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## Iwakiite, $Mn^{2+}(Fe^{3+}, Mn^{3+})_2O_4$ , a new tetragonal spinelloid mineral from the Gozaisho mine, Fukushima Prefecture, Japan

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### ABSTRACT

Iwakiite,  $Mn^{2+}(Fe^{3+}, Mn^{3+})_2O_4$ , is tetragonal, space group  $P4_2/nnm$ ,  $a$  8.509,  
 $c$  8.534Å,  $Z=8$ , and is found in rhodonite-quartz ore from the regionally  
metamorphosed manganese ore deposit of the Gozaisho mine, Fukushima Pre-  
fecture, Japan.

Wet chemical analysis by K.N. gave MnO 30.34, MgO 0.61,  $Mn_2O_3$  22.70,  
 $Fe_2O_3$  44.42,  $Al_2O_3$  0.32,  $SiO_2$  0.47,  $TiO_2$  0.30, SrO 0.04,  $Na_2O$  0.02,  $K_2O$  0.02,  
total 99.24%, corresponding to  $(Mn^{2+}_{0.982}Mg_{0.035})\Sigma=1.017(Fe^{3+}_{1.277}Mn^{3+}_{0.660}Si_{0.018}$   
 $Al_{0.014}Ti_{0.009})\Sigma=1.978O_4$  on the basis of  $O=4$ , or ideally  $Mn^{2+}(Fe^{3+}, Mn^{3+})_2O_4$ ,  
where  $Fe^{3+} : Mn^{3+} \approx 2:1$ . The strongest X-ray powder diffractions are 4.929  
(30) (111), 3.016 (40) (202, 220); 2.570 (100) (113, 311), 1.640 (30) (333), 1.509 (35)  
(404), 1.506 (40) (440). They are similar to but distinct from those of jacobsite  
due to splitting of (404)-(440).

It is opaque. Colour is greenish black, lustre metallic. Streak is black.  
No cleavage. Density ( $g/cm^3$ ) 4.85 (meas.), 4.89 (calc.). Strongly magnetic.  
It is optically anisotropic. The reflection colour in air is olive grey like  
jacobsite without discernible reflection pleochroism. Between crossed polars  
the colour changes from yellowish to brownish grey.

The name is for the city name Iwaki, in which the locality is situated.  
Type material is deposited in National Science Museum, Shinjuku, Tokyo,  
Japan.

### Introduction

In weakly to strongly metamorphosed manganese ores from bedded manganese ore deposits in Japan, the occurrence of jacobsonite is not uncommon (Sakurai, 1957), especially in high grade manganese ores comprising such manganese minerals as hausmannite, rhodochrosite and sonolite (Watanabe *et al.*, 1970). These ores are generally characterized by the absence of quartz and such silica saturated manganese silicates as rhodonite, pyroxmangite, bustamite, spessartine, manganese-bearing pyroxenes and amphiboles, except jacobsonite containing appreciable amount of magnetite molecule (Harada and Hirowatari, 1959).

Accordingly, a black and strongly magnetic jacobsonite-like mineral in a rhodonite-quartz ore from the Gozaisho mine, Iwaki city, Fukushima Prefecture, attracted the authors' attention. Its ore microscopic study disclosed the presence of anisotropism and the subsequent wet chemical and X-ray studies informed the presence of high  $Mn_2O_3$  content in place of  $Fe_2O_3$  in jacobsonite and pseudocubic tetragonal cell respectively. On account of these differences, the mineral was considered to be a new species distinct from jacobsonite. The mineral and name have been approved by the Commission on New Minerals and Mineral Names, I. M. A.

### Occurrence

The Gozaisho mine is located about 18 km WSW of Taira station of Japan National Railway, Iwaki city, Fukushima Prefecture, Japan (Fig. 1). The bedded manganese ore deposits are in Paleozoic crystalline schists belonging to epidote-amphibolite facies and a part of Gozaisho metamorphic rocks. The geology and petrology of mining and adjacent area have been studied by Kano *et al.* (1973) and the outline of ore deposits was described by Nambu and Okada (1959). It is said that the mining activity had ceased since 1955.

