Many minerals such as siderite (= chalybite) and ferriferrous chlorites give rise to ferrous oxide on heating. At high temperatures this will be immediately oxidized to hematite if free access to air is permitted. The case of siderite requires special consideration as it is a constituent of coals and the 2·79 Å line may well be due to residual siderite. On heating siderite in vacuum at 550° C, both FeO and Fe_3O_4 have been reported to form (Bernal, Dasgupta, and Mackay, 1959), the latter from FeO itself by disproportation. The same process may give rise to hematite and wüstite with differing cell size.

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Geological Survey of India,
29 Chowringhee,
Calcutta K. C. CHANDY

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Thermal transformation of β-ferric oxyhydroxide

AKAGANÉITE, β-FeOOH, has been shown to be tetragonal[1],[2] unlike the other two monohydrates of Fe_2O_3, goethite and lepidocrocite, which are orthorhombic. The author reported[3] a natural occurrence of β-FeOOH and in the course of an investigation on artificial preparations, it was found that it becomes orthorhombic on heating before the final transformation to hematite, which is the final product of heating all the three polymorphic forms.

β-FeOOH prepared by hydrolysing ferric chloride solution at 80° C, repeated washing, and drying at 90° C, was heated to different temperatures and durations. Changes in the X-ray patterns recorded with a 57·3 mm diameter powder camera using Fe-$K\alpha$ radiation were observed.

For accurate spacing measurements, the sample was mixed with high purity aluminium powder and a 114·6 mm diameter camera was used.

TABLE I. X-ray diffraction spacings observed in β-FeOOH before and after heating

1				2				
d	*I*	*hkl*		*d*	*I*	*hkl*	$1/d^2$ obs.	$1/d^2$ calc.
7·41 Å	vs	110		5·4	vvw, i	200	0·0343	0·0349
5·25	m	200	(5·16)	4·9	vvw, i	020	0·0416	0·0416
3·71	vw	220		3·34	w	310	0·0896	0·0890
3·33	vs	310	(3·27)	3·14	m, d	130	0·1014	0·1024
2·63	m	400		2·68	m	400	0·1392	0·1397
2·55	s	211		2·50	vs, b, d	211	0·1600	0·1592
2·48	vw	330		2·28	m	301	0·1924	0·1924
2·35	w	420		2·19	m	031	0·2085	0·2075
2·29	m	301	(2·26)	1·948	w	411	0·2635	0·2640
2·099	w	321		1·858	w	141	0·2897	0·2891
2·061	w	510		1·634	s, b, d	{521 / 060}	0·3745	{0·3738 / 0·3748}
1·952	m	411	(1·92)					
1·859	vw	440		1·482	m, sh	002	0·4553	0·4552
1·753	m	600		1·452	m, sh	112	0·4743	0·4743
1·729	vw	501, 431						
1·642	s	521						
1·516	m	002						
1·503	vw	611						
1·488	w	112, 710						
1·458	vw	640						
1·446	s	541						

1. β-FeOOH prepared by the hydrolysis of ferric chloride solution. The figures in brackets are the corresponding spacings after heating the specimen for 20 hours at 200° C. Tetragonal, *a* 10·52 Å, *c* 3·033 Å.

2. β-FeOOH heated successively for 20 hour periods at 140, 200, and 220° C, and then for 8 hour periods at 280 and 320° C. Orthorhombic *a* 10·70 Å, *b* 9·80 Å, *c* 2·964 Å.

b, broad; d, diffuse; sh, sharp; i, ill-defined.

Little change was noticeable in patterns up to about 150° C. On heating, the diffraction lines at first showed a shift to higher angles indicating a contraction of the tetragonal cell (table I and fig. 1). Significant changes took place from about 220° C; the lines 411, 301, 310, and 200 were found to broaden out and finally split up to form pairs of lines. The pair formed by the 200 line was vanishingly faint but the others were quite evident at 320° C. Column 2 of table I shows that the new structure is orthorhombic with *a* 10·70 Å, *b* 9·80 Å, *c* 2·964 Å.

