stantaneous orientations of the four twofold molecules which touch it. The phenomenon of molecular over-crowding has been observed in several disordered organic crystals (Dunning, 1961).

Table 1. *Observed and calculated structure factors for cyclo-octane*

hkl	F_o	F_c	*hkl*	F_o	F_c
110	5·8	10·7	330	14·6	10·9
200	49·2	45·0	420	15·4	−14·5
210	54·0	−52·2	421	17·1	8·8
211	47·3	47·1	332	—	−15·5
220	21·3	−22·1	422	4·7	7·9
310	< 16·4	18·6	430	—	9·9
222	12·7	12·9	431	—	−4·4
320	25·8	−29·3	510	—	4·3
321	21·7	−17·1	520	—	−10·9
400	38·2	−46·2	432	15·0	−5·7
410	26·2	23·6	521	13·3	−7·9
411	< 19·4	−10·6			

The discrepancy index for this structure is 0·16 (Table 1). The agreement is poorer at higher angles where the calculations are more sensitive to such details as partial ordering, molecular distortions, and the nature of the displacements. These details cannot be deduced from the limited data. Preliminary computations using the tub and the butterfly molecular configurations (Bellis & Slowinski, 1959) did not fit the data.

Acknowledgement is made to the donors of The Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

References

Allinger, N. L., Jindal, S. P. & DaRooge, M. A. (1962). *J. Org. Chem.* **27**, 4290.
Anet, F. A. L. & Hartman, J. S. (1963). *J. Amer. Chem. Soc.* **85**, 1204.
Bellis, H. E. & Slowinski, E. J., Jr. (1959). *Spectrochim. Acta*, **15**, 1103.
Chiurdoglu, G., Doehaerd, T. & Tursch, B. (1960). *Bull. Soc. Chim. Fr.* p. 1322.
Danford, M. D. (1954). *Dissert. Abst.* **14**, 1936.
Dunitz, J. D. & Prelog, V. (1960). *Angew. Chem.* **72**, 896.
Dunning, W. J. (1961). *Phys. Chem. Solids*, **18**, 21.
Finke, H. L., Scott, D. W., Gross, M. E., Messerly, J. F. & Waddington, G. (1956). *J. Amer. Chem. Soc.* **78**, 5469.
Jordan, T. H., Streib, W. E., Smith, H. W. & Lipscomb, W. N. (1964). *Acta Cryst.* **17**, 777.
Zachariasen, W. H. (1945). *Theory of X-ray Diffraction in Crystals*, pp. 223–224. New York: Wiley.

Acta Cryst. (1965). **19**, 279

The unit-cell dimensions of potassium myristate and 1:1 acid potassium myristate.

By J. H. Dumbleton*, *Physics Department, The University, Edgbaston, Birmingham* 15, England.

(*Received* 18 *December* 1964)

Piper (1929) investigated a series of neutral and 1:1 acid potassium soaps and reported the long-spacings of these compounds. Since that time a number of papers have been published on the structure and nature of neutral potassium soaps and it has been established that the neutral soaps studied by Piper were of the *B* form discussed by Lomer (1952). The same interest does not appear to have been shown in the 1:1 acid potassium soaps although studies of other acid salts have been undertaken to investigate the nature of the hydrogen bonding in these substances (Mills & Speakman, 1960). The study of the crystal structures of soaps has been hindered by the difficulty in obtaining single crystals and it was decided to try to grow single crystals of a *B* form potassium soap as a preliminary to a structure determination and potassium myristate was chosen. At the same time the growth of 1:1 acid potassium soap crystals was attempted.

Crystals of potassium myristate were grown from ethanol solution, the soap being first formed in solution by the reaction of potassium hydroxide and myristic acid. Phenolpthalein was used as indicator and the neutral solution was evaporated to dryness over calcium chloride *in vacuo* at room temperature. The crystals which formed were mostly needle-like with the *b* axis of the unit cell along the needle axis, but one or two twinned platelets were found, the large faces being (001). The twin components were found to have a common axis and this effect has been reported by Müller (1927). The crystal plates were very thin and malformed; no interedge angles could be measured. Although the crystals were not suitable for structure determination it was possible to obtain the unit-cell dimensions. The unit cell was triclinic with:

$$\begin{array}{ll} a = 4{\cdot}13 \pm 0{\cdot}02\ \text{Å} & \alpha = 91^\circ 6' \pm 20' \\ b = 5{\cdot}65 \pm 0{\cdot}02 & \beta = 91^\circ 25' \pm 20' \\ c = 34{\cdot}28 \pm 0{\cdot}08 & \gamma = 92^\circ 21' \pm 20' \\ d_{001} = 34{\cdot}26 \pm 0{\cdot}08 & \end{array}$$

These dimensions are in good agreement with those obtained by Lomer (1952) from powder photographs. The calculated density assuming 2 molecules of $CH_3(CH_2)_{12}COOK$ per unit cell was 1·105 g.cm^{-3} whilst the density

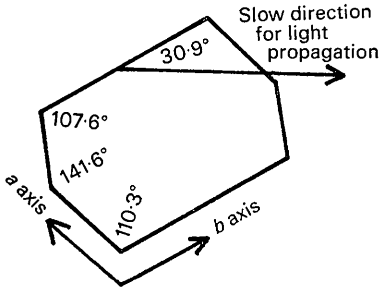

Fig. 1. Crystal of 1:1 acid potassium myristate on the (001) face.

