



**TNG LTD.**  
**Mount Peake Project**  
**Metallurgical Testwork Summary Report**

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## 1. EXECUTIVE SUMMARY

Mr Paul Burton, Exploration Director of TNG Limited contacted Mr Damian Connelly from Mineral Engineering Technical Services Pty Ltd (METS) to develop a sighter test program of the Mount Peake Project in the Northern Territory, Australia. This sighter test will serve as an initial metallurgical investigation to determine the amenability of Mount Peake ore to an extraction process. It aims to form the basis from which more detailed studies can be designed to produce the optimised process flowsheet.

Two samples, labelled as MPM001 and MPM003, were submitted to Amdel and AMMTEC. The MPM001 and MPM003 samples were originated from drill holes, 09MPRC002 and 09MPRC004, respectively.

Both MPM001 and MPM003 returned head grades that are worthy of further investigation, assaying 30.6-34.2% Fe, 8.1-10.6% TiO<sub>2</sub> and 0.44-0.56% V<sub>2</sub>O<sub>5</sub>. The samples were amenable to magnetic separation, as supported by the Davis tube and low intensity wet magnetic separation (LIMS) tests.

For MPM001, the LIMS test on P<sub>100</sub> -75 µm generated a magnetic concentrate, assaying 51.2% Fe, 1.1% V<sub>2</sub>O<sub>5</sub>, 14.7% TiO<sub>2</sub>, 5.98% SiO<sub>2</sub> and 2.9% Al<sub>2</sub>O<sub>3</sub>. While an economic grade of vanadium was produced, the silica level was higher than the recommended value of 3% SiO<sub>2</sub>. The Davis tube recovery test performed at 45 µm produced a magnetic concentrate at 56.1% Fe, 15.8% TiO<sub>2</sub>, 1.2% V<sub>2</sub>O<sub>5</sub>, 1.5% SiO<sub>2</sub> and 2.2% Al<sub>2</sub>O<sub>3</sub>. On the basis of the Davis tube results, it indicates that fine grinding can be successfully applied to improve the silica rejection.

For MPM003, the LIMS test on P<sub>100</sub> -75 µm generated a magnetic concentrate grading 53.5% Fe, 1.12% V<sub>2</sub>O<sub>5</sub>, 17.2% TiO<sub>2</sub>, 2.91% SiO<sub>2</sub> and 2.56% Al<sub>2</sub>O<sub>3</sub>. The silica level was within the acceptable limit for the roasting test.

The QEMSCAN analysis performed on the LIMS magnetic concentrates showed that the vanadium is locked within titanomagnetite with minor quantities found in association with ferromagnesian silicates, magnetite, ilmenite and sphene. Unlike magnetite where low temperature treatment at 900-1100°C is efficient to oxidise the spinel magnetite, it is anticipated that higher temperature treatment would be needed to oxide the titanomagnetite in order to achieve high vanadium recovery.

Some free ferromagnesian silicates particles were found in the MPM001 sample, which are not removed by LIMS separation. This could be the result of entrainment. Re-treating the magnetic concentrate through the LIMS separator could assist the removal of the free ferromagnesian silicates (which contribute to the high silica level in the magnetic concentrate). Nevertheless, there is a potential that a small percentage of the vanadium will be rejected along with the ferromagnesian silicate. About 3.61% of the vanadium occurs within the ferromagnesian silicates.

Furthermore, there also exist some simple binary and ternary complexes in both the MPM001 and MPM003 samples. Fine grinding could be employed to liberate the gangue minerals from the valuable titanomagnetite.

The magnetic concentrates generated from the LIMS test were subjected to salt roasting followed by water leaching. The roasting and water leach results were not encouraging, giving poor vanadium water leach recoveries. However, the results are preliminary at this stage. The high silica level in sample MPM001 and the low alumina:silica ratio in the concentrates, in particular the MPM001 sample, are both known to cause problems in the roasting process. In addition, there is evidence of partial reaction of the charge with the silica crucible.

Following these observations, a new test program was developed to improve the vanadium recovery. The magnetic concentrate was reground to  $P_{100} \sim 45 \mu\text{m}$  and subjected to magnetic separation for silica rejection, before salt roasting.

The LIMS results on the regrind samples were encouraging, producing a magnetic concentrate with a combined silica and alumina grade of less than 5% and the ratio of alumina-to-silica greater than 1. Grinding the magnetic concentrate to  $P_{100} \sim 45 \mu\text{m}$  also improved the vanadium grade in the magnetic concentrate. For MPM001, the vanadium grade is upgraded from 1.07%  $\text{V}_2\text{O}_5$  to 1.26%  $\text{V}_2\text{O}_5$  with about 88.1% of the  $\text{V}_2\text{O}_5$  recovered in 33.8% of the mass. Similarly, with MPM003, an upgrade in the vanadium grade from 1.1%  $\text{V}_2\text{O}_5$  to 1.2%  $\text{V}_2\text{O}_5$  was attained at a vanadium recovery of 84.7% in 40.9% of the mass.

Subsequently when the sample is treated in the furnace at 1150°C and subjected to water leach at 70°C for 1 hour,  $\text{V}_2\text{O}_5$  leach recoveries of 69% and 73% were recorded, respectively for MPM001 and MPM003.

Comparing to the first roast/water leach test, the  $V_2O_5$  recovery of MPM001 was improved from 17% to 69%. The positive effect is mainly attributed to the drastic decrease in the silica content and to a lesser extent, the presence of extra oxidant resulting from the decomposition of sodium percarbonate. The decrease in the silica level reduces the formation of a glassy silicate complex and also the competing side reaction from silica for the sodium salt.

For MPM003, the  $V_2O_5$  recovery improved from 67% to 73% when compared to the first roast/water leach test. Hydrogen peroxide generated from the decomposition of sodium percarbonate can promote the oxidation of the titanomagnetite to hematite, and more importantly, oxidise the vanadium to a water soluble form.

It is anticipated that the remaining vanadium could be locked within the titanium oxide phase in the titanomagnetite. If this is the case, liberating vanadium from the titanium phase will be needed to further improve the  $V_2O_5$  recovery.

## 2. RECOMMENDATIONS

There was about 69-73% of  $V_2O_5$  recovered from the roast/water leach reaction. The vanadium recovery aligned well with the industry norm for titanomagnetite ore. Nonetheless, METS strongly recommends repeat testwork, to optimise the vanadium extraction from the salt roast test.

METS recommends that:

- A QEMSCAN analysis is performed on the leach residues to study the vanadium deportment to confirm the conjecture that the remaining vanadium is locked with the titanium phase of the titanomagnetite.
- The roasting test should be repeated using sodium oxalate as the flux. Oxalate ion has been reported to be capable of dissolving titanium dioxide, where this could improve the liberation of vanadium from titanomagnetite. The Windimurra vanadium project used sodium oxalate as the sodium salt.
- A separate test using sodium chloride as the sodium salt should also be explored. The HCl vapour generated from the decomposition of sodium chloride could assist the dissolution of titanium dioxide which subsequently enhances the liberation of vanadium from the titanium phase of the titanomagnetite.

In addition, due to the high cost of the salt roast process, testwork should investigate alternative processes, such as strong acid leaching, to produce separate iron, vanadium and titanium products.



### 3. INTRODUCTION

TNG has acquired the exclusive rights for the Mount Peake project within EL23074, which covers a total area of 169.2 km<sup>2</sup>. The Mount Peake project is strategically located in close proximity to existing infrastructure, including the Alice Springs-Darwin Railway, Stuart Highway and the new LPG pipeline (approximately 20 km to its east). It is situated within the north-central portion of the Paleoproterozoic Arunta Province, 80 km north east of Alice Springs (Figure 3.1). Geological assessment of the region identified the central Arunta as a prospective area for Proterozoic, intrusive related, magnetic Ni-Cu-(platinum group element, PGE) sulfide mineralisation.

