

## Naldrettite, Pd<sub>2</sub>Sb, a new intermetallic mineral from the Mesamax Northwest deposit, Ungava region, Québec, Canada

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### ABSTRACT

Naldrettite, Pd<sub>2</sub>Sb, is a new intermetallic mineral discovered in the Mesamax Northwest deposit, Cape Smith fold belt, Ungava region, northern Québec. It is associated with monoclinic pyrrhotite, pentlandite, chalcopyrite, galena, sphalerite, cobaltite, clinochlore, magnetite, sudburyite (PdSb), electrum and altaite. Other rarer associated minerals include a second new mineral (ungavaite, Pd<sub>4</sub>Sb<sub>3</sub>), sperrylite (PtAs<sub>2</sub>), michenerite (PdBiTe), petzite (Ag<sub>3</sub>AuTe<sub>4</sub>) and hessite (Ag<sub>2</sub>Te). Naldrettite occurs as anhedral grains, which are commonly attached or moulded to sulphide minerals, and also associated with clinochlore. Grains of naldrettite vary in size (equivalent circle diameter) from ~10 to 239 μm, with an average of 74.4 μm ( $n = 632$ ). Cleavage was not observed and fracture is irregular. The mineral has a mean micro-indentation hardness of 393 kg/mm<sup>2</sup>. It is distinctly anisotropic, non-pleochroic, has weak bireflectance, and does not exhibit discernible internal reflections. Some grains display evidence of strain-induced polysynthetic twinning. Naldrettite appears bright creamy white in association with pentlandite, pyrrhotite, clinochlore and chalcopyrite. Reflectance values in air (and in oil) for  $R_1$  and  $R_2$  are: 49.0, 50.9 (35.9, 37.6) at 470 nm, 53.2, 55.1 (40.3, 42.1) at 546 nm, 55.4, 57.5 (42.5, 44.3) at 589 nm and 58.5, 60.1 (45.4, 47.2) at 650 nm. The average of 69 electron-microprobe analyses on 19 particles gives: Pd 63.49, Fe 0.11, Sb 35.75, As 0.31, and S 0.02, total 99.68 wt.%, corresponding to  $(\text{Pd}_{1.995}\text{Fe}_{0.007})_{2.002}(\text{Sb}_{0.982}\text{As}_{0.014}\text{S}_{0.002})_{0.998}$ . The mineral is orthorhombic, space group  $Cmc2_1$ ,  $a$  3.3906(1),  $b$  17.5551(5),  $c$  6.957(2) Å,  $V$  414.097(3) Å<sup>3</sup>,  $Z = 8$ .  $D_{\text{calc}}$  is 10.694(1) g/cm<sup>3</sup>. The six strongest lines in the X-ray powder-diffraction pattern [ $d$  in Å ( $I$ )( $hkl$ )] are: 2.2454(100)(132), 2.0567(52)(043), 2.0009(40)(152), 1.2842(42)(115), 1.2122(50)(204) and 0.8584(56)(1.17.4).

**KEYWORDS:** PGE, PGM, naldrettite, Pd-Sb, new intermetallic mineral species, platinum-group elements, platinum-group minerals, hydroseparation, Mesamax Northwest deposit, Cape Smith fold belt, Québec, Ungava, Canada.

### Introduction

NALDRETTITE, Pd<sub>2</sub>Sb, (НАЛДРЕТИТ [naldrettit]) is a new mineral discovered in drill-core rejects

(MXNW 02-50) from the Mesamax Northwest deposit, Cape Smith fold belt of northern Québec, Ungava region, Canada at N61°34'25" and W73°15'36". Non-magnetic and magnetic heavy mineral concentrates of sample 196406 (0.563 kg) were subjected to heavy mineral separation using a HS-02 Hydroseparator (Rudashevsky *et al.*, 2001, 2002; Cabri, 2003, 2004). More than 1000

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grains of precious metal minerals were found in these heavy mineral concentrates, the majority being naldrettite.

The name is for Professor Anthony J. Naldrett (b. 1933), University of Toronto, who has made significant contributions to our understanding of the genesis of platinum-group element (PGE) deposits, and has served as President of both the Mineralogical Association of Canada and the International Mineralogical Association (IMA). Both the mineral data and mineral name were approved (by a vote of 23 for, none against, and no abstentions) by the Commission on New Minerals and Mineral Names (CNMMN) of the IMA (IMA 2004-007). Holotype material is catalogued in the collections of the Natural History Museum, Cromwell Road, London SW7 5BD, UK (polished section; catalogue no BM 2004,34) and the Canadian Museum of Nature, Ottawa (polished section; catalogue no. CNMMN 84397).

