# MINERALOGY AND GEOCHEMISTRY OF THE TIN-TUNGSTEN DEPOSIT IN SINTOK, KEDAH, MALAYSIA

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Abstract. The Sintok mine in northern Kedah is known for tin-tungsten mineralization in quartz veins and intrusive rocks as well as in sheeted vein hosted within metasedimentary rock. This study focuses on the mineralogy and geochemistry of ore body within the intrusive that is currently being mined. The ore is dominated by cassiterite and wolframite in a northsouth trending ore zone (Zone 1-unoxidized and Zone 2-oxidized). Zone 1 consists of narrow, whitish quartz porphyry with disseminated cassiterite-wolframite. Oxidized Zone 2 consists of a brownish ore zone with intense veining and hosts significant Sn mineralization with up to 1.2% Sn, mainly at the contact with the western wall rock. The samples were analyzed by XRD analysis, scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) and inductively coupled plasma mass spectrometry (ICP-MS) for the mineral composition of the ore and multi-element analysis. Based on the XRD analysis, the typical metallic minerals that make up the ore mineralization are cassiterite, wolframite with sulphide mineral that oxidized to copper(ii) oxide, arsenic oxide, todorokite (hydrous manganese oxide) and other secondary iron oxyhydroxides. The SEM-EDX study confirms that the ore mineral composition is Sn and W with invariable Fe and minor As and Mn. Multi-element analysis of the bulk samples shows that the Sn-W mineralization is associated with Mn, As, Cu, Zn, Rb and Pb. The Sintok deposit has similar characteristics to other hydrothermal tin-tungsten vein deposits with part of the deposit are hosted in intrusive rocks.

Keywords: Geochemistry, mineralogy, cassiterite, wolframite, hydrothermal vein

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#### 1. INTRODUCTION

Tin is one of the first metals to be used by humans. Tin metal is almost invariably used as an alloy. Because of the toughening effect of copper, tin metal was used in bronze objects as early as 3500 BC [1]. Today, tin is mainly used for building materials, cans and containers, transportation goods and solders. The sheet metal industry and the development of tin metal in wire coating in the wake of the expansion of the electricity industry have triggered an increase in demand for tin ores. Peninsular Malaysia contributed significantly to tin production until the 1980s, particularly from tin deposits in the Kinta Valley in Ipoh [2]. The revival of the tin price and increased demand for metals, including tin, is now evident in the growth of several former tin fields, such as in the Sintok area. It has therefore been increasingly recognised that more attention needs to be paid to this area.

The aim of this study is to understand the mineralogy and geochemistry of the tintungsten deposit in Sintok, Kedah based on a new exposure from the mine area. Geological mapping and ore observation were studied on the pit wall which provides better continuity of the ore zone. Previous authors such as [3-4] provided early geological observations and interpretations of tin mineralization in the Sintok area as a vein type based on the limited surface exposure and underground workings. The other major tin deposits of similar type are Sg. Lembing and Klian Intan [4-6]. This study provides much detail near surface geology and lab analysis by different characterization techniques to understand the research topic.

### 1.1 Tectonic Setting and Regional Geology

The Southeast Asian tin belt is known worldwide for its wealth of tin deposits. Since 1800, more than half of the world's tin production has been mined here [7]. The Southeast Asian Tin Belt stretches over 3000 km, from Myanmar, Thailand, Malaysia, and Singapore to Indonesia [8]. The Southeast Asian Tin Belt is centred on both the tectonostratigraphic terranes of the Sibumasu Block (Sino-Burma, Malaya, and Sumatra) and the East Malaya Block during the Paleozoic and Mesozoic [9-12]. This Southeast Asian tin belt comprises at least three major granitoid provinces: (i) the Western Province, which extends from southwest Thailand to eastern Burma; (ii) the Main Mountain Province (main mountain granite of Peninsular Malaysia), which extends from western and southern Thailand to western Malaysia; (iii) the Eastern Province, which extends from Laos through eastern and central Thailand to eastern Malaysia [10].

