

# Article

# Implications of the unusually high degrees of Pb and Se substitutions in bismuth tellurides from the Stall Lake VMS deposit, Canada

Paul Alexandre (D)



Geology Department, Brandon University, Brandon, Manitoba, Canada

#### **Abstract**

Five discrete bismuth telluride compositions, characterised by high and variable degrees of Pb and Se substitutions, were observed at the Stall Lake VMS deposit in the Snow Lake area, Canada. The major cation substitutions are Pb (3.0 to 11.0 wt.%), Fe (0.2 to 1.4 wt.%), Cu (up to 0.9 wt.%) and Ag (up to 3.2 wt.%). The main anion substitution is Se (0.3 to 7.9 wt.%); S never exceeds 0.3 wt.%. These results were compared to a literature data compilation of all publicly available data for the pure bismuth tellurides tsumoite and tellurobismuthite, and the Pb-bearing rucklidgeite and kochkarite. On the basis of these new data and the literature compilation, a few generalisations about the substitutions in bismuth tellurides can be made. The major conclusion is that bismuth tellurides always contain at least some substitutional cations (Pb, Ag, Fe, Cu, Sb and Au), typically combining to ~2 wt.% if Pb is excluded, and anions (mostly Se and some S, typically <1 wt.% combined). Another conclusion is that bismuth tellurides have highly variable compositions, which can be quite far from their theoretical ones, to the point of defining specific mineral varieties such as high-Pb tsumoite, low-Pb kochkarite, and high-Se rucklidgeite. Two high-Se bismuth telluride compositions were observed at Stall Lake (average Se ≈ 4.9 and ≈ 7.2 wt.%), which had never been documented before. This observation, in conjunction with the bismuth tellurides literature data, emphasises the high potential for both cation and anion substitutions in these minerals.

**Keywords:** bismuth tellurides; kochkarite; rucklidgeite; tsumoite; tellurobismuthite; poubaite; element substitutions; bismuth; tellurium; lead; selenium

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#### Introduction

Pure bismuth tellurides are a group of five minerals (ehrigite, hedleyite, pilsenite, tsumoite and tellurobismuthite) with varying characteristics, including their compositions and crystallinity (Anthony et al., 1990). One of the ways to differentiate them from each other is the Bi/Te ratio of their structural formulas (e.g. Cook et al., 2007; Gu et al., 2001), which varies from 0.667 for tellurobismuthite (Bi<sub>2</sub>Te<sub>3</sub>) to 2.667 for the newly discovered ehrigite (Bi<sub>8</sub>Te<sub>3</sub>; Ciobanu et al., 2024). Most bismuth tellurides belong to the trigonal/hexagonal crystal system and have a layered structure (Anthony et al., 1990). Their diversity is controlled by different factors, specifically the order of layer alternation, the tolerance of their crystalline structure to incorporating substitution elements, and the possibility of an ordered distribution of substitution elements in their structure (Belogub et al., 2011; Cook et al., 2007).

Email: alexandrep@brandonu.ca

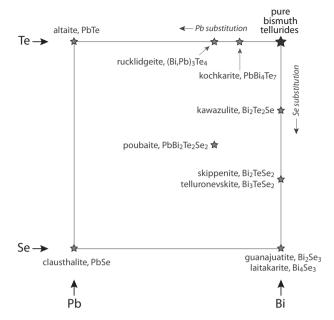
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In terms of the composition of these minerals, Bi is commonly substituted by Pb, whereas Te is substituted by S and less commonly by Se, leading to the existence of several minerals, many of which are rather rare, with varying complexity, including a few solid solutions (e.g. Anthony et al., 1990; Cook et al., 2007; Gu et al., 2001; Alexandre et al., 2019). However, when only Pb and Se substitutions in bismuth tellurides are considered (Fig. 1), it becomes apparent that it is much more common for one or the other of these substitutions to exist to the point of defining a distinct mineral, but it is rather uncommon for both Pb and Se substitutions to define a specific discrete mineral, with poubaite (PbBi<sub>2</sub>Te<sub>2</sub>Se<sub>2</sub>) being the unique example (Fig. 1). Other cations, such as Fe, Cu, Sb, Sn and Ag, can also substitute for Bi in pure bismuth tellurides to varying degrees, from very low to sufficiently high to form distinct minerals (Anthony et al., 1990). In general terms, the compositions of bismuth tellurides and its substitutions are relatively well studied, even though some knowledge gaps remain; the most notable of these involve the compositional variability of these minerals and the extent of the cation and anion substitutions.

As a part of a large-scale project on the comparative ore mineralogy of several volcanogenic massive sulfide deposits in the Flin

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2

**Figure 1.** Theoretical positions of minerals in Bi–Pb–Te–Se space. The pure bismuth tellurides, tsumoite, tellurobismuthite and the Pb-bearing kochkarite and rucklidgeite were subject of a literature data compilation.

Flon – Snow Lake Mineral Belt (Manitoba, Canada; Fig. 2), a bismuth telluride mineral, or rather a range of compositions, with significant and highly variable degrees of both Pb and Se substitutions was observed at the Stall Lake VMS deposit. It was soon appreciated that the degree of Pb substitution was comparable to that of other Pb-rich bismuth tellurides such as rucklidgeite ((Bi,Pb)<sub>3</sub>Te<sub>4</sub>) and kochkarite (PbBi<sub>4</sub>Te<sub>7</sub>). However, the degree of Se substitution in this phase was significantly higher than that of any of these minerals, without reaching the levels of kawazulite (Bi<sub>2</sub>Te<sub>2</sub>Se), skippenite (Bi<sub>2</sub>TeSe<sub>2</sub>), or telluronevskite (Bi<sub>3</sub>TeSe<sub>2</sub>).

It was the unusually high degrees of Pb and Se substitutions in this phase that prompted a comprehensive comparison with the all literature data available for similar minerals, specifically the almost pure bismuth tellurides tsumoite and tellurobismuthite, and the Pb-rich bismuth tellurides rucklidgeite and kochkarite. Tsumoite and tellurobismuthite were preferred for this comparison rather than other pure bismuth tellurides, as their Bi/Te ratios are very similar to those of the Stall Lake phase. Rucklidgeite and kochkarite were selected because their Pb contents are very similar to that of the Stall Lake phase. The conclusions of this comparison are presented in this paper, together with observations regarding Pb and Se substitutions in bismuth tellurides.

