Petrography and Mineralogy Results

Empire sent 29 samples for petrographic analysis and 16 samples for Tescan Integrated Mineral Analyzer ("TIMA") analysis from samples collected from both RC and diamond drill holes. These samples represent a point sample from a known depth in a drill hole and the information generated is interpreted to provide a first order understanding of the geology and mineralogy at only very specific locations.

Petrology and TIMA results illustrate that the dominant detrital grains of quartz and feldspar in the sandstones and conglomerate matrix show angular to sub-angular shapes (refer Figure 1), indicative of immature sediments that have not been extensively abraded but fairly well sorted and so are inferred to have been deposited in one relatively brief period of transport and sedimentation, possibly from a nearby source. The quartz, feldspar and mica grains within the siltstones are angular to sub angular and also preferentially aligned.

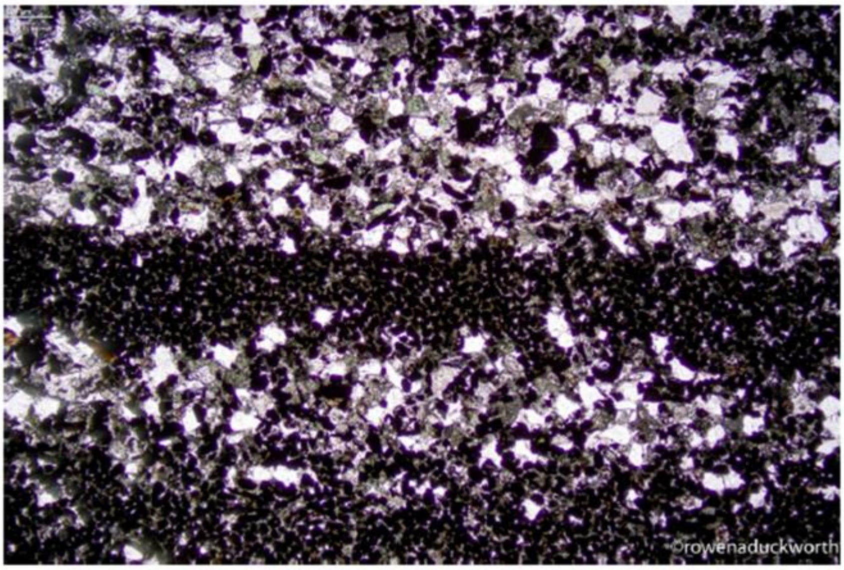

Figure 1: Plane polarised light photomicrograph showing the angular to subangular nature of the grains.

The sediments within the Yandanooka Basin have been highly altered by hydrothermal fluids, petrographic evidence suggesting two separate hydrothermal events that each caused significant mineralogical changes. The alteration paragenesis has been developed by detailed petrography and TIMA analysis on RC chips and slices of drill core.

This work shows the initial alteration fluid produced an assemblage of epidote-titanite-carbonate and chlorite. The geochemical signature of this hydrothermal alteration event is strong Ca-Ti-Mg-Al-Fe rich with elevated Co-Cr-Co-Cu-Ni-Zn indicting that the fluid interacted with a mafic source. The composition of the hydrothermal fluid is not yet understood but silica was readily available from the primary siliciclastic components and so wasn't necessarily introduced to form the secondary silicate minerals.

This initial alteration mineral assemblage was subsequently overprinted by an iron-mobile hydrothermal fluid which was notable for the formation of hematite, largely as replacement of the primary magnetite grains. Such an extensive marmatisation event indicates that the first alteration event may not have sealed off the porosity and permeability within the sediments, or that this later hydrothermal fluid may have generated its own permeability as a result of fluid-rock interactions. The hematite from this second alteration event has subsequently been altered much later to goethite presumably by recent meteoric ground waters.

