APPENDIX I

Raw Analytical Data and Methods

International Metallurgical and Environmental Inc.
Acid-Base Accounting

Project : Klohn Crippen - Calgary
Mine: ULU Project

Sample	Paste pH	Total S %	S as SO_4^{-2} %	Acid Pot. kg/t $CaCO_3$	Neut. Pot. kg/t $CaCO_3$	Net Neut. Pot kg/t $CaCO_3$	NP: AP Ratio	CO_2 kg/t $CaCO_3$
KC-1	8.63	0.26	<0.01	8	45	37	5.6	24.1
KC-2	8.62	0.09	0.01	3	27	24	10.6	0.2
KC-3	8.11	3.91	0.02	122	13	-109	0.1	0.4
KC-4	9.26	0.49	0.01	15	22	7	1.5	1.7
KC-5	8.56	2.65	0.01	83	21	-61	0.3	6.5
KC-6	8.34	2.8	0.01	87	14	-73	0.2	2.2
KC-7	8.38	3.15	0.01	98	21	-77	0.2	3.8
KC-8	8.69	2.29	0.02	71	24	-47	0.3	6.7
KC-9	8.76	0.15	0.01	4	30	26	6.9	6.5
KC-10	8.87	0.28	0.01	8	26	18	3.1	8.8
KC-11	8.79	0.93	0.02	28	33	5	1.2	12.5
KC-12	8.75	1.17	0.01	36	33	-3	0.9	6.3
KC-13	8.60	2.24	0.01	70	30	-39	0.4	5.3
KC-14	8.60	1.60	0.01	50	18	-31	0.4	5.3
KC-15	9.43	0.28	0.02	8	24	15	2.9	1.0
KC-16	9.18	0.22	0.03	6	16	10	2.7	1.6
KC-17	8.37	1.19	0.06	35	23	-13	0.6	<0.1
KC-18	7.23	1.29	0.03	39	26	-14	0.6	0.2
KC-19	8.90	0.93	0.01	29	18	-11	0.6	3.5
KC-20	8.96	0.11	0.01	3	22	19	7.2	1.7
KC-21	8.82	0.07	0.01	2	21	19	11.3	1.2
KC-22	8.54	0.22	0.01	7	25	19	3.9	<0.1
KC-23	8.95	0.13	0.01	4	18	14	4.7	1.2
KC-24	9.27	0.06	0.01	2	15	14	9.8	1.0

International Metallurgical and Environmental Inc.
Acid-Base Accounting

Project : Klohn Crippen - Calgary
Mine: ULU Project
Date: Oct 25, 1996

Sample	Paste pH	Total S %	S as SO_4^{-2} %	Acid Pot. kg/t $CaCO_3$	Neut. Pot. kg/t $CaCO_3$	Net Neut. Pot kg/t $CaCO_3$	NP: AP Ratio	CO_2 kg/t $CaCO_3$
KC-25	9.03	1.49	0.01	46	22	-25	0.5	3
KC-26	9.03	0.58	<0.01	18	19	1	1.1	3
KC-27	9.24	0.80	0.01	25	23	-1	0.9	6
KC-28	9.00	0.31	<0.01	10	23	13	2.4	6
KC-29	9.21	0.73	0.01	23	23	1	1.0	11
KC-30	9.18	0.37	<0.01	12	20	9	1.8	2
KC-31	8.96	0.32	<0.01	10	19	9	1.9	4
KC-32	9.07	0.20	0.01	6	19	13	3.2	1

CANADIAN CERTIFIED REFERENCE MATERIALS PROJECT (CCRMP)

Catalogue of Certified and Provisional Reference Materials

CCRMP is a project of the Mineral Sciences Laboratories of CANMET
Natural Resources Canada, 555 Booth Street, Ottawa, ON, Canada K1A 0G1

NBM-1	ARD Acid Base Accounting	100 g	AVAILABLE	60.00

CERTIFIED ELEMENTS

Element	Certified Value	Uncertainty
S	0.298 %	± 0.015

PROVISIONAL ELEMENTS

Element	Provisional Value	Element	Provisional Value
NNP	42 t/1000 t	NP	52 t/1000 t

GA/GC Results.

