expected that this »external« distorting effect will be more pronounced in structures with large and »clumsy« ligands. Indeed, in the structure of bis(1,10-phenanthroline)mercury(II) nitrate⁴, the angle between the ligand planes is only 24.5°, so that the coordination of mercury in this complex is more conveniently described as a twisted square planar than as a flattened tetrahedral coordination. Still, the question about the coordination type in the free [(phen)₂Hg] cation remains open, although a tetrahedron, based upon sp³ hybrid orbitals, seems more probable than a square, for which sp²d hybrid orbitals would be necessary. Moreover, the square planar coordination around mercury has not been observed yet. The octahedral effective coordination, which is rather common in the structure of mercury compounds, is based in most cases upon two bonds with additional four equatorial bonds (the effective two-plus-four coordination) rather than upon four bonds with additional two bonds from ligands in trans-position (the effective four-plus-two coordination). Yet, there are three known crystal structures with hexa-coordinated mercury in a regular octahedron formed by six oxygen atoms, i. e. hexakis(pyridine-*N*-oxide)mercury(II) perchlorate,¹¹ hexakis(dimethylsulphoxide)mercury(II) perchlorate¹² and hexa-aquamercury(II) perchlorate¹³, while some others with other ligands¹⁴ are likely to be found. Since »distorted« coordination polyhedra around mercury occur more frequently than the regular ones, it seems that the hybrid mercury bond orbitals, favourable for the given geometry, do not differ markedly in energy. There is still one fact which has to be taken into account when the chelation of the mercury atom is concerned. Because of a proportionally large mercury-to-donor atom distance, the small »bite« of the chelate ligands, as measured by the angles of 72.2(4)° and 71.8(4)° between the N(1)-Hg-N(2) and N(3)-Hg-N(4) bonds, respectively, in the present structure, is more appropriate for a planar or octahedral coordination than for a tetrahedral one. E. g., in the one-to-two complex of mercury(II) thiocyanate with 1,10-phenanthroline, where the mercury atom is in a distorted octahedral coordination, the thiocyanate groups are

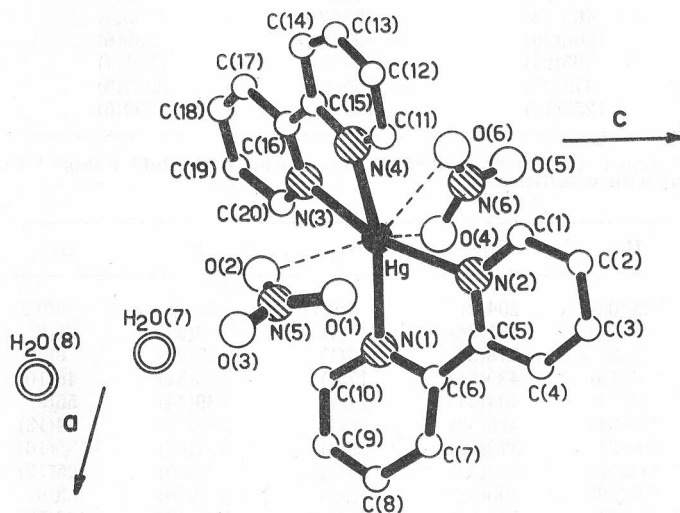


Figure 1. One asymmetric unit in the crystal structure of bis(2,2'-bipyridyl)mercury(II) nitrate dihydrate as viewed along the *b* axis. The additional mercury-to-oxygen contacts smaller than the sum of van der Waals radii are shown by dashed line.

TABLE II

Interatomic Distances (Å) and Angles (°), with Standard Deviations in Parentheses

(a) Distances and Angles about the Mercury Atom

Hg-N(1)	2.284(10)	Hg...O(1)	2.970(19)
Hg-N(2)	2.286(9)	Hg...O(2)	2.777(17)
Hg-N(3)	2.274(10)	Hg...O(4)	2.673(13)
Hg-N(4)	2.299(11)	Hg...O(6)	2.720(16)
N(1)-Hg-N(2)	72.2(4)	N(2)-Hg...O(2)	123.0(3)
N(1)-Hg-N(3)	117.1(4)	N(3)-Hg...O(4)	80.1(3)
N(1)-Hg-N(4)	152.9(4)	N(3)-Hg...O(6)	78.1(3)
N(2)-Hg-N(3)	159.5(4)	N(3)-Hg...O(2)	77.4(3)
N(2)-Hg-N(4)	108.7(4)	N(4)-Hg...O(4)	120.8(3)
N(3)-Hg-N(4)	71.8(4)	N(4)-Hg...O(6)	76.9(3)
N(1)-Hg...O(4)	86.3(3)	N(4)-Hg...O(2)	81.6(3)
N(1)-Hg...O(6)	128.9(3)	O(4)...Hg...O(6)	46.3(4)
N(1)-Hg...O(2)	76.2(3)	O(4)...Hg...O(2)	140.8(5)
N(2)-Hg...O(4)	82.5(3)	O(6)...Hg...O(2)	151.4(4)
N(2)-Hg...O(6)	82.1(3)		

