



Industrial Mineral Testing Program

December 1996

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Prepared for James Bay Lowlands Gypsum Development Group

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By New Worlds Engineering

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Industrial Mineral Testing Program - Moose River Gypsum

<u>Goal</u>

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Perform processing and physical property testing on samples of Moose River gypsum, to determine the type and form of hemi-hydrate (stucco) that can be produced. A materials testing program, which includes the measurement of calcining temperatures and time, water up-take and setting times, will reveal the specific properties of the stucco to determine marketing potential.

Gypsum

Gypsum is a non-metallic mineral, found as a rock composed of 79.1% calcium sulfate and 20.9% chemically bonded water by weight (calcium sulfate dihydrate CaSO₄ \cdot 2H₂O). Gypsum was formed during the Silurian period of the Paleozoic Era of the geological calendar. This corresponds to approximately 300 million years ago. In absolutely pure form, gypsum rock is white. However, as found in nature, it most often contains impurities whose presence makes the rock appear gray, brown, pink and even black.

The water contained in the structure gives this mineral some particularly useful properties. Gypsum readily gives up, or takes on this crystalline water. With the application of a moderate amount of heat in a process known as calcining, gypsum is converted into what is commonly called "Plaster of Paris" or stucco. This hemihydrate calcium sulfate (CaSO₄ · $\frac{1}{2}$ H₂O) can easily be changed back to the dihydrate form by simply mixing it with liquid water. Conveniently, there is a certain "setting" time between the addition of water, and when the dihydrate becomes fully solid. This means that the mixture can be shaped before it hardens.

Gypsum and its products have been known and used from the earliest times. The Ancient Assyrians called it Alabaster and used it for sculpting. Five thousand years ago the Egyptians had learned to make plaster from it. The ancient Greeks named this mineral "Gypsos", from which came the name "Gypsum". One particular form of the mineral, clear and transparent in layers like mica, was used as temple windows many centuries before glass was invented. The Greeks called it "Selene" after the moon goddess - today we call it Selenite.

In modern times gypsum is used in the manufacturing of many products including: drywall board, plaster, sheathing, toothpaste, blackboard chalk, and as filler in paints. It is also used as a surgical casting material and molds for false teeth.

In the gypsum industry, calcining is the step of reducing dihydrate to hemihydrate or anhydrous (CaSO₄) form. In the very early history of gypsum plaster production, pieces of rock were simply heated in an open wood fire to bring about the dehydration process. Today, stucco is produced using sophisticated vertical kilns or fluid bed roasters. Quality control of mined material has become critical in terms of impurities, sizing and separation of natural anhydrates. These factors have been found to significantly alter the form and usefulness of the stucco produced. Laboratory tests, such as the type performed for this report, are the first step in determining the possible uses of the gypsum found in the Moose River area.

Hemihydrate

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Hemihydrate calcium sulfate has two basic forms, alpha and beta, the processes and properties of each are outlined below:

Alpha Hemihydrate - produced in a water saturated atmosphere at temperature >97°C. This is usually accomplished at elevated temperature in an autoclave in the presence of steam. It is more stable, or less reactive, than the beta form, and has a slower rate of strength development. This is a disadvantage in many uses of stucco, but nevertheless rehydrated alpha hemihydrate makes a denser, stronger plaster which has advantages in other uses.

Beta Hemihydrate - can be produced in a vacuum or non-saturated atmosphere at 100°C. Usually it is produced in a less than saturated environment at atmospheric pressure. The beta form is cheaper to produce and is usually used for wall board. It requires more water to form, but it sets quicker. Beta form was produced during this testing program.

	Normal Consistency *	Setting Range (minutes)	Typical set expansion (in per in)	Avg. compresive strength dry (psi)
Alpha	40-43	20-30	0.003	5500
Beta	64-66	10-20	0.0018	2000

*parts water to 100 parts stucco by weight to make a pourable slurry.

