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The Crystal Structure of the Cubic Cadmium Phosphorus Sulphide Iodide $Cd_{13}P_4S_{22}I_2$

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Crystals of $Cd_{13}P_4S_{22}I_2$ have been grown by vapour transport. They are cubic, space group $F\bar{4}3m$; $a = 9{\cdot}969$ (1) Å; $Z = 1$. The intensities were measured on a Nonius CAD-4 diffractometer. A Patterson synthesis revealed the basic structure. A least-squares refinement, taking into account anisotropic temperature factors, isotropic extinction and anomalous scattering for the Cd atoms, led to a final R of 0·065. The structure consists of a framework of interpenetrating S–I icosahedra, forming a tetrahedrally close-packed anion sublattice which is closely related to the Laves phase $MgCu_2$. The P atoms are exactly tetrahedrally coordinated by four S atoms. The Cd atoms occupy two different positions: Cd(1) is situated in a distorted tetrahedron consisting of two S and two '(S,I) atoms', *i.e.* positions containing S and I in a statistical distribution. Cd(2) is in triangular coordination by three anions.

Introduction

In the system Cd–P–S only one compound, cadmium hypothiosulphate $Cd_2[P_2S_6]$ (with formally tetravalent P), has been reported (Klingen, Ott & Hahn, 1973; Klingen, Eulenberger & Hahn, 1973). Recently, we have found (Nitsche, Grieshaber & Bubenzer, 1976) that another compound, $Cd_{14}P_4S_{24}$ (with pentavalent P), exists. It is monoclinic ($Z = 1$) and X-ray data indicate that its structure is nearly identical to the also monoclinic (space group Cc) structure of $Cd_{16}Ge_4S_{24}$ (Nitsche, 1964; Susa & Steinfink, 1971) and to the isomorphous $Cd_{16}Si_4S_{24}$ (Krebs & Mandt, 1972). Furthermore, we have found that a closely related cubic structure can be obtained if one replaces two S atoms of the anion sublattice of $Cd_{14}P_4S_{24}$ by two I atoms. The resulting compound, $Cd_{13}P_4S_{22}I_2$, contains, for electrochemical neutrality, only 13 Cd atoms.

Crystals of $Cd_{13}P_4S_{22}I_2$ (bright yellow tetrahedra up to 2 × 2 × 2 mm) are obtained by reacting the elements in the proper ratio at 650 °C and vapour-transporting the product in a temperature gradient of 650 to 575 °C. This paper presents the structure of $Cd_{13}P_4S_{22}I_2$ and describes its relation to the monoclinic (although pseudocubic) $Cd_{16}Ge_4S_{24}$ structure.

Experimental

Crystal data

Vapour grown, cubic $Cd_{13}P_4S_{22}I_2$ crystals show $\{111\}$ combined with $\{1\bar{1}1\}$ and occasionally $\{100\}$ faces, and are yellow and transparent. The formula was confirmed by chemical analysis; F.W. 2544·31. Lattice parameters (20 °C): $a = 9{\cdot}969\ (1)$ Å, $V = 990{\cdot}6\ (2)$ Å^3, $Z = 1$, $D_m = 3{\cdot}98$, $D_c = 4{\cdot}26$ g cm^{-3}. Reflexion conditions: hkl: $h+k$, $k+l$, $l+h = 2n$; space group: $F\bar{4}3m$. Mo $K\alpha$, $\lambda = 0{\cdot}7109$ Å; $\mu(\text{Mo } K\alpha) = 96$ cm^{-1}. Crystal size: $140 \times 140 \times 190$ μm. The cell parameters were obtained by a least-squares refinement of Guinier powder data [$\lambda(\text{Cu } K\alpha_1) = 1{\cdot}54051$ Å] with the X-RAY 70 system (*PARAM*; Stewart, Kundell & Baldwin, 1970).

