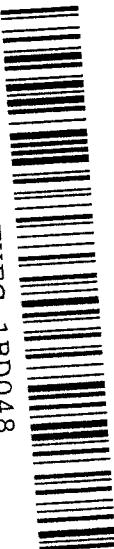


**GEOCHEMICAL CHARACTERIZATION OF
RAFFINATE AND SHALE
FROM THE DURITA URANIUM-RAFFINATE
DISPOSAL SITE**



RRMOTHEC 1BD048

GEOCHEMICAL CHARACTERIZATION OF
RAFFINATE AND SHALE FROM THE DRITA UR/
STORAGE LOC: [REDACTED] DOCDATE: 02/11/1993

RAD-42

Prepared for:

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Project No. 33-0014

February 11, 1993

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1.0 INTRODUCTION

The evaporation ponds located at the Durita disposal facility hold liquid waste from uranium and vanadium processing. Sulfuric acid leach liquors were treated for uranium and vanadium recovery using conventional solvent extraction procedures. The raffinate from the solvent extraction circuit was discharged as waste into the evaporation ponds. Over the years, this raffinate liquid waste has evaporated, leaving behind an acid solution saturated in the inorganic salts that were either leached from the ore rock or introduced during hydrometallurgical processing.

GEOCHEM, a Division of Terra Vac, was asked to review and interpret laboratory analyses, and to supervise scoping testwork on raffinate samples and shale samples from the Durita uranium-raffinate disposal site. The objective of the geochemical analyses and testwork was three-fold:

- (1) characterize the chemical and radiochemical composition of the raffinate stored in the evaporation ponds;
- (2) characterize the mineralogical and geochemical properties of locally-available Mancos shale; and
- (3) perform bench-scale testwork to determine whether the Mancos shale could be used to neutralize and stabilize the raffinate liquids so as to effect in-place remediation of the evaporation ponds in an environmentally safe manner.

2.0 RAFFINATION CHARACTERIZATION

The raffinate stored in the evaporation ponds at the Durita site consists of three liquid and semi-liquid phases: an acidic solution concentrated in dissolved salts; a very wet salt-crystal phase; and an intermediate gelatinous phase of previously unknown composition. Samples of the three phases were collected by Dr. Alan Kuhn of AK GeoConsult and shipped under chain-of-custody to Accu-Labs Research, Golden, Colorado, for chemical and radiochemical analyses. The results of the analyses are summarized in Tables 1 and 2; the actual analytical reports from Accu-Labs Research are included as Appendix A to this report.

2.1 Chemical Composition of Raffinate Samples

Chemical analyses of the raffinate phases indicate that the solution phase and the gelatinous phase are very similar in composition. The liquid and semi-liquid can be characterized as an acidic, saturated, magnesium-sodium-aluminum sulfate solution containing traces of ammonia and nitrate, arsenic, cadmium, lead and zinc. The salt crystal phase differs from the more-liquid material only by the absence of magnesium. Magnesium is apparently not incorporated into the secondary minerals that form upon evaporation of the raffinate.

The similarity in composition between the solution phase and gelatinous phase samples is shown in the trilinear and Stiff diagrams of Figures 1 and 2. The trilinear plotting technique, as illustrated in Figure 1, consists of a graphic presentation of the major-ion concentrations in solutions. The relative percentages of the cations and anions are plotted in corresponding cation and anion triangles at the bottom of the trilinear diagram. The locations of these points are then projected and plotted on a central plotting rhomb which defines a solution's characteristic composition. The Stiff diagram shown in Figure 2 is another way of plotting the major-ion concentrations of solutions to illustrate characteristic composition.

The plotting location of the various raffinate samples confirms the chemical similarity between the solution and gelatinous phase samples. The major-ion composition of the salt crystal material, and notably the absence of magnesium, locates the plotting position on the trilinear diagram away from the solution and the gelatinous material (Figure 1). The geometric shapes of the Stiff diagrams corresponding to the solution phase samples and gelatinous phase samples are essentially the same (Figure 2). The geometric shape of the salt crystal samples is similar, except for the absence of a magnesium component to the geometric shape.

2.2 Radiochemical Composition of Raffinate Samples

Radiochemical analyses of the raffinate phases indicate that the gelatinous phase and salt crystal samples are elevated in radionuclide content compared to the solution phase samples. This observation is reasonable, considering the tendency of radium and thorium to coprecipitate with various secondary minerals that form as the raffinate solution evaporates.

TABLE 1

CHEMICAL CHARACTERIZATION OF RAFFINATE SAMPLES

Sample No.	Sample Description	Solution Phase				Gelatinous Phase			Salt Crystal Phase	
		1A	3A	4A	1G	3G	4G	1S	3S	4S
Constituents:										
pH (pH Units)	2.9	2.8	2.5	3.1	2.5	2.8	2.9	2.9	2.8	2.8
TDS (%)	37	32	57	80	63	63	67	68	69	69
Organic Carbon (%)	<0.05	0.011	<0.05	0.13	0.08	0.06	0.06	0.05	<0.05	<0.05
Major Ions (mg/l):										
Sodium	22,000	18,000	29,000	18,000	2,200	25,000	29,000	32,000	32,000	32,000
Potassium	50	5	8	1,100	240	390	1,100	1,100	1,100	320
Calcium	380	380	320	9,900	3,200	1,400	3,000	6,300	6,900	6,900
Magnesium	38,000	26,000	33,000	29,000	31,000	25,000	950	1,000	1,000	1,000
Aluminum	17,000	18,000	22,000	28,000	25,000	30,000	43,000	42,000	42,000	37,000
Iron	340	140	520	13,000	3,300	2,200	5,800	7,700	7,700	3,200
Manganese	1,900	1,400	3,400	820	1,200	1,600	950	1,000	1,000	1,000
Chloride	1,900	16,000	37,000	530	7,100	11,000	7,100	11,000	11,000	11,000
Sulfate	280,000	250,000	280,000	98,000	190,000	330,000	330,000	330,000	330,000	440,000
Silica	190	<50	<50	220	81	130	120	160	160	82

TABLE 1

CHEMICAL CHARACTERIZATION OF RAFFINATE SAMPLES (Continued)

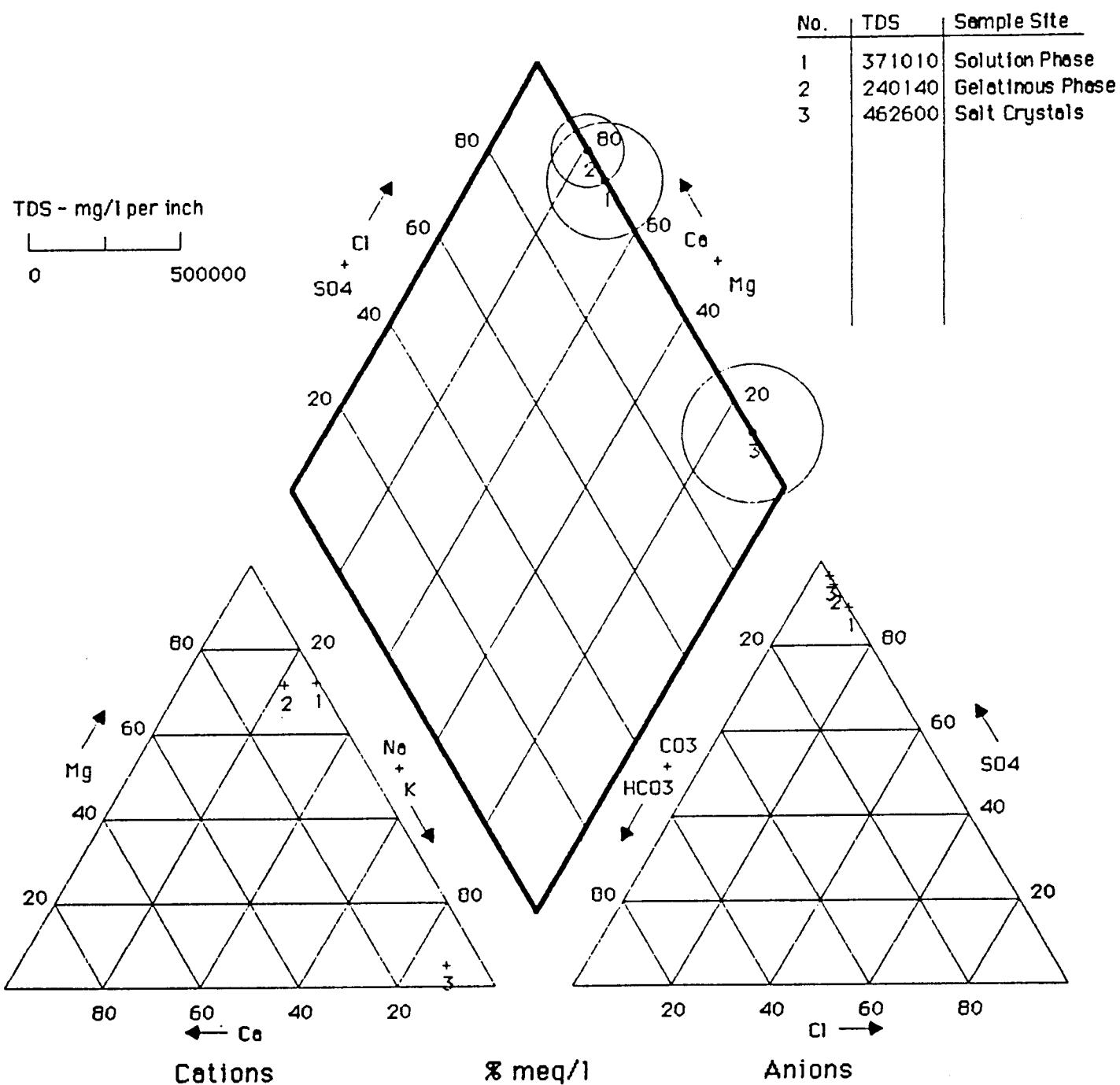
Sample Description	Solution Phase				Gelatinous Phase		Salt Crystal Phase		
Sample No.	1A	3A	4A	1G	3G	4G	1S	3S	4S
Trace Elements (mg/L):									
Ammonia (as N)	500	590	180	290	150	340	1,100	500	250
Nitrate (as N)	35	35	91	28	37	44	18	24	20
Arsenic	27	23	50	74	38	45	60	70	40
Cadmium	36	28	82	15	23	36	18	21	16
Molybdenum	<1	<1	<1	0.9	<0.5	<0.5	<1	<1	<1
Lead	9	8	8	6.2	4.8	5.2	13	21	9
Zinc	1,300	930	1,600	780	930	900	940	850	1,200

TABLE 2

RADIOCHEMICAL CHARACTERIZATION OF RAFFINATE SAMPLES

Sample No.	Sample Description	Solution Phase				Gelatinous Phase				Salt Crystal Phase			
		1A	3A	4A		1G	3G	4G		1S	3S	4S	
Constituent:													
Uranium (mg/kg)		120	100	2.0		44	94	63		25	32	22	
Gross Alpha (pCi/g)	84±17	120±30	220±20		3700±100	1200±100	1000±100		610±50	1400±100	540±50		
Gross Beta (pCi/g)	7±15	17±24	28±6		93±27	18±17	58±22		22±18	39±21	11±5		
Thorium-230 (pCi/g)	220±10	370±10	570±20		2600±100	1200±100	1400±100		1200±100	1900±100	600±20		
Radium-226 (pCi/g)	0.6±0.2	0.0±0.2	0.4±0.2		16±1	6.5±0.4	5.5±0.4		7.2±0.5	12±1	3.6±0.4		
Lead-210 (pCi/g)	1.9±0.6	0.1±0.4	1.0±0.7		6.1±0.8	6.1±0.8	4.8±0.8		7.2±1.3	9.3±0.9	3.9±0.7		
Polonium-210 (pCi/g)	0.4±0.2	0.5±0.5	1.1±0.4		9.0±1.4	2.0±0.5	2.4±0.7		6.5±1.0	4.1±0.7	2.0±0.5		
Thorium-232 (pCi/g)	0.1±0.2	0.80.5	0.1±0.2		9.6±1.9	0.8±0.9	0.2±0.4		0.2±0.8	1.7±1.6	0.6±0.8		
Radium-228 (pCi/g)	0.0±1.0	0.0±1.0	2.1±0.6		0.0±0.9	0.0±1.0	0.0±1.1		1.7±0.6	0.0±1.5	0.0±0.8		

