135552

FINAL
FEASIBILITY STUDY REPORT

VOLUME III of IV

APPENDICES A - C

WHITMOYER LABORATORIES SITE
LEBANON COUNTY, PENNSYLVANIA

FEBRUARY 1990
W.A. NO. 200-3LC9

FEBRUARY 2, 1990

FINAL
FEASIBILITY STUDY REPORT
(VOLUME III OF IV)

WHITMOYER LABORATORIES SITE
LEBANON COUNTY, PENNSYLVANIA

EPA WORK ASSIGNMENT NUMBER 200-3LC9
UNDER
CONTRACT NUMBER 68-01-7250

PREPARED BY:
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PITTSBURGH, PENNSYLVANIA

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APPENDIX A

MEDIUM-SPECIFIC CONTAMINATED VOLUME AND QUANTITY CALCULATIONS

A.1 QUANTITY CALCULATIONS FOR THE VAULT AND LAGOONS

NUS CORPORATION AND SUBSIDIARIES — **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DDB	PAGE 1 OF 2
SUBJECT: A.1 Quantity Calculations - Vault, Lagoons		CHECKED BY: MPS	DATE: 8/29/89

Upper vault.

Arsenic:

Assumed density ← ; ↙ conc.

3000 CY x 27 CF/CY x 120 lb/CF x 0.12 = 1,166,400

Say 1,200,000 lb.

Aniline

3000 x 27 x 120 x 0.092 = 894,240

Say 900,000 lb.

Other (N-Nitrosodiphenylamine)

3000 x 27 x 120 x 0.047 = 456,840

Say 450,000

PCE (Not Detected)

3000 x 27 x 120 x < 1×10^{-6} = < 9.7 lb

Say < 10 lb.

Lower Vault

Arsenic

1500 CY x 27 x 120 x 0.157 = 763,020

Say 763,000 lb.

Aniline @ 0.2% = 9720

Say 9700 lb.

Benzene, Xylene, Phenol -N-NDPA, summed avg: 220 mg/Kg

⇒ 0.022% = 1069 lb

Say 1100 lb.

NUS CORPORATION AND SUBSIDIARIES			**STANDARD CALCULATION SHEET**
CLIENT: EPA	FILE NO.: 1317	BY: DDB	PAGE 2 OF 2
SUBJECT: A.1		CHECKED BY: MPS	DATE: 8/29/89

Lagoons

Arsenic 24,000 CY × 27 CF/CY × 90 lb/CF × 0.025 = 1,458,000

assumed density (above 90 lb/CF); 2.5% conc (above 0.025)

Say 1,500,000

Aniline 24,000 × 27 × 90 $<1\times10^{-6}$ = <58 lbs

Others (see Table 4-3 in the RI)

	Vol	Summed Avg Conc. - Organics
Capping Material	930 CY	615 ug/Kg
Soil/Sludge	9700	877
Sludge	5210	49,773
Liner	1310	3,395
Exc. Lagoons	6990	860
	24,140	

$$\text{Vol. Weighted Avg} = \frac{\Sigma\, \text{Vol}_i\, \text{Conc}_i}{\Sigma\, V_i}$$

$$= 11{,}551\ \text{ug/Kg}$$

Total lbs =

24,000 CY × 27 × 90 × 11,551 $\times10^{-9}$ = 673 lb

Say 700 lbs

PCE Avg conc is < 4 ug/Kg

Total lbs PCE < 0.23 lbs

Aniline Avg Conc (Vol wted is) = 138 ug/Kg

Total lbs = 8.1 lb. (Say 8.0 lbs)

A.2 EXTENT OF GROUNDWATER CONTAMINATION

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1$17	BY: LEK	PAGE OF 9
SUBJECT: A.2 - Extent of GW Contamination (Revision 1)		CHECKED BY: DJB	DATE: 9/9/89

AREAL EXTENT

(Refer to attached Appendix Figures)

Arsenic and aniline are most prevalent at the site. Consider outline of shallow and deep contamination for Aniline greater than 10 µg/l and As greater than 50 µg/l

Planimeter Setting / Factor

Planimeter Serial No. 76740

Vernier setting on tracer bar = 14.4

Planimeter Conversion Factor (developed from measurements on graph paper)

Known Areas*	Planimeter Reading		
4 sq in	0.266 0.266 0.263	0.265	0.06625/sq in
10 sq in	0.666 0.660	0.663	0.0663/sq in

Say 0.0663/sq in

* Note: All figures and dimensions presented were based on 11x17 figures. These figures were reduced for incorporation into this report.