#### Geological background of the Stall Lake deposit

The mined-out Stall Lake deposit is one of the 12 volcanogenic massive sulfide (VMS) deposits from the Snow Lake arc assemblage in Manitoba, Canada, itself the easternmost part of the Flin Flon – Snow Lake Mineral Belt (Fig. 2). The Snow Lake assemblage was formed at ca. 1.87–1.85 Ga as the result of intraoceanic accretion (Lewry and Collerson 1990; Lucas *et al.*, 1996).

The Stall Lake deposit was the third largest in the Snow Lake area, with ~6.4 million tonnes total ore containing 4.41 wt.% Cu, 0.5 wt.% Zn, 0.01 wt.% Pb, 12.34 g/t Ag and 1.41 g/t Au

(Pehrsson *et al.*, 2016). It was discovered in 1956 by ground electromagnetics followed by drilling and mined between 1964 and 1982. The host lithology is dominated by gneisses (quartz-eye gneiss, staurolite-garnet gneiss and sillimanite gneiss) and schists (chlorite-garnet schist and chlorite schist), with minor andesitic tuffs and amphibolite sills (Studer, 1982).

The ore at the Stall Lake deposit was contained in seven sulfide lenses, each dipping between  $\sim\!\!30$  and  $60^\circ$  to the SW, with a maximum length between  $\sim\!\!50$  and 300 m, and thickness varying between  $\sim\!\!10$  and 50 m (Studer, 1982). These lenses were zoned and heterogeneous, dominated by massive coarse-grained pyrrhotite with lesser pyrite, chalcopyrite and sphalerite, minor galena, magnetite and gahnite, and traces of hessite (Ag2Te) and the Bi–Te phase that is the subject of this paper (Studer, 1982; our observations; Fig. 3). The main ore mineral, chalcopyrite, is typical, with minor Pb (0.36 wt.%) and Bi (0.09 wt.%); sphalerite contains 6.38 wt.% Fe, 0.37 wt.% Cu and 0.35 wt.% Pb (EMPA, our unpublished data).

# Sampling and analytical methodology

Approximately 50 representative drill core samples from the Stall Lake deposit main ore bodies were collected from the Manitoba Geological Survey's Centennial core storage facility at Baker Lake, ~20 km south of Flin Flon (Manitoba, Canada), as a part of a larger study of the Flin Flon – Snow Lake Mineral Belt VMS deposits ore mineralogy. Of these, 20 high-grade ore samples, composed mostly of sulfide minerals, were selected for further analyses. Polished thin sections were prepared from these samples and were used for petrographic observations and electron microprobe analyses (EMPA).

Optical microscopy (reflected light) and scanning electron microscopy (SEM) were used to identify the ore minerals present at Stall Lake. The SEM instrument used was a Jeol JSM-6390 at Brandon University, Canada.

Compositions of the Bi–Te phase were obtained using wavelength dispersive spectroscopy (WDS) at the University of Manitoba with a Cameca SX100 electron microprobe. The elements analysed were: Se (X-ray line used La, analytical crystal used TAP); Pb (Ma, LPET); S (Ka, LPET); Sb (La, PET); Ag (La, PET); Bi (Mb, PET); Cu (Ka, LLIF); Ni (Ka, LLIF); Co (Ka, LLIF); Fe (Ka, LLIF); and Te (La, LLIF). The analytical conditions were: 15 keV, 20 nA and 1 mm beam; peak counting times were 20 s (except for Te, Se and S: 30 s), with the background counted for half of that time, once on each side of the peak. The ZAF corrections followed the PAP procedure (Pouchou and Pichoir, 1984; Pouchou and Pichoir, 1985). Reference materials are natural and synthetic CdSe (for Se), PbTe (for Pb and Te), pyrite (for S and Fe), stibnite (for Sb), silver (for Ag), bismuthinite (for Bi), chalcopyrite (for Cu), pentlandite (for Ni) and cobalt (for Co).

It was impossible to collect any structural data for the Bi–Te phase from the Stall Lake deposit, due to its small grain size (Fig. 3) and very low quantities. If X-ray diffraction analysis had been performed on a bulk sample, the signal emanating from the small Bi–Te phase grains would be too low to be distinguished from the background noise. Further, micro-Raman is impossible to apply, because the method is not applicable to telluride minerals, which oxidise under the laser beam and thus do not produce a recognisable spectrum or signature (Alexandre and Aisida, 2023). In the absence of structural data, this work relies only on the composition of the Bi–Te phase observed at the Stall Lake deposit.

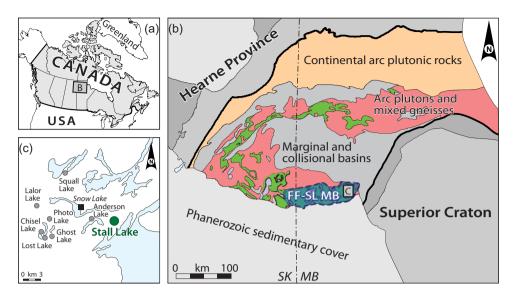


Figure 2. Simplified geological context of the Stall Lake and other VMS deposits within North America (a), the Trans-Hudson Orogen of Canada (b), and the Snow Lake area (c). The approximate extent of the Flin Flon – Snow Lake Mineral Belt (FF-SL MB) is indicated in (b). SK=Saskatchewan, MB=Manitoba.

A significant component of this work is the literature data compilation of the composition of Bi-Te minerals comparable to the Stall Lake phase, used to situate these new data within a relevant compositional context. To that end, all publicly available literature on tsumoite, tellurobismuthite, rucklidgeite and kochkarite are summarised here. These minerals were selected as they are comparable to the Stall Lake phase, on the basis of the Bi/Te (or total cation/total anion) ratio of the structural formula (0.73 to 1.05) and Pb contents. The results of this compilation are provided, together with the Stall Lake phase data, in Table 1 and will be discussed in the Results section; the references used are Rucklidge (1969); Zav'yalov and Begizov (1977); Haranczyk (1978; Spiridonov et al. (1989); McQueen (1990); McQueen and Solberg (1990); Kase et al. (1993); Gu et al. (2001); Cepedal et al. (2006); Cook et al. (2007); Ivashchenko et al. (2008); Sejkora et al. (2009); Belogub et al. (2011); Voloshin et al. (2012); Maslennikov et al. (2013); Jian et al. (2014); Sabau et al. (2014); Xue and Campbell (2014); Stenco et al. (2014); Roberts (2017); Haing (2019); Kondratieva et al. (2021); Kasatkin et al. (2022); Alfonso et al. (2023); Kasprowicz (2023); and Ma et al. (2024).