The intrusion of highly developed Late Triassic post-collisional granites was the most important event for the Main Range Granitoid [13]. In the western belt, the granitoid consists mainly of biotite granite with localised hornblende-biotite adamellites. Tin deposits associated with the biotite granite that make up the Main Range Granitoid and the northern extension account for more than 55% of the tin produced in Peninsular Malaysia, the southern peninsula and central Thailand [7,14]. Primary tin ore deposits are associated with magmatic-hydrothermal systems that are invariably associated with end-stage granitic phases such as pegmatites and tin intrusive porphyries [2]. There is overwhelming evidence that one metal predominates over the other in hydrothermal granite-related Sn and W deposits [15].

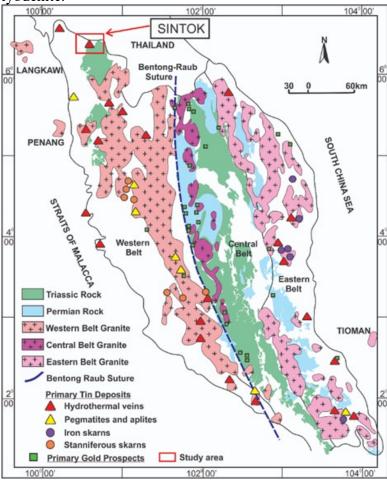
### 1.2 Granitoids of Malaysia

The intrusive rocks of Peninsular Malaysia consist of three major granitic provinces: the Main Range Granitoid, the Eastern Belt Granite and the Central Belt Sub-unit Granite

[16-17]. The Late Triassic Main Range granitoid province west of the Bentong-Raub suture consists predominantly of biotite granite with features of the S-type and ilmenite series [7,16,18]. Magma sources and redox states are important during the tectonic and orogenic episode that controls the completion of the Paleo-Tethys and play an important role in controlling tin formation and tin-bearing granite in Peninsular Malaysia [4]. The studies by [19] also show that granites and tungsten-tin deposits are inextricably linked.

# 1.3 Geology and Tin Mineralization

The tin-tungsten mineralization in Sintok mine is mainly distributed in the quartz vein zone and disseminated in the white to yellowish alteration zone in narrow intrusive. The alteration zone has a clear boundary with the barren host rock and may have formed within the shear zone. The location of Sintok and other tin deposits in Peninsular Malaysia is shown in Figure 1. This figure shows various types of tin deposit in Peninsular Malaysia including hydrothermal veins, pegmatites and aplites, Sn-Fe skarns and stanniferous skarns. Structural deformation and veins are associated with late-stage hydrothermal fluids that are believed to have formed shortly after the major granite intrusion. The veins run parallel to the shear and differ from the Klian Intan tin deposit where the veins run perpendicular to the main shear [6-7]. The tin-tungsten deposit in the Sintok is associated with chalcopyrite, pyrite, arsenopyrite, scheelite and molybdenite.



**Figure 1**: Geological map of Peninsular Malaysia and the distribution of the various tin deposits including Sintok, modified after [11].

In the Sintok area, significant quantities of cassiterite and wolframite have been recovered from numerous shear-dominated veins. As no granite outcrops in the vicinity of this area, no radiometric age is available. The closest data are from the Langkawi Island granite with a date of 215.3 +/- 2.6 Ma [8]. The age of tin mineralization at Sintok is 218.9+/- 3.4 Ma based on cassiterite U-Pb age [4], which is consistent with magmatic activity in the northern region.

A narrow quartz porphyry intrusive is observed within the active mine in this study. The exposed north-south trending orebody is interpreted as a hydrothermally altered and sheared narrow intrusive consisting of quartz veins and mica-rich on several occasion. Within underground working, series of north-south trending sheeted quartz vein in metasedimentary rock. Figure 2(A) shows the location of the bulk samples in the mine area. The area was cleaned with the excavator prior to sampling. The thickness of Zone 1 and Zone 2 varies in the narrow intrusive rock. Zone 1 is wider in the southern part, as can be seen in Figure 2B. The close-up of Zone 1 shows the coarse-grained intrusive rock with disseminated dark minerals (Figure 2C). Zone 2 is slightly weathered and contains intense quartz veins.



