

NEW DATA FOR HISINGERITE AND NEOTOCITE

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ABSTRACT

New chemical and *x*-ray data are given for three samples of hisingerite from northern Minnesota. The variable chemical composition of hisingerite, a hydrous iron silicate, is shown by the new analyses representing two samples from gabbros of the Beaver Bay complex and one from veins in the Biwabik iron-formation at Babbitt, Minnesota.

The *x*-ray diffraction patterns consist of a few diffuse lines that resemble the pattern for nontronite and also bear some similarity to the published data for iron-rich saponite. Structural formulas computed for the Beaver Bay hisingerites fit a saponite structure reasonably well; however, the hisingerite from Babbitt, as well as analyzed samples from Parry Sound, Ontario, and from Montauban, Quebec, show excess Fe_2O_3 for the saponite structure.

Hisingerite occurs in a variety of geologic environments, and commonly results from alteration of pyroxene and olivine. So-called hisingerites may represent mixtures of two or more minerals or stages in the alteration of ferromagnesian minerals.

A sample of hisingerite-like material from the Montreal Mine, Iron County, Wisconsin, closely resembling hisingerite in physical properties, was found to be a hydrous manganese silicate, neotocite. A chemical analysis and *x*-ray data are given for the neotocite.

INTRODUCTION

Hisingerite is a soft, black, hydrous iron silicate with a characteristic resinous luster and conchoidal fracture. It has been reported throughout the world in rocks of various ages and in many geologic settings. It occurs with ores of uranium, tin, copper, lead, zinc, iron, and manganese. Recent studies of hisingerite are by Bowie (1955), Nikolsky (1953), Sudo and Nakamura (1952). The present study is concerned with occurrences of hisingerite in gabbros in the vicinity of Beaver Bay and in iron formation at Babbitt in northeastern Minnesota. Some new data are presented for hisingerite from Parry Sound, Ontario, previously described by Schwartz (1924) and for hisingerite from the Montauban Mines, Quebec, described by Osborne and Archambault (1950).

DESCRIPTION OF SAMPLES

Beaver Bay complex. Hisingerite is a common alteration produced in the iron-rich gabbroic rocks of the Beaver Bay complex. The present study was started on material collected by R. B. Taylor who noted the widespread occurrence of hisingerite in the Beaver Bay area during the course of field work in 1953. Additional material was collected by H. M. Gehman, Jr., in 1955. The mineral described by Muir (1954) as bowlingite, an alteration product of olivine in iron-rich diabase from Beaver Bay,

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is the hisingerite of the present paper. In describing the olivine in the Beaver Bay specimen, Muir notes that it "is invariably surrounded either by a reddish-brown highly-birefringent alteration product which is mostly bowlingite, or, more rarely, by deep-green isotropic chlorophaeite."

The Beaver Bay complex was named by Grout and Schwartz (1939, p. 13) and was related by them to the Middle Keweenawan intrusive action at the time of emplacement of the Duluth gabbro. The gabbroic intrusives, where concordant with the Keweenawan flows, are sills, but Grout and Schwartz (1939, p. 33) noted that transgressive and dike-like masses of diabase form some high bluffs. More recent studies by H. M. Gehman, Jr. (unpublished Ph.D. thesis, University of Minnesota, 1957) show that the Beaver Bay complex consists of a number of intrusions.

Sample No. 1 (Table 1, etc.) was collected by R. B. Taylor from laminated ferrogabbro in a quarry that supplied large blocks for the construction of the breakwaters of the Silver Bay harbor. The quarry is situated west of Highway 61 in SW $\frac{1}{4}$ Sec. 6, T. 55 N., R. 8 W., approximately one mile southwest of the Reserve Mining Company plant at Silver Bay.

Sample No. 2 was collected by H. M. Gehman, Jr. from ferrogabbro in a freshly blasted cut behind the thickening plant of the taconite concentrator of the Reserve Mining Company. The locality is east of Highway 61 in SE $\frac{1}{4}$ Sec. 31, T. 56 N., R. 7 W.

East Mesabi. Hisingerite has been reported in very small amounts in the Biwabik formation in the Morris Mine near Hibbing, Minnesota (Gruner, 1946, p. 22), but it is a common and locally abundant mineral in the Eastern Mesabi district where it occurs in veinlets in the iron formation. We are indebted to J. N. Gundersen who collected the material for this study from the Peter Mitchell Pit, Reserve Mining Company, Babbitt, Minnesota. According to Gundersen, the hisingerite veinlets average about 1 mm. in width, with a maximum of 5 mm. The veinlets are usually more common in phases of the iron formation which contain more fayalite and hypersthene than average for the locality.

