

## Structure of Bis{2'-[ $\alpha$ -(2-pyridyl)benzylidene]salicylohydrazido}nickel(II) Monohydrate [Ni(C<sub>19</sub>H<sub>14</sub>N<sub>3</sub>O<sub>2</sub>)<sub>2</sub>].H<sub>2</sub>O

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**Abstract.**  $M_r = 709.4$ , orthorhombic,  $Pbca$ ,  $a = 16.268$  (5),  $b = 19.670$  (7),  $c = 23.070$  (7) Å,  $V = 7382.2$  Å<sup>3</sup>,  $D_m = 1.28$  (1) (by flotation in xylene/chloroform),  $D_x = 1.276$  Mg m<sup>-3</sup>,  $\mu(\text{Mo } K\alpha) = 0.585$  mm<sup>-1</sup>,  $Z = 8$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $F(000) = 2944$ ,  $T = 295$  K, final  $R = 0.064$  for 2103 observed diffractometer data. In the complex the metal is surrounded by pairs of O, azo N and pyridine N atoms in a distorted octahedron; the two ligands are in *cis* positions. Ni–N(py) distances are relatively longer than the Ni–N(azo) distances, owing to the considerable  $\pi$ -backbonding power of the azo function. The two azo N atoms are *trans* [N(azo)–Ni–N(azo) = 178.2 (4)°] and the two pyridine N atoms are *cis* [N(py)–Ni–N(py) = 92.1 (4)°]. The bond lengths and angles in the ligands are as expected. The N–N and the adjacent C–N lengths are all intermediate between single- and double-bond lengths. Intramolecular hydrogen bonds stabilize the ligand while the molecules are held together by van der Waals forces. The anisotropic thermal parameters for the water O atom indicate large thermal motion.

**Introduction.** Recently, Dutta & Hossain (1984) have successfully synthesized the tridentate ligand 2'-[ $\alpha$ -(2-pyridyl)benzylidene]salicylohydrazide which has an added structural feature in having a phenolic OH group *ortho* to the hydrazone group. A number of coordination complexes of this ligand with transition and non-transition elements [Ni, Co, Cu and Zn, Cd, Mg] have also been synthesized. The present X-ray study was undertaken to establish the coordination geometry of the title complex, to ascertain the relative  $\pi$ -backbonding ability of N(py) and N(azo) towards Ni<sup>II</sup>, to see whether the tridentate ligand is planar and to study the bonding in the molecule.

**Experimental.** Title compound synthesized by refluxing an aqueous solution of nickel chloride hexahydrate with ethanolic solution of the ligand, which is the condensation product of 2-benzylpyridine and salicylohydrazide. Thin plate-shaped, deep-red crystals obtained by slow evaporation from acetone/alcohol at room temperature. Approximate lattice constants from rotation and Weissenberg photographs, accurate values

by least squares from the  $2\theta$  values of 25 reflexions. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation. Crystal  $ca$  0.25 × 0.23 × 0.20 mm. Intensity data for  $4.1 \leq 2\theta \leq 43.9^\circ$ , index range  $0 \leq h \leq 17$ ,  $0 \leq k \leq 20$ ,  $0 \leq l \leq 24$  (beyond this range no intensities observed).  $\omega$ - $2\theta$  scans. Three standard reflexions, no significant variation. 4506 independent reflexions recorded, 2103 [ $I \geq 2\sigma(I)$ ] employed for the structure determination. Lorentz-polarization corrections but no absorption correction. Approximate coordinates of the Ni atom from unsharpened three-dimensional Patterson map, positions of the remaining nonhydrogen atoms from successive Fourier syntheses. Block-diagonal least-squares refinement,  $F$  magnitudes, anisotropic thermal parameters for C, N, O and Ni; H atoms (excluding phenolic H and water H atoms) included in calculated positions (C–H = 0.95 Å) and kept fixed during the refinement; each H atom given the isotropic temperature factor of its associated C atom. A very large value of the thermal parameter of O(W) ( $B_{eq} = 21.9$  Å<sup>2</sup>) compared to those of the other O atoms is not unexpected for a loosely bound water of crystallization (the crystal loses the water molecule when placed in a desiccator). Refinement discontinued when all parameter shifts  $< \sigma$ . Final  $R = 0.064$  and  $R_w = 0.099$  for 2103 observed reflexions. Weighting scheme based on  $\sigma(F)$  given by (Seal & Ray, 1981): for  $11.03 < |F_o| \leq 12.67$ ,  $\sigma(F) = 0.24|F_o|$ ; for  $12.67 < |F_o| \leq 17.07$ ,  $\sigma(F) = 0.21|F_o|$ ; for  $17.07 < |F_o| \leq 19.88$ ,  $\sigma(F) = 0.18|F_o|$ ; for  $19.88 < |F_o| \leq 23.99$ ,  $\sigma(F) = 0.15|F_o|$ ; for  $23.99 < |F_o| \leq 29.44$ ,  $\sigma(F) = 0.13|F_o|$ ; for  $29.44 < |F_o| \leq 42.32$ ,  $\sigma(F) = 0.11|F_o|$ ; and for  $|F_o| > 42.32$ ,  $\sigma(F) = 0.09|F_o|$ . The  $|F_o| - |F_c|$  values for strong low-order reflexions were randomly positive and negative and no corrections for secondary extinction therefore applied. Phenolic H and water H atoms could not be located and were not included in the structure factor calculations.  $\Delta\rho$  in final difference map within  $\pm 0.5$  e Å<sup>-3</sup>, largest deviations in the vicinity of O(W). Final value of  $S$  1.65, a reasonable value for a model where H atoms are not refined. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on the Burroughs B 6700 computer of the Regional Computer

