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

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 unit-cell 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 Fe K radiation, showed that the crystal belongs to the triclinic system with the following cell dimensions.

$$a = 11{\cdot}83, \quad b = 9{\cdot}09, \quad c = 9{\cdot}36 \text{ Å};$$
$$x = 94°, \quad \beta = 96°, \quad \gamma = 100° .$$

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

The observed density 1·35 g.cm^{-3}, determined by the flotation method with a mixture of ethylbenzene and bromobenzene, agrees with the value 1·33 g.cm^{-3} calculated for two molecules per unit cell.

No further work on this crystal is contemplated.

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

An X-ray study of the γ-Cu_4Cd_3 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 γ-Cu_4Cd_3 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 γ phase which according to them corresponded to a very complicated structure. Later Laves & Mueller (1938) reported that the powder photographs of both β-Al_3Mg_2 and γ-Cu_4Cd_3 are very similar and conclude that the two structures are isomorphous. The structure of the β-Al_3Mg_2 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 electron-diffraction study of the alloy. However, no independent study of the γ-Cu_4Cd_3 phase seems to have been made yet.

According to *Metals Handbook* (Smith, 1948) the γ-Cu_4Cd_3 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_4Cd_3 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 γ 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 $a = 13{\cdot}701$ Å and $c = 9{\cdot}944$ Å, taking wave lengths $^{\lambda}k_{\alpha_1} = 1{\cdot}5405$ Å and $^{\lambda}K_{\alpha_2} = 1{\cdot}5443$ Å for copper radiation.

The density of the alloy measured by a density bottle is 9·09 g.cm^{-3}. The calculated density on the basis of 120 atoms per unit cell is 9·00 g.cm^{-3}. By considering the extinctions the space group appears to be $P4_2/ncm$.

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

Table 1. *Powder diffraction data for* γ-Cu_4Cd_3; Cu $K\alpha$ *radiation*

hkl	$\sin^2\theta$ (calc.)	$\sin^2\theta$ (obs.)	Intensity
200	0·0126	0·0126	2
002	0·0240	0·0235	4
220	0·0252	0·0250	2
112	0·0303	0·0300	4
202	0·0366	0·0366	2
302	0·0524	0·0525	6
003	0·0540	0·0538	3
411	0·0600	0·0606	2
113	0·0603		
322	0·0651	0·0655	6
402	0·0745	0·0742	10
332	0·0808	0·0806	3
431	0·0850	0·0853	3
511	0·0881	0·0879	25
422	0·0872		
323	0·0950	0·0950	12
004	0·0960	0·0962	10
502	0·1030	0·1022	10
114	0·1023		
440	0·1011		
600	0·1138	0·1138	65
423	0·1172	0·1173	40
304	0·1244	0·1244	100
532	0·1314	0·1311	50
541	0·1355	0·1355	25
523	0·1456	0·1452	20
334	0·1529	0·1524	20
115	0·1563	0·1572	5
550	0·1580		
710	0·1580		
424	0·1592	0·1594	3
640	0·1643	0·1650	5
215	0·1658		
641	0·1703	0·1703	5
613	0·1709		
623	0·1804	0·1803	8
315	0·1816	0·1812	2
524	0·1876	0·1873	3
006	0·2160	0·2162	3
652	0·2167		
831	0·2367	0·2372	8
733	0·2372		
306	0·2444	0·2443	2
841	0·2590	0·2589	8
910	0·2591		
743	0·2594		
724	0·2634	0·2637	10
753	0·2878	0·2878	10
851	0·2872		
814	0·3014	0·3014	8
860	0·3160	0·3158	35
10,0,0	0·3160		
861	0·3220	0·3220	28
763	0·3226		
772	0·3337	0·3339	3
624	0·3424	0·3430	10
655	0·3427		
10,1,2	0·3431		
844	0·3488	0·3496	25
10,3,1	0·3504		
825	0·3648	0·3655	8
10,4,0	0·3665		
10,4,1	0·3725	0·3717	2
646	0·3803	0·3798	2
008	0·3840	0·3835	2
218	0·3998	0·4005	15
537	0·4014		
10,5,1	0·4010		
765	0·4180	0·4183	2
10,5,2	0·4190		
627	0·4204	0·4209	2
746	0·4214		
10,6,0	0·4297	0·4295	2
408	0·4346	0·4351	10
10,6,1	0·4358		
666	0·4435	0·4431	2
10,6,2	0·4436		
12,0,0	0·4550	0·4544	2
10,2,5	0·4785	0·4785	14
12,0,2	0·4790		
009	0·4860	0·4866	2
119	0·4923	0·4925	2
936	0·5004	0·4995	5
884			
219	0·5018	0·5018	2
11,6,1	0·5021		
12,2,3	0·5216	0·5214	2
12,4,2	0·5295	0·5291	2
339	0·5429	0·5431	10
10,2,6	0·5446	0·5450	10
995	0·6619	0·6623	15
12,0,6	0·6710	0·6714	15
669	0·7135	0·7133	10
6,3,10	0·7422	0·7424	15
13,0,6	0·7500	0·7496	15
13,13,4	0·8606	0·8611	8
16,3,2	0·8613		

work. One of the authors (B.N.D.) is grateful to C.S.I.R. (New Delhi) for partial support of the work.

