results in: $a = 3{\cdot}872$, $b = 3{\cdot}826$ and $c = 24{\cdot}315$ Å. The structure of $Lu_3Co_2Si_7$, shown in Fig. 5, is described with these permutated unit-cell parameters. The structure consists of AlB_2-type slabs intergrown with double the number of $BaAl_4$-type slabs and strongly deformed Cu_3Au-type slabs. In rough approximation the structure thus resembles the $La_3Co_2Sn_7$ structure (Fig. 3).

Whether or not an $Ho_3Co_2Si_7$-type phase exists in the Lu–Co–Si phase diagram is a difficult question to answer. We can report, however, from microprobe analyses, the existence of a phase with a composition compatible with 5:4:14, but not with 3:2:7, in three samples prepared at nominal compositions: $Lu_3Co_2Si_7$ (1073 K/7 d), $Lu_3Co_2Si_8$ (1073 K/7 d) and $Lu_5Co_4Si_{14}$ (levitated). We cannot exclude the possibility that an $Ho_3Co_2Si_7$-type phase is stable in a different temperature range. In our opinion, however, the coordination around the rare-earth atoms in the Cu_3Au-type slab is unlikely and the crystal structure of $Ho_3Co_2Si_7$ should be reinvestigated. We have tried, on the other hand, to correlate the lattice constants reported for '$Lu_3Co_2Si_7$' with those of $Lu_5Co_4Si_{14}$ assuming, on a hypothetical basis, that a displacement by $\frac{1}{2}c$ of the triple slab (II–III–II) shown in Fig. 1 is possible. This shift will not change the interaction with the neighboring AlB_2 type slabs but should lead, when randomly repeated in the crystal, to an A-centered pseudo-tetragonal orthorhombic unit cell with a metric very similar to the unit-cell parameters given by Yarovets. Naming the original orthorhombic cell parameters A, B and C, the corresponding monoclinic cell parameters of $Lu_5Co_4Si_{14}$ are obtained as follows: $a = \frac{1}{2}(A^2 + B^2)^{1/2}$, $b = 2C$, $c = 2A$, $\beta = 90° + \mathrm{arctg}\, A/B$. Applying these equations to the original orthorhombic data given by Yarovets for the '$Lu_3Co_2Si_7$' compound leads to the following monoclinic parameters: $a = 12{\cdot}307$, $b = 7{\cdot}744$, $c = 7{\cdot}652$ Å and $\beta = 98{\cdot}94°$. These values are in acceptable agreement with the lattice parameters reported for $Lu_5Co_4Si_{14}$ in this work.

We would like to thank Dr J. Steinmetz (Laboratoire de Microanalyse, Université de Nancy I, France) for the electron microprobe analyses. We acknowledge the help of Mrs Birgitta Künzler with the preparation of the drawings and technical assistance from F. Liniger. The study was supported by the Swiss National Science Foundation under contract 2·604–0·85.

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Acta Cryst. (1986). C**42**, 949–952

Structure of Hexasodium *trans*-Diaquatetrasulfatozinc(II)

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(*Received* 21 *November* 1985; *accepted* 17 *March* 1986)

Abstract. $Na_6[Zn(SO_4)_4(H_2O)_2]$, $M_r = 623{\cdot}6$, triclinic, $P\bar{1}$, $a = 6{\cdot}052\,(1)$, $b = 6{\cdot}815\,(1)$, $c = 10{\cdot}141\,(2)$ Å, $\alpha = 70{\cdot}07\,(2)$, $\beta = 77{\cdot}29\,(2)$, $\gamma = 74{\cdot}72\,(2)°$, $V = 375{\cdot}41\,(1)$ Å^3, $Z = 1$, $D_x = 2{\cdot}758$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, μ(Mo $K\alpha$) $= 25{\cdot}1$ cm^{-1}, $F(000) = 308$, room temperature, $R = 0{\cdot}029$ for 1272 observed

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reflections. The structure consists of discrete octahedral $[Zn(SO_4)_4(H_2O)_2]^{6-}$ anions with the Zn^{II} ion occupying a crystallographic center of symmetry. The Zn—O(sulfate) lengths [2·134 (2), 2·135 (2) Å] are significantly longer than the $Zn—OH_2$ distance [2·032 (2) Å].

