carbons in the microcrystalline waxes from petroleum. Since very little is known about the crystalline structures of acids with side chains in definitely established positions on the main aliphatic chains, we have undertaken the study of single crystals of a series of such acids. Preliminary results are reported herewith on 14-ethylhexadecanoic, 16-methyloctadecanoic and 17-methyloctadecanoic acids, samples of which were very kindly furnished by Dr James Cason, University of California, Berkeley, California. These three acids crystallize in the form of flakelets and with the *c* axis parallel to the thin direction of the flake. The crystals studied were so characteristically imperfect in spite of repeated efforts at improvement on recrystallization that only a Buerger precession camera could be used to give interpretable patterns from which the unit-cell dimensions and space-group symmetries were determined. It is an added tribute to this technique that imperfection in packing of molecules is not an insuperable obstacle to analysis, though complete Fourier analysis of molecular structures is still nearly hopeless because of great uncertainties in intensity evaluation. The densities of the crystals were determined by pycnometric measurements of solutions which would just suspend the crystals. The results are assembled in Table 1.

17-Methyloctadecanoic acid is an extraordinary case; it is triclinic but has four molecules per unit cell. Its probable space group is $P\bar{1}$, which requires two asymmetric units; thus a group of two molecules must constitute the asymmetric unit of structure. These two molecules are connected by a pseudo glide plane perpendicular to the *a* axis, whose presence is strongly suggested by the existence of an almost perfect halving of the $0kl$ planes when k is odd. 17-Methyloctadecanoic acid has a positive optic sign. This indicates that the chains are not crossed; therefore the chain is not tilted toward the pseudo glide plane, the *bc* plane. Moreover, the $h00$ reflections are weak, indicating that the plane of the chain must possess a pitch relationship to the *bc* plane.

Further work is being continued on a whole series of these acids with the primary object of establishing the diffraction criteria for side-chain positions along the aliphatic chain.

Table 1. *Crystallographic properties*

Compound	a (A.)	b (A.)	c (A.)	α	β	γ	V (A.^{3})	$\rho_{\text{exp.}}$	$\rho_{\text{X-ray}}$	n	Space group	Optic sign
14-Ethylhexadecanoic acid	5·58	6·79	27·96	93° 15′	111° 30′	100° 14′	960·6	0·989	0·982	2	$P\bar{1}$	−
16-Methyloctadecanoic acid	5·43	6·91	28·00	91° 16′	94° 16′	104° 56′	1011·3	0·988	0·979	2	$P\bar{1}$	−
17-Methyloctadecanoic acid	5·64	9·66	37·10	90° 24′	91° 38′	102° 33′	1972·7	0·995	1·004	4	$P\bar{1}$	+

Acta Cryst. (1951). **4**, 471

The crystal structure of lanarkite, $PbO.PbSO_4$.

By W. P. Binnie, *Department of Physics, Purdue University, Lafayette, Indiana, U.S.A.*

(*Received* 3 *May* 1951)

Treatment, such as baking in oxygen, of PbS deposits produces or increases the photo-sensitivity of such cells and also gives rise to an oxidation product on the surface which has been identified as the mineral lanarkite, $PbO.PbSO_4$ (Wilman, 1948; Doughty, Lark-Horovitz, Roth & Shapiro, 1950). It has been assumed by some workers in the field that the barrier between the PbS crystallites may be in part lanarkite. Therefore Lark-Horovitz suggested that a detailed examination of the crystal structure of lanarkite would possibly provide information which would elucidate the mechanism of the photo-sensitivity.

The presence of the heavy atoms in the molecule does not allow clear resolution of the lighter atoms, but the lead atoms and a single oxygen atom have been located; these, together with a tentative position which has been given to the sulphate group, form an interesting arrangement similar to that found in PbO.

Natural lanarkite crystals were obtained from Scotland, and the following crystal data were obtained. System, monoclinic; unit-cell dimensions,

$$a = 13{\cdot}75 \pm 0{\cdot}04, \quad b = 5{\cdot}68 \pm 0{\cdot}02, \quad c = 7{\cdot}05 \pm 0{\cdot}02 \text{ A},$$
$$\beta = 116{\cdot}2^\circ \pm 0{\cdot}2^\circ;$$

density, found 6·92 g.cm.$^{-3}$, giving 3·9 molecules of $PbO.PbSO_4$ per unit cell. Extinctions are consistent with the space groups, Cm, $C2$ and $C2/m$. Pyro-electric tests do not show the absence of a centre of symmetry in the crystals, so space group $C2/m$ was chosen. These crystal data agree well with those of Richmond & Wolfe (1938).

Series of Weissenberg and precession films were taken about the three principal axes, using copper and molybdenum characteristic radiations and crystals of no greater size in one dimension than 0·15 mm. Intensities, corrected for absorption, were used to sum Patterson projections, and interpretation of these led to the assignment of x, y and z co-ordinates to the lead atoms. Successive Fourier syntheses failed to resolve the sulphate group, but one oxygen atom was located at the origin of the b-axis projection and an approximate y co-ordinate could be given to it.

