

JUNE 9, 1978

FIXATION OF ARSENICAL WASTES

at

Giant Yellowknife Mines
Yellowknife, Northwest Territories
Canada

-- BY --

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*(Formerly Known As:
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-- FOR --

FISHERIES AND ENVIRONMENT CANADA
ENVIRONMENTAL PROTECTION
WATER POLLUTION CONTROL DIRECTORATE
MINING AND METALLURGICAL PROGRAMS

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<u>TABLE OF CONTENTS</u>	<u>PAGE</u>
LIST OF TABLES	iii
LIST OF FIGURES	iv
ACKNOWLEDGEMENT	v
ABSTRACT	vi
1. INTRODUCTION	1
2. INITIAL PROJECT DEVELOPMENT	3
3. LABORATORY WORK	8
a) Sample Solidification	8
b) Original Sample Analysis	8
c) Sample Leaching and Analysis	11
d) Freeze-Thaw Test and Leaching Dates	16
e) Lab Data Analysis	16
4. OBSERVATIONS AND CONCLUSIONS	19
5. RECOMMENDATIONS	20
6. APPENDICES	
A. PROJECT PROPOSAL	22
B. SOLIDIFICATION PATENT	23
C. PRELIMINARY U OF T REPORT	24
D. METHODS OF SAMPLE ANALYSIS	25
7. BIBLIOGRAPHY	26

LIST OF TABLES

<u>TABLE</u>		<u>PAGE</u>
2-1	SAMPLES REQUESTED FROM GIANT MINES LTD., YELLOWKNIFE, N.W.T.	4
3-1	COMPOSITION OF SOLIDIFICATION FORMULATIONS FOR ARSENIC FIXATION	9
3-2	COMPOSITION OF BARREN SLURRY AND CALCINED RESIDUE FILTER CAKE	10
3-3	LEACH TESTS ON SAMPLE 85-02 (BARREN SLURRY PLUS SILT)	12
3-4	LEACH TESTS ON SAMPLE 86-01 (COMBINATION OF BOTH WASTES AND NO SILT)	13
3-5	LEACH TESTS ON SAMPLE 86-02 (COMBINATION OF BOTH WASTES PLUS SILT)	14
3-6	LEACH TEST DATA ON CALCINED RESIDUE FILTER CAKE - AS RECEIVED	15
3-7	LEACH TEST DATA ON FREEZE-THAW SAMPLES (5-CYCLES)	17
3-8	CALCULATED PERCENTAGES OF AVAILABLE ARSENIC PICKED UP BY THE LEACHING SOLUTION DURING THE LEACH TEST	18

LIST OF FIGURES

Page(iv)

Figure

Page

2-1 Milling Flowsheet to be used for
DKL Sampling Program.

6

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Erik Seraphim,	Noranda Group
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Neil Cook,	Geochemist, CWT
Giant Yellowknife Staff	
L. Price,	Falconbridge, Toronto

Many others could be included, but it is felt that the major portion of the work was accomplished by those listed above.

ABSTRACT

The Report discusses receiving wastes from Giant Yellowknife Mines containing arsenic, laboratory work involved in solidifying the arsenic waste with the objective of tying up the arsenic so that it will not leach out and pose a pollution problem. While the work was not conclusive and successful, it indicates that the solidification process could be an answer to the arsenic problem.

Solidification is a method whereby materials are tied up or compounded into silicate compounds to form stone-like materials analogous to the forms the elements are naturally found in the earth.

1. INTRODUCTION

This report evolved as a result of an unsolicited proposal (a copy of which can be found in Appendix A) directed to the Environmental Protection Service arm of Environment Canada.

The unsolicited proposal from David Krofchak Limited* was prompted by three factors. The first being the fact that a reasonably successful study of a similar nature, had been carried out on another mining operation. Secondly, the recent flurry of publicity in the news media about the problems to do with arsenic in the environment in the town of Yellowknife focused attention on the element arsenic. Thirdly, very recent work by DKL specifically directed at arsenic appeared to be very promising. For example, in Appendix C, can be found the report titled "Preliminary Report on the Technical Assessment of a Solidification Process for Treating Industrial Liquid Wastes". This paper was developed by the Department of Metallurgy and Materials Science at the University of Toronto. Liquid samples had been doped with arsenic, along with other heavy metals for the independent evaluation of the solidification process. The result was encouraging with respect to arsenic. Confidential work on some arsenic bearing streams from two Ontario Gold mines proved extremely encouraging. Further work on arsenic fixation is being carried out with J.B.F. Scientific Ltd., who are under contract to the U.S. Environmental Protection Agency to evaluate arsenic fixation systems. Results from this study are still not available at present.

* The David Krofchak Limited company name has been changed to Canadian Waste Technology Incorporated.

1. INTRODUCTION (CONT.)

The emphasis of this work on arsenic is mainly because of the recent interest in arsenic as a pollutant of concern. This is witnessed by observing the bibliography. None of the references dealing with arsenic as a pollutant were published prior to 1969 (Ref. #1, 2,10,11,12,13,14,15,16). While some of the references deal with items other than arsenic, the emphasis is usually on arsenic.