Introduction. The ternary system sodium sulfate/zinc sulfate/water forms several stable compounds including Tutton's salt $[Na_2Zn(SO_4)_2.6H_2O]$ and $Na_2Zn(SO_4)_2.$-$4H_2O$. Recently we have successfully prepared single crystals of a dihydrate compound, $Na_6Zn(SO_4)_4.$-$2H_2O$. Our interest in this compound is twofold. The vibrational dynamics of water molecules in ionic crystal lattices is particularly important in understanding the nature of the intermolecular hydrogen bond. The availability of a series of compounds in which the H···O—S bond distances vary provides a useful basis for a comparative spectroscopic study, and the possibility of better defining frequency shift–bond distance correlations. The second motive for studying these compounds lies in their electrical conductivity properties. Significant conductivity has been observed in the anhydrous compound prepared from $Na_6Zn(SO_4)_4.$-$2H_2O$, and tentative measurements suggest that the conductivity is of mixed ionic/electronic nature. In order to elucidate more fully the origin and mechanism of this conductivity, it is useful to know the structure of the starting material and any intermediate phases or compounds.

Experimental. Single crystals of $Na_6Zn(SO_4)_4.2H_2O$ were grown by slow evaporation at 371 K from a saturated aqueous solution prepared by adding 70·223 g of $ZnSO_4.7H_2O$ (Mallinckrodt, Inc.) and 50·019 g of Na_2SO_4 (Fisher Scientific Co.) to 150 ml of hot doubly-distilled water. This amount of solute corresponds to a point on the $Na_2SO_4/ZnSO_4/H_2O$ ternary phase diagram from which only the anhydrous compound $Na_6Zn(SO_4)_4$ is reported to crystallize (Benrath & Benrath, 1929; Benrath, 1930). The solution was carefully filtered into a 500 ml Erhenmeyer flask and placed in a thermostated oven. Evaporation was allowed to proceed at a slow constant rate for 14 d. The resulting crystalline mass was composed of many small crystals of no well defined morphology. Imbedded in this mass were a considerable number of larger clear crystals that were of a general rhombohedral shape. The largest of these was about 5 × 6 × 4 mm. Upon exposure to the atmosphere, the crystal faces clouded significantly, suggesting that the dihydrate is unstable with respect to the formation of a higher hydrate. Precautions were taken to prevent this reaction in all manipulations of the compound.

Colorless, irregular multifaceted crystals, 0·25 × 0·25 × 0·25 mm, coated with epoxy to retard possible dehydration. Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Lattice parameters from least-squares fit of 25 reflections with $2\theta \geq 18°$. No absorption corrections applied; 1354 total reflections measured with $\theta/2\theta$ scans in the sphere $2 \leq 2\theta \leq 50$, $0 \leq h \leq 7$, $-8 \leq k \leq 8$, $-12 \leq l \leq 12$. Three standard reflections monitored every 200 reflections for orientation and additionally measured every 2 h for intensity, intensity variation 2% throughout. 1313 unique reflections, 1272 observed reflections with $I_o \leq 2\sigma(I)$. Structure solved by heavy-atom methods, full-matrix least-squares refinement on F, minimizing $\sum w(F_o - |F_c|)^2$. H atoms placed on observed positions and refined with $U(H)$ held at 0·05 Å^2, all non-H atoms refined anisotropically. $R = 0·029$, $wR = 0·040$ for observed reflections, $R = 0·030$, $wR = 0·040$ including weak reflections, $S = 1·82$, $w = (\sigma_F)^{-2}$, max. Δ/σ in final least-squares cycle = 0·02, largest ΔF peak represents 1·0 e Å^{-3} located near the Zn atom. Neutral-atom scattering factors and corrections for anomalous dispersion from *International Tables for X-ray Crystallography* (1974), calculations performed with local modifications of *SHELX* crystallographic programs (Sheldrick, 1976).

Discussion. Fractional atomic coordinates are presented in Table 1;* Table 2 contains interatomic distances and selected bond angles. Fig. 1 illustrates the geometry.

