Carruthers, J. R., Watkin, D. J. & Betteridge, P. W. (1990). *CRYSTALS User Guide*. Chemical Crystallography Laboratory, Univ. of Oxford, England.
Day, V. W., Fredrich, M. F., Klemperer, W. G. & Shum, W. (1977). *J. Am. Chem. Soc.* **99**, 952-953.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Larson, A. C. (1967). *Acta Cryst.* **23**, 664-665.
Masters, A. F., Gheller, S. F., Brownlee, R. T. C., O'Connor, M. J. & Wedd, A. G. (1980). *Inorg. Chem.* **19**, 3866-3868.
Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Strouse, C. E. (1982). *UCLA Crystallographic Software*. Univ. of California, Los Angeles, USA.

Acta Cryst. (1993). C**49**, 1902–1905

Structure of Diaquatris(5,7-dimethyl[1,2,4]-triazolo[1,5-*a*]pyrimidine-N^3)copper(II) Perchlorate Dihydrate

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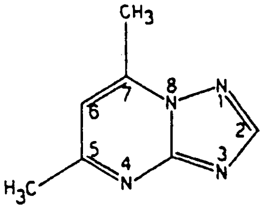
Abstract

The synthesis and crystal structure of the title compound obtained by reaction of copper perchlorate with 5,7-dimethyl[1,2,4]triazolo[1,5-*a*]pyrimidine (dmtp) are reported. The structure consists of $[Cu(dmtp)_3(H_2O)_2]^{2+}$ coordination cations, ClO_4^- anions and interstitial water molecules. The Cu atom is coordinated by three N(3) atoms of triazolopyrimidine ligands [Cu—N = 1.985 (5), 1.993 (4), 2.024 (4) Å] and two aqueous O atoms [Cu—O = 1.979 (4), 2.245 (4) Å]. The Cu polyhedron is a trigonal bipyramid. This compound is the first dmtp complex with a ClO_4 anion and the first mononuclear complex involving three dmtp ligands, and whose crystal structure has been solved by X-ray diffraction.

Comment

Transition-metal complexes of purine analogs have been studied in order to elucidate the role of such metal ions in interactions with nucleic acids. Among these purine analogs, the [1,2,4]triazolo[1,5-*a*]-pyrimidine ring (tp) and its 5,7-dimethyl derivative (dmtp) have been studied as ligands (Dillen, Lenstra, Haasnoot & Reedijk, 1983; Biagini Cingi, Manotti Lanfredi, Tiripicchio, Haasnoot & Reedijk, 1983, 1984; Biagini Cingi, Manotti Lanfredi, Tiripicchio, Reedijk & Haasnoot, 1986; Biagini Cingi, Manotti Lanfredi, Tiripicchio, Cornelissen, Haasnoot & Reedijk, 1986, 1987; Bamidele Sanni, Smits, Beurskens, Haasnoot, Schild & Lenstra, 1986). Since both ligands have no acidic H atoms on the ring N atoms, the isolated metal compounds involve negative species such as ligands or counteranions.



dmtp

Different copper(II) complexes of tp and dmtp as ligands have been described in the literature and characterized by X-ray diffraction methods. Thus, in $[Cu(tp)_2(H_2O)Br_2]$ (van Albada, de Graaff, Haasnoot, Schild & Reedijk, 1991) the tp ligand is monodentate through N(3), whereas in $[Cu(tp)_2(NCS)_2]$ and $[Cu(tp)_2Cl_2]$ (Biangini Cingi *et al.*, 1987) the tp ligand is bidentate through N(3) and N(1). Nevertheless, in both compounds the Cu···N(1) contacts are long: 2.633 (6) and 2.692 (4) Å. In the complexes $[Cu(dmtp)_2(NCS)_2]$ (Biagini Cingi *et al.*, 1983), $[Cu(dmtp)_4(H_2O)_2](PF_6)_2$ (Favre, Haasnoot & Reedijk, 1986) and $[Cu(NCS)_2(dmtp)_2]_2$ (Haasnoot, Driessen & Reedijk, 1984) N(3) is the sole metal-binding site, whereas in $[Cu_4(dmtp)_4Cl_2][Cu_2Cl_4]$ (Haasnoot, Favre, Hinrichs & Reedijk, 1988) the dmtp acts as a bridging bidentate ligand *via* N(3) and N(4).