RAFFINATE SAMPLES



PROJECT: Durita Evaporation Ponds

DATE: January 18, 1993

GEOCHEM, A Division of
Terra Vac

Lakewood, Colorado

Figure 1

Trilinear Diagram of raffinate samples

RAFFINATE SAMPLES

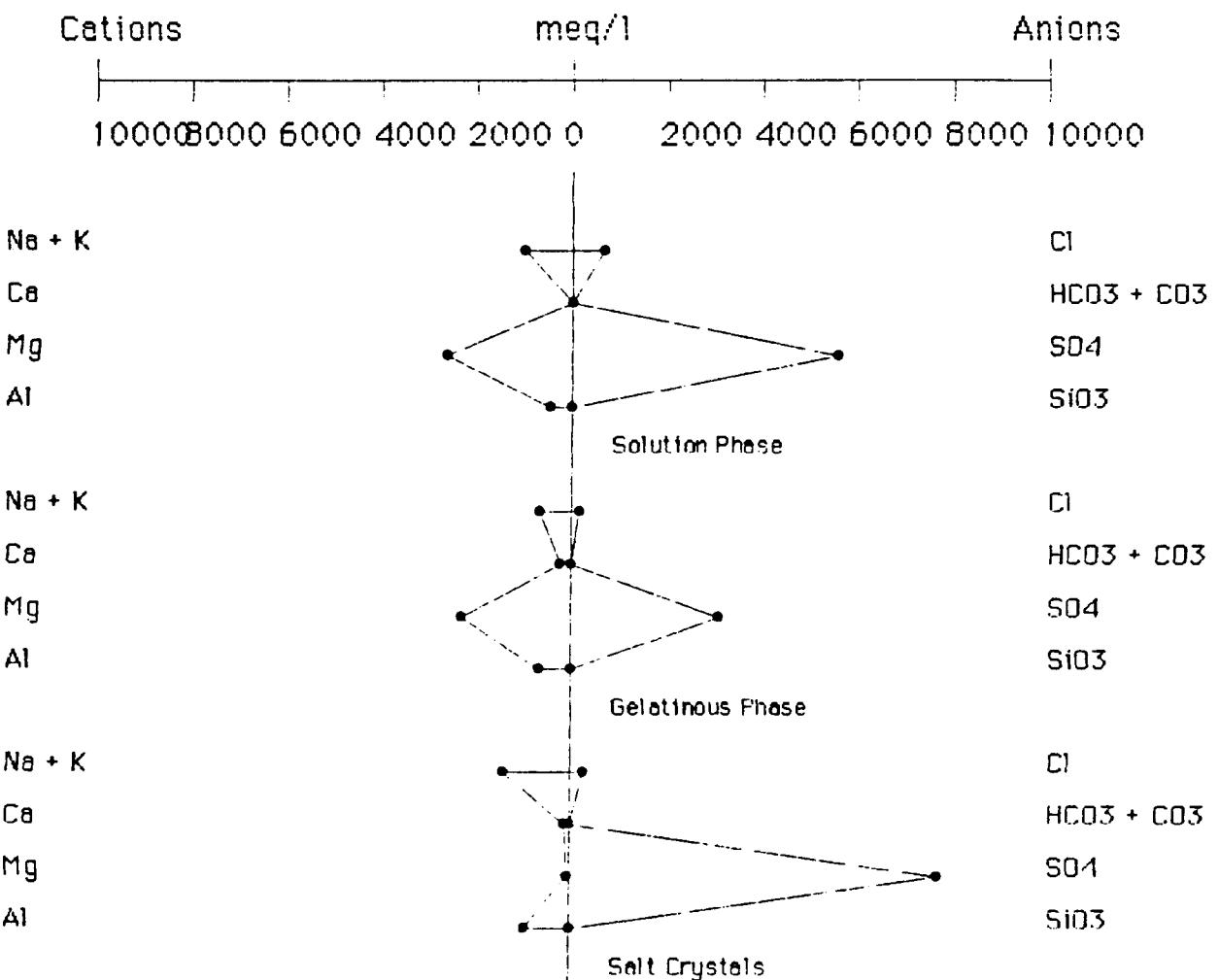


Figure 2 Stiff diagram of raffinate samples

3.0 MANCOS SHALE CHARACTERIZATION

Two samples of Mancos shale were collected by Dr. Alan Kuhn from the immediate vicinity of the evaporation ponds. The samples were shipped under chain of custody to Accu Labs Research for mineralogical and geochemical analysis. The mineralogical analysis was completed at The Mineral Lab in Lakewood, Colorado; the geochemical analyses were performed at Accu Labs Research. Analytical reports on the Mancos shale samples are included in Appendix A.

The results of x-ray diffraction analysis are summarized in Table 3 and indicate material which would be expected to neutralize the acidity of the raffinate. Bulk-mineral analysis showed calcite (CaCO_3) content between 50 and 60 percent by weight, and clay-mineral content between 15 and 31 percent by weight. The principal clay-forming minerals include kaolinite, illite, and sepiolite.

The geochemical properties of the Mancos shale samples are summarized in Table 4 and indicate a material that would be expected to interact favorably with the raffinate, neutralize acidic pH and, in the process, immobilize potential ground-water contaminants including heavy metals and natural radionuclides. Neutralizing the acidity of the raffinate would render potential contaminants insoluble through any number of chemical reactions, including precipitation and coprecipitation. Secondly, the near-neutral pH conditions that would result from the shale/raffinate interaction would promote geochemical reactions that bind and fix potential contaminants to the shale. Geochemical mechanisms affecting this fixation include, for example, cation and anion exchange with kaolinite and illite clays, or sorption onto the traces of iron and manganese hydrous oxides or organic matter present in the shale. The effectiveness of such mechanisms is well known.

TABLE 3

Mineralogical Properties of Mancos Shale SamplesBulk Mineralogy (Wt %):

	<u>9014 K Mancos 1</u>	<u>9014 K Mancos 2</u>
Calcite	60	50
Quartz	14	15
Kaolinite	15	7
Mica/Illite	<10	14
"Unidentified"	<5	<5
Clay-Size Material (<2 µm)	6	6

Clay-Size Fraction (Wt %):

Calcite	27	10
Quartz	<5	<5
Kaolinite	30	50
Mica/Illite	20	20
Sepiolite	15	15
"Unidentified"	<5	<5

$$\begin{aligned}
 & \text{est. clay \%} \\
 & = \sum (\text{cl. min. + clay size \%}) \\
 & = 15 + (<10)/2 + 6 \times \\
 & \quad \frac{(30+20+15)}{100} = 23.9\%
 \end{aligned}$$

$$\begin{aligned}
 & = 7 + 14 + 6 \times \frac{(5.5+20+15)}{100} \\
 & = 26.1\% \text{ for } \# 2 \\
 & \text{ave} = 25\%
 \end{aligned}$$

TABLE 4
Geochemical Properties of Mancos Shale Samples

<u>Geochemical Parameters</u>	<u>9014 K Mancos 1</u>	<u>9014 K Mancos 2</u>
Particle Size/Texture		
% Sand	36	38
% Silt	45	46
% Clay	19	16
Acid Neutralizing Potential (% CaCO ₃)	64	50
Soil pH (pH units)	7.4	7.7
Cation-Exchange-Capacity (meq/100g)	23	21
Exchangeable Cations:		
Calcium (meq/100g)	66 (1)	76(1)
Magnesium (meq/100g)	1	4
Sodium (meq/100g)	<1	<1
Potassium (meq/100g)	2	2
Organic Carbon Content (%)	0.34	0.34
<u>Radiochemical Characterization</u>		
Gross Alpha (pCi/g)	14±6	10±6
Radium-226 (pCi/g)	2.8±0.3	1.8±0.3
Radium-228 (pCi/g)	0.9±0.5	0.9±0.5

(1) Anomalously high exchangeable calcium due to CaCO₃ interference in analytical method.

4.0 NEUTRALIZATION AND STABILIZATION TESTWORK

Bench-scale laboratory testwork was initiated at IC Technologies, Wheat Ridge, Colorado, to ascertain whether mixing raffinate with Mancos shale would neutralize and stabilize the raffinate, converting the saturated solution into a solid, low-permeability mixture. Successfully demonstrating that the raffinate stored in the Durita evaporation ponds could be treated with locally-available Mancos shale would offer an environmentally safe alternative to remediating the raffinate ponds. Final disposition of the raffinate could take place on-site, eliminating the need to concentrate chemical and radiochemical contaminants into another waste product that would require off-site transportation and disposal.

4.1 Stabilization Testwork

Mixtures of crushed (minus 1/4-inch) Mancos shale and raffinate solution were prepared in beakers. The mixtures represented different volume ratios of shale to raffinate, ranging from 0.33 to 1.0. The effects of the shale addition on the raffinate were noted. As expected, the reaction between the shale and raffinate was rapid and intense, evolving carbon dioxide gas as the calcium carbonate in the shale reacted with sulfuric acid in the raffinate. Observations from the beaker tests were as follows:

- crushed Mancos shale was very effective at neutralizing the acidic pH of the raffinate; pH neutralization occurred in shale to raffinate mixtures at volume ratios of 0.33 and above;
- for shale to raffinate mixtures at volume ratios of 0.44 through 0.67, the mass solidified after 72 hours of standing;
- solidification occurred within 24 hours with shale to raffinate mixtures at volume ratios of 0.83 and higher.

Photographs of the solidified mass are appended to this report.

A bulk sample of shale/raffinate mixture at a 0.50 volume ratio was prepared by IC Technologies for shipment to Vinyard & Associates, Albuquerque, New Mexico. The sample will be utilized in follow-up geotechnical testwork.

During the beaker tests, the reaction of the Mancos shale with raffinate evolved copious amounts of carbon dioxide gas. At the same time, an increase in the volume of the mixtures, estimated between 10 and 25%, was noted as the reaction went to completion. In order to quantify the amount of gas liberated as well as the volume change caused by the reaction, the beaker test with a shale/raffinate mixture at 0.50 volume ratio was repeated. Careful measurements of weight changes and volume changes were taken which are reported below:

Mancos Shale to Raffinate Mixture of 0.50:1.0 by Volume

	<u>Volume</u>	<u>Weight</u>
Raffinate	1,000 ml	1,284 gm
Shale	495 ml (cm^3)	750 gm
Initial Mixture (@ Time 0 Hours)	1,475 ml	2,034 gm
After 2 Hours	1,700 ml	1,980 gm
After 6 Hours	1,750 ml	1,974 gm

Volume Increase (%)

After 2 Hours $(225/1,475) = 15\%$

After 6 Hours $(275/1,475) = 19\%$

Weight Decrease(%)

After 2 Hours $(54/2,034) = 3\%$

After 6 Hours $(60/2,034) = 3\%$

At the end of the reaction, after 6 hours into the test, the volume of the Mancos shale/raffinate mixture increased by about 19%, and the weight of the mixture

decreased by 3% when compared to the initial volume and weight measured at the start of the test.

4.2 Leaching Testwork

Sequential batch-contact leaching tests were conducted on solidified material representing a shale to raffinate volume ratio of 0.50. In the sequential batch testwork (SBT) procedure, weighed amounts of solidified material were contacted with measured volumes of demineralized water, and the chemical constituents solubilized from the solidified material were determined by chemical analyses of the resultant leachate solutions. The SBT procedure simulates continuously leached columns, wherein, successive leach solution (demineralized water) comes in contact with fresh solid material (shale/raffinate solidified mass).