The purpose of this report was basically to use the tested approach to problem solution that had been developed over the years by DKL. This method is elaborated on in detail in Appendix A of this report. Unfortunately, due to problems and situations out of the control of DKL, the methodology as developed was not applied. Instead, a somewhat limited look was taken at two samples sent down from the Giant Mine in Yellowknife, N.W.T.

This report deals only with fixation of the two samples received from Giant Mines Limited.

2. INITIAL PROJECT DEVELOPMENT

Early on in the project initiation, it was decided at a staff meeting that the program as outlined in the proposal was not going to achieve the maximum in benefits. The decision was made on the basis that some time would be necessary for the staff members involved to become familiar with the gold mining operations at the Giant Mine in Yellowknife.

This time would be also used to request and receive some samples from the Giant Mine. The samples would be worked on in the lab and any preliminary data produced could only help at the time of visiting Giant.

With the foregoing in mind, a literature search was conducted with some of the more pertinent material dealing with the Giant operation included in the bibliography (Ref. #2,3,4,5,6,7,8,9) of this report. Two valuable references dealing with arsenic chemistry are also included (Ref. #1,10). Numerous notes and other private correspondence, although on hand, are not detailed due to questions of confidentiality.

A request for samples was quickly formulated (Table 2-1). Table 2-1 is keyed to Figure 2-1, a flow-sheet of the Giant Mill operation. The total Giant operation was well bracketed by the requested samples. Data acquired from these samples would have been invaluable to our staff visiting the Giant Mine.

However, as matters turned out, the communications situation between Giant Mine, Environment Canada, and DKL personnel was such that much time was wasted. Also, because of the same situation at this time, a visit was out of the question.

TABLE 2-1 SAMPLES REQUESTED FROM GIANT MINES LTD.
YELLOWKNIFE, N.W.T.

Sample Code	Sample Location Name	Sample Volume Required	Sampling Comments	pH When Sample Taken	Date & Time Sample Taken
A	Mine Discharge Water	5 gal. (1x5 gal.)*	Sample should include solids if any.		
B-1	Raw Tailings (Total Gangue)	10 gal. (2x5 gal.)	Sample should be taken prior to desliming or any other treatment. See attached flowsheet.		
B-2	Tailings Slime	10 gal. (2x5 gal.)	Sample should be taken immediately after desliming operation, prior to any other treatment. See flowsheet attached.		
D-1	Calcine Wash No. 1	5 gal. (1x5 gal.)	Sample should include solids if any. See flowsheet attached.		
D-2	Calcine Wash No. 2	5 gal.	Sample should include solids if any. See flowsheet attached.		
E-2	Dust Leach	5 gal. (1x5 gal.)	Sample should include solids if any. See flowsheet attached.		

TABLE 2-1 (CONTINUED)

Sample Code	Sample Location Name	Sample Volume Required	Sampling Comments	pH When Sample Taken	Date & Time Sample Taken
E-1	Carbon Plant Barren	5 gal. (1x5 gal.)*	Sample should include solids if any. See flowsheet attached.		
F	Calcine Residue	10 gal. (2x5 gal.)	See flow sheet attached.		
G	Barren Solution Bleed-off	5 gal. (1x5 gal.)	Sample should include solids if any. See flowsheet attached.		
H	As Treatment Sludge	5 gal. (1x5 gal.)	See flowsheet attached.		
J	Baghouse Dust	25 pounds (1x5 gal.)	See flowsheet attached. Please send release form for shipment of arsenic waste.		
K	Tailings Discharge	10 gal. (2x5 gal.)	Sample should be a blend of all streams entering the tailings impoundment area.		

* Note: The 5 gallon sample pails requested should be lined with an acid resistant lining, perhaps epoxy or polyethylene.

