tances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances in Table 2.* KAu_2 crystallizes with the hexagonal $MgZn_2$ (C_{14})-type structure (Friauf, 1927). The axial ratio $c/a = 1·7412$ is rather high compared to the ideal value $c/a = (8/3)^{1/2} = 1·633$ for C_{14}-type Laves–Friauf phases. The increased c/a value in KAu_2 causes some distortion of the CN 12 and CN 16 polyhedra.

Related literature. For the phase diagram of the Au–K system see Kienast & Verma (1961) and Massalski (1986). The related compound $NaAu_2$ (Mathewson, 1911; Kienast & Verma, 1961; Massalski, 1986) has a wide range of homogeneity and crystallizes with the cubic $MgCu_2$ (C_{15}-type) structure (Perlitz & Aruja, 1937; Haucke, 1937).

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* Lists of structure factors, anisotropic thermal parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. 44905 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Monoclinic Nickel(II) Sulfate Hexahydrate

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Abstract. $NiSO_4.6H_2O$, $M_r = 262·86$, monoclinic, $C2/c$, $a = 9·878$ (2), $b = 7·214$ (2), $c = 24·065$ (6) Å, $\beta = 98·37$ (2)°, $V = 1696·6$ Å^3, $Z = 8$, $D_x = 2·06$ g cm^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0·71069$ Å, $\mu = 25·596$ cm^{-1}, $F(000) = 1088$, $T = 294$ K, $R = 0·046$ for 2913 unique reflections. Each of the two independent Ni atoms is octahedrally coordinated to six water molecules and has an average Ni—O bond distance of 2·048 Å. The hydrogen bonding occurs mainly between the water molecules and the sulfate O atoms, but there is a single hydrogen bond which occurs between water molecules on adjacent nickel octahedra. The sulfate ions are nearly ideally tetrahedral, but have one S—O bond which is shorter than the rest: 1·449 *vs* 1·473 Å.

Experimental. Nickel sulfate crystals were formed from aqueous solution by slow evaporation at room temperature during our preparation of crystalline nickel perbromate (Gallucci, Gerkin & Reppart, 1988), owing to the presence of a sulfate impurity in our preparation of nickel hydroxide. The dark green crystal chosen for intensity measurements had eleven faces ($1\bar{1}2$, $\bar{1}1\bar{2}$, 001, $00\bar{1}$, $\bar{1}11$, $11\bar{1}$, $\bar{1}\bar{1}1$, $10\bar{2}$, 112, 101, $\bar{2}0\bar{1}$) and approximate dimensions 0·34 × 0·56 × 0·34 mm.

Intensity data were measured with a Syntex $P\bar{1}$ automated four-circle diffractometer. Unit-cell parameters were determined from a least-squares fit of the setting angles for 25 reflections with $20 < 2\theta < 30°$. Intensity data were collected, using the ω–2θ technique, for 5623 reflections ($h_{\max} = 14$, $k_{\max} = 10$, $l_{\max} = 36$) over two ranges: (1) $+h,+k,\pm l$ and $4 < 2\theta < 65°$, and (2) $-h,-k,\pm l$ and $4 < 2\theta < 45°$. Scan widths were from $2\theta(\text{Mo } K\alpha_1) - 1·0°$ to $2\theta(\text{Mo } K\alpha_2) + 1·2°$ and the background/scan time ratio was 0·5. Uncertainties were assigned to each reflection using the formula $\sigma_I^2 = R^2(C+4B) + (0·02I)^2$, where R is the variable scan rate, C is the total number of counts, B is the total number of background counts and I is the integrated intensity $[I = R(C-2B)]$. The six standard