The Mesamax Northwest deposit was first identified as a gossan in the 1960s. In 1970 a series of diamond drill-holes revealed the presence of a body of disseminated to net-textured sulphide mineralization hosted by amphibolite and serpentinite replacing pyroxenite and peridotite. In 2002, Canadian Royalties Inc. tested a prominent geophysical anomaly at the site and intersected over 50 m of massive sulphide in suboutcrop under several metres of overburden. Subsequent drilling has delineated an indicated resource of 1.84 Mt grading 1.9% Ni, 2.3% Cu, 0.08% Co, 0.9 g/t Pt, 4.3 g/t Pd and 0.3 g/t Au (Canadian Royalties Inc., press release, 10 June, 2004).

The host rocks comprise a portion of a regionally-extensive swarm of internally differentiated dykes of komatiitic basalt and basaltic komatiite up to several hundred metres thick. Mineralization is present at dyke margins and along the keel-shaped lower extremities of the blade-shaped dykes. The Mesamax Northwest body was formed at an irregularity in the basal portion of a dyke, where it intersected a bed of silicate-facies iron formation ~20 m thick. Mineralization is largely confined to a broadening of the dyke within the partially assimilated iron formation.

### Occurrence and associated minerals

The sample in which the new mineral was discovered had a very high PGE concentration,

with a notable Pd enrichment, and >1000 platinum-group mineral (PGM) particles were concentrated by hydroseparation. The number of PGM actually measured was limited to 791 because these were more than adequate to characterize the sample mineralogically. In addition, only PGM in non-magnetic fractions (<45, 45–75, 75–125, 125–180 and 180–350 µm) and one magnetic fraction (75–125 µm) were examined in detail because sufficient PGM had been measured to characterize the sample. The PGM in the magnetic fraction were not attached to magnetite, but had been entrained in the magnetic concentrate.

The precious metal minerals are dominated by Pd antimonides, primarily naldrettite, which is the dominant PGM in non-magnetic fractions (where it represents 87% of the total PGM observed) and is the only PGM found in one section of magnetic concentrate. None of the 10 particles of naldrettite in the magnetic fraction was attached to magnetite; however, a single grain of magnetite in the non-magnetic fraction was attached to naldrettite. Other PGM and precious-metal-bearing minerals are present (both as inclusions and discrete grains) and include 67 particles of sudburyite, PdSb, (9% of PGM), the new mineral ungavaite (Pd<sub>4</sub>Sb<sub>3</sub>, McDonald *et al.*, submitted), sperrylite (PtAs<sub>2</sub>), and michenerite (PdBiTe), as well as 67 particles of electrum (8.5% of all precious metal minerals) and two particles each of the tellurides petzite (Ag<sub>3</sub>AuTe<sub>4</sub>) and hessite (Ag<sub>2</sub>Te). Sudburyite has variable contents of Sb, Te and Bi, with an average ( $n = 14$ ) composition of Pd<sub>0.99</sub>(Sb<sub>0.64</sub>Te<sub>0.22</sub>Bi<sub>0.22</sub>)Σ<sub>1.08</sub>. The electrum is silver-rich (Au<sub>1.02</sub>Ag<sub>0.98</sub>), based on the average result of 10 analyses by energy dispersive spectrometry (EDS).

The common base metal sulphides (BMS) comprise pyrrhotite, pentlandite, chalcopyrite, galena, sphalerite and cobaltite. Altaite (PbTe) sometimes also occurs when sudburyite is present. The pyrrhotite is considered to be monoclinic in symmetry since the average of seven analyses by EDS corresponds to (Fe<sub>6.95</sub>Ni<sub>0.04</sub>)Σ<sub>6.99</sub>S<sub>8.01</sub>, and the Ni content averages 0.4 wt.%, with a range from 0 to 0.6 wt.% Ni. Pentlandite has Ni>Fe, with an average composition of (Ni<sub>4.74</sub>Fe<sub>4.24</sub>Co<sub>0.35</sub>)Σ<sub>9.33</sub>S<sub>8.09</sub>, based on the average result of seven analyses by EDS. The average of three EDS analyses of cobaltite gives (Co<sub>0.76</sub>Ni<sub>0.16</sub>Fe<sub>0.06</sub>)Σ<sub>0.98</sub>As<sub>0.95</sub>S<sub>1.06</sub>. One analysis of sphalerite by EDS gave 6.8 wt.% Fe.