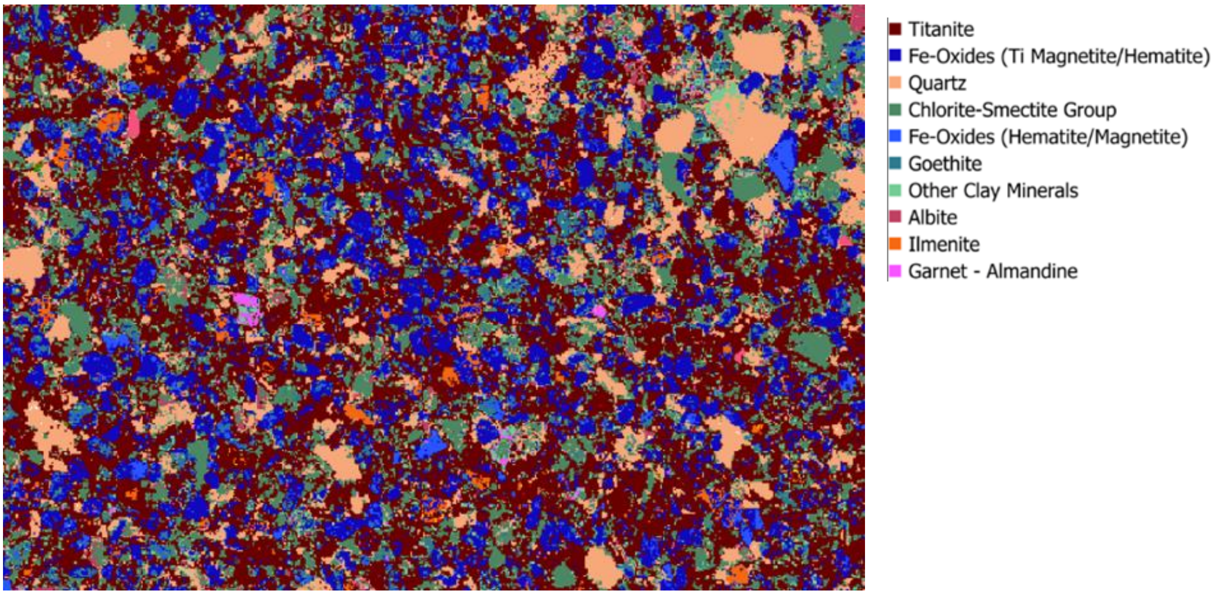

Figure 2. TIMA image showing abundance of titanite (maroon) and Ti-bearing Fe oxides (dark blue). Titanite is showing unusual morphology and appears to be infilling around quartz (flesh colour) grains.

A much later carbonate veining event occurred which appears to be coeval with deformation structures as evidenced within drill core and RC chips by carbonate filled fractures and veins cross cutting conglomerate casts and sedimentary fabrics. This carbonate event is therefore interpreted as part of the structural-metamorphic event rather than a third distinct hydrothermal event.

The mineralogical analyses completed to date has identified titanite as the most abundant titanium mineral followed by Ti bearing Fe oxides, ilmenite and then rutile group minerals. Titanite's abundance within these point samples averages approximately 20% of the rock mass. Literature searches have established this level of titanite concentration to be extraordinary and perhaps unprecedented. Titanite is usually an accessory metamorphic mineral and rarely gets above 1-2% of the rock mass. The abundance of hydrothermal titanite at Pitfield suggests extreme Ti mobility and highlights the unique nature of this giant mineral system. Of the remaining Ti minerals, Ti bearing Fe oxides make up around 15% of the rock mass with ilmenite forming 2%.

CSIRO Microprobe Analysis Results

Ten RC chip sample were collected from the maiden drilling programme carried out in April 2023. Electron microprobe work was performed at CSIRO Division of Minerals. Combined with other mineralogical work, this work shed light on how the titanium occurs and represents the start of technical studies as the basis for future metallurgical test work to identify an appropriate ore processing flowsheet.

CSIRO were contracted to perform microprobe work on the samples (previously analysed by AXT's TIMA method for mineral analysis) to try and determine the levels of TiO_2 within the various minerals. CSIRO probed 384 grains and captured images of each grain which would then allow for an interpretation of the grains. What can be clearly seen from the CSIRO BSE images is the euhedral and subhedral nature of the grains of hematite/magnetite with ilmenite exsolution lamellae and those grains which show good crystal lattice (refer Figure 3). The titanite grains show poor morphology and are often partially replaced by hematite; importantly they often appear to be filling the spaces between grains.