Areal Extent

From planimeter trace of Figure A-1, Drawing scale 1" = 600 ft

[illegible]
[illegible]
[illegible] } 1.721

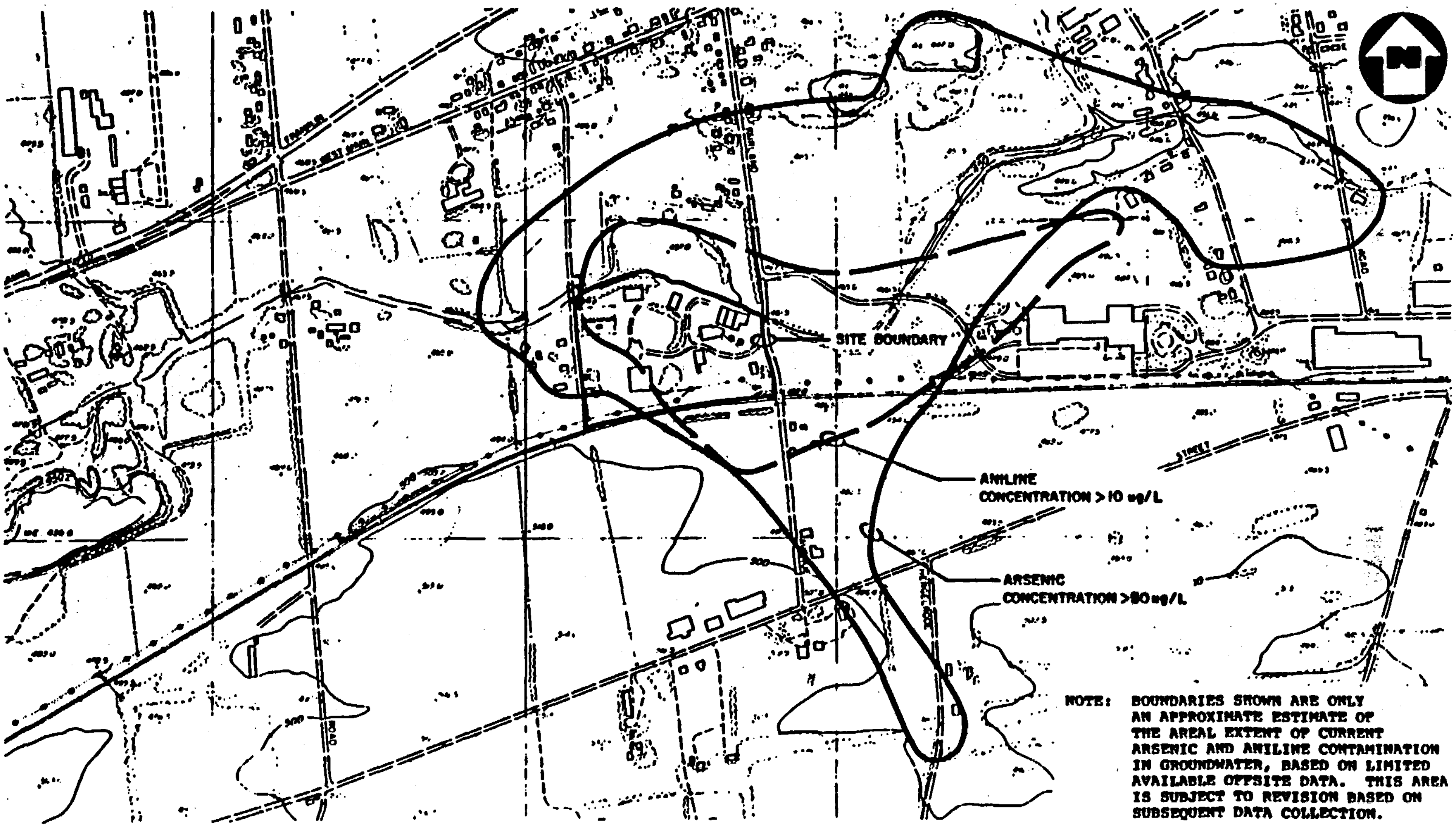
$$\frac{1.721}{0.0663/\text{sq in}} = 25.958 \text{ sq in}$$