Im+E
12/9/96

NP		RV	52 t/1000t	Im+E	55.5 51.2
S	UTS-1	RV	1.0 %	Im+E	1.06
	NBM-1	RV	0.30 %	Im+E	0.32
SO4		RV	0.32 %	Im+E	0.29
		RV	0.01 %	Im+E	0.02

INTERNATIONAL METALLURGICAL AND ENVIRONMENTAL INC.

Modified Acid Base Accounting

OBJECTIVES:

- to determine the balance between acid producing and acid consuming components of a mine waste.

PRINCIPLES OF TEST:

The fundamental principles of the modified acid base accounting procedure are essentially the same as for the standard procedure, and involve: (1) determination of the neutralization potential (NP) of a sample and (2) calculation of the acid potential (AP) of the sample. The difference between the two values, the net neutralization potential (Net NP), allows classification of the sample as potentially acid consuming or producing. A positive value for the Net NP indicates that the sample is a net consumer of acid. To facilitate comparison of values, NP, AP, and Net NP are all expressed in units of tonnes $CaCO_3$ equivalent per 1000 tonnes.

In the modified method, the neutralization potential is determined by treating a sample with excess standardized hydrochloric acid at ambient, or slightly above (25-30 C) ambient temperatures for 24 hours. Acid is added as required during the acid-treatment stage to maintain sufficient acidity for reaction. After treatment, the unconsumed acid is titrated with standardized base to pH 8.3 to allow calculation of the calcium carbonate equivalent of the acid consumed. The sample is analyzed for total sulphur and sulphate sulphur, and sulphide sulphur is calculated by difference. AP is determined from the calculated sulphide sulphur analysis, assuming (1) total conversion of sulphide to sulphate, and (2) production of 4 moles H^+ per mole of pyrite oxidized. In some cases, other sulphur species might be determined.

The modified acid base accounting test is still under development, although the test as described herein has been in use essentially since 1986.

A. Reagents

1. Distilled and deionized water.
2. Certified grade, 1.0 N hydrochloric acid, for standardization of bases.
3. Certified grade, 1.0 N sodium hydroxide for standardization of acids.

4. *0.1 N hydrochloric acid* - using a 100 ml burette, dispense 16.5 ml of concentrated HCl into a 2000 ml volumetric flask and bulk to the mark with deionized water.

5. *0.5 N hydrochloric acid* - using a 250 ml burette, dispense 82.5 ml of concentrated HCl into a 2000 ml volumetric flask and bulk to the mark with deionized water.

6. *0.1 N sodium hydroxide* - measure 8.0 grams of granular NaOH into a plastic weighing boat and transfer the NaOH into a 1000 ml beaker containing 500 ml of deionized water. Place on magnetic stirrer until dissolved. Transfer into a 2000 ml volumetric flask and bulk to volume with deionized water.

7. *0.5 N sodium hydroxide* - measure 40 grams of granular NaOH into a plastic cup and transfer the NaOH into a 1000 ml beaker containing 500 ml of deionized water. Place on a magnetic stirrer until dissolved. Transfer into a 2000 ml volumetric flask and bulk to volume with deionized water.

NOTE: granular NaOH should only be handled and measured with a plastic spatula and weighing boat. Take extra care not to allow contact with metal.

8. *Phenolphthalein Indicator* - dissolve 0.5g. Phenolphthalein in 50 ml. ethyl or isopropyl alcohol and bulk to 100 ml. with distilled water.

B <u>Reagent Standardization Method</u>:

-Pipette 20 ml of 0.1 N HCl (4.) into a 50 ml beaker. Add 4 to 5 drops phenolphthalein indicator and place on a magnetic stirrer. Titrate with a certified 1.0 N NaOH standard solution from a conditioned 50 ml burette until the endpoint (from a clear to a pink color) is reached. <u>Approximately</u> 2 ml. (lets say 2.1 ml NaOH was used for the example equation).

Duplicate this step to standardize the 0.5 N HCl (5.). The endpoint should take approximately 10 ml of the certified 1.0 N NaOH standard solution.

