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Structures of 26 New Polytypes of Tin Disulfide*

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Abstract. Structures of 26 new polytypes of SnS_2 grown from the vapour phase are reported (*t–o–f*/ Zhdanov notations): 12 hexagonal polytypes: $12H_3$: $tf5f1f2f1f1/21211212$; $14H_3$: $f5f1f2f1f1$-$f2f1/1211212112$; $16H_1$: $(tf5f1f1)_2/(21212)_2$; $24H_2$: $(t)_2f5f1f1f5f1of1f5f1f1/2212121211121212$; $24H_3$: $(f5f1of1)_2f2f2f1f1/(121112)_2111212$; $38H_1$: $(f1f5f1)_6o/(2121)_611$; $44H_2$: $(f1f5f1)_4[f1f5$-$f1(o)_2]_2/(2121)_4[2121(11)_2]_2$; $58H_1$: $(f1f5f1)_9(o)_2/$ $(2121)_9(11)_2$; $76H_1$: $[f1f5f1(o)_3]_6(o)_2/[2121(11)_3]_6$-$(11)_2$; $78H_1$: $(t)_2(f5f1f1)_6f5f1of1(f5f1f1)_5/22$-$(1212)_6121112(1212)_5$; $80H_1$: $[f1f5f1(o)_6]_2(o)_4f1f5$-$f1(o)_{15}/[2121(11)_6]_2(11)_42121(11)_{15}$; $92H_1$: $(f1f5$-$f1)_{15}o/(2121)_{15}11$; and 14 rhombohedral polytypes: $42R_3$: $(f1f5f1)_2o/(2121)_211$; $48R_3$: $f5f1f1tf5tf1f1/$ 1212212212; $60R_1$ $(f1f5f1)_2(o)_4/(2121)_2(11)_4$; $78R_3$: $(f1f5f1)_4o/(2121)_411$; $90R_1$: $f5f1f1f2f2f1f1f5$-$f1f1f2of2f1f1/121211121212121(11)_2212$; $90R_2$: $(t)_2(f5f1f1)_2f5f1of1f5f1f1/22(1212)_2$-$1211121212$; $96R_3$: $(f1f5f1)_5o/(2121)_511$; $102R_2$: $[f1f5f1(o)_2]_2(o)_7/[2121(11)_2]_2(11)_7$; $138R_2$: $[f1f5$-$f1(o)_3f1f5f1o]_2f1f5f1/[2121(11)_3212111]_22121$; $144R_2$: $(f1f5f1)_6of1f5f1(o)_2/(2121)_6112121(11)_2$; $156R_2$: $(t)_2(f5f1f1)_8/22(1212)_8$; $192R_1$: $[f1f5f1$-$(o)_3]_4(o)_8/[2121(11)_3]_4(11)_8$; $246R_1$: $(f1f5f1)_{13}(o)_2/$ $(2121)_{13}(11)_2$; $258R_1$: $(f1f5f1)_{14}o/(2121)_{14}11$.

Editorial note: The Zhdanov notation used in this paper is a simplified version of the recommendations approved by the International Union of Crystallography *Ad-Hoc* Committee on the Nomenclature of Disordered, Modulated and Polytype Structures [Guinier, Bokij, Boll-Dornberger, Cowley, Ďurovič, Jagodzinski, Krishna, de Wolff, Zvyagin, Cox, Goodman, Hahn, Kuchitsu & Abrahams (1984). *Acta Cryst.* A40, 339–404].

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Introduction. Although only a little is known about the origin of polytypism, in some cases it is possible to predict the structures (stacking sequences) which can form in crystals under given conditions. As discussed many times in the literature most of the large-period polytypes may be divided into structural series: groups of structures having similar construction. To explain these regularities a screw-dislocation mechanism has been suggested by numerous authors; but the geometrical rules for constructing polytypes on the basis of a growth mechanism appear to have only limited use. According to the geometrical concepts of polytypism, to create a polytype in the crystal it should be enough to have one structure grown in given conditions. In most cases, however, the structures observed in real crystals do not conform with those predicted in this way.

