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PARKERITE (Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>) FROM SUDBURY, ONTARIO: REDEFINITION OF THE SPECIES<sup>1</sup>

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#### Abstract

Parkerite is orthorhombic with the probable space-group  $Pmm 2-C_{2s}^{1}$ . The unit cell with a=4.02, b=5.52, c=5.72 Å, contains Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>. Found only in grains and cleavage fragments, tabular on (001) and striated or stepped parallel to [110] by prevalent lamellar twinning on (111). Cleavage (001), perfect. Parting (111). Fracture uneven. Brittle. Hardness 3 (B+). Specific gravity 8.4 (meas.), 8.50 (calc.). Colour, bright bronze, tarnishing darker. Lustre metallic. Streak black, shining. Opaque. Non-magnetic. Good electrical conductor. Polished sections light cream coloured, noticeably pleochroic, strongly anisotropic, showing lamellar twinning. Composition Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>, requiring Ni 26.7, Bi 63.6, S.9.7 = 100. Analysis: Ni 26.8, Bi 63.6, S.9.2, Pb trace=99.6. Soluble in concentrated HNO<sub>3</sub>. Strongest *x*-ray powder lines: (10) 2.85, (9) 2.33, (7) 1.645 Å. Occurs sparingly embedded with galena, sulphides, arsenides, and tellurides, in the extremities of one of the ore-bodies in the Sudbury district, Ontario. Obtained in crystalline masses by fusing the elements in nitrogen or in a vacuum.

Although the original description of parkerite from South Africa (Scholtz, 1936) is too indefinite to permit an exact comparison, the Sudbury mineral can be identified with parkerite on the basis of x-ray powder data.

A recent study of the ore minerals of the Sudbury area (Michener, 1940) revealed more than thirty species containing a considerable variety of metallic elements. Of particular interest was the prevalence of the element bismuth which enters the composition of several of the observed minerals. One of these bismuth minerals was noticed in polished sections as a pale cream coloured mineral with strong anisotropism and striking lamellar twinning in almost every section. On further study this mineral proved to be a nickel bismuth sulphide which could not be identified with any described species.

The unknown mineral was later received by the second author for

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further examination. As the work progressed it was noted at Copper Cliff that the unknown mineral might be parkerite (Scholtz, 1936), an imperfectly known mineral tentatively described as a new nickel sulphide from the nickel ores of South Africa. A closer comparison of the properties of the Sudbury mineral with the description of parkerite, to be given later, showed that there can be no reasonable doubt of the identity of the two minerals, even though our mineral contains 63.6 per cent of bismuth which appears to have been overlooked in parkerite. Although the introduction of a new name might be justified under these circumstances we have decided to retain the name parkerite and offer the present account as a redefinition of the species.

This joint work was done in co-operation with Dr. A. B. Yates, Chief Geologist, and Dr. G. A. Harcourt, Acting Superintendent of Research, of The International Nickel Company at Copper Cliff, Ontario. Dr. F. G. Smith, formerly at the University of Toronto and now at the Geophysical Laboratory, Washington, D. C., made an important contribution to the work done in Toronto, by preparing synthetic parkerite which not only provided abundant material to compare with the scanty mineral, but indicated the exact composition which was subsequently confirmed by a new analysis of the mineral. The mineral was recognized and isolated at Copper Cliff where the physical and chemical properties were determined; the crystallographic and synthetic work and some confirmatory and supplementary observations were carried out in Toronto, where the present account was prepared from the assembled information. In this way we have obtained a full description which is largely verified by independent observations.

Since natural and artificial parkerite are alike in all their specific properties it will be of interest to give parallel descriptions of the two materials before giving an account of the preparation of the synthetic compound.

#### Occurrence

Parkerite is found in small stringers and veinlets in the extremities of the ore-body in one of the Sudbury mines,<sup>2</sup> where it occurs sparingly in association with galena, native bismuth, bismuthinite, tetradymite, hessite, cubanite, maucherite, niccolite, sperrylite, and gold, together with the prevailing chalcopyrite, pyrrhotite, and pentlandite. The association with galena is particularly persistent and characteristic. As a group these minerals occur around the margins of the main copper-nickel-iron sulphides; parkerite has not been found in the central part of any of the larger sulphide masses.