### Physical and optical properties

In the concentrates, naldrettite occurs as liberated anhedral grains (Fig. 1*b,f,h,l*), but is also often found attached to or moulded on sulphide minerals (Fig. 1*a,c,d*), and commonly associated with clinocllore (Fig. 1*e,g,i,j,k,m*), rarely with magnetite (Fig. 1*n*). Grains of naldrettite vary in size (equivalent circle diameter) from ~10 to 239 μm, with an average of 74.4 μm (*n* = 632). The mineral is metallic, opaque and the macroscopic colour and streak were not examined. Cleavage was not observed and the fracture is irregular. Deformation induced by indentation suggests almost ductile behaviour, certainly flexibly inelastic. The mineral has a micro-indentation hardness VHN<sub>50</sub> of 393 (*n* = 10 indentations on five grains; range of 358–418 kg/mm<sup>2</sup>), which corresponds to a Mohs hardness of 4 to 5.

In plane-polarized reflected light, naldrettite appears bright creamy white in association with pentlandite, pyrrhotite, chalcopyrite and clinocllore, and shows no discernible internal reflections. It has a higher reflectance and is less yellow than pentlandite. The mineral shows weak bireflectance, no pleochroism, and distinct aniso-

tropy, with rotation tints that are deep bright blue, lemon-buff, and mauve to pale pink. Some grains display evidence of what is considered to be strain-induced polysynthetic twinning. Reflectance was measured (*n*<sub>oil</sub> 1.515, DIN 58.884 at 20°C) relative to a WTiC standard (Zeiss 314), following the methodology of Stanley *et al.* (2002). The reflectance data and colour values for the naldrettite grain that was analyzed (#193) are given in Tables 1 and 2.

### Chemical composition

Chemical analyses originally carried out on seven particles of naldrettite using energy dispersion spectrometry (EDS) and a Camscan Microspec-4DV scanning electron microscope with a Link AN-10 000 detector, gave essentially a stoichiometric composition. The operating conditions included an accelerating beam voltage of 30 kV, a beam current of 1–2 nA, a beam diameter of 1 μm and counting times of 50–100 s. Electron microprobe analyses (WDS) were later carried out using a JEOL 8900 instrument, an accelerating beam voltage of 20 kV, a beam current of 20 nA, a beam diameter of 5 μm and counting times of 50–100 s. The mean analytical results on 19 different particles in polished sections (<45 μm, 75–125 μm) are given in Table 3. The empirical formula (based on three atoms) is: (Pd<sub>1.995</sub>Fe<sub>0.007</sub>)Σ2.002(Sb<sub>0.982</sub>As<sub>0.014</sub>S<sub>0.002</sub>)Σ0.998 and the ideal formula is Pd<sub>2</sub>Sb. The electron microprobe analysis (WDS) of liberated grain #193 (Fig. 1*l*) corresponds to (Pd<sub>1.997</sub>Fe<sub>0.004</sub>)Σ2.001(Sb<sub>0.977</sub>As<sub>0.014</sub>S<sub>0.002</sub>)Σ0.993.

TABLE 1. Reflectance data (%) for naldrettite.

λ	R <sub>1</sub> (%)	R <sub>2</sub> (%)	λ	<sup>im</sup> R <sub>1</sub>	<sup>im</sup> R <sub>2</sub>
400	44.5	46.3	400	32.0	33.8
420	45.9	47.7	420	33.1	34.8
440	47.1	49.0	440	34.2	35.8
460	48.3	50.2	460	35.4	37.0
<b>470</b>	<b>49.0</b>	<b>50.9</b>	<b>470</b>	<b>35.9</b>	<b>37.6</b>
480	49.7	51.5	480	36.4	38.3
500	50.8	52.6	500	37.6	39.5
520	51.9	53.7	520	39.0	40.7
540	53.0	54.8	540	40.1	41.9
<b>546</b>	<b>53.2</b>	<b>55.1</b>	<b>546</b>	<b>40.3</b>	<b>42.1</b>
560	53.8	55.8	560	40.9	42.8
580	54.9	57.1	580	42.0	43.8
<b>589</b>	<b>55.4</b>	<b>57.5</b>	<b>589</b>	<b>42.5</b>	<b>44.3</b>
600	56.0	58.0	600	43.0	44.8
620	57.2	59.0	620	44.0	45.8
640	58.1	59.7	640	45.0	46.8
<b>650</b>	<b>58.5</b>	<b>60.1</b>	<b>650</b>	<b>45.4</b>	<b>47.2</b>
660	58.9	60.5	660	45.8	47.7
680	59.6	61.1	680	46.5	48.4
700	60.5	62.0	700	47.2	49.2

Values shown in bold are those recommended by COM, the IMA Commission on Ore Mineralogy.