Processing Tests

Material that was ground to 100% passing 100 mesh was heated in open crucibles to determine the temperatures and times at which steam is produced. Steam should be produced when the material has reached the temperature range of 116°C to 121°C. Once the temperature range is determined, the time required to evolve the water can be determined also. This can be done by measuring the time between when the material first starts to produce steam and when it stops.

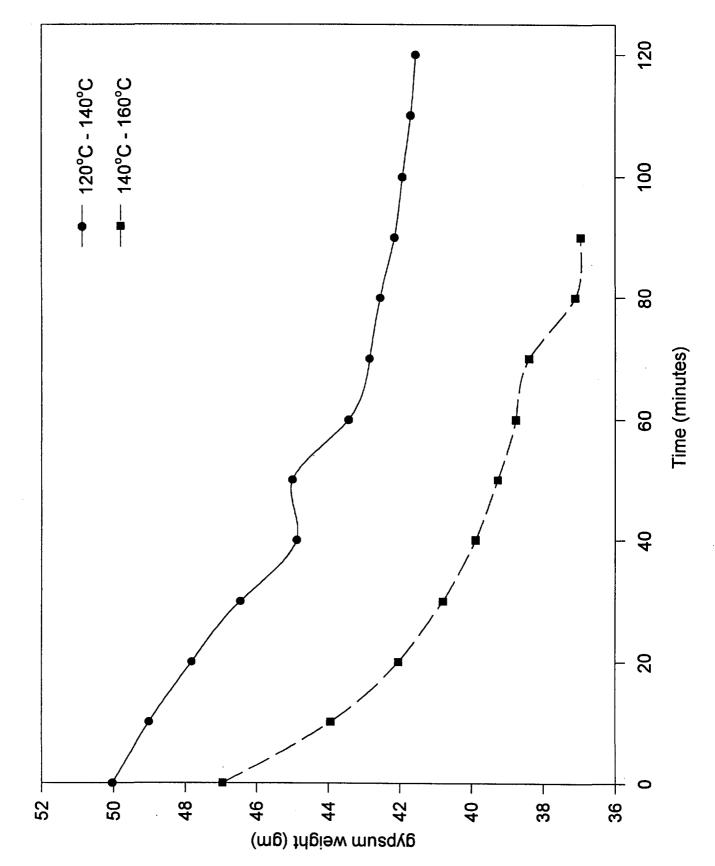
The gypsum used in the these trials was obtained from General Surveys and Exploration. Approximately 50 kg of gypsum was selected from the gypsum provided. Selection was made based upon the apparent anhydrate content of the pieces. Pieces containing primarily anhydrate were not included in the crushing and grinding process. After crushing and grinding the material was screened to100% passing 100 mesh before use.

To determine the temperature and time range in which water evolution begins, and to also determine the time required to fully evolve the water, a series of 5 sets of 2 samples each were placed in a temperature controlled, atmospheric pressure oven. Each 50 g sample was placed in a crucible and removed at the appropriate time. Removal times were chosen to start when steam was first produced. Confirmation of stream production was determined by observing a glass plate covering a hole in the top of the oven. Water droplets form on the cooler surface of the glass as soon as steam appears in the oven. Because the temperature would rise and fall when each sample was removed, the precise temperature at which steam was first produced was not determined. The oven temperature controller was set at 180°C to 200°C.

Steam production began after 20 minutes of heating. The droplets disappeared from the window at approximately 35 minutes. Samples removed after only 20 minutes of heating did not set properly when water was added after heating and cooling of the sample. This confirms that little or no conversion to hemihydrate had occured before 20 minutes. Samples removed after 35 minutes of heating set within 1 to 2½ minutes when water was added after heating and cooling of the powdered gypsum. The amount of water added also increased substantially as the heating time was increased. Table #1 shows the results of the tests performed.

