Acta Cryst. (1966). **21**, 705

A Neutron Diffraction Analysis of The Crystal Structure of Tetragonal Nickel Sulphate Hexadeuterate*

By B. H. O'Connor†
Physics Department, University of Western Australia, Nedlands, Western Australia

and D. H. Dale†
Australian Institute of Nuclear Science and Engineering, Lucas Heights, Sydney, Australia

(*Received* 26 *February* 1966)

The deuterium positions in nickel sulphate hexadeuterate have been located from two-dimensional neutron diffraction data, and the positional and thermal parameters of all atoms in the structure have been refined by full-matrix least-squares analysis to yield a final R index of 0·082. The structure proposed by Beevers & Lipson (1932) for the non-hydrogen atoms is confirmed. The crystals are tetragonal, space group either $P4_12_12$ or $P4_32_12$ (enantiomorphic), 4 molecules in a unit cell with parameters $a=b=6·790 \pm 0·003$, $c=18·305 \pm 0·004$ Å. The deuterium atoms all participate in non-linear hydrogen bonds. None of these bonds is bifurcated or of abnormal length. The mean O–D length is 0·97 Å (e.s.d. 0·02 Å).

Introduction

Much attention has been directed recently to the arrangement of hydrogen bonds in crystalline hydrates (*e.g.* Baur, 1965). One of these, tetragonal $NiSO_4 . 6H_2O$, was first examined by Beevers & Lipson (1932). Their X-ray analysis was based on assumed Ni–O and S–O bond distances, and did not provide locations for the hydrogen atoms. The purpose of the authors' two-dimensional neutron diffraction analysis of $NiSO_4 . 6D_2O$ was to establish the deuterium positions and then to refine independently the coordinates and thermal parameters of all atoms in the structure.

Experimental

Since deuterium gives a greatly reduced amount of spin incoherent background scattering and has a higher coherent scattering length as compared with hydrogen it is more suitable for neutron diffraction studies. Slow evaporation of a concentrated solution of nickel sulphate in heavy water produced large deep-green crystals with tetragonal pyramidal habit, and with a (001) cleavage plane. The unit-cell constants were determined by the back-reflexion Weissenberg technique with Cu $K\alpha$ X-radiation.

Crystal data

Tetragonal
$a=b=6·790 \pm 0·003$, $c=18·305 \pm 0·004$ Å
$U=843·9$ Å^3
Space group $P4_12_12$ (No. 92) or $P4_32_12$ (No. 96) (enantiomorphic)
$Z=4$

The neutron intensity data were measured with the A.I.N.S.E.2-Tan single-crystal spectrometer at the Australian Atomic Energy Commission HIFAR reactor, and a monochromatic neutron beam, with a flux of approximately 10^7 thermal neutrons $min^{-1} . cm^{-2}$ and wavelength 0·95 Å, reflected from the (200) planes of a lead monochromator. The spectrometer is a conventional two-circle instrument employing the 2θ-scan technique (Alexander & Smith, 1962). The intensities were recorded by continuous scanning of the reflected neutron beam with a high pressure BF_3 proportional counter. The reflexions 008 and 220 were used as standards. Results showed that the neutron flux varied little during the experiment.

The crystal used was approximately square in cross-section, with 2·8 mm sides, and 3·8 mm deep, the longest dimension being parallel to [001]. Within the range $\theta < 60°$ a total of 265 $hk0$ and $h0l$ reflexions were measured and 191 of these had intensities significantly greater than background. Lorentz factors were applied but no absorption or extinction corrections were made to the recorded intensities.

Location of deuterium atoms and initial structure refinement

Initially a set of $h0l$ structure factors was computed on the Beevers & Lipson model, using scattering lengths equal to 10·3, 3·1 and 5·77 Fermi units (1 Fermi $\equiv 10^{-13}$ cm) for the nickel, sulphur and oxygen atoms respectively (Bacon, 1962). A plot of $\ln(F_o/F_c)$ *versus* $\sin^2 \theta/\lambda^2$ provided values for the scale factor and overall isotropic temperature factor. The first R index was 0·49. A Fourier synthesis of neutron scattering density

---

* Research performed under the auspices of the Australian Institute of Nuclear Science and Engineering.