Data collection

The crystal was set with [110] parallel to the axis of the goniometer. Intensities were collected on a computer-controlled four-circle diffractometer (Nonius CAD-4) with Mo $K\alpha$ radiation from a graphite monochromator. The intensities were measured with a scintillation counter. The ω–2θ scan method was used for an octant of the sphere of reflexion between $2\theta = 7{\cdot}07$ and 89·65°. 1126 reflexions were measured of which 247 were independent. Of these, 90 had intensities $I \geq 3\sigma(I)$, where $\sigma(I)$ is the variance of observations. The internal $R = \sum|I - I_m|/\sum I_m$ was 0·044 (I_m = mean intensity).

Data reduction

The X-RAY 70 system (Stewart, Kundell & Baldwin, 1970) was used for data reduction, structure solution and refinement. Lorentz–polarization and absorption corrections were applied. The absorption corrections, based on the measured crystal dimensions, varied between 4·0 and 3·3. Scattering factors for Cd, P, S and I were taken from *International Tables for X-ray Crystallography* (1968).

Structure determination and refinement

Wilson statistics and physical measurements (a positive piezo effect and second-harmonic generation on irradiation with a pulsed Nd-glass laser) indicated noncentrosymmetry and uniquely determined the space group as $F\bar{4}3m$.

A Patterson map displayed peaks suggesting occupancy of the 48-fold equipoints (h), the 16-fold equipoints (e) and at least two fourfold equipoints. We assigned 16 of the 22 S atoms to the position (e). The remaining six S atoms, with the two I atoms, were statistically distributed over the two fourfold positions (a) and (c), with mean atomic scattering factors corresponding to an S:I ratio of 6:2. Atoms in these positions will be designated by (S, I). The 13 Cd atoms were assumed to occupy approximately 27% of the 48-fold equipoints (h) in a random fashion.

Fourier and difference maps were calculated with these atomic positions. The maps clearly revealed the four P atoms on the fourfold equipoints (b). In addition, partial occupancy of the 96-fold general position (i) was indicated. This must have been caused by part of the Cd atoms because all other atoms were already on fully occupied positions. Thus a reasonable distri-

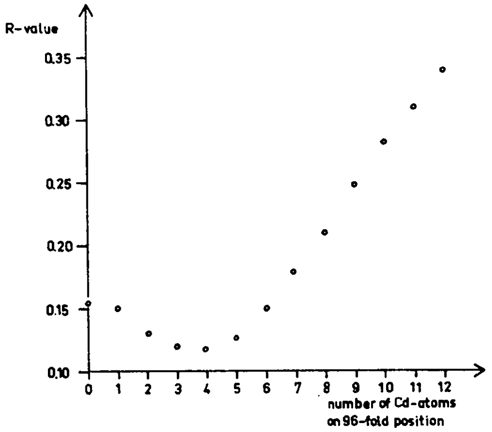

Fig. 1. R as a function of the Cd distribution on 48-fold and 96-fold positions. (Isotropic temperature factors, all parameters fixed.)

Table 1. *Fractional coordinates and thermal parameters*

All values are $\times 10^4$. Estimated standard deviations are in parentheses. The Debye–Waller terms are defined as

$$T = \exp\left[-2\pi^2 \sum_i^3 \sum_j^3 a_i^* a_j^* h_i h_j U^{ij}\right]$$

for Cd and $T = \exp[-2\pi^2 U^{11}(2 \sin \theta/\lambda)^2]$ for S, (S,I) and P. W = Wyckoff position, P = % occupancy.

