01-4538

827001

Experimental Development
of a
Process to Produce Magnesium
from MC36 Magnesite Ore

Final Report
(Progress Report # 4)

Project No.: LR 5398

NOTE: This report refers to the samples as received.
The practice of this Company in issuing reports of this nature is to require the recipient not to publish the report or any part thereof without the written consent of Lakefield Research.

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EXECUTIVE SUMMARY

The primary objectives of this laboratory development of a magnesite to magnesium process for Golden Triangle Resources NL (GTR) were firstly to confirm the findings of previous testwork and secondly to experimentally develop a preliminary practical flowsheet which comprises the operations of leaching, neutralisation, purification, filtration, brine evaporation and magnesium chloride crystal dehydration to $MgCl_2.1.2H_2O$. Laboratory testing of the proposed flowsheet has yielded two fully dehydrated magnesium chloride products for preliminary characterisation as feedstock for the production of magnesium metal in high efficiency electrolysis cells.

The present final report provides a proposed preliminary process flowsheet, a summary of the overall project results and the conclusions and recommendations of Lakefield Research Limited (LRL) for further flowsheet development by GTR. It also includes, in appendix, the three progress reports which were previously submitted to GTR in draft and completed forms, at various times during the first quarter of 1999.

Progress report # 1 contains results of leaching MC-36 and MC-32 magnesite ore samples, under varying conditions of ore particle size, acid concentration and addition sequence, acid to ore ratio, temperature, time and reflux condenser capacity (Constant conditions: ore addition rate; initial solid weight to liquid volume ratio; slurry agitation).

Progress report # 2 deals with purification process development and included an investigation of three purification schemes for heavy metal removal and methods for removal of calcium and sulphur from the leach slurry or brine. It also investigated three oxidation agents (chlorine, hydrogen peroxide, air) and four neutralising reagents (calcined ore, MgO, slaked $Mg(OH)_2$, NaOH) within a number of neutralisation-oxidation schemes, before proposing a simplified flowsheet for this part of the overall process. It was recognised, but not tested, that magnesite ore and caustic-calcined MgO could effectively and economically substitute for tested samples of calcined ore, $Mg(OH)_2$ and less reactive MgO.

Progress report # 3 (originally submitted as the draft report on dehydration) includes a review of eight existing dehydration technologies (including consultations with the industry), recommendation of two dehydration processes to GTR for scoping experiments (Alcan's patented methanol-ammonium chloride-ammonia process and an HCl thermal dehydration method inspired by Norsk-Hydro Bécancour practice). Preliminary experimentation of these methods was undertaken, with the aim of selecting one of these two processes for future process development. Both practical and business considerations led to the choice of Alcan technology.

Proposed flowsheet

Figure 1A presents the flowsheet developed in the present experimental laboratory program, and includes both optional purification and alternative dehydration unit operations (including the preferred Alcan route). Figure 1B, based on this flowsheet, provides approximate mass balances for magnesium and solid and liquid phases, based on non-integrated laboratory test results. Table 1 presents magnesium and impurity assays of the different products of Test 38 (chlorine gas oxidation), Test F01 (thermal dehydration) and Test Alcan 2 (methanol-ammonium chloride-ammonia dehydration), non fully integrated tests undertaken in this first phase of the magnesite project. Purified brine solution specifications have been indicated in Progress Report # 3 (Appendix A.2, p.17).

EXECUTIVE SUMMARY

The laboratory flowsheet development has successfully investigated the process chemistry and has yielded products capable of satisfying most of the purity requirements of feedstock for Alcan's MPC operation, or of similar modern high efficiency electrolytic cells and their electrolytic magnesium products. The work also succeeded in developing some optimum process conditions, but will require further intensive pre-pilot experimentation at small scale to develop the most efficient and economic conditions of operation of the several process unit operations that are destined for continuous small scale or full-scale piloting.

Leaching and dehydration, in the form of multi-stage rather than single stage operations in order to reduce metal or acid losses and reduce process costs, were not investigated in the present work and will require study in succeeding phases of the process development/implementation. Future work should also take into account recycle stream design and behaviour, optimisation of reagent addition and reaction kinetics, optimum pH-emf-temperature regimes, influence of oxidation on leaching economy, acid utilisation strategy, development of overall mass, energy, water and chlorine utilisation balances, the desirability or not of a second S/L separation, the Ca-S purification trade-off and improved purification concepts and testwork, and the development of other process parameters which are required to provide information for the continuous piloting of the process and to enable more confident engineering evaluation.

Such future development of the GTR magnesite process is considered key to its success. The recommendations are therefore set out in greater detail in the "Conclusions and Recommendations" chapter which appears at the beginning of the present report. The principal results and discussions issuing from each of the three experimental stages of Phase 1 (namely, magnesite leaching, neutralisation/ oxidation/purification/solid-liquid separation and brine evaporation/crystal dehydration) are indicated in the Summary chapter of this report.

Table 1: Chemical Analyses of Major Products of the Proposed Flowsheet (Figures 1A-1B)

A. MC-36 Magnesite Ore Sample (wt. %)

Mg	26.9	Cr	0.01
Mn	0.08	V	<0.01
Ca	1.48	Ni	<0.001
Fe	0.65	Cu	0.002
Na	<0.05	Zn	0.008
Si	1.11	S	....
B	27 ppm	Ba	....
Al	0.02		

B. Purified Leach Solution Analysis (mg/l) (Test No. 38)

g	94100	Cr	<0.10
Mn	<0.05	V	<0.10
Ca	1510	Ni	<0.10
Fe	0.18	Cu	0.1
Na	6.75	Zn	2.49
Si	0.59	S	818
B	2.07	Ba	0.37
Al	<0.50		

C. $MgCl_2.6NH_3$ From Alcan 2 Dehydration Test (ppm, unless indicated otherwise)

(Preliminary analysis data)

Mg	12.8 %	Cr	
Mn	< 50	V	
Ca	1500	Ni	< 20
Fe	< 50	Cu	
Na		Zn	< 10
Si	< 200	S	< 100 *
B		Ba	
Al		MgO	....

* S analysis is approximate; accurate assay will require method adapted to the product.

D. $MgCl_2$. From Thermal Dehydration Test F01 (ppm, unless indicated otherwise)

Mg	23.5 %	Cr	7.4
Mn	< 5	V	....
Ca	4150	Ni	6.7
Fe	49	Cu	5.3
Na	62	Zn	11
Si	300	S	800
B	....	Ba	< 5
Al	71	MgO	9800

N.B.: The reader is referred to Table 13 of Progress report # 2 (Appendix A.2, p.17) for purified brine specifications. Comparison of those specifications with the dehydration products must consider the brine solution $MgCl_2$ composition and the form of the dehydration product.

Figure No. 1A
Preliminary
Proposed GTR
Process Flowsheet

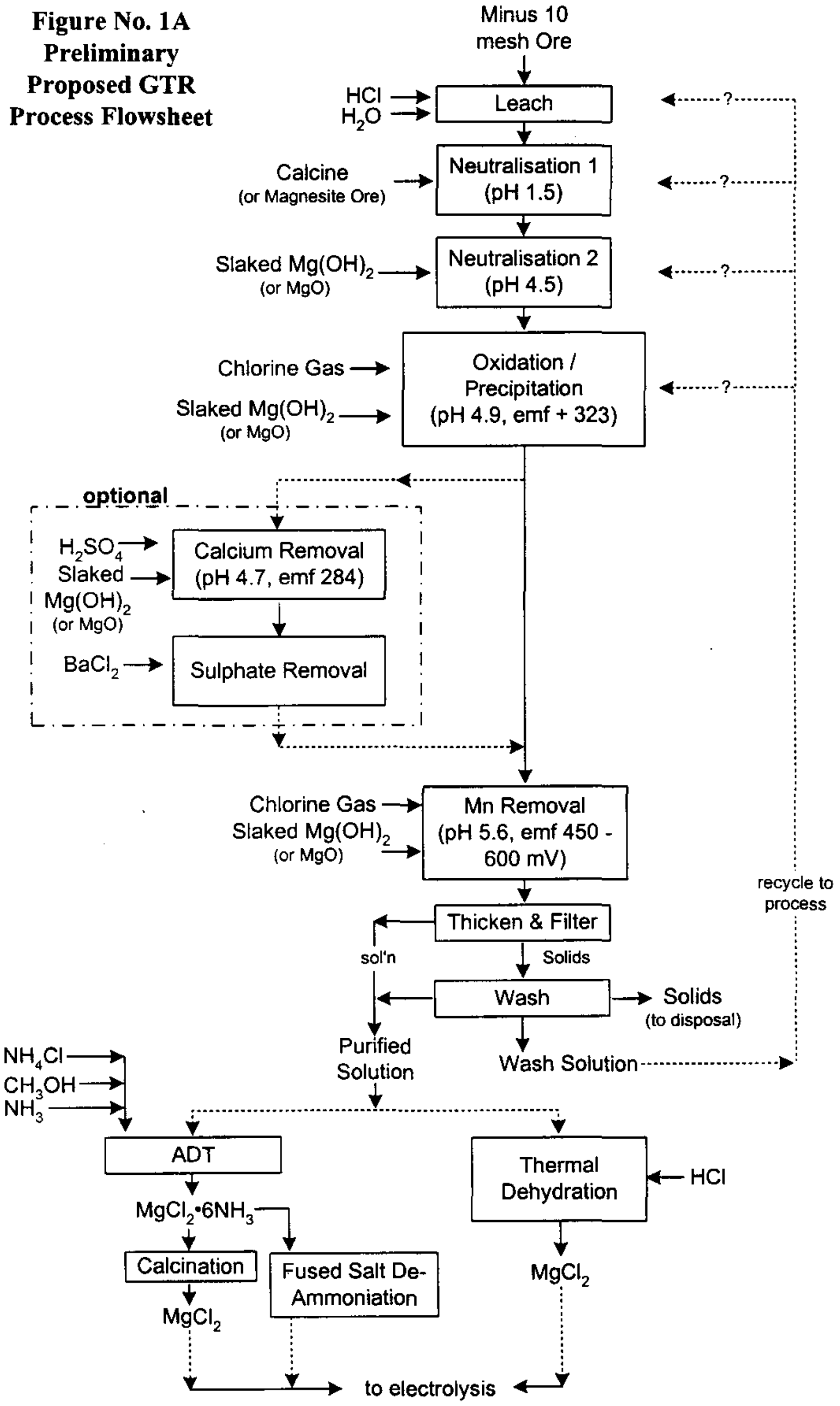
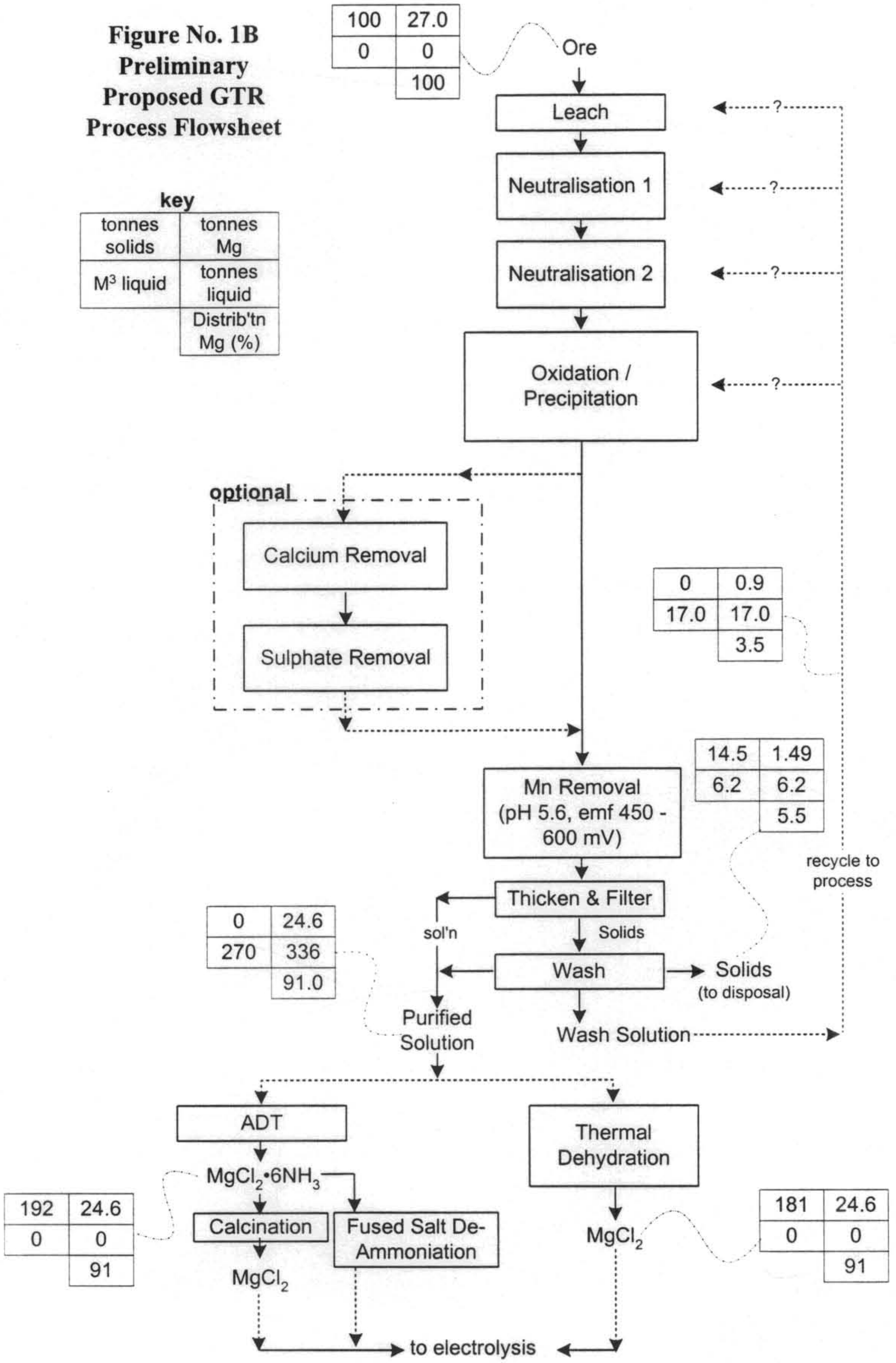


Figure No. 1B
Preliminary
Proposed GTR
Process Flowsheet



INTRODUCTION

At the request of Mr. Chris Laughton, General Manager of Golden Triangle Resources N.L., Melbourne, Australia, Lakefield Research Limited undertook an experimental laboratory program to develop a new magnesite ore to magnesium process. The aim of the program has been to research and develop each of the successive unit operations required to produce anhydrous magnesium chloride feedstock for magnesium electrowinning in a fused salt, specifically targeted at using the multipolar cell (MPC) technology of Alcan International. Alternative unit processes and conditions of operation were investigated and compared in order to provide the most economical and efficient flowsheet. Although these results are intended in part as preliminary data for an initial pre-feasibility study, conversely, such engineering feasibility information was not available to LRL to guide the various phases of this laboratory work, which simply sought to apply known industrial practices and know-how to the laboratory development of the most practical process capable of ensuring a product of suitable quality for the Alcan MPC cell.

This final report summarises the most significant results, discussions and conclusions appearing in the three progress reports which have been previously issued in both draft and completed form to Golden Triangle Resources during the period of December 1998 through to the end of the first quarter of 1999 (the final dehydration testwork was found to be particularly arduous, requiring the best part of two months to research and execute). This final report, progrss report # 4, summarises the progress, establishes a preliminary flowsheet and provides detailed conclusions and recommendations for further testwork to the goal of process implementation.

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Testwork by G. Toole, technologist

1. Conclusions and Recommendations

1.1 Conclusions

The objectives and deliverables of this Phase 1 study of a new magnesite to magnesium process (LR 5398) have been presented in the previous progress reports #1, #2 and #3, which were communicated to GTR in draft and finalised forms between December 1998 and March 1999.

The present progress report # 4 or Final report presents a proposed practical flowsheet (See Figures 1A and 1B of the Executive Summary) for the transformation of Main Creek magnesite to Mg electrolysis feedstock of appropriate composition. This experimentally developed flowsheet should serve as basis for the further development of an economical process. Specific conclusions, related to the various unit processes of the proposed flowsheet, have been presented in the previous progress reports. This report presents further results and conclusions on the performance of the two dehydration technologies (Alcan ADT and a generic thermal dehydration process) scoped in Phase 1 of this project. It provides data to improve pre-feasibility calculations and makes detailed recommendations on the required testwork to carry the project forward to both feasibility and engineering cost study stages, preparatory to process implantation.

The comparative dehydration processes testwork of Phase 1 permits the conclusion that the methanol-ammonium chloride-ammonia process (Alcan "ADT") provides the potentially better chance of industrial success for GTR. It is recommended that priority in further development be first given to further experimentation of that technology, to determine if it is indeed suitable for the transformation of purified brine treatment from magnesite leaching to high purity feedstock for the MPC electrolysis cell or similar high efficiency cells.

This conclusion and recommendation follow from:

1. The satisfactory preliminary scoping test result of ADT during Phase 1, coupled with Alcan assurances regarding MgO content of an ammoniated product (as low as 0.10 and 0.05 %);
2. The accessibility of Alcan dehydration technology to emerging Mg producers;
3. Probability of a comparatively inexpensive non-exclusive licensing from Alcan; and
4. The synergy of an Alcan-LRL collaboration with GTR, notably:
    - The proximity of the experimental research teams;
    - Alcan's desire to undertake testwork off-site;
    - Alcan's familiarity with LRL capabilities (present and past collaborations);
    - LRL's familiarity with the ADT process.

At this point, the case for the thermal dehydration route appears less convincing: The scale-up of thermal dehydration technology from laboratory scale to large pilot scale, even in the case of the relatively simple HCl process successfully scoped in this work, cannot be easily realised (see further discussion below). Accordingly, thermal dehydration should probably be considered as a second alternative to ADT, only if the continued tests of the latter uncover unexpected problems that are difficult to surmount.

Specifically, thermal dehydration success would require overcoming the following obstacles:

1. The acknowledged difficulty of obtaining thermal dehydration know-how and licensing (Norsk-Hydro has declared to us that it is very adamantly protective of its technology and know-how; collaborating engineering companies have been obliged to sign "life-of-plant" absolute secrecy agreements with Hydro; key engineering is believed to have been undertaken directly by Hydro);
2. Several carbonaceous reductant thermal technologies carry specific environmental burdens, such as chlorinated hydrocarbon production/emissions, or unknown commercial performance.
3. The piloting of a pyrometallurgical operation (Ex.: difficulties of design and scale-up of gas-solid and gas-solid-liquid reactors) can be onerous and require several scale-up intervals from pilot test to pilot test (1:10 scale-up interval is typical for many such pyrometallurgical processes);
4. Unless GTR can buy the technology, know-how, equipment and performance guaranties "off-the-shelf", the retention of the thermal dehydration route could represent a potentially important financial burden for GTR and add significantly to the delays from piloting to production.

These comments are made notwithstanding the very good performance, and readily perfectible conditions and chemistry, of the laboratory thermal dehydration scoping experiment undertaken in this Phase of the project.

1.2 Recommendations

The following experimental development and process demonstration activities are *strongly* recommended in order to carry the work successfully from the results of this present Phase 1 of the program through to confident feasibility and engineering cost studies and equipment / technology acquisition, prior to the process implementation stage:

Phase 2: Further development/optimisation of laboratory batch scale testwork

Based upon this Phase 1 laboratory testwork to establish a proposed basic flowsheet, Phase 2 would further the development of an economic flowsheet through additional laboratory/miniplant batch detailed testing and optimisation of the following unit processes: leaching; neutralisation and purification; solid-liquid separation; solution evaporation and $MgCl2$ dehydration.

The principal objectives of this recommended Phase 2 testwork are detailed below:

Key objectives of the Phase 2 proposal:

In regard to the leaching unit process:

- Determine the effect of multi-stage leaching and co-current vs.counter-current operation on acid utilisation efficiency (minimise acid losses rather than best possible Mg recovery);

CONCLUSIONS / RECOMMENDATIONS

- Investigate present Norsk-Hydro practice of variable particle size magnesite feeds on leaching operation efficiency; investigate HCl gas recycle to leaching (in respect of water balance and costs);
- Investigate reflux condenser needs and potential losses due to foaming and effluent gas displacement of charge; effect on temperature-time optimisation and selection;
- Investigate the effect of oxidising leaching vs. reductive leaching, on acid utilisation efficiency and on multivalent impurity solubilisation (effect on Mn, Fe, etc.) and impact on residue form (impact on slurry viscosity and eventual settling and filtration behaviour);
- Obtain further results on the effect of acid concentration, of S/L ratio in leaching, of excess magnesite ore addition (acid deficiency in final stage), and the effects of reaction temperature and time and their optimisation (leach kinetics);
- Undertake locked cycle testwork with wash solution recycle to leaching and other recycle streams to leaching (choice dependent upon results of work on subsequent unit processes); establish impurity deportment and effect on metallurgy;

In regard to the neutralisation-purification-filtration unit process:

- Investigate conditions for proposed magnesite ore – magnesia – NaOH neutralisation steps (slight concentrations of Na in brine from slight excess NaOH are advantageous);
- Optimise the direct neutralisation and oxidising precipitation procedure developed and selected in phase 1, including optimisation of chlorine oxidation conditions and minimum Mg loss, temperature, emf, pH, nucleation conditions and crystal growth period (time), to maximise the elimination of iron, manganese and other elements, and to control the forms of the precipitates (impact on settling, filtration);
- Optimise settling and filtration in regard to one (preferred) or two (initial bulk + polishing filter) S/L separations;
- Pursue Phase 1 experimentation of $CaSO_4$ precipitation, effect on bulk precipitation;
- Investigate alternative Ca and S elimination operations, in addition to those commercial practices (Dow, Norsk-Hydro) studied in Phase 1;
- Repeat for the optimised conditions further small scale settling and filtration tests, with or without settling or filtration reagents; prepare large batch of slurry under best conditions and perform larger scale filtration tests on pilot scale filters;
- Develop best wet waste cake (WWC) porosity and filtration/washing characteristics;
- Establish best washing practice of the optimum WWC; balance water evaporation costs against Mg losses and effluent treatment costs;
- Determine chlorine losses and water balances; study stream recycle (wash water, other) implications;

The metallurgical target for this work will be the purified brine specifications compatible with each of: dehydration technology, high efficiency electrolysis cell operation constraints and magnesium alloy impurity requirements (the development of these values will be made in collaboration with Alcan, Sitec, and on the basis of LRL information on current commercial brine specifications.

In regard to purified brine evaporation and $MgCl_2$ crystal dehydration unit processes:

- Testwork on the Alcan process would pursue batch scale tests to optimise the NH_4Cl to water ratios, NH_3 consumption, rate of reagent addition and precipitation (crystalliser) conditions, temperature of the CH_3OH-NH_3-NH_4Cl-H_2O bath, rate of heat extraction and the effect of different feed preparation (brine vs. crystallised $MgCl_2.nH_2O$);

- Once batch test conditions are optimised, further tests on the stripping of spent NH_3-CH_3OH-H_2O-$MgCl_2$ solution and on the recycle of the $MgCl_2$-NH_4Cl-H_2O solution to the brine contacting stage should be undertaken. Small continuous process tests or locked cycle tests should be considered;
- The product should be dried and calcined to differing nNH_3 stages to determine effect on energy balance; material and energy balances may also be obtained from the other constituent operations;
- The use of a spray drier to provide $MgCl_2.nH_2O$ from purified brine should be questioned, and possibly be circumvented by a direct contacting with the process reagents;
- Ca, S and other impurities will be followed through the various process streams and products; conditions favourable to Ca and S rejection will be experimented, consistent with maximum recovery of MgO-free $MgCl_2.6NH_3$.
- Sufficient material will be produced for initial electrolysis feed characterisation tests; heat, water and mass balances will be obtained at small scale to guide feasibility evaluation.

This work will be undertaken in close co-operation with Alcan, which has developed a positive working relationship with LRL on former Alcan projects and on dehydration scoping testwork over the past two months.

Phase 3: Small scale continuous testwork

Phase 4: Full-scale pilot testwork of critical operations and process demonstration

These two phases (3 and 4) correspond to the overall recommendations of Bateman, Brown and Root for their proposed phases indicated as “2 and 3”, as summarised in a note to LRL from GTR in March 1999. Specific LRL recommendations for Phases 3 and 4 will be provided later and will refer to the nature of the required testwork and the process issues and strategy to be invoked in regard to the overall process development and implementation (A summary preliminary proposal on these phases of the project was forwarded to GTR in March 1999).

2. Summary and Discussion of Project Results

2.1 Leaching Unit Process

Scoping leach tests were undertaken on MC36 sample under the following range of conditions:

- Constant S/L ratio (0.4, as initial g. of solids weight to ml of solution);
- 3, 6, 10 and 28 mesh crush size (100 % passing);
- 60, 90 and 120 minutes leach period;
- Specific dry solids addition period (5 to 10 minutes; consistent with limited foaming);
- Leach temperature of 50, 80 and 95 °C (azeotropic boiling point estimated as 107 °C);
- Acid addition of 110, 120 or 130 % stoichiometric (based upon Mg and Ca dissolution only);
- Staged acid addition (t_0 and t_{60}) and / or increased reflux condensation capacity (2X normal).

Additional tests were undertaken at the request of GTR to remove calcium using a 20 minute pre-leach, as suggested by Oretest Pty.

10 mesh crush size appears to be close to optimum under the conditions tested. When the magnesite ore samples were crushed to 100 % – 10 mesh, and leached with 110 % of the calculated stoichiometric quantity (ignoring $FeCO_3$) of HCl, for 60 minutes at 80 °C, the highest extractions were obtained (94 % Mg in the case of sample MC36; 97 % Mg for sample MC32).

The acid requirements to reach these extractions were 2519 kg of 36 % HCl per tonne of feed,or 907 kg on a 100 5 HCl basis (Maximum leach acid concentration in the pulp was of the order of 32 %). In practice, Cl2 would be recycled from electrolysis, transformed to HCl and returned to leaching as a solution and possibly as HCl gas. The latter use would likely be influenced by the water balance considerations (Ex: Quantity of recycle water from filtration washing).

Lower stoichiometric ratios were not tested, nor were lesser leach times at the higher temperatures (80 and 95 °C). Future work would consider these and optimum S/L ratios, as well as replication of Norsk Hydro type 3 stage variable crush size feed leaching or other leach strategies, such as LRL's suggestion of magnesite ore leach pre-neutralisation (Ref: LRL proposal to GTR on asbestos/magnesite leaching, September 23, 1999). Possible solids and liquid losses resulting from high velocity expulsion of CO_2 from the reactor were not detected, within the experimental errors of combined solution and residue Mg contents.

The pre-leach was not very effective (best result of 34 % Ca extraction) in removing calcium as was the use of the Dow Chemical method of Ca removal, as tested in the later solution purification work. The separate treatment of the pre-leach solution presents a problem. On the other hand, although more efficient in removing calcium, the Dow method leaves residual sulphates which would require removal using barium chloride (Norsk-Hydro method).

Recommendations for further leaching testwork in Phase 2 have been mentioned in Chapter 1.2.

2.2 Neutralisation/Oxidation/Precipitation and Solid-Liquid Separation Unit Processes

the objectives of this stage of flowsheet development was to confirm prior testwork, to test three LRL-proposed purification routes and to develop a simplified flowsheet for the leach slurry neutralisation / oxidation / purification and solid liquid separation operations of a future process.

A simplified flowsheet based upon a "DOP" (Direct neutralisation and oxidising precipitation of impurities) purification scheme, potentially coupled with known methods for calcium (Dow Chemical) and sulphur (Norsk Hydro) elimination, provide the preferred neutralisation , oxidation and purification flowsheet. Neutralisation would be accomplished to pH 2 using calcined ore or (preferably) magnesite, with brucite ($Mg(OH)_2$) or (preferably) low boron caustic-calcined MgO being used to raise the pH to 4 or 5. Further neutralisation would be undertaken using very small additions of NaOH (Note that the presence of Na in the brine may not be a major concern, given the probable impact of Ca impurity and the combined Na-Ca presence in the Mg cell electrolyte).

Best results of twelve purification tests provided purified brine of the following composition:

Mg	985000 mg/l
Ca	1250 mg/l
Fe	<0.10 mg/l
Mn	0.2 mg/l
Zn	6.96 mg/l
Al	1.2 mg/l
Cu	0.9 mg/l
B	2.4 mg/l
Ni	<0.1 mg/l
Cr	< 0.1 mg/l
Na	6.8 mg/l
Si	0.8 mg/l
S(T)	926 mg/l

The use of H_2SO_4 could precipitate up to 76% of the leach-dissolved calcium from the brine as gypsum. This option will depend upon the success of removing sulphate ion with the Norsk-Hydro ($BaCl_2$) or other method.

The precipitated gypsum product improved the filtration of the difficult to filter iron products and this may constitute a novel advantage of the GTR process. However, further leaching testwork may well provide more easily filterable iron.

The optimisation and selection of best reagents for this part of the process would be pursued in Phase 2 testwork amongst other objectives which are described in the Recommendations chapter (1.2) of this report.

2.3 Dehydration Phase

The testwork advanced beyond the initially proposed and limited experimentation of a single dehydration process which would yield either $MgCl_2.2H_2O$ or $MgCl_2.1.2H_2O$. An anhydrous product, $MgCl_2$, has been experimentally produced using HCl gas-thermal dehydration. Whereas the MgO content was high (0.98 %), the possibility of obtaining an otherwise pure $MgCl_2$ product was demonstrated.

The Alcan dehydration method has been scoped and yields a product, the Mg content of which (12.8 %) suggests the formation of $MgCl_2.6NH_3$. Although no known diffraction lines are available to characterise $MgCl_2.6NH_3$, the analytical method demonstrated the absence (at a detection level of about 2 %) of oxides, oxychlorides or hydroxychlorides. A white precipitate was observed to form under the ammonia injection tube within the $MgCl_2/NH_4Cl/NH_3/H_2O$ crystalliser bath. Whereas further experimentation will be required to investigate best operating conditions for the production of a low MgO product, to test the effect of impurities in the recycle streams and to transfer the operation to larger scale, the preliminary results suggest that the basic chemistry of the method should be applicable to GTR purified brine.

At this stage of knowledge and experimentation, the preferred method for further optimisation testwork would appear to be the Alcan ADT process. In regard to thermal dehydration, the combination of HCl atmosphere and a reducing substance (CH_4, CO, C), or of a CO + Cl_2 atmosphere, has not been studied, but neither should be dismissed as potentially viable routes to MPC cell feedstock dehydration, although carbo-chlorination is known to yield difficult environmental problems. Unfortunately, there is also no indication that these technologies can be easily acquired from their potential suppliers. The scale-up of thermal processes, such as the Norsk-Hydro dehydration, involves mass and heat transfer considerations which must be treated at large scale, as well as the usually difficult scale-up of pyrometallurgical reactors.

APPENDICES

A.1: Progress report # 1, Project LR 5398

A.2: Progress report # 2, Project LR 5398

A.3: Progress report # 3, Project LR 5398

Appendix A1

Progress Report No. 1
LR-5398

827016

An Investigation

of

HCl Leaching of Magnesite Ore Samples

from

Golden Triangle Resources

Progress Report No. 1

Project No.: LR 5398

NOTE: This report refers to the samples as received.
The practice of this Company in issuing reports of this nature is to require the recipient not to publish the report or any part thereof without the written consent of Lakefield Research.

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March 23, 1999

ABSTRACT

Two magnesite samples denoted MC32 and MC36 were received by Lakefield Research Limited from Golden Triangle Resources. The samples assayed as follows:

Sample	Assay (%)			
	Mg	Ca	Si	Fe
MC32	26.2	1.51	0.95	1.62
MC36	26.9	1.48	1.11	0.65

A series of hydrochloric acid leach tests were performed on the two samples, for the extraction of magnesium. The majority of the tests were performed on sample MC36, experimenting varying conditions of ore particle size (100 5 passing "crush size"), acid concentration and addition sequence, acid to ore ratio, temperature, time and reflux condenser capacity. The conditions maintained constant were ore addition rate, initial solid weight to liquid volume ratio and slurry agitation.

When the samples were crushed to 100% minus 10 mesh, and leached with 110% of the calculated stoichiometric quantity of HCl, for 60 minutes at 80 °C then 94% of the magnesium was extracted from sample MC36, and 97% from sample MC32. The acid requirements to achieve these extractions are; 2519 kg of 36% HCl per tonne of feed, or on a 100% HCl basis 907 kg/t.

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INTRODUCTION

At the request of Mr. Chris Laughton of Golden Triangle Resources, Lakefield Research Limited under took a test program to investigate the hydrochloric acid leaching of two magnesite samples from their Tasmanian Main Creek deposit (sample MC32 and MC36). Test results were forwarded electronically, and by fax to Mr. Laughton and Golden Triangle Resource's consultant Mr. Andrew Firek.

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Experimental Work by: G. Toole

1.0 SUMMARY of Results

1.1 Sample Preparation and Head Analysis

Two samples were received at Lakefield Research Limited on Nov 17th 1998 (LR 9803120). The samples were received in four bags, two for each sample. The samples were labelled MC32 and MC36. They consisted of quarter sawn 1-3/4 inch diameter drill core. The total weight of each sample was approximately 50 kg.

The individual samples were blended, one half of the material was split out and set aside in the as received state. The other half of the sample was crushed to minus 10 mesh, and split into 1000 gram test charges.

Head samples (10 grams each) were removed from the first eight charges (minus 10 mesh) used in the test program. The average of these analyses is shown in Table No. 1, whereas the individual sample analysis are presented in Appendix A.

Table No. 1 - Head Analysis

Element	MC36 (XRF)	MC32 (XRF)
Mg [%]	26.9	26.2
Ca [%]	1.48	1.51
Si [%]	1.11	0.95
Fe [%]	0.65	1.62
Mn [%]	0.08	0.07
Al [%]	0.02	0.02
Cr [%]	0.01	<0.01
Na [%]	<0.05	<0.05
K [%]	<0.01	<0.01
Ti [%]	<0.01	<0.01
P [%]	<0.01	<0.01
V [%]	<0.01	<0.01
LOI [%]	50.0	49.9
SUM [Oxide %]		
Cl [g/t]	110	38
Ni (%)	<0.001	<0.001
Zn (%)	0.008	0.003
Cu (%)	0.002	<0.001
B (g/t)	27	<20

The client requested that the analysis of iron, calcium and silica be confirmed, as they were not at the levels that were expected. These analyses were repeated several times by X-ray fluorescence and by atomic absorption techniques, and the values returned were within the acceptable range for all of the analytical techniques employed. The results of the duplicate analysis are shown in Table No. 2.

Table No. 2 - Re-Analysis of MC 36 Heads
(minus 10 mesh feed samples from Leach Tests 1 to 8)

Feed Samples		Assay				
		Ca (%)	Fe (%)	Mg (%)	Si (%)	B* (g/t)
LCH 01	Original (XRF)	1.49	0.67	26.8	1.19	...
	Re-assay (XRF)	1.50	0.61	26.0	1.15	32
	Re-assay (AA)	1.51	...	...	1.12	...
LCH 03	Original (XRF)	1.44	0.66	26.5	1.17	...
	Re-assay (XRF)	1.44	0.65	26.9	1.21	24
	Re-assay (AA)	1.42	...	...	1.15	...
LCH 06	Original (XRF)	1.46	0.65	27.0	1.04	...
	Re-assay (XRF)	1.47	0.66	27.2	1.07	24
	Re-assay (AA)	1.44	...	...	1.02	...
LCH 08	Original (XRF)	1.51	0.66	26.9	1.02	...
	Re-assay (XRF)	1.51	0.66	27.1	1.05	27
	Re-assay (AA)	1.49	...	...	1.04	...
Average	Original (XRF)	**1.48**	**0.66**	**26.8**	**1.11**	**...**
	Re-assay (XRF)	**1.48**	**0.65**	**26.8**	**1.12**	**27**
	Re-assay (AA)	**1.47**	**...**	**...**	**1.08**	**...**

*analysis by ICP

1.2 Leach Conditions

The majority of the leach tests were performed on Sample MC36, the sample with the highest purity. Twenty tests were performed on this sample, examining the effect of leach time, temperature, acid dosage, crush size, stage addition of acid and increased acid reflux. The conditions used in the tests are summarised in Table No. 3. Three additional tests were performed on MC36 to evaluate the possibility of pre-leaching the calcium. Two leach tests were performed on sample MC32, to evaluate its amenability to the HCl leach process.

The leach tests were performed in covered glass reaction vessels, equipped with glass reflux condensers. The combined water acid mixture was heated to the test temperature and the dry solids were added slowly, over a period of 5 to 10 minutes. The pulp frothed considerably for the first 30 minutes of the leach, as the CO_2 was driven off. The frothing generally increased the pulp volume by at least 50%.

Table No. 3 - Leach Conditions

Test No.	Sample	Crush Size	Temp (°C)	Time (min)	HCl (Stoich %)	Initial Density (w/w %)	Initial L/S (ml sol'n/ /g solids)	Other
1	MC-36	10 m	80	90	120	25.7	2.48	Effect of time, temp & acid
2	MC-36	10 m	80	90	110	25.7	2.48	" " " " " "
3	MC-36	10 m	80	60	120	25.5	2.48	" " " " " "
4	MC-36	10 m	80	60	110	25.7	2.48	" " " " " "
5	MC-36	10 m	50	90	120	25.5	2.48	" " " " " "
6	MC-36	10 m	50	90	110	25.7	2.48	" " " " " "
7	MC-36	10 m	50	60	120	25.5	2.48	" " " " " "
8	MC-36	10 m	50	60	110	25.7	2.48	" " " " " "
9	MC-36	28 m	80	90	120	25.5	2.48	Effect of crush size
10	MC-36	28 m	80	120	120	25.5	2.48	" " " "
11	MC-36	10 m	20	30	pH 3	28.2	2.54	Ca pre-leach
12	MC-36	10 m	20	30	pH 2	27.3	2.64	Ca pre-leach
13	MC-36	10 m	20	30	pH 1	26.3	2.75	Ca pre-leach
14	MC-36	10 m	50	240	120	25.5	2.48	Kinetic samples
15	MC-36	10 m	80	90	110	28.7	2.14	Use low boron glass
16	MC-36	6 m	95	60	110	25.8	2.48	Effect of crush size & temp
17	MC-36	3 m	95	60	110	25.8	2.48	" " " " " "
21	MC-36	10 m	95	60	120	25.5	2.48	Effect of temp
22	MC-36	10 m	95	90	120	25.5	2.48	Effect of temp
23	MC-36	10 m	95	60 + 30	120 + 10	25.5	2.48	extra acid added after 60 minutes
25	MC-32	10 m	80	60	110	25.8	2.48	Other sample
26	MC-32	10 m	80	120	110	25.8	2.48	Other sample, effect of time
31	MC-36	10 m	90	90	130	25.5	2.48	increased reflux capacity
32	MC-36	10 m	80	60	110	25.7	2.48	increased reflux capacity
33	MC-36	10 m	80	60	110	25.7	2.48	residue sample for mineralogy

SUMMARY

The acid dosage for each test was based on the head analysis of the sample and the calculated stoichiometric quantity of acid required for the dissolution of all of the contained magnesium and calcium. The quantity of hydrochloric acid varied from test to test, ranging from 907 to 989 kg/t (on a 100% HCl basis). The variable that was kept constant throughout the tests was the initial ratio of liquid volume to solids weight, which was generally maintained at 2.5 to 1, as shown in Table No. 3. The tests were generally performed using 200 grams of solids and 500 ml of liquid.

At the end of the tests the samples were filtered hot, using a water-jacketed porcelain buckner funnel. The filter cake was washed with de-ionised water, dried at approximately 110 °C, and pulverised for chemical analysis.

The details for each leach test may be found in Appendix A.

Test numbers 18-20, 24, and 27-30 were solution purification tests, the results of which are given under separate cover (Progress Report No. 2).

The pregnant leach solutions were assayed by standard ICP methods. The residues were analysed by XRF.

1.3 Leach Results

The leach test results are summarised in the tables on the following pages. The pregnant solution and residue assays are given in Table No. 4, and the calculated metal extractions and heads are shown in Table No. 5.

