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The Crystal Structure of α-CdP_2

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The crystal structure of α-CdP_2 is orthorhombic with $a=9{\cdot}9_0$, $b=5{\cdot}40_8$ and $c=5{\cdot}17_1$ Å. The space group is $Pna2_1$ with four molecules in the unit cell. The phosphorus atoms are closely bonded in chains parallel to the *c* axis, the chains being linked together by tetrahedrally coordinated cadmium atoms. The average P–P bond length (2·22 Å) in a chain is much less than that (4·14 Å) in a Cd–P tetrahedron.

Introduction

The unit cell of CdP_2 was shown by Stackelberg & Paulus (1935) to be tetragonal with dimensions $a=5{\cdot}28$ and $b=19{\cdot}70$ Å. Recently, Berak & Pruchnik (1968) carried out a detailed investigation of phase equilibria in the cadmium–phosphorus system and found that CdP_2 could exist in two crystalline phases: an orthorhombic low temperature form (α-CdP_2) with $a=9{\cdot}89$, $b=5{\cdot}168$ and $c=5{\cdot}403$ Å, and a high temperature modification (β-CdP_2) with cell parameters similar to those reported by Stackelberg & Paulus. The equilibrium temperature between these two phases was estimated to be 420 °C.

Samples of Cd_2P_3 recently prepared in this department have been found to contain dark red crystals which were identified as α-CdP_2 by X-ray analysis. The specimen had been prepared by heating stoichiometric amounts of cadmium and phosphorus for 72 hours in a sealed evacuated tube, the phosphorus being maintained at 500 °C at one end of the tube and the cadmium at 850 °C at the other. After heating, the tube was slowly cooled to room temperature over a period of four hours. The resulting grey polycrystalline mass of Cd_2P_3 was found to contain some crystals of α-CdP_2 which were of suitable size for a structural investigation by the Weissenberg technique.

X-ray data

A crystal, elongated parallel to [012] and of cross-section 0·065 × 0·040 mm perpendicular to the *c* axis, was selected for intensity measurements. Five layer lines were recorded on equi-inclination Weissenberg photographs taken about the *c* axis with Cu *Kα* radiation. Accurate values of cell parameters were determined from X-ray powder data taken with Cu *Kα* radiation by a crystal-focusing camera of 22·9 cm effective diameter.

Crystal data

Orthorhombic with $a=9{\cdot}90\pm0{\cdot}01$, $b=5{\cdot}408\pm0{\cdot}005$, $c=5{\cdot}171\pm0{\cdot}005$ Å.
$Z=4$, $V=276{\cdot}9$ Å^3.
$D_x=4{\cdot}18$ g.cm^{-3}, $D_m=4{\cdot}19\pm0{\cdot}16$ g.cm^{-3}
$\mu=739$ cm^{-1} (Cu *Kα* radiation).
Space group $Pna2_1$ or *Pnam* (non-standard setting of *Pnma*). Systematic absences *0kl* for $k+l$ odd, *h0l* for *h* odd. $Pna2_1$ is consistent with the deduced structure.

The intensities of 289 reflexions were estimated visually from multiple-film exposures, observed data being corrected for the Lorentz-polarization factor and for spot shape on upper layer lines (Phillips, 1954). An allowance was made for absorption by applying the correction factors given by Bond (1959), assuming the specimen to be cylindrical in shape with the axis of the cylinder parallel to **c** and an average radius of 0·0288 mm ($\mu r=2{\cdot}13$).

Because of the uncertainty resulting from high absorption corrections, no attempt was made to collect intensity data about another axis for the purpose of scaling. Scaling was, in fact, achieved by carefully controlling exposure and photographic processing for each layer.

The structure analysis began with a Patterson synthesis projected along [001]. A minimum function map was then constructed to help locate the phosphorus atoms in addition to the heavy Cd atom. This showed the presence of the Cd atom at (0·16, 0·10, *z*) and two small peaks which could be attributed to non-equivalent P atoms, P(1) and P(2), at approximately (0·10, 0·45, *z*) and (0·00, 0·25, *z*), respectively. An electron density projection along [001], calculated by the use of

Table 1. *Atomic parameters*

Origin on 2_1. Standard deviations are given in brackets.
Equipoints are all 4(*a*).

