CATTIERITE AND VAESITE: NEW Co-Ni MINERALS FROM THE BELGIAN CONGO

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Abstract

The group which includes CoS_2 , NiS_2 , and FeS_2 forms an isostructural series following the pyrite lattice. Heretofore, the formation of the artificial end members of the series has been established, but approximations to CoS_2 and NiS_2 have not been found in nature. At the Shinkolobwe mine in the Belgian Congo, CoS_2 having a pyrite structure is now known. To this mineral is assigned the name *cattierite*. Material approaching NiS_2 is composition and having a pyrite type of lattice has been found in the Kasompi mine of the Belgian Congo. This mineral is tentatively called *vaesite*. The lattice constants for the natural end members of the series in angstroms are pyrite $5.40667 \pm .00007$, vaesite $5.66787 \pm .00008$, and cattierite $5.52346 \pm .00048$. The minerals are named after important contributors to the mineral development of the Belgian Congo. The four members of the pyrite series would be vaesite, cattierite, bravoite, and pyrite.

INTRODUCTION

In September 1943 Mr. Johannes Vaes of the Union Minière du Haut Katanga submitted two metallic sulfide minerals upon which he had previously made independent mineralogical studies including chemical analyses and an examination of polished surfaces. One sample was a nickel sulfide with an analysis corresponding to the formula NiS₂; the other a cobalt sulfide corresponding to CoS₂. The nickel di-sulfide had been obtained in a core during the progress of diamond drilling at the Kasompi mine about 70 kilometers west-southwest of Kambove in Haut Katanga of the Belgian Congo. The cobalt sulfide was from the Shinkolobwe mine near Jadotville, also in Haut Katanga. Preliminary chemical and microscopic investigation had led Mr. Vaes to the interpretation that the two minerals belonged to the pyrite group and there was good reason to believe represented new species. In the interval which has elapsed since the specimens were first submitted, the two minerals have been studied in the laboratory at Columbia University. The following account is intended to set forth the results of this examination.

The identity of the two sulfides has been confirmed and it seems appropriate to suggest that they represent species worthy of special names. It seems proper that the cobaltian sulfide receive the name *cattierite* after Mr. Felicien Cattier, Chairman of the Board, Union Minière du Haut Katanga, who has had a life-long connection with colonial affairs of the Belgian Congo and whose activities have contributed so largely to the stimulation of wartime metal production. He has also taken a great

interest in scientific research and the welfare of Belgian universities. It is proposed that the nickelian sulfide receive the name *vaesile* after Mr. Johannes Vaes, mineralogist for the Union Minière du Haut Katanga of the Belgian Congo, who has shown such a keen interest in the study Katanga minerals.

RS₂ Compounds

Identification of the two minerals in question depends to a considerable degree upon comparison with material previously described. This includes both minerals and artificial compounds of the RS_2 type which contain cobalt, nickel, and iron.

ARTIFICIAL CO-NI-FE DISULFIDES

It is reported that CoS_2 was made by von Setterberg (1826), although the melting and dissociation points of the synthetic product were not determined. Von Fellenberg (1840) is credited with having made NiS₂ in a melt of NiCO₃ and K₂CO₃. Both syntheses were completed before the days of adequate metallographic or x-ray studies.

de Jong and Willems (1927) report the successful synthesis of CoS_2 and NiS₂. Powdered NiS or CoS under atmospheric pressure were heated 30–40 hours with a mixture of molten sulfur at 170°, followed by heating at 150°C. for 24 hours. The nickel sulfide formed contained Ni=45.6% and S=54.4%, as compared with a theoretical Ni=47.8% and S=52.2%. X-ray diffraction measurements by de Jong and Willems (1927) yielded determinations of the dimensions of the unit cell as follows: NiS₂=5.74 and $CoS_2=5.64$ Å. The specific gravities were: NiS₂=4.31 and CoS_2 =4.55. Both synthetic products were reported to yield an x-ray diffraction pattern of the pyrite type.

In a recent paper Michener and Yates (1944) have called attention to a series of synthetic fusion products covering the range from FeS₂ to NiS₂. As reported, polished surfaces and x-ray diffraction patterns indicate that the intermediate products synthesized were mechanical mixtures, although the end-members were homogeneous with the pyrite type of structure. This apparently represents a set of synthetic products differing in character from homogeneous minerals of intermediate composition which show lattice variation as recorded in the work of Bannister (1940).