As part of our work on the coordination chemistry of purine analogs (Sirera, Romero, Salas, Sanchez & Moreno, 1991), we now report a new structure with the dmtp ligand.

The *PLUTO* drawing (Motherwell & Clegg, 1978) shows the asymmetric unit (Fig.1) with the complex cation $[Cu(C_7H_8N_4)_3(H_2O)_2]^{2+}$, the two ClO_4^- anions and the two non-coordinated O(*w*3) and O(*w*4) water molecules. The Cu atom is coordinated by three dmtp N(3) atoms and the two aqueous O atoms. The polyhedron is a trigonal bipyramid (Fig. 2); the base is defined by Cu, N(3*A*), N(3*B*) and O(*w*2); the apices are N(3*C*) and O(*w*1).

The dmtp ligand is as monodentate through N(3). The Cu—N(3) distances of 1.985 (5), 1.993 (4) and

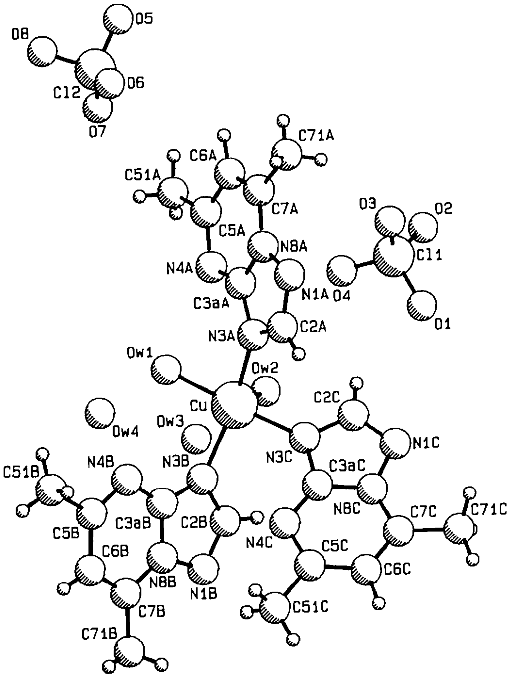


Fig. 1. The asymmetric unit with the $[Cu(dmtp)_3(H_2O)_2]^{2+}$ complex cation, the two ClO_4 anions and the two non-coordinated water molecules.

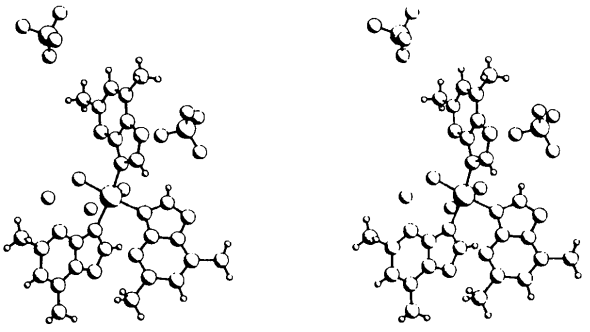


Fig. 2. Stereoscopic view.

2.024 (4) Å are in agreement with those found with the tp, mtp or dmtp ligands in other copper complexes: 1.918 (8) in $[Cu_4(dmtp)_4Cl_2][Cu_2Cl_4]$ (Haasnoot *et al.*, 1988); 1.969 (3) in $[Cu(tp)_2(H_2O)Br_2]$ (van Albada *et al.*, 1991); 1.975 (2) in $[Cu(NCS)_2(6\text{-}mtp)_2]$ (Cornelissen, de Graaff, Haasnoot, Prins, Reedijk & Haasnoot, 1989); 1.995 (2) and 2.058 (2) in β-$[Cu(NCS)_2(dmtp)_2]_2$ (Haasnoot *et al.*, 1984); 2.000 (7) and 2.019 (8) in $[Cu(dmtp)_2(NCS)_2]$ (Biagini Cingi *et al.*, 1983); 2.019 (3) and 2.050 (3) in $[Cu(dmtp)_4(H_2O)_2](PF_6)_2$ (Favre *et al.*, 1986); 2.021 (4) in $[Cu(tp)_2Cl_2]$ (Biagini Cingi *et al.*, 1987); 2.035 (5) in $[Cu(tp)_2(NCS)_2]$ (Biagini Cingi *et al.*, 1987); 2.06 (2) in $[CuHg(\mu\text{-}SCN)_3(dmtp)_2]$ (Biagini Cingi *et al.*, 1986); 2.020 (4) Å in $[Cu(NCS)_2(5\text{-}mtp)_2]$ (Cornelissen *et al.*, 1989).