A much more useful and reasonable approach for understanding the rules governing polytypism of crystals is to consider polytype structures as mixed phases – the structures intermediate between simple (basic) polytypes. On developing this concept for polytype structure determination it was found that the formation of structural series of polytypes as well as the growth of polytypes in only some specific conditions may be easily explained. Using the same assumptions (rules) to predict the stacking sequences for different MX_2 compounds the structure determination of tens of CdI_2, PbI_2, $SnSe_2$ and SnS_2 polytypes has been performed.

In the present work several very-large-period polytypes of SnS_2 are given. As discussed the large number of layers in the polytypes of SnS_2, much larger than in typical polytypes of CdI_2 or PbI_2, is a direct consequence of the formation of mixed phases from the basic polytypes of this material: $2H$, $4H$ and $18R$. The polytypes composed only of $2H$ and $4H$ stackings were not found in SnS_2.

Experimental. Crystals of SnS_2 were grown by chemical transport: starting materials were powders of SnS_2 (or powdered S and Sn) and SnI_4, temperature of crystallization was in the range 670–1170 K (Pałosz, Pałosz & Gierlotka, 1984, 1986). The crystals were examined by X-rays in a cylindrical camera of radius 43 mm and with a 0·7 mm collimator. The oscillation method was used with the [12$\bar{3}$0] direction as a rotation axis and with the angle between the incident beam (Co K radiation) and the c axis varying between 15 and 30°. The crystals were usually platelets with well defined bipyramidal (or rhombohedral) faces and the orientation of the samples was accomplished with an optical goniometer. The intensities of the reflections 10.l and 1$\bar{1}$.l registered in the patterns were measured photometrically and corrected for the Lorentz–polarization factor and for sample absorption (Pałosz et al., 1985a,b). The intensity values of the reflections were plotted in diagrams and their comparison with similarly plotted F^2 values calculated for a certain number of theoretical models of the structure was the basis for the structure determination. The method used here for the determination of the structures of 26 polytypes of SnS_2 is similar to that used earlier for 45 SnS_2 polytypes (Pałosz et al., 1985a,b).

The stacking sequences of 26 new polytypes of SnS_2 are given, in Zhdanov and t–o–f notations, in Table 1 together with the growth temperatures; the intensity diagrams for the 92 H_1 and 258 R_1 polytypes are given in Fig. 1.* A list of R values calculated for the polytypes identified is given in Table 2. The t–o–f notation was introduced by Pałosz (1980) to make easier the derivation of the possible structures for MX_2 polytypes and the elimination of the equivalent structures. To construct the layer sequences which had to be considered to identify the polytypes under investigation we applied the multiphase model of polytype structures, considering large-period polytypes as structures intermediate between basic polytypes of SnS_2: 2H, 4H and 18R, with Zhdanov sequences 11, 22, and $(2121)_3$ and t–o–f sequences o, t and $(f1f5f1)_3$, respectively, according to Pałosz et al. (1984, 1985a, 1986). The formulae of the stacking sequences of the structural series of polytypes predicted by this model were given for SnS_2 polytypes by Pałosz et al. (1985a). The models corresponding to these simple structural series were considered first. 20 of 26 polytypes were found to have structures belonging to these simple series. They were identified quickly after consideration of no more than 20 models for each one. Six polytypes had more complicated structures containing, beside the basic

layer arrangements o, t, and $f1f5f1$ ($f5f1f1$), also the stackings $f1f2$ ($f2f1$) and/or $f2f2$. It was found that to construct the structural models of the polytypes with the stackings $f1f2$ and $f2f2$ one can use the formulae derived for the stacking sequences of simple series (Pałosz et al. 1985a). For this purpose, one should replace one or several t and/or $f5$ layers with the sequences $f1f2$ ($f2f1$) and/or $f2f2$, respectively, as indicated schematically in Fig. 2. For example, starting with the series SnH–2: $(t)_{2N+1}f5(t)_{2N'+1}f1(o)_nf1$ one