The Babbitt hisingerite is black, resinous, and brittle, with a hardness of 3 and a specific gravity of approximately 2.67. Under the microscope it appears to be isotropic, greenish-brown in color, and in very thin fragments at high magnification (400 \times) is apple green. The refractive index is 1.66.

Wilcox Mine, Parry Sound, Ontario. Hisingerite from the Wilcox Mine was generously supplied by G. M. Schwartz, and the description that follows is taken from the earlier paper by Schwartz (1924). In the Wilcox Mine hisingerite is intimately associated with pyrite and chalcopyrite and forms a matrix for the sulfides. The ore occurs as masses and dis-

seminations in Precambrian garnet-biotite schists. In addition to the garnet and biotite, minerals of the schists include plagioclase, hypersthene, hornblende, apatite, quartz, and others. Schwartz (1924, p. 144) concluded that the hisingerite was formed for the most part by the alteration of the hypersthene.

The Parry Sound hisingerite is black, resinous, and brittle. The hardness is approximately 2.5. The specific gravity is given as 2.50 by Schwartz (1924, p. 142) who noted that the "streak is yellow and similar to that of limonite." Schwartz estimated that three-fourths of the material he studied is isotropic and one-fourth anisotropic. He found a range in refractive index, $n = 1.50-1.56$, which was confirmed, $n = 1.50-1.57$, in the present investigation.

Montauban Mines, Quebec. The hisingerite was first noted in the Tetreault mine by Osborne in 1938 and described in detail by Osborne and Archambault (1950), and the notes that follow are taken from their description. The sample was presented by Osborne to J. W. Gruner who made it available for the present study.

The zinc and lead ores at Montauban occur in crystalline limestone in a gangue that is largely diopside and tremolite. The hisingerite, according to Osborne and Archambault (1950, p. 289), is largely a replacement of diopside, and complete pseudomorphs after diopside were found. Some of the hisingerite apparently also formed as a replacement of calcic plagioclase and of siderite. The hisingerite is cut by veinlets of pyrite from one-tenth to one inch wide and by paper-thin veinlets of calcite. The calcite veinlets are attributed to fillings of contraction cracks by Osborne and Archambault.

The Montauban hisingerite is black, resinous, and brittle. Osborne and Archambault give the hardness as 2.5 and the specific gravity as 2.53-2.55. They found the material to be isotropic with variable refractive index which they determined as $n = 1.65$ in 1948 and as $n = 1.51$ in 1951. The sample in the Minnesota collection is variable in refractive index, but the results generally were close to $n = 1.66$.

Montreal Mine, Wisconsin. A sample labeled hisingerite in the Minnesota collection comes from the 94th crosscut on the 30th level of the Montreal Mine, Gogebic range, Iron County, Wisconsin. This sample was collected by J. W. Gruner. It is dark brown, resinous, brittle with good conchoidal fracture, and has a hardness of 3 and specific gravity of 2.43. The mineral is neotocite as shown by its chemical analysis (Table 1). The sample is essentially isotropic with small amounts anisotropic when viewed at high magnification. The index of refraction is variable but is close to 1.62.

CHEMISTRY

Three new chemical analyses of hisingerite from Minnesota (Table 1) were made for the present investigation in the Rock Analysis Laboratory at the University of Minnesota. For comparison, the published analyses of hisingerite from the Wilcox Mine, Parry Sound, Ontario, and from the Tetreault Mine at Montauban, Quebec, are included. The variability in chemical composition of so-called hisingerite is apparent from these analyses. The molecular ratio of $\text{Fe}_2\text{O}_3/\text{FeO}$ in these samples ranges from 3.67 for the Parry Sound sample to 0.35 for the sample from Babbitt. The chemical variability is further discussed in a later section concerned with the probable structural formula.