The contact was accomplished by mixing demineralized water with solidified material at desired liquid to solids ratios. The resulting slurries were gently agitated in containers over a period of 24 hours. At the conclusion of each 24-hour period, the slurry was decanted and filtered. The solids and appropriate volume of leachate solution from the previous contact were advanced in sequence according to the matrix illustrated in Figure 3. Leachate solutions from each batch contact were analyzed for the following chemical parameters and constituents (on a dissolved basis):

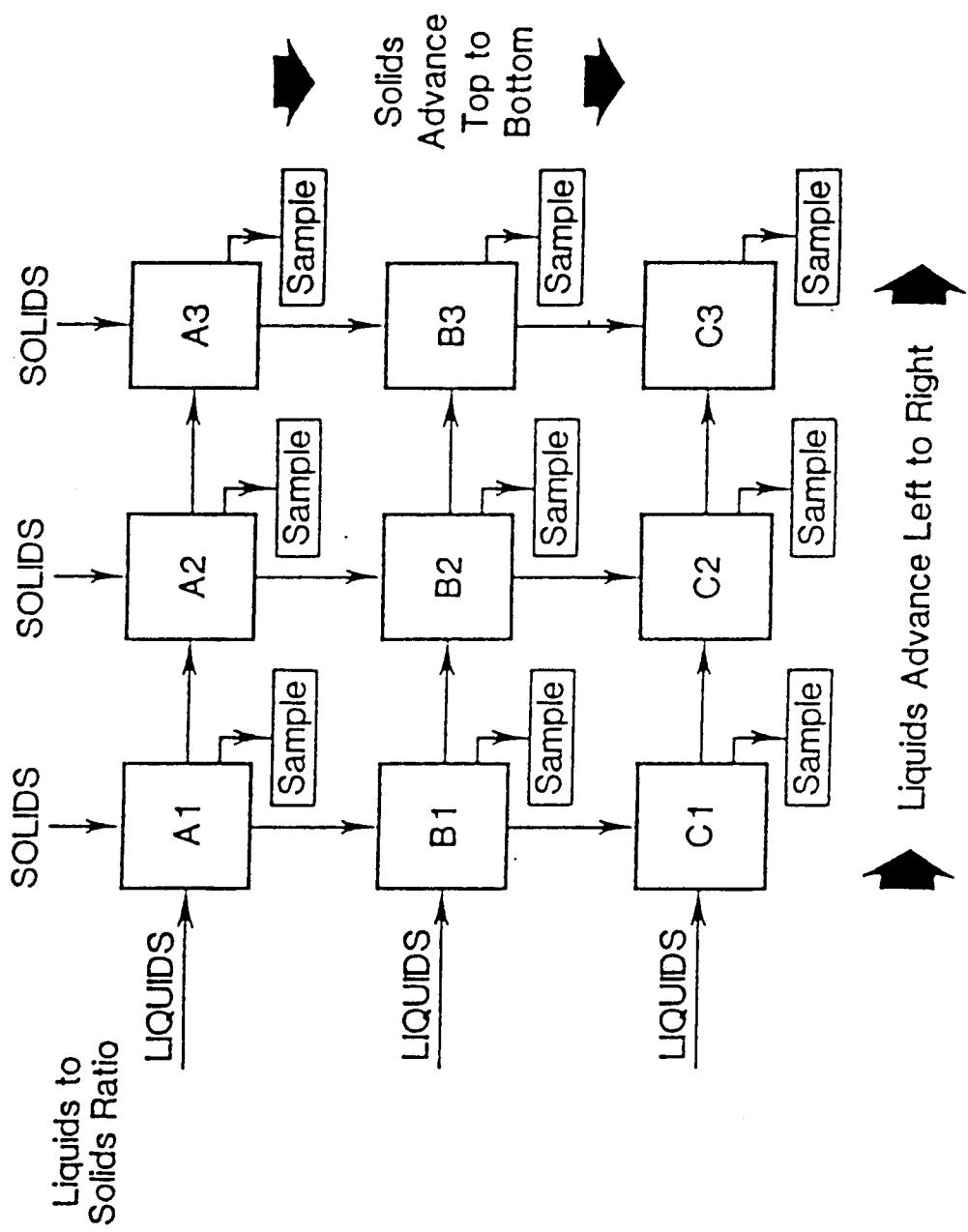
- pH and TDS;
- calcium, magnesium, sodium, chloride and sulfate;
- ammonia and nitrate; arsenic, cadmium, iron, manganese, lead and zinc.

The results of the analyses are presented in the analytical reports included in Appendix A. Figure 4 through 12 summarize the analyses completed on the leachate samples for selected chemical parameters and constituents.

Analyses of the leachate samples from the leaching testwork indicated the following:

- multiple contacts with demineralized water consistently gave leachate solutions that were near-neutral in pH, ranging between 6.9 and 7.6 (Figure 4);
- much of the sulfate in the original raffinate solution was precipitated and immobilized by the reaction with the shale material; leachate solutions were consistently lower in sulfate than the starting raffinate (Figure 5);
- reaction of the raffinate with shale precipitated and immobilized much of the ammonia and nitrate, and most of the arsenic, cadmium, iron, manganese and zinc present in the raffinate from the Durita evaporation ponds. (Figures 6 through 12).
- reaction of the raffinate with shale had nominal effects on the solubility of conservative ions such as sodium, magnesium, and chloride.

Leachate samples were not analyzed for radionuclides. However, laboratory and field experience has repeatedly shown that thorium and radium are rapidly immobilized when acidic raffinate is pH-neutralized through contact with calcareous clay-bearing material such as natural clay liners. Thorium, for example, is prevented from migrating in ground water by cation-exchange reactions with clay minerals and natural organics found in aquifer material; radium is attenuated in its movement by precipitation and coprecipitation reactions with sulfate ion, for example. Numerous other geochemical reactions can contribute to essentially trapping the radionuclides in the subsurface.

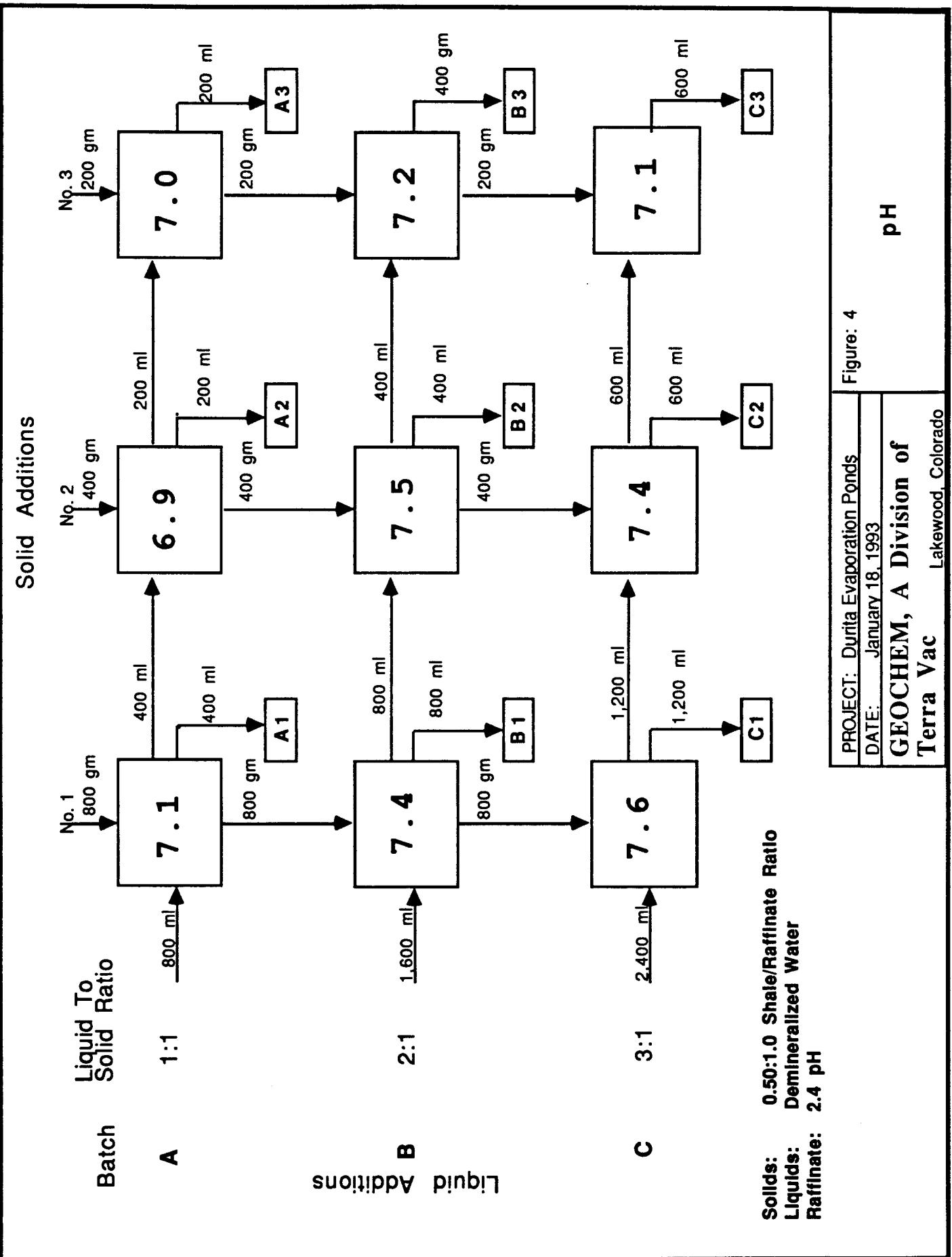


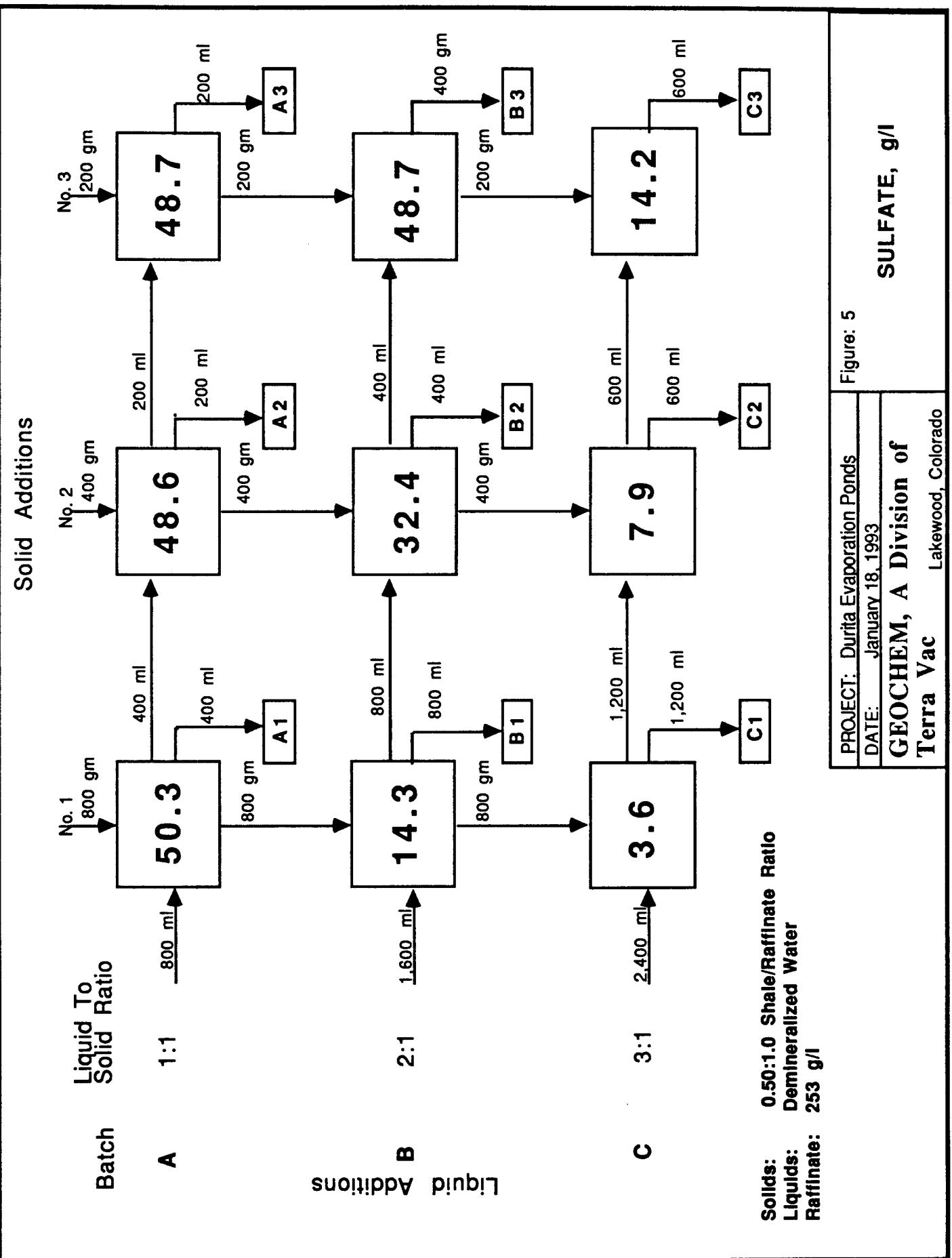
PROJECT: Durita Evaporation Ponds
DATE: January 18, 1993