Table 1. Final fractional atomic coordinates ( $\times 10^4$ , for Ni  $\times 10^5$ ) with e.s.d.'s in parentheses and equivalent isotropic thermal parameters for the non-hydrogen atoms

$$B_{eq} = (B_{11} + B_{22} + B_{33})/3.$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Ni	79098 (10)	24031 (7)	56254 (7)	
N(1)	7098 (6)	1370 (4)	6296 (4)	3.6
N(2)	7838 (6)	1526 (4)	6049 (4)	3.7
N(3)	9059 (6)	1980 (5)	5470 (4)	3.8
N(4)	8377 (7)	3047 (5)	6267 (5)	4.5
N(5)	8015 (6)	3267 (5)	5196 (4)	3.5
N(6)	7759 (7)	3278 (5)	4623 (4)	4.0
O(1)	6714 (5)	2433 (4)	5951 (4)	4.2
O(2)	6066 (6)	688 (4)	6891 (4)	5.6
O(3)	7469 (6)	2143 (4)	4804 (4)	4.1
O(4)	7329 (6)	3798 (4)	3664 (4)	5.7
O(W)	3783 (14)	645 (11)	1105 (13)	21.9
C(1)	4139 (9)	1620 (8)	6916 (7)	5.8
C(2)	4322 (9)	2174 (9)	6591 (7)	6.2
C(3)	5113 (8)	2267 (7)	6358 (6)	4.8
C(4)	5716 (7)	1776 (6)	6457 (5)	3.6
C(5)	5520 (8)	1183 (6)	6789 (6)	4.3
C(6)	4730 (10)	1150 (8)	7005 (7)	6.0
C(7)	6552 (8)	1883 (6)	6219 (5)	4.0
C(8)	8438 (8)	1105 (6)	6047 (5)	3.7
C(9)	8433 (8)	414 (6)	6297 (6)	3.9
C(10)	8598 (9)	-146 (7)	5967 (7)	5.8
C(11)	8552 (11)	-811 (8)	6194 (9)	7.6
C(12)	8352 (12)	-867 (8)	6770 (9)	8.1
C(13)	8182 (11)	-338 (9)	7120 (8)	7.2
C(14)	8217 (10)	314 (8)	6869 (7)	6.3
C(15)	9168 (8)	1382 (6)	5743 (5)	3.7
C(16)	9928 (9)	1052 (7)	5724 (7)	5.6
C(17)	10567 (10)	1328 (8)	5420 (7)	6.2
C(18)	10452 (9)	1961 (8)	5149 (7)	6.0
C(19)	9685 (9)	2249 (7)	5177 (7)	5.4
C(20)	8560 (10)	2923 (7)	6821 (6)	4.9
C(21)	8930 (12)	3366 (8)	7167 (7)	6.7
C(22)	9130 (11)	3995 (8)	6965 (7)	6.9
C(23)	8950 (9)	4168 (6)	6394 (5)	4.7
C(24)	8594 (8)	3687 (6)	6046 (6)	3.7
C(25)	8373 (8)	3795 (5)	5426 (5)	3.2
C(26)	8614 (7)	4416 (6)	5097 (5)	3.5
C(27)	8403 (8)	5055 (6)	5299 (6)	4.3
C(28)	8657 (9)	5622 (6)	4994 (6)	5.2
C(29)	9123 (10)	5553 (7)	4491 (6)	5.1
C(30)	9332 (8)	4906 (7)	4297 (6)	4.8
C(31)	9079 (9)	4345 (6)	4600 (5)	4.1
C(32)	7458 (8)	2653 (6)	4474 (5)	3.8
C(33)	7089 (8)	2615 (6)	3904 (5)	4.2
C(34)	7005 (9)	3185 (7)	3527 (7)	5.3
C(35)	6620 (11)	3093 (8)	2984 (6)	6.7
C(36)	6290 (12)	2489 (9)	2848 (7)	8.2
C(37)	6367 (12)	1936 (8)	3195 (7)	7.3
C(38)	6766 (10)	1999 (7)	3716 (6)	5.4