# Analytical results and literature data compilation findings

In this section, the Stall Lake Bi–Te phase is described (Fig. 3) together with its composition, followed by the results of the compilation of all available literature data on the composition of tsumoite, tellurobismuthite, kochkarite and rucklidgeite. The summary data for all minerals, using the minimum, maximum and mean values (both in terms of wt.% and apfu), are provided in Table 1; all data are also represented in Figs 4 and 5.

#### Mineral description

On the basis of optical microscope (reflected light; Fig. 3a, b) and SEM observations (Fig. 3c-f), the Stall Lake Bi-Te phase has the following characteristics. The colour is pale yellow to white (Fig.

3a, b). Due to the small grain size, anisotropy was difficult to estimate, though appears to be very low. Its reflectivity is very high, though not as high as galena and is estimated to be  $\sim$ 80% (Fig. 3a, b). The phase varies from a few µm to  $\sim$ 30 µm across (Fig. 3). Its morphology varies between mostly isomorphic (Fig. 3e, f) to moderately elongated (Fig. 3d); it is mostly anhedral and occasionally subhedral (Fig. 3c, f).

The Stall Lake Bi–Te phase is associated with the major minerals present at this deposit, mostly with pyrite and pyrrhotite, though also chalcopyrite and magnetite (Fig. 3). It is also sometimes closely associated with hessite (Ag<sub>2</sub>Te; Fig. 3c, e). On the basis of its textural relationships with other minerals, it probably formed later than the main stage (coarse grained) minerals and together with other (minor and rather small grained) sulfides, possibly during the lower to middle almandine–amphibolite-facies metamorphism that affected the deposit area (maximum conditions 5 kb and 535°C; Froese and Moore, 1980), in imitation of other deposits in the near vicinity (Alexandre *et al.*, 2019; Alexandre and Aisida, 2023).

## The composition of the Stall Lake deposit Bi-Te phase

At a first glance, this phase is dominated by Bi and Te, with concentrations anywhere between ~41 and 56 wt.% for the former and between ~35 and 41 wt.% for the latter, when all analyses are considered. The major cation substitutions are Pb, varying between 3.0 and 11.0 wt.%, Fe (0.2 to 1.4 wt.%), Cu (up to 0.9 wt.%), Ag (up to 3.2 wt.%), and traces of Sb and Au (Table 1). Copper, Ni, Zn, Mn, As, Sn and Hg were also analysed, but were only just at the quantification limit or below. The main anion substitution is Se, varying overall from 0.3 to 7.9 wt.%; S never exceeds ~0.3 wt.% (Table 1).

When these data are considered from the point of view of the two main substitutions, Pb for Bi and Se for Te (Fig. 4), five clearly discrete compositional groups can be empirically defined (Fig. 5), as described below.

Compositional groups 1 and 2 are fairly similar to each other and are defined by the two lowest amounts of Pb substitutions ( $\sim$ 3.3 wt.% and 5.1 wt.% on average) and the lowest Se substitution ( $\sim$ 0.3 wt.% for both groups, on average; Figs 4

**Table 1.** Stall Lake Bi-Te minerals. Summary (min, mean, max) of the analytical results for the Stall Lake bismuth telluride compositional groups seen in Figs 4 and 5 (1 to 5) and the literature data compilation for tsumoite, tellurobismuthite, rucklidgeite, and kochkarite (6 to 12 in Fig. 5; based on the references in the text) Cobalt, Ni, Zn, Mn, As, Sn, and Hg were also analysed in the Stall Lake phase, but were always just about or below the quantification limit; Zn, As and Hg were occasionally reported in the literature data, however the values were always very near the quantification limit for the respective method (predominantly electron microprobe), and are not reported here.