W	P		x	y	z	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
48(h)	18·8	Cd(1)	1780 (10)	1780 (10)	180 (10)	520 (40)	520 (40)	420 (40)	−110 (50)	−90 (20)	−90 (20)
96(i)	4·2	Cd(2)	1260 (40)	2000 (30)	470 (30)	1300 (400)	300 (100)	700 (200)	−10 (150)	−250 (250)	170 (140)
4(b)	100	P	5000 (0)	5000 (0)	5000 (0)	280 (40)					
16(e)	100	S	6186 (4)	6186 (4)	6186 (4)	410 (20)					
4(a)	100	(S,I) (1)	0 (0)	0 (0)	0 (0)	550 (30)					
4(c)	100	(S,I) (2)	2500 (0)	2500 (0)	2500 (0)	550 (30)					

bution of the 13 Cd on (*i*) and (*h*) had to be found. Therefore, after finding approximate atomic coordinates by isotropic refinement, *R* was calculated as a function of the distribution of the Cd atoms on the positions (*i*) and (*h*). The result is shown in Fig. 1. A distinct minimum is seen to occur when four Cd are statistically distributed on the 96-fold position (*i*) and nine on the 48-fold position (*h*).

A least-squares refinement was carried out with isotropic temperature factors for the S, I and P atoms. For the Cd atoms anisotropic temperature factors were used; isotropic extinction (Larson, 1967) was introduced as an additional parameter, and the *f* values were corrected for anomalous scattering. The values $\Delta f'$ and $\Delta f''$ were taken from *International Tables for X-ray Crystallography* (1974).

The function minimized was $\sum w||F_m - F_c||^2$, where $w = 1/\sigma^2(F)$. Refinement was stopped when the shifts of all parameters were less than one third of the corresponding standard deviations. The final $R(=\sum||F_m| - |F_c||/\sum F_m)$ was 0·065.*

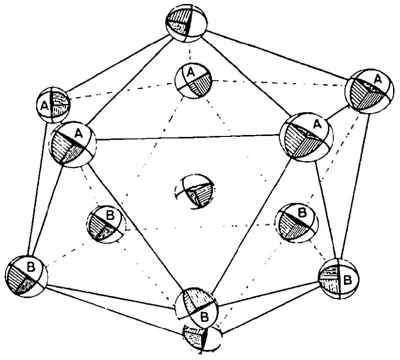

Fig. 2. (Drawn by *ORTEP*: Johnson, 1965). An anion icosahedron. Two of the twelve possible pentatomic rings are marked as *A* and *B*.

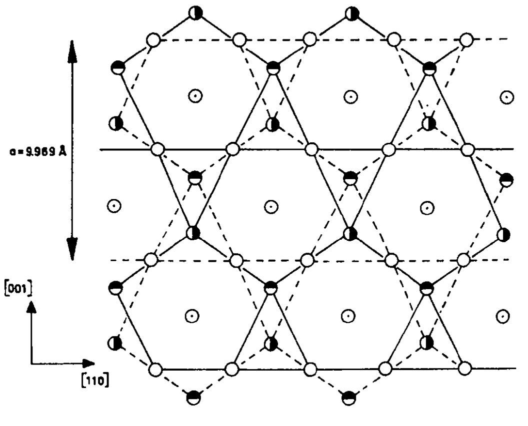

Fig. 3. Projection of the anion sublattice along [1$\bar{1}$0]. Two neighbouring primary layers (pentagon–triangle nets – – – – and ——); and three secondary layers (triangle nets) are shown. For explanation of atomic symbols see Figs. 4 and 5. The S atoms with dots constitute the centres, the upper and the lower points of the icosahedra generated.

Structural results and discussion

Table 1 gives the atomic coordinates, the percentage occupancy of the various equipoints and the thermal parameters. The 13 Cd atoms are distributed statistically in a ratio of 9:4 between the 48-fold position (*h*): (*x*,*x*,*z*) [Cd(1) with $x = 0{\cdot}178$; $z = 0{\cdot}018$] and the 96-fold position (*i*): (*x*,*y*,*z*) [Cd(2) with $x = 0{\cdot}126$; $y = 0{\cdot}200$, $z = 0{\cdot}047$]. The P atoms are located in the fourfold position (*b*): $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$. 16 (of the 22) S atoms occupy the 16-fold position (*e*): (*x*,*x*,*x*) [S with $x = 0{\cdot}6186$]. The remaining six S atoms, with the two I atoms, are statistically distributed among the two fourfold positions (*a*): (0,0,0) [(S,I) (1)] and (*c*): $(\frac{1}{4},\frac{1}{4},\frac{1}{4})$ [(S,I) (2)].

