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ORETEST
Metallurgical Testwork and Research

QUALITY MANAGEMENT SYSTEM

RECOVERY OF MAGNESIUM CHLORIDE

FROM

MAGNESITE COMPOSITES

CLIENT: Golden Triangle Resources NL

JOB No.: 7740b

DATE: 27 August 1998

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SUMMARY

Mr Andrew Firek, on behalf of Golden Triangle Resources NL, requested Oretest Pty Ltd to undertake a two-part investigation into the recovery of magnesium as magnesium chloride, from magnesite.

The first part entitled, "Flotation Testwork on Two Composite Magnesite Ores from Main Creek", Report No. 7740 was dated 22 July 1998. The second part, the subject of this report, dealt with the leaching of magnesite and the recovery of crystalline magnesium chloride.

The objectives of the investigation were to:

- Characterise the samples by size analysis, chemical analysis and DTA/TG.
- Determine optimum conditions for the calcination of the samples and the generation of magnesia products.
- Leach the magnesite samples with concentrated hydrochloric acid and establish optimum conditions over a given range of temperature, time and % acid stoichiometry.
- Neutralise the leach liquors with calcine and measure the elemental concentrations in the filtered solutions.
- Recover the magnesium chloride from the neutralised solutions and to determine the quality of the product.

SAMPLE CHARACTERISATION

Two samples were used in this work: *Comp. 1 (Conc.)* and *Comp. 1 (-2 mm)*. Comp. 1 (Conc.) was a flotation upgraded product and was produced during stage 1 of the project. The analyses of the two samples were as follows:

Table S1. Head Analysis of Magnesite Samples

Sample	LOI, %	Mg, %	Ca, %	Si, %	Fe, %	Mn, ppm
Comp 1 (-2 mm)	50.1	26.47	1.66	1.32	0.56	412
Comp 1 (Flot Conc.)	51.43	27.2	1.68	<0.02	0.48	427

Sample	Ni, ppm	Cu, ppm	Zn, ppm	Cr, ppm	Al, ppm	B, ppm
Comp 1 (-2 mm)	<10	11	25	<10	<100	<20
Comp 1 (Flot Conc.)	<10	<5	32	<10	<100	<20

The main difference between the two was the silicon content, (1.32% in the –2 mm material versus <0.02% in the concentrate).

Sizing analyses of the two showed that the coarser of the two (the -2 mm material) consisted of particles, approximately half of which were greater than 0.5 mm in diameter. The concentrate was much finer, with approximately 55% less than 30 μm.

The DTA/TG curves demonstrated the characteristic endothermic peaks for the decomposition of magnesium carbonate (peak value at approximately 690 - 700°C) and calcium carbonate (peak at approximately 770°C). Both samples also indicated an apparent exothermic peak at approximately 800°C.

CALCINATION

Calcination tests were carried out on Comp. 1 (Conc.) at temperatures of 600, 650 and 700°C. For a calcination period of 60 minutes, it was shown that, with increasing temperature: the mass loss and the % Mg increased, the LOI of the calcined products demonstrated decreasing values and the calcines sustained a decrease in activity. Although tests at 700°C resulted in higher mass losses and higher % Mg values, the activity was lower than that obtained at 650°C. Optimum conditions selected for the calcination of Comp. 1 (Conc.) were 650°C and 60 minutes.

Calcination of the –2 mm material at 650°C and for 60 minutes, returned an activity of 600 seconds. This value was improved to 32 seconds after pulverising. A summary of the calcination results is given in the table below:

TABLE S2. MAGNESITE CALCINATION

Calcine No.	Magnesite Sample	Temperature °C	Time mins.	Mass Loss, %
1	Comp 1 (-2mm)	650	60	45.5 45.7
2	Comp 1 (Flot Conc.)	650	60	46.2
3	Comp 1 (Flot Conc.)	650	120	46.8
4	Comp 1 (Flot Conc.)	600	60	46.3 45.2
5	Comp 1 (Flot Conc.)	700	60	47.0
6	Comp 1 (Flot Conc.)	700	120	47.5

ACID LEACHING

A summary of the acid leach results is presented in Table S3 overleaf.

A maximum magnesium recovery of 90.7% was obtained at 50°C, 120 minutes and an acid stoichiometry of 120%. During this leach a corresponding 86.8% calcium was recovered. Another leach resulted in recoveries of magnesium and calcium of 86.7% and 92.8% respectively. The conditions for this test were 75°C, 120 minutes and 110% acid stoichiometry. The optimum conditions for leaching could possibly be derived from a combination of the parameters used in these two tests.

The higher silica content in the –2 mm material did not appear to contribute to higher soluble silica values. The maximum value recorded for the soluble silica (expressed as Si) in the leach liquor was 7 mg L^{-1}. Boron was also shown to be low with values in the leach liquors which ranged from 3.1 – 3.8 mg L^{-1}.

Tests showed that calcium leached readily from both the –2 mm and concentrate samples. During a room temperature leach for 60 minutes, calcium dissolution was as high as 85% while magnesium recovery was only 17%. This result suggested that a preleach would be a possible means of selectively removing a large proportion of calcium from the magnesite.

TABLE S3 ACID LEACH RESULTS

Leach No.	Magnesite Sample	Temperature °C	Time mins.	Acid Stoich., %	P.D. w/v %	Mass, % Dissolved	Recovery, % Mg	Recovery, % Ca
1	Comp 1 (Flot conc.)	RT	60	110	35.2	21.2	15.7	88.8
2	Comp 1 (Flot conc.)	50	60	110	35.2	75.7	77.5	86.5
4	Comp 1 (Flot conc.)	50	120	110	35.2	84.6	83.3	86.6
10	Comp 1 (-2mm)	RT	60	110	36.0	21.1	16.9	85.5
11	Comp 1 (-2mm)	50	60	110	36.0	72.0	72.4	82.0
12	Comp 1 (-2mm)	50	120	110	36.0	79.6	77.9	81.9
13	Comp 1 (-2mm)	75	60	110	36.0	91.1	82.3	92.3
14	Comp 1 (-2mm)	75	120	110	36.0	92.1	86.7	92.8
15	Comp 1 (-2mm)	50	60	120	33.4	68.3	77.1	85.5
16	Comp 1 (-2mm)	50	120	120	33.4	78.0	90.7	86.8

NEUTRALISATION

The leach liquors were neutralised with magnesia generated from the –2 mm sample calcined at 650°C for 60 minutes. The analyses of the leach liquors are summarised in Table S4 overleaf.

The neutralisation curves showed that neutralisation occurred at a pH of approximately 6. During the tests calcine additions were made to the liquor in an attempt to adjust the pH to 7 but it was shown later that this only contributed to an excess of MgO in solution. On standing, this excess resulted in a precipitate of either $Mg(OH)_2$ or Sorel cement (represented by the formula, $MgCl_2.3Mg(OH)_2.8H_2O$).

During neutralisation, iron precipitated mainly as a dark blue Fe(II) hydroxide. However in some cases it was observed that some Fe(III) had precipitated as the hydrated oxide (typified by a yellow/brown colour). Evidence of Fe(III) precipitation was recorded in some of the neutralisation curves with a pH plateau at approximately 2. The lowest level of iron in the neutralised liquors was 12 mg L^{-1} and this corresponded to the test in which the yellow/brown precipitate was the most apparent. This result would indicate that the extent of iron removal would increase should oxidation be employed to precipitate the iron in the Fe(III) condition.

Calcium removal would be enhanced by treating liquors (leach or neutralised liquors) with sulphuric acid. The principle of this treatment depends on the insolubility of calcium sulphate. The step would be warranted should lower levels of calcium be required.

CRYSTALLISATION

The crystallisation process resulted in a purification of the magnesium chloride with an approximate 90% rejection of calcium, iron and manganese. Table S5 summarises the results of the crystallisation tests and Table S6 provides some data regarding the rejection of impurities during crystallisation.

The deliquescent nature of magnesium chloride hexahydrate required that the crystals be dried at an elevated temperature. However the temperature needed to be maintained at a value below which decomposition commenced.

TABLE S4. ANALYSIS OF NEUTRALISED LEACH LIQUORS

Test No	Leach Soln. Vol., mL	Total Calcine Addition, g	Final pH	[Mg] g L^{-1}	[Ca] g L^{-1}	[Fe] mg L^{-1}	[Mn] mg L^{-1}	[Ni] mg L^{-1}	[Zn] mg L^{-1}	[Cu] mg L^{-1}	[Cr] mg L^{-1}	[Al] mg L^{-1}
1	200	57.28	6.58	109	13.60	40	70	2.3	0.7	0.8	0.1	2.6
2	200	50.27	6.40	114	8.07	30	158	<0.1	1.2	1.0	1.0	1.6
4	200	47.38	6.47	113	7.44	35	159	<0.1	1.7	0.9	1.7	2.0
10	200	43.66	7.00	100	13.00	12	104	0.3	1.6	0.3	<0.1	2.8
11	200	33.94	6.92	108	7.74	44	112	<0.1	1.0	0.1	<0.1	2.0
12	200	65.42	6.47	115.4	7.30	32	72	<0.1	0.4	<0.1	0.1	3.6
13	200	61.36	6.63	108.2	6.60	36	62	<0.1	0.4	<0.1	<0.1	3.8
14	200	40.46	6.53	108	7.84	45	134	<0.1	0.7	0.2	0.2	2.0
15	200	42.46	6.59	104	6.36	62	142	<0.1	1.5	0.2	0.2	2.5
16	200	49.76	6.44	110.8	7.35	47	162	<0.1	1.0	0.1	0.2	3.0

TABLE S5. MAGNESIUM CHLORIDE CRYSTALLISATION AND THEIR ANALYSES

Test No.	Vol. of Neutralised Leach Liquor	Mass of Liquor prior to Crystallisation, g	Mass of Crystals g	Make-up Solution Vol, mL	Analysis, ppm[+]								
					Mg	Ca	Mn	Fe	Ni	Cu	Zn	Cr	Al
1	100	105.3	9.7	150	6150	85 74	1 0.4	<0.1	0.1	<0.1	0.2	<0.1	<1
2	100	112.77	2.87	150	2850	20 20	0.7 0.3	<0.1	0.1	<0.1	0.2	<0.1	<1
4	100	107.33	5.33	150	4070	32 30	0.9 0.6	<0.1	0.1	<0.1	0.2	<0.1	<1
10	100	70.98	3.08	50	7700	13.7	<0.1	0.4	<0.1	<0.1	0.1	<0.1	<1
11	70	63.66	0.46	50	1130	2.4	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<1
12	50	63.72	0.12	50	285	1.7	<0.1	0.2	<0.1	<0.1	<0.1	<0.1	<1
13	70	75.55	5.15	150	4150	19 18	0.6 <0.1	<0.1	0.1	<0.1	0.2	<0.1	<1
14	100	92.05	5.95	150	4700	31 29	1.1 0.5	<0.1	0.1	<0.1	0.2	<0.1	<1
15	100	91.94	13.24	150	10000	74 63	2 0.8	<0.1	0.1	<0.1	0.2	<0.1	<1
16	100	109.92	14.52	150	12000	73 62	2.1 1.1	<0.1	0.1	<0.1	0.2	<0.1	<1

[+] Second set of values are those from Analabs

TABLE S6. ELEMENTAL REJECTION DURING CRYSTALLISATION

Test No.	Solution	Element Conc., expressed as a ratio relative to [Mg]			
		Mg	Ca	Mn	Fe
1	Neutralised Leach Liquor	1	0.125	0.00037	0.00064
	Crystal Solution	1	0.014	0.00000016	0.00000002
	% Rejection	-	88.9	100.0	100.0
2	Neutralised Leach Liquor	1	0.071	0.00139	0.00026
	Crystal Solution	1	0.007	0.000246	0.000035
	% Rejection	-	90.1	82.3	86.7
4	Neutralised Leach Liquor	1	0.066	0.00141	0.00031
	Crystal Solution	1	0.008	0.000221	0.000025
	% Rejection	-	88.1	84.3	92.1
10	Neutralised Leach Liquor	1	0.130	0.00104	0.00012
	Crystal Solution	1	0.002	0.000013	0.000052
	% Rejection	-	98.6	98.8	56.7
11	Neutralised Leach Liquor	1	0.072	0.00104	0.00041
	Crystal Solution	1	0.002	0.000088	0.000088
	% Rejection	-	97.0	91.5	78.3
12	Neutralised Leach Liquor	1	0.063	0.00062	0.00028
	Crystal Solution	1	0.006	0.000351	0.000702
	% Rejection	-	90.6	43.8	-153.1
13	Neutralised Leach Liquor	1	0.061	0.00057	0.00033
	Crystal Solution	1	0.005	0.000145	0.000024
	% Rejection	-	92.5	74.8	92.8
14	Neutralised Leach Liquor	1	0.073	0.00124	0.00042
	Crystal Solution	1	0.007	0.000234	0.000021
	% Rejection	-	90.9	81.1	94.9
15	Neutralised Leach Liquor	1	0.061	0.00137	0.00060
	Crystal Solution	1	0.007	0.000200	0.000010
	% Rejection	-	87.9	85.4	98.3
16	Neutralised Leach Liquor	1	0.066	0.00146	0.00042
	Crystal Solution	1	0.006	0.000175	0.000008
	% Rejection	-	90.8	88.0	98.0

1 INTRODUCTION

Mr Andrew Firek, on behalf of Golden Triangle Resources NL, requested Oretest Pty Ltd to undertake a testwork program involving the recovery of magnesium as magnesium chloride from magnesite samples.

This report corresponds to the second part of an overall investigation into magnesite which relates to the leaching, neutralisation and the precipitation of crystalline magnesium chloride.

The first part entitled, "Flotation Testwork on Two Composite Magnesite Ores from Main Creek", Report No. 7740, was dated 22 July 1998.