	x	*y*	*z*	*B*
Cd	0·1529 (3)	0·1016 (5)	0·2606 (26)	1·95 (5) Å^2
P(1)	0·1186 (10)	0·4458 (21)	0·5850 (30)	1·34 (16)
P(2)	−0·0074 (10)	0·2697 (19)	−0·0676 (33)	1·16 (15)

the observed structure factors and phases determined from the position of the Cd atom, confirmed the coordinates of Cd, P(1) and P(2) suggested by the minimum function map. With 8 phosphorus atoms in the cell, P(1) and P(2) would have to be situated at the equipoint 4(*c*) of the space group *Pnam*, so that all the atoms in the cell would lie in two planes at $z=\frac{1}{4}$ and $z=\frac{3}{4}$. This structure would possess some unacceptably short interatomic distances and an unreasonable coordination of the Cd atom. In the space group $Pna2_1$ these difficulties do not arise, since *z* can take a general value for each set of atoms at the equipoint 4(*a*). On these grounds the space group *Pnam* was rejected and the structure was solved in the space group $Pna2_1$.

Two cycles of Fourier refinement reduced the reliability index, $R=\Sigma\ ||F_o|-|F_c||/\Sigma\ |F_o|$, from 0·28 to 0·1 for the *hk*0 data. The structure factor calculations were made by the use of atomic scattering factors for the neutral atoms listed in *International Tables for X-ray Crystallography* (1962) and assuming temperature factors of 2·5 Å^2 for Cd and 1·5 Å^2 for P.

Assuming reasonable values for Cd–P and P–P separations, packing considerations suggested the approximate *z* parameters for all the atoms, which, when used with the more accurate *x* and *y* parameters gave a value of $R=0{\cdot}29$ for the full three-dimensional intensity data. The structure was now refined by means of the three-dimensional least-squares program of Daly, Stephens & Wheatley (1963) and after several cycles the value of *R* was reduced to 0·15. The observed structure factors were now rescaled to the calculated data for each layer, and a further cycle of refinement reduced *R* to 0·136; at this stage the shifts in the atomic parameters were less than the estimated standard deviations. The final values of atomic parameters are given in Table 1. Values of *R* and scale factors for each layer are given in Table 2, and observed and calculated structure factors are listed in Table 3.

Table 2. *Final R indices and scale factors*

Layer (*l*)	*R*	Final scale factors
0	0·124	1·001
1	0·132	1·195
2	0·133	0·972
3	0·145	0·954
4	0·164	1·016

Description of the structure

A projection of the structure along [001] is shown in Fig. 1, in which it is seen that each P atom is bonded to two Cd atoms in such a way that each Cd is surrounded by four P atoms lying at the corners of a somewhat distorted tetrahedron. The Cd–P and P–P bond lengths and angles in such a tetrahedron are given in Table 4;

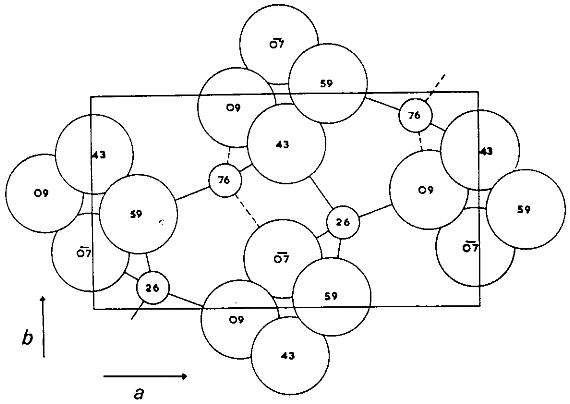

Fig. 1. Projection of the structure viewed along the *c* axis. Large and small circles represent P and Cd atoms, respectively. The numbers are the *z* coordinates of the atoms expressed as hundredths of the *c* parameter. The *c* axis is directed into the plane of the diagram. Broken lines indicate bonds from Cd to P atoms at a height *c* above those shown in the diagram.