The structure consists of discrete octahedral $[Zn(SO_4)_4(H_2O)_2]^{6-}$ moieties linked by Na^+ ions and hydrogen bonds. The Zn^{2+} ion occupies a crystallographic inversion center and is coordinated to four O atoms each from distinct sulfato groups and two *trans* water molecules. The $Zn—OH_2$ length of 2·032 (2) Å is within the range of $Zn^{II}—OH_2$ distances noted for other precise structural determinations: 2·091 and 2·048 (5) Å in diaquabis(glycolato)zinc(II) (Fischinger & Webb, 1969), 2·088 (5) Å in bis(oxamato)zinc dihydrate (Braibanti, Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1971) and 1·999 (20) Å in monoaquabis(acetylacetonato)zinc (Montgomery & Lingafelter, 1963) which contains a five-coordinate Zn ion. The Zn—O(sulfate) lengths of 2·134 (2), 2·135 (2) Å are significantly longer than the Zn—O(water) distance. A related complex is polymeric $[ZnSO_4.H_2O]$ (LeFur, Coing-Boyat & Bassi, 1966) whose Zn coordination is identical to that of the title compound, *i.e.* two *trans* water oxygen ligands and four sulfato-oxygen ligands. The Zn—O distances reported for $[ZnSO_4.H_2O]$ (at $R = 0·06$) are 2·05, 2·08 Å for Zn—O(sulfate) and

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* Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42920 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

2·15 Å for Zn—O(water). These values differ significantly from those for $[Zn(SO_4)_4(OH_2)_2]^{6-}$ and illustrate the flexibility of the Zn—O bond and its dependence on packing forces. Three-dimensional arrays of alternating Zn and sulfate ions are also seen in the structure of $\{Zn[C(NH_2)_3]_2(SO_4)_2\}$ (Morimoto & Lingafelter, 1970). There the Zn ion is tetrahedrally coordinated to sulfato-O atoms at a distance of 1·94 Å, and when compared to the title structure confirms our expectations of decreasing metal–ligand distance with decreasing coordination number.

Table 1. *Atomic parameters*

	x	y	z	U_{eq}†
Zn(1)	1·0	0·0	0·0	0·0128 (1)
S(1)	1·12357 (9)	−0·26617 (9)	0·32546 (6)	0·0108 (2)
S(2)	0·55463 (9)	0·20474 (9)	0·19979 (6)	0·0111 (2)
O(11)	1·1750 (3)	−0·1156 (3)	0·1815 (2)	0·0152 (5)
O(12)	0·9882 (3)	−0·1442 (3)	0·4207 (2)	0·0198 (5)
O(13)	0·9874 (3)	−0·4122 (3)	0·3179 (2)	0·0195 (5)
O(14)	1·3465 (3)	−0·3860 (3)	0·3727 (2)	0·0199 (5)
O(21)	0·3656 (3)	0·3519 (3)	0·1268 (2)	0·0210 (5)
O(22)	0·4630 (3)	0·0976 (3)	0·3459 (2)	0·0164 (5)
O(23)	0·6667 (3)	0·0426 (3)	0·1238 (2)	0·0198 (5)
O(24)	0·7289 (3)	0·3231 (3)	0·1984 (2)	0·0191 (5)
O(1)	0·9656 (3)	−0·3041 (3)	0·0376 (2)	0·0162 (5)
Na(1)	0·5617 (2)	0·7081 (2)	0·1072 (1)	0·0251 (3)
Na(2)	1·0409 (2)	0·2150 (1)	0·3332 (1)	0·0207 (3)
Na(3)	0·6081 (2)	−0·2621 (2)	0·4636 (1)	0·0224 (3)
H(1)	1·012 (7)	−0·356 (7)	0·119 (5)	0·05
H(2)	1·083 (7)	−0·356 (6)	−0·019 (4)	0·05

† $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$ for non-H atoms; $U_{eq} = B/8\pi^2$ for H atoms.