One of the two Cu—O bonds is short [1.979 (4) Å], the other being much longer [2.245 (4) Å].

This compound is the first dmtp complex with the ClO_4^- anion that has been solved by X-ray diffraction. The shorter contacts involve water O atoms: $O(3)\cdots O(w4^{i}) = 2.783$, $O(5)\cdots O(w4^{ii}) = 2.935$ and $O(8)\cdots O(w4^{ii}) = 3.074$ Å [symmetry code: (i) $x-1, y, z$; (ii) $1-x, -y, 1-z$. Another copper(II) perchlorate complex of dmtp, $Cu(dmtp)_{3.67}(ClO_4)_2(H_2O)_{2.67}$, has been synthesized and characterized (Favre *et al.*, 1986). These authors indicate in a preliminary X-ray study on this complex that one third of the copper ions appears to be six-coordinated by four dmtp ligands and two water molecules, having the same coordination geometry as $[Cu(dmtp)_4(H_2O)_2](PF_6)_2$ (Favre *et al.*, 1986), whereas two thirds of the copper ions are five-coordinated, one third being surrounded by four equatorial dmtp ligands and one axial water molecule and one third by three dmtp ligands and two water molecules, as in our case.

On the other hand, $[Cu(dmtp)_3(H_2O)_2](ClO_4)_2.2H_2O$ is the second mononuclear complex involving three dmtp ligands. Another example of coordination by three dmtp ligands is the heterotetranuclear complex $[CoHg(dmtp)_3(SCN)_4(H_2O)]_2$ (Biagini Cingi *et al.*, 1984).

Experimental

Crystal data

$[Cu(C_7H_8N_4)_3(H_2O)_2]$-$(ClO_4)_2.2H_2O$
$M_r = 779.0$
Triclinic
$P\bar{1}$
$a = 11.176\ (2)$ Å
$b = 11.879\ (2)$ Å
$c = 14.026\ (2)$ Å
$\alpha = 96.39\ (1)°$
$\beta = 92.84\ (1)°$
$\gamma = 112.80\ (1)°$
$V = 1697.1\ (8)$ Å^3
$Z = 2$
$D_x = 1.53$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 12.2\text{-}19.0°$
$\mu = 0.9$ mm^{-1}
$T = 295$ K
Plate
0.45 × 0.40 × 0.15 mm
Blue

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-5/3θ scans
Absorption correction: None
10 244 measured reflections
10 244 independent reflections
5055 observed reflections $[I > 3\sigma(I)]$
$\theta_{max} = 30°$
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity variation: 3.2%

Refinement

Refinement on F
Final $R = 0.054$
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.8$

$wR = 0.062$
$S = 1.6$
5055 reflections
506 parameters
Only coordinates of H atoms refined

