Table 1. *Powder data for* Zn_2Zr_3

hkl	I/I_1	*d* (obs.)	*d* (calc.)
112	20	2·92 Å	2·926 Å
220	44	2·69	2·699
022	75	2·57	2·572
221	38	2·52	2·516
122	100	2·44	2·438
130	63	2·41	2·414
131	63	2·28	2·281
013	31	2·23	2·221
222, 113	15	2·13	2·133, 2·133
230	18	2·12	2·117
132	20	1·98	1·984
140	22	1·85	1·851
232	18	1·81	1·809
331, 004	22	1·74	1·742, 1·741
033, 240	5	1·71 *B*	1·715, 1·707
042, 133	3	1·67	1·673, 1·673
114	3	1·65	1·657
142	31	1·63	1·635
332	31	1·595	1·598
124	22	1·550	1·551
242, 340	22	1·529	1·533, 1·527
—	3	1·511	—
—	20	1·490 *B*	—
—	38	1·460	—
—	31	1·447	—
—	15	1·421	—
—	38	1·410	—
—	5	1·387	—
—	38	1·372	—
—	31	1·346	—
—	31	1·310	—
—	44	1·271	—
—	8	1·252	—
—	18	1·236	—
—	5	1·218	—

Table 1 (*cont.*)

hkl	I/I_1	*d* (obs.)	*d* (calc.)
—	20	1·205	—
—	15	1·187	—
—	5	1·170	—
—	3	1·160	—
—	10	1·135 *B*	—
—	8	1·109	—
—	5	1·097	—
—	10	1·078 *B*	—
—	10	1·065	—
—	13	1·042	—
—	5	1·033	—
—	5	1·027	—
—	8	1·018	—
—	8	1·010	—
—	5	1·000	—
—	8	0·990	—
—	5	0·983	—
—	8	0·974	—
—	8	0·960	—

Note that *B* indicates a *broad* line.

Relative intensities were established by visual comparison against calibrated standards.

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Acta Cryst. (1961). **14**, 329

X-ray investigation of the anhydrous cadmium and mercuric sulphates.

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Continuing the systematic X-ray investigation of the unstable anhydrous sulphates of bivalent metals undertaken by our Department, we are studying the structure determination of $CdSO_4$ and $HgSO_4$. The two substances were found to be isostructural and different from the sulphates studied so far. In this preliminary communication are given the unit-cell dimensions and the space group of the compounds.

According to the literature both substances crystallize in the orthorhombic system. Crystallographic measurements are given only for $CdSO_4$, which, however, owing to the chance approximation of the angle $(011):(01\bar{1})$ to that of $ZnSO_4$, was erroneously assumed to be isomorphous to it (Groth, 1908). The values of the angles $(110):(1\bar{1}0) = 89° 58'$ and $(011):(01\bar{1}) = 70° 52'$ given by Groth, differ considerably from the values 89° 48′ and 71° 26′ respectively, measured on crystals prepared by us. The latter represent the mean of a series of measurements on different crystals, which yielded somewhat differing values because of imperfect face growth. Their approximation to the values 89° 49′ and 71° 20′ respectively, calculated from the lattice constants, is closer than that of the values given in Groth.

No crystallographic measurements on $HgSO_4$ crystals have been reported in the literature. On the crystals prepared by us the values of the interfacial angles, corresponding to those of $CdSO_4$ given above, are 89° 27′ and 72° 28′, in satisfactory agreement with the values 89° 34′ and 72° 27′ calculated from the lattice constants.

The systematic extinctions on Weissenberg and precession photographs made necessary a reorientation of the crystal axes, so as to make the space-group symbol agree with that given in the *International Tables* (1952). The changes are as follows:

$$\text{Groth } a \rightarrow c_0, \quad \text{Groth } b \rightarrow a_0, \quad \text{Groth } c \rightarrow b_0 .$$

The cell dimensions given below were obtained from powder diagrams, taken with a calibrated 9 cm. Unicam camera for $CdSO_4$ and with a Norelco diffractometer for $HgSO_4$. The indexing of the powder diagrams was carried out by using the lattice constants obtained from

Weissenberg and precession diagrams. Cu $K\alpha$ radiation was used ($\lambda = 1{\cdot}5418$ Å).