The objectives of the testwork program were to:

- Characterise the samples by size analysis, chemical analysis and TGA/DTA.
- Determine optimum conditions for the calcination of the samples and the generation of magnesia products.
- Leach the magnesite samples with concentrated hydrochloric acid and establish optimum conditions over a given range of temperature, time and % acid stoichiometry.
- Neutralise the leach liquors with calcine and measure the elemental concentrations in the solutions.
- Recover the magnesium chloride from the neutralised solutions and to determine the quality of the product.

2 SAMPLE PREPARATION

During the flotation program, two composites (*Comp. 1* and *Comp. 2*) were prepared to –2 mm and from these, two concentrates (with lower levels of silica) were generated. The current testwork was carried out using *Comp. 1*.

3 TESTWORK PROCEDURES

3.1 SAMPLE CHARACTERISATION

Subsamples of each of *Comp. 1 (-2 mm)* and *Comp. 1 (Conc.)* were obtained by riffling and were analysed for Mg, Ca, Fe, Ni, Mn, Cu, Zn, Cr, Al, Si and B and Loss on ignition (LOI) at 1100°C.

Other subsamples were subjected to a sizing analysis. The screen sizes used on the –2 mm material was as follows: 1.0, 0.71, 0.50, 0.355 mm. Because the concentrate was finer, the following screen sizes were used: 212, 150, 106, 75 and 38 µm. The –38 µm product was treated further by cyclosizing to determine the size distribution of the sub-sieve sized material.

3.2 CALCINATION

100 g of magnesite sample were placed in a large silica container (approximately 300 mm long, 140 mm wide and 25 mm deep) and spread evenly to a depth of no more than 5 mm. A muffle furnace, which was fitted with a thermocouple whose tip was located immediately adjacent to the location of the silica container, was preheated such that the thermocouple registered the desired calcination temperature. At this stage, the silica container was positioned inside the furnace for the required calcination period, at the end of which it was removed and allowed to cool. The calcined product was weighed immediately, sealed in a plastic bag and stored in a desiccator to prevent any absorption of moisture.

The activity of the calcine was determined using the citric acid method. This involved preparing a solution of 0.5 g of sodium benzoate, 28 g of citric acid monohydrate and 0.1 g of phenolphthalein in water and diluting to 1 litre. 100 mL of this solution was pipetted into a beaker and 2.00 g of sample was added with stirring. The time taken for the solution to turn pink (in seconds) was the citric acid reactivity.

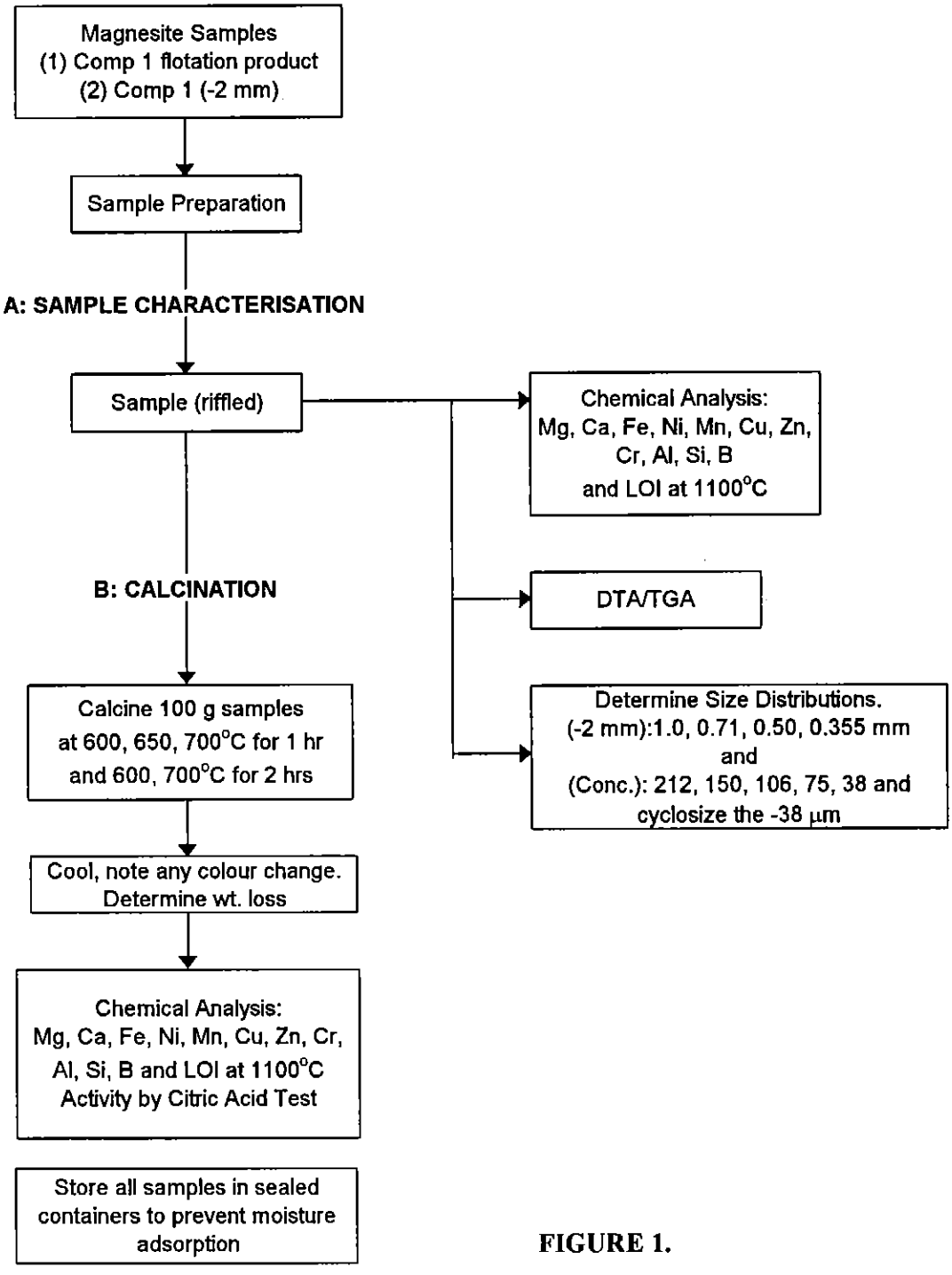

Magnesite Samples
(1) Comp 1 flotation product
(2) Comp 1 (-2 mm)
Sample Preparation
A: SAMPLE CHARACTERISATION
Sample (riffled)
Chemical Analysis:
Mg, Ca, Fe, Ni, Mn, Cu, Zn,
Cr, Al, Si, B
and LOI at 1100°C
DTA/TGA
Determine Size Distributions.
(-2 mm):1.0, 0.71, 0.50, 0.355 mm
and
(Conc.): 212, 150, 106, 75, 38 and
cyclosize the -38 µm
B: CALCINATION
Calcine 100 g samples
at 600, 650, 700°C for 1 hr
and 600, 700°C for 2 hrs
Cool, note any colour change.
Determine wt. loss
Chemical Analysis:
Mg, Ca, Fe, Ni, Mn, Cu, Zn, Cr,
Al, Si, B and LOI at 1100°C
Activity by Citric Acid Test
Store all samples in sealed
containers to prevent moisture
adsorption

FIGURE 1.

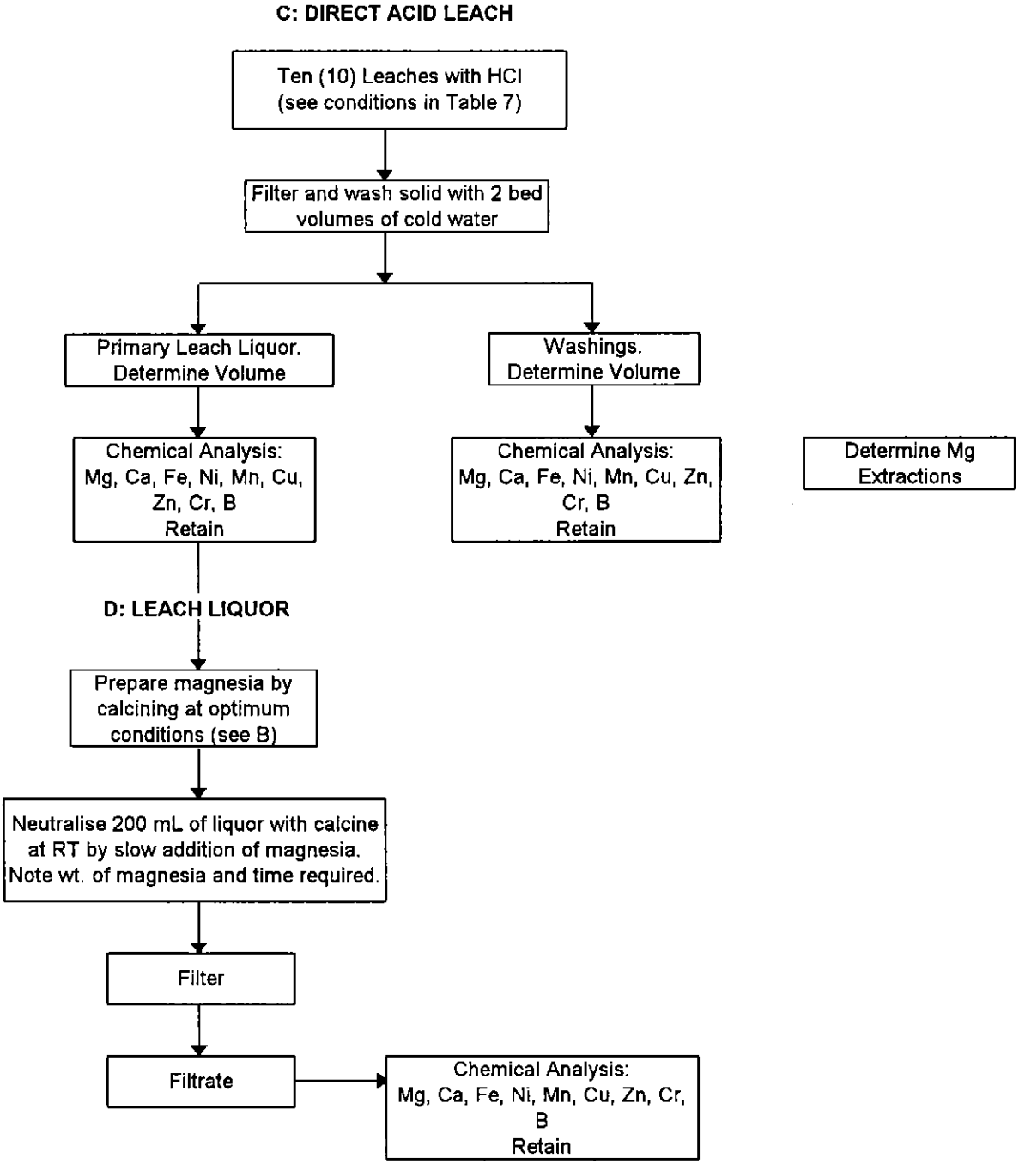

C: DIRECT ACID LEACH
Ten (10) Leaches with HCl
(see conditions in Table 7)
Filter and wash solid with 2 bed volumes of cold water
Primary Leach Liquor.
Determine Volume
Washings.
Determine Volume
Chemical Analysis:
Mg, Ca, Fe, Ni, Mn, Cu, Zn, Cr, B
Retain
Chemical Analysis:
Mg, Ca, Fe, Ni, Mn, Cu, Zn, Cr, B
Retain
Determine Mg Extractions
D: LEACH LIQUOR
Prepare magnesia by calcining at optimum conditions (see B)
Neutralise 200 mL of liquor with calcine at RT by slow addition of magnesia.
Note wt. of magnesia and time required.
Filter
Filtrate
Chemical Analysis:
Mg, Ca, Fe, Ni, Mn, Cu, Zn, Cr, B
Retain

FIGURE 2.

E: EVAPORATION AND CRYSTALLISATION

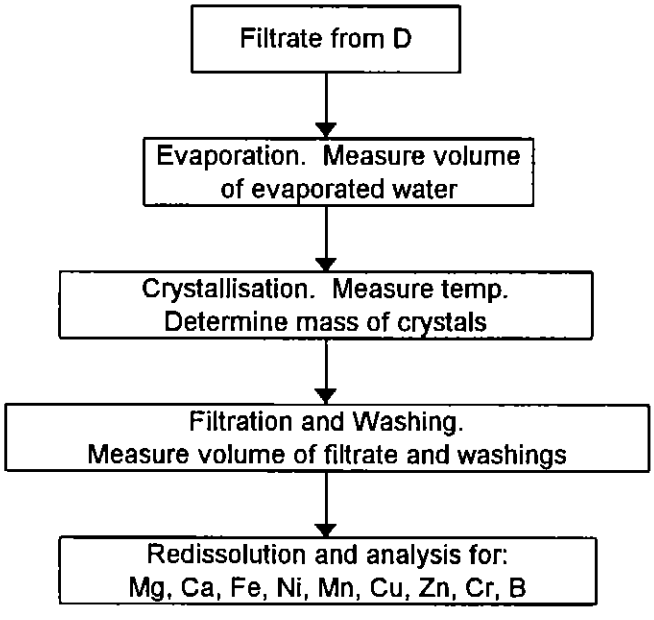


FIGURE 3.

3.3 ACID LEACHING

The leaching vessels employed were glass, equipped with ground glass lids. The lids contained a sufficient number of ports to house a stirrer, a water-cooled condenser and a thermometer. In addition, a stoppered port was used to supply the magnesite sample. The enclosed vessel, fitted with a water-cooled condenser, was utilised to minimise any loss of HCl vapour during leaching. To ensure close control of the leaching temperature, the vessels were immersed in a temperature-controlled water bath.

The required amount of concentrated hydrochloric acid (AR) was added to the vessel and its temperature adjusted to the required value. At this point the mass of magnesite (200 g) was added through the stoppered port taking care to minimise the level of frothing which occurred. Usually a period of approximately two minutes was required for this operation.