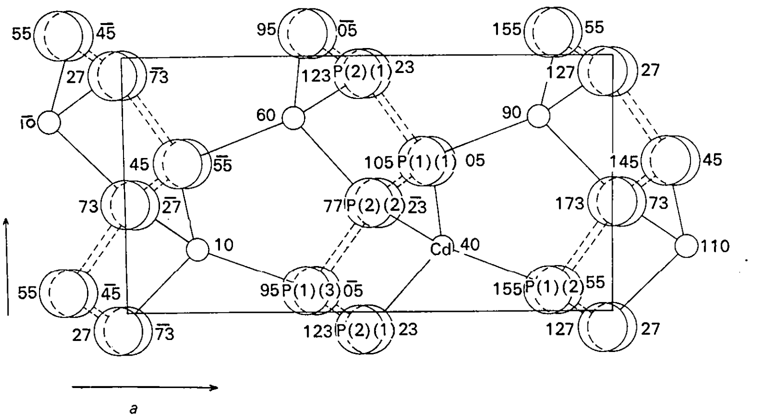

Fig. 2. Projection of the structure viewed along the *b* axis. Short P–P bonds are shown by broken lines. Chains of closely linked P atoms, one above the other in the projection, have been displaced slightly from each other parallel to the *a* axis. The numbers are the *y* coordinates of the atoms expressed as hundredths of the *b* parameter. The *b* axis is directed out of the plane of the diagram.

Table 3. *Calculated and observed structure factors on an absolute scale*

Nine unobserved reflexions were assigned a value of $\frac{1}{2}I_{\min}$.

