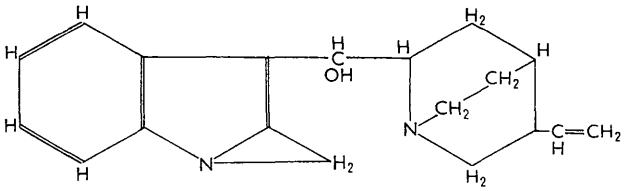
Acta Cryst. (1962). **15**, 512

Crystallographic data for apoquinamine.

By J. G. Scane, *Physics Department, College of Technology, Portsmouth, England*

(*Received* 12 *December* 1961)

Apoquinamine ($C_{19}H_{22}ON_2$) (m.pt. 115–117 °C.) has the following constitution. (Henry, Kirby & Shaw, 1945).



Good crystals, obtained from the specimen as supplied, were in the form of cream-coloured plates with (100) prominent and (001) and (10$\bar{1}$) also present.

A full survey of the reciprocal lattice, using a stationary-film single-crystal camera and Cu $K\alpha$ radiation, gave the results listed below. The density was measured by flotation.

$$a = 10{\cdot}5,\ b = 10{\cdot}3,\ c = 8{\cdot}0\ \text{Å};$$
$$\beta = 109°,\ d_c = 1{\cdot}19\ \text{g.cm.}^{-3},\ d_o = 1{\cdot}18\ \text{g.cm.}^{-3},\ Z = 2\,.$$

The only systematic absences were $0k0$, $k \neq 2n$, and, since apoquinamine exhibits optical activity, the space group is $P2_1$.

It is intended to examine the hydrohalides with a view to undertaking more detailed work on apoquinamine.

I am indebted to Dr D. Rogers for suggesting this problem, and to the Wellcome Foundation for the specimen.

Reference

Henry, T. A., Kirby, K. S. & Shaw, G. E. (1945). *J. Chem. Soc.* 524.

Acta Cryst. (1962). **15**, 512

The space group of β-In_2S_3.

By G. S. D. King, *Union Carbide European Research Associates, s.a., 95, rue Gatti de Gamond, Brussels 18, Belgium*

(*Received* 8 *January* 1962)

There has recently been a considerable interest (Rooymans, 1959; Hatwell *et al.*, 1961; Huber, 1961; Goodyear & Steigmann, 1961) in β-In_2S_3 both as regards its relation to In_3S_4 which has a spinel structure and in its twinning mechanism. As some apparently single crystals had been obtained by Hatwell and at that time the only published X-ray results were the powder measurements of Hahn & Klinger (1949) and of Rooymans, it was decided to determine the space group from Weissenberg photographs.

Preliminary experiments showed that most of the crystals were disordered but one was found that gave good Weissenberg photographs. (All photographs were taken with Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å). These photographs could be indexed in terms of a cubic lattice with $a = 32{\cdot}2$ Å, which is three times that of the In_3S_4 cell. Two of the three indices of all observed reflexions on a-axis Weissenberg photographs with $h = 0, 1, 2, 3, 4, 5, 6, 9, 12$ were multiples of three. The crystal could thus be considered to be a twin consisting of three mutually perpendicular components in which the unit cell is tetragonal with $c = 32{\cdot}2$ Å and $a = c/3$. This system of twinning is that which would be expected according to Hatwell. The reflexions from one twin component, having an a axis parallel to the Weissenberg oscillation axis, were very much more intense than corresponding reflexions from either of the other two components.

Another single-crystal fragment $0{\cdot}1 \times 0{\cdot}05 \times 0{\cdot}04$ mm. was mounted about the same axis as the first crystal. Weissenberg photographs of layer lines with $h_{\text{cubic}} \neq 3n$ showed no spots and there was no trace of these layer lines on a 60-hour rotation photograph. The crystal was therefore essentially a single component. All spots could be indexed on the basis of Rooymans' face-centred lattice so that the simple cell is body-centred and the oscillation axis was [110]. Weissenberg photographs of layer lines $0 \rightarrow 8$ inclusive led to the following results:

$$a = 7{\cdot}61,\ c = 32{\cdot}24\ \text{Å}\,.$$

Systematic absences:

hkl when $h + k + l = 2n + 1$,
$hk0$ when $h, (k) = 2n + 1$,
$h0l$ none,
hhl when $2h + l \neq 4n$.

The space group is thus $I4_1/amd$. According to the density (4·613 g.cm.$^{-3}$) there are 15·93 formula units of In_2S_3 per unit cell. This space group is that of Rooymans' proposed structure in which eight In atoms are missing from three spinel cells stacked one on top of another.

A complete structure determination will be carried out in due course.

I am grateful to Mr J. P. Pauwels for carrying out the experimental work.

References

Goodyear, J. & Steigmann, G. A. (1961). *Proc. Phys. Soc.* **78**, 491.
Hahn, H. & Klinger, W. (1949). *Z. anorg. Chem.* **260**, 97.
Hatwell, H., Offergeld, G., Hérinckx, C. & Van Cakenberghe, J. (1961). *C. R. Acad. Sci., Paris*, **252**, 3586.
Huber, M. (1961). *C. R. Acad. Sci., Paris*, **253**, 471.
Rooymans, C. J. M. (1959). *J. Inorg. Nucl. Chem.* **11**, 78.