At the end of the leach period the leach pulp was cooled and filtered. The filtrate was collected and its volume measured. The remaining solid was washed with approximately two bed volumes of cold, deionised water and the washings collected separately after recording their volume.

The remaining solids were dried and weighed. Both the liquor and washings were analysed for Mg, Ca, Fe, Ni, Cr, Mn, Cu, Zn and B.

Boron analysis by the normal ICP method was limited to a 20 ppm detection limit. However specialised techniques with ICP-AES which included readings in triplicate with frequent zero checks permitted a much lower detection limit of approximately 0.4 mg L^{-1} to be attained.

3.4 LEACH LIQUOR NEUTRALISATION

200 mL of each of the primary leach liquors were treated with stagewise additions of magnesia until a pH of 7 was obtained. The cumulative mass of magnesia was recorded and with each addition, the pH and time was noted.

Immediately after neutralisation, the pulp was filtered and the filtrate retained for magnesium chloride crystallisation.

3.5 CRYSTALLISATION OF MAGNESIUM CHLORIDE

Solubility data for magnesium chloride hexahydrate suggested that there is a significant change with temperature: 367 g per 100 cc in hot water compared with 167 g per 100 cc in cold water (*Handbook of Chemistry and Physics, 58th ed.*). It was intended to recover magnesium chloride crystals by supersaturating the solution through heating until the onset of crystal formation, followed by cooling to promote growth.

A sample of the neutralised, filtered leach liquor was placed in a flask and was gently heated by immersion in a water bath maintained at approximately 60 - 70°C and the concentration of the solution was increased to point of supersaturation. At this point the flask was removed from the water bath and placed in chilled water and left over night. The resulting product was a quantity of acicular magnesium chloride crystals together with a volume of supernatant liquor. The crystals were recovered by filtration, dried and weighed. Later they were dissolved in a known volume of water and the solution was analysed for Mg, Ca, Fe, Ni, Cr, Mn, Cu and Zn to determine the magnesium chloride purity.

4 TESTWORK RESULTS AND DISCUSSION

4.1 ANALYSIS OF SAMPLES

The following (Table 1) summarises the analysis of *Comp. 1 (-2 mm)* and *Comp. 1 (Conc.)*.

It may be seen that the most significant difference between the two is the silica concentration: 1.32% for the –2 mm material and <0.02% for the concentrate.

Table 1. Head Analysis of Magnesite Samples

Sample	LOI, %	Mg, %	Ca, %	Si, %	Fe, %	Mn, ppm
Comp 1 (-2 mm)	50.1	26.47	1.66	1.32	0.56	412
Comp 1 (Flot Conc.)	51.43	27.2	1.68	<0.02	0.48	427

Sample	Ni, ppm	Cu, ppm	Zn, ppm	Cr, ppm	Al, ppm	B, ppm
Comp 1 (-2 mm)	<10	11	25	<10	<100	<20
Comp 1 (Flot Conc.)	<10	<5	32	<10	<100	<20

The sizing distributions of the *Comp. 1 (-2 mm)* and *Comp. 1 (Conc.)* were determined as follows:

Table 2. COMP.1 (-2 mm) SIZE ANALYSIS

Screen Size mm	Mass		Cum % O/S
	g	%	
+1000	91.4	23.4	23.4
-1000 +710	54.9	14.1	37.4
-710 +500	45.9	11.7	49.2
-500 +355	35.1	9.0	58.2
-355	163.4	41.8	100.0
	390.7	100.0	

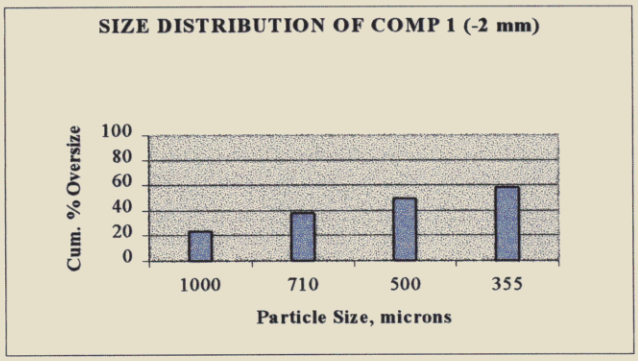
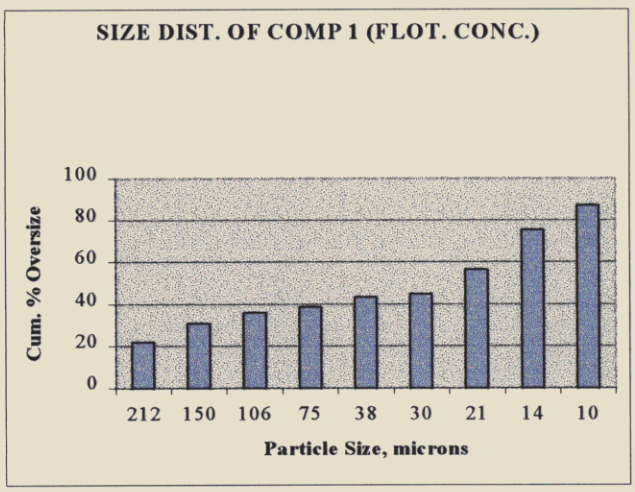


Table 3. COMP 1 (Conc.) SIZE ANALYSIS

Size µm	Mass		Cum % O/S
	g	%	
+212	81.6	22.0	22.0
-212 +150	32.9	8.9	30.8
-150 +106	19.8	5.3	36.1
-106 +75	9.1	2.4	38.6
-75 +38	17	4.6	43.2
-38 +30	5.7	1.5	44.7
-30 +21	44.6	12.0	56.7
-21 +14	69.6	18.7	75.4
-14 +10	43.6	11.7	87.1
-10	47.8	12.9	100.0
	371.7	100.0	



The results show that of the –2 mm material, approximately half of the mass existed as particles which were greater than 0.5 mm in diameter. The flotation concentrate was very much finer in its size distributions with approximately 55% less than 30 µm.

The Differential Thermal Analysis/Thermogravimetric curves for Comp.1 (Conc.) and Comp.1(-2 mm) are provided in Section 6.

The curves, which are similar, demonstrate the typical endothermic decomposition reactions of magnesium carbonate (peak at 690 - 700°C) and calcium carbonate (peak at 770 - 790°C). In both traces an exothermic peak occurred at approximately 800°C but because it was not accompanied by any noticeable weight change, it is possible it corresponded to a recrystallisation or phase change.

4.2 CALCINATION

The results of the calcination tests are detailed in Tables 4 and 5.

TABLE 4. MAGNESITE CALCINATION (1)

Calcine No.	Magnesite Sample	Temperature °C	Time mins.	Mass, g		Mass Loss, %
				Initial	Final	
1	Comp 1 (-2mm)	650	60	306.1 303.0	166.8 164.6	45.5 45.7
2	omp 1 (Flot Conc.	650	60	102.0	54.9	46.2
3	omp 1 (Flot Conc.	650	120	102.5	54.5	46.8
4	omp 1 (Flot Conc.	600	60	109.5 127.0	58.8 69.6	46.3 45.2
5	omp 1 (Flot Conc.	700	60	126.9	67.3	47.0
6	omp 1 (Flot Conc.	700	120	104.7	55.0	47.5

Although most of the tests were carried out on the concentrate, some comments could be made regarding the results of the tests. To gauge the effect of temperature, the results for the calcination of the concentrate for 60 minutes were plotted against temperature. The variables considered were % Mass Loss, % Mg, % LOI and the Activity.

TABLE 5. MAGNESITE CALCINATION (2)

Calcine No.	Magnesite Sample	Calcine Analysis												
		LOI, %	Activity, sec	Mg, %	Ca, %	Fe, %	Al, %	Si, %	Ni, mg kg^{-1}	Mn, mg kg^{-1}	Cu, mg kg^{-1}	Zn, mg kg^{-1}	Cr, mg kg^{-1}	B, mg kg^{-1}
1	Comp 1 (-2mm)	8.61	600 32*	47.92	2.98	0.83	0.35	2.41	22	657	11	47	11	<20
2	Comp 1 (Flot Conc.)	10.15	55	49.76	3.2	0.78	0.31	0.4	32	646	11	54	14	<20
3	Comp 1 (Flot Conc.)	9.05	47	50.24	3.36	0.75	0.28	0.28	39	679	11	54	21	<20
4	Comp 1 (Flot Conc.)	11.18	51	49.49	3.23	0.9	0.42	0.23	35	685	12	54	15	<20
5	Comp 1 (Flot Conc.)	8.9	75	50.23	3.36	0.96	0.45	0.23	39	718	13	65	22	<20
6	Comp 1 (Flot Conc.)	4.54	100	53.38	3.45	1.02	0.47	0.25	46	781	15	58	19	<20

* Value obtained after pulverising the calcined product

FIGURE 4. EFFECT OF TEMPERATURE ON CALCINATION
Comp.1 (Conc.)
Time: 60 mins

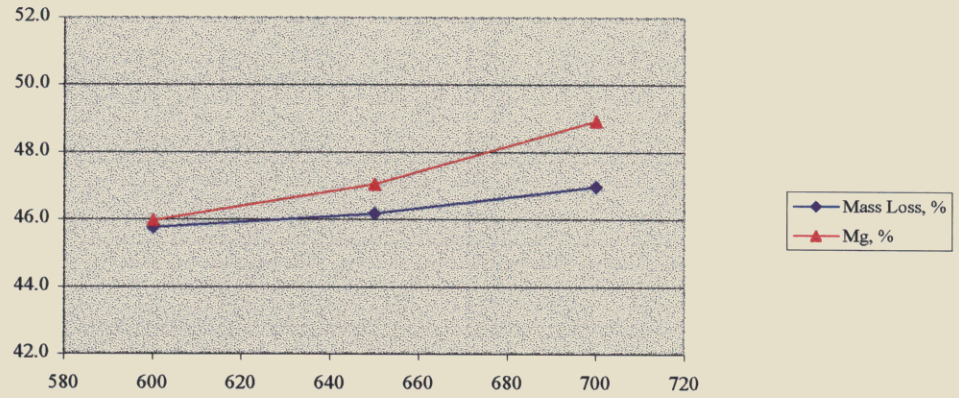
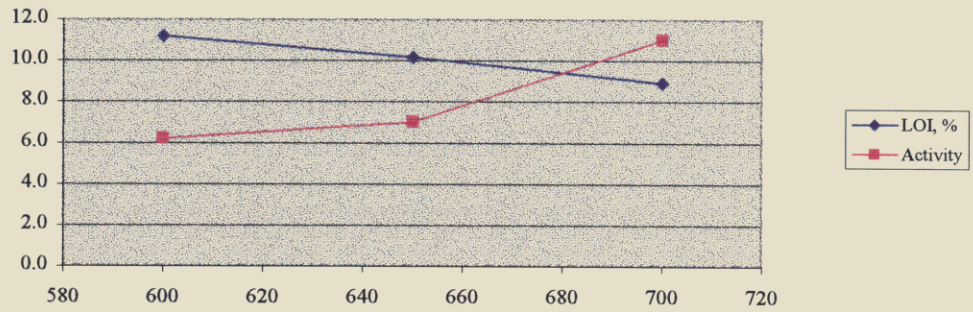


FIGURE 5. EFFECT OF TEMPERATURE ON CALCINATION
Comp.1 (Conc.)
Time: 60 mins



It may be seen from the Figures 4 and 5 that with increasing temperature, from 650 to 750°C, the mass loss and the % Mg decreased. Also, as expected, the LOI decreased with increasing temperature. However the activity of the calcine decreased (an increase in the absolute value of activity (reported in seconds) represents a decrease in the calcine's activity). This effect was also noted with longer times (e.g. 2 hours) at 700°C.

The calcination carried out on the –2 mm material returned an activity of 600 seconds. When this calcine was pulverised and retested, the activity value was improved markedly to only 32 seconds. Generally a value which is less than 100 seconds is regarded of high activity.

It was demonstrated that, as the extent of calcination increased, so did the concentration of magnesium. This pattern was also evident for the impurities, such as Ca, Fe, Al, Ni, Mn, Cu, Zn and Cr.

Although tests at 700°C resulted in higher mass losses and higher % Mg values, the activity was lower than that obtained at 650°C. Optimum conditions selected for the calcination of Comp. 1 (Conc.) were 650°C and 60 minutes.

4.3 ACID LEACHING

The original program, as proposed, was based on leaching the Comp.1 (Concentrate) and consisted of the following tests:

Table 6. Proposed Leaching Program

Leach No.	Temperature °C	Duration hrs	Acid (% Stoich.)	Pulp Density %, (w/v)
1	RT	1	110	30
2	50	1	110	30
3	75	1	110	30
4	50	2	110	30
5	50	2	120	30
6	50	3	110	30
7	50	1	110	40
8	50	1	120	40
9	50	2	120	40
10*	RT	1	110	30

* Acid leach on material prior to flotation

However, on commencement of the testwork, a higher priority was given to the –2 mm material and as a consequence the program was altered. The leach test conditions selected were as follows:

Table 7. Actual Leaching Program.

Leach No.	Sample Comp. 1	Temp. °C	Duration mins	Acid (% Stoich.)	Pulp Density %, (w/v)*
1	Conc.	RT	60	110	35.2
2	Conc.	50	60	110	36.0
4	Conc.	50	120	110	35.2
10	-2 mm	RT	60	110	36.0
11	-2 mm	50	60	110	36.0
12	-2 mm	50	120	110	36.0
13	-2 mm	75	60	110	36.0
14	-2 mm	75	120	110	36.0
15	-2 mm	50	60	120	33.4
16	-2 mm	50	120	120	33.4

The pulp density was determined by the % acid stoich. and the sample composition (see below)

Calculation of the Acid Requirement and Pulp Density Values.