<sup>2</sup> Under present conditions we are not permitted to define the locality more closely.

# PREPARATION OF MATERIAL

Parkerite occurs in grains rarely larger than 1 mm. in diameter. A sample of the mineral was obtained by a combination of methods. Samples of the ore containing parkerite were crushed and sized to a 65–100 mesh fraction and concentrated on the Haultain superpanner, which gave a product consisting largely of galena, with parkerite, pyrrhotite, and chalcopyrite. From its behaviour on the superpanner parkerite appeared to have a specific gravity similar to that of galena. Most of the galena was removed from this concentrate by depressing it in a flotation cell; the magnetic pyrrhotite was taken out with a magnetic separator; and the chalcopyrite and gangue were separated with Clerici solution. The concentrate thus obtained consisted largely of parkerite, with some galena, non-magnetic pyrrhotite, niccolite, and pentlandite. These impurities were removed with dilute nitric acid in which parkerite is insoluble. The resulting concentrate, still showing grains of gold and sperrylite, was finally cleaned by hand.

## PHYSICAL PROPERTIES

Single grains of parkerite (Figs. 1 and 3) are bright bronze coloured with brilliant metallic lustre on fresh fractures, becoming darker and dull on tarnished surfaces. There is an eminent cleavage in one direction and a good parting parallel to the twin lamellae which intersect the perfect cleavage in one or two directions, giving plates bounded by parallel edges or rhombic outlines. The mineral is brittle, with an uneven fracture, and it gives a black shining streak, barely marking paper. The hardness is 3 and the specific gravity is 8.4, measured by F. G. Smith with the Berman balance, on a few milligrams of selected grains. The mineral is nonmagnetic and a good conductor of electricity.

Fragments of artificial parkerite (Fig. 2) are slightly brighter and more sharply crystallized than the natural material but otherwise indistinguishable from the mineral. On larger samples the specific gravity is 8.44 (Smith), 8.43 (Peacock).

In polished sections (Fig. 4) parkerite appears in shapeless areas with a light cream colour that contrasts sharply with that of adjoining galena. Reflection pleochroism is perceptible and the anisotropism is strong, greenish-grey to yellowish-brown, even slate-blue to salmon-pink, depending on the adjustment of the nicols. Crossed nicols almost invariably show strong twin lamellae in one direction, usually traversed obliquely by weaker lamellae. The hardness is B+, as indicated by a needlescratch traversing galena, parkerite, and chalcopyrite, under uniform pressure. Etch reactions: HNO<sub>3</sub>, blackens instantly; HCl, negative;

KCN, negative; FeCl<sub>3</sub>, blackens instantly; KOH, negative; HgCl<sub>2</sub>, stains iridescent.

A polished section of the artificial product (Fig. 5) shows a coarse mozaic of grains differing in no significant way from those of the mineral. The twin lamellae in the synthetic product are sharp and narrow and the second direction is almost equally well developed, indicating that the two directions may be crystallographically equivalent.



FIGS. 1, 2. Parkerite: fragments of natural and artificial crystals striated or stepped in one or two directions by lamellar twinning. Inclined illumination and equal magnification; the largest fragment is 1.4 mm. long. Fig. 1 (left), natural fragments. Fig. 2 (right), artificial fragments.



FIG. 3. Parkerite: natural fragments. Ring illumination; the larger fragment is 0.9 mm. long.

## **CRYSTALLOGRAPHIC** PROPERTIES

Unit cell. For the determination of the symmetry and lattice dimensions of parkerite the following x-ray photographs were taken on a

# PARKERITE FROM SUDBURY, ONTARIO



FIGS. 4, 5. Parkerite: polished sections of natural and artificial materials showing strong anisotropism and twin lamellae in two directions. Vertical illumination, crossed nicols, and equal magnification; each field is  $1.17 \times 0.73$  mm. Fig. 4 (left), natural material. Fig. 5 (right), artificial material.

cleavage fragment (0.2 mm.) of the mineral, with copper radiation: rotation about the axis [ $\overline{1}10$ ] (edge between the perfect cleavage and the parting), and Weissenberg resolutions of the zero, first, and second layerlines; rotation about the axis [001] (normal to the perfect cleavage), and Weissenberg resolution of the zero layer-line. A series of single crystal photographs were also taken on a cleavage fragment of the artificial compound: rotation about [001] and Weissenberg resolutions of the zero, first, and second layer-lines; and rotation about [100] with Weissenberg resolution of the zero layer-line.