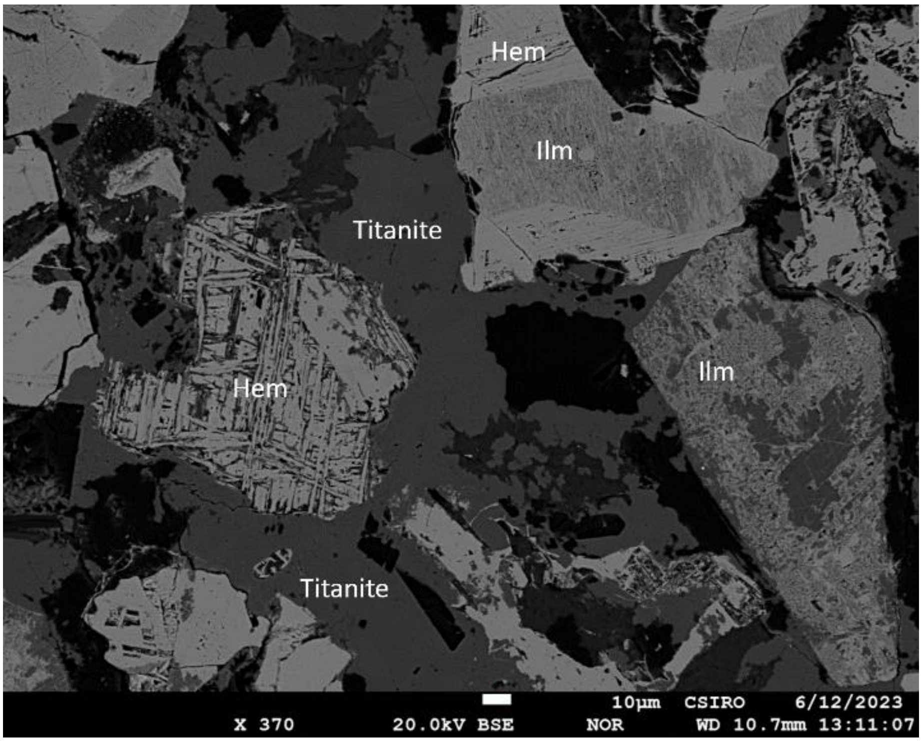

Figure 3. CSIRO Microprobe BSE image. Titanite is seen filling pore spaces. Hematite (Hem) replacing relict magnetite grains and intergrown with ilmenite.

The microprobe data obtained from CSIRO highlights several distinct groups of minerals and also hybrid minerals that have formed due to the alteration assemblage. There are distinct groups of titanite, hematite, Mg-Al-Fe silicates and ilmenite. However, the alteration of minerals by hydrothermal fluids has led to hybrid minerals such as hemo-ilmenite which is made up of more Fe than Ti and the titano-hematite which has more Ti than Fe. This is due to substitution within the lattice either allowing for more Ti or Fe.

By grouping data from multiple analysis points, the main Ti bearing minerals tend towards the following approximate compositions.

Table 1: Approx. Compositions of Ti Bearing Phases from EMPA (Electron Microprobe) Analyses

Element	**Titanite**	**Hematite**	**Titano-Hematite to Hemo-Ilmenite Series**		
			Ti End	**Average**	**Fe End**
TiO_2	36.7	1.6	74.2	22.4	6.4
Fe_2O_3†	2.5	97.1	27.2	78.7	92.6
CaO	29.6	0.1	0.3	0.1	0.2
SiO_2	31.0	0.7	0.0	0.2	0.3
Al_2O_3	1.9	0.5	0.0	0.17	0.5

† All Fe expressed as Fe_2O_3. Oxidation state not specified at this stage. Some assay totals will exceed 100%.

Ilmenite, an iron-titanium oxide mineral ($FeTiO_3$) containing approximately 50-60% TiO_2, lies somewhere within the mid-range of the titano-hematite to hemo-ilmenite series measured above. The actual abundance of ilmenite is difficult to quantify as hematite has not only replaced relict magnetite grains but has also intergrown with ilmenite. The TIMA analysis identified the presence of Ti-bearing Fe oxides making up to ~15% of the rock mass. These Ti-bearing Fe oxides are best seen in the CSIRO microprobe images as anhedral grains of hematite with exsolution lamellae of ilmenite as well as subhedral grains of hematite with ilmenite within a more ragged crystal structure.

Preliminary Metallurgical Assessment

Wet Gravity Separation Diagnostic Testwork

Two samples were selected from the maiden RC drill campaign (one from the Thomas property and one from the Mount Scratch area). The two head samples were generated from the 2m sample intervals collected during the RC drilling and are representative of an 88-metre depth interval for the Thomas sample and between 170 – 190 metres depth interval for the Mount Scratch sample.