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
Ni(1)	0	5000	0	22 (<1)
O(5)	926 (2)	7216 (2)	430 (1)	31 (<1)
O(6)	−1898 (2)	5607 (3)	206 (1)	45 (1)
O(7)	297 (2)	3397 (3)	705 (1)	40 (1)
Ni(2)	0	4504 (1)	2500	19 (<1)
O(8)	1169 (2)	6528 (2)	2191 (1)	25 (<1)
O(9)	1441 (2)	4510 (2)	3200 (1)	32 (1)
O(10)	1150 (2)	2548 (2)	2173 (1)	31 (1)
S	3715 (1)	4517 (1)	1240 (1)	22 (<1)
O(1)	3021 (2)	2710 (2)	1183 (1)	33 (1)
O(2)	2739 (2)	5984 (2)	1337 (1)	31 (1)
O(3)	4218 (2)	4920 (2)	706 (1)	37 (1)
O(4)	4835 (2)	4477 (2)	1702 (1)	45 (1)
H(1)	1352	6898	760	64 (11)
H(2)	550	8183	534	71 (11)
H(3)	−2026	6250	548	124 (17)
H(4)	−2591	5211	−13	110 (17)
H(5)	1130	3122	880	53 (9)
H(6)	−116	2213	637	58 (10)
H(7)	623	7398	2030	52 (9)
H(8)	1761	6124	1988	75 (12)
H(9)	1550	3589	3407	76 (12)
H(10)	1561	5467	3394	48 (9)
H(11)	1892	2297	2379	86 (14)
H(12)	624	1717	2030	102 (15)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $NiSO_4.6H_2O$ *with e.s.d.'s in parentheses*

Ni(1)—O(5)	2·047 (2)	O(5)—Ni(1)—O(6)	93·91 (7)
Ni(1)—O(6)	2·055 (2)	O(5)—Ni(1)—O(7)	91·32 (7)
Ni(1)—O(7)	2·039 (2)	O(6)—Ni(1)—O(7)	86·91 (8)
Ni(2)—O(8)	2·066 (2)	O(8)—Ni(2)—O(9)	86·38 (7)
Ni(2)—O(9)	2·042 (2)	O(8)—Ni(2)—O(10)	88·72 (7)
Ni(2)—O(10)	2·040 (2)	O(9)—Ni(2)—O(10)	87·69 (7)
S—O(1)	1·471 (2)	O(1)—S—O(2)	110·2 (1)
S—O(2)	1·473 (2)	O(1)—S—O(3)	107·8 (1)
S—O(3)	1·473 (2)	O(1)—S—O(4)	110·4 (1)
S—O(4)	1·449 (2)	O(2)—S—O(3)	107·9 (1)
		O(2)—S—O(4)	109·7 (1)
		O(3)—S—O(4)	110·9 (1)
Hydrogen bonds			
O(5)···O(2)	2·761 (2)	O(8)···O(2)	2·776 (3)
O(5)···O(3^{i})	2·724 (3)	O(8)···O(4^{i})	2·683 (2)
O(6)···O(1^{i})	2·809 (3)	O(9)···O(1^{iv})	2·754 (2)
O(6)···O(3^{iii})	2·957 (3)	O(9)···O(2^{v})	2·847 (2)
O(7)···O(1)	2·812 (3)	O(10)···O(4^{ii})	2·729 (2)
O(7)···O(3^{ii})	2·725 (3)	O(10)···O(8^{v})	2·954 (2)

Symmetry codes: (i) $x-\frac{1}{2}, y+\frac{1}{2}, z$; (ii) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (iii) $-x, 1-y, -z$; (iv) $\frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z$; (v) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

reflections ($\bar{3}\bar{1}1$, $\bar{5}\bar{3}\bar{4}$, $\bar{2}16$, $11\bar{7}$, 425, $71\bar{4}$) had relative intensity variations which were less than ±8%. Averaging of equivalent reflections resulted in 2920 unique reflections for which $R_{int} = 0{\cdot}027$. An absorption correction was applied using the program of de Meulenaer & Tompa (1965) and Alcock (1970); transmission factors ranged from 0·35 to 0·52.