The acid requirements and the pulp densities were calculated as shown in Table 8.

Table 8. Calculation of Acid Requirements.

	Comp. 1 (-2 mm)	Comp. 1 (Conc.)
% MgO	43.9	45.11
% CaO	2.32	2.35
% $MgCO_3$	91.8	94.4
% $CaCO_3$	4.1	4.2
FOR 110% STOICH.		
Mass of HCl/100 g of ore		
For Mg component	87.4	89.8
For Ca component	3.3	3.4
Mass of AR grade HCl, g		
For Mg component	273.0	280.5
For Ca component	10.4	10.5
Vol of AR grade HCl, mL		
For Mg component	235.3	241.8
For Ca component	8.9	9.1
TOTAL ACID REQUIREMENT		
mL of conc. AR HCl	244.3	250.9
PULP DENSITY, w/v %	**36.0**	**35.2**
FOR 120% STOICH		
Mass of HCl/100 g of ore, g		
For Mg component	95.3	97.9
For Ca component	3.6	3.7
Mass of AR grade HCl, g		
For Mg component	297.8	306.0
For Ca component	11.3	11.5
Vol of AR grade HCl, mL		
For Mg component	256.7	263.8
For Ca component	9.7	9.9
TOTAL ACID REQUIREMENT		
mL of conc. AR HCl	266.5	273.7
PULP DENSITY, w/v %	**33.4**	**32.6**

The results of the leach tests are summarised in Tables 9 and 10. Table 9 details the results for the magnesium and calcium. Table 10 is a continuation of Table 9 but provides the analyses of the impurities.

To illustrate the effect of temperature and acid concentration, Figures 6 and 7 were constructed. Both of these figures were produced from data obtained from leaching the sample, Comp. 1 (-2 mm).

TABLE 9. LEACH TEST RESULTS (1)

Leach No.	Magnesite Sample	Temperature °C	Time mins.	Acid Stoich., %	P.D. w/v %	Mass, % Dissolved	Filtrate Vol. mL	Wash Vol. mL	$[Mg]_{Filtrate}$ g L^{-1}	$[Mg]_{Wash}$ g L^{-1}	Recovery, % Mg	$[Ca]_{Filtrate}$ g L^{-1}	$[Ca]_{Wash}$ g L^{-1}	Recovery, % Ca
1	Comp 1 (Flot conc.)	RT	60	110	35.2	21.2	472	177	16.1	5.27	15.7	5.73	1.58	88.8
2	Comp 1 (Flot conc.)	50	60	110	35.2	75.7	495	202	82.2	7.36	77.5	5.61	0.64	86.5
4	Comp 1 (Flot conc.)	50	120	110	35.2	84.6	493	235	88.4	7.23	83.3	5.61	0.61	86.6
10	Comp 1 (-2mm)	RT	60	110	36.0	21.1	455	187	18.0	3.99	16.9	5.78	1.11	85.5
11	Comp 1 (-2mm)	50	60	110	36.0	72.0	470	158	77.4	12.20	72.4	5.44	1.05	82.0
12	Comp 1 (-2mm)	50	120	110	36.0	79.6	475	180	83.0	10.10	77.9	5.41	0.83	81.9
13	Comp 1 (-2mm)	75	60	110	36.0	91.1	485	220	85.9	8.75	82.3	6.08	0.525	92.3
14	Comp 1 (-2mm)	75	120	110	36.0	92.1	482	205	94.0	9.13	86.7	6.23	0.56	92.8
15	Comp 1 (-2mm)	50	60	120	33.4	68.3	530	225	75.1	9.51	77.1	5.10	0.755	85.5
16	Comp 1 (-2mm)	50	120	120	33.4	78.0	532	324	89.0	6.17	90.7	5.20	0.46	86.8

TABLE 10. LEACH TEST RESULTS (2)

Leach No.	Lab	[Fe] Filtrate mg L^{-1}	[Fe] Wash mg L^{-1}	[Mn] Filtrate mg L^{-1}	[Mn] Wash mg L^{-1}	[Ni] Filtrate mg L^{-1}	[Ni] Wash mg L^{-1}	[Zn] Filtrate mg L^{-1}	[Zn] Wash mg L^{-1}	[Cu] Filtrate mg L^{-1}	[Cu] Wash mg L^{-1}	[Cr] Filtrate mg L^{-1}	[Cr] Wash mg L^{-1}	[Al] Filtrate mg L^{-1}	[Al] Wash mg L^{-1}	[Si] Filtrate mg L^{-1}	[Si] Wash mg L^{-1}	[B] Filtrate* mg L^{-1}
1	Analabs	329	128									3.61	1.42	24	15	<5	<5	
	Oretest	452	147	36.2	10.3	21.1	6	3.6	1.4	1.9	0.4	4.80	1.4					3.5
2	Analabs	1090	132									3.92	0.95	34	17	6	<5	
	Oretest	1640	156	95.4	12.3	5.4	1.1	8.1	1.4	1.8	0.3	4.90	0.9					3.6
4	Analabs	1221	127									4.23	0.64	35	17	7	<5	
	Oretest	1850	141	105	11	6	1	9.6	1.5	1.9	0.2	6.60	0.7					3.8
10	Analabs	361	84									0.65	0.32	24	14	<5	<5	
	Oretest	467	95	37	8	4.3	3.2	3.6	1.1	0.3	0.1	0.80	<0.1					3.6
11	Analabs	1083	202									0.93	0.36	33	23	7	<5	
	Oretest	1551	238	95	20	4	1	8.1	2.0	0.5	0.1	0.80	0.3					2.5
12	Analabs	1174	168									0.96	0.32	34	19	7	<5	
	Oretest	1652	186	109	15.1	4	0.7	8.8	1.7	0.3	<0.1	1.00	<0.1					3.5
13	Analabs	1303	108	102	10.3	<0.1	<0.1	10.0	1.1	<0.05	<0.05	1.0	0.1	30	3	7	<5	
	Oretest	1710	142	144	11.6	1.4	0.4	11.1	1.5	0.4	0.1	<0.1	<0.1					3.5
14	Analabs	1354	114	106	10.9	<0.1	<0.1	9.7	1.5	<0.05	<0.05	1.2	0.1	32	3	7	<5	
	Oretest	1730	157	158	12.9	1.1	0.1	11.7	2.0	0.1	0.1	0.1	<0.1					3.6
15	Analabs	926	118	74.8	11.4	<0.1	<0.1	5.3	0.9	<0.05	<0.05	1.0	0.1	26	3	<5	<5	
	Oretest	1280	163	102	13.6	0.7	0.1	7.2	1.4	0.1	0.1	0.1	<0.1					3.2
16	Analabs	1071	95	85.2	8	<0.1	2.2	5.9	2.0	<0.05	0.75	0.8	3.0	28	3	6	<5	
	Oretest	1370	123	112	9.2	0.4	2.8	7.1	2.5	0.2	0.9	<0.1	2.9					3.1

* Boron Analyses determined by Chemistry Centre (WA)

Figure 6 shows that leaching at room temperature for 60 minutes resulted in a low mass loss and magnesium recovery (both approximately 20%) but significantly higher calcium dissolution of 85%. Similar results were obtained on the Comp. 1 (Conc.) sample (see results of Leach Nos. 1 and 2). These results clearly demonstrate that calcium leached much more readily from the samples than did magnesium. The results for the longer leaching time of 120 minutes did not yield any significant differences between the mass loss and the recoveries of calcium and magnesium.

This difference may be utilised to control calcium. For example, a low temperature leach for a relatively short time may be employed as a pretreatment to selectively remove a large proportion of calcium. Further testwork would be needed to investigate this option.

Figure 7 illustrates the difference of acid concentration (110% and 120% stoichiometry). The first part of the diagram was prepared from 60 minute data while the second part was constructed from the 120 minute results. The difference between the two leach times may be gauged readily – the mass loss and magnesium recoveries were higher at the longer leach times. However there was little difference between the calcium dissolution plots at 60 and 120 minutes. The higher acid strength resulted in a small increase in magnesium recovery for both leach periods.

The impurity concentration in the leach liquor generally increased with the intensity of the leach conditions. For example, iron, manganese and zinc showed a steady increase with increasing temperature (compare the figures in tests 10, 11 and 13).

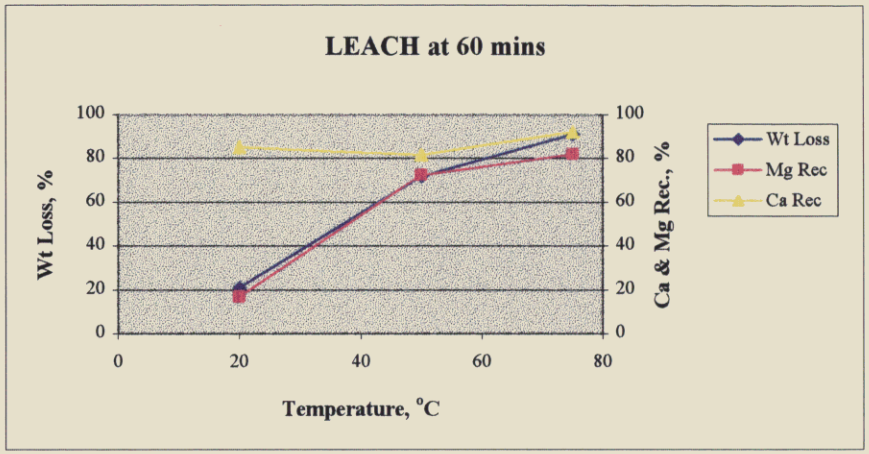
The higher concentration of silica in the –2 mm material, compared with that in the flotation concentrate, was of some concern. It was suspected that higher levels of soluble silica may have resulted during leaching of the –2 mm sample. However in all the leaches, the soluble Si levels were approximately the same and never exceeded 7 mg L^{-1} in the primary leach liquor.

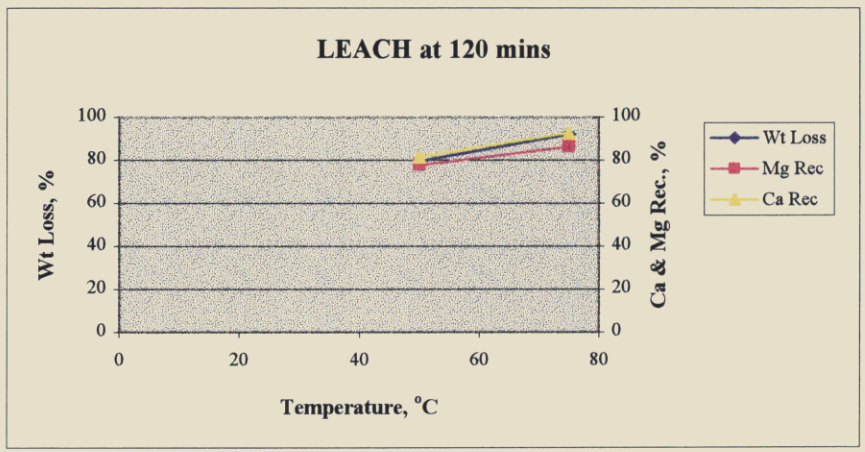
Boron was another element whose concentration needed to be maintained at low levels. It was essential that levels less than approximately 7 mg L^{-1} (personal communication) be maintained in the leach liquors to ensure that the resulting magnesium chloride met the required specification. The detection limit of boron in the solid magnesite samples was 20 ppm and as shown in the analyses, all the results were less than this value. Specialised techniques with the ICP enabled lower detection limits to be obtained for the solution analysis. All of the leach liquors were shown to have values which ranged from 2.5 to 3.8 mg L^{-1}.

Sample:	Comp 1 (-2 mm)
Acid Stoich.:	110%
Pulp Density, % (w/v):	36

FIGURE 6. EFFECT OF TEMPERATURE

Leach No.	Time mins	Temperature °C	Mass, % Dissolved	Recovery, % Mg	Recovery, % Ca
10	60	20	21.1	16.9	85.5
11	60	50	72.0	72.4	82.0
13	60	75	91.1	82.3	92.3
12	120	50	79.6	77.9	81.9
14	120	75	92.1	86.7	92.8