Corresponding photographs of the natural and artificial compounds are alike in the positions and relative intensities of the spots. The films taken with [001] as the rotation axis show orthorhombic symmetry and measurements on the films from the mineral fragment give the cell dimensions:

a = 4.02, b = 5.52, c = 5.72 Å

which yield the ratios:

a:b:c=0.7283:1:1.0362.

This mineral is naturally oriented with the perfect cleavage as the basal plane; in this position the axis normal to the perfect cleavage, which is probably polar, is conveniently vertical.

Diffraction spots due to the twin lamellae were recognized by their slight progressive displacement and lack of orthorhombic symmetry



FIG. 6. Parkerite: stereographic projection of c(001) and p(111) twinned by reflection in (111).

FIGS. 7, 8, 9. Parkerite: cleavage fragments of the artificial material, with cleavage c(001) and twinning and parting on p(111). The largest fragment is 0.8 mm. long. Figs. 7, 9, plan on (001). Fig. 8, plan on the plane normal to [I10].

with reference to the axes of the main crystal. A general condition of spectral omission, (hkl) present only with (k+l) even, is approached but not strictly realized except in the zone [001], where (hk0) is present only with k even. These conditions do not serve to fix the space-group, but a study of the atomic arrangement, now in progress, indicates a structure with the symmetry of the space-group  $Pmm2-C_{2n}^{1}$  (pyramidal class-mm2).

The unit cell of parkerite contains one molecule of  $Ni_3Bi_2S_2$ , giving the calculated density 8.50, as compared with 8.4 measured on the mineral and 8.44 measured on the artificial compound.

Cleavage, parting, twinning. Fragments of natural and artificial parkerite are all twinned intergrowths consisting of tablets bounded by the eminent basal cleavage and traversed by twin lamellae which are parallel to planes of the form (111) of the main tablet. The lamellae cut the basal plane in the directions [ $\overline{110}$ ] and [110] which make the calculated angle 72°08', as compared with 72 $\frac{1}{2}$ ° measured under the microscope on a plate of the mineral, 72°17' measured with the goniometer on a plate of the artificial compound. The basal plates are usually bevelled by one or more planes which are parallel or nearly parallel to planes of the form (111). These planes proved to be either planes of parting parallel to the twin lamellae, or planes of the basal cleavage on twinned parts of the intergrowth. The calculated angle (001):(111) is 60°24', as compared to 60°02', 60°32', measured on natural fragments, 60°24', 60°30', measured on artificial plates. The calculated angle (001):(001) twinned is 59°12' which compares with 59°20' measured on the mineral, 59°14'-59°17' noted on the synthetic compound.

Figure 6 gives a stereographic projection of the cleavage c(001) and the parting p(111) of parkerite, twinned by reflection in the plane (111) whose trace is marked *TTP*. The projection shows that  $p(\overline{111})$  in twin position is nearly parallel to c(001) in normal position, and vice versa. Professor J. D. H. Donnay has been good enough to send us a mathematical treatment of the twin law in the form developed by Friedel (1926, p. 245). According to this discussion the twin law is an example of twinning by reticular pseudomerohedry, with index 2 and obliquity  $1^{\circ}35\frac{1}{2}'$ . The prevalence of the twinning on this law is explained and predicted by the small values of the index and the obliquity.

Figures 7, 8, 9 show typical fragments of artificial parkerite with twin lamellae parallel to p(111) and  $p(\overline{1}11)$ . Similar but less perfect forms were found among the natural fragments. No case was noted of twinning on three or four of the equivalent planes in the orthorhombic holohedry and no certain indication of the crystal class was obtained from the morphology.

