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SURFACE CHEMISTRY OF COMPOSITE TAILINGS SAMPLES FROM THE FARO DEPOSIT, YUKON

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Core Technologies and Services Laboratory

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Cost Recovery

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SURFACE CHEMISTRY OF COMPOSITE TAILINGS SAMPLES FROM THE FARO DEPOSIT, YUKON

by

L.J. Cabri*

EXECUTIVE SUMMARY

Laser Ionization Mass Spectrometry (LIMS) was used to obtain semi-quantitative data on the surface chemistry of a series of 15 composite tailings samples from the Faro deposit, Yukon. Optical microscopy of polished sections indicated that the sulphides are not altered and consist of the following minerals, in decreasing order of abundance: pyrite, marcasite, sphalerite, pyrrhotite, chalcopyrite, and galena. Sphalerite, pyrite, and pyrrhotite occur mostly as liberated particles.

LIMS analyses of selected sphalerite grains showed that in all 15 samples, the sphalerite grains were activated with Cu (percent of Cu-activated grains range from 33.3 to 100%) with no obvious correlation with pH at the sample site. On the other hand, there was a negative correlation between Pb-activated sphalerite grains (percent of Pb-activated grains range from 12.5 to 50%) at low pH (2.6 to 5.5). This was followed by erratic values in the intermediate zone (pH 6.3 to 6.9) and, at high pH values (7.5 to 8.3) with increased sample depths, by fairly consistent highly Pb-activated sphalerite grains (percent of Pb-activated grains range from 62.5 to 100%). The dominance of Pb-activated liberated sphalerite particles should guide processing options, but the common occurrence of marcasite should also be noted.

Keywords: Flotation tailings, sulphides, LIMS analyses, surface chemistry, Cu-activation, Pb-activation

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INTRODUCTION

Curragh Resources is investigating the possibility of reprocessing old flotation tailings from the Faro deposit, Yukon, prior to transfer of the tailings for final deposition in the mined-out Faro open pit. Results obtained from laboratory tests carried out previously on the old tailings indicated a limited response to standard sulphide flotation. The study of the surface chemistry of particles from the tailings is part of a more comprehensive study by CANMET (Project 50854, T. Cienski, Project Leader). It was decided to use a Laser Ionization Mass Spectrometer (LIMS) for the surface characterization, based on earlier unpublished analyses and tests on base metal flotation products.

METHODOLOGY

Fifteen composite samples were provided by Mr. T. Cienski (RUL/CANMET). The samples had been screened to +200 mesh with distilled water and dried (Table 1). A polished section was made for each composite in order to obtain mineralogical information by optical microscopy, necessary for guiding sample selection and preparation for the LIMS analyses. Sphalerite and pyrite/marcasite grains were hand-picked under a binocular microscope by Mr. D.R. Owens (CTSL/CANMET) and carefully placed on top of thin pieces of pure indium foil (~5 mm square), because the grains stick to the indium surface. The indium pieces themselves were very lightly stuck

Table 1. Curragh Resources: Faro Down Valley Tailings Composite Samples

Composite Sample	Curragh Sample	pH	Average pH	Composite Sample	Curragh Sample	pH	Average pH
C1	D-2-1	2.32		C10	D-2-10	7.54	
	E-7-1	3.19			E-7-10	8.46	
	F-10-1	2.20	2.57		F-10-10	6.21	
					F-11-10	8.18	7.60
C2	E-7-2	4.51					
	F-10-2	2.67	3.59	C11	D-2-11	7.81	
					E-7-11	7.83	
C3	E-7-3	4.43			F-10-11	6.47	
	F-10-3	4.66	4.55		F-11-11	7.94	7.51
C4	D-2-4	5.62		C12	D-2-12	7.66	
	E-7-4	4.64			E-7-12	8.77	
	F-10-4	5.30	5.19		F-10-12	7.22	
					F-11-12	8.20	7.96
C5	F-9-5	5.69					
	F-10-5	5.24	5.47	C13	D-2-13	7.62	
					E-7-13	8.54	
C6	D-2-6	6.56			F-10-13	7.46	
	F-10-6	6.01	6.29		F-11-13	8.29	7.98
C7	D-2-7	7.18		C14	D-2-14	7.55	
	E-7-7	6.64	6.91		E-7-14	8.78	
					F-10-14	7.79	
C8	D-2-8	8.00			F-11-14	9.08	8.30
	E-7-8	7.43					
	F-11-8	8.84	8.09	C15	D-2-15	7.68	
					E-7-15	8.51	
C9	D-2-9	7.44			F-10-15	6.40	
	E-7-9	8.46			F-11-15	8.58	7.79
	F-10-9	5.21					
	F-11-9	8.85	7.49				

with double-sided tape to the base of small plastic sample boxes. The laser ionization mass spectrometer used was a LIMA-2A laser microprobe manufactured by Cambridge Mass Spectrometry Ltd. (Cambridge, England). The indium pieces were attached to steel sample holders with conducting carbon paint and were evacuated to about 10^{-7} Torr (in about 5-10 min). A He-Ne pilot laser, mounted co-axially with the primary Nd-YAG ablation laser, was used for focusing. The primary laser was set at 28.7% transmission with a 1.4 μsec delay time (Δ^t) for the post-ionization laser. The ions produced in the laser/solid interaction (focused to $\sim$2 to 5 μm) were then analyzed using a time-of-flight (TOF) mass spectrometer. The acquired spectrum was deconvoluted and calibrated in terms of mass, with the entire process, from "firing" the laser to display of the mass spectrum, taking less than about one minute. The spectra are then saved on diskettes.

The procedure followed was to analyze all sphalerite grains stuck to each indium substrate, as well as analyzing a few grains of pyrite/marcasite. From one to five areas were analyzed with a surface "shot" on each sphalerite grain, followed by one to two (in rare cases, fifteen) further shots penetrating into deeper layers, until neither Cu nor Pb could be detected. This was done in order to assure that the Cu and Pb were due to surface coatings and not to other mineral impurities. A series of successive LIMS spectra demonstrating sampling depth is shown in Figure 1. A CuS layer thick enough to make sphalerite float is, typically, a few atomic layers thick (Mellgran et al., 1973).

