

TECHNICAL MEMORANDUM: AM/TM/2013_06_18

SUBMITTED TO: TUC Resources

SUBJECT: Mineralogy of the Stromberg Deposit STDH03 Sample

PREPARED BY: B. Young and K. E. Prince

DATE: 18th June 2013

ANSTO Minerals, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia

Ph: +61 2 9717 3858 Fax: +61 2 9717 9129

E-Mail: ansto.minerals@ansto.gov.au

Web: www.anstominerals.com

1. INTRODUCTION

A core sample from TUC Resources' Stromberg Deposit was submitted for mineralogical characterisation. The sample, labelled STDH-03, is described as a "Bleached Breccia". The major REE containing phase, xenotime-Y is said to have leached more readily than expected.

This report presents the results of the mineralogical characterisation of the Stromberg Deposit STDH-03 core sample. Major and minor mineralogical constituents were assessed using QEMSCAN (an automated mineralogical technique). Manual scanning electron microscopy (SEM) was undertaken to identify the REE bearing minerals, their association and liberation characteristics.

2. SAMPLES

A portion of the STDH-03 sample was submitted for chemical assay using XRF.

A portion of the STDH-03 sample was prepared for mineralogical characterization using QEMSCAN and manual SEM. The sample was mixed with similarly sized particles of graphite to ensure good separation of the particles for examination using QEMSCAN. The sample/graphite blend was impregnated with epoxy resin to form a resin block with a polished surface for examination. The polished surface was coated with a thin layer of carbon (a few nanometres thick), prior to analysis, to maintain electrical conductivity during examination.

3. ANALYSIS

3.1 Chemical Analysis

The results of the chemical analysis of the STDH-03 sample are provided in **Table 1**.

TABLE 1

Chemical Analysis of the Stromberg STDH-03 Sample (wt%)

Ce	Dy	Er	Eu	Gd	Ho	La
0.046	0.191	0.12	0.016	0.099	0.034	0.015
Lu	Nd	Pr	Sm	Tb	Tm	Y
0.018	0.066	0.013	0.033	0.029	0.014	1.26
Al	As	Ba	Ca	Co	Cr	Cs
16.1	<0.001	1.43	0.34	0.001	0.001	0.004
Cu	Fe	Hf	K	Mg	Mn	Mo
0.022	1.33	0.003	0.082	0.04	<0.001	<0.001
Na	Nb	Ni	P	Pb	Rb	S
0.01	0.017	0.072	1.76	0.027	<0.001	0.14
Sc	Si	Sn	Sr	Ta	Th	Ti
0.018	21.2	0.001	0.28	0.002	<0.001	0.067
U	V	Zn	Zr			
0.14	0.024	0.006	0.001			

3.2 QEMSCAN

QEMSCAN particle mineralogical analysis (PMA) was carried out using a Quanta 650 electron microscope with dual Bruker XFlash 5030 energy dispersive detectors, controlled by iDiscover and iMeasure image analysis hardware/software.

Some of the REE-containing minerals in the sample could not be conclusively identified due to their small particle size and/or being intergrown with other phases. These unidentified REE-containing phases are reported as “REE Minerals” in the QEMSCAN results.

The QEMSCAN technique is unable to differentiate conclusively between some minerals with similar chemistry. For this reason minerals with similar chemistry have been grouped, e.g. kaolinite and halloysite; “Fe oxide/hydroxide” (magnetite, haematite and goethite). Similarly the crandallite group minerals gorceixite, goyazite, and crandallite have been grouped together due to the QEMSCAN having difficulty in distinguishing between them on account of their finely intergrown nature.

A summary of the modal mineralogy is shown in **Figure 1**. A list of the minerals identified in the sample by QEMSCAN, their empirical chemical formulae and modal abundance is given in **Table 2**.

The STDH-03 sample is dominated by kaolinite/halloysite (64.1 wt%). Minor constituents include the crandallite group minerals (12.5 wt%), quartz (11.8 wt%), smectite (5.5 wt%), xenotime-Y (2.5 wt%) and chamosite (1.8 wt%).

