

Heat treatment of chevkinite and perrierite.

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Summary. Standard heat treatments of 700° C for 3 hours, 1000° C for 1 hour, and 1300° C for 1 hour, both in a nitrogen atmosphere and in air, were applied to several samples of chevkinite and perrierite, and X-ray powder photographs were taken of the unheated and the heated materials.

Metamict chevkinites from the Urals, USSR, and from Ifasina, Madagascar, give the chevkinite phase when heated in nitrogen at 1000° C for 1 hour, but when heated in air at 1300° C for 1 hour give the perrierite phase. Perrierites only give the perrierite phase when heated in air or in nitrogen. A criterion for the identification of metamict samples of these two minerals was deduced from these experiments.

DURING the last years chevkinite and perrierite have been studied by several authors, particularly by Kauffman and Jaffe (1946) and Jaffe, Evans, and Chapman (1956) in the U.S.A.; Bonatti and Gottardi (1950, 1954) and Bonatti (1959) in Italy; Takubo and Nishimura (1953) in Japan; and Makarochkin, Gonibesova, and Makarochkina (1959) in the USSR. Although very detailed work has been carried out by these authors, some discrepancies and controversy arise from their results.

This paper is concerned with a study of the thermal behaviour of several samples of chevkinite and perrierite under different conditions of heat treatment. This work leads to a criterion for the identification of metamict chevkinites and perrierites, and explains the discrepancies observed in the work of previous authors.

Standard heat treatments (Lima-de-Faria, 1956, 1958) of 700° C for 3 hours, 1000° C for 1 hour, and 1300° C for 1 hour, both in an inert atmosphere (nitrogen) and in air, were applied to five samples of chevkinite and six samples of perrierite from different localities.¹ X-ray powder photographs were taken of the unheated and the heated materials. The results are shown in tables Ia, Ib, and II.

In these tables C indicates the chevkinite phase as determined from the unheated partially metamict material from New Hampshire, U.S.A..

¹ During the standard heat treatments the minerals were used as powder when heated in air, and as fragments when heated in nitrogen.

or from Arizona, U.S.A.; P indicates the perrierite phase as determined from the unheated perrierite from Nettuno, Italy (the type locality).

It can be seen from table Ia that the two samples of chevkinite from the Urals, USSR (the type locality), were completely metamict. These

TABLE I. Thermal behaviour of chevkinite and perrierite, as shown by X-ray powder photographs. C, chevkinite; C', a chevkinite-like phase with some slight differences and a few extra lines; P, perrierite; X, unidentified phase; cub., a cubic phase with a 5.44 Å (CeO₂); M, metamict (no diffraction pattern obtained); tr., a very weak pattern, indicating only a small amount of the phase.

B.M., British Museum; USNM, United States National Museum.

Locality and number.	Un-heated	700° C 3 hrs.		1000° C 1 hr.		1300° C 1 hr.		
		in N ₂	in air	in N ₂	in air	in N ₂	in air	
<i>a. Chevkinites.</i>								
Ilmen Mts., Urals ...	M	M	M	C	P (tr.) + cub.	C'	P	
B.M. 35265								
Urals								
B.M. 39244 ...	M	M	M	C	P (tr.) + cub.	C'	P	
Ifasina, Ambatofinandrahana								
Madagascar ...	M	C	M	C	P (tr.) + cub.	C	P + cub.	
Devil's Slide, New Hampshire, U.S.A. ...	C	C	C	C	C	X	C*	
Mohave Co., Arizona, U.S.A. ...	C	C	C	C	C	C	C*	
USNM 105344								
<i>b. Perrierites.</i>								
Near Bangalore, India ...	M	P	M	P	P + cub.	P	P	
USNM 106630								
Kanjamalai, near Salem, Madras, India ...	P (tr.)	M	M	P	P	P	P	
B.M. 34167								
Kobe, Kyoto prefecture, Japan ...	P	P	P	P	P	P	P	
Bingre, Tete, Mozambique	P	P	P	P	P	P	P	
Nettuno, Rome, Italy ...	P	P	P	P	P	P	P	
Bedford Co., Virginia, U.S.A. ...	P (tr.)	P	P	P	P	P	P	
B.M. 86189								

* Perrierite phase is formed on recrystallization after melting at 1350° C for 1 hour in air.

samples do not give any diffraction pattern when examined after heating at 700° C for 3 hours either in nitrogen or in air. When heated in nitrogen at 1000° C for 1 hour they show the same phase as that observed from the unheated American samples and this is accepted as chevkinite (see also table II). When heated in air at 1000° C for 1 hour they show a very small amount of the perrierite phase, and a face-centred cubic phase (a 5.44 Å), which can be ascribed to CeO₂, as already pointed out by Bonatti (1959) for a chevkinite from Madagascar.