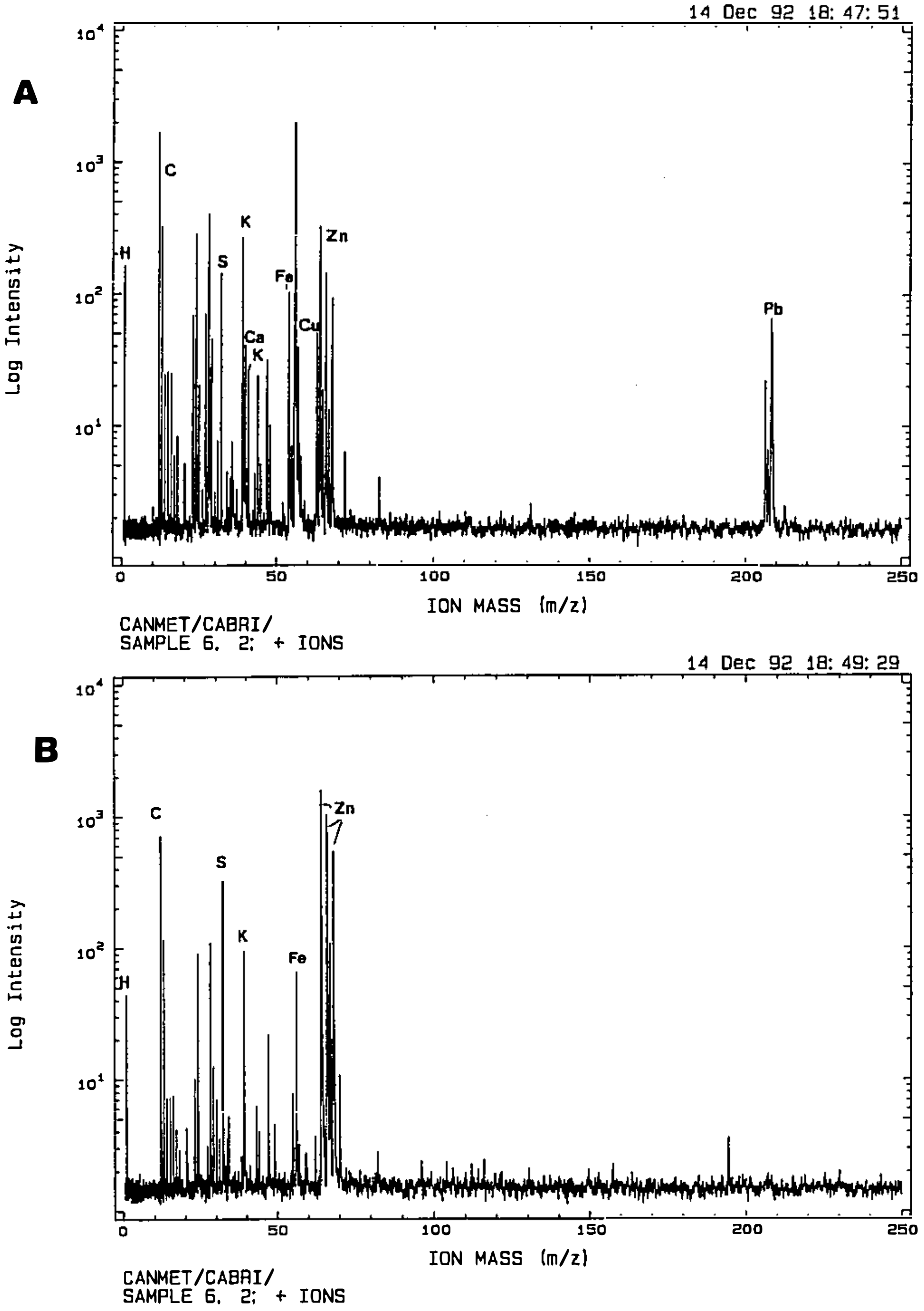

Figure 1. Two LIMS spectra obtained from the upper surface layers (A) of a Cu- and Pb-activated sphalerite grain and (B) obtained by a third firing of the primary laser on the same spot, penetrating into deeper layers. Note that Ca, Cu and Pb have disappeared, Zn has increased.

RESULTS

A total of 568 spectra were recorded and the peak area signal intensity values computed for most common elements found at the surface of sphalerite grains: K, Ca, Fe, Cu, Zn, and Pb (see Fig. 1). It must be emphasized that element sensitivities are highly variable, up to one order of magnitude, and thus relative peak heights for different elements in the LIMS spectra are not proportional to their concentrations. Thus, all the results must be considered only as semi-quantitative. The relative quantity present for each element is obtained by computing the peak signal intensity value, i.e., the sum of areas under the peak corresponding to a specific isotope. Calibration of results for specific minerals is possible, but was beyond the scope of this study. Other elements such as Ba, In, Cd, and Sn, were occasionally found and noted.

Optical microscopy of polished sections revealed that the sulphides are not obviously altered and consist of the following minerals, in decreasing order of abundance: pyrite, marcasite, sphalerite, pyrrhotite, chalcopyrite, and galena. Pyrite is by far the most abundant sulphide and marcasite is quite common, and, from this cursory examination, appears to be primary in origin. Sphalerite, pyrite, and pyrrhotite occur mostly as liberated particles. Sphalerite often contains micro-inclusions of chalcopyrite and no liberated galena was observed. Typical grains are shown in Figure 2.

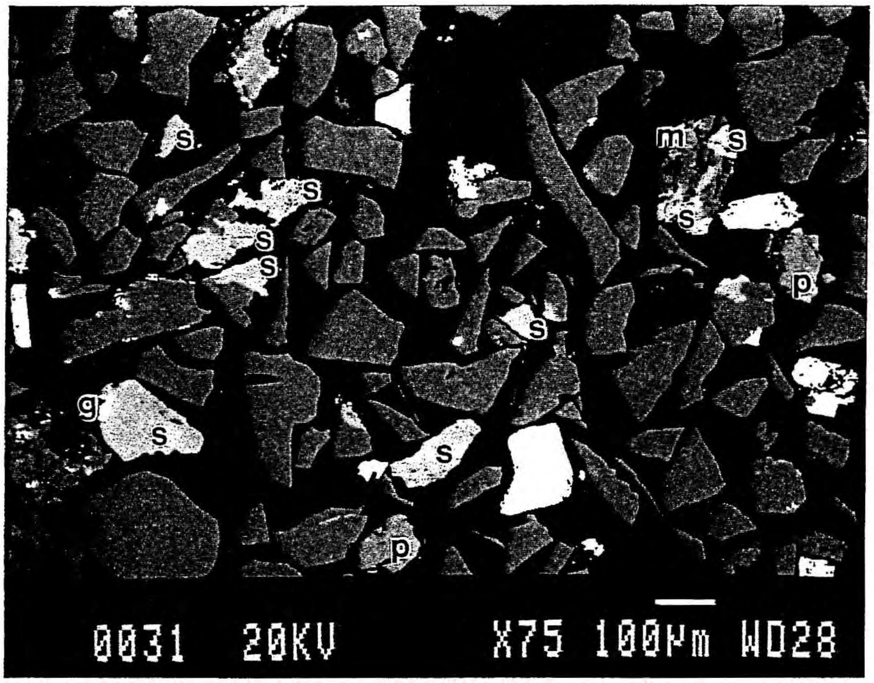

Figure 2. Backscattered Electron Image showing typical liberated sphalerite (s), attached to galena (g), pyrite (grey), and marcasite (m) as well as free pyrrhotite (p) and barite (white). Sample C-5.

The percent of sphalerite grains with detectable Cu and/or Pb for all 15 samples is shown graphically in Figure 3. It can be observed that Pb is most erratic, partly due to its greater sensitivity, and that the number of grains with Pb is low for samples 2 to 5, whereas only sample 5 had significantly fewer grains coated with Cu.