Table 2: Origin and Lithology of RC Metallurgical Samples

Hole ID	Sample Type	Sample Interval (m)	Depth From (m)	Depth To (m)	Lithology
Sample 1 - Thomas					
RC23TOM001	Continuous	88	60	148	Conglomerate (60-122m) Sandstone (122-148m)
Sample 2 – Mount Scratch					
RC23MTS001	2m Every 10m	188	8	196	Sandstone (8-196m)
RC23MTS002	2m Every 5m	174	4	178	Sandstone (4-178m)
RC23MTS003	2m Every 10m	170	8	178	Sandstone (8-178m)

In order to examine the potential for gravity concentration, the two head samples were ground to produce three sub-samples at differing size ranges: minus 500-micron, 300-micron and 150-micron. Each sub-sample was initially deslimed, by removing the minus 38 micron material, and then separated over a wet table. Three or four concentrate streams were produced (depending on sample) and a tailings stream.

High mass recoveries to the concentrate streams were achieved on all samples, indicating that grind size needs to be further optimised. The geochemical analysis results showed 90% TiO_2 recovery was achieved with approximately 80%, 81% and 84% mass recovery for the 150-micron, 300-micron and 500-micron size fractions, respectively.

The wet table streams for the 300-micron grind size tests for Sample 1 and Sample 2 were sent to AXT for mineralogical analysis. The abundance of four Ti bearing mineral phases in the wet table streams was measured (see **Error! Reference source not found.**).

The results show that around 90% of the heavy minerals such as ilmenite, Ti-oxides and Fe-oxides, which have an SG in the range of 4.2 to 4.7, are recovered in the first three concentrate streams. The lighter titanite (around 3.5 SG) has about 84% of the mass reporting to the first three concentrate streams. The performance of the wet table in recovering these minerals was encouraging and provides confidence that these minerals can be recovered by gravity separation.

At present there is a significant amount of gangue minerals reporting to the third concentrate stream. Further optimisation of the wet gravity separation is required to improve selectivity of titanite recovery.

Table 3: Mass Deportment of Minerals to Wet Table Streams

Stream	Titanite	Ilmenite	Ti Oxides	Fe Oxides	Gangue Silicates	Other
Sample 1						
Con 1	16.7%	31.8%	36.8%	29.5%	3.0%	3.5%
Con 2	25.3%	28.6%	25.2%	24.5%	12.2%	9.7%
Con 3	43.9%	33.8%	31.1%	36.8%	55.1%	49.8%
Con 4	12.2%	5.5%	6.1%	8.6%	26.1%	31.1%
Tail	1.9%	0.4%	0.7%	0.7%	3.6%	5.8%
Sample 2						
Con 1	9.4%	38.3%	32.9%	28.7%	1.1%	1.4%
Con 2	27.6%	30.2%	24.5%	26.9%	8.3%	7.3%
Con 3	47.3%	23.6%	29.3%	35.5%	54.0%	55.3%
Con 4	13.4%	7.2%	11.5%	8.3%	31.2%	31.2%
Tail	2.3%	0.7%	1.7%	0.7%	5.4%	4.8%

Titanium Distribution Amongst Main Ti-Bearing Minerals

The mineralogical information presented above can be combined with the CSIRO microprobe information to estimate the distribution of TiO_2 with respect to the various Ti bearing minerals in the two samples.

For this analysis, the following TiO_2 contents were applied to the minerals:

- Titanite set as 36.7% TiO_2 based on CSIRO microprobe analyses.
- Ilmenite set at 54% TiO_2 based on typical assay for WA primary ilmenite.
- Ti-oxides set at 100% TiO_2 for rutile and anatase minerals.

By applying the above TiO_2 values to each mineral phase, and using the head sample TiO_2 analysis, the average TiO_2 value for the various Fe-oxide minerals (which contained varying content of TiO_2 from the CSIRO microprobe work) can be calculated. By using this approach, the optimised TiO_2 value for the Fe-oxides was determined to be 5.7%.

Table 4: TiO_2 Distribution Across Ti-bearing Minerals

Mineral Type	**% of TiO_2 Units in Mineral Type**	
	Sample 1	**Sample 2**
Titanite	66.9%	67.1%
Ilmenite	16.1%	6.9%
Ti-Oxide	3.3%	4.5%
Fe-Oxide	13.7%	21.6%

The analysis shows that the titanite phase is estimated to contain around 67% of the TiO_2 units in these two samples.

Metallurgical Processing Options

Explorative metallurgical investigation to date has focused mainly on wet gravity separation as a way to beneficiation the ore before further processing and froth flotation needs to be considered as a further mineral concentration step after wet gravity separation. Given the preliminary nature of the metallurgical testwork to date, flotation has been tentatively included as a processing step in the developing flowsheet. Further investigation is required to determine whether flotation concentration is worth pursuing.

