

Flambeau Mining Company  
N4100 Highway 27  
Ladysmith, WI 54848  
(715) 532-6690  
FAX (715) 532-6885

October 30, 1996

Mr. Larry Lynch  
Wisconsin Department of Natural Resources  
Bureau of Waste Management  
101 South Webster Street  
P.O. Box 7921  
Madison, WI 53707-7921

**Kennecott  
Minerals**

Dear Mr. Lynch:

Re: Flambeau Open Pit Backfilling - Field And Laboratory Work Plan

As described in the report titled *Fall 1996 Backfilling Plan for Stockpiled Type II Material* submitted to the Wisconsin Department of Natural Resources earlier this month, Flambeau Mining Company (Flambeau) has completed a series of tests to support limited backfilling of material from the Type II stockpile to its open pit. Flambeau outlined in its report a framework for additional testing during the 1996-97 winter season to prepare for 1997 backfilling. A detailed work plan for this subsequent testing is presented in this letter.

## **1.0 INTRODUCTION**

### **1.1 Background**

The waste rock removed from the Flambeau open pit has been classified and stored in two categories. The categories have been defined on the basis of sulfur content, with Type I rock containing less than 1.0 % sulfur. This category includes the overburden that was stripped during the development of the open pit. All other material produced from the open pit was classified as Type II, and is stored in a lined facility.

As part of the 1989 Mine Permit Application (MPA), Flambeau planned to backfill stored rock and overburden into the open pit during reclamation. The Type II material will be backfilled in the bottom of the mined-out pit followed by the Type I material.

#### **1.1.1 Type II Material**

Placement of stockpiled Type II material in the open pit first, and subsequent saturation, will ensure that the Type II rock is located in an environment in which future oxidation and acid generation is controlled. Because the Type II rock will be below the future water table, oxygen entry to the rock will be limited, and anoxic conditions will develop. During the period of groundwater recovery, groundwater gradients will be directed towards the open pit so that very little release of water from the pit is expected. As the groundwater table recovers, the pore water of the waste rock will rapidly become anoxic, and increasing release of pore water to groundwater will occur.

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Site water is treated in a lime treatment system with sulfide polishing to remove metals prior to discharge. The resulting metal oxy-hydroxide solids, referred to as clarifier underflow (CUF) solids, have been co-disposed with the Type II material. The CUF solids are to be backfilled with the Type II material.

As stated in the MPA, the Type II rock will be amended with an alkali before backfilling to the open pit. The objective of the alkali amendment is to control dissolved metal concentrations at an acceptably low level in the waste rock pore water after flooding, i.e. after anoxic conditions have developed. The alkali amendment is required to neutralize:

- stored oxidation products and available acidity already contained in the Type II material; and,
- acidity and oxidation products that may be generated from future oxidation during the reflooding period.

The stored acidity and oxidation products are a result of the oxidation that has already occurred during the on-surface storage. Lime was originally specified as the alkali of choice in the 1989 MPA. At the time of the permit application, a preliminary estimate of the alkali requirement was made on the basis of partially oxidized Type II rock which was generated under short term laboratory testing conditions. However, limestone has replaced lime as the alkali of choice, and the alkali requirement of the oxidized material is to be determined directly using an alkali demand test, as described in the October 1996 report.

Co-disposal of the CUF solids may impact the geochemistry of the Type II material after saturation. First, the CUF solids may introduce excess alkalinity in the form of unreacted lime, and second, the sludges contain iron oxy-hydroxides that may become more soluble under anoxic conditions.

Recent testing completed by Flambeau in support of the planned Fall 1996 relocation program has shown that:

1. limestone can be used as an alternative to lime as an alkali source;
2. limestone blinding appears to be insignificant;
3. variability in alkali demand exists within the Type II material stockpile; and,
4. estimated long term pore water quality will be similar to that predicted in the MPA.

The investigation completed in support of the Fall 1996 relocation program was based on short term, rapid test methods. Short term testing was required in order that the small scale fall relocation program might be undertaken, and in that purpose satisfied the objectives of the investigation. As discussed in the October 1996 report, the short term investigation did not address all of the issues

related to the relocation of the Type II stockpile as a whole. As part of that investigation, some issues were identified that required further investigation, and/or confirmation. This work plan has been developed to address relevant outstanding issues, and includes both field and laboratory investigations that will be completed in support of the planned 1997 backfilling program.

#### 1.1.2 Type I Stockpile

During the construction of the Type I stockpile, rock has been placed in three distinct cells on top of a till layer, containing respectively sandstone, saprolite, and Type I rock. The Type I stockpile is characterized by a sulfur content of less than 1 %.

To date, a geochemical investigation of the Type I stockpile comprising eleven drillholes has been completed. Drilling was performed using a percussion drill rig with reverse air circulation to recover rock samples. The drillholes were located outside of the sandstone area, with one hole located in the saprolite area, as shown in Figure 1. The results showed that copper is present predominantly in the oxide form, while sulfur contents are typically less than 0.3%. There are, however, a few pockets of material that exhibit a sulfur content between 0.3% and 1%.

Since the Type I rock is relatively low in sulfur content, it is not anticipated to be net acid generating. However, ongoing oxidation of any sulfides may lead to the release of soluble metals, as indicated by the dissolved copper concentration observed for seep T1-1, location shown in Figure 1. The seepage quality varies according to the flow rate; low pH values and peak copper concentrations are associated with peak flow rates for the period March to October, 1996. While the average copper concentration is less than 10 mg/L, peak concentrations in excess of 50 mg/L associated with slightly acidic pH values have been observed. This suggests that there are probably some pockets of material that may require alkali amendment on relocation.

The copper concentration in this seep may be an isolated occurrence, in which case no marked effect is expected on the pore water quality after backfilling and saturation of the Type I material. However, should a significant portion of the Type I materials contain similar soluble constituents, it may be necessary to add alkalinity during the backfilling. Furthermore, the reactivity of the backfilled Type I material, i.e. oxygen consumption rate, will determine the rate of oxygen entry into the underlying Type II materials. If the Type I rock is highly reactive with oxygen, less oxygen will enter the underlying Type II rock, and its alkali amendment could potentially be reduced. However, should a portion of the Type I rock prove to be potentially acid generating, it will be necessary to amend that material to neutralize acidity generated during groundwater recovery.

The program proposed below, therefore, also includes tests to assess the reactivity of the Type I materials, and, to address the potential need for alkali amendment to neutralize stored acidity if required.