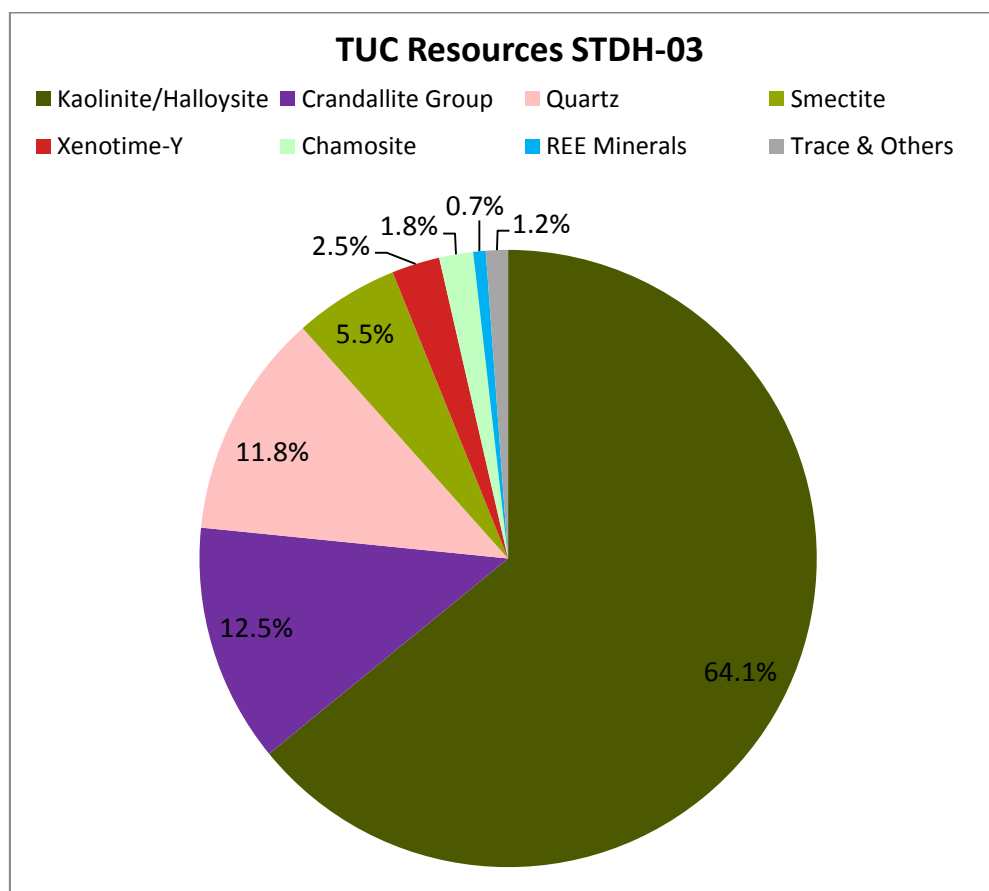


FIGURE 1 Modal Mineralogy of the STDH-03 sample by QEMSCAN Analysis

TABLE 2

Mineral phases identified by QEMSCAN (wt%) in the STDH-03 sample

Mineral Name	Chemical Composition	Wt. %
Kaolinite/Halloysite	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	64.1
Crandallite Group	$(\text{Ba}, \text{Sr}, \text{Ca})\text{Al}_3(\text{PO}_4)(\text{PO}_3\text{OH})(\text{OH})_6$	12.5
Quartz	SiO_2	11.8
Smectite	$(\text{Na}, \text{Ca})_{0.3}(\text{Al}, \text{Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \bullet n\text{H}_2\text{O}$	5.49
Xenotime-Y	YPO_4	2.52
Chamosite	$(\text{Fe}, \text{Mg})_5\text{Al}(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH}; \text{O})_8$	1.78
REE Minerals	Y, REE, P, O	0.65
Muscovite	$\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH}; \text{F})_2$	0.16
K-Feldspar	KAlSi_3O_8	0.14
Other Silicates		0.13
Fe Oxide/Hydroxide	$\text{Fe}_x\text{O}_y/\text{FeO}(\text{OH})$	0.02
Others		0.01
Unclassified		0.69

A comparison of the QEMSCAN calculated assay with the bulk chemical analytical data for the sample is shown in **Table 3**, and a plot of the QEMSCAN assay against the chemical analysis is shown in **Figure 2**. The QEMSCAN assay data is calculated from empirical formulae for the minerals, and can therefore be prone to errors caused by the measured phases varying in composition from the empirical formulae (caused by elemental substitution). The data presented in **Table 3** and **Figure 2** shows there is a reasonably good correlation between the QEMSCAN calculated assay and the measured chemical analysis, particularly for the major and minor elements (*i.e.* concentrations > 1 wt%) in the sample.