Table 1. *Structure and growth conditions of 26 polytypes of tin disulfide*

Ramsdell symbol*	t–o–f sequence	Zhdanov sequence	Growth temperature (K)
Hexagonal polytypes			
12H_3	$tf5f1f2f1f1$	21211212	935
14H_3	$f5f1f2f1f1f2f1$	1211212112	945
16H_1†	$(tf5f1f1)_2$	$(21212)_2$	825
24H_2†	$(t)_2f5f1f1f5f1of1f5f1f1$	2212121211121212	825
24H_3‡	$(f5f1of1)_2f2f2f1f1$	$(121112)_2111212$	825
38H_1	$(f1f5f1)_6o$	$(2121)_611$	860
44H_2	$(f1f5f1)_4(f1f5f1(o)_2)_2$	$(2121)_4 2121(11)_2 l_2$	885
58H_1	$(f1f5f1)_9(o)_2$	$(2121)_9(11)_2$	860
76H_1	$f1f5f1(o)_1 l_4(o)_2$	$l2121(11)_1 l_4(11)_2$	935
78H_1	$(t)_2(f5f1f1)_6f5f1of1(f5f1f1)_5$	22(1212)$_6$121112(1212)$_5$	945
80H_1	$f1f5f1(o)_5 l_3(o)_4 f1f5f1(o)_{15}$	$l2121(11)_6 l_3(11)_4 2121(11)_{15}$	875
92H_1§	$(f1f5f1)_{15}o$	$(2121)_{15}11$	935
Rhombohedral polytypes			
42R_3	$(f1f5f1)_2o$	$(2121)_211$	860
48R_1§	$f5f1f1f5f1f1$	1212212212	945
60R_1	$(f1f5f1)_2(o)_4$	$(2121)_2(11)_4$	945
78R_3	$(f1f5f1)_4o$	$(2121)_411$	935
90R_1	$f5f1f1f2f2f2f1f1f5f1f1f2$ $of2f1f1$	121211121212121(11)$_2$12	875
90R_2	$(t)_2(f5f1f1)_2f5f1of1f5f1f1$	22(1212)$_2$1211121212	1025
96R_3§	$(f1f5f1)_5o$	$(2121)_511$	885
102R_2	$f1f5f1(o)_5 l_2(o)_7$	$l2121(11)_5 l_2(11)_7$	885
138R_2	$f1f5f1(o)_5 f1f5f1of_3 f1f5f1$	$l2121(11)_5 2121111 l_2 2121$	860
144R_2	$(f1f5f1)_6 of1f5f1(o)_2$	$(2121)_6 112121(11)_2$	860
156R_2†	$(t)_2(f5f1f1)_8$	22(1212)$_8$	825
192R_1	$f1f5f1(o)_5 l_4(o)_8$	$l2121(11)_5 l_4(11)_8$	875
246R_1§	$(f1f5f1)_{13}(o)_2$	$(2121)_{13}(11)_2$	1005
258R_1	$(f1f5f1)_{14}o$	$(2121)_{14}11$	945

* Indices of Ramsdell symbols are after Pałosz, Pałosz & Gierlotka (1985a,b).

† Nominal chemical composition of the crystal $Sn_{0.99}Ga_{0.01}S_2$.

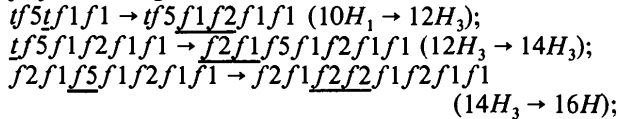‡ Nominal chemical composition of the crystal $Sn_{0.99}Sb_{0.01}S_2$.

§ Centrosymmetric structures with space groups $P\bar{3}m$ (hexagonal polytypes) or $R\bar{3}m$ (rhombohedral polytypes). All other polytypes belong to the space groups $P3m1$ (hexagonal) or $R3m$ (rhombohedral).