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
2	0	0	6	4	-4	0
4	0	0	104	108	-108	0
6	0	0	122	118	118	0
8	0	0	61	60	60	0
10	0	0	53	35	-35	0
12	0	0	29	22	22	0
1	1	0	50	38	38	0
2	1	0	92	103	-103	0
3	1	0	88	97	-97	0
4	1	0	55	57	57	0
5	1	0	33	30	30	0
6	1	0	54	49	49	0
7	1	0	58	53	53	0
8	1	0	47	36	-36	0
9	1	0	65	60	-60	0
10	1	0	13	9	9	0
11	1	0	17	14	-14	0
12	1	0	31	27	27	0
0	2	0	43	35	35	0
1	2	0	91	97	-97	0
2	2	0	54	48	-48	0
3	2	0	21	16	-16	0
4	2	0	74	82	-82	0
5	2	0	78	91	91	0
6	2	0	7	7	-7	0
7	2	0	52	50	-50	0
8	2	0	7	1	-1	0
9	2	0	42	40	-40	0
10	2	0	36	26	-26	0
11	2	0	51	48	48	0
12	2	0	16	15	-15	0
1	3	0	22	23	-23	0
2	3	0	101	129	-129	0
3	3	0	54	55	55	0
4	3	0	53	52	52	0
5	3	0	17	18	18	0
6	3	0	47	52	52	0
7	3	0	20	20	-20	0
8	3	0	55	61	-61	0
9	3	0	20	11	11	0
10	3	0	12	11	-11	0
11	3	0	12	13	13	0
0	4	0	46	48	-48	0
1	4	0	27	24	-24	0
2	4	0	46	49	49	0
3	4	0	10	8	8	0
4	4	0	56	66	66	0
5	4	0	28	31	31	0
6	4	0	29	31	-31	0
7	4	0	25	27	-27	0
8	4	0	9	10	10	0
9	4	0	12	9	-9	0
10	4	0	42	46	46	0
1	5	0	45	50	-50	0
2	5	0	18	15	-15	0
3	5	0	42	49	49	0
4	5	0	7	2	-2	0
5	5	0	20	18	-18	0
6	5	0	18	18	18	0
7	5	0	47	51	-51	0
8	5	0	10	11	11	0
0	6	0	54	60	-60	0
1	6	0	34	36	36	0
2	6	0	5	0	0	0
3	6	0	16	17	17	0
4	6	0	26	22	22	0
5	6	0	28	36	-36	0
6	6	0	29	35	-35	0
2	0	1	102	141	-130	-54
4	0	1	82	87	87	-6
6	0	1	46	40	35	20
8	0	1	92	87	-87	-7
10	0	1	34	27	16	-22
12	0	1	43	38	36	-10
0	1	1	72	74	-70	23
1	1	1	115	122	-121	19
2	1	1	72	64	49	40
3	1	1	50	49	-44	23
4	1	1	68	81	67	45
5	1	1	79	101	101	-7
6	1	1	47	49	-43	24
7	1	1	36	28	-20	-19
8	1	1	24	14	5	13
9	1	1	54	42	-42	5
10	1	1	54	45	42	16
11	1	1	41	33	29	15
12	1	1	18	17	-9	14
1	2	1	96	38	-88	2
2	2	1	38	31	-20	-23
3	2	1	103	115	113	-11
4	2	1	26	26	26	3
5	2	1	25	20	-4	-20
6	2	1	24	25	6	24
7	2	1	67	77	-77	-4
8	2	1	25	22	-20	10
9	2	1	44	36	35	8
10	2	1	17	12	12	-3
11	2	1	23	19	18	-5
12	2	1	15	15	15	3
0	3	1	107	119	-105	-56
1	3	1	34	27	25	11
2	3	1	30	35	24	-25
3	3	1	12	10	2	9
4	3	1	42	47	47	1
5	3	1	30	29	-27	-10
6	3	1	64	78	-76	-20
7	3	1	22	17	14	-10
8	3	1	31	31	-9	-30
9	3	1	11	10	10	2
10	3	1	40	43	40	-14
11	3	1	19	17	-17	2
1	4	1	44	41	-32	25
2	4	1	63	69	69	-2
3	4	1	58	56	56	1
4	4	1	44	43	-42	-7
5	4	1	17	11	9	-6
6	4	1	32	31	-31	-3
7	4	1	32	33	-30	13
8	4	1	37	40	40	-1
9	4	1	25	23	14	19
10	4	1	13	10	-7	-8
0	5	1	28	21	21	-1
1	5	1	54	56	56	3
2	5	1	19	15	7	14
3	5	1	23	18	18	1
4	5	1	26	30	-5	29
5	5	1	48	55	-55	-0
6	5	1	17	17	6	16
7	5	1	24	21	20	5
8	5	1	14	11	11	-1
1	6	1	7	8	8	-0
2	6	1	33	34	32	9
3	6	1	37	35	-29	-19
4	6	1	23	22	-22	1
5	6	1	14	18	4	-17
6	6	1	12	11	-11	-3
2	0	2	56	75	73	-19
4	0	2	87	103	95	-40
6	0	2	84	76	-67	-36
8	0	2	7	12	11	1
10	0	2	65	67	67	1
12	0	2	31	28	-17	-21
1	1	2	90	82	-77	-27
2	1	2	79	69	69	-3
3	1	2	101	106	101	33
4	1	2	41	42	-40	-12
5	1	2	25	21	1	21
6	1	2	24	18	-18	-2