Results for Cu and Pb for individual samples are shown graphically in Figures 4 to 6, illustrating, respectively, typical distribution of Cu- and Pb-activated sphalerite grains with dominance of Cu-activation and low degree of Pb-activation near the surface of the tailings (low pH from 2.6 to 5.5) through a "mixed" zone at intermediate pH (6.3 to 6.9) to

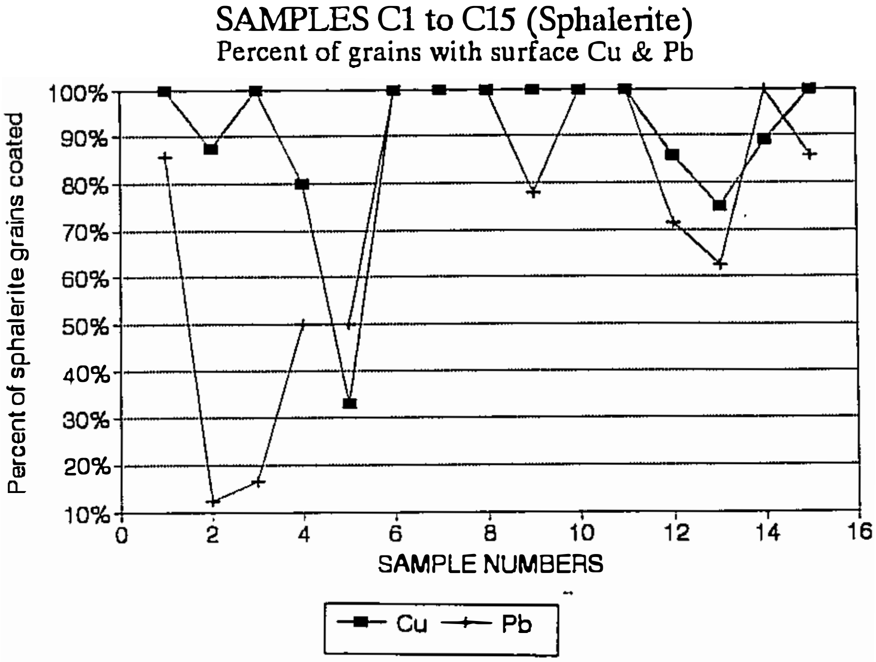

Figure 3. Percent of Cu- and Pb-activated sphalerite grains.

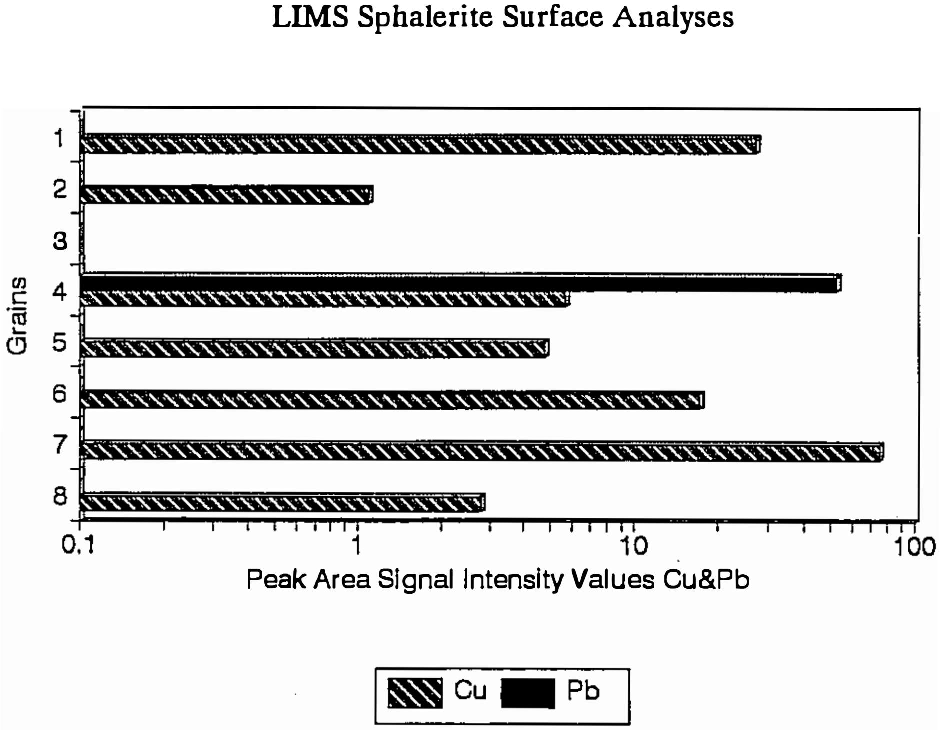

Figure 4. Relative distribution of Cu- and Pb-activated sphalerite, Sample C-2.

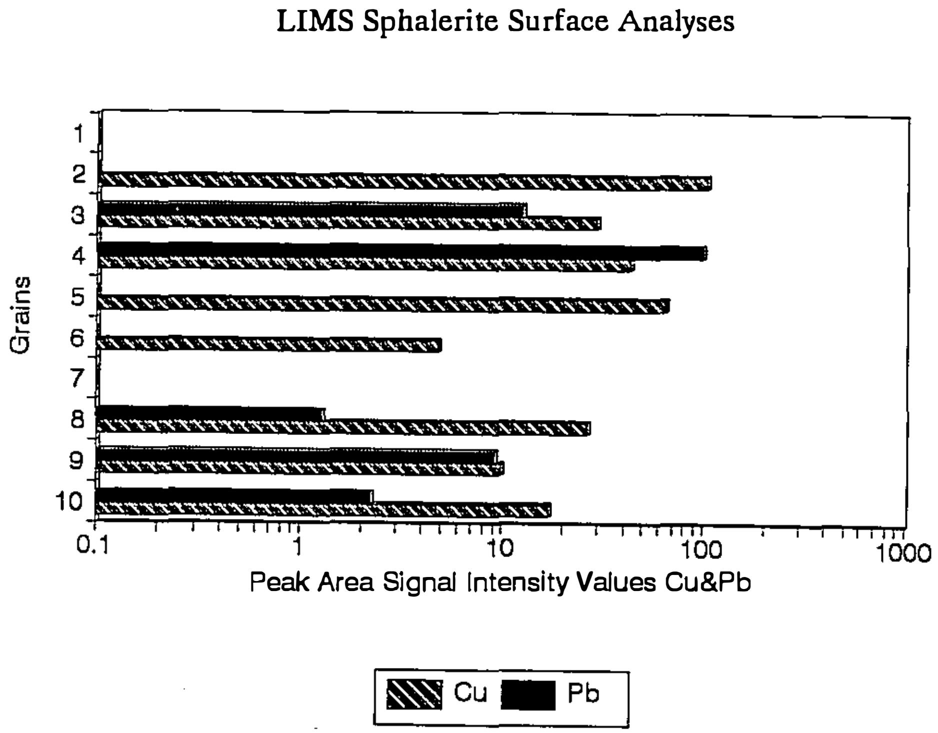

Figure 5. Relative distribution of Cu- and Pb-activated sphalerite, Sample C-4.

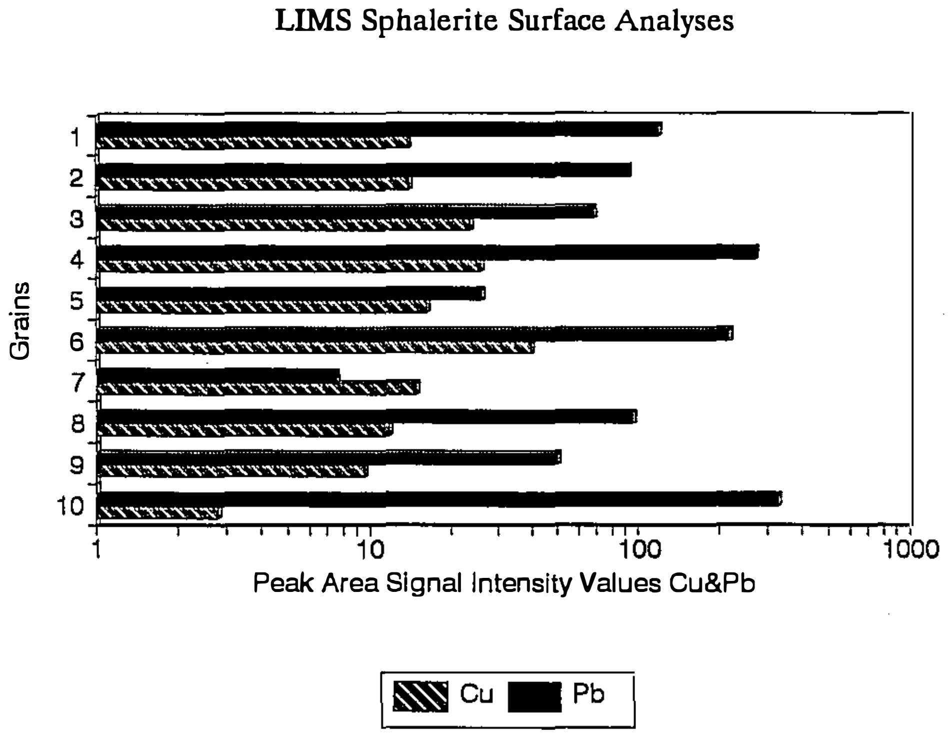

Figure 6. Relative distribution of Cu- and Pb-activated sphalerite, Sample C-10.

typical Pb-activated dominant samples at higher pH (7.5 to 8.3). These results are summarized in Table 2 and Figure 7.