† Present address: Ceramics Division, United Kingdom Atomic Energy Research Establishment, Harwell, Berkshire, England.

was computed* and this showed that the deuterium atoms lie, as expected, close to sulphate oxygen and water oxygen short contacts. A deuterium scattering length of 6·5 Fermi units (Bacon, 1962) was introduced into the calculation of a second set of structure factors which was based upon all the atoms in the structure. The *R* index fell to 0·41. A difference Fourier synthesis was then calculated. On this map, peaks of negative sign appeared at the deuterium atom sites. It was suspected on this basis that the crystal was partially hydrated. A spectroscopic analysis (Whateley, 1964) of the crystal used, and of the mother liquor from which it was grown, confirmed this, and fixed the D:H ratio at 3·1:1·0. Accordingly, in initial refinement calculations, a scattering length of 4·0 Fermi units was assigned to the 'deuterium' atoms. Undoubtedly the accuracy of the analysis was adversely affected by the presence of H_2O in the crystal.

Six cycles of structure-factor–difference-Fourier refinement on the positional parameters and isotropic temperature factors lowered the *R* index to 0·21.

Least-squares refinement

The final refinement was carried out by the full-matrix least-squares method. A modification of the program

Table 1. *Observed and calculated structure amplitudes in Fermi units*

H	K	L	|F(OBS)|	|F(CAL)|
0	0	4	67.6	63.4
		8	103.9	110.5
		12	37.1	34.3
		16	122.2	140.5
		20	87.3	121.0
1	0	1	37.5	34.4
		2	11.9	10.2
		3	11.7	10.5
		4	U	0.9
		5	33.2	34.5
		6	31.9	33.9
		7	23.6	22.8
		8	54.4	55.4
		9	U	1.3
		10	52.6	53.6
		11	16.7	16.6
		12	17.2	18.1
		13	19.7	18.2
		14	45.1	43.6
		15	17.3	17.8
		16	46.8	50.8
		17	U	8.7
		18	U	15.4
		19	40.8	41.9
1	1	0	16.2	13.5
	2		48.1	48.5
	3		37.9	38.2
	4		U	8.6
	5		29.4	30.9
	6		35.2	37.9
	7		13.8	8.6
	8		49.8	46.7
	9		U	9.1
	10		19.6	14.6
	11		U	4.0
	12		19.9	16.3
2	0	0	24.6	21.1
		1	U	2.8
		2	58.0	57.9
		3	112.1	117.8
		4	40.6	40.3
		5	35.2	36.8
		6	28.5	29.2
		7	12.1	12.9
		8	96.0	98.2
		9	U	5.4
		10	U	2.2
		11	27.5	24.2
		12	41.9	42.0
		13	23.3	21.2
		14	47.4	47.9
		15	32.1	27.7
		16	U	11.9
		17	52.5	45.7
		18	17.6	17.4
		19	U	5.2
		20	31.0	33.8
		21	14.3	18.9
2	2	0	63.8	64.2
	3		63.8	68.0
	4		42.5	34.2
	5		55.8	54.5
	6		36.2	34.5
	7		41.2	36.9
	8		55.0	50.8
	9		U	8.0
	10		40.8	36.6
	11		23.4	23.1
	12		24.7	23.9
3	0	1	62.7	62.1
		2	U	5.1
		3	55.9	49.8
		4	34.2	30.3
		5	U	11.0
		6	36.0	30.9
		7	11.5	9.2
		8	U	9.6
		9	U	4.3
