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Potassium Hexafluororhenate (IV)

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Abstract. K_2ReF_6, $M_r = 378{\cdot}4$, trigonal, $P\bar{3}m1$, $a = 5{\cdot}879$ (1), $c = 4{\cdot}611$ (3) Å, $U = 138{\cdot}02$ Å^3, $T = 292$ K, $Z = 1$, $F(000) = 164{\cdot}96$, $d_c = 4{\cdot}55$ g cm^{-3}, μ(Mo $K\alpha$) = 225·72 cm^{-1}. The crystals are of the K_2GeF_6 type. Re—F = 1·953 (4) Å but the ReF_6^{2-} octahedron is slightly compressed giving F—Re—F angles for adjacent F atoms of 93·8 (2) and 86·2 (2)°. The K^+ ions are not coplanar with the hexagonal-close-packed layers of F atoms. The 12 F neighbours to each K^+ are in a 3 + 6 + 3 arrangement at 2·789, 2·957 and 2·998 (4) Å.

Introduction. Crystals were supplied by Mr J. Rogers and Professor R. D. Peacock of the University of Leicester as pale pink hexagonal plates developed on {100} and {001}. Photographs showed the crystals to be trigonal, with diffraction symmetry $P\bar{3}m$, and no systematic absences. Cell dimensions were derived from the observed 2θ values for axial reflections on a Stoe Weissenberg diffractometer (graphite-monochromated Mo $K\alpha$ X-rays). The crystal used for data collection was 0·20 × 0·20 × 0·10 mm. Intensities were recorded for $hk0$ through $hk7$ to $\theta_{\max} = 40°$ with an ω-scan technique. The optimum scan width was computed for each reflection (Freeman, Guss, Nockolds, Page & Webster, 1970). The scan rate was 1° s^{-1} with stationary-background measurements of 20 s at each end of the scan range. Every accessible quadrant was examined to give between 4 and 12 measurements for each unique reflection. Attenuators were not required as the maximum count was 8000 s^{-1}. The data were corrected for Lorentz, polarization, and absorption effects (maximum and minimum transmission coefficients applied to F were 0·507 and 0·444) before equivalent reflections were averaged to yield 329 unique reflections, all of which were of significant intensity. The index for internal consistency, $\{\Sigma(N\Sigma\{w[F(\mathrm{mean}) - F]^2\})/\Sigma[(N-1)\Sigma(wF^2)]\}^{1/2}$ [where the inner summations are over the N equivalent reflections averaged to give F(mean), and the outer summations are over all unique reflections], was 0·040.

The possible space groups were $P321$, $P3m1$, and $P\bar{3}m1$. The Patterson map was consistent with Re (0,0,0); K ($\frac{1}{3}$,$\frac{2}{3}$,0·3); F (0·16,–0·16,–0·23) in $P\bar{3}m1$, and refinement was undertaken on that basis. There was no indication at any stage that the symmetry should be relaxed. The full least-squares matrix was used to minimize $\Sigma w(|F_o| - |F_c|)^2$. Scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974).

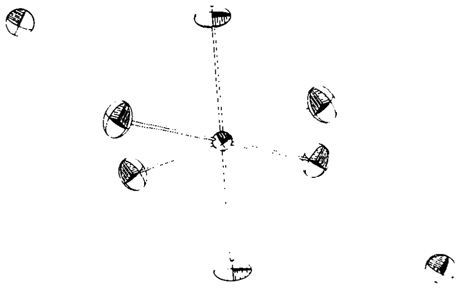

Fig. 1. Anisotropic thermal ellipsoids for K_2ReF_6 enclosing 50% probability volumes.