The chemical analysis (Table 1, No. 6) of neotocite was made of two small fragments which appeared to be homogeneous, and no impurities

TABLE 1. CHEMICAL ANALYSES OF HISINGERITES AND OF NEOTOCITE

	1	2	3	4	5	6
SiO_2	39.11	42.35	38.19	35.57	37.54	31.74
Al_2O_3	3.89	3.65	0.00	0.38	0.56	0.50
TiO_2	0.16	0.13	0.01	0.12	0.00	0.01
Fe_2O_3	22.22	23.28	19.91	39.20	37.02	1.88
FeO	5.90	5.54	24.64	4.80	4.66	0.00
MnO	0.92	0.38	0.66		0.75	12.29
MnO_2						27.05
MgO	5.21	8.22	2.36	1.60	2.81	9.55
CaO	2.52	2.06	0.61	0.85	1.52	0.28
Na_2O	0.27					0.02
K_2O	0.07					0.01
$\text{H}_2\text{O}+$	5.82	6.85	8.40	11.60	9.20	8.86
$\text{H}_2\text{O}-$	13.54	6.95	5.53	6.00	6.00	7.04
CuO						0.78
Organic Matter					0.05	
Total	99.63	99.41	100.31	100.12	100.11	100.01
S.G.			2.67—.02	2.50	2.53-2.55	2.43—.02

1. Hisingerite, gabbro, Beaver Bay complex. S. S. Goldich, analyst.
2. Hisingerite, gabbro, Beaver Bay complex. S. S. Goldich, analyst.
3. Hisingerite, Biwabik iron-formation, East Mesabi range. C. O. Ingamells, analyst.
4. Hisingerite, Parry Sound, Ontario. R. J. Leonard, analyst (Schwartz, 1924, p. 142).
5. Hisingerite, Montauban Mines, Quebec. H. Boileau, analyst (Osborne and Archambault, 1950, p. 286).
6. Neotocite, Montreal Mine, Wisconsin. Doris Thaemlitz and C. O. Ingamells, analysts.

were recognizable in the powder used for determination of the refractive index.

X-RAY DIFFRACTION DATA

Samples for powder photographs were obtained by hand picking under a binocular microscope. The hisingerite (No. 2) from gabbro of the Beaver Bay complex was further purified by magnetic separation with a Franz magnetic separator. Data from the powder photographs, together with the data of Sudo and Nakamura (1952) and of Bowie (1955), are given in Table 2. The lines on all patterns were broad and diffuse.

The x -ray diffraction pattern of neotocite is similar enough to those of the hisingerites that considering the broad and diffuse lines in the patterns of both of these minerals it is impossible to differentiate them by x -ray diffraction.

INFRA-RED ABSORPTION SPECTRA

The infra-red absorption spectra of the chemically analyzed samples of hisingerite and of neotocite are shown in Fig. 1. The silicate bands of the spectra at approximately 10 microns are poorly defined as is typical of the montmorillonite group. The maxima at about 6.2 microns represents O-H bending.

DIFFERENTIAL THERMAL ANALYSES

The DTA curves of the hisingerites and neotocite are shown in Fig. 2. The runs were made in air with a heating rate of 10° C. per minute. The endothermic reactions between 100° C. and 200° C. on these curves represent the loss of water. The only other characteristic reaction is exothermic and occurs at about 1000° C. This reaction probably represents the formation of ferrites. The exothermic reactions between 400° C. and 500° C. on the curves of the Montauban Mines and Parry Sound hisingerites represent the oxidation of a very small amount of pyrite contaminating these samples. The strong exothermic reaction between 700° C. and 800° C. on the DTA curve of neotocite represents the formation of braunite.

STRUCTURE

The fact that hisingerites give poor x -ray diffraction patterns makes a unique determination of structure impossible. Gruner (1935), Sudo and Nakamura (1952), and Bowie (1955) all have noted the general similarity between the x -ray diffraction patterns of hisingerites and those of nontronites. The strong 12–17 Å basal reflection of nontronites, however, is absent in x -ray diffraction patterns of hisingerite. The poor x -ray diffraction patterns of hisingerites, together with their variable ferric-ferrous

TABLE 2. X-RAY DIFFRACTION PATTERNS OF HISINGERITE AND NEOTOCITE

1	2	3	4	5	6	7	8	9	10	11	12
d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I	d(Å) I
		7.53 w									
4.42 w	4.51 w	4.33 w	4.55 w		4.36 w	4.45 s	4.40 s	4.40 s	4.39 s	4.43 s	4.40 ss
		3.50 w			3.59 w	3.53 s	3.55 s	3.55 s	3.58 s		
2.48 w	2.54 w	2.55 w	2.59 w	2.51 2	2.59	2.56 s	2.56 s	2.58 s	2.57 s	2.56 s	2.60 ss
	1.70 w					1.71 w	1.70 w	1.70 w	1.71 w		
1.54 vw	1.53 w	1.55 w	1.54 w	1.54 w	1.54 w	1.54 s	1.53 s	1.53 s	1.54 s	1.53 s	1.50 ss
1.21 vw		1.21 w									

1-6 unfiltered iron radiation, camera radius 57.3 mm., 24-hour exposures.