Table No. 4 - Leach Results – Product Analysis

Test	Pregnant Solution								Residue							
No.	Mg (g/l)	Ca (g/l)	Mn (mg/l)	Fe (mg/l)	F.A. (g/l)	pH	emf (mV)*	Vol (ml)	Wgt (g)	Mg (%)	Ca (%)	Si (%)	Mn (%)	Fe (%)	Cl (g/t)	LOI (%)
1	98	5.2	260	2020	<1.0	-0.7	404	495	14.9	23	0.1	12	0.1	0.8	93	36
2	97	5.0	253	1950	<1.0	0.0	426	515	13.5	23	0.1	13	0.1	0.8	64	33
3	93	4.9	249	1920	2.0	-0.9	362	510	14.7	23	0.1	12	0.1	0.8	101	35
4	95	4.9	245	1860	<1.0	1.3	351	500	16.7	23	0.1	11	0.1	0.7	86	36
5	77	4.9	203	1570	106	-1.8	284	580	49.3	27	0.1	3.7	0.1	0.7	3739	47
6	69	4.8	188	1460	106	-1.6	319	510	55.6	27	0.1	3.3	0.1	0.7	982	47
7	70	4.7	184	1420	110	-1.6	236	510	51.6	26	0.1	3.3	0.1	0.7	15805	49
8	67	4.9	183	1465	116	-1.7	250	495	63.5	27	0.1	3.0	0.1	0.7	709	48
9	96	5.4	254	1920	<1.0	-0.1	384	455	22.7	25	0.1	8.0	0.1	0.8	603	41
10	99	5.3	255	1930	<1.0	1.3	398	460	19.2	24	0.1	9.6	0.1	0.9	731	38
11	0.5	0.5	...	9.0	...	3.0	...	240	97.7	27	1.3	...	...	0.60	...	...
12	1.8	1.2	...	44.8	...	2.0	...	245	95.5	28	1.1	...	...	0.6	...	...
13	2.6	1.7	...	63.9	...	1.0	...	260	94.7	28	1.0	...	...	0.6	...	...
14	81	4.5	138	1670	60	-1.6	349	440	21.7	25	0.1	8.5	0.1	0.8	402	40
15	72	4.9	198	1500	<1.0	0.7	377	120	11.0	27	0.1	4.2	0.1	0.7	1192	46
16	91	5.5	249	1890	<1.0	1.5	398	455	32.8	26	0.1	5.6	0.1	0.8	305	44
17	91	5.2	246	1850	<1.0	1.7	354	475	27.0	26	0.1	6.3	0.1	0.7	507	43
21	94	6.4	288	2230	...	...	395	480	27.4	25	0.1	7.0	0.1	0.8	486	42
22	89	5.3	221	1680	<1.0	1.1	401	460	36.9	26	0.1	5.3	0.1	0.80	570	44
23	86	4.6	223	1730	5.2	-1.7	370	540	13.4	22	0.1	14	0.1	0.8	1949	32
25	100	6.0	209	5040	2.0	-0.9	304	480	12.5	18	0.1	15	0.1	3.5	1080	34
26	103	6.1	216	5320	<1.0	-0.8	367	470	9.0	16	0.1	16	0.1	12	615	32
31	99	5.5	224	1760	7.0	-1.1	372	520	15.0	22	0.1	12	0.1	0.8	2801	35
32	98	5.5	242	1880	15.0	-1.4	342	510	16.4	23	0.1	12	0.1	0.8	3438	36
33	100	5.6	252	1960	12.0	-1.7	342	490	15.3	n/a	n/a	n/a	n/a	n/a	n/a	n/a

* vs Ag/AgCl electrode

Table No. 5 – Extractions and Calculated Heads

Test	Extraction (%)					Calc Heads (%)			
No.	Mg	Ca	Mn	Fe	LOI*	Mg	Ca	Mn	Fe
1	93.5	99.4	92.7	89.9	94.7	26.6	1.33	0.07	0.57
2	94.4	99.9	93.4	92.3	95.5	27.1	1.32	0.07	0.56
3	93.6	99.4	92.7	90.0	94.8	26.2	1.31	0.07	0.56
4	92.6	99.5	90.4	88.1	93.9	26.3	1.28	0.07	0.55
5	78.1	98.7	73.6	97.5	76.9	30.1	1.52	0.08	0.49
6	71.0	98.5	69.4	67.4	73.7	25.9	1.31	0.07	0.58
7	73.2	97.9	70.2	72.8	75.0	25.1	1.26	0.07	0.55
8	67.7	98.1	63.1	61.9	69.7	26.6	1.33	0.08	0.58
9	89.1	99.3	87.0	83.7	90.8	25.7	1.30	0.07	0.55
10	91.3	99.4	87.8	85.1	92.7	26.3	1.30	0.07	0.55
11	0.5	9.7	...	0.4	...	26.6	1.35	...	0.59
12	1.7	22.9	...	1.9	...	26.8	1.33	...	0.59
13	2.7	33.9	...	2.9	...	26.9	1.37	...	0.61
14	87.6	99.2	77.8	81.7	91.3	24.1	1.08	0.04	0.45
15	76.3	98.5	74.7	70.8	94.9	24.7	1.31	0.07	0.56
16	83.3	98.7	79.9	78.4	85.7	25.8	1.31	0.07	0.57
17	86.5	98.5	84.9	83.3	88.5	25.9	1.29	0.07	0.55
21	87.2	99.2	85.3	84.0	88.6	...	1.60	0.08	0.66
22	81.6	98.2	76.3	73.3	83.7	26.4	1.30	0.07	0.55
23	94.3	99.3	93.0	89.9	95.7	25.6	1.29	0.07	0.54
25	95.7	99.7	91.1	85.1	95.8	25.9	1.48	0.06	1.47
26	97.2	99.8	93.6	88.1	97.1	25.8	1.48	0.06	1.47
31	94.1	99.9	92.0	88.3	94.8	28.3	1.48	0.07	0.54
32	93.1	99.5	90.6	88.4	94.1	27.5	1.46	0.07	0.56
33	...	...	...	...	...	...	...	...	...

*calculated from head and residue values

SUMMARY

The effect of leach time and temperature on the extraction of magnesium from Sample MC36 is shown in Figure No. 1. The results shown are for leaches of minus 10 mesh material using a 120 percent stochiometric acid addition. The highest extractions were obtained at 80 °C. Under these test conditions, at 80 and 95 °C, there was little correlation between extraction and terminal free acidity, indicating that excess acid may not be required at the end of the leach.

The magnesium and calcium leach kinetics for Sample MC36, at 50 °C were measured at 1, 2, 3, and 4 hours (Test 14). The results are shown in Figure No. 2. The maximum magnesium extraction was reached after about 2 hours at this temperature. The results at higher temperatures suggest that shorter reaction times may be possible at 80 or 95 °C.

Figure No. 1 - Magnesium Extraction vs Temperature (MC36)

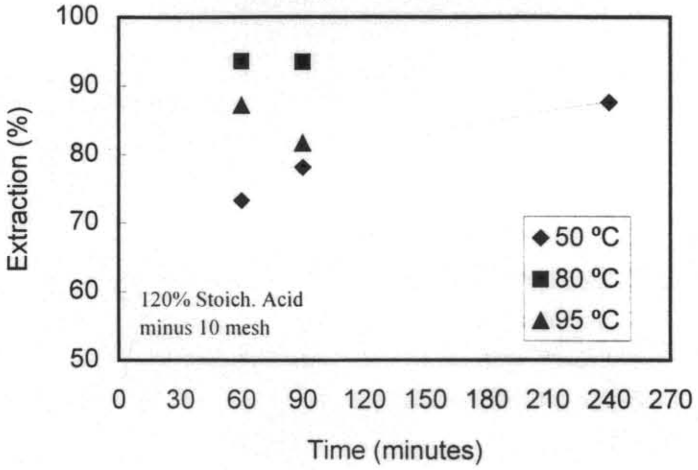
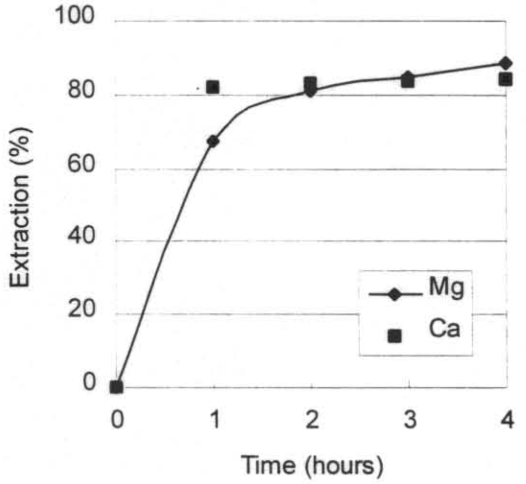


Figure No. 2 - MC36 Leach Kinetics at 50 °C



5 cm

Figure No. 3 - Ca & Mg Extractions from MC36 in a Cold Acid Leach

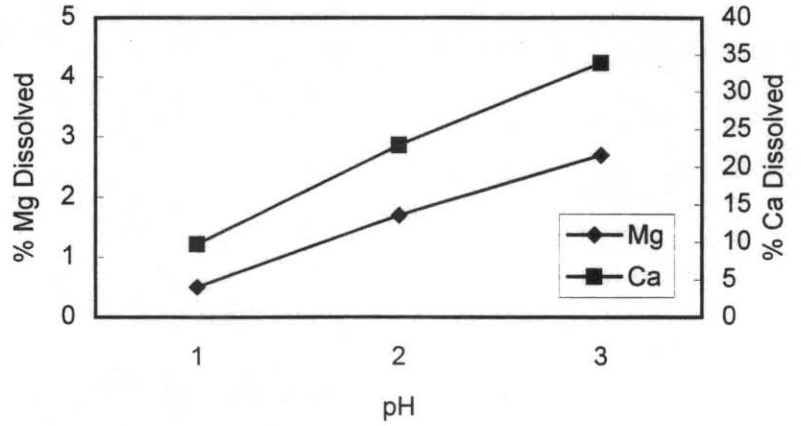
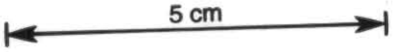


Figure No.3 illustrates the results obtained in the “Calcium Pre-Leach” tests. Up to 34 percent of the calcium could be leached from sample MC36, while only removing 3 percent of the magnesium. Other methods of controlling the quantity of calcium reporting to the down-stream metal recovey processes were investigated, and are reported in the solution purification report. .

Thickening and filtration tests were performed on various leach residues. As a majority of this type of test work was performed on leach pulps that had been treated using various solution purification schemes, the results of this work are presented under separate cover, in the solution purification report (Progress Report No. 2).



1.4. Physical Separation Tests

An extensive investigation of magnesite enrichment using physical separation techniques, particularly flotation, had been performed earlier by other investigators. Two additional physical separation methods were examined very briefly in this project.

Separations by Size Fraction:

The literature makes reference to industrial magnesite plants using a simple screening process to preferentially remove quartz from the plant feed. The silica apparently concentrates in the fine fraction, perhaps due to differential fracture mechanics.
A sample of MC36 that had been crushed to 100% minus 10 mesh was wet screened into 5 fractions, and each fraction was submitted for silica and magnesium analysis. The results are shown in Table No. 6

Table No. 6 – Mg & Si Distribution in MC36 Head by Size Fraction

Size Fraction	Weight (%)	Assay (%)		Distribution (%)	
		Mg	Si	Mg	Si
+ 65 mesh	65.8	27.2	0.58	66.2	51.2
- 65 +100	3.23	26.3	0.95	3.1	4.1
- 100 +200	4.42	25.7	1.21	4.2	7.2
- 200 + 400	3.57	25.6	1.89	3.4	9.0
- 400 mesh	23.0	27.1	0.92	23.1	28.4
Head (calc)	100	27.0	0.74	100.0	100.0

A concentration of silica occurs in the intermediate size fraction (100 to 400 mesh). The concentration of silica in the minus 200 plus 400 mesh fraction is 2.5 times higher then the head grade. However the degree of concentration of silica in any given fraction is not sufficiently high to suggest that this may be used as a means of silica rejection.

Magnetic Separation:

The MC36 material contains a magnetic component. A scoping type magnetic separation test was performed to determine if this method could be used to reduce the iron content in the feed prior to leaching. A sample was separated at 200 mesh, and the fine and coarse material treated separately. The full details for this test can be found in Appendix B.

The plus 200 mesh fraction was processed dry on a belt magnetic separator equipped with a permanent rare earth magnet (20,000 gauss). About 3.5 weight percent of the total feed was removed as a magnetic concentrate. A brief examination of this material indicated that it consisted of particles of biotite schist, magnetite, chalcopyrite, and locked particles of magnesite/dolomite with solid solution iron.

SUMMARY

The minus 200 mesh material was treated in a wet high intensity magnetic separator (approximately 20,000 gauss). The magnetic fraction represented about 0.7 weight percent of total feed. The weights and metal distributions are shown in Table No. 7

Table No. 7 - Metal Distribution in Mag Sep Products

Product	Wgt	Assay (%)			Distribution (%)		
	(g)	Mg	Fe	Ca	Mg	Fe	Ca
-200 Mag	1.6	25.6	2.67	1.07	0.74	2.8	0.6
-200 Non Mag	49.1	27.4	0.67	1.23	24.4	21.4	20.2
+ 200 Mag	7.4	26.7	1.41	1.74	3.58	6.8	4.3
+200 Non Mag	145.3	27.1	0.73	1.54	71.3	69.0	74.9
Head (calc)	203.4	27.1	0.76	1.47	100	100	100

The non-magnetic fractions show only about a 5% lowering of calcium and 10% lowering of iron levels in this magnetically purified fraction, whereas 4.3% of the magnesium is lost to the waste magnetic fraction. There is little concentration effect. The waste magnetic fractions represent 9% of the total weight, and contain only 9.6% of the total iron.

1.5 Mineralogical Results

The best magnesium extractions obtained from Sample MC 36 were in the range of 92 to 94.4%. A sample of pulverised leach residue was submitted for examination by x-ray diffraction analysis, to determine the mineral types present in the residue. The examination indicated the presence of magnesite and talc. The presence of talc in an HCl leach residue is not to be unexpected, as talc is less reactive in this system then magnesite.

A leach test was performed especially to produce a residue sample for further mineralogical examination. The leach conditions (Test No. 33) were as follows:

Sample:	MC36
Crush Size:	minus 10 mesh
Temperature:	80 °C
Leach Time:	60 minutes
Acid Addition:	110%

The residue contained both magnesite, and talc grains. There was some rimming of talc on magnesite grains, but this phenomenon was not extensive enough to explain the presence of the unleached magnesite. Many of the magnesite grains observed were liberated and their surfaces appeared "quite fresh" (no evidence of acid surface attack).

The full mineralogical report is presented in Appendix C

2.0 DISCUSSION of Results

2.1 Leaching

The maximum magnesium extractions from sample MC36 were in the range of about 90 to 94%. These results were obtained from the minus 10 mesh sample, using 110% of the stoichiometric acid addition, while leaching at 80 °C for 60 to 90 minutes. Increasing the leach time, the acid addition or the leach temperature did not improve the results beyond this level. Nor did increasing or decreasing the top crush size improve the magnesium extractions. It was also noted that excess free acid at the end of the leach had little effect on the final magnesium extraction. The use of a shorter leach time at a temperature of 80 °C or higher could be applicable and should be examined.

Mineralogical examination of the leach residue indicated the presence of both magnesite and talc. Talc is known to be refractory under the leach conditions employed[1]. The magnesite in the residue was generally "clean", with only moderate amounts having discontinuous rims of talc. The incomplete leaching of the magnesite present in the sample may be due to a combination of locking with talc, and with the depletion of the acid concentration of the leach solution.

The major impurities in the pregnant leach solution were calcium, iron and manganese. When the highest magnesium extractions were obtained the extraction of calcium was 98% or greater, while iron and manganese extractions were varied from 85 to 93%. The significance of these and other impurities is discussed further under separate cover, in Progress Report No.2.

As shown previously in Figure No. 3 it was possible to remove about 34% of the calcium from sample MC36, while only leaching 3% of the magnesium. The benefit gained by the early removal of some calcium from the process must be weighed against the added cost of an additional solid-liquid separation stage, and the treatment of the calcium rich solution produced. Current thinking favours the application of the Dow technology or other processes to remove the calcium at the solution purification stage, after magnesium leaching (see Progress Report No. 2).

The weight loss during the leach was typically 93 to 95%, leaving a leach residues similar in composition to the feed material, except depleted in calcium and carbonate and enriched in silica.

	Typical Residue	Feed
Mg (%)	23	26
Fe (%)	0.8	0.65
Ca (%)	0.1	1.5
LOI (%)	35	50

---

[1] Paper submitted for publication, M. Nagarmori and A. Plumpton

The two leaches performed on sample MC32 indicated that a magnesium extraction of 97% could be expected from this material, when it was leached at 80 °C for 60 minutes using a 110% stochiometric acid addition. The higher extraction of magnesium from this sample over MC36 may be due to ratio of magnesium to talc in the two ores. This should be clarified by performing a comparative mineralogical examination of the two samples.

One major difference in the response of MC32 over MC36 was the concentration of iron in the leach solution. For MC36 the iron was generally 1.5 to 2 g/l whereas for MC32 the iron concentration was 5 g/l. The calcium level was also slightly higher, being 6 g/l. Although the solutions from MC32 were significantly different than MC36, the solution purification procedures as developed and described in Progress Report No. 2 should be able to handle the observed variation.

2.2 Physical Separation Tests

As described in Section 4, the use of physical separation methods to "clean" the ore prior to leaching was very briefly examined. The rejection of silica based on its concentration in a given size fraction was suggested in the literature as a simple means of up-grading magnesite. This is obviously dependent on the deposit characteristics, and in this case the separation on the basis of size fraction was not appropriate.

The rejection of iron, or iron bearing minerals was suggested by an initial physical examination of the sample. The material contains a small proportion of magnetic material. However the results of the magnetic separation tests indicate that the iron is not only associated with the magnetic particles, but occurs in solid solution in magnesite/dolomite grains. Minimal elimination of iron or calcium occurred, rendering the method inappropriate in this case.

CONCLUSIONS AND RECOMMENDATIONS

3.0 Conclusions and Recommendations

The MC36 and MC32 samples leached very well using the HCl leach technique.

When the samples were crushed to 100% minus 10 mesh, and leached with 110% of the calculated stoichiometric quantity of HCl, for 60 minutes at 80 °C then 94% of the magnesium was extracted from sample MC36, and 97% from sample MC32. The acid requirements to achieve these extractions are;

2519 kg of 36% HCl per tonne of feed,

or on a 100% HCl basis 907 kg/t.

Increasing the acid addition, temperature, crush fineness, or leach time had no beneficial effect on the leaching of magnesium from MC36. Nor did increasing the reflux conditions or staged acid additions increase magnesium extraction. However the leach kinetics at higher temperatures warrant additional investigation, as it may be possible to use a leach time of less then 60 minutes.

Leach density was not examined in this test program. It should be examined and optimised in the next phase of the development test program.

During this testwork no attempt was made to measure the amount of HCl lost due to CO_2 off-gassing during the leach. The volume and rate of CO_2 evolved during the initial addition of the solids was very high, and probably carried with it some HCl, despite the use of reflux condensers during leaching. Calculated gas velocities were very high and may have taxed the capacity of the two in-series reflux condensers. The quantity of HCl lost from the leach in this manner should be minimised in further testwork. The gas flowrates, and acid content should also be determined so that appropriate methods may be designed to capture and re-use the acid in an operating plant.

Consideration should be given to stage leaching and counter-current leaching to optimise acid utilisation, and to reduce possible entrainment of solution or fine solids in the off-gasses. A counter-current leach system may also be conducive to the development of a talc leach process, which may be worthy of attention.

Only two samples were tested during this investigation. It is recommended that additional samples be tested to determine the variability of the ore body with respect to the proposed leach procedure.

APPENDIX A

TEST DETAILS

5398 - Golden Triangle Resources

MC36 Head Analysis

minus 10 mesh feed samples from Leach Tests 1 to 8

Element	T1	T2	T3	T4	T5	T6	T7	T8	T1 RA	T3 RA	T6 RA	T8 RA	Average of 8 Samples	Range (95% confid.)	
														Low	High
Mg [%]	26.8	27.0	26.5	26.9	27.1	27.0	27.1	26.9	26.0	26.9	27.2	27.1	26.9 ± 0.18	26.7	27.1
Ca [%]	1.49	1.42	1.44	1.53	1.53	1.46	1.47	1.51	1.50	1.44	1.47	1.51	1.48 ± 0.02	1.46	1.50
Si [%]	1.19	1.20	1.17	1.22	1.00	1.04	0.97	1.02	1.15	1.21	1.07	1.05	1.11 ± 0.05	1.06	1.16
Fe [%]	0.67	0.64	0.66	0.66	0.66	0.65	0.67	0.66	0.61	0.65	0.66	0.66	0.65 ± 0.01	0.65	0.66
Mn [%]	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.09					0.08 ± 0.00	0.08	0.08
Al [%]	0.02	< 0.01	0.02	0.02	0.02	< 0.01	0.02	< 0.01					0.02 ± 0.00	0.02	0.02
Cr [%]	0.01	0.01	< 0.01	0.01	0.01	0.01	0.01	0.01					0.01 ± 0.00	0.01	0.01
Na [%]	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					<0.05	n/a	n/a
K [%]	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01					<0.01	n/a	n/a
Ti [%]	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01					<0.01	n/a	n/a
P [%]	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01					<0.01	n/a	n/a
V [%]	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01					<0.01	n/a	n/a
LOI [%]	49.6	49.8	49.7	49.8	50.4	50.4	50.4	50.2					50.0 ± 0.22	49.8	50.3
SUM [Oxide %]	99.8	100.2	99.3	100.2	100.7	100.5	100.6	100.3					100.2 ± 0.30	99.9	100.5
Cl [g/t]	93	64	101	86	89	107	279	61					110 ± 45.5	64.5	155.5

Project: 5398
Test: LCH 01

Technologist: G. Toole
Date: Nov. 25, 1998

Purpose: To determine the amenability of MC-36 to HCl acid leaching for the extraction of Mg.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	549.6 g
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required). .
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	245	
30	81	322	frothing stopped after 20 min
60	82	354	
90	81	404	

** vs Ag/AgCl

Final preg pH -0.72 preg colour: yellow wash colour: clear

Results:

Product	Amount (ml, g)	Assay (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	495	97900	5170	2020	260	0.3	5.42	0.94	0.24	6.59	1.85
Wash	240	5600	359	128*	16*	0*	0*	0*	0*	0*	0*
Residue	14.9	23.2	0.1	0.78	0.07	...	...	...	<0.01	0.05	...
Head (cal)		26.6	1.33	0.57	0.07	...	...	...	0.0	0.0	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	91.0	96.2	87.2	89.9	...	...	...	7.4	30.2	...
Wash	2.5	3.2	2.7	2.8	...	...	...	0.2	0.9	...
Residue	6.5	0.6	10.1	7.3	...	...	...	92.4	68.9	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	35.5	999	11.6	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	<0.5 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	987 kg/t

Project: 5398
Test: LCH 02

Technologist: G. Toole
Date: Nov. 25, 1998

Purpose: To determine the effect of acid addition on Mg extraction from MC-36.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	503.8 g
Water added:	73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	238	
30	81	301	frothing stopped after 20 min
60	82	380	
90	81	426	

** vs Ag/AgCl

Final preg pH: 0 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	515	96700	4950	1950	253	0.21	4.98	0.83	0.23	5.87	1.65
Wash	210	6400	393	142*	18*	0*	0*	0*	0*	0*	0*
Residue	13.5	22.6	0.01	0.64	0.07	...	...	...	<0.01	0.05	...
Head (cal)		27.1	1.32	0.56	0.07	...	...	...	0.00	0.00	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	91.9	96.8	89.6	90.7	...	...	...	8.0	30.7	...
Wash	2.5	3.1	2.7	2.7	...	...	...	0.2	0.9	...
Residue	5.6	0.1	7.71	6.6	...	...	...	91.7	68.4	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	33.3	202	13.1	

Reagents:

HCl (36%) added:	503.8 g
HCl (100%) residual:	<0.2 g
HCl (100%) Addition:	907 kg/t
HCl (100%) Consumption:	906 kg/t

Project: **5398**
Test: **LCH 03**

Technologist: G. Toole
Date: Nov. 25, 1998

Purpose: To determine the effect of time on the extraction of Mg from MC-36

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 549.6 g
Water added: 34 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 120 %
Initial Percent Solids: 25.5 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	198	
15	81	267	frothing stopped
30	82	290	
45	81	338	
60	81	362	

** vs Ag/AgCl

Final preg pH: -0.89 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	510	93100	4910	1920	249	0.25	5.13	0.87	0.24	5.95	1.59
Wash	280	5340	349	123*	16*	0*	0*	0*	0*	0*	0*
Residue	14.7	22.9	0.1	0.77	0.07	...	...	...	<0.01	0.07	...
Head (cal)		26.2	1.31	0.56	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	90.7	95.7	86.9	89.6	...	...	...	7.7	22.6	...
Wash	2.9	3.7	3.1	3.2	...	...	...	0.3	0.8	...
Residue	6.4	0.6	10.0	7.3	...	...	...	92.1	76.6	...
	100.0	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay(%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				2.0
Residue	35.4	2748	11.8	

Reagents:

HCl (36%) added:	549.6	g
HCl (100%) residual:	1.0	g
HCl (100%) Addition:	360	kg/t
HCl (100%) Consumption:	358	kg/t

Project: 5398
Test: LCH 04

Technologist: G. Toole
Date: Nov. 25, 1998

Purpose: To determine the effect of acid on the extraction of Mg from MC-36.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	503.8 g
Water added:	73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	192	
15	81	285	frothing stopped
30	82	317	
45	81	333	
60	81	351	

** vs Ag/AgCl

Final preg pH: 1.26 preg colour: yellow wash colour: clear

5398
LCH 04

Metallurgical Balance:

Product	Amount (mL, g)	Assays (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	500	94500	4890	1860	244	0.21	4.87	0.72	0.22	4.99	1.49
Wash	240	6380	391	137*	18*	0*	0*	0*	0*	0*	0*
Residue	16.7	23.2	0.08	0.78	0.08	...	...	...	0.01	0.08	...
Head (cal)		26.3	1.28	0.55	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	89.7	95.8	85.1	87.3	...	...	...	6.2	15.6	...
Wash	2.9	3.7	3.0	3.1	...	...	...	0.2	0.6	...
Residue	7.4	0.5	11.9	9.6	...	...	...	93.6	83.8	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	36.3	375	10.9	

Reagents:

HCl (36%) added:	503.8 g
HCl (100%) residual:	0.5 g
HCl (100%) Addition:	907 kg/t
HCl (100%) Consumption:	904 kg/t

Project: 5398
Test: LCH 05

Technologist: G. Toole
Date: Nov. 26, 1998

Purpose: To determine the effect of temperature on the extraction of Mg from MC-36

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	549.6 g
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120 %
Initial Percent Solids:	25.5 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	50 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	48	206	
30	50	214	frothing stopped
60	50	271	
90	50	284	seemed to be still reacting reacting

** vs Ag/AgCl

Final preg pH: -1.82 preg colour: yellow wash colour: clear

5398
LCH 05

Results:

Product	Amount (mL, g)	Assay (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	580	77300	4920	1570	203	0.38	4.01	0.98	0.33	5.31	2.35
Wash	230	9190	643	196*	25*	0*	1*	0*	0*	1*	0*
Residue	49.3	26.7	0.08	0.05	0.09	...	...	...	0.01	0.05	...
Head (cal)		30.1	1.52	0.49	0.08	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	74.6	93.8	92.9	70.1	...	...	...	3.7	11.0	...
Wash	3.5	4.9	4.6	3.5	...	...	...	0.2	0.5	...
Residue	21.9	1.3	2.5	26.4	...	...	...	96.1	88.4	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				106
Residue	46.9	3739	3.73	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	61.5 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	682 kg/t

Project: 5398
Test: LCH 06

Technologist: G. Toole
Date: Nov. 26, 1998

Purpose: To determine the effect of acid addition at lower temperature.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	503.8 g
Water added:	73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	50 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	48	225	no apperent frothing
30	49	220	
60	50	240	
90	50	319	

** vs Ag/AgCl

Final preg pH: -1.64 preg colour: yellow wash colour: pale straw yellow

5398
LCH 06

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	510	68700	4810	1460	188	0.23	3.7	0.78	0.21	4.88	1.58
Wash	220	7800	579	171*	22*	0*	0*	0*	0*	1*	0*
Residue	55.6	27.0	0.07	0.68	0.08	...	...	...	0.01	0.04	...
Head (cal)		25.9	1.31	0.58	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	67.7	93.7	64.2	66.0	...	...	...	1.9	10.0	...
Wash	3.3	4.9	3.2	3.3	...	...	...	0.1	0.5	...
Residue	29.0	1.5	32.6	30.6	...	...	...	98.0	89.5	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				106
Residue	47.4	982	3.33	

Reagents:

HCl (36%) added:	503.8 g
HCl (100%) residual:	54.1 g
HCl (100%) Addition:	907 kg/t
HCl (100%) Consumption:	637 kg/t

Project: 5398
Test: LCH 07

Technologist: G. Toole
Date: Nov. 26, 1998

Purpose: To determine the effect of time at lower temperature.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	549.6 g
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120 %
Initial Percent Solids:	25.5 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	50 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	48	220	
15	49	224	
30	50	220	frothing stopped
45	50	227	
60	50	236	

** vs Ag/AgCl

Final preg pH: -1.58 preg colour: yellow wash colour: clear

Results:

Product	Amount (mL, g)	Assay (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	510	69600	4650	1420	184	0.23	3.7	0.78	0.26	4.91	1.42
Wash	250	5140	357	107*	14*	0*	0*	0*	0*	0*	0*
Residue	51.6	26.1	0.1	0.69	0.09	...	...	...	0.01	0.02	...
Head (cal)		25.1	1.26	0.55	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	70.6	94.4	65.4	65.3	...	...	...	2.5	19.4	...
Wash	2.6	3.6	2.4	2.4	...	...	...	0.1	0.7	...
Residue	26.8	2.1	32.2	32.3	...	...	...	97.4	79.9	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				110
Residue	48.5	15805	3.34	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	56.1 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	709 kg/t

Project: 5398
Test: LCH 08

Technologist: G. Toole
Date: Nov. 26, 1998

Purpose: To determine the effect of acid addition at lower temperature.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 503.8 g
Water added: 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.7 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 50 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	50	236	no apparent frothing
15	50	220	
30	50	223	
45	50	244	
60	50	250	

** vs Ag/AgCl

Final preg pH: -1.67 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	495	68100	4910	1360	184	0.24	3.56	0.66	0.28	4.69	1.31
Wash	250	9180	728	192*	26*	0*	1*	0*	0*	1*	0*
Residue	63.5	27.1	0.08	0.70	0.09	...	...	...	0.01	0.03	...
Head (cal)		26.6	1.33	0.58	0.08	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	63.3	91.3	57.7	58.9	...	...	...	2.1	10.8	...
Wash	4.3	6.8	4.1	4.2	...	...	...	0.2	0.8	...
Residue	32.3	1.9	38.1	36.9	...	...	...	97.7	88.5	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				116
Residue	47.8	709	2.99	

Reagents:

HCl (36%) added:	503.8	g
HCl (100%) residual:	57.4	g
HCl (100%) Addition:	907	kg/t
HCl (100%) Consumption	620	kg/t

Project: **5398**
Test: **LCH 09**

Technologist: G. Toole
Date: Nov. 27, 1998

Purpose: To determine the effect of crush size on Mg extraction.

Leach Conditions:

Leach Feed: MC36
Crush Size: motar and petastal to -28M
Feed weight: 200 g
HCl added: 549.6 g
Water added: 34 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 120 %
Initial Percent Solids: 25.5 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 90 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	236	
30	80	332	froth stopped after 18 min
60	80	354	
90	80	384	

** vs Ag/AgCl

Final preg pH -0.12 preg colour: yellow wash colour: clear

5398
LCH 09

Results:

Product	Amount (mL, g)	Assay (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	455	96100	5380	1920	254	0.48	8.45	5.63	0.4	4.52	1.27
Wash	225	9330	621	204*	27*	0*	1*	1*	0*	0*	0*
Residue	22.7	24.8	0.08	0.79	0.08	...	...	...	0.03	0.08	...
Head (cal)		25.7	1.30	0.55	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	85.0	93.9	79.5	82.7	...	...	...	2.6	10.1	...
Wash	4.1	5.4	4.2	4.3	...	...	...	0.1	0.5	...
Residue	10.9	0.7	16.3	13.0	...	...	...	97.3	89.3	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	40.5	603	8.02	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	0.5 g
HCl (100%) Addition:	989 kg/t
HCl (100%)Consumption:	987 kg/t

Project: **5398**
Test: **LCH 10**

Technologist: G. Toole
Date: Nov. 27, 1998

Purpose: To determine the effect of crush size on Mg extraction.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	motar and petastal to -28M
Feed weight:	200 g
HCl added:	549.6 g
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120 %
Initial Percent Solids:	25.5 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	120 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	235	
30	80	314	frothing stopped after 25 min
60	80	336	
90	80	379	
120	80	398	

** vs Ag/AgCl

Final preg pH 1.25 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	460	99200	5250	1930	255	0.33	7.94	5.73	0.39	4.19	1.21
Wash	260	8971	621	201*	27*	0*	1*	1*	0*	0*	0*
Residue	19.2	23.8	0.08	0.86	0.09	...	...	...	0.02	0.12	...
Head (cal)		26.3	1.30	0.55	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	86.9	93.2	80.3	82.9	...	...	...	4.5	7.7	...
Wash	4.4	6.2	4.7	4.9	...	...	...	0.3	0.5	...
Residue	8.7	0.6	14.9	12.2	...	...	...	95.3	91.9	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	38.2	731	9.63	

Reagents:

HCl (36%) added: 549.6 g
HCl (100%) residual: 0.5 g
HCl (100%) Addition: 989 kg/t
HCl (100%) Consumption: 987 kg/t

Project: 5398
Test: LCH 11

Technologist: G. Toole
Date: Nov. 30, 1998

Purpose: To determine calcium and magnesium solubility verses pH at 20°C

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	100 g
HCl added:	4.65 g
Water added:	250 g
HCl Concentration:	12 %
Initial Percent Solids:	28.2 %w/w
Initial L/S	2.54 ml sol'n / g solid
Temperature:	20 °C
Time:	30 min
pH:	3.0
Mixer Speed:	650 rpm

Procedure:

1. The sample was weighed into a 600mL beaker with 250mL of DI water and mixed
2. The intial pH was taken. The acid was added to the sample to the desired pH. Once pH was reached the test was started
3. The pH was maintained with constant additions of acid for 30 minutes
4. At this point the sample was filtered and washed
5. All products were assayed for Mg, Ca, and Fe.

Data:

initial pH	10.17
final pH	3.0
range throughout test	2.85-3.06

Results:

Product	Amount	Assay (mg/L, %)			Distribution (%)		
	(mL, g)	Mg	Ca	Fe	Mg	Ca	Fe
Preg	240	485	522	8.98	0.4	9.3	0.4
Wash	80	92.5	73.8	1.42	0.0	0.4	0.0
Residue	97.7	27.1	1.25	0.60	99.5	90.3	99.6
Head (cal)		26.6	1.35	0.59	100	100	100
Head (dir)	100	26.9	1.48	0.65			

Project: 5398
Test: LCH 12

Technologist: G. Toole
Date: Nov. 30, 1998

Purpose: To determine calcium and magnesium solubility verses pH at 20°C

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 100 g
HCl added: 16.48 g
Water added: 250 g
HCl Concentration: 12 %
Initial Percent Solids: 27.3 %w/w
Initial L/S 2.64 ml sol'n / g solid
Temperature: 20 °C
Time: 30 min
pH: 2.0
Mixer Speed: 650 rpm

Procedure:

1. The sample was weighed into a 600mL beaker with 250mL of DI water and mixed
2. The intial pH was taken. The acid was added to the sample to the desired pH. Once pH was reached the test was started
3. The pH was maintained with constant additions of acid for 30 minutes
4. At this point the sample was filtered and washed
5. All products were assayed for Mg, Ca, and Fe.

Data:
initial pH 10.06
final pH 2.0
range throughout test 1.93 - 2.07

Results:

Product	Amount	Assay (mg/L, %)			Distribution (%)		
	(mL, g)	Mg	Ca	Fe	Mg	Ca	Fe
Preg	245	1780	1170	44.8	1.6	21.6	1.8
Wash	110	238	159	3.9	0.1	1.3	0.1
Residue	95.5	27.6	1.07	0.61	98.3	77.1	98.1
Head (cal)		26.8	1.33	0.59	100	100	100
Head (dir)	100	26.9	1.48	0.65			

Project: 5398
Test: LCH 13

Technologist: G. Toole
Date: Nov. 30, 1998

Purpose: To determine calcium and magnesium solubility verses pH at 20°C

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	100 g
HCl added:	29.83 g
Water added:	250 g
HCl Concentration:	12 %
Initial Percent Solids:	26.3 %w/w
Initial L/S	2.75 ml sol'n / g solid
Temperature:	20 °C
Time:	30 min
pH:	1.0
Mixer Speed:	650 rpm

Procedure:

1. The sample was weighed into a 600mL beaker with 250mL of DI water and mixed
2. The intial pH was taken. The acid was added to the sample to the desired pH. Once pH was reached the test was started
3. The pH was maintained with constant additions of acid for 30 minutes
4. At this point the sample was filtered and washed
5. All products were assayed for Mg, Ca, and Fe.

Data: initial pH 10.02
final pH 1.0
range throughout test 0.98 - 1.03

Results:

Product	Amount	Assay (mg/L, %)			Distribution (%)		
	(mL, g)	Mg	Ca	Fe	Mg	Ca	Fe
Preg	260	2560	1650	63.9	2.5	31.4	2.7
Wash	110	430	311	9.64	0.2	2.5	0.2
Residue	94.7	27.6	0.96	0.63	97.3	66.1	97.1
Head(cal)		26.9	1.37	0.61	100	100	100
Head(dir)	100	26.9	1.48	0.65			

Project: **5398**
Test: **LCH 14**

Technologist: G. Toole
Date: Dec. 01, 1998

Purpose: To determine Mg leach kinetics at 50 °C with 120% acid addition.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 549.6 g
Water added: 34 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 120 %
Initial Percent Solids: 25.5 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 50 °C
Time: 240 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. Kinetic samples were taken during the test at 1, 2, 3 hours
7. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
8. The final solution volumes, and residue dry weight were measured.
9. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	49	195	
30	50	200	
60	50	212	
90	50	252	
120	50	272	
150	50	299	
180	50	311	
210	50	332	
240	50	349	

** vs Ag/AgCl

Final preg pH: -1.61 preg colour: orangy yellow wash colour: straw yellow

5398
LCH 14

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
1hr preg	21	64900	4850	...	...	...	...	...	...	...	...
2hr preg	22	76900	4770	...	...	...	...	...	...	...	...
3hr preg	23	79000	4650	...	...	...	...	...	...	...	...
Preg	440	81200	4540	1670	138	<0.1	4.82	0.7	0.24	6.49	0.13
Wash	260	8525	550	189*	0*	0*	0*	0*	0*	0*	0*
Residue	21.7	24.8	0.08	0.76	0.08	...	...	...	0.01	0.07	...
Head (cal)		24.1	1.24	0.47	0.04	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
1hr preg	2.8	4.12	...	...	...	...	...	...	...	...
2hr preg	3.5	4.25	...	...	...	...	...	...	...	...
3hr preg	3.8	4.33	...	...	...	...	...	...	...	...
Preg	74.1	80.8	77.4	77.8	...	...	...	4.6	15.8	...
Wash	4.6	5.8	5.2	0.0	...	...	...	0.0	0.0	...
Residue	11.2	0.7	17.4	22.2	...	...	...	95.4	84.2	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				60
Residue	39.9	402	8.51	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	26.4 g
HCl (100%) Addition:	989 kg/t
HCl Consumption:	857 kg/t

Project: 5398
Test: LCH 15

Technologist: G. Toole
Date: Dec. 2, 1998

Purpose: To examine the effect of using a low boron silicate glass beaker (repeat of LCH02)

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 50 g
HCl added: 106 g
Water added: 18 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 28.7 %w/w
Initial L/S 2.14 ml sol'n / g solid
Temperature: 80 °C
Time: 90 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A low boron glass beaker without a lid was used for the test. No condenser was used.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	320	
30	80	350	frothing stopped after 20 min
60	80	305	
90	80	377	

** vs Ag/AgCl

Final preg pH: 0.71 preg colour: yellow wash colour: clear

5398
LCH 15

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	120	72200	4900	1500	198	0.21	7.57	0.72	0.2	5.24	0.45
Wash	90	8610	649	189*	25*	0*	1*	0*	0*	1*	0*
Residue	11	26.6	0.09	0.74	0.08	...	...	...	<0.01	0.02	...
Head (cal)		24.7	1.31	0.56	0.07	...	...	...	0.00	0.01	...
Head (dir)	50	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	70.1	89.6	64.7	68.3	...	...	...	2.1	21.8	...
Wash	6.3	8.9	6.1	6.4	...	...	...	0.2	2.1	...
Residue	23.7	1.5	29.2	25.3	...	...	...	97.7	76.2	...
	100.0	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<0.1
Residue	46	1192	4.19	

Reagents:

HCl (36%) added: 106 g
HCl (100%) residual: 0.01 g
HCl (100%) Addition: 763 kg/t
HCl (100%) Consumption: 763 kg/t

Project: 5398
Test: LCH 16

Technologist: G. Toole
Date: Dec. 2, 1998

Purpose: To determine the effect of temperature and crush size on Mg extraction.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 6 mesh
Feed weight:	200 g
HCl added:	503.6 g
Water added:	73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.8 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	95 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	1000 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	92	228	
15	94	306	frothing stopped after 10 min
30	95	375	
45	95	386	
60	95	398	filtered quickly(~2min)

** vs Ag/AgCl

Final preg pH: 1.54 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	455	91100	5450	1890	249	0.3	16.9	1.73	0.24	2.51	0.54
Wash	230	6390	440	143*	19*	0*	1*	0*	0*	0*	0*
Residue	32.8	26.2	0.1	0.75	0.09	...	...	...	<0.01	0.06	...
Head (cal)		25.8	1.31	0.57	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	80.5	94.9	75.5	77.0	...	...	...	3.2	5.5	...
Wash	2.9	3.9	2.9	2.9	...	...	...	0.1	0.2	...
Residue	16.7	1.3	21.6	20.1	...	...	...	96.7	94.3	...
	100.0	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<0.1
Residue	43.7	305	5.61	

Reagents:

HCl (36%) added: 503.6 g
HCl (100%) residual: 0.05 g
HCl (100%) Addition: 906 kg/t
HCl (100%) Consumption: 906 kg/t

Project: 5398 **Technologist:** G. Toole
Test: LCH 17 **Date:** Dec. 2, 1998

Purpose: To determine the effect of temperature and crush size on Mg extraction.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 3 mesh
Feed weight: 200 g
HCl added: 503.6 g
Water added: 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.8 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 95 °C
Time: 60 min
pH: not controlled
Mixer Speed: 1000 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	95	238	
15	95	268	frothing stopped after 10 min
30	95	311	
45	95	330	
60	95	354	filtered quickly(~2min)

** vs Ag/AgCl

Final preg pH: 1.66 preg colour: yellow wash colour: clear

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	475	91100	5150	1850	246	0.19	8.26	0.31	0.22	1.44	0.54
Wash	250	6160	420	138*	18*	0*	1*	0*	0*	0*	0*
Residue	27	25.9	0.14	0.68	0.08	...	...	...	0.01	0.03	...
Head (cal)		25.9	1.29	0.55	0.07	...	...	...	0.00	0.00	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	83.5	94.5	80.1	81.7	...	...	...	3.7	7.8	...
Wash	3.0	4.1	3.1	3.2	...	...	...	0.1	0.3	...
Residue	13.5	1.5	16.7	15.1	...	...	...	96.1	91.9	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<0.1
Residue	42.8	512	6.33	

Reagents:

HCl (36%) added: 503.6 g
HCl (100%) residual: 0.05 g
HCl (100%) Addition: 906 kg/t
HCl (100%) Consumption: 906 kg/t

5398
Test 18

This test may be found in Progress Report No. 2 - Solution Purification

5398
Test 19

This test may be found in Progress Report No. 2 - Solution Purification

5398
Test 20

This test may be found in Progress Report No. 2 - Solution Purification

Project: 5398
Test: LCH 21

Technologist: G. Toole
Date: Dec. 10, 1998

Purpose: To determine the effect of leaching at 95 °C on extraction of Mg.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 549.6 g
Water added: 34 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 120 %
Initial Percent Solids: 25.5 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 90-95 °C
Time: 120 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time min	Temp °C	emf mV	Comments
0	90	256	
30	91	352	Frothing stopped after 10 min
60	90	395	filters within 1 minute

** vs Ag/AgCl

Final preg pH: preg colour: yellow wash colour: clear
preg SG (g/mL): 1.267

Results:

Product	Amount (mL, g)	Assay (mg/L, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	480	93581**	6370	2230	288	0.43	6.25	0.92	<0.10	6.27	<0.10
Wash	210	9540	540	208*	27*	0*	1*	0*	0*	1*	0*
Residue	27.4	25.1	0.09	0.77	0.09	...	...	...	0.01	0.05	...
Head (cal)		26.9	1.60	0.66	0.08	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.01	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

**original assay too high - calculated from head

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	83.5	95.7	80.8	82.0	...	...	...	1.7	17.9	...
Wash	3.7	3.5	3.3	3.3	...	...	...	0.1	0.7	...
Residue	12.8	0.8	15.9	14.6	...	...	...	98.2	81.4	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<0.1
Residue	41.6	486	27.4	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	0.05 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	989 kg/t

Project: 5398
Test: LCH 22

Technologist: G. Toole
Date: Dec. 9, 1998

Purpose: To determine the effect of time on Mg extraction, when leaching at 95 °C.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	549.6 g
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120 %
Initial Percent Solids:	25.5 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	90-95 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	90	252	
30	92	339	frothing stopped after 10 minutes
60	93	384	
90	93	401	filters within 1 minute

** vs Ag/AgCl

Final preg pH: 1.12 preg colour: yellow wash colour: clear
preg SG (g/mL): 1.259

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LCH 22

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	460	89400	5290	1680	221	<0.10	8.28	0.97	<0.10	5.56	<0.10
Wash	230	8450	551	167*	22*	0*	1*	0*	0*	1*	0*
Residue	36.9	26.3	0.13	0.8	0.09	...	...	...	0.01	0.06	...
Head (cal)		26.4	1.30	0.55	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	77.9	93.3	69.8	72.7	...	...	...	1.2	10.3	...
Wash	3.7	4.9	3.5	3.6	...	...	...	0.1	0.5	...
Residue	18.4	1.8	26.7	23.7	...	...	...	98.7	89.2	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<0.1
Residue	44.1	570	5.3	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	0.05 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	989 kg/t

Project:	**5398**	**Technologist:**	G. Toole
Test:	**LCH 23**	**Date:**	Dec. 8, 1998

Purpose: To determine the effect of a two stage acid addition on Mg extraction.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	549.6 g
Additonal HCl added after 60 minutes:	45.8
Water added:	34 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	120% at time zero, and 10% after 60 minutes
Initial Percent Solids:	25.5 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	90-95 °C
Time:	90 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	90	245	
30	90	344	
60	90	385 / 357	45.8g conc. HCl added
75	90	372	
90	90	370	

** vs Ag/AgCl

Final preg pH: -1.74 preg colour: yellow wash colour: clear

preg SG (g/mL): 1.2557

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	540	86100	4560	1730	223	<0.10	5.68	0.89	<0.10	8.46	<0.10
Wash	230	7510	417	155*	20*	0*	1*	0*	0*	1*	0*
Residue	13.4	21.8	0.13	0.81	0.07	...	...	...	<0.01	0.12	...
Head (cal)		25.6	1.29	0.54	0.07	...	...	...	0.00	0.01	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	90.9	95.6	86.6	89.6	...	...	...	3.9	21.9	...
Wash	3.4	3.7	3.3	3.4	...	...	...	0.1	0.8	...
Residue	5.7	0.7	10.1	7.0	...	...	...	96.0	77.2	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				5.2
Residue	32.4	1949	13.6	

Reagents:

HCl (36%) added:	549.6	g
HCl (100%) residual:	2.81	g
HCl (100%) Addition:	989	kg/t
HCl (100%) Consumption:	975	kg/t

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Test 24

This test may be found in Progress Report No. 2 - Solution Purification

Project: 5398
Test: LCH 25

Technologist: G. Toole
Date: Dec. 11, 1998

Purpose: To determine the amenability of MC-32 to HCl acid leaching for the extraction of Mg.