MINERAL CO-NI-FE DISULFIDES

Although it would appear from the work of de Jong and Willems (1927)

that artificial CoS_2 with the structure of pyrite may be prepared, the existence of this material in nature has not heretofore been confirmed. The same appears to be true for naturally occurring NiS_2 . On the other hand, compounds of the pyrite type in two instances containing slightly more nickel than iron, but usually containing less nickel than iron, are well established. Pyrite may occur free from nickel or with small amounts distributed through the unit cell. The nickel-bearing mineral may be known as nickelian pyrite where the amount is small, or bravoite where it is considerable. The point at which nickelian pyrite ends and bravoite begins must of necessity rest on an arbitrary decision.

Hillebrand (1907) in studying small isometric crystals associated with the vanadium ores of Minasragra, Peru, discovered a pyrite-like material which contained about 18 per cent nickel. He made a complete analysis and suggested the name bravoite. The formula given was (Fe, Ni)S₂ with the ratio of nickel to iron about 3 to 5. Later Hewitt (1909) in a general description of the ores showed a photomicrograph of a polished surface in which the roughly square outlines of bravoite crystals appeared to stand out in moderate to strong relief from a soft ground mass of patronite. The crystals pictured would measure up to one and one-half millimeters in cross section.

Villamanite from Villamanin station near Carmenes province of Leon, Spain, has been described by Schoeller and Powell (1920) as a member of the pyrite group. The mineral has been given the formula (Cu, Ni, Co, Fe)S, Se. It is a black mineral, granular and crystalline, which occurs in dolomite. Isometric crystals have been observed. In the absence of x-ray and microscopic data, the role of the mineral is uncertain.

Buddington (1924) identified a nickel-iron sulfide from the vicinity of Chitina, in the Copper River region, Alaska, which corresponded to the bravoite of Hillebrand. The Chitina material contained substantially more nickel with the iron to nickel ratio being roughly of the order of 4 to 5. Buddington also compared the properties of the two on polished surfaces as follows:

Bravoite, C	chitina, Alaska
Color:	dull gray
Cleavage:	3 directions
Hardness:	$4\frac{1}{2}$
HNO ₃ :	effervesces
HCl:	turns yellow (rubs clean)
KCN, FeCl ₃ , HgCl ₂ :	negative
KOH:	negative
Sp. Gr.:	4.31

Bravoite, Minasragra, Peru pallid gray (violet hue) 3 directions 5 (a little less) effervesces (turns blue gray) negative negative slight reaction 4.33 (?) Henglein (1914) described a mineral belonging to the pyrite group with the formula $(Co,Ni,Fe)S_2$. The mineral was found at Musen in Westphalia, Germany, and occurred in small steel gray pyritohedral crystals. Through crystal measurement the forms $\{023\}$, $\{001\}$, and $\{111\}$ were identified.

Kalb and Meyer (1926) mounted crushed ore from Mechernich in a cementing medium, polished the briquettes and studied them with a microscope. Small isometric crystals with cross sections indicating a pyritohedral habit were observed. Goniometric measurements of isolated crystals showed the forms $\{750\}$ and $\{320\}$. The crystals exhibited a distinct zonal banding on polished surfaces. Yellow nickel-poor bands were nickelian pyrite, while steel gray bands containing 3.28 per cent cobalt, 24.73 per cent nickel and 17.00 per cent iron were classed as bravoite. Later de Jong and Willems (1927) examined the bravoite from Mechernich which contained 24.73 per cent nickel and 3.28 per cent cobalt by means of x-rays. The cell edge was determined as 5.57 A.U., and the pattern was of the pyrite type.

Scholtz (1936) on the basis of microscopic study reported bravoite from the magmatic nickel-bearing ore deposits of East Griqualand and Pondoland in Africa. The mineral was observed on polished surfaces where it replaced pentlandite and vallerite. Bannister (1940) described bravoite from the Mill Close mine, Derbyshire. The mineral contained a little less nickel in proportion to iron than the Peruvian bravoite. The specific gravity was also higher, 4.82. From x-ray studies Bannister concluded that the mineral had the pyrite structure wth the unit-cell-edge equal to 5.49 A.U. Another bravoite in the same mine was said to contain 28 per cent nickel with a cell edge of 5.47 A.U.