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**Discussion.** The final atomic coordinates are given in Table 1,\* bond lengths and angles in Table 2. The asymmetric unit, consisting of one molecule, is shown

\* Lists of structure factors, anisotropic thermal parameters, least-squares-planes calculations, H-atom coordinates and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39410 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected interatomic distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) involving non-hydrogen atoms with e.s.d.'s in parentheses

Ni-N(2)	1.985 (9)	Ni-N(3)	2.077 (10)
Ni-N(4)	2.090 (11)	Ni-N(5)	1.974 (9)
Ni-O(1)	2.086 (8)	Ni-O(3)	2.090 (8)
N(1)-N(2)	1.37 (1)	N(5)-N(6)	1.39 (1)
N(1)-C(7)	1.36 (2)	N(2)-C(8)	1.28 (2)
N(3)-C(15)	1.35 (2)	N(3)-C(19)	1.33 (2)
N(4)-C(20)	1.33 (2)	N(4)-C(24)	1.40 (2)
N(5)-C(25)	1.30 (1)	N(6)-C(32)	1.37 (1)
O(1)-C(7)	1.27 (1)	O(2)-C(5)	1.34 (2)
O(3)-C(32)	1.26 (1)	O(4)-C(34)	1.35 (2)
C(1)-C(2)	1.36 (2)	C(1)-C(6)	1.35 (2)
C(2)-C(3)	1.41 (2)	C(3)-C(4)	1.40 (2)
C(4)-C(5)	1.43 (2)	C(4)-C(7)	1.48 (2)
C(5)-C(6)	1.38 (2)	C(8)-C(9)	1.48 (2)
C(8)-C(15)	1.48 (2)	C(9)-C(10)	1.37 (2)
C(9)-C(14)	1.38 (2)	C(10)-C(11)	1.41 (2)
C(11)-C(12)	1.37 (3)	C(10)-C(13)	1.35 (3)
C(13)-C(14)	1.41 (2)	C(15)-C(16)	1.40 (2)
C(16)-C(17)	1.37 (2)	C(17)-C(18)	1.41 (2)
C(18)-C(19)	1.37 (2)	C(20)-C(21)	1.33 (2)
C(21)-C(22)	1.36 (2)	C(22)-C(23)	1.39 (2)
C(23)-C(24)	1.37 (2)	C(24)-C(25)	1.49 (2)
C(25)-C(26)	1.49 (2)	C(26)-C(27)	1.38 (2)
C(26)-C(31)	1.38 (2)	C(27)-C(28)	1.38 (2)
C(28)-C(29)	1.39 (2)	C(29)-C(30)	1.39 (2)
C(30)-C(31)	1.37 (2)	C(32)-C(33)	1.45 (2)
C(33)-C(34)	1.43 (2)	C(33)-C(38)	1.39 (2)
C(34)-C(35)	1.41 (2)	C(35)-C(36)	1.34 (2)
C(36)-C(37)	1.36 (2)	C(37)-C(38)	1.37 (2)
N(2)-Ni-N(3)	77.9 (4)	N(3)-Ni-O(3)	93.1 (4)
N(2)-Ni-N(4)	101.5 (4)	N(4)-Ni-N(5)	78.6 (4)
N(2)-Ni-N(5)	178.2 (4)	N(4)-Ni-O(1)	93.8 (4)
N(2)-Ni-O(1)	78.0 (4)	N(4)-Ni-O(3)	156.2 (4)
