Acta Cryst. (1970). B**26**, 1184

Crystallographic data for testosterone hydrate and anhydrate.

By A. L. THAKKAR, N. D. JONES, H. A. ROSE, L. G. TENSMEYER and N. A. HALL,* *Lilly Research Laboratories, Eli Lilly and Company, Indianapolis, Indiana* 46206, *U.S.A.*

(*Received* 15 *January* 1970)

Testosterone hydrate crystallizes in the space group $P2_12_12_1$ with four molecules in a unit cell having the dimensions $a=13{\cdot}63$, $b=15{\cdot}95$ and $c=7{\cdot}94$ Å. Anhydrous testosterone crystallizes in the space group $P2_1$ with four molecules in the unit cell. The proper cell dimensions are $a=14{\cdot}45$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=110{\cdot}5°$.

In a previous study on the solution behavior of testosterone in aqueous media, conversion of the anhydrate form to a hydrate was reported (Thakkar & Hall, 1969). Since testosterone is a natural hormone and exists in an aqueous environment, characterization of this form is important. We wish to report here the crystallographic parameters of the hydrate.

Small single-crystals were grown by a continuous fall method from saturated aqueous solution cooled from 33·0 to 29·5°C at 0·1°C per hour. Elemental analysis, Karl Fischer titration and thermogravimetric analysis showed this crystalline form to be the monohydrate.

From Weissenberg and precession photographs taken with Cu $K\alpha$ radiation the space group has been found to be $P2_12_12_1$ (systematic absences: $h00$, $0k0$, and $00l$ for h, k or l odd); there are four molecules in a unit-cell having the dimensions $a=13{\cdot}63$, $b=15{\cdot}95$ and $c=7{\cdot}94$ Å. The density measured by displacement is 1·181 g.cm^{-3}, which agrees well with the calculated density for $C_{19}H_{28}O_2.H_2O$ of 1·179 g.cm^{-3}.

For comparison we have measured the crystal parameters for anhydrous testosterone and have obtained values which differ from those reported by Bernal & Crowfoot (1936). The space group is $P2_1$ with four molecules in a unit-cell having the dimensions $a=14{\cdot}73$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=113{\cdot}3°$, which agree fairly well with the values given by Olhrt, Haner & Norton (1965). There is, however, an alternative cell with β closer to 90°. The dimensions for this proper cell are $a=14{\cdot}45$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=110{\cdot}5°$. These cells give a calculated density of 1·173 g.cm^{-3}, which is identical with the experimentally measured value given by Bernal & Crowfoot.

The indexed powder data for these two forms of testosterone will be submitted for inclusion in the *ASTM Powder Diffraction File*.

References

BERNAL, J. D. & CROWFOOT, D. (1936). *Z. Kristallogr.* **93**, 464.

OHRT, J. M., HANER, B. A. & NORTON, D. A. (1965). *Acta Cryst.* **19**, 479.

THAKKAR, A. L. & HALL, N. A. (1969). *J. Pharm. Sci.* **58**, 68.

* Present address: College of Pharmacy, University of Washington, Seattle, Washington 98105.

Acta Cryst. (1970). B**26**, 1184

Crystal data of $BaSrFe_4O_8$.

By S. MERIANI and G. SLOCCARI, *Istituto di Chimica Applicata dell'Università di Trieste, via Valerio* 2, *Trieste, Italy.*

(*Received* 20 *January* 1970)

The dimensions of the orthorhombic unit cell of $BaSrFe_4O_8$, which contains two formula units, are $a=5{\cdot}516$, $b=8{\cdot}265$, $c=9{\cdot}188$ Å. The sapce group is *Pnna*.

A previous report on the phase equilibrium diagram, $BaO–SrO–Fe_2O_3$, shows that a new stable compound, having the composition $BaSrFe_4O_8$, may occur as a single phase above 1100 ± 10°C (Batti, 1962). It undergoes thermal transformation at about 1200°C and melts incongruently at 1240 ± 10°C. A further investigation by Barbariol & Batti (1968) established that this new phase forms a solid solution with the binary compound $BaFe_2O_4$, which is reported to be orthorhombic (Okazaki, Mori & Mitsuda, 1963; DoDinh & Bertaut, 1965). They display complete solubility above 1200°C whereas at lower temperatures a solid-solution gap of increasing width was reported.

Single crystals of $BaSrFe_4O_8$ were grown, by solid-state reaction, from a pressed pellet mixture of 1 $BaCO_3$: 1 $SrCO_3$: 2 Fe_2O_3 which was heated on a platinum strip in a resistance furnace to about 950°C. The sintered pellet was reground and refired to assure complete reaction. The microcrystalline specimen was brought to 1200°C and left in the furnace

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 $K\bar{\alpha}=1\cdot5418$, Cu $K\alpha_1=1\cdot5405$, Fe $K\bar{\alpha}=1\cdot9373$, Fe $K\alpha_1=1\cdot9360$ 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 $a=5\cdot516$, $b=8\cdot265$ and $c=9\cdot188$ Å ($\pm0\cdot001$ Å) at 25°C. Systematic extinctions occurred for reflexions $0kl$ with $k+l\neq2n$, $h0l$ with $h+l\neq2n$ and $hk0$ with $h\neq2n$; 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). **B26**, 1185

The crystal structure of $KNaThF_6$.*

By GEORGE BRUNTON, *Reactor Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee* 37830, *U.S.A.*

(*Received* 19 *November* 1969)

Crystals of $KNaThF_6$ are hexagonal $P\bar{3}$ with $a_0=6\cdot3073\,(2)$ and $c_0=7\cdot8907\,(2)$ 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 $N_O=1\cdot454$ and $N_E=1\cdot448$.

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.