7	1	2	71	67	-66	-15
8	1	2	46	48	48	2
9	1	2	35	27	26	-8
10	1	2	20	12	-3	-11
11	1	2	23	18	16	7
12	1	2	17	18	-16	-10
0	2	2	74	61	-44	42
1	2	2	98	103	99	25
2	2	2	35	28	-8	26
3	2	2	39	37	33	17
4	2	2	13	8	-6	5
5	2	2	78	95	-93	-19
6	2	2	31	40	-38	11
7	2	2	27	24	23	-5
8	2	2	32	28	-8	26
9	2	2	45	40	39	11
10	2	2	24	18	7	17
11	2	2	34	34	-33	4
1	3	2	30	25	21	-14
2	3	2	70	72	72	-7
3	3	2	26	20	-19	-4
4	3	2	49	57	-56	-7
5	3	2	12	13	13	3
6	3	2	29	32	-28	15
7	3	2	22	23	22	-7
8	3	2	50	53	51	16
9	3	2	21	17	-12	-12
10	3	2	17	18	-17	-6
0	4	2	66	83	83	-3
1	4	2	51	47	44	18
2	4	2	27	20	-8	-18
3	4	2	23	25	20	15
4	4	2	42	41	-34	-24
5	4	2	37	42	-39	-16
6	4	2	42	53	53	-7
7	4	2	14	15	7	-13
8	4	2	15	18	17	-6
9	4	2	22	21	21	5
1	5	2	37	32	28	15
2	5	2	23	21	-11	-18
3	5	2	54	63	-63	-1
4	5	2	6	5	2	-4
5	5	2	9	7	-3	6
6	5	2	10	15	11	10
7	5	2	34	35	33	12
0	6	2	32	26	24	8
1	6	2	22	20	-20	5
2	6	2	16	21	-20	7
3	6	2	11	9	-3	9
4	6	2	25	30	-28	10
5	6	2	18	20	20	-1
2	0	3	84	129	128	21
4	0	3	54	63	-62	-14
6	0	3	60	68	-68	-11
8	0	3	58	49	48	10
10	0	3	5	4	-1	-3
0	1	3	119	106	103	22
1	1	3	65	53	51	14
2	1	3	20	[illegible]	6	4
3	1	3	8	4	0	4
4	1	3	32	25	-25	-1
5	1	3	54	55	-54	-13
6	1	3	54	61	59	14
7	1	3	41	41	41	5
8	1	3	31	30	29	7
9	1	3	29	25	24	6
10	1	3	[illegible]	11	-11	-1
11	1	3	35	39	-39	-6
1	2	3	43	35	34	8
2	2	3	54	49	48	6
3	2	3	72	80	-78	-18
4	2	3	13	8	-8	-2
5	2	3	16	13	13	0
6	2	3	20	20	-20	-1
7	2	3	42	45	44	9
8	2	3	22	18	18	5
9	2	3	44	46	-45	-8
10	2	3	13	14	14	2
0	3	3	77	77	76	10
1	3	3	37	32	-32	-5
2	3	3	49	47	-46	-12
3	3	3	16	17	-17	-2
4	3	3	76	91	-89	-17
5	3	3	25	27	26	4
6	3	3	28	29	29	5
7	3	3	6	3	-2	-2
8	3	3	12	7	7	-2
9	3	3	14	16	-15	-3
1	4	3	21	17	16	7
2	4	3	51	49	-48	-11
3	4	3	21	13	-12	-4
4	4	3	25	30	29	6
5	4	3	22	25	25	3
6	4	3	16	14	14	3
7	4	3	24	23	23	6
8	4	3	35	39	-39	-8
0	5	3	29	28	28	4
1	5	3	43	42	-41	-8
2	5	3	19	16	16	4
3	5	3	11	10	-10	-2
4	5	3	8	4	-1	4
5	5	3	36	45	44	9
6	5	3	7	6	6	3
1	6	3	27	33	-32	-6
2	6	3	32	33	-33	-6
3	6	3	17	21	21	2
2	0	4	45	45	-32	-31
4	0	4	56	73	-47	-56
6	0	4	45	54	54	-9
8	0	4	23	2	-2	-1
10	0	4	45	45	-41	-18
1	1	4	55	47	47	-1
2	1	4	48	45	-40	-21
3	1	4	57	65	-65	-2
4	1	4	26	28	27	2
5	1	4	15	18	-4	17
6	1	4	12	15	14	5
7	1	4	48	44	44	6
8	1	4	39	32	-30	-12
9	1	4	24	23	-17	-15
10	1	4	6	10	5	-8
0	2	4	40	46	15	43
1	2	4	70	64	-63	-8
2	2	4	18	22	-5	21
3	2	4	26	23	-23	5
4	2	4	15	5	-3	3
5	2	4	48	62	61	11
6	2	4	32	26	18	18
7	2	4	20	19	-15	-12
8	2	4	18	24	-2	24
9	2	4	31	28	-28	-3
1	3	4	21	20	-9	-17
2	3	4	46	54	-46	-28
3	3	4	14	16	15	3
4	3	4	40	38	36	11
5	3	4	12	6	-6	-0
6	3	4	23	27	16	22
7	3	4	18	17	-12	-12
8	3	4	32	38	-38	-2
0	4	4	63	55	-49	-25
1	4	4	40	30	-30	3
2	4	4	11	17	13	-11
3	4	4	22	16	-14	8
4	4	4	25	31	30	-9
5	4	4	33	28	28	-2
6	4	4	40	37	-31	-21
7	4	4	8	14	-3	-14
1	5	4	24	24	-24	3
2	5	4	16	15	9	-12
3	5	4	43	44	40	18