Table 2. Sphalerite grains analyzed

Sample	Cu grains*	Pb grains*	Cu**	Pb**	pH
1	7/7	6/7	25.5	15.7	2.57
2	7/8	1/8	16.7	6.6	3.59
3	6/6	1/6	17.8	0.2	4.55
4	8/10	5/10	30.2	12.5	5.19
5	2/6	3/6	12.9	7.8	5.47
6	8/8	8/8	39.0	225.9	6.29
7	10/10	10/10	39.4	18.2	6.91
8	10/10	10/10	27.6	173.9	8.09
9	9/9	7/9	24.3	83.8	7.49
10	10/10	10/10	17.2	127.8	7.6
11	10/10	10/10	58.1	188.9	7.51
12	6/7	5/7	60.9	65.9	7.96
13	6/8	5/8	23.6	81.2	7.98
14	8/9	9/9	16.3	155.8	8.3
15	7/7	6/7	27.4	174.3	7.79

* Number of grains with detectable Cu or Pb/total number of grains examined.

** These represent the median peak area signal intensity values for Cu and Pb for all grains in each sample.

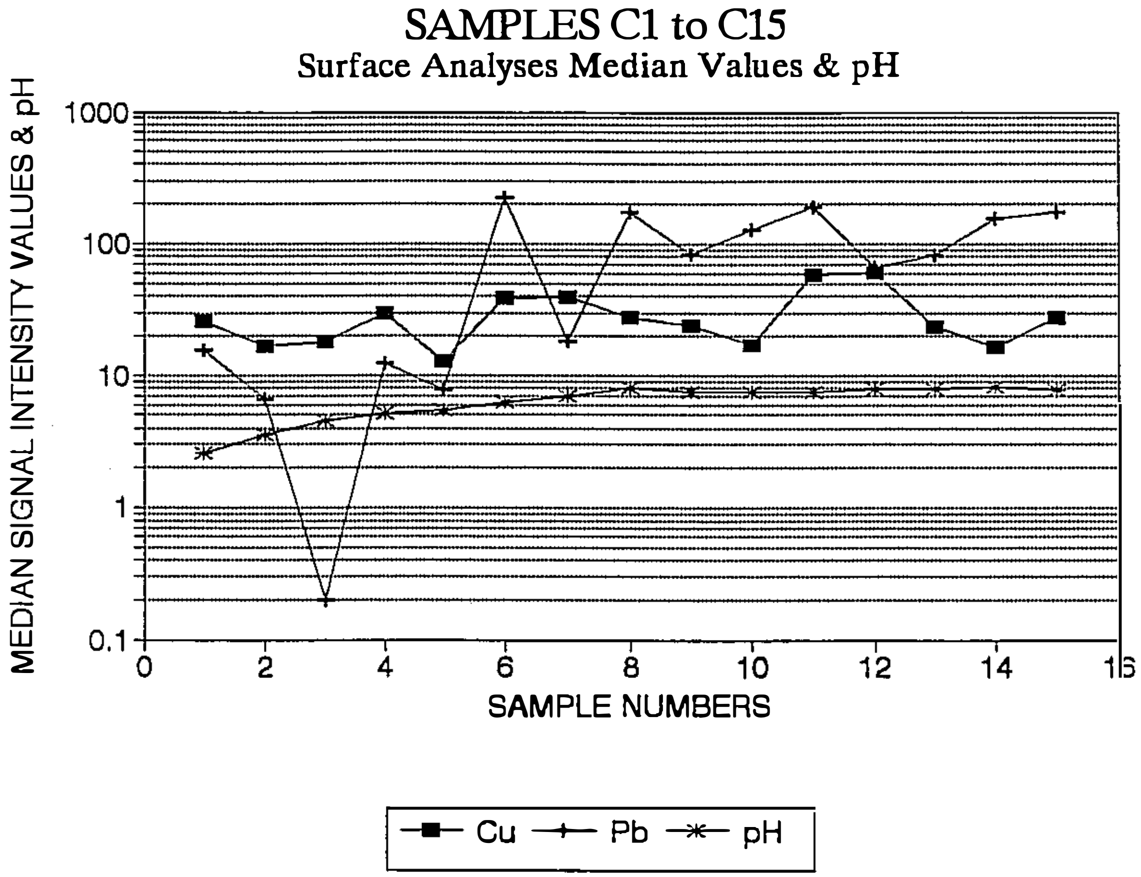

Figure 7. Summary illustrating pH versus relative degree of Cu- and Pb-activation for all 15 composite samples.

DISCUSSION AND CONCLUSIONS

Though Pb-activated sphalerite is a minor factor for the first five samples derived from the surface zones of the tailings pile with lowest pH, it becomes dominant in the other samples with neutral to slightly alkaline pH. This suggests that the liberated sphalerite grains in the tailings may represent grains that were Pb- and Cu-activated during the original processing.

The reactions leading to Pb-activation of sphalerite may be (after Fuerstenau, 1982):

$$PbS + 2O_2 \rightarrow PbSO_4 \rightarrow Pb^{2+} + SO_4^{2-} \quad (1)$$

and

$$ZnS + Pb^{2+} \rightarrow PbS \text{ (on sphalerite)} + Zn^{2+} \quad (2)$$

It is possible that the lower degree of Pb-activation evident on the sphalerite surface in the upper zones of the tailings pile may be due to dissolution of the PbS as indicated by equation 1. Analysis of water samples for Pb in the upper parts of the tailings pile may contribute to verification of this hypothesis.

ACKNOWLEDGEMENTS

The technical assistance of Messrs. M. Beaulne and D.R. Owens in polished section preparation, selection of sphalerite grains, and SEM photography is gratefully acknowledged.

REFERENCES

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May 20, 1993

Mr. G. McDonald
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M5J 2N7

Dear Godfrey:

As discussed I am enclosing descriptions of the Phase II work. The purpose of these descriptions, originally dated April 6 and May 11, 1993 is to present the to-date information on the on-going work on Curragh tailings retreatment. Please bear in mind that this is not a report and the data and information contained should be used for discussion only.