Treatment Time	Temperature Range	Observations
20 min.	100-170°C	- water vapour @ 20 min.
40 min.	140 - 200°C	- water vapour stopped @ 35 min.
60 min.	160 - 200°C	- no vapour
90 min.	180 - 200°C	- no vapour
120 min.	180 - 200°C	- no vapour

To more precisely determine the processing times, two samples were heated at different temperatures for up to 120 minutes and weighed every 10 minutes during that time. Figure 1 shows the decrease in mass of the samples as they were heated. Based upon the total mass lost by each sample (8.485 g for Sample A, and 10.006 g for Sample B) significant amounts of water was evolved from the chemical structure. To produce hemihydrate 15.7% of the total mass should be lost. Sample A lost 16.9% of its mass, Sample B lost 21.3% of its mass. Mass losses greater than 15.7% are generally caused by the evaporation of water vapour in the material, before the loss of crystalline water occurs. In other words, the samples dried out in the oven before water was evolved from the dihydrate. Table 2 shows a breakdown of mass loss results accounting for loss of moisture in the sample.



GYPSUM TEST (weight loss)

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Table 2

	Sample A (120-140°C)	Sample B (140-160°C)
Starting Weight	50.035 g	46.955 g
Moisture loss (typical 5%)	2.502 g	2.348 g
Dry Weight	47.533 g	44.607 g
Hemihydrate weight	40.070 g	37.604 g
Actual final weight	41.550 g	36.951 g

Property Testing

Once a specific range of processing parameters is defined, some physical properties of the stucco can be examined. Of particular interest are the normal consistency (ie. water up-take in parts of water per 100 parts of stucco by weight) and the setting time.

Normal consistency can be determined by adding various amounts of water to a set of constant mass samples. Beta plaster usually requires 64 to 66% by weight of water to make a pourable slurry. Normal consistencies as calculated are shown in Table 3.

Table 3			
Treatment	Water to	Normal	Observations
Time	Form Slurry	Consistency	
20 min.	30 ml.	60	Not fully set in 24 hours
40 min.	40 ml.	80	 set time 1 minute noticeable heat produced during setting
60 min.	45 ml.	90	 set time 2 minutes noticeable heat produced during setting
90 min.	50 ml.	100	 set time 2.5 minutes noticeable heat produced during setting
120 min.	50 ml.	100	 set time 2.5 minutes noticeable heat produced during setting

The normal consistency values compare favourably for beta hemihydrate. Since the test is subjective in nature, (i.e. subjective judgment is required to determine when a slurry is formed) it is not unusual to have higher values reported for normal consistency. Beta hemihydrate usually requires >60% water by weight to form a slurry.

Setting times, however, are unusually quick. Beta hemihydrate generally takes more than 10 minutes to set. Since the numbers remain consistent throughout the program though, it is clear that the setting times must be a result of some additional component in the natural gypsum used. Small levels of certain impurities are known to produce rapid setting times. It would be advisable to perform elemental analysis of the gypsum used to determine the type of impurities contained. Fast setting times have considerable advantages for some types of applications such as drywall and other manufactured materials.

Conclusions

The Moose River gypsum tested in this program can be used to form beta hemihydrate using common and traditional methods and techniques. Evolution of water was shown to occur at a temperature greater than 100°C after 20 minutes of heating. Normal consistency was determined to be >60. These factors are normal for gypsum commonly used to form beta hemihydrate.

Unusually quick setting times were demonstrated for the hemihydrate produced. This factor might be caused by natural impurities in the gypsum, something which could on one hand be exploited as a production advantage in the manufacture of formed materials, while on the other hand could prove to be a limiting factor for usage. It will be critical to examine this aspect before further recommendations can be made with respect to possible products and formulations.

Beta hemihydrate is most suited for the manufacture of wallboard and the formulation of plasters for building markets. The two key factors for success in these applications are early strength development and cost. Beta hemihydrate is considerably less expensive to produce than the alpha form, which requires expensive autoclave equipment. The Moose River gypsum clearly appears to be suited for general, simple calcining to produce common beta hemihydrate. At very least this material is ideally suited for use as wallboard filler and building plaster applications which make up more than 80% of the use of gypsum in North America.