While pyrite is one of the oldest of our common minerals and was recognized in the early Greek literature, the discovery of nickel-bearing pyrite is more recent. Cobalt-nickel pyrite was reported from Canada by Hunt (1866) and was later listed by Vernadsky (1914). Thomson and Allen (1939) in connection with studies of the nickelian pyrite from the Denison mine of the Sudbury district in Canada called attention to the existence of several localities in which pyrite contains a small amount of nickel. A sample from the Denison mine yielded 6.50 per cent nickel; from the Emmens Metal Company 3.5; from the Shepherd mine 5.40; and from the Murray mine 4.34. Material described by Emmens (1892) may have been of the same type. Later Peacock and Smith (1941) made x-ray precision measurements of common pyrite and nickelian pyrite in order to study the variation in structure with composition. They furnished a correlation of cube-edge measurements as determined by x-ray measurements.

X-ray Study

X-ray diffraction patterns of the Debeye type were taken of both the cobaltian and nickelian sulfides and associated metallic minerals. Measurements of pyrite free from nickel and cobalt, and the nickel and cobalt sulfides of the pyrite type are shown in Table 2. Lattice constants based on the measurements of the powder diffraction patterns are included in the data shown in Table 1. The associated sulfides, aside from pyrite and chalcopyrite, are shown by x-ray diffraction patterns to belong to the linnaeite-polydymite group with the formula R_3S_4 and the spinel type of lattice. In the absence of chemical analyses of the R_3S_4 minerals no attempt is made at placement in the group.

In addition to photographs with the Debye type cameras recorded in Table 2, precision photographs were taken in a symmetrical focusing camera of the type described by Jette and Foote (1935). The latter have been computed as set forth in a separate paper on the lattice constants written in cooperation with Mr. Ralph Holmes and Mrs. Margaret Knox.

	Locality		Per Cent		D (
Mineral		<i>a</i> ₀	Ni	Co	Keierence
Pyrite	Leadville, Colo.	5.40667 (1)	2.2	-	
Pyrite	Rio Morina, Elba	5,406	-		Peacock and Smith (1941)
Pyrite	Leadville, Colo.	5.408		—	Peacock and Smith (1941).
Nickelian pyrite	Denison mine	5.419	6.50		Peacock and Smith (1941).
Bravoite	Mill Close Mine	5.49	16.69		Bannister (1940).
Bravoite	Mechernich, Germany	5.57	24.73	3.28	de Jong and Willems (1927).
Vaesite	Kasompi, Katanga	5.66787(2)	41.24	3.41	
Artificial NiS ₂		5.74	45.6	-	de Jong and Willems
					(1927).
Cattierite	Shinkolobwe, Katanga	5.52346 (3)	1.53	40.6	
Artificial CoS ₂	_	5.64	_		de Jong and Willems
					(1927).
(1)	$\pm .00007$ (2)	$\pm.00008$	(3) $\pm .0$	0048

TABLE 1. SUMMARY OF LATTICE CONSTANTS

Series (Co, Ni, Fe)S₂

$(h^2+k^2+l^2)$	Miller Indices	1*	Pyrite (FeS ₂) d	Cattierite (CoS ₂) d	Vaesite (NiS ₂) d
3	(111)	84	3.080		3.238
4	(200)	100	2,673	2.750	2.809
5	(210)	60	2.394	2.463	2.515
6	(211)	48	2.186	2.249	2.298
8	(220)	34	1.896	1.950	1.992
9	(221)	40	1 11 -1	-	1.902
11	(311)	55	1.618	1.663	1,702
12	(222)	17	1.550	1.592	1.629
13	(230)	17	1.490	1.529	1.566
14	(231)	22	1.436	1.474	1.509
16	(400)	 .	-		
19	(331)	14	1.238	1.267	1.297
20	(420)	6	1.204	1.235	1.265
21	(421)	5	1.175	1.205	1.234
22	(332)	3	1.148	1.178	1.201
24	(242)	17	1.100	1.128	1.155
26	(150)				
27	(333)	55	1.038	1.063	1.080
29	(520)		1.002	1.026	1.041
30	(521)		.986	1.009	1.035
32	(440)	-		.977	1,002

TABLE 2. X-RAY DIFFRACTION MEASUREMENTS OF TWO NICKELIAN AND COBALTIAN MINERALS COMPARED WITH PYRITE

* Intensities on the basis of photo-cell measurements.