N(2)-Ni-O(3)	102.3 (4)	N(5)-Ni-O(1)	103.7 (4)
N(3)-Ni-N(4)	92.1 (4)	N(5)-Ni-O(3)	77.6 (4)
N(3)-Ni-N(5)	100.4 (4)	O(1)-Ni-O(3)	90.8 (3)
N(3)-Ni-O(1)	155.8 (4)	Ni-N(2)-N(1)	116.9 (7)
Ni-N(5)-N(6)	117.8 (7)		
N(2)-N(1)-C(7)	111 (1)	C(15)-N(3)-C(19)	119 (1)
N(1)-N(2)-C(8)	122 (1)	C(20)-N(4)-C(24)	117 (1)
N(6)-N(5)-C(25)	121 (1)	N(5)-N(6)-C(32)	109 (1)
C(2)-C(1)-C(6)	119 (1)	C(1)-C(2)-C(3)	121 (1)
C(2)-C(3)-C(4)	119 (1)	C(3)-C(4)-C(5)	120 (1)
C(3)-C(4)-C(7)	119 (1)	C(5)-C(4)-C(7)	121 (1)
O(2)-C(5)-C(6)	123 (1)	O(2)-C(5)-C(6)	121 (1)
C(4)-C(5)-C(6)	116 (1)	C(1)-C(6)-C(5)	125 (1)
N(1)-C(7)-O(1)	124 (1)	N(1)-C(7)-C(4)	117 (1)
O(1)-C(7)-C(4)	119 (1)	N(2)-C(8)-C(9)	126 (1)
N(2)-C(8)-C(15)	112 (1)	C(9)-C(8)-C(15)	122 (1)
C(8)-C(9)-C(10)	122 (1)	C(8)-C(9)-C(14)	120 (1)
C(10)-C(9)-C(14)	118 (1)	C(9)-C(10)-C(11)	122 (1)
C(10)-C(11)-C(12)	117 (2)	C(11)-C(12)-C(13)	125 (2)
C(12)-C(13)-C(14)	117 (2)	C(9)-C(14)-C(13)	122 (1)
N(3)-C(15)-C(8)	116 (1)	N(3)-C(15)-C(16)	121 (1)
C(8)-C(15)-C(16)	124 (1)	C(15)-C(16)-C(17)	120 (1)
C(16)-C(17)-C(18)	119 (1)	C(17)-C(18)-C(19)	118 (1)
N(3)-C(19)-C(18)	124 (1)	N(4)-C(20)-C(21)	124 (1)
C(20)-C(21)-C(22)	120 (2)	C(21)-C(22)-C(23)	120 (2)
C(22)-C(23)-C(24)	118 (1)	N(4)-C(24)-C(23)	121 (1)
N(4)-C(24)-C(25)	115 (1)	C(23)-C(24)-C(25)	124 (1)
N(5)-C(25)-C(24)	113 (1)	N(5)-C(25)-C(26)	124 (1)
C(24)-C(25)-C(26)	123 (1)	C(25)-C(26)-C(27)	120 (1)
C(25)-C(26)-C(31)	119 (1)	C(27)-C(26)-C(31)	121 (1)
C(26)-C(27)-C(28)	119 (1)	C(27)-C(28)-C(29)	121 (1)
C(28)-C(29)-C(30)	119 (1)	C(29)-C(30)-C(31)	120 (1)
C(26)-C(31)-C(30)	120 (1)	N(6)-C(32)-O(3)	124 (1)
N(6)-C(32)-C(33)	115 (1)	O(3)-C(32)-C(33)	121 (1)
C(32)-C(33)-C(34)	124 (1)	C(32)-C(33)-C(38)	119 (1)
C(34)-C(33)-C(38)	117 (1)	O(4)-C(34)-C(33)	121 (1)
O(4)-C(34)-C(35)	120 (1)	C(33)-C(34)-C(35)	119 (1)
C(34)-C(35)-C(36)	120 (2)	C(35)-C(36)-C(37)	122 (2)
C(36)-C(37)-C(38)	119 (2)	C(33)-C(38)-C(37)	122 (1)