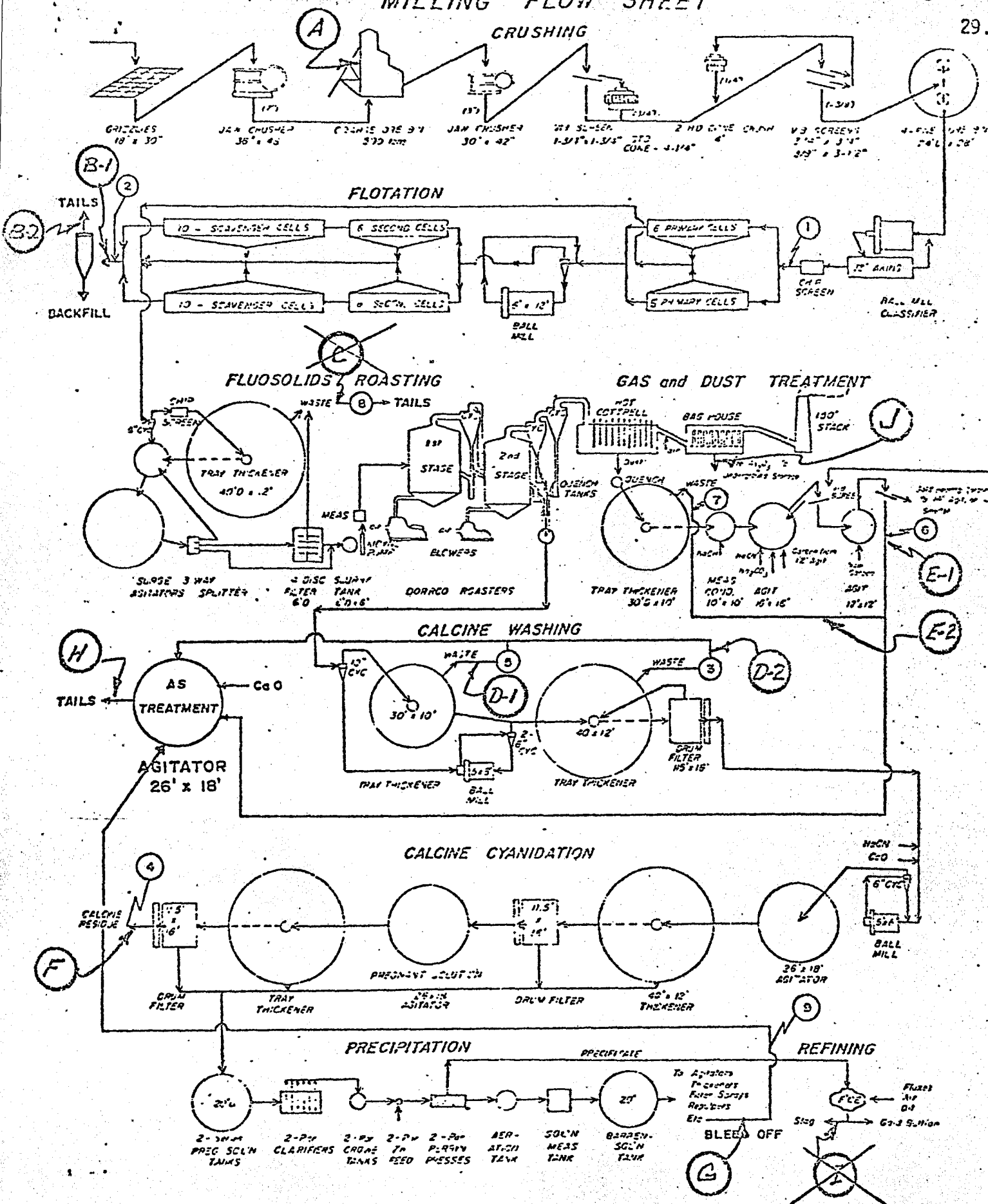


Figure 2-1: Milling flowsheet at Giant Yellowknife Mines, as of May 1969 and modified in 1972, after the Canadian Mining Journal 1969 published by National Business Publication Ltd Gardenvale, Québec.

(K) - TAILINGS DISCHARGE AT END OF PIPE IN TAILINGS POND.

2. INITIAL PROJECT DEVELOPMENT (CONT.)

One sample of dust barren slurry and one sample of Calcine residue filter cake were finally received late in February by the DKL laboratories. These two samples were sent after a general understanding of the project was achieved between parties.

DKL staff decided that the best approach to evaluating the two samples received was by means of a standard laboratory fixation. This is written up in the next section on sample Solidification.

3. LABORATORY WORK

a) SAMPLE SOLIDIFICATION

The initial fixation done on a new sample in the laboratory is usually tailored very closely to the original process as patented. (See appendix 8) This procedure is usually not deviated from unless something more specific is know about the sample in question.

The samples from the Giant Mine fell into the vague area of the unknown. They were handled by the standard process. The formulation data is shown in Table 3-1.

b) ORIGINAL SAMPLE ANALYSIS

The original samples were sent out for an ultimate analysis. (See Appendix D). These were sent out at the same time as the leachate samples. The analysis results are tabulated in Table 3-2.

Of most interest are the rather unexpected high levels of arsenic in the two samples. Nowhere in the literature, published or otherwise, is there any hint that arsenic levels would be in the percentage range. This factor may have created some of the problems later encountered.

Secondly, it is noted that the levels of SiO_2 in the samples are high. With this information we can see that additional SiO_2 in the form of silt is really unnecessary as there is sufficient SiO_2 in the samples themselves. In any case, the additional SiO_2 (Silt) put into the solidification formulation would not normally adversely affect the samples. It should only cause a dilution of the reacting chemicals.

44

TABLE 3-1 COMPOSITION OF SOLIDIFICATION
FORMULATIONS FOR ARSENIC
FIXATION

SAMPLE NUMBER	SAMPLE COMPOSITION IN %		
	85-02	86-01	86-02
CALCINE RESIDUE FILTER CAKE		46 %	44 %
BARREN SLURRY (38.8 SOLIDS)	35.5%	46 %	44 %
SILT	57.2%		4.3%
ACIDIC IRON SULPHATE SOLUTION	3.6%	3.5%	3.4%
LIME (AS $\text{Ca}(\text{OH})_2$)	3.7%	4.5%	4.3%
FINAL pH	12+	12+	12+

TABLE 3-2 COMPOSITION OF BARREN SLURRY
AND CALCINED RESIDUE FILTER
CAKE.