Figure 3

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Lakewood, Colorado

Schematic of Sequential Batch
Testwork of Matrix



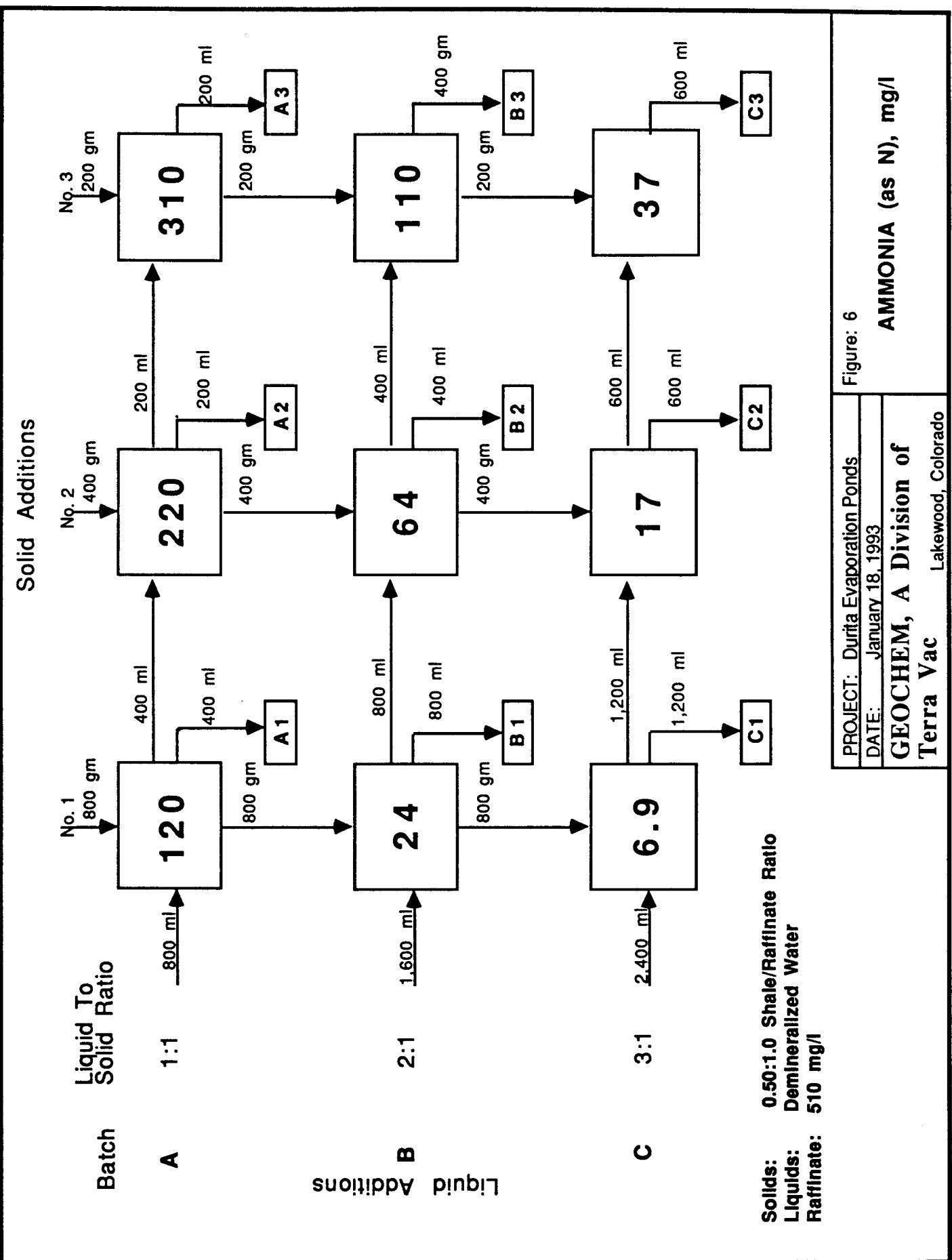


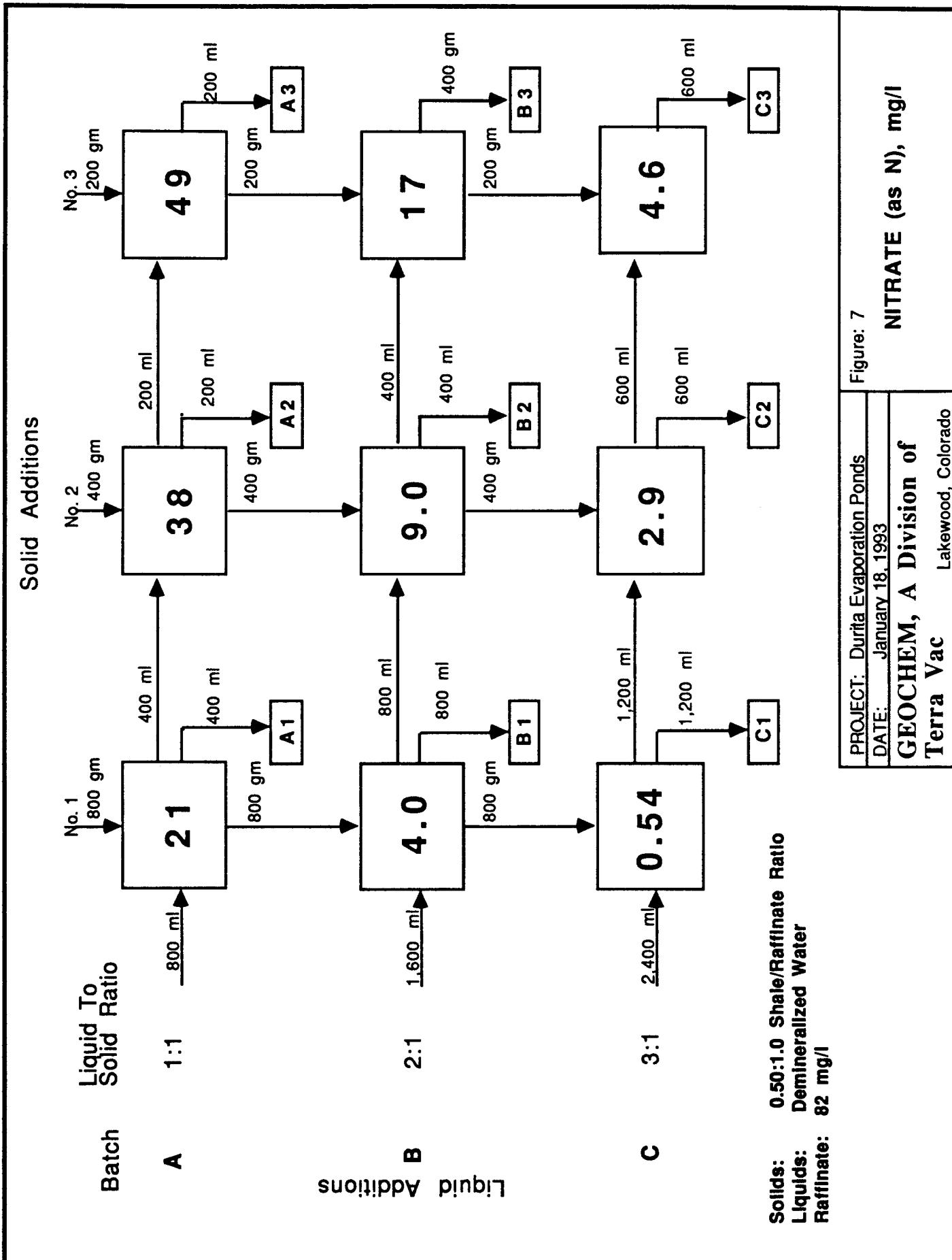
Solids: 0.50:1.0 Shale/Raffinate Ratio
Liquids: Demineralized Water
Raffinate: 253 g/l

