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References

ABRAHAMS, S. C. & KEVE, E. T. (1971). *Acta Cryst.* A**27**, 157–165.

CULLEN, D. L. & LINGAFELTER, E. C. (1971). *Inorg. Chem.* **10**, 1264–1268.

HARROWFIELD, B. V., DEMPSTER, A. J., FREEMAN, T. E. & PILBROW, J. R. (1973). *J. Phys.* (*C*): *Solid State Phys.* **6**, 2058–2065.

HARROWFIELD, B. V. & PILBROW, J. R. (1973). *J. Phys.* (*C*): *Solid State Phys.* **6**, 755–766.

JOESTEN, M. D., TAKAGI, S. & VENABLE, J. H. (1975). *Chem. Phys. Lett.* **36**, 536–538.

LENHERT, P. G. (1975). *J. Appl. Cryst.* **8**, 568–570.

MILLER, P. T., LENHERT, P. G. & JOESTEN, M. D. (1972). *Inorg. Chem.* **11**, 2221–2227.

REINEN, D., FRIEBEL, C. & REETZ, K. P. (1972). *J. Solid State Chem.* **4**, 103–114.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1975*a*). *J. Amer. Chem. Soc.* **97**, 444–445.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1975*b*). *Acta Cryst.* B**31**, 596–598.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1975*c*). *Acta Cryst.* B**31**, 1968–1970.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1975*d*). *Acta Cryst.* B**31**, 1970–1972.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1975*e*). Unpublished results.

TAKAGI, S., JOESTEN, M. D. & LENHERT, P. G. (1976). *Acta Cryst.* B**32**, 326–328.

TAKAGI, S., LENHERT, P. G. & JOESTEN, M. D. (1974). *J. Amer. Chem. Soc.* **96**, 6606–6609.

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Tetraaquobis(succinimidato)nickel(II) Dihydrate

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Abstract. Orthorhombic, *Pnnm*, $a = 8{\cdot}6580$ (8), $b = 7{\cdot}1118$ (8), $c = 12{\cdot}522$ (1) Å, $C_8H_8N_2O_2Ni.6H_2O$, $Z = 2$, $D_m = 1{\cdot}57$, $D_c = 1{\cdot}563$ g cm^{-3}. Two of the coordinated water molecules form intramolecular hydrogen bonds to the carbonyl O atoms, whereas the other two are involved with water of crystallization in intermolecular bonding.

Introduction. Pale blue prismatic crystals were prepared as previously described (Ley & Werner, 1906). Mean weight loss on heating, 29·7%; calculated for $6H_2O$ per molecule of complex, 29·8%. Data were collected using a Hilger–Watts automated diffractometer, Mo $K\alpha$ radiation, to a θ limit of 33°, giving a set of 481 observed data. Systematic absences were in $hk0$ when $h+k$ odd, $0kl$ when $k+l$ odd. Space group *Pnnm* was assumed, and no feature of the structure gave cause to doubt it. The Ni atom position was fixed by symmetry, and all non-hydrogen atoms were readily placed from the heavy-atom phased density synthesis. H atoms were subsequently located from difference syntheses. Refinement was by full-matrix least squares, minimizing $\sum w(F_o - F_c)^2$, with weights based on the relationship $\sigma(I) = [\sigma^2(c) + (0{\cdot}04I)^2]^{1/2}$ where $\sigma^2(c)$ is the variance from counting statistics. Anisotropic thermal parameters were adopted, except for the H atoms which were given a constant isotropic parameter of 5·0 Å^2. The final R was 0·043. A final difference synthesis confirmed that no atom had been overlooked. Atom coordinates and thermal parameters are listed in Table 1,