Dwg Scale 1" = 600 ft, ∴ 1 sq in = 360,000 sq ft

$$(25.958 \text{ sq in})\left(\frac{360{,}000 \text{ sq ft}}{\text{sq in}}\right)\left(\frac{\text{acre}}{43{,}560 \text{ sq ft}}\right) = 214.6 \text{ acres}$$

Say 215 acres

NUS 155A REVISED 0286



NOTE: BOUNDARIES SHOWN ARE ONLY AN APPROXIMATE ESTIMATE OF THE AREAL EXTENT OF CURRENT ARSENIC AND ANILINE CONTAMINATION IN GROUNDWATER, BASED ON LIMITED AVAILABLE OFFSITE DATA. THIS AREA IS SUBJECT TO REVISION BASED ON SUBSEQUENT DATA COLLECTION.

* Figure reduced from original 11x17

NUS CORPORATION

Figure A-1 *

WHITMOYER LABORATORIES SITE, LEBANON COUNTY, PA

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1$17	BY: LEK	PAGE 2 OF 9
SUBJECT: A.2 - Extent of GW Contamination		CHECKED BY: DB	DATE: 9/9/89

QUANTITY OF CONTAMINATION

(Refer to Appendix figures illustrating contours, Dwg Scale 1" = 600 ft)

Measure contours 50 µg/l and greater with planimeter

Arsenic (Shallow) - Figure A-2

50 µg/l Contour
1.344
1.366 } 1.348
1.334

100 µg/l Contour
1.040
1.022 } 1.029
1.024

1000 µg/l Contour
0.480
0.490 } 0.489
0.496

10,000 µg/l Contour
0.317
0.320 } 0.317
0.315

100,000 µg/l Contour
0.005
0.010 } 0.008 plus 0.005
0.008
0.010 } 0.008
0.008

0.016

Arsenic (Medium/Deep) - Figure A-3

50 µg/l Contour
1.480
1.468 } 1.482
1.497

100 µg/l Contour
1.226
1.212 } 1.214
1.205

1000 µg/l Contour
0.658
0.665 } 0.664
0.670

10,000 µg/l Contour
0.321
0.321 } 0.320
0.317

100,000 µg/l Contour
0.095
0.090 } 0.093
0.095

Contours For Total Arsenic Shallow (μg/l)

