The Pt—Cl bonds [2·289 (3), 2·302 (3) Å for (I) and 2·312 (5), 2·317 (5) Å for (II)] and the Pt—S bond distances [2·263 (3), 2·247 (3) Å for (I) and 2·257 (5), 2·272 (5) Å for (II)] are normal [the standard deviations calculated as in Melanson & Rochon (1975) are probably underestimated] and agree well with the values found in cis-[Pt(Me₂SO)₂Cl₂] (Melanson & Rochon, 1975) and cis-[Pt(tetrahydrothiophene sulfoxide)₂-Cl₂] (Melanson, de la Chevrotière & Rochon, 1985). The S atoms in the sulfoxide ligands are in a tetrahedral environment. The Pt—S—O angles [117·4 (3), 116·4 (3)° for (I) and 119·0 (6), 116·6 (6)° for (II)] are larger than the tetrahedral value as observed in the Pt–sulfoxide structures described above and other published structures (Rochon & Guay, 1987, and references therein). The Pt—S—C angles [110·1 (4)–111·6 (4)° for (I) and 108·1 (6)–111·3 (6)° for (II)] are normal as in Pt—Me₂SO complexes (Melanson & Rochon, 1975; Rochon, Kong & Melanson, 1985). The S—O, S—C and C—C bond distances agree well with published values except for a few terminal C—C bonds. The thermal factors of most terminal C atoms are high, indicating some disorder. All the C—C—C angles in cis-[Pt(dpso)₂Cl₂] are slightly larger than expected [112 (1)–118 (2)°], resulting in a flattening of the propyl groups.

The density of the crystal cis-[Pt(dpso)₂Cl₂] (1·857 Mg m⁻³) is smaller than expected. The corresponding densities for cis-[Pt(Me₂SO)₂Cl₂], cis-[Pt(tetrahydrothiophene sulfoxide)₂Cl₂] and cis-[Pt(emso)₂Cl₂] are 2·620, 2·381 and 2·352 Mg m⁻³. The low density in cis-[Pt(dpso)₂Cl₂] results in high thermal factors of the carbon atoms, which tend to occupy more space, resulting in a flattening of the propyl groups.

The packing of the molecules is shown in Figs. 2 and 3.* No hydrogen bonding is expected for these compounds. In cis-[Pt(dpso)₂Cl₂], the propyl groups are almost perpendicular to the platinum plane which also contains the O atoms. The propyl groups are also almost parallel to the b axis. In cis-[Pt(emso)₂Cl₂], the platinum planes with the O atoms are nearly parallel to the ab plane.

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Structure of Pentaaqua(hypoxanthine)nickel(II) Sulfate*

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Abstract. [Ni(C₅H₄N₄O)(H₂O)₅]SO₄, $M_r = 380·96$, triclinic, $P\bar{1}$, $a = 7·498$ (1), $b = 12·901$ (2), $c = 6·633$ (1) Å, $\alpha = 92·77$ (1), $\beta = 90·15$ (1), $\gamma = 92·23$ (1)°, $V = 640·4$ (3) Å³, $Z = 2$, $D_m = 1·98$ by flotation, $D_x = 1·98$ g cm⁻³, $\lambda(Mo\,K\alpha) = 0·71073$ Å, $\mu = 17·4$ cm⁻¹, $F(000) = 392$, room temperature, $R = 0·032$ for 5408 observed unique reflections. The structure contains [Ni(hypoxanthine)(H₂O)₅]²⁺ cations and non-coordinating sulfate anions. Nickel(II) exhibits a slightly distorted octahedral coordination by five water molecules and a monodentate hypoxanthine ligand binding through N(7). Hypoxanthine is protonated at N(1) and N(9); metal coordination at N(7) induces about 30% of the shifts in the bonding angles of the imidazole ring compared to the shifts by protonation of guanine derivatives at N(7). The structure is

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* Hypoxanthine is 1,7-dihydro-6H-purin-6-one.

