puted the 'difference' Patterson function, with coefficients $(F_D^2 - F_H^2)$, where $|F_D|$ and $|F_H|$ are the respective neutron structure amplitudes, each on an absolute scale. The outcome is represented on the left-hand side of Fig. 1.

As these acid salts are almost identically isomorphous, as the single hydrogen atom lies at a centre of symmetry (I2/a, Z=4), and as the neutron-scattering lengths of D and H are +6.5 and -3.8 fermi, the map shows a complete image of the structure. The nine peaks shown are the nine highest, and only significant, independent peaks of the Patterson function. (The peak representing the hydrogen atom has a poor profile in the y direction, but this (origin) peak is sensitive to slight errors in the data.)

This exercise is a logical, and obvious, extension of a method of absolute structure determination developed in the 1930's (Beevers & Lipson, 1934; Robertson, 1936; Robertson & Woodward, 1940). Whether we refer to the heavy-atom method or to that of isomorphous replacement may be debated. We prefer to regard the 'difference atom', (D-H), as 'heavy', with a scattering power of +10.3 fermi.

A crystal belonging to the space group I2/a yields a vector pattern in I2/m; hence any image of the true structure is doubled by a mirror-plane. Fortunately all the atoms are free from overlap except for the one oxygen atom which, consequently, has a peak of nearly double height. So we easily recognize an acceptable molecule in a correct position in the cell. The mirror ambiguity would equally have compromised the absolute structure determination of platinum phthalocyanine $(P2_1/a \rightarrow P2/m)$ had it been possible, at the time, to apply the method three-dimensionally.



Fig. 1. On the left-hand side are sections from the three-dimensional 'difference' Patterson function, with positive contours at 4,6,8... in arbitrary units. On the right is the crystal structure established and refined by normal X-ray and neutron diffraction analysis. (The asterisk symbolizes a centre of inversion at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$.)

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Acta Cryst. (1971). B27, 2290

The crystal structure of K₇Th₆F₃₁.* By GEORGE BRUNTON, Reactor Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830, U.S.A.

(Received 26 April 1971)

The complex fluoride $K_7 Th_6 F_{31}$ is isostructural with $Na_7 Zr_6 F_{31}$. It is trigonal, $R\overline{3}$, with hexagonal axes $a_0 = 15 \cdot 293$ (2) and $c_0 = 10 \cdot 449$ (2) Å, Z = 3 and $\varrho_{cal} = 5 \cdot 307$ g.cm⁻³. The eight Th-F distances range from 2.24 (1) to 2.41 (1) Å. The K-F distances range from 2.63 (2) to 3.36 (2) Å.

The structure of $K_7 Th_6 F_{31}$ is identical to that of $Na_7 Zr_6 F_{31}$ (Burns, Ellison & Levy, 1968) and was determined to con-

* Research sponsored by the U.S. Atomic Energy Commission under contract with the Union Carbide Corporation. firm the supposition that all $7AF.6MF_4$ complexes are probably isostructural, (A=Na, K, Rb and NH₄, and M=most of the tetravalent lanthanides and actinides) (Thoma, 1962).

The reflection data were collected from an ellipsoidal

Table 1. Lattice and atomic parameters for K₇Th₆F₃₁

Space	group R3, $a_0 = 15.29$	93 (2), c ₀ =10·449 (2) A, $Z=3$, $\rho_{cal}=5\cdot3^{\circ}$	07 g.cm ⁻³ .
Standard errors in parenthe	eses, corresponding t	to the last significant	digit, are given by	the variance-covariance matrix.

	x	У	Z	$^*\beta_{11} imes 10^4$	$\beta_{22} \times 10^4$	$\beta_{33} \times 10^4$	$\beta_{12} = 104$	$\beta_{13} imes 10^4$	$\beta_{23} \times 10^4$
K(1)	0.0824 (4)	0.3088 (4)	0.4948 (5)	30 (3)	37 (3)	53 (4)	20 (2)	-4 (3)	1 (3)
K(2)	0	0	1/2	26 (4)	β_{11}	37 (8)	$(\frac{1}{2}\beta_{11})$	0	0
Th	0.18917 (4)	0.05122 (4)	0.17732 (6)	16(1)	14 (1)	24 (1)	7 (1)	1 (1)	1 (1)
F(1)	0.3548 (7)	0.1126 (7)	0.0938 (9)	1.4 (2)†					
F(2)	0.1834 (8)	0.0551 (8)	0.392 (1)	1.8 (2)					
F(3)	0.2747 (8)	0.3717 (8)	0.425(1)	1.7 (2)					
F(4)	0.2103 (9)	0.1602 (9)	0.004 (2)	2.8 (3)					
F(5)	0.2442 (9)	0.544 (1)	0.446 (2)	2.9 (3)					
F(6)	0	0	0.03 (2)	10 (6)					