Mineral association data for xenotime-Y was calculated using QEMSCAN and is presented in **Table 4**. The QEMSCAN analysis software considers two minerals to be “associated” if a pixel of one mineral occurs adjacent to a pixel of the other mineral. The measured particles are scanned, and the mineral transitions or “associations” are counted. The values presented in **Table 4** are the proportions of transitions between xenotime-Y with associated minerals (listed down the side of the table). For example, 65 % of the transitions of xenotime-Y with other mineral phases occur with kaolinite/halloysite.

The “Background” in the mineral association table represents the epoxy mounting resin. Associations with the background are a result of the mineral being adjacent to the outer edge of the particle, or being located adjacent to an internal void in the particle (*i.e.* porosity, a crack, or a sample preparation artefact). “Unclassified” refers to pixels the QEMSCAN could not assign to a particular mineral, possibly due to mixtures of phases within the analysis volume at the boundary between phases.

The mineral association data in **Table 4** indicates that the majority of associations of xenotime-Y occur with kaolinite/halloysite.

TABLE 3**A Comparison of the QEMSCAN Assay and Chemical Analysis (wt%) of STDH-03**

Element	Chemical Assay	QEMSCAN Assay
Al	16.1	16.4
Ba	1.43	1.61
Ca	0.34	0.43
Ce	0.046	0.044
Dy	0.19	0.11
Er	0.12	0.08
Fe	1.33	1.08
K	0.082	0.042
La	0.015	0.017
Mg	0.040	0.044
Na	0.010	0.009
Nd	0.066	0.017
P	1.76	1.87
S	0.14	0.16
Si	21.2	21.2
Sr	0.28	0.31
U	0.14	0.09
Y	1.26	1.10
Yb	0.092	0.053
Zr	0.001	0.003

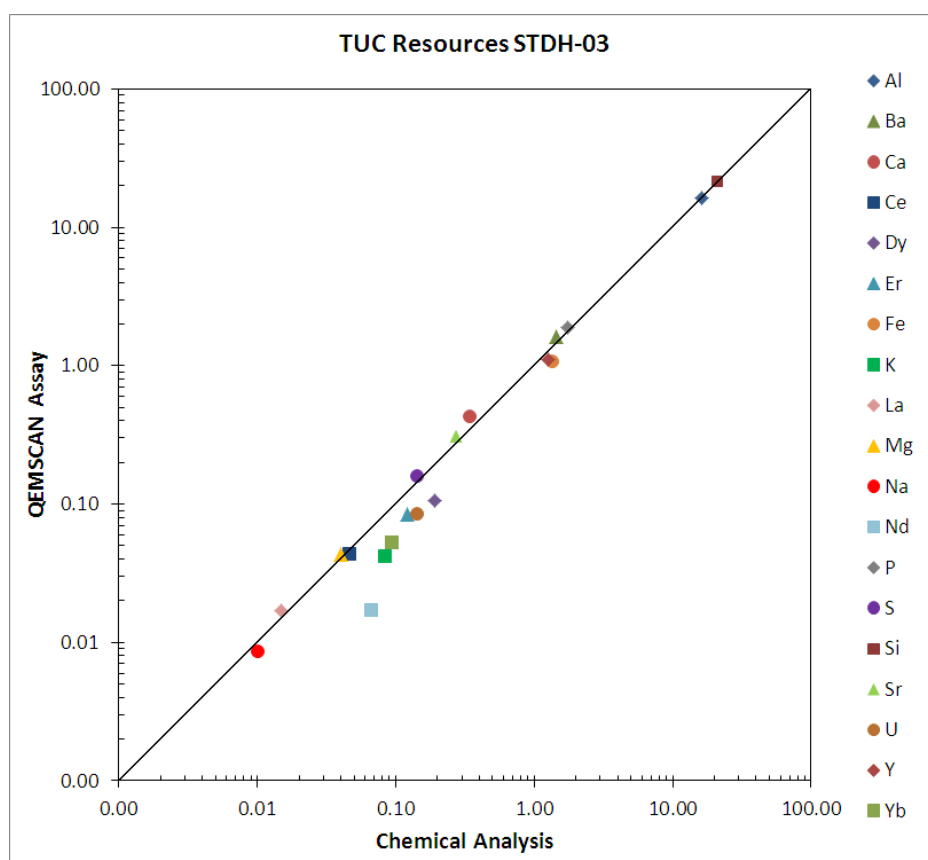
**FIGURE 2 A Comparison of the QEMSCAN Assay and Chemical Analysis (wt%) of the STDH-03 Sample**