---

[*] Present address: Chemstrand Research Center, Research Triangle Park, Durham, North Carolina, U.S.A.

observed was 1·125 g.cm^{-3}. Subsequently crystals of form *B* potassium palmitate were prepared by the above method and these crystals were well-formed and suitable for X-ray analysis. Dumbleton & Lomer (1965) have determined the crystal structure of this soap.

Crystals of 1:1 acid potassium myristate were grown by the same method used for the neutral soaps, with the difference that only half the fatty acid molecules were neutralized. The crystals formed as plates with the large faces (001) and were many times thicker than the neutral potassium myristate soap crystals. It was possible to measure interedge angles and Fig. 1 shows a drawing of a typical crystal with these measurements given. It was found that crystals could be persuaded to cleave parallel to the (001) face by pressing down on the (001) face with a sharp razor blade, and in that way crystals of suitable thickness for X-ray work were obtained. Investigation under polarized light showed that the crystals were optically positive and biaxial with the optic axial plane perpendicular to the (001) face. The unit cell was triclinic with:

$$\begin{array}{ll} a = 13{\cdot}52 \pm 0{\cdot}04\ \text{Å} & \alpha = 87°36' \pm 20' \\ b = 8{\cdot}78 \pm 0{\cdot}03 & \beta = 93°12' \pm 20' \\ c = 40{\cdot}40 \pm 0{\cdot}08 & \gamma = 110°0' \pm 20' \\ d_{001} = 40{\cdot}36 \pm 0{\cdot}08 & \end{array}$$

The value obtained by Piper for the long spacing was 40·40 Å. The calculated density assuming 6 molecules of $(CH_3[CH_2]_{12}COOK)(CH_3[CH_2]_{12}COOH)$ was 1·104 g.cm^{-3} and the observed density was 1·096 g.cm^{-3}.

This work was carried out during the tenure of a D.S.I.R. research studentship.

References

Dumbleton, J. H. & Lomer, T. R. (1965). *Acta Cryst.* In the press.

Lomer, T. R. (1952). *Acta Cryst.* **5**, 14.

Mills, H. H. & Speakman, J. C. (1961). *J. Chem. Soc.* p. 1164.

Müller, A. (1927). *Proc. Roy. Soc.* A, **114**, 542.

Piper, S. H. (1929). *J. Chem. Soc.* p. 234.

Acta Cryst. (1965). **19**, 280

Crystal data (I) for some halogenated steroids*.

By Jean M. Ohrt, Barbara A. Haner and Dorita A. Norton,
Biophysics Department, Roswell Park Memorical Institute, Buffalo, New York, U.S.A.

(*Received* 13 *October* 1964 *and in revised form* 21 *January* 1965)

In conjunction with the steroid structure program currently underway in our laboratory, single-crystal data have been obtained for ten halogenated steroids. The crystal data for two of the compounds (4-bromoestrone and 4-bromoestradiol) have already been reported in the structure papers (Norton, Kartha & Lu, 1963, 1964). Since the structures of the 16- and 21-bromo isomers of 5α-pregnan-3β,17α-diol-11,20-dione, and of androsterone 3-bromoacetate are being determined, the crystal data for these substances will be reported with the final structures. The crystal data for five halogenated steroids on which no further work is anticipated at the present time are given in Table 1.

Lattice constants were determined on a General Electric XRD-5 X-ray diffraction unit equipped with a goniostat using Cu *Kα* radiation. Space groups were established on the basis of systematic absences and optical activity. Flotation density measurements were used to calculate the number of molecules per unit cell. Compound 5 crystallized with one molecule of solvent per molecule of steroid.

References

Norton, D. A., Kartha, G. & Lu, C. T. (1963). *Acta Cryst.* **16**, 89.

Norton, D. A., Kartha, G. & Lu, C. T. (1964). *Acta Cryst.* **17**, 77.

Table 1. *Crystal data* (I) *for some halogenated steroids*

	1	2	3	4	5
Formula	$C_{21}H_{29}O_3Br$	$C_{21}H_{29}O_3Br$	$C_{19}H_{27}O_2Br$	$C_{27}H_{45}Cl$	$C_{23}H_{33}O_3Br\ .\ C_7H_{16}$
Mol. wt.	409·38	409·38	367·34	405·11	537·63
D_m (g.cm.$^{-3}$)	1·380	1·320	1·373	1·040	1·519
D_x (g.cm.$^{-3}$)	1·385	1·369	1·353	1·047	1·544
Space group	$P2_1$	$C2$	$P2_12_12_1$	$P2_1$	$P2_12_12_1$
Z	4	8	8	2	4
a(Å)*	12·413	29·243	16·442	16·333	11·419
b (Å)*	15·645	6·186	29·364	7·553	25·654
c (Å)*	10·164	22·307	7·455	10·691	7·895
β (°)	96·03	100·21	—	102·95	—
V (Å^3)	1963	3971	3599	1285	2313
Solvent	Ether	Hexane-ether	Toluene-heptane	Methanol	Heptane

* ±0·009 Å.

1. 4-Androsten-17β-ol-3-one 17-bromoacetate (17β-testosterone 17-bromoacetate)
2. 4-Androsten-6-bromo-17β-ol-3-one 17-acetate (6-bromotestosterone 17-acetate)
3. 5β-Androstan-4β-bromo-3,17-dione (4β-bromoetiocholane-3,17-dione)
4. 5-Cholestene,3β-chloro(cholesteryl chloride)
5. 5-Pregnen-17α-bromo-3β-ol-20-one 3-acetate (17α-bromopregnenalone 3-acetate)

---

* This investigation was supported in part by Public Health Service Research Grant CA-06183 from the National Cancer Institute.