## 1.2 Work Plan Objectives

Both field and laboratory investigations will be undertaken. The objectives of the field investigation are to:

1. Provide additional data to assess the spatial variability of paste parameters (pH and conductivity) within the Type II stockpile;
2. Develop sampling and testing protocols to determine the limestone addition rate which during relocation provides the minimum required neutralization potential;
3. Provide necessary samples for continuing laboratory investigations; and,
4. Establish the requirements to implement a variable alkali application rate for future relocation;

The objectives of the laboratory investigation can be summarized as follows:

1. Demonstrate the effectiveness of limestone over lime to produce secondary mineral phases that will remain stable under anoxic conditions;
2. Determine the availability of the alkali under anticipated short- and long-term conditions in the backfilled pit;
3. Quantify the alkali addition rates to control metal release from stored oxidation products (in pore water) and meta-stable secondary mineral phases;
4. Evaluate the stability of the CUF solids under anticipated long term saturated (anoxic) conditions; and,
5. Assess field test methods for the control of alkali application rates.

A final objective is to investigate the reactivity of the Type I materials, and to assess the need for alkali addition, should this become necessary to control metal release during and after saturation.

## 2.0 FIELD INVESTIGATIONS

### 2.1 Type II Stockpile

Sampling of the Type II stockpile will be undertaken to reconfirm that the calculated limestone addition rate is adequate for the fall 1996 relocation. This will be accomplished by sampling first ahead of the relocation, and second, sampling the excavation face itself. In addition to this, the remainder of the stockpile will be sampled to provide additional information on the spatial variability and to provide the necessary samples to complete the proposed laboratory investigations. The field sampling and investigation programs are described below.

### 2.1.1 Material Characterization Ahead of Excavation

The Type II stockpile material to be relocated in 1996 will be sampled at a fixed grid of about 60 feet by 60 feet in plan on the plateau, to provide a total of about 26 samples. The slopes will also be sampled by extending the grid pattern laterally to provide an additional 12 samples, for a total of about 38 samples. The sampling locations are shown in Figure 2. The location of each test pit will be surveyed. Test pits will be excavated to a depth of about 15 feet, which is equal to the relocation lift height. A representative sample will be obtained for the entire depth of the test pit by scraping the face of one side of the test pit from bottom to top, at an even 'slice', to provide a bulk sample. The full depth of the test pit will be logged to describe significant variations in material type, coloration, size distribution, moisture content, etc. with depth.

A representative sub-sample will be obtained from each bulk sample, and will be taken from the <1/4 inch size fraction. The sub-sample will be tested under laboratory conditions to determine its alkali demand, as described in Section 3.1 below under the laboratory investigation program.

Paste pH and conductivity will be obtained in the field on each quarter of the bulk sample, to provide a total of four per bulk sample. Methods for obtaining field parameters are provided in Attachment A.

### 2.1.2 Material Characterization at the Excavation Face

Similar to sampling the test pits, face samples will be obtained, as excavation proceeds during the fall 1996 relocation, by scraping the entire length of the face with the backhoe to provide an even 'slice'. Care will be taken to prevent limestone contamination, or over representation of any portion of the excavation height in the sample. One sample will be taken on every second day of excavation.

The bulk sample will be placed on a clean surface (limestone free) or a tarpaulin, and will be characterized as described above for the test pit samples. A representative sample of the < 1/4 inch material will be removed and an alkali demand test will be completed, as described below. Field parameters (paste pH and conductivity) will be obtained as described in Section 2.1.1.

### 2.1.3 Additional Samples

An additional 30 test pits will be completed on a random grid (Figure 2) over the remainder of the Type II Stockpile. The test pits will be excavated as described above in Section 2.1.1. Bulk sampling and sub-sampling will be undertaken as described in Section 2.1.1 and the test pit will be logged. Field parameters will also be obtained for the bulk sample as described in Section 2.1.1

At random, representative samples of the < 3 inch size fraction will be taken from six test pits. Leach extraction tests will be completed on these samples to determine the immediately extractable oxidation product distribution, as described below in Section 3.1.4 for the laboratory investigation.

#### 2.1.4 CUF Solids

One representative sample of the CUF solids will be obtained from the deposition location currently in use, and one from a previous deposition location (Figure 2). Should it not be possible to identify a prior deposition location, then two samples will be taken from the current location. The pH and conductivity of the pore water will be obtained in the field, using the procedures described in Attachment A.

Characterization of the CUF solids will be completed as described in the laboratory investigation program described in Section 3.2 below.

### 2.2 Type I Stockpile

The objectives for sampling the Type I stockpile are to provide information on the current state of the contained materials, and the spatial variability of the field parameters and pore water quality within the stockpile. While the pockets of material which have higher than average sulfur grades, based on the drillhole investigation, are generally below the depth to which test pit excavation is possible, oxidation is expected to be more significant at the near surface. Test pitting and sampling will provide an indication of the amount of oxidation products present in the Type I material.

The sampling program for the Type I stockpile will comprise a total of 20 test pits located on a random grid to represent each of the different rock types. A total of 5 test pits will be excavated within the saprolite, 5 within the sandstone, and 10 within the remainder of the Type I stockpile.

Test pits will be excavated, logged and sampled as described in Section 2.1.1 above for the Type II stockpile sampling program. Test pit excavation will be to a depth of 10 feet. Field parameters (paste pH and conductivity) will be obtained as described in Attachment A. Leach extraction tests will be completed on the representative sub-samples of the less than 1/4 inch material, as described in the laboratory investigation program discussed in Section 3.4.

### 2.3 Backfilled Material

The objective for sampling the backfilled material is to confirm that the calculated rate of limestone addition for the fall 1996 relocation satisfies the minimum requirements, and that the limestone is effectively blended during the relocation process.

Sampling of the backfilled material will be undertaken for each two days of placement. Three test pits, evenly distributed over the material that has been placed over the two days, will be excavated to three feet in depth (i.e. equal to the placement lift height) by backhoe or similar means. The sample will be taken, and the test pits will be logged for material characteristics and variations as discussed in Section 2.1.1. The three <1/4 inch samples obtained will be mixed in equal proportions (by weight) to provide a single composite sample. A confirmation extraction test will be completed for a period of 72 hours on each sample, as described in the laboratory investigation, discussed in Section 3.3.

#### 2.4 Summary of Field Investigations

The field investigation program is summarized in Table 2.1.