**Figure 2**: (A) Location of the bulk samples in the mine area. Zone 1; No 1 (sample BSWA-whitish ore, brownish ore from Zone 2; (sample BSO1B), 3 (BSO2C), 4 (BSO3D) and 5 (BSO4E). Location of samples 3-5 consist of dominant quartz veins, (B) The zone 1 area locally forms a broader zone to the north and (C) A close-up of the rock in Zone 1 shows quartz grains in a clayey mineral forming a texture of quartz porphyry with disseminated Sn-W mineralization

#### 2. MATERIALS AND METHODS

Field work was conducted at the Sintok tin-tungsten mine to collect the representative samples for mineralogical study. Mapping of the ore body, host rock, veins distribution and structural geology for better understanding of geological setting. A total of four samples were taken from the ore body at approximately 1m intervals. One additional sample was collected from the same vein of the western footwall contact of Zone 2 on the north side to investigate the geochemical composition of the ore and to perform XRD and SEM-EDX analysis. Selected samples for the polished section were taken from both the veins and the host rock. Samples were also taken from the processing plant concentrates.

A small portion of each bulk sample was subjected to X-ray diffraction (XRD) analysis to determine the type of minerals present in the samples. Quantification and qualification of minerals was performed using X'Pert HighScore software based on phase identification and unit cell size. Five samples were selected for mineral identification. The polished samples of the representative vein with visible Sn-W mineralization, especially cassiterite with wolframite, and the altered zone were examined under a reflected light microscope to observe the mineral association. The most important analysis is the scanning electron microscope with energy dispersive X-ray microprobe (SEM-EDX, Carl Zeiss, SUPRA35VP, Carl Zeiss, Oberkochen, Germany). Surface mapping and spot analyses were performed to observe the elemental composition and texture of the mineral phases.

All samples for geochemical analysis were crushed, pulverised, and split. A total of 100 g of each sample was sent to an Actlab laboratory (Canada) for multi-element analysis by ICP-MS (inductively coupled plasma mass spectrometry. Following the Actlab laboratory (Canada) procedure, the samples were digested with a combination of concentrated hydrochloric acid and nitric acid using the aqua regia dissolution method to leach sulphides, some oxides, and some silicates. Sixty elements, including rare earths elements, were analysed. This method provides high accuracy in the analysis of sulphide elements (As, Cu, Zn, Pb).

However, the resistant minerals such as cassiterite and rare earth elements cannot be effectively determined using the previous technique. Therefore, the replicate samples were re-analysed for Sn and other elements including rare earth elements using a lithium borate fusion technique prior to acid dissolution and then by ICPMS analysis with an upper limit of 10,000 ppm Sn. One sample exceeded this limit and was re-analysed using the sample fusion technique commonly used for rare earth elements with a limit of 50,000 ppm Sn. Only significant results and those related to tin-tungsten are presented in this study.

# 3. RESULTS AND DISCUSSION

In the past, many old tunnels were dug to extract the tin-tungsten contained in the veins of the Sintok mine. A new adit is currently being developed by the company to observe the continuity of the veins in the open pit mine. The shear zones currently being mined in the open pit extend to depth and exposed in the new adit consisting of Zone 1 and Zone 2. This study focuses only on the main ore body currently exposed in the open pit. The ore body runs in a north-south direction and has a steep dip of approximately 80° to the east.

### 3.1 Ore Minerals by Hand Specimen Study

Tungsten, cassiterite, quartz and mica were discovered in a hand sample (Figure 3). Photomicrographs of the selected cassiterite, wolframite and outcrop samples from the site are shown in Figure 3. Mineralization is restricted to the north-south trending shear zone, which is filled like an orebody with shear-parallel veins and hydrothermally altered zones. The ore body consists of an altered whitish zone of kaolinite with quartz grains (zone 1), which resembles a porphyry texture. The brownish coloured zone with mica-like minerals and thin, foliated, north-south trending veins is dominated by shear-parallel veins at the western footwall contact. Coarse wolframite and fine cassiterite can be observed in the veins, but both minerals are only clearly visible in section in the whitish alteration zone. The shear zone cuts through the foliation, which is oriented approximately 320 °NW and dips 45° to the east. Further series of quartz veins up to 1 m width and running in the same direction can be observed in the adit.