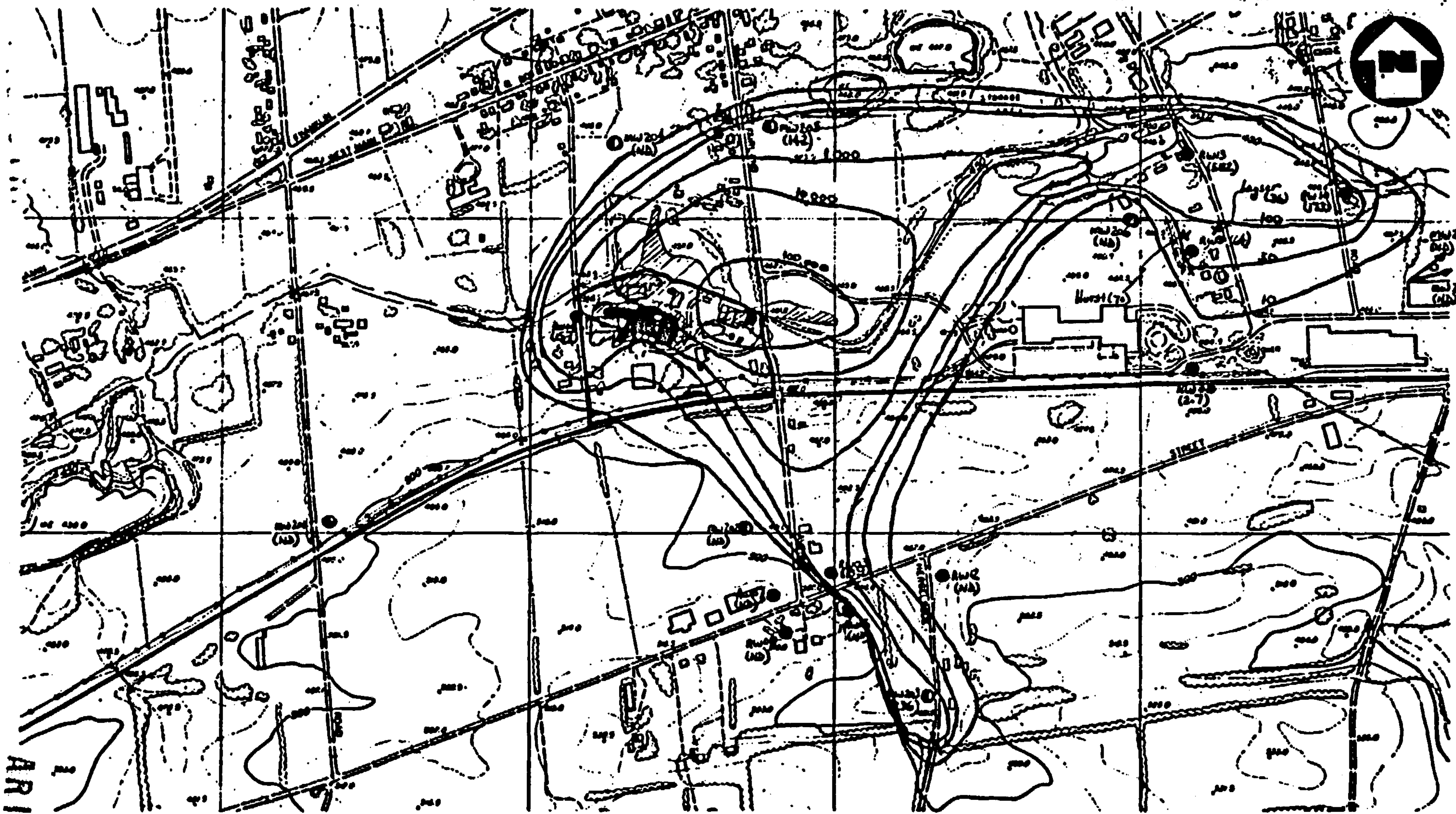
Note: Station 18 & 19 (Quarries) are surface water samples believed to be contaminated via groundwater.

Figure A-2*

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

NUS CORPORATION

* Figure reduced from orginal 11x17"



Contours for Total Arsenic
Medium / Deep (µg/l)

Figure A-3 *

Shaded areas represent
Soil hot spots for arsenic
● 10,000 mg/kg
▨ 1,000 mg/kg

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

NUS CORPORATION

* Figure reduced from
original 11X17

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1$17	BY: LEK	PAGE 3 OF 9
SUBJECT: A.2 Extent of GW Contamination		CHECKED BY: DWB	DATE: 9/9/89