these were calculated by the use of a program written by Daly, Stephens & Wheatley (1963).

The interesting feature of the structure is that the phosphorus atoms are densely populated in chains parallel to the *c* axis; this situation can be seen at positions $(\frac{1}{2},0)$ and $(0,\frac{1}{2})$ in Fig. 1. In each chain the atoms lie in two sets at the equivalent positions of a 2_1 screw axis of the space group, and alternate pairs of atoms are separated by distances of 2·05 and 2·39 Å, respectively. These bonds lengths are much shorter than the average P–P separation (4·14 Å) in a Cd–P tetrahedron, suggesting strong covalent bonding between adjacent atoms in a chain.

The way in which the chains are linked together by Cd atoms is shown in Fig. 2. Although this diagram is

Table 4. *Bond lengths and angles*

Cd–P tetrahedron
Bond lengths

Standard deviations are 0·015 Å for Cd–P and 0·018 Å for P–P.

Cd–P(1)(1)	2·529 Å	P(1)(1)–P(1)(2)	4·557 Å
Cd–P(1)(2)	2·579	P(1)(1)–P(2)(1)	3·722
Cd–P(2)(1)	2·495	P(1)(1)–P(2)(2)	4·100
Cd–P(2)(2)	2·627	P(1)(2)–P(2)(1)	4·302
		P(1)(2)–P(2)(2)	4·278
		P(2)(1)–P(2)(2)	3·901

Table 4 (*cont.*)

Angles

Standard deviations are 0·5° in each case.

P(1)(1)–Cd–P(1)(2)	126·3°
P(1)(1)–Cd–P(2)(1)	95·6
P(1)(1)–Cd–P(2)(2)	105·4
P(1)(2)–Cd–P(2)(1)	115·9
P(1)(2)–Cd–P(2)(2)	110·5
P(2)(1)–Cd–P(2)(2)	99·2

Short P–P bonds

Bond lengths

Standard deviations are 0·018 Å in each case.

P(1)(1)–P(2)(1)	2·386 Å
P(1)(1)–P(2)(2)	2·050

Angles

Standard deviations are 0·7° in each case.

P(2)(1)–P(1)(1)–P(2)(2)	108·0°
P(1)(1)–P(2)(2)–P(1)(3)	105·7

For location of atoms see Fig. 2.

really a projection of the structure along [010], P atoms at the same position in the projection, but a distance *b* apart, are slightly displaced from each other to give the diagram a three-dimensional effect. The atoms in each chain are joined by broken lines and the diagram shows three rows of chains, the chains in each row being one above the other perpendicular to the plane of the projection. Such rows are joined together by one quarter of the tetrahedral bonds from Cd to P(1) in each case. The chains in each row are joined by the remaining tetrahedral bonds, two to P(2) atoms and one to a P(1) atom from each Cd.

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On the Additivity of Crystal Radii in Alkali Halides

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The additivity of crystal radii in alkali halides has been studied by applying the method of least squares to the experimental nearest-neighbour distances, d_{ah}. The result is a basic set of crystal radii, r_a and r_h ($r_{\mathrm{Li}}=1{\cdot}038$, $r_{\mathrm{Na}}=1{\cdot}295$, $r_{\mathrm{K}}=1{\cdot}617$, $r_{\mathrm{Rb}}=1{\cdot}759$, $r_{\mathrm{C}}=2{\cdot}017$, $r_{\mathrm{F}}=1{\cdot}019$, $r_{\mathrm{Cl}}=1{\cdot}534$, $r_{\mathrm{Br}}=1{\cdot}695$, $r_{\mathrm{I}}=1{\cdot}934$ Å), and an additive correction c (r_a+c, r_h-c). Theoretically the constant c has the value zero. The best agreement of the least-square radii with the experimental radii was achieved with the value $c_{\mathrm{exp}}=(-0{\cdot}114\pm0{\cdot}014)$ Å. The cation radii decreased and anion radii increased in comparison with the basic values ($c=0$) resulting from the ionic character of alkali halides. The root-mean-square deviation of the radii sum, r_a+r_h, from the experimental d_{ah} values is 0·021 Å, and the r.m.s. deviation of the values r_a+c_{exp}, r_h-c_{exp} from the experimental radii, 0·058 Å.

Crystal radius

Disregarding the small non-sphericity of atoms in a crystal lattice and taking into account the low compressibility of crystalline solids, a crystal can be approximated to a solid composed of hard atomic spheres. Strictly, of course, those spheres are neither hard nor spherical, as can be seen quite clearly from electron density maps (see *e.g.* Witte & Wölfel, 1955). Therefore, the radius of an atom in a crystal – crystal radius – does not have any accurate meaning and can have various definitions.

The classical way to determine the crystal radii in a family of salts, as in the alkali halides, is to fix the radius ratios of the alkali and halogen ions in one crystal by use of a suitable physical criterion. Well known examples are the radii evaluated by Wasastjerna (1923), Goldschmidt (1926), Pauling (1927) and Zachariasen (1931).

Fumi & Tosi (1964) have reported a set of crystal radii based on the Pauling and Huggins–Mayer forms (Fumi & Tosi, 1964), and on the Born–Mayer form (Tosi & Fumi, 1964) of the repulsive energy (see also Tosi, 1964). The procedure is cumbersome, but yields values in fairly good agreement with the experimental ones derived from the X-ray measurements of electron distributions in crystals (Inkinen & Järvinen, 1968; Järvinen & Inkinen, 1967; Krug, Witte & Wölfel,