Table 2. *R values for the 26 identified polytypes of SnS_2*

Polytype/ Ramsdell symbol	No. of measured reflexions	R^*	Polytype/ Ramsdell symbol	No. of measured reflexions	R^*
12H_3	26	0·29	43R_3	32	0·20
14H_3	30	0·27	60R_1	44	0·27
16H_1	34	0·20	78R_3	54	0·33
24H_2	52	0·36	90R_2	60	0·29
24H_3	50	0·34	90R_1	60	0·28
38H_1	78	0·36	96R_3	64	0·20
44H_2	110	0·30	102R_2	70	0·30
58H_1	116	0·21	138R_2	92	0·20
76H_1	152	0·30	144R_2	96	0·29
78H_1	158	0·29	156R_2	104	0·24
80H_1	162	0·38	192R_1	128	0·24
92H_1	186	0·17	246R_1	164	0·23
42R_3	30	0·28	258R_1	172	0·17

* $R = (\Sigma|I_o - I_c|/\Sigma I_o)^{1/2}$, where I_o is the measured and I_c the calculated intensity.

can obtain a family of structures with $f1f2$ and/or $f2f2$ stackings:

$tf5tf1f1 \rightarrow tf5\underline{f1f2}f1f1$ $(10H_1 \rightarrow 12H_3)$;

$tf5f1f2f1f1 \rightarrow \underline{f2f1}f5f1f2f1f1$ $(12H_3 \rightarrow 14H_3)$;

$f2f1\underline{f5}f1f2f1f1 \rightarrow f2f1\underline{f2f2}f1f2f1f1$

$(14H_3 \rightarrow 16H)$;

where we have underlined the layers which substitute each other in each step.

Discussion. As discussed earlier by Pałosz *et al.* (1985*b*) the structures with stackings $f1f2$ and $f2f2$ may be regarded as multiphase structures with inter-polytype interfaces more complicated than those for the structures of simple series.

Once the set of stacking sequences corresponding to the structural series is known, the identification of a polytype only requires consideration of the models composed of these sequences. So far all 71 known polytypes of SnS_2 have structures satisfying the multiphase model. Yet, it cannot be excluded that further studies on the polytype structure of SnS_2 will introduce additional rules of construction of the polytypes. There is one polytype, $6H$: $f3f3f4/33$ (t–o–f/Zhdanov), reported by Whitehouse & Balchin (1979) which seems to have a strange composition. Most probably the identification of this polytype was performed erroneously; the comparison of the experimental reflection intensities with those calculated by the authors for two structures (1122 and 33) should lead them to the conclusion that the structure observed
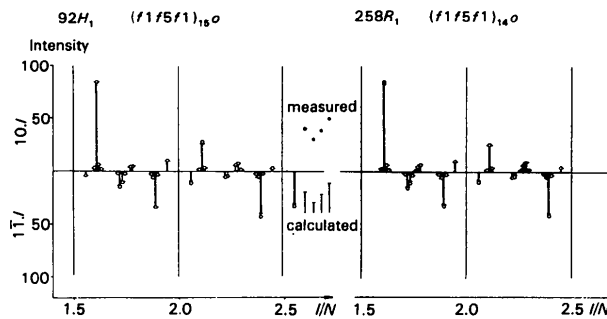

Fig. 1. Diagram of intensities of reflections $10.l$ and $1\bar{1}.l$ measured experimentally and calculated theoretically for the SnS_2 polytypes $92H_1$ and $258R_1$.

Fig. 2. Schematic drawing of the substitution of (*a*) $f5$ with $f2f2$ and (*b*) t with $f1f2$ (or $f2f1$) in the layer stackings of the polytypes of SnS_2. The stacking sequences are represented in the plane (11.0).

is neither 1122 nor 33. From our experience with SnS_2 polytypes, no $6H$ polytype is possible for SnS_2. The polytype $6H$: $tf1f2/1122$ may be regarded as a $2H$–$4H$ intermediate structure and it occurs frequently in CdI_2 crystals. $2H$ and $4H$ are also basic structures of SnS_2, but neither the polytype $6H$ nor other structures of the type $2H$–$4H$ have been found in hundreds of SnS_2 crystals we have investigated. However, why the intermediate structures $2H$–$18R$, $4H$–$18R$ and $2H$–$4H$–$18R$ do form in SnS_2 crystals and the structures $2H$–$4H$ do not, is not known yet.