# CHEMICAL PROPERTIES

Parkerite is completely soluble in concentrated HNO<sub>3</sub> but is insoluble in HCl. A spectrographic analysis showed the presence of Ni and Bi, with slight traces of Cu and Sn; not present: Fe, Mn, W, Ti, Sc, Co, Pd, Ag, Cd, Ir, Pt, Te, As, Sb. S was revealed by a microchemical test. Quantitative analyses on small picked samples gave the values in Table 1. These were obtained by first estimating Ni (by dimethylglyoxime) and Bi (by electrolysis) on a sample of the artificial product. The values thus obtained were used to standardize the dithizone method for Ni and Bi, which was then applied to a sample of the mineral weighing 2.75 mg. S was determined on a separate sample of 30 mg.

As shown in Table 1 parkerite and the synthetic compound conform closely to the composition Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>, which appears to be a new type of mineral composition. The formula shows a resemblance to the spinel

TABLE 1					
	1	2	3		
Ni	26.8	27.42	26.7		
Bi	63.6	64.00	63.6		
S	9.2		9.7		
РЪ	trace				
			() <del></del> )		
	99.6		100.0		

1. Parkerite, Sudbury, Ontario. Microanalysis by W. Wagner.

2. Artificial parkerite. Partial analysis by W. Wagner.

3. Percentage composition of Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>.

formula, RO · R<sub>2</sub>O<sub>3</sub>, but there is no suggestion of a structural relationship to this cubic type. Ullmannite (NiSbS) is also cubic, and kallilite, to which Dana (1892, p. 1039) gave the hypothetical formula NiBiS, is merely a bismuthian ullmannite (Peacock and Berry, 1940, p. 60) with the composition Ni(Sb,Bi)S. With no close mineral relatives parkerite might be appended to the Arsenopyrite Group in a system of descriptive mineralogy.

# X-RAY POWDER DATA

Parkerite gives a distinctive x-ray powder pattern which is shown in the films from natural and artificial materials reproduced without reduction in Figs. 10, 11. Table 2 gives for each line the relative intensity I estimated visually<sup>3</sup> on a film from the natural material; the measured glancing angle  $\theta(Cu)$ ; the corresponding measured interplanar spacing d (meas.); the indices (hkl) of the reflecting lattice planes; and the calculated spacing of the reflecting planes d (calc.). For determinative purposes the columns I and d (meas.) suffice (Harcourt, 1942); the addition of  $\theta$  is a convenience, while (*hkl*) and *d* (calc.) give the desirable verification of a standard powder pattern (Peacock, 1941).

The powder photographs (Figs. 10, 11) were made in Toronto with equipment which appears to give better than ordinary resolution. On films made for rapid determinative purposes some of our slightly separated lines may coalesce and some of our faint lines may not appear.

<sup>3</sup>We estimated the intensities independently with differences which only rarely amounted to 2 degrees on a 10 degree scale.

# TABLE 2. PARKERITE: X-RAY POWDER SPECTRUM (CU/NI)

I	θ (Cu)	d (meas.)	(hkl)	d (calc.)
4	7.6°	5.8 Å	(001)	5.72 Å
7	11 05	4 01	∫(100)	4.02
,	11.05	1.01	$\left( \begin{array}{c} (011) \\ (101) \end{array} \right)$	3.97
1	13.5	3.29	(101)	3.29
10	15.65	2.85	(002)	2.80
1	17 5	2 56	(012)	2.54
0	19.25	2.33	(102)	2.33
4	19.7	2.28	(120)	2.28
12	20.9	2.15	(112)	2.15
12	21.3	2.12	(121)	2.11
5	22.4	2.02	(200)	2.01
5	22.8	1.984	(022)	1.986
12	23.9	1.897	(201)	1.890
0	25.25	1.802	(013)	1.002
2	25.55	1.782	(122) (103)	1 723
2	20.5	1.725	(113)	1.645
7 .	27.85	1.645	(202)	1.645
1	28.5	1.611	(131)	1.606
4	32.5	1.431	(004)	1.430
5	32.9	1.415	(222)	1.413
1	22 7	1 385	$\int (014)$	1.384
2	55.7	1,000	(203)	1.383
3	34.85	1.345	$\int (104)$	1.347
1	01.00 05 5	1 204	(213)	1.342
2	35.5	1.324	((311)	1.324
2	37.25	1.270	(024)	1.270
			((302))	1,213
5	39.4	1.211	(124)	1.211
1	40.4	1.186	(312)	1.185
2	41.3	1.165	(141)	1.173
1	42.4	1.140	(214)	1.140
12	43.4	1.119	(015)	1.120
4	45.5	1.078	(115)	1.079
2	51.8	0.978	(215)	0.979
			((304)	0.978
1	54 5	0 944	(135)	0.944
1	J1.J	0.711	(333)	0.942
1/2	56.0	0.927	(106)	0.928
12	56.4	0.923	(324)	0.922
12	57.3	0.913	(116)	0.915
1	58.6	0.901	(026)	0.901
1	61.0	0.879	(126)	0.8/9
2	0110		((413)	0.875
1	61 6	0.874	(054)	0.874
2	01.0	0.0/4	(342)	0.873
		0.044	(206)	0.862
12	63.2	0.861	(315)	0.860
C	(0.0	0 012	J(226)	0.823
1	09.0	11.87.0	5 (101)	0 000