in Fig. 1. Since the intensities were only recorded up to  $2\theta = 43.9^\circ$ , the e.s.d.'s in atomic coordinates and in bond lengths and angles are rather large. However, the main interest of this crystal structure is to establish the

coordination geometry of the complex. 2'-[ $\alpha$ -(2-Pyridyl)benzylidene]salicylhydrazide is a tridentate ligand; the enolic O, azo N and pyridine N atoms are coordinated to Ni to form two five-membered chelate rings. In the coordination octahedron, the Ni—O distances are normal and the N(py) atoms are *cis* [N(3)—Ni—N(4) = 92.1 (4)°] and the two N(azo) atoms are *trans* [N(2)—Ni—N(5) = 178.2 (4)°]. The average bite angle of the five-membered chelate ring is ~78°, thus introducing a considerable distortion in the octahedron. The ligands in the octahedron are in *cis* positions and are chemically identical but crystallographically different.

A consistent and interpretable pattern of Ni to N distances appears. Where pyridine N atoms are involved, the bond lengths of 2.077 (10) and 2.090 (11) Å (see Table 2) are similar to that found in (bis{2-[(2-pyridyl)methylamino]ethyl} disulfide)-bromonickel(II) perchlorate (Warner, Kadooka & Seff, 1975). Noticeably shorter Ni—N distances [1.985 (9) and 1.974 (9) Å] occur for azo N. Pyridine N is normally a better donor than azo N. Evidently the  $\pi$ -backbonding power of the azo function is very considerable, resulting in the net ( $\sigma + \pi$ ) bond order being larger in Ni—N(azo) than in Ni—N(py). A similar result has been obtained in two isomers of dichlorobis(2-phenylazopyridine)ruthenium(II) (Seal & Ray, 1984).

The four five-membered chelate rings are planar while individual ligands are non-planar. Excluding the phenyl ring atoms, the mean planes through the first and second ligand atoms are almost perpendicular to

each other; C(2) and O(1) show the greatest deviations from the first plane [0.10 (2) and -0.10 (1) Å], and C(36) from the second plane [-0.27 (2) Å]. The angle between these two planes is 83 (1)°. The mean geometry of the pyridine rings is also similar to that found in other pyridyl groups and in metal complexes of di-2-pyridyl disulfide (Raghavan & Seff, 1977; Warner, Otterson & Seff, 1974), with a similar bond angle at N. The small C—N—C angle is due to the absence of an extra-annular H atom, as suggested by Singh (1965). The two pyridine rings are reasonably planar exhibiting an interplanar angle of 82 (1)°. The N(3) ring is almost perpendicular [85 (1)°] while the N(4) ring is almost parallel [6 (1)°] with respect to the best plane defined by N(4), N(5), Ni, O(3) and N(2). The phenyl rings are essentially planar and almost parallel to each other [dihedral angle 18 (1)°] and are twisted out of the plane of the associated chelate rings as indicated by the N(2)—C(8)—C(9)—C(14) and N(5)—C(25)—C(26)—C(31) torsion angles of 55 (1) and 52 (1)° respectively. A coplanar arrangement of the phenyl and pyridine rings is impossible owing to steric effects. The salicyloyl rings are strictly planar and are approximately parallel to the pyridine rings within the same ligand [dihedral angles 0.3 (10) and 11 (1)° with the N(3) and N(4) pyridine rings respectively].

The N—N distances are similar to those found in Ni<sub>3</sub>(trz)<sub>6</sub>(NO<sub>3</sub>)<sub>6</sub>(H<sub>2</sub>O)<sub>8</sub>\* (Reimann & Zocchi, 1971) and are intermediate between single- and double-bond lengths indicating that the azo group is involved in resonance. A number of factors such as the electron-withdrawing power of the metal, resonance possibility involving the ligand, the nature of the ligands and different coordination polyhedra may all influence the character of the azo bond. It seems that the double-

