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Bond-valence summation for borates: errata. By GABRIELLE DONNAY and J. D. H. DONNAY, Department of Geological Sciences, McGill University, P.O. Box 6070, Station A, Montreal, Quebec, Canada H3C 3G1

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In Table 1 of Donnay & Donnay [Acta Cryst. (1973), B29, 1417–1425], (O–H) should read (B–O); on p. 1424, $L \leq L$ should read $L \geq L$.

The following errata should be corrected in Donnay & Donnay (1973): p. 1418, Table 1: in the title and first column heading, instead of (O–H), read (B–O); p. 1424, 1st column, line 14 from bottom of page: instead of $L \le \overline{L}$ read $L \ge \overline{L}$.

Reference

DONNAY, G. & DONNAY, J. D. H. (1973). Acta Cryst. B29, 1417–1425.

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New compounds of Yb_3Fe_4O_{10} and Yb_4Fe_5O_{13}. By NOBORU KIMIZUKA, KATSUO KATO, ISAMU SHINDO and ISAO KAWADA, National Institute for Researches in Inorganic Materials, Sakura-mura, Niihari-gun, Ibaraki 300-31, Japan and TAKASHI KATSURA, Faculty of Science, Tokyo Institute of Technology, Ookayama, Meguroku, Tokyo 152, Japan

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New compounds of $Yb_3Fe_4O_{10}$ [(YbFeO₃)₃FeO] and $Yb_4Fe_5O_{13}$ [(YbFeO₃)₄FeO] have been found in the YbFeO₃-FeO pseudo-binary system and their crystallographic data determined. The structural relations of $Yb_3Fe_4O_{10}$ and $Yb_4Fe_5O_{13}$ to the previously determined YbFe₂O₄ and Yb₂Fe₃O₇ are presented.

Introduction

In the pseudo-binary system of $(A/B)FeO_3$ -FeO, the $(AFeO_3)FeO$ and the $(BFeO_3)_2FeO$ types of compound (A = Y, Ho, Er, Tm, Yb and Lu; B = Yb and Lu) have so far been synthesized under controlled oxygen partial pressures at 1200°C (Kimizuka, Takenaka, Sasada & Katsura, 1975*a*, *b*; Tannières, Evrard & Aubry, 1974) and crystal structure analyses have been performed on YbFe₂O₄ (Kato, Kawada, Kimizuka & Katsura, 1975) and Yb₂Fe₃O₇ (Kato, Kawada, Kimizuka, Shindo & Katsura, 1976). The crystal structure of (Yb,Eu)Fe₂O₄ has been independently determined by Malaman, Evrard, Tannières & Aubry (1975). The present paper reports the existence of the new compounds of Yb₃Fe₄O₁₀ and Yb₄Fe₅O₁₃ along with their crystallographic data and their structural relations to the compounds of YbFe₂O₄ and Yb₂Fe₃O₇.

Sample preparation

99.99 % purity grade Yb₂O₃ (Shinetsu Chemical Co.) and guaranteed reagent grade Fe₂O₃ (3/4 in mole ratio) were thoroughly mixed in an agate mortar under ethyl alcohol and heated at 1200 °C in air for two days. The mixture was then placed in a crucible ($10 \times 10 \text{ mm } \varphi$) of 20 % Rh–Pt alloy and heated at 1650 ± 1 °C in a CO₂ gas (99.99 %) atmosphere using a vertical Mo-wire wound furnace. Reaction time was 30 min and the sample was rapidly cooled to ice temperature.

X-ray powder diffraction by a counter diffractometer (Mn-filtered Fe $K\alpha$ radiation and Si standard) revealed that

the polycrystalline material thus obtained was a mixture of $Yb_2Fe_3O_7$, $Yb_3Fe_1O_{10}$ and $Yb_4Fe_5O_{13}$. The crystals of the former two phases were grown large enough to be examined by single-crystal methods.

Table 1. Spacings and relative intensities of Yb₃Fe₄O₁₀

	h	k l	d_{obs} (Å)	d_{calc} (Å)	I_{obs}
I	0	0 3	19.8	20.3	1
2	0	06	10.07	10.13	15
3	0	09	6.726	6.754	80
4	0	0 12	5.055	5.066	80
5	0	0 15	4.054	4.053	30
6	0	0 18	3.359	3.377	5
7	0	0 21	2.8929	2.8948	100
8	1	0 10	2.7013	2.7063	20
9	1	0 13]	2.5277	2.5383	10
10	0	0 24	2.3311	2.5329	10
11	0	0 27	2.2548	2.2515	2
12	ī	0 20	2.1411	2.1432	3
13	1	0 22	2 ·0419	2.0394	5
14	1	0 25	1.8933	1.8945	3
15	ī	0 26	1.8474	1.8493	10
16	1	1 0	1.7452	1.7450	20
17	1	0 31	1.6430	1.6451	3
18	ī	0 32	1.6086	1.6084	3
19	1	0 34	1.5407	1.5388	3
20	$\overline{2}$	0 1	1.5110	1.5107	5
21	1	1 21	1.4963	1.4955	10
22	$\overline{2}$	0 10	1.4659	1.4666	8
23	0	0 42	1.4467	1.4474	30
24	ī	0 38	1.4141	1.4139	3