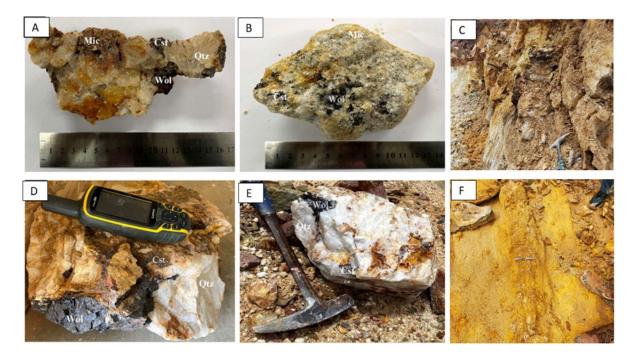


Figure 3: Hand specimens of tungsten (Wol), cassiterite (Cst), quartz (Qtz) and mica (Mic). (A) Quartz vein with wolframite, cassiterite and mica in the rim zone of the altered host rock, (B) Hydrothermally altered whitish zone with scattered mica (most abundant) and occurrences of cassiterite, wolframite and other minerals, (C) Zone 2 at the rock boundary (footwall), which hosts the main veins and is the richest zone in Sn and W, (D) Quartz vein with north-south orientation dominated by wolframite and less cassiterite, (E) Quartz vein with wolframite and cassiterite and (F) Brownish Zone 2 consists of oxidized, silty rock with a thin, shear-parallel, foliated vein at the boundary with Zone 1

### 3.2 XRD Analysis

The mineral phase of selected mineralized veins of Sintok oxide ore by XRD analysis is shown in Figure 4. Distinct 20 peaks with diffraction angles of 26.97, 21.19, 18.08, 12.59 and 45.69 were found. Using XRD, distinct peaks can be found that indicate related minerals

that are important for tin mineralization. The main minerals are quartz and dominate in all samples followed by kaolinite in samples BSWA and BCO1B which are less quartz vein and rich in clay-rich mineral. Minor compositions are tungsten (IV) oxide, tin oxide, clauditite (As2O3), todorokite (complex hydrous manganese oxide) and others (see Table 1, Figure 4).

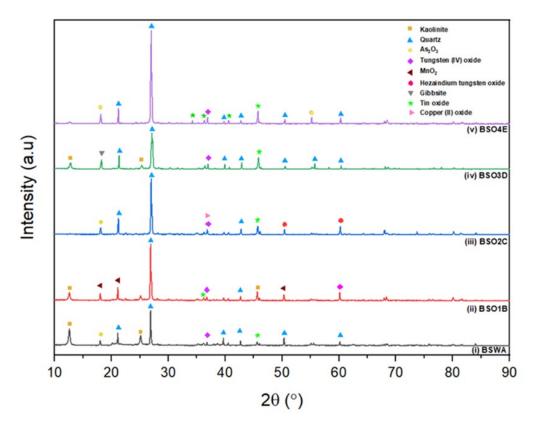


Figure 4: Mineral phases of selected mineralized veins from Sintok using XRD analysis

**Table 1**: Quantification of mineral based on the XRD analysis of bulk samples in percentage. BSWA (Zone 1), BSO1B and others are in veins dominant samples of Zone 2

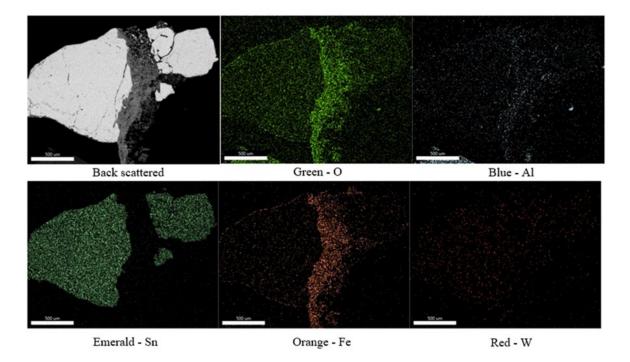
Sample no.	Zone	Kaolinite	Quartz	$(\mathrm{As}_2\mathrm{O}_3)$	Tungsten (IV) oxide	(MnO <sub>2</sub> )	tungsten oxide	Gibbsite	Birnessite	Tin oxide	Arsenic		Krautite	Copper (II) oxide
BSWA	1	68.2	30.4	0.2	0.1					0.1			1.0	
BSO1B	2	38.3	58.4		0.2	3.0				0.1				
BSO2C	2		94.3	2.4	0.5		0.9			0.2	0.3	1.2		0.2
BSO3D	2	0.8	76.7		0.3			17		0.3				
BS04E	2		89.7	7.1	0.2	2.1			0.1	0.8				