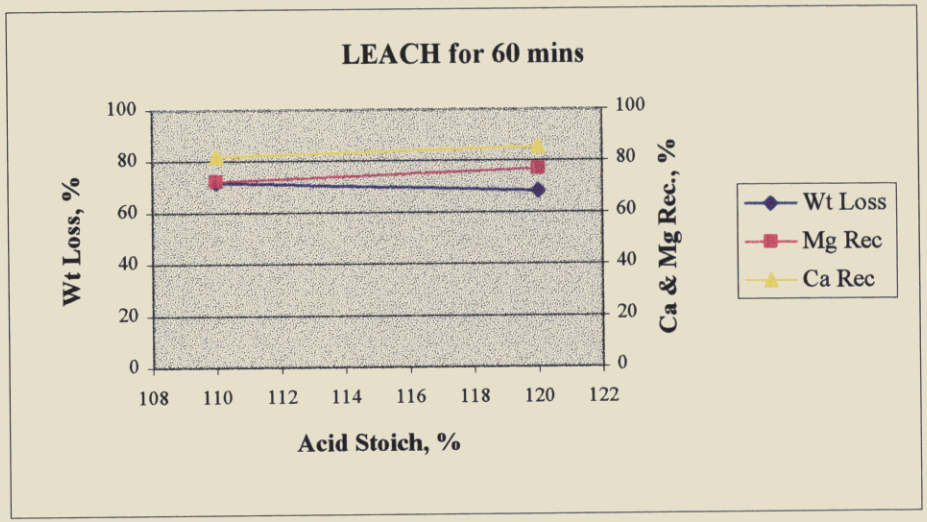


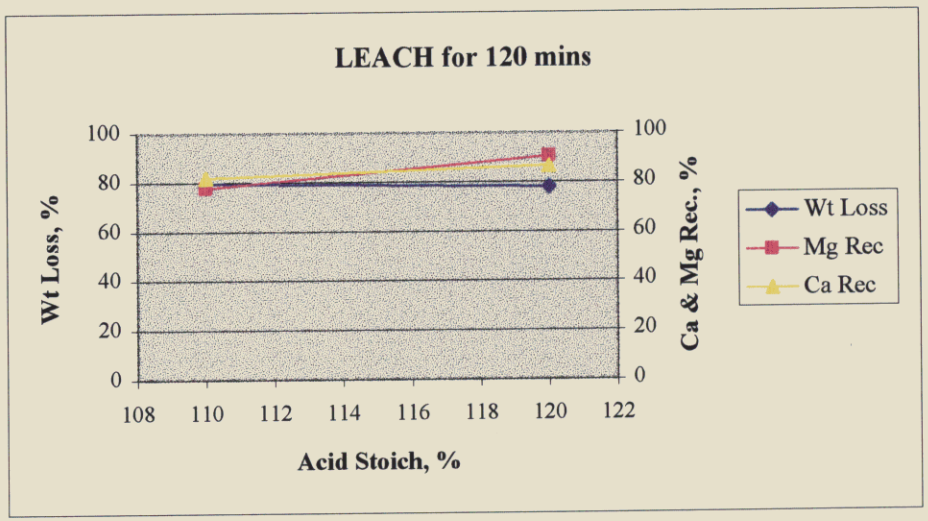


Sample: Comp 1 (-2 mm)
Temperature, °C: 50

FIGURE 7. EFFECT OF ACID CONCENTRATION

Leach No.	Time mins	Acid Stoich. %	Mass, % Dissolved	Recovery, % Mg	Recovery, % Ca
11	60	110	72.0	72.4	82.0
15	60	120	68.3	77.1	85.5
12	120	110	79.6	77.9	81.9
16	120	120	78	90.7	86.8





From the limited number of tests conducted, it was shown that a maximum magnesium recovery of 90.7% was possible using leaching conditions of: 50°C, 120 minutes and an acid stoichiometry of 120%. The corresponding calcium value was 86.8%. A lower acid stoichiometry of 110%, but with a temperature of 75°C and a leach period of 120 minutes, resulted in a magnesium recovery of 86.7% and a corresponding calcium value of 92.8%.

Only three leaches were carried out on the flotation concentrate and a maximum magnesium recovery of 83.3% was obtained (86.6% calcium was extracted during this leach). The conditions were less intense than those quoted above - 50°C, 110% acid stoichiometry and for 120 minutes leach duration. It is possible that the finer particle size of the flotation concentrate would have had an effect on leaching rates. Further tests are suggested to assess particle size.

4.4 LEACH LIQUOR NEUTRALISATION

Leaching of the magnesite was carried out using concentrated HCl. After filtration to remove any remaining solid, the primary leach liquor was acidic and needed to be neutralised. Calcined magnesite was used for this purpose. The calcine selected for this application was the –2 mm product after the following treatment:

Temperature: 650°C
Time: 60 minutes.

It was noted that after calcination, the colour changed from a near white to that of a slight pink-brown. This suggests that the iron and manganese had oxidised during heating to the Fe(III) and to Mn(IV) conditions respectively.

The detailed results of the neutralisation tests are given in Figures 8 – 17 in Section 6 and the analyses of the neutralised leach liquors are given in Table 11.

The conditions outlined in the original proposal included neutralisation to a pH of 7. From the plots in Figures in 8 – 17 it may be seen that neutralisation occurred at a pH lower than this value. The long tail on the end of the curves represents attempts to achieve a pH of 7.

From the comments regarding the neutralisation tests (in Table 12) it was evident that iron precipitated predominantly as ferrous hydroxide (characterised by the typical dark blue precipitate). Because of the similarities between the Fe(II) and Mg(II) ions it is understood that iron exists in the magnesite lattice as Fe(II). Therefore it would be expected that on leaching magnesite, iron would be solubilised in the Fe(II) form.

However in some cases during neutralisation, a yellow/brown precipitate was observed and this suggested that the iron precipitate was present as Fe(III). Because Fe(III) precipitates from solution at a pH of approximately 2, evidence of its precipitation could be obtained from the deflection (at a pH of 2) in the neutralisation curves (e.g. Figures 11, 12 and 13). In these cases oxidation of iron had occurred during processing of the solutions. Perhaps further tests could be undertaken to determine whether oxidation of iron by bubbling air (or oxygen) through the solution would assist in removing iron to a greater extent.

During neutralisation of leach solutions with calcine, it was understood that a reaction between MgO and $MgCl_2$ was possible and that immediate filtration after neutralisation was necessary to avoid such a reaction. The formula for such a compound, known as Sorel cement, has been expressed as $MgCl_2.3Mg(OH)_2.8H_2O$ (Heslop R. and P Robinson, *Inorganic Chemistry*. Elsevier Pub. Co, 1963). Although procedures were adopted to filter immediately, it was noted that some white precipitate formed (unlike magnesium chloride crystals) while the solutions were left to stand overnight. It was also found that with some of the solutions, during the next stage of crystallisation, instead of magnesium chloride crystals forming, the solution transformed into a gelatinous, white mass. Both these effects confirmed the presence of an excess of MgO. The excess calcine added during neutralisation had solubilised and remained in solution during filtration. However, when left to stand (or when heated during crystallisation) precipitation as $Mg(OH)_2$ or as Sorel cement occurred.

The analyses of the neutralised leach solutions, in Table 11, indicate that the solutions contain approximately 100 – 115 g L^{-1} Mg and 7 – 13 g L^{-1} Ca. The other major impurities were iron (10 – 60 mg L^{-1}) and manganese (60 – 160 mg L^{-1}). Some soluble aluminium (approximately 2 – 3 mg L^{-1}) was also present. It was interesting to note that the low iron level (12 mg L^{-1} in Test No 10 (Figure 11)) coincided with the conditions in which precipitation of iron occurred mostly as Fe(III). This suggests that soluble iron levels could be reduced by oxidising to the Fe(III) condition.

4.5 MAGNESIUM CHLORIDE CRYSTALLISATION

In order to neutralise the excess MgO, concentrated HCl was added dropwise with stirring until the precipitate disappeared. At this point the pH was noted.

TABLE 11. ANALYSIS OF NEUTRALISED LEACH LIQUORS

Test No	Leach Soln. Vol., mL	Total Calcine Addition, g	Final pH	[Mg] g L^{-1}	[Ca] g L^{-1}	[Fe] mg L^{-1}	[Mn] mg L^{-1}	[Ni] mg L^{-1}	[Zn] mg L^{-1}	[Cu] mg L^{-1}	[Cr] mg L^{-1}	[Al] mg L^{-1}
1	200	57.28	6.58	109	13.60	40	70	2.3	0.7	0.8	0.1	2.6
2	200	50.27	6.40	114	8.07	30	158	<0.1	1.2	1.0	1.0	1.6
4	200	47.38	6.47	113	7.44	35	159	<0.1	1.7	0.9	1.7	2.0
10	200	43.66	7.00	100	13.00	12	104	0.3	1.6	0.3	<0.1	2.8
11	200	33.94	6.92	108	7.74	44	112	<0.1	1.0	0.1	<0.1	2.0
12	200	65.42	6.47	115.4	7.30	32	72	<0.1	0.4	<0.1	0.1	3.6
13	200	61.36	6.63	108.2	6.60	36	62	<0.1	0.4	<0.1	<0.1	3.8
14	200	40.46	6.53	108	7.84	45	134	<0.1	0.7	0.2	0.2	2.0
15	200	42.46	6.59	104	6.36	62	142	<0.1	1.5	0.2	0.2	2.5
16	200	49.76	6.44	110.8	7.35	47	162	<0.1	1.0	0.1	0.2	3.0

TABLE 12. LEACH LIQUOR NEUTRALISATION COMMENTS

Test No	Liquor Description	Soln. Vol. mL	Total Calcine Addition, g	Final pH	Comment
1	Liquor from Conc. Leached at RT for 1 hr (110% stoich.)	200	57.28	6.58	Fe^{3+} (as brown-orange foam on top) at pH of approx. 4
2	Liquor from Conc. Leached at 50°C for 1 hr (110% stoich.)	200	50.27	6.4	Brown colour formed at pH = 4.68; Dark blue at pH of 5.54
4	Liquor from Conc. Leached at 50°C for 2 hr (110% stoich.)	200	47.38	6.47	Green/Grey colour at pH = 1.86; Dark blue at pH of 6.08
10	Liquor from -2 mm Leached at RT for 1 hr (110% stoich.)	200	43.66	7	Brown-red colour at end, suggesting Fe^{3+}
11	Liquor from -2 mm Leached at 50°C for 1 hr (110% stoich.)	200	33.94	6.92	Colour grey/green at end
12	Liquor from -2 mm Leached at 50°C for 2 hr (110% stoich.)	200	65.42	6.47	Dark green colour at pH of 1.54; Dark blue/green colour at end
13	Liquor from -2 mm Leached at 75°C for 1 hr (110% stoich.)	200	61.36	6.63	Dark blue colour at pH = 5.28; Dark blue/green colour at end
14	Liquor from -2 mm Leached at 75°C for 2 hr (110% stoich.)	200	40.46	6.53	Blue colour at pH = 5.2
15	Liquor from -2 mm Leached at 50°C for 1 hr (120% stoich.)	200	42.46	6.59	Golden yellow at pH = -1.57; Dark green/grey at pH = 5.5
16	Liquor from -2 mm Leached at 50°C for 2 hr (120% stoich.)	200	49.76	6.44	Golden colour at pH = -1.22; Brown colour at pH = 1.4: Dark green/grey at pH of 4.97

After crystallisation, the mass of $MgCl_2.6H_2O$ crystals was determined and after dissolving in a known volume of water, the solution was analysed.

The results of these analyses are summarised in Table 13.

This table provides the values of the volume of neutralised leach liquor and the mass of liquor prior to crystallisation for each test. The differences between the two varied from test to test. The reason for this arose from the need, in some cases, to supply some water to the solution to ensure that a sufficient volume of solution was present above the crystals after their formation. In each test the solution was heated to the water bath temperature (70°C) and, just before crystallisation, the beaker containing the solution was transferred to an ice/water mixture (0°C).

In order to determine the improvement in the reduction of impurity levels, each of the neutralised leach liquor concentrations of Mg, Ca, Mn and Fe was ratioed to the Mg concentration. The same was carried out for the solutions formed from the magnesium chloride crystals. A comparison between these values for each of the elements provides an indication of the upgrading achieved during crystallisation. Table 14 sets out these values. In general, it may be seen that there was a ten-fold reduction in the level of calcium, relative to that of magnesium, during crystallisation. In some cases the reduction in manganese and iron was higher.

An example of a solution which were known to fulfil specification requirements were supplied by the client. These were as follows:

	$MgCl_2$	NaCl	KCl	Ca	SO_4^{2-}	Cr	Fe	Mn	Ni	B
% (w/v)	32	0.06	0.3	0.1	0.03	-	-	-	-	-
ppm	-	-	-	-	-	0.3	1	1	1	2

A comparison of some of these impurity levels with those obtained from the crystal solutions is given in Table 15. These values show that almost all the calcium concentrations were less than those provided in the above table. The iron and manganese levels were slightly higher.

It would be possible to remove calcium to a greater extent by treating the solution with sulphuric acid and precipitating as calcium sulphate. Further tests would be necessary to evaluate such a reaction.

TABLE 13. MAGNESIUM CHLORIDE CRYSTALLISATION AND THEIR ANALYSES

Test No.	Vol. of Neutralised Leach Liquor	Addition of HCl, mL*	Final pH	Mass of Liquor prior to Crystallisation, g	Mass of Crystals g	Make-up Solution Vol, mL	Analysis, ppm+								
							Mg	Ca	Mn	Fe	Ni	Cu	Zn	Cr	Al
1	100	6.5	4.3	105.3	9.7	150	6150	85 74	1 0.4	<0.1	0.1	<0.1	0.2	<0.1	<1
2	100	9.2	4.7	112.77	2.87	150	2850	20 20	0.7 0.3	<0.1	0.1	<0.1	0.2	<0.1	<1
4	100	9	4.9	107.33	5.33	150	4070	32 30	0.9 0.6	<0.1	0.1	<0.1	0.2	<0.1	<1
10	100	2	5.1	70.98	3.08	50	7700	13.7	<0.1	0.4	<0.1	<0.1	0.1	<0.1	<1
11	70	2.8	5.4	63.66	0.46	50	1130	2.4	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<1
12	50	6.5	4.7	63.72	0.12	50	285	1.7	<0.1	0.2	<0.1	<0.1	<0.1	<0.1	<1
13	70	7.3	4.7	75.55	5.15	150	4150	19 18	0.6 <0.1	<0.1	0.1	<0.1	0.2	<0.1	<1
14	100	6.8	5.2	92.05	5.95	150	4700	31 29	1.1 0.5	<0.1	0.1	<0.1	0.2	<0.1	<1
15	100	5.5	4.3	91.94	13.24	150	10000	74 63	2 0.8	<0.1	0.1	<0.1	0.2	<0.1	<1
16	100	9.2	4.9	109.92	14.52	150	12000	73 62	2.1 1.1	<0.1	0.1	<0.1	0.2	<0.1	<1

* Acid required to neutralise precipitated $Mg(OH)_2$, arising from excess of calcine added during neutralisation