		10	53.1	54.8
		11	12.4	10.3
		12	31.5	30.5
		13	U	4.1
		14	26.4	23.7
		15	14.9	19.7
		16	27.8	21.9
		17	63.6	65.0
		18	23.5	22.8
		19	43.0	42.8
		20	U	8.9
		21	15.7	22.4
3	3	0	11.5	11.1
	4		91.8	93.8
	5		51.9	46.8
	6		44.7	45.0
	7		U	10.1
	8		U	2.7
	9		31.5	31.6
	10		U	9.6
	11		18.6	21.4
4	0	0	U	7.1
		1	69.6	64.7
		2	54.2	51.1
		3	31.1	32.1
		4	16.0	11.2
		5	80.0	82.1
		6	U	8.6
		7	27.2	35.1
		8	44.1	42.6
		9	35.0	27.5
		10	18.8	18.7
		11	43.1	42.8
		12	U	0.4
		13	37.6	41.2
		14	15.3	13.7
		15	43.5	49.1
		16	44.2	37.8
		17	55.3	57.3
		18	21.3	14.5
		19	72.8	72.5
		20	16.2	16.5
4	4	0	80.2	82.6
	5		15.9	18.7
	6		21.6	27.9
	7		16.5	15.0
	8		22.9	21.4
	9		U	5.0
	10		16.0	15.5
	11		22.4	17.5
5	0	1	19.2	19.9
		2	11.6	10.3
		3	10.0	14.7
		4	U	5.1
		5	U	2.2
		6	15.0	13.5
		7	22.1	17.2
		8	U	13.4
		9	18.1	16.6
		10	23.2	23.0
		11	21.0	20.0
		12	14.1	13.7
		13	36.6	38.7
		14	27.3	25.0
		15	17.5	22.2
		16	U	8.7
		17	U	0.6
		18	22.9	18.0
		19	17.4	19.7
		20	U	8.9
		21	19.9	24.1
5	5	0	26.0	37.8
	6		22.3	22.5
	7		35.2	27.0
	8		32.8	38.3
	9		U	1.0
	10		20.0	16.7
6	0	0	19.7	18.2
		1	U	15.1
		2	12.2	10.5
		3	39.2	33.8
		4	U	8.1
		5	26.9	30.2
		6	U	2.7
		7	45.0	39.2
		8	U	9.9
		9	33.5	35.4
		10	U	3.1
		11	40.7	40.3
		12	U	10.6
		13	31.1	30.9
		14	46.6	44.5
		15	U	2.9
		16	15.0	14.3
		17	U	5.9
		18	15.1	20.2
6	6	0	58.7	50.9
	7		U	2.4
	8		21.6	21.9
7	0	1	U	2.7
		2	57.4	58.2
		3	11.7	12.2
		4	15.7	16.5
		5	16.1	19.5
		6	U	2.6
		7	37.4	40.2
		8	15.9	13.6
		9	U	4.0
		10	45.8	39.6
		11	13.5	17.2
		12	12.5	15.1
		13	U	14.9
		14	44.9	51.3
		15	23.9	26.3
		16	U	1.1
		17	13.0	16.6
		18	U	10.5
		19	12.9	14.5
		20	11.8	12.6
7	7	0	37.0	46.3
8	0	0	U	2.6
		1	36.9	34.5
		2	29.9	28.0
		3	20.2	19.9
		4	44.1	42.8
		5	17.2	19.9
		6	40.2	36.9
		7	18.0	20.3
		8	45.5	45.8
		9	19.7	12.3
		10	14.3	11.7
		11	U	18.9
		12	U	27.3
		13	U	10.0
		14	U	13.2
		15	U	13.1
		16	U	1.9
		17	19.1	16.1
9	0	1	U	2.0
		2	U	0.6
		3	27.9	29.2
		4	U	0.1
		5	19.8	25.6
		6	30.4	30.0
		7	19.1	19.1
		8	15.1	8.6
		9	U	6.1
		10	26.2	25.7
		11	U	4.9
		12	U	3.9
		13	U	9.7
		14	12.7	14.7
		15	U	6.1
		16	14.7	12.0
10	0	0	21.4	24.0
		1	U	5.0
		2	U	10.0
		3	U	8.0
		4	19.9	19.2
		5	36.2	32.4
		6	18.3	15.6
		7	14.6	13.5
		8	51.2	49.6
		9	17.1	16.0
		10	U	1.6
		11	18.4	21.1
		12	17.1	22.1
		13	U	10.9
		14	U	13.0
		15	14.6	16.0
		16	13.9	13.1
11	0	1	U	2.4
		2	U	3.1
		3	19.2	17.9
		4	11.7	11.7
		5	33.4	30.0
		6	23.7	23.8
		7	18.0	17.6
		8	20.9	21.1
		9	14.9	17.9
		10	U	6.4
		11	11.9	15.7
12	0	0	18.5	13.1
		1	U	6.6
		2	U	3.0
		3	11.0	16.2
		4	11.2	12.1