$\Delta\rho_{\max} = 1.3$ e Å^{-3}
$\Delta\rho_{\min} = -0.9$ e Å^{-3}
Atomic scattering factors from *SDP* (B. A. Frenz & Associates, Inc., 1982)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Cu	0.71822 (6)	0.49513 (6)	0.22950 (5)	2.81 (1)
N(1A)	0.4226 (4)	0.5369 (4)	0.3887 (4)	4.0 (1)
C(2A)	0.5266 (5)	0.5731 (5)	0.3410 (4)	3.9 (1)
N(3A)	0.5689 (4)	0.4836 (4)	0.3092 (3)	3.14 (9)
C(3aA)	0.4852 (4)	0.3824 (4)	0.3403 (4)	3.0 (1)
N(4A)	0.4846 (4)	0.2702 (4)	0.3304 (3)	3.5 (1)
C(5A)	0.3869 (5)	0.1853 (5)	0.3667 (4)	4.1 (1)
C(6A)	0.2909 (5)	0.2128 (5)	0.4127 (4)	4.2 (1)
C(7A)	0.2937 (5)	0.3284 (5)	0.4236 (4)	3.9 (1)
N(8A)	0.3956 (4)	0.4138 (4)	0.3868 (3)	3.3 (1)
C(51A)	0.3842 (7)	0.0581 (6)	0.3570 (6)	6.0 (2)
C(71A)	0.1982 (6)	0.3701 (7)	0.4693 (5)	5.7 (2)
N(1B)	1.0645 (4)	0.6290 (4)	0.1015 (3)	3.3 (1)
C(2B)	0.9390 (4)	0.5662 (5)	0.1040 (4)	3.2 (1)
N(3B)	0.8998 (3)	0.5465 (4)	0.1925 (3)	2.81 (9)
C(3aB)	1.0107 (4)	0.6026 (4)	0.2520 (4)	2.7 (1)
N(4B)	1.0256 (4)	0.6102 (4)	0.3477 (3)	3.4 (1)
C(5B)	1.1466 (5)	0.6712 (5)	0.3878 (4)	3.7 (1)
C(6B)	1.2529 (5)	0.7234 (5)	0.3337 (4)	3.9 (1)
C(7B)	1.2361 (4)	0.7148 (4)	0.2369 (4)	3.4 (1)
N(8B)	1.1101 (4)	0.6522 (4)	0.1974 (3)	2.85 (9)
C(51B)	1.1696 (7)	0.6827 (7)	0.4947 (5)	5.9 (2)
C(71B)	1.3376 (5)	0.7635 (6)	0.1711 (5)	5.2 (2)
N(1C)	0.5699 (4)	0.6857 (4)	0.0614 (3)	3.25 (9)
C(2C)	0.5725 (4)	0.5980 (4)	0.1089 (4)	3.2 (1)
N(3C)	0.6873 (4)	0.6194 (4)	0.1597 (3)	2.99 (9)
C(3aC)	0.7656 (4)	0.7327 (4)	0.1425 (4)	2.8 (1)
N(4C)	0.8902 (4)	0.7993 (4)	0.1757 (3)	3.4 (1)
C(5C)	0.9415 (5)	0.9115 (5)	0.1484 (5)	4.1 (1)
C(6C)	0.8707 (5)	0.9565 (5)	0.0891 (5)	4.1 (1)
C(7C)	0.7447 (5)	0.8866 (5)	0.0556 (4)	3.7 (1)
N(8C)	0.6953 (4)	0.7730 (3)	0.0833 (3)	2.89 (9)
C(51C)	1.0792 (7)	0.9883 (7)	0.1849 (6)	6.8 (2)
C(71C)	0.6551 (6)	0.9232 (5)	−0.0052 (5)	5.2 (2)
Cl(1)	0.2033 (1)	0.3564 (1)	0.1376 (1)	3.79 (3)
O(1)	0.2602 (5)	0.4610 (4)	0.0897 (4)	7.3 (1)
O(2)	0.0969 (4)	0.2658 (4)	0.0759 (4)	5.8 (1)
O(3)	0.1578 (5)	0.3923 (5)	0.2238 (4)	7.8 (2)
O(4)	0.3004 (4)	0.3124 (4)	0.1598 (4)	7.1 (1)
Cl(2)	0.2887 (2)	0.0352 (2)	0.6567 (2)	8.01 (6)
O(5)	0.1570 (7)	−0.0397 (7)	0.6516 (8)	13.7 (3)
O(6)	0.309 (1)	0.1454 (6)	0.6895 (8)	25.0 (3)
O(7)	0.3486 (8)	0.0240 (8)	0.5817 (6)	16.6 (3)
O(8)	0.332 (1)	−0.013 (1)	0.7312 (8)	22.2 (6)
O(w1)	0.7411 (3)	0.3669 (3)	0.2982 (3)	3.68 (8)
O(w2)	0.6246 (4)	0.3480 (3)	0.1022 (3)	4.24 (9)
O(w3)	0.7924 (4)	0.2392 (4)	0.0511 (3)	4.9 (1)
O(w4)	0.8975 (4)	0.2528 (4)	0.2445 (4)	5.7 (1)

Table 2. *Geometric parameters* (Å, °)