<u>Appendix A</u>

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Report from Mikro-Tek - attached

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GYPSUM TEST #1

- gypsum ground and screened to 100% < 100 mesh (approx. 50 kilos)
- heated 40 gm. for 3.5 hr. @ 180 200°C
- added water to hydrated gypsum and non-heated gypsum (control) to form uniform slurry

HYDRATED SAMPLE (40 gm)	GYPSUM CONTROL (40 gm)
10 ml. H ₂ O	15 ml. H ₂ O
 set in 4 min heat produced edges brown/burnt colour 	 hardened in 30 min. still not set in 24 hrs.

GYPSUM TEST #2

- gypsum ground and screened to 100% < 100 mesh
- 50 gm samples x 2 reps per treatment = 100 g/treatment
- temperature set @ 180 200°C

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- samples were heated for specificied times
- after heating and cooling water was added to form consistent slurry

TREATMENT TIME	TEMPERATURE RANGE	H ₂ O TO FORM SLURRY	OBSERVATIONS
20 min	100 - 170°C	30 ml.	- water vapour @20 min - not fully set in 24 hrs.
40 min	140 - 200℃	40 ml.	 vapour stopped @ 35 min set time 1 min. heat produced
60 min	160 - 200⁰C	45 ml.	- set time 2 min - heat produced
90 min	180 - 200°C	50 ml.	- set time 2.5 min. - heat produced
120 min	180 - 200°C	50 ml.	- set time 2.5 min. - heat produced

GYPSUM TEST SUMMARY #3 (A & B)

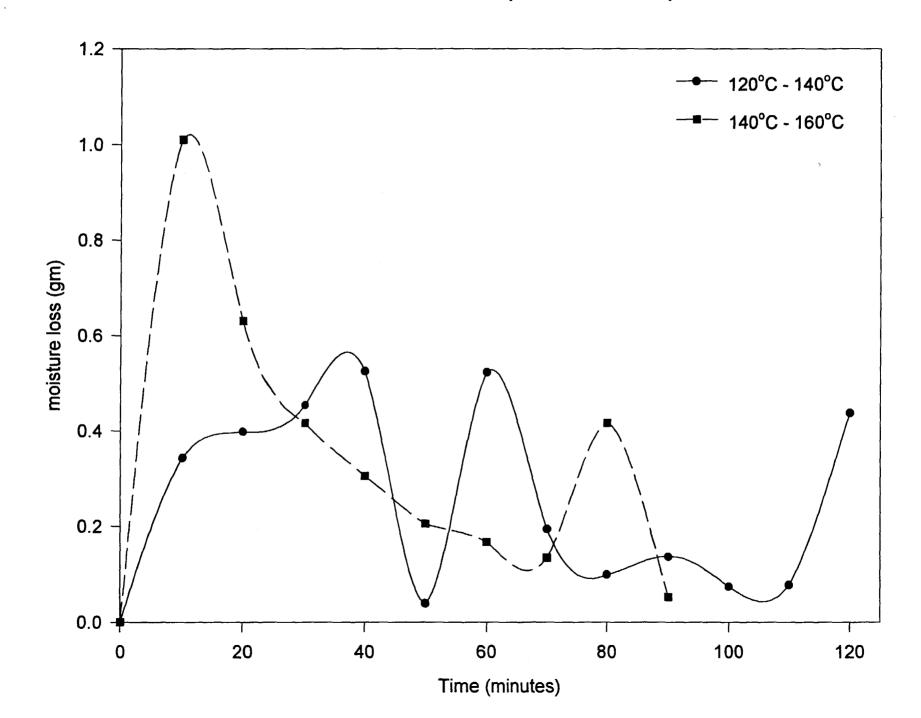
- ground and screened gypsum to 100% <100 mesh
- starting weight of gypsum : A 50.035 g (total of 3 reps) B - 46.957 g (total of 3 reps)
- samples were heated at set temperature and weights taken every 10 minutes
- set temperature for Test A 120°C 140°C Test B - 140°C - 160°C