Intensities are as follows: s, strong; w, weak; vw, very weak.

1. Hisingerite No. 1, Beaver Bay complex.
2. Hisingerite No. 2, Beaver Bay complex.
3. Hisingerite, East Mesabi range.
4. Hisingerite, Parry Sound.
5. Hisingerite, Montauban Mines.
6. Neotocite, Montreal Mine.
7. Hisingerite, Riddarhyttan, Sweden (Bowie, 1955).
8. Hisingerite, Blaine County, Idaho (Bowie, 1955).
9. Hisingerite, Nicholson Mine, Saskatchewan (Bowie, 1955).
10. Hisingerite, Langanshyttan, Sweden (Bowie, 1955).
11. Hisingerite, Weal Jane, Cornwall (Bowie, 1955).
12. Hisingerite, Kawayma Mine, Japan (Sudo and Nakamura, 1952)

ratio, lead the writers to suggest that hisingerites may form as a metastable iron saponite which then undergoes oxidation in different degrees. On this basis the analyzed samples described in this paper were fitted to saponite structures by the method of Ross and Hendricks (1945). Trivalent iron was equated to divalent iron. The results of these calculations are given in Table 3. The analyses of the hisingerites from the Beaver Bay complex fit a saponite structure reasonably well. Hisingerite No. 2, for example, gives a structural formula of $(\text{Fe}''_{1.90}\text{Mn}_{0.03}\text{Mg}_{1.05}\text{Al}_{0.01})\text{Al}_{0.36}\text{Si}_{3.64}\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$, $\text{Ca}/2=0.38$ (analysis), $\text{X}=0.37$ (calculated).

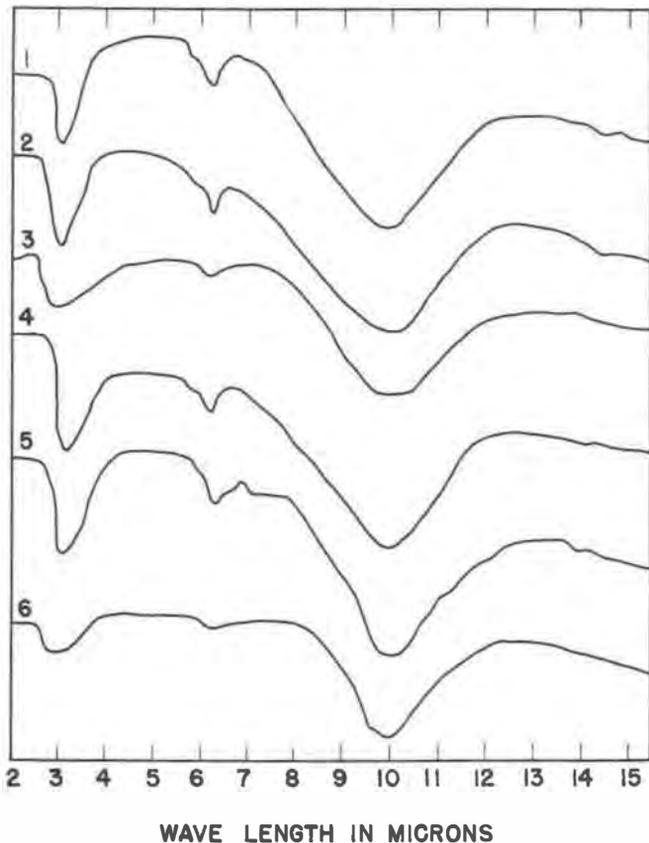


FIG. 1. Infra-red Spectra.