Special problems in polytype identification: hexagonal and rhombohedral structures

Identification of polytypes having not more than 30 layers in the unit cell ($30H/90R$) is rather simple, requiring consideration of at most few tens of models. The problem becomes much more complicated when the number of layers in the cell is larger than 40–50, because of the very high number of models which have to be considered to identify a polytype uniquely and also because of experimental conditions. For large-period polytypes it is often difficult to separate hexagonal (NH) and rhombohedral ($3NR$) polytypes because the resolution of the reflections on the patterns may not be sufficient. Therefore, in the case of polytypes having more than 30 layers in the cell, both hexagonal and rhombohedral structural models were considered, *e.g.* to determine the structure of the polytype $58H_1$ the models $58H$ and $174R$ were tried. The most intense reflections co-incide with the reflections of the $18R$ polytype. It follows that the structure is basically composed of $18R$-type stackings: $f1f5f1$ (or $f5f1f1$). From the comparison of the intensity diagram of the polytype with the diagrams of already known polytypes it was deduced that the polytype belongs to the series of $2H$–$18R$ structures: SnH–1 or SnR–1 (Pałosz *et al.*, 1985*a*). The general stacking sequence of the polytypes of these two series is given by the same formula:

$$(f1f5f1)_N(o)_n(f1f5f1)_{N'}(o)_{n'}(f1f5f1)_{N''}(o)_{n''}\cdots$$

A hexagonal form of the above sequence should contain $3M$ sequences $f1f5f1$ ($N + N' + N'' + \cdots = 3M$); a rhombohedral form should contain $3M \pm 1$ such sequences. The unit cell of the polytype was established to be composed of 29 molecular sandwiches S–Sn–S. The hexagonal cell of such a polytype should have 3, 6 or 9 sequences $f1f5f1$ with 20, 11 or 2 o layers, respectively. As the structure was based on the $18R$ polytype, only the models with two o layers were considered: $(f1f5f1)_9(o)_2$, $(f1f5f1)_8of1f5f1o$, $(f1f5f1)_7o(f1f5f1)_2o$, $(f1f5f1)_6o(f1f5f1)_3o$, $(f1f5f1)_5o(f1f5f1)_4o$.

The possible rhombohedral cells should have 2, 4, 5, 7 or 8 $f1f5f1$ sequences with 23, 17, 12, 8 or 5 o layers, respectively. In this case only the models with
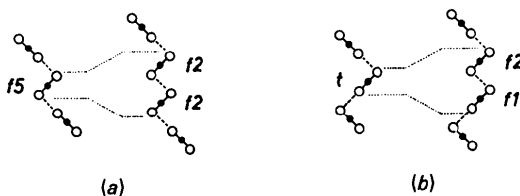
five o layers were considered: $(f1f5f1)_8(o)_5$, $(f1f5f1)_7(o)_4 f1f5f1o$, $(f1f5f1)_6(o)_4(f1f5f1)_2o$, $(f1f5f1)_5(o)_4(f1f5f1)_3o$, $(f1f5f1)_4(o)_4(f1f5f1)_4o$, $(f1f5f1)_7(o)_3 f1f5f1(o)_2$, $(f1f5f1)_6(o)_3(f1f5f1)_2(o)_2$, etc.

The intensities for the hexagonal and rhombohedral models were calculated and it was found that only the structure $58H$: $(f1f5f1)_9(o)_2$ gave the intensity diagram corresponding to that of the polytype under investigation. So, one can say that in this case the symmetry of the polytype cell was established indirectly, by comparing the measured and calculated intensities.

The case of a very-large-period polytype $258R_1$, Fig. 1, is similar. This polytype is very close to the basic structure $18R$ and, similar to the polytype $58H_1$ discussed above, belongs to the series $SnH-1$ or $SnR-1$. The polytype contains 43 molecular sandwiches in the cell ($86H$ or $258R$), and the simplest hexagonal polytype with the minimal number of o layers has seven such layers in the unit cell: $(f1f5f1)_{12}(o)_7$. This model can be excluded because it contains a relatively large number of o layers and the polytype under investigation is very close to $18R$. The rhombohedral forms with minimal number of o layers are: $(f1f5f1)_{14}o$ and $(f1f5f1)_{13}(o)_4$, and the model with one o layer was identified as the correct one for the polytype under consideration.

