approximation each symmetry-independent water molecule has the same orientation with respect to the other two. It is thus surprising, notwithstanding the susceptibility of the metal–oxygen bonds to deformation, that the geometry of the hexaaquametal group is less ideal than that of either the ammonium or the sulfate group.

The asymmetry in the hexaaquametal moiety does not relate directly to differences in X-ray and neutron structural geometry, nor to the hydrogen-bond network. It may be due to longer-range interactions – which are considered with the study of the electron density of Maslen, Ridout & Watson (1988).

Financial support was received from the Australian Institute for Nuclear Science and Engineering and the Australian Research Grants Scheme. SCR acknowledges support from the Commonwealth Postgraduate Awards Scheme. Thanks are due to B. W. Skelton, who prepared the crystals for this work, to A. H. White for assistance with the X-ray data and to the AINSE Neutron Diffraction Group for assistance with neutron diffraction facilities.

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The Structures of Tutton's Salts. II. Diammonium Hexaaquanickel(II) Sulfate

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(*Received* 7 *February* 1987; *accepted* 3 *November* 1987)

Abstract. The crystal structure of the title compound at 295 K was refined using both neutron and X-ray data. $[NH_4]_2[Ni(H_2O)_6](SO_4)_2$, $M_r = 395{\cdot}00$, $P2_1/a$, monoclinic, $a = 9{\cdot}179\ (3)$, $b = 12{\cdot}462\ (6)$, $c = 6{\cdot}241\ (2)$ Å, $\beta = 106{\cdot}95\ (3)°$, $V = 682{\cdot}90$ Å^3 (X-ray results), $Z = 2$, $D_x = 1{\cdot}921$ Mg m^{-3}, $F(000) = 412$. Neutron data: $\lambda = 0{\cdot}9840$ Å, $\mu = 0{\cdot}22$ mm^{-1}, $R = 0{\cdot}038$ and $wR = 0{\cdot}033$ for 1225 reflections. X-ray data: Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}772$ mm^{-1}, $R = 0{\cdot}047$ and $wR = 0{\cdot}033$ for 7025 reflections. Differences between the lengths of bonds which would be equal for isolated groups and small changes from the X-ray to the neutron structure are consistent with the effects of interactions between neighbouring groups.

Introduction. The title compound is a member of the isomorphous series $(NH_4)_2M^{II}(SO_4)_2(H_2O)_6$, known as Tutton's salts. M is a divalent metal. X-ray structures have previously been reported by Grimes, Kay & Webb (1963), by Montgomery & Lingafelter (1964) and by Treushnikov, Kuskov, Soboleva & Belov (1978). The last-named authors also made a study of the electron density distribution based on X-ray data only. The structural parameters from an X-ray refinement are biased by the redistribution of electron density due to bonding. The resulting bias, which potentially contains information on bonding, can be determined by comparison with a neutron diffraction experiment. Neutron diffraction data and more complete X-ray data were collected. The structure was refined with these data as the first stage of a charge density study.

Experimental. Crystals by evaporation of a solution containing stoichiometric quantities of ammonium and magnesium sulfates in water.

Neutron measurements on the 4-circle diffractometer at the HIFAR reactor at the Australian Atomic Energy Commission Research Establishment at Lucas Heights,

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New South Wales. Crystal showing twelve (110), (011), (001) and (111) faces with minimum and maximum dimensions 1·52 and 3·55 mm respectively, and volume 13·6 mm^3. Neutron cell dimensions determined by least-squares fit to the angular setting of 36 reflections fully centred in the counter aperture: $a = 9{\cdot}175$ (2), $b = 12{\cdot}455$ (4), $c = 6{\cdot}242$ (3) Å, $\beta = 106{\cdot}98$ (5)°.

2504 intensities measured with an ω–2θ scan reduced by statistical averaging to 1225 unique reflections. 51 with negative measured values classed as 'less-thans' during structure refinement. $0 \le h \le 11$, $0 \le k \le 15$, $-5 \le l \le 5$. $0{\cdot}07 < (\sin\theta)/\lambda < 0{\cdot}63$ Å^{-1}. Data corrected for absorption using the analytical method with $\mu = 0{\cdot}22$ mm^{-1}, calculated from coefficients by Melkonian (1949) for H and by Bacon (1975) for the remaining atoms. Transmission factor range 0·56–0·73. Standard $\bar{4}04$ reflection repeated after 26 other measurements, with 11% long-term variation. $\sigma(I)$ estimated from counting statistics and probable error in the absorption correction, modified where necessary from a comparison of equivalent reflections. Data reduction programs by Elcombe, Cox, Pryor & Moore (1971). Neutron form factors from *International Tables for X-ray Crystallography* (1974). Parameters determined by full-matrix least-squares minimization of $\sum w(|F_o| - F_c|)^2$ with $w = 1/\sigma^2(|F_o|)$. Coordinates for atoms in general positions, anisotropic thermal parameters, a scale factor and a secondary-extinction coefficient refined until $S = 1{\cdot}24$, $R = 0{\cdot}038$, $wR = 0{\cdot}033$, largest shift $< 0{\cdot}1\sigma$. $r^* = 0{\cdot}127$ (2) (Larson, 1970).

