

observed was  $1.125 \text{ g.cm}^{-3}$ . 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:

$$\begin{aligned} a &= 13.52 \pm 0.04 \text{ \AA} & \alpha &= 87^\circ 36' \pm 20' \\ b &= 8.78 \pm 0.03 & \beta &= 93^\circ 12' \pm 20' \\ c &= 40.40 \pm 0.08 & \gamma &= 110^\circ 0' \pm 20' \\ d_{001} &= 40.36 \pm 0.08 \end{aligned}$$

The value obtained by Piper for the long spacing was  $40.40 \text{ \AA}$ . The calculated density assuming 6 molecules of  $(\text{CH}_3[\text{CH}_2]_{12}\text{COOK})(\text{CH}_3[\text{CH}_2]_{12}\text{COOH})$  was  $1.104 \text{ g.cm}^{-3}$  and the observed density was  $1.096 \text{ g.cm}^{-3}$ .

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

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**Crystal data (I) for some halogenated steroids\***. By JEAN M. OHRT, BARBARA A. HANER and DORITA A. NORTON, *Biophysics Department, Roswell Park Memorial Institute, Buffalo, New York, U.S.A.*

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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  $5\alpha$ -pregnan- $3\beta,17\alpha$ -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  $\text{Cu K}\alpha$  radiation. Space groups were established on the basis of systematic absences and optical activity. Floation 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

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

	1	2	3	4	5
Formula	$\text{C}_{21}\text{H}_{29}\text{O}_3\text{Br}$	$\text{C}_{21}\text{H}_{29}\text{O}_3\text{Br}$	$\text{C}_{19}\text{H}_{27}\text{O}_2\text{Br}$	$\text{C}_{27}\text{H}_{45}\text{Cl}$	$\text{C}_{23}\text{H}_{33}\text{O}_3\text{Br} \cdot \text{C}_7\text{H}_{16}$
Mol. wt.	409.38	409.38	367.34	405.11	537.63
$D_m$ ( $\text{g.cm}^{-3}$ )	1.380	1.320	1.373	1.040	1.519
$D_x$ ( $\text{g.cm}^{-3}$ )	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
$a$ ( $\text{\AA}$ )*	12.413	29.243	16.442	16.333	11.419
$b$ ( $\text{\AA}$ )*	15.645	6.186	29.364	7.553	25.654
$c$ ( $\text{\AA}$ )*	10.164	22.307	7.455	10.691	7.895
$\beta$ ( $^\circ$ )	96.03	100.21	—	102.95	—
$V$ ( $\text{\AA}^3$ )	1963	3971	3599	1285	2313
Solvent	Ether	Hexane-ether	Toluene-heptane	Methanol	Heptane

\*  $\pm 0.009 \text{ \AA}$ .

- 4-Androsten- $17\beta$ -ol-3-one 17-bromoacetate (17 $\beta$ -testosterone 17-bromoacetate)
- 4-Androsten-6-bromo- $17\beta$ -ol-3-one 17-acetate (6-bromotestosterone 17-acetate)
- 5 $\beta$ -Androstan-4 $\beta$ -bromo-3,17-dione (4 $\beta$ -bromoetiocholane-3,17-dione)
- 5-Cholestene,3 $\beta$ -chloro(cholesteryl chloride)
- 5-Pregnen-17 $\alpha$ -bromo-3 $\beta$ -ol-20-one 3-acetate (17 $\alpha$ -bromopregnenalone 3-acetate)