TABLE 2. Colour values for naldrettite.

C illuminant	R <sub>1</sub> air	R <sub>2</sub> air	<sup>im</sup> R <sub>1</sub>	<sup>im</sup> R <sub>2</sub>
<i>x</i>	0.325	0.324	0.329	0.329
<i>y</i>	0.329	0.329	0.334	0.333
Y%	53.8	55.7	40.8	42.6
λ <sub>d</sub>	579	579	579	579
P <sub>e</sub> %	7.4	7.2	9.7	9.5
A illuminant	R <sub>1</sub> air	R <sub>2</sub> air	<sup>im</sup> R <sub>1</sub>	<sup>im</sup> R <sub>2</sub>
<i>x</i>	0.460	0.460	0.464	0.464
<i>y</i>	0.41	0.41	0.411	0.411
Y%	54.6	56.5	41.6	43.4
λ <sub>d</sub>	588	588	588	588
P <sub>e</sub> %	10.9	10.6	14.2	13.8

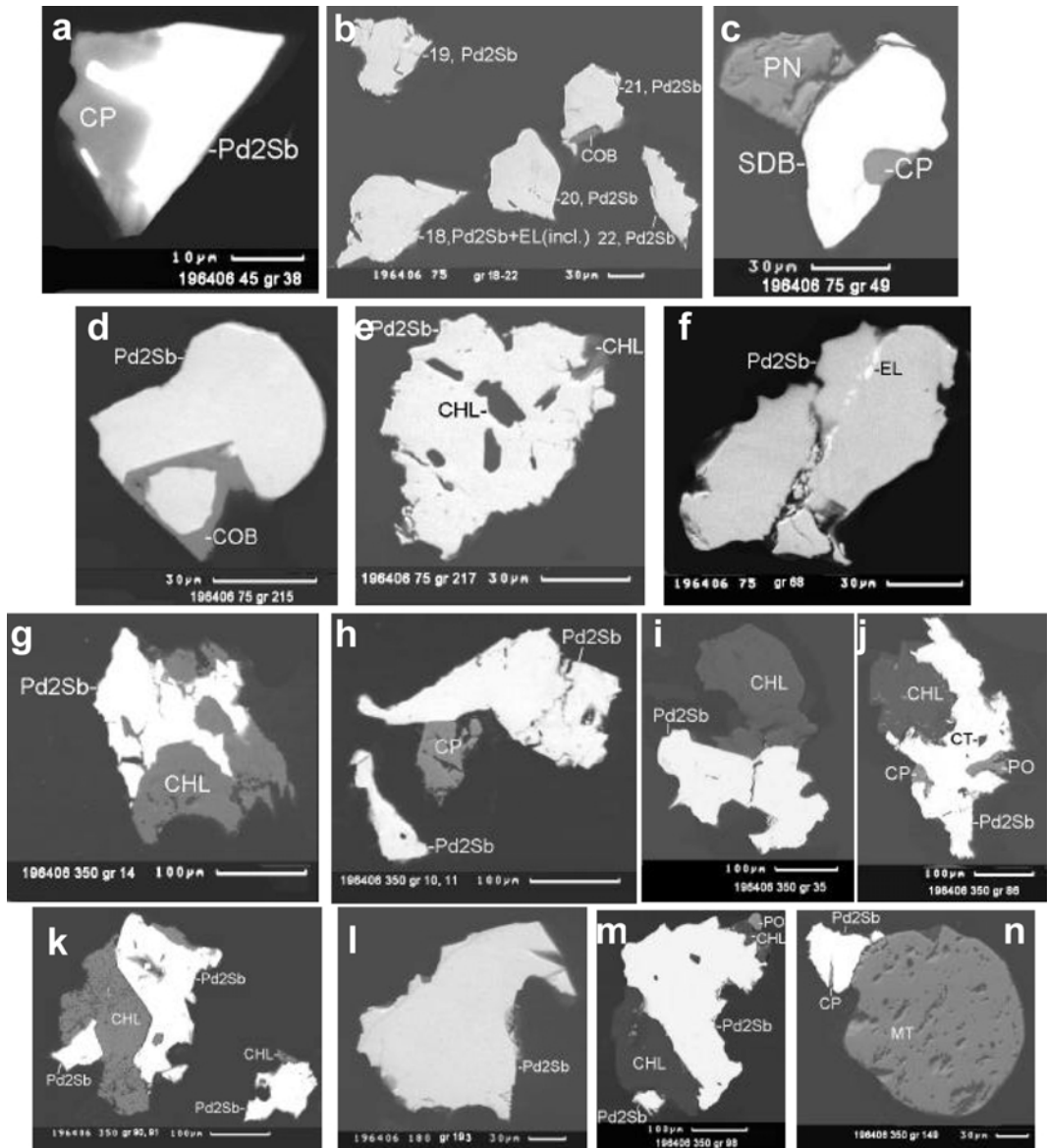


FIG. 1. SEM images of naldrettite (Pd<sub>2</sub>Sb) showing different associations from four size fractions (in parentheses). (a) Naldrettite-chalcopyrite (CP) intergrowth (<45 μm); (b) five liberated particles of naldrettite, one with attached cobaltite (COB) and another with electrum (EL) inclusions (45–75 μm); (c) naldrettite associated with pentlandite (PN) and chalcopyrite (45–75 μm); (d) naldrettite associated with cobaltite (45–75 μm); (e) naldrettite intergrown with clinocllore (CHL) (45–75 μm); (f) naldrettite with vein-like inclusions of electrum (45–75 μm); (g) naldrettite intergrown with clinocllore (180–350 μm); (h) two particles of naldrettite; one with attached chalcopyrite and the other one liberated (180–350 μm); (i) naldrettite with attached clinocllore (180–350 μm); (j) complex intergrowth of naldrettite, chalcopyrite, calcite (CT), pyrrhotite (PO) and clinocllore (180–350 μm); (k) two naldrettite grains, both associated with clinocllore (180–350 μm); (l) liberated particle of naldrettite used for reflectance measurements (125–180 μm); (m) naldrettite intergrown with clinocllore, with attached pyrrhotite (180–350 μm); (n) a rounded grain of magnetite (MT) with attached naldrettite and chalcopyrite (180–350 μm).

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TABLE 3. Electron probe analyses (wt.%) of naldrettite. Average of 69 separate analyses from 19 grains\*.