POLISHED SURFACES

The cobaltian mineral from Shinkolobwe forms a granular intergrowth with another sulfide, but it may be distinguished by a cubic cleavage and pinkish color. The associated sulfide lacks the cleavage and yields x-ray diffraction patterns of the spinel lattice type. According to qualitative tests it is a sulfide of Co, Ni, and Fe. It is believed to be a member of the linnaeite-polydymite series of the R_3S_4 type. The mineral is a pale corinthian pink of the color shown in the color dictionary of Maerz and Paul (Plate 3, 1B, 1930). The two sulfides occur in the dolomitic gangue.

The nickelian mineral from Kasompi occurs in crystals disseminated through dolomite. These are both octahedral and cubic in habit. The cleavage is cubic and on polished surfaces the color is gray. Specimens develop a coarse cleavage pattern. Associated with the nickelian mineral is a member of the linnaeite-polydymite series. The mineral yields qualitative tests for Co, Ni, and Fe, and is a metallic sulfide. According to x-ray diffraction patterns it has the spinel type of structure. On polished surfaces it is a pale corinthian pink but does not show cleavage. Presumably it represents a mineral of the R_3S_4 type. Small crystals of pyrite are also scattered through the ore. These are euhedral with a pyritodehral habit and may be isolated from the associated sulfides. The gangue material is chiefly dolomite. It is easily disintegrated with acid and small crystals of the nickelian sulfide may be isolated from the decomposed carbonate.

Photomicrographs of polished surfaces of the two minerals of the pyrite series are shown in Figs. 1 and 2. The cleavage of each is clearly shown in the area photographed.

Polished surfaces of both the mineral from Kasompi and the mineral from Shinkolobwe were tested electrochemically by the print method in order to confirm the distribution of cobalt, nickel, and iron throughout the area of the polished surface.

In the case of the Shinkolobwe material the minerals of the polished surface are too intergrown to make more than general application of the print method. Strong qualitative tests for cobalt and iron are furnished by most of the metallic area of the polished surface. Nickel is also broadly distributed but the test secured was not as strong.

The tests on the K.asompi material were less satisfactory due to the irregular distribution of the metallic constituent throughout the gangue. Crystals of the metallic minerals were found to be isolated in a number of instances and hence not in a position to complete the circuit. Also the dolomitic gangue was strongly reactive to the iron test while the Kasompi nickel mineral hardly reacted at all. The R₃S₄ mineral associated with the Kasompi nickel mineral reacted for iron.

On the whole the print tests merely confirm the other observations to the effect that qualitatively cobalt, nickel, and iron are probably present in each of the gray metallic minerals of the polished surfaces. The texture is not sufficiently coarse and well defined to employ the method to best advantage.

Nomenclature

The minerals under discussion furnish an interesting problem in nomenclature. The (RS_2) sulfides of pyritohedral symmetry with varying amounts of nickel, cobalt and iron have been described under the names pyrite, nickelian pyrite, and bravoite. The (R_3S_4) sulfides of the same elements with a lattice of the spinel type have been described under the names linnaeite, siegenite, polydymite, and violarite.



FIG. 1. Photomicrographs of a polished surface of vacsite NiS_2 from Kasompi showing well developed cubic cleavage resulting in both triangular and rectangular patterns. (Magnification $\times 180$.)

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FIG. 2. Photomicrographs of a polished surface of cattierite CoS_2 from Shinkolobwe showing irregularly developed cubic cleavage. An area free from cleavage in the upper view represents a member of the linnaeite-polydymite series. (Magnification $\times 180$.)

Where minerals belong to isomorphous groups, the distinction between species of like group structure is of necessity arbitrary. Palache, Berman, and Frondel (1944) in their review of the series NiS_2 -FeS₂ propose to apply the name *bravoite* only to members which contain more nickel than iron. Specimens containing less nickel than iron would be called nickelian pyrite. Under this system of nomenclature the original bravoite of Hillebrand (1907), as well as samples high in nickel described by Henglein (1914), referred to by Doelter and Leitmeier (1926) and mentioned by Bannister (1940), would no longer qualify as bravoite but would be known as nickelian pyrite. It is assumed that a parallel system of nomenclature would apply in the case of cobalt, and any member of the group with less cobalt than iron would be cobaltian pyrite. Presumably a member of the series with an excess of cobalt would also be called bravoite, although on this point the system to be applied is indefinite.