Aniline (Shallow) - Figure A-4

10 µg/l Contour
0.264
0.266 } 0.267
0.272

10,000 µg/l Contour
0.064
0.071 } 0.068
0.068

100 µg/l Contour
0.210 0.006
0.195 } 0.201 plus 0.009 } 0.008
0.199 0.010
0.209

1000 µg/l Contour
0.111
0.110 } 0.112
0.114

Aniline (Medium/Deep) - Figure A-5

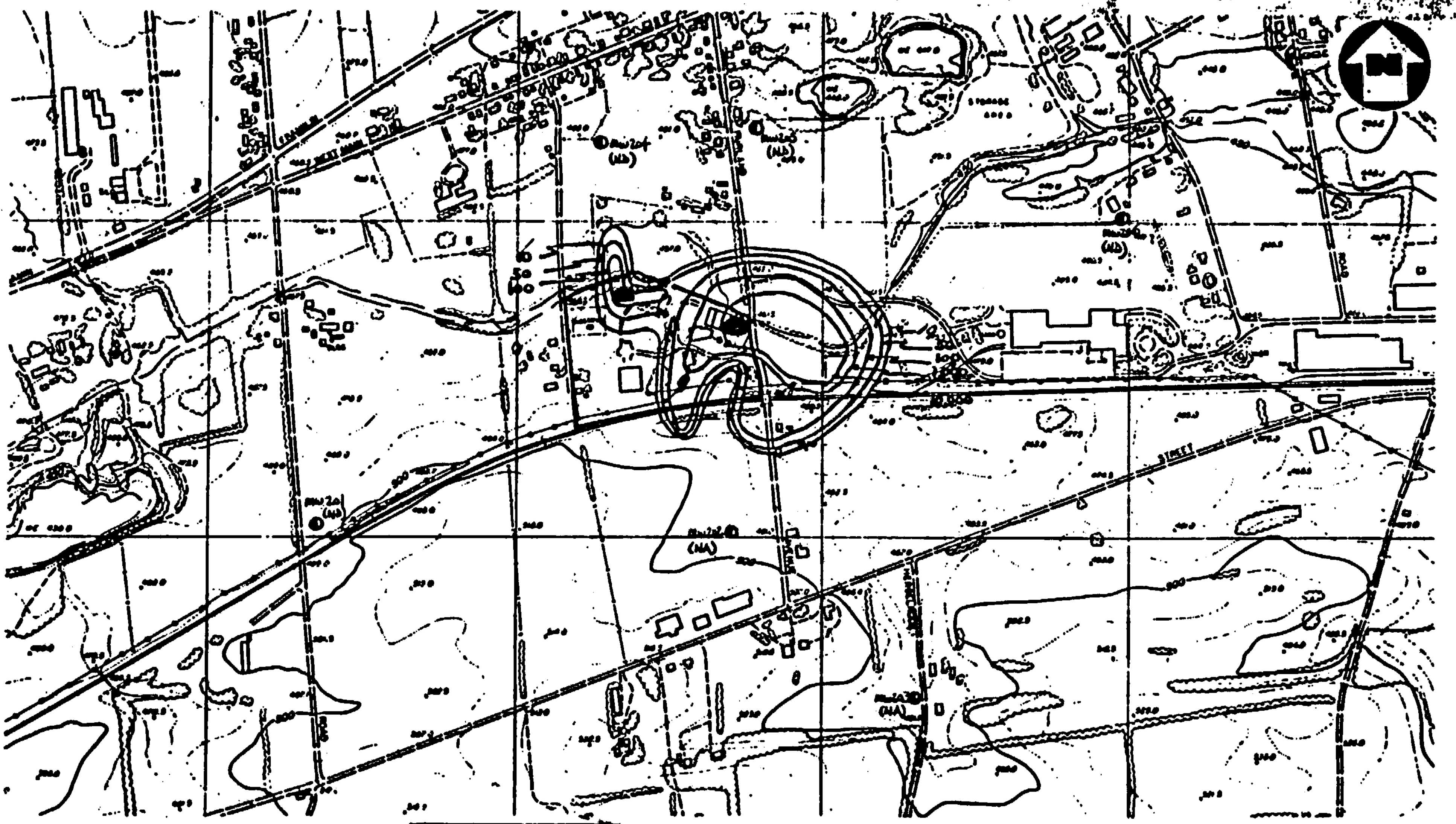
10 µg/l Contour
0.553
0.552 } 0.558
0.570

10,000 µg/l Contour
0.127
0.128 } 0.128

100,000 µg/l Contour
0.040 0.003
0.040 } 0.040 plus 0.005 } 0.004
0.040 0.004
0.044

100 µg/l Contour
0.474
0.470 } 0.471
0.469

1000 µg/l Contour
0.232
0.228 } 0.228
0.224



Contours for Aniline
Shallow (µg/l)