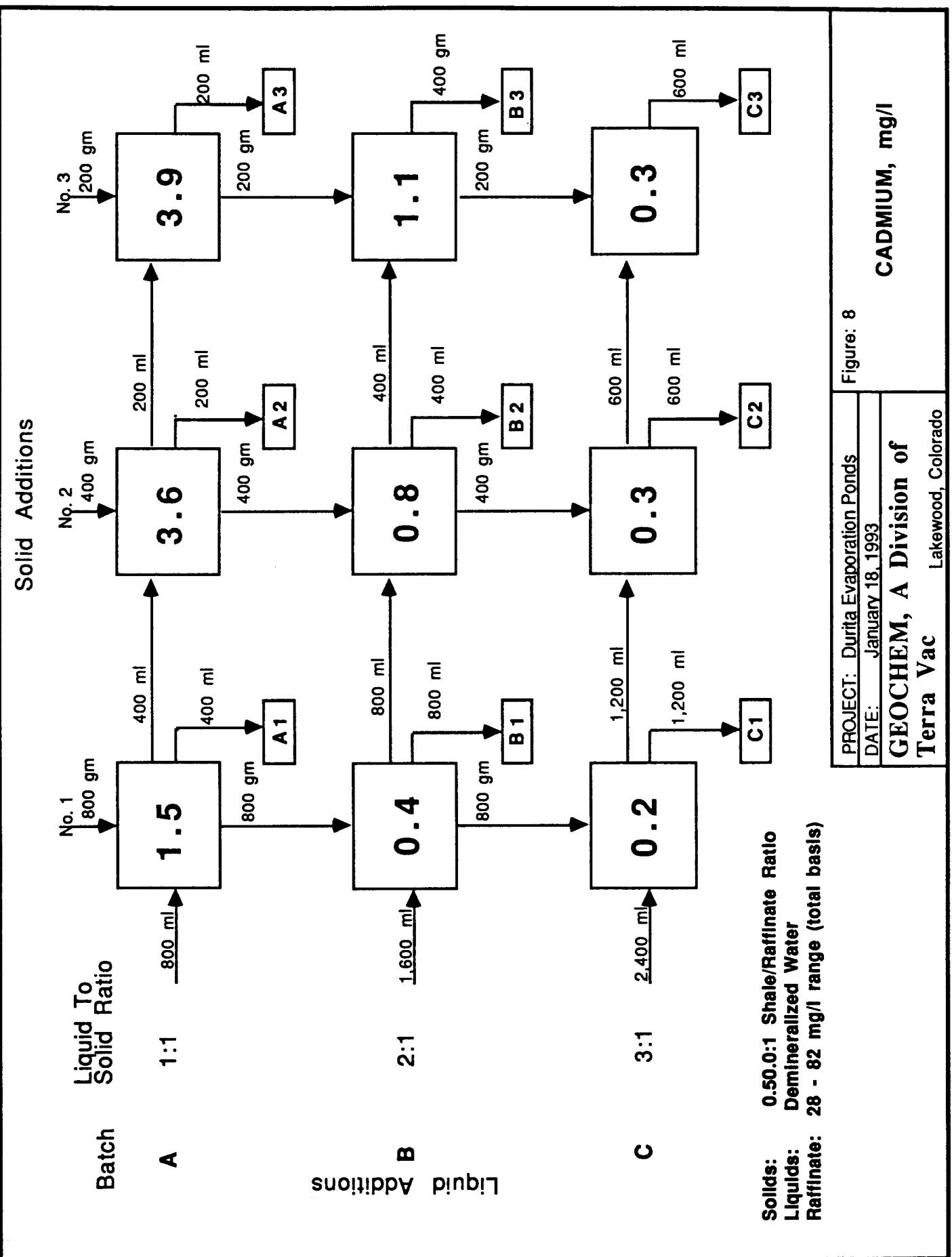
Figure: 5

SULFATE, g/l

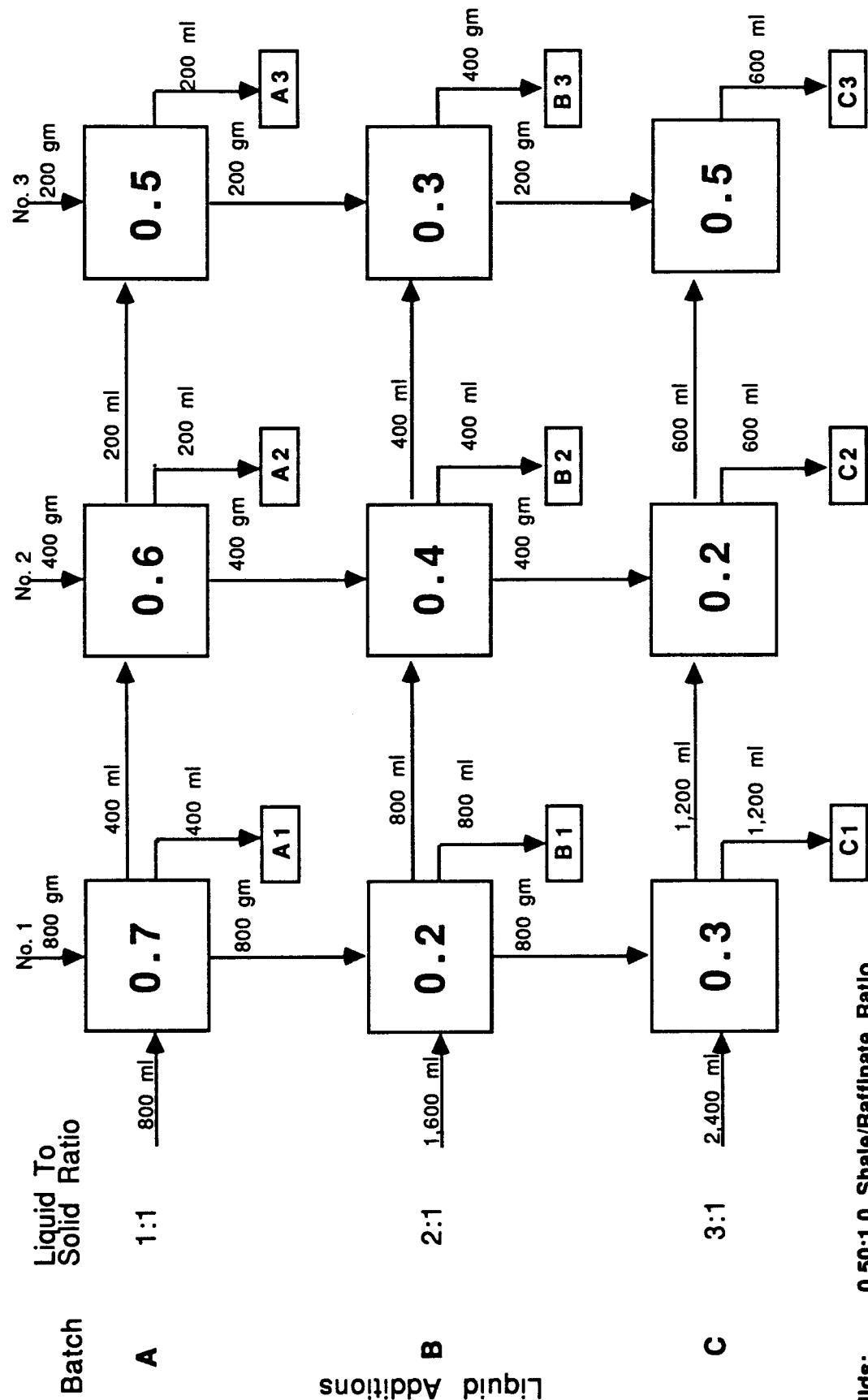
PROJECT: Durita Evaporation Ponds	DATE: January 18, 1993
GEOCHEM, A Division of Terra Vac	
Lakewood, Colorado	







Solid Additions



Solids: 0.50:1.0 Shale/Raffinate Ratio
Liquids: Demineralized Water
Raffinate: 140 - 520 mg/l range (total basis)

PROJECT: Durita Evaporation Ponds	Figure: 9
DATE: January 18, 1993	
GEOCHEM, A Division of Terra Vac Lakewood, Colorado	

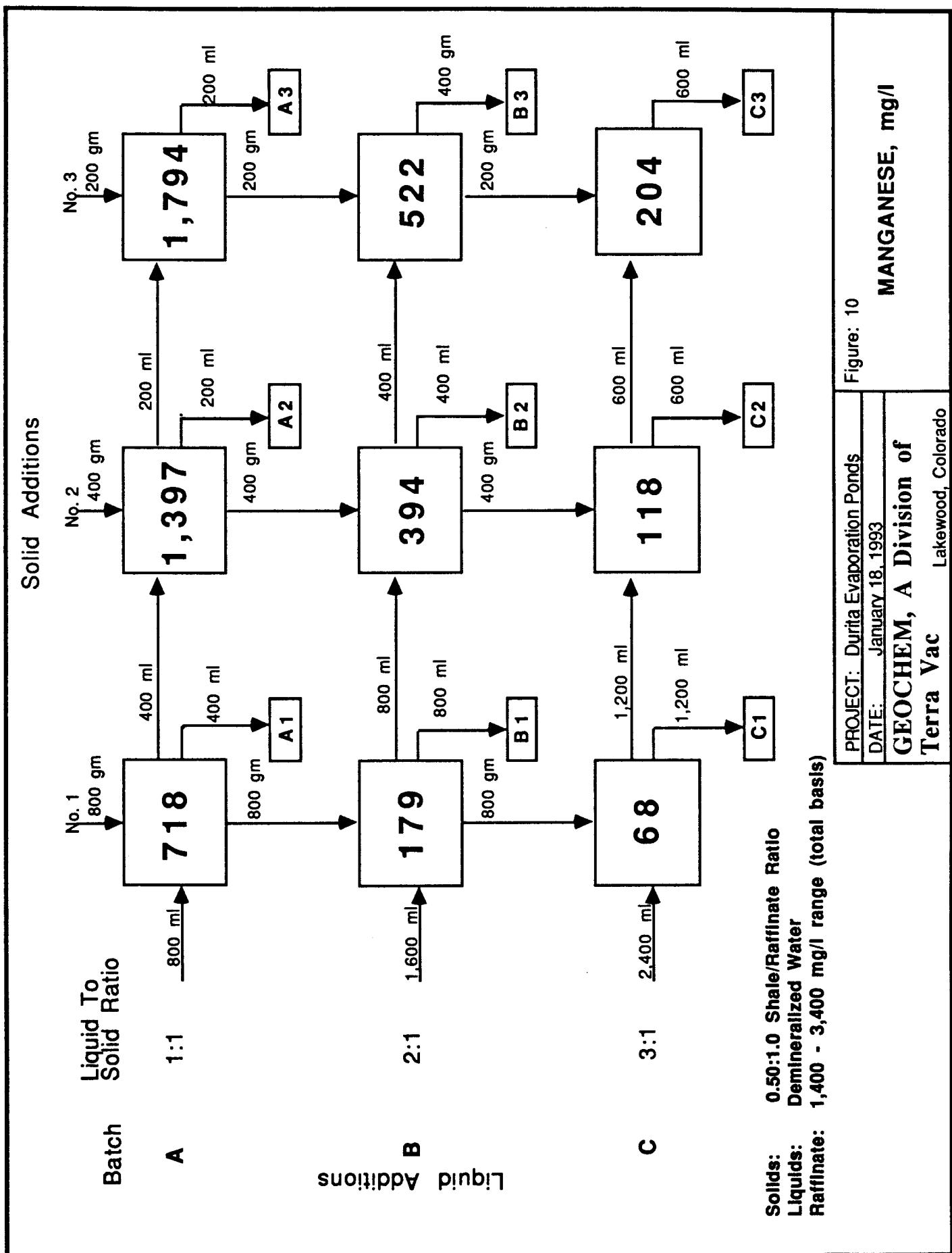
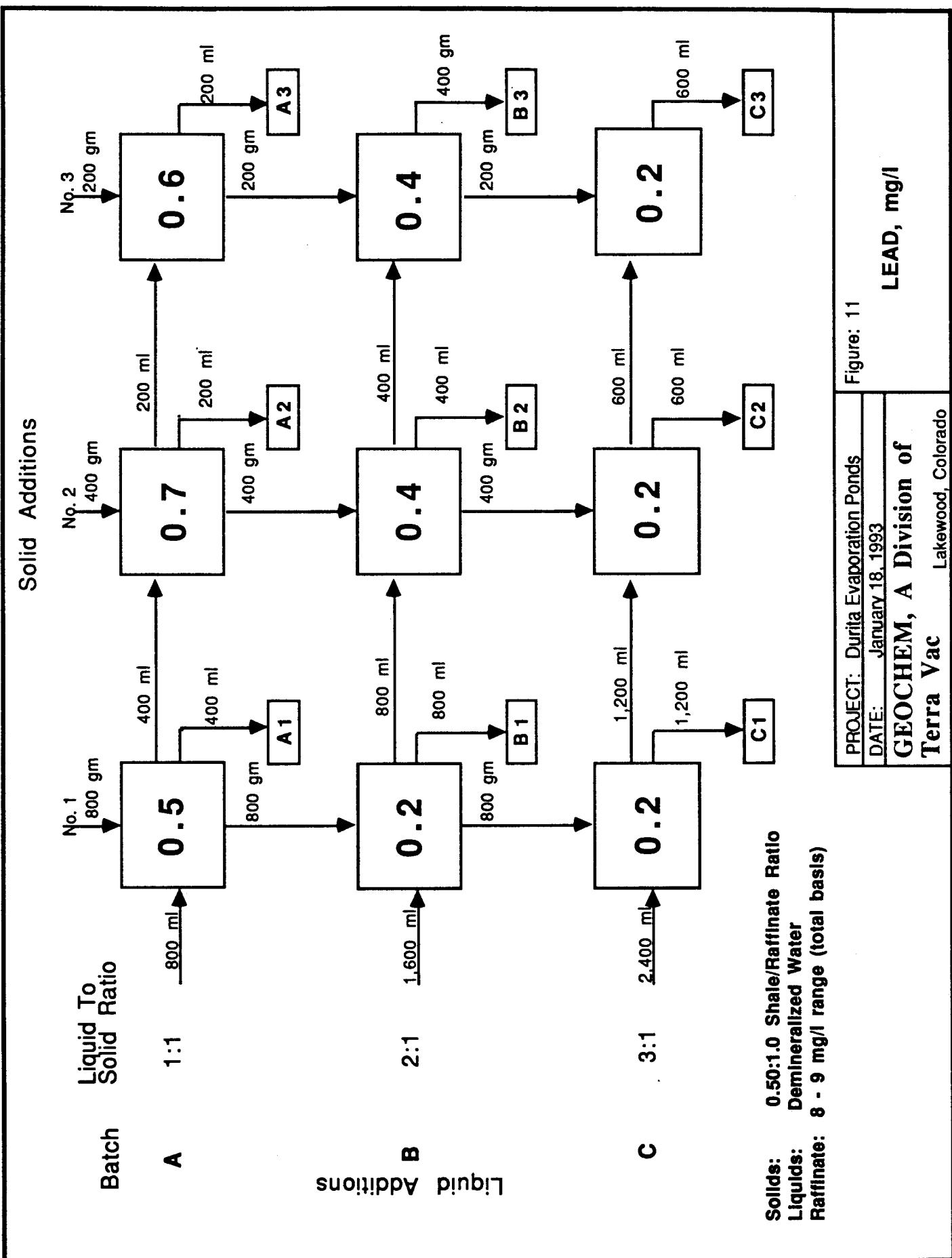


