different, and if the space group in either compound is indeed *Cmc*2_1, they may be very different; the best we can do is to report the averages, which we will call the M–CN distances. If we assume that the C–N distance is 1·16 Å (Britton, 1967), then we calculate from the *c* values for the average M–CN distances, 2·45, 2·40, and 2·55 Å for $(CH_3)_3SnCN$, $(C_2H_5)_3SnCN$, and $(CH_3)_3PbCN$, respectively. The difference between the first two numbers is an indication of the sensitivity of this bond distance to the environment. The difference between the first and third is what we would expect from the univalent radii for Sn^{4+} and Pb^{4+}, 0·96 and 1·06 Å, respectively (Pauling, 1960).

In the absence of low temperature facilities we do not anticipate any further work on either of these compounds.

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Polytypism and spiral growth in cadmium iodide.

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A new measurement of the refractive index of CdI_2 has been used to re-evaluate the step heights of growth spirals on the surfaces of CdI_2 crystals reported by Trigunayat & Verma. There is now good correlation between step heights and crystal structure since the corrected step height is an integral multiple of the *c* dimension of the unit cell for 30 out of 36 spirals on 33 crystals with polytype structures having between two and twenty iodide layers in the unit cell.

A careful study of polytypism in cadmium iodide was reported by Trigunayat & Verma (1962) who used an interference technique to measure the step heights of growth spirals on the surfaces of CdI_2 single crystals whose structure type was then determined by X-ray diffraction. They concluded that in most crystals there was no correlation between step height and the *c* dimension of the unit cell determined by X-ray methods. The step heights were measured by the internal interference method (Forty, 1952) which gives the *optical* height of a step (*i.e.* height × refractive index). A value of 1·849 ± 0·001 was used for the ordinary refractive index of CdI_2 at the wavelength of the mercury green line $\lambda = 5461$ Å. (The ordinary refractive index is appropriate for microscope observations, using narrow pencil illumination, of CdI_2 platelets with *c* axes perpendicular to their habit plane.)

Recent studies of the refractive index and dispersion of CdI_2 by interference methods (Tubbs, 1969; Lee, Said, Davies & Lim, 1969) show that the ordinary refractive index at $\lambda = 5461$ Å is 2·39 ± 0·04 and the birefringence 0·2. This value is also consistent with 2·5 ± 0·1 quoted by Fotland (1959) for observations in 'white light'. A more accurate value for the ordinary index may be obtained from measurements of step heights since all steps must be integral multiples of $M = 6{\cdot}84$ Å, the thickness of the basic I–Cd–I layer.

A set of step height measurements is shown in Table 1 for eight crystals where the steps were less than 15 I–Cd–I

Table 1. *Corrected step heights* (*using refractive index of* 2·41) *for small steps on* CdI_2 *crystals where the step height is known to an accuracy of* ± 0·26 *basic layers or better*

Crystal number	Step height* in multiples of $M = 6{\cdot}84$ Å	Corrected step height (units of *M*)	Structure type
24	19·33 ± 0·26	14·8	4*H*
35	12·00 ± 0·22	9·2	unidentified
37	13·09 ± 0·26	10·0	unidentified
42	13·71 ± 0·22	10·5	resembles 4*H*
47	17·01 ± 0·05	13·0	resembles 2*H*
56	10·29 ± 0·25	7·9	16*H*
62	18·19 ± 0·12	14·0	2*H* + 14*H*
66	15·66 ± 0·23	12·0	22*H*

* From Trigunayat & Verma (1962)

layers high and the error in step measurement was quoted as being 0·26 layer or less. The first column gives the crystal number following Trigunayat & Verma (1962) while the second gives their measurement of step height. The corrected step heights in column three have been calculated from those in column two using a value of 2·41 for the refractive index since this gives the smallest deviations of step heights from integral numbers of I–Cd–I layers. With the exception of crystal number 42 (for which the step height is never integral for an index in the range 2·39 ± 0·04), all the corrected step heights are integers within the quoted errors. The structure type for each crystal determined by X-ray diffraction is given in column 4 of Table 1 and it is particularly satisfying to note that crystal 56 shows a step height of 7·9 basic layers and a c dimension of eight basic I–Cd–I layers (the notation $16H$ means that there are 16 iodide layers or 8 I–Cd–I layers in the unit cell). Crystal 62 showed coalescence and polytypes $2H$ and $14H$ were identified; the corrected step height is 14 basic layers in this case.