In addition, options for preliminary separation of crushed product are being evaluated including the use of gravity jigs and/or ore-sorting.

At this early development stage, the processing flowsheet steps being considered include the following:

1. Crushing
2. Preliminary Separation (Ore-Sorting or Jig)
3. Grinding
4. Wet gravity separation
5. Flotation (to be confirmed)
6. Acid Leach
7. Final product treatment (including calcination)

The preliminary flowsheet is shown below in Figure 4.

One of the key differences between the processing options being considered for the titanite ore from the Pitfield Titanium Project and those commonly found in the ilmenite industry is that a low temperature acid digestion process is likely all that is needed to extract the titanium and produce a high quality product.

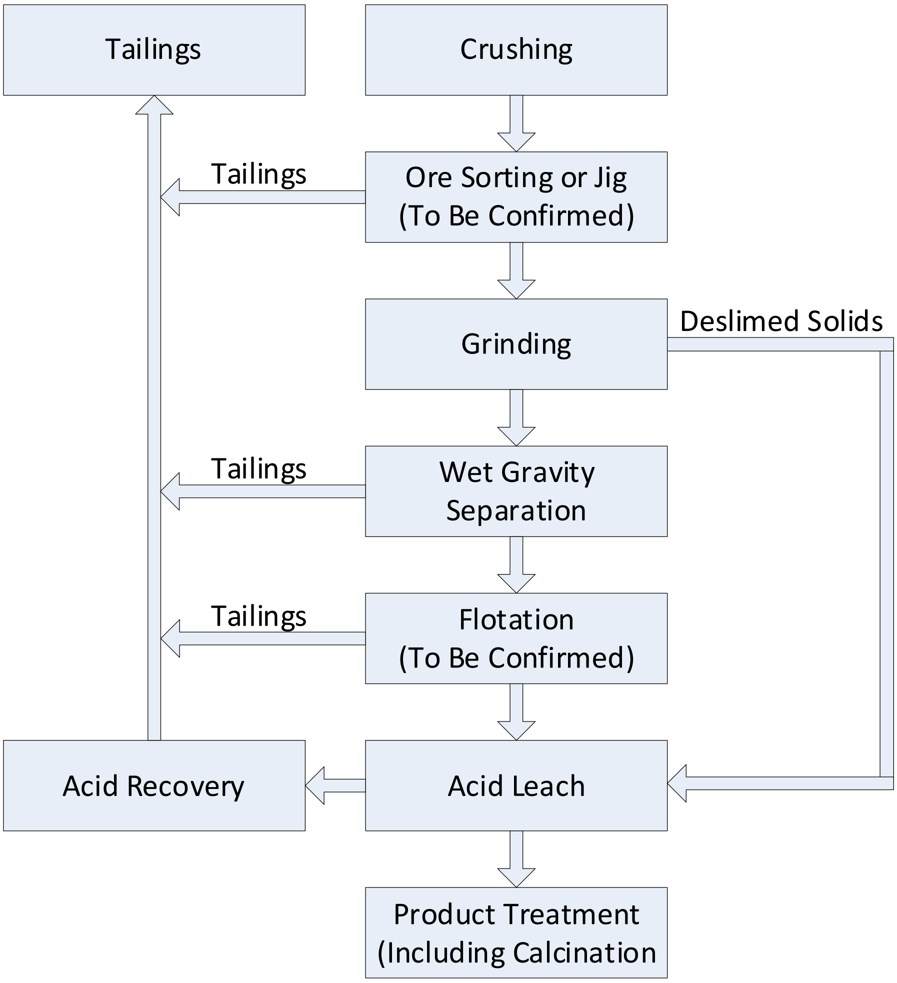

Figure 4. Preliminary Metallurgical Process Flowsheet.

The literature reports a considerable amount of work done on the leaching of titanite particularly from the Murmansk Region, in Russia.

Much of the Russian work considers the use of a sulphuric acid leach to dissolve the Ti in the titanite into the liquid phase (refer Equation 1). The contained Ca and Si are separated as solids (calcium sulphate and amorphous silica). Purification of the $TiOSO_4$ rich liquor and subsequent precipitation of TiO_2 by hydrolysis is possible. This processing route is one option for treatment of titanite.