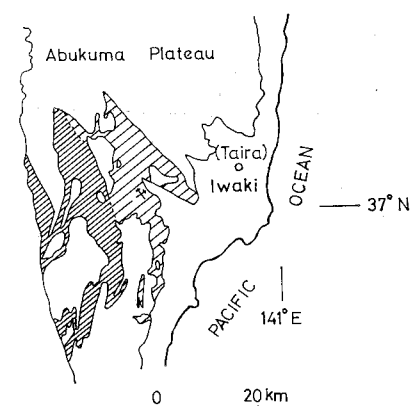


Fig. 1. Index map of the Gozaisho mine (crossed hammar mark). Coarse hatch: Gozaisho metamorphic rocks. Dense hatch: Takanuki metamorphic rocks. (After Kano *et al.* (1973) with minor corrections)

Iwakiite is found in rhodonite-quartz, hematite, rhodochrosite and minor species. It forms nearly monomineralic aggregates of various sizes in the ore (Fig. 2). The aggregates consist of anhedral grains with a mosaic to granular texture, grain size being 0.1 mm across (Fig. 3). Microscopic inclusions of copper and arsenic are seen as thin film of a manganese dioxide mineral. Due to the scarcity of this mineral impedes further study.

The manganese minerals from the Gozaisho mine include, in addition to those mentioned above, jacobsonite, hausmannite, tephroite, sonolite, aegirine, nambulite, a lithian-sodian tirobatite, helvine, pyrophanite and manganberzelite. These minerals were reported by Matsubara (1975) together with the presence of

Introduction

metamorphosed manganese ores from deposits in Japan, the occurrence of jacobsonite (Kato, 1957), especially in high grade manganese ores as hausmannite, rhodochrosite (Kato *et al.*, 1970). These ores are generally composed of quartz and such silica saturated minerals as pyroxmangite, bustamite, spinel, pyroxenes and amphiboles, except jacobsonite and magnetite molecule (Harada and

strongly magnetic jacobsonite-like mineral from the Gozaisho mine, Iwaki city, Fukushima Prefecture, attracted the authors' attention. Its ore microscopic characteristics of anisotropism and the subsequent wet etching informed the presence of high  $Mn_2O_3$  jacobsonite and pseudocubic tetragonal cell. Despite these differences, the mineral was considered distinct from jacobsonite. The mineral was named by the Commission on New Minerals and

Occurrence

located about 18 km WSW of Taira station, Iwaki city, Fukushima Prefecture, manganese ore deposits are in Paleozoic to epidote-amphibolite facies and a part of the rocks. The geology and petrology of the deposits have been studied by Kano *et al.* (1973) and were first described by Nambu and Okada (1959). Mining activity had ceased since 1955.

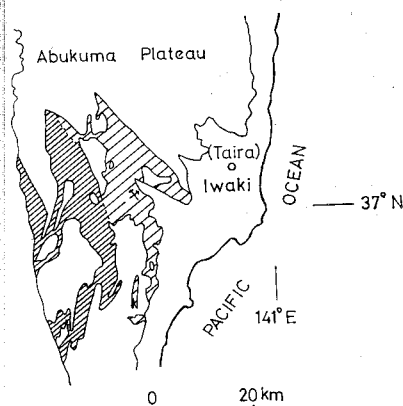


Fig. 1. Index map of the Gozaisho mine (crossed hammar mark). Coarse hatch: Gozaisho metamorphic rocks. Dense hatch: Takanuki metamorphic rocks. (After Kano *et al.* (1973) with minor corrections)

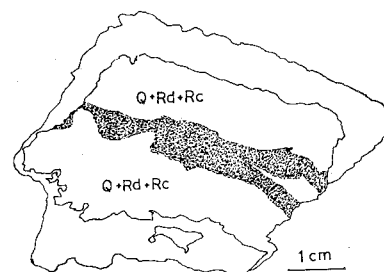


Fig. 2. Sketch of a partially polished surface of iwakiite-bearing specimen. Dotted: Iwakiite aggregate. Q+Rd+Rc: Quartz (stained by a manganese dioxide) with minor amounts of rhodonite and rhodochrosite. (No indication: unpolished)

Iwakiite is found in rhodonite-quartz ores containing braunite-hematite, rhodochrosite and minor spessartine as visual associates. It forms nearly monomineralic aggregates reaching centimeter-order size in the ore (Fig. 2). The aggregates are composed of minute sub-anhedral grains with a mosaic to granular texture, the maximum grain size being 0.1 mm across (Fig. 3). A few grains contain microscopic inclusions of copper and are superficially coated by a very thin film of a manganese dioxide mineral containing strontium. But the scarcity of this mineral impedes further study.