Leach Conditions:

Leach Feed:	MC32
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	496 g
Water added:	80 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.8 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	208	not as much frothing as MC36 sample
30	80	286	
60	80	304	

** vs Ag/AgCl

Final preg pH: -0.88 preg colour: yellow wash colour: clear
Solution SG: 1.286 g/L

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LCH 25

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	480	100000	5950	5040	209	1.34	6.59	0.61	0.1	10.2	<0.10
Wash	235	6470	389	328*	14*	0*	0*	0*	0*	1*	0*
Residue	12.6	17.5	0.08	3.48	0.08	...	...	...	0.02	0.27	...
Head (cal)		25.9	1.48	1.47	0.06	...	...	...	0.0	0.0	...
Head (dir)	200	26.2	1.51	1.62	0.07	<0.00	0.003	<0.00	<0.01	0.02	<20 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	92.8	96.6	82.4	88.3	...	...	...	1.9	12.5	...
Wash	2.9	3.1	2.6	2.8	...	...	...	0.1	0.4	...
Residue	4.3	0.3	14.9	8.9	...	...	...	98.1	87.1	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				2.0
Residue	33.8	1080	14.6	

Reagents:

HCl (36%) added:	495.7 g
HCl (100%) residual:	0.96 g
HCl (100%) Addition:	892 kg/t
HCl (100%) Consumption:	887 kg/t

Project: 5398
Test: LCH 26

Technologist: G. Toole
Date: Dec. 11, 1998

Purpose: To determine time on the extraction of Mg from MC32.

Leach Conditions:

Leach Feed:	MC32
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	495.7 g
Water added:	80 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.8 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	120 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
7. The final solution volumes, and residue dry weight were measured.
8. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	214	not as much frothing as MC36 sample
30	80	276	
60	80	293	
90	80	321	
120	80	367	

** vs Ag/AgCl

Final preg pH: -0.77 preg colour: yellow wash colour: clear
Solution SG: 1.2902

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LCH 26

Results:

Product	Amount	Assay (mg/L, %)									
	(mL, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	470	103000	6070	5320	216	0.58	7.91	0.61	0.14	10.2	<0.10
Wash	230	7980	471	412*	17*	0*	1*	0*	0*	1*	0*
Residue	9.0	16	0.07	3.88	0.08	...	...	...	0.02	0.32	...
Head (cal)		25.8	1.48	1.47	0.06	...	...	...	0.00	0.017	...
Head (dir)	200	26.2	1.51	1.62	0.07	<0.00	0.003	<0.00	<0.01	0.02	<20 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	93.7	96.1	84.9	90.2	...	...	...	3.5	14.2	...
Wash	3.6	3.7	3.2	3.4	...	...	...	0.1	0.5	...
Residue	2.8	0.2	11.9	6.4	...	...	...	96.3	85.3	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				<1.0
Residue	31.7	615	16.1	

Reagents:

HCl (36%) added:	495.7 g
HCl (100%) residual:	0.47 g
HCl (100%) Addition:	360 kg/t
HCl (100%) Consumption:	359 kg/t

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Test 27

This test may be found in Progress Report No. 2 - Solution Purification

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Test 28

This test may be found in Progress Report No. 2 - Solution Purification

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Test 29

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Test 30

This test may be found in Progress Report No. 2 - Solution Purification

Project: 5398
Test: LCH 31

Technologist: G. Toole
Date: Dec. 22, 1998

Purpose: To determine the effect of additional acid and increased reflux condensing on the extraction of Mg from MC36

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 549.6 g
Additional HCl added after 60 minutes: 45.8
Water added: 34 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 130 %
Initial Percent Solids: 25.5 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 90-95 °C
Time: 90 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. The condenser consisted of 2 condensers on top of one another. Condenser water flow was set at 2650 mL/min
3. Hydrochloric acid was added to the reactor and heated to the desired temperature.
4. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
5. Once all the sample was in the test was started.
6. Periodic redox readings were taken during the test.
7. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
8. The final solution volumes, and residue dry weight were measured.
9. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	90	241	
30	90	316	
60	91	74/35	45.8g conc. HCl
90	91	372	filter ~4 minutes

** vs Ag/AgCl

Final preg pH: -1.13 preg colour: yellow wash colour: clear
preg SG (g/mL): 1.2656

Results:

Product	Amount	Assay (mg/L, %)									
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	520	99000	5460	1760	224	0.32	5.61	0.81	<0.01	8.16	<0.01
Wash	155	11600	641	206*	26.3*	0.0*	0.7*	0.1*	0.0*	1.0*	0.0*
Residue	15	22.3	0.1	0.84	0.07	...	...	...	0.01	0.36	...
Head (cal)		28.3	1.48	0.54	0.07	...	...	...	0.00	0.03	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	90.9	96.1	85.3	88.9	...	...	...	0.3	7.3	...
Wash	3.2	3.4	3.0	3.1	...	...	...	0.0	0.3	...
Residue	5.9	0.5	11.7	8.0	...	...	...	99.7	92.5	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				7.0
Residue	34.5	2801	12.2	

Reagents:

HCl (36%) added:	549.6 g
HCl (100%) residual:	3.64 g
HCl (100%) Addition:	989 kg/t
HCl (100%) Consumption:	971 kg/t

Project: 5398
Test: LCH 32

Technologist: G. Toole
Date: Dec. 23, 1998

Purpose: To determine the effect of increased reflux condensing with 110% acid addition.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight:	200 g
HCl added:	503.8 g
Water added:	73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. The condenser consisted of 2 condensers on top of one another. Condenser water flow was set at 2100 mL/min
3. Hydrochloric acid was added to the reactor and heated to the desired temperature.
4. Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
5. Once all the sample was in the test was started.
6. Periodic redox readings were taken during the test.
7. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes of D.I. water.
8. The final solution volumes, and residue dry weight were measured.
9. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	249	
30	80	305	frothing ceased after 10 min
60	81	342	filter form ~5min

** vs Ag/AgCl

Final preg pH: -1.40 preg colour: yellow wash colour: clear
Sloution SG: 1.274 g/mL

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LCH 32

Results:

Product	Amount	Assay (mg/L, %)									
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	510	97600	5530	1880	242	0.65	5.45	0.71	0.1	10	<0.1
Wash	205	7280	421	142*	18*	0*	0*	0*	0*	1*	0*
Residue	16.4	23.3	0.09	0.79	0.08	...	...	...	0.01	0.13	...
Head (cal)		27.5	1.46	0.56	0.07	...	...	...	0.0	0.0	...
Head (dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Product	Distribution (%)									
	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	90.4	96.5	85.8	88.0	...	...	...	3.0	19.2	...
Wash	2.7	3.0	2.6	2.7	...	...	...	0.0	0.6	...
Residue	6.9	0.5	11.6	9.4	...	...	...	97.0	80.2	...
	100	100	100	100	...	...	...	100	100	...

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				15.0
Residue	35.6	3438	11.5	

Reagents:

HCl (36%) added:	503.8 g
HCl (100%) residual:	7.65 g
HCl (100%) Addition:	907 kg/t
HCl (100%) Consumption:	869 kg/t

Project: 5398
Test: LCH 33

Technologist: G. Toole
Date: Dec. 23, 1998

Purpose: To produce a MgCl2 solution for further testwork
To produce residue for mineralogical examination.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight: 200 g
HCl added: 504 g
Water added: 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.7 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
2. The condenser consisted of 2 condensers on top of one another.
   Condenser water flow was set at 2100 mL/min
3. Hydrochloric acid was added to the reactor and heated to the desired temperature.
4. Once at temperature the sample was added slowly to avoid sample loss
   due to excessive frothing (3 to 5 minutes typically required).
5. Once all the sample was in the test was started.
6. Periodic redox readings were taken during the test.
7. At the conclusion of the test the sample was filtered and washed with ~ 2 bed volumes
   of D.I. water.
8. The final solution volumes, and residue dry weight were measured.
9. Samples of the tests solutions and residue were submitted for chemical analysis.

Data:

Time (min)	Temp (°C)	emf (mV)**	Comments
0	80	242	
30	80	302	frothing ceased after 10 min
60	81	342	filter form ~5min

** vs Ag/AgCl

Final preg pH: -1.66 preg colour: yellow wash colour: clear
Sloution SG: 1.283 g/mL

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LCH 33

Results:

Product	Amount (ml, g)	Assay (mg/l, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	B
Preg	490	100000	5610	1960	252	<0.01	4.7	2.0	<0.01	8.66	<0.10
Wash	240	8310	446	159*	20*	0*	0*	0*	0*	1*	0*
Residue	15.3	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Head (cal)											
Head(dir)	200	26.9	1.48	0.65	0.08	<0.00	0.008	0.002	0.01	0.02	27 g/t

* estimated based on ratio of Mg and Ca in Preg and Wash

Additional Assays:

Product	Assay (%, g/L)			
	LOI	Cl	Si	Free Acid
Preg				12.0
Residue				

Reagents:

HCl (36%) added:	503.8	g
HCl (100%) residual:	5.88	g
HCl (100%) Addition:	907	kg/t
HCl (100%) Consumption:	877	kg/t

827091

APPENDIX B

MAGNETIC SEPARATION

827092

LAKEFIELD
RESEARCH

Mineralogical Services

Magnetic Separation and Mineralogical Analysis of MC-36 Magnesite

Submitted By:
Golden Triangle Resources

Report by:	**Geoff Lane, Project Mineralogist** **Bruce Jago Ph.D., Senior Mineralogist**
Submission Date:	**December 9, 1998**
Project No:	**5398 LIMS#Nov3601**

Summary Report

One sample identified as MC-36 was submitted to Mineralogical Services, Lakefield Research Limited, for magnetic separation to remove iron contaminants. Further investigation was carried out to identify the composition of the magnesite grains reporting to the magnetic fraction.

Procedure

The sample was dry screened at 200 mesh so that the two fractions could be treated separately. The +200 mesh fraction was submitted for a Dry High Intensity Magnetic Separation (DHIMS) using the permanent roll magnetic separator developed by International Process Systems. This separation was carried out at a field strength of approximately 20,000 gauss.

The –200 mesh fraction was submitted for a Wet High Intensity Magnetic Separation (WHIMS) using the WHIMS manufactured by Eriez. This separation was also carried out at a field strength of approximately 20,000 gauss. All products from both separations were submitted for whole rock analysis. The materials distribution for iron, calcium and magnesium are presented in Tables 1 to 3.

After the +200 mesh fraction was separated the magnetic fraction was observed to contain a large quantity of magnesite. A portion of these grains and a selection of the mafic and oxide minerals that reported to the magnetic fraction were selected for analysis using the Jeol E5C Scanning Electron Microscope equipped with a Tracor Northern Energy Dispersive X-ray Analyzer (SEM-EDX).

Results

Table 1: Iron Distribution

Sample ID	**Weight**			**Assay Fe**		**% Distribution**	
	wt. (g)	**% Individual**	**% Cumulative**	**% Individual**	**% Cumulative**	**Fe Individual**	**Fe Cumulative**
-200 mesh Mag	1.6	0.76	0.76	2.67	2.67	2.7	2.7
-200 mesh Non-Mag	49.1	24.15	24.92	0.67	0.73	21.4	24.1
+200 mesh Mag	7.4	3.62	28.54	1.41	0.82	6.7	30.8
+200 mesh Non-Mag	145.3	71.46	100.00	0.73	0.75	69.2	100.0
Head (Calc.)	**203.3**	**100.00**		**0.75**		**100.0**	

Table 2: Magnesium Distribution

Sample ID	Weight			Assay Mg		% Distribution	
	wt. (g)	**% Individual**	**% Cumulative**	**% Individual**	**% Cumulative**	**Mg Individual**	**Mg Cumulative**
-200 mesh Mag	1.6	0.76	0.76	25.63	25.63	0.7	0.7
-200 mesh Non-Mag	49.1	24.15	24.92	27.38	27.33	24.4	25.1
+200 mesh Mag	7.4	3.62	28.54	26.66	27.24	3.6	28.7
+200 mesh Non-Mag	145.3	71.46	100.00	27.08	27.13	71.3	100.0
Head (Calc.)	**203.3**	**100.00**		**27.13**		**100.0**	

Table 3: Calcium Distibution

Sample ID	Weight			Assay Ca		% Distribution	
	wt. (g)	**% Individual**	**% Cumulative**	**% Individual**	**% Cumulative**	**Ca Individual**	**Ca Cumulative**
-200 mesh Mag	1.6	0.76	0.76	1.07	1.07	0.6	0.6
-200 mesh Non-Mag	49.1	24.15	24.92	1.23	1.22	20.3	20.8
+200 mesh Mag	7.4	3.62	28.54	1.74	1.29	4.3	25.1
+200 mesh Non-Mag	145.3	71.46	100.00	1.54	1.47	74.9	100.0
Head (Calc.)	**203.3**	**100.00**		**1.47**		**100.0**	

The materials distribution in Table 1 indicates that an iron bearing phase is still present in the +200 mesh and –200 mesh non-magnetic size fractions.

The magnetic fraction was determined to contain mainly impure magnesite grains. SEM-EDX analysis (Figures 1, 2 and 3) shows that the magnesite can contain minor to trace amounts of iron and calcium. Since magnesite occurs in a solid solution series with siderite [$FeCO_3$], dolomite [$Ca(Mg,Fe)(CO_3)_2$] and calcite [$CaCO_3$], iron and calcium may be stoichiometrically bound rather than occurring as discrete iron and calcium rich phases. This fraction also contains minor to moderate amounts of magnetite, garnet and biotite, which was intergrown with fine-grained chalcopyrite and quartz.

827095

Figures 1, 2 and 3 show the X-ray spectra obtained from a single magnesite grain. They show that iron and calcium may not be evenly distributed on a millimeter scale. This suggests that unmixing of discrete iron and calcium rich phases may have occurred, rather than iron and calcium being bound stoichiometrically. This is suggested by the peak intensities of calcium and magnesium in Figure 3. If the X-ray beam were to impinge on dolomite, the calcium and magnesium peaks would have similar peak strengths. However, the greater strength of the calcium peak suggests that the X-ray beam unevenly overlapped the contact between a discrete calcium bearing phase, such as calcite and a magnesium bearing host, such as magnesite.

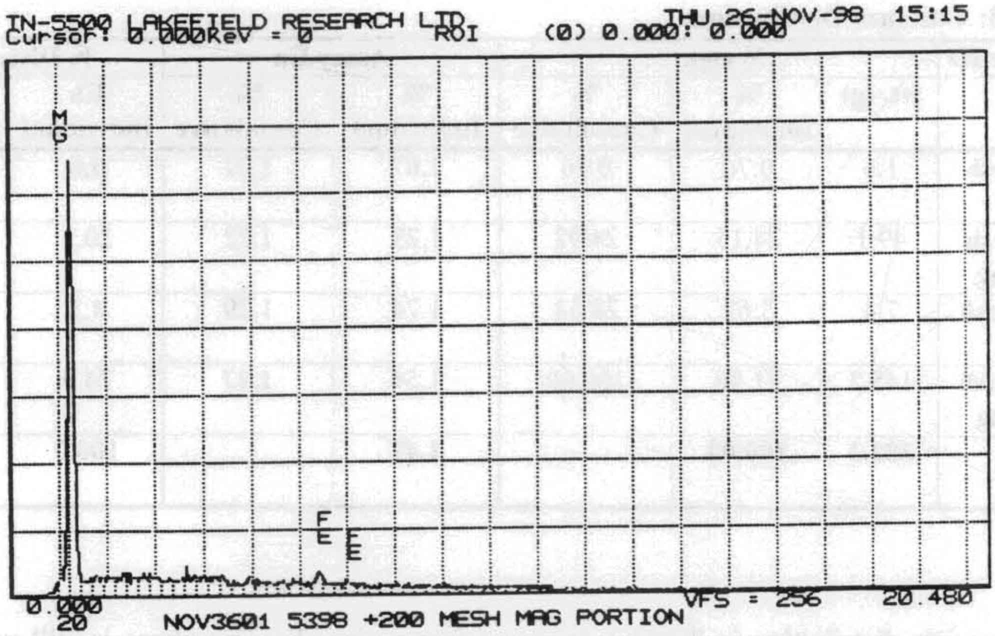
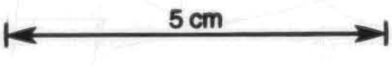


Figure 1: SEM-EDX spectra, 0-20keV, 30second acquire time, 25kv beam. Portion of a magnesite grain showing a high magnesium peak and a small iron peak.



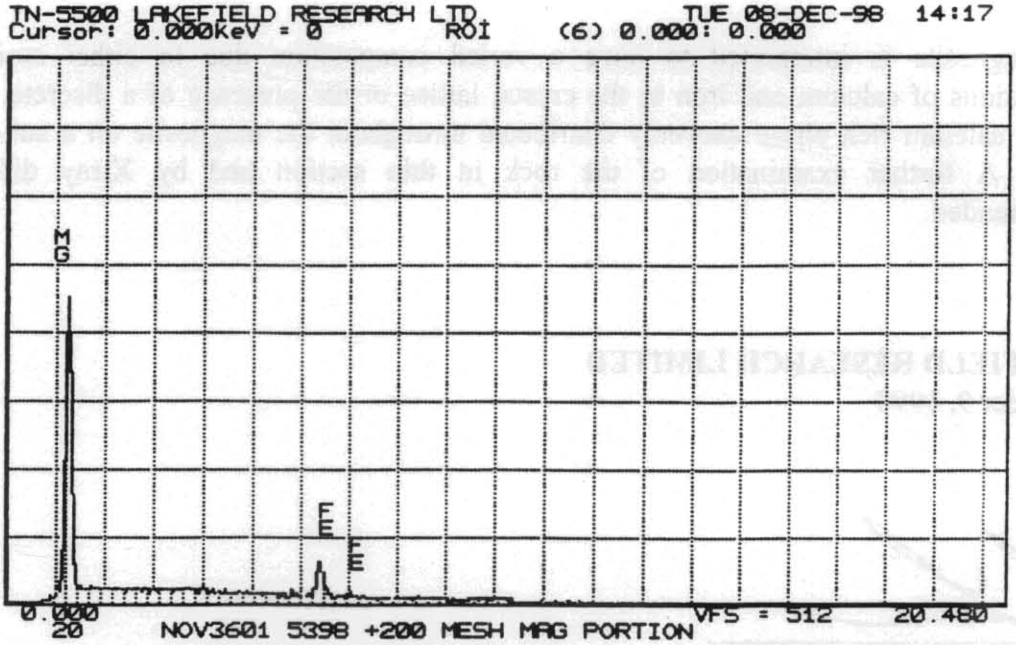


Figure 2: SEM-EDX spectra, 0-20keV, 30second acquire time, 25kv beam. Portion of the same magnesite grain from Figure 1, above, showing a high magnesium peak and a moderate iron peak.

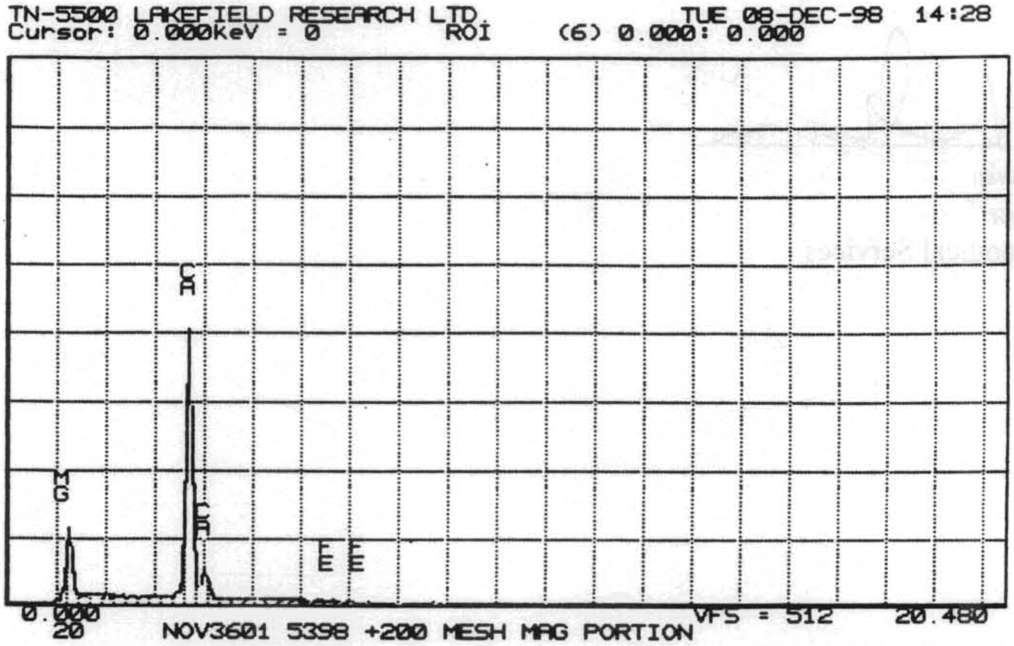


Figure 3: SEM-EDX spectra, 0-20keV, 30second acquire time, 25kv beam. Portion of the same magnesite grain as Figures 1 and 2 showing a strong calcium peak and a moderate magnesium peak.

5 cm

Remarks

The magnesite is interpreted to have a varied composition due to either stoichiometric substitutions of calcium and iron in the crystal lattice or the presence of a discrete, very fine-grained calcium rich phase unevenly distributed throughout the magnesite on a sub-millimeter scale. A further examination of the rock in thin section and by X-ray diffraction is recommended.

LAKEFIELD RESEARCH LIMITED
December 9, 1998

Geoff Lane
Project Mineralogist

Bob Irwin
Manager
Mineralogical Services

827098

APPENDIX C

MINEROLOGY

March 1999 *Lakefield Research Limited*

827099

LAKEFIELD
RESEARCH

Mineralogical Services

Mineralogical Analysis of a Magnesite Leach Residue

Submitted by:
Golden Triangle Resources N.L.

Report by: **Geoff Lane, Project Mineralogist**

Submission Date: **January 13, 1999**

Project No: **5398 LIMS#Jan6000.R99**

Note

This report refers to the samples as received. The practice of this Company in issuing reports of this nature is to require the recipient not to publish the report or any part therof without the written consent of Lakefield Research Limited.

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Summary

One sample identified as Leach Residue was submitted to Mineralogical Services, Lakefield Research Limited, for mineralogical analysis. The purpose was to determine the textural relationship between magnesite and talc, and explain the reason why some magnesite remained un-leached.

The sample is primarily composed of liberated magnesite, with moderate amounts having discontinuous rims of talc. Talc was observed in the sample in minor amounts as fine (<50 to 150 µm), liberated grains, fine discontinuous rims on magnesite, as larger composite grains with quartz, and as talc aggregates. Minor amounts of quartz and trace amounts of pyrite, chalcopyrite, and ilmenite were also noted.

Procedure

One polished thin section was prepared and was systematically scanned using a petrographic microscope at 160x magnification in transmitted light. Further analysis was carried out with the Jeol 35C Scanning Electron Microscope equipped with a Tracor Northern Energy Dispersive X-ray Analyzer (SEM-EDX) to determine if any elemental unconformity was characteristic between the magnesite grains that would prohibit leaching.

Results

Eighty to ninety percent of the magnesite grains were subhedral to euhedral, ranging in size from 40 µm to 300 µm and were free of any intergrowths. Moderate amounts (10-20%) of the magnesite occurred with fine discontinuous talc rims. Most of the magnesite grains showed little to no effect from leaching and appeared quite fresh (Figure 1).

Talc occurred as discrete liberated grains, and as rims on some magnesite and quartz grains. Talc rims on magnesite are discontinuous and range in thickness from 10 µm to 100 µm. Figure 2 illustrates the rimming of the talc on the magnesite. In this illustration, talc is not continuous around the grain, therefore leaving enough exposed magnesite to be leached. Scarce occurrences of magnesite had more continuous rims covering 75% of the circumference of the grain (Figure 3). Talc also occurred in minor amounts as large composite grains with quartz or as large talc aggregates (Figure 4).

Pyrite occurred in trace amounts as liberated grains, as attachments and inclusions in magnesite. Trace amounts of chalcopyrite and ilmenite were observed as liberated grains, and as attachments to pyrite.

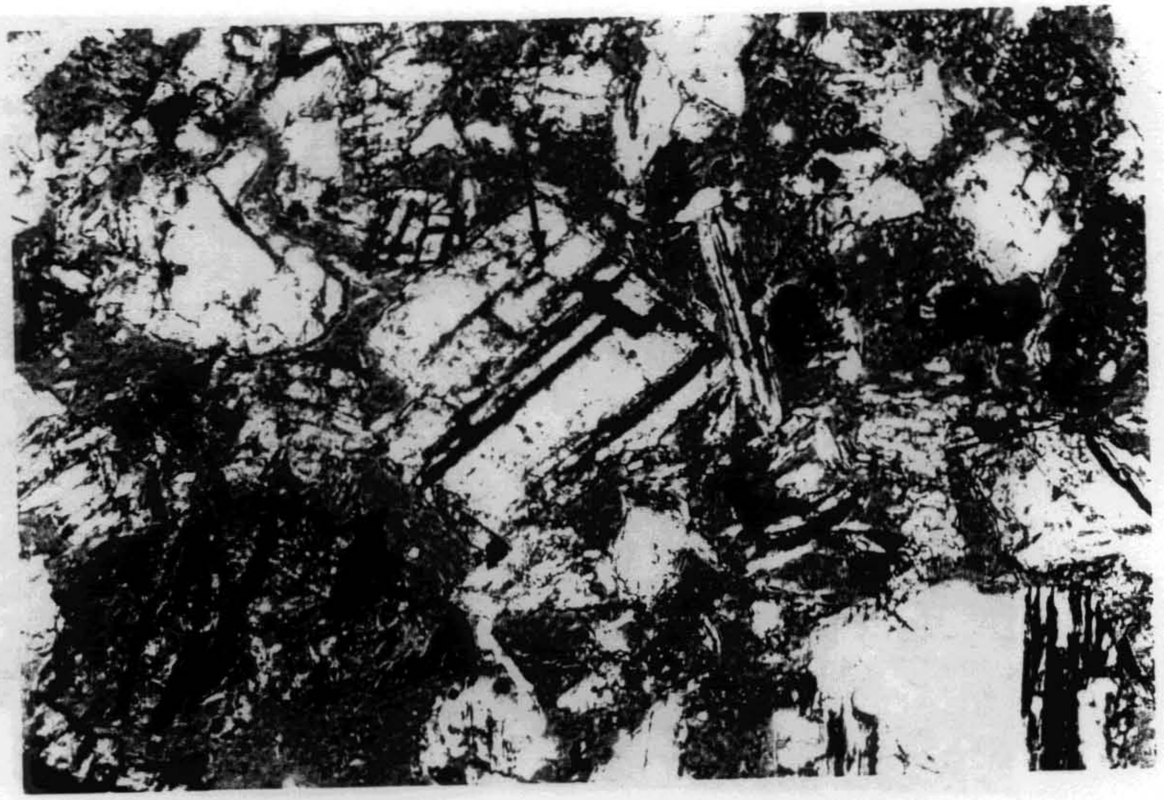


Figure 1: **Leach Residue** PTS5167
Magnification 160x Transmitted Cross Polarized Light
Liberated euhedral magnesite (black arrow) without talc rimming.
A liberated talc grain is viewed to right of the magnesite grain (red arrow).

5 cm

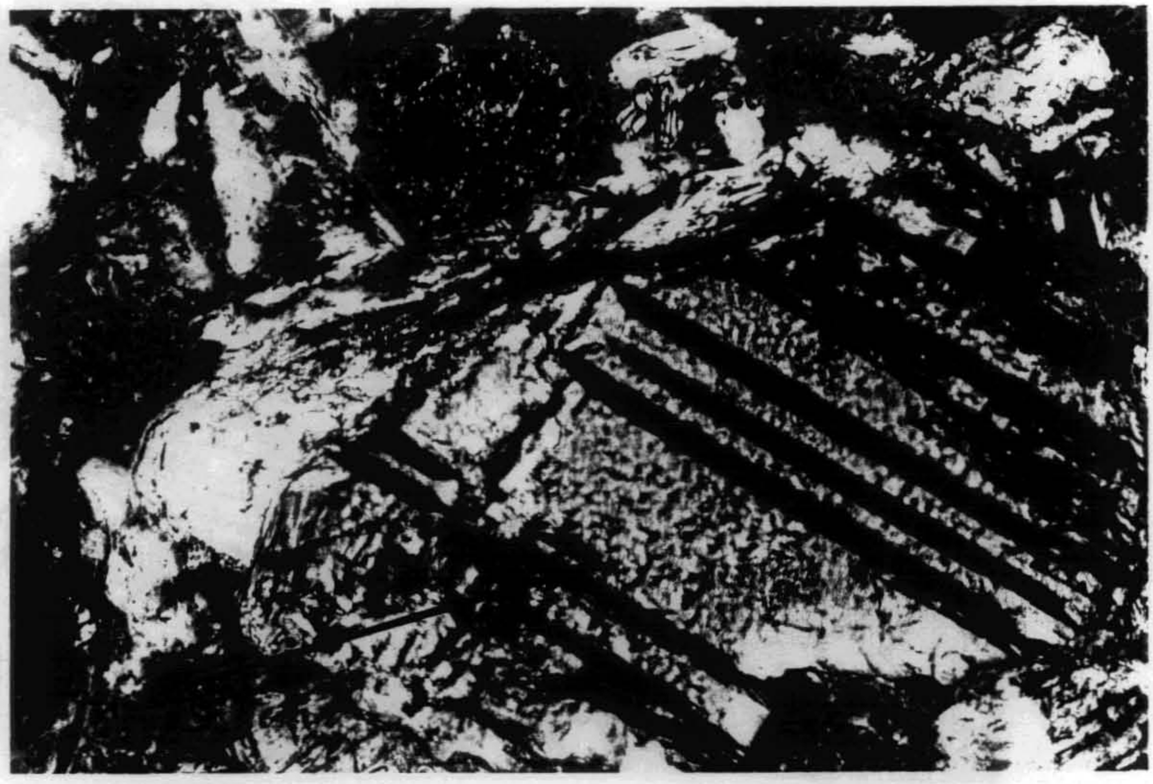


Figure 2: **Leach Residue** PTS5167 160 µm
Magnification 160x Transmitted Cross Polarized Light
Discontinuous talc (red arrow) rimming magnesite (black arrow).

5 cm

Figure 3: **Leach Residue** PTS5167 160 µm
Magnification 160x Transmitted Cross Polarized Light
Talc rim (red arrow) covering more of the circumference of the magnesite grain (black arrow).

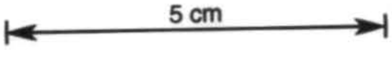
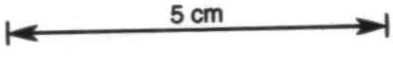


Figure 4: **Leach Residue** PTS516 160 μm
Magnification 160x Transmitted Cross Polarized Light
A large liberated talc aggregate grain.



The magnesite grains were analyzed with the SEM-EDX to determine if there was an elemental difference between partially leached grains, in contrast to non-reacted grains. The iron content varied between magnesite grains, however no correlation was noted between the amount of iron present and the degree of leaching that the grain had undergone. Figures 5, 6 and 7 illustrate the SEM-EDX spectra from the magnesite grains. The iron content varies in different grains from trace (<1%) in Figure 3 to moderate (1-3%) in Figure 4, and high in Figure 5 (3-5%).

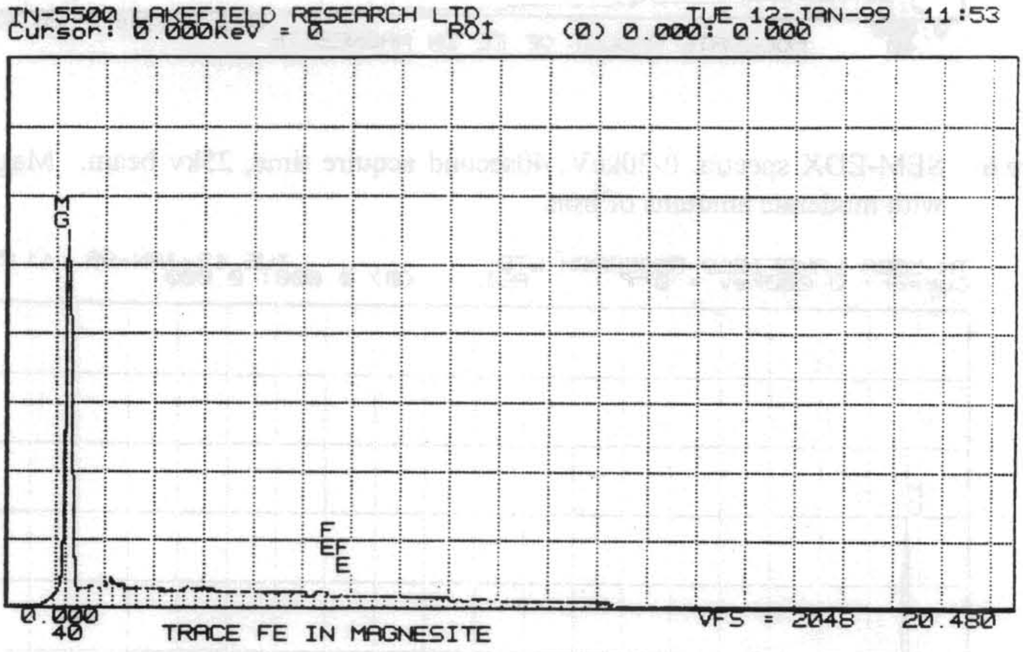
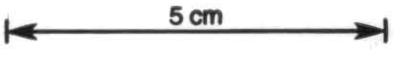


Figure 5: SEM-EDX spectra, 0-20keV, 40second acquire time, 25kv beam. Magnesite grain showing a high magnesium peak and a trace amounts of iron.



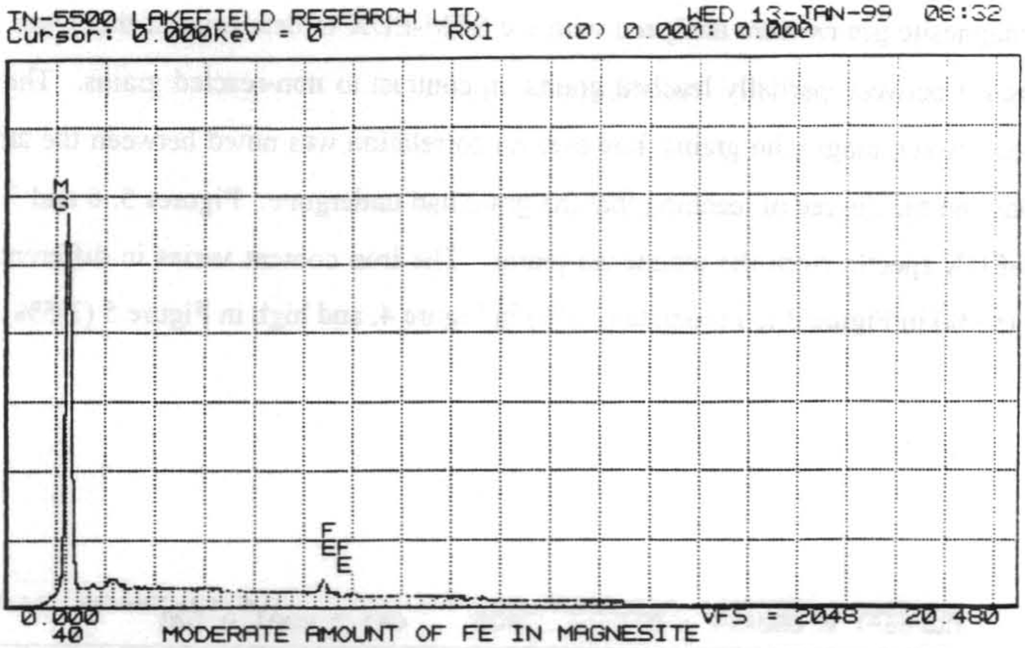


Figure 6: SEM-EDX spectra, 0-20keV, 40second acquire time, 25kv beam. Magnesite grain with moderate amounts of iron.