There is no record of Ni exploration until TNG acquired the license and commenced exploration. Falconbridge, on commencement of the Joint Venture agreement with Tennant Creek Gold (NT) Pty Ltd, a wholly owned subsidiary of TNG, has identified six prospective areas of Ni-S mineralisation. In 2003, Discovery Nickel Ltd (DNL), under a Heads of Agreement with Falconbridge, conducted a ground EM (SMART-EM) survey which detected no evidence of Ni-S mineralization. DNL reported the anomalies were related to massive magnetic gabbro. They found that the drill core showed little variation and predominantly contained medium to coarse grained, massive, magnetite-bearing gabbro, with a mineralogy of magnetite-pyroxene-feldspar-quartz with traces of biotite.

In 2006, TNG resumed the management of the tenement following the decision from Falconbridge and DNL to terminate the joint venture partnership. Reprocessing and interpretation of the aeromagnetic and GEOTEM data along with the assay analyses on the diamond drill hole, ARD02 revealed that the magnetite mineralisation is extended for at least 80 m from a depth of 30 to 111 m, with a grade of 28.7% Fe, 0.43% V<sub>2</sub>O<sub>5</sub> and 7.5% TiO<sub>2</sub> at 31 to 80 m.

Davis Tube recovery tests were also conducted on ARD02 to investigate the amenability of the ore to magnetic separation. The Davis Tube results have shown economic grade of vanadium-magnetite-titanium in the magnetite-gabbro, yielding magnetite concentrates with grade in excess of 55% Fe, 1.2% V<sub>2</sub>O<sub>5</sub> and 15.6% TiO<sub>2</sub>.

Following the encouraging Davis Tube recovery test results, a reverse circulation drilling program was commenced. Six new holes, 09MPRC001, 09MPRC002, 09MPRC003, 09MPRC004, 09MPRC005 and 09MPRC006 were drilled, as shown in Figure 3.2.

The RC Chip samples from holes, 09MPRC002 and 09MPRC004, were investigated in the current sighter test program.