The reduction in peaks was found to correspond to the uniform distribution of elements in the diffraction pattern matrix. It was also observed that the centre of the peaks shifted towards the lower value. Stressed evaluation in the matrix causes the amplitude of the peaks to decrease. Simple quartz vein of cassiterite-wolframite can be observed and cassiterite-wolframite with a minor copper oxide and arsenic oxide (claudetite-As2O3) due to

oxidation of the original sulphide mineral. Mn, which is thought to be related to tin mineralization, can be abundant in late intrusive rocks such as pegmatite and can be converted to hydroxide/oxide mineral by oxidation with water (Post, 1999), including todorokite (a type of manganese hydroxide mineral).

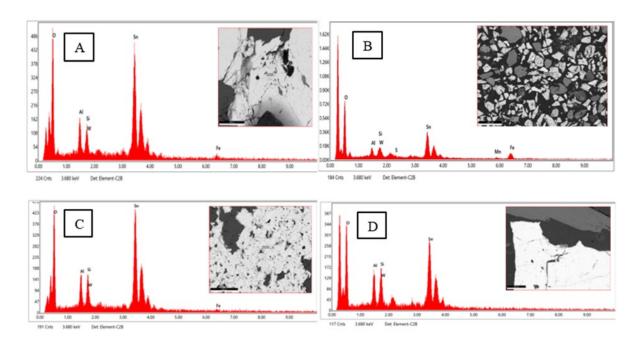
# 3.3 SEM-EDX Analysis

Examination by SEM-EDX shows minerals containing the elements Sn, W, Si, O, Al as well as Fe and the presence of Mn, As, In and S in the samples as indicated in Figures 5 and 6 and Table 2.



**Figure 5**: SEM-EDX elemental mapping of the cassiterite in vein sample BSO3D (scale bar = 500 micron) shows secondary Fe-Oxide infilled the fracture in cassiterite

There are minor occurrences of indium in samples BSO3(D). The most common gangue minerals and secondary minerals after cassiterite and wolframite are quartz and iron hydroxide. The fracture of cassiterite is filled with iron oxides. Cassiterite and quartz often exhibit strong brecciation and the spaces between the fracture grains are often filled with quartz. This shows that quartz veins containing cassiterite can be found in the samples within the shear and hydrothermally altered zone. Tiny and small hairline cracks in the host minerals filled with secondary iron oxide.



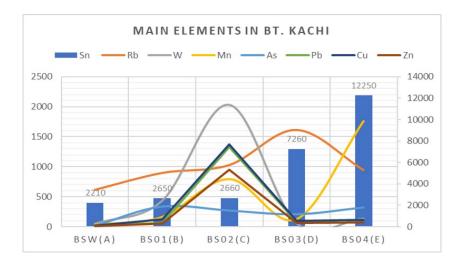
**Figure 6**: The SEM-EDX analyses shown in (A), (B), (C) and (D) indicate the presence of W, Si, Al, Fe and Sn-related minerals in the selected sample by EDX analysis. Figure B shows the cassiterite concentrate from the plant process with Sn, W, Al, and others

**Table 2**: Chemical composition (wt %) by SEM-EDX microprobe of the selected mineralized veins and tin concentrates from the Sintok area

