Table 1. *Debye–Scherrer diagram of* $BaSrFe_4O_8$

h	k	l	d_{obs}	d_{calc}	I_{obs}
0	1	1	6·14	6·145	*vw*
1	0	1	4·734	4·729	*w*
1	1	2	3·247	3·246	*m*
1	2	1	3·112	3·112	*vs*
0	2	2	3·073	3·072	*s*
2	0	0	2·758	2·758	*ms*
1	0	3	2·679	2·678	*s*
2	1	0	2·616	2·616	*vw*
1	1	3	2·549	2·547	*vw*
2	1	1	2·517	2·516	*vw*
0	0	4	2·297	2·297	*w*
0	4	0	2·067	2·066	*ms*
2	2	2	2·051	2·052	*ms*
0	2	4	2·008	2·008	*m*
2	3	1	1·906	1·907	*w*
1	4	1	1·895	1·893	*vw*
1	3	4	1·678	1·680	*w*
3	2	1	1·652	1·652	*ms*
1	4	3	1·635	1·636	*m*
2	2	4	1·623	1·623	*w*
1	2	5	1·606	1·606	*m*
3	0	3	1·576	1·576	*ms*
0	0	6	1·531	1·531	*w*
1	5	2	1·497	1·497	*vw*
0	6	0	1·377	1·377	*w*
2	0	6	1·339	1·339	*vw*
2	1	6	1·322	1·322	*vw*

Intensities: *vs*=very strong, *s*=strong, *m*=medium, *ms*=medium strong, *w*=weak, *vw*=very weak. Wavelengths: Cu $K\bar{\alpha}=1·5418$, Cu $K\alpha_1=1·5405$, Fe $K\bar{\alpha}=1·9373$, Fe $K\alpha_1=1·9360$ Å.

for about five days. The resulting black material looked like a coarse-grained disk with slightly rounded edges.

Fragments were chiselled out of the sintered mass. They were characterized by sharp edges and highly reflecting surfaces, although no characteristic shape could be recognized. They were inspected by means of a Laue flat camera. Precession photographs were taken with Co $K\alpha$ X-radiation. The unit cell is orthorhombic with lattice parameters $a=5·516$, $b=8·265$ and $c=9·188$ Å ($\pm 0·001$ Å) at 25°C. Systematic extinctions occurred for reflexions $0kl$ with $k+l\neq 2n$, $h0l$ with $h+l\neq 2n$ and $hk0$ with $h\neq 2n$; no extinction was observed for the general reflexions hkl. The space group therefore is *Pnna*, No. 52 of *International Tables for X-ray Crystallography* (1952). Assuming two formula units per unit cell the theoretical density is 4·57 g.cm^{-3} which may be compared with the observed value of 4·62 g.cm^{-3} measured by pycnometry methods.

From these unit-cell dimensions and this space group it has been possible to index the powder pattern (Table 1). The observed spacings are mean values obtained with Cu $K\alpha$ and Fe $K\alpha$ radiations.

No further structural work on this compound is contemplated at present.

References

BARBARIOL, I. & BATTI, P. (1968). *Univ. Trieste Ist. Chim. Appl.* No. 25.

BATTI, P. (1962). *Ann. Chim. (Rome)*, **52**, 1227.

DODINH, C. & BERTAUT, E. F. (1965). *Bull. Soc. franç. Minér. Crist.* **88**, 413.

International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.

OKAZAKI, C., MORI, S. & MITSUDA, H. (1963). *Acta Cryst.* **16** (13), A23.

Acta Cryst. (1970). B**26**, 1185

The crystal structure of $KNaThF_6$.*

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Crystals of $KNaThF_6$ are hexagonal $P\bar{3}$ with $a_0=6·3073$ (2) and $c_0=7·8907$ (2) Å. The structure of $KNaThF_6$ is a framework of Na octahedra and K and Th 9-coordinated polyhedra.

The complex fluoride compound $KNaThF_6$ melts incongruently to $Na_7Th_6F_{31}$ and liquid at 674°C (Brunton, Insley, McVay & Thoma, 1965). It is uniaxial negative with $N_O=1·454$ and $N_E=1·448$.