**TABLE 2.1**  
**Summary of Field Investigation Program**

Material type	Number of Samples							
	Type II				CUF Solids	Type I Stockpile		
	Ahead of Relocation	Relocation Face	Remainder	Fall 1996 Backfill		Sandstone	Saprolite	Type I Rock
Test Pits	38	na	30	45	2	5	5	10
Depth (ft)	15	na	10	3	na	10	10	10
Samples <1/4 inch < 3 inch	38	15	30 6	15	2	5	5	10
Field Parameters								
Paste pH	152	60	120	60	2	20	20	40
Paste cond.	152	60	120	60	2	20	20	40

na - not applicable

### **3.0 LABORATORY INVESTIGATIONS**

#### **3.1 Type II Materials**

##### **3.1.1 Effect of Size Distribution**

The objective of the testing is to confirm the results from earlier testing which indicated that the majority of the stored oxidation products are associated with the < 1/4 inch size fraction, and that testing to determine alkali demand on this size fraction is conservative.

The six < 3 inch samples will be screened at 1/4 inch to provide < 1/4 inch, and 1/4 to 3 inch splits. Leach extraction tests, conducted at a liquid to solid ratio of 1:1 using distilled water, will be completed on each of the splits. The procedure for the leach extraction test is described in Attachment B.

##### **3.1.2 Alkali Demand**

The objective of the testing is to establish the correlation between field characteristics (paste pH and conductivity) and the alkali demand of the Type II material, for the potential implementation of a control program for the application of alkali during relocation at a variable rate. Lime is used to determine the alkali demand in this test because it reacts rapidly. Test results can be obtained within 24 hours, as has been shown in the October 1996 report. As also shown in that report, the alkali requirement obtained from the lime demand test can be easily converted to a limestone requirement.

Alkali demand testing will be undertaken on all the samples from the Type II Stockpile that have a paste pH equal to or lower than 6.5. The method is provided in Attachment B.

##### **3.1.3 Confirmation of Alkali Amendment Type**

The objective of the testing is to confirm the effectiveness of limestone versus lime as the alkali source for the neutralization of the stored acidity contained in the Type II materials. Limestone has been selected as the alkali of choice on the basis of its buffering capacity, and the stability of the secondary minerals phases that are formed, as discussed in the October 1996 report. The testing will be completed only to confirm this.

A total of 6 samples will be selected for confirmation testing of the alkali type. The samples will be selected to represent a low, intermediate and high alkali demand. Testing will be completed on the <1/4" size fraction, using the anoxic column test procedure described in Attachment B. A representative sample will be taken from the larger bulk sample, blended and then split in half (by



cone and quarter). The first split will be amended with lime at the rate indicated by the alkali demand test. The second split will be amended with limestone at the stoichiometric requirement, but corrected for the availability and moisture content of the limestone.

#### 3.1.4 Alkali Amendment Rate and Long-Term Pore Water Quality Confirmation

The objectives of the testing are first to confirm that the limestone demand calculated on the basis of the alkali demand test results, and appropriate correction factors, is accurate, and secondly that the long term pore water quality will approximate that predicted for the saturated backfill.

A total of 12 samples will be selected from the Type II material samples and tested using the anoxic column test procedure, provided in Attachment B. The limestone amendment will be calculated from the alkali demand, determined as before. The test will be completed on the < 1/4 inch size fraction. Limestone will be blended with the sample at the calculated rate prior to loading into the column.

### 3.2 Stability of CUF Solids Under Anoxic Conditions

The objective of the testing is to determine the stability of the CUF solids under anoxic conditions, such as those anticipated after saturation of the backfill.

Testing of the CUF solids will be undertaken in two stages. First, leach extraction tests will be completed at a solids to liquid ratio of 1:1, using distilled water. The first of two extraction tests will be completed at the natural pH, and, the second at a pH of about 6.5 using sulfuric acid to modify the leachate pH, recording the total amount of acid added to achieve a stable pH. The eluates will be analyzed for dissolved constituents. The acid consumption to 6.5 will be back-calculated from the amount of acid added. The acidification is completed to determine the stability of the CUF solids for a slight change in pH, and to determine its neutralization potential, should it be contacted with existing acidity. In the second stage, column tests will be completed on the two CUF solids samples, using the anoxic column test procedure provided in Attachment B.

### 3.3 Fall 1996 Backfilled Material

The objective of the testing is to demonstrate that the limestone amendment for the material relocated during Fall 1996 is sufficient to satisfy the minimum neutralization requirements, and that adequate mixing has been achieved during relocation.

Anoxic leach extraction tests will be completed on the composite samples taken from the placed materials. The extraction tests will be completed at a solids to liquid ratio of 1:1 using distilled water, as described in Attachment B, but using argon to maintain anoxic conditions during the extraction period. The eluate will be monitored for pH, redox and conductivity only. The relationship between pore water composition as a function of the pH will be known from the column testing, and therefore,

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analysis of dissolved constituents will not be completed on the eluates. Upon completion of the leachate extraction tests, an acid consumption test using sulfuric acid will be performed on the solids using the procedures described in Attachment B.

Three composite samples will be selected for longer term testing, using the anoxic column test procedure provided in Attachment B.

### **3.4 Type I Materials**

#### **3.4.1 Pore Water Quality**

The Type I material pore water quality will be established by completing leach extraction tests on the < 1/4 inch size fractions of the samples taken from the test pits. The leach extraction test procedure is provided in Attachment B. The leachate from the extraction will be analyzed for dissolved constituent concentrations.

#### **3.4.2 Reactivity**

Acid base account (ABA) testing will be completed on each of the samples taken from the Type I stockpile. The ABA will include a sulfur speciation, i.e. total sulfur and sulfate sulfur will be determined analytically, and the sulfide sulfur by difference.

### 3.5 Summary of the Laboratory Investigation

The laboratory investigation is summarized in Table 3.1.

**TABLE 3.1**  
**Summary of Laboratory Investigation Program**

Material Type	Number of Samples							
	Type II				CUF Solids	Type I Stockpile		
	Ahead of Relocation	Relocation Face	Remainder of Stockpile	Fall 1996 Backfill		Sandstone	Saprolite	Type I Rock
Alkali Demand	38	15	30	-	-	5	5	10
Anoxic Column - limestone	-	-	18	3	2	-	-	-
Anoxic Column - lime	-	-	6	-	-	-	-	-
Leach Extraction	-	-	12	-	-	5	5	10
Anoxic Extraction	-	-	-	15	-	-	-	-
Acid Consumption	-	-	-	15	2	-	-	-
ABA	-	-	-	-	-	5	5	10

### 4.0 DATA INTERPRETATION AND PRESENTATION

The work will be performed in accordance with the schedule shown in Figure 3.