Element	Average wt.%	Range	St. dev.	Standard	LOD (ppm)	Ideal wt.%
S	0.02	0.00–0.04	0.009	chalco33	119	
Fe	0.11	0.00–0.61	0.142	chalco33	294	
As	0.31	0.00–0.59	0.130	461Pd3As	665	
Sb	35.75	34.96–39.63	0.42	NiSb	550	36.39
Pd	63.49	62.61–64.37	0.329	Pd metal	653	63.61
Totals	99.68	98.30–101.29				100

\*Grains #1–10 (polished section 75–125 µm M); grains #17, 18, 22, 24, 25, 27, 32, 33 (polished section 75–125 µm KT1); grain #193 (polished section 125–180 µm).

In addition, the following elements were sought but not detected: Se, Te, Ni, Bi, Ag, Zn, Hg, Cu, Rh, Ir, Ru, Co and Pt. LOD: limit of detection.

### X-ray crystallography

Powder X-ray diffraction (XRD) data (Table 4) were collected on grain #43 (previously analysed by SEM-EDX) with a 114.6 mm diameter Gandolfi camera employing Ni-filtered Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). Naldrettite is considered to be isostructural with synthetic Pd<sub>2</sub>Sb (PDF 23-0036) based on similarities in their powder XRD patterns and compositions (Bälz and Schubert, 1969). On this basis, naldrettite crystallizes in the space-group *Cmc*2<sub>1</sub> with  $V = 414.097(3) \text{ \AA}^3$  and  $Z = 8$ . The Pearson Symbol Code (PSC) is tC24. The unit-cell edges,  $a = 3.3906(1)$ ,  $b = 17.5551(5)$ ,  $c = 6.957(2) \text{ \AA}$ , were determined on the basis of 47 reflections for which unambiguous indexing was possible. The observed intensities were determined from a scanned powder XRD film. These are in good agreement with those calculated from the cell dimensions refined in this study, the refined crystal structure for synthetic Pd<sub>2</sub>Sb (Bälz and Schubert, 1969) and the program *POWDERCELL* (Nolze and Kraus, 1998).

The density could not be measured because of the small grain size. Based on the empirical average formula from analyses of 19 grains and unit-cell parameters refined from powder XRD data, the calculated density is  $10.694(1) \text{ g/cm}^3$ , a value consistent with many PGM.

