0.148262, and we take  $\theta$ , or sin  $\theta$ , to be negative for a spot on the lower half of the film. The results are set out in Table 1.

On our photographs the spots extend over about  $1^{\circ}$  in the  $\omega$  direction. This extension must mainly be due to crystal size. We do not know the true mosaic spread. Table 1 suggests that conjunction within 5' in the  $\omega$  values is adequate to cause double reflexion, which seems a reasonable conclusion.

If a tolerance of this order applies generally, double reflexions must be fairly common. With a cell of moderate size,  $\sim 10^3$  reflexions may occur in the region covered, and the chance must be statistically high that two with at least moderate intensities will have  $\omega$  values within 5' of each other. The consequent double reflexion will be obvious only in the comparatively rare case where it leads to a breach of the space-group absences. In general it will merely add to the intensity of a normal reflexion. As was pointed out by Lipson & Cochran (see also Yakel & Fankuchen, 1962), this is yet another source of error in the measurement of intensities. It may deserve consideration when an accuracy better than 5% is being sought.

#### References

- GOLIČ, L. & SPEAKMAN, J. C. (1965). J. Chem. Soc. To be published.
- International Tables for X-ray Crystallography (1959). Vol. II, Chapter 4. Birmingham: Kynoch Press.
- JAMES, R. W. (1950). The Optical Principles of the Diffraction of X-rays. London: Bell.
- LIPSON, H. & COCHRAN, W. (1953). The Determination of Crystal Structures. London: Bell.
- YAKEL, H. L. & FAUNKUCHEN, I. (1962). Acta Cryst. 15, 1188.

## Acta Cryst. (1965). 18, 571

# A new polytype of zinc sulfide crystals. By MARIA FARKAS-JAHNKE, Research Institute for Technical Physics of the Hungarian Academy of Sciences, Budapest, Hungary

#### (Received 18 September 1964)

As is well known, zinc sulphide is liable to form polytype modifications similarly to silicon carbide, during the growing process, in addition to the wurzite and sphalerite structures. This problem has been investigated by several authors (Frondel & Palache, 1947; Strock & Brophy, 1955; Buck & Strock, 1955; Smith, 1955; Evans & McKnight, 1959; Ramsdell, 1947; Verma, 1957), and polytypes containing 2, 3, 4, 6, 8, 9, 10, 12, 15, 21, 54, ... layers have been already found among natural and artificially made zinc sulphide crystals. Investigating the structure of zinc sulphide single crystals, grown from the vapour phase, we have found, besides the known polytypes, one crystal containing a new polytype modification of 24 layers. The lattice parameters of the new modification are:

$$a_0 = 3,853, c_0 = 75,067$$
 Å;  $a_0/c_0 = 1:19,483$ ,

with cell contents  $Zn_{24}S_{24}$ . In the same crystal there are several kinds of the numerous possible 24-layer structures, as seen in the 25° oscillation patterns in Fig. 1. The X-ray diffraction patterns were made in a cylindrical camera of 57.3 mm diameter, with unfiltered Fe K radiation.

These interesting structures will be discussed later in detail.

The author expresses her thanks to Academician G. Szigeti and Dr E. Pócza for their interest. She is also very grateful to Mr P. Kovács and Mr E. Lendvay for the preparation of the crystals (Kovács & Szabo, 1962).



Fig. 1. 25° oscillation patterns taken from different parts of the 24-layer ZnS polytype crystal.

## References

BUCK, D. C. & STROCK, L. W. (1955). Amer. Min. 40, 192.

Evans, H. T. & McKnight, E. T. (1959). Amer. Min. 44, 1210.

FRONDEL, C. & PALACHE, C. (1947). Science, 107, 602.

Acta Cryst. (1965). 18, 572

Kovács, P. & Szabó, J. (1962). Acta Phys. Hung. 14, 131.

RAMSDELL. L. S. (1947). Amer. Min. 32, 64.

SMITH, F. G. (1955). Amer. Min. 40, 658.

- STROCK. L. W. & BROPHY, V. A. (1955). Amer. Min. 40, 94.
- VERMA, A. R. (1957). Proc. Roy. Soc. A. 240, 462.

Crystal data for robustic acid methyl ether. By K. V. KRISHNA RAO and P. VENKATESWARA RAO, Physics Department, Osmania University, Hyderabad 7, India

(Received 9 September 1964)

In the course of the study of scandenin (Krishna Rao & Venkateswara Rao, 1963) and related compounds, the authors have determined the space group and the unitcell dimensions of robustic acid methyl ether  $(C_{23}H_{22}O_6)$ . This compound has been obtained as platy crystals with a pinacoidal habit, by Khan (1960) during his studies on the structure of robustic acid.