The structure is best understood by first considering the sublattice formed by S^{2-} and I^-. The anions form a tetrahedrally close-packed structure, consisting of face-sharing, distorted tetrahedra, some of which are potential sites for the cations P^{5+} and Cd^{2+}. Within this anion structure, a framework of centred, interpenetrating, slightly distorted icosahedra can be discerned. Each anion is thus surrounded by 12 nearest anion neighbours, situated at the vertices of an icosahedron. In Fig. 2 such a centred anion icosahedron is shown. It is seen that its triangular faces, together with the lines connecting its vertices with the centre, form 20 pseudotetrahedral pyramids. Additional pseudotetrahedra arise as interstices between the icosahedra.

The spatial connexion of the anion icosahedra is best described by a general scheme for coding layered, tetrahedrally close-packed structures (Pearson & Shoemaker, 1969; Shoemaker & Shoemaker, 1972).† According to these authors, a large variety of icosahedral structures (*e.g.* many intermetallic phases) can be generated by the alternate stacking of so-called primary layers (consisting of pentagon–triangle nets of atoms) and secondary layers (consisting of triangle nets or their tessellations only). The stacking is understood to be such that all pentagons of successive primary layers are covered asymmetrically by the pentagons of neighbouring primary layers, whereas the atoms of the interleaving secondary layers centre all (and only) the pentagons of the primary layers.

Fig. 3 shows a projection along the [1$\bar{1}$0] directions of two primary and three secondary layers of the present structure. Each primary layer consists of triangles and two kinds of slightly distorted pentagons (Fig. 4), differing in angles and edge lengths. In both

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31833 (2pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

† Another classification of close-packed tetrahedral groupings of atoms occurring in complex alloys has been given by Frank & Kasper (1958, 1959).

pentagons two neighbouring vertices are 'pure' S positions [16(*e*)] whereas the other three are 'mixed' (S, I) positions, corresponding to (S, I) (1) in 4(*a*) and (S, I) (2) in 4(*c*). The distance between two primary nets is 3·52 Å. The triangular, secondary nets consist of 'pure' S atoms only. From Fig. 3 it is seen how the layers generate a network of icosahedra, partially sharing vertices, partially sharing faces. The pentagonal S–(S, I) rings in the primary layers are perfectly flat, their planes corresponding to ⟨110⟩. The secondary nets are not exactly planar, the deviation from the middle between the primary nets being less than 0·1 Å.

In the coding scheme developed by Pearson & Shoemaker (1969), the icosahedral anion sublattice of $Cd_{13}P_4S_{22}I_2$ has the (simplified) code $P(0;L)$, which applies also, for example, for the Laves phase $MgCu_2$.

Fig. 5 is a projection of the structure along [1$\bar{1}$0], giving the heights of the various atoms above the (110) plane. The distortion of each icosahedron is such that of the 20 (pseudotetrahedral) pyramids formed by its triangular faces and the centre (as their common apex), two are perfect tetrahedra, although of slightly different sizes. Both are formed by 'pure' S positions only. In the midpoints of the smaller tetrahedra [4(*b*)] the P atoms are located, the P–S distances being 2·05 Å. The larger tetrahedra [4(*d*)] remain empty. For clarity these perfect tetrahedra are not outlined in Fig. 5. However, in the icosahedron at the lower left, around the P atom at 0·262, a tetrahedron, formed by two S at 0·262, one S at 0·025 and one S at 0·500 can be seen. The corresponding empty tetrahedron is formed by the two S atoms at 0·762, and those at 0·025 and 0·500.