The usage suggested is somewhat at variance with the literature. In the analyses of Table 1, according to accepted usage, bravoite has been recognized in five well-established localities. The work of Hillebrand, de Jong and Willems, Buddington and Bannister has established bravoite as a mineral with definite properties, isostructural with pyrite but containing substantial amounts of nickel. At the same time through the work of Thomson and Allen, and Peacock and Smith nickelian pyrite with smaller amounts of nickel has been recognized. The line between the two has not been definitely established, but the usage of Bannister, a recent contributor, would class material on the order of 36.3 Ni to 63.7 Fe as bravoite and no objection is expressed to considering material with a lower nickel content as bravoite. Even an excess above 20 Ni to 80 Fe would have a significance which would warrant more than adjectival description, particularly when accompanied by a definite change in lattice constant and physical properties.

It is believed this report shows that NiS_2 and CoS_2 are more than artificial end-members. Minerals with compositions close to the theoretical values are now known. Under these circumstances a triangular relationship of the type illustrated in Fig. 3 seems more appropriate as a guide to the nomenclature.

 CoS_2 and NiS_2 each occupy a vertex of the diagram with FeS₂ occupying the third. Chemically the mineral from Shinkolobwe fits nicely into the cobalt corner. At the nickel corner the mineral from Kasompi corresponds as closely as one would expect a mineral to agree with the theoretical NiS₂. Thus there is good reason to seriously consider the claim of Mr. Vaes that the two minerals might properly be considered valid species.



FIG. 3. A triangular diagram indicating the relative proportions of nickel, cobalt, and iron in various members of the pyrite group.

- 1. Artificial CoS₂, von Setterberg (1826).
- 2. Bravoite, Minasragra, Peru, Hillebrand)1907).
- 3. Bravoite, Musen, Henglein (1914).
- 4. Artificial NiS₂, Fellenberg (1840).
- 5. Bravoite, Chitina, Alaska, Buddington (1924).
- 6. Bravoite, Mechernich, Kalb and Meyer (1926).
- 7. Nickelian pyrite, Mechernich, Kalb and Meyer (1926).
- 8. Artificial NiS₂, de Jong and Willems (1927).
- 9. Artificial CoS₂, de Jong and Willems (1927).
- 10. Nickelian pyrite, Denison Mine, Thomson and Allen (1939).
- 11. Bravoite, Mill Close Mine, Bannister (1940).
- 12. Cattierite, Shinkolobwe Mine, Ledoux and Co. (1944).
- 13. Cattierite, Shinkolobwe Mine, Vaes (1943).
- 14. Vaesite, Kasompi Mine, Ledoux and Co. (1944).
- 15. Vaesite, Kasompi Mine, Vaes (1943).

If one should arbitrarily truncate the species of the triangular diagram at a line corresponding to 80%, the end members would be clearly delineated. In the center of the diagram is a comparatively large area which seemingly contains too few examples as yet to justify subdivision. Since bravoite is well established and has been confirmed from several sources, it is suggested that this name be retained for the compositions represented by the entire central area of the triangle. Thus the RS₂ triangular domain would be divided into the three vertex areas in which occur pyrite, cattierite, and vaesite, while the entire interior would be bravoite.

If the same treatment is applied to the R_3S_4) group, which has the spinel structure, representatives among minerals may be found for the Ni and Co vertices. Here the Ni-vertex would appear to warrant the name polydymite and the Co-vertex the name linnaeite. Artificial Co_3S_4 with spinel structure has been made, but artificial Ni₃S₄ is yet to be confirmed. The Fe-vertex does not appear to be represented among minerals and artificial Fe₃S₄, although claimed, has not been confirmed. The large central area would contain the R-proportions for minerals previously described as siegenite, violarite, and linnaeite. This diagram is much less definite than the one for RS₂.