Leach extraction results from the <1/4 inch, and 1/4 to 3 inch size fractions will be compared to reconfirm that the small size fraction can be used to conservatively represent the whole sample in further tests. Alkali demand results will be used to estimate limestone and lime requirements for subsequent tests. The alkali demand results will be compared with the field parameters. This will be done to establish if there is a correlation which can be used, as a simple predictive tool, to establish the alkali demand of the weathered rock. The spatial correlation in field parameters will then be determined. Simple statistical analyses are not valid for spatial correlation and therefore visual presentations, e.g. by color coded plots, will be used to assess the feasibility for variable alkali application rates. Also, a manual cross check of the field parameters and alkali demand with the test pit logs and field observations will be completed.

The results from the column tests performed on the test pit samples will be plotted. The trends will be evaluated to establish the effectiveness of limestone to control metal solubility initially, and in the long term. The results from the lime amended and limestone amended column tests will be compared directly, as appropriate, to provide an indication of the relative effectiveness of each alkali to control

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metal solubility. Some geochemical modeling will be undertaken using the MINTEQA2 model to identify solubility controlling phases.

The variability in the equilibration (extraction) test results performed on the backfilled material will be used to establish the effectiveness of alkali blending during relocation. A determination of whether suitable blending is achieved will be obtained from the combined results of the column tests to be completed on the backfilled material. The combined results will be used to establish future quality assurance sampling and testing requirements.

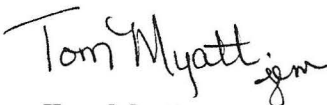
The acid generation potential will be compared with the acid consumption potential for the Type I samples. The net potential for acid generation will be determined by establishing the NP:AP ratios. The results from the extraction tests to be performed on the Type I materials will be used to quantify the immediately extractable oxidation products, and thus determine the need for alkali amendment to neutralize pore water and stored oxidation products.

A report will be prepared in which the results from the field and laboratory investigations will be summarized and discussed.

## 5.0 CLOSING COMMENTS

As you know, to avoid confusion once backfilling begins, we have already collected the 38 samples within the Type II stockpile Fall 1996 excavation area. Per the schedule shown in Figure 3, the remaining test pits in both the Type II and Type I stockpiles are to be collected during the week of October 28, 1996, with testing to commence immediately thereafter. Therefore, we would appreciate any comments on our work plan at your earliest convenience. I will contact you shortly to discuss our plan.

Sincerely,

A handwritten signature in cursive script that reads "Tom Myatt" followed by a small monogram "jm".

Tom Myatt  
Flambeau Mining Company

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cc: Al Christianson, City of Ladysmith  
Jeff Earnshaw, Flambeau  
Daryl Hockley, Steffen Robertson and Kirsten  
Jeane Hull, Kennecott Corp.  
Jim Hutchison, Foth & Van Dyke  
Ken Markart, Wisconsin Department of Natural Resources  
Jana E. Murphy, Flambeau  
Thure Osuldsen, Rusk County  
Tom Riegel, Town of Grant  
Jerry Sevick, Foth & Van Dyke  
Melvin Spencer, Rusk Co. Zoning