Sample no.	Zone	0	Al	W	Sn	Mn	Fe	As	In	S
CBK		20.46	4.50	6.23	46.01	3.56	15.23	-	-	2.09
BSO3(D)		16.94	5.78	3.62	66.37	-	5.03	-	-	-
BSO3(D)		26.47	14.94	5.42	36.13	-	5.34	-	-	-
BSO3(D)		36.05	14.43	9.09	8.35	-	4.37	1.58	11.14	-
BSO3(D)		36.28	18.53	5.34	2.79	-	5.05	1.18	14.13	-
PK4		34.33	18.10	7.42	5.71	17.95	3.68	-	-	-
PK4		32.88	6.22	7.62	3.08	34.99	2.66	-	-	-
PK4		12.58	1.85	2.19	82.99	-	-	-	-	-
PO1		15.90	5.96	3.61	67.22	-	4.04	-	-	-
PO1		28.88	9.23	5.91	31.27	-	4.04	-	-	-
TCPY		10.72	1.23	2.51	85.54	-	-	-	-	-
TCPY		16.78	7.47	4.64	60.47	-	3.67	-	-	1.61
TCPY		22.77	9.70	30.06	6.76	9.24	11.71	-	-	-
TCPY		15.96	3.20	4.02	61.59	-	12.98	2.25	-	-
TCPY		9.97	0.91	1.63	84.19	-	2.43	0.87	-	-

### 3.4 ICP-MS Analysis

The geochemical analysis of sixty-three elements such as tin (Sn), tungsten (W), arsenic (As), zinc (Zn), lead (Pb), copper (Cu), manganese (Mn), lead (Pb) from bulk samples is shown in Table 3 and a few elements are plotted as in Figure 7.



**Figure 7**: Distribution pattern of main elements in Sintok. Sn is shown as histogram on the right axis. All results are in ppm

**Table 3**: All data from two analyses of ICPMS with different samples dissolution. All units in ppm except those indicated with the analyte

	Ac		Lithi	Lithium borate fusion for resistive mineral							
Analyte	BSW (A)	BS01 (B)	BS02 (C)	BS03 (D)	BS04 (E)	Analyte	BSW (A)	BS01 (B)	BS02 (C)	BS03 (D)	BS04 (E)
Fe (%)	0.19	1.3	1.04	0.83	1.02	Sn	2210	2650	2660	7260	12250
Al% Ti (%)	1.41	0.68	0.22	0.58	0.26	Ce Cr	49.4 10	45.8 30	23.9 50	50.1 20	42.4 20
S (%) Cu	0.01 0.008 14.5	0.01 0.011 125	0.01 0.178 1370	0.01 0.013 101	0.01 0.01 115	Cs Dy	28.6 0.66	47.3 1.39	62.6 1.24	97.2 0.78	58.3 0.96
As	18	334	268	207	319	Er	0.19	0.65	0.67	0.24	0.34
Zn	10.7	54.6	953	64.1	67.6	Eu	0.16	0.28	0.25	0.3	0.25
Pb	29.1	59.3	1330	83.1	118	Ba	26	140	189	73.1	139
Co	0.2	0.4	0.4	0.5	8.6	Ga	53	41.6	36.9	65.7	36.4
Ni	2.2	2.1	3.8	3.1	2.6	Gd	0.88	1.55	1.25	1.41	1.34
Sb	3.8	17.7	20.2	4.41	13.1	Hf	5.7	4.1	6.6	4.6	3.3
Mo	0.83	3.33	4.91	4.57	4.39	Ho	0.08	0.23	0.22	0.09	0.14
Mn	39	165	795	128	1760	La	40.8	35	13.7	55.2	24.9
B	2	3	4	3 0.3	3	Lu	0.07	0.17	0.09	0.07	0.08
Se	0.3	0.6	1.7		0.9	Nb	90	61.9	54.2	51.4	46.3
Sc	0.6	1.2	0.8	1.4	1.7	Nd	13.5	13.3	8.8	25.7	13.3
Mo	0.83	3.33	4.91	4.57	4.39	Sn	2210	2650	2660	7260	12250
Ag	0.032	0.263	7.43	0.388	1.62	W	46	430	2030	58	133
Cd	0.02	0.15	6.08	0.25	0.53	Pr	5.31	4.89	2.61	8.77	4.51
In	0.12	0.51	2.1	0.39	0.71	Rb	617	898	1030	1615	946
Sb Te Re	3.8 0.02 <	17.7 0.24 <	20.2 0.23	4.41 0.13	13.1 0.57 <	Sm Ta Tb	2.1 80.3 0.14	2.71 60.9 0.27	1.75 58.8 0.23	3.37 71.8 0.18	2.47 79.6 0.2
Au	0.001 0.7	0.001 10	0.001 338	0.001 8.6	0.001 4.6	Th	9.09	7.97	7.17	6.18	6.03
Tl	0.1	0.15	0.16	0.21	1.88	Tm	0.04	0.13	0.1	0.04	0.06
Bi	6.09	21.3	41.6	8.62	38.4	U	3.76	5.15	3.68	2.63	2.45
Hg	10	< 10	< 10	< 10	< 10	V	<5	14	24	<5	6
Ge	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	Sr	5.7	19.6	21.6	9.8	7
Li	4	4.7	11.7	11.4	7.7	Y	2.4	6.7	6	2.2	3.7
Be	1.4	1.1	0.4	1.1	0.5	Yb	0.47	1.25	0.66	0.46	0.56