Parameters measured from Fourier projections and referred to the crystallographic axes are:

	x (A.)	y (A.)	z (A.)
Pb_1	2·012	0	0·729
Pb_2	0·324	2·84 ($\frac{1}{2}b$)	1·915
O_5	0	1·42 ($\frac{1}{4}b$)	0

The co-ordinates of O_5 mean that a row of oxygen atoms in van der Waals contact lie along the b axis. Fig. 1, a Fourier projection down the b axis, shows that O_5 is surrounded tetrahedrally by four lead atoms at distances of 2·30 and 2·29 A. This arrangement and these distances are similar to those found in PbO (Dickinson & Friauf, 1924; Moore & Pauling, 1941). Furthermore, in PbO each lead atom is bonded to four oxygen atoms which form a square to one side of it. This arrangement can also be approximated in lanarkite by giving the sulphate group a plane of symmetry and the following parameters, again referred to the crystallographic axes:

	x (A.)	y (A.)	z (A.)
S_1	4·97	2·84 ($\frac{1}{2}b$)	1·99
O_1	3·41	2·84	1·72
O_2	5·97	2·84	3·63
O_3	5·28	1·63	1·27
O_4	5·28	4·05	1·27

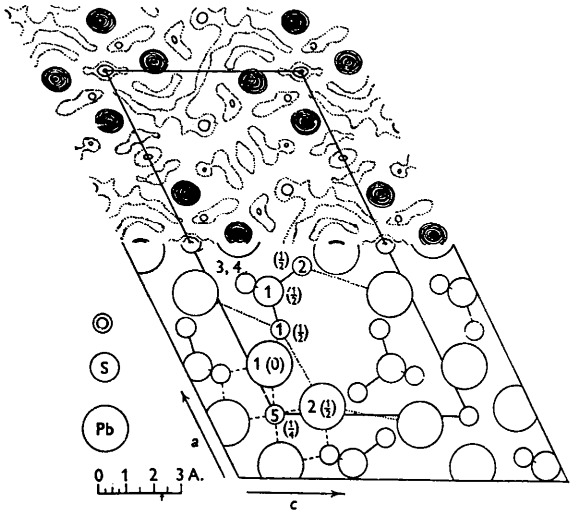

Fig. 1. Electron-density projection along b axis on (010). The unit-cell projection is outlined by solid lines forming a parallelogram. Pb–O bonds are shown by broken lines, van der Waals distances by dotted lines; y co-ordinates of atoms are indicated in brackets in the explanatory diagram.

In this manner, four oxygen atoms (two O_5 atoms at $\pm\frac{1}{4}b$, O_3 and O_4) form approximately a square on one side of each lead atom, at an average distance of 2·3 A. This position of the sulphate group cannot be confirmed from the Fourier map, but it is noteworthy that with this arrangement, a very close network of van der Waals forces exists between O_1, O_2 and the lead atoms. The reliability index, calculated from the usual formula and using these parameters, is less than 0·35 for each zone.

Work is being continued in order to check other possible positions of the sulphate group and to obtain more accurate bond distances.

References

Dickinson, R. G. & Friauf, J. B. (1924). *J. Amer. Chem. Soc.* **46**, 2457.

Doughty, J., Lark-Horovitz, K., Roth, L. M. & Shapiro, B. (1950). *Phys. Rev.* **79**, 203.

Moore, W. & Pauling, L. (1941). *J. Amer. Chem. Soc.* **63**, 1392.

Richmond, W. E. & Wolfe, C. W. (1938). *Amer. Min.* **23**, 799.

Wilman, H. (1948). *Proc. Phys. Soc.* **60**, 117.

Acta Cryst. (1951). **4**, 472

Crystal orientation on the Weissenberg goniometer.

By B. Jerslev, *The Royal Danish School of Pharmacy, Universitetsparken 2, Copenhagen Denmark*

(*Received* 24 *May* 1951)

Winchell (1950) has described a modification of the X-ray method given by Bunn (1945) for setting a crystal accurately with a zone axis parallel to the axis of rotation. A somewhat similar technique, though based upon a different theoretical aspect, was used by Arnfelt (1939).

Another modification of Bunn's method, which is considered to be quicker and as accurate, is given here. The crystal is placed as correctly as possible on the goniometer head of a Weissenberg camera. The goniometer head is turned so that the X-ray beam bisects the angle between the two arcs. A narrow strip of film wrapped in black paper is placed around the layer-line screen covering the slit, and a short exposure is made, during which the crystal is oscillated some 10–15° about the above position.

If the plane of the slit in the layer-line screen is assumed to be perpendicular to the axis of rotation and parallel to the X-ray beam, then the shadow of the slit on the film caused by the background blackening may be used as a reference line with respect to the equatorial zone, and with a 57·3 mm. diameter Weissenberg camera the adjustments α° and β° of the arcs I and II respectively are given by

$$\alpha^\circ \approx C - D \text{ mm.},\quad \beta^\circ \approx A - B \text{ mm. (see Fig. 1).}$$

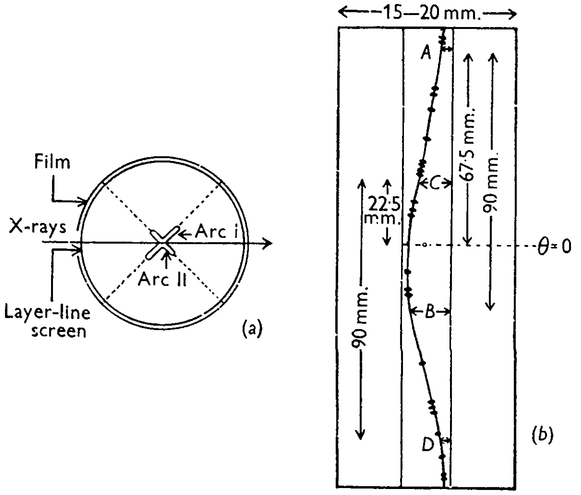

Fig. 1. (*a*) Diagrammatic drawing of the film strip and goniometer head viewed along the axis of rotation of the crystal. (*b*) Film strip flattened out. Horizontal dimensions are expanded as compared with the vertical in the figure. Spots from the equatorial zone are shown.