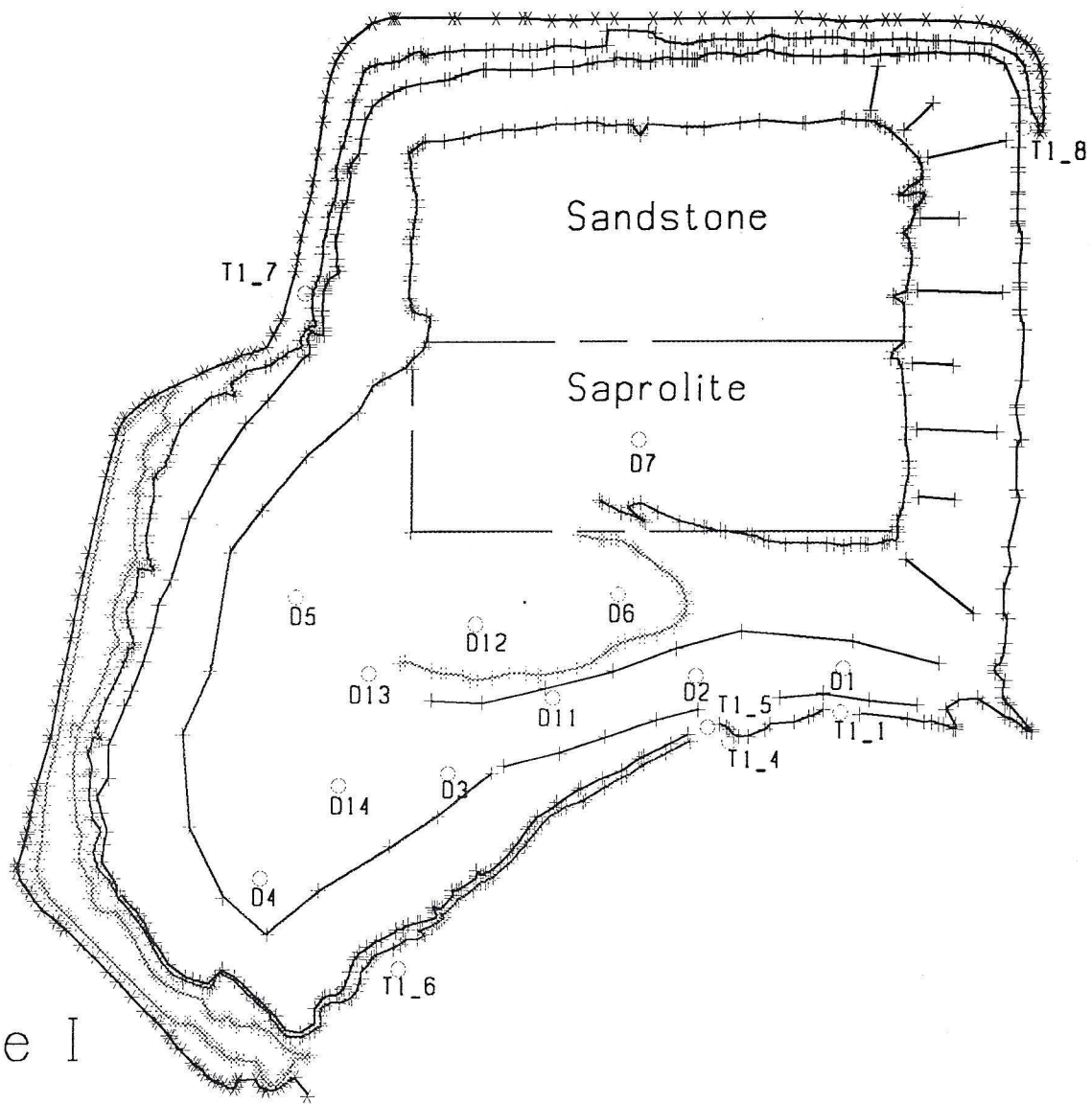


Figure I

Type I Stockpile: Drill Hole & Seep Locations

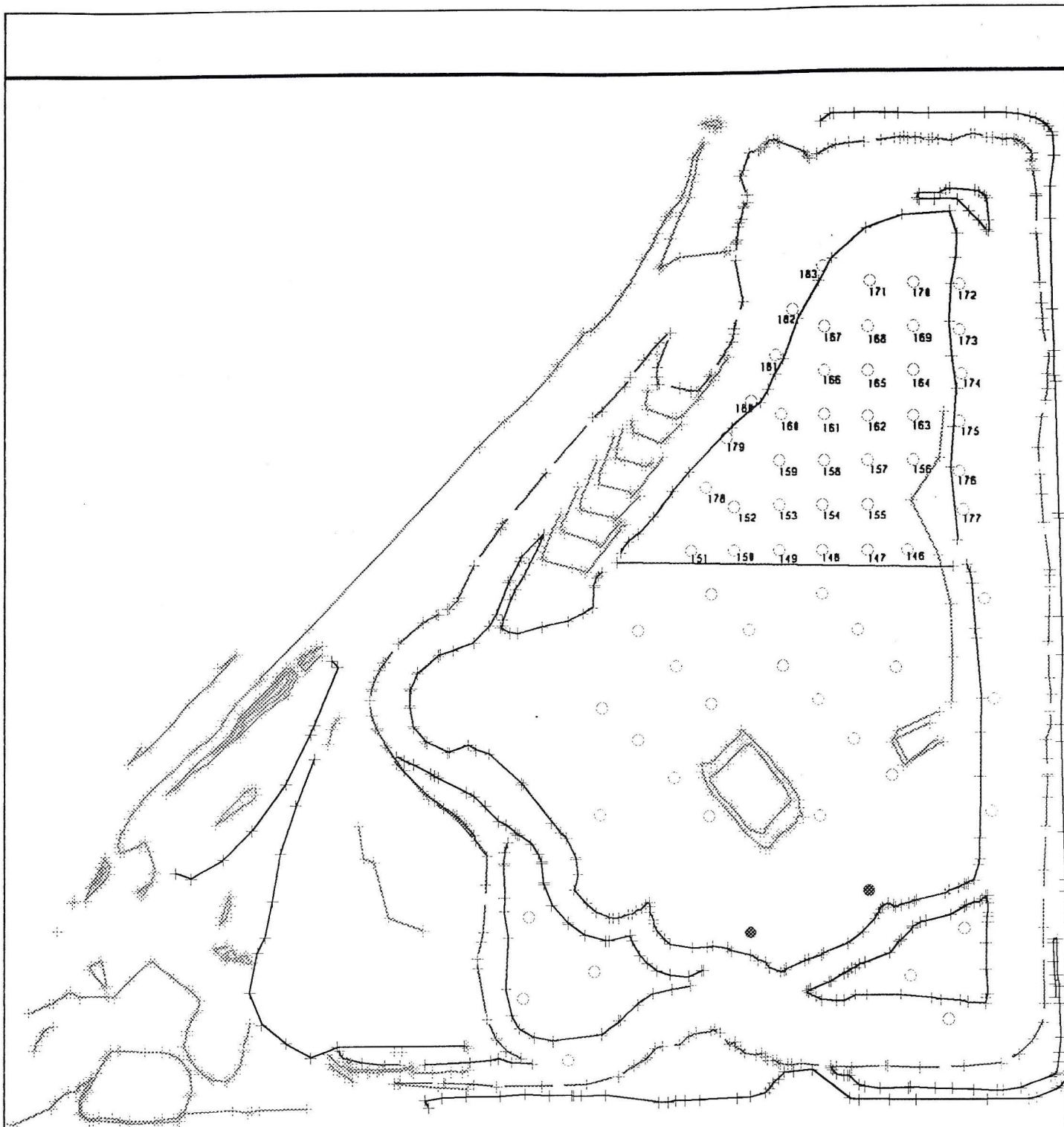
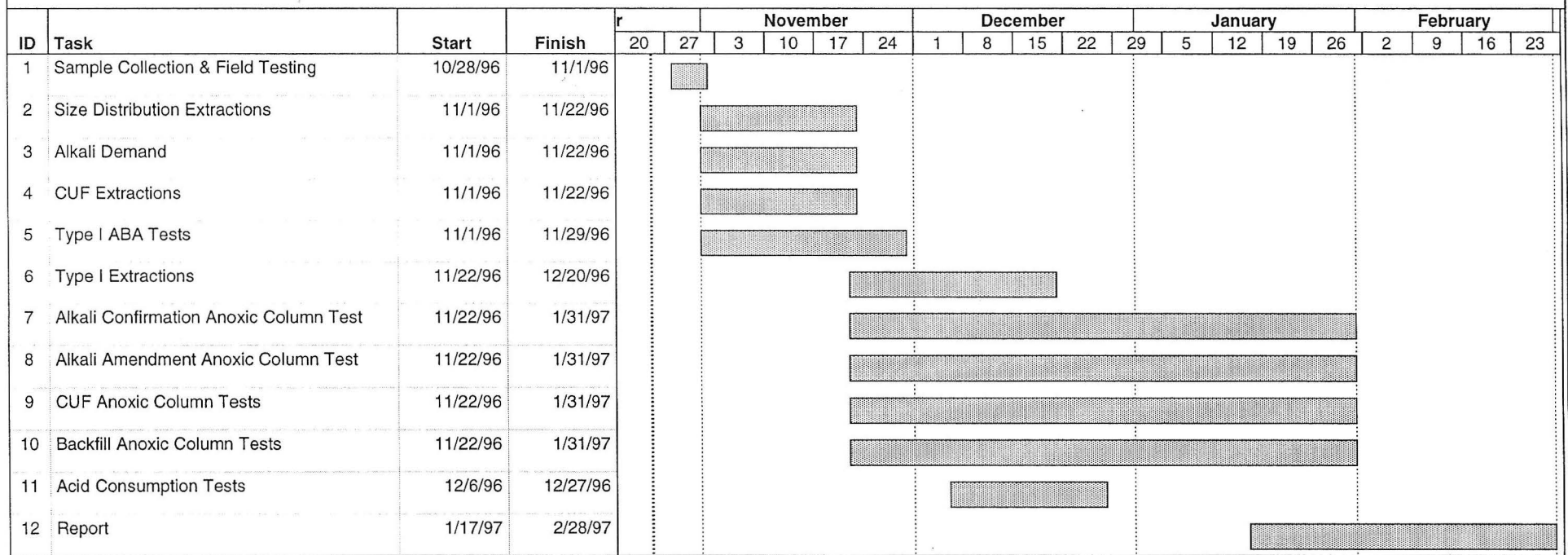


