Acta Cryst. (1955). **8**, 739

The crystal structure of $KAsF_6$.

By Raymond B. Roof Jr., *Mineralogical Laboratory, University of Michigan, Ann Arbor, Michigan, U.S.A.*

(*Received* 2 *May* 1955 *and in revised form* 1 *July* 1955)

Potassium arsenic hexafluorine is the first of an extensive series of arsenic–fluorine compounds being prepared for investigation by the Chemistry Department at the University of Michigan. The purpose of the investigation of the crystal structure of $KAsF_6$ was to establish the interatomic distance between As and F and to examine the role of K in the bonding of the crystal.

Minute single crystals were grown from a water solution and these crystals exhibited the symmetry $\bar{3}$. Weissenberg photographs established the space group as $R\bar{3}$. The hexagonal unit cell lattice constants are

$$a = 7{\cdot}352,\ c = 7{\cdot}235\ \text{Å}.$$

From a measured density of 3·29 g.cm.$^{-3}$ the number of formula weights per unit cell was determined as $Z = 3$. Intensity measurements were made by a photodensitometer trace of a powder photograph of the material which was taken with Cu $K\alpha$ radiation.

Table 1. *Comparison of observed and calculated powder line intensities*

(Cu $K\alpha$ radiation)

Line No.	θ (°)	*hk.l*	F_o^2p	F_c^2p
1	9·2	10.1	30	25
2	12·1	11.0	69	82
3	14·1	01.2	72	61
4	15·3	02.1	27	33
5	18·7	00.3, 20.2	27	30
6	19·7	21.1, $\bar{2}$3.1	15	8
7	21·3	30.0	21	19
8	22·6	12.2, 11.3, $\bar{1}$2.3, $\bar{1}$3.2	228	285
9	24·8	22.0	48	67
10	26·1	10.4	24	25
11	26·7	13.1, $\bar{1}$4.1	42	34
12	29·0	4$\bar{1}$.2, 03.3, 31.2, 30.3, 02.4	105	113
13	32·0	04.2, 22.3, 21.4, 3$\bar{1}$.1	198	155
14	32·6	32.1, 5$\bar{2}$.1	24	28
15	33·7	41.0, 14.0	78	72
16	34·6	23.2, $\bar{2}$5.2, 01.5	81	69
17	35·5	20.5	15	18
18	37·4	05.1, 13.4, $\bar{1}$4.4	54	36
19	39·0	33.0, 12.5, $\bar{1}$3.5	63	86
20	39·8	00.6, 50.2, 41.3, 14.3	90	97

Table 1 compares the observed and calculated powder line intensities. The observed intensities have been corrected for the Lorentz and polarization factors. The factor p is the multiplicity factor. An experimentally determined temperature factor, $B = 0{\cdot}7$ Å^2, was applied to the calculated intensities. The interpretation of the powder photograph is also given.

The crystal structure of $KAsF_6$ can be described as follows:

$$(0, 0, 0),\ (\tfrac{1}{3}, \tfrac{2}{3}, \tfrac{2}{3}),\ (\tfrac{2}{3}, \tfrac{1}{3}, \tfrac{1}{3}) +$$
$$1\ \text{As at}\ 0, 0, 0;$$
$$1\ \text{K at}\ 0, 0, \tfrac{1}{2};$$
$$6\ \text{F at}\ x, y, z\ \text{etc.},$$

where

$$x = 0{\cdot}117,\ y = 0{\cdot}234,\ z = 0{\cdot}152\ .$$

These coordinates were determined from Fourier projections on 00.1 for x and y and $\bar{1}$2.0 for z. The As–F distance was determined to be 1·85 Å. The value of the residual computed from the structure factors is $R = 0{\cdot}19$.

The six F atoms are arranged in a very slightly distorted octahedral coordination around the arsenic atom. The attractive forces of the K atom have caused the F atoms to move parallel to the 111 direction of the octahedron so that the F–As–F angle is reduced to 88°. The structure can be compared to that of an ideal simple cubic structure (the AsF_6 groups at the corners of the cube and the K atom at the center of the cube) in which the attractive forces of the K atom have reduced the c/a ratio of the ideal cubic by 20%.

Acta Cryst. (1955). **8**, 739

Crystallographic data for dioxospartalupine hydrate, $C_{15}H_{22}N_2O_2 . H_2O$.*

By A. E. Lessor, Jr.,† *Chemistry Department, Indiana University, Bloomington, Indiana, U.S.A.*

(*Received* 15 *July* 1955)

Crystals grown from a solution in hot *n*-hexane were long needles with well formed pinacoids {001}. Precession and Weissenberg photographs were taken with the crystal mounted parallel to the needle axis, and gave the orthorhombic unit-cell dimensions:

$$a = 6{\cdot}19,\ b = 10{\cdot}39,\ c = 21{\cdot}92\ \text{Å}.$$

There are four molecules per unit cell. Density: calculated 1·32 g.cm.$^{-3}$; observed (flotation) 1·28 g.cm.$^{-3}$.