TABLE 4

Xenotime-Y Associations in the STDH-03 Sample (% of total associations)

	Xenotime-Y
Kaolinite/Halloysite	65.1
Background	9.6
Smectite	6.8
Quartz	6.2
Crandallite Group	4.0
REE Minerals	3.3
Chamosite	0.8
Muscovite	0.2
Other Silicates	0.2
K-Feldspar	0.1
Fe Oxide/Hydroxide	0.1
Zircon	0.01
Others	0.03
Unclassified	3.5

The liberation statistics for xenotime-Y are presented in **Table 5**. Liberation is calculated for a particular mineral by determining the proportion of the surface area of each grain of that mineral that is in contact with the background (*i.e.* the outer surface of the particle, the epoxy mounting resin). For example, a xenotime-Y grain that exhibits 100% liberation will have its entire surface area in contact with the epoxy mounting resin. 50% liberation means that half of the grain's surface is in contact with the mounting resin, while the other half of the grain's surface area is in contact with other minerals.

The horizontal axis in **Table 5** is divided into ranges that refer to the degree of liberation. The numbers in the table in each of the liberation ranges refer to the proportion (wt%) of xenotime-Y that falls in each range. For example, 2.3 wt% of the xenotime-Y in the STDH-03 sample is contained in grains that have a degree of liberation of 90 to 100%, *i.e.* in grains where more than 90% of their surface area is exposed to the surface of the particle (resin).

The liberation statistics indicate that the majority of the xenotime-Y in the sample is poorly liberated, with 75.7 wt% of the xenotime falling in the 0-10% liberation category. This is consistent with the association data, which suggests that the majority of associations occur with kaolinite/halloysite.

TABLE 5

Liberation Statistics of Xenotime-Y (wt%)

	0-10%	10-20%	20-30%	30-40%	40-50%	50-60%	60-70%	70-80%	80-90%	90-100%
Xenotime (wt%)	75.7	14.0	3.6	0.2	0.3	1.4	0.1	1.4	1.1	2.3

The particle/grain size distribution of the xenotime-Y in the sample as calculated by QEMSCAN is shown in **Table 6**, and is also presented graphically in **Figure 3**. Particle size is calculated by QEMSCAN as the equivalent sphere diameter, i.e. the diameter of a sphere of equivalent area to the particle/grain being measured.

There appears to be two distinct size populations of xenotime-Y; one consisting of particles greater than 30 microns in size and another consisting of particles less than 30 microns in size. This is supported by the observations made in the manual SEM examination of the sample, detailed below in section **3.3 Scanning Electron Microscopy**, with two distinct types of Y-phosphate observed: large, well-liberated particles/grains of crystalline xenotime-Y, and smaller grains of Y-phosphate that does not have the crystalline appearance of xenotime-Y.

TABLE 6

Particle/Grain Size Distribution of Xenotime-Y in the STDH-03 Sample by QEMSCAN (normalised mass %)

	size > 40	size > 35	size > 30	size > 25	size > 20	size > 15	size > 10	size > 5	size < 5
STDH-03	6.7	3.8	0.8	3.0	4.7	9.2	13.5	25.3	33.0