The manganese minerals from the Gozaisho mine recorded to date include, in addition to those mentioned above, manganosite, jacobsonite, hausmannite, tephroite, sonolite, a manganese-bearing aegirine, nambulite, a lithian-sodian tirodite, manganian phlogopite, helvine, pyrophanite and manganberzeliite. The last one is described by Matsubara (1975) together with the principal mineral associations

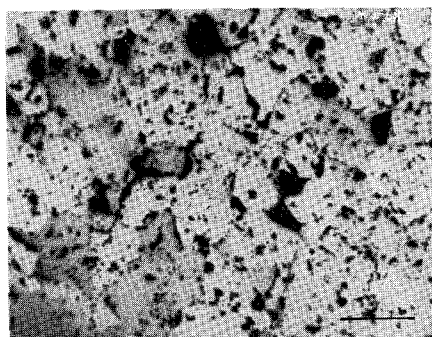


Fig. 3. Photomicrograph of polished section of iwakiite. Crossed polars. Scale 0.05 mm. Air system.

in the manganese ores found in the dump.

#### Physical and optical properties

The mineral is greenish black in colour with metallic lustre. The greenish tint can be noticed due to the direct comparison with ordinary jacobsite. Streak is black and cleavage is absent. Measured

Table 1. Reflectances of iwakiite in air.

wave length (nm)	$R_{\max}$	$R_{\min}$
420	20.0%	19.4%
440	19.9	19.3
460	20.3	19.5
480	20.4	19.6
500	20.8	19.8
520	21.2	20.1
540	21.4	20.3
560	21.3	20.4
580	21.2	20.1
600	21.0	20.0
620	20.9	19.7
640	20.5	19.6
660	20.4	19.2
680	20.2	19.1

density  $4.85 \text{ g/cm}^3$  is close to the calculated value  $4.89 \text{ g/cm}^3$ .

It is more strongly magnetic than ordinary jacobsite by a simple hand magnet examination.

Under the ore microscope the reflection colour in air is olive grey like jacobsite without discernible reflection dichroism.

The reflectances measured on fourteen wave lengths are given in Table 1. Vickers micro-indentation hardness ranges from

681 to  $772 \text{ kg/mm}^2$  (load 100 g), corresponding to  $6\frac{1}{2}$  in Mohs' scale. Between crossed polars it is anisotropic with colours changing from yellow to black. In a few grains trace of simple twin is observed. The orientation of it could not be directly determined. It is not a twin with  $\{101\}$  as a twinning plane as in jacobsite.

Observation of other polished sections of iwakiite is less anisotropic variety than the studied material. The powder study by the diffractometer method suggests it to be less distorted than analysed material. It contains a lesser amount of  $\text{MnMn}_2\text{O}_4$  molecule than the

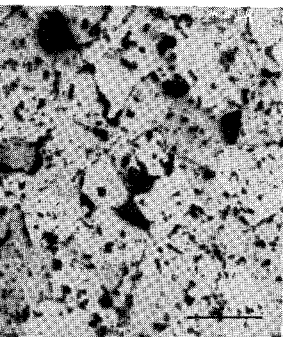
#### X-ray studies

Precession photographs of the single crystal of iwakiite are tetragonal, space group  $P4_2/nmm$ ,  $a_0=8.52$ ,  $c_0=8.52$  Å into  $a_0=8.519$ ,  $c_0=8.540$  Å after the X-ray powder study by the diffractometer method. The given tetragonal cell in terms of the analogy with jacobsite. If reduced to the  $a$ -axis,  $a_0$  becomes  $6.024$  Å. The indexed powder diffraction pattern is given in Table 2. It is very similar to that of jacobsite. This is due to the split of some diffraction peaks of jacobsite (404)-(440) (Fig. 4). The X-ray powder diffraction of the material exposed in air for a month after the preparation shows significant changes of diffraction angles and the appearance of subsidiary peaks together with the lower order peaks of iwakiite itself. This suggests that the material in air and suffers oxidation very probably.

#### Chemical analysis

About 1 gramme of purified material was analysed by the author. The result is given in Table 3 and the ideal formula  $\text{Mn}^{2+}(\text{Fe}^{3+}, \text{Mn}^{3+})_2\text{O}_4$ , where

tetragonal spinelloid mineral



Micrograph of polished section of iwakiite.  
Scale 0.05 mm. Air system.

found in the dump.

#### Physical and optical properties

Iwakiite is black in colour with metallic lustre. It is noticed due to the direct comparison with hausmannite, which is black and cleavage is absent. Measured density 4.85 g/cm<sup>3</sup> is close to the calculated value 4.89 g/cm<sup>3</sup>. It is more strongly magnetic than ordinary jacobsite by a simple hand magnet examination.