-Pipette 20 ml of 0.1 N NaOH (6.) into a 50 ml beaker. Add 4 to 5 drops phenolphthalein indicator and place on a magnetic stirrer. Titrate with a certified 1.0 N HCl standard solution from a conditioned 50 ml burette until the endpoint (from a pink to a clear solution) is reached. <u>Approximately</u> 2 ml.

Duplicate this step to standardize the 0.5 N NaOH (7.). The endpoint should take approximately 10 ml of the certified 1.0 N HCl standard solution.

Normality (NaOH/HCl) : V(base) X N(base) = V(acid) X N(acid)
e.g.: 20 ml of ? N HCl ,2.1 ml of 1.0 N NaOH was used. Then;

$$(2.1 \text{ ml NaOH})(1.0 \text{ N NaOH}) = (20 \text{ ml HCl})(\ ?\ \text{N HCl})$$

$$\text{Therefore: } ?\ \text{N HCl} = \frac{(2.1 \text{ ml})(1.0 \text{ N})}{20 \text{ ml}}$$

NOTE: Record Normality to 3 decimal places.

C. Procedure

a) Weigh 2.00 grams of sample into a 250 ml Nalgene wide mouth bottle, and add 40 ml (using a 100 ml burette) of 0.1 N standardized HCl.

b) Cover with a film of saran wrap and secure with a punctured lid.

c) Agitate the contents for 24 hours by placing on a mechanical shaker.

d) After 6 hours check the pH of the pulp.

d.i) If the pH of the pulp is below 1.0 at this point, the sample must be rerun starting with a smaller amount of 0.1 N HCl.

d.ii) If the pH of the pulp is above 1.3 at this point, carefully add 0.5 N HCl until the pH drops to 1.2-1.25. Return it to the shaker and continue shaking for the remainder of the test period.

NOTE: The Normality of the hydrochloric acid added at this point should be of the same strength originally added. However, the volume of the bottles may not accommodate the added amount of HCl , therefore, you can use 0.5 N HCl at this point as long as it is accounted for in the final equation.

d.iii) If the pH of the pulp is between 1.0 and 1.3 return it to the shaker and continue for the remainder of the test period.

e) At the end of the shaking period, check the pulp pH. If the total volume and strength of acid was appropriate, the end pH will be in the range 1.0 - 1.5. If the pH is above this range, the amount of acid added is judged to be insufficient for the reaction. If the pH is below the range, the amount of acid added is judged to be too high, causing over- reaction. In either case, repeat the test using the next higher or lower volume or strength of HCl as required.

f) If the pH is within the 1.0-1.5 range titrate the contents using 0.5 N NaOH until a pH of 8.3 is reached. Titrate with NaOH until a *constant reading* of 8.3 remains for at least 30 seconds.

NOTE: The Normality of the sodium hydroxide added at this point should be of the same strength originally added. However, if additional acid of 0.5 N was added at the 6 hour mark then it is best to titrate with 0.5 N NaOH at this point as long as it is accounted for in the final equation.

h) All samples must be run in duplicate.

i) The NBM-1 CANMET standard is run with each set of samples to insure the accuracy of the procedure.

D. Calculations:

1. The neutralization potential, NP, of the sample given by:

$$NP = \frac{50a\,[x - (b/a)y]}{c}$$

where:NP = neutralization potential in tonnes $CaCO_3$ equivalent per 1000 tonnes of material
a = normality of HCl
b = normality of NaOH
c = sample weight in grams
x = volume of HCl added in ml
y = volume of NaOH added to pH 8.3 in ml

2. Different concentrations :

2.1) e.g. If you started with 40 ml 0.1 HCl and 6 ml of 0.489 N HCl was added at the 6 hour mark then the following calculation would be used to determine:

$$\underline{a}:\quad \frac{0.489\text{ N}}{5} = 0.098\text{ N} \qquad \frac{0.098\text{ N} + 0.1\text{ N}}{2} = \underline{0.099\text{ N HCl}}$$

$$\underline{x}:\quad 6\text{ ml} \times 5 = 30\text{ ml} \qquad 40\text{ ml} + 30\text{ ml} = \underline{70\text{ ml of }.099\text{N HCl}}$$