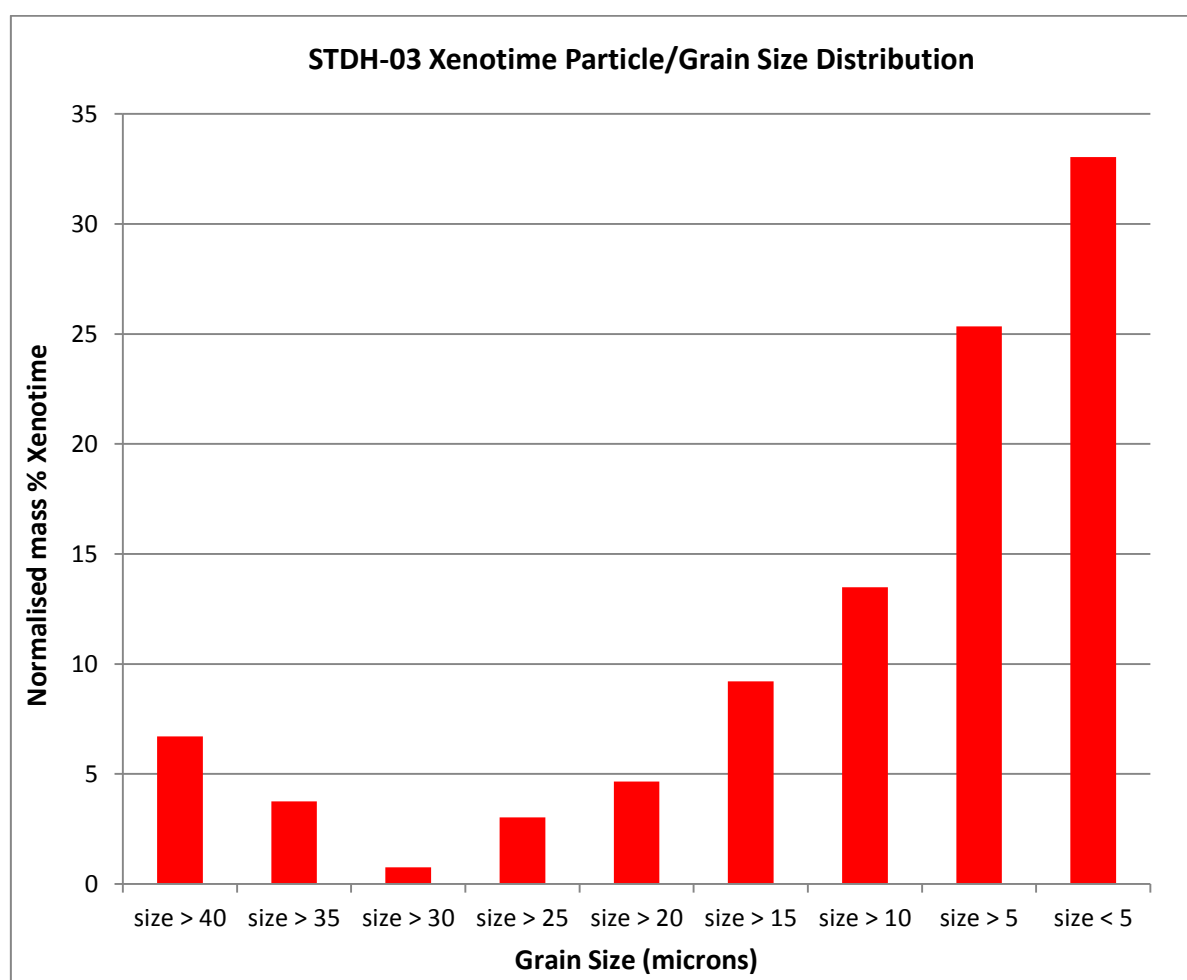


FIGURE 3 Particle/Grain Size Distribution of the Xenotime-Y in the STDH-03 Sample

3.3 Scanning Electron Microscopy

SEM and X-ray microanalysis were carried out on an epoxy resin impregnated portion of the sample using a Quanta 650F electron microscope with dual Bruker XFlash 5030 energy dispersive detectors. The SEM was operated at an accelerating voltage of 15 keV with a working distance of 13 mm. The images were acquired in backscattered electron (BSE) imaging modes. BSE imaging is commonly used to examine variation in chemical composition (evidenced through changing mean atomic number) between and within minerals. Lighter grey levels in the micrographs indicate compositions containing higher mean atomic number elements (e.g. Y, REE) than compositions producing the darker grey levels (e.g. Al, Si, Mg). The black background in the images is the epoxy resin grain mount and any fine detail visible in the black background is most likely from the graphite particles added to aid separation of the bulk concentrate particles. X-ray analysis (energy dispersive system – EDS) was undertaken to confirm mineralogy as far as possible given the small grain size and the intimately intergrown nature of some of the material. The Bruker EDS system's standardless analysis package was used to calculate the normalised elemental composition of the minerals.

The detailed results of the SEM examination of the REE containing phases in the sample are presented in **Appendix A**. A summary of findings is given below.

3.3.1 Summary of Findings

The STDH-03 sample is dominated by kaolinite/clay minerals, with minor amounts of crandallite group minerals (predominantly gorceixite with some crandallite and goyazite), quartz and xenotime-Y. Traces of chamosite and Fe-oxide/hydroxide are also present.