	CALCINED RESIDUE FILTER CAKE	DUST TREATMENT BARREN SLURRY (38.8% SOLIDS)	
		SOLIDS	LIQUID
As	2.40%	3.76%	0.173%
Ag, ppm	6.3	30.8	2.21
Al	3.5-%	7.12%	0.38 ppm
Be, ppm	0.02	0.80	N.D.
Ca	2.56%	3.66%	559 ppm
Co, ppm	360	273	3.00
Cy, ppm	308	654	606
Cr, ppm	60	195	0.175
Fe	27.5 %	18.7 %	20.9 ppm
K	1.10%	1.83%	43.2 ppm
Mg	1.53%	2.70%	208 ppm
Mn, ppm	464	822	0.0134
Na, ppm	1800	6100	1670
Ni, ppm	447	396	30.6
Pb, ppm	1300	2840	0.4
Sr, ppm	26.5	41.2	0.356
Ti, ppm	2890	4110	0.071
V, ppm	145	259	0.037
Zn, ppm	2280	2080	0.18
Zr, ppm	62	94	N.D.
SiO ₂	29.4 %	29.9 %	----
SO ₄ ⁼ , mg/l	----	----	7800
Cl ⁻ , mg/l	----	----	91.7

N.D. - Not Detected

3. LABORATORY WORK (CONT.)

c) SAMPLE LEACHING AND ANALYSIS :

The leachate tests used to determine the chemical stability of the solidified samples were as follows:

1. One hundred and twenty-five (125) grams of the solidified sample was pulverized and packed into a 40 x 600 mm chromatography column containing 1 inch of glass wool at the bottom.

2. The leaching solution (de-ionized water) was then added at the top of the column.

3. The leachate was collected in 500ml portions, an amount equivalent to a 40 cm high column of water passing through the sample. It took 24 hours for each 500 ml of leaching solution to pass through the leaching column.

4. Each leachate sample was stored at 4°C until analysed by Barringer Research Ltd. See Appendix D for methods of sample analysis.

The results are tabulated in Tables 3-3, 3-4, and 3-5.

Table 3-6 shows the leachate composition when the untreated Calcined Residue filter cake was leach tested for comparison purposes. The filter cake was leached to provide a check on the effect and/or efficiency of the solidification process.

TABLE 3-3 LEACH TESTS ON SAMPLE 85-02
(BARREN SLURRY ALONE)

	STANDARD LEACH TEST			
	0-40 cm	40-80 cm	80-120 cm	120-160 cm
	CENTIMETERS OF LEACHATE WATER			
pH	11	10	9	10
As	1.88	34.6	31.3	28.9
Ag	0.198	0.007	0.011	0.008
Al	0.18	0.42	1.05	1.83
Be	ND	ND	ND	ND
Ca	504	183	104	100
Co	0.04	0.03	0.05	0.06
Cu	27.4	2.74	2.57	1.69
Cr	ND	ND	0.012	0.010
Fe	2.70	0.355	0.201	0.230
K	9.4	7.1	10.1	7.6
Mg	ND	ND	ND	ND
Mn	ND	ND	ND	ND
Na	173	5	7	6
Ni	2.77	ND	ND	ND
P	4.6	ND	ND	ND
Pb	0.2	ND	ND	ND
Si	1.81	4.02	3.82	3.31
Sr	0.44	0.119	0.0741	0.0741
Ti	ND	ND	0.002	0.002
V	0.003	0.006	0.020	0.012
Zn	ND	ND	ND	ND
Zr	ND	0.003	0.008	0.007
SO ₄ =	2440	756	415	415
Cl-	50.0	41.7	52.8	75.0

ALL ELEMENT CONCENTRATIONS IN mg/l

ND = NOT DETECTIBLE

TABLE 3-4 LEACH TESTS ON SAMPLE 86-01
(COMBINATION OF BOTH WASTES AND NO SILT)

	STANDARD LEACH TEST			
	0-40 cm	40-80 cm	80-120 cm	120-160 cm
	CENTIMETERS OF LEACHATE WATER			
pH	9	10	8.5	8.5
As	6.68	10.0	13.1	11.2
Ag	0.20	0.012	0.010	ND
Al	0.62	1.99	3.18	3.68
Be	ND	ND	ND	ND
Ca	160	96.8	66.0	66.9
Co	0.06	0.05	0.06	0.06
Cu	24.7	2.04	1.16	0.63
Cr	0.010	0.011	0.011	0.012
Fe	2.28	1.57	1.53	1.13
K	14.2	9.3	7.4	7.4
Mg	ND	ND	ND	ND
Mn	ND	ND	ND	ND
Na	106	9	6	7
Ni	1.99	ND	ND	ND
P	3.7	2.5	ND	0.9
Pb	ND	0.2	ND	0.3
Si	2.64	2.61	2.37	2.53
Sr	0.196	0.0889	0.0704	0.0741
Ti	0.002	0.002	0.001	0.001
V	0.006	0.007	0.007	0.007
Zn	ND	ND	ND	ND
Zr	0.005	0.007	0.008	0.007
SO ₄ =	2440	585	537	366
Cl-	86.1	103	88.9	83.3

ALL ELEMENT CONCENTRATIONS IN mg/l

ND = NOT DETECTIBLE

TABLE 3-5 LEACH TESTS ON SAMPLE 86-02
(COMBINATION OF BOTH WASTES, PLUS SILT)