Table 2. *Geometric parameters*

Anionic bond distances (Å)			
Zn—O(11)	2·135 (2)	S(2)—O(21)	1·457 (2)
Zn—O(23)	2·134 (2)	S(2)—O(22)	1·467 (2)
Zn—O(1)	2·032 (2)	S(2)—O(23)	1·493 (2)
S(1)—O(11)	1·493 (2)	S(2)—O(24)	1·482 (2)
S(1)—O(12)	1·462 (2)	O(1)—H(1)	0·85 (4)
S(1)—O(13)	1·479 (2)	O(1)—H(2)	0·88 (4)
S(1)—O(14)	1·468 (2)		
Selected bond angles (°)			
O(23)—Zn—O(11)	92·9 (1)	O(22)—S(2)—O(21)	109·5 (1)
O(1)—Zn—O(11)	87·5 (1)	O(23)—S(2)—O(21)	108·8 (1)
O(1)—Zn—O(23)	86·1 (1)	O(23)—S(2)—O(22)	109·5 (1)
O(12)—S(1)—O(11)	109·1 (1)	O(24)—S(2)—O(21)	109·7 (1)
O(13)—S(1)—O(11)	109·6 (1)	O(24)—S(2)—O(22)	110·5 (1)
O(13)—S(1)—O(12)	109·0 (1)	O(24)—S(2)—O(23)	108·8 (1)
O(14)—S(1)—O(11)	107·1 (1)	S(1)—O(11)—Zn	131·8 (1)
O(14)—S(1)—O(12)	111·4 (1)	S(2)—O(23)—Zn	129·7 (1)
O(14)—S(1)—O(13)	110·6 (1)	H(1)—O(1)—H(2)	104 (3)
Sodium–oxygen distances* (Å)			
$Na(1^{i})$—O(1)	2·383 (2)	$Na(2^{i})$—O(13)	2·430 (2)
$Na(1^{ii})$—O(11)	2·418 (2)	$Na(2^{v})$—O(22)	2·486 (2)
$Na(1^{iii})$—O(21)	2·470 (2)	$Na(2^{v})$—O(21)	2·634 (2)
Na(1)—O(24)	2·474 (2)	Na(3)—O(22)	2·353 (2)
$Na(1^{i})$—O(23)	2·589 (2)	$Na(3^{iv})$—O(14)	2·392 (2)
$Na(1^{ii})$—O(14)	2·660 (2)	$Na(3^{vi})$—O(14)	2·438 (2)
$Na(2^{iv})$—O(12)	2·350 (2)	$Na(3^{vii})$—O(22)	2·456 (2)
Na(2)—O(12)	2·384 (2)	Na(3)—O(12)	2·535 (2)
Na(2)—O(24)	2·386 (2)	Na(3)—O(13)	2·608 (2)
Hydrogen-bond contacts (Å)			
H(1)···O(13)	1·903	$H(2^{viii})$···O(24)	1·897

* Only those Na—O contacts <2·7 Å are listed. Transformations of indicated atoms: (i) $x, y-1, z$; (ii) $1+x, y-1, z$; (iii) $1-x, 1-y, -z$; (iv) $2-x, -y, 1-z$; (v) $x-1, y, z$; (vi) $1+x, y, z$; (vii) $1-x, -y, 1-z$; (viii) $2-x, -y, -z$.

The geometric parameters collected in Table 2 illustrate nonequivalence in the S—O bonds. The longest S—O distance in each of the two independent sulfate groups involves the O atom that is additionally bound to Zn [S(1)—O(11) = 1·493 (2) and S(2)—O(23) = 1·493 (2) Å]. Of the remaining sulfate O atoms, the next longest S—O bonds are to those O atoms participating in hydrogen bonds [S(1)—O(13) = 1·479 (2) and S(2)—O(24) = 1·482 (2) Å]. Overall however the average S—O length [1·475 (14) Å] and O—S—O angle [109·5 (1·0)°] are well within the range of values for other inorganic metal sulfate complexes: 1·41–1·51 Å for S—O and 107–112° for O—S—O (LeFur, Coing-Boyat & Bassi, 1966).

The sodium sulfate dihydrate compound provides a very useful point in the series of compounds which form in the ternary sodium sulfate/zinc sulfate/water system. In Tutton's salt, $Na_2Zn(SO_4)_2.6H_2O$, the crystal system is monoclinic (space group C_{2h}^5, $Z = 2$) with the Zn ion occupying the special position C_i and surrounded by a slightly distorted octahedron of water molecules (Hofmann, 1930, 1931). The tetrahydrate, $Na_2Zn(SO_4)_2.4H_2O$, also occurs in the same space group as Tutton's salt (Giglio, 1958) and the Zn atom also occupies the special position C_i; however, it is surrounded by a slightly distorted octahedron consisting of four water molecules and two O atoms from nearby sulfate ions. In the dihydrate studied here this trend of Zn-ion coordination continues. Now the space group is triclinic, but as in the case of the tetrahydrate and the hexahydrate, all atoms except Zn occupy general sites. Here the Zn is also found in a site of C_i symmetry; however, the octahedral coordination is defined by the two water molecules and four O atoms of four nearby sulfate ions.