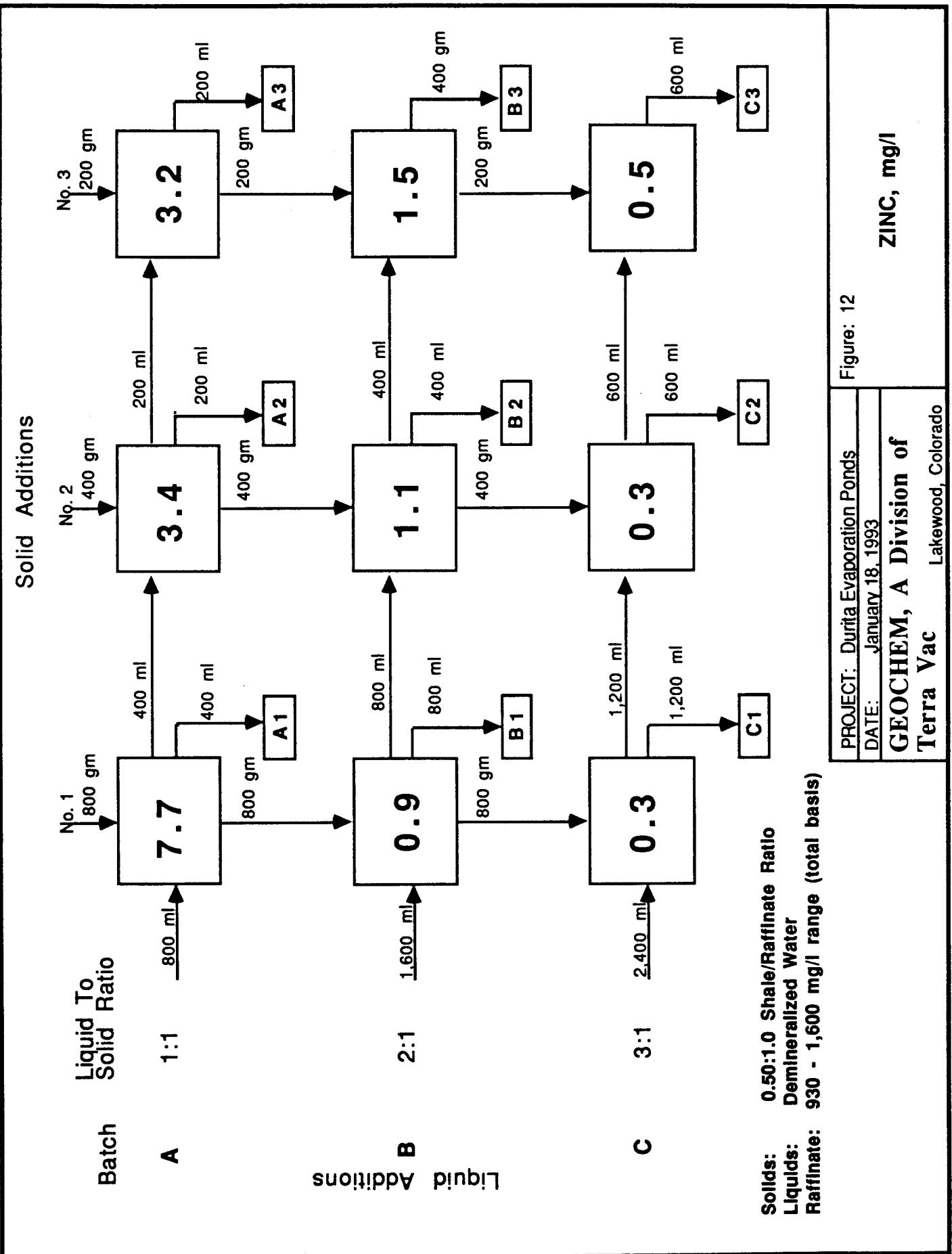
Figure: 10

MANGANESE, mg/l

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DATE: January 18, 1993

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Terra Vac
Lakewood, Colorado





Appendix A

Analytical Reports

**Accu-Labs Research
IC Technologies
The Mineral Lab**



Accu-Labs Research, Inc.

4663 Table Mountain Drive Golden, Colorado 80403-1650
(303) 277-9514 FAX (303) 277-9512

October 7, 1992
Page 1 of 3

Mr. Gary Gamble
Hecla Mining Co.
P.O. Box 8000
Coeur d'Alene, ID 83816-1931

RE: 9963-44395-9
Date Samples Rec'd 8-31-92
Proj. No. 9014

ADDITIONAL ANALYSIS

REPORT OF ANALYSIS

ALR Designation	9963-44395-9-1	9963-44395-9-2	9963-44395-9-3
Sponsor Designation	DUR601A 1,2,3,4	DUR603A 1,2,3,4	DUR604A 1,2,3,4
	8-27-92	8-27-92	8-27-92

Chloride, µg/mL	19,000	16,000	37,000
Ammonia (as N), µg/mL	500	590	180
Nitrate (as N), µg/mL	35	35	91
pH, pH units	2.9	2.8	2.5
Sulfate (as S), µg/mL	280,000	250,000	280,000
Total Solids, %	--	--	57

ICP METALS, $\mu\text{g/mL}$:

Arsenic, total	27	23	50
Calcium, total	380	380	320
Cadmium, total	36	28	82
Iron, total	340	140	520
Potassium, total	50	5	8
Magnesium, total	38,000	26,000	33,000
Manganese, total	1900	1400	3400
Molybdenum, total	<1	<1	<1
Sodium, total	22,000	18,000	29,000
Lead, total	9	8	8
Zinc, total	1300	930	1600
Aluminum, total *	17,000	18,000	22,000
Silica, total *	190	<50	<50

Accu-Labs Research, Inc.

October 7, 1992
Page 2 of 3

REPORT OF ANALYSIS

ALR Designation	9963-44395-9-4	9963-44395-9-5	9963-44395-9-6
Sponsor Designation	DUR601G 1,2,3,4	DUR603G 1,2,3,4	DUR604G 1,2,3,4
	8-27-92	8-27-92	8-27-92

Chloride, µg/g	530	7100	11,000
Ammonia (as N), µg/g	290	150	340
Nitrate (as N), µg/g	28	37	44
pH, pH units	3.1	2.5	2.8
Sulfate (as S), µg/g	98,000	190,000	160,000
Total Solids, %	80	63	63

ICP METALS, µg/g:

Arsenic, total	74	38	45
Calcium, total	9900	3200	1400
Cadmium, total	15	23	36
Iron, total	13,000	3300	2200
Potassium, total	1100	240	390
Magnesium, total	29,000	31,000	25,000
Manganese, total	820	1200	1600
Molybdenum, total	0.9	<0.5	<0.5
Sodium, total	18,000	2200	25,000
Lead, total	6.2	4.8	5.2
Zinc, total	780	930	900
Aluminum, total *	28,000	25,000	30,000
Silica, total *	220	81	130

Accu-Labs Research, Inc.

October 7, 1992
Page 3 of 3

REPORT OF ANALYSIS

ALR Designation	9963-44395-9-7	9963-44395-9-8	9963-44395-9-9
Sponsor Designation	DUR601S 1,2 8-27-92	DUR603S 1,2 8-27-92	DUR604S 1,2 8-27-92

Chloride, µg/g	7100	11,000	11,000
Ammonia (as N), µg/g	1100	500	250
Nitrate (as N), µg/g	18	24	20
pH, pH units	2.9	2.9	2.8
Sulfate (as S), µg/g	330,000	330,000	440,000
Total Solids, %	67	68	69

ICP METALS, µg/g:

Arsenic, total	60	70	40
Calcium, total	3000	6300	690
Cadmium, total	18	21	16
Iron, total	5800	7700	3200
Potassium, total	1100	1100	320
Magnesium, total	35,000	29,000	44,000
Manganese, total	950	1000	1000
Molybdenum, total	<1	<1	<1
Sodium, total	29,000	32,000	32,000
Lead, total	13	21	9
Zinc, total	940	850	1200
Aluminum, total *	43,000	42,000	37,000
Silica, total *	120	160	82

* Additional Analysis

Notes: Samples #1, 2 and 3 were analyzed as water samples.
Samples #4, 5 and 6 were analyzed as wet solid samples.
Samples #7, 8 and 9 were prepped prior to analysis.

These samples are scheduled to be discarded 30 days after the date of this report.

Eyda Hergenreder
Eyda Hergenreder
Soils/RCRA Laboratory Supervisor

EH/ep



Accu-Labs Research, Inc.

4663 Table Mountain Drive Golden, Colorado 80403-1650
(303) 277-9514 FAX (303) 277-9512

A N A L Y S I S R E P O R T
DATE: 10/09/92 PAGE 1

GARY GAMBLE
HECLA MINING COMPANY
P.O. BOX 8000
COEUR d'ALENE, ID 83816-1931

Lab Job Number: 9963-44395-9
Date Samples Received: 08/31/92
Customer PO Number: PROJ#9014

These samples to be disposed of 30 days after the date of this report.

ALR Designation -	9963-44395-9-1	9963-44395-9-2	9963-44395-9-3	9963-44395-9-4
Sponsor Designation -	DUR601A 1,2,3,4	DUR603A 1,2,3,4	DUR604A 1,2,3,4	DUR601G 1,2,3,4
Date Collected -	08/27/92	08/27/92	08/27/92	08/27/92

Determinations in % unless noted

Total Organic Carbon

(mg/L) ----- 110 ----- -----

Total Carbon <0.05 ---- <0.05 0.13

Total Dissolved Solids (@180 °C)

(mg/L) 370,000 320,000 ----

ALR Designation - 9963-44395-9-5 9963-44395-9-6 9963-44395-9-7 9963-44395-9-

Sponsor Designation - DUR603G 1,2,3,4 DUR604G 1,2,3,4 DUR601S 1,2 DUR603S 1,2

Date Collected - 08/27/92 08/27/92 08/27/92 08/27/92

Total Carbon 0.08 0.06 0.06 0.05

ALR Designation - 9963-44395-9-9
Sponsor Designation - DUR604s 1,2
Date Collected - 08/27/92

Total Carbon <0.05

By: Eyda Hergenreder
Eyda Hergenreder
Water Laboratory Supervisor

EH/dh
cc: Bill Bond, Geochem



Accu-Labs Research, Inc.

4663 Table Mountain Drive Golden, Colorado 80403-1650
 (303) 277-9514 FAX (303) 277-9512

A N A L Y S I S R E P O R T

DATE: 12/08/92 PAGE 1

GARY GAMBLE
 HECLA MINING COMPANY
 P.O. BOX 8000
 COEUR d'ALENE, ID 83816-1931

Lab Job Number: 9963-44395-9
 Date Samples Received: 08/31/92
 Customer PO Number: PROJ#9014

These samples to be disposed of 30 days after the date of this report.