Table 1. *Atomic coordinates and temperature factors*

	x	y	z	B or B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ni	0	0	0	0·0089 (2)	0·0180 (3)	0·0033 (1)	−0·0005 (4)	0	0
N	0	0	0·1679 (3)	0·0082 (6)	0·017 (1)	0·0028 (3)	−0·0000 (20)	0	0
O	0·2565 (4)	−0·0555 (6)	0·1974 (3)	0·0099 (5)	0·037 (1)	0·0042 (2)	0·0018 (7)	−0·0010 (3)	−0·0014 (4)
C(1)	0·1257 (6)	−0·0270 (9)	0·2317 (3)	0·0111 (7)	0·019 (2)	0·0036 (2)	−0·0011 (9)	−0·0000 (3)	0·0007 (6)
C(2)	0·0853 (7)	−0·0165 (14)	0·3484 (3)	0·0152 (8)	0·029 (2)	0·0029 (2)	0·0004 (17)	−0·0008 (4)	0·0012 (8)
O(*W*1)	0·2332 (7)	0·0898 (9)	0	0·0105 (8)	0·024 (2)	0·0056 (4)	−0·0048 (9)	0	0
O(*W*2)	−0·0820 (7)	0·2712 (7)	0	0·0132 (8)	0·015 (1)	0·0040 (3)	−0·0014 (8)	0	0
O(*W*3)	0	0·5	0·1685 (3)	0·0105 (6)	0·023 (1)	0·0035 (3)	0·0010 (17)	0	0
H(C1)	0·120 (7)	−0·137 (9)	0·392 (4)	5·0					
H(C2)	0·164 (7)	0·076 (8)	0·386 (4)	5·0					
H(*W*1)	0·272 (7)	0·036 (9)	0·053 (4)	5·0					
H(*W*2)	0·452 (6)	0·149 (8)	0·441 (4)	5·0					
H(*W*3)	0·085 (6)	0·464 (9)	0·200 (4)	5·0					

Table 2. *Interatomic distances and angles*

Ni—N	2·102 (2) Å	C(2)–H(C1)	1·06 (6) Å
Ni—O(*W*1)	2·118 (5)	C(2)–H(C2)	1·06 (6)
Ni—O(*W*2)	2·055 (5)	O(*W*1)–H(*W*1)	0·83 (6)
N—C(1)	1·364 (6)	O(*W*2)–H(*W*2)	0·96 (5)
C(1)–C(2)	1·505 (8)	O(*W*3)–H(*W*3)	0·87 (6)
C(1)–O	1·228 (6)		
C(2)–C(2)′	1·496 (8)		
O(1)···O(*W*1)	2·686 (6)	O(1)···H(*W*1)	1·93 (6)
O(1)···O(*W*3)	2·724 (4)	O(1)···H(*W*3)	1·89 (6)
O(*W*3)···O(*W*2)	2·758 (5)	O(*W*3)···H(*W*2)	1·78 (5)
O(*W*1)-Ni—O(*W*2)	92·7 (2)°	H(C1)—C(2)-H(C2)	90 (4)°
Ni—N—C(1)	125·9 (2)	H(C1)—C(2)-C(1)	113 (3)
N—C(1)—O	123·7 (5)	H(C1)—C(2)-C(2)′	114 (3)
N—C(1)—C(2)	112·2 (3)	H(C2)—C(2)-C(1)	108 (3)
C(1)—C(2)—C(1)′	103·7 (5)	H(C2)—C(2)-C(2)′	123 (3)
O(*W*1)-H(*W*1)···O(1)	150·7 (5)	H(*W*1)-O(1)-H(*W*1)′	105 (8)
O(*W*2)-H(*W*2)···O(*W*3)	176·1 (6)	H(*W*2)-O(2)-H(*W*2)′	98 (4)
O(*W*3)-H(*W*3)···O(1)	160·1 (6)	H(*W*3)-O(3)-H(*W*3)′	127 (8)

bond lengths and angles in Table 2.* The complex molecule and the hydrogen-bonding scheme are depicted in Fig. 1.