Rotation and zero-layer Weissenberg photographs, taken with FeK radiation, showed that the crystal belongs to the triclinic system with the following cell dimensions.

a = 11.83,	$b = 9 \cdot 09$ ,	c = 9.36  Å;
$x = 94^{\circ}$ ,	$\beta = 96^{\circ}$ ,	$\gamma = 100^\circ$ .

Of the two possible space groups P1 and  $P\overline{1}$ , it is likely that the crystal belongs to  $P\overline{1}$ , in view of its pinacoidal habit.

The observed density  $1.35 \text{ g.cm}^{-3}$ , determined by the flotation method with a mixture of ethylbenzene and bromobenzene, agrees with the value  $1.33 \text{ g.cm}^{-3}$  calculated for two molecules per unit cell.

No further work on this crystal is contemplated.

#### References

KHAN, W. A. (1960). Ph. D. Thesis, Osmania University, Hyderabad, India.

KRISHNA RAO, K. V. & VENKATESWARA RAO, P. (1963). Indian J. Phys. 37, 546.

## Acta Cryst. (1965). 18, 572

An X-ray study of the  $\gamma$ -Cu<sub>4</sub>Cd<sub>3</sub> phase alloy. By B. N. DEY and M. A. QUADER, Indian Association for the Cultivation of Science, Calcutta 32, India

(Received 8 June 1964 and in revised form 4 August 1964)

The present note reports some crystallographic data of the y-Cu<sub>4</sub>Cd<sub>3</sub> phase alloy based on X-ray powder diffraction studies. Earlier Owen & Pickup (1933) while studying the copper-cadmium alloy system observed a diffraction pattern for the  $\gamma$  phase which according to them corresponded to a very complicated structure. Later Laves & Mueller (1938) reported that the powder photographs of both  $\beta$ -Al<sub>3</sub>Mg<sub>2</sub> and  $\gamma$ -Cu<sub>4</sub>Cd<sub>3</sub> are very similar and conclude that the two structures are isomorphous. The structure of the  $\beta$ -Al<sub>3</sub>Mg<sub>2</sub> phase was found by Perlitz (1944) to be face-centred cubic with  $a = 28 \cdot 16$  kX and 1166 atoms per unit cell. This has also been confirmed by Soulnier & Mirand (1960) from an electrondiffraction study of the alloy. However, no independent study of the  $\gamma$ -Cu<sub>4</sub>Cd<sub>3</sub> phase seems to have been made yet.

According to *Metals Handbook* (Smith, 1948) the  $\gamma$ -Cu<sub>4</sub>Cd<sub>3</sub> phase does not form on solidification but appears only after prolonged annealing at about 500 °C. The alloy investigated was made from spectroscopically pure Johnson-Matthey copper and cadmium to the composition Cu<sub>4</sub>Cd<sub>3</sub> by melting them in evacuated silica tubes. The alloy thus prepared was annealed for several days at a temperature of 470 °C, after which filings were obtained with a No. 1 file. The filings were then annealed at 470 °C for two hours to make them strain-free. All the annealing processes mentioned above were performed in evacuated and sealed Pyrex tubes. X-ray powder diffraction photographs of the alloy were taken on a 114.6 mm camera with filtered Cu  $K\alpha$  radiation. Good photographs could be obtained after 30 hours of exposure at 36 kV and 20 mA in the Philips PW 1010 X-ray unit. It was found that the photographs did not contain lines of phases other than the  $\gamma$  phase. A diffractometric study was also made. The powder diffraction data are given in Table 1. All intensities below 4 were obtained with the diffractometer. All the lines could be indexed on the basis of a tetragonal unit cell with a = 13.701 Å and c = 9.944 Å, taking wave lengths  ${}^{\lambda}k_{\alpha_1} = 1.5405$  Å and  ${}^{\lambda}K_{\alpha_2} = 1.5443$  Å for copper radiation.

The density of the alloy measured by a density bottle is  $9.09 \text{ g.cm}^{-3}$ . The calculated density on the basis of 120 atoms per unit cell is  $9.00 \text{ g.cm}^{-3}$ . By considering the extinctions the space group appears to be  $P4_{g/ncm}$ .

We wish to express our gratitude to Prof. B. N. Srivastava, D.Sc., F.N.I., for his constant interest in this