A further 25 crystals are listed in Table 2. In all cases the structure type was unambiguously determined by Trigunayat & Verma (1962) and the step height is either known to better than 0·4 basic layers or the c dimension is so large that an error of one or two layers introduces no ambiguity. The first four columns give crystal number *etc.* as in Table 1, while the fifth column notes the degree of correlation between step height and X-ray c dimension. In Table 3 step heights and structure type are listed for a further 19 crystals where the step height was accurately determined but the structure type could only be classified as 'resembling' $2H$ or $4H$. The $4H$ polytype is expected to occur frequently since steps of increasing height may be constructed by adding alternate ($A\gamma B$) and ($C\alpha B$) layers and the $4H$ polytype occurs whenever the step is 2,4,6, … $2n$ basic layers high.

There are eight crystals of $2H$ type in Tables 2 and 3 and in all cases there is good correlation between step heights and X-ray c dimensions. This is not surprising since the step height need only be an integral number of basic I–Cd–I layers to give correlation for the $2H$ polytype consists of iodide ions in hexagonal close packing ($A\gamma B$). There is, however, correlation within the experimental errors (using the errors of column two rather than the slightly smaller ones that could be placed on step heights in column three) which are as small as $0{\cdot}05M$ in one case. For the sixteen $4H$ crystals, ($A\gamma B$) ($C\alpha B$), there is correlation in twelve cases, one is doubtful and there is no correlation within the quoted errors for the remaining three crystals. The $6H$ polytype (crystal 45) shows correlation but the 'resembles $8H$' (crystal 65) does not. However there is good correlation between step height and an integral multiple of the c dimension for the seven other crystals (8 growth spirals) with polytype structures between $10H$ and $20H$. This agreement is particularly convincing because the c dimensions of the unit cells are large in these cases and the step height is equal to the c dimension for crystals 23, 56 and 76. For crystals with polytypes between $22H$ and $52H$ there is correlation in one case (crystal 48), one doubtful case (crystal 54) and no correlation in the remaining nine cases. However, in three of these nine crystals the step height differs by only one or two layers from the value expected from the measured c dimension.

One may conclude from these results that there is good correlation between the correct step heights of spiral growth

Table 2. *Corrected step heights for accurately measured growth steps on* CdI_2 *crystals whose structure type was determined by X-ray methods*

Crystal number	Step height* in multiples of M = 6·84 Å	Corrected step height (M units)	Structure type and c dimension of unit cell	Is there a correlation between step height and unit cell dimension?
7	32·32 ± 0·20	24·8	$2H$	Yes
71	36·85 ± 0·23	28·3	$2H$	Yes
9	27·75 ± 0·15	21·3	$4H$	No
24	19·33 ± 0·26	14·8	$4H$ (c = 13·7 Å)	No
43	52·43 ± 0·25	40·2	$4H$	Yes
51	36 ± 1	28	$4H$	Yes
63	22·82 ± 0·39	17·5	$4H$ (c = 13·7 Å)	Perhaps
45	54 ± 1	41·4	$6H_c$	Yes
38	27 ± 0·85	20·7	$10H_b$(c = 34·2 Å)	Yes
46	36 ± 3	28	$10H_a$	Yes
77	38 ± 1	29·2	$12H_d$(c = 41·0 Å)	Yes
56	10·29 ± 0·25	7·9	$16H$ (c = 54·7 Å)	Yes
23	12 ± 0·7	9·2	$18H$ (c = 61·5 Å)	Yes
76	(i) 54 ± 2·2	41·5	$20H$ (c = 68·4 Å)	Yes
	(ii) 13 ± 0·7	9·9	$20H$ (c = 68·4 Å)	Yes
29	25·69 ± 0·23	19·7	$22H_a$(c = 75·2 Å)	No
66	15·66 ± 0·23	12·0	$22H_b$(c = 75·2 Å)	No
78	36 ± 2	28	$32H$ (c = 109·4 Å)	No
16	24·88 ± 0·45	19·1	$34H_a$(c = 116·3 Å)	No
54	23·38 ± 0·19	17·9	$34H_b$(c = 116·2 Å)	Almost
48	23·92 ± 0·20	18·35	$36H$ (c = 123·1 Å)	Yes
50	29·06 ± 0·16	22·3	$38H_a$(c = 129·9 Å)	No
55	35·90 ± 0·46	27·5	$38H_b$(c = 129·9 Å)	No
58	54·23 ± 0·28	41·6	$38H_c$(c = 129·9 Å)	No
79	38·89 ± 0·15	29·8	$50H$ (c = 171·0 Å)	No
72	42·24 ± 0·12	32·4	$52H$ (c = 177·8 Å)	No