# Orthorhombic; a = 4.02, b = 5.52, c = 5.72 Å

#### Syntheses

The compound  $Ni_3Bi_2S_2$  was obtained in pure, coarsely crystallized masses by fusing the elements in the proper proportions in a sealed pyrex tube in which the air had been displaced by nitrogen at low pressure (Smith), or entirely removed (Burr). On gentle heating with an open



FIGS. 10, 11. Parkerite: x-ray powder photographs of natural and artificial materials with Cu/Ni radiation. Full size reproductions of contact prints (1 mm. on film=1°  $\theta$ ). Fig. 10, natural material. Fig. 11, artificial material.

flame the charge combined vigorously, giving a liquid which crystallized with the liberation of much heat after the flame was withdrawn. When broken in halves the product was found to be a solid crystalline aggregate showing brilliant continuous cleavage surfaces up to 5 mm. in width. A polished section showed that the product was pure and homogeneous.

This satisfactory result was not, however, immediately obtained, since the first purpose of the synthetic work was to check a simpler but, as it proved, incorrect formula, NiBiS, suggested by preliminary analyses. The product of a charge with this composition proved to be a mixture of parkerite, bismuthinite, and bismuth, representing the reaction:

9 (Ni+Bi+S) $\rightarrow$ 3Ni<sub>3</sub>Bi<sub>2</sub>S<sub>2</sub>+Bi<sub>2</sub>S<sub>3</sub>+Bi.

This showed that parkerite contains relatively more Ni than NiBiS, and, incidentally, that a Bi-analogue of ullmannite probably cannot exist.

With this clue, the proper composition of parkerite was eventually found by preparing charges with the limited number of simple rational compositions permitted by the then roughly known molecular weight of the cell content and the approximate composition. Only the composition  $Ni_3Bi_2S_2$  gave pure parkerite; the others showed substantial amounts of impurities. Special care was taken to exclude the composition  $Ni_4Bi_2S_2$ which gave a product consisting of parkerite together with a white magnetic impurity resembling nickel.

# PARKERITE FROM SUDBURY, ONTARIO

It is noteworthy that the powder pattern of parkerite given by various impure products showed only very slight displacements of the lines as compared with the pattern of pure  $Ni_3Bi_2S_2$ . This showed that the compound has a very limited solid solution range.

After these experiments had been completed, and the foregoing account written, we found a reference to a published equilibrium study by Schenck and Forst (1939) bearing directly on our work. Using the established method of isothermal reduction with hydrogen, these authors studied the systems Sb-Ni-S and Bi-Ni-S. In the former system they found NiSbS (ullmannite) as the only ternary compound; in the latter system they unexpectedly found  $Ni_3Bi_2S_2$  as the only ternary compound, with only a slight indication that NiBiS might appear as a metastable phase.

# Identity of the Sudbury Mineral and Parkerite of Scholtz

The name parkerite was given by Scholtz (1936, p. 186) to one of sixteen unidentified ore minerals found in the nickeliferous deposits of East Griqualand and Pondoland, South Africa. Limited material from Waterfall Gorge, Insizwa, studied in Zürich with the help of Professors Niggli and Parker, led to a description which may be condensed as follows:

Symmetry probably monoclinic, as indicated by a Laue photograph (Niggli). Composition  $Ni_2S_3$  or  $NiS_2$ , suggested by a partial analysis (Schilz) of a few milligrams. Cleavage in three directions, one easier than the others, giving rhomboidal plates. Polished sections creamy white, with a faint mauvish tinge, noticeably pleochroic and strongly anisotropic, always showing lamellar twinning which causes zigzag traces in sections across the best cleavage. Hardness apparently slightly less than that of galena. Negative etch-reactions only to KCN and KOH. Soluble in 50 per cent HNO<sub>3</sub>. Associated with galena; chalcopyrite, maucherite, sperrylite, pentlandite, niccolite, bismuth, and silicates.