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stabilized by an extended hydrogen-bonding system and by stacking interactions, with mean stacking distances between the hypoxanthine planes of 3·25 and 3·33 Å.

Introduction. Metal complexes of bi- and trivalent metal ions with adenine and guanine, the major purine bases present in DNA and RNA, have been described in a number of crystallographic studies and reviews (Hodgson, 1977; Sletten, 1977). In contrast only a few structures are known involving hypoxanthine, which occurs as a constituent of inosine in tRNA.

The coordination properties of hypoxanthine and xanthine, intermediate products in the reduction of nucleic acid to uric acid in man, are of medical interest in cases of diseases like gout and xanthinuria, both resulting from disturbances in purine metabolism. Molybdenum complexes of xanthine are thought to be involved in the molecular mechanism of action of xanthine oxidase, a molybdenum–iron enzyme catalysing the oxidation of xanthine to uric acid (Stiefel, 1977).

The crystal structures and results of thermal analysis, IR and electronic spectral studies of Cu^{II}-(hypoxanthine)$SO_4.H_2O$ and of Co^{II}(hypoxanthine)-$SO_4.5H_2O$ have been presented in a recent study (Dubler, Hänggi & Bensch, 1987). According to similar cell parameters and on the basis of IR and thermo-analytical data the nickel complex Ni^{II}(hypoxanthine)-$SO_4.5H_2O$ was thought to be isostructural with its cobalt analogue. To verify this assumption and to elucidate details of the ligand geometry, hydrogen-bonding parameters and stacking properties, we performed the structure determination of the title compound.

Experimental. Green–blue crystals of distorted hexagonal prismatic shape, synthesized by boiling a mixture of hypoxanthine (hyxa) with an excess of $NiSO_4.6H_2O$ in $0.25M$ H_2SO_4, crystallized at 310 K. Composition: Calculated for $Ni(hyxa)SO_4.5H_2O$ C 15·76, H 3·70, N 14·71, S 8·42, H_2O 23·6%; observed C 15·73, H 3·65, N 14·50, S 8·15, H_2O 23·6% (thermogravimetric analysis). Most of the crystals twinned along [010]. A single crystal of dimensions 0·38 × 0·32 × 0·11 mm was prepared for the measurements by carefully cutting a twinned prism parallel to the (010) plane. Enraf–Nonius CAD-4 diffractometer. Cell dimensions measured by least-squares refinement of 25 reflections in the interval $18 < \theta < 24°$. Graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scan method, zigzag mode, variable scan rate 1·25 to 20·0° min⁻¹. Max. $(\sin\theta)/\lambda = 0.904$ Å⁻¹, range of *hkl*: $-13 \le h \le 13$, $-23 \le k \le 23$, $0 \le l \le 12$. 2·1% reduction of intensities of four standard reflections measured approximately every three hours. Orientation controlled every 250 reflections using four standard reflections. 8410 reflections measured (excluding

Table 1. *Positional and isotropic or equivalent isotropic thermal parameters for* Ni(*hyxa*) $SO_4.5H_2O$