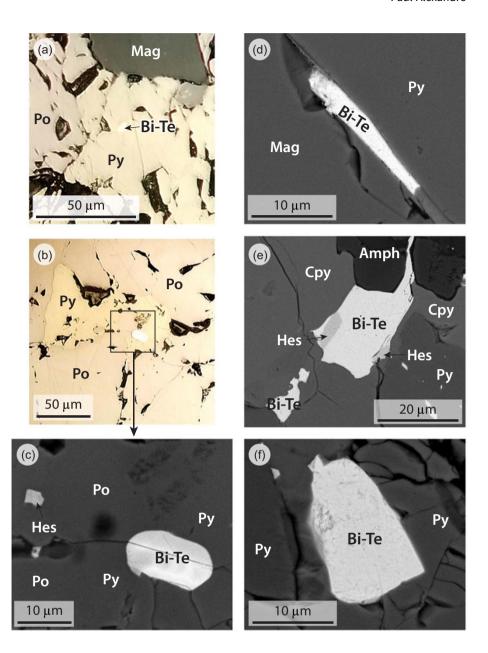
	STALL LAKE Bi-Te MINERALS														LITERATURE DATA Bi-Te MINERALS						
		High-Pb tsumoite				Low-Pb kochkarite					High-Se kochkarite					Tsumoite			High-Pb tsumoite		
		1			2			3			4			5			6			7	
wt.%	min	mean	max	min	mean	max	min	mean	max	min	mean	max	min	mean	max	min	mean	max	min	mean	max
Bi	54.35	55.25	56.19	52.56	54.67	55.91	43.61	45.03	47.60	40.99	43.24	46.30	42.04	43.55	45.04	55.55	62.31	65.71	54.65	57.21	62.4
Pb	2.97	3.31	3.78	4.80	5.13	5.52	6.18	7.24	8.39	8.79	10.92	13.35	9.33	10.96	13.55	bdl	0.39	1.20	3.16	4.60	5.91
Fe	1.08	1.20	1.33	1.03	1.20	1.38	0.74	0.92	1.25	0.22	0.42	0.79	0.33	0.42	0.55	0.42	0.45	0.48		bdl	
Sb		bdl		bdl	0.06	0.17	bdl	0.02	0.18	bdl	0.02	0.16	bdl	0.02	0.13	0.14	0.35	0.67		bdl	
Cu	0.51	0.68	0.93	0.40	0.62	1.11	0.03	0.13	0.30	0.07	0.16	0.27	0.13	0.27	0.45		bdl			bdl	
Ag	bdl	0.03	0.11	bdl	0.02	0.14	0.18	0.30	0.47	0.34	0.63	0.87	0.28	0.66	3.23	0.01	0.34	2.25		bdl	
Au	bdl	0.01	0.06	bdl	0.01	0.07	bdl	0.02	0.06	bdl	0.01	0.07	bdl	0.02	0.06	0.01	0.09	0.19		bdl	
Te	38.48	39.33	40.79	35.06	37.77	39.77	42.56	45.49	47.17	37.44	39.56	42.34	35.39	36.75	38.33	32.48	36.43	41.36	34.36	37.59	39.34
Se	0.28	0.30	0.34	0.27	0.30	0.32	0.68	0.75	0.94	4.12	4.94	6.18	6.90	7.21	7.85	0.02	0.22	0.80		bdl	
S	0.07	0.11	0.19	0.07	0.12	0.21	0.05	0.10	0.27	bdl	0.06	0.11	0.00	0.11	0.16	bdl	0.09	0.80		bdl	
Total		100.24			99.94			100.02			99.97			99.99			99.32	1.61		99.39	
APFU		2			2			12			12			12			2			2	
		atoms			atoms			atoms			atoms			atoms			atoms			atoms	
Bi		0.841			0.841			4.034			3.819			3.775			0.984			0.927	
Pb		0.051			0.080			0.654			0.973			0.958			0.006			0.075	
Fe		0.068			0.069			0.307			0.140			0.138			0.027				
Sb					0.002			0.003			0.004			0.004			0.009				
Cu		0.034			0.031			0.039			0.045			0.076							
Ag		0.001			0.001			0.053			0.108			0.112			0.010				
Au											0.001			0.002			0.001				
S <sub>CAT</sub>		0.996			1.024			5.089			5.089			5.064			1.038			1.002	
Te		0.981			0.952			6.674			5.722			5.217			0.943			0.998	
Se		0.012			0.012			0.179			1.154			1.655			0.009				
S		0.011			0.012			0.056			0.034			0.064			0.010				
S <sub>AN</sub>		1.004			0.976			6.909			6.911			6.936			0.962			0.998	
Cat/An		0.99			1.05			0.74			0.74			0.73			1.08			1.00	

(Continued)

Table 1. (Continued.)

						LITERATU	RE DATA Bi-Te N	IINERALS						
Low-Pb kochkarite				Kochkarite			Rucklidgeite		H	igh-Se rucklidge	eite	Tellurobismuthite		
8			9			10			11			12		
min	mean	max	min	mean	max	min	mean	max	min	mean	max	min	mean	max
42.79	43.74	44.81	35.66	41.06	44.29	30.05	39.98	44.34	34.73	39.03	44.03	48.12	51.88	53.84
6.54	6.98	7.62	9.46	11.78	15.98	10.77	13.39	20.91	13.11	16.07	20.52	bdl	0.26	2.27
bdl	0.06	0.20	0.17	1.04	1.76	bdl	0.53	1.36	0.06	0.51	0.99	0.02	0.95	2.24
0.43	0.54	0.63	0.05	0.35	0.70	0.06	0.47	1.15	0.18	0.27	0.44	0.28	0.39	0.72
bdl	0.01	0.01	bdl	0.12	0.45	bdl	0.06	0.45	bdl	0.17	0.59	bdl	0.38	1.47
0.82	0.95	1.10	bdl	1.34	3.70	0.37	1.56	4.69	1.15	1.41	1.99	bdl	0.15	0.36
bdl	0.03	0.13		bdl		bdl	0.01	0.07				bdl	0.01	0.03
45.89	46.57	47.51	41.46	44.98	48.11	42.55	44.44	46.29	39.05	41.10	42.38	42.90	47.07	49.65
bdl	0.03	0.07	0.10	0.20	0.46	bdl	0.27	1.11	2.27	3.06	3.56	bdl	0.45	2.71
bdl	0.00	0.01	bdl	0.08	0.30	bdl	0.06	0.14	0.04	0.08	0.19	bdl	0.06	0.28
	98.92			100.41			100.83			101.72			99.90	
	12 atoms			12 atoms			7 atoms			7 atoms			5 atoms	
	4.031			3.645			2.098			1.997			1.899	
	0.648			1.055			0.709			0.829			0.009	
	0.021			0.346		0.104			0.097			0.130		
	0.086			0.054			0.043			0.024			0.025	
	0.002			0.036			0.011			0.028			0.046	
	0.170			0.231			0.159			0.140			0.011	
	0.003						0.001							
	4.962			5.365			3.124			3.114			2.120	
	7.029			6.539			3.820			3.444			2.822	
	0.007			0.047			0.037			0.414			0.043	
	0.002			0.049			0.019			0.027			0.015	
	7.038			6.635			3.876			3.886			2.880	
	0.71			0.81			0.81			0.80			0.74	

Notes: bdl = below detection limit



**Figure 3.** Examples of the Stall Lake VMS deposit bismuth tellurides (Bi–Te) in reflected light (a and b) and in back-scattered electron images (c–f), together with other minerals present (Po: pyrrhotite; Py, pyrite; Cpy, chalcopyrite; Mag, magnetite; Hes, hessite, and Amph, amphibole).

and 5). Iron and Cu are present in both groups ( $\sim$ 1.2 wt.% for Fe,  $\sim$ 0.7 and  $\sim$ 0.6 wt.% for Cu); Au, Ag and Sb are present in trace amounts (Table 1). Their average structural formulae, on the basis of two atoms in imitation of tsumoite, can be written as (Bi<sub>0.841</sub>Fe<sub>0.068</sub>Pb<sub>0.051</sub>Cu<sub>0.034</sub>)<sub> $\Sigma$ 0.996</sub>(Te<sub>0.981</sub>Se<sub>0.012</sub>So<sub>0.011</sub>) $\Sigma$ 1.004 for group 1 and (Bi<sub>0.841</sub>Pb<sub>0.080</sub>Fe<sub>0.069</sub>Cu<sub>0.031</sub>) $\Sigma$ 1.024</sub>(Te<sub>0.952</sub>Se<sub>0.012</sub>So<sub>0.012</sub>) $\Sigma$ 1.0296 for group 2.