Figure 2

Type II Test Pits

- 146 Denotes Sample Number
- Additional Test Pit
- ◆ CUF Solids Test Pit

Figure 3  
Flambeau Mining Company  
Backfilling Field and Laboratory Work Plan




Project: Flambeau Mining  
Date: 10/25/96

Task 

Progress 

Milestone 

Summary 

Rolled Up Task 

Rolled Up Milestone 

Rolled Up Progress 



## **ATTACHMENT A FIELD METHODS**

### **A.1 FIELD SAMPLING AND TESTING**

#### **TEST PIT LOGGING**

1. Survey the location of the test pit
2. Log horizons in the test pit walls, noting in particular:
  - color / weathering
  - lithological composition
  - primary and secondary mineralization
  - particle size distribution
  - moisture content
3. Photograph the wall of the test pit

#### **FIELD SAMPLING**

A representative sub-sample of the bulk sample is obtained, and the obvious oversize is (i.e. > 3 inches) is discarded. The sample is then screened at 1/4 inch at the field moisture content. Agglomerated material in the oversize should be broken up to maximize recovery of the < 1/4 inch size fraction. The approximate size distribution of the bulk sample is estimated. Approximately 25 - 30 lb (12 to 14 kg) of the <1/4 inch sample is required to complete subsequent laboratory investigations described in Attachment B.

#### **FIELD TESTING**

Paste parameters are obtained, using the method described below for one sample from each quarter of the bulk sample. The tests may be performed either in the field, or if inclement weather conditions dictate, in the laboratory. To complete testing in the laboratory, samples of the < 1/4 inch material taken from each quarter of the bulk sample are to be prepared, bagged and marked individually.

## **A.2 PASTE pH AND CONDUCTIVITY**

### **EQUIPMENT AND REAGENTS**

1. Portable pH and conductivity meters
2. 2x Glass/plastic beakers (150 to 250 ml)
3. 1/4 inch hand sieve
4. Measuring cup or scoop (approximately 60 to 65 ml)
5. Distilled water
6. Spatula

### **METHOD**

1. A sample of approximately 1-2 kg is taken with a small shovel, discarding the obvious oversize (> 3 inch) material.
2. The sample is sieved through a 1/4 inch screen, and from the undersize material, a sample of 65 ml, using a scoop that can be scraped level to remove excess sample, is obtained.
3. The sample is placed in a 150 to 250 ml beaker, the scoop is rinsed and the residue is added to the beaker, using exactly 100 ml of water.
4. The prepared sample is mixed thoroughly using a spatula
5. The pH and conductivity of the supernatant is measured with calibrated field equipment.  
IMPORTANT : Do not contact the solids with the glass pH probe - it is fragile and may scratch. Ensure that the readings have stabilized before recording the values.

## ATTACHMENT B LABORATORY TESTING METHODS

### B1 ALKALI DEMAND

#### EQUIPMENT AND REAGENTS

1. 1.0 to 1.5 liter Erlenmeyer flasks or roll bottles
2. Distilled water
3. Hydrated lime ( $\text{Ca}(\text{OH})_2$ )
4. Sulfuric Acid
5. pH and conductivity meters
6. Rotary shaker or rollers