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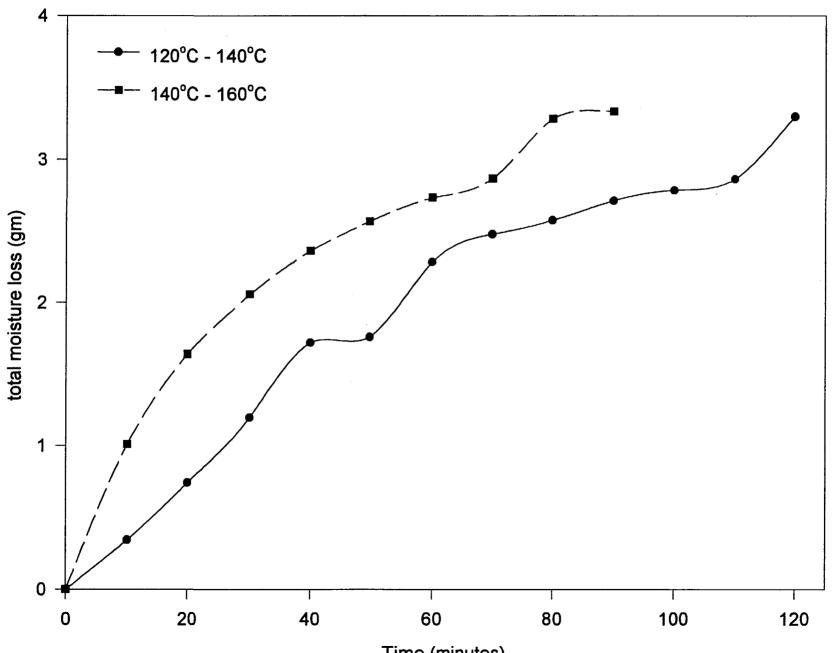
TEST B

TIME (min.)	Avg. Wt. (gm)	H ₂ O lost (gm)	Avg. Wt. (gm)	H ₂ O lost (gm)
0	50.034	-	46.956	-
10	49.005	0.343	43.926	1.010
20	47.811	0.398	42.036	0.630
30	46.449	0.454	40.788	0.416
40	44.874	0.525	39.873	0.305
50	44.994	0.039	39.258	0.205
60	43.425	0.523	38.757	0.167
70	42.843	0.194	38.355	0.134
80	42.546	0.099	37.107	0.416
90	42.138	0.136	36.951	0.052
100	41.916	0.074		
110	41.685	0.077		
120	41.550	0.437		

GYPSUM TEST (moisture loss)



GYPSUM TEST (total moisture loss)



Time (minutes)

Appendix B

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Direct Cost:

Wages: Consultants		
New Worlds Engineering, 14 hours x \$95 per hour		\$1330.00
-	GST	\$ 93.10
		\$1423.10
Laboratory Services:		
Mikro-Tek		
Crushing a	nd Grinding - 50 kg	\$ 400.00
Screening '	100% passing 100 mesh	\$ 250.00
Hydration to	ests, setting time tests, processing tests	<u>\$4800.00</u>
·	Sub-total	\$5450.00
	GST	\$ 381.50
		\$5831.50

Total Cost

\$7254.60

 New Worlds Engineering, 3234665 Canada Inc.

 68 Castlewood Ave. , Timmins, ON, Canada, P4R 1L5

 Phone: 705 268 0948, 267 5363
 Fax: 705 264 1161

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New Worlds Engineering

This report is submitted by New Worlds Engineering, 3234665 Canada Inc.

To: James Bay Lowlands Gypsum Development Group

Date: December 6, 1996.

Author: Grant R. Cool, Ph.D., P.Eng.



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MIKRO-TEK

DIVISION OF M. KBAN RESOURCES INC.