Compositional group 3 is notable for increased Pb and Se substitutions, with average Pb of  $\sim$ 7.2 wt.% and average Se of  $\sim$ 0.75 wt.% (Table 1). There is less Fe ( $\sim$ 0.9 wt.%), Ag ( $\sim$ 0.3 wt.%) and Cu ( $\sim$ 0.1 wt.%); Sb and Au are present at trace level (Table 1). Sulfur is relatively low, at  $\sim$ 0.1 wt.%. The average structural formula for this compositional group can be written, on the basis of 12 atoms as in kochkarite, as (Bi<sub>4.034</sub>Pb<sub>0.654</sub>Fe<sub>0.307</sub>Cu<sub>0.039</sub>)<sub>25.089</sub>(Te<sub>6.674</sub>Se<sub>0.179</sub>S<sub>0.056</sub>)<sub>26.909</sub>.

Compositional groups 4 and 5 contain higher Pb ( $\sim$ 10.9 wt.% on average) than group 3, however they are remarkable by the largest degree of Se substitutions, with, on average,  $\sim$ 4.9 wt.% for

group 4 and ~7.2 wt.% for group 5 (Table 1; Fig. 4). Though Se in these two groups is indeed high, it does not reach the levels of poubaite (~13.6 wt.%; Anthony et~al., 1990; Johan et~al., 1987; Kalinin, 2021). There is less Fe (~0.4 wt.%) and Cu (~0.2 and ~0.3 wt.%, respectively) than the other groups, but more Ag (~0.6 and ~0.7 wt.%, on average; Table 1); Sb and Au are present at trace level. Sulfur is also the lowest among all Stall Lake analyses, at ~0.1 wt.%; Table 1). The average structural formula for these two compositional groups can be written, also based on 12 atoms, as  $(Bi_{3.819}Pb_{0.973}Fe_{0.140}Cu_{0.045})_{\Sigma5.089}(Te_{5.722}Se_{1.154}S_{0.034})_{\Sigma6.911}$  for group 4 and  $(Bi_{3.775}Pb_{0.958}Fe_{0.138}Cu_{0.076})_{\Sigma5.064}$   $(Te_{5.217}Se_{1.655}S_{0.064})_{\Sigma6.936}$  for group 5.

The main point of these observations is the continuous increase in both Pb and Se substitution from group 1 to group 5, without Se reaching poubaite levels (Fig. 5). Iron and Cu are always present in measurable amounts but tend to decrease with increasing degree of Pb and Se substitution; Ag is the highest in groups 4 and 5, whereas Au and Sb are always present at trace amounts (Table 1).

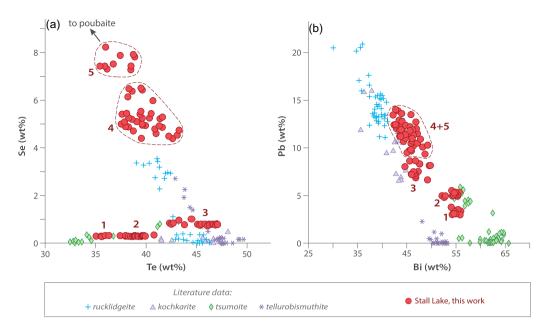
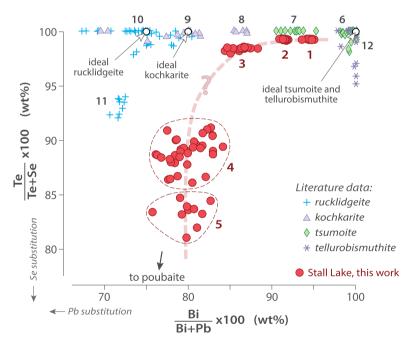


Figure 4. Se and Pb substitutions in the Stall Lake bismuth tellurides (a and b, respectively), compared to those of the pure bismuth tellurides tsumoite and tellurobismuthite and the Pb-bearing kochkarite and rucklidgeite, based on the references given in the text (Sampling and analytical methodology). The empirically defined Stall Lake compositional groups are numbered 1 to 5. Note that weight percent (wt.%) are used rather than atoms per formula unit (apfu), as minerals with different numbers of atoms in their structural formula (2, 5, 7 and 12) are being compared.



**Figure 5.** The empirically defined Stall Lake bismuth tellurides compositional groups (numbered 1 to 5), compared to the literature data for tsumoite, tellurobismuthite, kochkarite, and rucklidgeite (numbered 6 to 12), in a Pb substitution vs. Se substitution diagram, based on the references given in the text (Sampling and analytical methodology). The extent of substitution was based on the weight percent (wt.%), even though fairly similar results would have been obtained if atoms per formula unit (apfu) were used, given that the substitutions are expressed as ratios.

It should also be noted that the average Bi/Te (or total cations/total anions) ratio for groups 1 and 2 is close to 1, whereas in the other three groups this ratio is closer to 0.74 (Table 1), justifying using the tsumoite and kochkarite structural formula patterns (2 and 12 atoms, respectively), as indicated above.

# Literature data compilation

Tsumoite literature data fall into two compositional groups. The first corresponds to a fairly typical tsumoite with 62.3 wt.% Bi and 36.4 wt.% Te, and relatively low degrees of

cation and anion substitutions ( $\sim$ 1.6 wt.% and  $\sim$ 0.33 wt.% in total, respectively; Table 1). The main substitution cations are Pb, Fe, Sb and Ag, all at  $\sim$ 0.4 wt.%; Au is also present ( $\sim$ 0.1 wt.%). Its structural formula can be written as (Bi<sub>0.984</sub>Fe<sub>0.027</sub>Sb<sub>0.010</sub>Sb<sub>0.009</sub>)<sub>Σ1.038</sub>(Te<sub>0.943</sub>S<sub>0.010</sub>Se<sub>0.009</sub>)<sub>Σ0.962</sub>.