(b) Bond Lengths and Angles within the Bipyridyl Ligands

N(2)-C(1)	1.372(18)	N(4)-C(11)	1.368(26)
N(2)-C(5)	1.315(13)	N(4)-C(15)	1.356(17)
C(1)-C(2)	1.382(19)	C(11)-C(12)	1.420(32)
C(2)-C(3)	1.369(20)	C(12)-C(13)	1.348(35)
C(3)-C(4)	1.388(23)	C(13)-C(14)	1.434(39)
C(4)-C(5)	1.410(16)	C(14)-C(15)	1.389(25)
C(5)-C(6)	1.488(17)	C(15)-C(16)	1.456(21)
C(6)-C(7)	1.380(17)	C(16)-C(17)	1.416(23)
C(7)-C(8)	1.387(25)	C(17)-C(18)	1.374(34)
C(8)-C(9)	1.360(23)	C(18)-C(19)	1.367(32)
C(9)-C(10)	1.386(22)	C(19)-C(20)	1.403(27)
N(1)-C(6)	1.341(14)	N(3)-C(16)	1.330(16)
N(1)-C(10)	1.351(20)	N(3)-C(20)	1.368(23)
C(1)-N(2)-C(5)	119.1(10)	C(11)-N(4)-C(15)	123.9(13)
N(2)-C(1)-C(2)	122.1(12)	N(4)-C(11)-C(12)	118.7(18)
C(1)-C(2)-C(3)	119.0(15)	C(11)-C(12)-C(13)	115.5(25)
C(2)-C(3)-C(4)	119.1(13)	C(12)-C(13)-C(14)	127.7(24)
C(3)-C(4)-C(5)	119.4(11)	C(13)-C(14)-C(15)	113.0(18)
C(4)-C(5)-C(6)	121.5(10)	C(14)-C(15)-C(16)	121.3(14)
C(4)-C(5)-N(2)	121.3(12)	C(14)-C(15)-N(4)	121.2(16)
N(2)-C(5)-C(6)	117.1(9)	N(4)-C(15)-C(16)	117.5(12)
C(5)-C(6)-N(1)	116.7(9)	C(15)-C(16)-C(17)	122.6(13)
C(7)-C(6)-N(1)	121.0(12)	C(17)-C(16)-N(3)	120.5(14)
C(5)-C(6)-C(7)	122.3(10)	C(15)-C(16)-N(3)	116.9(18)
C(6)-C(7)-C(8)	118.2(12)	C(16)-C(17)-C(18)	117.4(17)
C(7)-C(8)-C(9)	120.9(15)	C(17)-C(18)-C(19)	122.9(21)
C(8)-C(9)-C(10)	118.7(17)	C(18)-C(19)-C(20)	117.5(23)
C(9)-C(10)-N(1)	120.6(13)	C(19)-C(20)-N(3)	120.3(16)
C(10)-N(1)-C(6)	120.6(10)	C(20)-N(3)-C(16)	121.3(12)

(c) Bond Lengths and Angles within the Nitrate Ions

N(5)-O(1)	1.279(35)	N(6)-O(4)	1.246(15)
N(5)-O(2)	1.215(31)	N(6)-O(5)	1.227(24)
N(5)-O(3)	1.209(52)	N(6)-O(6)	1.270(22)
O(1)-N(5)-O(2)	119.2(24)	O(4)-N(6)-O(5)	119.3(15)
O(1)-N(5)-O(3)	122.4(29)	O(4)-N(6)-O(6)	114.9(15)
O(2)-N(5)-O(3)	118.4(31)	O(5)-N(6)-O(6)	125.7(14)

Table II cont.