X-ray diffractometry: Nicolet $P\bar{1}$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. Crystal face development similar to that of the neutron crystal with volume 0·004 mm^3. Cell dimensions from 12 reflections with $13 < 2\theta < 20°$. $\mu = 1{\cdot}772$ mm^{-1}. Three standards, measured every 100 reflections, showed a total fluctuation of 6% during the data collection. Of these the 200 reflection, which is extinction affected, increased in intensity by 4%. The $\bar{1}34$ reflection showed a 3% decrease, and the fluctuations in the $2\bar{4}1$ reflection were random. 20 076 intensities measured with an ω–2θ scan reduced to 7025 unique reflections in the range $(\sin\theta)/\lambda < 1{\cdot}073$ Å^{-1}, $0 \le h \le 19$, $0 \le k \le 26$, $-13 \le l \le 13$. $R_{\rm int} = 0{\cdot}022$. Absorption corrections evaluated using Gaussian integration with coefficients from *International Tables for X-ray Crystallography* (1974). Transmission-factor range 0·61–0·72. $\sigma(I)$ determined following the procedure for the neutron data.

Atomic scattering factors for H taken from Stewart, Davidson & Simpson (1965) and for C, Ni, N, O and S with dispersion corrections from *International Tables for X-ray Crystallography* (1974). Isotropic thermal parameters for H and other parameters as in the neutron analysis refined by full-matrix least squares until $S = 1{\cdot}82$, $R = 0{\cdot}047$, $wR = 0{\cdot}033$, largest shift $< 0{\cdot}1\sigma$. $r^* = 0{\cdot}0091$ (1) (Larson, 1970). Difference density range $-0{\cdot}68 < \Delta\rho < 0{\cdot}35$ e Å^{-3} near the Ni atom. Absorption and structure refinement programs from the *XRAY76* system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).†

Discussion. Atomic positions are listed in Table 1, with bond lengths and angles in Table 2. A diagram of the structure including the hydrogen-bond lengths is shown in Fig. 1.

The relationship between the bond lengths for the neutron structure closely resembles that in the magnesium complex (Maslen, Ridout, Watson & Moore, 1988). S(2)—O(4) is short, in comparison with S(2)—O(3), S(2)—O(5) and S(2)—O(6), as expected because of O(4)'s weaker involvement in the hydrogen bonding.

In the hexaaquametal moiety, the hydrogen-bond connections for the water molecules are equivalent, to a first approximation. There are minor differences in the O—H and H···O distances involving O(8) and O(9), similar to those in the magnesium structure. The differences between the hydrogen-bond networks for O(8) and O(9) are quite small, but the Ni—O(7) and Ni—O(8) lengths are approximately 0·03 Å longer than that involving O(9). The reasons for this are examined by Maslen, Ridout & Watson (1988).

Although the trends in the magnesium complex are confirmed, the X-ray results have lower precision because of the dominance of the Ni atom on the X-ray scattering. A comparison of the positions from the X-ray and neutron structures is included in Table 3. Each displacement of an H atom (X-ray—neutron position) is approximately antiparallel to the corresponding N—H or O—H bond. The average displacements are 0·168 and 0·195 Å in length, compared with mean bond-length differences of 0·162 and 0·183 Å respectively. The corresponding values for the O—H bonds have a larger range, and are inversely correlated with the O—H lengths, especially with those of the X-ray structure. Thus large H atom displacements reflect weaker involvement of these atoms in the hydrogen-bond network. The more asymmetrical is its environment, the larger is the *X*—*N* shift for the H atom.

For the O atoms, the displacements are much smaller than those for the H atoms. Whereas the values for the sulfate O atoms are of limited significance, those for the water molecules are larger, and close to the corresponding values for the magnesium complex. The displacements for O(7), O(8) and O(9) are close to the external bisector of the corresponding H—O—H angle.