The second compositional group for tsumoite, observed in two deposits (from the Ural mountains and from the Karelian Peninsula in Russia (Belogub *et al.*, 2011; Ivashchenko *et al.*, 2008, is characterised by the presence of Pb substitution (~4.6 wt.% of Pb on average, ranging from 3.2 to 5.9 wt.%) and was labelled

8 Paul Alexandre

Pb-rich tsumoite. No other substitution cations or anions were observed (Table 1); its structural formula can be written as  $(Bi_{0.927}Pb_{0.075})_{\Sigma 1.002}Te_{0.998}$ .

Kochkarite literature data also define two discrete two compositional groups, distinguished from each other by their Pb concentrations (Figs 4 and 5, Table 1). The major group corresponds to typical kochkarite, with, on average,  $\sim$ 43.7 wt.% Bi, 11.8 wt.% Pb, 1.3 wt.% Ag, and 1.0 wt.% Fe; Sb and Cu are also present (Table 1). The anions are dominated by Te ( $\sim$ 45.0 wt.%); the amount of Se is relatively low (0.2 wt.%; Table 1). Significantly, the data show a high degree of variation of all elements present, with Pb, for instance, varying from  $\sim$ 9.5 to  $\sim$ 16.0 wt.% (Table 1). The average structural formula for this group is (Bi<sub>3.645</sub>Pb<sub>1.055</sub>Fe<sub>0.346</sub>Ag<sub>0.231</sub>)<sub>25.365</sub>(Te<sub>6.539</sub>S<sub>0.049</sub>Se<sub>0.047</sub>)<sub>26.635</sub>.

The second kochkarite group, observed at the Deer Horn intrusion-related polymetallic deposit in British Columbia, Canada (Roberts, 2017), contains more Bi ( $\sim$ 43.7 wt.% on average), less Pb ( $\sim$ 7.0 wt.% on average), some Ag ( $\sim$ 1.0 wt.%), Sb ( $\sim$ 0.5 wt.%) and traces of Fe, Au and Cu (Table 1); its anion substitutions are very low (Se  $\approx$  0.03 wt.% on average, no S). This compositional group was labelled 'low-Pb kochkarite' and its average structural formula is (Bi $_{4.031}$ Pb $_{0.648}$ Ag $_{0.170}$ Sb $_{0.086}$ ) $_{\Sigma4.962}$ (Te $_{7.029}$ Se $_{0.007}$ ) $_{\Sigma7.038}$ .

Rucklidgeite is another mineral for which two distinct subpopulations were observed in the compilation dataset; it is also the one with the highest variation in the concentrations of most elements (Fig. 4b; Table 1). Most analyses form a group of more or less typical rucklidgeite, with ~40.0 wt.% Bi on average, ~16.1 wt.% Pb (varying from ~10.8 to ~20.9 wt.%), ~1.6 wt.% Ag (~0.4 to ~4.7 wt.%), and some Fe and Sb (~0.5 wt.% for both; Table 1). This group contains ~44.4 wt.% Te on average, and limited Se (~0.3 wt.% on average, ranging from below detection to ~1.1 wt.%); S is minimal. Its average structural formula is  $(Bi_{2.098}Pb_{0.709}Ag_{0.159}Fe_{0.104})_{\Sigma3.124}(Te_{3.820}Se_{0.037}S_{0.007})_{\Sigma3.876}$ .

A minor subpopulation of rucklidgeite, from a VMS deposit in Quebec, Canada (Kasprowicz, 2023), is characterised by high Se ( $\sim$ 3.1 wt.% on average: Table 1) and was labelled high-Se rucklidgeite. The concentrations of all other elements in this group are similar to those in the main rucklidgeite population (Table 1). Its average structural formula is (Bi<sub>1.997</sub>Pb<sub>0.829</sub>Ag<sub>0.140</sub>Fe<sub>0.097</sub>)<sub> $\Sigma$ 3.114</sub>(Te<sub>3.444</sub>Se<sub>0.414</sub>S<sub>0.027</sub>)<sub> $\Sigma$ 3.886</sub>.

Tellurobismuthite also shows some compositional variation, in particular in its Se concentrations, which vary from below detection to  $\sim$ 2.7 wt.% ( $\sim$ 0.45 on average; Table 1). The average Bi concentrations are  $\sim$ 51.9 wt.%; Te is present at  $\sim$ 47.1 wt.% on average (Table 1). Tellurobismuthite has the lowest Pb concentrations of all the minerals considered ( $\sim$ 0.3 wt.% on average); Fe, Sb, Cu and Ag are also present ( $\sim$ 1.0 wt.%,  $\sim$ 0.4, wt.%,  $\sim$ 0.4 wt.%, and  $\sim$ 0.2 wt.%, on average, respectively). The average structural formula of tellurobismuthite, based on this compilation, is (Bi<sub>1.899</sub>Fe<sub>0.130</sub>Cu<sub>0.046</sub>Ag<sub>0.011</sub>)<sub>Σ2.120</sub>(Te<sub>2.822</sub>Se<sub>0.043</sub>S<sub>0.015</sub>)<sub>Σ2.880</sub>.

#### Discussion

The literature compilation of all publicly available compositional data for tsumoite, tellurobismuthite, kochkarite and rucklidgeite (based on the references at the end of the section on Sampling and analytical methodology), provide some interesting insights and some general comments can be made about their chemical purity and their compositional variability; these questions have never been addressed. The composition of the Stall Lake deposit

phases will also be discussed, in the context of the other bismuth tellurides (e.g. Cook *et al.*, 2007; Gu *et al.* 2001).

#### Extent of substitutions in bismuth tellurides

All minerals considered and documented by both the literature compilation and the new data from Stall Lake contain at least some Pb, Fe, Cu, Sb, Ag and Au (Table 1). Silver is most abundant in rucklidgeite ( $\sim$ 1.6 wt.%) and kochkarite ( $\sim$ 1.4 wt.%); Fe is most abundant in kochkarite and tellurobismuthite ( $\sim$ 1.0 wt.% on average). Tellurobismuthite also has the most Sb and Cu, both at  $\sim$ 0.4 wt.% on average. The average concentrations of Pb vary from  $\sim$ 0.3 wt.% in tellurobismuthite to  $\sim$ 16.1 wt.% in high-Se rucklidgeite. Overall, the cations observed in these minerals, other than Bi and Pb, typically combine to  $\sim$ 2 wt.%.