U • UNOBSERVED REFLECTION

---

* In the course of calculating the Fourier summations it was discovered that the expression listed in *International Tables for X-ray Crystallography* (1952) for space group $P4_12_12$ is in error. The correct formula is given by

$$
\begin{aligned}
\varrho(x,y,z) = {} & \frac{8}{V_c}\Bigg\{ \underset{h,k,l=0}{\overset{H\;K\;L\quad 2h+2k+l=4n}{\Sigma\;\Sigma\;\Sigma}} |F(hkl)|\,[\cos 2\pi hx \cos 2\pi ky \cos 2\pi lz \cos\alpha \\
& \qquad - \sin 2\pi hx \sin 2\pi ky \sin 2\pi lz \sin\alpha] \\
& + \tfrac{1}{2}\overset{2h+2k+l=4n+1}{\Sigma\Sigma\Sigma} |F(hkl)|[\sin 2\pi(hx+ky)\sin(\alpha-2\pi\, lz) \\
& \qquad + \sin 2\pi(hx-ky)\cos(\alpha+2\pi lz)] \\
& + \overset{2h+2k+l=4n+2}{\Sigma\Sigma\Sigma} |F(hkl)|[\cos 2\pi hx \cos 2\pi ky \sin 2\pi lz \sin\alpha \\
& \qquad - \sin 2\pi hx \sin 2\pi ky \cos 2\pi lz \cos\alpha] \\
& + \tfrac{1}{2}\overset{2h+2k+l=4n+3}{\Sigma\Sigma\Sigma} |F(hkl)|[\sin 2\pi(hx+ky)\sin(\alpha-2\pi lz) \\
& \qquad - \sin 2\pi(hx-ky)\cos(\alpha+2\pi lz)]\Bigg\}.
\end{aligned}
$$

ORFLS (Busing, Martin & Levy, 1962) for the IBM 7030 machine was employed to refine the atomic parameters and scale factors for the $h0l$ and $hk0$ structure factor sets and the effective deuterium scattering length. The analysis was based on minimization of the quantity $\Sigma w(|F_o|-k|F_c|)^2$ in which the observed terms were assigned unit weights and from which the unobserved reflexions were excluded. After several cycles of refinement it was obvious that the intensity measured for the 0,0,20 reflexion was considerably lower than expected. This reflexion was therefore excluded from the subsequent calculations. All parameter shifts were insignificant after six cycles and the refinement was terminated. The refined value for the effective deuterium scattering length, 4·3 Fermis (e.s.d. 0·3), confirmed the value given by spectroscopic analysis. For the final structural model the weighted R index, defined as $\Sigma\, w(||F_o|-k|F_c||)/\Sigma\, w|F_o|$, was 0·082. The observed structure amplitudes and the calculated amplitudes for the final structural model are tabulated in Table 1. Final values for the atomic coordinates and isotropic temperature factors are listed in Table 2, the standard deviation estimates being those derived from the diagonal elements of the inverse to the least-squares matrix.

Discussion

Bond distances and angles

The interatomic bond distances and angles are listed in Table 3. They have not been corrected for thermal vibration effects. The distance and angle standard deviation estimates were computed with a program based on the error formulae given in *International Tables for X-ray Crystallography* (1959). Individual Ni–O bond distances do not show significant departures from their mean value, 2·06 (2) Å. The mean agrees well with that of 2·04 (2) Å found in $(NH_4)_2Ni(SO_4)_2.6H_2O$ (Grimes, Kay & Webb, 1963) and the average distance of 2·08 (2) Å given in *International Tables for X-ray Crystallography* (1962).

