Table 2. *Structure parameters of average cell*

	Coordinates of $P4_132$ space group			Occupation number (experimental)*			Occupation number
				$VC_{0.863}$	$VC_{0.863}$	$VC_{0.865}$	
	x	y	z	(4·2°K)	(300°K)	(300°K)	(ideal)†
V	$\frac{3}{8}$	$\frac{3}{8}$	$\frac{3}{8}$				0·333
V	$\frac{1}{8}$	$\frac{3}{8}$	$\frac{1}{8}$				1·000
C′	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$	0·186 ± 0·008	0·181 ± 0·017	0·183 ± 0·007	0·165
C″	$\frac{1}{8}$	$\frac{5}{8}$	$\frac{5}{8}$	0·480 ± 0·010	0·472 ± 0·024	0·478 ± 0·010	0·493
C″	$\frac{1}{8}$	$\frac{3}{8}$	$\frac{7}{8}$	0·497 ± 0·009	0·503 ± 0·023	0·475 ± 0·009	0·493
Temperature factor (carbon)				0·1 Å^2	0·2 Å^2	0·2 Å^2	
Reliability (R) value				3·4%	5·8%	3·4%	

* Refinements based on a carbon scattering length of $0·663 \times 10^{-12}$ cm.

† Calculated assuming small vacancy excess on ideal V_8C_7 composition is randomly distributed.

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A note on the crystal structure of α-CdP_2. By Olle Olofsson and Jan Gullman, *Institute of Chemistry, University of Uppsala, S-751 21 Uppsala, Sweden*

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The single-crystal data recorded by Goodyear & Steigmann for α-CdP_2 have been further refined. The resulting distances are in better agreement with distances in other known phosphides of similar type.

Recently, Goodyear & Steigmann (1969) described the determination of the crystal structure of α-CdP_2. One characteristic feature of the structure is chains of phosphorus atoms running parallel to each other in the **c** direction. There are two different P–P distances within the chains. According to Goodyear & Steigmann these distances are 2·05 and 2·39 Å respectively. It seemed to us that these distances deviated considerably from what was to be expected from a consideration of other known polyphosphides with similar arrangements of phosphorus atoms. In particular, the 2·05 Å distance is very short in comparison with the normal P–P bond distance of 2·20 Å.

Despite the fact that Goodyear & Steigmann claim that the least-squares refinement was stopped when the last shifts were less than the standard deviations, we decided to try to see if it was possible to make a further refinement using their observed structure factors. We used a local version of the least-squares program *LALS* originally written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Los Angeles, U.S.A.

Table 1. *Atomic parameters with standard deviations*

For each atom the result from the present refinement is given in the first row, and the result of Goodyear & Steigmann in the second row.

	x	y	z	B
Cd	0·1529 (2)	0·1011 (3)	0·2606*	1·89 (8) Å^2
	0·1529 (3)	0·1016 (5)	0·2606 (26)	1·95 (5)
P(1)	0·1185 (7)	0·4442 (14)	0·5957 (23)	1·43 (12)
	0·1186 (10)	0·4458 (21)	0·5850 (30)	1·34 (16)
P(2)	−0·0064 (7)	0·2693 (12)	−0·1036 (24)	1·24 (12)
	−0·0074 (10)	0·2697 (19)	−0·0676 (33)	1·16 (15)

* Arbitrary.

Starting with the published atomic parameters it was obvious that after a few cycles of full-matrix refinement considerable changes of some coordinates had taken place and at the same time the R value had dropped appreciably. The refinement was continued until all shifts were less than one tenth of the estimated standard deviations. The data were weighted according to $w=1/(a+|F_o|+c|F_o|^2+d|F_o|^3)$.