All minerals considered show some degree of Se substitution, with Se varying from below detection limit to  $\sim$ 3.6 wt.% for high-Se rucklidgeite (VMS deposit in Quebec; Kasprowicz, 2023). The amounts of Se are low for tsumoite and kochkarite (both at  $\sim$ 0.2 wt.% on average), higher for tellurobismuthite ( $\sim$ 0.5 wt.% on average, up to 2.7 wt.%; Cook *et al.*, 2007), and highest for high-Se rucklidgeite ( $\sim$ 3.1 wt.% on average; Kasprowicz, 2023; Table 1). Overall, Se and S typically combine to just under 1 wt.% in these minerals.

From this perspective, it could be argued that there is no such thing as pure bismuth telluride (in clear contradiction of the first three words of this paper): all and any of them will always contain a certain amount of both cation and anion substitutions, as demonstrated by the literature data compilation and the new data obtained at Stall Lake. The cation substitutions are mostly Pb, but also, in decreasing amounts, Ag, Fe, Sb, Cu and Au; the anion substitutions are predominantly Se and much less S (Table 1). As a practical corollary, these are the bare minimum of elements that should always be analysed in any bismuth telluride.

### Compositional variability of bismuth tellurides

Another important observation originating from the literature data compilation is the significant compositional variability observed for all the minerals concerned, to the point of defining distinct sub-groups (high-Pb tsumoite, low-Pb kochkarite, high-Se rucklidgeite; Fig. 5). That variability, in particular for the Pb concentrations, is the highest for kochkarite and rucklidgeite: in fact, their Pb concentrations overlap to a very high extent (Fig. 5). This observation raises questions about the chemical definition of these two minerals (and also about labelling them in any particular investigation, as discussed below; see also Cook et al., 2007 and Gu et al., 2001). For instance, it should be possible to distinguish kochkarite and rucklidgeite from each other by the Bi/Pb ratio in their structural formulae as this is ideally 0.750 for rucklidgeite ((Bi,Pb)<sub>3</sub>Te<sub>4</sub>) and 0.714 for kochkarite (PbBi<sub>4</sub>Te<sub>7</sub>) (Anthony et al., 1990). However, the average total cation/total anion ratios resulting from this compilation are on the order of 0.80 for kochkarite, rucklidgeite and high-Se rucklidgeite, and ~0.71 for low-Pb kochkarite (Table 1): these are significantly different from the theoretical

## Labelling, or naming, of bismuth tellurides

A direct and practical consequence of the above comments is that sometimes it might be problematic, in the absence of structural

information, to know which bismuth telluride is observed when new data are collected. The present investigation is a pertinent example of this difficulty.

The Stall Lake deposit bismuth tellurides fall into five empirically defined compositional groups, as described above (Mineral description; Table 1; Fig. 5), and the question that emerged immediately was to what minerals these compositions correspond (again, in the absence of structural data). This question is most easily answered for compositional groups 1 and 2, which seem to closely resemble the high-Pb tsumoite visible in the literature data compilation (Fig. 5; Table 1). Specifically, (1) the cation/anion ratios in their structural formulas are very similar, at  $\sim 1$  (no other bismuth tellurides have the same ratio; telluronevskite is not an option because of its high Se; Anthony et al., 1990); and (2) the amounts of Pb in the Stall Lake groups 1 and 2 (~3.3 and ~5.1 wt.%) are fairly comparable to those of literature data high-Pb tsumoite (~4.6 wt.%; Table 1; VMS deposits in the Ural, Russia: Belogub et al., 2011). It seems that these two compositional groups can be regarded as high-Pb tsumoite (e.g. Cook et al., 2007).

The other three compositional groups from Stall Lake are more difficult to label. The total cation/total anion ratio of their average structural formulae are ~0.74, which is closest to theoretical rucklidgeite (0.75; Anthony *et al.*, 1990). However, the literature compilation (Table 1; references at the end of Sampling and analytical methodology) reveals that kochkarite and rucklidgeite have cation/anion ratios between 0.71 (low-Pb kochkarite) and 0.81 (kochkarite, rucklidgeite), indicating that it may be indeed challenging to decide what the Stall Lake compositions are.

Based on its amount of Pb, the Stall Lake deposit compositional group 3 (Pb  $\approx$  7.2 wt.%) is closest to low-Pb kochkarite (compilation group 8: Pb  $\approx$  7.0 wt.%; Roberts, 2017; Table 1); the amount of Pb is significantly lower than that of rucklidgeite (ranging from  $\sim$ 10.8 to  $\sim$ 20.9 wt.%; references at the end of Sampling and analytical methodology). Hence, this bismuth telluride compositional group can probably be named low-Pb kochkarite.

The greatest difficulty is deciding what the Stall Lake compositional groups 4 and 5 are, the high-Se bismuth tellurides. The average cation/anion ratio of their structural formulas ( $\sim$ 0.73) are not sufficiently close to either literature data kochkarite or rucklidgeite ( $\sim$ 0.8) to be of any help. Based uniquely on the Pb contents of these two groups (at  $\sim$ 10.9 wt.%; Table 1), kochkarite was preferred, as its Pb content is  $\sim$ 11.8 wt.% on average (Table 1). This is not ideal and serves to illustrate the difficulties in naming unknown bismuth tellurides.

## The high-Se bismuth telluride phases from Stall Lake

The difficulty described above is compounded by the very high degree of Se substitution observed in the Stall Lake deposit compositional groups 4 and 5 (average Se  $\approx$  4.9 and  $\approx$  7.2 wt.%, respectively; Figs 4 and 5), which had never been observed in either kochkarite or rucklidgeite. These concentrations are lower than in poubaite (Se  $\sim$ 13.6 wt.% on average; Anthony  $et\ al.$ , 1990; Kalinin, 2021; Johan  $et\ al.$ , 1987); poubaite also has more Pb ( $\sim$ 20.1 wt.% on average). The high amounts of Se in the Stall Lake compositional groups 4 and 5 clearly demonstrate the potential for anion substitutions in bismuth tellurides, as noted above, however this also raises the question of what mineral(s) these two groups correspond to.