### Other possible occurrences of naldrettite

An unnamed mineral with possible composition of (Pd,Pt,Sn)<sub>2</sub>(Sb,Sn) was reported from a heavy fraction of crushed Cu-Ni ore in a two-pyroxene deposit from China (location not given) by Jen and Huang (1973), and discussed by Fleischer *et*

*al.* (1975), and by Cabri (1981, who referred to the mineral as UN1973-4). This mineral, which occurs as grains 0.05–0.1 mm in size, was reported to have a composition corresponding to (Pd<sub>1.18</sub>Pt<sub>0.77</sub>Ni<sub>0.15</sub>) $\Sigma$ 2.00(Sb<sub>0.90</sub>Sn<sub>0.10</sub>) $\Sigma$ 1.00 and to be strongly anisotropic, with rotation tints from yellowish white to yellowish brown. Jen and Huang (1973) also reported a 26-line XRD pattern (including 6  $\beta$ -lines) with the strongest lines [ $d(I)$ ] 3.53 (5), 2.980 (10), 2.343 (5), 2.243 (9), 2.198 (7), 2.164 (8) and 2.040 (7). These data were indexed as orthorhombic Pd<sub>2</sub>Sb (PDF 29-0134) based on the similarity in the powder XRD patterns for the two. These authors also commented that the few reflections that did not fit well "...may be due to second phases".

A strongly anisotropic mineral with a composition of approximately Pd<sub>2</sub>Sb (no other elements reported) is one of several reported by the Platinum Metal Mineral Research Group in 1974 and abstracted by Fleischer *et al.* (1976). This mineral occurs in Cu-Ni sulphide deposits in a clinopyroxenite intruding Permian sandy shales and volcanics (location "W") in northeastern China and in a serpentinite intrusion in Permian metamorphic rocks (location "Y") in southwestern China. Cabri (1981) listed the mineral as UN1974-8 and suggested that this may be a Pt-free variety of UN1973-4.

A single particle of Pd<sub>2</sub>Sb was reported from the vein-type Cu-Ni sulphide ores of the Ioko-Dovyren massif located in the northern Baikal region, southern Siberia, Russia, by Rudashevsky *et al.* (2003). The sample was crushed, sieved and concentrated by hydroseparation (HS-01) and 60 particles of precious-metal minerals were found, mostly of sperrylite, but also sudburyite, gever-

TABLE 4. Powder XRD data for naldrettite<sup>1</sup>.

$I_{\text{obs}}$	$I_{\text{calc}}$	$d_{\text{meas}}$ (Å)	$d_{\text{calc}}$ (Å)	$h$	$k$	$l$	$I_{\text{obs}}$	$I_{\text{calc}}$	$d_{\text{meas}}$ (Å)	$d_{\text{calc}}$ (Å)	$h$	$k$	$l$
5	5	3.4963	3.4785	0	0	2		3		1.0744	1	9	5
2	4	3.2418	3.2338	0	2	2		2		1.0729	3	1	2
6	20	3.0065	3.003	1	1	1	14	2	1.0607	1.0623	2	8	4
4	2	2.9386	2.9337	1	3	0		10		1.0572	3	3	2
21	45	2.7123	2.7032	1	3	1	6	1	1.0539	1.0554	2	12	2
17	44	2.441	2.439	1	5	0		4		1.0543	1	15	2
34	34	2.4067	2.4051	1	1	2	15	2	1.0434	1.0472	1	5	6
35	93	2.3029	2.3016	1	5	1		1		1.0464	0	16	2
100	100	2.2454	2.2426	1	3	2		5		1.0446	2	4	5
	43		2.2421	0	2	3	9	2	1.0312	1.0304	3	7	0
	24		2.2391	0	6	2	6	3	1.0166	1.0175	1	13	4
27	27	2.1971	2.1944	0	8	0		1		1.0171	0	14	4
28	33	2.0979	2.0927	0	8	1	11	3	1.0095	1.0095	2	6	5
52	40	2.0567	2.0504	0	4	3		1		1.0081	2	14	0
40	20	2.0009	1.997	1	5	2	9	3	1.0052	1.0051	1	7	6
14	12	1.9414	1.9366	1	7	1	15	1	0.9982	1.001	3	3	3
30	20	1.9058	1.9028	1	1	3		1		0.9994	2	12	3
11	4	1.8620	1.8559	0	8	2		1		0.9985	1	15	3
22	5	1.8226	1.8193	1	3	3		1		0.9977	2	14	1
	7		1.8174	0	6	3	8	3	0.9912	0.9918	0	16	3
12	1	1.7472	1.7444	1	7	2		1	0.9745	0.9753	0	18	0
	3		1.7392	0	0	4	12	1	0.9745	0.9759	3	5	3
9	1	1.7059	1.7061	0	2	4	16	1	0.9665	0.9693	0	4	7
	6		1.7022	0	10	1		8		0.9683	2	14	2
17	35	1.6857	1.6951	2	0	0		1		0.9675	0	10	6
11	4	1.5986	1.5939	0	8	3		1		0.9658	2	8	5
11	5	1.5715	1.5672	0	10	2	17	7	0.9506	0.9514	2	2	6
12	2	1.5433	1.5421	2	4	1	28	3	0.9478	0.9463	3	1	4
	5		1.5415	1	1	4	17	8	0.9335	0.9342	2	12	4
22	2	1.5229	1.5239	2	0	2	9	1	0.9245	0.9245	2	14	3
	3		1.5216	1	7	3	13	1	0.9201	0.9204	1	5	7
	1		1.5206	1	9	2	5	6	0.9127	0.9131	2	16	1
15	5	1.4983	1.4961	1	3	4	30	7	0.9089	0.9096	2	6	6
	1		1.4669	2	6	0		1		0.9088	1	17	3
	1		1.4629	0	12	0		3		0.9087	0	12	6
7	1	1.4350	1.4353	2	6	1	7	5	0.9025	0.9011	3	9	3
	1		1.4316	0	12	1	4	3	0.8989	0.9042	1	11	6
10	1	1.4170	1.4161	1	5	4		1		0.899	0	18	3
	5		1.4138	1	11	1	10	7	0.8912	0.8914	1	7	7
12	5	1.4006	1.3997	0	10	3		2		0.8904	2	16	2
35	11	1.3674	1.3662	1	9	3	8	8	0.8835	0.8842	1	19	1
23	15	1.3513	1.3522	2	2	3	5	8	0.8749	0.8762	3	1	5
	8		1.3516	2	6	2	8	4	0.8697	0.8696	0	0	8
	2		1.3485	0	12	2	17	1	0.8658	0.8667	3	13	0