* From Trigunayat & Verma (1962)

Table 3. *Corrected step heights for growth steps on crystals with structures resembling known polytypes where the step height is known to better than ±0·5 basic layers*

Crystal number	Step height* in multiples of $M=6{\cdot}84$ Å	Corrected step height (M units)	Structure type	Is there a correlation between step height and unit cell dimension?
33	(i) 33·34 ± 0·38	25·6	Resembles $2H$	Yes
	(ii) 22·19 ± 0·49	17·0	Resembles $2H$	
39	59·21 ± 0·48	45·4	Resembles $2H$	Yes
47	17·0 ± 0·05	13·05	Resembles $2H$	Yes
49	30·02 ± 0·11	23·0	Resembles $2H$	Yes
60	47·34 ± 0·43	36·3	Resembles $2H$	Yes
61	48·27 ± 0·30	37·0	Resembles $2H$	Yes
6	34·38 ± 0·23	26·4	Resembles $4H$	Yes?
12	44·36 ± 0·34	34·0	Resembles $4H$	Yes
17	17·20 ± 0·49	13·2	Resembles $4H$	No
18	52·33 ± 0·40	40·1	Resembles $4H$	Yes
26	(ii) 25·86 ± 0·13	19·8	Resembles $4H$	Yes
32	31·33 ± 0·35	24·0	Resembles $4H$	Yes
34	24·02 ± 0·20	18·4	Resembles $4H$	Yes?
36	13·51 ± 0·42	10·4	Resembles $4H$	Yes
41	59·94 ± 0·16	46·0	Resembles $4H$	Yes
44	38·92 ± 0·14	29·9	Resembles $4H$	Yes
68	(i) 18·00 ± 0·37	13·8	Resembles $4H$	Yes
	(ii) 16·30 ± 0·31	12·5	Resembles $4H$	Perhaps
65	18·94 ± 0·32	14·5	Resembles $8H$	No
62	18·19 ± 0·12	14·0	$2H+14H$	Yes

* From Trigunayat & Verma (1962)

hills and the c dimension of the unit cell for most CdI_2 crystals with structure types $2H$ to $20H$. These results are thus consistent with the dislocation theory of spiral growth (Frank, 1951).

The crystals where correlation is not observed may be those in which slip has occurred in the last stages of growth. The measured step height in such cases would not be representative of the structure of the bulk of the crystal and correlation would not be expected. The available results suggest that slip may occur more readily in crystals having unit cells with large c dimensions.

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The unit cell and space group of the antiferroelectric phase of copper formate tetrahydrate.

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X-ray single-crystal photographs of copper formate tetrahydrate, taken at temperatures below the antiferroelectric transition temperature (−38 °C) showed that at these temperatures the unit cell was doubled, due to a doubling of the c axis, compared to the room temperature cell. The low temperature space group was $P2_1$ instead of $P2_1/a$ at room temperature.

It has been shown by Kiriyama (1954) that at room temperature copper formate tetrahydrate is monoclinic with space group $P2_1/a$, and that the structure contains layers of water molecules which alternate with layers made up from copper and formate ions, all layers being parallel to (001). Neutron diffraction studies by Okada, Kay, Cromer & Almodovar (1966) indicated that the orientation of the water molecules was disordered. The antiferroelectric phase transition at −38 °C was first observed by Kiriyama (1962) during a study of dielectric properties. X-ray diffraction studies of this low-temperature phase by Kiriyama (1962) and by Okada *et al.* (1966) failed to show any changes in the number or intensity of diffracted spectra, but Turberfield (1967) using neutrons detected additional reflexions which indicated a doubling of the c axis and the loss of the glide plane. More recently, Molineaux (1969) working in this laboratory