Single crystals of $KNaThF_6$ were obtained from a quench (Friedman, Hebert & Thoma, 1962) of the stoichiometric composition. The crystals were ground to approximately spherical shape in an air driven race. An ellipsoid of the

Table 1. *Atomic parameters for* $KNaThF_6$

The number in parentheses is the standard error in terms of the last significant digit as derived from the variance-covariance matrix.

	x	y	z	β_{11}[a]	β_{22}	β_{33}	β_{12}	[b]
Th	$\frac{1}{3}$	$\frac{2}{3}$	0·1221 (2)	0·0034 (4)	(c)	0·0039 (3)	(c)	
K	$\frac{1}{3}$	$\frac{2}{3}$	0·608 (2)	0·014 (2)	(c)	0·004 (2)	(c)	
Na	0	0	0·236 (3)	0·014 (3)	(d)			
F(1)	0·104 (3)	0·381 (3)	0·322 (2)	0·011 (2)	(d)			
F(2)	0·395 (3)	0·319 (3)	0·097 (2)	0·010 (2)	(d)			

[a] Coefficients in the temperature factor: $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$.
[b] $\beta_{13}=\beta_{23}=0$.
[c] $2\beta_{12}=\beta_{22}=\beta_{11}$.
[d] The temperature factors for Na, F(1) and F(2) were constrained to be isotropic.

dimensions 0·060 × 0·120 × 0·120 mm was selected for single-crystal diffraction. The intensity data were collected with a Picker four-circle goniostat and a scintillation counter detector using unfiltered Ag $K\alpha$ radiation. The linear absorption coefficient of $KNaThF_6$ is $\mu = 289{\cdot}0$ cm^{-1} for Ag $K\alpha$ and an absorption correction was made for each reflection.

The unit-cell parameters were determined by a least-squares adjustment of 22 high angle ($116° < 2\theta < 140°$) Cu $K\alpha_1$ ($\lambda = 1{\cdot}54050$ Å) reflections; $a_0 = 6{\cdot}3073$ (2), $c_0 = 7{\cdot}8907$ (2) Å, $Z = 2$ and $\varrho = 4{\cdot}985$ g.cm^{-1}. The diffraction symmetry is $\bar{3}$ and there are no extinction conditions. Space groups $P3$ (143) and $P\bar{3}$ (147) are consistent with these conditions. The final structure has the space group $P\bar{3}$.

The structure was refined by iterative least-squares using a modification of the Busing, Martin & Levy (1962) computer program. The initial z parameter for Th atom at $2(d)$ in $P\bar{3}$ was determined from the three-dimensional Patterson function. The quantity minimized by the least-squares program was $\sum w||sF_o^2| - |F_c^2||^2$ with weights, w, derived from the empirical equation (Brown & Levy, 1964):

$$\sigma^2(F_o^2) = \lfloor T + B + \{0{\cdot}05\,(T - B)\}^2 \rfloor / [A(\mathrm{Lp})^2]\,,$$

where T = total counts, B = background counts, A = absorption correction and Lp = Lorentz–polarization factor. Anisotropic temperature factors were calculated for Th^{4+} and K^+ and isotropic factors for Na^+ and F^-. An extinction correction was made on F_c by the method of Zachariasen (1967). The scattering factors for the ions were taken from