* Coefficients in the temperature factor: exp $\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right]$. † Isotropic B.

Table 2. Interatomic distances for K₇Th₆F₃₁

Atom labels correspond to those of Burns et al. (1968).

		cons conserver		,	
Th - F(3)	2·24 (1) Å	ThF(2)	2·24 (2) Å	ThF(5)	2·32 (2) Å
Th - F(5)	2.33(2)	Th - F(4)	2.34 (2)	ThF(4)	2.37 (2)
Th - F(1)	2.38 (1)	Th - F(1)	2·41 (1)	K(1) - F(2)	2.63 (2)
K(1) - F(3)	2.66(2)	K(1) - F(3)	2.67 (2)	K(1)F(3)	2.70 (2)
K(1) - F(2)	2.72(2)	2[K(1)-F(1)]	2.78 (2)	K(1)F(5)	3.23 (2)
K(1) - F(4)	3.28 (2)	K(1) - F(4)	3.31 (2)	6[K(2)-F(2)]	2.74 (2)
6[K(2)-F(5)]	3.36 (2)	F(1) - F(1)	2.56 (2)	F(1) - F(3)	2.79 (2)
F(1) - F(2)	2.80(2)	2[F(1)-F(4)]	2.81 (2)	F(1) - F(4)	2.95 (2)
F(1) - F(5)	2.96 (2)	F(1) - F(3)	3.28 (2)	F(2) - F(3)	2.75 (2)
F(2) - F(5)	2.80 (2)	F(2) - F(1)	2.80 (2)	F(2) — F(5)	2.87 (2)
F(2) - F(3)	3.24 (2)	2[F(2)-F(2)]	3.37 (2)	F(3) — F(4)	2.84 (2)
F(3) - F(5)	2.90 (2)	F(4) - F(5)	2.80 (2)	F(4) - F(5)	2.91 (2)
2[F(4) - F(4)]	2.91 (2)	F(4) - F(6)	2.93 (3)	F(4) —F(6)	2.94 (4)
F(5) - F(6)	2.6 (2)	2[F(5)-F(5)]	2.91 (3)		

Table 3. Observed and calculated structure factors and extinction correction for K₇Th₆F₃₁

$ECOR = 10^3 \times extinction$ correction.

t ≠0	BS FCAL L	FOBS FCAL L	FORS FCAL LF	OBS FCAL L	FOOS STALL L	FOBS FCAL 1	FOBS FCAL L	FORS FCAL L	FORS FEAL L F	CBS FCAL	L FORS FEAL L	FORS *CAL L	FORS FCAL L	FORS FEAS L	COS FCAL L	FORS FCAL L	FORS FLAN I	FORS FEAL	L FORS PEA	L 1085 174	•
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crystal $0.26 \times 0.241 \times 0.26$ mm using unfiltered Ag Ka radiation. The experimental and processing details are identical to those previously described by Brunton (1971). Because of the identity of the K₇Th₆F₃₁ and Na₇Zr₆F₃₁ structures, the description of the structure by Burns *et al.* (1968) need not be repeated here. The lattice and atomic parameters are listed in Table 1, interatomic distances in Table 2, and the 1853 observed and calculated structure factors in Table 3. An isotropic extinction correction was applied to F_o , and the reflections with an extinction correction different from 1.0 are also listed in Table 3 (Coppens & Hamilton, 1970). Tables 1, 2 & 3.

The Th–F distances range from 2.24 (1) to 2.41 (1) Å and the K–F distances range from 2.63 (2) to 3.36 (2) Å. The

Th-F and K-F distances average respectively 0.21 and 0.30 Å longer than corresponding Zr-F and Na-F distances, and the corresponding F-F distances average 0.30 Å longer for K₇Th₆F₃₁. The discrepancy indices $R(F_o)$ and weighted $R(F_o)$ are 0.0660 and 0.1188 respectively for all reflections less than $\sigma(F_o^2)$.