Sincerely,

Tad Cienski
Project Leader

TC:lp
Encl.

c.c. M.C. Campbell
C.A. Hamer

CURRAGH INC. TAILINGS RETREATMENT

Preliminary Description of Phase II Testwork
April 6, 1993

Statistical Design

Variables prescreening was carried out by performing bench flotation tests as required by a fractional statistical design. To obtain the initial information on effects 32 test runs were done studying 11 variable sat at two levels. Variables investigated and the levels used are listed below:

Variable		High	Low
A.	agitation intensity	1800 rpm	700 rpm
B.	agitation time	20 min.	5 min.
C.	slurry density	40%	35%
D.	aeration time	10 min.	5 min.
E.	alkalinity agent*	lime	soda ash
F.	lime/soda rate	3000 g/t	750 g/t
G.	$CuSO_4$	300 g/t	150 g/t
H.	SO_2	600 g/t	250 g/t
J.	NaCN	100 g/t	50 g/t
K.	collector type*	3418A	SAX
L.	collector level	35 g/t	20 g/t

*Lime and dithiophosphinate (3418A) were used when the design required as "+", while soda ash and xanthate substitute for "-".

Procedure

Reagentized slurries were agitated in high intensity conditioner. They were then transferred into an automated flotation cell where 4 concentrates were produced: 2 min, 2 min, 4 min, and 8 min for a total flotation time of 16 minutes. Also head and tails samples were removed from the cell.

Wet and dry weights of all the products were obtained, samples were pulverized and analyzed for Pb and Zn.

Table 1 shows test conditions (variables levels) for each of the 32 runs.

Table 1. Tests Variables and Their Levels

	Variables										
Test No.	A	B	C	D	E	F	G	H	J	K	L
1	−	−	−	−	−	−	−	−	−	−	−
2	+	−	−	−	−	+	−	−	+	+	−
3	−	+	−	−	−	+	+	−	−	−	+
4	+	+	−	−	−	−	+	−	+	+	+
5	−	−	+	−	−	+	+	+	+	−	−
6	+	−	+	−	−	−	+	+	−	+	−
7	−	+	+	−	−	−	−	+	+	−	+
8	+	+	+	−	−	+	−	+	−	+	+
9	−	−	−	+	−	−	+	+	+	+	+
10	+	−	−	+	−	+	+	+	−	−	+
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27	−	+	−	+	+	+	−	−	+	−	+
28	+	+	−	+	+	−	−	−	−	+	+
29	−	−	+	+	+	+	−	+	−	−	−
30	+	−	+	+	+	−	−	+	+	+	−
31	−	+	+	+	+	−	+	+	−	−	+
32	+	+	+	+	+	+	+	+	+	+	+

Results

Table 2 shows lead and zinc grades and recoveries in the combined bulk rougher concentrates.

Table 2. Variables pre-screening factorial design tests results

Test No.	Combined Conc.		Recovery %	
	% Pb	% Zn	Pb	Zn
1	1.23	4.01	54.8	42.7
2	1.50	4.91	58.7	59.9
3	1.73	3.65	56.8	42.8
4	1.30	1.95	35.3	26.6
5	1.58	3.96	16.5	39.3
6	1.28	2.03	44.1	49.2
7	1.93	3.52	50.7	21.2
8	1.61	4.78	30.0	45.4
9	1.46	2.92	38.6	28.6
10	1.33	3.10	56.8	33.2
11	1.44	3.31	51.4	32.7
12	1.26	2.94	57.0	46.5
13	1.52	4.24	14.2	44.8
14	1.24	4.45	24.1	40.0
15	1.28	2.59	50.5	26.4
16	1.55	3.83	26.1	41.6
17	1.21	2.09	54.4	23.5
18	1.42	3.57	38.3	37.8
19	1.49	3.13	46.4	44.9
20	1.34	2.38	43.6	17.1
21	1.80	7.03	13.0	59.2
22	1.20	3.01	43.0	62.7
23	1.33	3.64	29.3	32.7
24	1.29	2.06	38.6	50.7
25	1.39	2.91	55.0	21.3
26	1.37	3.05	49.8	46.8
27	1.79	3.02	53.4	28.4
28	1.44	5.50	16.0	37.8
29	1.63	3.25	16.4	30.6
30	0.88	1.63	59.9	28.4
31	1.06	1.97	63.3	11.5
32	1.36	2.89	59.3	48.7

Statistical analysis of results were carried out in respect of zinc recovery. The main conclusion concerning process conditions needed for increased recovery of zinc are:

A. agitation intensity: high
B. agitation time: low
C. slurry density: high
D. aeration time: immaterial
E. soda/lime: lime
F. level soda/lime: high
G. $CuSO_4$: low
H. SO_2: detrimental
J. NaCN: immaterial
K. collector type: dithiophosphinate
L. collector level: preferred high

Within the investigated parameter space, only intensity, conditioning time, aeration time, soda versus lime and sulphur dioxide levels have been found to have influence on zinc recovery. The only parameters to have a positive effect on zinc recovery are intensity of agitation and the level of lime or soda used.

Except for B, the conditioning time, which has a significance level of only 91%, the remaining four parameters are all significant at higher that 95%. Under the present conditions and within the investigated parameter space the highest practically possible Zn recovery is 57.5% ±1.6%.

Regarding the parameter screening tests the following could be concluded with a very high certainty:

1. Only: Intensity
   Agitation time
   Aeration time
   Level of soda or lime
   Level of sulphur dioxide

have to have statistically significant effect on zinc recovery;

2. Within the multi-parameter space the highest Zn recovery expected is 57.5 ±1.6%. There is a physical constraint that limits the value to which the intensity of agitation can be increased. There is also a minimum conditioning time required for an interaction between mineral surfaces and reagents. The complete elimination of aeration time and sulphur dioxide from the process and an increase of soda or lime addition beyond 3000g/t may under favourable conditions improve zinc recovery by further 7% to about 62% to 65% at best.

The main effect plots for each of the significant parameters are shown on the following pages.

Statistical analysis of results were carried out in respect of zinc recovery. The main conclusion concerning process conditions needed for increased recovery of zinc are:

A. agitation intensity: high
B. agitation time: low
C. slurry density: high
D. aeration time: immaterial
E. soda/lime: lime
F. level soda/lime: high
G. $CuSO_4$: low
H. SO_2: detrimental
I. NaCN: immaterial
J. collector type: dithiophosphinate
K. collector level: preferred high

Main effect line graphs and interaction cube graphs illustrating effects of variables on the recovery of zinc are included,together with the table of coefficients.

Confirmatory tests in triplicate were carried out using the recommended set of conditions aimed at a high range of zinc recovery. Results of these tests are not available at the time of writing.

Statistical analysis of pre-screening data will be carried out with respect of lead recovery and lead and zinc recoveries together.