2.2) e.g. con't At the end of the test period 8.6 ml of 0.498 N NaOH was added, then the following calculation would be used to determine:

y: 8.6 ml X 5 = 43 ml

b: 0.498 N / 5 = 0.100 N NaOH

Therefore, the NP would be:

$$\frac{50(0.099\ \mathrm{N})\,[70\ \mathrm{ml} - (0.100\ \mathrm{N} / 0.099)\ 43\ \mathrm{ml}]}{2.00\ \mathrm{grams}} = 65.75$$

3. The acid potential, AP, of the sample in tonnes $CaCO_3$ equivalent per 1000 tonnes, is given by:

AP = Percent sulphide sulphur X 31.25

where sulphide sulphur = total sulphur - sulphate sulphur

4. The net neutralization potential, Net NP, in tonnes $CaCO_3$ equivalent per 1000 tonnes of material is given by:

Net NP = NP X AP

REFERENCES

1. Coastech Research, Modified Acid Base Accounting, *AMD Prediction Procedures*, Vancouver, B.C. July (1990).

Remark: This procedure is a modification of Coastech Research method (Modified Acid Base Accounting, July 1990)

INTERNATIONAL METALLURGICAL AND ENVIRONMENTAL INC.

Carbonate Analysis

The following procedure is used for the analysis of met-lab samples for the determination of carbonate.

Glassware Cleaning Procedure

All glassware must be cleaned with 10% HCl. DI water rinsed and acetone dried.

Preparation of CO2 Free Water

1) Boil DI Water for 15 Minutes
2) Transfer to a 2 Litre flask with the tube containing glass wool and CO_2 removing agent Ascarite II. Rapid cooling can be accomplished by running cold water over flask or putting into an ice bath.

Preparation of Reagents

0.1N HCl:(Pipette) 8.25 ml concentrated HCl into 1000 ml flask and bring to the mark with CO_2 Free water.

0.6N HCl: (Pipette) 50 ml concentrated HCl into 1000 ml flask and bring to the mark with CO_2 Free water.

0.1N NaOH: weigh 4gm of NaOH into 1000 ml flask and bring to the mark with CO_2 Free water.

40% Potassium Hydroxide:
40% in 100 ml flask , weigh 40.0 gms.
40% in 300 ml flask , weigh 120.0 gms.
40% in 200 ml flask , weigh 80.0 gms.

15% $BaCl_2$ Solution: weigh 150 grams $BaCl_2$ crystals into 1000 ml flask and bring to the mark with CO_2 Free water.

Indicator: dissolve 0.5 gms of Phenolphthalein in 50 ml Iso Propyl alcohol and add 50 ml CO_2 Free water.

Procedure:

1) Weigh out 1.0 to 5.0 grams of sample into a flat bottom flask.

2) Into a 1 Litre filtering flask Pipette exactly 50 ml of 0.1N NaOH and then add 4 to 5 drops of indicator. The indicator will turn the solution pink when added.

3) Connect a separatory funnel to the flat bottom flask and the filtering flask so a vacuum can be established between both flasks. Evacuate the system for about 5 minutes. Don't forget to close the stop cock to maintain a vacuum throughout the test.

4) Add 50 ml of 0.6N HCl (using a graduate cylinder) to the separatory funnel and slowly add to the flat bottom flask. (i.e., add three drops then shake, add 3 drops then shake etc.). Stop flow when a few drops remain in the bottom of the separatory funnel.

5) After contact for a few minutes, shake every 2 min. for 10 minutes.

6) Connect the top of the separatory funnel to the Gas Washing Tower containing 40% KOH and very slowly draw in CO_2 free air until the vacuum is destroyed. This should take about 10 minutes. Shake flasks for about 10 minutes.

7) Disconnect filter flask:
   - Rinse down sides with CO_2 free water.
   - Add 10 ml of $BaCl_2$ solution to the filter flask.
   - Titrate with 0.1N HCl until indicator becomes colorless.

NOTE: A reagent blank must be carried through the entire operation.