+ Second set of values are those from Analabs

TABLE 14. ELEMENTAL REJECTION DURING CRYSTALLISATION

Test No.		Mg	Ca	Mn	Fe
1	Neutralised Leach Liquor, mg L^{-1}	109000	13600	40	70
	Relative to [Mg]	1	0.125	0.00037	0.00064
	Crystal Solution, mg L^{-1}	6150	85	1	0.1
	Relative to [Mg]	1	0.014	0.00000016	0.00000002
	% Rejection	-	88.9	100.0	100.0
2	Neutralised Leach Liquor, mg L^{-1}	114000	8070	158	30
	Relative to [Mg]	1	0.071	0.00139	0.00026
	Crystal Solution, mg L^{-1}	2850	20	0.7	0.1
	Relative to [Mg]	1	0.007	0.000246	0.000035
	% Rejection	-	90.1	82.3	86.7
4	Neutralised Leach Liquor, mg L^{-1}	113000	7440	159	35
	Relative to [Mg]	1	0.066	0.00141	0.00031
	Crystal Solution, mg L^{-1}	4070	32	0.9	0.1
	Relative to [Mg]	1	0.008	0.000221	0.000025
	% Rejection	-	88.1	84.3	92.1
10	Neutralised Leach Liquor, mg L^{-1}	100000	13000	104	12
	Relative to [Mg]	1	0.130	0.00104	0.00012
	Crystal Solution, mg L^{-1}	7700	13.7	0.1	0.4
	Relative to [Mg]	1	0.002	0.000013	0.000052
	% Rejection	-	98.6	98.8	56.7
11	Neutralised Leach Liquor, mg L^{-1}	108000	7740	112	44
	Relative to [Mg]	1	0.072	0.00104	0.00041
	Crystal Solution, mg L^{-1}	1130	2.4	0.1	0.1
	Relative to [Mg]	1	0.002	0.000088	0.000088
	% Rejection	-	97.0	91.5	78.3
12	Neutralised Leach Liquor, mg L^{-1}	115400	7300	72	32
	Relative to [Mg]	1	0.063	0.00062	0.00028
	Crystal Solution, mg L^{-1}	285	1.7	0.1	0.2
	Relative to [Mg]	1	0.006	0.000351	0.000702
	% Rejection	-	90.6	43.8	-153.1
13	Neutralised Leach Liquor, mg L^{-1}	108200	6600	62	36
	Relative to [Mg]	1	0.061	0.00057	0.00033
	Crystal Solution, mg L^{-1}	4150	19	0.6	0.1
	Relative to [Mg]	1	0.005	0.000145	0.000024
	% Rejection	-	92.5	74.8	92.8
14	Neutralised Leach Liquor, mg L^{-1}	108000	7840	134	45
	Relative to [Mg]	1	0.073	0.00124	0.00042
	Crystal Solution, mg L^{-1}	4700	31	1.1	0.1
	Relative to [Mg]	1	0.007	0.000234	0.000021
	% Rejection	-	90.9	81.1	94.9
15	Neutralised Leach Liquor, mg L^{-1}	104000	6360	142	62
	Relative to [Mg]	1	0.061	0.00137	0.00060
	Crystal Solution, mg L^{-1}	10000	74	2	0.1
	Relative to [Mg]	1	0.007	0.000200	0.000010
	% Rejection	-	87.9	85.4	98.3
16	Neutralised Leach Liquor, mg L^{-1}	110800	7350	162	47
	Relative to [Mg]	1	0.066	0.00146	0.00042
	Crystal Solution, mg L^{-1}	12000	73	2.1	0.1
	Relative to [Mg]	1	0.006	0.000175	0.000008
	% Rejection	-	90.8	88.0	98.0

TABLE 15. A COMPARISON OF THE IMPURITY LEVELS IN THE CRYSTAL SOLUTIONS WITH A SPECIFICATION PROVIDED

	$MgCl_2$	Mg	NaCl	KCl	Ca^{2+}	SO_4^{2-}	Cr	Fe	Mn	Ni	B
% (w/v)*	32	8.17	0.06	0.3	0.1	0.03					
mg L^{-1}*		81700	-	-	1000	300	0.3	1	1	1	2
Expressed as a ratio rel. to Mg		1			0.0122399	0.003671971	0.0000037	0.0000122	0.0000122	0.0000122	0.0000245
Test No											
1		1			0.014			0.00000002	0.00000016		
2		1			0.007			0.000035	0.000246		
4		1			0.008			0.000025	0.000221		
10		1			0.002			0.000052	0.000013		
11		1			0.002			0.000088	0.000088		
12		1			0.006			0.000702	0.000351		
13		1			0.005			0.000024	0.000145		
14		1			0.007			0.000021	0.000234		
15		1			0.007			0.000010	0.000200		
16		1			0.006			0.000008	0.000175		

* Data provided by client

The small mass of crystals recorded for test numbers 11 and 12 arose after the production of a much larger quantity of crystals. The neutralised leach solutions from these tests were among the first to be crystallised and the crystals from both tests had been collected, filtered and had been left to air dry on the filter paper before weighing. (The choice to air dry was made based on the concern that magnesium chloride was known to dissociate into magnesium oxide on heating.) However the fact that $MgCl_2.6H_2O$ was deliquescent had been overlooked and when next inspected for weighing, the crystals had absorbed sufficient moisture from the atmosphere to form a solution. What solution could be recovered was dried at a low temperature and the crystals recovered and their weight was recorded in Table13.

Subsequent treatment of magnesium chloride crystals involved drying in a low temperature oven (approximately 60 - 70°C) before weighing.

It is necessary to supersaturate the solution with respect to magnesium chloride before crystallisation. Lowering of the temperature will result in a decrease in its solubility and magnesium chloride will precipitate to adjust to the new condition. However in order to utilise the crystallisation process as a means of purification, a suitable volume of solution needs to be maintained above the crystals to act as a "reservoir" for the impurities. Therefore the reduction in the water content of the solution before crystallisation is critical. Too much water removal would result in crystallisation of the total mass and hence would eliminate the chance for purification. Too little water removal would cause little or no crystal formation. The optimum would be decided on the analysis of the solution and more extensive knowledge of the solubility behaviour of magnesium chloride hexahydrate (particularly as a function of temperature).

The primary purpose of these tests was to establish the relative purity of the magnesium choride product. Any calculated yields from these data would only be approximate and therefore would not be indicative of optimum values.

5 CONCLUSIONS

1. The head analyses of the Comp.1 (Conc.) and Comp.1 (-2 mm) revealed that the major difference was the silicon content (1.32% in the –2 mm material versus <0.02% in the Comp.1 (Conc.)).

2. Sizing analyses showed that approximately half of the mass of Comp. 1 (-2 mm) material existed as particles which were greater than 0.5 mm in diameter. Comp. 1 (Conc.) was much finer with approximately 55% less than 30 μm.

3. Calcination testing on Comp. 1 (Conc.) was carried out at temperatures of 600, 650 and 700 °C. It was shown that for a calcination period of 60 minutes, the mass loss and the % Mg increased with increasing temperature. LOI tests carried out on the calcined product also demonstrated decreasing values with increasing temperatures. However the activity showed a decrease. Mass losses which ranged from 45.2 to 47.5% were recorded. Calcination conditions of 650°C and 60 minutes were selected as optimum for the Comp. 1 (Conc.) sample.

4. Calcination of the –2 mm material at 650°C and for 60 minutes, returned an activity of 600 seconds. After pulverising the calcine, the activity had improved to 32 seconds. The sample sustained a mass loss of 45.6% during calcination.

5. Ten leaches were carried out – seven on the Comp.1 (-2 mm) and three on the Comp.1 (Conc.). A maximum magnesium recovery of 90.7% was obtained at 50°C , 120 minutes and an acid stoichiometry of 120%. A corresponding calcium recovery of 86.8% was noted during this leach. Generally, there was a correlation between the mass loss and the % Mg dissolution.

6. The tests indicated that calcium was leached readily from the sample. At room temperature, calcium recoveries as high as 85% were obtained from the –2 mm feed after 60 minutes but the magnesium dissolution value under the same conditions was only 17%. This result suggested a possible means of selectively removing a large portion of the calcium by employing a preleach.

7. The higher silica content of Comp. 1 (-2 mm) did not appear to result in higher levels of soluble silica in the leach liquor. For all the tests the concentrations were relatively low with values not exceeding 7 mg L^{-1}.

8. Boron concentrations in the solid magnesite samples were recorded at <20 ppm (the detection limit for this element in a solid). However specialised techniques using ICP-AES resulted in boron measurements which ranged from 3.1 to 3.8 mg L^{-1}.

9. The leach liquors were neutralised with calcine generated from the –2 mm material treated at 650°C for 60 minutes. The experimental curves showed that neutralisation occurred at a pH of approximately 6. Any additional calcine contributed to an increase in the level of dissolved MgO and a possible precipitation as $MgCl_2.3Mg(OH)_2.8H_2O$ (Sorel cement).

10. During neutralisation, iron precipitated mainly as Fe(II) hydroxide. However in some cases a yellow/brown precipitate formed confirming the oxidation of iron to the Fe(III) condition. Evidence of this reaction was noted in a plateau, occurring at a pH of approximately 2 in some of the neutralisation curves.

11. Analysis of the neutralised leach liquors indicated the following levels: 100 – 115 g L^{-1} Mg and 7 – 13 mg L^{-1} Ca. The major impurities present were iron (10 – 60 mg L^{-1}), manganese (60 – 160 mg L^{-1}) and aluminium (2 – 3 mg L^{-1}).

12. The lowest level of iron in the neutralised leach solutions (12 mg L^{-1}) corresponded to the test in which precipitation of iron (as Fe(III)) was the most evident. This suggested that oxidation of the iron during neutralisation would contribute to an elimination a larger proportion of the soluble iron.

13. Conditions required for the crystallisation of magnesium chloride were very much dependent on the change in solubility with temperature. The crystal yield and purity would be determined initially by the solution concentration and secondly by the change in temperature during crystallisation. More detailed knowledge of the variation of magnesium chloride solubility with temperature would assist with the choice of conditions.

14. The crystallisation process resulted in a purification of the magnesium chloride with an approximate ten-fold decrease in the calcium concentration (relative to that of magnesium). In some of the tests, the reduction in the manganese and iron concentrations was higher.

15. Because of the deliquescent nature of the magnesium chloride hexahydrate the crystals needed to be dried at an elevated temperature. However this temperature should be low enough to prevent any decomposition.

Appx. 1

475041

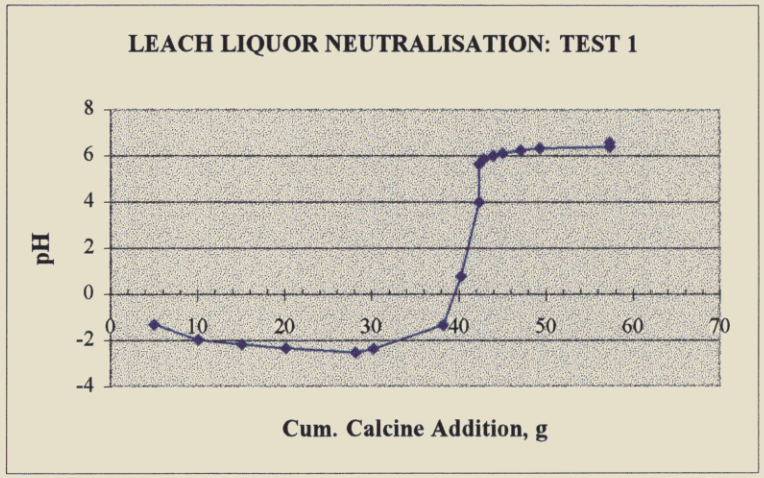
6 DETAILED TEST RESULTS

Neutralisation Test Results

Figure 8: Neutralisation Test

Test No: 1
Liquor from Conc. Leached at RT for 1 hr (110% stoich.)
Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	5.03	5.03	-1.31
2	5.01	10.04	-1.96
4	5.02	15.06	-2.18
6	5.03	20.09	-2.32
8	8.01	28.1	-2.54
10	2.08	30.18	-2.36
12	8.01	38.19	-1.33
14	2.06	40.25	0.78
16	2.05	42.3	4
18	0	42.3	5.63
20	0.55	42.85	5.86
22	1.07	43.92	6.01
24	1.07	44.99	6.12
26	2.07	47.06	6.24
28	2.21	49.27	6.32
30	8.01	57.28	6.38
42	0	57.28	6.58



Corrected Calcine Addition: 44 g (Adjust pH to 6)

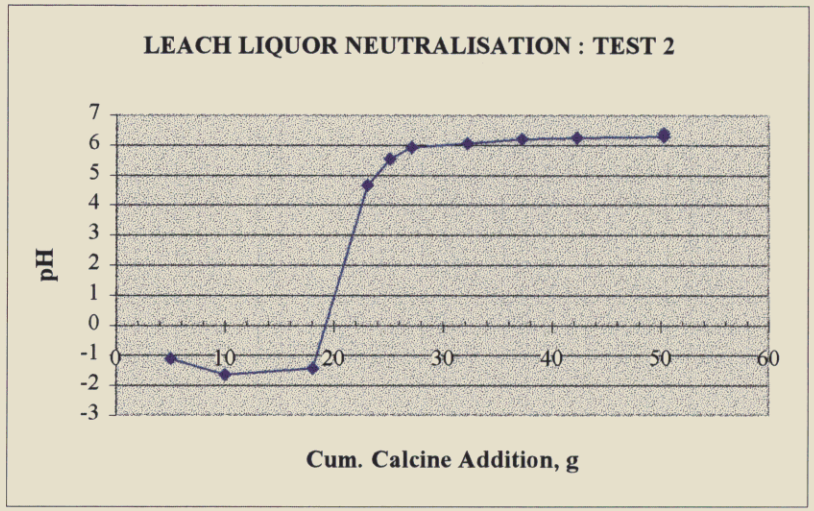
Figure 9: Neutralisation Test

Test No: 2

Liquor from Conc. Leached at 50°C for 1 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	5	5	-1.1
2	5.01	10.01	-1.64
4	8.01	18.02	-1.42
6	5.03	23.05	4.68
8	2.02	25.07	5.54
10	2.05	27.12	5.93
12	5.06	32.18	6.07
14	5.03	37.21	6.2
16	5.05	42.26	6.25
18	8.01	50.27	6.3
27	0	50.27	6.4