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44486 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$$B_{\text{eq}} = (8\pi^2/3)\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	Neutron ($\times 10^4$)				X-ray ($\times 10^5$ N, Ni, O, S; $\times 10^3$ H)			
	x	y	z	B_{eq}(Å^2)	x	y	z	B_{eq}(Å^2)
Ni	0	0	0	1·20	0	0	0	1·25
S(2)	4070 (3)	1364 (2)	7389 (5)	1·26	40778 (2)	13646 (2)	73898 (3)	1·46
O(3)	4145 (2)	2276 (1)	5902 (3)	2·16	41456 (9)	22762 (5)	59037 (12)	2·52
O(4)	5486 (2)	749 (1)	7868 (4)	2·78	54882 (8)	7486 (6)	78666 (15)	3·04
O(5)	2791 (2)	671 (1)	6205 (3)	1·86	27898 (7)	6709 (5)	62088 (11)	1·95
O(6)	3838 (2)	1773 (1)	9488 (3)	2·12	38343 (8)	17734 (5)	94828 (11)	2·35
O(7)	1687 (2)	1069 (1)	1630 (4)	1·72	16794 (8)	10710 (6)	16267 (12)	1·91
O(8)	−1610 (2)	1100 (1)	338 (3)	1·74	−16029 (8)	10983 (5)	3457 (12)	1·97
O(9)	18 (2)	−665 (1)	2998 (3)	1·65	233 (8)	−6622 (5)	29938 (11)	1·87
N(10)	1345 (1)	3464 (1)	3566 (2)	2·20	13477 (12)	34646 (8)	35652 (17)	2·33
H(11)	649 (6)	3339 (4)	2020 (9)	5·01	79 (2)	335 (1)	225 (3)	4·17
H(12)	2265 (5)	2981 (3)	3978 (9)	4·69	212 (2)	304 (1)	388 (3)	4·49
H(13)	726 (6)	3294 (4)	4606 (9)	5·45	84 (2)	336 (1)	449 (3)	4·00
H(14)	1688 (5)	4240 (3)	3671 (8)	4·59	161 (2)	410 (1)	358 (3)	4·55
H(15)	2165 (4)	894 (2)	3190 (7)	2·85	199 (2)	90 (1)	284 (3)	3·19
H(16)	2467 (4)	1225 (2)	915 (6)	2·91	223 (2)	117 (1)	102 (3)	4·07
H(17)	−2653 (3)	962 (2)	−552 (6)	2·76	−246 (2)	95 (1)	−37 (3)	4·27
H(18)	−1389 (3)	1839 (2)	32 (6)	2·75	−139 (2)	175 (1)	15 (2)	3·26
H(19)	−925 (4)	−579 (3)	3370 (6)	3·18	−69 (2)	−58 (1)	339 (3)	3·62
H(20)	269 (3)	−1422 (2)	3228 (6)	2·63	29 (2)	−130 (1)	319 (2)	3·04

Table 2. *Bond lengths* (Å) *and angles* (°)

	X-ray	Neutron		X-ray	Neutron
Ni–O(7)	2·068 (1)	2·071 (2)	O(7)–Ni–O(8)	88·44 (4)	88·75 (7)
Ni–O(8)	2·066 (1)	2·071 (2)	O(7)–Ni–O(9)	90·24 (4)	90·35 (8)
Ni–O(9)	2·037 (1)	2·042 (2)	O(8)–Ni–O(9)	89·42 (4)	89·45 (7)
S(2)–O(3)	1·479 (1)	1·481 (3)	O(3)–S(2)–O(4)	109·57 (5)	109·23 (22)
S(2)–O(4)	1·459 (1)	1·462 (3)	O(3)–S(2)–O(5)	108·10 (5)	108·15 (19)
S(2)–O(5)	1·477 (1)	1·471 (3)	O(3)–S(2)–O(6)	109·52 (5)	109·57 (19)
S(2)–O(6)	1·479 (1)	1·478 (4)	O(4)–S(2)–O(5)	109·11 (5)	109·10 (19)
N(10)–H(11)	0·841 (16)	1·002 (5)	O(4)–S(2)–O(6)	110·78 (5)	110·41 (21)
N(10)–H(12)	0·858 (17)	1·007 (4)	O(5)–S(2)–O(6)	109·71 (5)	110·35 (22)
N(10)–H(13)	0·851 (18)	1·002 (6)	H(11)–N(10)–H(12)	110·0 (15)	113·8 (4)
N(10)–H(14)	0·828 (17)	1·012 (4)	H(11)–N(10)–H(13)	109·5 (16)	105·5 (4)
O(7)–H(15)	0·759 (16)	0·969 (5)	H(11)–N(10)–H(14)	105·5 (15)	108·0 (4)
O(7)–H(16)	0·721 (20)	0·967 (4)	H(12)–N(10)–H(13)	109·2 (15)	107·4 (4)
O(8)–H(17)	0·804 (15)	0·972 (3)	H(12)–N(10)–H(14)	111·4 (16)	109·5 (3)
O(8)–H(18)	0·859 (14)	0·975 (3)	H(13)–N(10)–H(14)	111·2 (16)	112·6 (5)
O(9)–H(19)	0·770 (18)	0·965 (4)	Ni–O(7)–H(15)	108·2 (10)	113·8 (2)
O(9)–H(20)	0·828 (13)	0·971 (3)	Ni–O(7)–H(16)	112·7 (12)	116·8 (2)
			H(15)–O(7)–H(16)	117·2 (17)	109·2 (3)
			Ni–O(8)–H(17)	113·5 (12)	116·0 (2)
			Ni–O(8)–H(18)	114·6 (10)	114·2 (2)
			H(17)–O(8)–H(18)	111·8 (14)	106·4 (3)
			Ni–O(9)–H(19)	116·9 (10)	114·0 (2)
			Ni–O(9)–H(20)	116·5 (11)	117·9 (3)
			H(19)–O(9)–H(20)	109·2 (15)	105·3 (3)