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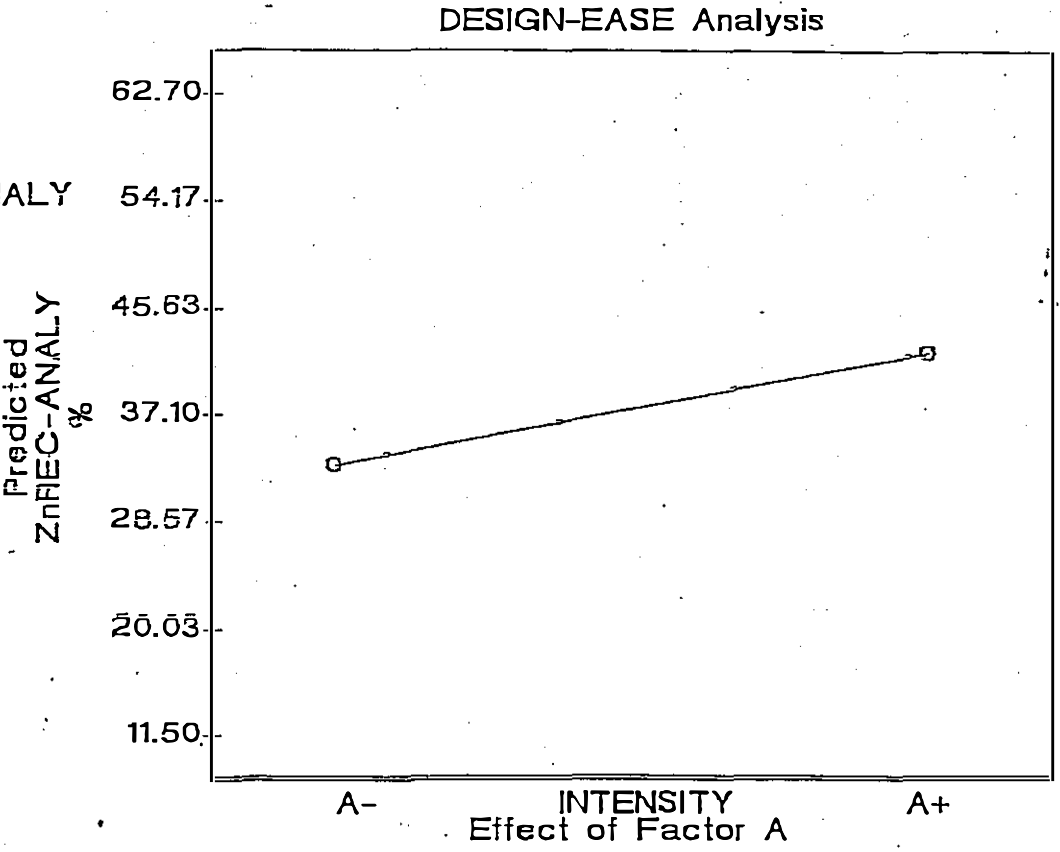
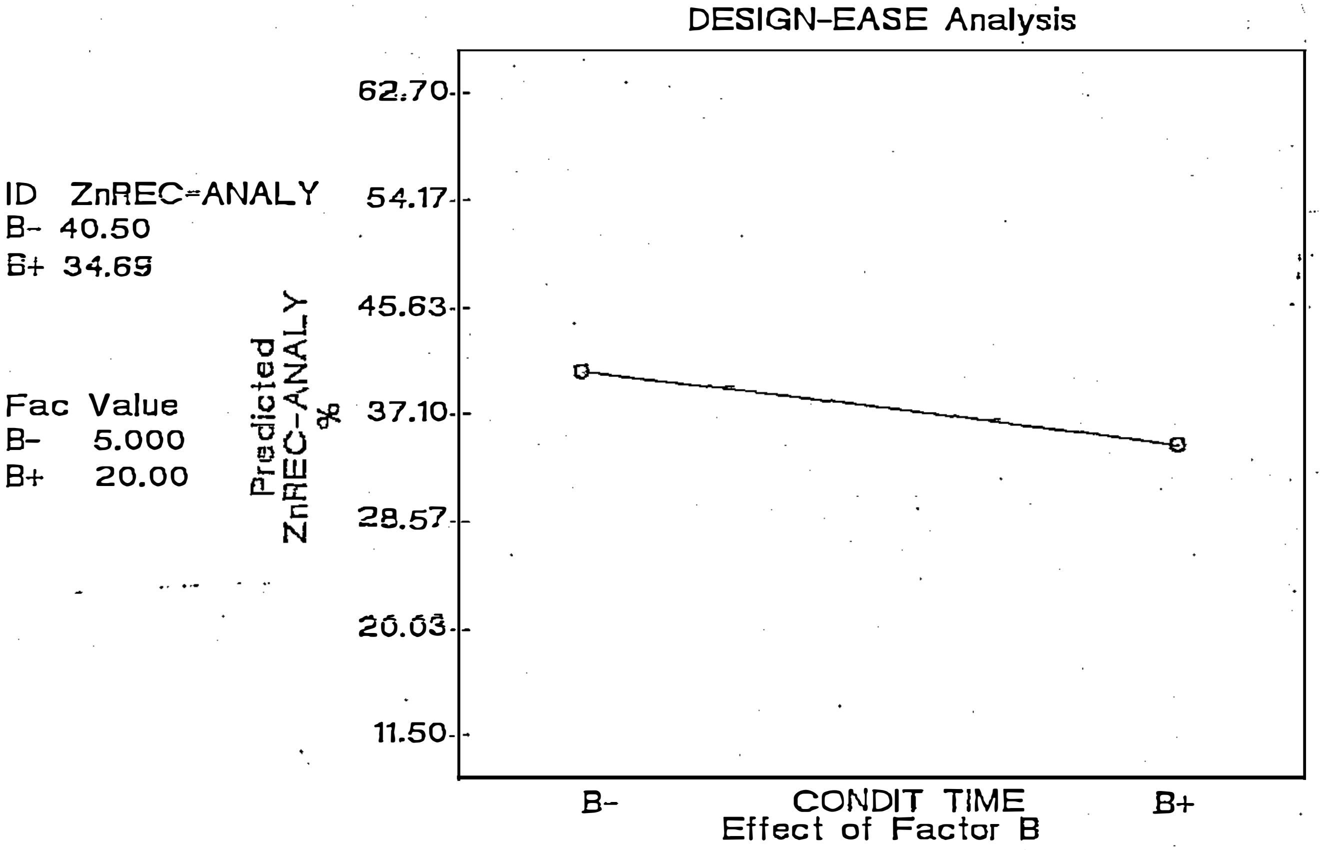
A- 33.16
A+ 42.03