site, mertieite-II, and Ag-Au alloys, in addition to an anhedral particle of Pd<sub>2</sub>Sb 48 μm long.

#### Synthesis, genetic implications and discussion

Bälz and Schubert (1969) were able to synthesize orthorhombic Pd<sub>2</sub>Sb by reacting Pd<sub>66.6</sub>Sb<sub>33.4</sub> for 120 h at 500°C, followed by grinding and

reheating for 48 h at 500°C. Those authors also noted that heating the synthetic Pd<sub>2</sub>Sb above 580°C resulted in the formation of Pd<sub>5</sub>Sb<sub>2</sub> + hexagonal Pd<sub>5</sub>Sb<sub>3</sub>. In the same paper, Bälz and Schubert (1969) were able to synthesize and refine the crystal structure of orthorhombic Pd<sub>2</sub>As (no *R* or atomic displacement factors given), which they considered to be isostructural with

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Table 4 (contd.)

<i>I</i> <sub>obs</sub>	<i>I</i> <sub>calc</sub>	<i>d</i> <sub>meas</sub> (Å)	<i>d</i> <sub>calc</sub> (Å)	<i>h</i>	<i>k</i>	<i>l</i>	<i>I</i> <sub>obs</sub>	<i>I</i> <sub>calc</sub>	<i>d</i> <sub>meas</sub> (Å)	<i>d</i> <sub>calc</sub> (Å)	<i>h</i>	<i>k</i>	<i>l</i>
19	12	1.3332	1.3336	1	11	2		4		0.8665	2	12	5
	5		1.3263	0	4	5		1		0.8659	1	15	5
11	12	1.3170	1.3173	2	8	1		3		0.8654	0	2	8
	5		1.317	1	7	4		3		0.8649	0	10	7
23	20	1.3049	1.3065	2	4	3	4	1	0.8631	0.8635	1	19	2
42	15	1.2842	1.2838	1	1	5		1		0.8616	0	16	5
27	1	1.2565	1.2572	1	3	5	56	24	0.8584	0.859	1	17	4
	2		1.2565	0	6	5		12	0.8584	0.86	3	13	1
	3		1.2546	1	13	0		1		0.857	3	11	3
	1		1.2539	0	14	0		1		0.8568	1	9	7
	2		1.2517	2	8	2		1		0.8561	2	16	3
34	10	1.2356	1.2397	2	6	3		11		0.8524	3	9	4
	1		1.2373	0	12	3	7	1	0.8498	0.8515	1	13	6
	1		1.2355	0	10	4		3		0.8513	0	14	6
	17		1.2346	1	13	1		3		0.8511	0	20	2
	1	1.2356	1.234	0	14	1		2		0.8507	0	18	4
5	1	1.2253	1.2258	1	11	3	10	4	0.8445	0.8454	2	18	0
50	1	1.2122	1.214	2	0	4		10		0.8437	4	0	0
	15		1.2123	1	9	4	51	10	0.8409	0.8414	1	1	8
	9		1.2086	1	5	5		1		0.841	3	13	2
10	3	1.1991	1.2012	2	10	1		3		0.8403	2	10	6
	2		1.2025	2	2	4		6		0.8392	2	18	1
14	1	1.1803	1.1801	1	13	2		2		0.8376	4	2	1
	6		1.1796	0	14	2	14	2	0.8334	0.8338	1	3	8
	1		1.1751	0	8	5		1		0.8336	0	6	8
23	3	1.1466	1.1508	2	10	2	2	2	0.8307	0.8321	1	19	3
	5		1.1495	0	2	6	8	3	0.8264	0.8264	4	4	1
	5		1.1452	1	7	5							
25	5	1.1199	1.1195	0	12	4							
9	1	1.1022	1.1034	1	13	3							
	1		1.0972	0	16	0							
	3		1.0958	3	3	1							
5	1	1.0906	1.095	1	1	6							
	2		1.0938	2	12	1							
	2		1.0926	1	15	1							
	4		1.0904	0	10	5							
21	3	1.0784	1.0793	2	10	3							
	4		1.0783	1	3	6							
	4		1.0779	0	6	6							
	2		1.0758	3	5	0							