MICROBIAL TECHNOLOGIES FOR THE ENVIRONMENT MINING FORESTRY AGRICULTURE

FAX TRANSMISSION FORM

FAX TO:	MNDM	FAX #:	705-670-5863
ATTENTION:	Steve Benetcau	DATE:	February 24/97
FAX FROM:	Mark Kenn	PAGES:	1 (including cover sheet)

RE: Gypsum laboratory work for claims in Canfield and Carrol townships Transaction Numbers W9660.00596&7

The analytical work for the report was conducted at:

Mikro-Tek 36 Emerald St P.O. Box 2120 Thumins Ontario P4N 7X8

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Contact: Mark Kean Phone: 705-268-3536 Fax: 705-268-7411

36 EMBRALD ST., P.O. BOX 2120, TIMMINS, ONTARIO PAN 7X8 • PHONE 705-268-3536 • FAX 705-268-7411

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I, <u>MARK KEAN</u> <u>AND</u> <u>KEVIN</u> <u>Cool</u>, do hereby certify that I have personal knowledge of the facts set (Print Name) forth in this Declaration of Assessment Work having caused the work to be performed or witnessed the same during or after its completion and, to the best of my knowledge, the annexed report is true.

Signature of Recorded Holder Or Agent		Dec. 9/96
Agent's Address	Telephone Number	Fax Number
	above	above

5. Work to be recorded and distributed. Work can only be assigned to claims that are contiguous (adjoining) to the mining land where work was performed, at the time work was performed. A map showing the contiguous link must accompany this form.

wo mir col	ork wa ning li lumn	Claim Number. Or if as done on other eligible land, show in this the location number d on the claim map.	Number of Claim Units. For other mining land, list hectares.	Value of work performed on this claim or other mining land.	Value of work applied to this claim.	Value of work assigned to other mining claims.	Bank. Value of work to be distributed at a future date.
- 1	eg :	TB 7827	2 16 ha	\$26, 825	N/A	\$24,000	\$2,825
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; ; ;			Column Totals	7254	6800	5654	4-54-

I, $\underline{Kev_{IN}}$ \underline{Cool} + Mark Kean, do hereby certify that the above work credits are eligible under (Print Full Name) subsection 7 (1) of the Assessment Work Regulation 6/96 for assignment to contiguous claims or for application to the claim where the work was done.

Signature of Recorded Holder or Agent Authorized in Vritina 96 lão Cor

6. Instructions for cutting back credits that are not approved.

Some of the credits claimed in this declaration may be cut back. Please check (\checkmark) in the boxes below to show how you wish to prioritize the deletion of credits:

1. Credits are to be cut back from the Bank first, followed by option 2 or 3 or 4 as indicated.

Och 10 2. Credits are to be cut back starting with the claims listed last, working backwards; or

3. Credits are to be cut back equally over all claims listed in this declaration; or

4. Credits are to be cut back as prioritized on the attached appendix or as follows (describe):

Note: If you have not indicated how your credits are to be deleted, credits will be cut back from the Bank first, followed by option number 2 if necessary.

		•	
For Office Use	MECEIVED		
Received Stamp		Deemed Approved Date 9/97	Date Notification Sent
	1105 C CR	Date Approved	Total Value of Credit Approved
	PORCUPINE MINING DIVISION	Approved for Recording by Mining Reporter (Signature)

ATTN: DARLENE

Ministry of Northern Development

🕅 Ontario

Statement of Costs for Assessment Credit

GEN. SURVEYS & EXPL.

section Number (office 19660.00630

PAGE 01

FROM: KEVIN GOOL, C.

CLIENT # 120650

Personal information collected on this form is obtained under the authority of subsection 6(1) of the Assessment Work Regulation 8/96. Under section 8 of the Mining Act, the information is a public record. This information will be used to review the assessment work and correspond with the mining land holder. Questions about this collection should be directed to the Chief Mining Recorder, Ministry of Northern Development and Mines, 6th Floor, 933 Ramsey Lake Road, Sudbury, Ontario, PSE 685.