	x	y	z	$B/B_{eq}(\text{Å}^2)$
Ni	−0·19388 (2)	0·72626 (1)	0·75915 (2)	1·411 (2)
S	0·25497 (4)	0·63381 (2)	1·25700 (5)	1·469 (4)
O(11)	0·1526 (1)	0·73027 (8)	1·2621 (2)	2·00 (2)
O(12)	0·2111 (2)	0·57115 (9)	1·0725 (2)	2·66 (2)
O(13)	0·2148 (2)	0·57559 (9)	1·4366 (2)	2·58 (2)
O(14)	0·4469 (1)	0·6648 (1)	1·2574 (2)	2·50 (2)
N(1)	−0·4074 (2)	1·09985 (9)	0·7368 (2)	1·87 (2)
C(2)	−0·5856 (2)	1·1090 (1)	0·7546 (2)	2·08 (2)
N(3)	−0·7015 (2)	1·0325 (1)	0·7722 (2)	2·13 (2)
C(4)	−0·6219 (2)	0·9389 (1)	0·7692 (2)	1·61 (2)
C(5)	−0·4422 (2)	0·9200 (1)	0·7542 (2)	1·47 (2)
C(6)	−0·3181 (2)	1·0064 (1)	0·7345 (2)	1·75 (2)
O(6)	−0·1554 (2)	1·00674 (9)	0·7166 (2)	2·94 (2)
N(7)	−0·4177 (1)	0·81416 (9)	0·7607 (2)	1·65 (2)
C(8)	−0·5801 (2)	0·7723 (1)	0·7758 (2)	1·93 (2)
N(9)	−0·7068 (1)	0·8440 (1)	0·7819 (2)	1·99 (2)
O(1)	0·0334 (1)	0·64520 (9)	0·7597 (2)	2·17 (2)
O(2)	−0·2895 (2)	0·63276 (9)	0·9774 (2)	3·06 (2)
O(3)	−0·2933 (2)	0·62678 (9)	0·5369 (2)	3·43 (2)
O(4)	−0·0591 (1)	0·82189 (8)	0·9831 (2)	2·20 (2)
O(5)	−0·0757 (2)	0·81526 (9)	0·5445 (2)	3·05 (2)
H(1)	−0·337 (3)	1·159 (2)	0·738 (3)	3·1 (5)*
H(2)	−0·626 (3)	1·181 (2)	0·759 (3)	3·2 (5)*
H(8)	−0·607 (3)	0·706 (2)	0·785 (3)	2·0 (4)*
H(9)	−0·813 (3)	0·830 (2)	0·806 (4)	4·1 (6)*

* Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

standards). Lorentz–polarization correction, numerical absorption correction, max. and min. transmission coefficients are 0·83 and 0·53, $R_{int} = 0.012$ for 723 averaged reflections. 7915 unique reflections, 5408 with $I > 3\sigma$ (*I*), σ(*I*) based on counting statistics.

Refinement was started with the atomic coordinates of the non-hydrogen atoms of the model for the cobalt analogue (Dubler, Hänggi & Bensch, 1987). All hydrogen atoms were located from difference Fourier maps and were included in the refinements with varying positional and isotropic temperature parameters. 5408 reflections in final refinement cycle; 246 parameters refined minimizing $\sum w(F_o - F_c)^2$; $w = 1/[\sigma^2(F) + 0.0004F^2]$, $S = 1.446$, $R = 0.032$, $wR = 0.043$, $(\Delta/\sigma)_{max} = 0.23$ (H-atom parameter) and 0·05 (non-hydrogen parameters) in final refinement cycle, max. height in final difference Fourier synthesis 0·93 e Å⁻³ near the Ni atom. All calculations performed on a PDP 11/34 computer using the *SDP* program system (Frenz, 1983). The final atomic parameters are listed in Table 1.*

Discussion. Ni(hyxa)$SO_4.5H_2O$ represents the first example of a structurally characterized nickel

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters of the water molecules and hydrogen-bonding parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44069 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

hypoxanthine complex. It is built up from $[Ni(hyxa)(H_2O)_5]^{2+}$ cations and non-coordinating sulfate anions (Fig. 1). Bond lengths and angles are listed in Table 2. The nickel ion is situated at the centre of a slightly distorted $NiNO_5$ octahedron. The average Ni—O bond length within the equatorial plane determined by four water molecules is 2·059 (1) Å. One diagonal atom pair O(3)/O(4) is 0·019 (1) Å above the plane, and the second diagonal atom pair O(2)/O(5) is 0·019 (1) Å below this plane. The nickel ion is displaced from the plane by 0·107 Å towards the axial N atom. The axial positions are occupied by another water molecule [Ni—O(1) = 2·034 (1) Å] and by the hypoxanthine ligand. Ni—O—H angles range from 109(2) to 129(1)°.