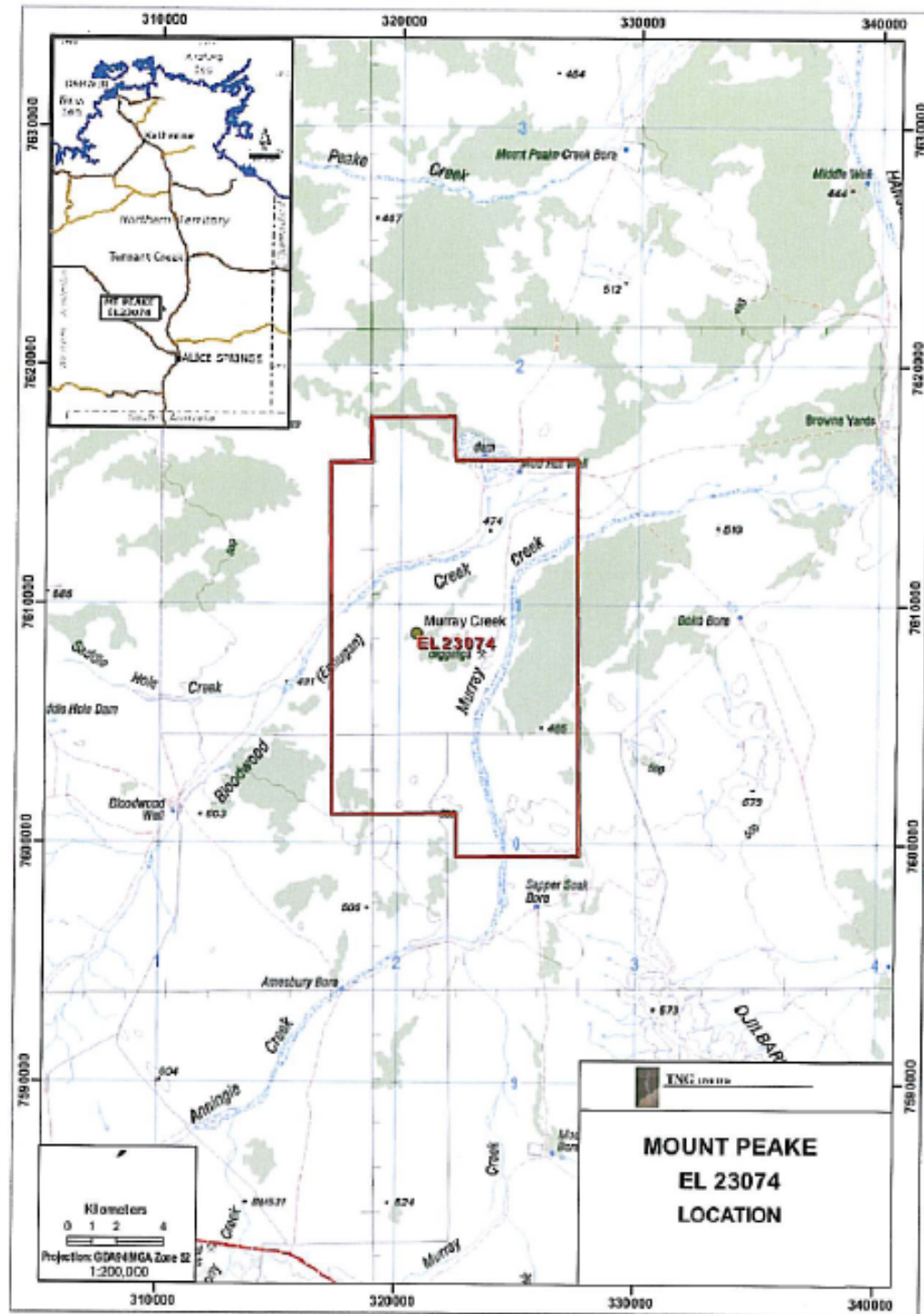


Figure 3.1: Map showing the location of the Mount Peake project

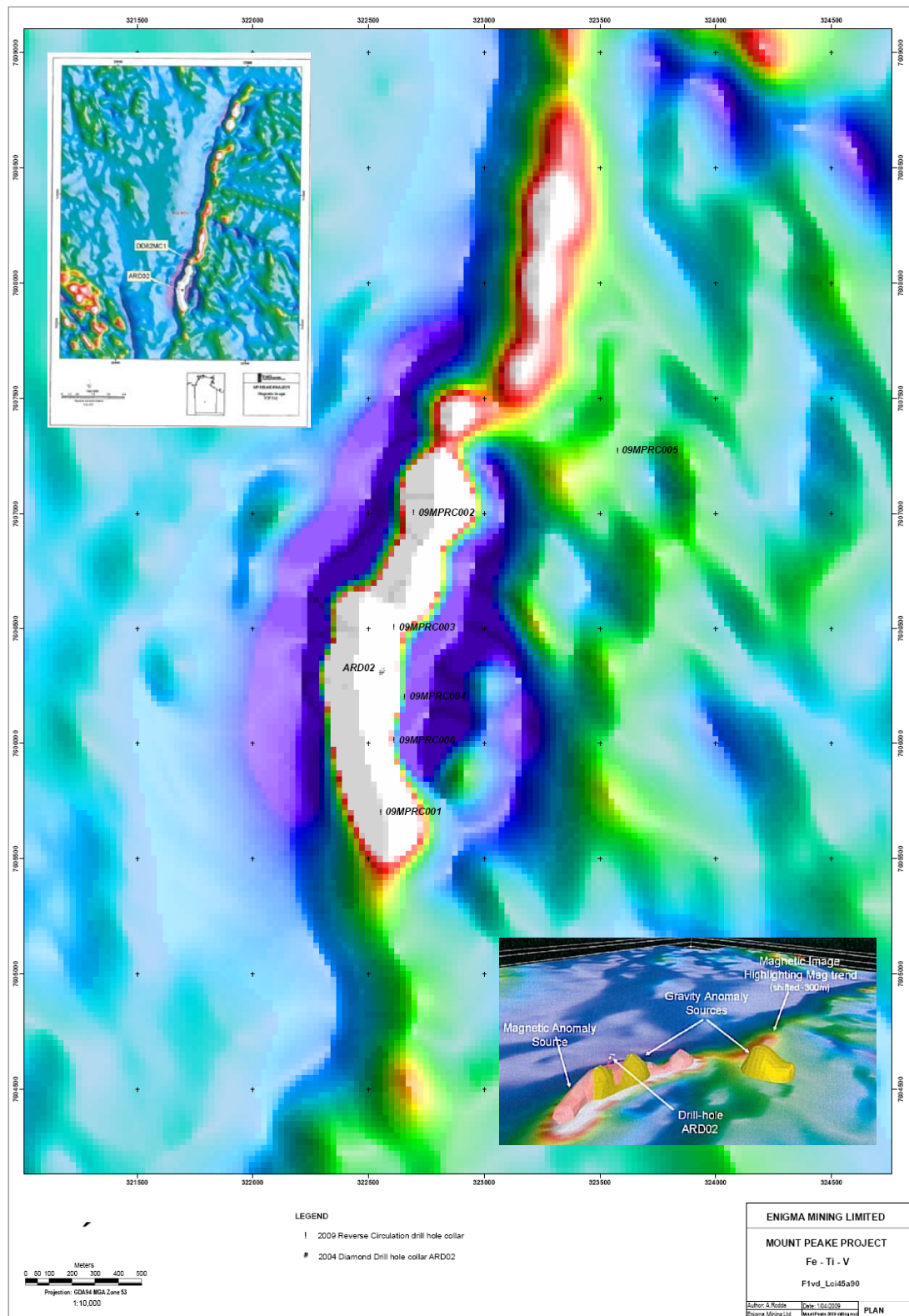


Figure 3.2: Map showing the 2009 drill hole locations

#### **4. SCOPE OF WORK**

The objective of the Mount Peake Project is to produce a magnetic concentrate to feed into a salt roast and water leach process and determine the vanadium extraction. Apart from that, the potential for iron and titanium dioxide extraction in the magnetic fraction will also be examined.

The scope of work for the sighter test includes, but is not limited to:

- Develop a testplan for the sighter test to examine the upgrading of the Mount Peake ore by magnetic separation and roasting
- Obtain quotations for the testwork from the testing laboratories, analyse the quotations and make a recommendation to TNG Limited
- Manage the testwork through the selected laboratory
- Analyse and interpret the testwork results, and
- Write a report on the testwork and make a recommendation on further testwork based on the findings of the sighter test.

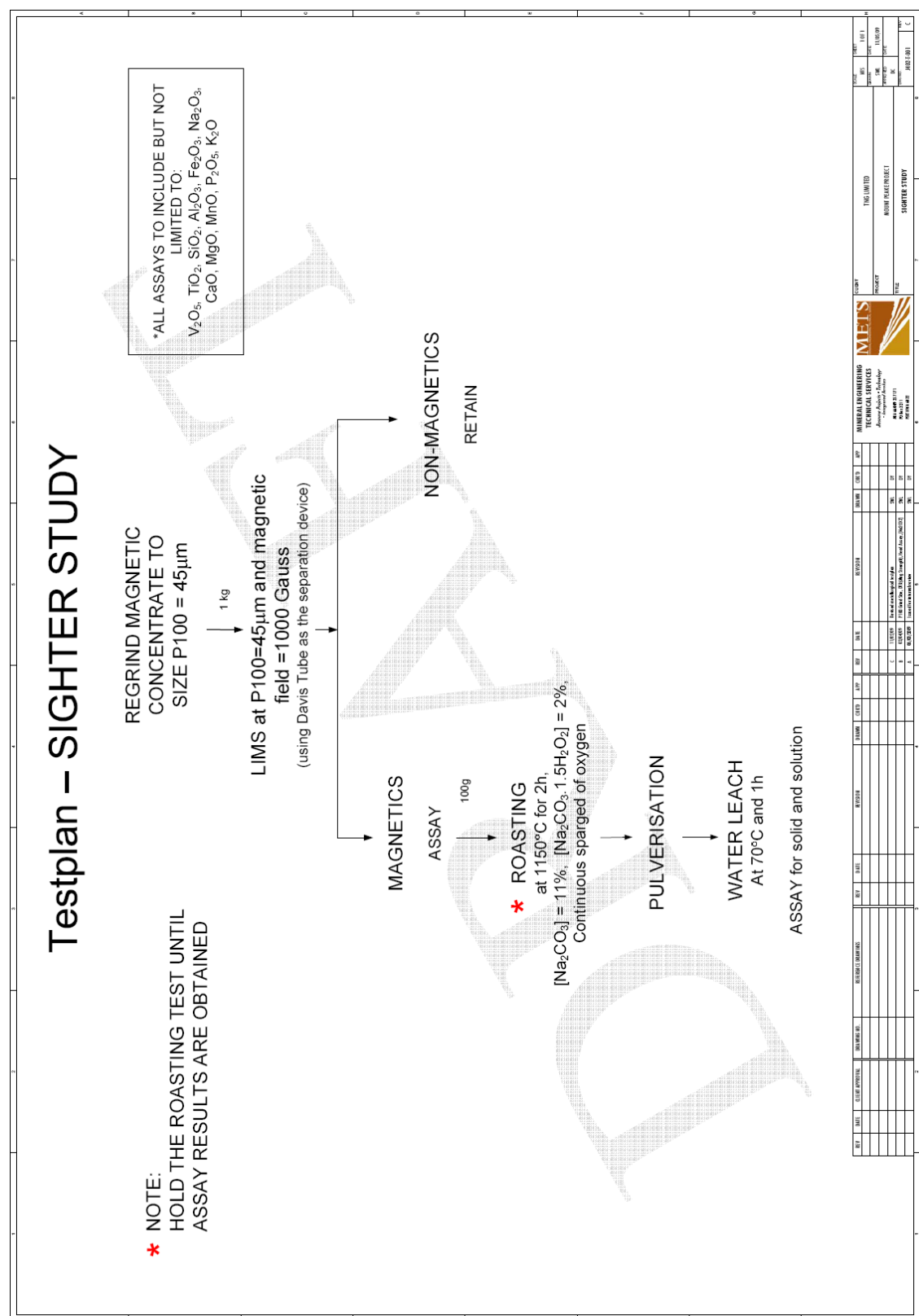
Excluded from the scope of work is:

- Marketing
- Environmental
- OPEX
- CAPEX
- Flowsheets, equipment lists, engineering specifications.

This report contains the testwork results of the sighter test on Mount Peake samples, performed by Amdel and AMMTEC. A concise analysis and interpretation of the testwork findings was provided by METS.

Two testwork programs were developed to obtain a preliminary performance response of the Mount Peake samples to typical industry processing. Schematics of the Phase 1 and Phase 2 testwork programs are shown in Figure 5.1 and Figure 5.2, respectively.





**Figure 5.2: Testplan for Mount Peake Sighter Test Phase 2**

Two RC Chip samples, identified as MPM001 and MPM003, were submitted to Amdel for the testwork. The MPM001 and MPM003 samples originated from drill holes 09MPRC002 (interval 46-47 m) and 09MPRC004 (interval 49-50 m), respectively.

The full Amdel and AMMTEC reports are available in Appendix A and B, respectively. A summary of the results is presented below.

## **5.1 Phase 1 Testwork**

### **5.1.1 Head Assay**

The samples were crushed to -0.5 mm prior to the multi-element head analysis. The multi-element head analysis was performed using X-ray fluorescence (XRF). This method allows the identification of many elements, both the prospective element (such as vanadium oxide) as well as those that might be problematic during processing (such as silica, alumina, phosphorus, sulfur and calcium). Furthermore, fire assay analysis was also carried out to determine the gold content in the samples.

The results of the head assay analyses of the samples are presented in Table 5.1. Both MPM001 and MPM003 showed similar head grade. The obtained results revealed that the samples contained 0.44-0.56%  $V_2O_5$ , a grade that is worthy of further investigation.