Corrected Calcine Addition: 27 g (Adjust pH to pH of 6)

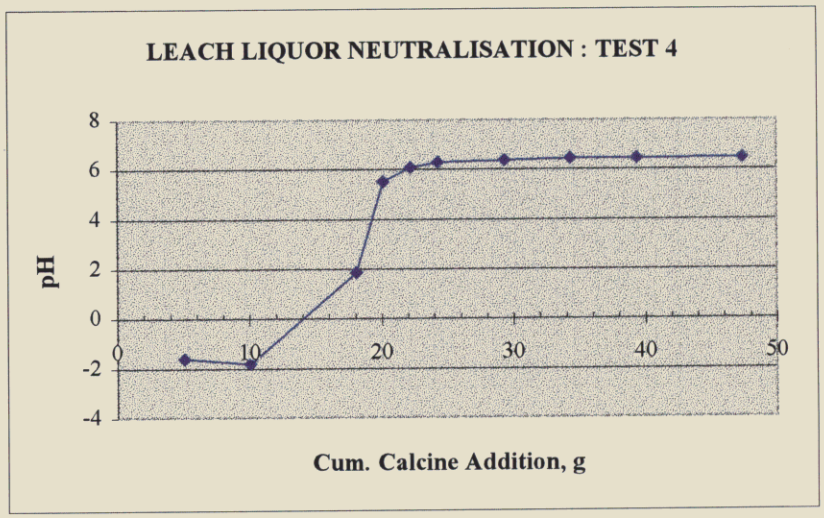
Figure 10: Neutralisation Test

Test No: 4

Liquor from Conc. Leached at 50°C for 2 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	5.03	5.03	-1.6
2	5.02	10.05	-1.79
4	8.01	18.06	1.86
6	2.06	20.12	5.53
8	2.08	22.2	6.08
10	2.06	24.26	6.29
12	5.03	29.29	6.38
14	5.03	34.32	6.44
16	5.03	39.35	6.45
18	8.03	47.38	6.47
27	0	47.38	6.47

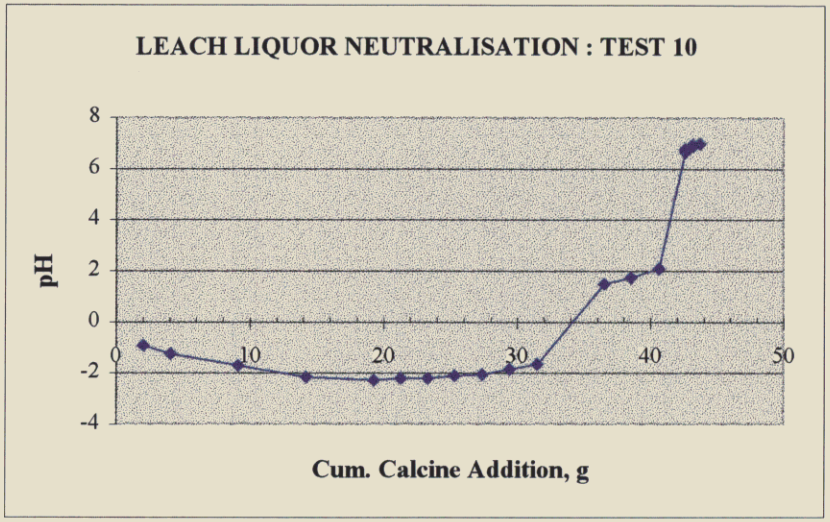


Corrected Calcine Addition: 24 g (Adjust pH to 6.3)

Figure 11: Neutralisation Test

Test No: 10
Liquor from -2 mm Leached at RT for 1 hr (110% stoich.)
Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	2.01	2.01	-0.9
2	2.06	4.07	-1.24
4	5.04	9.11	-1.7
8	5.06	14.17	-2.14
12	5.06	19.23	-2.28
14	2.02	21.25	-2.2
18	2.01	23.26	-2.2
23	2.05	25.31	-2.1
24	2.05	27.36	-2.05
25	2.05	29.41	-1.85
28	2.07	31.48	-1.64
30	5.01	36.49	1.5
35	2.03	38.52	1.76
37	2.07	40.59	2.09
40	1.99	42.58	6.68
43	0	42.58	6.8
46	0.55	43.13	6.9
49	0	43.13	6.96
50	0.53	43.66	7



Corrected Calcine Addition: 43 g (Adjust pH to 7)

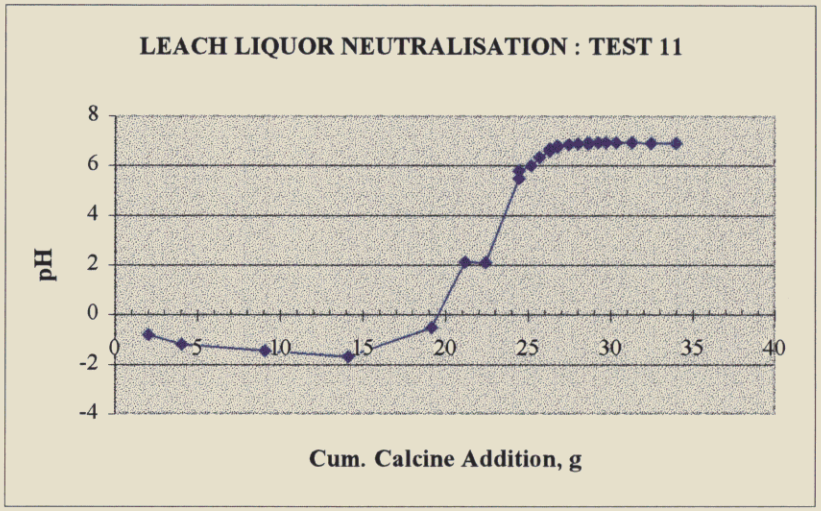
Figure 12: Neutralisation Test

Test No: 11

Liquor from -2 mm Leached at 50°C for 1 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	2.02	2.02	-0.8
2	2.02	4.04	-1.18
4	5.02	9.06	-1.43
8	5.05	14.11	-1.68
12	5.05	19.16	-0.5
14	2	21.16	2.12
18	1.23	22.39	2.11
23	2.04	24.43	5.5
25	0	24.43	5.8
27	0.73	25.16	6.02
29	0.5	25.66	6.35
33	0.62	26.28	6.6
37	0	26.28	6.68
39	0.48	26.76	6.81
42	0	26.76	6.75
45	0.7	27.46	6.85
49	0.52	27.98	6.89
53	0.58	28.56	6.91
56	0.11	28.67	6.92
58	0.56	29.23	6.93
60	0.45	29.68	6.94
62	0.64	30.32	6.94
64	0.94	31.26	6.94
66	1.18	32.44	6.92
70	1.5	33.94	6.92



Corrected Calcine Addition: 28 g, (Adjust pH to 7)

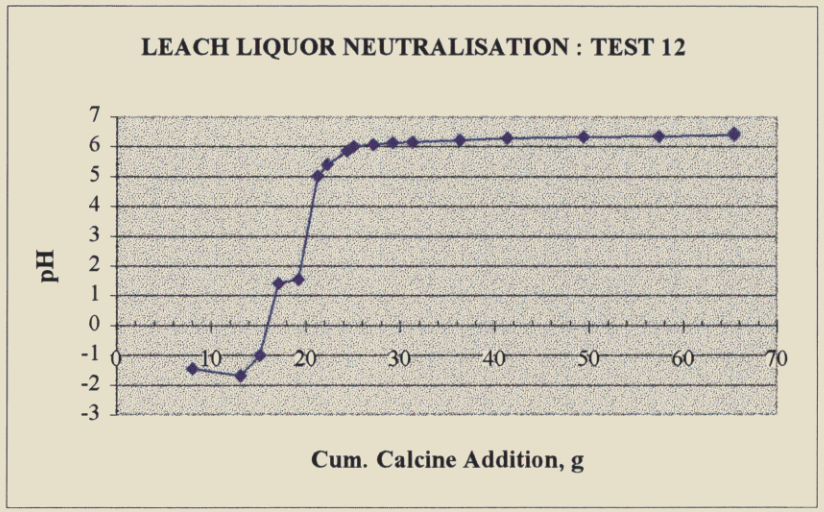
Figure 13: Neutralisation Test

Test No: 12

Liquor from -2 mm Leached at 50°C for 2 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	8.01	8.01	-1.45
2	5.05	13.06	-1.69
4	2.05	15.11	-1
6	2.01	17.12	1.41
8	2.06	19.18	1.54
10	2.02	21.2	5.01
12	1.07	22.27	5.41
14	2.06	24.33	5.86
16	0.69	25.02	6.01
18	2.09	27.11	6.08
22	2.06	29.17	6.14
24	2.08	31.25	6.17
26	5.02	36.27	6.23
28	5.06	41.33	6.29
32	8.03	49.36	6.33
34	8.03	57.39	6.36
38	8.03	65.42	6.39
42	0	65.42	6.4
55	0	65.42	6.47



Corrected Calcine Addition: 26 g (Adjust pH to 6)

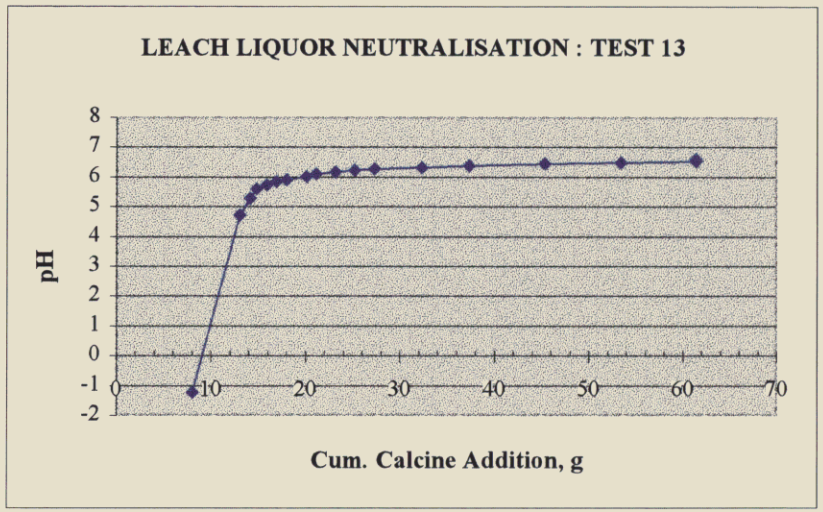
Figure 14: Neutralisation Test

Test No: 13

Liquor from -2 mm Leached at 75°C for 1 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	8.01	8.01	-1.22
2	5.01	13.02	4.73
4	1.07	14.09	5.28
6	0.68	14.77	5.6
8	1.07	15.84	5.73
10	1.06	16.9	5.84
12	1.07	17.97	5.92
14	2.09	20.06	6.01
16	1.01	21.07	6.1
18	2.06	23.13	6.17
22	2.06	25.19	6.23
24	2.1	27.29	6.27
26	5.03	32.32	6.32
28	5.01	37.33	6.39
32	8.01	45.34	6.45
34	8.01	53.35	6.49
38	8.01	61.36	6.53
42	0	61.36	6.56
55	0	61.36	6.63



Corrected Calcine Addition: 22 g, (Adjust pH to 6)

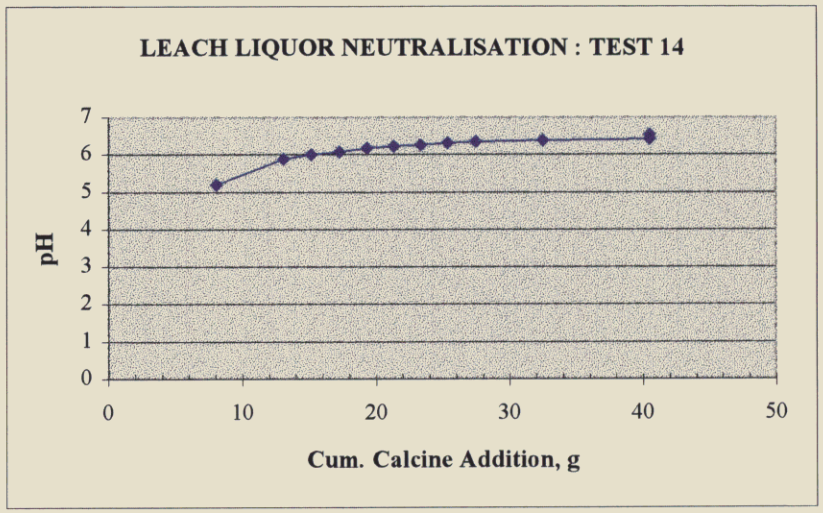
Figure 15: Neutralisation Test

Test No: 14

Liquor from -2 mm Leached at 75°C for 2 hr (110% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	8.03	8.03	5.2
2	5.06	13.09	5.89
4	2.06	15.15	6
6	2.08	17.23	6.08
8	2.07	19.3	6.17
10	2.02	21.32	6.23
12	2.01	23.33	6.27
14	2.02	25.35	6.31
16	2.09	27.44	6.35
18	5.01	32.45	6.39
20	8.01	40.46	6.42
29	0	40.46	6.53



Corrected Calcine Addition: 18 g (Adjust pH to 6)