Equation 1. $CaTiSiO_5 + 2H_2SO_4 = TiOSO_4 + CaSO_4 + SiO_2.H_2O + H_2O$

There is also the well-known Ti sulphate pigment production process where primary ilmenite is dissolved in sulphuric acid to produce a prified liquor rich in $TiOSO_4$ (refer Equation 2). The TiO_2 is then hydrolysed from the solution.

Equation 2. $FeTiO_3 + 2H_2SO_4 = TiOSO_4 + FeSO_4 + 2H_2O$

The process conditions required for these sulphuric acid leaching processes are well understood. Test work is required to confirm conditions and performance results on the Pitfield titanite concentrate.

The University of Minnesota have also developed a hydrochloric acid dissolution process for the processing of a low-grade ilmenite concentrate. The ilmenite concentrate assayed at 39% TiO_2 and 30% Fe (as predominantly Fe^{2+}). The concentrate contained elevated Mg and Mn as the main impurity elements.

The Minnesota processing circuit consisted of a mixed chloride leach (HCl and $MgCl_2$) followed by an iron oxidation step prior to solvent extraction stages aimed at concentrating an iron rich liquor and a titanium rich liquor (refer Equation 3). Titanium dioxide is precipitated from the Ti rich liquor by thermal precipitation.

Equation 3. $FeTiO_3 + 5HCl = HFeCl_3 + 2H_2O + TiOCl_2$

Hydrochloric acid leaching systems have been reported extensively in the published literature for ilmenite leaching. The Benilite process also used a hydrochloric acid leaching process to partially solubilise Ti in ilmenite. The Benelite process involves the reduction of ilmenite to convert the ferric iron into the ferrous state and hydrochloric acid is subsequently used to leach the iron.

Further Russian studies carried out on titanite ore from apatite deposits of the Khibiny Massif demonstrated that the reaction of titanite with concentrated hydrochloric acid produces hydrated titanosilicate precipitate (TSP) which, in turn, can be a precursor in titanosilicate synthesis.

Metallurgical Summary

Titanite is not considered to be a “refractory” mineral and should dissolve readily under low temperature acidic conditions. The ilmenite and titano-hematite minerals present in the ore may require slightly elevated temperatures. The flowsheet should consist initially of one or more beneficiation stages to generate a titanium rich concentrate, and to remove acid consuming gangue minerals, followed by a simple acid leaching stage to extract the titanium and produce a high value product.

Options exist for the development of either a sulphuric acid or hydrochloric acid-based leaching process to treat the Pitfield titanite rich concentrate. Test work and investigation is needed to evaluate the effectiveness of the leaching systems and to target conditions. Further research and evaluation of industry and researchers’ know-how is required.

There is scope to combine features from several related applications to suit Empire's unique requirements for treatment of the Pitfield concentrate. Either acid digestion system will require the recycle of metal sulphates or chlorides to recover acid back to the leaching process. In both sulphuric acid and hydrochloric acid processing there are commercially operating technologies in operation in industry.

The key advantage that Pitfield has over the current industry producers is that the ore lends itself to a simple hydrometallurgical processing route to achieve a high grade TiO_2 product for sale and does not require an energy intensive, smelting process as used by the igneous sourced ("hard rock") ilmenite miners.

Potential TiO_2 Products

The final product from the leaching stage is expected to be a fine precipitated material. This material should have a high TiO_2 concentration, approaching the same grade as rutile. However, the material will likely require calcination and probably some consolidation to make it an attractive handleable product for customers. The consolidation process could take the form of a high pressure pelletisation or adjustment of thermal treatment conditions to improve the physical integrity of the product.

Based on Q4 2023 rutile market supply and demand forecasts the price range for rutile is in the order of US$1,880/t to US$2,180/t, reflecting a tightening of the supply chain despite increasing demand over the next decade *(Source: Sovereign Metals Ltd. February 2024 Investor Presentation - Prices per TZMI; Ilmenite price: November 2023 FOB Mozambique; Rutile price: "weighted average price for bagged rutile shipments")*.

Alternatively, the Company could consider the potential to further refine the TiO_2 product and look to make a pigment quality product for marketing directly to the final end users, in other words for use in the manufacture of paints and coatings, plastics and paper.

Based on Q4 2002 prices the expected price range for TiO_2 pigments is in the order of US$4,340/t to US$5,180/t. *(Source:https://medium.com/intratec-products-blog/titanium-dioxide-prices-latest-historical-data-in-several-countries-24ec1864a198)*