The samples also contained about 30.6-34.2% Fe, which is a typical head grade of NT magnetite deposits. There is a possibility for the iron to be recovered into a saleable form to off-set some of the operating costs.

The gold content was low, assaying 20 ppb. Its recovery would not lead to any economic benefit to the project.

Gangue minerals, including silica, alumina, calcium oxide and phosphorus were at high levels with the samples grading 22.2-26.4%  $SiO_2$ , 5.7-5.8%  $Al_2O_3$ , 3.6% CaO and 0.08-0.11% P. A pre-treatment process is necessary for the rejection of gangue minerals prior to the roasting process.

Other deleterious elements, such as sulfur, are in low percentages and of no major concern.

**Table 5.1: Head grade of MPM001 and MPM003**

Sample ID	Chemical Assay (%)								
	Fe <sub>(tot)</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	P	TiO <sub>2</sub>	S	V <sub>2</sub> O <sub>5</sub>	Au (ppb)
MPM 001	30.6	26.4	5.8	3.6	0.11	8.1	0.04	0.44	20
MPM 003	34.2	22.2	5.7	3.6	0.08	10.6	0.04	0.56	20

### 5.1.2 Davis Tube Recovery (DTR) Test

The Davis Tube is a special laboratory magnetic separator used to separate ferro-magnetic and non-magnetic fractions of small crushed magnetic iron ore at intensely controlled laboratory conditions. This technique is ideally suited to establish the recoveries expected from a magnetic separation process. The Davis Tube tests were performed using the constant conditions summarised below:

- Field Strength                      3000 Gauss
- Stroke length                        38 mm
- Tube Angle                            45 degrees
- Tube Diameter                        25 mm
- Sample mass                         20 g

The testwork was carried out at a grind size of 45 µm. This grind size is finer than what was used in the historical testwork ( $P_{100} \sim 75 \mu\text{m}$ ) and was designed to establish the optimum grind size for the LIMS testwork. A finer grind size will improve mineral liberation to allow a higher rejection of gangue minerals. However, grinding finer than the optimum grind size will increase the energy costs for little recovery and grade advantage.

A comparison of the Davis Tube results for MPM001 and MPM003 is presented in Table 5.2. Overall, the Davis Tube at a  $P_{100}$  of 45 µm was encouraging; though the assay results were not available before the low intensity magnetic separation (LIMS) tests were carried out. The LIMS tests were carried out at the grind size of 75 µm, based on similar mass recoveries for the DTR at 45 and 75 µm.



The DTR assays showed that for MPM001, high  $V_2O_5$  recovery at 87.1% with a grade of 1.23%  $V_2O_5$  was attained. Similarly, MPM003 also achieved high  $V_2O_5$  recovery at 91.7%, assaying 1.17%  $V_2O_5$ . The obtained  $V_2O_5$  grade was within the recommended feed grade for the roasting process.

As shown in Table 5.2, Fe and  $TiO_2$  were upgraded along with the  $V_2O_5$ . This is aligned with subsequent QEMSCAN analysis, which showed that the vanadium is hosted within titanomagnetite.

Low silica content, grading at 1.5%  $SiO_2$  for MPM001 and 2.0%  $SiO_2$  for MPM003, was detected in the Davis tube magnetic concentrates. The Davis tube magnetic concentrates also contained low levels of calcium oxide, assaying 0.25% CaO for MPM001 and 0.17% for MPM003. The low levels of gangue minerals are expected to pose no major issue to the roasting process. The recommended condition for the vanadium roasting process is to maintain the  $SiO_2$  and CaO levels below 3% and 1%, respectively.

Compared to the historical DTR result performed on ARD02 at a grind size of 75  $\mu m$ , the grade profile was found to be similar to that of MPM001 and MPM003, with an exception for silica. The ARD02 contained a slightly higher content of  $SiO_2$  than what was recorded for MPM001 and MPM003. Nevertheless, the  $SiO_2$  grade was within the acceptable limit of 3%.

**Table 5.2: Davis Tube of MPM001 and MPM003 magnetic concentrates**

Sample	Grind size ( $\mu m$ )	Mass Dist (%)	Fe (tot)		$SiO_2$		$Al_2O_3$		$TiO_2$		$V_2O_5$	
			Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)
Historical Data (ARD02)	75	31.3	54.8	62.3	3.1	3.6	2.7	n/a	15.6	62.3	1.17	83.3
MPM 001	45	30.9	56.1	56.8	1.5	1.7	2.2	11.7	15.7	59.7	1.23	87.1
MPM 003	45	44	55.4	71.3	2	4.1	2.4	18.3	16.8	70.2	1.17	91.7

### 5.1.3 Low Intensity Wet Magnetic Separation (LIMS) Testwork

This test aims to produce a vanadium rich magnetic concentrate, while minimising the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{CaO}$  levels in the magnetic concentrate. The wet magnetic separation is an effective technique for upgrading the vanadium feed grade as well as rejecting gangue minerals that can potentially increase the salt consumption and decrease the water leach recoveries.

The LIMS testwork was conducted on a feed size of  $P_{100}$  75  $\mu\text{m}$  operating at 1000 Gauss. The LIMS testwork results for MPM001 and MPM003 are presented in Tables 5.3 and 5.4, respectively.

As shown in Table 5.3, the LIMS test result of MPM001 was not as encouraging as the Davis tube test result. A higher content of the gangue minerals was detected in the LIMS magnetic concentrate when compared to the Davis tube magnetic concentrate. The LIMS magnetic concentrate contained about 5.94%  $\text{SiO}_2$ . This value is approximately four times greater than what was observed in the Davis tube magnetic concentrate. The results indicated that there could still be a strong interlocking between the gangue minerals and valuable minerals at 75  $\mu\text{m}$ . Regrinding the magnetic concentrate would be needed to improve the minerals liberation and so the rejection of silica. It could also enhance the leach kinetics by providing an increased surface area. For MPM003, the LIMS test result was found to be comparable to the Davis tube test result. The observation is clearly illustrated in Table 5.4.

Examining the LIMS testwork results, MPM003 was found to yield a magnetic concentrate containing a higher content of  $\text{V}_2\text{O}_5$  and lower content of gangue minerals when compared to MPM001. The laboratory technician observed that sample MPM003 was relatively easier to grind than MPM001. These findings imply that both MPM001 and MPM003 could have distinct ore mineralogy from each other. A mineralogy investigation on the head sample is recommended to confirm this.

**Table 5.3: Comparison of DTR and LIMS test results for MPM001**

MPM 001												
Sample ID	Grind size ( $\mu\text{m}$ )	Mass Dist (%)	Fe		$\text{SiO}_2$		$\text{Al}_2\text{O}_3$		$\text{TiO}_2$		$\text{V}_2\text{O}_5$	
			Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)
DTR Mags	45	30.9	56.1	56.8	1.48	1.74	2.18	11.7	15.72	59.7	1.23	87.1
LIMS Mags	75	38.08	51.2	62	5.98	8.9	2.86	20.1	14.69	65	1.07	88.8

**Table 5.4: Comparison of DTR and LIMS test results for MPM003**

MPM 003												
Sample ID	Grind size (µm)	Mass Dist (%)	Fe		SiO <sub>2</sub>		Al <sub>2</sub> O <sub>3</sub>		TiO <sub>2</sub>		V <sub>2</sub> O <sub>5</sub>	
			Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)	Grade (%)	Dist (%)
DTR Mags	45	44	55.4	71.3	2.05	4.1	2.35	18.3	16.84	70.2	1.17	91.7
LIMS Mags	75	42.5	53.5	67.1	2.91	5.5	2.56	19.6	17.17	68.6	1.12	85.3

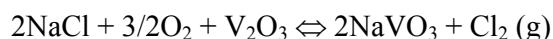
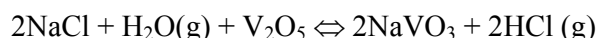
#### 5.1.4 Roasting/Water Leach Testwork