Reflections of types (h00), (0k0) and (00l) were ob-

* Contribution No. 681, Chemistry Department, Indiana University.

† Present address: Chemistry Department, University of Cincinnati, Cincinnati, Ohio, U.S.A.

Table 1. *Powder diffraction data*

d (Å)	I/I_0	d (Å)	I/I_0
4·62	0·46	2·25	0·42
3·46	1·00	2·18	0·64
3·21	0·42	2·01	0·26
3·02	0·42	1·86	0·20
2·60	0·55	1·69	0·30

served only when $h = 2n$, $k = 2n$, $l = 2n$, fixing the space group as D_2^1–$P2_12_12_1$. The powder pattern was run using nickel-filtered Cu $K\alpha$ radiation and Eastman Kodak No-Screen X-ray film. The ten most intense lines, estimated visually by comparison with standard intensity strips, are summarized in Table 1.

The twofold symmetry of the molecule has been proven by the direct synthesis of the compound and no X-ray structure determination is being contemplated.

I wish to thank Dr Marvin Carmack and Miss Hanna Suss, who kindly furnished the crystals used in this work. This work was supported by the O. O. R.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. Copy should be sent direct to the British Co-editor (R. C. Evans, Crystallographic Laboratory, Cavendish Laboratory, Cambridge, England).

Acta Crystallographica

Readers are reminded that current subscriptions expire on the appearance of Part 12 to be published on 10 December 1955. To ensure continuity of supply orders for Volume 9, with remittance, should be placed through the usual channels as soon as possible, and in any case in time to reach the publishers in Copenhagen not later than 31 December 1955.

International Union of Crystallography

Open Meeting of the Commission on Crystallographic Teaching

As already announced, an open meeting of the Commission will be held in Madrid, Spain, during the period 2–7 April 1956. A circular will shortly be sent to all academic departments known to the Commission to be engaged in the teaching of crystallography, but any person who wishes to ensure receipt of this circular should apply to the Secretary (Dr H. Judith Grenville-Wells, Department of Chemistry, University College, Gower Street, London W.C. 1, England). This circular will give particulars of the sessions and speakers and of arrangements for accommodation, travel and excursions.

The five open sessions of the meeting will be as follows:

1. How crystallography is now taught in several countries; crystallographic teaching in Spain.
2. Apparatus and books for teaching (joint session with the Commission on Crystallographic Apparatus).
3. Crystal geometry: morphological; stereochemical.
4. Structure analysis: general; mathematical techniques and machines.
5. Crystal physics: general and optics; metals.

Short written communications are invited on any topic connected with teaching, and those received by the Secretary not later than 1 February 1956 will be circulated before the meeting.

Books Received

The undermentioned works have been received by the Editors. Mention here does not preclude review at a later date.

Diffusion in Metallen. By W. Seith and T. Heumann. Pp. vi+306 with 238 figs. Berlin, Göttingen, Heidelberg: Springer. 2nd ed. 1955. Price DM. 39.

Dielectric Behavior and Structure. By C. P. Smyth. Pp. 441. New York; Toronto; London: McGraw-Hill. 1955. Price \$9·00; 64*s.* 6*d.*

Report of the Conference on Defects in Crystalline Solids. Pp. 429 with many figs. and plates. London: The Physical Society. 1955. Price 40*s.*

Petrographic Mineralogy. By E. E. Wahlstrom. Pp. 408. New York: Wiley; London: Chapman and Hall. 1955. Price \$7·75; 62*s.*

Praktische Edelsteinkunde. By W. Fischer. Pp. 187 with 96 figs. and 3 tables. Kettwig/Ruhr: Gustav Feller-Nottuln. 2nd ed. 1954. Price DM. 16·80; 30*s.*; \$4·00.

X-Ray Diffraction by Polycrystalline Materials. Edited by H. S. Peiser, H. P. Rooksby and A. J. C. Wilson. Pp. 725 with 263 figs. London: Institute of Physics. 1955. Price 63*s.*; \$9.

College Chemistry. By L. Pauling. Pp. xii+697 with 202 figs. San Francisco: Freeman; London: Bailey Brothers and Swinfen. 2nd ed. 1955. Price \$6·00; 51*s.*