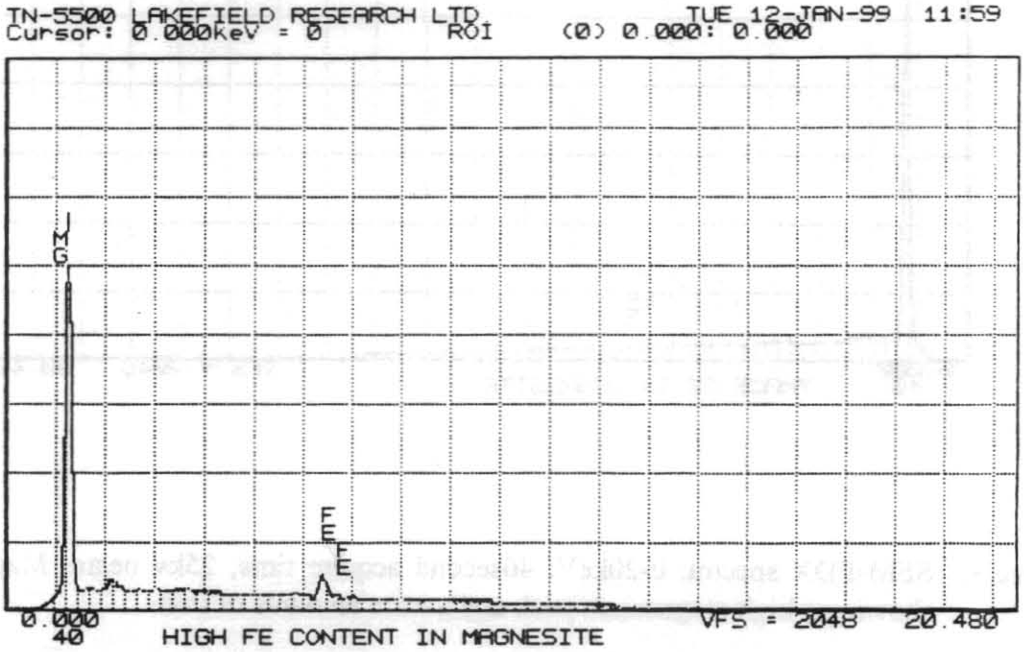
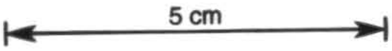


Figure 7: SEM-EDX spectra, 0-20keV, 30second acquire time, 25kv beam. Magnesite grain showing a high amounts of iron.



Remarks

The mineralogical observations made of the magnesite grains indicate:

- The grains are euhedral-subhedral without noticeable evidence of leaching.
- The chemistry of the magnesite grains is not such that it would prohibit leaching.
- The rimming of the magnesite by talc is discontinuous and therefore will not greatly inhibit leaching.

The above points indicate that the magnesite remaining in the leach residue is most likely due to the terminal acid leach concentration.

LAKEFIELD RESEARCH LIMITED
January 13, 1999

G. Lane
Geoff Lane
Project Mineralogist

Bob Irwin
Bob Irwin
Business Manager
Mineralogical Services

Appendix A2

Progress Report No. 2
LR-5398

827110

An Investigation of

the

Purification of Leach Liquor

from

HCl Leaching of Golden Triangle Resources MC36 Magnesite Ore

Progress Report No. 2

Project No.: LR 5398

NOTE: This report refers to the samples as received.
The practice of this Company in issuing reports of this nature is to require the recipient not to publish the report or any part thereof without the written consent of Lakefield Research.

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March 26, 1999

ABSTRACT

The objective of this phase of laboratory development of a magnesite to magnesium process, for Golden Triangle Resources NL, has been to confirm prior testwork, to test three proposed purification routes and to develop a simplified flowsheet for the leach slurry neutralisation, purification and solid liquid separation operations of a future process.

This progress report follows upon the submission of original drafts on January 20th and 27th. Recent information from both Alcan International and the inferred specifications of a magnesium producer, have permitted the re-evaluation of purified brine composition targets for the GTR process.

A simplified flowsheet based upon a "DOP" (direct neutralisation and oxidising precipitation of impurities) purification scheme, potentially coupled with known methods for calcium (Dow Chemical) and sulphur (Norsk Hydro) elimination, provide the preferred neutralisation and purification flowsheet. Neutralisation would be accomplished to pH 2 using calcined ore or (preferably) magnesite, with low boron MgO or brucite ($Mg(OH)_2$) being used to raise the pH to 4 or 5. Further neutralisation would be undertaken using very small additions of NaOH.

Best results of twelve purification tests provided purified brine of the following composition:

Mg	985000 mg/l
Ca	1250 mg/l
Fe	<0.10 mg/l
Mn	0.2 mg/l
Zn	6.96 mg/l
Al	1.2 mg/l
Cu	0.9 mg/l
B	2.4 mg/l
Ni	<0.1 mg/l
Cr	< 0.1 mg/l
Na	6.8 mg/l
Si	0.8 mg/l
S(T)	926 mg/l

The use of H_2SO_4 could precipitate up to 76% of the leach-dissolved calcium from the brine as gypsum. The precipitated gypsum product improved the filtration of the difficult to filter iron products.

The optimisation and selection of best reagents for this part of the process would be pursued in Phase 2 testwork, which is more fully described in the final summary report.

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INTRODUCTION

At the request of Mr. Chris Laughton, General Manager of Golden Triangle Resources N.L., Australia, Lakefield Research Limited undertook a preliminary laboratory test program to develop a magnesite ore to electrolytic magnesium process for their Tasmanian Main Creek deposit. The results of the first phase of this program, on magnesite leaching, have been summarised in Progress Report No 1. The present Progress Report No 2 covers the laboratory investigation of the purification of a magnesium chloride solution generated from the hydrochloric acid leaching of ore sample MC-36.

The results of this work have been forwarded previously by fax, electronic transmission and in the form of a draft report in January 1999. The information was sent to Mr. Laughton and to Dr. Andrew Firek, consultant to Golden Triangle Resources.

The aim of the solution purification testwork was to examine various process options for the removal of the main impurities in the leach solution, in order to produce a feedstock suitable for use in the Alcan Multi-Polar Cell. The solid-liquid separation characteristics of various precipitates and residues produced in the solution purification process were also examined. A simplified flowsheet was prepared following the investigation of purification process options.

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Senior Engineer
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Arthur Plumpton

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John Geldart, B.Sc., B.Eng.
Project Metallurgist
Hydrometallurgy Group

Experimental Work by: Gavin Toole

SUMMARY

1. Summary of Results

This section includes the results of leach slurry purification scoping experiments, experimental flowsheet development and characterisation of the solid-liquid separation behaviour of the leach residue. Section 1.1, which addresses solution purification methods and raw materials, is followed by Section 1.2, dealing with the specific tests, their conditions and results. The section also includes a partial crystallisation experiment on the purified brine and solid-liquid separation tests on various samples. Section 2 provides a discussion of the results in terms of flowsheet development and of industrial specifications for purified brine destined for electrolysis cell feedstock following evaporation and dehydration stages. Section 3 contains the conclusions related to purification and solid-liquid separation. The details of the testwork conducted in the area of the dehydration may be found in Progress Report No. 3.

1.1 Solution Purification Methodology - materials and conditions

Hydrochloric acid leaching of the MC-36 magnesite sample resulted in magnesium extractions in the range of about 92 – 94.5%. Under similar conditions the MC-32 sample yielded as much as 97 % extraction of magnesium, possibly owing to the absence of difficult-to-leach talc.

The average chemical analysis of the resulting leach solution of the MC-36 magnesite sample is shown in Table No. 1. The major impurities in the solution are calcium, iron and manganese. These impurities must be removed from the leach solution before processing by solution evaporation, $MgCl_2$ dehydration and electrolysis. The solution purity guidelines are discussed in section 1.2.7.

Various potential flowsheet options for removing these main impurities from the leach solution were examined. These included three basic purification procedures: Direct neutralisation without oxidation (DP); Direct neutralisation and oxidising precipitation (DOP), and Pre-reduction, neutralisation and oxidising precipitation ("U-Process"). The latter two purification strategies were sometimes accompanied by calcium and sulphate removal operations.

SUMMARY

Table No. 1 - Average Pregnant Solution Analysis

Element	Concentration
Mg	95.8 g/l
Ca	8 mg/l
Si	4 mg/l
Na	5.29 g/l
Fe	2.33 g/l
Mn	240 mg/l
Zn	6.7 mg/l
Al	6.3 mg/l
Cu	1.5 mg/l
B	0.8 mg/l
Ni	0.4 mg/l
Cr	4 mg/l
S	0.2 mg/l

The various test conditions for the application of these purification options are outlined in Table No. 2. More detailed test conditions and results are contained in the body of this report, and in Appendix A.

Some of the tests were performed so as to generate solids (slurry) for thickening and filtration characterisation testwork.

Table No. 2 – Solution Purification Test Conditions

Test No.	Pulp or Sol'n	Temp °C	Procedure			Removal of		S / L Tests	Comment
			DP	DOP	"UP"	Ca	S		
18	Pulp	50	✓					yes	Calcine to pH 4.7 & NaOH to pH 6.8
19	Sol'n	50		✓		✓		no	Calcine to pH 4.8
20	Pulp	50		✓		✓	✓	yes	Calcine to pH 4.6
24	Sol'n	50		✓				no	MgO to pH 6.4
27	Pulp	50			✓	✓	✓	no	$Mg(OH)_2$ as neutraliser, H_2SO_4 addition was prior to reduction stage
28	Pulp	50			✓	✓	✓	no	Calcine to pH 1.5, $Mg(OH)_2$ to pH 6.1, H_2SO_4 addition prior to reduction, SO_2 used as reducer
29	Pulp	80			✓			no	as per Nagamori et al
30	Pulp	80			✓	✓	✓	no	as per Nagamori et al plus removal of Ca & S after U-process
34	Pulp	80			✓				Iron metal (6.75kg/t) added, in place of ferrous chloride
35	Pulp	80			✓			no	As above, but 1.25 kg/t addition of iron metal
36	Pulp	80		✓		✓	✓	no	Chlorine gas oxidant; $Mg(OH)_2$ neutraliser; Ca and S removal at end
37	Pulp	80		✓		✓	✓	no	Oxygen gas oxidant; $Mg(OH)_2$ neutraliser; Ca and S removal at end

Note on abbreviations:

DP - Direct neutralisation and precipitation

DOP - Direct neutralisation and oxidising precipitation

"UP" - "U - Process", or Pre-reduction, neutralisation and oxidising precipitation

S/L - solid liquid separation

SUMMARY

1.1.1 Reagents:

The following reagents were used in the solution purification testwork:

Calcine – calcined magnesite, prepared by heating MC-36 ore for 60 minutes at 650 °C, the calcine had a LOI of 8.1% and a measured "activity" of 72 seconds.
MgO – ACS grade by Fisher Scientific
$Mg (OH)_2$ – Lab grade by Nymoc
$NaOH$ – Lab grade by Nymoc
HCl – A.R. grade by Anachemia – 36%
H_2SO_4 –A.R. grade by Anachemia – 96%
Fe – A.R. grade - Fisher (100 mesh)
$FeCl_2$ – A.R. grade by Mailinckrodt
$BaCl_2$ – A.R. grade by BDH
H_2O_2 – A.R. grade by Spectrum – 35%
Chlorine gas – BOC Gases
Compressed air – BOC Gases
Water – distilled water was use throughout testwork

1.2 Purification Test Results

1.2.1 Summary of Test Results

Twelve purification tests were performed in total. The final solution analysis for each of these tests is given in Table No. 3. The results are presented in detail in the following sections.

Table No. 3 – Final Purified Solution Analysis

Test	Final Solution Analysis (mg/L)												
No.	Mg	Ca	Fe	Mn	Zn	Al	Cu	B	Ni	Cr	Na	Si	S(t)
18	63300	4790	0.34	6.32	<5.00	<0.50	<1.00	<0.50	<0.50	<0.10	16800	<5.00	220
19	94100	274	7.95	192	4.86	...	0.35	<0.10	<0.10	...	25.8	0.92	...
20	101000	744	<0.1	203	...	...	...	...	...	...	...	1.05	1050
24	90400	...	...	56.3	...	...	...	...	...	...	...	...	...
27	101000	1300	4.20	54.5	8.6	<0.50	<0.10	<0.10	<0.10	<0.10	<0.50	8.30	443
28	94100	1400	1.90	357	6.4	2.5	<0.10	<0.50	<0.10	<0.10	6.10	2.30	615
29	84300	4800	0.68	20.5	0.17	3.0	<0.05	1.5	<0.05	<0.05	8.56	<2.0	...
30	108000	702	<0.2	1.34	3.26	1.02	<0.2	2.83	0.56	<0.2	6.98	<1.0	984
34	90500	5040	0.84	15.3	...	...	...	...	...	...	...	...	...
35	91300	5400	0.54	3.27	...	...	...	...	...	...	...	...	...
36	98500	1250	<0.10	61.9*	6.96	1.21	0.86	2.37	<0.10	<0.10	6.75	0.84	926
37	106000	1190	<0.10	144	6.06	1.21	<1.00	1.78	<0.10	<0.10	6.62	<0.50	943

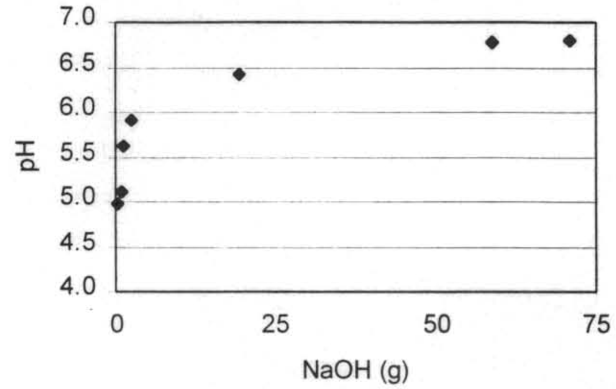
*0.2 mg/l Mn when re-chlorinated

1.2.2 Direct Neutralisation and Precipitation

In Test No 18 the direct neutralisation and precipitation (DP) procedure was employed. Calcined MC-36 ore and HCl were used to adjust the leach pulp pH to 2.5. The neutralised pulp was thickened and filtered. The solution was then further neutralised with calcined ore to pH 4.6. Thereafter, calcine would not raise the pH above 4.6 and NaOH was thereby used to obtain a higher pH.

A pH higher than 6.8 could not be obtained with NaOH, despite large additions as shown in Figure No. 1. This is indicative of the occurrence of a precipitation reaction. During the NaOH additions the solution took on a blue colour at about pH 5.1. With further caustic addition the colour intensified, solids formed and the solution took on the consistency of a thin paste. The final solids produced were thick, waxy and gelatinous, and resisted filtration (see solid liquid separation section). This precipitate is thought to have been Sorel cement ($MgCl_2 \cdot 3Mg(OH)_2 \cdot 8H2O$), although, given this unwanted result, no attempt was made at its identification.

Figure No. 1 - pH vs NaOH Addition



5 cm

During the test, solution samples were withdrawn at various pH levels. The analysis of the aliquot solutions is shown in Table No. 4. It may be noted that impurity levels were not reduced significantly until a pH of 6.8 was reached. However, at this pH and lower levels (pH 5.0) a large portion of Mg was also lost from solution. It thus appears that a direct neutralisation and precipitation method is unable to provide the conditions necessary for selective impurity removal from the magnesium chloride leach solution. This approach was not investigated further.

Table No. 4 – Solution Analysis – Test 18

	Assay (mg/l)			
pH	Mg	Ca	Fe	Mn
-1.5	107100	6390	2060	263
2.0	103000	5970	1760	259
5.0	71400	6140	1410	245

6.8	63300	4790	0.34	6.34

1.2.3 Direct Neutralisation and Oxidising Precipitation

The "direct neutralisation and oxidising precipitation" (DOP) procedure was experimentally evaluated as an option to improve the removal of iron from the leach solution. The first solution purification test (Test 18), performed using the "direct neutralisation and precipitation" (DP) procedure, indicated that a majority of the iron, about 68%, was in the ferrous state (Fe+2). This is illustrated by the values given in Table No. 4, where there remains 1.4 g/L iron in solution at a pH of 5.0. In the DOP procedure the iron was first oxidised to the ferric (Fe+3) state before the pH was raised, so that a majority of the iron would precipitate below a pH of 2.5 to 3.0.

Two tests were initially performed using the DOP procedure and hydrogen peroxide oxidant; Test 19 was conducted using a composite leach solution, and Test 20 was performed on a freshly leached pulp sample. The procedure was enacted in a stage-wise fashion, so as to permit a study of the chemical and physical nature of the various products.

The basic procedure is illustrated in Figure No. 2. Two additional purification steps were added to the basic DOP procedure, to investigate the removal of calcium and sulphate ions.

The results of Test 19 on a composite leach solution are shown in Table No. 5. The concentration of iron in solution was reduced 250 fold from 1940 to 7.7 mg/l by using the DOP procedure. The iron precipitate assayed 36.0 % Fe and 8.15% Mg. No net magnesium was lost from solution during the iron precipitation. The amount of magnesium increased slightly, due to its addition to the solution through the use of calcined ore (magnesite) in the neutralisation of the excess acid.

In Test 19 the sulphuric acid was added in stages to the leach solution to determine the effect on calcium removal. The additions were approximately 1.5, 5.0, 25, 45 and 66% of the calculated stoichiometric quantity required for the reaction;

$$CaCl_2 + H_2SO_4 \rightarrow CaSO_4 + 2HCl$$

Barium chloride was not added for sulphate removal in this test.

The results shown in Table No. 5 indicate that the calcium concentration can be reduced substantially using a sub-stoichiometric addition of acid. This would suggest that a co-precipitation reaction is occurring with magnesium. The quantity to be used in practice will be dictated by the purity requirements, and by the added cost of removing any excess sulphate. The final pH of the solution in Test 19 was 4.7.

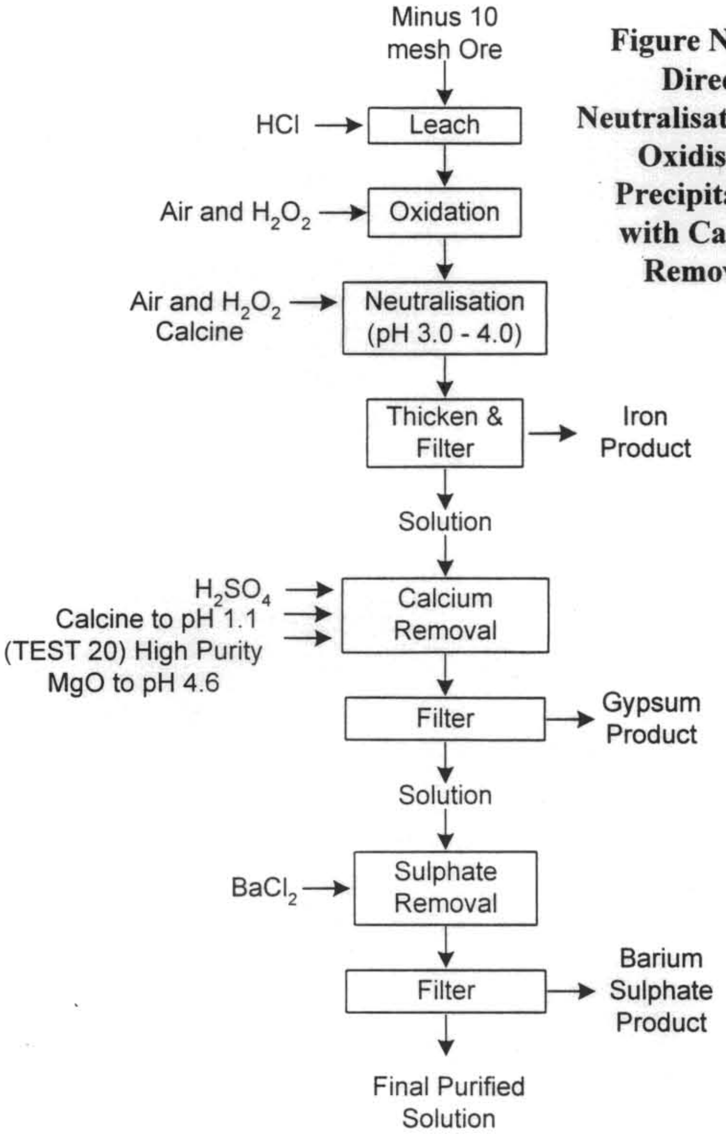


Figure No. 2 - Direct Neutralisation and Oxidising Precipitation with Ca & S Removal

Table No. 5 – Results of Direct Oxidation and Neutralisation, and Calcium Removal Test on a Composite Leach Solution (Test 19)

Product	Amount	Assays (mg/L, %)			
	(ml, g)	Mg	Ca	Fe	Mn
pH 3.0 filtrate	465	105000	5210	7.72	223
pH 3.0 wash	98	24000	1420	7.79	
pH 3.0 solids	2.7	8.15	1.65	36.3	0.9
acid 1 preg	25	105000	3210	0.95	
acid 2 preg	25	104000	1610	0.93	
acid 3 preg	25	93500	616	1.05	
acid 4 preg	25	100000	353	2.30	
acid 5 preg	385	94100	274	7.95	192
acid 5 wash	37	41000	1360	40.5	
Final solids	7.7	4.06	22.3	0.45	0.02
Head (dir)	500	94800	5260	1940	251

The DOP procedure was repeated in Test No. 20, using approximately the same procedure as in Test 19, but treated leach slurry instead of leach liquor. The thickening and filtration characteristics of the resultant products were examined. The solution analysis results for this test are shown in Table No. 6

Table No. 6 – Solution Analysis for Test 20

Product	Amount	Assays(mg/L, %)				
		Mg	Ca	Mn	Fe	S
Preg Sol'n		96400	5210	270	2030	<0.5
pH 4.8 Sol'n		101000	689	217	3.51	1000
Decant & Filtrate	955	101000	744	203	<0.1	1050
Total Wash	296	11214	...	...	...	...
Residue	50.9	13.6	6.23	13.8	4.68	

As in Test 19, the iron and calcium levels were greatly reduced (iron significantly) using this procedure, without any reduction in the concentration of magnesium in solution. However in both Tests 19 and 20, the manganese levels remain unchanged at about 200 mg/L, still considered too high a brine specification for modern electrolysis cells. To determine if the Mn concentration could be lowered by simple pH adjustment an additional test (No. 24) was performed on the solution from Test 20.

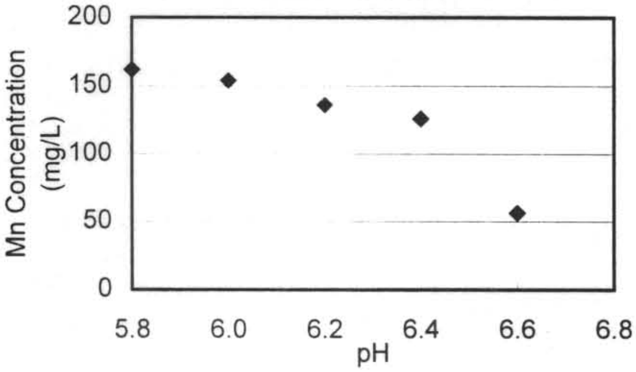
In Test No. 24 the Test No. 20 solution was neutralised with high purity MgO instead of calcine, followed by NaOH addition. Solution samples were withdrawn at several pHs for Mn and Mg analysis. The results are shown in Table No. 7 and Figure No.3.

The more complete neutralisation of the oxidized solution in this case provided additional Mn removal without significant loss of magnesium. However, the final Mn level remained higher than desired. Further development of the DOP procedure was thus temporarily suspended in favour of the "U-Process" which reportedly could remove manganese (as well as chromium and vanadium) from concentrated magnesium solutions. Later DOP purification tests were undertaken, substituting chlorine gas and air for hydrogen peroxide (see section 1.2.6).

Table No. 7 – Results of Test 24 – Mn Precipitation by Simple pH Adjustment

Product	Amount	Assays (mg/L, %)		Distribution (%)	
	(ml, g)	Mg	Mn	Mg	Mn
pH 5.8	5	90000	162		
pH 6.0	10	92200	154		
pH 6.2	10	89300	136		
pH 6.4	10	87600	126		
pH 6.6	245	90400	56.3	77.8	75.6
Wash	95	62900	0.45	21.0	0.23
Precip	14.7	23.5	0.3	1.21	24.2
Head (dir)	380	94650	271		
Head (calc)				100	100

Figure No. 3 - Mn Precipitation following a DOP Procedure



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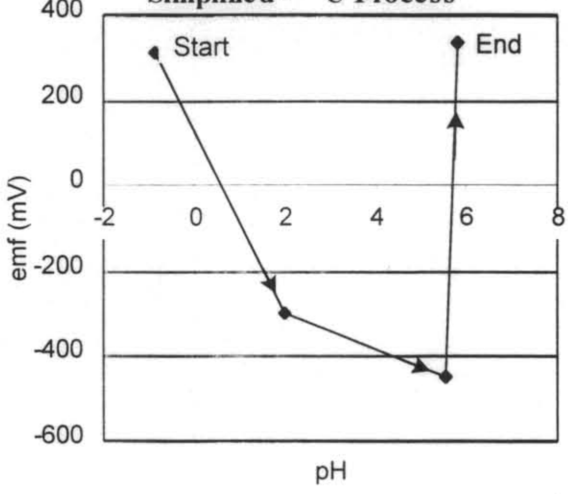
1.2.4 U-Process

The "U-Process", developed by M. Nagamori et al[1], was developed to purify acid solutions generated by the oxidising acid leaching of magnesite ore or serpentine asbestos tailings. It takes its name from the pattern formed on the E – pH diagram during the process; the solution starts as acidic and oxidising, and goes through 3 distinct stages: acid and reducing, alkaline and reducing, and finally alkaline and oxidising. A simplified sketch illustrating this path is shown in Figure No. 4.

Figure No. 4
Simplified - "U Process"



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Six tests were performed, one following the U-Process as described in the literature, three combined the U-Process with the previously mentioned methods for removing calcium and sulphate and two employed varying additions of cheaper metallic iron rather than ferrous chloride reducing agent. In Test No. 27 and 28, the tests were performed at 50 °C with the calcium removal stage coming before the "U". Tests 29, 30, 34 and 35 were performed at 80 °C, and in Test 30 the calcium removal stage came after the "U". Only in Tests 29, 30, 34 and 35 were the manganese levels of the final solutions significantly reduced (below about 20 ppm). The procedure used in Test 30 is shown in Figure No. 5. The flowsheet would ultimately be simplified, as in other cases, through reduction of the number of filtration steps. The final purified solution analyses for the six tests are shown in Table No. 8.

The procedure used in Test 30 needs further optimisation to minimise reagent use and improve the later stages of calcium and sulphate removal. The lower amount of metallic iron used in Test 35 provided equally good if not better purification results than Test 34 (with the exception of Na, which was introduced for pH control).

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[1] Nagamori, M., Plumpton, A.J. and Panneton, J.J., "Purification of Magnesium Salt solutions with Ferrous Salts and Magnesium Hydroxide", Hydrometallurgy, 30 (1992), pp. 499 – 513.

Table No. 8 – Final Purified Solution Analysis

Test No.	Final Solution Analysis (mg/L)												
	Mg	Ca	Fe	Mn	Zn	Al	Cu	B	Ni	Cr	Na	Si	S(t)
27	101000	1300	4.20	54.5	8.6	<0.50	<0.10	<0.10	<0.10	<0.10	<0.50	8.30	443
28	94100	1400	1.90	357	6.4	2.5	<0.10	<0.50	<0.10	<0.10	6.10	2.30	615
29	84300	4800	0.68	20.5	0.17	3.0	<0.05	1.5	<0.05	<0.05	8.56	<2.0	...
30	108000	702	<0.2	1.34	3.26	1.02	<0.2	2.83	0.56	<0.2	6.98	<1.0	984
34	90500	5040	0.84	15.3	...	...	...	...	...	...	1550	...	...
35	91300	5400	0.54	3.27	...	...	...	...	...	...	10	...	...

SUMMARY

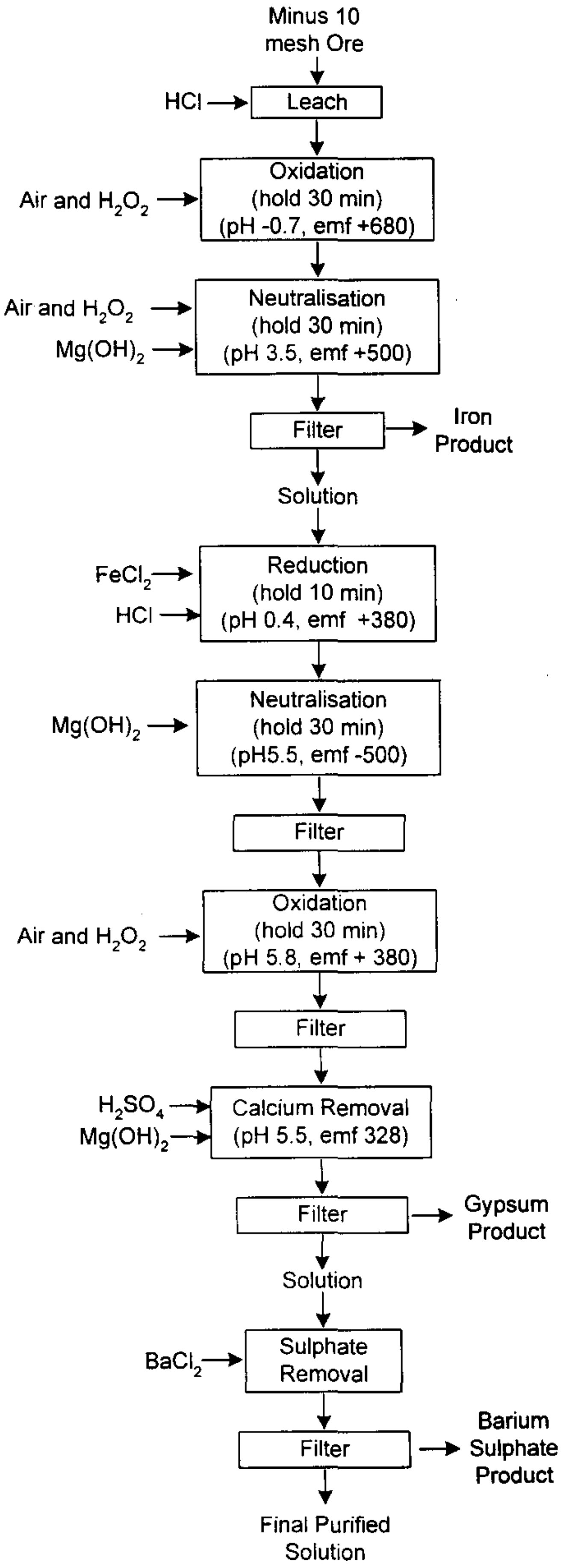

Figure No. 5
Modified U- Process

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1.2.5 Direct Oxidation and Precipitation, using chlorine gas or oxygen rather than hydrogen peroxide

Chlorine would be available from the electrolysis stage of a proposed Golden Triangle magnesium plant. Whereas most of the chlorine would likely be recycled transformed as HCl solution or gas for leaching, a part of the chlorine could be used for solution oxidation (as proposed by Magnola Metallurgy Inc.). A DOP procedure using chlorine gas was thereby investigated, as was an oxygen gas DOP purification of the leach solution, similar to a practice reported in industry to prepare purified brine for synthetic MgO production.

Test 36 investigated the use of chlorine as oxidant and Test 37 studied oxygen gas in that role. These tests were also characterised by an attempt to reduce the complexity of the flowsheet, using a **single** filtration step, after all precipitation reactions had been completed.

1.2.6 Partial Evaporation and Crystallisation of Purified Solution

The purified solution from Test 30 (see Table 10) was used as feed to a partial crystallisation scoping test, to determine if further purification could be achieved by means other than those considered during solution purification tests.

The initial leach solution had a volume of about 1000 ml. The volume was reduced throughout the purification stages (at 80 °C) as no reflux condensers were used. The volume reporting to crystallisation was 670 ml. An initial attempt was made to reduce this volume to 350 ml, however this resulted in the formation of a solid mass. An addition of 300 ml of de-ionised water was added to these solids, and the mixture was allowed to mix overnight at about 15 °C in order to dissolve all of the crystals. The mixture was then slowly heated to about 100 °C, and the volume reduced to 500 ml. This solution was allowed to cool to about 50 deg C, which produced a "slushy" mixture. The mixture filtered rapidly, leaving 245 ml of solution. The crystals were air dried, and submitted for analysis. A balance for this procedure is shown in Table No. 9. The chemical analysis of the purified solution and crystals are shown in Tables 10 and 11. The Mg and Cl concentrations shown in Table No. 11 are consistent with the compound $MgCl_2 \cdot 6H_2O$.

Enhanced purification, beyond that achieved in solution purification Test 30, is indeed possible using this partial crystallisation technique. However, as about one-half of the magnesium reports to the purified crystals and one-half to the impure supernatant, the application of this method would be impeded by the need to recycle an important quantity of supernatant (about 25 % of the weight of the purified brine of Test 30) to the front end of the purification flowsheet, thereby increasing the quantity of solution to be handled and purified, and increasing the potential for impurity build-up.

Table No. 9 – Test 30 - Crystallisation

Product	Amount (g, ml)	Assay (mg/l, %)		Distribution (%)	
		Mg	Ca	Mg	Ca
Crystals	258.7	12.0	0.045	52.1	44.5
Supernatant	245	116400	592	47.9	55.5
Head (calc)	670	88899	390		
Head (dir)		108000	720		

Table No. 10
Analysis of Purified Solution

Element	Conc (mg/l)
Mg	108000
Ca	702
Si	<1.0
Na	6.98
B	2.83
Mn	1.34
Al	1.02
Cr	<0.2
V	<0.10
Ni	0.56
Cu	<0.2
Zn	3.26
Fe	<0.2
S	984
Ba	65.5

Table No 11
Chemical Analysis of $MgCl_2 \cdot 6H_2O$
Produced by the Partial Crystallisation
of MC36 Leach Solution

Quantitative Analysis			
Wgt Loss		Fe	<0.0005%
@ 200 °C		Cu	<0.0005%
@ 300 °C		B	<0.0005%
@ 400 °C		C	0.01%
Mg	12.0	S	<0.01 %
Cl	34.3	P	<0.0020%
Ca	0.045%	Na	<0.0010%
Si	<0.005%	K	<0.0100%
Mn	0.0005%	Cr	<0.0005%
Al	<0.0020%	Ni	<0.0005%
		Zn	<0.0005%

Semi-quantitative Analysis			
Ag	< 0.5 ppm	As	< 100 ppm
Pd	< 5 ppm	Au	< 100 ppm
Be	< 10 ppm	Ba	< 100 ppm
Bi	< 10 ppm	In	< 100 ppm
Co	< 10 ppm	Nb	< 200 ppm
V	< 10 ppm	Ta	< 200 ppm
Ge	< 25 ppm	W	< 200 ppm
Pb	< 25 ppm	Rb	< 200 ppm
Mo	< 25 ppm	Pt	< 200 ppm
Sr	< 25 ppm	Te	< 300 ppm
Sn	< 25 ppm	Y	< 300 ppm
Ti	< 25 ppm	Ce	< 300 ppm
Zr	< 25 ppm	U	< 1000 ppm
Sb	< 50 ppm	Th	< 1000 ppm
Cd	< 50 ppm		
Ga	< 50 ppm		
Li	< 50 ppm		
Tl	< 50 ppm		

1.2.7 Comparison of purification test results and magnesium industry specifications

The following Table (12) compares the results of purification Test No. 27 with specifications for $MgCl_2$ cell feedstock as discussed with Golden Triangle in January 1999. Note that the former should be multiplied by a factor of 3 to compare to the stated cell feedstock specification. Note also that the Kh. Strelets and Andreassen references are very likely outdated, as they are 20 or more years old. At that time, the high energy efficiency cells were either not in operation, or just beginning to appear. The new cells, by virtue of their close electrode design and other factors, require more stringent impurity specifications than previously accepted. Based upon the older information, and some Alcan data provided by the client, it would appear that test 27 comes very close to meeting most of the requirements. In fact, solely on that basis, it could be argued that the attained Fe and Mn levels are unnecessarily low.

However, a second more realistic comparison with industrial data may be made (see Table 13), based upon some fragmentary and preliminary but more recent data acquired in discussions with Alcan International (Kingston, Ontario) and from a current magnesium producer who uses the more modern cells. The latter specification relates to a pH 4 purified brine, which may or may not have been subjected to additional purification. The Alcan data is for MPC cell feedstock and has been transposed to the estimated brine values, which would exist prior to solution evaporation and MgCl2 crystal dehydration.

Table No. 12 – Specifications for $MgCl_2$ Cell Feed / Purified Brine*

Element	Alcan [1]	Strelet [1]	Andreassen 1978 [2]	LRL Test 27
MgO	...	5000	2000	...
C	...	...	1500	...
H_2O	1000	1000	1000	...
Mn	600	600	600	55
Fe	400	138 [3]	200	4.2
S	...	120 [4]	100	443
Si	...	...	100	8.3
Ti	...	80	...	...
P	...	...	50	...
B	10 - 20	6.2 [5]	10	0.1
Notes:	[1]	data reported by Andrew Fire		
	[2]	data from Erzmetall Vol. 31, No. 7/8, pp301 – 309, 1978		
	[3]	reported as 440 $FeCl_2$		
	[4]	reported as 300 SO_3		
	[5]	reported as 20 B_2O_3		

* all data is for new cell feed (MgCl2) except for LRL Test 27 which is purified brine (the latter values should be multiplied 3 fold to compare directly with cell feedstock specifications)

Table No. 13 – Revised Specifications for $MgCl_2$ Purified Brine Destined for High Efficiency Mg Cells

Element	Alcan MPC [1]	Mg producer (2)	Spec. indicated in report 7740B Oretest (3)	LRL Test 36	LRL Test 27
MgO	...	...	...		...
Na	...	5000	140	6.8	<0.5
Ca	300	5000	1000	1250	1300
Mn	0.2	10-20	1	62→0.2	55
Fe	0.2	1-5	1	<0.1	4.2
S	...	1-2	100	926	443
Si	5000	10	...	0.84	8.3
Ti	...	...	...		...
P	...	1-5	...		...
B	10 – 20	15	2	2.4	<0.1
Ni	<1?	...	1	<0.1	<0.1
Al	2-3	...	...	1.2	<0.5
Zn	5		...	7	8.6
K			1050		

Notes: (1) preliminary data from Drs. D. Creber and G. Holywell of Alcan, Feb. 5, 1999 meeting at LRL(2) data from a commercial magnesium brine purification operation (ranges quoted to mask precise data)
(3) Specifications from "client", as indicated in ORETESTreport 7740B, August 1998.

1.2.8 Solid- Liquid Separation

As the solution purification procedure was not defined at the beginning of the test program, and was developed through experimentation,
the basic solid liquid separation characteristics of the various precipitates were examined as the test work progressed. The qualitative values are listed below. Some of the filtration rates are very rapid, for example the pH 2.5 pulp residue from Test 18 had a filtration rate of 2293 kg of dry filter cake per hour per meter square. In such cases, filtration requirements are minimised.

SUMMARY

Qualitative Results:

Test 18

- no gypsum precipitation

Stage 1 (leach pulp neutralised to pH 2.5 with calcine, no oxidation)

- thickening at pH 2.5:
  - no flocculant added & no visible mudline (bottom up)
  - settled density after 1 hour = 847 grams dry solids per litre of pulp
  - liquid density: 1.284
  - est. solid density: 2.93
  - final pulp density: 1760 g/l or 48.2% (grams solids/grams pulp)
- filtration of thickened solids:
  - dry cake rate = 2293 kg/h/m^2
  - filtrate rate (initial) = 778 l/h/m^2
  - filtrate rate (wash) = 584 l/h/m^2
  - filter cake moisture = 17.0%

Stage 3 (neutralise solution from previous stage to 5.0 with calcine, and 6.8 with NaOH)

- thickening at pH 6.8:
  - no flocculant added & two distinct mudlines
  - settled density after 1 hour = 87.9 grams dry solids per litre of pulp
  - liquid density: 1.252
  - solid densty: not determined (est 2.80)
  - estimated final pulp density 1300 g/l or 6.8% solids (grams solid/grams pulp)
- Filtration of thickened solids:
  - dry cake rate = 246 kg/hr/m2
  - filtrate rate (initial) = 246 l/h/m2
  - filtrate rate (wash) = 45 l/h/m2
  - filter cake moisture = 65%

Test 19

Stage 1 (oxidation of leach solution and precipitation of iron at pH 4.4 with calcine)

- Filtration of un-thickened solids
- dry cake rate = 0.75 kg/h/m2
- filtrate rate = 130 l/h/m2

Stage 2 (gypsum precipitation and neutralisation to pH 4.8 with calcine)

- Filtration of un-thickened solids
- dry cake rate 17.2 = kg/h/m2
- filtrate rate = 859 l/h/m2

SUMMARY

Test 20

(pulp oxidation, neutralisation, gypsum and sulphate precipitation)

- thickening
  - settled density after 1 hour = 318 grams dry solids per litre of pulp
  - liquid density: 1.250
  - solid density: not determined (est 2.80)
  - estimated final pulp density 1424 g/l or 22.3% solids (grams solid/grams pulp)
- filtration of thickened solids
  - dry cake rate = 771 kg/h/m2
  - filtrate rate (initial) = 2121 l/h/m2
  - filtrate rate (wash) = 943 l/h/m2
  - filter cake moisture: 45.7

Test 30

Stage 1 (pulp oxidation, neutralisation to pH 3.5 with Mg(OH)2)

- filtration of un-thickened solids
  - dry cake rate = 18.5 kg/h/m2
  - filtrate rate (initial) = 569 l/h/m2
  - filtrate cake moisture = 20.8%

Stage 2 (solution from previous, reduced with FeCl2 and neutralised with Mg(OH)2 to 5.5)

- filtration of un-thickened solids
  - dry cake rate = 4.7 kg/h/m2
  - filtrate rate (initial) = 212 l/h/m2

Stage 3 (solution from previous, oxidised and neutralised to pH 5.8 with Mg(OH)2)

- filtration of un-thickened solids
  - dry cake rate = 1.0 kg/h/m2
  - filtrate rate (initial) = 31 l/h/m2

Stage 4 (solution from above with H2SO4 and Mg(OH)2 to pH 5.5 for gypsum precipt'n)

- filtration of un-thickened solids
  - dry cake rate = 44.3 kg/h/m2
  - filtrate rate (initial) = 1652 l/h/m2

Stage 5 (solution from above with BaCl2 for sulphate removal)

- filtration of un-thickened solids
  - dry cake rate = 0.8 kg/h/m2
  - filtrate rate (initial) = 150 l/h/m2

2. Discussion of Purification Results

The various purification operations applied to the leach solution of GTR's MC 36 ore sample were comprehensively experimented in this test program and have led to the conception and construction of a simplified flowsheet which is capable of yielding a satisfactorily purified brine. The purified brine requirements for the production of anhydrous magnesium chloride have been set out in various commercial specifications to which the products of this testwork have been compared.