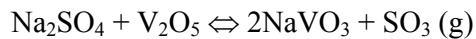
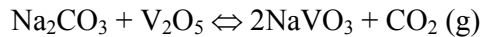
Magnetite is an inverse spinel structure where Fe<sup>3+</sup> ions occupy the tetrahedral sites and Fe<sup>2+</sup> and Fe<sup>3+</sup> occupying the octahedral sites. Ti<sup>4+</sup> can substitute for the Fe<sup>3+</sup> in the octahedral positions and the charge difference is compensated for by divalent ions. Complete substitution of Fe<sup>3+</sup> by titanium leads to the mineral ulvospinel (Fe<sup>2+</sup><sub>2</sub>TiO<sub>4</sub>), one end of the ulvospinel-magnetite solid solution series referred to as titanomagnetites or titaniferous magnetites. Other tri-valent cations such as V<sup>3+</sup>, Cr<sup>3+</sup>, and Al<sup>3+</sup> can substitute for the Fe<sup>3+</sup> in the magnetite or titanomagnetite structure. When the vanadium substitution is significant (>1%) the vanadiferous titanomagnetites can be a viable ore resource for vanadium. To extract the vanadium from these ores, the tri-valent vanadium ion (V<sup>3+</sup>) must be oxidised to the penta-valent ion (written as V<sub>2</sub>O<sub>5</sub>) and reacted with a sodium salt to form a water soluble sodium metavanadate.

The purpose of roast testwork is to convert the insoluble vanadium into readily water soluble sodium metavanadate, where the vanadium is then extracted by water leaching. The salt roasting followed by water leaching is the most common method used to recover vanadium from titaniferous magnetite concentrates. This method offers the advantage of being highly selective in comparison to acid and alkaline leaching.

Generally, the roasting test is performed at 800-1200°C in the presence of an oxidising atmosphere and a sodium salt. Sodium chloride, sodium sulfate and sodium carbonate are the commonly used sodium salts. The reactions involved are:

##### NaCl roasting



Na<sub>2</sub>SO<sub>4</sub> roastingNa<sub>2</sub>CO<sub>3</sub> roasting

Both sodium chloride and sodium sulfate can selectively attack vanadium, however hazardous gases/fumes such as HCl/Cl<sub>2</sub> vapour and SO<sub>3</sub> are generated during the roasting process. Sodium carbonate roasting is a more environmental friendly method. It is the most widely used sodium salt in current industry practice. It has been found to generate the best vanadium water leach recovery for magnetite when compared to Na<sub>2</sub>SO<sub>4</sub> or NaCl. On this basis, sodium carbonate is chosen as the sodium salt for this testwork.

In this study, the vanadium-rich magnetic concentrates produced from the LIMS magnetic separation are blended with sodium carbonate and then roasted at 1100°C for 2 hours. The samples were placed in fused silica crucibles. METS was concerned about the stability of the fused silica crucibles during the roasting process and requested the Amdel laboratory to conduct a blank test to determine the presence of side reactions between sodium carbonate and the container, at the test temperature.

Amdel reported that no apparent reaction occurred between the sodium carbonate and the fused silica crucible in the blank test. However, following the salt roast and leach test, the leach residue was found to contain a significantly higher SiO<sub>2</sub> content than was present in the roast feed. It was apparent that the crucible had reacted with roast charge, giving an increase in the silica content. This increase in the silica content is undesirable as it indicates some consumption of the salt in side reactions.

At the high roast temperature, silica can react with sodium carbonate to produce glassy silicate complexes. These reaction products can incorporate vanadium within its structure, thereby reducing the vanadium water leach recovery. Ideally, the vanadium-rich magnetic concentrate must have a combined SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> grade below 5% and a ratio of Al<sub>2</sub>O<sub>3</sub> to SiO<sub>2</sub> grade greater than 1 to achieve a good conversion of vanadium pentoxide into sodium metavanadate.

For MPM001, the leach residue contained a significant silica content of 10.71% (Table 5.5). The value is approximately twice that detected in the roast feed. The stoichiometric calculation reveals that there is an insufficient amount of salt in the system to drive the

conversion of vanadium pentoxide to form sodium metavanadate. Under a sodium salt deficient environment, the vanadium pentoxide is converted into sodium vanadate with Na/V ratio < 1. This compound tends to lose oxygen easily and forms water-insoluble species known as “bronze”. As a consequence, a poor V<sub>2</sub>O<sub>5</sub> recovery at 17.1% was recorded from the water leach test at 70°C.

For MPM003, while the leach residue was found to contain about 4.66% SiO<sub>2</sub> (Table 5.6), the amount of sodium carbonate added (9% w/w) satisfied the stoichiometric salt requirement of ~8.2% w/w. Hence, a higher V<sub>2</sub>O<sub>5</sub> recovery at 66.9% was recorded for MPM003 when compared to MPM001. However, this value is lower than what METS expects. There could be an insufficient level of oxygen in the furnace to create an oxidising environment for the roasting process. No oxygen was sparging during the roasting process. The roasting was carried out in a muffle furnace in air.

**Table 5.5: Roast and water leach test results for MPM001**

Sample	MPM001										
	Solids Assays (%)										
	V <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	Mn	P	K <sub>2</sub> O	Na <sub>2</sub> O
Roast Feed	1.07	14.7	51.2	5.98	2.86	2.99	0.79	0.34	0.02	0.1	0.16
Leach Residue	0.77	12.5	43.6	10.7	2.93	2.6	0.69	0.29	0.02	0.14	6.5
Extraction (%)	17	2	2	-106	-18	0	0	2	9	-59	-4662
Sample	Solution Assays (mg/L)										
	V	Ti	Fe	Si	Al	Mg	Ca	Mn	P	K	Na
Leach PLS Solution	360	1	4.5	355	60	-0.5	0.5	0.5	15	10	2480

**Table 5.6: Roast and water leach test results for MPM003**

Sample	MPM003										
	Solids Assays (%)										
	V <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	Mn	P	K <sub>2</sub> O	Na <sub>2</sub> O
Roast Feed	1.12	17.2	53.5	2.91	2.56	1.68	0.28	0.35	0.008	0.04	0.05
Leach Residue	0.35	15.5	48.7	4.66	2.48	1.58	0.26	0.32	0.003	0.064	4.55
Extraction (%)	67	3	2	-72	-4	-1	0	2	60	-72	-9698
Sample	Solution Assays (mg/L)										
	V	Ti	Fe	Si	Al	Mg	Ca	Mn	P	K	Na
Leach PLS Solution	1545	1	5.25	238	121	-0.5	1.25	-0.5	20	22.5	3465

The low vanadium recoveries were attributed to the roasting conditions rather than the quality of the ores. The low extraction efficiency of  $V_2O_5$  was due to:

- the noticeable increase in the silica content, where the amount of sodium carbonate (9% w/w) added was insufficient to meet the stoichiometric salt requirement to produce sodium metavanadate
- the absence of highly oxidising atmosphere to drive the conversion of vanadium pentoxide to form sodium metavanadate

METS strongly recommend that the testwork be repeated under an oxygen-enriched environment using an inert container (zirconium oxide) so that a true recovery of vanadium can be determined. Alternatively, an alumina crucible can also be employed. It has been known that alumina is beneficial in suppressing the formation of low melting point glassy silicates complexes. Aluminium exhibits the tendency to form complex with silicates that has a higher melting temperature than most the glassy silicate complexes. These alumina-based complexes incorporate less vanadium in the lattice, and thus improving the water leach recovery of vanadium.

## **5.2 QEMSCAN Analysis on the LIMS Concentrate**

A mineralogical investigation on the LIMS magnetic concentrates from the Mount Peake deposit was conducted using Quantitative Evaluation of Minerals by Scanning Electron Microscope (QEMSCAN) Particle Mineralogical Analysis (PMA) and Energy Dispersive Spectrometer (EDS). The analysis was to examine the mineralogical reasons for the poor vanadium extraction, especially for sample MPM001. The QEMSCAN-PMA gave information on the quantitative mineralogy and metal deportment, while the QEMSCAN-EDS was used to establish chemical composition and impurities of the key minerals.