Under the ore microscope the reflection colour in air is olive grey like jacobsite without discernible reflection dichroism. The reflectances measured on fourteen wave lengths are given in Table 1. Vickers micro-indentation hardness ranges from

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681 to 772 kg/mm<sup>2</sup> (load 100 g), corresponding approximately to 6 to 6½ in Mohs' scale. Between crossed polars it is moderately anisotropic with colours changing from yellowish to brownish grey. In a few grains trace of simple twin is observed. Although the orientation of it could not be directly determined, it will be due to a twin with {101} as a twinning plane as in case of hausmannite.

Observation of other polished sections disclosed the existence of less anisotropic variety than the studied material. Preliminary X-ray powder study by the diffractometer method informed the unit cell to be less distorted than analysed material. It is expected to contain lesser amount of MnMn<sub>2</sub>O<sub>4</sub> molecule than the studied material.

#### X-ray studies

Precession photographs of the single crystal showed it to be tetragonal, space group  $P4_2/nmm$ ,  $a_0=8.52$ ,  $c_0=8.54\text{Å}$ , which are refined into  $a_0=8.519$ ,  $c_0=8.540\text{Å}$  after the X-ray powder diffraction study by the diffractometer method. The given tetragonal cell was taken in terms of the analogy with jacobsite. If reduced by taking [110] as  $a$ -axis,  $a_0$  becomes 6.024 Å. The indexed powder pattern is given in Table 2. It is very similar to that of jacobsite but distinct from it due to the split of some diffraction peaks such as (004)-(400) and (404)-(440) (Fig. 4). The X-ray powder diffraction pattern of the material exposed in air for a month after pulverization shows significant changes of diffraction angles and appearance of some subsidiary peaks together with the lowering of intensity of some peaks of iwakiite itself. This suggests that the mineral is unstable in air and suffers oxidation very probably.

#### Chemical analysis

About 1 gramme of purified material was analysed by the third author. The result is given in Table 3 and can be recalculated into the ideal formula  $Mn^{2+}(Fe^{3+}, Mn^{3+})_2O_4$ , where  $Fe^{3+} : Mn^{3+}$  is about 2:1.

Table 2. X-ray powder data for jacobsite and iwakiite.

1		2				
<i>I</i>	<i>d</i> (Å)	<i>I</i>	<i>d</i> (Å)	<i>Q</i> <sub>obs</sub>	<i>Q</i> <sub>cal</sub>	<i>hkl</i>
20	4.88	30	4.929	0.0412	0.0413	111
40	3.00	40	3.016	0.1099	0.1100	202
					0.1102	220
100	2.56	100	2.570	0.1514	0.1510	113
					0.1515	311
5	2.45	6	2.461	0.1651	0.1651	222
50	2.13	16	2.135	0.2194	0.2194	004
		16	2.130	0.2204	0.2204	400
20	1.734	15	1.739	0.3308	0.3304	422
60	1.632	30	1.640	0.3717	0.3714	333
70	1.499	35	1.509	0.4391	0.4398	404
		40	1.506	0.4408	0.4409	440
5	1.340					026
20	1.294	10	1.299	0.5927	0.5918	533
5	1.279	8	1.284	0.6062	0.6060	622
<i>a</i> <sub>0</sub> =8.474Å		<i>a</i> <sub>0</sub> =8.519Å				
		<i>c</i> <sub>0</sub> =8.540Å				

1. Jacobsite. Jacobsberg, Sweden. Fe/Mn radiation. Camera method. After Berry and Thompson (1962).
2. Iwakiite. Gozaisho mine, Fukushima Pref., Japan. Fe/Mn radiation. Diffractometer method. This study.

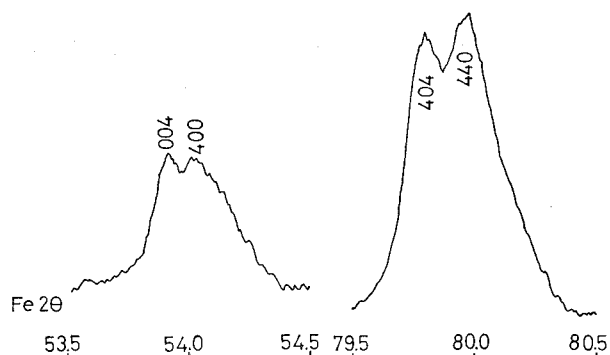


Fig. 4. X-ray powder diffraction peaks of (004)-(400) and (404)-(440) showing the split nature. Fe/Mn rad.