Cd atoms occur in two typical positions. The Cd(1) which (partially) occupy 48(*h*) can reside in several of the pseudotetrahedral interstices in or between the anion icosahedra. In Fig. 5 one of several possible Cd distributions is shown. In the lower left, a Cd atom (height 0·567) is located in a pseudotetrahedron within an icosahedron. At the right, in the middle, another pseudotetrahedron is outlined which lies between two icosahedra and contains a Cd atom at height 0·406. The Cd(2) atoms, which (partially) occupy 96(*i*), lie on some of the triangular faces of the icosahedra, *i.e.* their coordination by S and (S, I) is nearly triangular with Cd–S distances ranging from 2·40–2·45 Å. The Cd(2) atoms thus occupy a triangular face shared by two pseudotetrahedra. This situation is outlined by dotted lines in Fig. 5: the Cd(2) at 0·589 lies practically in the plane formed by S and (S, I) at height 0·762 and (S, I) at height 0·262.

Table 2 contains the bond angles and the Cd–S, Cd–(S, I) distances of all Cd atoms occurring in the structure. As mentioned before, the structure of $Cd_{13}P_4S_{22}I_2$ is closely related to the monoclinic (but pseudocubic) structure (*Cc*) of $Cd_{16}Ge_4S_{24}$ (Susa & Steinfink, 1971) and the isomorphous $Cd_{16}Si_4S_{24}$ (Krebs & Mandt, 1972), although it may seem surprising that these chemically simpler compounds are of lower symmetry. Their structures can be described by stacking the same primary and secondary layers as in $Cd_{13}P_4S_{22}I_2$, the only difference being that the pentatomic rings are no longer planar and the coordination for Ge (Si) is not exactly tetrahedral (as it is for P). However, the maximum deviation in each direction from the corresponding cubic positions is not more than 0·2 for the S and 0·3 Å for the Ge (Si) atoms. Furthermore it is remarkable that in $Cd_{16}Ge_4S_{24}$ all 16 Cd atoms are in (pseudotetrahedral) positions that correspond to the equipoint 48(*h*) in $Cd_{13}P_4S_{22}I_2$. Positions corresponding to the triangular coordination of 96(*i*) are not occupied. If one surveys all pseudotetrahedra in $Cd_{16}Ge_4S_{24}$, containing equipoints corresponding to 48(*h*), one finds that neighbouring pseudotetrahedra sharing faces or edges are never occupied simultaneously by Cd. This can be understood on the basis of the Cd–Cd distances involved which would be less than

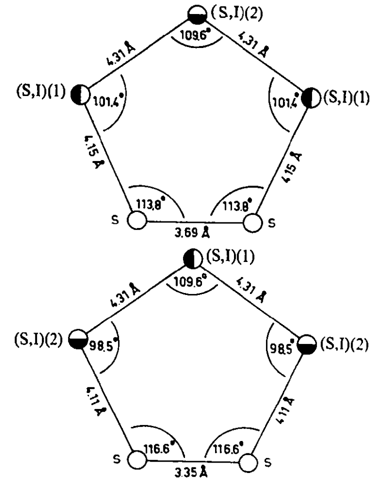

Fig. 4. The two different pentagons of the primary nets.

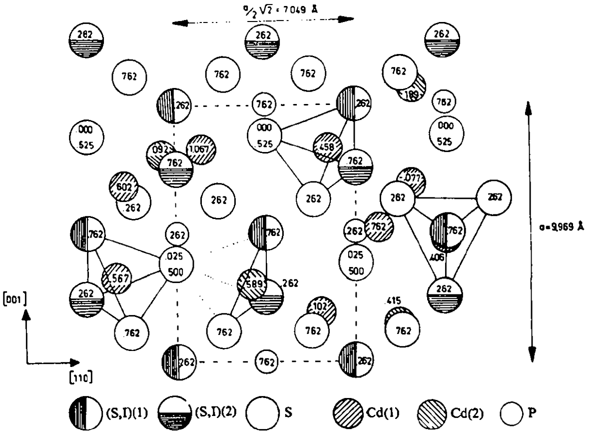

Fig. 5. Projection of the $Cd_{13}P_4S_{22}I_2$ structure along [1$\bar{1}$0]. Note the perfect tetrahedral coordination of P by four S atoms and the two possible coordinations of Cd: (*a*) distorted tetrahedral (solid lines) by two S and two (S, I) atoms and (*b*) trigonal planar (dotted lines) by S and (S, I). The Cd distribution shown has been arbitrarily chosen; many others are possible.