Further experimental work in conjunction with Golden Triangle Resources metallurgical and engineering consultants will permit further optimisation and development of the simplified process flowsheet established in this phase of the process development.

2.1 Basic Flowsheet development

The basic flowsheet developed from this work is depicted in Figure No. 6 on the following page. The stages in the development of this flowsheet are discussed in the following.

In the only known currently operating electrolytic magnesium industrial plant that uses magnesite as feed stock, the solution purification plant is limited or non-existent. The requisite solution purity is achieved primarily by using very high purity ore feed stock (98.5 % + magnesite), which contains very low iron and silicon as well as little calcium and acid insoluble material. The use of this type of feed material all but eliminates solution purification and solid-liquid separation unit processes from the plant. Solution purification is a necessary part of other magnesium plants using alternative feedstock, such as the Magnola pilot plant , which uses low purity asbestos tailings.

The proposed Golden Triangle Resources feed materials contained acid soluble iron, calcium and manganese, as well as other impurities at lower levels. There has been reported also a small percentage of difficulty soluble material, principally talc, which would separated from the pregnant leach liquor, or subjected to re-leaching (or counter-current leaching) using fresh HCl. The present work dealt primarily with the removal of the acid-soluble impurities to acceptable low levels.

The solution purification process that is to be developed for the treatment of this material must be kept as simple as possible, with the minimum number of solid-liquid separation steps to minimise soluble losses and the addition of excess water to the system. Hand in hand with this must be the minimisation of reagent additions to keep down costs, if this process is to be cost competitive with current processes. As stated above, at least one such competitive operation is believed to practice very little or no purification.

Calcium, iron and likely manganese are present in the ore as carbonates. $CaCO_3$ is more leachable than $MgCO_3$, until the concentrated brine reaches a certain level of $CaCl_2$ (approximately 0.35 mass %) at which point $MgCO_3$ can be dissolved preferentially[2]. In the

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[2] From a technical paper by M. Nagamori and A. Plumpton, recently submitted for publication.

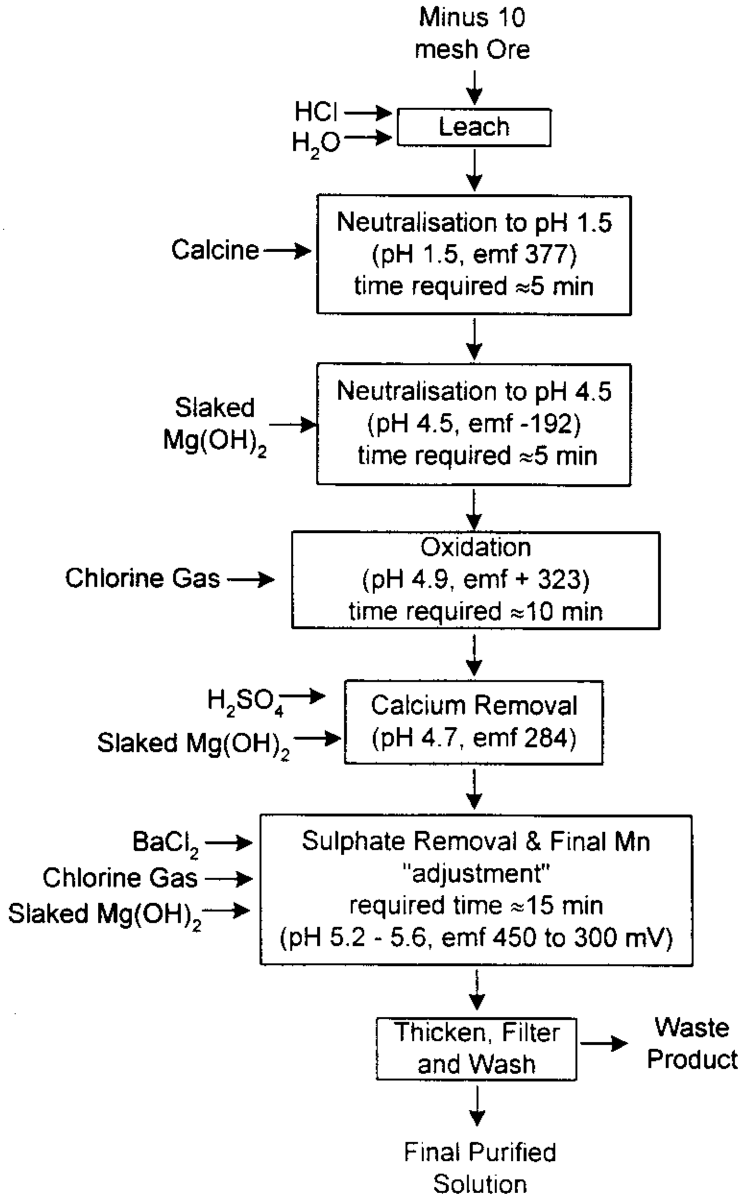


Figure No. 6
Simplified Process

case of $FeCO_3$, even very low concentrations of $FeCl_2$ in solution (about 0.01 mass %) create conditions favourable to $MgCO_3$ dissolution. However, as the saturation pH values (defined as the equilibrium pH values of the $MgCl_2 \bullet H2O$ solutions saturated with a mineral) of these salts are all very close together[2], all of the salts dissolve readily during leaching and must be removed in purification.

These constituted the overall objectives which have guided this testwork, while recognising the necessity to develop different process chemistries in the search for one which would achieve the result of a sufficiently pure $MgCl_2$ solution for subsequent solution evaporation and $MgCl_2$ dehydration.

Purification testwork has been primarily concerned with the removal of major leached impurities as Ca, Fe, and Mn and their elimination to levels at or below those specified for molten $MgCl_2$ cell feed (Table 13).

The purification results have shown that with a modification of the "U-Process" a solution of relative high purity can be produced. However, the U-process adds unnecessary complexity if high valence state Mn and Cr are not initially present. It was found that the use of the simpler "DOP" (direct neutralisation and oxidising precipitation) method, with chlorine gas oxidation (Test 36), yielded equal or better results to the "U process" with this leach solution (Table 3), whether the "U process" was based upon a ferrous chloride or metallic iron reduction step.

The level of calcium and sulphur that can be tolerated in the feed to the electrolysis system must also be addressed. Within the context of a simplified flowsheet, the precipitation of calcium with H_2SO_4 (or possibly $MgSO_4$) permits a significant reduction of calcium in the solution and provides the added advantage of creating a gypsum precipitate the filtration qualities of which may aid in the slurry filtration, particularly in respect to the iron based precipitates. Although not successfully tested in this work (a single experiment was devoted to sulphate removal), the use of barium chloride should be further investigated in a later stage as Norsk Hydro is believed to have mastered its use to lower the sulphate content of the purified brine.

Elsewhere, the solid liquid separation characteristics of the leach residue and precipitation products are important to the success of this process. The data in the solid-liquid separation section of this report indicates that the nature of the precipitates formed at various stages of this process vary widely in there thickening and filtration characteristics. The products with the best filtration characteristics are the gypsum and the leach residue. The iron products and high pH manganese precipitates generally filter poorly. The best alternative is to combine all of these materials in a single filter product as presented in the simplified process flowsheet proposed in Figure No. 6. If the conditions of gypsum precipitation can be manipulated so as to incorporate the precipitated iron hydroxides, a material of moderate filterability, and low residual moisture, should be produced.

2.2 Acid, Oxidants and Miscellaneous Reagents of the Process

Chlorine from electrolysis should be transformed to HCl and recycled to the leaching stage. However, recycling chlorine gas between the $MgCl_2$ molten salt electrolysis cell and the HCl leach cannot make up the amount of HCl consumed (lost) in the leach by $CaCO_3$. For each increment of 1 mass % $CaCO_3$ in the ore, it may be calculated that a 50,000 t/y Mg plant will require the addition of 4990 tons of 26 mass % HCl (at an estimate of 120$/t, this translates to about $600,000) to accommodate this shortfall[2].

On the other hand, some of the HCl consumed by $FeCO_3$ (and possibly by $MnCO_3$) may be recovered in the neutralisation operation, at the expense of MgO. To accomplish this, the ferrous iron must be first oxidised to the ferric state (ferric oxides, and ferric hydroxides will also filter more readily then there ferrous counter parts). This requires the use of an oxidiser, which was pursued in the DOP and U process alternatives studied in this work.

As suggested by the present work, the E-pH relationship during neutralisation will require careful attention, in order to avoid high potentials at which the impurity Mn is transformed from an insoluble form, Mn_2O_3, to the more soluble ion MnO_2^-.

Several oxidisers were considered in the present testwork, including hydrogen peroxide, and chlorine and oxygen gases (present work). Chlorine was found to be the most efficient, and potentially the most cost effective, through the results of this work. Chlorine is less expensive then hydrogen peroxide and would be available on-site. It's role in neutralisation is less well known than that of peroxide or oxygen, and, despite positive results in this present preliminary work (namely, Test 36), further work (in Phase 2) will be required to establish an optimum chlorine oxidation scheme and to complete the assessment of engineering and economic issues which will allow final choice of the most economic purification flowsheet.

2.3 Neutralising Agent Considerations

Neutralising agents are required in the process to neutralise residual acid from the leach, and for pH control during impurity precipitation. The precipitation of calcium, and the oxidation and precipitation of iron in the leach liquor generate acid as shown in the reactions written below. This acid must be neutralised to allow the precipitation reactions to proceed. Thus it can be seen that the neutralising agents costs will principally depend on the quantity of residual acid from the leach, and the quantity of iron and calcium to be removed from the leach liquor.

$$2FeCl_2 + 2H_2O + \frac{1}{2}O_2 \rightarrow Fe_2O_3 + 4HCl$$

$$CaCl_2 + H_2SO_4 \rightarrow CaSO_4 + 2HCl$$

The choice of agents will be based on economic as well as metallurgical considerations. The neutralising agents must be inexpensive and readily available at the proposed plant site. They also must not add to the impurity burden and must be sufficiently reactive at the required pH levels.

Purification testwork on the leach slurry has investigated a number of neutralising agents, including low temperature calcined magnesite (to simulate a caustic calcined MgO of low boron content), reagent grade MgO, NaOH and $Mg(OH)_2$.

Theoretical calculations have indicated the respective saturation pH values of brucite ($Mg(OH)_2$) and magnesite to be approximately 6.6 and 5.4 in a solution containing 30 mass % $MgCl_2$ at 75 °C. These chemical equilibrium values are obtained only after uneconomically long neutralisation times, so that the practical normal upper pH limits on the application of calcined MgO and magnesite are limited to 4.0 and 2.0 respectively. $Mg(OH)_2$ is more reactive and we have attempted neutralisation with $Mg(OH)_2$ at higher values than 4.0, as was occasionally attempted and realised with reactive MgO calcine.

However, the high activity of the latter has a tendency to produce a Sorel cement type reaction, thus precipitating a mixed oxide + chloride salt of magnesium during the attempt to precipitate the impurity heavy metals (especially Mn).

In a practical flowsheet for the GTR ore, the sequence of addition will likely be magnesite to pH 2, MgO to pH 4 (or $Mg(OH)_2$ to pH 5, approximately), followed by a very small amount of NaOH, to attain pH 6 or so.

The use of magnesite, commonly available at about 50 $ per tonne (excluding shipment), is recommended for the GTR process flowsheet, in which incomplete leaching of a final magnesite addition would allow minimisation of HCl usage and preliminary neutralisation to about pH 2.

Future testwork on solution purification should include further testwork on this suite of neutralising agents, which also follows the logical order of increasing reagent unit cost and decreasing neutralising agent requirements.

An example to support this recommendation is set forth in Table No. 14 wherein the added cost of using calcined MgO at low pH, rather than high purity pulverised magnesite ore, is presented.

Phase 2 testwork on process optimisation and development of engineering data is required to distinguish the alternative reagents and secondary purification operations (Ex. Ca and S), to simplify the process and to choose the most economical overall process for the GTR ore.

Table No. 14

Economical benefit of using pulverized magnesite to neutralize a 30 % MgCl2 acid brine to pH 2, for differing free acid concentrations

(Basis: 50,000 tpy Mg plant)

Mass % Free HCl	Initial pH (approximate)	Magnesia $ / (kg Mg)	Magnesite $ / (kg Mg)	Economy $ million / year
3.0	-0.01	0.0700	0.0335	1.83
2.5	0.07	0.0580	0.0277	1.52
2.0	0.16	0.0461	0.0220	1.20
1.5	0.29	0.0343	0.0164	0.90
1.0	0.46	0.0225	0.0108	0.59
0.5	0.77	0.0109	0.0052	0.29

Note: Data assumes caustic-calcined magnesia (94% MgO) is available at a price of 300 $/t, and pulverized magnesite (96 % $MgCO_3$) at a price of 70 $/t.

3. Conclusions on Purification Testwork

The present solution purification test program has been detailed, yet scoping, in nature. The work has amply demonstrated that there are a number of valuable options available to produce a high quality purified leach solution from Golden Triangle magnesite ore for further processing to anhydrous magnesium chloride. The most attractive of those tested is based upon a DOP method using chlorine gas oxidiser. Further testwork should be of both optimising and of engineering nature, to select the final most economic flowsheet and to design the process to meet electrolytic cell feed specifications, typified by the Alcan MPC electrolysis cell.

Recommendations for further development work have been outlined in the preceding section (indicated generally as Phase 2) and will be presented collectively and in additional detail in the final summary report.

APPENDIX A

TEST DETAILS

Dec 2 Calcine

MC-36 minus 10 mesh feed
two charges of 250 g
in round bottom alumina dish, cm in diameter
about 15 min warm-up
60 minutes at 650, stirred once
30 minute cool down, with door open
weight of batches after 135.8 g
133.2 g
% weight loss: 53.8 %

pulverised each lot in swing pots, approx. 30 sec
submitted sample for assay, whole rock, Cl, B

measured activity: 72 sec
"Percent Calcined" 91.3
(LOI in calcine / LOI in feed)

Sample ID	Head-Dec 2 Calcine
Si [%]	1.83
Al [%]	0.04
Fe [%]	1.20
Mg [%]	47.5
Ca [%]	2.74
Na [%]	< 0.05
K [%]	< 0.01
Ti [%]	< 0.01
P [%]	< 0.01
Mn [%]	0.14
Cr [%]	0.02
V [%]	< 0.01
LOI [%]	8.11
SUM [Oxide	96.7
Cl [g/t]	80
B_tot [g/t]	<20

5398 - Golden Triangle **INSTRUCTIONS**
Test No . LCH 18

Purpose: To determine the solid-liquid separation characteristics of the neutralized leach liquor, and measure the basic impurity levels of the levels of the final and intermediate solutions.

1) Conduct 2 leaches simultaneously using the following conditions:

crush size: - 10 mesh
Acid Stoich.: 110 %
Temp: 80 °C
Time: 60 minutes

Stage 1

2) At end combine both pulps in one vessel, and record weight
Remove a thief solution sample for analysis. Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T), Free HCl
3) Return to water bath at 70 °C.
4) Neutralise to pH 2.0 with the slow addition of MC-36 calcine record rate of addition and pH. Hold at this pH for 10 minutes.
5) Pour into 1000 ml graduated cylinder and thicken in hot fish tank (50 °C)
6) Record settling rate (no floc addition) for about 60 minutes (keep covered to minimise evaporative losses).
7) Decant solution. Remove thief sample and assay as for initial solution.
8) Conduct filtration and washing tests on thickened solids see attached details.

Stage 2

9) Return solution (decant + filtrate) to water bath (50 °C), and neutralise to pH 5.0 with MC-36 calcine. Hold for 10 minutes. Record rate of addition and pH.
10) Remove a thief solution sample for analysis (as for initial solution).

Stage 3

11) Neutralize to pH 7.0 with an NaOH solution (50 g/l)
Record rate of addition and pH. Hold at this pH for 10 minutes.
12) Remove a thief solution sample for analysis (as for initial solution).
13) Thicken slurry in hot fish tank, and record settling rate (no floc)
14) Decant solution, and perform filtration and washing tests on thickened solids. see attached details.
15) Submit decant + filtrate solution for assay as for initial solution
16) Submit wash solutions for Mg, Ca, Cl
17) Submit washed solids for moisture, whole rock, s.g. and Cl.

Solution samples for analysis:

Test 18 - Preg	Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T), Free
Test 18 - pH 2.0 sol'n	Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T)
Test 18 - pH 2.0 Wash 1 to	Mg, Ca
Test 18 - pH 2.0 clean up	Mg, Ca
Test 18 - pH 4.0 sol'n	Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T)
Test 18 - pH 7.0 sol'n	Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T)
Test 18 - pH 7.0 Wash 1	Mg, Ca
Test 18 - pH 7.0 Wash 2	Mg, Ca
Test 18 - pH 7.0 Wash 3	Mg, Ca
Test 18 - pH 7.0 Wash 4	Mg, Ca

Project: **5398**
Test: **LCH 18**

Technologist: G. Toole
Date: Dec. 3, 1998

Purpose: To determine the solid-liquid separation characteristics of the neutralized leach liquor, and measure the basic impurity levels of the final and intermediate solutions.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight 2 x 200 g
HCl added: 2 x 503 g
Water added: 2 x 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.7 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

Leach:

1) Two duplicate leaches were performed to produce sufficient material for thickeneing.
2) A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for the sample addition.
3) Hydrochloric acid was added to the reactor and heated to the desired temperature.
4) Once at temperature the sample was added slowly to avoid sample loss due to excessive frothing (3 to 5 minutes typically required).
5) Once all the sample was in the test was started.
6) Periodic redox readings were taken during the test.

Stage 1:

7) At end of the leach both pulps were combined, and the pulp weight was recorded. A thief solution sample was taken for analysis (Mg, Ca, Fe, Mn, Ni, Zn, Cu, Cr, Al, Na, Si, B, S(T), Free HCl)
8) The combined pulp was placed in a bath at 70 °C.
9) The pulp was neutralise to pH 2.0 with the slow addition of MC-36 calcine This pH was held for 10 minutes.
10) The pulp was placed in a 1000 ml graduated cylinder and submerged in a 50 °C water bath.
11) The settling rate was recorded for about 60 minutes (no floc addition).
12) The supernatant solution was decanted and the thickened solids filtered using a "pour-on" filter apparatus.

Stage 2

13) The combined decant and initial filtrate solution were recombined and returned to a 50 °C water bath.
14) The solution was neutralised to pH 5.0 with MC-36 calcine. The pH was maintained for 10 minutes with calcine addition as required.
15) A thief solution sample was removed for analysis (assays as for initial solution).

Stage 3

16) The solution was further neutralize to pH 7.0 with an NaOH solution (50 g/l) The pH was held for 10 minutes.
17) A thief solution sample was removed for analysis (assays as for initial solution).

Procedure: - continued

18) The resultant slurry was thickened in a hot bath, and and the settling rate was noted.
19) The supernatant solution was decanted, and a "pour-on" filtration test was performed on the thickened solids. Washing tests were also performed.

Leach Data:

Leach A				Leach B			
Time min	Temp °C	emf mV Ag/AgCl	Comments	Time min	Temp °C	emf mV Ag/AgCl	Comments
0	80	213		0	80	213	
15	81	265	frothing stopped	15	80	257	frothing stopped
30	82	302		30	80	286	
45	81	309		45	80	299	
60	81	325		60	80	316	

Results:

Pulp weight: 1325 g
Pulp pH: -1.47 @ 67.1°C
Pulp emf 348 mV
Solution SG: 1.292 g/mL

Neutralization:

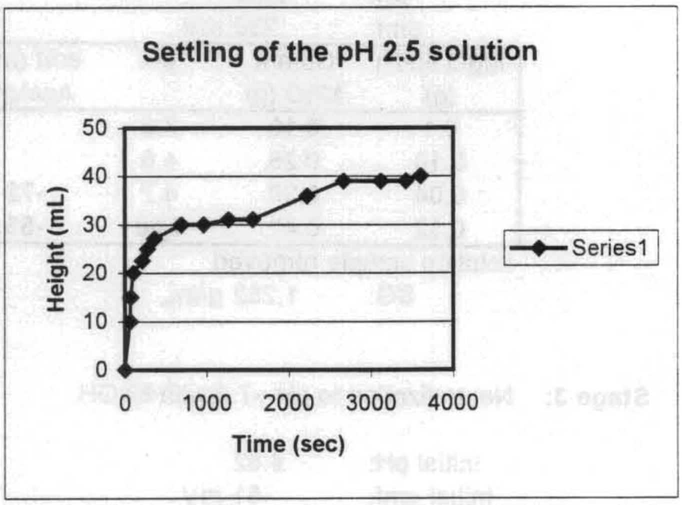
Agent: MC36 ore calcined at 650°C for 1 hour
used 2.11g of calcine to pH 3.8
added 0.86g of 12% HCl to bring pH back to2.5
Solution SG: 1.284 g/mL
Final pH: 2.7
Final emf: 216 mV

Thickening:

Pulp weight: 1235 g
Pulp Volume: 950 mL
Pulp height: 356.3 mm

Time sec	Mud Line mL
0	0
60	10
70	15
100	20
210	22.5
270	25
360	27
690	30
960	30
1260	31
1560	31
2220	36
2670	39
3120	39
3420	39
3600	40

Comments
Mud line is from the bottom



After settling for 1 hour poured of the solution
weight: 1163 g

5 cm

Filtration: **Filter cloth**

Media No.: 1820
Weave: 3-1 twill
Thread Count: 68X60
Weight: 4.4 oz/sq. yd
Air Flow 22 cfm/sq. ft
Finish: heat set

Sample ID	Time (sec)		Vol (ml)		Comments
	filter	dry	in	out	
filtrate	7	7		11.5	about 80% coverage on the cloth
1st wash	11		11.5	11.5	
2nd wash	16		11.5	20	
3rd wash	19		20	21.5	
4th wash	15		20	18.5	
5th wash	15		20	21	
6th wash	15		20	20	

Clean up solution from washing out of grad cylinder and beaker 260mL
All washes filtrate and clean up solution assayed for Mg and Ca

Moisture:

wet weight: 40.86 g
dry weight: 33.9 g
Percent Moisture: 17.0 %

Stage 2: Neutralization to pH ~5.0 with calcine MC36

Initial pH: 3.75
Initial emf: 153 mV
Mixed for 20 minutes
pH: 2.64
emf: 230 mV

MgO add'n (g)	Cumm MgO (g)	pH	emf (mV) Ag/AgCl
0.1	0.10	3.5	
0.16	0.26	4.6	
0.04	0.30	4.7	-72
0.12	0.42	4.66	-58

solution sample removed
SG: 1.252 g/mL

Stage 3: Neutralization to pH ~7.0 with NaOH

Initial pH: 4.52
Initial emf: -51 mV

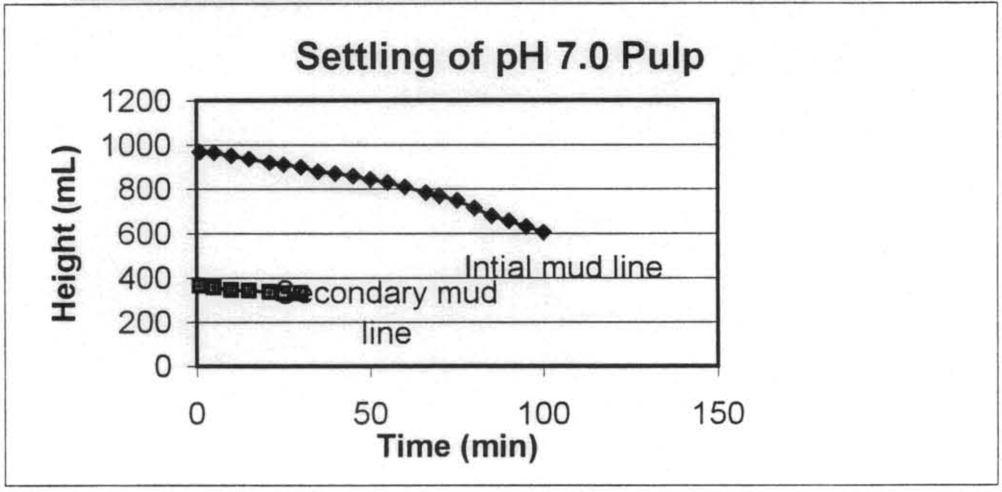
aOH add' 50g/L (g)	Cumm NaOH (g)	pH	emf (mV) Ag/AgCl	Comments
6.62	6.62	4.98	-150	
10.02	16.64	5.11	-164	mixture is now blue
10.14	26.78	5.62	-306	
20.45	47.23	5.92	-454	changed to neutralizing with 50%w/w NaOH
34.16	34.16	6.43	-549	
78.98	113.14	6.78	-570	
24.13	137.27	6.81	-499	

Total amount of NaOH used is 71 g
gelatinous like precipitate grey bluish colour
solution colour is clear
Solution SG: 1.252 g/mL

Thickening:

Pulp weight: 848.9 g
Pulp Volume: 975 mL
Pulp height: 365.63 mm

Time min	initial Mud Line mL	Secondary Mud Line mL	Comments
1	970		
5	965		
10	950		
15	937		
21	920		
25	910		
30	898		
35	880		
40	870		
45	860		
50	845		
55	830		
60	810		
66	785		
70	770	365	Secondary mud line appeared
75	750	355	
80	715	345	
85	680	340	
90	655	335	
95	630	330	
100	605	330	



5 cm

Filtration:

Using a 18.5cm heat buckner(50°C) with Watman 541 filter paper and plant vacuum

Volume of Decant: 370 mL

Sample ID	Time(min)		Vol (ml)		Comments
	filter	dry	in	out	
filtrate	10	2.5		390	volume out during dry 31.5mL
1st wash	14	2.5	100	122.5	
2nd wash	38	6	100	163	started to see a lot of cracks
3rd wash	37	2.5	300		Partially repulped with 300mL*
4th wash	21	2.5	100	152	

* probably increased grey precipitate(silica?) in volume, blue precipitate stuck everywhere

Moisture:

wet weight:	152.96 g
dry weight:	53.2 g
Percent Moisture:	65.2 %

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Stage 1 Residue

Filtration:

Dry Cake Weight:	33.9	Filter Diameter	11 cm
Wet Cake Weight:	40.86	Filter Area	0.00950332 m^2
Moisture Content:	17.0		(about 80% utilized)

Stage	Time (sec)		Vol (ml)	Cake Rate (kg/hr/m^2) form only		Filtrate Rate (l/hr/m^2) form only
	Form	Dry		wet	dry	
Filtration	7	7	11.5	2764	2293	778
Wash 1	11		11.5			495
Wash 2	16		20.0			592
Wash 3	19		21.5			536
Wash 4	15		18.5			584
Wash 5	15		21.0			663
Wash 6	15		20.0			631

Wash (ave) 584

Stage 3 Residue

Filtration:

Dry Cake Weight:	53.2	Filter Diameter	11 cm
Wet Cake Weight:	152.96	Filter Area	0.00950332 m^2
Moisture Content:	65.2		

Stage	Time (sec)		Vol (ml)	Cake Rate (kg/hr/m^2) form only		Filtrate Rate (l/hr/m^2) form only
	Form	Dry		wet	dry	
Filtration	600	150	390.0	96.6	34	246
Wash 1	840		122.5			55
Wash 2	2280		163.0			27
Wash 3	2220		300.0			51
Wash 4	1260		152.0			46

Wash (ave) 45

Product	Amount	Assays (mg/l, %)															
	mL/g	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Cr	Al	Na	Si	B	S(t)	Cl	Free Acid (g/l)	s.g.
Leach Preg	~980	107100	6390	2060	263	<0.50	<5.00	<1.00	<0.10	<0.50	26.2	<5.00	<0.50	700	na	1.3	na
pH 2.0 solution	~980	103000	5970	1760	259	<0.50	<5.00	<1.00	<0.10	6	26.5	<5.00	<0.50	700	na	na	na
pH 2.0 filtrate	11.5	92900	5530	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 1	11.5	73300	4360	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 2	20	43100	2730	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 3	21.5	14100	742	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 4	18.5	2770	158	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 5	21	768	52.5	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 wash 6	20	317	58.3	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 clean up	260	2900	29.7	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 2.0 solids	33.9	107000	0.19	1.53	0.09	0.004	0.004	0.002	0.01	0.07	<0.05	10.42	na	na	1916	na	2.82
pH 5.0 solution	~750	71400	6140	1410	245	<0.50	<5.00	<1.00	<0.10	<0.50	23.1	<5.00	<0.50	664	na	na	na
pH 7.0 solution	791.5	63300	4790	0.34	6.32	<0.50	<5.00	<1.00	<0.10	<0.50	16800	<5.00	<0.50	220	na	na	na
pH 7.0 wash 1	122.5	98200	4290	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 7.0 wash 2	163	42.0	644	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 7.0 wash 3		296.0	16.3	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 7.0 wash 4	152	296.0	12.9	na	na	na	na	na	na	na	na	na	na	na	na	na	na
pH 7.0 solids	53.2	36.4	0.04	3.27	0.40	0.001	0.020	0.003	0.01	0.04	0.36	0.68	na	na	23226	na	2.63

5398 - GTR **INSTRUCTIONS**

Test 19

Purpose: Determine effect of oxidation on precipitation of iron,
and the addition of H_2SO_4 on the precipitation of gypsum
from "Combine Leach Solution (1-4, 9-10)"

Procedure:

Stage 1 - Iron oxidation and precipitation

Place 500 ml of solution in beaker in 50 °C water bath.
Heat to temperature
Mix, inject low flow of air
Raise emf of solution as high as possible (emf = 600 mV?)
    with the addition of hydrogen peroxide
Slowly and carfully raise pH to 2.0 with addition of calcine.
    keep emf high by adding air and peroxide
Mix, and maintain for 15 minutes
Raise pH to 3.0 with calcine, maintain for 15 minutes
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg, Ca, and Fe
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, Si, Mn, Cu, Zn, Ni, Mn, B

Stage 2 - Gypsum Precipitation

Place solution in beaker back in water bath (record volume)
Raise pH to 4.8 with calcine
add 25 ml of 100 g/l H_2SO_4, mix 5 minutes
remove 25 ml of solution, filter into container
    assay for Mg and Ca
repeat acid addition and sampling procedure 3 more times
measure and record pH and emf during procedure.
make one last addition of acid, mix 5 minutes
filter total slurry
submit filtrate solution for assays as above
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Tech. G. Toole

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LCH 19

Time	Temp °C	pH	emf mV	Additions (g, mL)					Comments
				Air (mL/min)	Calcine MgO	H2O2 35%w/w	H2SO4 100g/L	HCl 12%	
Stage 1									
8:20	42.0	-0.6	394	268		0.41			
8:25	48.2	0.87	460	268		0.51			
8:27	49.2	1.06	877	268					
8:33	49.5	0.99	873	268					
8:35	49.6	0.98	860	268					
8:38	49.6	0.96	838	268					
8:40	49.6	1.03	806	268	0.09				
8:43	49.6	1.10	780	268	0.19				
8:45	49.6	1.12	757	268	0.25				
8:48	49.6	1.17	732	268					
8:52	49.9	1.22	722	268					
8:54	49.9	1.28	712	268	0.18				
8:56	49.9	1.35	697	268	0.17				
8:58	49.9	1.38	845	268		0.10			
9:00	49.9	1.46	795	268	0.08				
9:01	49.9	1.66	671	268	0.1				
9:03	49.9	2.74	564	268		0.22			
9:06	49.9	3.62	491	268					
9:08	49.9	4.23	459	268		0.57			
9:09	49.9	4.40	444	268					
9:11	49.9	4.11	459	268				0.31	
9:15	49.9	4.43	435	268				0.12	
Stage 2									
9:51	41.7	5.46	300						
10:16	48.5	5.48	284						
10:20	48.5	0.5	646				1		
10:26	48.5	2.32	566		0.1				
10:38	49.5	2.19	896		1.5				
10:40	48.9	3.74	466						
10:42	48.9	5.81	320				2		
10:50	48.9	5.77	373						
11:12	48.9	4.88	416		1.18				
11:14	48.9	5.79	315						
11:16	48.9	-0.62	918				12		
11:47	48.9	4.83	316		1.18				
11:50	47.6	5.62	306						
11:52	47.3	-0.45	933				12		
12:23	47.9	4.6	427		1.12				Fisher MgO
12:26	48.1	5.44	359						
12:32	47.8	-0.44	862				12.5		
13:00	48.4	4.7	431		1.2				

5398
LCH 19

Stage 1
Filtered on 18.5cm buchner (50°C) with Whatman 541 paper
Filtrate Form time 8min, dry 1.5min
Wash ~1min form and ~1min dry
Fitrate volume: 465 mL 25mL sample taken
Filtrate weight: 591.9 g
Solution SG: 1.273 g/L
Wash volume: 98 mL

Stage 2
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form ~1min, dry 2min

Fitrate volume: 385 mL
Filtrate weight: 476.1 g
Solution SG: 1.237 g/L
Wash volume: 37 mL

Product	Amount (ml /g)	Assays (mg/l, %)									
		Mg	Ca	Fe	Mn	Ni	Zn	Cu	Na	Si	B
pH 3.0 filtrate	10	105000	5210	7.72	223	<0.10	5.25	0.39	20.3	<0.50	<0.1
pH 3.0 wash	98	24000	1420	7.79							
pH 3.0 solids	2.7	8.15	1.65	36.3	0.9				0.05	1.25	
acid 1 preg	25	105000	3210	0.95							
acid 2 preg	25	104000	1610	0.93							
acid 3 preg	25	93500	616	1.05							
acid 4 preg	25	100000	353	2.3							
acid 5 preg	385	94100	274	7.95	192	<0.10	4.86	0.35	25.8	0.92	<0.10
acid 5 wash	37	41000	1360	40.5							
Final solids	7.7	4.06	22.3	0.45	0.02				<0.05	0.77	
Head (calc)	500	103485	4507	2041							
Head (dir)		94800	5260	1940	251	< 0.10	8.06	2.53	5.4	7.6	< 0.10

Product	Distribution (%)		
	Mg	Ca	Fe
pH 3.0 filtrate	2.0	2.3	0.0
pH 3.0 wash	4.5	6.2	0.1
pH 3.0 solids	0.4	2.0	96.1
acid 1 preg	5.1	3.6	0.0
acid 2 preg	5.0	1.8	0.0
acid 3 preg	4.5	0.7	0.0
acid 4 preg	4.8	0.4	0.0
acid 5 preg	70.0	4.7	0.3
acid 5 wash	2.9	2.2	0.1
Final solids	0.6	76.2	3.4
Feed Sol'n	100	100	100

5398 - GTR
Test 20

INSTRUCTIONS

Purpose: Determine solid liquid separation characteristics of leach pulp which has been oxidized, de-calcified and neutralised.

Procedure:

Leach

- conduct two - 200 gram leaches @
  60 minutes
  80 °C
  110% stoichiometric acid addition.
- at the end of leach mix two slurries together, measure total weight
- remove solution sample for assay (Mg, Ca, Si, Mn, Fe, S, Cl)
- return slurry to water bath at 50 °C

Purification

- inject air in slurry at about 300 ml/min
- raise emf to approx 800 with addition of peroxide,
  record amount of H_2O_2 used.
- continue to inject air, mix 10 minutes
- raise the pH to 4.8 with calcine, mix 10 minutes
- add 17.2 **grams** H_2SO_4 (96% reagent grade)
- mix 10 minutes
- raise pH slowly to about 2.5 to 3.0 with calcine
- mix, and maintain for 10 minutes
- raise pH to 4.8 with high grade MgO
  maintain for 10 minutes (add dilute HCl if necessary)
- remove solution sample for assay (Mg, Ca, Si, Mn, Fe, S, Cl)
- add 5.33 g $BaCl_2 \cdot 2H_2O$, mix 10 minutes,
  then remove solution sample for S assay

Solid / Liquid Separation

- transfer slurry to 2000 ml graduated cylinder
- place in fish tank at 50 °C
- mix up solids and record settling rate.
- after 60 to 90 minutes remove supernatant, record volume and s.g.
- remove solution sample for assay (Mg, Ca, Si, Mn, Fe, S, Cl)
- conduct pour-on filtration and washing tests on thickened solids
- follow Mg, and Cl in wash solutions
- submit washed cake for moisture, s.g., and whole rock

Project: **5398**
Test: **LCH 20**

Technologist: G. Toole
Date: Dec. 3, 1998

Purpose: To determine the solid-liquid separation characteristics of the neutralized leach liquor, and measure the basic impurity levels of the levels of the final and intermediate solutions.