Cu—N(3A)	2.024 (4)	C(3aB)—N(8B)	1.358 (6)
Cu—N(3B)	1.993 (4)	N(4B)—C(5B)	1.321 (6)
Cu—N(3C)	1.985 (5)	C(5B)—C(6B)	1.413 (8)
Cu—O(w1)	1.979 (4)	C(5B)—C(51B)	1.491 (9)
Cu—O(w2)	2.245 (4)	C(6B)—C(7B)	1.347 (9)
N(1A)—C(2A)	1.318 (7)	C(7B)—N(8B)	1.364 (5)
N(1A)—N(8A)	1.371 (7)	Cl(2)—O(5)	1.387 (7)
C(2A)—N(3A)	1.363 (8)	Cl(2)—O(6)	1.266 (8)
N(3A)—C(3aA)	1.340 (6)	Cl(2)—O(7)	1.297 (9)
C(3aA)—N(4A)	1.323 (7)	C(7B)—C(71B)	1.479 (8)
C(3aA)—N(8A)	1.365 (7)	N(1C)—C(2C)	1.307 (8)
N(4A)—C(5A)	1.336 (7)	N(1C)—N(8C)	1.376 (5)
C(5A)—C(6A)	1.403 (9)	C(2C)—N(3C)	1.354 (6)
C(5A)—C(51A)	1.49 (1)	N(3C)—C(3aC)	1.347 (6)
Cl(1)—O(2)	1.421 (4)	C(3aC)—N(4C)	1.335 (5)
Cl(1)—O(3)	1.420 (6)	C(3aC)—N(8C)	1.361 (7)
Cl(1)—O(4)	1.411 (6)	N(4C)—C(5C)	1.339 (7)
C(6A)—C(7A)	1.352 (9)	C(5C)—C(6C)	1.40 (1)
C(7A)—N(8A)	1.369 (6)	C(5C)—C(51C)	1.480 (8)
C(7A)—C(71A)	1.48 (1)	C(6C)—C(7C)	1.355 (7)
N(1B)—C(2B)	1.313 (6)	C(7C)—N(8C)	1.355 (7)
N(1B)—N(8B)	1.374 (6)	C(7C)—C(71C)	1.50 (1)
C(2B)—N(3B)	1.352 (7)	Cl(1)—O(1)	1.419 (5)
N(3B)—C(3aB)	1.345 (5)	Cl(2)—O(8)	1.39 (1)
C(3aB)—N(4B)	1.334 (7)		
N(3A)—Cu—N(3B)	159.8 (1)	C(6B)—C(5B)—C(51B)	119.7 (5)
N(3A)—Cu—N(3C)	90.6 (2)	C(5B)—C(6B)—C(7B)	121.4 (4)
N(3A)—Cu—O(w1)	87.7 (2)	C(6B)—C(7B)—N(8B)	114.6 (5)
N(3A)—Cu—O(w2)	104.0 (1)	C(6B)—C(7B)—C(71B)	127.4 (4)
N(3B)—Cu—N(3C)	91.9 (2)	N(8B)—C(7B)—C(71B)	118.0 (5)
N(3B)—Cu—O(w1)	90.2 (2)	N(1B)—N(8B)—C(3aB)	110.5 (3)
N(3B)—Cu—O(w2)	96.0 (1)	N(1B)—N(8B)—C(7B)	127.3 (4)
N(3C)—Cu—O(w1)	177.6 (1)	C(3aB)—N(8B)—C(7B)	122.2 (4)
N(3C)—Cu—O(w2)	91.1 (2)	C(2C)—N(1C)—N(8C)	101.9 (4)
O(w1)—Cu—O(w2)	87.6 (1)	N(1C)—C(2C)—N(3C)	116.2 (4)
C(2A)—N(1A)—N(8A)	101.8 (5)	Cu—N(3C)—C(2C)	123.4 (3)
N(1A)—C(2A)—N(3A)	115.6 (5)	Cu—N(3C)—C(3aC)	133.0 (3)
Cu—N(3A)—C(2A)	129.2 (3)	C(2C)—N(3C)—C(3aC)	103.4 (4)
Cu—N(3A)—C(3aA)	127.0 (4)	N(3C)—C(3aC)—N(4C)	128.7 (5)
C(2A)—N(3A)—C(3aA)	103.8 (4)	N(3C)—C(3aC)—N(8C)	108.1 (4)
N(3A)—C(3aA)—N(4A)	128.4 (5)	N(4C)—C(3aC)—N(8C)	123.2 (4)
N(3A)—C(3aA)—N(8A)	107.9 (5)	C(3aC)—N(4C)—C(5C)	115.1 (5)
N(4A)—C(3aA)—N(8A)	123.7 (4)	N(4C)—C(5C)—C(6C)	122.9 (4)
C(3aA)—N(4A)—C(5A)	115.7 (5)	N(4C)—C(5C)—C(51C)	116.5 (6)
N(4A)—C(5A)—C(6A)	122.4 (6)	C(6C)—C(5C)—C(51C)	120.6 (6)
N(4A)—C(5A)—C(51A)	116.9 (6)	C(5C)—C(6C)—C(7C)	120.8 (5)
C(6A)—C(5A)—C(51A)	120.7 (5)	C(6C)—C(7C)—N(8C)	115.2 (6)
C(5A)—C(6A)—C(7A)	121.3 (5)	C(6C)—C(7C)—C(71C)	127.1 (5)
C(6A)—C(7A)—N(8A)	115.1 (6)	N(8C)—C(7C)—C(71C)	117.7 (4)
C(6A)—C(7A)—C(71A)	127.0 (5)	N(1C)—N(8C)—C(3aC)	110.5 (4)
N(8A)—C(7A)—C(71A)	117.9 (6)	N(1C)—N(8C)—C(7C)	126.8 (5)
N(1A)—N(8A)—C(3aA)	110.9 (4)	C(3aC)—N(8C)—C(7C)	122.7 (4)
N(1A)—N(8A)—C(7A)	127.3 (5)	O(1)—Cl(1)—O(2)	108.8 (3)
C(3aA)—N(8A)—C(7A)	121.8 (5)	O(1)—Cl(1)—O(3)	109.0 (4)
C(2B)—N(1B)—N(8B)	101.9 (4)	O(1)—Cl(1)—O(4)	108.0 (3)
N(1B)—C(2B)—N(3B)	115.7 (4)	O(2)—Cl(1)—O(3)	109.7 (3)
Cu—N(3B)—C(2B)	127.0 (3)	O(2)—Cl(1)—O(4)	111.4 (3)
Cu—N(3B)—C(3aB)	126.7 (3)	O(3)—Cl(1)—O(4)	109.9 (4)
C(2B)—N(3B)—C(3aB)	103.7 (4)	O(5)—Cl(2)—O(6)	110.4 (6)
N(3B)—C(3aB)—N(4B)	127.9 (4)	O(5)—Cl(2)—O(7)	117.0 (5)
N(3B)—C(3aB)—N(8B)	108.0 (4)	O(5)—Cl(2)—O(8)	97.7 (6)
N(4B)—C(3aB)—N(8B)	124.1 (4)	O(6)—Cl(2)—O(7)	114.5 (6)
C(3aB)—N(4B)—C(5B)	114.9 (4)	O(6)—Cl(2)—O(8)	106.4 (8)
N(4B)—C(5B)—C(6B)	122.9 (5)	O(7)—Cl(2)—O(8)	109.0 (7)
N(4B)—C(5B)—C(51B)	117.4 (5)		