*****If titer is less than 5 ml re-run on a lower sample weight.**

*****Also re-run on a lower weight if solution turns milky when $BaCl_2$ solution is added.**

CALCULATIONS:

$$\frac{(\text{Blank titer, ml} - \text{Sample titer, ml}) \times \text{Normality of HCl} \times 5}{\text{Weight of sample, (grams)}} = \text{Calcium carbonate equivalent, \%}$$

Standardize 0.1 N HCl against standard 1.00 Normality NaOH solution and check 0.1 N NaOH against standard 1.00 Normality HCl solution.

Reagent Standardization Method:

-Pipette 20 ml of 0.10 N HCl into a 50 ml beaker. Add 4 to 5 drops phenolphthalein indicator and place on a magnetic stirrer. Titrate with a certified 1.0 N NaOH standard solution from a conditioned 50 ml burette until the endpoint (from a clear to a pink color) is reached. Approximately 2 ml. (lets say 2.1 ml NaOH was used for the example equation).

-Pipette 20 ml of 0.1 N NaOH into a 50 ml beaker. Add 4 to 5 drops phenolphthalein indicator and place on a magnetic stirrer. Titrate with a certified 1.0 N HCl standard solution from a conditioned 50 ml burette until the endpoint (from a pink to a clear solution) is reached. Approximately 2 ml.

Normality (NaOH/HCl) : V(base) X N(base) = V(acid) X N(acid)

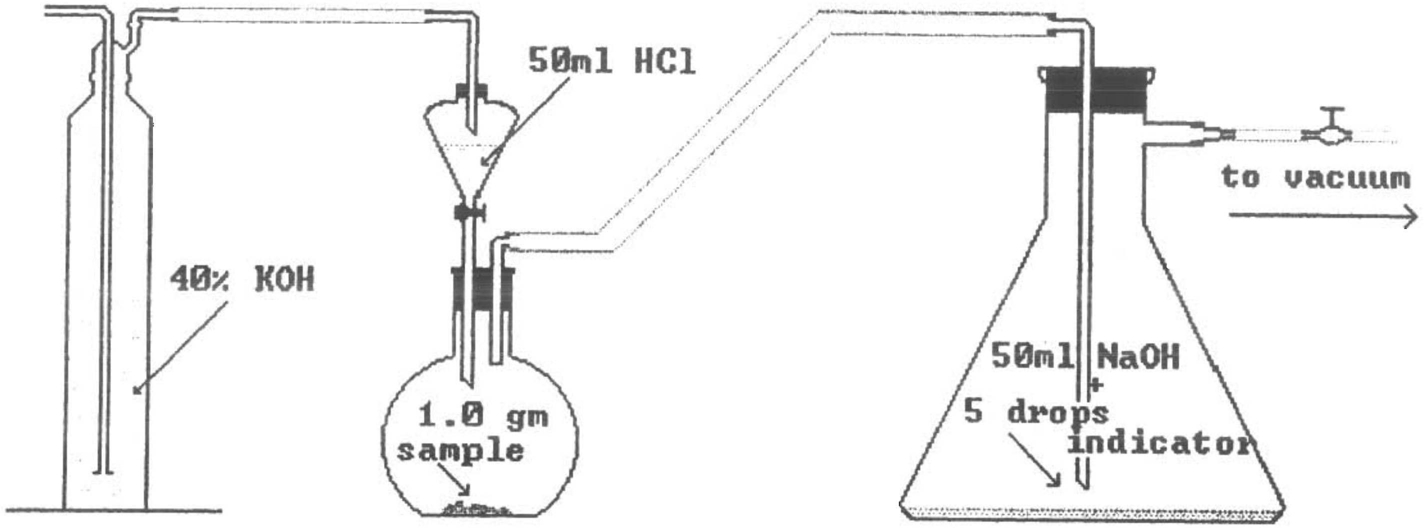
e.g.: 20 ml of ? N HCl ,2.1 ml of 1.0 N NaOH was used. Then;

$$(2.1 \text{ ml NaOH})(1.0 \text{ N NaOH}) = (20 \text{ ml HCl})(\text{ ? N HCl})$$

$$\text{Therefore: ? N HCl} = \frac{(2.1 \text{ ml})(1.0 \text{ N})}{20 \text{ ml}}$$

NOTE: Record Normality to 3 decimal places.

Carbonate Apparatus Setup:



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