Elimination of similar structures

Structures having similar construction give similar diffraction patterns: therefore, to determine uniquely the structure of a polytype it is not sufficient to find the model for which the calculated reflection intensities agree well with those measured. It is necessary to check the other possible models similar to that first identified to correspond to the structure under investigation. The above procedure: (1) selection of one model best fitted with the structure being identified and (2) elimination of other, similar models, was always used in the polytype identification. For example, for the polytype $258R_1$ identified as $(f1f5f1)_{14}o$, two other models were tried: $(f1f5f1)_{13}f1f2f2f1$ and $(f5f1f1)_{13}(f2f1)_2$. Similarly, for the polytype $58H_1$ having the structure $(f1f5f1)_9(o)_2$ several other models were tried: $(f5f1f1)_9(t)_2$, $(f1f5f1)_8 f1f2of2f1$, $(f1f5f1)_8$-$f1f2f2f1o$, $(f5f1f1)_8 f2of1f2f1$ and $(f5f1f1)_8$-$f2f1f2of1$.

Series of polytypes: origin of polytypism

Before the actual structure determination of SnS_2 crystals, expected layer sequences in these crystals have been speculated upon. Mitchell, Fujiki & Ishizawa (1982), presenting preliminary results of the structural analysis of SnS_2 polytypes (including merely determination of symmetry and cell dimensions of the polytypes), suggested several structural series according

to the screw-dislocation growth of the polytypes:

(i) $4H$-based polytypes (Zhdanov/t–o–f): $[(22)_n 11]_n/$ $(t)_n f2f1$, $[(22)_n 1111]_n/(t)_n f1of2$, $[(22)_n 33]_n/(t)_n f3$-$f3f4$, $[(22)_n 2123]_n/(t)_n f4tf5$;

(ii) $18R$-based structures: $[(2121)_n 22]_n/(f5f1f1)_n$-$(t)_2$, $[(2121)_n 11]_n/(f1f5f1)_n o$.

No SnS_2 structures classified by Mitchell *et al.* as $4H$-based polytypes have been found so far. Some of the polytypes identified may be considered as originating from $18R$ and these are, according to our model, $2H-18R$ and $4H-18R$ structures. As only a few of the structures predicted by Mitchell *et al.* do occur in the crystals, the suggestion that large-period polytypes originate from single basic structures does not seem to be true. This does not mean that the polytypes cannot (or do not) grow by a screw-dislocation mechanism, but that the structures of the polytypes do not depend on the mechanism by which they form in the crystals.

In contrast to the screw-dislocation theory of polytypism, leading frequently to results contradictory with experiment, the multiphase model of polytypism well describes the structures of the crystals: CdI_2, PbI_2 (Pałosz, 1983), $SnSe_2$ (Pałosz, Gierlotka & Lévy, 1985) and also SnS_2. The model assumes that multilayer polytypes are mixed structures, intermediate between basic structures (*i.e.* those occurring in the crystals of a given compound far more frequently than the others). According to the recent investigations by Mitchell *et al.* (1982) and Pałosz *et al.* (1984, 1986) the basic polytypes of SnS_2 ($2H$, $4H$ and $18R$) form in the crystals in certain ranges of growth temperatures only, *e.g.* in the crystals grown by chemical transport, with S and Sn as substrates and SnI_4 as transporting agent, $2H$ grows frequently below 1050 K, $18R$ in the range 750 to 1100 K and $4H$ above 900 K (Pałosz *et al.*, 1986). As seen, in certain temperature ranges, two or three basic polytypes grow simultaneously. It was found (Pałosz *et al.*, 1984) that only in these temperature ranges do multilayer polytypes grow. Moreover, the composition of the complex polytypes changes with growth temperature according to the changes of the relative frequencies of occurrence of the basic structures. These results demonstrate the correctness of the multiphase approach to polytypism.