1. Hisingerite No. 1, Beaver Bay complex
2. Hisingerite No. 2, Beaver Bay complex
3. Hisingerite, East Mesabi range
4. Hisingerite, Parry Sound
5. Hisingerite, Montauban Mines
6. Neotocite, Montreal Mine

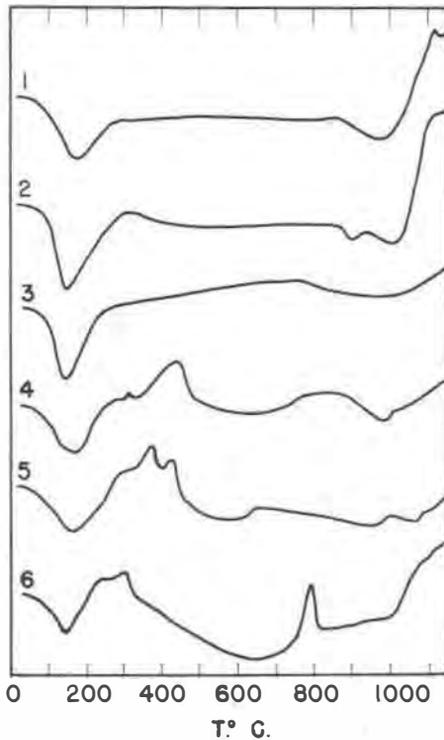


FIG. 2. DTA Curves.

1. Hisingerite No. 1, Beaver Bay complex
2. Hisingerite No. 2, Beaver Bay complex
3. Hisingerite, East Mesabi range
4. Hisingerite, Parry Sound
5. Hisingerite, Montauban Mines
6. Neotocite, Montreal Mine

The analyses of the other hisingerites, however, indicate more cations than can be fitted into a saponite structure. In these, Al + Si was assumed to occupy the four tetrahedral positions of the sheet structures, and Fe'', Mn'', Mg, and Fe''' recalculated to Fe'' were used to fill three octahedral positions. This method of calculation left excess Fe₂O₃. No goethite or hematite appeared in the *x*-ray diffraction patterns of these hisingerites. The DTA curves likewise do not show an endothermic reaction at about 400° C. which would be characteristic of goethite. In the Parry Sound and Montauban Mines hisingerites, however, this reaction may have been masked by the exothermic reactions due to the oxidation of sulfides contaminating the samples.

The *x*-ray diffraction pattern of the East Mesabi hisingerite has a 7.5 Å

TABLE 3. STRUCTURAL FORMULAS OF HISINGERITES CALCULATED AS SAPONITE^a

	1	2	3	4	5
Octahedral					
Fe'' ^b	2.01	1.90	2.57	2.73	2.49
Mn''	0.07	0.03	0.06		0.07
Mg''	0.72	1.05	0.37	0.27	0.44
Al'''	0.06	0.01			
Tetrahedral					
Al'''	0.36	0.36		0.05	0.07
Si''''	3.64	3.64	4.00	3.95	3.93
Exchangeable					
Ca''/2	0.50	0.38	0.14	0.20	0.34
Na'	0.05				
K'	0.01				
Calculated	0.58 ^c	0.37	0.00	0.05	0.07
Excess Fe ₂ O ₃ (%)			14.63	11.87	10.55

^a Calculated by the method of Ross and Hendricks (1945) using analytical results of alkaline earths and alkalis as "X." When excess cations for octahedral positions were indicated in analyses, Si+Al was considered as 4.00 and excess cations considered to be Fe₂O₃. Oxygen considered as 10, hydroxyl as 2.

^b Fe''' recalculated as Fe'' in structure.

^c 0.22 octahedral and 0.36 tetrahedral.

1. Hisingerite No. 1, Beaver Bay complex.

2. Hisingerite No. 2, Beaver Bay complex.

3. Hisingerite, East Mesabi range.

4. Hisingerite, Parry Sound.

5. Hisingerite, Montauban Mines.

line which suggests a possible incipient chlorite structure. This sample has a high ferrous to ferric ratio. The other hisingerites whose analyses cannot be fitted to a saponite structure have low ferrous to ferric ratios, and their x-ray diffraction patterns did not show 7.5 Å lines. For these reasons, a chlorite structure is not considered probable.