Hypoxanthine is coordinated to the metal atom through N(7) and is protonated at N(1) and N(9), but not at N(3). Binding sites of neutral unsubstituted hypoxanthine established by X-ray structure determinations are N(7) likewise in the cobalt analogue (Dubler, Hänggi & Bensch, 1987) of the nickel complex described here and in $Ru^{III}(hyxa)(NH_3)_5Cl_3.3H_2O$ (Kastner, Coffey, Clarke, Edmonds & Eriks, 1981), N(3) and N(9) in the dimeric complex $Cu^{II}(hyxa)_2Cl_2.3H_2O$ (Sletten, 1970), and N(3) and N(7) in the chain-type structure of $Cu^{II}(hyxa)SO_4.H_2O$ (Dubler, Hänggi & Bensch, 1987). Although in unsubstituted hypoxanthine N(9) is potentially also a metal coordination site, N(7) binding is favored over N(9), probably a consequence of the indirect chelation possibility *via* hydrogen-bonded O(6).

It is interesting to note that the principal structural features of $Ni(hyxa)SO_4.5H_2O$, namely octahedral coordination of the metal atom by N(7)-bonded

hypoxanthine and by five water molecules, have also been established in Ni(inosine 5'-monophosphate).$7H_2O$ and in Co(inosine 5'-monophosphate).$7H_2O$ (Clark & Orbell, 1974; Aoki, 1975). The molecular packing in the unit cell of Ni(hyxa)-$SO_4.5H_2O$ can be seen in Fig. 2.

As shown by the data given in Table 2, interatomic bond distances and angles of the hypoxanthine ligand in the nickel and in the cobalt complex are identical within the limits of errors. Taylor & Kennard (1982) have summarized the effect of protonation of N atoms on the geometry of purine base derivatives. Although only limited crystallographic data were available for guanine, the purine most similar to hypoxanthine, the following statements could be derived: Protonation of guanine derivatives mainly influences the bonding angles at N(7) (the protonation site) and at the adjacent atoms of the imidazole ring, and minor changes occur in the corresponding bond lengths. No significant trends were observed in the bond distances and angles within the pyrimidine ring and in the displacement of the atoms from the best plane through the purine system. Mean protonated/unprotonated bonding angles (°) of guanine given by Taylor & Kennard (1982) are 107·3 (9)/110·8(2) for C(4)—C(5)—N(7), 108·0(2)/104·2(3) for C(5)—N(7)—C(8), 109·4(2)/113·5(4) for N(7)—C(8)—N(9), 108·7(4)/106·0(2) for C(8)—N(9)—C(4) and 106·7(7)/105·6(1) for N(9)—C(4)—C(5). Since bond distances and angles of the nickel complex described here and of the isostructural cobalt compound (Dubler, Hänggi & Bensch, 1987) have been determined with high precision, an analysis of the effect of metal coordination on the ligand geometry may be of interest. All corresponding bond angles of the imidazole ring in the nickel complex are slightly shifted from the values of a neutral form towards the values of a protonated form. Metal coordination at N(7) obviously induces the same changes in the imidazole ring geometry as a (hypothetical) protonation would do, but to a much lower extent. An estimation based on the bonding angles given above and in Table 2 shows that metal coordination at N(7) induces only about 30% of the shifts associated with protonation at N(7).