	• •				204	k 5
Work Type		of Work of work, list the number metree of drilling, kilo- mber of samples, etc.		t Per Unit of work	Total C	ost
INDUSTRIAL MINERAL						· · ·
TESTING	(CONSULTINGT)	IA-HRS	§	35	1330	
	G.S.T.			93	93	
CANSHING, GRIMANG, SCABENING	(LAB SERVICES)	ST HRS	<u> </u>	5.41	5450	
+ HYDEATION TESTS	G.S.T.			<u> 81</u>	381	
			·			
Associated Costs (e.g. supplie	a, mobilization and	demobilization).				-
	<u> </u>				<u>.</u>	
			<u> </u>			···
						
Trans	portation Costs					
			<u> </u>			
				RECE	IVED	
Ened	and Lodeline Costs					
	and Lodging Costs	· · · · · · · · · · · · · · · · · · ·		FEB 1	2 1997	
	· · · · · · · · · · · · · · · · · · ·			MINING LANE	S BRANCH	
		Total Value of		mant Work		
		I DIGI YAINY OF	1996 2 3	INGIL WORK	7254	.00

Calculations of Filing Discounts:

1. Work filed within two years of performance is claimed at 100% of the above Total Value of Assessment Work. 2. If work is filed after two years and up to five years after performance, it can only be claimed at 50% of the Total

Value of Assessment Work. If this situation applies to your claims, use the calculation below:

TOTAL VALUE OF ASSESSMENT WORK × 0.50 = Total \$ value of worked claimed.

Note:

- Work older than 5 years is not eligible for credit.

- A recorded holder may be required to verify expenditures claimed in this statement of costs within 45 days of a request for verification and/or correction/clarification. If verification and/or correction/clarification is not made, the Minister may reject all or part of the assessment work submitted.

Certification verifying costs:

I, <u>KEVIN</u> Cool, , do hereby certify, that the amounts shown are as accurate as may (please print full name) reasonably be determined and the costs were incurred while conducting assessment work on the lands indicated on

the accompanying Declaration of Work form as

the accompanying Declaration of Work form as

to make this certification.

Signature

Date

r, sgent, or state company position with signing authority)

Ministry of Ministère du Ontario Northern Development Développement du Nord et des Mines and Mines Geoscience Assessment Office February 27, 1997 933 Ramsey Lake Road 6th Floor Sudbury, Ontario P3E 685 Gary White Mining Recorder Telephone: (705) 670-5853 60 Wilson Avenue, 1st Floor Fax: 670-5863 (705) Timmins, ON P4N 2S7 Dear Sir or Madam: Submission Number: 2,17045 Status Approval Subject: Transaction Number(s): W9660.00630

We have reviewed your Assessment Work submission with the above noted Transaction Number(s). The attached summary page(s) indicate the results of the review. WE RECOMMEND YOU READ THIS SUMMARY FOR THE DETAILS PERTAINING TO YOUR ASSESSMENT WORK.

If the status for a transaction is a 45 Day Notice, the summary will outline the reasons for the notice, and any steps you can take to remedy deficiencies. The 90-day deemed approval provision, subsection 6(7) of the Assessment Work Regulation, will no longer be in effect for assessment work which has received a 45 Day Notice.

Please note any revisions must be submitted in DUPLICATE to the Geoscience Assessment Office, by the response date on the summary.

NOTE: This correspondence may affect the status of your mining lands. Please contact the Mining Recorder to determine the available options and the status of your claims.

If you have any questions regarding this correspondence, please contact Steve Beneteau by e-mail at beneteau_s@torv05.ndm.gov.on.ca or by telephone at (705) 670-5855.

Yours sincerely,

ncodit.

ORIGINAL SIGNED BY Ron C. Gashinski Senior Manager, Mining Lands Section Mines and Minerals Division

Correspondence ID: 10599 Copy for: Assessment Library

Work Report Assessment Results

Date Correspondence Sent: February 27, 1997			Assessor: Steve Beneteau		
Transaction Number	First Claim Number	Township(s) / Area(s)	Status	Approval Date	
W9660.00630	1190295	CARROLL, CANFIELD	Approval	February 24, 1997	
Section: 18 Other INDUS					
Correspondence	to:		Recorded Holde	r(s) and/or Agent(s):	
Mining Recorder Timmins, ON			Kevin Cool T IMMI NS, ONTARIO)	
Resident Geologist Timmins, ON			KEVIN SCOTT COOL TIMMINS, Ontario	L	
Assessment Files Sudbury, ON	Library				

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