(d) *Hydrogen Bonded Atoms*

O(2) ... O(7)	2.908(25)	O(3) ... O(8) ¹	2.702(32)
O(7) ... O(8)	2.654(32)	O(7) ... O(8) ¹¹	2.742(44)

The Positions are Denoted as Follows:

No label x, y, z,

$$(i) \quad -x + 1, \frac{1}{2} + y, \frac{1}{2} - z \qquad (ii) \quad -x + 1, y - \frac{1}{2}, \frac{1}{2} - z$$

in *cis*-position with the S-Hg-S angle of 95.2°, while the N-Hg-N angles are 66.3° and 68.0°. ³

The mean value of 2.29 Å for the length of the Hg-N bonds in the present structure is in very good agreement with the values found previously in the structure with the tetra-coordinated mercury, i. e. 2.32 Å and 2.34 Å in the ethylenediamine¹ and phenanthroline⁴ complexes, respectively. It is larger than the Hg-N bond length in di-coordinated (~ 2.08 Å)⁸ as well as in tri-coordinated (~ 2.21 Å)⁷ mercury, but shorter than 2.47 Å, the bond length in *cis*-dithiocyanatobis(1,10-phenanthroline)mercury(II)³, as expected according to the regular increase in bond length with increasing coordination number.

The geometry of the bipyridyl ligand is in agreement with the known values.^{15,16} The pyridine rings are planar, the departure of the atomic positions from the least-squares best plane is less than the e. s. d.'s of the atomic coordinates. The pyridine rings within the given bipyridyl entity are twisted by an angle of 4.8° and 3.3° in the N(1)N(2) and N(3)N(4) ligands, respectively (Table III).

TABLE III

Equations of Least-squares Best Planes, Distances (Å) of Atoms from the Planes (in Square Brackets) and Dihedral Angles (°)

The equations are referred to orthogonal axes a, b and c*, where X, Y, Z are in Å.

Plane (1): ring N(2) to C(5)

$$-0.0843X - 0.9333Y + 0.3491Z - 3.1389 = 0$$

[N(2)0.000, C(1)-0.004, C(2)0.001, C(3)0.007, C(4)-0.011, C(5)0.007]

Plane (2): ring N(1) to C(10)

$$-0.0197X - 0.9169Y + 0.3987Z - 4.1414 = 0$$

[N(1)0.004, C(6)-0.003, C(7)-0.002, C(8)0.006, C(9)-0.005, C(10)0.000]

Plane (3): ring N(3) to C(20)

$$-0.4149X - 0.4913Y + 0.7658Z - 8.1962 = 0$$

[N(3)0.004, C(16)0.006, C(17)-0.006, C(18)-0.004, C(19)0.013, C(20)-0.014]

Plane (4): ring N(4) to C(15)

$$-0.3637X - 0.4906Y + 0.7919Z - 8.4195 = 0$$

[N(4)-0.010, C(11)0.011, C(12)-0.007, C(13)0.002, C(14)0.000, C(15)0.004]

Dihedral angles:

Planes (1) — (2)	4.8
Planes (3) — (4)	3.0

The mercury atom in such a »flat« cation is open to close intermolecular contacts with the nitrate oxygen atoms: the Hg...O(2), Hg...O(4) and Hg...O(6) distances of 2.78, 2.67 and 2.72 Å, respectively, are less than the sum of the van der Waals radii (1.50 Å⁸ or 1.55 Å¹⁷ for mercury, 1.40 Å¹⁸ for oxygen) and have to be included into the coordination of mercury, so that its effective coordination number is seven in a distorted capped octahedron.¹⁹ The water molecules do not belong to the mercury coordination sphere. They form a chain along the screw axis linking together the nitrate ions by O(2)...O(7), O(7)...O(8), O(3)...O(8)ⁱ and O(7)...O(8)ⁱⁱ hydrogen bonds of 2.91, 2.65, 2.70, and 2.74 Å, respectively.

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SAŽETAK

Kristalna i molekulska struktura bis(2,2'-bipiridil)živa(II)-nitrata dihidrata

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Kristalna struktura bis(2,2'-bipiridil)živa(II)-nitrata dihidrata određena je na osnovi 3589 nezavisnih refleksa sakupljenih na rendgenskom automatskom difraktometru i utočnjena metodom najmanjih kvadrata do faktora $R = 0.069$. Kristali su monoklinski holodrijski, prostorne grupe $P2_1/c$ i sadrže četiri formulske jedinice

u jediničnoj ćeliji veličine $a = 10,091(2)$, $b = 7,378(3)$, $c = 32,990(6)$ Å, $\beta = 105,49(3)^\circ$. Izmjerena gustoća iznosi $D_m = 1,87$ g cm⁻³, izračunata $D_x = 1,888$ g cm⁻³. Atomi dušika iz bipiridila, koji imaju funkciju helatnih liganda, stvaraju oko atoma žive spljošteni tetraedar na srednjem razmaku Hg-N od 2,29 Å. Tri atoma kisika iz nitrata na srednjem Hg...O razmaku od 2,72 Å upotpunjuju koordinaciju oko atoma žive na deformiranu oktaedarsku s jednim dodatnim vrhom. Molekule vode su izvan koordinacijske sfere atoma žive.

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