The familiar solid-state stacking (Bugg, 1972), found in most purine structures, is also retained in $Ni(hyxa)SO_4.5H_2O$. The purine rings are stacked on top of each other rotated by about 180° along the *c* axis. The mean stacking distances between hypoxanthine molecules, calculated as the mean distances of all atoms of one molecule from the least-squares plane through the stacking molecule, are 3·25 (symmetry operation $-x-1$, $2-y$, $2-z$) and 3·33 Å ($-x-1$, $2-y$, $1-z$). Calculations of least-squares planes reveal a very small but significant deviation from planarity of the hypoxanthine molecule, with maximum distances from the best plane of +0·016 (1) Å for N(9) and −0·019 (1) Å for N(7).
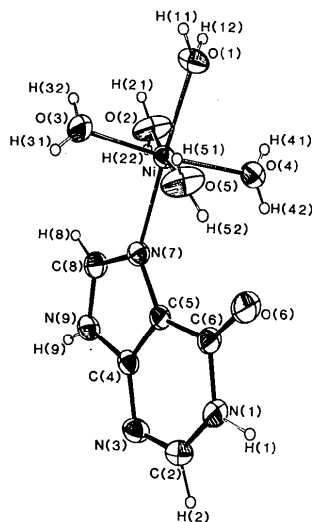
Fig. 1. *ORTEP* plot (Johnson, 1976) of the coordination polyhedra of Ni(hyxa)SO₄.5H₂O and atom-numbering scheme of the hypoxanthine ligand. The thermal ellipsoids are drawn at the 60% probability level. H atoms are shown as spheres of arbitrary size.

Table 2. *Interatomic bond distances* (Å) *and angles* (°) *with their e.s.d.'s.*

For comparison, results concerning the hypoxanthine ligand geometry in the analoguous cobalt complex (Dubler, Hänggi & Bensch, 1987) are given in square brackets.