β-FeOOH has the α-MnO_2 structure,[2, 4] with *a* 10·48 Å, *c* 3·023 Å. This structure contains channels parallel to the *c*-axis where Cl'/H_2O molecules can be statistically accommodated. A loss of the contents of the

channels on heating will account for the contraction of the tetragonal cell. Beginning at about 220° C, an expansion along a in one direction and a contraction in the perpendicular direction changes the cell to an

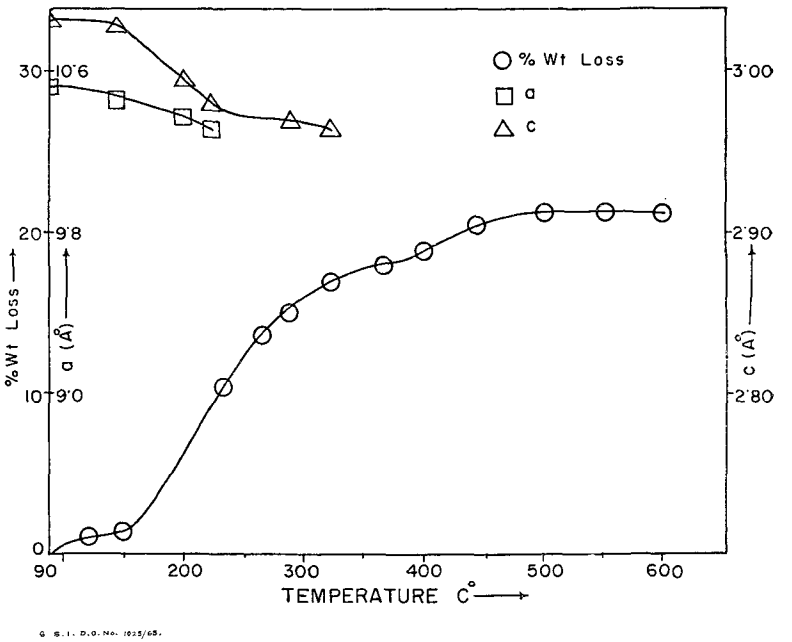

FIG. 1. Weight-loss curve and variation of the cell dimensions of β-FeOOH on heating.

orthorhombic one. The new structure still appears to contain OH and some Cl' and the step in the weight loss curve at about 390° C indicates the final transformation to hematite.

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Mineral Physics section,
Petrology Division,
Geological Survey of India,
29 Chowringhee,
Calcutta

K. C. CHANDY

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An occurrence of kyanite pseudomorphs after andalusite from Amb state, West Pakistan

THE Amb State area makes part of the western limb of a regional syntaxial arc of the north-western Himalayas. It consists of pelitic and psammitic schists and quartzites, intruded by granitic gneiss, while the whole area is cut sporadically by doleritic minor bodies. The highest grade of metamorphism reached by the schists is marked by almandine garnet while andalusite and cordierite-bearing hornfelses are developed at the granite contact. Nothing has yet been published on this area although a preliminary account on the adjoining Mansehra area has been published (Shams, 1961).

Recently, during field investigation of the aureole rocks near Choian (grid 896394 1″ topographic sheet No. 43B/15, Survey of Pakistan), certain hornfelses were found in which the andalusite had suffered transformation into kyanite in such a manner that the original shape and twinning of the andalusite are remarkably well preserved. These rocks are heavy, tough and dark coloured and, in addition to pseudomorphs, consist of quartz, muscovite, biotite, garnet, and tourmaline etc. The pseudomorphs lie preferably within weak foliation planes of the rock and show rough parallelism of their longer dimensions, although all orientations are present (fig. 1). Their size varies from very tiny to as big as $2'' \times \frac{3}{4}'' \times \frac{3}{4}''$ and rarely even bigger individuals are present. Their surface is generally coated with a brownish micaceous material while the freshly broken surfaces show the typical bluish colour of kyanite. Inside the pseudomorphs the kyanite blades are arranged in completely haphazard manner, minor amounts of quartz and muscovite are also present, the latter being at least partly of alteration origin. The kyanite gave: α 1·713, β 1·720, γ 1·725 (all $\pm$0·002, measured in filtered white light), $2V_\alpha = \sim 82°$, sp. gr. = 3·43.

The shape and the interpenetration twinning of the andalusite is so well preserved (fig. 2) that shearing stress, in the sense of Harker