Although this description lacks precision in almost all the specific characters, there is a suggestive similarity between the described appearance and associations of parkerite and those of the Sudbury mineral. However, the identity of our mineral with parkerite might not have been suspected, and certainly could not have been proved, without the *x*-ray powder data (Niggli) which is appended to the description of parkerite. Table 3 compares Niggli's intensities and spacings for the South African mineral with our data for the Canadian occurrence.

It will be seen that there is substantial agreement in the relative intensities and absolute spacings in the two lists. Only five of Niggli's lines do not appear in our list. Since Niggli noted that there might be lines in his pattern due to impurities, the source of these extra lines was sought with the help of Harcourt's *Tables* (1942). Three of the extra lines  $(d \ 3.02, \ 1.854, \ 1.5905)$  were at once identified with the three strongest lines of chalcopyrite  $(d \ 3.03, \ 1.86, \ 1.59)$ , leaving only two lines unidentified. Seven of our lines are missing in Niggli's list, but these are all very

Insizwa <sup>4</sup>		Sudbury		Insizwa <sup>4</sup>		Sudbury	
I	d(Å)	I	<i>d</i> (Å)	I	d(Å)	Ι	$d(\text{\AA})$
m	5.66	4	5.8	m	1.413	5	1.415
m	3.96	7	4.01	wb	1.376	$\frac{1}{2}$	1.385
		1	3.29	m	1.343(5)	3	1.345
m	3.02 cp			w	1.322	12	1.324
S	2.84	10	2.85	mwb	1.270	2	1.270
		1	2.56	S	1.209(5)	5	1.211
ms	2.32	9	2.33	w	1.187	1	1.186
w	2.27(5)	4	2.28	mw	1.165	2	1.165
		$\frac{1}{2}$	2.15	w	1.140	1	1.140
		12	2.12	w	1.115(5)	12	1.119
mw	2.013	5	2.02	s	1.075	$\frac{1}{4}$	1.078
m	1.979	5	1.984	m	0.9788	2	0.978
w	1.897	$\frac{1}{2}$	1.897	wb	0.9440	1	0.944
mw	1.854 cp					1	0.927
m	1.792	6	1.802	m	0.9240	10	0.923
w	1.782	2	1.782			1	0.913
w	1.714	12	1.723	m	0.8987	1	0.901
s	1.640	7	1.645	mw	0.8912	******	
w	1.606	$\frac{1}{2}$	1.611			12	0.879
mw	1.590(5) cp			mw	0.8755	1/2	0.874
w	1.568			w	0.8589	12	0.861
S	1.427	4	1.431	mwb	0.8217	2	0.823

TABLE 3. PARKERITE: COMPARISON OF X-RAY POWDER DATA (CU/NI)

<sup>4</sup> Niggli, in Scholtz (1936, p. 188). Intensities: w, weak; m, medium; s, strong; b, broad line. *cp*, line due to chalcopyrite.

weak. The agreement is in fact better than is usually obtained on one and the same mineral by different observers using different appliances, and thus the minerals from Canada and South Africa represent a single mineral species. Lack of orthorhombic symmetry in a Laue photograph of a basal plate of parkerite would be expected, since the ever-present twin lamellae would give unsymmetrical reflections. The serious discrepancy in the chemical composition suggested by Scholtz can be attributed to lack of sufficient material for adequate chemical study.

#### PARKERITE FROM SUDBURY, ONTARIO

When these conclusions were reached (November, 1941) we wrote Dr. Scholtz (Pretoria) and Professor Parker (Zürich) outlining our results and suggesting a test for essential Bi in parkerite. Up to the present time (March, 1943) we have had no reply and therefore the verification of the composition  $Ni_3Bi_2S_2$  on the South African mineral must be left to the future.

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