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(2-Furoato)trimethyllead

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Abstract. [Pb(C$_5$H$_3$O$_3$)(CH$_3$)$_3$], $M_r = 363 \cdot 4$, monoclinic, $P2_1/n$, $a = 10 \cdot 246$ (9), $b = 10 \cdot 297$ (9), $c = 10 \cdot 651$ (8) Å, $\beta = 106 \cdot 19$ (5)°, $V = 1079$ Å^3, $Z = 4$, $D_x = 2 \cdot 237$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 15 \cdot 4$ mm^{-1}, $F(000) = 664$, $T = 291$ K, final $R = 0 \cdot 039$ for 1077 unique observed [$I > 3\sigma(I)$] X-ray diffractometer data. In the crystalline state nearly planar (CH$_3$)$_3$Pb groups are linked by bridging carboxylate groups [Pb—O(1) 2·353 (9), Pb—O(2) 3·17 (1), Pb—O(2^i) 2·534 (9) Å] to form infinite chains parallel to **b**. An additional interaction between the O atom of the furan ring and a Pb atom is observed [Pb⋯O 3·55 (1) Å].

Introduction. Structures of organo Group IV element derivatives of carboxylic acids R'COOH (R' = heterocyclic remainder) are interesting, since there arises the possibility of coordinative interaction between the central atom, *e.g.* Sn or Pb, and the heteroatom in R', *e.g.* N or O. Recently, Harrison & Philips (1979) reported the structure of Me$_3$SnO$_2$CC$_5$H$_4$N.H$_2$O, but the heteroatom in the pyridine group is involved in hydrogen bonding, and there is no interaction with Sn. In the title compound on the other hand this is not the case and an interaction between the O atom of the furan ring and the Pb atom is observed.

Experimental. Colourless, fibrous crystals were prepared from equimolar amounts of Me$_3$PbOH and 2-furoic acid in methanol. The compound was recrystallized from methanol/di-*n*-butyl ether (1/1). Crystal size 0·14 × 0·30 × 0·18 mm, $\omega/2\theta$ scan, ω-scan speed 2·5° min^{-1}, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit using 25 reflections with $2\theta_{max} = 26 \cdot 6°$; intensities of four standard reflec-

tions recorded every 2·5 h showed decay of 50% during the course of data collection; 4127 reflections, $1° \leq \theta \leq 25°$, $-12 \leq h \leq 12$, $-12 \leq k \leq 12$, $0 \leq l \leq 12$; after averaging ($R_{int} = 0 \cdot 026$) 2015 unique reflections, 1077 with $I > 3\sigma(I)$, Lorentz–polarization correction, absorption correction *via* ψ scans (max./min. correction: 1·00/0·58), and decay correction; systematic absences showed space group to be $P2_1/n$; structure solution by Patterson methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for non-H atoms, H atoms in geometrically calculated positions (C—H 0·95 Å) and refined with one common isotropic temperature factor for these atoms; 110 variables, $w^{-1} = [\sigma^2(I) + (0 \cdot 1F_o^2)^2]^{1/2}$, $S = 0 \cdot 79$, $R = 0 \cdot 039$, $wR = 0 \cdot 047$, max. $\Delta/\sigma = 0 \cdot 13$, largest peak in final ΔF map = $\pm 2 \cdot 0$ (3) e Å^{-3} near Pb; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *Enraf–Nonius Structure Determination Package* (Frenz, 1981), *ORTEP*II (Johnson, 1976).

Discussion. The structure of the title compound with the numbering scheme is shown in Fig. 1 and a stereoscopic view of the unit cell is shown in Fig. 2 (*ORTEP*II). Atomic parameters are given in Table 1,* selected bond lengths and angles in Table 2. In the crystalline state Pb(CH$_3$)$_3$ groups are linked *via* bridging carboxylate groups of the 2-furoate ligands to form an infinite chain similar to that observed by

* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42732 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.