Table 3. Chemical analysis of iwakiite (an

	Wt. %	molecular quotient	metal number
MnO	30.34	0.2477	0.4277
MgO	0.61	0.0151	0.0151
Mn <sub>2</sub> O <sub>3</sub>	22.70	0.1438	0.2876
Fe <sub>2</sub> O <sub>3</sub>	44.42	0.2781	0.5562
Al <sub>2</sub> O <sub>3</sub>	0.32	0.0031	0.0062
SiO <sub>2</sub>	0.47	0.0078	0.0078
TiO <sub>2</sub>	0.30	0.0038	0.0038
SrO	0.04		
Na <sub>2</sub> O	0.02		
K <sub>2</sub> O	0.02		
Total	99.24%		

Empirical formula on the basis of O=4:  
 $(\text{Mn}^{2+}_{0.963}\text{Mg}_{0.035})_{\Sigma=1.018}(\text{Fe}^{2+}_{1.278}\text{Mn}^{3+}_{0.661}\text{Si}_{0.0078}\text{Ti}_{0.0038})_{\Sigma=2.950}\text{O}_4$   
 Ideal formula:  
 $\text{Mn}^{2+}(\text{Fe}^{3+}, \text{Mn}^{3+})_2\text{O}_4$  with  $\text{Fe}^{3+}:\text{Mn}^{3+}=2:1$

This is different from that of ordinary jacobite in the presence of Mn<sup>3+</sup> partially substituting Fe<sup>3+</sup> in magnojacobsite which has the Fe<sup>3+</sup>:Mn<sup>3+</sup> ratio (1964). If expressed in terms of the end member, the mineral contains (Mn<sup>2+</sup>Fe<sub>2</sub><sup>3+</sup>O<sub>4</sub>)<sub>66</sub>(Mn<sup>2+</sup>Mn<sub>2</sub><sup>3+</sup>O<sub>4</sub>)<sub>34</sub> hausmannite 34% after the neglect of Mn<sup>2+</sup>. The assumption that all the iron is in trivalent state is

#### Allied minerals

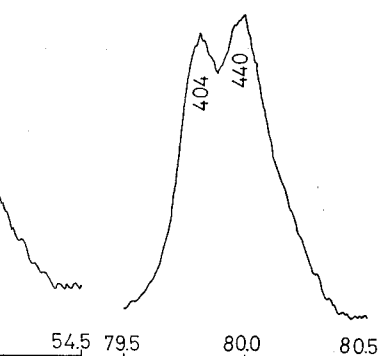
Although the present mineral is readily identified as a spinelloid site under the ore microscope, the reflectance spectra are very similar to those of ordinary jacobite. Moreover, X-ray powder and ore magnetic iron manganese oxide mineral. The locality show the existence of less anisotropic

new tetragonal spinelloid mineral

X-ray powder data for jacobsite and iwakiite.

2			
$d(\text{Å})$	$Q_{\text{obs}}$	$Q_{\text{cal}}$	$hkl$
4.929	0.0412	0.0413	111
3.016	0.1099	0.1100	202
		0.1102	220
2.570	0.1514	0.1510	113
		0.1515	311
2.461	0.1651	0.1651	222
2.135	0.2194	0.2194	004
2.130	0.2204	0.2204	400
1.739	0.3308	0.3304	422
1.640	0.3717	0.3714	333
1.509	0.4391	0.4398	404
1.506	0.4408	0.4409	440
			026
1.299	0.5927	0.5918	533
1.284	0.6062	0.6060	622

Sweden. Fe/Mn radiation. Camera method. (Lian, 1962).  
 Fukushima Pref., Japan. Fe/Mn radiation. This study.



split nature. Fe/Mn rad.

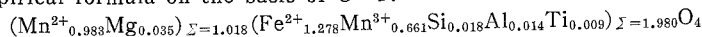
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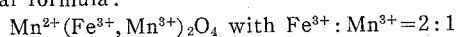
Table 3. Chemical analysis of iwakiite (analyst: K. Nagashima).