The mean S–O bond distance of 1·47 (1) Å agrees closely with the mean value of 1·473 (2) Å obtained

Table 2. *Fractional atomic coordinates and isotropic thermal parameters* (*standard deviations in parentheses*)

	x/a	y/b	z/c	B
Ni	0·2101 (5)	0·2101	0·0000	1·2 (1) Å^2
S	0·7126 (23)	0·7126	0·0000	1·7 (3)
O(1)	0·1714 (13)	−0·0489 (14)	0·0518 (5)	2·3 (1)
O(2)	0·4720 (11)	0·2449 (11)	0·0564 (4)	1·4 (1)
O(3)	0·0641 (13)	0·3564 (12)	0·0852 (5)	1·7 (1)
O(4)	0·6226 (18)	0·6191 (16)	0·0648 (5)	2·7 (2)
O(5)	0·9248 (12)	0·6757 (14)	−0·0003 (4)	1·9 (1)
D(11)	0·0754 (20)	−0·1409 (21)	0·0399 (7)	2·4 (3)
D(12)	0·2493 (19)	−0·0862 (20)	0·0959 (8)	2·8 (3)
D(21)	0·5679 (32)	0·1411 (27)	0·0493 (9)	3·3 (5)
D(22)	0·5423 (18)	0·3686 (16)	0·0565 (7)	2·2 (3)
D(31)	−0·0115 (25)	0·4675 (20)	0·0681 (8)	3·0 (3)
D(32)	−0·0178 (21)	0·2733 (21)	0·1155 (8)	2·3 (3)

Table 3. *Bond distances and angles** (*standard deviations in parentheses*)

Bond	Distance
Ni–O(1)	2·02 (1) Å
Ni–O(2)	2·07 (1)
Ni–O(3)	2·10 (1)
S–O(4)	1·48 (2)
S–O(5)	1·46 (2)
O(1)–D(11)	0·93 (2)
O(1)–D(12)	1·00 (2)
O(2)–D(21)	0·97 (2)
O(2)–D(22)	0·97 (2)
O(3)–D(31)	0·96 (2)
O(3)–D(32)	0·96 (2)

Bonds	Angle
O(1)–Ni–O(2)	88°38′ (20′)
O(1)–Ni–O(3)	90 8 (21)
O(1)–Ni–O(1′)	89 37 (23)
O(1)–Ni–O(3′)	89 55 (23)
O(2)–Ni–O(3)	88 53 (20)
O(2)–Ni–O(2′)	93 3 (19)
O(2)–Ni–O(3′)	91 4 (20)
O(1)–Ni–O(2′)	178 0 (21)
O(3)–Ni–O(3′)	179 55 (23)
Ni–O(1)–D(11)	124 23 (67)
Ni–O(1)–D(12)	122 14 (63)
Ni–O(2)–D(21)	115 21 (78)
Ni–O(2)–D(22)	121 40 (56)
Ni–O(3)–D(31)	112 23 (67)
Ni–O(3)–D(32)	114 58 (63)
O(4)–S–O(5)	109 43 (55)
O(4)–S–O(4′)	106 50 (68)
O(4)–S–O(5′)	110 29 (53)
O(5)–S–O(5′)	109 45 (53)
D(11)–O(1)–D(12)	112 58 (90)
D(21)–O(2)–D(22)	107 32 (94)
D(31)–O(3)–D(33)	109 41 (93)

* Atom symbols which have a primed numeral represent the equivalent position ($y,x,\bar{z}$).

for all S–O bond lengths, uncorrected for thermal motion, in $Li_2SO_4.H_2O$, $MgSO_4.4H_2O$, $FeSO_4.7H_2O$, $MgSO_4.7H_2O$ and $(NH_4)_2Mg(SO_4)_2.6H_2O$ (Baur, 1964*b*). As in the above mentioned sulphates the O–S–O angles in $NiSO_4.6D_2O$ are tetrahedral. Mean values of 0·97 (2) Å for the O–D bonds and 110°(1°) for the O–D–O angles conform with those tabulated for other hydrates (Baur, 1965). The angles Ni–O–D scatter over a range of 12°, from 112° to 124°.