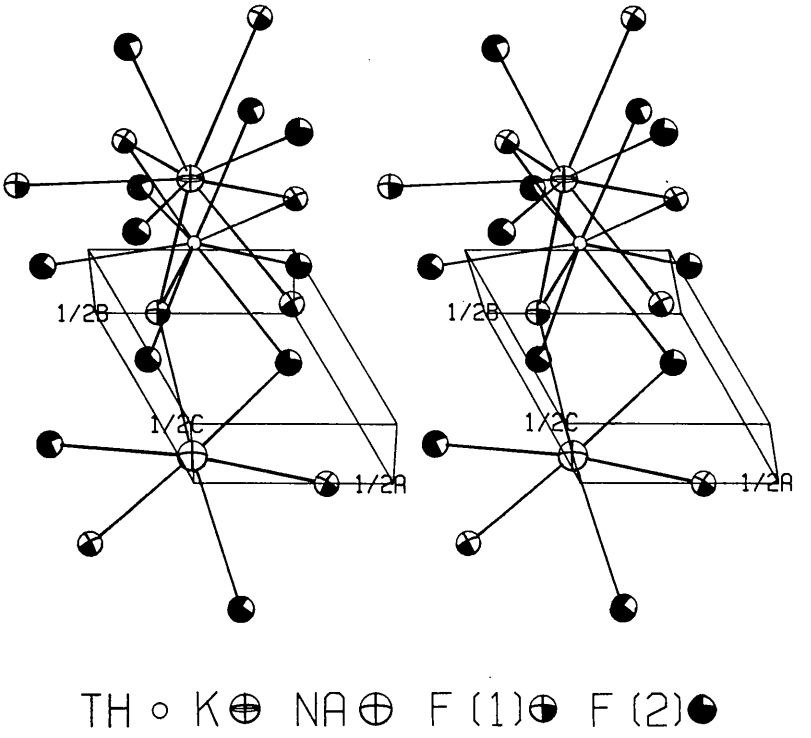


Fig. 1. One asymmetrical unit of $KNaThF_6$ – one quarter cell outlined. There is a triad inversion axis parallel to the cell edge through Na and a triad axis through Th and K.

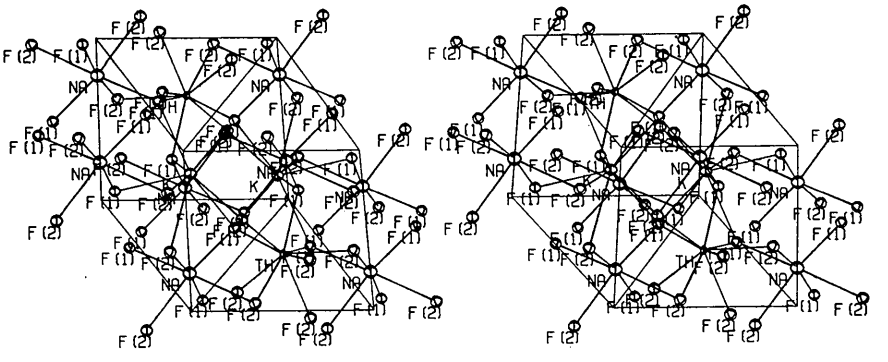


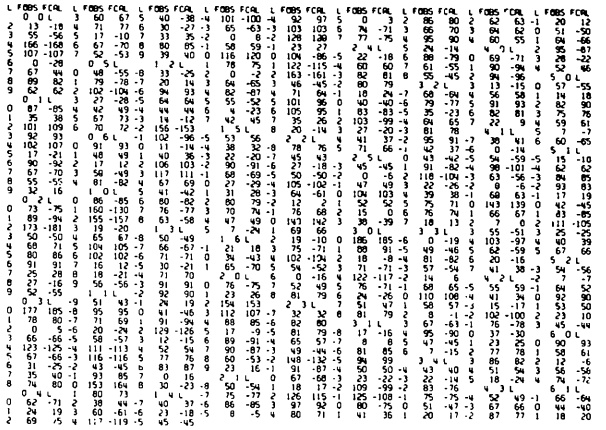
Fig. 2. One unit cell of $KNaThF_6$.

Table 2. *Interatomic distances for* $KNaThF_6$

3[Th–F(1)]*	2·28 (2) Å	3[K–F(1)]	2·68 (2) Å
3[Th–F(2)]	2·40 (2)	3[K–F(1)]	2·79 (2)
3[Th–F(2)]	2·42 (2)	3[K–F(2)]	2·87 (2)
3[Na–F(1)]	2·26 (2)	3[Na–F(2)]	2·54 (2)
F(1)–F(2)	2·72 (2)	2[F(1)–F(1)]	2·86 (3)
F(1)–F(2)	2·84 (2)	F(1)–F(2)	2·97 (2)

* The coefficient in front of the bond denotes the number of equal bonds in the structure.