Fac	Value
A-	700.0
A+	1800

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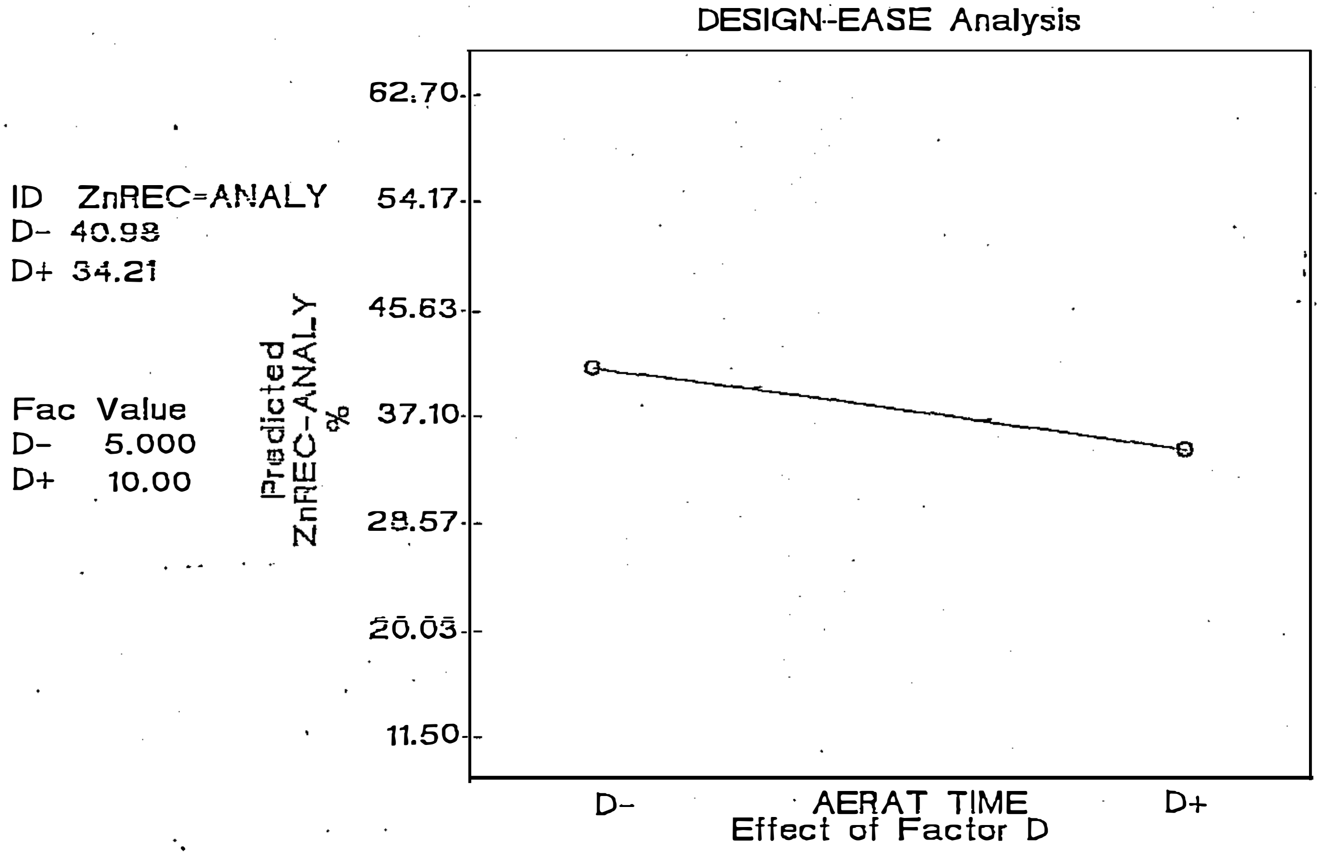
Effect of Factor B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D-
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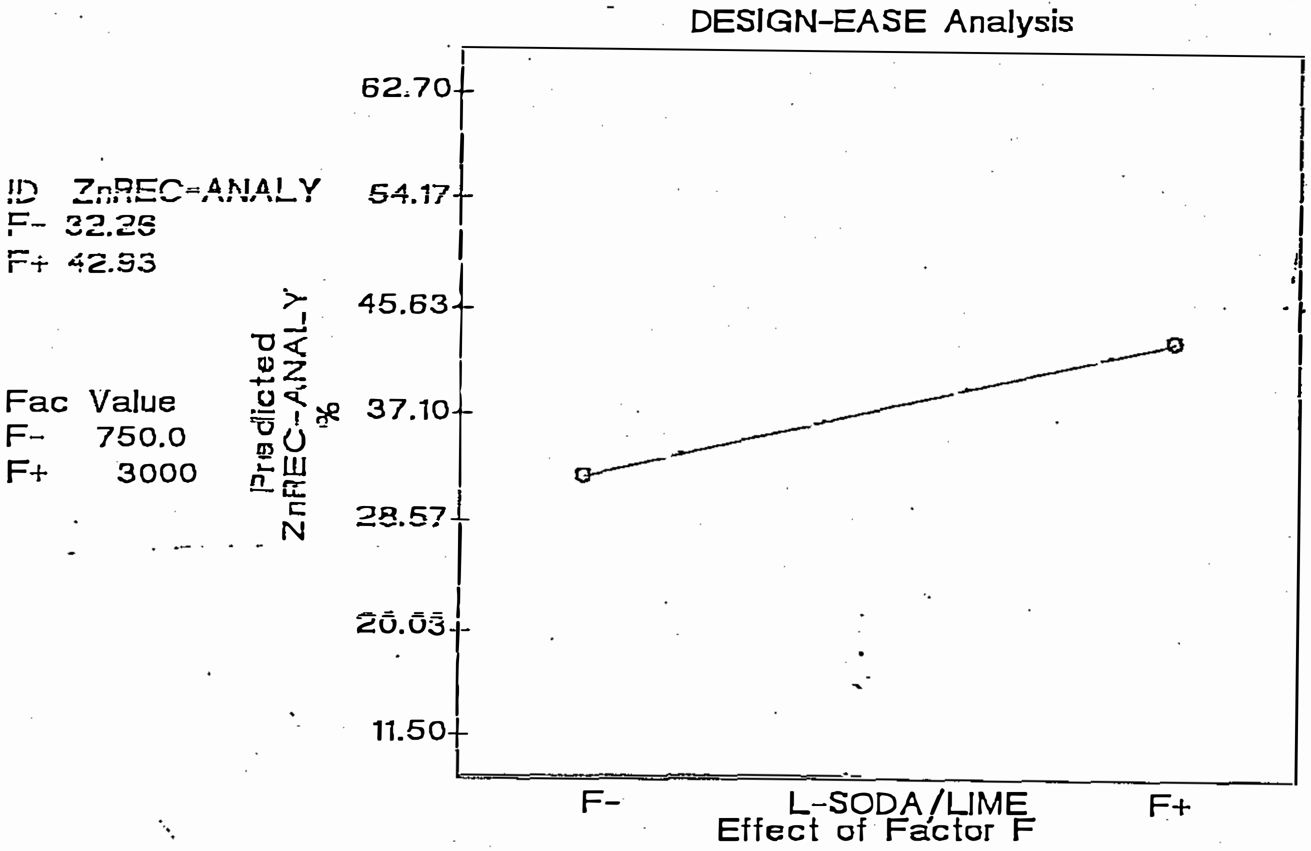
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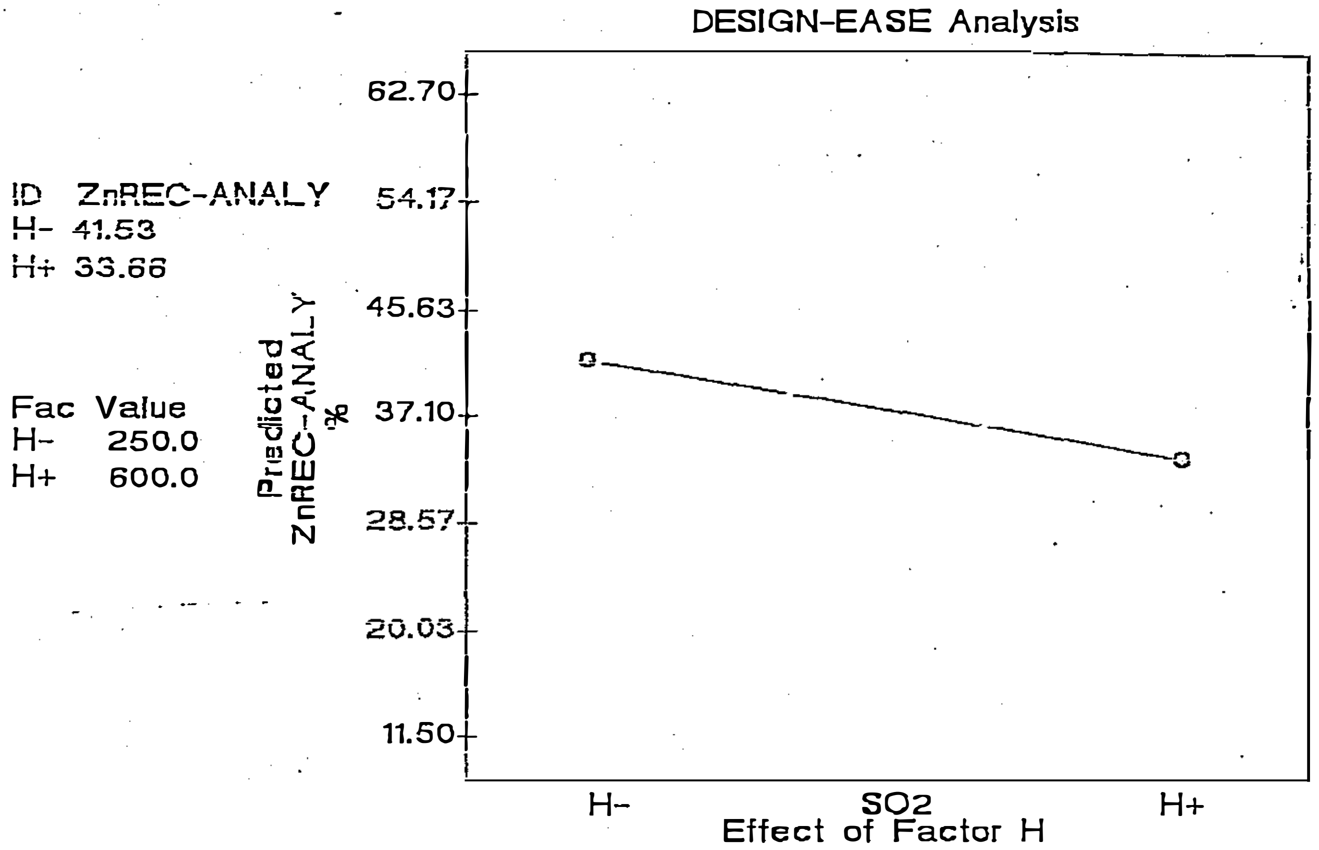
F+
Effect of Factor F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H- 250.0
H+ 600.0
Predicted ZnREC-ANALY %
62.70
54.17
45.63
37.10
28.57
20.03
11.50
H-
SO2
H+
Effect of Factor H