	STANDARD LEACH TEST			
	0-40 cm	40-80 cm	80-120 cm	120-160 cm
	CENTIMETERS OF LEACHATE WATER			
pH	8.5	8.5	8.5	8.5
As	12.7	13.1	10.6	12.2
Ag	0.031	0.020	0.020	0.023
Al	1.02	2.73	3.30	3.59
Be	ND	ND	ND	ND
Ca	279	71.1	64.4	64.2
Co	0.20	0.13	0.12	0.12
Cu	45.2	2.18	0.729	0.289
Cr	0.008	0.016	0.022	0.017
Fe	5.72	1.74	1.27	0.856
K	18.5	11.2	9.9	10.8
Mg	0.096	ND	ND	ND
Mn	ND	ND	ND	ND
Na	225	12	9	9
Ni	4.66	ND	ND	ND
P	0.9	8.9	0.7	2.6
Pb	0.2	ND	0.3	0.2
Si	3.37	3.02	2.76	2.41
Sr	0.274	0.0815	0.0704	0.0741
Ti	0.002	0.003	0.003	0.005
V	0.010	0.022	0.022	0.022
Zn	ND	ND	ND	ND
Zr	0.002	0.010	0.014	0.016
SO ₄ =	3900	561	479	537
Cl-	55.6	66.7	69.4	69.4

ALL ELEMENT CONCENTRATIONS IN mg/l

ND = NOT DETECTIBLE

TABLE 3-6 LEACH TEST ON UNTREATED CALCINED
RESIDUE FILTER CAKE

STANDARD LEACH TEST
0-40 CM.
LEACHATE WATER

pH	
As	122
Ag	0.289
Al	0.24
Ba	ND
Be	ND
Ca	94.0
Cd	ND
Co	0.40
Cr	0.029
Cu	0.729
Fe	8.57
K	15.6
Mg	4.14
Mn	ND
Na	45.0
Ni	0.33
P	1.8
Pb	0.3
Si	4.72
Sr	0.037
Ti	0.003
V	0.027
Zn	ND
Zr	0.014
SO ₄ ⁼	2440
Cl ⁻	97.2

ALL ELEMENT CONCENTRATIONS IN mg/l

ND = NOT DETECTIBLE

3. LABORATORY WORK (CONT.)

d) FREEZE-THAW TEST AND LEACHING DATA

Each of the three solidification formulations was passed through a quick freeze-thaw test procedure to establish fixation stability under winter/summer conditions. The test simply put a portion of each formulation through five cycles of freezing and thawing. Theoretically, this should represent at least five years of this form of stress.

Immediately after this cycling was completed the three formulations were subjected to their own leachate tests. The testing conducted was identical to that carried out in the previous section of this report.

The results are tabulated in Table 3-7.

e) LAB DATA ANALYSIS

The raw data compiled in the preceeding sections was evaluated to establish the overall efficiency of arsenic fixation. Calculations were made of the arsenic leached as a percentage of total arsenic in the sample being leached. Table 3-8 compiles the results of the calculations.

Of most interest is the first general impression. The arsenic in the untreated residue cake leached out at a level one magnitude higher than the arsenic in the fixed samples. In actuality only the leachate samples of the 86-01 and the 86-02 formulations showed this low leaching rate. The percentage of arsenic leaching out of sample 85-02 almost as high as was leaching out of the untreated residue. Unfortunately, the calcined residue filter cake was not solidified by itself. It would have been interesting to compare it with sample 85-02 which was Barren Slurry alone (along with a major portion of silt). Such a test might have uncovered some characteristic of the Barren Slurry which may have made the arsenic in it more leachable.

TABLE 3-7: LEACH TEST DATA ON FREEZE-THAW
SAMPLES (5 CYCLES)

(40cm Leachate Water used in each case)

	85-02	86-01	86-02
pH	7	7	7
As	54.7	4.25	11.2
Ag	5.25	0.348	0.049
Al	0.94	1.03	0.91
Be	ND	ND	ND
Ca	651	599	603
Co	0.19	0.31	0.28
Cu	28.6	41.6	21.0
Cr	0.13	0.27	0.24
Fe	1.92	7.31	4.51
K	16.3	24.9	19.9
Mg	3.69	2.28	4.34
Mn	ND	ND	ND
Na	144	228	137
Ni	ND	4.83	2.52
P	2.06	8.3	3.8
Pb	3.9	ND	0.3
Si	ND	2.55	1.69
Sr	2.63	0.504	0.344
Ti	0.615	0.005	0.007
V	0.003	0.025	0.025
Zr	ND	0.009	0.015
SO ₄ ⁼	7320	7320	6340
Cl ⁻	108	131	131

NOTE: EACH 40cm OF LEACHATE WATER REPRESENTS
APPROXIMATELY 500ml OF LEACHING SOLUTION

ND = Not Detectable

ALL ELEMENT CONCENTRATIONS IN mg/l

TABLE 3-8: CALCULATED PERCENTAGES OF AVAILABLE ARSENIC PICKED UP BY THE LEACHING SOLUTION DURING THE LEACH TEST

SAMPLE	NOTES	LEACHATE INTERVAL	% As PICK UP BY THE LEACH SOLUTION*
85-02	Barren	0-40 cm	0.094
"	Slurry	40-80 cm	1.73
"	Only	80-120cm	1.57
"		120-160cm	1.45
86-01	Combination	0-40 cm	0.099
"	of	40-80 cm	0.15
"	wastes with	80-120cm	0.19
"	no silt	120-160cm	0.16
86-02	Combination	0-40 cm	0.20
"	of wastes	40-80 cm	0.21
"	plus silt	80-120cm	0.17
"		120-160cm	0.19
CALCINED RESIDUE FILTER CAKE	Raw waste untreated	0-40 cm	2.03

* Based on dry sample weight.