The sample was scanned for:

- Mineralogical Content
- Elemental Deportment
- Liberation Characteristic
- Association of various minerals.

The AMMTEC report on the QEMSCAN analysis is available in Appendix B.

### 5.2.1 Mineral Abundances

The mineral content, measured by QEMSCAN-EDS, are summarised in Table 5.7. The QEMSCAN analysis indicated that both MPM001 and MPM003 primarily contained titanomagnetite and accessory magnetite and ferromagnesian silicates. The titanomagnetite is a solid solution of the ulvospinel-magnetite series with an approximate formula of  $\text{Fe}_2\text{Ti}_{(0.3-0.5)}\text{O}_4$ . There were also some minor quantities of feldspars, ilmenite, sphene, quartz and sulfides. Compared to MPM001, MPM003 was found to contain more titanomagnetite and less ferromagnesian silicate. The minor silicates, feldspar and quartz, are lower in MPM003 than in MPM001. This is in agreement with the LIMS assay analysis, which also showed that the MPM001 sample contained a considerably higher silica content. Other minerals, such as carbonates and apatite are of low content.

**Table 5.7: Mineral Mass Percent in Sample**

Mineral Grouping	Mineral mass percent (%) in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	81.11	86.63
Magnetite	5.65	5.65
Ilmenite	0.52	0.51
Sphene	0.53	0.03
Pyrite	0.29	0.27
Pyrrhotite	0.07	0.06
Chalcopyrite	0.01	0.01
Ferromagnesian	9.85	6.03
Feldspars	1.62	0.68
Quartz	0.22	0.11
Carbonates	0.02	0.02
Apatite	0.04	0
Minor Phases	0.06	0.01

### 5.2.2 Vanadium Deportment

In both MPM001 and MPM003, the vanadium is found in association with titanomagnetite. Minor quantities of vanadium are locked in the ferromagnesian silicates, magnetite, ilmenite and sphene. It is important to note from Table 5.8 that there is a significant proportion of vanadium in MPM001 sample is occurs within ferromagnesian silicates and sphene compared to MPM003. These minerals are non-magnetic. The rejection of these minerals could lead to lower vanadium recovery in the MPM001 when compared to MPM003.

**Table 5.8: Vanadium Deportment**

Mineral Grouping	Mass V % in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	94.81	97.57
Magnetite	0.57	0.54
Ilmenite	0.16	0.15
Sphene	0.85	0.04
Ferromagnesian	3.61	1.17

### 5.2.3 Iron Deportment

Similar to vanadium, the iron is also associated mainly with the titanomagnetite and magnetite. The remaining iron is contained within the ferromagnesian silicate. This is clearly demonstrated in Table 5.9.

**Table 5.9: Iron Deportment**

Mineral Grouping	Mass Fe % in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	89.8	90.85
Magnetite	7.46	7.11
Ilmenite	0.34	0.32
Sphene	0.03	0
Ferromagnesian	2.03	1.42
Pyrite	0.25	0.22
Pyrrhotite	0.08	0.07
Chalcopyrite	0.01	0
Feldspars	0	0
Carbonates	0	0



### 5.2.4 Titanium Deportment

As shown in Table 5.10, the majority of the titanium is found within titanomagnetite. Minor amounts of titanium were also detected in ilmenite and the ferromagnesian silicates.

**Table 5.10: Titanium Deportment**

Mineral Grouping	Mass Ti % in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	96.07	97.57
Magnetite	0.08	0.03
Ilmenite	1.55	1.43
Sphene	0.83	0.03
Ferromagnesian	1.47	0.78

### 5.2.5 Silicon Deportment

The majority of the silicon is contained within ferromagnesian silicates (Table 5.11). About one tenth of the silicon is found within feldspars. A minor amount of silicon is locked within titanomagnetite. The removal of ferromagnesian silicates is highly desirable to reduce the silica level in the concentrate to enhance the vanadium extraction during the salt roast process.

**Table 5.11: Silicon Deportment**

Mineral Grouping	Mass Si % in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	1.52	2.88
Ilmenite	0	0
Sphene	2.54	0.2
Ferromagnesian	79.33	84.18
Feldspars	12.8	9.32
Quartz	3.8	3.42

### 5.2.6 Aluminium Deportment

As shown in Table 5.12, the detected aluminium is associated mainly in the titanomagnetite and ferromagnesian silicates and to a lesser extent with feldspar. When aluminium is locked

within the titanomagnetite, this could be beneficial as alumina can suppress the formation of glassy silicate complexes during the roasting process.

**Table 5.12: Aluminium Deportment**

Mineral Grouping	Mass Al % in sample	
	MPM 001 LIMS Mags	MPM 003 LIMS Mags
Titanomagnetite	48.76	64.73
Magnetite	1.02	1.3
Ilmenite	0	0
Sphene	0.71	0.04
Ferromagnesian	32.33	25.69
Feldspars	16.19	8.02
Apatite	0.08	0.03
Minor Phases	0.9	0.19

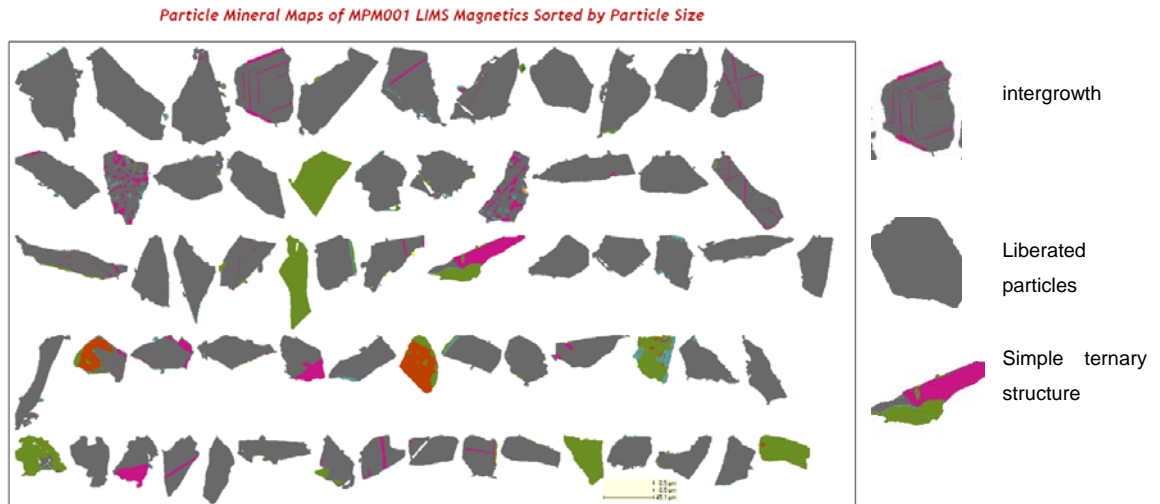
### **5.2.7 Liberation and Locking of Titanomagnetite, Magnetite, Ilmenite and Ferromagnesian Silicates**

As shown in Figure 5.3, titanomagnetite was found as liberated particles in both the MPM001 and MPM003 concentrates. Some of the well-liberated titanomagnetite retains some intergrowth with magnetite, ilmenite and ferromagnesian silicates. The intergrowths occur more frequently in MPM003 than in MPM001. These intergrowth structures are usually strongly bounded and difficult to liberate by fine grinding. This is aligned with the DTR observation which showed a similar grade at 45 µm and 75 µm grinds.