Leach Conditions:

Leach Feed:	MC36
Crush Size:	minus 10 mesh
Feed weight	2 x 20 g
HCl added:	2 x 50 g
Water added:	2 x 73 g
HCl Concentration:	36 %
HCl Stoichiometric concentration:	110 %
Initial Percent Solids:	25.7 %w/w
Initial L/S	2.48 ml sol'n / g solid
Temperature:	80 °C
Time:	60 min
pH:	not controlled
Mixer Speed:	900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for added the sample.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid frothing over.
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test both samples were combined and a thief sample was taken

Data:

Leach A				Leach B			
Time min	Temp °C	emf mV*	Comments	Time min	Temp °C	emf mV*	Comments
0	80	222		0	80	224	
15	80	266	frothing stopped	15	80	257	frothing stopped
30	80	293		30	80	293	
45	80	304		45	80	303	
60	80	325		60	80	325	

* vs Ag/AgCl electrode

Results:

Pulp weight:	1386	g	Solution SG:	1.274 g/mL
Pulp pH:	-1.41	@ 51.6°C		
Pulp emf:	347	mV		

5398
LCH 20

Time	Temp °C	pH	emf (mV) Ag/ AgCl	Additions (g, ml/min) Air	 H_2O_2 35%	 Calcine	 H_2SO_4 (conc)	 MgO 99.998%	Comments
8:15	51.6	-1.41	347	268					
8:22	53.2	-1.53	491	268	1.1				
8:24	53	-1.33	908	268	1				
8:34	52.1	-1.31	920	268					
8:40	51.1	-1.32	897	268		0.51			
8:47	50	-1.26	882	268		0.5			
8:50	50.2	-1.2	866	268		0.63			
8:54	50.1	-1.14	820	268		0.55			
8:56	50.1	-1.09	789	268		0.28			
9:00	50	-0.98	737	268		0.48			
9:04	49.9	-0.74	727	268		0.62			
9:06	49.8	-0.44	724	268		0.37			
9:10	50	0.61	723	268		0.35			
9:20	50.6	1.13	399	268					
9:23	50.7	1.29	688	268		0.22			
9:30	50.8	1.09	672	268		0.61			
9:35	51.4	1.19	661	268		1.37			
9:50	51.6	1.39	627	268		1.45			
9:45	51.6	3.55	391	268		0.69			
9:45	51.6	5.2	335	268			2 drops		
9:47	adding sulfuric drop wise to maintain pH before sample								
9:57	51.2	4.75	280	268					sample
10:00							17.2		
10:02	53.4	-1.28	917	268					
10:19	51.6	-1.21	771	268					sample
10:24	52	-1.09	837	268		1.28			
10:26	52.2	-0.94	857	268		0.64			
10:30	52.9	-0.78	833	268		0.36			
10:34	51.1	-0.22	860	268		0.92			
10:35	52	0.08	852	268		0.17			
10:43	51.7	1.05	804	268		0.22			
10:46	51.7	1.32	777	268		0.24			
10:48	51.7	1.15	737	268		0.09			
10:52	51.6	1.17	716	268		0.29			
10:53	51.6	1.13	708	268		0.12			
10:54	51.6	1.14	702	268		0.18			
10:57	41.6	1.11	697	268				0.15	
10:59	51.7	1.13	690	268				0.3	
11:02	51.7	1.14	683	268				0.41	
11:06	51.7	1.18	671	268				0.46	
11:09	51.8	1.19	659	268				0.18	
11:11	51.7	1.21	655	268				0.18	
11:13	51.7	1.25	649	268				0.17	
11:15	51.7	1.27	643	268				0.19	
11:17	51.6	1.32	633	268				0.12	

5398
LCH 20

Time	Temp °C	pH	emf (mV) Ag/ AgCl	Additions (g, ml/min) Air	H_2O_2 35%	Calcine	H_2SO_4 (conc)	MgO 99.998%	Comments
11:19	51.6	1.38	624	268				0.17	
11:22	51.6	1.49	601	268				0.17	
11:25	51.6	1.74	565	268				0.07	
11:27	51.3	2.5	468	268				0.06	
11:29	51.2	3.88	367	268					
11:30	51.2	4.44	337	268					
11:33	51.2	5.13	320	268					adding 12% HCl to maintain pH
11:35	51.2	4.85	300	268					sample
11:40	51.2	5.12	417	268	3.37				
11:52	51.2	4.64	427	268					sample
		4.61	425	268					5.33g $BaCl_2$•H_20+2.43g
11:56	51.2								and HCl to maintian pH

pH 4.8 sol'nSG: 1.265 g/mL
Mn sol'n SG: 1.262 g/mL
$BaSO_4$ sol'n SG: 1.247 g/mL

5398
LCH 20

Product	Amount	Assays(mg/L, %)								
	(ml, g)	Mg	Ca	Si	Mn	Fe	S	$S^=$	Cl	s.g.
Preg		96400	5210	6.1	270	2030	<0.5		318000	
pH4.8 sol'n		101000	689	1.29	217	3.51	1000		284000	
H2O2 sol'n					214					
BaSO4 sol'n							1050	<2		
Decant	815	10100	744	1.05	203	<0.1	1050		289000	
Filtrate	140	8260							233000	
Wash 1	140	19100							55000	
Wash 2	51	10100							33100	
Wash 3	50.5	1920							7240	
Wash 4	54	513							1490	
Residue	50.9	13.6	6.23	7.9	13.8	4.68				2.87

Lakefield Research Limited

SETTLING TEST REPORT

Project: **Golden Triangle Resources**
Project Number: LR-5398
Sample : Test 20 pulp
Test: 20
Date: 8-Dec-98
Operator: G.Toole

Flocculant 1: E-10
Flocculant 2: None
pH Modifier: None
Concentration: Wt.%
Weight Added: g
pH: (measured)

	Floc 1	Floc 2	
Concentration:	1.00		g/L
Volume Added:	1.0		mL

Initial Pulp Volume: 985 mL
Initial Pulp Height: 337.5 mm.
Initial Net Pulp Weight: 1287.7 g
Dry Solids Weight: 100.0 g
Meas. Dry Sol. Weight: 50.9 g
Settling Vessel Size: 0.0029 m3/m .... 890 mL/ft
Dry Solids S.G.: 2.87 g/cm^3
Liquid S.G.: 1.25 g/cm^3
Temperature: 54.0 Degrees C

Feed % Solids based on pulp density
Pulp Density: 1307 g/L
7.77 %Sol

Settling Test - Measurements

Time Min	Mud Line mL	Time Min	Mud Line mL	Time Min	Mud Line mL
0	985	2.9	410	8	255
0.2	950	3	400	9	245
0.4	900	3.3	380	10	238
0.6	850	3.5	370	11	230
0.8	800	3.6	360	15	210
1.2	750	3.8	350	20	190
1.4	650	4	340	25	183
1.6	600	4.3	330	30	179
1.8	550	4.6	320	40	170
2.1	500	4.9	310	50	165
2.3	470	5.3	300	60	160
2.5	450	6	282		
2.7	430	7	270		

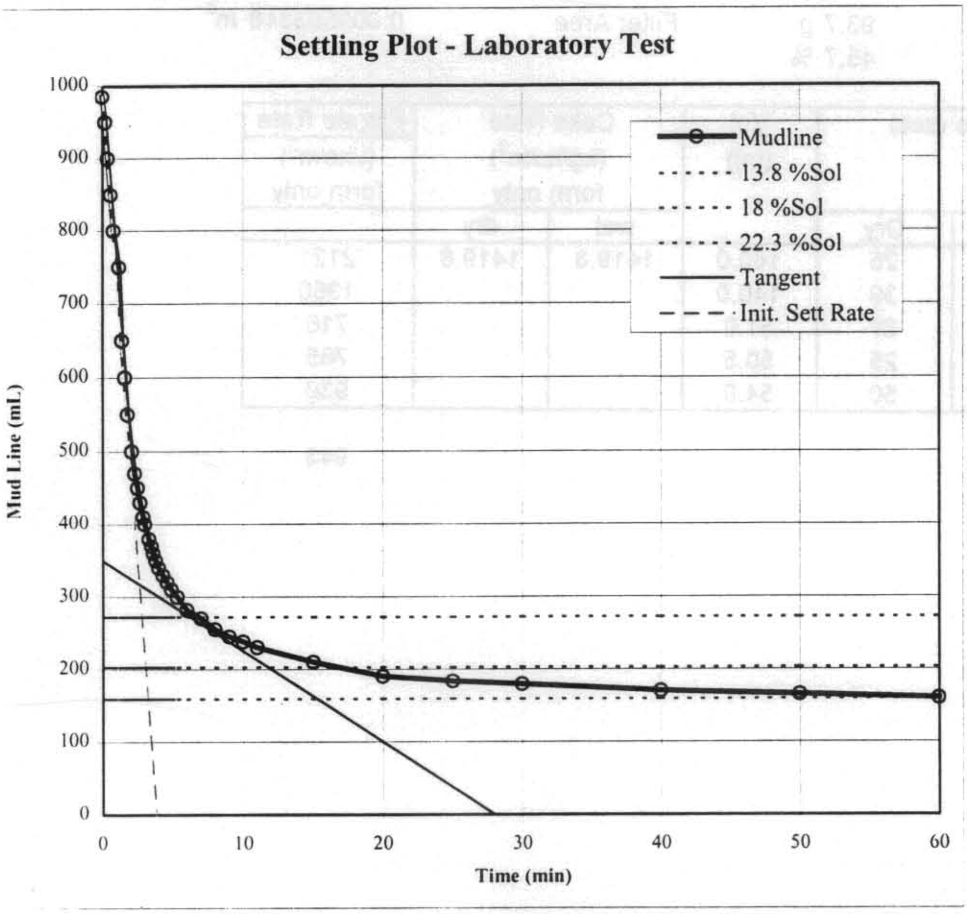
Supernatant after 1 hour: quite turbide fines moving around
Supernatant at final reading:

Observations:

Lakefield Research Limited
SETTLING TEST REPORT

Project : **Golden Triangle Resources**
Project Number : LR-5398
Test : 20
Sample : Test 20 pulp
Date : 8-Dec-98

Settling Plot - Laboratory Test



Reagents	Dosage (g/MT)
E-10	19.6

Dry Solids S.G.: 2.87 g/cm^3
Liquid S.G.: 1.25 g/cm^3
Temperature: 54.0 ^{0}C
pH:

5 cm

THICKENER UNIT AREA CALCULATION

Rise Rate :

Initial Settling Rate: 127.89 m^3/m^2.day
Safety factor: none
Rise Rate: **127.89** m^3/m^2.day **2.18** USGPM/ft^2

Feed and Discharge Data :

Feed Percent Solids: 4.0 Wt. % Final % Solids : 22.3 Wt. %
Fd Density: 1279 g/L

Underflow % Sol (Wt)	Density (g/L)	Underflow Intersect with tangent (min)	Thickener Underflow Unit Area (Talmage and Fitch) (No Safety Factor) (m^2/MTPD)	(ft^2/STPD)	Thickener Hydraulic Unit Area (Overflow Capacity) (No Safety Factor) (m^2/MTPD)	(ft^2/STPD)
13.8	1356	6.2	**0.248**	*2.42*	**0.137**	*1.34*
18.0	1391	11.7	**0.468**	*4.56*	**0.150**	*1.47*
22.3	1430	15.2	**0.606**	*5.92*	**0.158**	*1.55*

Note: The thickener unit area calculations contain no safety factor

All boxed data are operator input

5398
LCH 20

Filtration:

Cake Thickness: 4.0 mm
Dry Cake Weight: 50.9 g
Wet Cake Weight: 93.7 g
Moisture Content: 45.7 %

Filter Diameter 11 cm
Filter Area 0.009503318 m^2

Stage	Time (sec)		Vol (ml)	Cake Rate ($kg/hr/m^2$) form only		Filtrate Rate ($l/hr/m^2$) form only
	Form	Dry		wet	dry	
Filtration	25	25	140.0	1419.8	1419.8	2121
Wash 1	39	39	140.0			1360
Wash 2	27	27	51.0			716
Wash 3	25	25	50.5			765
Wash 4	22	50	54.0			930

Wash (ave)	943

5398
LCH 21

This test may be found in
Progress Report No. 1 - Leaching

5398
LCH 22

This test may be found in
Progress Report No. 1 - Leaching

5398
LCH 23

This test may be found in
Progress Report No. 1 - Leaching

Project: **5398**
Test: **Test 24**

Technologist: G. Toole
Date: Dec. 10, 1998

Purpose: To determine if Mn can be selectively precipitated from Mg at high pH.

Procedure:

Feed Solution: Test 20 preg
Feed volume: 380

1. 380 mL was placed in a 600mL beaker and heated to 50°C
2. Initial pH and emf readings were taken
3. High grade MgO(99.998%) was added slowly to raise pH
4. Samples were taken a different points between the starting pH and pH 6.6
5. Samples were analyzed for Mg and Mn
6. These samples were diluted with 12% HCl. The first was 5:100 and the rest were 10:100

Data:

Time min	Temp °C	emf mV Ag/AgCl	pH	Additions (g, mL)		
				MgO (99.998%)	NaOH (50g/L)	
11:54	42.8	290	5.14			
12:42	46.3	305	5.65			
13:00	46.3	307	5.65			
13:06	47.2	295	5.77	0.04		
13:09	47.2	292	5.80			Sample dilution 5:100
13:31	47.2	282	5.89	0.11		
13:35	46.7	278	5.92	0.21		
13:45	46.7	271	6.00	0.1		Sample dilution 10:100
13:57	47.8	260	6.03	0.24		
14:03	48.6	256	6.05	0.63		
14:15	48.6	246	5.99	0.16		
14:20	48.9	243	5.81			
14:30	48.9	237	5.90	0.21		
14:38	48.9	235	5.55			
14:40	49	239	5.72			
14:43	47.3	182	6.07			
14:46	48.3	229	6.11	0.32		
14:48	49.2	235	5.99	0.23		
14:55	48.9	236	5.45	0.29		
15:00	49.3	236	5.24			
15:05	49.5	239	5.88			
15:10	49.3		5.07		7	
15:15	49.3	226	5.33			
15:18	48.9	214	5.61		10	
15:20	48.9				10	
15:25	48.9		6.00		5.6	
15:30	48.9		6.12		4.4	
15:30	48.9		6.18		4	Sample dilution 10:100
15:45	48.3	163	6.39		11.8	Sample dilution 10:100

Solution was taken out of water bath and allowed to sit over night
pH in the morning was 6.60 another sample was taken dilution of 10:100
the solids were also analzed for Mg and Mn

Results

Product	Amoun (ml, g)	Assays (mg/l, %)		Distribution (%)	
		Mg	Mn	Mg	Mn
pH5.8	5	90000	162		
pH6.0	10	92200	154		
pH6.2	10	89300	136		
pH6.4	10	87600	126		
pH6.6	245	90400	56.3	70.1	23.8
wash	95	62900	0.45	18.9	0.1
precip	14.7	23.5	0.30	10.9	76.1
Head (dir)	380	94650	271		
Head (cal)		83100	152	100	100

5398
LCH 25

This test may be found in
Progress Report No. 1 - Leaching

5398
LCH 26

This test may be found in
Progress Report No. 1 - Leaching

5398 - GTR **INSTRUCTIONS**
Test LCH 27
Purpose: Attempt U-process for purification of MgCl solution with modifcations

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
combine pulp at conclusion of leach
take weight and sample for analysis
Mg, Ca, Si, Fe, Na, Mn, Cu, Zn, B, Ni, S, V, Cr, Al

Stage 1 - Iron oxidation and precipitation

Place pulp in beaker in 50 °C water bath.
Heat to temperature
Mix, inject low flow of air
Raise emf of solution as high as possible (emf = 600 mV?)
with the addition of hydrogen peroxide
Added 13 g of reagent grade sulfuric acid
Mix for 30 minutes
Raise pH to 2.0 with calcine
Add some peroxide
Raise pH to 3.0-4.0 with calcine, maintain for 15 minutes
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg, Ca, and Fe
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, Si, Mn, Cu, Zn, Ni, Mn, B, V

Stage 2 -

Added few drops of HCl to pH ~2.0 (measure emf)
Add 4g of $FeCl_2 \cdot H_2O$ mix
Add 5.33 g $BaCl_2 \cdot 2H_2O$
Mix 5 minutes
Added reagent grade $Mg(OH)_2$ to water(1gram) at a time to raise pH to ~5.5
Filter
submit filtrate solution for assays as above + Ba
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Stage 3-

Reheat filtrate to ~50°C
Raise emf to high as possible with peroxide and air
Add 2 g of $Mg(OH)_2$ mixed with DI water
mix 5 minutes and filter
Repeat above once more
submit filtrate solution(diluted in 12% HCl) for assays as above + Ba
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Tech. G. Toole

5398
LCH 27

Time	Temp °C	pH	emf mV	Additions (g, mL)					Comments
				Air (mL/min	Calcine MgO	H_2O_2 35%w/w	H_2SO_4 conc	HCl 12%	
Stage 1									
9:02	54	-1.05	363	268					
9:07	55	-1.04	694	268		1.3			
9:09	55	-1.20	897	268		0.38			
9:13	55	-1.12	947	268			13		
9:36	53	-1.15	736	268					
9:40	53	-1.13	950	268		0.57			
9:43	53	-1.13	971	268					
9:47	55	-1.05	942	268	4.16				
9:52	55	-0.75	884	268	5.62				
9:55	54	0.62	751	268	2.5				
10:00	54	1.45	883	268					
10:12	53	1.29	708	268					
10:17	53	1.42	693	268	2.68				
10:20	52	1.57	673	268	1.57				
10:24	52	1.75	749	268		0.26			
10:26	52	2.20	644	268					
10:27	52	2.80	622	268		0.4			
10:29	52	3.88	519	268					Filter
Stage 2									
11:09	49	5.09	378						
11:10	49	2.19	344					0.18	
11:11	49	2.17	303						4g $FeCl_2 \cdot H_2O$
11:16	50	2.13	305						
11:17	50	2.13	306						add 5.33g $BaCl_2 \cdot 2H_2O$
11:22	50	2.07	-204						
11:24	50	5.05	-235						< 1g $Mg(OH)_2$ + water
Stage 3									
11:27	50	5.14	-131						Filter
11:48	45	4.25	433	268					
11:50	45	1.64	-151	268		0.32			
11:53	45	5.22	396	268					2g $Mg(OH)_2$ + water
11:57	45	5.44	293	268		0.84			
13:35	50.3	5.92	380	268					
13:43	50.3	5.86	363	268					4g $Mg(OH)_2$ + water
13:45	50.3	5.95	362	268					Filter

5398
LCH 27

Stage 1

Preg and Calcine additon
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form time 5min, dry 2min

Fitrate volume:	960 mL	25mL sample taken	
Filtrate weight:	1222 g	wet weight:	100.93 g
Solution SG:	1.272 g/L	dry weight:	62.7 g
Wash volume:	63 mL	Percent Moisture:	37.878 %

Stage 2

Ferrous Chloride addition and Barium Chloride
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form time 6min, dry 1min

Fitrate volume:	905 mL	25mL sample taken
Filtrate weight:	1149 g	
Solution SG:	1.270 g/L	
Wash volume:	25 mL	

Stage 3

Peroxide and Magnesium hydroxide
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form 48 min

Stage 3b

Fitrate volume:	785 mL	diluted 10 in 100
Filtrate weight:	998.6 g	12% HCl
Solution SG:	1.272 g/L	

Product	Amount	Assays (mg/l, %)														
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Na	Si	B	Ba	V	Cr	S	Al
combined preg	~980	96800	5420	1820	233	<0.10	<6.32	0.79	2.14	10.1	<0.10			<0.10	5.09	9.11
filtrate 1	960	97100	1190	29.8	216	1.28	7.24	0.21	1.78	1.57	<0.10			<0.10	603	1.5
wash 1	63	78400	1730													
residue 1	62.7	14.3	7.12	3.97	0.12				<0.05	6.39			<0.01	0.01		0.08
filtrate 2	905	97400	1190	1080	216	1.15	7.32	0.23	1.66	0.55	<0.10	10.1		<0.10	450	1.36
wash 2	25	48700	670													
residue 2	2.4	1.42	0.13	0.88	0.01				0.05	0.5		57.9	<0.01	<0.01		0.3
residue 3	3.0	11.5	0.31	6.77	0.41				<0.06	0.43			<0.01	0.02		0.17
filtrate 4	785	101000	1300	4.20	54.50	<0.10	8.6	<0.10	<0.50	8.30	<0.10	2.40		<0.10	443	<0.50
residue 4	...	not assayed														

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5398 - GTR **INSTRUCTIONS**
Test 28
Purpose: Attempt U-process for purification of MgCl solution with modifcations

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
combine pulp at conclusion of leach
take weight and sample for analysis
Mg, Ca, Si, Fe, Na, Mn, Cu, Zn, B, Ni, S, V, Cr, Al

Stage 1 - Iron oxidation and precipitation

Place pulp in beaker in 50 °C water bath.
Heat to temperature
Mix, inject low flow of air
Raise emf of solution as high as possible (emf = 600 mV?)
with the addition of hydrogen peroxide
Raise pH to 3.0-4.0 with calcine, maintain for 15 minutes
Added 13 g of reagent grade sulfuric acid
Mix for 30 minutes
Raise pH to 1.5 with calcine
Add some peroxide
Raise pH to 3.0-4.0 with $Mg(OH)_2$, maintain for 15 minutes
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg, Ca, and Fe
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, Si, Mn, Cu, Zn, Ni, Mn, B, V, Al, Cr

Stage 2 -

Added few drops of HCl to pH ~2.0 (measure emf)
Sparge SO_2 into sample until emf is stable
Added reagent grade $Mg(OH)_2$ to water(1gram) at a time to raise pH to ~5.5
Mix 5 minutes
Add 5.33 g $BaCl_2 \cdot 2H_2O$
Filter
submit filtrate solution for assays as above + Ba
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Stage 3-

Reheat filtrate to ~50°C
Raise emf to high as possible with peroxide and air
Add 2.5 g of $Mg(OH)_2$ mixed with DI water
mix 5 minutes and filter
Repeat above once more
submit filtrate solution(diluted in 12% HCl) for assays as above + Ba
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

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Time	Temp °C	pH	emf mV	Additions (g, mL)					Comments
				Air (mL/min)	Calcine MgO	H2O2 35%w/w	H2SO conc	HCl 12%	
Stage 1									
9:04	52	-0.9	341	268					leach preg SG 1.287g/mL
9:08	54	-0.53	879	268		2.82			pulp weight 1349.5g
9:15	53	1.19	922	268	0.76				
9:23	53	1.53	892	268	4.05				
9:25	53	1.62	886	268	0.15				
9:29	53	2.52	784	268					
9:30	53	3.35	569	268		0.72			
9:31	53	4.00	498	268			13		
9:41	52	-0.74	872	268					
9:44	52	-0.29	852	268	1.46				
9:46	52	0.32	794	268	0.43				
9:50	52	1.5	885	268	0.48				
10:00	52	1.39	728	268	1				Change to Adding $Mg(OH)_2$
10:02	52	1.53	895	268	1				
10:05	50	1.76	829	268	1				
10:12	50	1.82	712	268	0.78				
10:16	50	3.28	566	268	0.2	0.82			
10:18	50	3.75	522	268					
10:20	50	3.93	504	268					
10:25	50	3.33	511	268				0.91	
10:29	50	3.59	526	268					
10:38	50	3.87	499	268					
10:43	50	3.97	488	268					filter
Stage 2									
11:39	44	3.52	467						
11:41	44	3.96	441						1drop conc HCl
11:42	44	1.32	531						add SO_2
11:49	44	1.15	376						
11:50	46	0.97	388						
11:52	46	4.72	183						.47 g $Mg(OH)_2$
11:56	46	5.89	64						add some dilute HCl no effect
12:00	48	5.95	82						filter
Stage 3									
13:03	50.3	6.1	187						
13:23	50	6.16	328			1.57			2.5g $Mg(OH)_2$
Stage 3b									
8:56	48	5.99	361						
9:02	49	5.98	350						
9:03	49	6.08	312			2.33			2.5g $Mg(OH)_3$
9:08	50	6.12	324						

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LCH 28

Stage 1

Preg and Calcine additon
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form time 7min, dry 2min

itrate volume:	1000 mL	25mL sample taken	
Filtrate weight:	1264 g	wet weight:	81.74 g
Solution SG:	1.264 g/L	dry weight:	g
Wash volume:	75 mL	Percent Moisture:	100 %

Stage 2

Sulfur Dioxide addition and Barium Chloride
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form time 6min,

itrate volume:	960 mL	25mL sample taken
Filtrate weight:	1211 g	
Solution SG:	1.261 g/L	
Wash volume:	93 mL	

Stage 3

Peroxide and Magnesium oxide
Filtered on 18.5cm buchner(50°C) with Whatman 541 paper
Filtrate Form 2 hours

itrate volume:	875 mL	10mL sample taken
Filtrate weight:	1117 g	diluted to 100mL
Solution SG:	1.276 g/L	in 12% HCl

Stage 3b

Peroxide and Magnesium oxide
Filtered on 18.5cm buchner(50°C) with Whatman 1 paper
Filtrate Form 3hours

itrate volume:	700 mL	10mL sample taken
Filtrate weight:	892.5 g	diluted to 100mL
Solution SG:	1.275 g/L	in 12% HCl

Product	Amount	Assays (mg/l, %)														
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Na	Si	B	Ba	V	Cr	S	Al
combined preg	~980	96800	5510	2150	248	<0.10	<6.12	0.90	5.79	7.37	<0.50	3.27		<0.10	3.31	8.77
filtrate 1	1000	10400	1250	2.35	237	<0.10	6.12	0.14	3.86	1.41	<0.50	0.1		<0.10	6.49	1.26
wash 1	75	58800	2940													
residue 1	56.2	15.5	7.52	4.58	0.09				<0.05	7.36			<0.01	<0.01		0.08
filtrate 2	960	93900	1280	0.66	219	<0.10	5.86	0.14	3.99	1.39	<0.50	30.1		<0.10	6.04	1.4
wash 2	93	20100	314													
residue 2	1.8	2.61	0.26	0.14	0.01				0.06	0.08		59.7	<0.01	<0.01		0.24
filtrate 3	875	96000	1400	2.5	198	<0.10	10.5	<0.10	5.9	6.2	<0.50	11.7		<0.10	697	2.9
residue 3	1.8	28.5	0.47	0.11	1.2				<0.05	0.12			<0.01	0.01		0.07
filtrate 4	700	94100	1400	1.90	357	<0.10	6.4	<0.10	6.10	2.30	<0.50	1.40		<0.10	615	2.5
residue 4	fused to paper	no assays possible														

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5398 - GTR
Test 29
Purpose: Attempt U-process for purification of $MgCl_2$ solution

INSTRUCTIONS

Procedure:

Conduct 1 leaches on MC36 110% stoich acid 80°C and 60 minutes
Take a solution sample for analysis
Mg, Ca, Si, Fe, Na, Mn, Cu, Zn, B, Ni, S, V, Cr, Al

Stage 1 - Iron oxidation and precipitation

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Raise emf of solution as high as possible (emf = 600 mV?)
with the addition of hydrogen peroxide every 10 minutes for 30 minutes
Raise pH to 3.5-4.0 with $Mg(OH)_2$, maintain for 15 minutes
while adding hydrogen peroxide
Mix 15 minutes
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, Si, Mn, Cu, Zn, Ni, Mn, B, V, Al, Cr

Stage 2 -

Added few drops of HCl to pH ~2.0 (measure emf)
Add 4 g of $FeCl_2 \cdot H_2O$
Added reagent grade $Mg(OH)_2$ to water(1gram) at a time to raise pH to ~5.5
Lower pH to 2.0 and mix for 10 minutes
Raise pH slowly with addtion of $Mg(OH)_2$ to above 5.5
Filter
submit filtrate solution for assays as above
Displacement wash filter cake
Submit wash for Mg
Submit solids for whole rock

Stage 3-

Reheat filtrate to ~80°C
Raise emf to high as possible with peroxide and air
Add 5 g of $Mg(OH)_2$ mixed with DI water
mix 5 minutes and filter
submit filtrate solutionfor assays as above
Displacement wash filter cake
Submit wash for Mg
Submit solids for whole rock

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Time	Temp °C	pH	emf mV	Additions (g, mL)					Comments
				Air (mL/min)	Mg(OH)$_2$	H2O2 35%w/w	FeCl$_2$•H$_2$0 conc	HCl 12%	
Stage 1									
12:15	80	-1.05	315			1			
12:25	80	-1.07	765			1			
12:35	80	-1.05	810			1.5			
12:45	81	-1.04	772						
12:50	81	-1.03	7385		7	4			
12:57	80	3.18	581						
13:00	81	3.67	522						Filter
Stage 2									
13:31	80	2.2	603					2drops	
13:35	80	2.19	327				4		
13:36		5.00	-24						
13:42	80	1.95	319	add HCl to maintain pH at ~2.0 for 10 minutes					
13:52	80	2.06	324						
14:12	80	5.3	-391		5				
14:15	80	5.38	-428		1				mix 2 min and filter
14:17	80	5.42	-466						
Stage 3									
14:39	80	3.91	12						
14:40	80	3.61	90						
14:42	80	3.42	124	268					
14:47	80	5.91	292	268	5	2.7			
14:48	80	5.93	311	268		0.3			Filter

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LCH 29

Stage 1

Preg and $Mg(OH)_2$ additon

Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form time 3.5min, dry 1min

Fitrate volume: 590 mL 25mL sample taken
Filtrate weight: 728.3 g wet weight: 121.15 g
Solution SG: 1.234 g/L dry weight: 56.2 g
Wash volume: 42 mL Percent Moisture: 53.6112 %

Stage 2

Ferrous chloride Addition

Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form time 1min, dry 1 min

Fitrate volume: 530 mL 25mL sample taken
Filtrate weight: 660.9 g
Solution SG: 1.247 g/L
Wash volume: 16 mL

Stage 3

Peroxide and Magnesium hydroxide

Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form 10 min, dry 1min

Fitrate volume: 480 mL
Filtrate weight: 594.8 g
Solution SG: 1.239 g/L

Product	Amount	Assays (mg/l, %)												
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Na	Si	B	V	Cr	Al
combined preg	500													
filtrate 1	590	87300	5340	0.88	160	<0.05	10	<0.05	6.74	2			<0.05	<2
wash 1	42	44700												
residue 1	14.1	18.8	0.14	7.89	0.16				0.07	12.9		<0.01	0.02	0.21
filtrate 2	530	86600	5190	720	162	<0.05	10.3	0.21	0.2	9			0.3	<2
wash 2	16	68700												
residue 2	3.8	28	0.61	10	0.09				0.14	0.61		<0.01	0.01	0.29
filtrate 3	480	84300	4800	0.68	20.5	<0.05	0.17	<0.05	8.56	<2			<0.05	3
wash 3	26.0	60100												
residue 3	7.3	28.4	0.72	6.26	1.26				<0.05	0.32		<0.01	0.01	0.17

5398 - GTR **INSTRUCTIONS**
Test 30
Purpose: Attempt U-process for purification of $MgCl_2$ solution with the removal of Calcium

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
combine two leaches and record weight
Take a solution sample for analysis
Mg, Ca, Si, Fe, Na, Mn, Cu, Zn, B, Ni, S, V, Cr, Al

Stage 1 - Iron oxidation and precipitation

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Raise emf of solution as high as possible (emf = 600 mV?)
with the addition of hydrogen peroxide every 10 minutes for 30 minutes
Raise pH to 3.5-4.0 with $Mg(OH)_2$, maintain for 15 minutes
while adding hydrogen peroxide
Mix 15 minutes
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg, Ca, and Fe
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, Si, Mn, Cu, Zn, Ni, Mn, B, V, Al, Cr

Stage 2 -

Added few drops of HCl to pH ~2.0 (measure emf)
Add 8 g of $FeCl_2 \cdot H_2O$
Added reagent grade $Mg(OH)_2$ to water(1gram) at a time to raise pH to ~5.5
Lower pH to 2.0 and mix for 10 minutes
Raise pH slowly with addtion of $Mg(OH)_2$ to above 5.5
Filter
submit filtrate solution for assays as above
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Stage 3-

Reheat filtrate to ~80°C
Raise emf to high as possible with peroxide and air
Add 5 g of $Mg(OH)_2$ mixed with DI water
mix 5 minutes and filter
submit filtrate solution for assays as above
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Stage 4- Calcium Removal
Reheat filtrate to ~80°C
Add Concentrate H_2SO_4(13 g)
Raise pH to ~1.0 and hold for 15 min
Raise pH to 5.5 and hold 5 minutes and filter
submit filtrate solution for assays as above
Displacement wash filter cake
Submit wash for Mg, and Ca
Submit solids for whole rock

Stage 5 SO_4 removal
Reheat filtrate to ~80°C
Add 5.33g $BaCl_2 \cdot 2H_2O$ and mix for five minutes
Filter submit filtrate solution for assays as above + Ba
Submit solids for whole rock + Ba

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Time	Temp °C	pH	emf mV	Additions (g, mL) Mg(OH)$_2$ 20%	H$_2$O$_2$ 35%w/w	FeCl$_2$•H$_2$0	H$_2$SO$_4$ conc	Comments
Stage 1								
7:43	76	-0.85	310		2			
7:53	78	-0.77	682		3			
8:03	78	-0.72	658		3			
8:13	78	-0.67	705	34	8.5			done adding at 8:20
8:27	78	3.38	553					
8:39	78	3.62	489					Thicken and filter
Stage 2								
9:55	75	3.90	439					
10:16	77	3.78	533					
10:18	77	2.10	487					2 drops 12% HCl
10:20	77	0.42	338			8		
10:30	78	0.42	339	30				
10:45	75	5.30	-500	30				
10:50	75	5.54	-555					
10:55	75	5.56	-557					Filter
Stage 3								
11:34	74	5.73	-451	25	3.00			
11:41	76	5.67	-388					
11:44	76	5.70	347	25	2.6			
11:49	76	5.82	336					Filter
Stage 4								
13:48	75	5.81	265				13	
13:53	75	-0.8	580	21				
14:05	75	1.06	608					
14:20	75	1.03	598					
14:21	75	4.88	463	1				
14:23	75	5.48	333	3				
14:32	75	5.54	328					Filter
Stage 5	**21-Dec**							
10:23	75	5.48	241					5.33g BaCl$_2$•2H$_2$O
10:34	75	5.55	254					Filter
The remaining solution went to crystalization								

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LCH 30

Preg and $Mg(OH)_2$ additon
Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form time 4min, dry 1min

Fitrate volume: 1019 mL
Filtrate weight: 1280 g wet weight: 41.91 g
Solution SG: 1.255 g/mL dry weight: 33.2 g
Wash volume: 44 mL Percent Moisture: 20.78 %
Ferrous chloride Addition
Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form time 10min, dry 1 min

Fitrate volume: 950 mL
Filtrate weight: 1203 g
Solution SG: 1.266 g/L
Wash volume: 46 mL

Peroxide and Magnesium hydroxide
Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form 1 hour

Fitrate volume: 830 mL
Filtrate weight: 1057 g
Solution SG: 1.273 g/L
Wash 65
Calcium removal
Filtered on 18.5cm buchner(80°C) with Whatman 541 paper
Filtrate Form 1 min, dry 1min

Fitrate volume: 740 mL
Filtrate weight: 934.6 g
Solution SG: 1.263 g/L

SO_4 removal
Filtered on 18.5cm buchner (80°C) with Whatman 541 paper
Filtrate Form 10 min, dry 1min

Fitrate volume: 670 mL
Filtrate weight: 852.8 g
Solution SG: 1.273 g/L

Product	Amount	Assays (mg/L, %)														
	(ml, g)	Mg	Ca	Fe	Mn	Ni	Zn	Cu	Na	Si	B	Ba	V	Cr	S	Al
combined preg	~1000	92300	6220	2060	187	<0.05	8.78	0.07	4.82	<2	<0.01	...	<0.01	0.07	...	4
filtrate 1	1019	91200	5800	0.13	159	0.05	9.54	0.05	3.97	<2	1.33	...	<0.01	0.06	...	<2
wash 1	44	45900	2780	0.71	99.5	0.2	4.06	0.12	3.97	5	1.03	...	<0.01	0.08	...	4
residue 1	33.2	18.9	0.19	6.97	0.09	...	...	...	<0.05	12.2	...	...	<0.01	0.01	...	0.13
filtrate 2	950	91700	5910	28.1	138	0.11	7.66	0.17	6.37	5	1.59	...	<0.01	0.8	...	8
wash 2	46	59800	3580	...	...	...	...	...	...	...	...	...	...	...	...	...
residue 2	21.1	26.7	0.63	11.2	0.15	...	...	...	<0.05	0.24	....	...	<0.01	0.01	...	0.09
filtrate 3	830	92500	5890	0.49	0.08	0.05	3.9	0.05	7.5	<2	2.04	...	<0.01	<0.05	...	<2
wash 3	65.0	63200	3760	...	...	...	...	...	...	...	...	...	...	...	...	...
residue 3	26.3	27	0.76	1.36	1.01	...	...	...	<0.05	0.14	...	...	<0.01	0.01	...	0.06
filtrate 4	740	98500	942	3.0	2.0	1.34	6.91	<0.05	12.3	14.0	3.42	...	<0.01	...	1520	...
residue 4	18.2	5.19	21.3	0.09	<0.01	...	...	...	<0.05	0.12	...	...	<0.01	<0.01	...	0.04
filtrate 5	670	108000	702	<0.2	1.34	0.56	3.26	<0.20	6.98	<1.0	2.83	65.5	<0.01	<0.20	984	...
residue 5	3.6	na	na	na	na	na	na	na	na	na	na	30.0	na	na	na	na

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LCH 30

Crystallisation

Solution from stage 5
Heat to boiling while mixing slowly
Evapourate off half the volume and cool
This produced a hard mass redissloved in 300mL of DI water
Mix overnight
Heat to ~100°C slowly and remove water to a volume of 500mL on beaker
Cool to ~50°C and filter
Remove filtrate from flask
Measure volume and place in beaker overnight
Place Buchner over a beaker overnight
Refilter filtrate

Initial Filtrate

volume of filtrate	245 mL
weight of filtrate	320.11 g
Solution SG	1.307 g/mL

Second Filtrate

volume of filtrate	245 mL
weight of filtrate	320.67 g
Solution SG	1.309 g/mL

weight of first crystals	239.3 g
weight of second crystals	19.4 g
total weight of crystals	258.7 g

The crystals were mixed well and ~60mL were removed for analysis
30.32g were taken for the chem lab

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LCH 30

Crystallisation

Product		Assay (mg/l, %)		Distribution (%)	
		Mg	Ca	Mg	Ca
Crystals	258.7	12.0	0.045	52.1	44.5
Supernata	245	116400	592	47.9	55.5
Head (calc	670	88899	390		
Head (dir)		108000	720		

Solution Analysis
For Test 30 - Crystallisation Step

	Purified Sol'n (mg/l)	Supernatant Sol'n (mg/l)
Vol (ml)	670	245
Mg	108000	116400
Ca	702	592
Si	<1.0	2.96
Na	6.98	...
B	2.83	...
Mn	1.34	...
Al	1.02	...
Cr	<0.2	...
V	<0.10	...
Ni	0.56	...
Cu	<0.2	...
Zn	3.26	...
Fe	<0.2	...
S	984	...
Ba	65.5	...

5398 - Golden Triangle Resources
Hydrated Magnesium Chloride from MC-36
$MgCl_2 \cdot 6H_2O$

Quantitative Analysis			
Wgt Loss		Fe	<0.0005%
@ 200 °C		Cu	<0.0005%
@ 300 °C		B	<0.0005%
@ 400 °C		C	0.01%
Mg	12.0	S	<0.01 %
Cl	34.3	P	<0.0020%
Ca	0.045%	Na	<0.0010%
Si	<0.005%	K	<0.0100%
Mn	0.0005%	Cr	<0.0005%
Al	<0.0020%	Ni	<0.0005%
		Zn	<0.0005%

Semi-quantitative Analysis			
Ag	< 0.5 ppm	As	< 100 ppm
Pd	< 5 ppm	Au	< 100 ppm
Be	< 10 ppm	Ba	< 100 ppm
Bi	< 10 ppm	In	< 100 ppm
Co	< 10 ppm	Nb	< 200 ppm
V	< 10 ppm	Ta	< 200 ppm
Ge	< 25 ppm	W	< 200 ppm
Pb	< 25 ppm	Rb	< 200 ppm
Mo	< 25 ppm	Pt	< 200 ppm
Sr	< 25 ppm	Te	< 300 ppm
Sn	< 25 ppm	Y	< 300 ppm
Ti	< 25 ppm	Ce	< 300 ppm
Zr	< 25 ppm	U	< 1000 ppm
Sb	< 50 ppm	Th	< 1000 ppm
Cd	< 50 ppm		
Ga	< 50 ppm		
Li	< 50 ppm		
Tl	< 50 ppm		

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LCH 31

This test may be found in
Progress Report No. 1 - Leaching

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LCH 32

This test may be found in
Progress Report No. 1 - Leaching

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LCH 33

This test may be found in
Progress Report No. 1 - Leaching

5398 - GTR **INSTRUCTIONS**
Test 34
Purpose: Direct Reducing Precipitation

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minute
Using double condeser on both leaches
combine two leaches and record weight
Take a solution sample for analysis
Mg and Free acid

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
2.7g Fe powder in 1g increments
Raise pH to 3.5-4.0 with MgO ~5
Raise pH and emf with NaOH and H_2O_2 respectivly
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg and Mn
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, and Mn

Stage 2 - Place stage 1 filtrate in beaker in 80 °C water bath.
Heat to temperature
Add H_2O_2 and NaOH to rasie emf and pH
Filter
submit filtrate solution for assays as above
Submit solids for whole rock

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Time	Temp °C	pH	emf mV	Additions (g, mL) Fe powder g	MgO 99.999% g	H2O2 35%w/w mL	NaOH 50g/L mL	NaOH conc g	Comments
Stage 1									
10:28	76	-0.54	385						
10:40	76	-0.07	386						
10:45	76	0.83	388						
10:48	76	0.85	391						
10:49	76	1.28	389	1					
10:50	76	1.62	387	1					
10:51	76	1.81	364						
10:52	76	1.94	366	0.7					
11:02	76	2.39	335						
11:06	76	4.41	51		0.18				Sol'n more clear than yello
11:12	76	4.79	-72		0.39				
11:15	76	4.85	-122		0.11				Turned blue ~ pH 4.5
11:18	76	4.9	-155				1.7g		
11:25	76	3.8-4.2	-50			1	28.6g		
11:33	76	4.45	-7					0.5	
11:40	76	4.86	-17					0.43	
11:42	77	4.95	-40						Filter
Stage 2									
1:47	73	4.43	92						
1:49	76	4.55	67			2.2	3		
1:51	76	4.89	430				37		
1:52	76	5.81	272			1.8			
1:53	76	5.81	361			0.4	20		
2:08	76	5.79	356						Filter

Stage 1
Filtered on 18.5cm buchner(80°C) with Whatman 4 paper
Filtrate Form time 5min, dry 1min

itrate volume: 940 mL 25mL sample taken
iltrate weight: 1205 g
Solution SG: 1.282 g/mL
Wash volume: 87 mL

Stage 2
Filtered on 18.5cm buchner(80°C) with Whatman 4 paper
Filtrate Form time 2min, dry 2min

itrate volume: 930 mL 25mL sample taken
iltrate weight: 1173 g
Solution SG: 1.262 g/L

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Product	Amount (ml, g)	Assays (mg/l, %)					Distribution(%)				
		Mg	Mn	Ca	Fe	Na	Mg	Mn	Ca	Fe	Na
filtrate 1	25	93500	268	5200	2120	9.5	29.1	43.6	50.8	99.1	0.6
wash 1	87	39500	94.9				42.8	53.7	0.0	0.0	0.0
residue 1	37.3	19.1	0.13	0.1	12.8	0.7	0.0	0.0	0.0	0.9	0.1
filtrate 2	25	90500	15.3	5040	0.84	1550	28.2	2.5	49.2	0.0	99.3
residue 2	5.6	14.8	3.24	0.57	1.08	0.35	0.0	0.1	0.0	0.0	0.0
Head (dir)		96000					100	100	100	100	100

5398 - GTR
Test 35
Purpose: Direct Reducing Precipitation

INSTRUCTIONS

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minute
Using double condeser on both leaches
combine two leaches and record weight
Take a solution sample for analysis
Mg and Free acid

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
0.5g Fe powder in 0.25g increments
Raise pH to ~1.5-2 with MC 36 calcine
Raise pH to ~4.5 with $Mg(OH)_2$ adding in 0.25g lots
Filter, set aside filtrate for stage 2.
Displacement wash cake, assay wash for Mg and Mn
Submit filter cake for whole rock
Submit a sample of the filtrate for Mg, Ca, Na, Fe, and Mn

Stage 2 - Place stage 1 filtrate in beaker in 80 °C water bath.
Heat to temperature
Add H_2O_2 and $Mg(OH)_2$ to rasie emf and pH
Tring to achieve a high pH(~5.8) and high emf(~350)
Filter
submit filtrate solution for assays as above
Submit solids for whole rock

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Time	Temp °C	pH	emf mV	Additions (g, mL) Fe powder g	$Mg(OH)_2$ g	H_2O_2 35%w/w mL	Calcine MC36 g		Comments
Stage 1									
9:47	75	-1.26	353						
10:15	75	-1.47	355						
10:25	75	-1.41	354	0.25					
10:26	75	-1.37	354	0.25					
10:29	75	-1.46	340				3.44		emf erratic from 288-340
10:40	75	-1.34	361						
10:44	75	0.26	389						
10:46	75	1.15	388						
10:48	75	1.54	384						
10:50	75	1.75	388		0.25				solution a caramel colour
10:52	75	2.18	322		0.25				
10:55	75	4.23	-40		0.25				solution more brown
10:57	75	4.52	-119						Filter
Stage 2									
11:26	75	2.31	311		0.25				
11:28	75	4.56	-112						
11:32	75	4.7	-212		1.25	0.6			
11:38	75	4.63	-148		0.75	0.2			
11:40	75	5.09	25		1	0.6			
11:45	75	5.58	2.75		1	5.8			
12:00	75	5.74	359						Filter

Stage 1
Filtered on 18.5cm buchner(80°C) with Whatman 4 paper
Filtrate Form time 8min, dry 1min

itrate volume: 960 mL 25mL sample taken
iltrate weight: 1230 g
Solution SG: 1.282 g/mL
ash volume: 39 mL

Stage 2
Filtered on 18.5cm buchner(80°C) with Whatman 4 paper
Filtrate Form time 20min, dry 3min

itrate volume: 935 mL 25mL sample taken
iltrate weight: 1185 g
Solution SG: 1.267 g/L
ash volume: 100 mL

Product	Amount (ml, g)	Assays (mg/l, %)						Distribution (%)				
		Mg	Mn	Ca	Fe	Na	FAT	Mg	Mn	Ca	Fe	Na
combined pre	25	96100					12	13.1	0	0	0	0
filtrate 1	25	97400	279	5880	2340	7.14		13.2	2.6	39.4	2.4	0.4
wash 1	39	50300	126					10.7	1.9	0	0	0
residue 1	21.1	20.9	0.08	0.27	2.08	0.18		24.0	6.4	15.3	18.1	87.8
filtrate 2	25	91300	3.27	5400	0.54	10		12.4	0.0	36.1	0.0	0.6
wash 2	100	40500	0.28					22.0	0.0	0	0	0
residue 2	6.9	12.3	3.41	0.5	27.9	0.07		4.6	89.1	9.2	79.5	11.2

5398 - GTR **INSTRUCTIONS**
Test 36
Purpose: Simplifification of purification flowsheet

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
Using double condeser on both leaches
combine two leaches and record weight

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Neutralize to pH 1.5 to 2.0 with calcine
Neutralize to pH 4.5 with slaked $Mg(OH)_2$
Raise emf by slowly bubbling Cl_2 gas into solution
Add slaked Mg(OH)2 to system to keep pH in range of 3.5 to 4.5
while adjusting emf with Cl_2
After a high emf is reached, add 14.6g of reagent grade H_2SO_4
and slaked Mg(OH)2 at the same time to keep pH in range 3.5 to 4.
When reading are stable (high pH ~ 4.5 and high emf ~ 350)
and slaked Mg(OH)2 to pH 5.5 to 5.8
Add 2.5g $BaCl_2 \cdot 2H_2O$, mix for 5 minutes
Add bit of Cl_2 to raise emf a bit
Filter
Submit filtrate for Mg, Ca, Si, Na, B, Mn, Al, Cr, V, Ni, Cu, Zn, Fe, S, and Ba
Submit wash for Mg and precipatate for whole rock, S, Ba, and Cl

Stage 2

Heat filtrate to 80°C
Add Cl_2 gas and slaked $Mg(OH)_2$
to rasie emf and pH
Filter
Submit solution assay for Mn

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Time	Temp °C	pH	emf mV	Additions (g, mL)				Comments
				Cl2 gas	Mg(OH)2 20%w/w (g)	Calcine MC36 (g)	$BaCl_2$ (g)	
Stage 1								
10:16	73	0.8	378					
10:20	75	1.41	382					
10:23	76	1.56	377		3.11			
10:27	76	4.37	-104		1.94			
10:32	77	4.48	-192	25				flow 13 on #4 flow meter
10:37	77	3.5	360	25				
10:41	77	3.23	398	25				
10:44	77	4.9	323					gas off and start adding 14.6g acid
10:55	78	4.23	147					
	78	4.12	154					
	78	3.86	172					
	78	4.3	168					
11:40	78	4.6	299					
	78	4.7	284					acid all in
	78	3.8	302					
	78	3.11	349					
	78	3.2	360					
	78	3.6	356					
	78	3.8	347					
	78	4	334					
	78	4.2	302					
11:44	76	4.39	268		16.5			
11:45	76	5.44	208		6.43			
11:49	76	5.54	200				2.5	
11:50	76	5.23	448	60	9.89			gas on 1 min
11:53	76	5.5	333					
11:58	76	5.6	297					
Stage 2								
1:51	73	5.71	262					
	73	5.62	914	68	15.33			Cl on ~ 30sec
2:00	73	5.65	909					pH not moving
2:05	74	5.71	896		1.81			

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LCH 36

Stage 1
Filtered on 18.5cm buchner(80°C) with Whatman 4 paper
Filtrate Form time 6min, dry 1min

Fitrate volume: 1010 mL 50mL sample taken
Filtrate weight: 1279 g wet weight: 82.46 g
Solution SG: 1.266 g/mL dry weight: 58 g
Wash volume: 94 mL Percent Moisture: 29.7 %

Stage 2
Filtered on 18.5cm buchner(80°C) with Whatman 42 paper
Filtrate Form time 6min, dry 3min

Fitrate volume: 915 mL 25mL sample taken
Filtrate weight: 1160 g
Solution SG: 1.268 g/L

Product	Amount (mL, g)	Assays (mg/L, %)														
		Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
Filtrate 1	1010	98500	61.9	1250	<0.10	6.75	0.84	2.37	1.21	<0.10	<0.10	<0.10	0.86	6.96	926	1.2
Wash 1	94	49300	31*	626*	0*	3*	0*	...	1*	0*	0*	...	...	...	463*	1*
Residue	58	15.8	0.38	6.99	4.17	0.06	6.68	...	0.14	<0.01	<0.01	...	...	...	5.17	2.13
Head (calc)	400	28.3	0.07	1.34	0.60	0.01	0.97	...	0.02	0.00	0.00	...	...	...	0.99	0.31
Head (dir)		26.9	0.08	1.48	0.65	<0.05	1.11	27	0.02	0.01	<0.01	<0.00	0.002	0.01	...	...