Diaquatris(5,7-dimethyl[1,2,4]triazolo[1,5-*a*]pyrimidine)copper(II) perchlorate dihydrate was prepared by slow evaporation at room temperature of an aqueous solution containing 2 mmol of dmtp and 2 mmol of $Cu(ClO_4)_2.6H_2O$ in 15 ml of water. Crystals of $[Cu(dmtp)_3(H_2O)_2](ClO_4)_2.2H_2O$ were obtained and dried in air.

Computing used the Enraf-Nonius *SDP* system (B. A. Frenz & Associates, Inc., 1982), on a DEC MicroVAX II computer at the Centre de Diffractométrie Automatique, Université Lyon I.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71276 (92 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1025]

References

Albada, G. A. van, de Graaff, R. A. G., Haasnoot, J. G., Schild, J. & Reedijk, J. (1991). *Acta Cryst.* C**47**, 946–949.

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package.* College Station, Texas, USA.

Bamidele Sanni, S., Smits, J. M. M., Beurskens, P. T., Haasnoot, J. G., Schild, J. & Lenstra, A. T. H. (1986). *J. Crystallogr. Spectrosc. Res.* **16**, 823–832.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Cornelissen, J. P., Haasnoot, J. G. & Reedijk, J. (1986). *Acta Cryst.* C**42**, 1296–1298.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Cornelissen, J. P., Haasnoot, J. G. & Reedijk, J. (1987). *Inorg. Chim. Acta*, **129**, 217–222.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Haasnoot, J. G. & Reedijk, J. (1983). *Inorg. Chim. Acta*, **72**, 81–88.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Haasnoot, J. G. & Reedijk, J. (1984). *Inorg. Chim. Acta*, **86**, 137–143.