ALR Designation -	9963-44395-9-1	9963-44395-9-2	9963-44395-9-3	9963-44395-9-4
Sponsor Designation -	DUR601A 1,2,3,4	DUR603A 1,2,3,4	DUR604A 1,2,3,4	DUR601G 1,2,3,4
Date Collected -	08/27/92	08/27/92	08/27/92	08/27/92

Determinations in pCi/g (dry) unless noted

Gross Alpha - total	84 ± 17 *	120 ± 30 *	220 ± 20 *	3700 ± 100 *
Gross Beta - total	7 ± 15 *	17 ± 24 *	28 ± 6 *	93 ± 27 *
Radium-226 - total	0.6 ± 0.2 *	0.0 ± 0.2 *	0.4 ± 0.2 *	16 ± 1 *
Radium-228 - total	0.0 ± 1.0 *	0.0 ± 1.0 *	2.1 ± 0.6 *	0.0 ± 0.9 *
Lead-210 - total	1.9 ± 0.6 *	0.1 ± 0.4 *	1.0 ± 0.7 *	6.1 ± 0.8 *
Polonium-210 - total	0.4 ± 0.2 *	0.5 ± 0.5 *	1.1 ± 0.4 *	9.0 ± 1.4 *
Thorium-228 - total	0.1 ± 0.2 *	0.2 ± 0.3 *	0.2 ± 0.5 *	1.4 ± 0.8 *
Thorium-230 - total	220 ± 10 *	370 ± 10 *	570 ± 20 *	2600 ± 100 *
Thorium-232 - total	0.1 ± 0.2 *	0.8 ± 0.5 *	-0.1 ± 0.2 *	9.6 ± 1.9 *
Uranium - total $(\mu\text{g/g (dry)})$	120	100	2.0	44

Accu-Labs Research, Inc.

A N A L Y S I S R E P O R T
DATE: 12/08/92 PAGE 2
Lab Job Number 9963-44395-9

These samples to be disposed of 30 days after the date of this report.

ALR Designation -	9963-44395-9-5	9963-44395-9-6	9963-44395-9-7	9963-44395-9-8
Sponsor Designation -	DUR603G 1,2,3,4	DUR604G 1,2,3,4	DUR601S 1,2	DUR603S 1,2
Date Collected -	08/27/92	08/27/92	08/27/92	08/27/92

Determinations in pCi/g (dry) unless noted

Gross Alpha - total	1200 ± 100 *	1000 ± 100 *	610 ± 50 *	1400 ± 100 *
Gross Beta - total	18 ± 17 *	58 ± 22 *	22 ± 18 *	39 ± 21 *
Radium-226 - total	6.5 ± 0.4 *	5.5 ± 0.4 *	7.2 ± 0.5 *	12 ± 1 *
Radium-228 - total	0.0 ± 1.0 *	0.0 ± 1.1 *	1.7 ± 0.6 *	0.0 ± 1.5 *
Lead-210 - total	6.1 ± 0.8 *	4.8 ± 0.8 *	7.2 ± 1.3 *	9.3 ± 0.9 *
Polonium-210 - total	2.0 ± 0.5 *	2.4 ± 0.7 *	6.5 ± 1.0 *	4.1 ± 0.7 *
Thorium-228 - total	1.0 ± 0.9 *	0.8 ± 0.9 *	1.0 ± 1.1 *	1.5 ± 1.3 *
Thorium-230 - total	1200 ± 100 *	1400 ± 100 *	1200 ± 100 *	1900 ± 100 *
Thorium-232 - total	0.8 ± 0.9 *	-0.2 ± 0.4 *	0.2 ± 0.8 *	1.7 ± 1.6 *
Uranium - total (μg/g (dry))	94	63	25	32

Accu-Labs Research, Inc.A N A L Y S I S R E P O R T
DATE: 12/08/92 PAGE 3
Lab Job Number 9963-44395-9

These samples to be disposed of 30 days after the date of this report.

ALR Designation - 9963-44395-9-9
Sponsor Designation - DUR604S 1,2
Date Collected - 08/27/92

Determinations in pCi/g (dry) unless noted

Gross Alpha - total	540 ± 50 *
Gross Beta - total	11 ± 5 *
Radium-226 - total	3.6 ± 0.4 *
Radium-228 - total	0.0 ± 0.8 *
Lead-210 - total	3.9 ± 0.7 *
Polonium-210 - total	2.0 ± 0.5 *
Thorium-228 - total	0.6 ± 0.9 *
Thorium-230 - total	600 ± 20 *
Thorium-232 - total	0.6 ± 0.8 *
Uranium - total	
(μg/g (dry))	22

* Variability of the radioactive disintegration process (counting error) at the 95% confidence level, 1.96σ.

Note: Thoriums were rerun to confirm results.

By: _____



Bud Summers
Radiochemistry Supervisor

BS/ep





Accu-Labs Research, Inc.

1651 1/2 Northw. Drive Golden, Colorado 80401-1650
1303) 277-9516 FAX (303) 277-9512CHAIN OF CUSTODY
SAMPLE TRANSMITTAL FORMCOMPANY NAME: Hercia Mining Co.
COMPANY CONTACT: Gerry Grun PHONE: 208-769-4400

ALR PROJECT #

PROJECT NO.: 9014

ALR USE ONLY	SAMPLER (SIGNATURE):	BOTTLE SIZE & PRESERVATIVE			ANALYSES	
		DATE	TIME	SAMPLE TYPE		NUMBER OF BOTTLES
1	8/21/92	1350	bulk gel	DUR 603A-1	✓	
2	8/21/92	1400	gel	DUR 603A-2	✓	
3	8/21/92	1430	acid	DUR 603A-1	✓	
4	8/21/92	1430	acid	DUR 603A-2	✓	
5	8/21/92	1430	acid	DUR 603A-3	✓	
6	8/21/92	1440	acid	DUR 604A-2	✓	
7	8/21/92	1440	acid	DUR 604A-3	✓	
8	8/21/92	1440	gel	DUR 604A-1	✓	
9	8/21/92	1440	gel	DUR 604A-2	✓	
10	8/21/92	1440	acid	DUR 604A-4	✓	
11	8/21/92	1530	gel	DUR 605A-3	✓	
RELINQUISHED BY:					DATE AND TIME RECEIVED BY:	
<u>Gerry Grun</u>					8/21/92 1400	RECEIVED FOR ALR BY:
RELINQUISHED BY:					DATE AND TIME RECEIVED FOR ALR BY:	REMARKS:
					8/21/92 1530	External gamma 2-growth background 8/21/92 1530 1/1/92
					DATE AND TIME RECEIVED BY:	



Accu-Labs Research, Inc.

1663 Fisher Mountain Drive Golden, Colorado 80403-1650
(303) 277-9514 FAX (303) 277-9512

CHAIN OF CUSTODY
SAMPLE TRANSMITTAL FORM

ALR PROJECT #:

COMPANY NAME: Hercules Chemical Co.
COMPANY CONTACT: Gerry Lembke **PHONE:** 303-769-4100

F8291 REV. C

PROJECT NO.: <u>8014</u> P.O.I.:				BOTTLE SIZE & PRESERVATIVE				ANALYSES			
SAMPLER (SIGNATURE):											
ALR USE ONLY	DATE	TIME	SAMPLE TYPE	SAMPLE IDENTIFICATION							
12	8/27/92	1350	salt	DUR 603 S-1	/	/	/	/	/	/	/
13	8/27/92	1350	salt	DUR 603 S-2	/	/	/	/	/	/	/
14	8/27/92	1440	salt	DUR 604 S-1	/	/	/	/	/	/	/
15	8/27/92	1440	salt	DUR 604 S-2	/	/	/	/	/	/	/
16	8/27/92	1520	salt	DUR 601 S-1	/	/	/	/	/	/	/
17	8/27/92	1520	salt	DUR 601 S-2	/	/	/	/	/	/	/
18	8/27/92	1525	acid	DUR 601 A-A	/	/	/	/	/	/	/
19	8/27/92	1530	gel	DUR 603 G-3	/	/	/	/	/	/	/
20	8/27/92	1530	gel	DUR 601 G-3	/	/	/	/	/	/	/
RELINQUISHED BY:				DATE AND TIME RECEIVED BY:				DATE AND TIME RECEIVED BY:			
<u>John R. Gandy</u> 8/27/92 1610											
RELINQUISHED BY:				DATE AND TIME RECEIVED FOR ALR BY:				DATE AND TIME REMARKS:			
				8/31/92 1530 <u>Mylene Shand</u>				8/31/92 1530 <u>External gamma 2-Sulfur + background</u>			



Accu-Labs Research, Inc.

6661 Table Mountain Drive **Golden, Colorado 80403-1450**
303) 277-9514 **[XXX] 1803 227 9514**

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LIBRARY

COMPANY NAME: Necla Printing Co.
COMPANY CONTACT: Beny Grampel

**SIMPLIFIED TRANSMITTAL FORM
CHAIN OF CUSTODY**

SILATH OE SISTANDU

卷之三

COMPANY NAME: Mocha Printing Co.
COMPANY CONTACT: Sammy Grunthal PHONE: 208-769-4100

Accu-Labs Research, Inc.

1665 Table Mountain Drive Golden, Colorado 80403-1650
(303) 277-9511

Digitized by srujanika@gmail.com

MAIL PROJECT /

**CHAIN OF CUSTODY
SAMPLE TRANSMITTAL FORM**

AL PROJECT # Weeks Mfg Co
COMPANY NAME: Weeks Mfg Co
COMPANY CONTACT: TENSE G. COOPER

COMPANY CONTACT: Mr. & Mrs. John G.
PHONE: 705-769-4141

PHONE: 108-169-2710

PROJECT NO.: 901
SAMPLED (SIGNATURE)
M.R.



September 25, 1992
Lab no. 920539

Mr. William R. Bond
Terra Vac
12265 W. Bayaud Ave., ste. 140
Lakewood, CO 80228

Dear Mr. Bond:

Two samples, ("9014 K Mancos 1&2") submitted earlier this month have been analyzed by x-ray diffraction (XRD) to determine their mineral compositions. The bulk samples and the clay size ($2\mu\text{m}$) fractions were analyzed.

Each sample was crushed to -1/4" before splitting and grinding.

A representative portion of each crushed sample was ground to approximately -400 mesh in a steel swing mill, packed into an aluminum holder and scanned with the diffractometer over the range, 3-61° 2θ using Cu-K α radiation. The enclosed table for the "bulk" samples summarizes the results of those XRD scans as approximate mineral weight percents. Estimates of mineral concentrations are based on relative peak heights and comparison to our XRF-determined elemental composition. The detection limit for an average mineral in these samples is ~3-5% and the analytical reproducibility is approximately equal to the square root of the amount. "Unidentified" accounts for that portion of the XRD scan which could not be resolved.

Each crushed sample was subjected to a size separation procedure based on Stokes' Law to concentrate the clay-size ($-2\mu\text{m}$) fraction for XRD analysis. A representative split of each sample was blended with distilled water and 10 ml of a 5% calgon solution (as an antiflocculant) and then brought up to volume in a 1000 ml graduated cylinder. Each suspension was allowed to settle for 19.5 hours and then 20 ml of the material suspended above the 300 ml mark in the cylinder were drawn into a preweighed beaker, air dried and the weight of the clay-size material determined. The weight percents of clay-size material concentrated by this procedure are listed on the enclosed table labelled, "Clay Separation Results".

Each remaining suspension was siphoned off for clay XRD analysis. A portion of each suspension was drawn onto a millipore filter and then rolled onto a glass slide forming an "oriented mount" for clay analysis. Each oriented mount was scanned with the diffractometer over the range, 2-61° 2θ , treated with glycol (to

William Bond
Terra Vac
Page 2

September 25, 1992
Lab no. 920539

expand any smectite present), and rescanned over the range, 2-30° 2θ. The results of the "clay" XRD scans are summarized on the enclosed table as approximate mineral weight percents. Concentration estimates are based on relative peak heights, and comparison to the bulk XRD results. The detection limit for an average mineral in these samples is ~3-5%. "Unidentified" accounts for that portion of the XRD scan which could not be resolved.