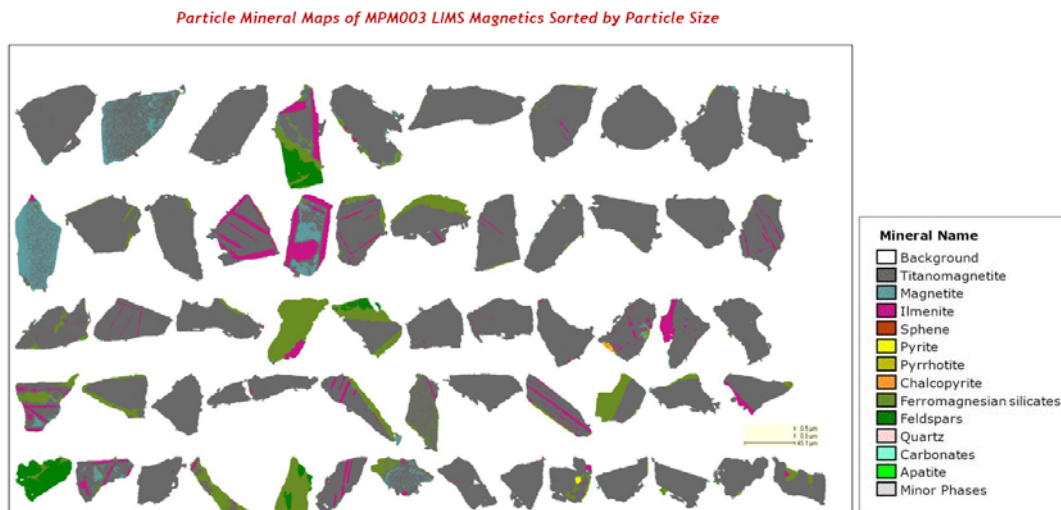
Ilmenite is effectively locked with titanomagnetite, forming oriented tabular exsolution within the titanomagnetite host. Sphene forms some association with titanomagnetite and magnetite, but predominantly with ferromagnesian silicates.

There are also some simple binary and ternary composites. The gangue minerals in these composites often can be liberated from the valuable minerals by simple fine grinding.

Some free particles of ferromagnesian silicates were also detected in MPM001. These particles are not removed during the LIMS wet magnetic separation, possibly due to an entrainment effect. A cleaner magnetic separation stage could assist the removal of these ferromagnesian silicates and other entrained gangue.



(a)



(b)

**Figure 5.3: Particle mineral maps of (a) MPM001 and (b) MPM003 magnetic concentrates**

Since vanadium is hosted predominantly within titanomagnetite, this can present a problem during the roasting process. Higher temperature treatment may be needed to oxidise the titanomagnetite, which is the host phase for vanadium. Essentially, successful magnetite oxidation will lead to high vanadium recovery.

## 5.3 Phase 2 testwork

### 5.3.1 Low Intensity Wet Magnetic Separation (LIMS) on the Regrind Samples

In an attempt to improve the vanadium recovery in the roast/leach process, a second phase of testwork was developed to improve the silica rejection. The magnetic concentrate was subjected to regrinding prior to magnetic separation, roasting and water leach tests. The aim is to produce a magnetic concentrate with a combined silica and alumina grade less than 5%, to minimise the competing reaction between the silica and vanadium for the sodium salt during the roasting process.

The phase one magnetic concentrates were reground to  $P_{100} \sim 45 \mu\text{m}$  followed by LIMS separation. The LIMS test results is presented in Table 5.13.

**Table 5.13: LIMS test result on the regrind samples**

MPM 001						
Sample	Grind size (mm)	Grade (%)				
		Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>
LIMS Mag Conc.	75	51.2	6	2.9	14.7	1.07
LIMS Regrind Mag	45	56.8	1.5	2.3	15.9	1.3
MPM 003						
Sample	Grind size (mm)	Grade (%)				
		Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>
LIMS Mag Conc.	75	53.5	2.9	2.6	17.2	1.1
LIMS Regrind Mag	45	55.6	1.6	2.4	17.2	1.2

Encouraging results were seen with the LIMS test results on the regrind samples. For MPM 001, the vanadium was upgraded from 1.07% to 1.26% V<sub>2</sub>O<sub>5</sub> following a high rejection of silica. The silica grade decreased from 6% to 1.5% SiO<sub>2</sub>. It was also noticed that the iron was upgraded from 51.2% to 56.8% Fe.

A similar observation was evidenced with MPM 003. An upgrade in the vanadium grade from 1.12% to 1.21%  $V_2O_5$  was recorded. The iron content was also upgraded from 53.5% to 55.6% Fe while the silica grade decreased from 2.9% to 1.6%  $SiO_2$ .

For both samples, the LIMS successfully produced a magnetic concentrate with a combined silica and alumina grade less than 5% and the ratio of alumina-to-silica greater than 1, which meets the feed requirement of the vanadium roast process.

A mass balance was also performed on the samples to determine the weight and metal recoveries. The results are summarised in Table 5.14.

**Table 5.14: Weight and metal recoveries on the regrind samples**

Sample	Mass recovery (%)	Recovery (%)				
		Fe	$SiO_2$	$Al_2O_3$	$TiO_2$	$V_2O_5$
MPM 001	33.8	59.5	2.2	14.2	62.4	88.1
MPM 003	40.9	66.0	3.1	17.5	66.9	84.7

For MPM001, about 88.1% of the  $V_2O_5$  was recovered in 33.8% of the mass. For MPM003, about 84.7% of the  $V_2O_5$  was recovered into 40.9% of the mass.

### 5.3.2 Roasting/Water Leach Test on the Regrind Samples

The LIMS magnetic concentrate of the regrind samples were subjected to roasting followed by water leach for vanadium recovery. To promote the conversion of vanadium to sodium metavanadate, excess sodium carbonate (~11% w/w) was introduced into the system. Sodium percarbonate (2% w/w) was also added to provide an additional source of oxidant to drive the oxidation of the titanomagnetite and the vanadium  $V^{3+}$  to  $V^{5+}$ . Under high temperature, sodium percarbonate decomposes, releasing  $H_2O_2$  to drive the oxidation mechanism. The roast test was also carried out at a higher temperature of 1150°C to facilitate the extraction of vanadium from the titanomagnetite. The roast test was conducted in a rotating furnace at CSIRO using a fused alumina tube to support the samples.

The roast/water leach results for MPM001 and MPM003 are presented in Table 5.15 and Table 5.16, respectively.

**Table 5.15: Roast and water leach test results for MPM001 regrind sample**

Sample	MPM001 (Regrind Sample)										
	Solids Assays (%)										
	V <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	Mn	P	K <sub>2</sub> O	Na <sub>2</sub> O
Roast Residue/ Leach Feed	1.02	14.0	51.1	1.48	2.74	1.29	0.23	0.42	0.010	0.020	6.52
Leach Residue	0.33	14.7	54.0	1.46	2.48	1.36	0.24	0.45	0.003	0.011	3.30
Extraction (%)	69	0	-1	6	14	-1	0	-2	71	47	52
Sample	Solution Assays (mg/L)										
	V	Ti	Fe	Si	Al	Mg	Ca	Mn	P	K	Na
Leach PLS Solution	1620	<0.2	1.25	205	800	<0.2	2.75	<0.2	25	40	9075

**Table 5.16: Roast and water leach test results for MPM003 regrind sample**

Sample	MPM003 (Regrind Sample)										
	Solids Assays (%)										
	V <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	CaO	Mn	P	K <sub>2</sub> O	Na <sub>2</sub> O
Roast Residue/ Leach Feed	0.98	15.2	50.3	1.61	3.12	1.08	0.09	0.41	0.009	0.017	6.23
Leach Residue	0.28	16.0	53.1	1.60	2.72	1.14	0.09	0.44	0.002	0.011	3.12
Extraction (%)	73	0	-1	6	17	0	5	-2	79	38	52
Sample	Solution Assays (mg/L)										
	V	Ti	Fe	Si	Al	Mg	Ca	Mn	P	K	Na
Leach PLS Solution	1830	<0.2	<0.5	160	1240	<0.2	2.25	<0.2	30	25	9485

After roasting, when the materials were removed from the fused alumina crucible, it was noticed that some of material stuck to the wall of the alumina tube. About 77.6% (or 77.6 g in 100 g) of the feed mass was recovered for MPM001. With MPM003, only 63.3% (or 63.3 g in 100 g) of the feed mass was recovered.