Table 1. *Final atomic parameters with estimated standard deviations in parentheses*

The temperature factor has the form $\exp[-2\pi^2(h^2U_{11}a^{*2} + \cdots + 2hkU_{12}a^*b^*)]$ and is given × 10^4.

	x	y	z	$U_{11} = U_{22}$	U_{33}	$U_{23} = -U_{13}$	U_{12}
Re	0·0	0·0	0·0	88 (2)	77 (2)	0·0	$\frac{1}{2}U_{11}$
K	$\frac{1}{3}$	$\frac{2}{3}$	0·3007 (5)	193 (5)	170 (7)	0·0	$\frac{1}{2}U_{11}$
F	0·1617 (4)	–0·1617 (4)	–0·2276 (9)	274 (14)	217 (15)	–39 (6)	202 (16)

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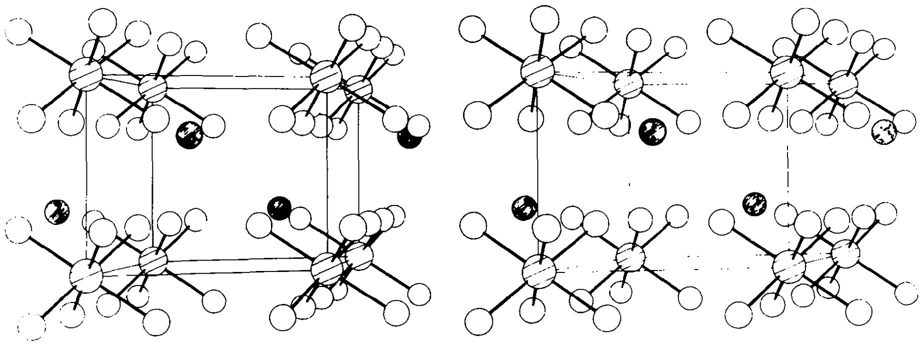

Fig. 2. Stereopair showing the packing arrangement.

Initial refinement, with unit weights and isotropic temperature factors, gave $R = 0{\cdot}078$. Subsequent cycles employed anisotropic thermal parameters, a weighting parameter, g, and an isotropic extinction parameter, x. [$w \propto 1/\sigma^2(F) + gF^2$; $F_c = F(1 - xF^2/\sin\theta)$.] After four cycles refinement converged. The maximum shift/error was 0·08 and there were no systematic trends in the calculated shifts. R was 0·032 and R_w $\{= [\Sigma\, w(|F_o| - |F_c|)^2/\Sigma\, w|F_o|^2]^{1/2}\}$ was 0·030.* Final values for g and x were $1{\cdot}79 \times 10^{-3}$ and $1{\cdot}20\,(6) \times 10^{-5}$ respectively.

Computer programs used were *SHELX* (Sheldrick, 1976) and *ABSCR*, a local version of *ABSCOR* (de Meulenaer & Tompa, 1965; Alcock, 1969). Final parameters are given in Table 1.

Discussion. Compounds belonging to the K_2GeF_6 structure type (Hoard & Vincent, 1939) include Cs_2ZrF_6, Rb_2ZrF_6, Cs_2HfF_6, Rb_2HfF_6 (Bode & Teufer, 1956), K_2ReF_6, $(NH_4)_2ReF_6$, Rb_2ReF_6, and Cs_2ReF_6 (Peacock, 1956; Weise, 1956; Schwochau, 1964). Unfortunately, only powder data were used for all these compounds, and since the F atoms make only a minor contribution to the X-ray intensities, the M—F distances have not been accurately determined. Calculations of crystal lattice energies require precise atomic coordinates, and when single crystals of K_2ReF_6 became available we undertook its structure determination.

K_2ReF_6 conforms to the K_2GeF_6 type (Hoard & Vincent, 1939). The geometry of one unit is shown in Fig. 1 while the overall packing can be seen in the stereopair of Fig. 2. The octahedral ReF_6^{2-} ions are not perfectly regular. The Re—F distances, 1·953 (4) Å, are required to be equal, but the F—Re—F angles are 180, 93·8 (2) or 86·2 (2)°, the octahedron being slightly compressed along **c**. A similar compression of the isoelectronic OsF_6^- anion has been found in $KOsF_6$ (Hepworth, Jack & Westland, 1956).

The K^+ ions are not coplanar with the *ABAB*... stacked layers of F atoms and therefore the 12 F neighbours to each K^+ are in a 3 + 6 + 3 arrangement with K—F = 2·789 (4), 2·957 (4) and 2·998 (4) Å. A similar arrangement has been observed in K_2TiF_6 where each K is surrounded by three F at 2·75, six F at 2·87, and three F at 3·08 Å (Siegel, 1952). In K_2GeF_6 (Hoard & Vincent, 1939) nine F atoms are at 2·85 and three are at 3·01 Å.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33148 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.