Cadmium sulphate ($CdSO_4$)

$$a_0 = 4{\cdot}709 \pm 0{\cdot}001,\ b_0 = 6{\cdot}562 \pm 0{\cdot}0015,$$
$$c_0 = 4{\cdot}694 \pm 0{\cdot}001\ \text{Å};\ V = 145{\cdot}05\ \text{Å}^3.$$

The axial ratios

$$a_0 : b_0 : c_0 = 0{\cdot}7176 : 1 : 0{\cdot}7153$$

are in fair agreement with those obtained from the crystallographic measurements

$$a : b : c = 0{\cdot}7190 : 1 : 0{\cdot}7165\ .$$

Mercuric sulphate ($HgSO_4$)

$$a_0 = 4{\cdot}821 \pm 0{\cdot}0005,\ b_0 = 6{\cdot}581 \pm 0{\cdot}0007,$$
$$c_0 = 4{\cdot}785 \pm 0{\cdot}0005\ \text{Å};\ V = 151{\cdot}83\ \text{Å}^3.$$
$$a_0 : b_0 : c_0 = 0{\cdot}7325 : 1 : 0{\cdot}7271$$
$$a : b : c = 0{\cdot}7328 : 1 : 0{\cdot}7258\ \text{(goniometrically)}.$$

For a unit-cell content of two units ($CdSO_4$) or ($HgSO_4$) densities of 4·757 and 6·487 g.cm.$^{-3}$ respectively are calculated, as compared with the values 4·691 and 6·470 g.cm.$^{-3}$ given in the literature (*Handbook of Chemistry and Physics*, 1958).

The only systematic extinctions are those of the $hk0$ reflexions when $h + k$ is odd.

Assuming from their appearance that the crystals are holohedral we deduce D_{2h}^{12}–$Pmmn$ as the most probable space group for both substances.

We wish to express our thanks to the Royal Hellenic Research Foundation for supporting our research program by donating a precession camera to our Department.

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The lattice constants of some metal-fluoroborate hexahydrates.

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Although it has been stated (West, 1935) that the fluoroborates, $M(BF_4)_2 . 6\ H_2O$ ($M = Mg^{2+}$, Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+}), are isomorphous with the corresponding perchlorates, none of their lattice constants appear to have been recorded. The lattice constants and measured and calculated densities are recorded in Table 1, where they are compared with the values obtained by West for the perchlorates. The Mg, Mn, Fe, Co, Ni, and Zn salts are hexagonal and are very similar in size to the corresponding perchlorates: the cadmium salts have a closely related trigonal structure with a one half of that shown in Table 1; the true value is doubled for comparison with the other salts. West has shown that the copper salts are not isomorphous with other divalent salts. Theory (Orgel & Dunitz, 1957) would predict a distortion of the octahedra of oxygen atoms about the Cu^{2+} ions.

Lithium fluoroborate exists in at least two forms. $LiBF_4 . H_2O$, stable above 23°, is tetragonal, $a = 5{\cdot}74$, $c = 4{\cdot}88$ Å. $LiBF_4 . 3\ H_2O$ crystallizes from aqueous solution below 23 °C. and is hexagonal, isomorphous with the corresponding perchlorate (West, 1935). The only phase that we could crystallize from such solutions is hexagonal, $a = 9{\cdot}90$, $c = 5{\cdot}53$ Å, but is not isomorphous with the perchlorate trihydrate.

The hydrates were prepared from solutions of the appropriate carbonates in fluoroboric acid. X-ray powder photographs were taken with a 9-cm. camera using Cu $K\alpha$, Co or Cr radiation.

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Table 1. *Lattice constants and densities*

	$M''(ClO_4)_2 . 6\ H_2O$				$M''(BF_4)_2 . 6\ H_2O$			
M''	a	c	Measured density	Calculated density	a	c	Measured density	Calculated density
Mg	15·52 Å	5·26 Å	1·981	1·99	15·36 Å	5·38 Å	1·849	1·85
Mn	15·70	5·30	2·102	2·10	15·46	5·44	1·982	1·98
Fe	15·58	5·24	2·147	2·17	15·49	5·33	2·038	2·02
Co	15·52	5·20	2·198	2·22	15·33	5·22	2·081	2·11
Ni	15·46	5·17	2·252	2·25	15·32	5·16	2·136	2·16
Zn	15·52	5·20	2·252	2·26	15·24	5·30	2·120	2·16
Cd	15·92*	5·30	2·368	2·38	15·96*	5·58	2·292	2·12

* See text.