Table	2.	Sp	acings	and	relative	intensities	of	Yb ₄ Fe ₅ O ₁₃
		$\sim r$					-,	

	h	k	l	d_{obs} (Å)	$d_{\rm calc}({\rm \AA})$	I_{obs}
1	0	0	4	13.1	13.2	3
2	0	0	6	8.72	8.83	5
3	0	0	8	6.55	6.63	20
4	0	0	10	5.25	5.30	50
5	0	0	12	4.386	4.420	3
6	0	0	14	3.740	3.788	5
7	0	0	16	3.311	3.315	3
8	0	0	18	2.962	2.946	50
9	0	0	20	2.672	2.652	100
10	1	0	12	2.493	2.501	5
11	0	0	22	2.3965	2.4107	4
12	0	0	24	2.2004	2.2098	5
13	1	0	22	1.8864	1.8874	3
14	1	0	23	1.8371	1.8358	5
15	0	0	30	1.7679	1.7678	10
16	1	0	26	1.6951	1.6928	10
17	1	0	27	1.6509	1.6488	5
18	1	1	22	1.4174	1.4171	12

Crystallographic data

 $Yb_3Fe_4O_{10}$

The spacings and relative intensities of the powder X-ray diffraction of $Yb_3Fe_4O_{10}$ (Table 1) were obtained by using Weissenberg photographs to identify the reflexions of this

Yb₄Fe₅O₁₃

Referring to the possible structures of the series $(YbFeO_3)_nFeO: n=1, 2, 3 \text{ and } 4$, derived by applying the same construction rules mentioned above, we could estimate their *c* lattice constants (Table 3). The *c* value of the compound $(YbFeO_3)_4FeO$ made it possible to assign indices to every powder diffraction line (Table 2) obtained by subtracting out the diffraction lines of $Yb_2Fe_3O_7$ and $Yb_3Fe_4O_{10}$ phases from the powder pattern of the mixture.

The de Wolff M_{17} value was estimated to be 8 (sin² $\theta_{17} = 0.348$, $\overline{\epsilon} = 0.00087$ and $N_{17} = 51$).

The possible structure of this compound has the symmetry $P6_3/mmc$ and the following layer sequence:

$$\frac{1}{c \simeq 53 \text{ Å}}$$

The crystallographic data and some structural information for $(YbFeO_3)_nFeO$ (n=1, 2, 3 and 4) and $YbFeO_3$ ($n=\infty$) are summarized in Table 3. The details of the crystal structure determinations of $Yb_3Fe_4O_{10}$ and $Yb_4Fe_5O_{13}$ will be published elsewhere (Kato & Kawada, 1976).

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Table 3. Crystal data

Compound	n	a (Å)	c (Å)	Z	Space group	c/p	p *	u†	$v\ddagger$	w§
YbFe ₂ O₄	1	3.455 ± 0.001	25.05 ± 0.01	3	R3m	2.09	12	3	0	3
Yb ₂ Fe ₃ O ₇	2	3.476 ± 0.001	28.43 ± 0.01	2	$P6_3/mmc$	2 ·03	14	4	2	2
Yb ₃ Fe ₄ O ₁₀	3	3.490 ± 0.001	60·79 ± 0·02	3	R3m	2·02 +	30	9	6	3
Yb ₄ Fe ₅ O ₁₃	4	3.503 ± 0.002	53.03 ± 0.02	2	$P6_3/mmc$	2.04	26	8	6	2
YbFeO ₃	∞	$a = 5 \cdot 231 \pm 0 \cdot 001$		4	Pbnm					
		$b = 5.562 \pm 0.001$								
		c = 7.569 + 0.001								

* Number of oxygen layers. † Number of YbO_{1.5} layers. ‡ Number of FeO_{1.5} layers. § Number of Fe₂O_{2.5} layers.

compound in the powder pattern of the mixture. The de Wolff M_{20} value, one of the figures of merit for an indexed powder diffraction pattern (Lipson & Steeple, 1970; de Wolff, 1968) was 25 (sin² $\theta_{20} = 0.438$, $\bar{\varepsilon} = 0.00040$, and $N_{20} = 44$).

According to the construction rules for the YbFe₂O₄ and Yb₂Fe₃O₇ structures (Kato *et al.*, 1975, 1976), a possible structure in $R\overline{3}m$ with the YbO_{1.5} (U), FeO_{1.5} (V) and Fe₂O_{2.5} (W) layers stacked in the following sequence is proposed for Yb₃Fe₄O₁₀:

$$\cdots UVUVUWUVUVUWUVUVUWUVUVU...$$

$$c \simeq 61 \text{ Å}$$

The systematic absences $-h+k+l \neq 3n$ observed on the Weissenberg photographs satisfy the extinction rule of the space group $R\overline{3}m$.

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