Nickelian Pyrite		Bravoite					
	Denison mine (10)	Mechernich (yellow) (7)	Mill Close mine (11)	Minasragra, Peru (recast) (2)	Musen, Germany (3)	Chitina, Alaska (5)	Mechernich (steel gray) (6)
Co	144			tr	10.6		3,28
Ni	6,50	4.40	16.69	18.23	11.7	24.81	24.73
Fe	34.54	38.02	29.30	29.46	22.8	20.68	17.08
Cu	1000	2.80					0.47
S	51.39	50.77	53.40	52.31	53.9	54.51	51.15
Insol.	3.80	2.50	04-14	<u></u>	0.7		0.40
		÷			<u></u>		1200
	96.23	98.49	99.39	100,00	99.7	100,00	97,11
Ni:Fe	15.8:84.2	10.5:89.5	36.3:63.7	38.2:61.8		54.6:45.4	
Co:Ni:Fe	-			·	23.5:26.0:50.5		7.3:54.9:37.

TABLE 3. CHEMICAL ANALYSES OF NICKELIAN PYRITE AND BRAVOITE

CHEMICAL COMPOSITION

Chemical analyses of cattierite and vaesite are shown in Table 4. One analysis of each mineral is substantially complete. Both confirm the cobaltian or nickelian nature of the two sulphides. The impurities for each are quartz, dolomite, calcite, mica, and chalcopyrite. The sulfides ana-

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lyzed were checked by x-ray diffraction patterns which yielded the pyrite type of structure. Additional lines due to other metallic minerals were not observed. The material analyzed by Ledoux and Co. was hand picked beneath a binocular microscope, grains showing an absence of

Cattierite Shinkolobwe mine		Vaesite Kasompi mine		
	Ledoux & Co. (1944)	Vaes (1943)	Ledoux & Co. (1944)	Vaes (1943)
	(12)	(13)	(14)	(15)
Co	31.60	41.40	2.82	_
Ni	1.19	3.20	32.93	44.58
Fe	3.40	3.27	1.85	0.95
Cu	.34	0.59	0.12	
S	42.25	51.32	42.61	51.22
SiO_2	.12	0.08	0.48	
Al_2O_3	0.52		0.28	
CaCO ₃	7.98	100	2.14	<u></u> 2
$MgCO_3$	7.63	3 -3	13.89	
	95.03	99.86	99.12	96.75

TABLE 4. CHEMICAL ANALYSES OF THE COBALTIAN AND NICKELIAN SULFIDES FROM KATANGA

TABLE 5. RECAST ANALYSES; CATTIERITE AND VAESITE

	Cattierite		Vaesite		
	(12)	(13)	(14)	(15)	
Со	40.60	42.20	3.41		
Ni	1.53	3.25	41.24	46.04	
Fe	3.97	2.80	2.20	.96	
S	53.90	51.75	53.15	53.00	
	100.00	100.00	100.00	100.00	
Computed Sp. Gr.	4.80		4.45		

cleavage being eliminated. Since both minerals show good cleavage this property was helpful in each case in making a separation. The material analyzed by Mr. Vaes was selected on the basis of examination on polished surfaces.

A recast of each analysis appears in Table 5. The nickelian mineral vaesite yields a comparative ratio of Co:Ni:Fe amounting to approxi-

mately 7.2:88.0:4.8 according to one analysis and —:98.0:2.0 according to the other. While it is not pure NiS₂ according to either analysis, it is close to the nickel apex of the triangle. The cobaltian mineral yields ratios of Co:Ni:Fe of approximately 88.1:3.8:8.6 and 87.5:6.7:5.8, according to the two analyses. Thus it would lie close to the cobalt apex of the triangle.

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CONCLUSION

On the basis of microscopic examination, chemical analysis, and x-ray study the two sulfides examined have been identified as lying close to NiS₂ and CoS₂ in the pyrite group. If the principle of nomenclature is adopted that the compositions corresponding to the apices of the triangular diagram are worthy of designation as separate mineral species, the nickelian and cobaltian disulfides would represent new minerals. The name bravoite would apply to the intermediate compositions. The boundary between bravoite and the end member in each case is placed at eighty per cent. Although arbitrary, this boundary lies close to established usage in distinguishing between bravoite and pyrite. In the light of this study the pyrite group forms an isostructural series with pyrite FeS₂, vaesite NiS₂ and cattierite CoS₂ as end members, while bravoite (Co, Ni, Fe)S₂ is intermediate.

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