At a higher temperature, 1300° C for 1 hour, these two samples from the Urals give in a nitrogen atmosphere a chevkinite-like phase with some slight differences and a few extra lines; this is marked C' in table Ia, and the *d* spacings are given in table II; heated in air at 1300° C for 1 hour they develop the perrierite phase (tables Ia and II).

These experiments form a link between the chevkinite phase and the perrierite phase, and suggest that perrierite is probably an oxidized form of chevkinite.¹ They also confirm the identity of the American and the Russian chevkinites; this is also supported by X-ray data given by Makarochkin, Gonibesova, and Makarochkina (1959) for an unheated partially metamict chevkinite from the Urals, USSR, which agrees well with the data determined from the American samples.

Metamict chevkinite from Ifasina, Madagascar, shows a similar thermal behaviour to the samples from the Urals. The two partially metamict chevkinites from New Hampshire and from Arizona, U.S.A., do not show the formation of the perrierite phase, even when heated in air at 1300° C for 1 hour, when they start to melt. However, at a higher temperature of 1350° C for 1½ hours in air, when melted completely, they develop the perrierite phase after slow cooling.

The chevkinite from New Hampshire, when heated in nitrogen at 1300° C for 1 hour, gives a badly defined X-ray pattern, which although it has some similarity with the perrierite phase, is significantly different from it. This unidentified phase has been labelled X (see tables Ia and II).

Table Ib shows the thermal behaviour of six samples of perrierite. With the exceptions of the perrierite from Nettuno, Italy, and of that from Mozambique, which was unclassified, all the other samples were originally classified as chevkinite. Direct comparison of the X-ray powder photographs of the unheated materials showed, however, that four of them give the perrierite diffraction pattern, and should consequently be called perrierites. Bonatti (1959) has already shown that the so-called chevkinite from Japan is in fact perrierite. The completely metamict sample from Bangalore, India (USNM 106630), shows a thermal behaviour very similar to the other perrierites examined, and is therefore considered here as a perrierite.

None of these perrierite samples when heated either in air or in

¹ Oxidation is carried almost to completeness when the material is heated as powder in air. If it is heated as fragments, the oxidation process is confined to the surface. When heated at 1000° C for 1 hour in air, fragments of the mineral from the Urals and from Madagascar show mainly the chevkinite phase. This is in agreement with the results obtained by Neumann, Sverdrup, and Saebo (1957).

nitrogen show the chevkinite phase; they always show a well-defined perrierite phase. Only in one heat treatment of one specimen (Bangalore, 1000° C 1 hour in air) does the cubic CeO_2 phase appear together with the perrierite phase.

On the basis of these experiments a criterion for the identification of metamict chevkinites and perrierites can be deduced: samples that after heating in air or in an inert atmosphere show only the perrierite phase should be called perrierites; samples showing the chevkinite phase when heated in an inert atmosphere should be called chevkinites. The heat treatment of 1000° C for 1 hour in an inert atmosphere would be, in this particular case, the best heat treatment for identification purposes.

The chevkinite sample from Ifasina, Madagascar, shows at 1000° C for 1 hour in air the cubic phase CeO_2 , with a few of the strongest lines of the perrierite phase; this is in agreement with the observation of Bonatti (1959) on a sample from Madagascar heated in air.

It has been shown here that the sample from Bedford Co., Virginia, U.S.A. (B.M. 86189), is a perrierite; Jaffe, Evans, and Chapman (1959) have also reported a chevkinite from this same region, and it should therefore be accepted that both minerals occur in Virginia.

The specimen of perrierite (B.M. 34167) from Kanjamalai, Madras, India, is a portion of the original 'Mineral de Coromandel' of Beudant; the change in synonymy should be noted.

Qualitative X-ray fluorescence analyses of these chevkinite and perrierite samples were made when sufficient material was available. In all cases they show the main constituents of these silicates to be Ti, Fe, and the cerium-group rare-earth elements. Variations of the minor constituents Th, Y, Zr, and Sr were observed, but no distinction between chevkinites and perrierites could be established on this basis. The complete chemical analyses already published for many samples from the same localities as those used in this work do not give any further clue to this problem, and the Fe_2O_3 and FeO contents do not enable the oxidation process to be traced back to the change in valency of the iron. Further chemical and structural studies must be carried out in the future to explain the difference in thermal behaviour of chevkinites and perrierites.

These experiments emphasize the need for complete description of the conditions under which the metamict samples are heated; and in the comparison of metamict minerals, rather than using only the X-ray powder pattern obtained after one heat treatment, it is often preferable to compare them from their thermal behaviour over a range of different and standard heat treatments, both in an inert atmosphere and in air.

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