Hydrogen bonding

Views of the hydrogen bond system down [001] and [010] are shown in Figs. 1 and 2 respectively. The $Ni(D_2O)_6^{2+}$ and SO_4^{2-} groups are distributed in hydrogen bonded layers lying parallel to the principal cleavage plane, (001). Adjacent layers are linked by hydrogen bonds between (i) the water oxygen atom O(3) and the sulphate oxygen atom $O(4^{v})$, and (ii) the atoms O(1) and $O(3^{v})$ in neighbouring $Ni(D_2O)_6^{2+}$ groups. There is one electron lone pair per water oxygen atom and there are two per sulphate oxygen atom. Thus, when crystal symmetry is considered, it is to be expected that all six deuterium atoms form hydrogen bonds. This is indeed the case and none of the bonds is bifurcated. Distances and angles defined by these bonds are given in Table 4. The hydrogen bonds are non-linear, the O–D···O angles falling within the range 153–168°. Although individual O–D distances show no significant differences from the mean value of 0·97(2) Å, the D···O and O···O distances show some small but significant differences from their mean values of 1·83 and 2·77 Å, respectively. However this phenomenon is not unusual in salt hydrates.

The most interesting hydrogen bond is undoubtedly that bridging the water oxygen atoms O(1) and O(3), in which O(3) acts as acceptor (Figs. 1 and 2). Beevers & Schwartz (1935) pointed out that two of the water oxygen atoms, at opposite corners of the octahedron, accept hydrogen bonds from outside water molecules. This happens in identical manner in both $NiSO_4.6H_2O$ and the orthorhombic $NiSO_4.7H_2O$. They also as-

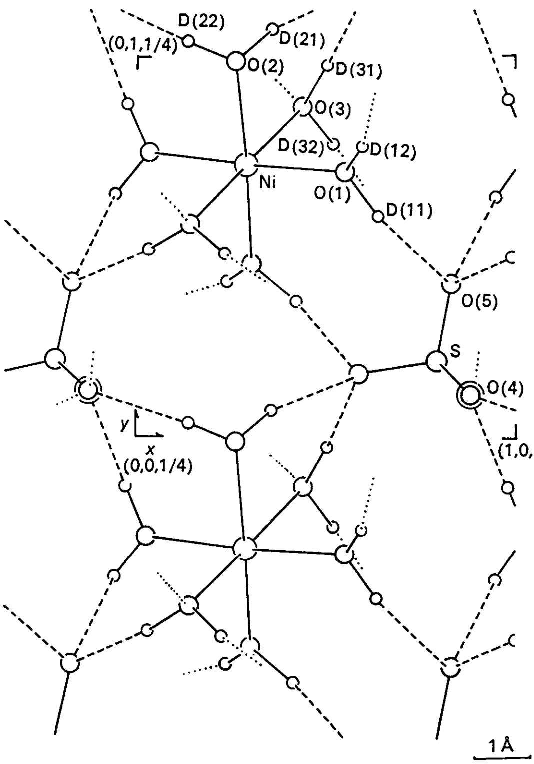

Fig. 1. The layer at $z=\frac{1}{4}$ from a projection of the structure along the unique axis of the crystal. Hydrogen bonds within the layer are represented by broken lines, those to adjoining layers by dotted lines.

Table 4. *Geometry of the hydrogen bonds* (*standard deviations in parentheses*)

Hydrogen bonds	Distances			Angles
	O–D	D···O	O···O	O–D···O
$D(11)\cdots O(5^{iv})$	0·93 (2) Å	1·77 (2) Å	2·69 (2) Å	167°17′ (1°22′)
O(1)				
$D(12)\cdots O(3^{v})$	1·00 (2)	1·83 (2)	2·81 (2)	168 5 (1 20)
$D(21)\cdots O(5^{ii})$	0·97 (2)	1·86 (2)	2·77 (2)	153 59 (1 46)
O(2)				
$D(22)\cdots O(4)$	0·97 (2)	1·79 (2)	2·74 (2)	167 16 (1 15)
$D(31)\cdots O(5^{iii})$	0·96 (2)	1·94 (2)	2·84 (2)	154 3 (1 29)
O(3)				
$D(32)\cdots O(4^{v})$	0·96 (2)	1·80 (2)	2·75 (2)	167 5 (1 27)

Equivalent positions: Unsuperscripted (x, y, z)

ii	$(y, -1+x, \bar{z})$
iii	$(-1+x, y, z)$
iv	$(-1+x, -1+y, z)$
v	$(\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{4}-z)$