4. OBSERVATIONS AND CONCLUSIONS

a) At this point it should be noted that all of the analytical data in the preceeding section was received at one time. All of the Solidification work, the freeze-thaw testing, and leaching were completed prior to any of the analytical work pertaining to the wastes, the formulations and the leachate samples. In other words, the solidification, the leaching, and the freeze-thaw tests were done "in the blind".

b) Based on the original sample analysis, a surprise came with the amount of arsenic available in the delivered samples. As noted earlier in this paper, the level of arsenic encountered in the delivered samples was very much higher than expected from the literature and other confidential data.

c) The amount of Si as available in the raw samples was a pleasant surprise. Because of the level of Si available, secondary sources of Si (such as silt) would not be necessary for future solidification of these materials.

d) The most unpleasant surprise came with the discovery that the solidified samples were unsatisfactory. The levels of arsenic leaching were on the high side, based on other work DKL had done with similar forms of arsenic. From previous work, arsenic leachate levels could be expected to improve by a factor of 1000 compared to the leachate of the untreated waste. In this case, the improvement was only by a factor of 10, from 2.0% for an unfixed sample to 0.2% for a fixed one.

e) Looking at the leachate levels of the other heavy metals, it is seen that copper (Cu) is leaching out at a relatively high level. Normally, copper

4. OBSERVATIONS AND CONCLUSIONS (CONT.)

is a metal that is tied up almost completely when solidification is successful. The fact that copper is leaching out of the fixed samples is indicative of extremely poor solidification.

f) The solidification was poor simply because nothing was known of the original composition of the samples. Had the arsenic level been known to be in the low percent range instead of ppm as assumed, a more rigorous treatment would have been carried out.

The explanation for the poor performance of the fixation reported on in this study is quite simple. The arsenic/ferrous-ferric sulphate and arsenic/lime reactions (Ref. #1) consumed too much of the reagents. What reagent was left was inadequate for promotion of the necessary silicate reactions.

5. RECOMMENDATIONS

Obviously the whole project did not proceed as planned, and as a result, the goals set out in the original proposal were not met.

However, considering the roadblocks and confusion met in the course of this project, it is a wonder that this report was written.

Despite the problems, it is felt that some good work has been done on arsenic fixation. At least, the work is indicative of possible future successes in this area. We are extremely optimistic on this score.

Our main and only recommendation in this report is quite simple. We feel that the original proposal should be reactivated and renegotiated. Most of the original confusion has been eliminated. Every one is more understanding of the concept of the proposal and the manner in which we would proceed with it. We feel that the

5. RECOMMENDATIONS (CONT.)

only possible recommendation that could be made at this time is to allow us to start and proceed with the proposal as originally developed, under terms to be renegotiated.

APPENDIX 'A'

-- ORIGINAL PROPOSAL --



**ENVIRONMENT
CANADA**

**Environmental Protection Service
Ottawa, Ontario
K1A 0H3**

MINE-MILL WASTE STREAMS

TREATMENT AND DISPOSAL BY SOLIDIFICATION

"A proposal for an engineering study to evaluate the economic and practical feasibility of using proprietary solidification techniques for the treatment and disposal of toxic Mine-Mill wastes and sludges at Giant Yellowknife Mines."

**Prepared by:
David Krofchak Limited
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Cambridge, Ontario
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Proposal Reference No. P-360

Date: February 1977

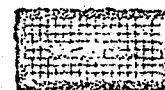
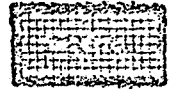


TABLE OF CONTENTS

Section	Page
1. Executive Summary	1
2. Introduction	2
3. General Information on Uses and Benefits of Solidification	3 & 4
4. Proposed Scope of Work	5 & 6
5. Engineering Study Schedule	7
6. Engineering Study Cost	8
7. Company Background and Capability	9 - 13

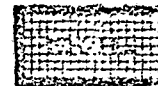


1. EXECUTIVE SUMMARY

This proposal to the Environmental Protection Service Branch of Environment Canada is for an engineering study to evaluate the economic and practical feasibility of using proprietary solidification techniques for the treatment and disposal of toxic Mine-Mill wastes and sludges at Giant Yellowknife Mines Ltd., Yellowknife, N.W.T.