Discussion. The crystal structure of the hexahydrate of *N,N′*-bis-(2-salicylideneaminoethyl)ethylenediamine-nickel(II) (Cradwick, Cradwick, Dodson, Hall & Waters, 1972) showed an unexpected structural feature in that the water molecules form linear columns which can be described in terms of stacked decahedra, these being akin to the pentagonal dodecahedra that form the unit of clathrate hydrate structures, but with square basal faces. One water molecule in three forms a donor hydrogen bond to a phenolic O of the ligand but otherwise the water structure and the hexadentate complex are independent structural units. Hydrates of metal compounds are not commonly of interest for the water structure *per se*, the water molecules usually being associated with the cation or anion, but the above result suggested that neutral chelates of high hydration number could be of unsuspected structural interest. In this context it was noted that bis(succinimidato)nickel(II) is reported to form an octahydrate, from which six of the water molecules are much easier to remove than the other two (Ley & Werner, 1906), suggesting that these six may not be coordinated to the metal. In fact this report proved not to be correct; the compound is a hexahydrate, with four of the six molecules coordinated to the Ni atom, and while the hydrogen-bonding scheme is of interest it is not of a novel type.

The complex molecule, which has required point group symmetry C_{2h}, is shown in Fig. 1. Bond lengths and angles are normal, and with regard to the succinimidato ligand, are remarkably similar to those of succinimide itself (Mason, 1961). The atoms of the ligand (other than the H atoms) and the Ni atom are coplanar within error. The Ni–O bonds to the two crystallographically distinct, coordinated water molecules differ significantly in length, and these two molecules have quite different hydrogen bonding roles. Molecule 1 is involved only in intramolecular hydrogen bonds to the carbonyl O atoms, and these bonds determine the conformation of the molecule, with regard to the rotation of the ligand about the Ni–N bond. Molecule 2 is involved in the intermolecular bonding scheme, forming donor hydrogen bonds to the molecules of water of crystallization. For their part these

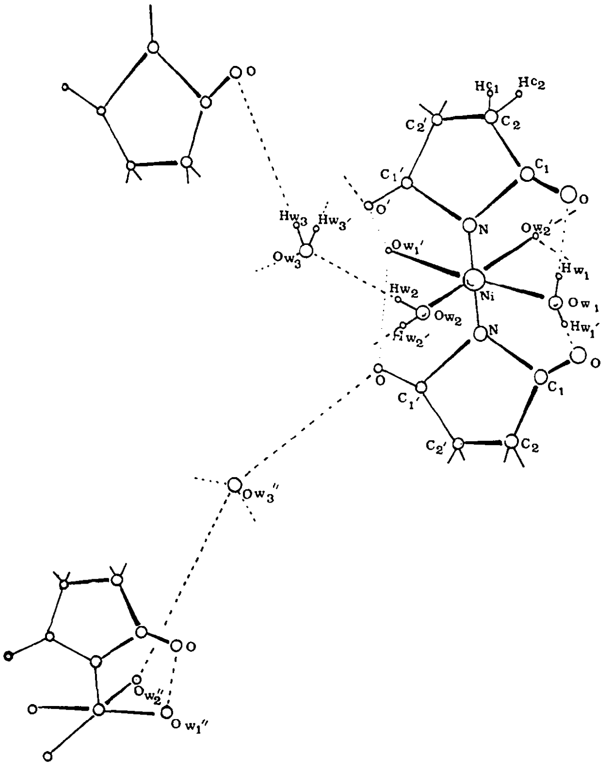

Fig. 1. The tetraaquobis(succinimidato)nickel(II) molecule, and the hydrogen-bonding scheme.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31555 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

water molecules are each involved in such linkages with two different complex molecules, and as well form donor linkages to a carbonyl O of two further complex molecules. Each complex molecule is thus involved in eight extramolecular hydrogen bonds, the carbonyl O atoms each being involved in one intra- and one intermolecular bond.

References

Cradwick, P. D., Cradwick, M. E., Dodson, G. G., Hall, D. & Waters, T. N. (1972). *Acta Cryst.* **B28**, 45–49.

Ley, H. & Werner, F. (1906). *Ber. dtsch. chem. Ges.* **39**, 2177–2180.

Mason, R. (1961). *Acta Cryst.* **14**, 720–724.