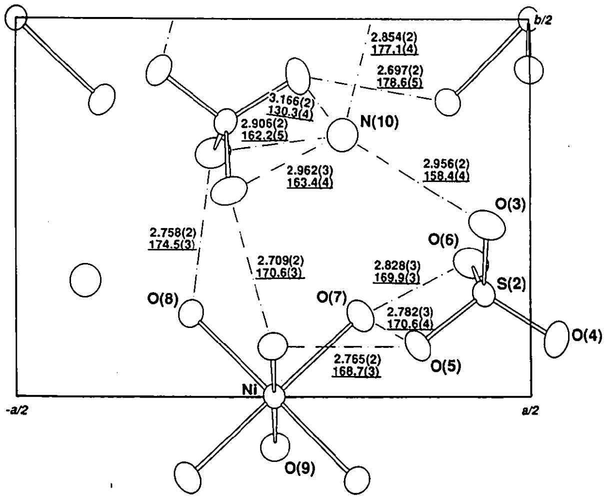

Fig. 1. Hydrogen-bond distances (Å) and angles (°) from the neutron structure. The A–H···B angles are underlined.

Table 3. *Comparison of X-ray* (*X*) *and neutron* (*N*) *structures*

X *N*	Distance (Å)	*X* *N* *N*	Angle (°)
S(2)–S(2)	0·007 (3)	O(7)–O(7)–Ni	66 (14)
O(3)–O(3)	0·001 (2)	O(7)–O(7)–*B**	156 (15)
O(4)–O(4)	0·003 (2)	O(8)–O(8)–Ni	50 (15)
O(5)–O(5)	0·003 (2)	O(8)–O(8)–*B**	149 (14)
O(6)–O(6)	0·004 (2)	O(9)–O(9)–Ni	46 (13)
O(7)–O(7)	0·007 (2)	O(9)–O(9)–*B*9*	165 (13)
O(8)–O(8)	0·007 (2)	O(3)–O(3)–S(2)	89 (85)
O(9)–O(9)	0·008 (2)	O(4)–O(4)–S(2)	150 (45)
N(10)–N(10)	0·002 (2)	O(5)–O(5)–S(2)	92 (27)
H(11)–H(11)	0·166 (16)	O(6)–O(6)–S(2)	69 (31)
H(12)–H(12)	0·151 (16)	H(11)–H(11)–N(10)	11 (7)
H(13)–H(13)	0·164 (18)	H(12)–H(12)–N(10)	11 (6)
H(14)–H(14)	0·190 (17)	H(13)–H(13)–N(10)	19 (6)
H(15)–H(15)	0·231 (14)	H(14)–H(14)–N(10)	13 (5)
H(16)–H(16)	0·258 (19)	H(15)–H(15)–O(7)	20 (4)
H(17)–H(17)	0·183 (15)	H(16)–H(16)–O(7)	12 (4)
H(18)–H(18)	0·127 (14)	H(17)–H(17)–O(8)	16 (5)
H(19)–H(19)	0·214 (17)	H(18)–H(18)–O(8)	22 (6)
H(20)–H(20)	0·158 (14)	H(19)–H(19)–O(9)	18 (4)
		H(20)–H(20)–O(9)	21 (5)

* *B*7, *B*8 and *B*9 are the internal bisectors of the H–O–H angles centred on O(7), O(8) and O(9) respectively.