The two most likely hypotheses are (1) the existence of new minerals or (2) the existence of a high-Se kochkarite or rucklidgeite. New minerals with these compositions would have the ideal structural formulas of  ${\rm Bi_4PbTe_6Se}$  (group 4) and  ${\rm Bi_4PbTe_5Se_2}$  (group 5). However, such a hypothesis cannot be addressed here because of the absence of structural data, highlighting in the process the absolute necessity to collect, when at all possible, crystal structure data for any mineral studied. The second hypothesis seems more likely and, on the basis of the Pb contents of these compositional groups (both are, on average,  $\sim 10.9$  wt.%; Table 1) the term 'high-Se kochkarite' was preferred for them. This labelling is far from satisfactory or conclusive and should be taken with a pinch of scepticism.

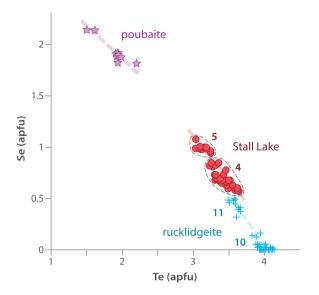
One corollary of the high Se observed in the Stall Lake bismuth tellurides is speculation regarding a possible solid solution between kochkarite and poubaite or between rucklidgeite and poubaite (e.g. Kase et al., 1993). The former possibility can be dismissed immediately, as kochkarite is cubic (point group  $4/m \ \bar{3}$ 2/m), whereas poubaite is hexagonal (point group  $\bar{3}$  2/m; Anthony et al., 1990). The latter possibility remains theoretically possible, as both poubaite and rucklidgeite are hexagonal, with the same point group  $(\bar{3} \ 2/m)$ . They also have comparable amounts of Pb (~20.1 for poubaite and ~16.1 for high-Se rucklidgeite) and the same cation/anion ratio of 0.75 (Anthony et al., 1990). In such a scenario, the high-Se phases from Stall Lake (compositional groups 4 and 5) could be considered as belonging to this hypothetical solid solution if they are in fact (low-Pb) rucklidgeite. The high-Se literature data for rucklidgeite (compositional group 11; Kasprowicz, 2023) would also belong to such a solid solution (Fig. 6). However, the lesser amount of Pb in Stall Lake compositional groups 4 and 5 compared to poubaite, and the far from consistent or continuous Te-Se anti-correlation (Fig. 6) are arguments against such a possibility. It can be concluded that while a poubaite-rucklidgeite solid solution might be theoretically possible, the present work, specifically the existence of a high-Se bismuth telluride compositions at Stall Lake, is not a sufficient proof.

# **Summary**

A few interesting generalisations can be made from the literature data compilation of four bismuth tellurides and new data obtained at the Stall Lake VMS deposit, the first is that these minerals have a highly variable composition (Fig. 4, Table 1). They all contain at least a modest degree of cation and anion substitutions, with (in decreasing concentrations) Pb, Ag, Fe, Cu, Sb and Au substituting for Bi, and Se and S substituting for Te. All these elements should be therefore routinely analysed for in bismuth tellurides. The amount of substitution cations other than Bi and Pb is in the vicinity of 2 wt.%, Se and S combine to under 1 wt.%.

Bismuth tellurides are not only more chemically complex than previously appreciated, but demonstrate fairly high compositional variability, to the point of defining specific mineral varieties such as high-Pb tsumoite, low-Pb kochkarite and high-Se rucklidgeite (Fig. 5). Their empirical compositions are commonly different from their ideal structural formulae (Fig. 5). The highest variability is observed in the amount of Pb in kochkarite and rucklidgeite, with the former varying between  $\sim 9.5$  and  $\sim 16.0$  wt.%, and the latter between  $\sim 10.8$  and  $\sim 20.9$  wt.% (Table 1). These two minerals' compositions overlap to such an extent that distinguishing them from each other conclusively might, in some cases, only be possible using crystal structural information. In practical terms, this compositional variability can sometimes represent a difficulty with the

10 Paul Alexandre



**Figure 6.** A speculative rucklidgeite-poubaite solid solution, visualised by the Se substitution of Te in rucklidgeite, high-Se rucklidgeite, the two high-Se Stall Lake compositional groups, and poubaite. The atoms per formula unit (apfu) were calculated on the basis of 7 atoms, for a generic formula (Bi,Pb)<sub>3</sub>(Te,Se)<sub>4</sub>, which describes both rucklidgeite and poubaite. The Se–Te anti-correlation is far from perfect in terms of consistency and connectivity, which, combined with the low Pb concentrations in the Stall Lake bismuth telluride and the lack of crystal structure data, renders the existence of such a solid solution currently unsubstantiated.

classification of an unknown bismuth telluride, as this investigation demonstrates.

Two new high-Se bismuth telluride compositions were discovered at the Stall Lake VMS deposit, containing, on average, ~43.4 wt.% Bi, 10.9 wt.% Pb, 39.6 and 36.8 wt.% Te, and 4.9 and 7.2 wt.% Se (Table 1; Fig. 5). These two compositional groups were tentatively labelled high-Se kochkarite. Such a high extent of Se substitution had never been observed for kochkarite or rucklidgeite and opens a few possibilities, one of which is the existence of two new minerals, with the ideal structural formulae of Bi<sub>4</sub>PbTe<sub>6</sub>Se and Bi<sub>4</sub>PbTe<sub>5</sub>Se<sub>2</sub>. It can be speculated that the Stall Lake high-Se bismuth telluride belongs to a hypothetical rucklidgeite–poubaite solid solution (Fig. 6), even though the available data, from literature or the newly obtained, do not conclusively support such a possibility.

As defining a new mineral or a solid solution would strongly rely on crystal structure data, which were not available for the Stall Lake phases, these speculations cannot be fully addressed at present. However, these new data, showing high and variable Pb and Se substitutions, emphasise the high compositional variability and the high extent of both cation and anion substitutions in bismuth tellurides, which is the main significance of this contribution.

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