Table 2. *Bond lengths and angles with standard deviations*

	This work	Goodyear & Steigmann (1969)
Cd–P(1)	2·562 (10) Å	2·529 (15) Å
P(1)	2·563 (8)	2·579 (15)
P(2)	2·619 (10)	2·495 (15)
P(2)	2·571 (8)	2·627 (15)
P(1)–P(2)	2·200 (14)	2·386 (18)
P(2)	2·167 (12)	2·050 (18)
P(1)–Cd–P(1)	125·6 (3)°	126·3 (5)°
P(1)–Cd–P(2)	98·9 (3)	95·6 (5)
P(1)–Cd–P(2)	107·7 (3)	105·4 (5)
P(1)–Cd–P(2)	114·0 (3)	115·9 (5)
P(1)–Cd–P(2)	109·3 (3)	110·5 (5)
P(2)–Cd–P(2)	97·3 (2)	99·2 (5)
P(2)–P(1)–P(2)	110·9 (4)	108·0 (7)
P(1)–P(2)–P(1)	108·4 (4)	105·7 (7)

The values $a=10{\cdot}0$, $c=0{\cdot}02$ and $d=0{\cdot}001$ finally used were suitable as was shown by a weighting analysis. In the space group $Pna2_1$, to which α-CdP_2 belongs, the z direction is polar and the z coordinate for the cadmium atom was accordingly kept constant during the refinement. The final R value based on the 282 reflexions used in the refinement was 0·110. For all 298 reflexions $R=0{\cdot}120$. ($R=\sum||F_o|-|F_c||/\sum|F_o|$). The reflexions excluded from the refinement were essentially very weak ones. The final atomic parameters are given in Table 1. Table 2 shows the bond lengths and angles obtained as compared with those given by Goodyear & Steigmann. The distances between the phosphorus atoms within the chains are now 2·17 and 2·20 Å. These distances, which deviate about ten standard deviations from those originally given, agree much better with the P–P distances found in other phosphides of similar type.

This result has also been confirmed independently by Goodyear and Steigmann.

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Etude radiocristallographique préliminaire de quelques thiazides.

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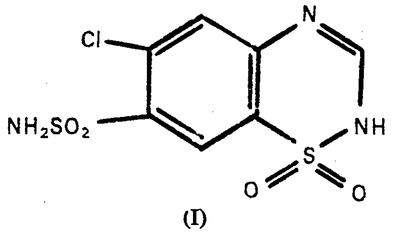
Crystal data for chlorothiazide, hydrochlorothiazide and polythiazide have been determinated from X-ray data by the Weissenberg method.

Les thiazides que nous étudions sont des sulfamides diurétiques de la série de la benzothiadiazine. Ils agissent en inhibant un ou plusieurs des mécanismes de transport qui assurent la réabsorption du sodium par le tube rénal (Gantt & Synek, 1961; Heinemann, Demartini & Laragh, 1959; Pignard, 1960).

L'étude qui suit concerne la détermination des paramètres des mailles cristallines et des groupes spatiaux du chlorothiazide ($C_7H_6ClN_3O_4S_2$), de l'hydrochlorothiazide ($C_7H_8ClN_3O_4S_2$) et du polythiazide ($C_{11}H_{12}ClF_3N_3O_4S_3$).

(*a*) *Le chlorothiazide: chloro-6-sulfamoyl-*7-*Δ*3*-benzothiazine-*1,2,4 *dioxyde-*1,1 (I).



(I)

Des cristaux de chlorothiazide, sous forme de plaquettes triangulaires incolores, ont été obtenus par évaporation lente, à température ambiante d'une solution de ce dérivé dans de l'éthanol.

Les données concernant la maille cristalline ont été déterminées par la méthode de Weissenberg avec la radiation Cu $K\alpha$ ($\lambda=1,5418$ Å). Le réseau cristallin est triclinique, de groupe spatial $P1$.

Les paramètres de la maille ont été affinés par la méthode des moindres carrés portant sur un nombre élevé de réflexions de $\theta>45°$, au moyen d'un programme de Cox (1967), adapté à l'ordinateur IBM 360/65. Les valeurs suivantes ont été obtenues:

$$a=7,423\pm0,010,\quad b=4,901\pm0,010,\quad c=9,024\pm0,010\text{Å}$$
$$\alpha=96,405\pm0,010,\quad \beta=99,504\pm0,010,\quad \gamma=121,182\pm0,010°$$

Volume de la maille, $V=269,3\pm1,2$ Å^3
Densité calculée, $D_c=1,81\pm0,01$ g.cm^{-3}
Coefficient d'absorption linéaire,
$\mu=66,7\pm0,3$ cm.$^{-1}$ (Cu $K\alpha$)
Nombre de molécules par maille, $Z=1$.