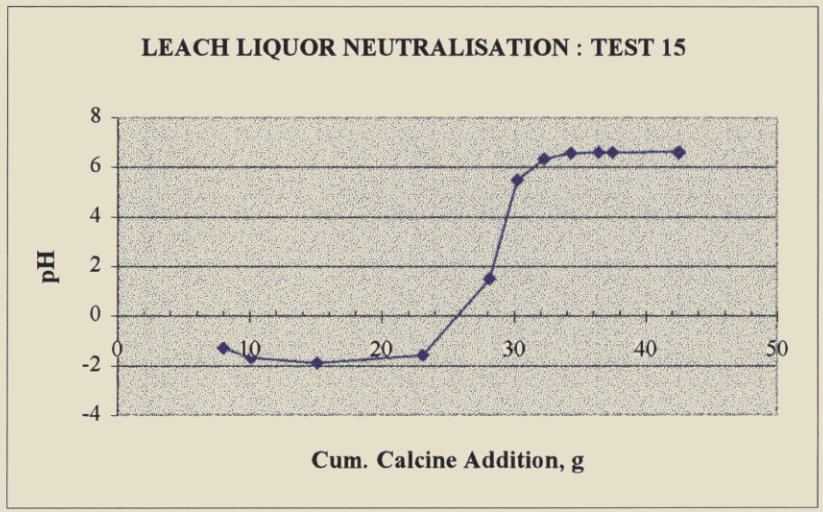
Figure 16: Neutralisation Test

Test No: 15

Liquor from -2 mm Leached at 50°C for 1 hr (120% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	8.01	8.01	-1.29
2	2.06	10.07	-1.66
4	5.01	15.08	-1.89
6	8.01	23.09	-1.57
8	5.05	28.14	1.51
10	2.06	30.2	5.5
12	2.05	32.25	6.35
14	2.06	34.31	6.58
16	2.06	36.37	6.61
18	1.07	37.44	6.6
20	5.02	42.46	6.62
29	0	42.46	6.59



Corrected Calcine Addition: 35 g (Adjust pH to 6.8)

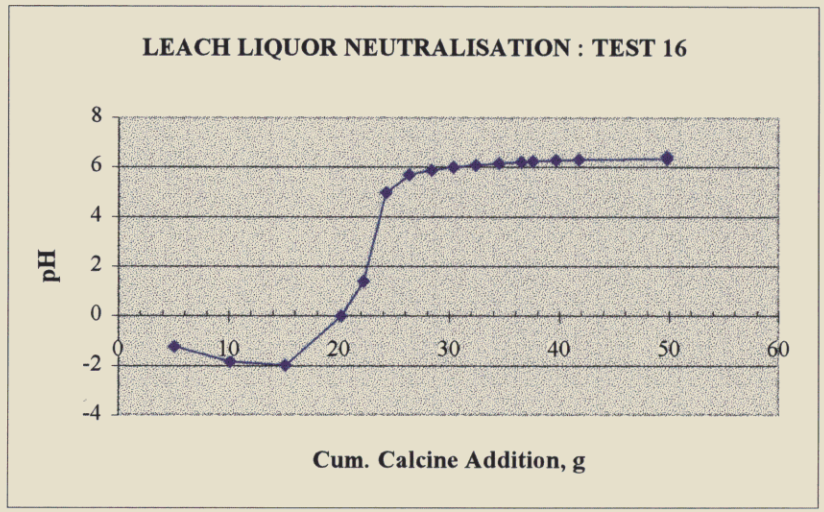
Figure 17: Neutralisation Test

Test No: 16

Liquor from -2 mm Leached at 50°C for 2 hr (120% stoich.)

Soln. Vol., mL: 200

Time, min	Calcine Addition, g	Cum. Calcine, g	pH
0	5.03	5.03	-1.22
2	5.06	10.09	-1.83
4	5.05	15.14	-1.98
6	5.01	20.15	0
8	2.06	22.21	1.4
10	2.06	24.27	4.97
12	2.07	26.34	5.7
14	2.02	28.36	5.89
16	2.01	30.37	6.01
18	2.02	32.39	6.09
20	2.06	34.45	6.16
22	2.06	36.51	6.21
24	1.07	37.58	6.24
26	2.09	39.67	6.29
28	2.06	41.73	6.32
30	8.03	49.76	6.35
42	0	49.76	6.44

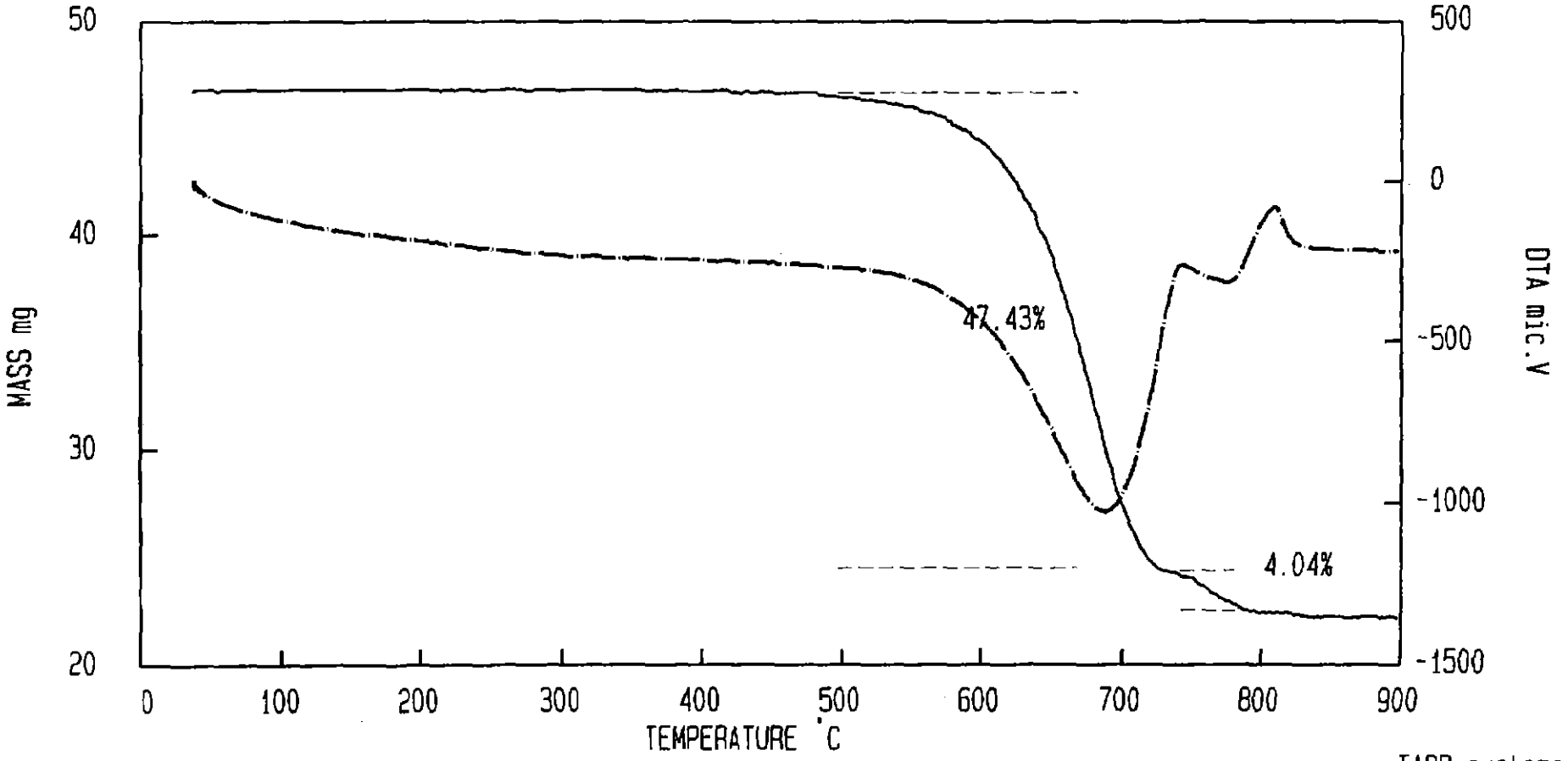


Corrected Calcine Addition: 30 g (Adjust pH to 6)

THERMAL CHARACTERISATION LABORATORY

CURTIN UNIVERSITY OF TECHNOLOGY

Sample:	COMP 1 (CONC)	REFERENCE:	EMPTY
Mass:	46.74 mg	Date/Time Plotted:	07-11-1998 17:58:21
Heating Rate:	20 C/MIN	Date/Time Collected:	07-11-1998 17:54:16
Crucible:	PLATINUM	File:	C:\IAN\T5017_1.DTA
Atmosphere:	NITROGEN Flow: 60.	Operator:	IAN

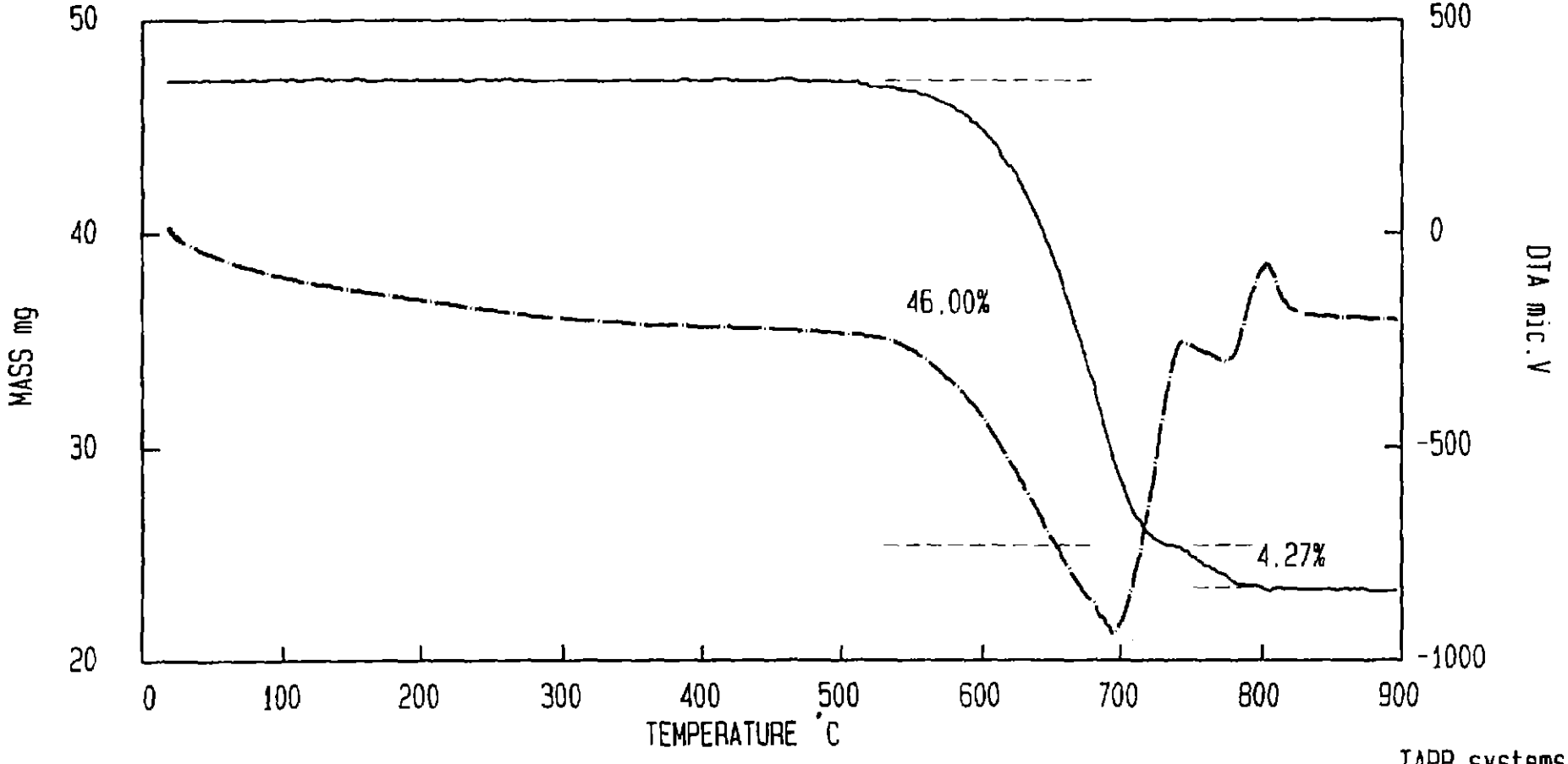


TAPP systems

THERMAL CHARACTERISATION LABORATORY

CURTIN UNIVERSITY OF TECHNOLOGY

Sample:	COMP 1 (-2MM)	REFERENCE:	EMPTY
Mass:	47.17 mg	Date/Time Plotted:	07-11-1998 19:44:16
Heating Rate:	20 °C/MIN	Date/Time Collected:	07-11-1998 19:41:43
Crucible:	PLATINUM	File:	C:\IAN\T5018_1.DTA
Atmosphere:	NITROGEN Flow: 60.	Operator:	IAN



TAPP systems

This report has been prepared by Oretest Pty Ltd (Oretest) on the request of Mr A Firek of Golden Triangle Resources NL.

This report documents the results of metallurgical testwork conducted by Oretest on the samples provided to Oretest by the Client as detailed in Section 2 of the report.

The testwork which Oretest was required by the Client to carry out on the Samples, the results of which are documented in this report, is detailed in Section 1.

This report is provided to the Client on the basis that the Client expressly acknowledges that:

(a) no representations have been made to Oretest as to the purpose for which the tests are required to be conducted; and

(b) the Testwork was carried out on Samples provided by the Client and that Oretest was not in any way involved in the drilling, collection or transportation of the Samples and, until the date upon which they were delivered to Oretest by the Client, was not involved in any way in the handling of the Samples

By this report, Oretest makes no representation or warranty (express or implied) as to the nature, source, completeness or handling of the Samples and Oretest and its directors, employees, agents and consultants denies and disclaims all liability (including for negligence) for any loss, cost, expense or damage arising from the opinions or conclusions contained in this report to the extent that loss, cost, expense or damage arises from the nature, source, completeness or handling of the Samples prior to their delivery to Oretest.

Oretest expressly denies liability for all damages for loss of opportunity, loss of revenue, loss of actual or anticipated profit or other consequential loss arising either directly or indirectly from reliance by the Client or any other person on the content and conclusions of this report.