|					
Ni–N(7)	2·061 (1)		S–O(11)	1·486 (1)	
Ni–O(1)	2·034 (1)		S–O(12)	1·463 (1)	
Ni–O(2)	2·042 (1)		S–O(13)	1·465 (1)	
Ni–O(3)	2·028 (1)		S–O(14)	1·479 (1)	
Ni–O(4)	2·114 (1)				
Ni–O(5)	2·054 (1)				
N(1)–C(2)	1·351 (2)	|1·348 (1)|	C(8)–N(9)	1·351 (2)	|1·354 (1)|
C(2)–N(3)	1·299 (2)	|1·303 (1)|	N(9)–C(4)	1·364 (2)	|1·361 (1)|
N(3)–C(4)	1·367 (2)	|1·362 (1)|	C(6)–O(6)	1·226 (1)	|1·229 (1)|
C(4)–C(5)	1·381 (1)	|1·382 (1)|	N(1)–H(1)	0·92 (2)	|0·83 (1)|
C(5)–C(6)	1·436 (1)	|1·432 (1)|	C(2)–H(2)	0·99 (2)	|1·03 (1)|
C(6)–N(1)	1·401 (2)	|1·397 (1)|	C(8)–H(8)	0·88 (2)	|0·89 (1)|
C(5)–N(7)	1·387 (1)	|1·384 (1)|	N(9)–H(9)	0·82 (2)	|0·86 (1)|
N(7)–C(8)	1·319 (1)	|1·318 (1)|			
N(7)–Ni–O(1)	177·56 (4)		O(1)–Ni–O(5)	87·27 (4)	
N(7)–Ni–O(2)	93·68 (4)		O(2)–Ni–O(3)	91·63 (6)	
N(7)–Ni–O(3)	92·68 (5)		O(2)–Ni–O(4)	89·75 (5)	
N(7)–Ni–O(4)	94·23 (4)		O(2)–Ni–O(5)	174·89 (5)	
N(7)–Ni–O(5)	91·25 (4)		O(3)–Ni–O(4)	172·85 (5)	
O(1)–Ni–O(2)	87·75 (4)		O(3)–Ni–O(5)	89·49 (6)	
O(1)–Ni–O(3)	89·24 (5)		O(4)–Ni–O(5)	88·53 (5)	
O(1)–Ni–O(4)	83·80 (4)				
O(11)–S–O(12)	109·64 (6)		O(12)–S–O(13)	111·01 (7)	
O(11)–S–O(13)	109·84 (5)		O(12)–S–O(14)	109·47 (6)	
O(11)–S–O(14)	107·64 (6)		O(13)–S–O(14)	109·18 (6)	
C(6)–N(1)–C(2)	125·5 (1)	|125·53 (6)|	N(7)–C(8)–N(9)	112·5 (1)	|112·44 (6)|
N(1)–C(2)–N(3)	125·4 (1)	|125·29 (6)|	C(8)–N(9)–C(4)	107·3 (1)	|107·13 (6)|
C(2)–N(3)–C(4)	111·8 (1)	|111·88 (6)|	N(9)–C(4)–C(5)	105·8 (1)	|106·01 (6)|
N(3)–C(4)–C(5)	128·1 (1)	|127·72 (6)|	N(3)–C(4)–N(9)	126·1 (1)	|126·27 (6)|
C(4)–C(5)–C(6)	118·6 (1)	|118·90 (6)|	C(6)–C(5)–N(7)	131·8 (1)	|131·53 (6)|
C(5)–C(6)–N(1)	110·7 (1)	|110·67 (6)|	N(1)–C(6)–O(6)	120·2 (1)	|120·45 (6)|
C(4)–C(5)–N(7)	109·6 (1)	|109·57 (6)|	C(5)–C(6)–O(6)	129·1 (1)	|128·88 (6)|
C(5)–N(7)–C(8)	104·7 (1)	|104·84 (5)|			
Ni–N(7)–C(5)	133·12 (7)	|133·85 (4)|	Ni–N(7)–C(8)	122·12 (8)	[121·20 (5)]
C(2)–N(1)–H(1)	118 (1)	|115·3 (8)|	N(7)–C(8)–H(8)	126 (1)	(126·3 (8)|
C(6)–N(1)–H(1)	116 (1)	|119·2 (8)|	N(9)–C(8)–H(8)	122 (1)	|121·3 (8)|
N(1)–C(2)–H(2)	115 (1)	|117·2 (7)|	C(4)–N(9)–H(9)	129 (2)	|129·1 (8)|
N(3)–C(2)–H(2)	120 (1)	|117·5 (7)|	C(8)–N(9)–H(9)	124 (2)	|123·6 (8)|
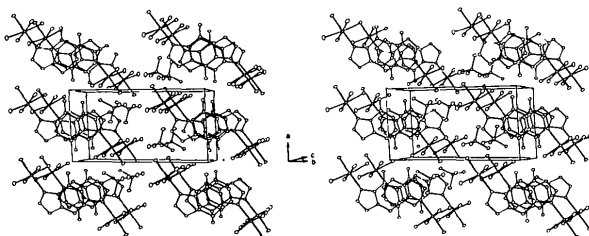
Fig. 2. *ORTEP* stereo plot (Johnson, 1976) of the packing diagram of Ni(hyxa)SO₄.5H₂O. The *ab* projection of the unit cell is rotated −17° around the *a* axis.

An analysis of hydrogen-bonded contacts shows that there are no hydrogen bonds between hypoxanthine molecules, but medium-strong interactions of the type N—H(hyxa)···O(SO₄) [N(1)···O(11) = 2·85 Å], N—H(hyxa)···O(H₂O) [N(9)···O(4) = 2·97 Å], O—H(H₂O)···O(SO₄) [O···O from 2·68 to 2·77 Å], O—H(H₂O)···N(hyxa) [O(4)···N(3) = 3·06 Å] and O—H(H₂O)···O(hyxa) [O(5)···O(6) = 2·76 Å]. The 'intramolecular' hydrogen bond between O(6) of hypoxanthine and the water molecule O(5) coordinated to the metal atom [O(5)—H···O(6) = 149°] is also observed in Cu(9-methylhypoxanthine)₂SO₄.4H₂O (Sletten

& Kaale, 1977) and can be considered as an indirect N(7)/O(6) chelation of nickel.

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