The most frequently occurring elements are Al and Fe with concentrations of 0.22 to 1.41% and 0.19 to 1.30% respectively. Al represents the clay host rock within the ore zone and Fe possibly originates from the oxidised zone due to weathering of the sulphide and iron enrichment. Sn and W range from 2,210 to 12,250 ppm and 46 to 2,030 ppm respectively. Significant Sn mineralization increases from the east towards the western contact (footwall) within the quartz veins that dominate Zone 2 at the contact with the footwall metasediment, e.g. in samples BS03(D) and BS04(E).

W shows a different trend across the orebody, with the higher grade centred in the brownish zone located in the quartz vein zone adjacent to the Sn-rich zone. The Sn and W curve (Figure 7) does not correlate well with the highest Sn grade. Wolframite often occurs in the vein as larger aggregates.

The vein has a more irregular shape than the vein in the Sn-rich zone at the base of the wall. Cu, As, Zn, Pb and Mn are the most important trace elements and lie between 14.5 and 1760 ppm. Only Rb generally shows an increasing trend in intensity from east to west, indicating the enrichment of this element related to the high intensity of the quartz vein (zone 2) compared to the whitish zone (zone 1). Arsenic shows an even distribution throughout the ore body and is slightly higher in the silty, brownish Zone 2 and the western quartz vein zone. The trend of Cu, Zn and Pb is similar to W, indicating a close relationship between these minerals. Mn also shows a similar trend, but its concentration is higher in the western quartz vein in association with Sn. Two sets of data from different samples dissolution are tabulated in Table 3.

#### 4. CONCLUSIONS

Tin mineralization at Sintok is generally confined to a narrow N-S trending shear zone characterized by strongly hydrothermally altered zones and quartz veins. Sintok is like other vein-type mineralization in Malaysia where mineralization is dominantly in quartz vein and partially disseminated within the host rock. Tin-tungsten mineralization fluid is originated from local granitic intrusive based on the temporal relationship between the earlier U-Pb cassiterite dating of Sintok and the age of local magmatism in the northern region, tin and tungsten could be interpreted as genetically related. The most common metallic minerals associated with tin-tungsten mineralization based on XRD analysis are arsenic oxide, oxidized manganese of todorokite (hydrous manganese oxide), while minor minerals are tungsten oxide, chalcopyrite, copper(iii) oxide, hematite, pyrrhotite and chalcocite. SEM-EDX analysis confirms the elements related to cassiterite and wolframite. In summary, based on ICPMS the elements associated with Sn-W include Cu, Pb, Zn, Rb, As and Mn, as in the analysis of the main samples. Sn value is up to 12,250.0 ppm, W up to 2,030.0 ppm and Rb up to 1,615.00 ppm.

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#### **Author Contributions**

All authors contributed toward data analysis, drafting, and critically revising the paper. and agree to be accountable for all aspects of the work.

#### **Disclosure of Conflict of Interest**

The authors have no disclosures to declare

## **Compliance with Ethical Standards**

The work is compliant with ethical standards

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