Acta Cryst. (1964). **17**, 1480

The crystal structure of β iron(III) trifluoride trihydrate, β-$FeF_3.3H_2O$,

By Gunter Teufer, *Engineering Materials Laboratory, Engineering Department, E. I. du Pont de Nemours & Co., Inc., Wilmington, Delaware, U.S.A.*

(*Received* 19 *May* 1964)

Maak, Eckerlin & Rabenau (1961) have reported a number of compounds which crystallize with the β-$AlF_3.3H_2O$ structure. Of these, the iron salt can be obtained in single crystals suitable for a structure determination. Crystals of β-$FeF_3.3H_2O$ were grown at 50 °C from a solution of freshly precipitated iron(III) oxide hydrate in dilute hydrofluoric acid according to the preparation of form I of $FeF_3.3H_2O$ given by Nielsen (1940). The crystals are pink and grow in square prisms or cubes.

Crystallographic data were derived from Weissenberg and precession camera photographs and are summarized in Table 1. The Laue symmetry is $4/m$, and $hk0$ reflections with $h+k+2n$ are absent. The lattice dimensions are in good agreement with the data reported by Maak *et al.* (1961).

Table 1. *Crystallographic data for* β-$FeF_3.3H_2O$

Space group $P4/n$

a = 7·846 Å (λ, Mo $K\alpha$ = 0·7107 Å)
c = 3·877 Å
Z = 2
D_x = 2·321 g.cm^{-3}
D_m (flotation) = 2·26 (Nielsen, 1940)

Approximate atomic coordinates were derived from Patterson projections along the a and c axes and from coordination considerations. Atomic scattering factors for Fe^{3+}, F^-, and O used for structure factor calculations were taken from *International Tables for X-ray Crystallography* (1962). A mean scattering factor for F^- and O was applied to the general positions xyz where fluorine atoms and water molecules are assumed to be statistically distributed. As a first approximation, isotropic temperature factors were assigned to all atoms in crystallographically different positions. Refinement of the atomic parameters was made through Fourier synthesis and least-squares analysis based on ($hk0$) and ($h0l$) data; the final R value for 123 reflections is 0·073. The observed and calculated structure factors can be obtained from the author. The final least-squares parameters are listed in Table 2.

A projection of the structure along the c axis is shown in Fig. 1 and the interatomic distances are listed in Table 3.

In β-$FeF_3.3H_2O$ each iron atom is surrounded by six ligands in the form of a nearly regular octahedron. Adjacent octahedra share apices in the direction of the c axis and fluorine atoms are assumed to form these bridges between iron atoms. The four other ligands of each octahedron are two fluorine atoms and two water molecules which occupy statistically the four positions of a square around the iron atom. Only the two water molecules in position b represent water of hydration; they are tetrahedrally surrounded by 4 F, H_2O ligands of four neighboring octahedra.

Table 3. *Interatomic distances in* β-$FeF_3.3H_2O$

Fe–(1) F	1·951 Å	H_2O, F–(2) H_2O, F	2·739 Å
(1) F	1·926	(1) F	2·733
(4) H_2O, F	1·937	(1) F	2·748
		(1) H_2O, F	2·58
H_2O–(4) H_2O, F	2·696	(1) H_2O	2·696

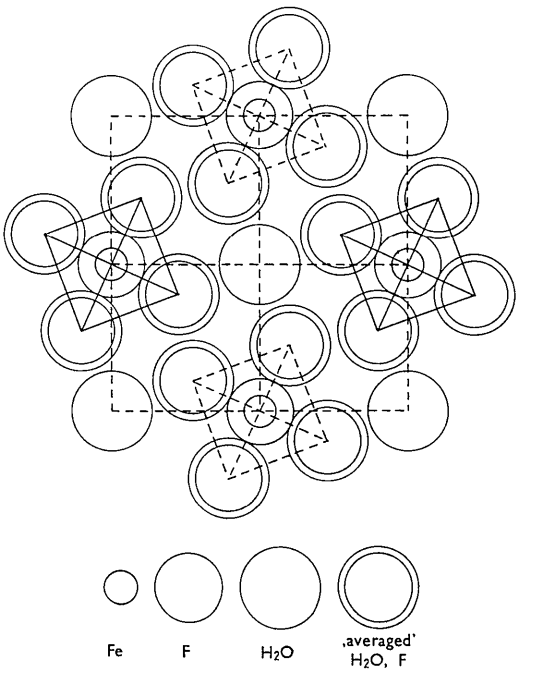

Fig. 1. The structure of β-$FeF_3.3H_2O$ viewed along the c axis. Double circles represent sites of randomly distributed fluorine atoms and water molecules.

The author is grateful to Miss P. M. G. Wong for assistance in computing.

References

International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.

Maak, I., Eckerlin, P. & Rabenau, A. (1961). *Naturwissenschaften*, **48**, 218.

Nielsen, A. H. (1940). *Z. anorg. Chem.* **244**, 85.

Table 2. *Least-squares parameters for* β-$FeF_3.3H_2O$ *with origin at* $\bar{4}$ *in space group* $P4n$

Atom	Set	x	y	z	B
Fe	2(*c*)	0	½	0·1453 ± 0·0012	1·29 + 0·07 Å^2
F	2(*c*)	0	½	0·6421 ± 0·0042	1·34 + 0·26
H_2O	2(*b*)	0	0	½	1·75 + 0·25
H_2O, F	8(*g*)	0·2754 ± 0·0009	0·1025 ± 0·0009	0·1394 ± 0·0021	2·08 ± 0·12