The Ni- and S-atom positions were determined from a Patterson map, and the O atoms were located by subsequent Fourier difference syntheses. Full-matrix least-squares refinement was performed using the *SHELX*76 (Sheldrick, 1976) program, which minimizes the function $\sum w(|F_o| - |F_c|)^2$, where $w = \sigma_F^{-2}$. All unique, non-systematically absent reflections with $I > 0$ were included in the calculations. The positions of the H atoms were determined from a difference map after anisotropic thermal parameters were introduced into the calculation. The H-atom positions could not be successfully refined, however, so these atoms were included in the refinement with fixed positions, but variable isotropic thermal parameters. The final results after convergence were: $R = 0{\cdot}046$, $wR = 0{\cdot}041$, $S = 2{\cdot}1$, $(\Delta/\sigma)_{max} = 0{\cdot}03$, $\Delta\rho_{max} = 0{\cdot}75$, $\Delta\rho_{min} = -0{\cdot}74$ e Å^{-3}. Efforts to refine the structure in the space group *Cc* were unsuccessful.

Neutral-atom scattering factors for Ni, S and O were taken from Cromer & Mann (1968), and the H-atom scattering factor was taken from Stewart, Davidson & Simpson (1965). Correction for anomalous dispersion was included for Ni and S (Cromer & Liberman, 1970).

The final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Selected bond lengths and angles are given in Table 2.

Related literature. The structure of monoclinic $NiSO_4.6D_2O$ has been previously reported by Ptasiewicz-Bak, McIntyre & Olovsson (1983). These authors collected only a minimal data set [max. $(\sin\theta)/\lambda = 0{\cdot}36$ Å^{-1}] and were forced to use a very constrained refinement model; consequently, detailed comparison of the present results with theirs appears unproductive. It should be noted, however, that the two determinations are concordant with respect to the general features of the structures. The structure of tetragonal $NiSO_4.6D_2O$ has been reported by O'Connor & Dale (1966) and that of tetragonal $NiSO_4.6H_2O$ by Stadnicka, Glazer & Koralewski (1987).

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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 44916 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[bis(pentamethylcyclopentadienyl)ytterbium(III)] Ditelluride

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Abstract. $[Yb(C_{10}H_{15})_2]_2Te_2$, $C_{40}H_{60}Te_2Yb_2$, $M_r = 1142{\cdot}20$, monoclinic, $P2_1/n$, $a = 15{\cdot}517\,(3)$, $b = 10{\cdot}611\,(2)$, $c = 13{\cdot}166\,(3)$ Å, $\beta = 114{\cdot}34\,(2)°$, $V = 1975{\cdot}1$ Å^3, $Z = 2$, $D_x = 1{\cdot}92$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 61{\cdot}71$ cm^{-1}, $F(000) = 1088$, $T = 296$ K, $R = 0{\cdot}036$ for 3330 $[F^2 > 2\sigma(F^2)]$ of 4560 total unique data. The Te_2^{2-} ion is on a center of symmetry and lies perpendicular to and between the two Yb atoms. Each Yb atom is bonded approximately tetrahedrally to two cyclopentadienyl ring centers and the Te_2^{2-} ion. Distances (Å) are: ave. Yb—C 2·626 (17), ave. Yb—Cp(centroid) 2·332 (18), Te—Te 2·7686 (11) Å.

