

## The Crystal Structure of Potassium Heptafluoroprotactinate(V)

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ALTHOUGH potassium heptafluoroprotactinate was one of the earliest of protactinium compounds to be prepared<sup>1</sup> and it is easy to crystallise, no studies of its crystal structure have yet been reported. We now present a preliminary account of such a study.

A well-formed elongated crystal was chosen and mounted in a Lindemann glass capillary. Single-crystal oscillation patterns and Guinier powder patterns showed the lattice to be *C*-centred monoclinic with  $a = 13.94$ ,  $b = 6.76$ ,  $c = 8.24$  Å,

$\beta = 125.5^\circ$ , and  $Z = 4$ . A *c*-glide was deduced from systematic absences and the space group  $C2/c - C_{2h}^{12}$  (No. 15) was chosen. (An attempt to refine in the alternative non-centric *Cc* led to no significant improvement.)

Filtered molybdenum  $K_\alpha$  radiation was used to produce Weissenberg films on which 589 independent reflections were measured; the [001] direction parallel to the longest crystal edges, was the oscillation axis. After correction of the measured intensities for Lorentz and polarization

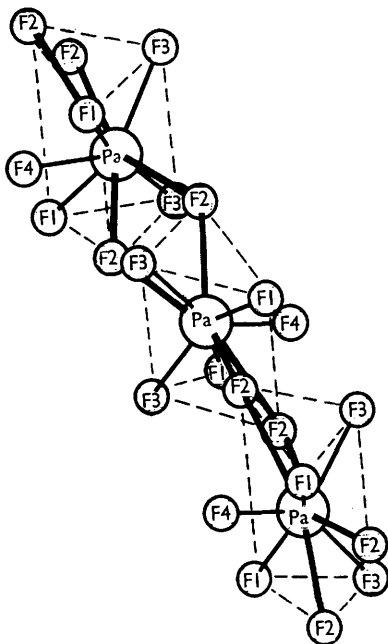
<sup>1</sup> A. von Grosse, *Proc. Roy. Soc.*, 1935, *A*, **150**, 363.

TABLE I  
Positional and thermal parameters in  $K_2PaF_7$  (space group  $C2/c$ )

	x	y	Z	$b_{11}$	$b_{22}$	$b_{33}$	$b_{23}$	$b_{31}$	$b_{12}$
Pa in (e)	0	0.0236	0.2500	0.0021	0.0057	0.0028	0	0.0006	0
K in (f)	0.3273	0.0085	0.2590	0.0044	0.0174	0.0151	-0.0030	0.0092	0.0009
F1 in (f)	0.8223	0.1221	0.1406	0.0026	0.0296	0.0195	0.0080	-0.0014	0.0047
F2 in (f)	0.0756	0.1260	0.5724	0.0096	0.0691	-0.0197	-0.0444	0.0200	-0.0273
F3 in (f)	0.8918	0.7799	0.0742	0.0055	0.0128	0.0107	0.0040	0.0025	-0.0078
F4 in (e)	0	0.3540	0.2500	0.0047	0.0163	0.0985	0	0.0397	0

effects and for X-ray absorption, the metal positions were obtained from a three-dimensional Patterson synthesis and the fluorines from a difference Fourier synthesis. Refinement of these positions using first isotropic, and later anisotropic, temperature parameters has yielded the

but not the imaginary part of the anomalous dispersion correction has been applied. The negative value of  $b_{33}$  for F2 shows that the anisotropic thermal parameters are not entirely meaningful, probably due to residual absorption errors.



results shown in Table I with an  $R$ -factor, based on observed reflections only, of 0.102. The real,

TABLE II

Interatomic distances in  $K_2PaF_7$

Pa-2 F1	2.19Å
Pa-2 F2	2.32
Pa-2 F2 (equatorial)	2.46
Pa-2 F3	2.13
Pa-F4	2.23
K-9F	2.66 to 3.04
K-3F	3.42 to 3.87

Each protactinium atom is surrounded by nine fluorines in an arrangement which may be idealised as a trigonal prism with three equatorial fluorines added. These  $PaF_9$  groups are linked in infinite chains parallel to  $[001]$  by two-fluorine bridges. Each bridging fluorine atom is equatorial to one and non-equatorial to the other protactinium, the angles  $FPaF$  and  $PaFPa$  within a bridge being  $60^\circ$  and  $120^\circ$  respectively. The non-bridging equatorial fluorine and the protactinium atom lie on a two-fold axis which is the only crystallographically imposed symmetry of the  $PaF_9$  group. The structure is illustrated in the Figure and the current values of the principal interatomic distances are listed in Table II. Refinement is continuing and full details of the structure will be published in due course.

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