This proposal shows:

- A new method of treating toxic inorganic mine-mill wastes in which the end product is an innocuous solid landfill material.
- It is possible to develop a solidification procedure at a minimal cost for treatment of mine-mill waste streams at Giant Yellowknife Mines.
- Toxic elements in the leachate from solidified material can be eliminated or otherwise substantially cut back, with specific reference to arsenic, lead, iron, etc.
- The solidification process, as applied to Giant Yellowknife Mines, is the subject of Patents granted or applied for in Canada and other foreign countries.
- The time required for completion of the study will be approximately eight weeks, at the end of which time a comprehensive evaluation report will be presented.
- The evaluation report will contain firm quotations for the implementation of the study recommendations on either a licensing or a lump sum purchase basis.
- The firm cost for the proposed engineering study is Eleven Thousand Dollars (\$11,000.00).



4. PROPOSED SCOPE OF WORK

a. Field Work

- Visit to Giant Yellowknife Mine
- Discuss and investigate all waste flows that are pertinent
- Discussions with Environment Canada and Giant Yellowknife Mine personnel to pinpoint all parameters of the waste streams under consideration
- Survey of local area for possible materials that would aid solidification
- Accumulation of a sufficient volume of samples for the laboratory work

b. Laboratory Work

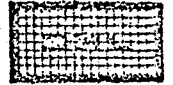
- Evaluation of the best solidification procedure, based on samples of materials on hand
- Preparation of several different formulations for the purpose of testing
- Analytical work on solidified formulations to establish critical parameters such as;
 1. Leachate metal levels
 2. Suitability of solidified material as landfill
 3. Effect of freeze-thaw cycling on leachate metal levels
 4. Evaluation of the most economical formulation based on acceptable leachate levels.

c. Economic and Engineering Appraisal and Recommendations

- An economic appraisal will be conducted for the waste treatment recommended in this study case. Such an appraisal will include both capital and annual cost.
- System design parameters will be used to size and cost process units of different capacities.
- The capital cost per unit flow will be presented in a graph form as a function of plant size.
- Chemicals and dosages, as well as power and labour, will be appraised for cost and presented in a graph form relative to the volume of treated waste.

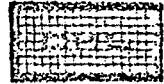
d. Implementation of Recommendations

- Firm price will be developed for the engineering and supply of a system to put the recommendations into practice.
- Alternate proposals for either licensing or purchasing outright the solidification technology pertinent to the recommendations will be presented.



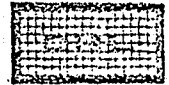
e. Final Report

- All the data, recommendations, engineering and economic evaluations along with comprehensive implementation costs will be developed into a final report.
- Also included will be equipment or process flow sheets as deemed necessary for a clearer understanding of the recommendations.
- The final report will be presented to Environment Canada personnel and other involved parties for its assimilation prior to a discussion meeting on it.



5. ENGINEERING STUDY SCHEDULE

	<u>Time Required</u>
a. Field Work	1 week
b. Laboratory Work	4 weeks
c. Economic and Engineering Appraisal and Recommendations	1 week
d. Implementation of Recommendations	1 week
e. Preparation of final report	1 week
<u>Total Time Required for Engineering Study</u>	<u>8 weeks</u>



6. ENGINEERING STUDY COST

For the development of an engineering study to evaluate the economic and practical feasibility of using proprietary solidification techniques for the treatment and disposal of toxic mine-mill wastes and sludges at Giant Yellowknife Mines, we are pleased to quote the following firm, lump sum price:

ELEVEN THOUSAND DOLLARS - - - - - \$11,000.00

APPENDIX 'D'

METHODS OF SAMPLE ANALYSIS

APPENDIX 'D'

ANALYSIS OF SAMPLES

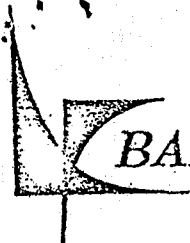
All analysis was done by Barringer Research Ltd.
(See accompanying literature)

They analysed the leachate samples for arsenic, sulphate and chloride, as well as all of the elements detectable with their Multielement Radio Frequency Coupled Plasma Emission Spectrometer (RFICP). For many elements the RFICP technologically supercedes atomic absorption techniques.

A hydrofluoric-perchloric acid extraction was done on the solid samples with the extract analysed by RFICP.

Sulphate and chloride concentrations were analysed using an ion chromatograph (Dionics).

Arsenic concentrations were determined by the silver diethyldithiocarbamate colorimetric method.



BARRINGER RESEARCH NEWS

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LOW COST MULTIELEMENT ANALYSIS WITH ATOMIC ABSORPTION PRECISION AND DETECTABILITY

SUMMARY

Barringer Research Limited has introduced a new multielement instrumental technique, which is capable of rapid low cost analysis. Samples can be analysed simultaneously for their major and trace element concentrations with typically the same precision and accuracy normally obtained by atomic absorption spectrophotometry. The sample is presented to the instrument in solution form which allows normal extraction techniques to be used and gives better precisions because sample inhomogeneity is reduced.