Y-phosphate/Xenotime-Y is the dominant REE containing mineral in the sample. Two distinct types of Y-phosphate are present in the sample: large crystalline xenotime-Y particles/grains that are typically well-liberated; and smaller Y-phosphate grains/particles that do not exhibit the typical crystalline appearance of xenotime-Y.

Examples of the large crystalline xenotime-Y particles/grains are shown in the BSE images in **Appendix A, Figure 1 A – F**, and EDS spectra and normalised elemental compositions from the xenotime-Y particles included in **Appendix A, EDS Spectra S1 – S4**.

The smaller Y-phosphates are typically present as fine inclusions in gangue phases such as kaolinite/clay or quartz. Examples of the smaller Y-phosphate particles/grains are shown in the BSE images in **Appendix A, Figure 2 A – F, Figure 3 A – F** and **EDS spectra S5 – S6**. A significant proportion of the Y-phosphate is in the form of spherical aggregates of very fine, granular Y-phosphate, such as the examples shown in **Appendix A, Figure 3 A – D**. Some examples of round, hollow Y-phosphate “shells” are also present, such as the examples in **Appendix A, Figure 3 E – F**.

The EDS spectra and normalised elemental compositions from the Y-phosphate and xenotime phases show minor and trace concentrations of uranium and also the heavy rare earth elements (HREE) Dy, Er and Yb (**Appendix A, EDS Spectra S1 – S6**). It is possible that there are also traces of other HREE in the xenotime and Y-phosphate phases that could not be detected by EDS analysis, such as Gd, Tb, Ho and Lu, given their detection in the sample in

the bulk chemical analysis (**Table 1**). Peak overlaps and the limits of detection may have prevented their identification in the EDS analysis of the xenotime and Y-phosphate phases. The regions of differing contrast within these structures are indicative of different compositions, however, no definitive statement can be made due to the limitation of the EDS analysis.

The EDS spectrum shown in **Appendix A, EDS Spectrum S4** is typical of the composition of the crandallite group minerals in the sample. Gorceixite, goyazite and crandallite are members of the crandallite subgroup of the Alunite Supergroup. Gorceixite and goyazite form a solid solution series and are isostructural with each other. Crandallite and goyazite also form a solid solution series. The normalised elemental composition from **EDS Spectrum S4** suggests a major Ba concentration (16.8 wt%), with a minor concentration of Sr (5.3 wt%) and trace Ca (2.5 wt%). The major Ba concentration suggests that most of the crandallite group minerals in the sample are closer to the gorceixite end member composition.

4. CONCLUSIONS

1. The STDH-03 sample is dominated by kaolinite/halloysite (64.1 wt% by QEMSCAN), with minor amounts of the crandallite group minerals (gorceixite, crandallite and goyazite, 12.5 wt% in total), quartz (11.8 wt%), smectites (5.5 wt%), xenotime-Y (2.5 wt%) and chamosite (1.8 wt%).
2. Y-phosphate/xenotime-Y is the dominant REE containing phase in the STDH-03 sample. Two distinct types of Y-phosphate are present: large ($> 30 \mu\text{m}$), well-liberated, crystalline xenotime-Y; and smaller ($< 30 \mu\text{m}$), poorly liberated, Y-phosphate particles/grains that do not exhibit the typical crystalline xenotime-Y appearance.
3. The particle size distribution data suggests the smaller Y-phosphate grains/particles are the dominant type of Y-phosphate/xenotime in the sample. This defines that grinding of the sample and thus, a grind assessment, will be required prior to any beneficiation test work.
4. Kaolinite/halloysite is the main mineral associated with the Y-phosphate/xenotime phases. Kaolinite/halloysite is present along the edges of some of the larger, well-liberated crystalline xenotime-Y particles. The small Y-phosphate phase is typically present as fine inclusions in kaolinite/halloysite. It is critical to determine whether the liberation of these Y-phosphate/xenotime inclusions with grinding is possible.
5. Minor and trace concentrations of U and heavy REE such as Dy, Er and Yb are present in the xenotime-Y and Y-phosphate phases. It is possible that traces of other HREE are also present in the xenotime-Y and Y-phosphate phases at levels that could not be identified by EDS analysis due to peak overlaps or the limits of detection.
6. The unusual spherical aggregates and small ($< 5 \mu\text{m}$) particles of Y-phosphate/xenotime may be the reason for partial leaching of Y+REE under relatively mild, acidic atmospheric leach conditions observed for other samples from the Stromberg deposit.