<sup>1</sup> Particle #43, using average of analyses of 19 particles given in Table 3.

their synthetic Pd<sub>2</sub>Sb phase. On this basis, Bälz and Schubert (1969) determined the crystal structure of orthorhombic Pd<sub>2</sub>Sb (space group *Cmc*2<sub>1</sub>, *a* = 3.354, *b* = 17.444, *c* = 6.909 Å) using atomic parameters obtained from Pd<sub>2</sub>As. However, since no *R* or atomic displacement factors were provided for the refined crystal structure of Pd<sub>2</sub>Sb, it is difficult to evaluate its correctness. On this note, a study to determine the crystal structure of naldrettite has been undertaken by one of us (AMM).

Synthetic Pd<sub>2</sub>Sb has more recently been synthesized by Kim and Chao (1996) at 500 and 570°C in their study of phase relations in the Pd-Sb system. They report the phase to be white under reflected light with a creamy tint in air and oil, and weakly birefractant. Anisotropism is described as strong, from brownish grey to greenish blue in air and from yellowish pink to dark greenish blue in oil, with *VNH*<sub>100</sub> = 293 (242–322) for *n* = 4. The powder XRD pattern for this synthetic Pd<sub>2</sub>Sb has *d* values slightly larger

than those of Bälz and Schubert (1969;  $a = 3.366(1)$ ,  $b = 17.523(3)$ ,  $c = 6.929(2)$  Å). In general, however, all these properties are in very good agreement with our data for naldrettite. The Pd-Sb binary phase diagram of Kim and Chao (1996) shows that below its incongruent melting point of 580°C, Pd<sub>2</sub>Sb can occur with either Pd<sub>5</sub>Sb<sub>2</sub> or PdSb, which helps to explain the occurrence of abundant sudburyite in the sample. A Pd<sub>5</sub>Sb<sub>3</sub> phase is shown to occur above 580°C, associated with either Pd<sub>5</sub>Sb<sub>2</sub> or PdSb. It should be noted that no phases of composition Pd<sub>4</sub>Sb<sub>3</sub> corresponding to the other new mineral in the sample, ungavaite (McDonald *et al.*, submitted), were reported. This suggests that ungavaite may be stable at lower temperatures (i.e. <500°C, which was the lowest temperature interval in Kim and Chao's (1996) experiments).

The Pd antimonide minerals are present within the contact zone separating massive and disseminated sulphide mineralization hosted by a pyroxenitic zone of a large gabbroic dyke. Palladium concentrations in the massive sulphide are anomalously low relative to other PGE and base metals when compared with the compositions of disseminated mineralization above the massive body. Palladium appears to have been removed from the massive sulphide body either during a late magmatic event or during regional metamorphism, and to have been redeposited around the edges of the massive sulphide body where it meets the enclosing ultramafic rock. Concentrations of Pd reach as high as 150 ppm in this contact facies, whereas the disseminated and massive sulphide facies each contain between 3 and 10 ppm Pd. The Pd antimonide mineral assemblage is largely limited to the Pd-enriched contact facies. The source of Sb in the mineralization remains unclear, but it is likely to have been externally derived. We infer that Pd was present in a relatively soluble form within the massive sulphide body and was removed by circulating fluids. When these fluids encountered Sb-rich rocks outside the massive sulphides, the Pd was precipitated at the point of contact to form the spatially-restricted Pd antimonide assemblage.

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