The roasted products were assayed before they were water leached. Compared to the roast feed, the roasted products of MPM001 and MPM003 contained a lower grade of V<sub>2</sub>O<sub>5</sub>. The drop in the V<sub>2</sub>O<sub>5</sub> grade is ascribed to the loss of vanadium on the wall of the alumina tube.

For MPM001, as the silica level in the magnetic concentrate reduced from 6% to 1.5%, the V<sub>2</sub>O<sub>5</sub> recovery increased from 17% to 69%. The decrease in the silica level suppresses the formation of a glassy silicate, improving the extraction of vanadium. It also eliminates the competing side reaction between silica and the sodium salt. The coupling of these effects leads to an increase in the vanadium recovery.

For MPM003, the vanadium recovery improved slightly from 67% to 73% when the silica level in the magnetic concentrate was reduced from 2.9% to 1.6%. The  $V_2O_5$  recovery of MPM003 was slightly higher than MPM001. Previously, the QEMSCAN analysis showed that about 3.61% of vanadium in the magnetic concentrate was contained within a ferromagnesian silicate. Liberating vanadium from ferromagnesian silicate during roasting can be quite challenging. Often, the insoluble vanadium species is encapsulated within the structure of ferromagnesian silicates.

It was expected that the presence of sodium percarbonate would promote the conversion of  $V^{3+}$  to  $V^{5+}$  and subsequent reaction with sodium to form sodium metavanadate, and hence improve  $V_2O_5$  recovery. However, the positive effect of the oxidant is only mild as suggested by the slight increase in the  $V_2O_5$  recovery of MPM003. This indicates that the oxidising conditions in the air atmosphere in the muffle furnace was adequate and that the low roast/leach recovery of MPM001 in the phase one leaches was related to the high silica content and starvation levels of the sodium salt.

It is anticipated that the remaining vanadium is locked with the titanium phase of the titanomagnetite. Effectively, extraction of vanadium from the titanium phase is critical to maximise the recovery of vanadium from titanomagnetite. QEMSCAN analysis on the leach residue would be required to confirm the vanadium deportment.

A study by Mukherjee et al.<sup>1</sup> (2005) showed that titanium dioxide is soluble in oxalic acid and ascorbic acid. The underlying mechanism behind the dissolution of titanium dioxide in organic acid is unknown. However, they observed about 40% of 0.05M titanium dioxide was dissolved in 0.15M oxalic acid under a mild stirring condition for 4 hours. They also reported that 50% of 0.05M titanium dioxide was dissolved in 50% HCl in 4 hours. On the basis of these findings, METS recommends the roast test be repeated using sodium oxalate or sodium chloride. While the sodium ion reacts with vanadium pentoxide to form sodium metavanadate, the oxalate ion will attack the titanium oxide, liberating the vanadium from the titanium phase of titanomagnetite.

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<sup>1</sup> A. Mukherjee, A. M. Raichur, J. M. Modak (2005) Chemosphere, vol. 61, p.585-588

## 6. CONCLUSION

A testwork program comprised of head assay, mineralogical investigation, Davis tube recovery test, wet magnetic separation, salt roasting and water leaching was undertaken to investigate the vanadium recovery from magnetite ore of the Mount Peake project.

Both the Davis tube recovery and LIMS showed that the ore can be successfully upgraded by magnetic separation. At 75  $\mu\text{m}$ , the LIMS test result on MPM001 showed that vanadium was upgraded from 0.44% to 1.2%  $\text{V}_2\text{O}_5$  at a recovery of 89%. However, the silica level (6%  $\text{SiO}_2$ ) was higher than the recommended level of 3%  $\text{SiO}_2$ . Fine grinding would be required to improve the gangue mineral rejection. The Davis tube test result, which was carried out at a grind size of 45  $\mu\text{m}$ , showed a comparatively low level of silica at 1.5%  $\text{SiO}_2$ .

For MPM003, the LIMS test achieved a  $\text{V}_2\text{O}_5$  recovery of 85% with the vanadium upgraded from 0.56% to 1.3%  $\text{V}_2\text{O}_5$ . The silica level (2.9%  $\text{SiO}_2$ ) was low and within the acceptable level of 3%  $\text{SiO}_2$ .

For both MPM001 and MPM003, mineralogical investigation on the LIMS magnetic concentrates showed that titanomagnetite is the predominant mineral, which also hosted the majority of the vanadium, titanium and iron. Silicon is mainly found as ferromagnesian silicates. There are some free ferromagnesian silicates in the MPM001. Re-treating the magnetic concentrate through another stage of magnetic separation (cleaner stage) could assist the removal of the ferromagnesian silicates. However, since the QEMSCAN mineralogy showed that 3.61% of the vanadium is locked within the ferromagnesian silicates, some vanadium will be rejected along with the ferromagnesian silicates..

It was also revealed in the mineralogical investigation that some of the titanomagnetite is intergrown with magnetite, ilmenite and the ferromagnesian silicates. Apart from that, some particles exist as simple binary and ternary composites. Fine grinding can be employed to improve the mineral liberation from these particles and hence improve the grade of the valuable mineral.

When the phase 1 LIMS magnetic concentrate was subjected to salt roasting and water leach test, the results were not encouraging. Poor vanadium water leach recoveries were recorded with 17% of the  $\text{V}_2\text{O}_5$  being recovered from MPM001 and 67% of the  $\text{V}_2\text{O}_5$  recovered from MPM003. Nonetheless, the test results were inconclusive. There was a high level of silica detected in the leach residue, possibly arising from contamination due to the reaction between



sodium carbonate and the fused silica crucible. With the increase in the silica content, there was insufficient salt to drive the formation of sodium metavanadate. Another potential reason for the poor water leach vanadium recoveries was the lack of oxygen in the furnace to induce the oxidation of vanadium.

Subsequent to this, a phase 2 testwork program was developed to improve the low  $V_2O_5$  recovery. In the new testwork program, the magnetic concentrate from phase 1 was subjected to regrinding, followed by LIMS separation for silica rejection. The magnetic concentrates were then roasted before being leached in water for vanadium recovery.

The LIMS test results on the regrind samples were positive. Not only that the silica level in the magnetic concentrate was reduced to an acceptable limit for the roast feed, but also the vanadium grade in the magnetic concentrate improved slightly. For MPM001, the vanadium grade was increased from 1.1% to 1.3%  $V_2O_5$ , and for MPM003, the vanadium grade of MPM003 increased from 1.1% to 1.2%  $V_2O_5$ .

The phase 2 roast test was carried out in a rotating furnace using a fused alumina crucible to support the sample. Sodium carbonate was used as the sodium salt. Sodium percarbonate was also added to provide the extra source of oxidant. When sodium percarbonate is decomposed,  $H_2O_2$  is released to promote the vanadium oxidation.

At the end of the roast process, some of the samples were bonded very strongly on the wall of the tube and not recovered for the water leach test. This led to a slight decrease in  $V_2O_5$  grade in the water leach feed.

For MPM001, decreasing the silica content in the magnetic concentrate from 6% to 1.5% led to an improvement in the  $V_2O_5$  recovery from 17% to 69%. The positive enhancement is attributed to the coupling effects of the reduced competing side reaction from the silica for the sodium salt, and the suppression of the low melting glassy silicate complex formation.

The MPM003 achieved a  $V_2O_5$  recovery of 73% from the roast/water leach test. Comparing with the first roast/water leach test, the  $V_2O_5$  recovery was only mildly enhanced. This indicates that the remaining vanadium could be locked within the titanium phase of the titanomagnetite and liberating vanadium from the titanium phase may be the key to maximising the recovery of vanadium.

## **Appendix A: AMDEL TESTWORK REPORT**

## **Appendix B: AMMTEC TESTWORK REPORT**