	Wt. %	molecular quotient	metal number	oxygen number	metal number (O=4)
MnO	30.34	0.2477	0.4277	0.4277	0.983
MgO	0.61	0.0151	0.0151	0.0151	0.035
Mn <sub>2</sub> O <sub>3</sub>	22.70	0.1438	0.2876	0.4314	0.661
Fe <sub>2</sub> O <sub>3</sub>	44.42	0.2781	0.5562	0.8343	1.278
Al <sub>2</sub> O <sub>3</sub>	0.32	0.0031	0.0062	0.0093	0.014
SiO <sub>2</sub>	0.47	0.0078	0.0078	0.0156	0.018
TiO <sub>2</sub>	0.30	0.0038	0.0038	0.0076	0.009
SrO	0.04				
Na <sub>2</sub> O	0.02				
K <sub>2</sub> O	0.02				
Total	99.24%				

Empirical formula on the basis of O=4:



Ideal formula:



This is different from that of ordinary jacobsite ( $\text{Mn}^{2+}\text{Fe}_3^{3+}\text{O}_4$ ) due to the presence of  $\text{Mn}^{3+}$  partially substituting  $\text{Fe}^{3+}$  and that of rhombo-magnojacobsite which has the  $\text{Fe}^{3+}:\text{Mn}^{3+}$  ratio about 1:1.2 (Lian, 1964). If expressed in terms of the end member molecules, the present mineral contains  $(\text{Mn}^{2+}\text{Fe}_3^{3+}\text{O}_4)_{66}(\text{Mn}^{2+}\text{Mn}_3^{3+}\text{O}_4)_{34}$  or jacobsite 66% and hausmannite 34% after the neglect of minor components and assumption that all the iron is in trivalent state.

#### Allied minerals

Although the present mineral is readily distinguished from jacobsite under the ore microscope, the reflection colour and reflectances are very similar to those of ordinary jacobsite or jacobsite solid solution. Moreover, X-ray powder and ore microscopical studies of magnetic iron manganese oxide mineral looking like iwakiite from the locality show the existence of less anisotropic material with less

distorted pseudocubic cell than the described material, suggesting the existence of minor crystallographic and compositional variation in iwakiite itself. If the anisotropism of the observed grain is weaker than the described iwakiite, it is highly probable that the mineral is mis-identified as jacobsite. Actually the occurrence of "anisotropic jacobsite" has been reported by Uytendogaardt and Burke (1975), Matsubara *et al.* (1976) and Hirowatari *et al.* (1978), and the occurrence of iwakiite might be more common than expected.

At least there are two pseudocubic spinelloid minerals to be mineralogically compared with iwakiite. One is donathite found by Seeliger and Mücke (1969) and later re-considered by Moore (1977). It is an intermediate member between magnetite and chromite, both being cubic and differing from the present mineral, which is an intermediate member between cubic jacobsite and tetragonal hausmannite. A compositional variation due to increase of  $Mn^{3+}$  replacing  $Fe^{3+}$  leads to the monotonous increase of degree of distortion in the iwakiite cell, whereas the compositional variation of donathite along the binary system  $FeFe_2O_4$ - $FeCr_2O_4$  is to be limited and the degree of distortion from cubic symmetry will not be so large as in hausmannite.

Another pseudocubic spinelloid is rhombomagnojacobsite described by Lian (1964), who obtained the symmetry as orthorhombic with larger degree of distortion from jacobsite and the chemical composition containing appreciable amount of magnesium probably replacing manganese. The role of magnesium should be significant as inferable from the largely distorted cell of synthetic  $MgMn_2O_4$  (Radhakrishnan and Biswas, 1975).

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new tetragonal spinelloid mineral

than the described material, suggesting the illographic and compositional variation in anisotropism of the observed grain is weaker than in iwakiite, it is highly probable that the mineral described by Uytenbogaardt and Burke (1975), and the occurrence of "anisotropic" reported by Hirowatari *et al.* (1978), and the occurrence of the mineral is more common than expected.

Two pseudocubic spinelloid minerals to be compared with iwakiite. One is donathite found by Hirowatari and later re-considered by Moore (1977). The difference between magnetite and chromite, both reported from the present mineral, which is an intergrowth of cubic jacobite and tetragonal hausmannite. The variation due to increase of  $Mn^{2+}$  replacing  $Fe^{2+}$  and the increase of degree of distortion in the structure with compositional variation of donathite along the  $FeCr_2O_4$  is to be limited and the degree of anisymmetry will not be so large as in haus-

mannite. The spinelloid is rhombomagnojacobite described by Hirowatari and the symmetry as orthorhombic with difference from jacobite and the chemical composition. The amount of magnesium probably replacing iron and the amount of magnesium should be significant as inferred from the unit cell of synthetic  $MgMn_2O_4$  (Radhakri-

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University, for measurement of reflectances.

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