Investigation based on Lakefield Research procedure

In addition to the statistical design study of 11 variables, two sets of tests were performed taking as a basis flotation conditions as reported by Lakefield Research when investigating Curragh tails retreatment. Three minute regrind at varying pH was followed with low intensity stage conditioning. Minerc M27 in combination with sodium isopropyl xanthate, were used as collectors. Zinc recoveries of 65% to 70% were obtained when low pH (~6.0) was applied.

In agreement with Curragh it was decided to investigate the possibility of replacing the effect of regrinding by slurry agitation. A statistical design set of tests was carried out with three variables (agitation intensity and time and slurry density) at two levels. This design required 11 runs which included 3 centre runs.

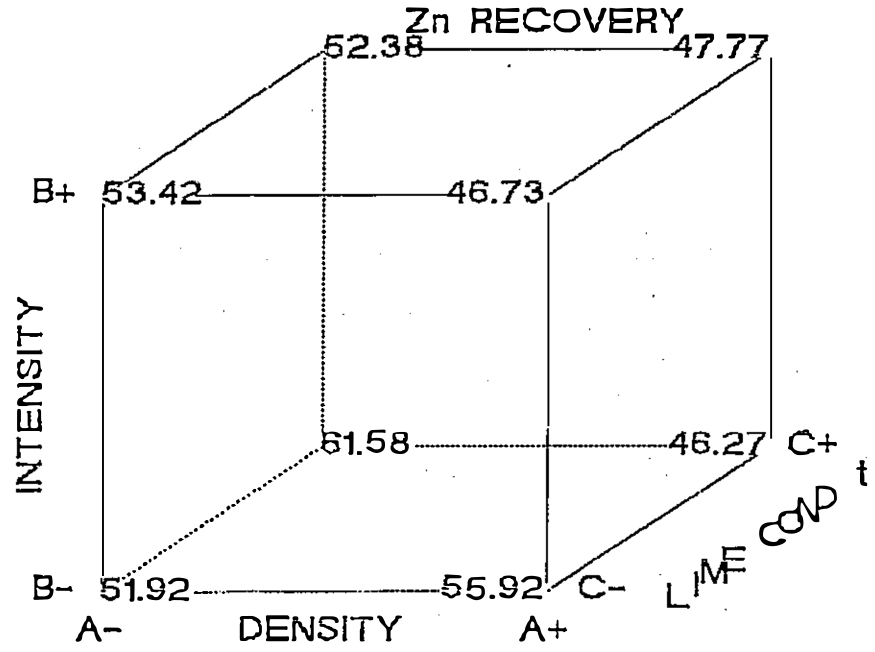
Results of the tests are shown in Table 3 on the following page while the conclusions obtained from the statistical analysis of the data are illustrated in Cube Plot. This plot indicates that for the conditions tested, best zinc recovery is obtained at low slurry density and low intensity of agitation and with the extended agitation time. Under those circunstances zinc recoveries of over 60% can be achieved.

Table 3. Results from the three variables, two level test design

ESIGN-EASE: Run Report
ata file: CURRAGH2.DAT

	Run		A	B	C	R1	R2	
			DENSITY	INTENSITY	LIME COND t	Zn RECOVERY	ZnCON GRADE	Dsn
Obs	Ord	Blk	%	RPM	min	%	%Zn	ID
3	1	1	35.000	1800.0	5.000	50.800	4.7800	3
10	2	1	50.500	1250.0	12.500	53.600	3.0500	0
4	3	1	66.000	1800.0	5.000	45.800	4.1400	4
9	4	1	50.500	1250.0	12.500	49.800	2.9100	0
2	5	1	66.000	700.0	5.000	55.000	4.8200	2
7	6	1	35.000	1800.0	20.000	53.300	3.1600	7
5	7	1	35.000	700.0	20.000	62.500	2.5400	5
6	8	1	66.000	700.0	20.000	45.500	4.8800	6
11	9	1	50.500	1250.0	12.500	56.400	3.3400	0
8	10	1	66.000	1800.0	20.000	50.400	2.7100	8
1	11	1	35.000	700.0	5.000	52.700	5.8600	1

DESIGN-EASE Analysis
Cube Plot of Predicted Values



CANMET PROJECT 584

Curragh Inc. Tailings Retreatment Update Description of Phase II Testwork

May 11, 1993

Mineralogical examination was carried out on two rougher concentrates and tails obtained from tests that gave highest and lowest zinc recoveries in a series of tests investigating roughing parameters. Examination shows the presence of significant proportion of free sphalerite in tails. It also shows that sphalerite coarser than 53 micrones (270 mesh) is not recovered (absent in concentrate) at short (5 minutes) agitation time. At the agitation extended to 20 minutes 36% of liberated sphalerite in +53 micron size fraction reported into concentrate. As a conclusion of this examination it can be stated that as a whole sphalerite is poorly activated and further work should focus on this aspect.

Influence of regrinding and pH in agitation and rougher flotation was investigated in detail. The results shows that at the ranges tested neither of these parameters has a significant effect on Zn or Pb recovery into the rougher concentrate.

A series of tests covered initial cleaning steps, with pH as the variable under investigation. Results obtained indicate that a neutral pH of agitation and rougher flotation (as against slightly acidic pH values) facilitates cleaning by improving depression of pyrite without a loss in zinc recovery. Further work will focus on pyrite depression.

Several tests were carried out on the material that was allowed to dry before repulping. It was observed that substantial changes in the chemistry of the solids took place as a result of the drying, significantly influencing its response to flotation. This aspect seems to be important and will need to be addressed.

A total of 83 flotation tests were conducted to date.

Zinc recoveries from the blended feed (25% <pH 5; 25% pH 5-7 and 50% pH >7) are mostly in the range between 50% and 60%.

The following testwork is now planned:

1. Two stage rougher flotation to allow for the presence of uncoated sphalerite particles and coated by lead sulphide (see report on surface chemistry);
2. Use of a new, stronger sphalerite collector;
3. Use of NaHS for the depression of pyrite in cleaning stages.