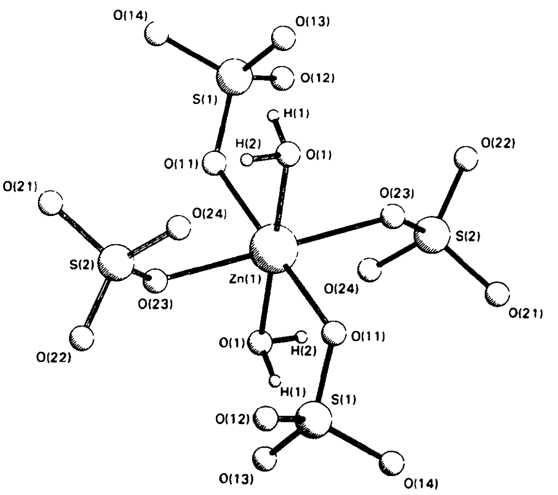

Fig. 1. Drawing by *PLUTO* (Motherwell, 1978) illustrating the numbering and geometry of the $[Zn(SO_4)_4(OH_2)_2]^{6-}$ anion. The central Zn atom occupies a crystallographic inversion center.

In terms of the vibrational dynamics of the water molecules this compound will probably be the most interesting in the series. Since there are only two water molecules per Zn ion, this is the simplest system in which intermolecular coupling effects between water molecules can be expected. Moreover, although the space group is $P\bar{1}$ with the two water molecules related through an inversion center, the water molecule sits on a site of general symmetry. Therefore this system provides an excellent test of the relative importance of site group effects *versus* correlation field effects.

This material is based upon work supported by the National Science Foundation under grant No. DMR 8319473.

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Acta Cryst. (1986). C**42**, 952–956

Structure of a Dihydrated, Di-*tert*-butyl Alcohol Solvated Zinc Acetate Complex with Erythromycin A at 188 K

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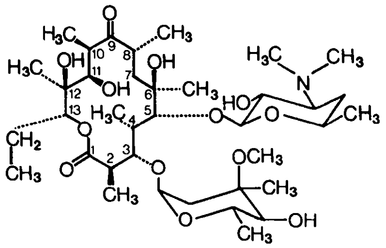
(*Received* 18 *September* 1985; *accepted* 22 *January* 1986)

Abstract. $[Zn(C_2H_3O_2)_2(C_{37}H_{67}NO_{13})(H_2O)].2C_4H_{10}O.2H_2O$, $M_r = 1119\cdot72$, orthorhombic, $P2_12_12_1$, $a = 13\cdot352\,(3)$, $b = 20\cdot013\,(6)$, $c = 22\cdot052\,(10)$ Å, $V = 5892\cdot6$ Å^3, $Z = 4$, $D_x = 1\cdot26$ g cm^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0\cdot71073$ Å, $\mu = 4\cdot95$ cm^{-1}, $F(000) = 2424$, final $R = 0\cdot053$ for 3344 unique reflections with $|F| > 3\sigma(|F_o|)$. The absolute structure of the complex was determined. The structure consists of an aquazinc complex and four molecules of solvation, two water molecules and two *tert*-butyl alcohol molecules. The complex has a six-coordinate Zn^{II} ion bonded to two acetate ligands, a water molecule, and the desosamine substituent of the erythromycin A molecule. The important bond lengths are Zn—N 2·124 (6), Zn—O(desosamine) 2·178 (4), Zn—O(water) 2·157 (5) and Zn—O(acetate) 2·024 (6), 2·107 (6) and 2·170 (6) Å.

Introduction. A formulation of erythromycin A (shown below and hereinafter referred to as ER) and zinc acetate has been developed by The Procter & Gamble Company for use as a topical anti-acne medication. The medication was shown in clinical studies (Feucht, Allen & Chalker, 1980) to be safe and effective for acne therapy. The results from a separate clinical investigation (Strauss & Stranieri, 1984) indicated that the mechanism of action of the zinc acetate/ER preparation is probably a direct antibacterial effect on *Propionibacterium acne*. The crystal structure determination was initiated to characterize the interaction between zinc acetate and ER. No other transition-metal complex with ER has been reported.



Erythromycin A

(I)

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