BACKGROUND AND DESCRIPTION

Analytical techniques are usually judged by several criteria, the most important of which are: precision, accuracy, sensitivity (or detectability), rapidity and cost of analysis. Over the past 25 years most major improvements in analytical performance have been achieved as a result of the introduction of new analytical instrumentation. For example, during the last decade trace metal analysis has become considerably cheaper, in terms of inflation adjusted dollars. There has also been considerable improvements in the precision and accuracy of the analytical data. Most of these improvements came as a consequence of the introduction of a single analytical technique: Atomic Absorption Spectrophotometry (AAS). In the mid 1960's Barringer Research was one of the pioneers of the application of this technique to geochemical and other analytical determinations. Since then AAS has grown in stature to a point where it is used very extensively for trace metal analysis.

During the past five years there has been a gradual increase in interest and use of multielement analysis. The environmentalist is no longer only concerned with mercury, the agricultural scientist now recognises that many elements affect land productivity, many different wear metals are now monitored in lubricants, to give but a few examples.

However, the potential user of multielement analysis is often in a quandary as to which analytical technique he should apply to his particular problem. Should he use a combination of atomic absorption spectrophotometry, colorimetry, and fluorimetry to achieve good precisions and detectabilities and incur the high attendant costs of this approach to multielement analysis; or should he sacrifice analytical performance, such as precision, sensitivity, and accuracy and use Emission or X-ray Spectroscopy to minimise his analytical costs (typically \$25.00 for 20 elements). This decision has always been difficult to make, some people have taken one route and some others the other. Unfortunately, all too often, the decision is not made and as a result, multielement analysis is discarded in favour of the more classical single, double or triple element approach.

Above a brief historical review has shown how there is a need for inexpensive, precise and sensitive, multielement analysis and how the introduction of a single instrument, such as an atomic absorption spectrometer, is capable of changing analytical performance. With these two factors in mind, Barringer Research has been looking for a solution for the last two years. During the summer of 1975 we started work on a new instrumental technique that provides such a solution; a Multielement Radio Frequency Induction Coupled Plasma Emission Spectrometer (RFICP), which realises inexpensive, sensitive, precise and accurate multielement analysis. This new technique will probably have a similar impact over the next decade as AAS did over the last. This instrument is capable of simultaneously measuring 32 elements with the same precisions and sensitivities that are currently obtained with AAS. For some elements such as the rare earths and refractory elements (U, Mo, W, etc.) its sensitivity is vastly superior to AAS and colorimetric techniques.

In addition to these advantages in analytical performance the technique is extremely cost effective, typical analytical costs for 32 elements are \$17.00 per sample. In May of 1975 we took delivery of a second instrument which also has a 32 element capability. Additionally, vast improvements have been made by the manufacturers in the instrumentation which has brought the sensitivities of the technique to a stage where generally its performance is superior to that obtained by atomic absorption.

However, many similarities still exist between this new technique and AAS especially when their instrumental parameters are considered. Like AAS, RFICP relies on a liquid presentation of a sample to the analytical system, this is an extremely important facet of the technique as it allows current total and selective extraction technology to be used without modification. The sample solution is nebulised into a radio frequency argon plasma, which has a physical temperature of 10,000° K (approximately 7,300° K hotter than a nitrous oxide/acetylene flame). The intense heat of the plasma core desolvates the solution aerosol, completely atomises the resulting salt particles and then excites and stimulates these atoms such that they emit their characteristic atomic spectra. The optical radiation emitted by the plasma is focussed onto the entrance slit of a polychromator, which simultaneously measures the emission intensity at several different wavelengths, each of which corresponds to a particular element. This intensity is proportional to the concentration of a particular element in the original sample solution.

Attached is a comparison of the detection limits for 32 elements obtained with this new technique to those obtained by atomic absorption spectrophotometry, for pure solutions. The actual detection limits that can be obtained for real samples, however, will depend on the extraction technique and the dilution used. We have analysed many different sample types on this instrument, including: waters (both natural and polluted), oils, soils, rocks, plants, urine, air particulates, Hi-Vol filters, feathers, sediments and sewage sludges.

The RFICP technique appears to be applicable to any analytical situation provided the sample can be put into solution.

In summary, this new multielement technique (RFICP) realises a trace and major element analysis for a total of 32 elements at a cost of \$17.00 per sample, when the sample is in a liquid. An additional \$4.00 per sample is charged for solid samples because of the additional time required for dissolution.

* - a 50 percent surcharge for batches less than 20 samples is charged.

COMPARISON OF DETECTION LIMITS

	RF ICP ¹ <u>ng/ml</u>	AAS ² <u>ng/ml</u>
Zn	2	2
Cd	2	2
Eu	15	500
Be	1	5
Se	60	100
Te	65	100
U	20	150,000
Co	10	6
Ti	0.9	100
Cr	6	4
Fe	12	5
Na	800	2
K	150	1
Ag	2	5
W	78	3,000
Au	3	50
Sn	45	50
V	0.8	20
Pb	88	25
Mo	6	40
P	110	*
Si	11	100
As	38	100
Al	2	40
Cu	0.4	3
Ni	10	5
Ca	<< 0.1	2
Mg	<< 0.1	1
B	0.3	*
Sr	< 1	10
Rb	< 1	6
Mn	1	3

* - These elements cannot be measured by Atomic Absorption

1 - These are the latest figures obtained on BRL's second instrument

2 - These figures taken from Perkin Elmer literature and are somewhat idealistic

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