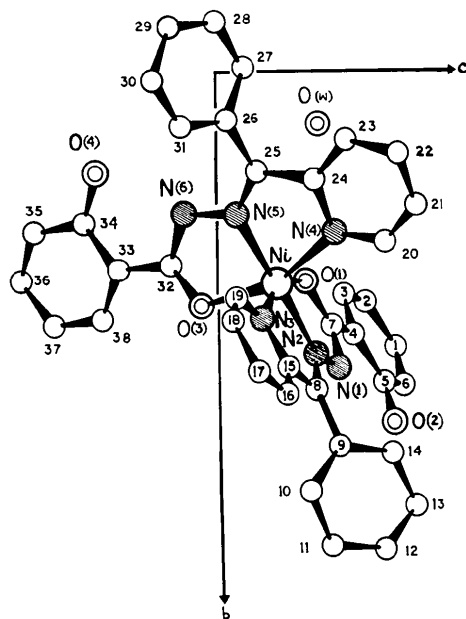


Fig. 1. View of the title compound along *a* and the atom-numbering scheme; H atoms have been omitted.

\* Abbreviations: trz = 1,2,4-triazolo, tan = 1-(2-thiazolylazo)-2-naphtholato.

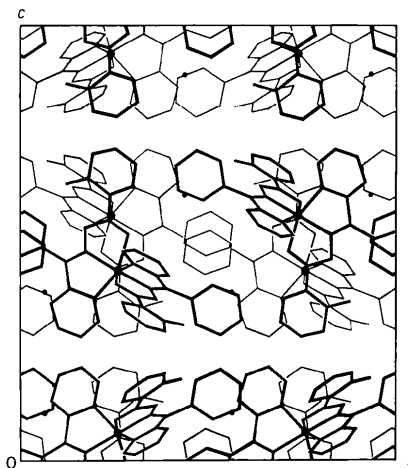


Fig. 2. Packing diagram. H atoms have been omitted for clarity.

bond character of the azo group is best preserved in the Ni(tan)<sub>2</sub> complex (Kurahashi, 1974b) [N—N = 1.26 (6) Å] while a very long N—N length exists in PdCl(tan) [1.39 (3) Å (Kurahashi, 1974a)], similar to those in the present structure.

The amide H atoms originally attached to N(1) and N(6) help to enolize the keto group so that the C—N and C—O bonds show partial double-bond character due to the delocalization of  $\pi$  electrons in this group.

There is one loosely bound water molecule of crystallization which is not hydrogen bonded. The H atoms attached to O(2) and O(4) are not well resolved; but judging from the O...N distances, two intramolecular hydrogen bonds may be assumed to exist between O(2) and N(1), and O(4) and N(6) at distances of 2.55 (2) and 2.53 (2) Å respectively. Fig. 2 illustrates the packing of the molecules in the unit cell; the structure consists essentially of a series of layers parallel to *ab* and the molecules are displaced with respect to one another to relieve short intermolecular contacts. Since all other H atoms are attached to C, it is unlikely that they are involved in hydrogen bonding; therefore, intermolecular bonding is probably of the van der Waals type. There are no unusually short intermolecular contacts so that packing forces are unlikely to influence the geometry of the molecule to an appreciable extent.

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## Organomercury Medicinal Chemistry. Synthesis and Structure of a ( $\beta$ -Methoxyethyl)mercury(II) Derivative of N(7)-Deprotonated Theophylline,\* [Hg(C<sub>3</sub>H<sub>7</sub>O)(C<sub>7</sub>H<sub>7</sub>N<sub>4</sub>O<sub>2</sub>)]

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**Abstract.**  $M_r = 438.84$ , monoclinic,  $P2_1/c$ ,  $a = 8.305$  (2),  $b = 9.349$  (3),  $c = 16.370$  (4) Å,  $\beta = 97.37$  (1)°,  $U = 1260.5$  Å<sup>3</sup>,  $Z = 4$ , density not measured as material very soluble,  $D_x = 2.31$  Mg m<sup>-3</sup>,

$\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 12.176$  mm<sup>-1</sup>,  $F(000) = 824$ ,  $T = 293$  K. Final  $R = 0.057$  for 1461 observed counter reflections. The structure consists of molecules of [Hg(CH<sub>2</sub>CH<sub>2</sub>OCH<sub>3</sub>)(C<sub>7</sub>H<sub>7</sub>N<sub>4</sub>O<sub>2</sub>)] linked into a three-dimensional network by weak intermolecular Hg...O (methoxyethyl) and Hg...N(9) (theophylline) interactions of 2.89 (1) and 2.91 (1) Å respectively.

\* (2-Methoxyethyl)(theophyllinato-*N*<sup>7</sup>)mercury(II).

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