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Acta Cryst. (1971). B27, 2292

Bond lengths and angles in the molecule of palladium n-propyl mercaptide. By N. R. KUNCHUR, Chemistry

Department, University of Tabriz, Tabriz, Iran

(Received 6 May 1971)

Revised values are given which supersede those in Acta Cryst. (1968) B24, 1623.

Bond lengths (Å)

The structure of palladium n-propyl mercaptide was solved by Kunchur (1968) and the values for the various bond lengths and angles within the molecule were reported. Owing to a fault in the program which was used in these calculations on a computer, errors were introduced in the values of bond lengths and angles reported. The author wishes to thank Dr R. O. Gould of the University of Edinburgh for pointing out this error. In Table 1 the corrected values of bond lengths and angles are given. The labelling scheme is the same as reported in the earlier work. However, a correction should be made in the labelling of two carbon atoms. The carbon atoms C(17) and C(18) in Table 1 of the previous work (Kunchur, 1968) should be read as C(18') and C(17') respectively.

Table 1. Dimensions of the molecule; corrected values

Bond lengt	hs (Å)	Angles (°)	
Pd(1)-S(2)	2.311	S(5) - Pd(1) - S(4)	82·2
Pd(1) - S(3)	2.332	S(5) - Pd(1) - S(3)	98·2
Pd(1) - S(4)	2.334	S(5) - Pd(1) - S(2)	177.8
Pd(1) - S(5)	2.336	S(4) - Pd(1) - S(3)	176.6
Pd(2)-S(1)	2.332	S(4) - Pd(1) - S(2)	97.6
Pd(2)S(4)	2.338	S(3) - Pd(1) - S(2)	81.9
Pd(2)S(5)	2.332	S(6) - Pd(2) - S(5)	97.1
Pd(2)-S(6)	2.318	S(6) - Pd(2) - S(4)	174.6
Pd(3)-S(1)	2.320	S(6) - Pd(2) - S(1)	81.8
Pd(3)-S(2)	2.326	S(5) - Pd(2) - S(4)	82.2
Pd(3)-S(3)	2.338	S(5) - Pd(2) - S(1)	177.7
Pd(3)-S(6)	2.327	S(4) - Pd(2) - S(1)	98.7
S(1) - C(1)	1.83	S(6) - Pd(3) - S(3)	174.3

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C(1)C(2)	1.52	S(6)Pd(3)-S(2)	97.5
C(2) - C(3)	1.48	S(6) - Pd(3) - S(1)	81.9
S(2) - C(4)	1.84	S(3) - Pd(3) - S(2)	81.9
C(4) - C(5)	1.62	S(2) - Pd(3) - S(1)	177.6
C(5) - C(6)	1.47	S(3) - Pd(3) - S(1)	98.5
S(3) - C(7)	1.82	C(1) - S(1) - Pd(3)	109.2
C(7) - C(8)	1.50	C(1) - S(1) - Pd(2)	111.1
C(8)—C(9)	1.54	C(4) - S(2) - Pd(1)	107.5
S(4) - C(10)	1.87	C(4) - S(2) - Pd(3)	109.4
C(10)-C(11)	1.52	C(7) - S(3) - Pd(3)	103.8
C(11)-C(12)	1.47	C(7) - S(3) - Pd(1)	104.5
S(5)C(13)	1.85	Pd(3)-S(1)-Pd(2)	85.1
C(13)-C(14)	1.45	Pd(3)-S(2)-Pd(1)	83.8
C(14) - C(15)	1.51	Pd(3)-S(3)-Pd(1)	83.5
S(6)—C(16)	1.86		
C(16)-C(17)	1.44		
C(17)-C(18)	1.55		
Pd(1)-Pd(3)	3.111		
Pd(1)-Pd(2)	3.088		
Pd(2)-Pd(3)	3.147		

Table 1 (cont.)

Angles (°)

The estimated standard deviations in the values of bond lengths and angles are not given here since they are not affected and are the same as reported previously.

Reference

KUNCHUR, N. R. (1968). Acta Cryst. B24, 1623.