#### METHOD

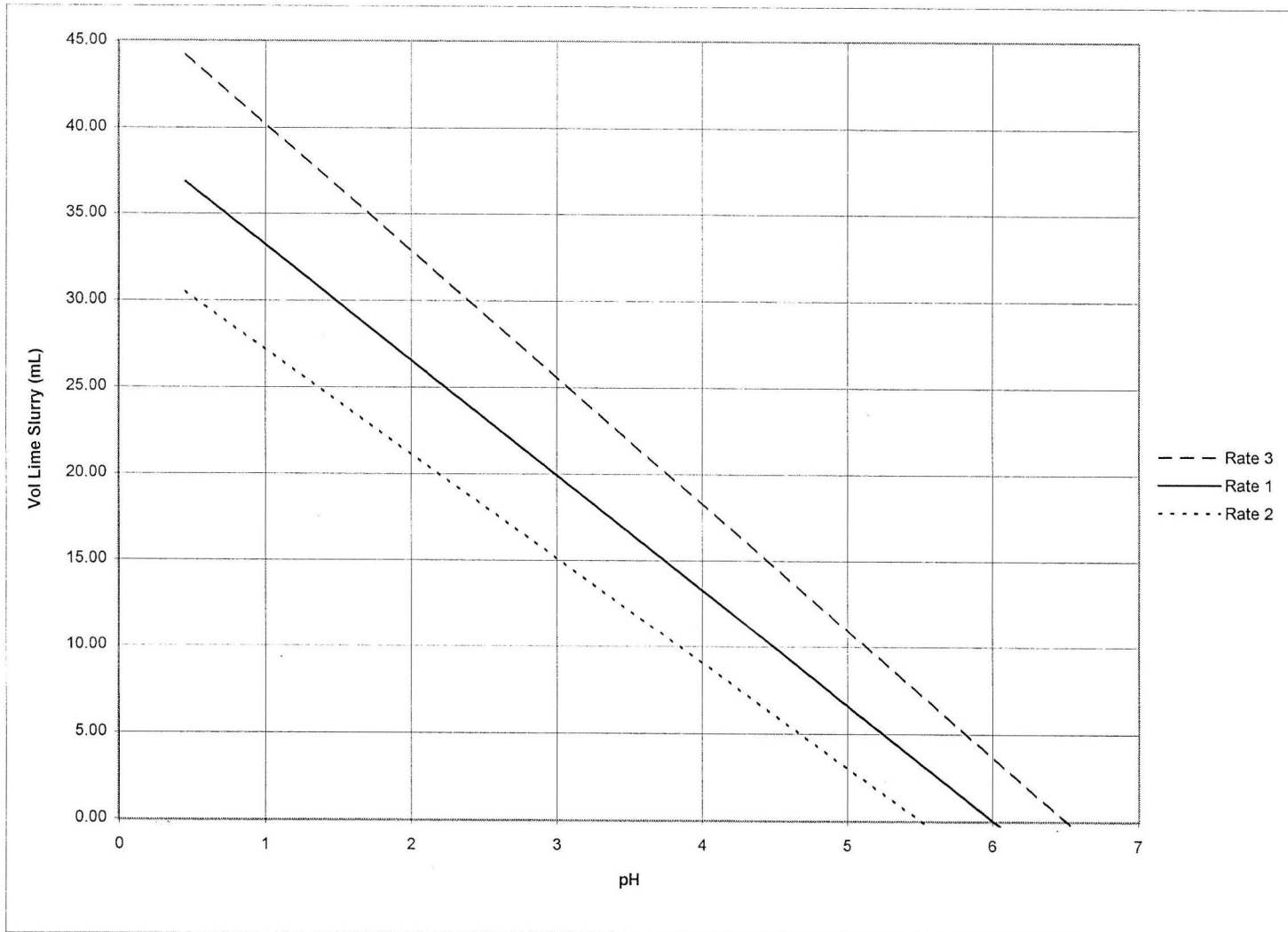
1. Prepare a 200 g/L hydrated lime slurry. The suspension is maintained by continuous mixing. A 10 ml sample of the hydrated lime slurry should be titrated with sulfuric acid at a known molarity, to determine the reactivity (R) of the lime slurry as mols  $\text{Ca}(\text{OH})_2$  per liter. The reactivity should be determined for each fresh batch of lime slurry that is prepared.
2. In triplicate, place 500 grams of the < 1/4 inch sample in a 1.0 to 1.5 liter Erlenmeyer flask or roll bottle.
3. Add 500 ml distilled water to each sub-sample, and lightly agitate for 1 hour.
4. Allow solids to settle for a few minutes and measure solute pH and conductivity.
5. Calculate the average pH for the three samples, and from Figure B-1 obtain the volumes of lime slurry to be added to each sample.
6. Add the indicated volumes of lime slurry to the respective vessels.
7. Gently agitate the samples for a period of 24 hours and record the final pH and conductivity.

## INTERPRETATION

The alkali demand for the sample is determined from the lime addition rate that yields a final pH nearest 7.0, but greater than 6.5. The alkali demand is back-calculated on the basis of the reactivity of the lime slurry and the weight of the sample as follows:

$$\text{Alkali demand as lime (mg/kg)} = R * V * 74 / m$$

where: R = reactivity in mols  $\text{Ca(OH)}_2$  /liter  
V = ml lime slurry added  
m = weight of sample in grams



**FIGURE B-1. LIME SLURRY ADDITION RATE**

Estimated lime addition rates have been established on the basis of the pH - alkali demand obtained in the short term testing program.

The central line represents the best fit to all the data.

The upper limit represents a 15% excess in alkali demand, and captures all samples but 2/29.

The lower is 15% below the average alkali demand, and only 2/29 samples fall below this line

## B.2 ANOXIC SATURATED COLUMN TEST

### EQUIPMENT

1. Column: ID = 4 inches  
Height = 16 inches  
sealable top and baseplate within and outlets as shown in Figure B-2.
1. Argon gas
2. Feed water reservoir
3. pH, redox and conductivity meters
4. Tubing and valves as required

### METHOD

1. *Column set-up:* The sample of < 1/4 inch material is blended to prevent particle size segregation. Where an amendment is required, approximately 16 lb (7.5 kg) of the amended and blended material is prepared. The column is prepared by placing a bottom screen (fine nylon mesh) on the baseplate of the column, as shown in Figure B-2. The weight of the column is obtained, and the sample is loaded to fill the column level with the top. The sample is then lightly compacted to ensure minimal settling during the saturation process. The bulk density of the sample should be checked by calculation prior to sealing the column. The another nylon mesh screen is placed on top of then sample, and the column is sealed.
2. *Operation:* Anoxic testing procedures are to be followed. Prior to flooding, the sample is flushed with argon to displace oxygen from the pore space. The feed solution is introduced from the base of the column and is allowed to percolate up through the sample at a slow rate (approximately 4 hours to fill) to displace contained pore gases. Once the solute is observed in the outlet tube, the inlet is sealed. The column is sealed by placing the outlet tube in a water trap to allow gas to escape from the column, but prevent oxygen entry into the column. The column is allowed to stand stationary for a period of 14 days.
3. *Leachate Displacement:* After the pore water has been allowed to equilibrate for 14 days, the outlet tube is removed from the water trap and placed in the leachate receptacle. To maintain anoxic conditions, the head space of the vessel is continuously flushed with argon gas. The pore water is then slowly displaced from the column with de-aerated (by vacuum) distilled water that is being sparged with argon to remove dissolved oxygen.

The pore water should be displaced at a continuous rate of about 4 ml/min or less (i.e. over a 4 hour or greater period), to minimize the effects of channel flow. The column is again sealed as before. The redox potential, pH and conductivity of the displaced pore water is to be obtained while maintaining anoxic conditions. The solute is then split, filtered and preserved as appropriate for dissolved constituent analysis.