3 Å for occupied, neighbouring pseudotetrahedra. This appears rather unrealistic in comparison with, for example, the Cd–Cd distance of 4·11 Å in CdS. In fact, the smallest Cd–Cd distance in $Cd_{16}Si_4S_{24}$ (Krebs & Mandt, 1972) is 3·83 Å. Thus the partial occupancy of 48(*h*) in $Cd_{13}P_4S_{22}I_2$ is understandable.

Table 2. *Bond lengths and angles of the* Cd *environments*

Standard deviations are in parentheses.

Distorted tetrahedral environment			
(S,I) (1)–Cd(1)	2·52 (1) Å	(S,I) (1)–Cd(1)–(S,I) (2)	117·6 (4)°
(S,I) (2)–Cd(1)	2·53 (1)	(S,I) (1)–Cd(1)–S	111·4 (4)
S———Cd(1)	2·51 (1)	(S,I) (1)–Cd(1)–S^i	111·4 (4)
S^i———Cd(1)	2·51 (1)	(S,I) (2)–Cd(1)–S	109·5 (4)
		(S,I) (2)–Cd(1)–S^i	109·5 (4)
		S———Cd(1)–S^i	95·0 (4)
Triangular environment			
(S,I) (1)–Cd(2)	2·40 (4) Å	(S,I) (1)–Cd(2)–(S,I) (2)	127 (2)°
(S,I) (2)–Cd(2)	2·42 (4)	(S,I) (1)–Cd(2)–S	117 (2)
S———Cd(2)	2·46 (4)	(S,I) (1)–Cd(2)–S^i	96 (1)
S^i———Cd(2)	3·14 (4)	(S,I) (2)–Cd(2)–S	115 (2)
		(S,I) (2)–Cd(2)–S^i	94 (1)
		S———Cd(2)–S^i	82 (1)

Superscript (i) in S^i refers to the transformation $\bar{x}+\frac{1}{2},\bar{x}+\frac{1}{2},x$ of the parameters in Table 1.

Assuming that also in this compound face- and edge-sharing pseudotetrahedra cannot be occupied simultaneously by Cd, then only 16 of the possible positions of 48(*h*) remain available. In $Cd_{16}Ge_4S_{24}$ a certain set (of several possible ones) of 16 positions is fully occupied by Cd. In $Cd_{13}P_4S_{22}I_2$, however, three of the 16 Cd sites would have to remain empty. These 'holes' would strain the lattice unduly and therefore a fraction of the 13 Cd shift to the positions 96(*i*) where they occupy a triangular plane common to two pseudotetrahedra. The partial occupancy in $Cd_{13}P_4S_{22}I_2$ of 48(*h*) and 96(*i*) by the 13 Cd thus 'stabilizes' the lattice and compensates for the 'loss' in Cd caused by partial substitution of the S sublattice by I. The question why the S and the (S,I) sublattices in these compounds prefer icosahedral rather than cubo-octahedral coordination (as in cubic and hexagonal close packings) cannot be answered unambiguously. Partly it seems connected with the asymmetric electronic d^{10} configuration of Cd (the isomorphous Hg compounds exist but the Zn analogues do not), and partly it appears to result from the large polarization power of the small, highly charged ions Si^{4+}, Ge^{4+} and P^{5+}.

Icosahedral sublattices in sulphides appear to be not uncommon. We shall soon report on other icosahedral sulphides containing [Cu+In] (instead of two Cd) or Cu only in combination with P^{5+} and Si^{4+}.

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