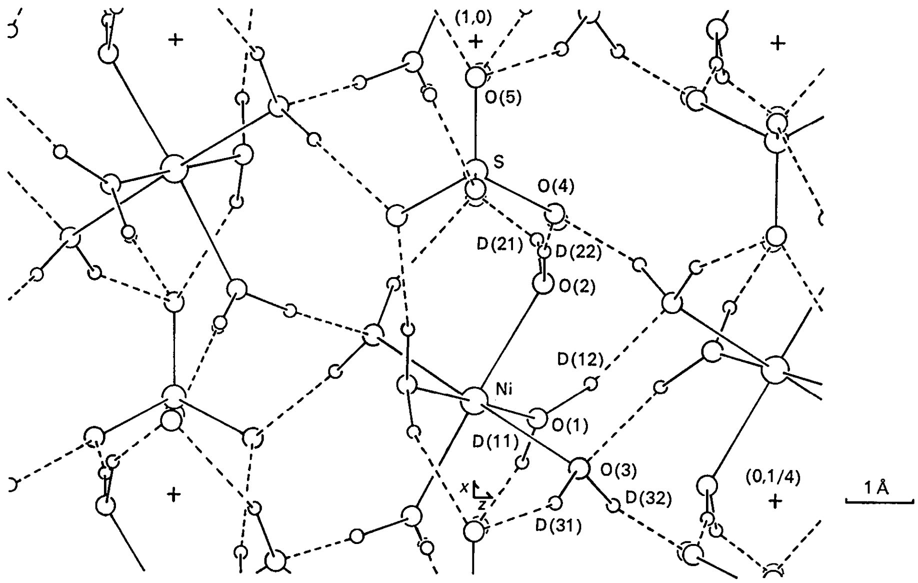

Fig. 2. A projection of the structure along the *b* axis of the crystal. Hydrogen bonds are represented by broken lines. Half circles represent atoms from a neighbouring cell.

sumed that no bond is formed between the atoms O(1) and O(4). The authors' analysis bears this out since the relevant angles and distances are:
O(1)–D(11)$\cdots$O(4^{v}) 75°, O(1)–D(12)$\cdots$O$^{\mathrm{v}}$ (4) 83°, O(1)$\cdots$O$^{\mathrm{v}}$ (4) 3·36 Å, D(11)$\cdots$O$^{\mathrm{v}}$ (4) 3·47 Å, and D(12)$\cdots$O(4^{v}) 3·33 Å.

The authors are very grateful to the Australian Institute of Nuclear Science and Engineering for providing facilities, to Dr G. A. Bottomley for advice on crystal growing procedures, and to Mr J. A. K. Battye and members of the A.A.E.C. staff for helping in the collection of diffraction data. One of us (B.H.O'C.) held an I.C.I. (Australia and New Zealand) Fellowship during the course of this work.

References

ALEXANDER, L. E. & SMITH, G. S. (1962). *Acta Cryst.* **15**, 983.

BACON, G. E. (1962). *Neutron Diffraction*, p. 61. Oxford: Clarendon Press.

BAUR, W. H. (1964*a*). *Acta Cryst.* **17**, 1167.

BAUR, W. H. (1964*b*). *Acta Cryst.* **17**, 1361.

BAUR, W. H. (1965). *Acta Cryst.* **19**, 909.

BEEVERS, C. A. & LIPSON, H. (1932). *Z. Kristallogr.* **83**, 123.

BEEVERS, C. A. & SCHWARTZ, C. M. (1935). *Z. Kristallogr.* **91**, 157.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). ORNL-TM-305. Oak Ridge National Laboratory, Oak Ridge, Tenn., U.S.A.

GRIMES, N. W., KAY, H. F. & WEBB, M. W. (1963). *Acta Cryst.* **16**, 823.

International Tables for X-ray Crystallography (1952). Vol. I, p. 426. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1959). Vol. II, p. 331. Birmingham: Kynoch Press.

International Tables for X-ray Crystallography (1962). Vol. III, p. 269. Birmingham: Kynoch Press.

WHATELEY, A. (1964). Private communication.