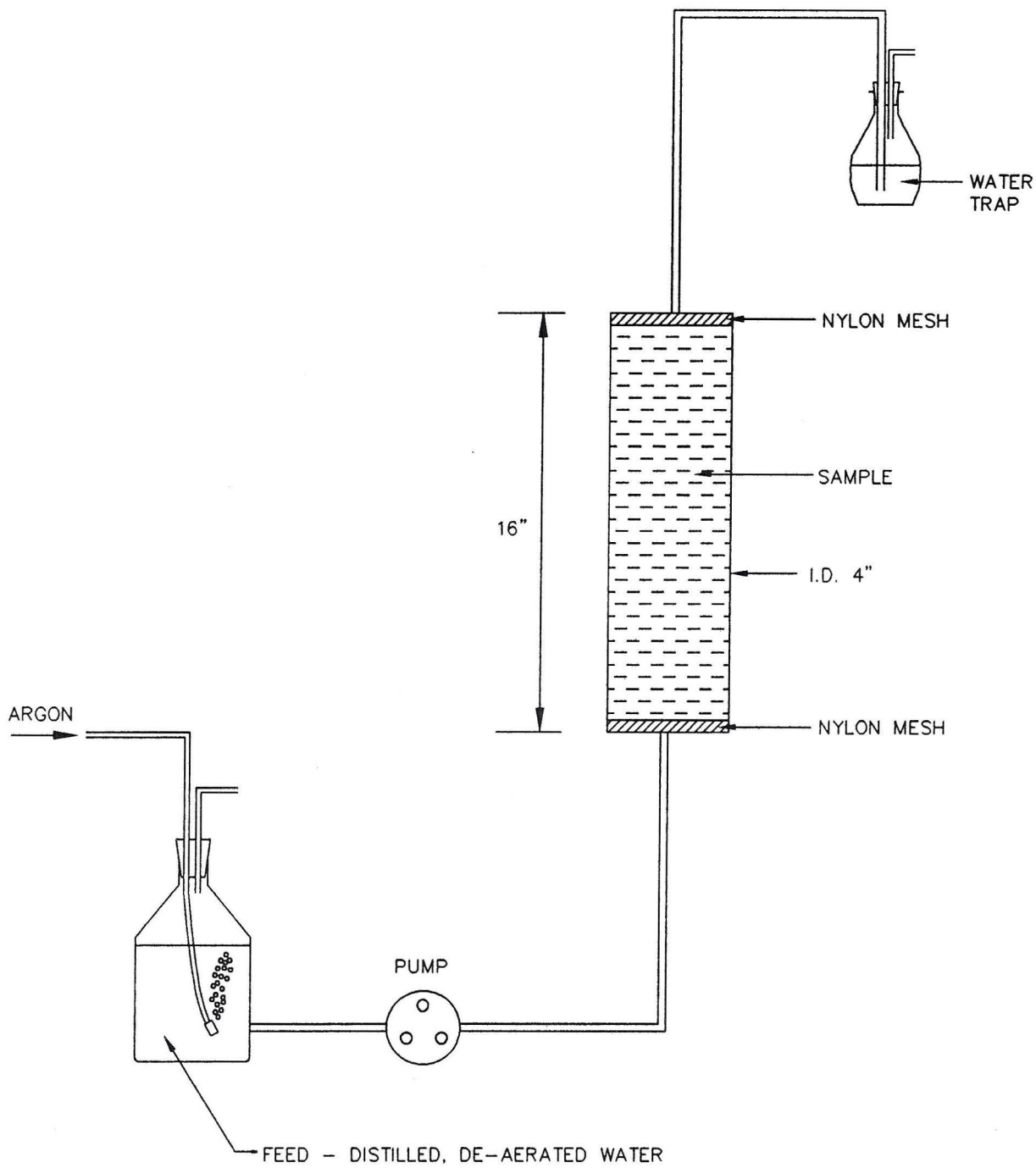
Steps 3 is repeated until steady solute concentrations are obtained. It is anticipated that this would be attained in about 3 to 4 cycles.

### **LIST OF ANALYTES**

pH, redox, conductivity, alkalinity/acidity

Anions:  $\text{SO}_4^{2-}$ ;  $\text{Cl}^-$

Cations: Al, Cd, Ca, Cr, Cu, Co,  $\text{Fe}^{2+}$ , Fe(T), K, Mn, Mg, Na, Tl.



STEFFEN ROBERTSON AND KIRSTEN  
Consulting Engineers

FLAMBEAU MINE BACKFILLING

TYPICAL ANOXIC COLUMN  
TEST SET-UP

FOTH AND VAN DYKE

PROJECT NO.

F107108

DATE

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APPROVED

FIGURE

B-2



### B.3 LEACH EXTRACTION TEST

#### EQUIPMENT

1. 1.5 liter Erlenmeyer flasks or roll bottles
2. distilled water
3. pH, redox and conductivity meters
4. rotary shaker or rollers as appropriate
5. filtration equipment and 0.45 $\mu$ m filter membranes

#### METHOD

1. Weigh out accurately 750 grams of the < 1/4 inch material and place in extraction vessel.
2. Add 750 ml distilled water to extraction vessel.
3. Gently agitate sample for 24 hours.
4. Allow solids to settle and measure solute pH, conductivity and redox potential.
5. Extract leachate, filter and preserve solute samples as required for analysis.

Note: The anoxic extraction test procedure is exactly the same as described above, but the head space of the test vessel is flushed with argon to displace oxygen, and is kept sealed at all times. The duration of the test (item 3) is extended from 24 hours to 72 hours.

#### LIST OF ANALYTES

pH, redox, conductivity, alkalinity/acidity

Anions:  $\text{SO}_4^{2-}$ ;  $\text{Cl}^-$

Cations: Al, Cd, Ca, Cr, Cu, Co,  $\text{Fe}^{2+}$ ,  $\text{Fe(T)}$ , K, Mn, Mg, Na, Tl.

## B.4 ACID CONSUMPTION TEST

### EQUIPMENT AND REAGENTS

1. 500 ml Erlenmeyer flask
2. pH meter
3. Burette
4. Spatula or magnetic stirrer
5. Distilled water
6. 0.4 molar sulfuric acid

### METHOD

1. The residue from the anoxic leach test is air dried and quartered using a sample splitter to yield a dry weight sample of about 188 grams. The exact weight of the sample is obtained. The moisture content of the sample is determined on a second quarter of the sample.
2. Place the sample in a 500 ml Erlenmeyer flask, and add distilled water to provide a 1:1 solid to liquid ratio (w/w).
3. Titrate the slurry with 0.4 molar sulfuric acid, while continuously stirring the slurry, to a stable endpoint of 4.5. The endpoint should not change within the period of 1 hour to be considered as stable.
4. The total volume of sulfuric added is recorded.

### INTERPRETATION

The residual neutralization potential (NP) for the sample is calculated as follows:

$$NP = m * V * 100 / w$$

where NP = the neutralization potential in mg CaCO<sub>3</sub> eq. / g  
m = molarity of the sulfuric acid  
V = volume titrant added to the sample  
w = weight of the sample in grams