* not assayed - estimated from Mg concentration in Preg and Wash solutions

Product	Distribution (%)														
	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
Filtrate 1	87.8	21.9	23.5	0.0	16.3	0.0	...	1.5	1.7	1.7	...	...	...	23.5	0.1
Wash 1	4.1	1.0	1.1	0.0	0.8	0.0	...	0.1	0.1	0.1	...	...	...	1.1	0.0
Residue	8.1	77.1	75.4	100.0	83.0	100.0	...	98.4	98.2	98.2	...	...	...	75.4	99.9
Total	100	100	100	100	100	100	...	100	100	100	...	...	...	100	100

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Re-Calculated Magnesium Balance

Product	Amount (mL, g)	Assays (mg/L, %)	Distr. based on re-calc head (%)
		Mg	Mg
Filtrate 1	1010	98500	90.3
Wash 1	94	49300	4.2
Residue	58	15.8	...
Mg added as neutralising agent	3.16	99.9	
Re Calculated	400	27.5	
Head (dir)		26.9	

Reagent Additions:			
Leach:	Ore	400.0	g
	H_2O	146.0	g
	HCl (36%)	1007.6	g
Purification:	Calcine	0.0	g
	$Mg(OH)_2$	11.0	g
	Cl_2 Gas	0.225	l
	H_2SO_4 (96%)	14.6	g
	$BaCl_2 \cdot 2H_2O$	2.5	g
Approximate Consumptions (per tonne of Leach feed)			
	HCl (100% basis)	907	kg/t
	Calcine	0	kg/t
	$Mg(OH)_2$	27.5	kg/t
	Cl_2 Gas	0.56	litres/kg
	H_2SO_4 (100%)	35.0	kg/t
	$BaCl_2 \cdot 2H_2O$	6.25	kg/t

5398 - GTR **INSTRUCTIONS**
Test 37
Purpose: Simplifification of purification flowsheet

Procedure:
Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
Using double condeser on both leaches
combine two leaches and record weight

Stage 1 -
Place pulp in beaker in 80 °C water bath.
Heat to temperature
Neutralize to pH 1.5 to 2.0 with calcine
Neutralize to pH 4.5 with slaked $Mg(OH)_2$
Raise emf by slowly bubbling O_2 gas into solution
Add slaked Mg(OH)2 to system to keep pH in range of 3.5 to 4.5
while adjusting emf with O_2
After a high emf is reached, add 14.6g of reagent grade H_2SO_4
and slaked Mg(OH)2 at the same time to keep pH in range 3.5 to 4.
When reading are stable (high pH ~ 4.5 and high emf ~ 350)
and slaked Mg(OH)2 to pH 5.5 to 5.8
Filter
Submit filtrate for Mg, Ca, Si, Na, B, Mn, Al, Cr, V, Ni, Cu, Zn, Fe, S, and Ba
Submit wash and Mg and precipatate for whole rock, S, Ba, and Cl

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Time	Temp °C	pH	emf mV	Additions (g, mL)			Comments
				02 gas mL/min	Mg(OH)2 (g)	Calcine MC36 (g)	
Stage 1							
9:05	73	-1.13	386				
9:37	75	-1.1	384			5.4	
9:42	78	1.78	392		1.99		
9:48	78	4.41	-158		1.56		
9:56	77	4.61	-232				
10:00	77	4.33	-114				
10:06	77	3.98	75	135			
10:11	77	4.17	59	135	2.18		
10:14	77	3.53	121	135	1.79		
10:15	77	4.34	37	135			
10:20	77	3.21	147	135	1.66		
10:22	77	4.34	40	135			
10:26	78	3.49	120	135	1.6		
10:27	78	4.40	25	135			
10:32	78	3.45	125	135	1.3		
10:33	79	4.47	18	135			
10:40	79	4.25	99	135			
10:44	79	4.60	200	135			
10:50	79	4.87	269	135			
11:05	79	4.93	287	135			Start adding acid
11:35	81	-0.06	627		37.6		half of acid in
	81	1.70	541				add the rest all at once
	81	3.12	290				
	81	3.60	256				
	81	3.80	224				
	81	4.00	163				
	81	4.10	116				
	81	4.20	103				
	81	4.30	82				
	81	4.40	76				
	81	4.50	62				
	81	4.60	45				
	81	4.70	39				
	81	4.80	38				
	81	4.90	25				
	81	5.00	20				
	81	5.10	24				

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Time	Temp °C	pH	emf mV	Additions (g, mL) 02 gas mL/min	Mg(OH)2 (g)	Calcine MC36 (g)	Comments
	81	5.20	43				
	81	5.30	100				
	81	5.40	153				
	81	5.50	183		27.4		
11:45	79	5.54	199	135			
11:50	79	5.54	235	135			
11:58	79	5.59	239	135			
12:15	79	5.61	246	135			
12:30	79	5.61	250	135			
12:50	79	5.61	250	135			filter

Stage 1
Filtered on 18.5cm buchner(80°C) with Whatman 42 paper
Filtrat Form time 9min, dry 1min

itrate volume:	1005 mL	25mL sample taken	
Filtrate weight:	1283 g	wet weight:	76.44 g
Solution SG:	1.276 g/mL	dry weight:	g
Wash volume:	145 mL	Percent Moisture:	100.0 %

Product	Amount (ml, g)	Assays (mg/l, %)														
		Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	1005	106000	144	1190	<0.10	6.62	<0.50	1.78	1.23	<0.10	<0.10	<0.10	1.02	6.06	943	
wash 1	145	31900														
residue	55	15.7	0.17	8.08	4.76	<0.05	7.76		0.14	<0.01	<0.01					

Product	Distribution (%)														
	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	88.9	60.8	21.2	0.0	19.5	0.0	100.0	1.6	1.8	1.8	100.0	100.0	100.0	100.0	
wash 1	3.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	
residue	7.2	39.2	78.8	100.0	80.5	100.0	0.0	98.4	98.2	98.2	0.0	0.0	0.0	0.0	
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	

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Test 38

INSTRUCTIONS

Purpose: Simplifification of purification flowsheet
To study the filtering and settling characteristics

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
Using double condeser on both leaches
combine two leaches and record weight

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Neutralize to pH 1.5 to 2.0 with calcine
Neutralize to pH 4.5 with slaked $Mg(OH)_2$
Raise emf by slowly bubbling Cl_2 gas into solution
Add slaked Mg(OH)2 to system to keep pH in range of 3.5 to 4.5
while adjusting emf with Cl_2
After a high emf is reached, add 14.6g of reagent grade H_2SO_4
and slaked Mg(OH)2 at the same time to keep pH in range 3.5 to 4.5
When reading are stable (high pH ~ 4.5 and high emf ~ 350)
and slaked Mg(OH)2 to pH 5.5 to 5.8
Add 5.0g $BaCl_2 \cdot 2H_2O$ mixed in water, mix for 5 minutes
Add bit of Cl_2 to raise emf a bit
Filter
Submit filtrate for Mg, Ca, Si, Na, B, Mn, Al, Cr, V, Ni, Cu, Zn, Fe, S, and Ba
Submit wash for Mg and precipatate for whole rock and Cl

Project: **5398**
Test: **LCH 38**

Technologist: G. Toole
Date: Dec. 3, 1998

Purpose: To determine the solid-liquid separation characteristics of the neutralized leach liquor, and measure the basic impurity levels of the levels of the final and intermediate solutions.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight 2 x 20 g
HCl added: 2 x 50 g
Water added: 2 x 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.7 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for added the sample.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid frothing over.
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test both samples were combined and a thief sample was taken

Data:

Leach A				**Leach B**			
Time min	Temp °C	emf mV Ag/AgCl	Comments	Time min	Temp °C	emf mV Ag/AgCl	Comments
0	80	230		0	80	236	
30	80	305		30	80	335	
60	80	357		60	80	398	

Time	Temp °C	pH	emf (mV) Ag/AgCl	Additions (g, mL/min)					Comments
				Calcine	Mg(OH)2 (20%)	Cl_2	H_2SO_4 (conc)	$BaCl_2 \cdot 2H_2O$	
10:20	72	-1.29	396						
10:32	77	-1.09	390	3.91					
10:40	78	1.93	376		2.61				
10:46	77	4.61	-177			47.6			
10:51	77	4.58	-180			72.4			
11:09	77	4.47	-92						
11:18	77	5.26	355		17.31				
11:40	77	1.96	727		37.6		14.6		gas off
11:45	78	5.55	280					5	
11:50	77	5.65	243			72.4			gas off 11:56
11:58	77	5.76	855		20.45				

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Filtration:

Filter cloth:

Media No	1820	Th. Count	68 X 60
Weave	3-1 twill	Wt	4.4 oz/sq.ft
Yarn	Polyester multifill	Air flow	22 cfm/sq.ft
		Finish	heat set

Dry Cake Weight:	52.4	Filter Diameter	11 cm
Wet Cake Weight:	81.21	Filter Area	0.00950332 m^2
Moisture Content:	35.5		

Stage	Time (sec)		Vol (ml)	Cake Rate ($kg/hr/m^2$) form only		Filtrate Rate ($l/hr/m^2$) form only
	Form	Dry		wet	dry	
Filtration	202	0	236.0	152.3	152.3	443
Wash 1	44	0	26.0			224
Wash 2	30	0	30.0			379
Wash 3	19	0	28.5			568
Wash 4	15	0	26.5			669
Wash 5	15	0	25.0			631

Wash (ave) 494

Cake thickness:	6 mm
Wet weight:	81.21 g
Dry weight:	57.16 g
Percent moisture:	29.6 % (air dried)

Product	Amount	Assays (mg/l, %)														
	(ml, g)	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	1080	94100	<0.05	1510	0.18	6.75	0.59	2.07	<0.50	<0.10	<0.10	<0.10	0.1	2.49	818	0.37
wash 1	26.0	52500	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 2	30.0	35000	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 3	28.5	22800	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 4	26.5	16700	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 5	25.0	16400	na	na	na	na	na	na	na	na	na	na	na	na	na	na
residue	52.4	13.1	0.56	6.88	4.75	0.08	7.6		0.13	<0.01	<0.01					

Product	Distribution (%)														
	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	90.4	0.02	31.1	0.01	14.8	0.02	100.0	0.8	2.0	2.0	100	100	100	100	100
wash 1	1.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
wash 2	0.9	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 3	0.6	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 4	0.4	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 5	0.4	na	na	na	na	na	na	na	na	na	na	na	na	na	na
residue	6.1	99.98	68.9	99.99	85.2	99.98	0.0	99.2	98.0	98.0	0.0	0.0	0.0	0.0	0.0
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	

Additional Assays

Product	Assays %	
	Cl	LOI
residue	1.82	23.4

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LCH 38

5398 - GTR **INSTRUCTIONS**
Test 39
Purpose: Simplification of purification flowsheet

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minute
Using double condeser on both leaches
combine two leaches and record weight

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Neutralize to pH 1.5 to 2.0 with calcine
Neutralize to pH 4.5 with slaked $Mg(OH)_2$
Raise emf by slowly bubbling Cl_2 gas into solution
Add slaked Mg(OH)2 to system to keep pH in range of 3.5 to 4.5
while adjusting emf with Cl_2
When emf is high(~700mV) add slaked Mg(OH)2 to pH 5.5 -5.8
while keeping emf high
Filter
Submit filtrate for Mg, Ca, Si, Na, B, Mn, Al, Cr, V, Ni, Cu, Zn, and
Submit wash for Mg and precipatate for whole rock and Cl

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LCH 39

Time	Temp °C	pH	emf mV	Additions (g, mL)		Comments
				Cl2 gas mL/min	Mg(OH)2 20%w/w g	
8:15	73	1.59	424		3.16	
8:20	75	4.64	-194			
8:21	75	2.03	371	72.4		
8:26	77	4.34	-88	72.4		
8:37	77	5.08	488	72.4		gas off for 30 sec
8:37	77	5.12	-79	72.4	34.73	
8:43	77	5.57	760			
8:45	77	5.64	686			gas on approximatly 60 sec
8:46	77	5.66	830		11.85	

Stage 1
Filtered on 18.5cm buchner(80°C) with Whatman 42 paper
Filtrate Form time 18min, dry 1min

Fitrate volume:	980 mL	25mL sample taken	
Filtrate weight:	1237.1 g	wet weight:	92.64 g
Solution SG:	1.262 g/mL	dry weight:	61.5 g
Wash volume:	160 mL	Percent Moisture:	33.6 %

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Product	Amount	Assays (mg/L, %)														
	(mL, g)	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
Filtrate 1	980	94900	<0.05	5440	<0.10	3.09	0.77	0.97	<0.10	<0.10	<0.10	<0.10	<0.10	2.4		
Wash 1	160	32800	0*	1880*	0*	1*	0*	...	0*	0*	0*	...	...	...	0*	0*
Residue	61.5	25	0.48	0.19	4.14	0.01	6.55	...	0.12	<0.01	<0.01	...	...	...		
Head (calc)	400	28.4	0.07	1.44	0.64	0.00	1.01	...	0.02	0.00	0.00	...	...	...	0.00	0.00
Head (dir)		26.9	0.08	1.48	0.65	<0.05	1.11	27	0.02	0.01	<0.01	<0.00	0.002	0.008	...	...

* not assayed - estimated from Mg concentration in Preg and Wash solutions

Product	Distribution (%)														
	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
Filtrate 1	81.8	0.0	92.7	0.0	32.4	0.0	...	0.1	1.6	1.6	...	...	...	...	...
Wash 1	4.6	0.0	5.2	0.0	1.8	0.0	...	0.0	0.1	0.1	...	...	...	...	...
Residue	13.5	100.0	2.0	100.0	65.8	100.0	...	99.9	98.3	98.3	...	...	...	...	...
Total	100.0	100.0	100.0	100.0	100.0	100.0	...	100.0	100.0	100.0	...	...	...	...	...

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LCH 39

Reagent Additions:		
Leach:	Ore	400.0 g
	H_2O	146.0 g
	HCl (36%)	1007.6 g
Purification:	Calcine	0.0 g
	$Mg(OH)_2$	9.9 g
	Cl_2 Gas	1.195 l
	H_2SO_4 (96%)	0 g
	$BaCl_2 \cdot 2H_2O$	0 g
Approximate Consumptions (per tonne of Leach feed)		
	HCl (100% basis)	907 kg/t
	Calcine	0 kg/t
	$Mg(OH)_2$	24.8 kg/t
	Cl_2 Gas	2.99 litres/kg
	H_2SO_4 (100%)	0.0 kg/t
	$BaCl_2 \cdot 2H_2O$	0 kg/t

Appendix A3

Progress Report No. 3
LR-5398

827218

An Investigation of
the
Evaporation of Purified Brine And $MgCl_2$ Crystal Dehydration
from
HCl Leaching of Golden Triangle Resources MC36 Magnesite Ore

Progress Report # 3

Project No.: LR 5398

NOTE: This report refers to the samples as received.
The practice of this Company in issuing reports of this nature is to require the recipient not to publish the report or any part thereof without the written consent of Lakefield Research.

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April 6, 1999

ABSTRACT

The primary objective of this stage of the laboratory development of a magnesite to magnesium process for Golden Triangle Resources NL (GTR) has been to experimentally develop the operations of purified brine solution evaporation and magnesium chloride crystal dehydration, referred to here simply as "dehydration". The work covered in this draft report includes a review by LRL of eight existing dehydration technologies (including contacts and consultations with industry and LRL consultants), the selection and proposal to GTR of two dehydration processes for scoping experiments; (1) Alcan's methanol-ammonium chloride-ammonia process, or Alcan Dehydration Technology (ADT), and (2) A generic, HCl-atmosphere, thermal dehydration technology, inspired by Norsk-Hydro's Bécancour, Quebec, practice); and the experimentation and production of preliminary metallurgical performance data on each of these methods.

This Progress Report No 3 follows upon several faxed and e-mailed technical notes and a draft report on test results of $MgCl_2$ dehydration, which were issued at various times to Golden Triangle Resources during 1998 and the first quarter of 1999. Unexpected difficulties in obtaining industry information, in the designing of scoping experiments, and in obtaining the desired anhydrous products, required more far-reaching effort than formerly predicted. Experimental work was undertaken during February and March 1999.

This report is appended to the Final report (Progress Report No 4) of the project, which includes Progress Reports No 1 (magnesite ore leaching) and No 2 (leach slurry neutralisation / oxidation / purification and solid-liquid separation). Together they complete the description of the laboratory development of a new process and a preliminary flowsheet for the GTR magnesite ore, undertaken in December 1998 and during the first quarter of 1999.

The dehydration stage testwork has ventured beyond the more limited experimentation of a single dehydration process to yield $MgCl_2{\bullet}2H_2O$ or $MgCl_2{\bullet}1.2H_2O$, as set out in the original proposal. Anhydrous $MgCl_2$ and $MgCl_2{\bullet}6NH_3$ have been experimentally produced, using the HCl gas-thermal dehydration and Alcan methanol-ammonium chloride-ammonia (ADT) processes, respectively.

The simulated chemistry of the Norsk-Hydro thermal dehydration (i.e., HCl atmosphere and reaction chemistry) appears to have provided a high percentage of the desired anhydrous magnesium chloride product, with an estimated removal of up to $1/3^{rd}$ of the feed impurity sulphur. More accurate methods of sulphur analysis of the specific chloride salts must be developed before reliable sulphur balance figures can be produced. The high MgO content of the anhydrous magnesium chloride product, 0.98 %, is expected to be reduced significantly if recognised further improvements are made to the preliminary procedure used for thermal dehydration experiments.

The precipitate from Alcan ADT experiments (Test "Alcan 2") possessed a Mg concentration (12.8 %) similar to that expected in the desired $MgCl_2{\bullet}6NH_3$ complex. Although there are no known X-ray diffraction patterns for this complex, there was no evidence of any oxide, oxychlorate or hydroxychloride compounds, within the limits of detection using X-ray diffraction. It is interesting to note that no X-ray diffraction evidence of the ammoniate product accompanied the original Alcan work, which turned to SEM analysis to detect its presence. The X-ray diffraction patterns of the precipitate in that Alcan work were similar to ours and inferred the presence of the hexa-ammoniate. The metallurgical balance shows that 97 percent of the magnesium reported to the precipitate. The residual methanol solution contained 35 percent of the calcium.

This comparative testwork of dehydration processes permits the drawing of the conclusion at this point that the methanol-ammonium chloride-ammonia process (Alcan ADT) should provide a better chance of industrial success for GTR than thermal dehydration.

For the reasons elaborated below, it is recommended that priority in further experimental development be first given to Alcan ADT technology, to determine if it is indeed suitable for the transformation of purified brine from magnesite leaching to high purity feedstock for the MPC electrolysis cell, or for similar high efficiency cells.

This conclusion and recommendation follow from:

1. The satisfactory preliminary scoping test result of ADT during Phase 1, coupled with Alcan assurances regarding MgO content of an ammoniated product (Alcan achieved MgO levels of 0.10 and 0.05 % in its prior laboratory transformation of pure MgCl2 brine or hexahydrate);
2. The accessibility of Alcan dehydration technology to emerging Mg producers;
3. Probability of a comparatively inexpensive non-exclusive licensing from Alcan; and
4. The synergy of an Alcan-LRL collaboration with GTR, notably:
    - The proximity of the experimental research teams;
    - Alcan's desire to undertake testwork off-site;
    - Alcan's familiarity with LRL capabilities (present and past collaborations);
    - LRL's familiarity with the ADT process.

At this stage of development, the argument for the thermal dehydration route appears less convincing. The scale-up of thermal dehydration technology from laboratory scale to large pilot scale, even in the case of the relatively simple HCl process successfully scoped in this work, cannot be easily realised (see the discussion below). Accordingly, thermal dehydration should probably be considered as a second alternative to ADT, to be further evaluated if the ADT technology provides unexpected problems that are difficult to surmount.

Specifically, thermal dehydration success would require overcoming the following obstacles:

1. The acknowledged difficulty of obtaining thermal dehydration know-how and licensing (Norsk-Hydro has declared to us that it is very adamantly protective of its technology and know-how; collaborating engineering companies have been obliged to sign "life-of-plant" absolute secrecy agreements with Hydro; key engineering is believed to have been undertaken directly by Hydro);
2. Several carbonaceous reductant thermal technologies carry specific environmental burdens, such as chlorinated hydrocarbon production/emissions, or unknown commercial performance.
3. The piloting of a pyrometallurgical operation (Ex.: difficulties of design and scale-up of gas-solid and gas-solid-liquid reactors) can be onerous and require several scale-up

ABSTRACT

intervals from pilot test to pilot test (1:10 scale-up interval is typical for many such pyrometallurgical processes);

4. Unless GTR can buy the technology, know-how, equipment and performance guaranties "off-the-shelf", the retention of the thermal dehydration route could represent a potentially important financial burden for GTR and add significantly to the delays from piloting to production.

Specific recommendations for further experimentation of the proposed Alcan ADT process are set out in the Final Report (Progress Report No 4; April 6, 1999)

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INTRODUCTION

At the request of Mr. Chris Laughton, General Manager of Golden Triangle Resources N.L., Melbourne, Australia, Lakefield Research Limited undertook a laboratory test program to develop a magnesite ore to electrolytic magnesium process. The first and second phases of this testwork have been described in Progress Report No 1 (magnesite leaching operation) and Progress Report No 2 (neutralisation and purification of the leach slurry).

The present draft report covers pre-evaluation of various technologies and experimentation of purified brine evaporation and magnesium chloride dehydration operations (referred to hereafter simply as "dehydration") of a proposed new process. Evaluation of dehydration process options and preliminary experimentation of two proposed methods, more far-reaching than previously predicted, were undertaken during January, February and March of 1999. Results have been previously forwarded electronically and by fax to Mr. Laughton and to Golden Triangle Resource's metallurgical consultant, Dr. Andrew Firek, in late 1998 and in the first quarter of 1999.

The aim of the dehydration testwork was originally to evaluate various process options for the removal of water and water of hydration of the targeted electrolysis cell feedstock, $MgCl_2$, and to undertake experimentation to lower the water of hydration of the crystal to $2H_2O$ or lower, thereby to further develop the overall process flowsheet through to the Mg electrolysis stage. As the testwork progressed, the product target was revised to the more desirable anhydrous $MgCl_2$.

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Testwork by G. Toole

SUMMARY

1. Summary of Results

This section presents the results of the following stages of the dehydration investigation:

- Pre-evaluation of existing industrial or pilot-tested dehydration technologies;
- Consultations with industry and a thermochemistry consultant;
- Selection and recommendation of two dehydration technologies for scoping experiments;
- Experimentation and comparison of Alcan ADT and thermal dehydration (HCl gas) methods.

The Discussion and Conclusions of testwork on dehydration process development are presented in Sections 2 and 3, respectively.

1.1 Pre-evaluation of Commercial and Piloted Dehydration Technologies

A review was made of available industrial or of previously pilot tested dehydration methods. These are listed in random order below, together with accompanying comments, most of which were previously communicated to GTR in January 1999:

Norsk-Hydro:	Anhydrous $MgCl_2$ via solution evaporation + fluid-bed dehydration of prills, in air and then in HCl gas (up to about 400 °C); Patents appear to have expired in 1973; technology is not readily available; no willingness of Norsk-Hydro to licence; Implementation would be difficult (pyrometallurgical operation), however basic chemistry is quite well understood (Ref.: Kh. Strelets, Ullmann's encyclopaedia, Kirk-Othmer encyclopaedia); Probably uses carbon to de-oxidise product (potential environmental issue).
Magnola:	Anhydrous $MgCl_2$ obtained via solution evaporation to xH_2O (about 2% H_2O, 2% MgO) in fluid bed dehydrators (sim. To Norsk) followed by super-chlorinator production of anhydrous $MgCl_2$ and low MgO (approximately 0.2 %,) with HCl gas, or alternatively, with CO + Cl_2 gases, at 450 to 850 °C (depending upon whether pure $MgCl_2$, or 10-20% $MgCl_2$ electrolyte is used as chlorinator bath); This process and its advantages and disadvantages was the subject of the previous LRL project report (LR 5318) to Golden Triangle Resources, August 1998. Potential environmental issue with carbo-chlorination alternative.
Esso Research:	Concentrated brine reacted with ammonium chloride in 3-stage fluid-bed reactors. Anhydrous magnesium chloride produced by decomposition of ammonium salt at 350-550 °C; Seemingly an elegant process, but possibly difficult to implement (fluid-bed reactor design).

SUMMARY

Nalco:	Anhydrous $MgCl_2$. As further developed by CSIRO and earmarked for another Australian venture. Absorption of brine by ethylene glycol, vacuum distillation to remove water + water of crystallisation, reaction of remaining solution with gaseous ammonia, cooling to 15-30 °C to precipitate $MgCl_2 \cdot 6NH_3$, washing of precipitate with methanol, two stage removal of methanol and water at 74 °C and 400 °C.
Dow:	$MgCl_2 \cdot 2H_20$ produced by drier or by spouting bed (brine sprayed onto solidified particles in upward moving air column). Technology available (Dow closed operations in November 98, following plant destruction by flooding, and other causes) but inappropriate for Alcan MPC cell or N-H cell types.
MagCorp:	Anhydrous $MgCl_2$, produced by spray drying of purified brine to 4% MgO, 4% H_2O, followed by carbo-chlorination at high temperature in a brick-lined vessel, to low water and MgO contents. Issue of chlorinated hydrocarbons.
Russian:	Anhydrous $MgCl_2$. Consequent to carnalite dissolution and combined K-Mg brine treatment (Hydrolysis problem with carnalite brine is somewhat less problematic than $MgCl_2$, but not entirely so); not appropriate for GTR because of KCl inconvenience.
Alcan:	Anhydrous $MgCl_2$, using a method reputedly simpler than the Nalco-CSIRO method. Absorption of brine by methanol-ammonia solution; Addition of ammonia gas and formation of insoluble $MgCl_2 \cdot 6NH_3$ product (precipitation from the methanol-ammonium chloride-water solution at low ambient temperatures); distilling of the methanol from water (rather than up-front removal of water from ethylene glycol, as in Nalco process); protected by 1995 and 1996 patents, but Alcan told me that they would make the process available to GTR or anyone else (Alcan is apparently not interested in exclusivity arrangement; they recognise that technology will be developed in piloting work and in use by licensee) and they would help in the piloting. Previously applied only to pure hexahydrate or lower hydrate salt, or to pure $MgCl_2$ brine. Probably applicable to impure brines, with possibility of some impurity rejection to the residual solution following insoluble hexa-ammoniate production. A recent meeting with Alcan (March 30, 1999) has indicated Alcan's willingness to work with LRL on process development for GTR.

The selection of processes for scoping testwork considered the following:

- Dow Chemical and Russian carnalite practice were both considered as inappropriate to the task of feedstock production for modern electrolytic cells (MPC, others), the first in view of its inability to produce anhydrous feedstock of low MgO (less than 0.1 %), the second because of the complication and undesirability of KCl production. The Dow electrolysis uses consumable electrodes with lower cell electrical efficiency, higher C consumption (higher water and MgO contents in the feedstock) and more frequent sludge removal (However, it is possible that lower cost elements of the operation may have rendered these compromises more palatable. It is understood, however, that the plant was quite basic, having evolved from WW2 needs, and that electrode positioning was not even automated).

- Magnola: Molten bath complexity and possible limitations on HCl utilisation efficiency of the Magnola super-chlorination route have been discussed in the report of project 5318; The technology is not marketed and is presently unproven; This is true also for the alternative CO + Cl_2 operation of the super-chlorinator, to which is added the environmental issue.
- Esso Research: more information is required on this interesting technology before testwork could be considered; this was not available to us (our patent and literature searches have not succeeded yet in finding the information, although this process was not a priority in more extended searches); The technology should perhaps be re-visited at some point;
- Nalco process patent is presently being developed (as is, or modified?) by CSIRO-QMC project in Australia; Probably exclusive to the QMC project; Alcan process might be preferable, in view of need for only one solvent, rather than two as in Nalco scheme;
- Magcorp process is interesting; However its availability is questionable (a competitor); Carbo-chlorination may lead to toxic organic products and costly environmental measures; Norsk-Hydro (and Magnola?) apparently also adds a little carbon to its HCl dehydration and may have similar problems.

Conclusion:

This summary evaluation left as likely dehydration technologies for the GTR magnesite process the Alcan ADT process and a simplified HCl atmosphere thermal dehydration route. These technologies were thereby recommended in early February (Feb. 1 and Feb. 7, 1999) to GTR as being the two best routes to investigate experimentally, within the scope of this preliminary laboratory development of a process flowsheet.

1.2 Consultations with Industry and a Thermochemistry Consultant

- Various contacts were undertaken with Mg industry personnel (including Mr. Brown of the Monthly Mg Newsletter and Byran Clow of the International Magnesium Association) and our thermochemistry consultant (Dr. M. Nagamori). Dow Chemical technology and MagCorp technology were recognised as being available to licence, particularly the former.
- Norsk-Hydro: Discussion between A. Plumpton and Norsk-Hydro Bécancour's Bertrand Dubreuil, present Manager of technology (recently replacing Mr. André Faucher in that task), indicated that Norsk-Hydro has no intention to liscence its process to a competitor. The construction of the Bécancour plant was undertaken by Norwegian employees and a Quebec province engineering company (the current expansion is apparently egineered by Monenco/Agra group), the latter signing a confidentiality agreement which apparently runs the length of the expected life of the plant (>75 years). It is very unlikely that Norsk-Hydro technology can be obtained (the patents are from 1970-73). However, the Norsk technology is the only one in use for the magnesite leaching route and uses a simpler form of solid-gas reaction (rather than Magnola's gas-solid-liquid reactor) and high reactivity prills moving through three fluidized beds in order to avoid formation of MgOHCl.

- An Australian metallurgical consultant was also contacted, as was a Hatch Associates Canadian office engineer involved in dehydration and Australian ventures. The latter, however, was in Australia and not available for comment at the time of this work.
- Drs. David Creber and George Holywell of Alcan International Limited were then contacted for limited technical information that permitted the initial experiments with Alcan's ADT process. Details of these meetings have been forwarded separately to Golden Triangle Resources.

The thermochemistry of the thermal dehydration method was well known and available in Chapters VII and VIII of the Strelets book. Consultations of one of the authors with Dr. M. Nagamori permitted the establishment and/or confirmation of some of the Russian data, and reception of additional data, prior to the testwork. The physical behaviour of $MgCl_2•6H_2O$ and $MgCl_2$ (both very deliquescent and requiring atmospheric protection) as well as of intermediate compounds and hydroxychlorides, completed the knowledge base for the design of the preliminary and quantitative thermal dehydration experiments.

1.3 Proposal of Two Dehydration Methods for Scoping Experiments

As indicated to GTR in separate communications in January and early February 1999, LRL proposed to undertake scoping testwork using the Alcan ADT (methanol-ammonia process) and Norsk-Hydro type chemistry thermal dehydration, the latter using a free-flowing bed of solid hydrated $MgCl_2$ crystals (initially $MgCl_2.6H_2O$, latterly, partially-dehydrated $MgCl_2•4H_2O$) in contact with a nitrogen-HCl atmosphere.

1.4 Laboratory Dehydration Scoping Test Conditions and Results

Following preliminary tests to establish equipment performance, phase transformations (especially, incongruent melting), and test conditions, three dehydration tests were then performed; one used a HCl atmosphere thermal route based on Norsk-Hydro chemistry, the other two followed the ADT process developed and patented by Alcan International, of Montreal, Canada. The tests were scoping in nature.

A single common feed material, derived from purified leach solution, was used for all three tests. It was prepared using parameters developed in earlier phases of the test program. (For development of leach conditions see Progress Report No. 1. The solution purification development is summarised in Progress Report No. 2.)

1.4.1 Feed Preparation

A sample of MC36 ore was leached with 110% of the stoichiometric quantity of HCl acid for 60 minutes, at 80 °C. The pulp was neutralised at 80 °C with calcine, to pH 2.0, and then to pH 4.5 with slaked $Mg(OH)_2$. The emf of the pulp was raised with the addition of chlorine gas. Slaked $Mg(OH)_2$ was added periodically to maintain the pH. H_2SO_4 and $BaCl_2$ were added to remove a portion of the calcium and sulphur. The full test procedure and results (Test No. 38) may be found in Appendix A. The final purified solution analysis is shown in Table No. 1.

The purified brine was heated to 100 °C and the volume reduced from approximately 1000 ml to 500 ml. The solution was then allowed to cool to room temperature, while stirring constantly. The resultant "mush" was placed in a 60 °C oven and left over-night to dry. However the material did not dry, but became partial liquid again. The material was reheated and "boiled" at 130 °C for about 30 minutes to drive off all of the residual "free" water. Again the material was cooled, while being constantly stirred. A "dry" coarse white crystalline product was obtained when the material reached room temperature.

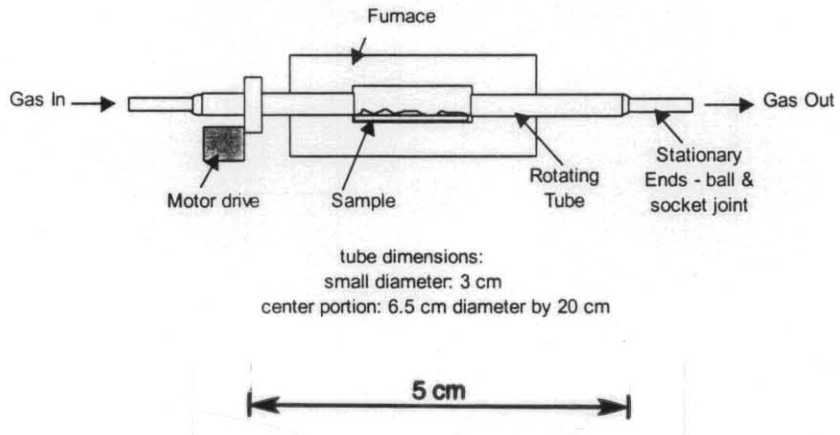
Table No. 1 – Purified Leach Solution Analysis (mg/l) (Test No. 38)

Mg	94100	Cr	<0.10
Mn	<0.05	V	<0.10
Ca	1510	Ni	<0.10
Fe	0.18	Cu	0.1
Na	6.75	Zn	2.49
Si	0.59	S	818
B	2.07	Ba	0.37
Al	<0.50		

1.4.2 Thermal Dehydration

The thermal dehydration test was performed using a Lindburg laboratory tube furnace. The sample was held in a sealed rotating glass tube as shown in Figure No 1. Nitrogen and dry HCl gas were injected into the tube. The flow rate of each gas was measured using acid resistant flowmeters. The off-gas was collected and drawn through scrubbers.

Figure No. 1 – Tube Furnace Schematic



SUMMARY

An initial attempt at dehydrating the "dry" crystals, prepared as described in Section 1.4.1, was unsuccessful. When the material was heated to about 120 °C it became molten and coated the rotating tube. The experiment was continued for a short time to determine what would occur at higher temperatures. Depending upon the temperature, the material either solidified and flaked off the walls, or remelted completely. The test was terminated.

The literature[1] makes reference to the melting of $MgCl_2 \bullet 6H_2O$ in its water of crystallisation at 117 °C. Magnesium chloride di-hydrate can also behave in this manner.

In light of this behaviour the "dry" crystals were dried further before a second tube furnace test was attempted. The crystals were spread in a flat-bottomed glass pan and placed in a muffle furnace at 90 °C for approximately 12 hours and 110 °C for 2 hours.

A second dehydration test in the rotating tube was performed using the "re-dried" crystals.

A 200 gram charge was placed in the glass tube, inside the furnace. The tube was rotated slowly. Air, nitrogen and dry HCl gas were injected using the schedule shown in Table No. 2.

The full details may be found in Appendix A (Test F-01). The assay of the dehydrated material is shown in Table No. 3. The theoretical magnesium content of anhydrous $MgCl_2$ is 25.5%. The lower than theoretical Mg content in the product is probably due to the impurities.

Table No. 2 – Gas and Temperature Regime used in Thermal Dehydration Procedure

Time	Temp	Gas Flow (l/min)		
(min)	(°C)	Air	N_2	HCl
180	90	3.0	0	0
60	120	"	0	0
60	140	"	0	0
60	140	0	2.6	0.7
60	185	0	2.6	0.7
60	350	0	2.6	0.7
cool down		0	2.6	0.05

---

[1] Kh L. Strelets – Electrolytic Production of Magnesium –1972, Moscow

Table No. 3 – Chemical Analysis of Thermal Dehydration Product

Mg (%)	23.5	Ca (%)	0.41	MgO (%)	0.98
Al (g/t)	***	Mn (g/t)	***	Cl (g/t)	***
As (g/t)	***	Mo (g/t)	***	Si (%)	0.03
Ba (g/t)	***	Na (g/t)	***	Mn (%)	0.01
Be (g/t)	***	Ni (g/t)	***	Al (%)	< 0.01
Cd (g/t)	***	P (g/t)	***	Fe (%)	0.01
Co (g/t)	***	Pb (g/t)	***	Cu (%)	0.001
Cr (g/t)	***	Sb (g/t)	***	B_tot (g/t)	***
Cu (g/t)	***	Se (g/t)	***	C(t) (%)	***
Fe (g/t)	***	Sn (g/t)	***	P (%)	< 0.01
K (g/t)	***	Te (g/t)	***	Na (%)	0.07
La (g/t)	***	Y (g/t)	***	K (%)	0.02
		S (%)	0.08	Cr (%)	< 0.01
				Ni (%)	< 0.001

1.4.3 Alcan Dehydration

Two dehydration tests were performed using the "re-dried" crystals as feed (the same feed as for the thermal dehydration test) to a patented Alcan dehydration process. The basis for the process is the conversion of the magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$), which is soluble in methanol, to magnesium chloride ammoniate ($MgCl_2{\cdot}6NH_3$), which is insoluble at low ambient temperatures.