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The Monoclinic Form of *p*-Hydroxyacetanilide

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Abstract. HO–$C_6H_4NHCOCH_3$, m.p. 168–170°C, monoclinic, $P2_1/a$, $a=12{\cdot}93$ (4), $b=9{\cdot}40$ (1), $c=7{\cdot}10$ (2) Å, $\beta=115{\cdot}9$ (2)°. $D_m=1{\cdot}30$, $D_x=1{\cdot}296$ g cm^{-3}, $Z=4$. The structure was refined to $R=0{\cdot}072$ for 1382 non-zero reflexions. The molecules are linked together by two kinds of hydrogen bonds [2·663 (3) Å, OH donates to O=C; 2·934 (3) Å, OH accepts from H–N] to form a pleated sheet parallel to the *ac* plane and the sheets are stacked along **b**. The dihedral angle between the benzene ring and the amide group is 21·2°. The structure is compared with that of the orthorhombic form.

Introduction. The monoclinic crystals obtained from an aqueous solution were prisms elongated along **c**. Intensity data were collected on Weissenberg photographs with specimens 0·3 × 0·3 × 0·6 mm for the layers *hk*0 to *hk*5, and 0·4 × 0·6 × 0·2 mm for the layers *h*0*l* to *h*7*l*. Visually estimated intensities were corrected for Lorentz and polarization factors and for spot shape, but no absorption correction was made [μ(Cu $K\alpha$) = 8·0 cm^{-1}].

Intensities of 1382 non-zero reflexions (78% of the reflexions within Cu $K\alpha$ sphere) were placed on an approximately absolute scale by a Wilson plot ($B=3{\cdot}19$ Å^2).

The structure was solved from a Patterson map. Block-diagonal least-squares refinement reduced R from 0·35 to 0·11. A difference Fourier synthesis revealed the H atoms except for those of the methyl group. Further refinement including the H atoms reduced R to 0·092. After correction for extinction for the six strongest reflexions, a difference Fourier synthesis revealed all the H atoms. Refinement including all the atoms gave a final R value of 0·072 for 1382 non-zero reflexions.* The weights adopted were $w=1{\cdot}0$ for $0<|F_o|\le 7{\cdot}0$ and $w=(7{\cdot}0/|F_o|)^2$ for $|F_o|>7{\cdot}0$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The computations were performed on an NEAC 2200-500 computer at the Okayama University Computer Center. The programs used were *HBLS*-5 and *DAPH* (Ashida, 1973).

Table 1. *The final positional and thermal parameters* (× 10^4) *with standard deviations in parentheses*

The anisotropic temperature factors have the form exp $[-\beta_{11}h^2-\beta_{22}k^2-\beta_{33}l^2-\beta_{12}hk-\beta_{13}hl-\beta_{23}kl]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	2228 (2)	851 (2)	560 (3)	70 (1)	138 (3)	233 (5)	72 (3)	151 (4)	94 (6)
O(2)	−1923 (2)	4977 (2)	−5458 (3)	69 (1)	168 (3)	170 (4)	3 (3)	83 (4)	47 (6)
N	−1599 (2)	4436 (2)	−2145 (3)	52 (1)	86 (2)	173 (5)	4 (3)	84 (4)	17 (5)
C(1)	−611 (2)	3547 (2)	−1519 (3)	44 (1)	68 (2)	164 (5)	−16 (3)	58 (4)	−9 (5)
C(2)	142 (2)	3562 (2)	−2442 (4)	57 (2)	82 (3)	178 (5)	−15 (3)	91 (5)	21 (6)
C(3)	1089 (2)	2656 (3)	−1721 (4)	56 (2)	101 (3)	206 (6)	−9 (4)	131 (5)	−2 (7)
C(4)	1290 (2)	1740 (3)	−69 (4)	51 (2)	86 (3)	183 (5)	−4 (3)	92 (5)	−13 (6)
C(5)	551 (2)	1744 (3)	887 (4)	62 (2)	103 (3)	215 (6)	21 (4)	116 (6)	90 (7)
C(6)	−387 (2)	2641 (3)	160 (4)	55 (2)	106 (3)	216 (6)	9 (4)	126 (5)	59 (7)
C(7)	−2196 (2)	5067 (2)	−4003 (4)	55 (2)	79 (3)	163 (5)	−23 (3)	58 (5)	1 (6)
C(8)	−3233 (3)	5896 (3)	−4176 (4)	75 (2)	119 (3)	233 (7)	46 (5)	66 (6)	33 (8)

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