Wilshire (1958), studying iddingsites which are similar chemically and physically to hisingerites, concluded they consist of mixed-layer chlorite-smectite minerals. Brown and Stephen (1959) found iddingsite from New South Wales, Australia, to consist of goethite and a layer silicate lattice. Sun (1957) found material considered iddingsite from basaltic rocks in New Mexico was cryptocrystalline goethite plus amorphous silica and other oxides. A possible explanation is that ferromagnesian

minerals first alter to an iron saponite, then to a mixture of goethite and a layer silicate lattice and finally to a mixture of goethite and amorphous oxides. Perhaps the hisingerites with iron in excess of that which can be fitted to a saponite structure represent an alteration intermediate between the first and second steps listed above. A. C. Waters (personal communication, November 23, 1955) suggests that hisingerite and chlorophaeite may be equivalent. Wilshire (1958) has published x -ray data on isotropic alteration products of olivines and orthopyroxenes which are in good agreement with the data for hisingerite, except that the chlorophaeite patterns have a 14 Å basal reflection. Since this basal reflection was not affected by treatment with ethylene glycol or heating, he postulated an incipient chlorite structure for chlorophaeite.

Canbyite (Hawkins and Shannon, 1920; Bowie, 1955) and an iron-rich saponite from Japan (Sudo, 1954) are also similar to hisingerite. Some typical chemical data for the various iron silicates, selected from the literature, are given in Table 4. X -ray data are given in Table 5.

TABLE 4. CHEMICAL ANALYSES OF CANBYITE, IRON-RICH SAPONITE, CHLOROPHAEITE, AND IDDINGSITE

	1	2	3	4
SiO ₂	32.85	39.68	40.35	38.63
Al ₂ O ₃	2.64	3.93	5.11*	1.78
TiO ₂	0.26	0.37	0.20	
Fe ₂ O ₃	40.70	19.82	24.99	32.49
FeO		1.12	3.55	
MnO	0.74	0.19	0.22	
MgO	2.05	11.21	5.48	6.64
CaO	1.50	2.37	1.32	2.79
Na ₂ O			0.18	
K ₂ O			1.44	
H ₂ O+	7.90	6.16	8.51	17.70
H ₂ O-	11.40	15.11	8.51	
Total	100.04	99.96	99.86	100.03

* With possible P₂O₅, CO₂, SO₃, S, NiO, BaO, SrO not found.

1. Canbyite, Brandywine Quarry, Wilmington, Delaware. E. V. Shannon, analyst (Hawkins and Shannon, 1924).
2. Iron-rich saponite, iron sand bed at Monuia, Oide-Mura, Natorigun, Miyagi Prefecture, Japan. J. Osaka, analyst (Sudo, 1954).
3. Chlorophaeite, New Reservoir, Holyoke, Massachusetts. G. Steiger, analyst (Emerson, 1905).
4. Iddingsite, La Jara Creek, Conejos quadrangle, Colorado (Ross and Shannon, 1925).

TABLE 5. X-RAY DIFFRACTION PATTERNS OF IRON-RICH SAPONITE, CANBYITE, AND CHLOROPHAEITE

Indices	$d(\text{kX})$	I	$d(\text{\AA})$	I	$d(\text{\AA})$	I
	1		2		3	
001	15.7	40-150			15.0-14.0	s
11·0·2	4.55	10	4.40	s	4.5	w
					4.48	vw
			3.54	s		
13·2·0	2.62	8	2.56	s	2.59	vw
	2.47	5				
31·15·24	1.70	5	1.70	w		
33·0·6	1.533	10	1.53	s	1.55	vvw
	1.323	5				

1. Iron-rich saponite, Japan (Sudo, 1954).
2. Canbyite, Wilmington, Delaware (Bowie, 1955).
3. Chlorophaeite, Walla Walla, Washington (Wilshire, 1958).

CONCLUDING REMARKS

Hisingerite is found in a number of differing geologic occurrences. The Beaver Bay material is attributed to deuteric and late-stage hydrothermal alteration of olivine, pyroxene, and possibly other minerals. The hisingerite that occurs in veinlets cutting the Biwabik iron-formation on the East Mesabi range is related to the later stages of metasomatism in connection with the metamorphism at the time of intrusion of rocks of the Duluth complex. The hisingerites at Parry Sound, Ontario, and at Montauban, Quebec, appear to be closely related to the sulfide ore deposition and probably represent late-stage hydrothermal activity.

The origin of hisingerite as an alteration product replacing a variety of minerals suggests that this material might easily be a mixture of two or more minerals. The fineness of grain makes optical and chemical studies difficult, and even the more strongly birefringent materials give poor x-ray diffraction patterns. Hisingerite, however, appears to be a rather common mineral that warrants further study.

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