Thank you for the opportunity to be of service.

Sincerely,



Peggy Dalheim

PD/tz

Terra Vac
XRD Results for Bulk "9014 K Mancos" Samples Given as Approx. Wt%.

September 25, 1992
Lab no. 920539

Mineral	SAMPLE	
	1	2
Calcite	60	50
Quartz	14	15
Kaolinite	15	17
Mica/illite	<10	14
"Unidentified"	<5	<5

Analysis performed by The Mineral Lab, Inc.

Terra Vac
Clay Separation Results for "9014 K Mancos" Samples.

September 25, 1992
Lab no. 920539

Sample	Wt% -2 μ m Material Concentrated
1	6
2	6

Analysis performed by The Mineral Lab, Inc.

Terra Vac
Clay-Size Fractions of "9014 K Mancos" Samples
XRD Results Given as Approx. Wt%.

September 25, 1992
Lab no. 920539

Mineral	SAMPLE	
	1	2
Calcite	27	10
Quartz	<5	<5
Kaolinite	30	50
Mica/illite	20	20
Sepiolite	15	15
"Unidentified"	<5	<5

Analysis performed by The Mineral Lab, Inc.



Accu-Labs Research, Inc.

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October 7, 1992

Page 1 of 1

Mr. Gary Gamble
Hecla Mining Co.
P.O. Box 8000
Coeur d'Alene, ID 83816-1931

RE: 9963-44533-2
Date Samples Rec'd 9-8-92
Project 33-0016

ADDITIONAL ANALYSIS

REPORT OF ANALYSIS

ALR Designation	9963-44533-2-1	9963-44533-2-2
Sponsor Designation	9014 K Mancos 1	9014 K Mancos 2
Date Collected	<u>8-27-92</u>	<u>8-27-92</u>

Determination: meq/100g unless noted

% Organic Matter	0.34	0.34
CEC	23	21
ph, pH units	7.4	7.7
Acid/Base Potential, %	64	50
Calcium, exchangeable	66	76
Iron (II), oxide	16,000	19,000
Potassium, exchangeable	2	2
Magnesium, exchangeable	1	4
Manganese (II), oxide	200	140
Sodium, exchangeable	<1	<1
Aluminum, total, µg/g *	12,000	16,000
Silica, total, µg/g *	1100	1300
Particle Size/Texture:		
% Sand	36	38
% Silt	45	46
% Clay	19	16
USDA Classification	L	L

* Additional Analysis

These samples are scheduled to be discarded 30 days after the date of this report.

EH/ep

Eyda Hergenreder
Eyda Hergenreder
Soils and RCRA Supervisor

Accu-Labs Research, Inc.

4663 Table Mountain Drive **Golden, Colorado 80403-1650**
(303) 277-9514 **FAX (303) 277-9512**

A N A L Y S I S R E P O R T
DATE: 10/08/92 **PAGE 1**

GARY GAMBLE
HECLA MINING COMPANY
P.O. BOX 8000
COEUR D'ALENE, ID 83816-1931

These samples to be disposed of 30 days after the date of this report.

ALL Designation - 9963-44533-2-1 9933-44533-2
Sponsor Designation - 9014 K MANCOS 1 9014 K MANCOS
Date Collected - 08/27/92 08/27/92

Determinations in $\mu\text{Ci/g}$ unless noted

Gross Alpha - total	$14 \pm 6 \text{ *}$	$10 \pm 6 \text{ *}$
Radium-226 - total	$2.8 \pm 0.3 \text{ *}$	$1.8 \pm 0.3 \text{ *}$
Radium-228 - total	$0.9 \pm 0.5 \text{ *}$	$0.9 \pm 0.5 \text{ *}$

* Variability of the radioactive disintegration process (counting error) at the 95% confidence level.

By: Bud Summers
Radiochemistry Supervisor

BS / ep



Accu-Labs Research, Inc.

1665 Table Mountain Drive Golden, Colorado 80403-1650
303/277-9314 FAX 303/277-9512

**CHAIN OF CUSTODY
SAMPLE TRANSMITTAL FORM**

AIR PROJECT /

COMPANY NAME: GEOCHIM-TERRA VAC
ADDRESS: 211 BAPTIST
CITY: NEW YORK STATE: NY ZIP CODE: 10036
PHONE: (212) 633-1000

VAC PHONE: 988-8902

BOTTLE SIZE & PRESERVATIVE

Laboratory Determination of Moisture Content
 (ASTM D 2216-90)
Durita Samples
 ICT Project 1197

Test #	Mix Ratio g Shale:g Raffinate	Wet Weight, g	Dry Weight, g	Weight Loss, g	Weight Loss, %
1	0.5:1	432.0	281.1	150.9	34.9%
2	0.67:1	411.0	254.3	156.7	38.1%
3 (1)	0.75:1	236.6	159.2	77.4	32.7%
4	1.00:1	416.3	270.2	146.1	35.1%
5	1.25:1	409.1	284.1	125.0	30.6%
6	1.50:1	422.6	298.5	124.1	29.4%

(1) The supply of this mix ratio was depleted by earlier work. The ASTM procedure's minimum quantity of 100g was satisfied.

Notes ASTM D 2216-90 specifies a minimum of 100g starting material for particle size of 100% minus 4 mesh.

Section 1.4 of ASTM D 2216-90 states that "Materials containing water with substantial amounts of soluble solids...when tested by this method will give a mass of solids which includes the previously soluble solids. These materials require special treatment to remove or account for the presence of precipitated solids in the dry mass of the specimen, or a qualified definition of water content must be used."

Accordingly, weight loss, rather than % moisture, is reported for these samples

IC Technologies

12325 West 52nd Avenue Wheat Ridge, CO 80033-1916 303/424-8000 FAX: 303/424-8580 1-800-275-0600

FACSIMILE TRANSMISSION COVER SHEET

DATE: December 21, 1992

TIME: 10:40 AM PM

TO: Dr. Roman Pyrih
TerraVac
12265 W. Bayaud Ave.
Lakewood, CO 80228
FAX: (303) 988-0288

FROM: R. Lee Schwartz
Senior Project Coordinator
IC TECHNOLOGIES, INC.

Total number of pages being transmitted, including this cover sheet: 2

Message:

Roman:

The data summary originally issued on Friday, 12/18 has been revised to reflect incorrect entries for the samples 1001-104 Decant and the raffinate sample. As we presumed on Friday, pH, TDS, and Cl⁻ values were switched for those samples.

The table shows two TDS values for sample 1001-100-4 and raffinate. The top value was generated by electrode method, while the bottom value is by dilution/filtration/evaporation. It appears that because the TDS electrode measures conductivity rather than true dissolved solids, accurate values on these samples may not be obtainable by electrode method. We will repeat these analyses by evaporation method. I'll re-issue the data summary following results of TDS analyses.

**IF YOU DO NOT RECEIVE ALL OF THE PAGES,
PLEASE CALL BACK AS SOON AS POSSIBLE.**

Applying tomorrow's technologies to today's problems.

Sequential Batch-Contact Leaching Tests
Summary of Solution Analyses
 ICI Project 1197

Sample Number	Stage	Element or Species	Units	Method		TDS	Ca	Mg	Na	Cl	SO4	NH3	NO3	As	Cd	Fe	Mn	Pb	Zn
				Electro	Elec/El/Evap														
-1	A-1: 800/800		mg/l	0.05	10	10.980	IP/OL	IP/OL	41.9	10.2	50.3	IP/OL	IP/OL	ND	1.5	0.7	718	0.5	7.7
-2	A-2: 400/400		mg/l	6.9	11.400	IP/OL	IP/OL	68.2	17.9	48.6	IP/OL	IP/OL	ND	3.6	0.6	1397	0.7	3.4	
-3	A-3: 200/200		mg/l	7.0	11.780	IP/OL	IP/OL	87.4	24.8	48.7	IP/OL	IP/OL	ND	3.9	0.5	1794	0.6	3.2	
-4	B-1: 800/1600		mg/l	7.4	23.300	IP/OL	IP/OL	7.3	1.8	14.3	IP/OL	IP/OL	<0.5	0.4	0.2	179	0.2	0.9	
-5	B-2: 400/800		mg/l	7.5	6.720	IP/OL	IP/OL	19.2	4.4	32.4	IP/OL	IP/OL	<1.0	0.8	0.4	394	0.4	1.1	
-6	B-3: 200/400		mg/l	7.2	7.690	IP/OL	IP/OL	33.3	7.9	48.7	IP/OL	IP/OL	<0.5	1.1	0.3	522	0.4	1.5	
-8	C-1: 800/2400		mg/l	7.6	2.580	IP/OL	IP/OL	1.1	0.3	3.6	IP/OL	IP/OL	ND	0.2	0.3	68	0.2	0.3	
-10	C-2: 400/1200		mg/l	7.4	4.130	IP/OL	IP/OL	3.9	0.8	7.9	IP/OL	IP/OL	ND	0.3	0.2	118	0.2	0.3	
-12	C-3: 200/600		mg/l	7.1	5.520	IP/OL	IP/OL	7.2	1.6	14.2	IP/OL	IP/OL	ND	0.3	0.3	204	0.2	0.5	
001-104 Decont				3.8	9.000	IP/OL	IP/OL	120.0	35.0	48.1	IP/OL	IP/OL	ND	0.7	1.4	5040	0.7	1578	
Raffinate				8.300	101.000			82.8	87.0	253.2	IP/OL	IP/OL	ND	0.9	2.5	3415	0.7	1410	

Notes: Elements or species shown in italic typeface will be analyzed by an outside laboratory.

In Progress at Outside Laboratory: ND = Not Detected at concentrations above method detection limit

Method Abbreviations:

Electro

Atomic Absorption: detection limit improved by evaporation of sample solution.

Titrimetry

Gravimetry



Accu-Labs® Research, Inc.

4663 Table Mountain Drive Golden, Colorado 80403-1650
(303) 277-9514 FAX (303) 277-9512

A N A L Y S I S R E P O R T
DATE: 01/11/93 PAGE 1

L. SCHWARTZ
IC TECHNOLOGIES INC
4880 ROBB ST #3
WHEAT RIDGE, CO 80033

Lab Job Number: 2291-46177-10
Date Samples Received: 12/18/92
Customer PO Number: 1197

These samples to be disposed of 30 days after the date of this report.

ALR Designation -	2291-46177-10-1	2291-46177-10-2	2291-46177-10-3	2291-46177-10-4
Sponsor Designation -	1001-100-1	1001-100-2	1001-100-3	1001-100-4
Date Collected -	12/18/92	12/18/92	12/18/92	12/18/92

Determinations in mg/L unless noted

Calcium - total	500	470	390	490
Magnesium - total	14,000	25,000	33,000	2,700
Ammonia (as N)	120	220	310	24
Nitrate (as N)	21	38	49	4.0

ALR Designation -	2291-46177-10-5	2291-46177-10-6	2291-46177-10-7	2291-46177-10-8
Sponsor Designation -	1001-100-5	1001-100-6	1001-100-8	1001-100-10
Date Collected -	12/18/92	12/18/92	12/18/92	12/18/92

Calcium - total	490	520	500	470
Magnesium - total	6,600	11,000	510	1,500
Ammonia (as N)	64	110	6.9	17
Nitrate (as N)	9.0	17	0.54	2.9

ALR Designation -	2291-46177-10-9	2291-46177-10-10
Sponsor Designation -	1001-100-12	1001-104
Date Collected -	12/18/92	12/18/92

Calcium - total	470	300
Magnesium - total	2,700	49,000
Ammonia (as N)	37	510
Nitrate (as N)	4.6	82

By: Eyda Hergenreder
Eyda Hergenreder
Water Laboratory Supervisor

EH/ep