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Acta Cryst. (1965). **18**, 574

Die Kristallstruktur des sauren Kaliummetasilikates $K_4(HSiO_3)_4$. Korrektur.

Von WALTRAUD HILMER, *Institut für anorganische Chemie der Deutschen Akademie der Wissenschaften zu Berlin, Berlin–Adlershof, Rudower Chaussee, Deutschland*

(*Eingegangen am* 16. *Oktober* 1964)

Auf Einwand von Frau Prof. Boll-Dornberger, im $K_4(HSiO_3)_4$ würde höhere Symmetrie als monoklin vorliegen, ergab ein Intensitätsvergleich von Aufnahmen verschiedener Kristalle, dass die Abweichungen der Intensitäten von rhombischer Symmetrie auf Absorption zurückzuführen sind. Intensitätssymmetrie, Auslöschungen und *I*-Statistik nach Howells, Phillips & Rogers der früher angegebenen Aufnahmen führen jetzt zur Raumgruppe *Bmab*. Dann ist $a_{\text{rhomb.}} = |\mathbf{a}-\mathbf{c}|_{\text{mon.}} = 11{,}50$ Å, $b_{\text{rhomb.}} = b_{\text{mon.}} = 11{,}25$ Å, $c_{\text{rhomb.}} = |\mathbf{a}+\mathbf{c}|_{\text{mon.}} = 9{,}65$ Å; $Z = 16$. Berücksichtigung der höheren Symmetrie führt zu folgenden Koordinaten in der rhombischen Zelle:

	x/a	y/b	z/c
K(1)	250	250	737
K(2)	0	043	822
Si(1)	500	184	986
Si(2)	302	0	0
O(1)	0	310	560
O(2) (=O(2, 3) mon.)	389	110	044
O(4)	500	199	825
O(5) (=O(5, 6) mon.)	227	029	870

Die in der Veröffentlichung angegebenen *R*-Faktoren ändern sich von 13,1% auf 12,8% und von 9,5% auf 9,3% (bzw. 17,6% auf 17,8%). Ein zur Kontrolle berechneter Bereich von (1*kl*)-Reflexen führte ebenfalls zu guter Übereinstimmung zwischen F_o und F_c.

Die Si–O-Abstände ändern sich im Mittel um 0,01 Å (maximal um 0,03 Å), ∢Si(1)–O(2,3)–Si(2) = 140,7°.

Ich danke Frau Prof. Boll-Dornberger für ihren Hinweis.

Acta Cryst. (1965). **18**, 574

Crystal data on $FeAlO_3$.

By R. R. DAYAL, J. A. GARD and F. P. GLASSER, *Department of Chemistry, University of Aberdeen, Scotland*

(*Received* 21 *September* 1964)

The $FeAlO_3$ phase was discovered by Richardson, Ball & Rigby (1954), who synthesized it by heating a 1:1 Fe_2O_3–Al_2O_3 mixture in oxygen at 1400° C. They also established that the product was a homogeneous single phase, distinct from α-Al_2O_3, α-Fe_2O_3 or α-$(Al, Fe)_2O_3$ solid solutions. This $FeAlO_3$ phase has subsequently been encountered by others, principally Atlas & Sumida (1958), Muan & Gee (1955) and Muan (1958). The latter workers explored the small range of stoichiometry and the limited range of thermodynamic stability of this phase; for example at $P_{O_2} = 1$ atm. the stability range is 1318°–1495 °C. The present authors encountered the $FeAlO_3$ compound during equilibrium studies in the system CaO–Al_2O_3–Fe_2O_3. Powder X-ray data were obtained from nearly pure preparations; these data (Table 1 (*c*)) agree very closely with those of Richardson *et al.* (1954), rather less well with those of Atlas (1958). Richardson *et al.* obtained a unit cell from trial-and-error procedures; the correctness of the cell was apparently supported by an analogy between $FeAlO_3$ and that of κ-Al_2O_3. Present efforts to improve the fit between d_o and d_c were, however, unsuccessful. Efforts to grow single crystals of $FeAlO_3$ would obviously be handicapped by the limited stability of this phase; the prospect of growing single crystals appeared uninviting. It was therefore decided to examine the relatively fine-grained powders by selected-area electron diffraction.

A 1:1 mixture of coprecipitated hydrous oxides of Fe^{3+} and Al^{3+} was prepared. Gentle ignition at *ca.* 500 °C produced a nearly amorphous starting material. Heating at 1370° ± 3 °C for four days in air gave a nearly pure $FeAlO_3$ preparation, judging by the near absence of α-Al_2O_3 and α-Fe_2O_3 reflexions in powder photographs. Fe_3O_4 (magnetite) was absent. The sample adhered strongly to a small permanent magnet.

Samples were crushed gently and dispersed in isopropyl alcohol using ultrasonic vibration. Drops of this suspension were transferred to specimen grids carrying carbon films lightly coated with evaporated aluminum for calibration of the camera constant λL. The specimens were examined with an A.E.I. type EM3 electron microscope equipped with a goniometric stage permitting rotation and a maximum tilt of 45°, modified from a design of Burge & Munden (1960).

Approximately 30 crystals were examined. These all