Experimental. The complex was isolated from the reaction of $[Yb\{C_5(CH_3)_5\}_2].(C_2H_5)_2O$ with a large excess of tellurium powder in hexane after stirring for 2 days at room temperature. Black air-sensitive crystals were sealed inside quartz capillaries under argon. Crystal, 0·25 × 0·25 × 0·33 mm, with eight faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 28 reflections, $20 < 2\theta < 54°$; analytical absorption correction, range 2·4–3·8; max. $(\sin\theta/\lambda) = 0{\cdot}65$ Å^{-1}; h −20 to 20, k 0 to 13, l −17 to 17; three standard reflections, average decay 0·8%, intensities adjusted accordingly; 8993 data, 4560 unique, $R_{\rm int} = 0{\cdot}023$; structure solved by Patterson and Fourier methods; refined on F, f' and f'' terms included, 199 parameters; H atoms not observed in difference maps and were not included; anisotropic thermal parameters for all atoms refined; $R = 0{\cdot}036$ for 3330 reflections for which $F^2 > 2\sigma(F^2)$; $R = 0{\cdot}055$ all data; $wR = 0{\cdot}052$; $S = 1{\cdot}38$; $w = 4F_o/[\sigma^2(F_o^2) + (0{\cdot}06F_o^2)^2]$; max. (shift/σ) < 0·05; extinction correction $F_{\rm corr} = (1 + 4{\cdot}8 \times 10^{-8}I)F_o$, extinction parameter estimated after each series of refinements, max. correction 11%; max. and min. of ΔF synthesis 1·6 and −1·4 e Å^{-3}; atomic f, f' and f'' for neutral Yb, Te and C from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

Atomic parameters are listed in Table 1, and distances and angles are listed in Table 2.* Fig. 1 shows the molecule and numbering scheme.

Related literature. Comparable Te—Te distances are 2·78 Å in $AuTe_2X$, X = Cl or I (Haendler, Mootz, Rabenau & Rosenstein, 1974), and 2·802 (1) Å in $[Ni\{CH_3C[CH_2P(C_6H_5)_2]_3\}]_2[\mu\text{-}Te_2].2C_4H_8O$ (Di Vaira, Peruzzini & Stoppioni, 1986). The latter is the only structurally characterized compound with the same

Table 1. *Atomic parameters*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Yb	0·16540 (2)	0·07854 (3)	−0·02377 (2)	2·656 (9)
Te	0·05403 (3)	−0·07689 (4)	0·08828 (4)	3·43 (2)
C1	0·1697 (6)	0·2522 (7)	0·1192 (7)	3·7 (2)
C2	0·1706 (5)	0·3167 (6)	0·0264 (7)	3·6 (2)
C3	0·2584 (6)	0·2928 (7)	0·0192 (7)	4·1 (3)
C4	0·3112 (6)	0·2094 (7)	0·1103 (8)	4·4 (3)
C5	0·2554 (6)	0·1844 (7)	0·1702 (7)	4·2 (3)
C6	0·1835 (5)	0·0476 (7)	−0·2110 (6)	3·5 (2)
C7	0·2764 (5)	0·0245 (8)	−0·1270 (6)	3·5 (2)
C8	0·2717 (6)	−0·0846 (7)	−0·0692 (7)	3·7 (2)
C9	0·1786 (6)	−0·1345 (7)	−0·1208 (7)	3·8 (3)
C10	0·1232 (5)	−0·0508 (7)	−0·2093 (6)	3·5 (2)
C11	0·0950 (8)	0·2726 (10)	0·1658 (10)	6·3 (4)
C12	0·0970 (9)	0·4095 (8)	−0·0467 (10)	6·7 (4)
C13	0·2962 (10)	0·3655 (10)	−0·0528 (12)	7·8 (6)
C14	0·4163 (7)	0·1799 (12)	0·1501 (12)	8·1 (5)
C15	0·2877 (10)	0·1090 (11)	0·2775 (9)	7·0 (5)
C16	0·1544 (7)	0·1523 (9)	−0·2991 (8)	5·1 (3)
C17	0·3649 (7)	0·0846 (9)	−0·1232 (9)	5·5 (4)
C18	0·3546 (7)	−0·1478 (10)	0·0237 (7)	5·5 (3)
C19	0·1504 (7)	−0·2631 (7)	−0·0948 (9)	5·1 (4)
C20	0·0263 (6)	−0·0778 (9)	−0·2988 (7)	4·8 (3)

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* Lists of structure factors, anisotropic thermal parameters, additional distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44931 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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