The test procedure used was derived from the patent (see Appendix B) which is in the public domain. As no agreement had been struck, by the time of the testwork, between Golden Triangle Resources and Alcan on the potential of obtaining a license for the use of the patented process, only information in the public domain could be used for this work. This was complemented by a few comments from Dr. Creber, Alcan, which were requested by LRL, prior to and following the first test. The details of the test procedure appear in Appendix A.

The first test, "Alcan 1", was not successful. Analysis of the precipitate by x-ray diffraction indicated that it was magnesium chloride hydroxide hydrate ($Mg_2(OH)_3Cl{\cdot}4H_2O$). It is not fully clear why this material formed, but it is most likely due to the fact that the precipitation system was not saturated with ammonia throughout the process (occasional blockage of the ammonia tube with precipitate, other manipulation problems during the test), thereby possibly.

The procedure was repeated in Test "Alcan 2", with minor modifications to the procedure following discussions of one of the authors with Dr. Creber of Alcan. The ammonia gas flow was increased, and other mechanical and chemical modifications were made to assure sufficient ammonia and the saturation of the solution with gas at all times.

The precipitate produced in this test had a Mg content similar to that expected for the ammoniate complex, 12.8 % Mg. The metallurgical balance appears in Table No. 4. The precipitate was examined by X-ray diffraction. There was no evidence of any oxychlorate or hydroxychloride compounds, as had plagued the first test. However, in view of the lack of ICDD X-ray diffraction patterns for the desired product, it could not be directly proven that the material was $MgCl_2 \bullet 6NH_3$. Various research laboratories, including Alcan, CRM, CANMET, were contacted, which confirmed the absence of the data. It is interesting to note that no Xray diffraction evidence of the ammoniate product accompanied the original Alcan work, which used SEM analysis to provide indirect confirmation. The Alcan Xray diffraction pattern shows similarity to the LRL pattern, insofar as the "$MnCl_2 \bullet 6NH_3$" lines go (there was no significant manganese present in *either* Alcan or LRL tests).

The metallurgical balance shows that 97 percent of the magnesium reported to the precipitate, indicating a very high recovery. The methanol solution contained 35 percent of the calcium. This was lower then was hoped for or predicted by Alcan.

Additional impurity analyses of the precipitate are given in Table No. 5.

Table No. 4 – Metallurgical Balance for "Alcan 2" Precipitation

Product	Amount	Assays (mg/L, %)			Distribution (%)		
	(ml, g)	Mg	Ca	S	Mg	Ca	S
Preg	270	141	74.9	n.a.*	1.2	35.1	...
Wash	60	1161	21.5	n.a.*	2.2	2.2	...
Precip't	24.1	12.8	0.15	<0.01	96.6	62.7	...
Head (cal)	25.0	12.8	0.23	0.00	100	100	...
Head (dir)	25.0	13.6	0.24	0.06			

* Difficulties in organic-aqueous assays led to erroneous S analysis

Table No. 5 – "Alcan 2" Precipitate Impurity Analysis

Product	Assay (%)				
	Fe	Mn	Ni	Zn	Si
Precip't	<0.005	<0.005	<0.002	<0.001	<0.02

Reagents employed in the test program

The following reagents were used in the testwork:

Calcine – calcined magnesite, prepared by heating MC-36 ore for 60 minutes at 650 °C, the calcine had a LOI of 8.1% and a measured "activity" of 72 seconds.

MgO – ACS grade by Fisher Scientific
$Mg(OH)_2$ – Lab grade by Nymoc
HCl – A.R. grade by Anachemia – 36%
H_2SO_4 –A.R. grade by Anachemia – 96%
$BaCl_2$ – A.R. grade by BDH
H_2O_2 – A.R. grade by Spectrum – 35%
Chlorine gas – A.R. grade bottled gas
Water – distilled water was use throughout testwork
NH_3 – A.R. grade bottled gas
Methanol, and Ammonium chloride – reagent grade

2. Discussion of Dehydration Test Results

A full discussion is presented in the Final report of this project (Progress Report No 4, April 6, 1999).

The products of both thermal dehydration in HCl atmosphere (Norsk-Hydro chemistry) and ammoniation of magnesium chloride in methanol-ammonia solutions (Alcan ADT process) yielded anhydrous final products, apparently consistent with the chemistry of anhydrous magnesium chloride and magnesium chloride ammoniate, respectively. Further data on the composition of the two products appears in the Abstract of the Final Report of this project (Progress Report No 4).

The thermal dehydration product yielded 0.98 % MgO, which is believed to be of the order of about five to ten times as high as that desirable in modern electrolytic cells, such as the Alcan MPC. The oxygen content of the thermal dehydration test was not due to the impurity level of the bottled gases used. It is likely caused by the difficulty of maintaining (1) high HCl-water vapour ratios at all temperatures and times of the test, and (2) sufficient impermeability to the outside atmosphere at rotating tube joints. The high MgO levels would be expected to decrease significantly in further improved experimental procedure.

Overall, the two processes acquitted themselves reasonably well in these preliminary scoping tests, notwithstanding the difficulties inherent in the testing of hitherto unexperimented methods. For the reasons set out in the Abstract of this report, the Alcan ADT process is recommended to GTR for further development and integration into the new magnesite to magnesium process.

Further experimental work in Phase 2 would permit further optimisation and development of the Alcan ADT dehydration process (See chapter 1.2 Recommendations in the Final Report, Progress Report No 4, April 6, 1999).

3. Conclusions on the Dehydration Testwork

Satisfactory scoping tests were performed with the two dehydration technologies subjected to experimental evaluation. Products obtained were largely as anticipated, and significantly better than that targeted in the project proposal. The high MgO content of the $MgCl_2$ product of thermal dehydration would be readily correctable in further experimentation at laboratory scale. However, the engineering of a successful large scale system presents a considerable challenge, which would require input from an experienced dehydration engineering company or lengthy and expensive pilot scale testwork, proceeding through various scale-up intervals, which is common to the development of many industrial pyrometallurgical systems.

The Alcan process is considered more easily adaptable to the GTR needs via further laboratory and pilot scale testwork. The primarily aqueous-organic system allows large scale-up factors, the technology is accessible (possibly at relatively low cost) and Alcan has expressed an interest in working with LRL in respect of this possible development for GTR. The product of Alcan dehydration is likely magnesium chloride ammoniate, as was targeted. Calcium was partially eliminated to the residual aqueous-organic stream. Further testwork, with the aid of Alcan Kingston, involving development of optimal reaction conditions and process stream recycling, is clearly necessary to further evaluate and integrate this dehydration option into the proposed (See Final Report, Figure 1A) GTR magnesite to magnesium flowsheet.

More detailed conclusions and recommendations appear in the Final Report (Progress Report No 4) of this project.

APPENDIX A

TEST DETAILS

5398

INSTRUCTIONS

Test 38

Purpose: Simplifification of purification flowsheet
To study the filtering and settling characteristics

Procedure:

Conduct 2 leaches on MC36 110% stoich acid 80°C and 60 minutes
Using double condeser on both leaches
combine two leaches and record weight

Stage 1 -

Place pulp in beaker in 80 °C water bath.
Heat to temperature
Neutralize to pH 1.5 to 2.0 with calcine
Neutralize to pH 4.5 with slaked $Mg(OH)_2$
Raise emf by slowly bubbling Cl_2 gas into solution
Add slaked Mg(OH)2 to system to keep pH in range of 3.5 to 4.5
while adjusting emf with Cl_2
After a high emf is reached, add 14.6g of reagent grade H_2SO_4
and slaked Mg(OH)2 at the same time to keep pH in range 3.5 to 4.5
When reading are stable (high pH ~ 4.5 and high emf ~ 350)
and slaked Mg(OH)2 to pH 5.5 to 5.8
Add 5.0g $BaCl_2 \cdot 2H_2O$ mixed in water, mix for 5 minutes
Add bit of Cl_2 to raise emf a bit
Filter
Submit filtrate for Mg, Ca, Si, Na, B, Mn, Al, Cr, V, Ni, Cu, Zn, Fe, S, and Ba
Submit wash for Mg and precipatate for whole rock and Cl

Project: 5398
Test: LCH 38

Technologist: G. Toole
Date: Dec. 3, 1998

Purpose: To determine the solid-liquid separation characteristics of the neutralized leach liquor, and measure the basic impurity levels of the levels of the final and intermediate solutions.

Leach Conditions:

Leach Feed: MC36
Crush Size: minus 10 mesh
Feed weight 2 x 20 g
HCl added: 2 x 50 g
Water added: 2 x 73 g
HCl Concentration: 36 %
HCl Stoichiometric concentration: 110 %
Initial Percent Solids: 25.7 %w/w
Initial L/S 2.48 ml sol'n / g solid
Temperature: 80 °C
Time: 60 min
pH: not controlled
Mixer Speed: 900 rpm

Procedure:

1. A glass reaction kettle with a lid was set up. The lid had 4 ports, one port was used for the impeller, one used for a condenser, and one for added the sample.
2. Hydrochloric acid was added to the reactor and heated to the desired temperature.
3. Once at temperature the sample was added slowly to avoid frothing over.
4. Once all the sample was in the test was started.
5. Periodic redox readings were taken during the test.
6. At the conclusion of the test both samples were combined and a thief sample was taken

Data:

Leach A				**Leach B**			
Time min	Temp °C	emf mV Ag/AgCl	Comments	Time min	Temp °C	emf mV Ag/AgCl	Comments
0	80	230		0	80	236	
30	80	305		30	80	335	
60	80	357		60	80	398	

Time	Temp	pH	emf (mV)	Additions (g, mL/min)					Comments
	°C		Ag/AgCl	Calcine	Mg(OH)2 (20%)	Cl_2	H_2SO_4 (conc)	$BaCl_2 \cdot 2H_2O$	
10:20	72	-1.29	396						
10:32	77	-1.09	390	3.91					
10:40	78	1.93	376		2.61				
10:46	77	4.61	-177			47.6			
10:51	77	4.58	-180			72.4			
11:09	77	4.47	-92			–			
11:18	77	5.26	355		17.31	–			
11:40	77	1.96	727		37.6	–	14.6		gas off
11:45	78	5.55	280			–		5	
11:50	77	5.65	243			72.4			gas off 11:56
11:58	77	5.76	855		20.45				

5398
LCH 38

Filtration:

Filter cloth:

Media No	1820	Th. Count	68 X 60
Weave	3-1 twill	Wt	4.4 oz/sq.ft
Yarn	Polyester multifill	Air flow	22 cfm/sq.ft
		Finish	heat set

Dry Cake Weight:	52.4	Filter Diameter	11 cm
Wet Cake Weight:	81.21	Filter Area	0.00950332 m^2
Moisture Content:	35.5		

Stage	Time (sec)		Vol (ml)	Cake Rate ($kg/hr/m^2$) form only		Filtrate Rate ($l/hr/m^2$) form only
	Form	Dry		wet	dry	
Filtration	202	0	236.0	152.3	152.3	443
Wash 1	44	0	26.0			224
Wash 2	30	0	30.0			379
Wash 3	19	0	28.5			568
Wash 4	15	0	26.5			669
Wash 5	15	0	25.0			631

Wash (ave) 494

Cake thickness: 6 mm
Wet weight: 81.21 g
Dry weight: 57.16 g
Percent moisture: 29.6 % (air dried)

Product	Amount (ml, g)	Assays (mg/l, %)														
		Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	1080	94100	<0.05	1510	0.18	6.75	0.59	2.07	<0.50	<0.10	<0.10	<0.10	0.1	2.49	818	0.37
wash 1	26.0	52500	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 2	30.0	35000	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 3	28.5	22800	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 4	26.5	16700	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 5	25.0	16400	na	na	na	na	na	na	na	na	na	na	na	na	na	na
residue	52.4	13.1	0.56	6.88	4.75	0.08	7.6		0.13	<0.01	<0.01					

Product	Distribution (%)														
	Mg	Mn	Ca	Fe	Na	Si	B	Al	Cr	V	Ni	Cu	Zn	S	Ba
filtrate 1	90.4	0.02	31.1	0.01	14.8	0.02	100.0	0.8	2.0	2.0	100	100	100	100	100
wash 1	1.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
wash 2	0.9	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 3	0.6	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 4	0.4	na	na	na	na	na	na	na	na	na	na	na	na	na	na
wash 5	0.4	na	na	na	na	na	na	na	na	na	na	na	na	na	na
residue	6.1	99.98	68.9	99.99	85.2	99.98	0.0	99.2	98.0	98.0	0.0	0.0	0.0	0.0	0.0
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	

Additional Assays

Product	Assays %	
	Cl	LOI
residue	1.82	23.4

16.

LCH 38

5398

Project: 5398
Test: F-01

Technologist: G. Toole
Date: Feb. 11, 1999

Purpose: To produce a dehydrated $MgCl_2$ product

Procedure:

1. 200g of crystals from test 38 were dried in the muffle furnace for 22 hours at 90°C and stored in a tightly seal bottle. They were then dried at 110°C for 2 hours
2. 25g were then loaded into the rotary tube and heated to 90°C for 2 hours with ~3 L/min air flow
3. After 2 hours the temperature was increased to 120°C with the same air flow
4. After 1 hour the temperature was increased to 140°C with the same air flow
5. After 1 hour the gas flows were changed to 100mL/min anhydrous HCl and 2.6L/min N_2
6. After 1 hour the temperature was increased to 185°C
7. After 1 hour the temperature was increased to 350°C
8. After 1 hour the temperature was increased to 250°C
9. After 1 hour the tube was cooled in 2.6L/min N_2 and 50mL/min HCl
10. Once cool the gases were stopped and the tube emptied into a glass jar purged with nitrogen and then sealed and put in a dessicator overnight

Data:

Time	Temp	Gas flow (ml/min)			Comments
(min)	(°C)	Air	N_2	HCl	
0	23	3000			
11	88	3000			
26	9	3000			
61	92	3000			
81	91	3000			
102	127	3000			
110	124	3000			
156	122	3000			
163	142	3000			
209	140	3000			
230	140		2600	100	
257	140		2600	100	
273	142		2600	100	
278	142		2600	100	
285	141		2600	100	
315	183		2600	100	
332	184		2600	100	
363	185		2600	100	
383	250		2600	100	
409	251		2600	100	
428	351		2600	100	
448	351		2600	100	
478	350		2600	100	

Assays

Product	Assays(mg/L)				
	Mg	Ca	MgO	Zn	S
Feed	13.6	0.26			0.06
Calcine	23.5	0.41	0.98	0.001	0.08

Additional Assays - Calcine

Al (g/t)	***	Mn (g/t)	***	Cl (g/t)	***
As (g/t)	***	Mo (g/t)	***	Si (%)	0.03
Ba (g/t)	***	Na (g/t)	***	Mn (%)	0.01
Be (g/t)	***	Ni (g/t)	***	Al (%)	< 0.01
Cd (g/t)	***	P (g/t)	***	Fe (%)	0.01
Co (g/t)	***	Pb (g/t)	***	Cu (%)	0.001
Cr (g/t)	***	Sb (g/t)	***	B_tot (g/t)	***
Cu (g/t)	***	Se (g/t)	***	C(t) (%)	***
Fe (g/t)	***	Sn (g/t)	***	P (%)	< 0.01
K (g/t)	***	Te (g/t)	***	Na (%)	0.07
La (g/t)	***	Y (g/t)	***	K (%)	0.02
				Cr (%)	< 0.01
				Ni (%)	< 0.001

Project: **5398**
Test: **Alcan 1**

Technologist: G. Toole
Date: Feb. 12, 1999

Purpose: To simulate the Alcan process in the production of dehydrated $MgCl_2$

Feed: Magnesiun chloride brine, heated as a liquid to 130 °C , cooled to solid, crushed, and dried in air at 90 °C for about 12 hours.

Procedure:

1. 200mL of analytical grade methanol was placed in a water bath a 20°C
   Anhydrous ammonia was sparged into the methanol to saturate it (flowrate of 280mL/min)
2. 25g of dried crystals were mixed with 140mL of analytical grade methanol and 1.53g of ammonium chloride
3. This mixture was pumped into the saturated methanol over a 1 hour period
4. Ammonia was continuously sparged into the pulp over the entire test period.
5. Once all the mixture was in the beaker was rinsed with 40mL of methanol (not ammonia saturated) and pumped into the slurry and mixed for an additional 15min.
6. The slurry was then filtered and wash with ammonia saturated methanol
7. The preg and wash were kept separate and assayed for Mg. Ca, and S
8. The residue was assayed wet for MgO and dry for Mg, Ca, and S
9. Samples of the feed and precipitate were submitted for XRD analysis.

Cake Moisture

Total wet weight (g)	26.7
Sub-sample wet weight(g)	5.2
Sub-sample dry weight(g)	1.8
Precent moisture	65.4

Metallurgical Balance:

Product	Amount	Assays (mg/L, %)			Distribution (%)		
	(mL, g)	Mg	Ca	S	Mg	Ca	S
Preg	330	1620	60.8	<3	15.7	34.0	25.4
Wash	50	791	31.8	<3	1.2	2.7	3.8
Residue	26.7	10.6	0.14	0.01	83.1	63.3	70.8
Head (cal)	25	13.6	0.24	0.02	100	100	100
Head (dir)	25	13.6	0.24	0.06			

Client: Golden Triangle Resources
LRL Project: 5398, LIMS # MAR4505.R99

Lakefield Research Limited
11-Mar-98

Summary of Qualitative X-ray Diffraction Results

	Crystalline Mineral Assemblage (relative proportions based on peak height)			
Sample	**Major**	**Moderate**	**Minor**	**Trace**
Oven Dried	magnesium chloride hydride	magnesium chloride hydrate	-	*chloromagnesite

*Tentative identification due to low concentrations, diffraction line overlap or poor crystallinity

Instrument:	**Siemens D5000 diffractometer**
Scan Conditions:	**Co radiation, graphite monochromator, 40Kv, 30mA, Step: .02°, Step time: 1.0 s**
Interpretations:	**JCPDS / ICDD powder diffraction files. Siemens Search / Match software.**
Detection Limit:	**0.5-2%. Strongly dependent on crystallinity.**

Interpretations do not reflect the presence of non-crystalline / amorphous compounds. Mineral proportions are based on relative peak heights and may be strongly influenced by crystallinity, structural group or preferred orientations.
Interpretations and relative proportions should be accompanied by supporting petrographic and geochemical data (WRA, ICP-OES).

Mineral	**Composition**
Chloromagnesite	$MgCl_2$
Magnesium Chloride hydrate	$MgCl_2.4H_2O$
Magnesium Chloride hydride	MgClH

Note:

Relative mineral proportions are difficult to determine by this method due to poor sample crystallinity. The main diffraction peaks of chloromagnesite overlap with those of magnesium chloride hydride. Bischofite ($MgCl_2.6H_2O$) was not identified in this sample.

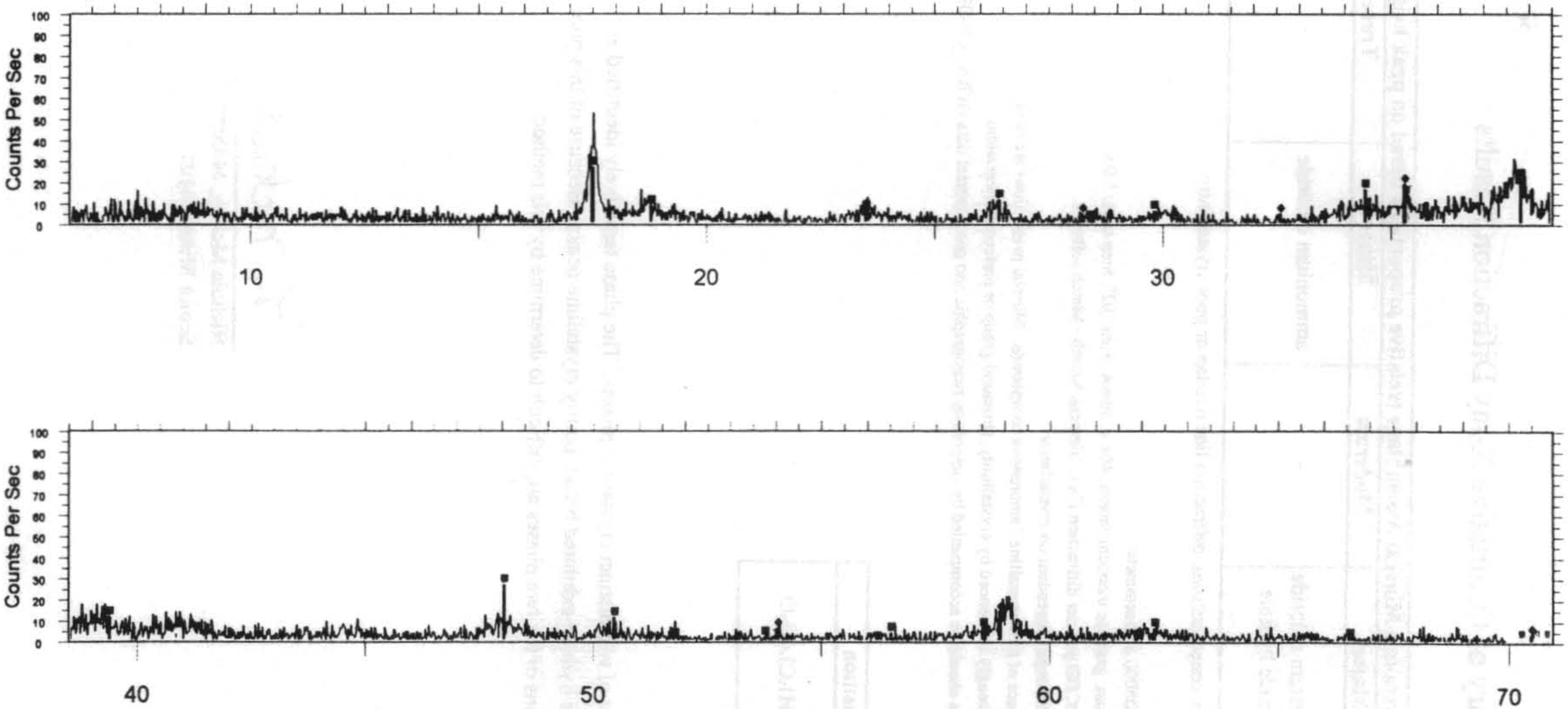
Lisa Ryan
XRD Technician

Nichola McKay
Nichola McKay, M.Sc.
Senior Mineralogist

The Qualitative XRD method (METH # 8-8-1) used by Lakefield Research Limited Mineralogical Services is accredited to the IEC/ ISO Guide 25 standard by the Standards Council of Canada. Mini-method available upon request.

827244

Oven Dried



Lakefield Research Limited

File: mar4505-1.RAW - Start: 6.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 1.0 s
37-0774 (I) - Chloromagnesite, syn [NR] - MgCl2
■ 01-1210 (N) - Magnesium Chloride Hydrate - MgCl2·4H2O
◆ 22-0702 (N) - Magnesium Chloride Hydride - MgClH

5 cm

Client: Golden Triangle Resources
LRL Project: 5398, LIMS # MAR4502.R99

Lakefield Research Limited
5-Mar-98

Summary of Qualitative X-ray Diffraction Results

Sample	Crystalline Mineral Assemblage (relative proportions based on peak height)			
	Major	**Moderate**	**Minor**	**Trace**
Alcan 1	*magnesium chloride hydroxide hydrate	-	ammonium chloride	-

*Tentative identification due to low concentrations, diffraction line overlap or poor crystallinity

Instrument:	Siemens D5000 diffractometer
Scan Conditions:	Co radiation, graphite monochromator, 40Kv, 30mA, Step: .02°, Step time: 1.0 s
Interpretations:	JCPDS / ICDD powder diffraction files. Siemens Search / Match software.
Detection Limit:	0.5-2%. Strongly dependent on crystallinity.

Interpretations do not reflect the presence of non-crystalline / amorphous compounds. Mineral proportions are based on relative peak heights and may be strongly influenced by crystallinity, structural group or preferred orientations. Interpretations and relative proportions should be accompanied by supporting petrographic and geochemical data (WRA, ICP-OES).

Mineral	Composition
Ammonium Chloride	NHCl
Magnesium Chloride hydroxide hydrate	$Mg_2(OH)_3Cl.4H_2O$

Note:

The sample appeared to consist of two distinct crystalline phases. The phase tentatively identified as magnesium chloride hydroxide hydrate exhibited broad, poorly crystalline peaks. Because of this poor crystallinity, relative proportions of the two phases are difficult to determine by this method.

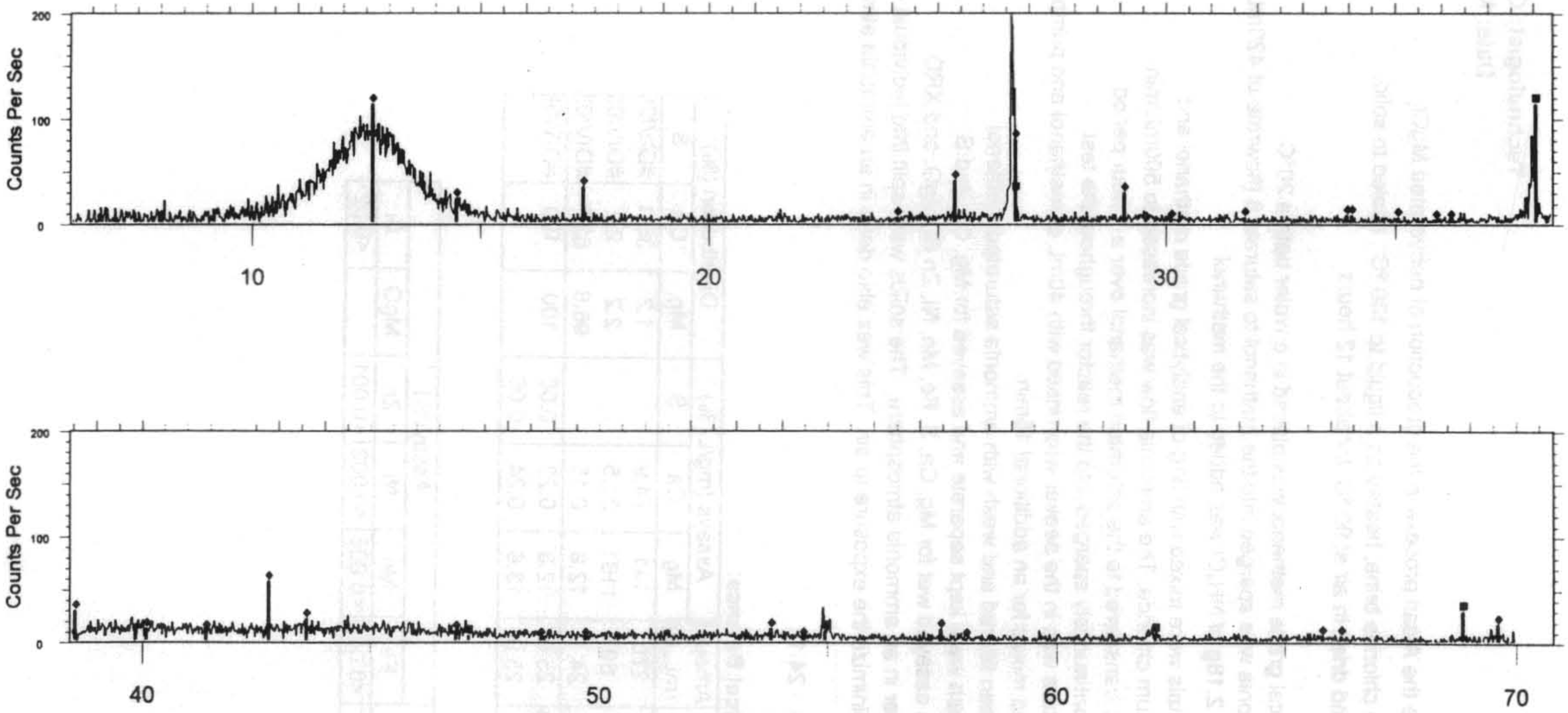
Lisa Ryan
XRD Technician

L. McKay
Nichola McKay, M.Sc.
Senior Mineralogist

The Qualitative XRD method (METH # 8-8-1) used by Lakefield Research Limited Mineralogical Services is accredited to the IEC/ ISO Guide 25 standard by the Standards Council of Canada. Mini-method available upon request.

827246

Alcan 1



Lakefield Research Limited

File: mar4502-1.RAW - Start: 6.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 1.0 s

◆ 07-0412 (I) - Magnesium Chloride Hydroxide Hydrate - $Mg_2(OH)_3Cl \cdot 4H_2O$

■ 07-0007 (I) - Salammoniac, syn - NH_4Cl

5 cm

23.

Project: **5398** **Technologist:** G. Toole
Test: **Alcan 2** **Date:** March 18/99

Purpose: To simulate the Alcan process in the production of dehydrated MgCl$_2$

Feed: Magnesiun chloride brine, heated as a liquid to 130 °C , cooled to solid, crushed, and dried in air at 90 °C for about 12 hours.

Procedure:

1. 200mL of analytical grade methanol was placed in a water bath a 20°C
   Anhydrous ammonia was sparged into the methanol to saturate it (flowrate of 420mL/min)
2. After 30 minutes 2.18g of NH$_4$Cl was added to the methanol
3. 25g of dried crystals were mixed with 31g of analytical grade methanol and 0.53g of ammonium chloride. The ammonia flow was increased to 500mL/min
4. This mixture was transferred to the saturated methanol over a 1 hour period
5. Ammonia was continuously sparged into the reactor throughout the test.
6. Once all the mixture was in the beaker was rinsed with 40mL of methanol and pumped into the slurry and mixed for an additional 15min.
7. The slurry was then filtered and wash with ammonia saturated methanol
8. The preg and wash were kept separate and assayed for Mg. Ca, and S
9. The residue was assayed wet for Mg, Ca, S, Fe, Mn, Ni, Zn Si, MgO, and XRD
10. The pulp was filter in an ammonia atmosphere. The solids were split into individual cups for each assay to minimize the exposure to air. This was also done in an ammonia atmosphere.

Total wet weight (g) 24.12

Metallurgical Balance:

Product	Amount (mL, g)	Assays (mg/L, %)			Distribution (%)		
		Mg	Ca	S	Mg	Ca	S
Preg	270	141	74.9		1.2	35.1	#DIV/0!
Wash	60	1161	21.5		2.2	2.2	#DIV/0!
Residue	24.1	12.8	0.15		96.6	62.7	#DIV/0!
Head (cal)	25.0	12.8	0.23	0.00	100	100	#DIV/0!
Head (dir)	25.0	13.6	0.24	0.06			

Additional Assays

Product	Assay(%)					
	Fe	Mn	Ni	Zn	MgO	Si
Residue	<0.005	<0.005	<0.002	<0.001		<0.02

Project: **5398**

Test: **Alcan2Calcine**

Technologist: G. Toole

Date: March 25, 99

Purpose: To produce a calcined $MgCl_2$ product from MgCl2.6NH3 (Alcan2 test product)

Procedure:

1. 11 g of product sample from test Alcan2 weighed (compare with weight of this same sample following the Alcan2 test); record weight and weight change;
2. Load sample into clean rotary tube (electric furnace)and purge out air with NH3 at 500 mL/min for 20 minutes;
3. Start heating and raise temperature to 400 C, while maintaining NH3 flow;
4. After 20 minutes at 400 C, and ammonia flow of 500 mL/min.switch from NH3 to Argon gas
5. Maintain at 400 C for 20 min. under Argon (500 mL/min.), thereafter raise temperature to 600 C
6. Maintain at 600 C for 20 minutes, then cool under Argon flow to room temperature;
7. When fully cooled, remove sample, weigh and transfer to Analysis (MgO)

Data:

Time (min)	Temp (°C)	Gas flow(mL/min) NH3	Argon	Comments
0	14	500		start NH3 flow
10	51	500		
20	60	500		start heat
30	323	500		
35/0	424	500	500	change to argon
10	382		500	
20	400		500	
40	404		500	start heat
50/0	604		500	
10	602		500	
20	598		500	
				approximatly 2.9 g of calcine
				start with 10.8g wet

Product	Assays (%)				
	Mg	Ca	MgO		
Feed Calcine					

25.

131

827249

Summary of Qualitative X-ray Diffraction Results

Sample	**Crystalline Mineral Assemblage (relative proportions based on peak height)**			
	Major	**Moderate**	**Minor**	**Trace**
Alcan 2	chloromagnesite	*manganese chloride ammonia, ammonium chloride	-	*magnesium chloride hydride

*Tentative identification due to low concentrations, diffraction line overlap or poor crystallinity

Instrument:	Siemens D5000 diffractometer
Scan Conditions:	Co radiation, graphite monochromator, 40Kv, 30mA, Step: .02°, Step time: 1.0 s
Interpretations:	JCPDS / ICDD powder diffraction files. Siemens Search / Match software.
Detection Limit:	0.5-2%. Strongly dependent on crystallinity.

Interpretations do not reflect the presence of non-crystalline / amorphous compounds. Mineral proportions are based on relative peak heights and may be strongly influenced by crystallinity, structural group or preferred orientations. Interpretations and relative proportions should be accompanied by supporting petrographic and geochemical data (WRA, ICP-OES).

Mineral	**Composition**
Ammonium Chloride	NH_4Cl
Chloromagnesite	$MgCl_2$
Magnesium Chloride Hydride	$MgClH$
Manganese Chloride Ammonia	$MnCl_2.6NH_3$

Note:

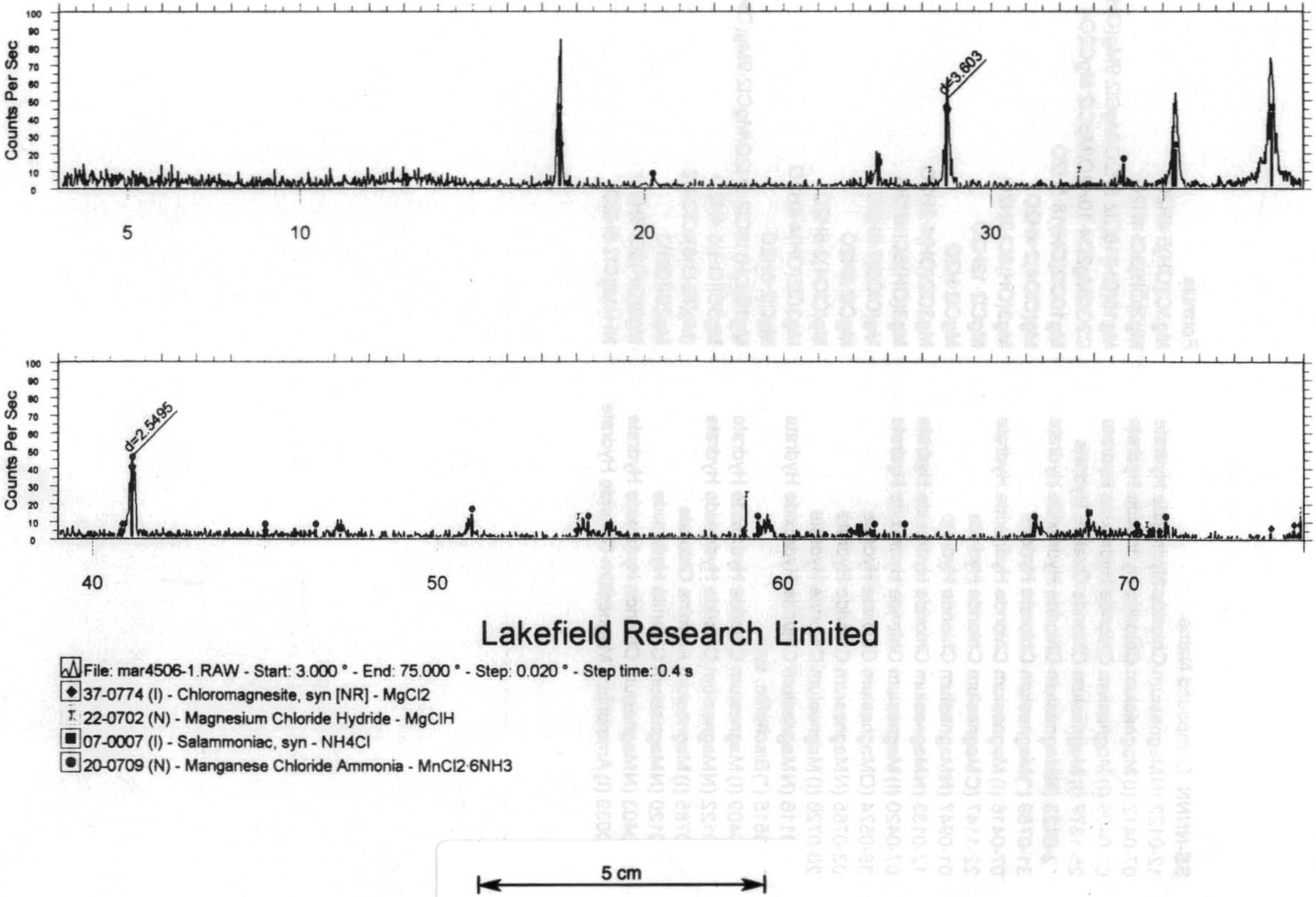
Relative mineral proportions are difficult to determine by this method due to poor sample crystallinity. The main diffraction peaks of chloromagnesite overlap with those of magnesium chloride hydride. The ICDD powder diffraction database does not contain a pattern for $MgCl_2.6NH_3$.
No other magnesium chloride phase was identified in the sample. Diffraction patterns for phases of magnesium chloride hydroxide hydrate, magnesium chlorate hydrate, magnesium chloride oxalate hydrate, magnesium chloride hydrate and ammonium magnesium chloride hydrate, were checked and did not correspond to the diffraction pattern of the sample.

Lisa Ryan
XRD Technician

Nichola McKay, ~~M.Sc.~~
Senior Mineralogist

The Qualitative XRD method (METH # 8-8-1) used by Lakefield Research Limited Mineralogical Services is accredited to the IEC/ ISO Guide 25 standard by the Standards Council of Canada. Mini-method available upon request.

Alcan 2


Counts Per Sec
0
10
20
30
40
50
60
70
80
90
100
d=3.603
5
10
20
30
d=2.5495
40
50
60
70
Lakefield Research Limited
File: mar4506-1.RAW - Start: 3.000 ° - End: 75.000 ° - Step: 0.020 ° - Step time: 0.4 s
◆ 37-0774 (I) - Chloromagnesite, syn [NR] - MgCl2
I 22-0702 (N) - Magnesium Chloride Hydride - MgClH
■ 07-0007 (I) - Salammoniac, syn - NH4Cl
● 20-0709 (N) - Manganese Chloride Ammonia - MnCl2·6NH3
5 cm

SS-NNNN	Compound Name	Formula
12-0122	(NMagnesium Chloride Hydroxide Hydrate	Mg3Cl(OH)5·4H2O
07-0412	(I) Magnesium Chloride Hydroxide Hydrate	Mg2(OH)3Cl·4H2O
07-0409	(I) Magnesium Chloride Hydroxide Hydrate	Mg10(OH)18Cl2·5H2O/MgCl2·9Mg(OH)2·5H2O
25-1377	(I) Magnesium Chloride Oxalate Hydrate	C2Cl2Mg2O4·10H2O/MgCl2·MgC2O4·10H2O
12-0123	(NMagnesium Chloride Hydroxide Hydrate	Mg10Cl2(OH)18·5H2O
31-0789	(*)Magnesium Chlorate Hydrate	Mg(ClO4)2·xH2O
07-0416	(I) Magnesium Chloride Hydroxide Hydrate	Mg3(OH)5Cl·3H2O
22-1147	(CMagnesium Chloride Hydrate	MgCl2·12H2O
01-0947	(NMagnesium Chloride Hydrate	MgCl2·H2O
12-0133	(NMagnesium Chloride Hydroxide Hydrate	Mg3Cl2(OH)4·2H2O
07-0420	(I) Magnesium Chloride Hydroxide Hydrate	Mg3(OH)5Cl·4H2O
38-0574	(QMagnesium Chlorate Hydrate	Mg(ClO3)2·6H2O
03-0765	(NMagnesium Chloride Hydrate	MgCl2·2H2O
20-0726	(I) Magnesium Chlorate Hydrate	Mg(ClO4)2·6H2O
12-0116	(NMagnesium Chloride Hydroxide Hydrate	Mg3Cl2(OH)4·4H2O
25-0515	(*)Bischofite, syn	MgCl2·6H2O
07-0409	(I) Magnesium Chloride Hydroxide Hydrate	Mg10(OH)18Cl2·5H2O/MgCl2·9Mg(OH)2·5H2O
12-0122	(NMagnesium Chloride Hydroxide Hydrate	Mg3Cl(OH)5·4H2O
31-0785	(I) Magnesium Ammine Chlorate	(Mg(NH3)6)(ClO4)2
12-0120	(NMagnesium Chloride Hydroxide	Mg2Cl(OH)3
07-0403	(NMagnesium Chloride Hydroxide Hydrate	Mg2(OH)3Cl·3H2O
25-0039	(I) Ammonium Magnesium Chloride Hydrate	NH4MgCl3·6H2O

APPENDIX B

Alcan Process

"U.S. Patent No. 5,514,359 – Process for Making Anhydrous Magnesium Chloride"

To be forwarded with
Final report

99-03-26