Biagini Cingi, M., Manotti Lanfredi, A. M., Tiripicchio, A., Reedijk, J. & Haasnoot, J. (1986). *Acta Cryst.* C**42**, 427–430.

Cornelissen, J. P., de Graaff, R. A. G., Haasnoot, J. G., Prins, R., Reedijk, J., Biagini Cingi, M., Manotti Lanfredi, A. M. & Tiripicchio, A. (1989). *Polyhedron*, **8**, 2313–2320.

Dillen, J., Lenstra, A. T. H., Haasnoot, J. G. & Reedijk, J. (1983). *Polyhedron*, **2**, 195–201.

Favre, T. L. F., Haasnoot, J. G. & Reedijk, J. (1986). *Polyhedron*, **5**, 1405–1411.

Haasnoot, J. G., Driessen, W. L. & Reedijk, J. (1984). *Inorg. Chem.* **23**, 2803–2807.

Haasnoot, J. G., Favre, T. L. F., Hinrichs, W. & Reedijk, J. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 856–858.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.

Sierra, R., Romero, M. A., Salas, J. M., Sanchez, M. P. & Moreno, M. N. (1991). *J. Inorg. Biochem.* **43**, 229.

Acta Cryst. (1993). C**49**, 1905–1906

An *N,N'*-Bis(salicylidene)-1,3-propanediamine–Nickel Complex

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Abstract

In the crystal structure of this Ni^{II} complex with bis(salicylidene)-1,3-diaminopropane, aqua{2,2'-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-nickel(II), nickel is five-coordinate with a water molecule at the apex of a square pyramid. The complex has symmetry *m* and the two Schiff base moieties, which in themselves are planar, are inclined at 38.6 (4)° to each other, forming a shallow umbrella form. The configuration at the N atoms is planar, as would be expected of sp^2 hybridization.

Comment

Metal derivatives of Schiff bases have been extensively studied and copper(II) and nickel(II) complexes have played a major role in both synthesis and structural research. Copper(II) and nickel(II) react with tridentate anionic Schiff bases, giving dimerized complexes with square-planar configurations (Maggio, Pizzino & Romano, 1974). *N*-(2-Hydroxyphenyl)salicylideneamine reacts with the nickel(II) ion giving a pseudo-octahedral complex by polymerization. The present study concerns the complex formed when nickel(II) reacts with bis(salicylidene)-1,3-diaminopropane.

The two Schiff base moieties containing O1, N1, C1, C2, C3, C4, C5, C6, C7 and O1*A*, N1*A*, C1*A*, C2*A*, C3*A*, C4*A*, C5*A*, C6*A*, C7*A*, which in themselves are planar [maximum deviation from planes being 0.042 (6) Å], are inclined at 38.6 (4)° to each other, giving a shallow umbrella form as reported in other similar structures. The angles at the C atoms of the propane bridge, C8 112.8 (5) and C9 115.4 (7)°, indicate a small deviation from a regular tetrahedral configuration. The water molecule contributes to the molecular packing through hydrogen bridges.

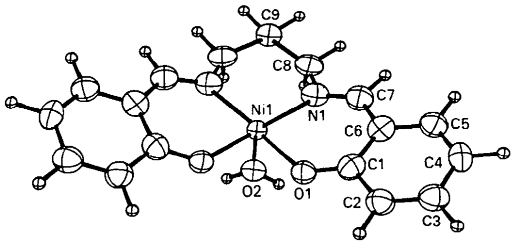

Fig. 1. Molecular structure of the title compound showing the numbering scheme and 50% probability thermal ellipsoids for the non-H atoms.

Experimental

Crystal data

$[Ni(C_{17}H_{16}N_2O_2)(H_2O)]$
M_r = 357.04
Orthorhombic
Pbnm
a = 7.539 (1) Å
b = 8.871 (1) Å
c = 24.108 (10) Å

Mo *Kα* radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 9–32°
μ = 1.22 mm^{-1}
T = 293 K