Table 3. *Observed and calculated factors for* $KNaThF_6$



Cromer & Waber (1965) and the values of $\Delta f' = -3{\cdot}95$ and $\Delta f'' = 10{\cdot}29$ electrons were used for the anomalous dispersion of Ag $K\alpha$ radiation by thorium (Cromer, 1965). The standard deviation of an observation of unit weight, $[\sum w(F_o - F_c)^2/(n_o - n_v)]^{1/2} = 1{\cdot}377$ where n_o is the number of reflections and n_v the number of parameters. The agreement index $R = \sum ||F_o^2| - |F_c^2|| / \sum |F_o^2|$ is 0·095 for 365 independent reflections.

The refined parameters are listed in Table 1, the interatomic distances in Table 2 and the structure factor data in Table 3. Fig. 1 is a stereoscopic pair of drawings showing one asymmetrical unit of $KNaThF_6$, and Fig. 2 shows a complete unit cell. The Na polyhedron is an irregular octahedron which appears to be squashed along one of the triad axes. The Th and K polyhedra are the trigonal prisms with pyramids on each prism face typical of 9 coordination.

The Th and K polyhedra alternate with one another along the *c* axis and share the bases of the trigonal prisms. The K polyhedra share edges with K and Th polyhedra in adjacent columns within the unit cell. The Na octahedra tie the unit cells together perpendicular to the *c* axis by sharing edges with the Th and K polyhedra.

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References

BROWN, G. M. & LEVY, H. A. (1964). *J. Phys.* **25**, 497.

BRUNTON, G. D., INSLEY, H., MCVAY, T. N. & THOMA, R. E. (1965). USAEC Report ORNL-3761, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). USAEC Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

CROMER, D. T. (1965). *Acta Cryst.* **18**, 17.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104.

FRIEDMAN, H. A., HEBERT, G. M. & THOMA, R. E. (1963). USAEC Report ORNL-3373, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558.

Acta Cryst. (1970). B**26**, 1187

The crystal structure of *p*-sulfobenzenediazonium inner salt.

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The crystal structure of *p*-sulfobenzenediazonium inner salt, $C_6H_4N_2O_3S$, has been determined. Observed unit-cell parameters are $a = 8{\cdot}10$ (1), $b = 9{\cdot}94$ (1), $c = 9{\cdot}46$ (1) Å and $\beta = 97{\cdot}4$ (2)°. The space group is $P2_1/n$. Three-dimensional visual data were collected with Cu $K\alpha$ radiation. The trial structure was obtained by Patterson methods and refined to a final R of 13%.

The crystal structure of *p*-sulfobenzenediazonium inner salt, $^{\ominus}O_3S$–C_6H_4–$N_2^{\oplus}$, has been determined. Needle-like crystals of the compound about 0·2 to 0·3 mm in diameter and suitable for X-ray analysis were grown from an aqueous solution and mounted on a glass fibre. Weissenberg photographs (Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å) indicated the space group to be $P2_1/n$–C_{2h}^5 ($h0l$ absent when $h+l = 2n+1$; $0k0$ absent when $k = 2n+1$). Unit-cell dimensions are $a = 8{\cdot}10$ (1), $b = 9{\cdot}94$ (1), $c = 9{\cdot}46$ (1) Å and $\beta = 97{\cdot}4$ (2)°. The observed density, measured by flotation, is 1·69 g.cm^{-3}; the density calculated from the X-ray data is 1·63 g.cm^{-3} assuming four molecules per unit cell. Multiple-film equi-inclination Weissenberg photographs were taken about the *a* axis ($h = 0$–6). Intensities were measured visually. The Lorentz–polarization factor was applied and the data were correlated by using a series of oscillation photographs in the usual way. No corrections were made for extinction or absorption. A total of 521 reflections in the region of reciprocal space explored had detectable intensities.

Patterson projections and the sharpened Patterson–Harker section $P(u\frac{1}{2}w)$ were synthesized and served to establish trial positional parameters for the sulfur atom. A fourfold sharpened superposition function based on the sulfur atom trial parameters was constructed. Although ra-