Financial support was received from the Australian Institute for Nuclear Science and Engineering and the Australian Research Grants Scheme. SCR acknowledges support from the Commonwealth Postgraduate Awards Scheme. Thanks are due to B. W. Skelton, who prepared the crystals for this work, to A. H. White for assistance with the X-ray data and to the AINSE Neutron Diffraction Group for assistance with neutron diffraction facilities.

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Acta Cryst. (1988). C**44**, 415–416

Synthèse et Structure du Borouranate de Sodium, $NaBUO_5$

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(*Reçu le* 22 *septembre* 1987, *accepté le* 3 *décembre* 1987)

Abstract. $M_r = 351{\cdot}89$, orthorhombic, *Pcam*, $a = 10{\cdot}712\,(3)$, $b = 5{\cdot}780\,(1)$, $c = 6{\cdot}862\,(2)$ Å, $V = 424{\cdot}86$ Å^3, $Z = 4$, $D_x = 5{\cdot}5$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 36$ mm^{-1}, $F(000) = 592$, $T = 290$ K, $R = 0{\cdot}075$, $wR = 0{\cdot}090$ for 1478 reflexions with $I \geq 3\sigma(I)$. U^{6+} is located inside a pentagonal bipyramid and forms a nearly collinear uranyl group normal to the plane of five secondary bonds. BO_3 triangles join these polyhedral chains through an edge and a corner. Na^+ ions ensure the crystal cohesion in the third direction. U—O distances vary between 1·804 and 2·407 Å, B—O between 1·347 and 1·387 Å and Na—O between 2·365 and 2·888 Å.

Introduction. Hoekstra (1967) signalait la synthèse, en poudre, de plusieurs borouranates alcalins: $LiBUO_5$, $NaBUO_5$ et $KBUO_5$, et en décrivait les spectres d'absorption infra-rouge, tous très voisins.

L'obtention de monocristaux de $NaBUO_5$ nous a permis d'en établir la structure.

Partie expérimentale. Chauffage à l'air à 1373 K pendant 15 h de U_3O_8, Na_2CO_3 et B_2O_3 avec 1U + 2Na + 11B. Après lavage à l'eau bouillante, produit homogène composé d'aiguilles plates jaune clair.

Symétrie orthorhombique *Pcam* ou $Pca2_1$ mise en évidence sur films. Plaquette en biseau limitée par les faces (001), (001), (301), (100), (100), (041) et (010). Longueur: 360 µm, épaisseur moyenne 60 µm. Diffractomètre Philips PW 1100, monochromateur en graphite, méthode d'intégration $\omega/2\theta$ avec 0,02° s^{-1}. Paramètres affinés par moindres carrés à partir de 25 réflexions entre 10 et 15° θ. 1896 réflexions indépendantes non nulles avec $5 \leq \theta \leq 45°$ et $0 \leq h \leq 21$, $0 \leq k \leq 11$, $0 \leq l \leq 13$. Trois réflexions de référence (413, 600, 413) mesurées toutes les heures avec une variation moyenne d'intensité de 0,002. Correction de l'absorption par la méthode analytique de Meulenaer & Tompa (1965). Transmission comprise entre 0,025 et 0,125.

En *Pcam*, les sections de Patterson tridimensionnelles donnent le site de l'uranium. Les séries différence de Fourier donnent les autres atomes. Affinements par moindres carrés [*AFINE*, modification du programme Busing, Martin & Levy (1963)] des coordonnées atomiques et des facteurs de température isotropes, puis anisotropes pour les cations. Le passage dans le groupe non centré $Pca2_1$ n'améliore pas les résultats.

Fonction minimisée: $\sum w(\Delta F^2)$ avec $w = 1/\sigma^2$. $R = 0{,}075$, $wR = 0{,}090$ pour les 1478 réflexions telles que $I \geq 3\sigma(I)$.* Cette valeur élevée de R est liée à une imprécision dans la correction d'absorption due à la forme et aux faibles dimensions du cristal. Au dernier cycle, $S = 5{,}25$; $\Delta/\sigma_{\max} = 0{,}15$ (β_{11} de U); $\Delta\rho_{\max} = 5$ e Å^{-3} localisé au voisinage du site de l'uranium. Facteurs de diffusion extraits de *International Tables for X-ray Crystallography* (1974), dispersion anomale prise en compte pour U. Paramètre d'extinction: $0{,}290 \times 10^{-6}$. Ordinateur utilisé: MATRA 570/CX.

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* Les listes des facteurs de structure et des paramètres thermiques anisotropes ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 44568: 12 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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