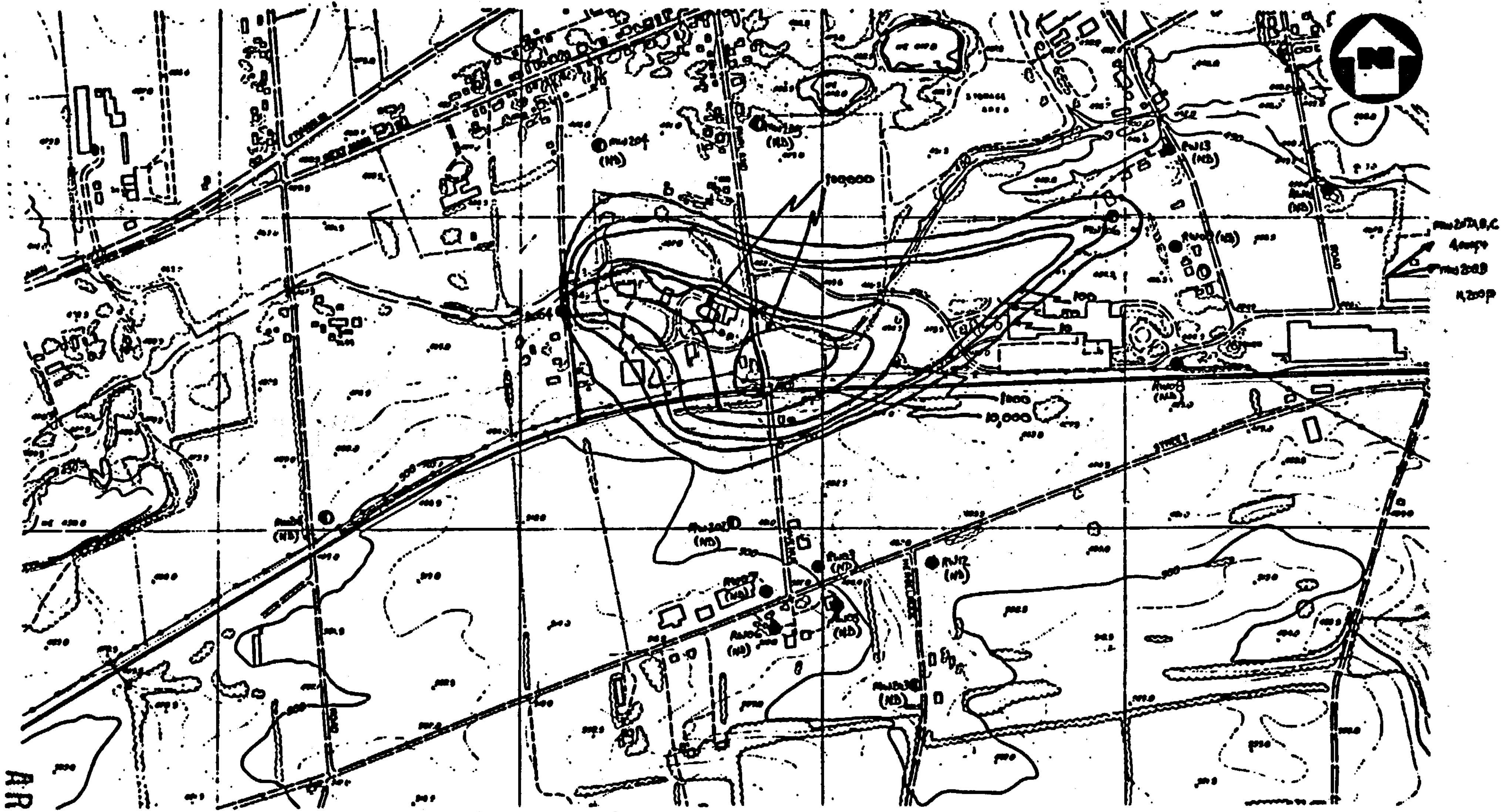
Shaded Areas
represent soil hot spots
for aniline

Figure A-4 *

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

* Figure reduced
from original 11x17"

NUS CORPORATION



Contours for Aniline
Medium/Deep (µg/l)

Figure A-5*

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

* Figure reduced
from original 11x17

0 600 1200
SCALE IN FEET

NUS CORPORATION

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

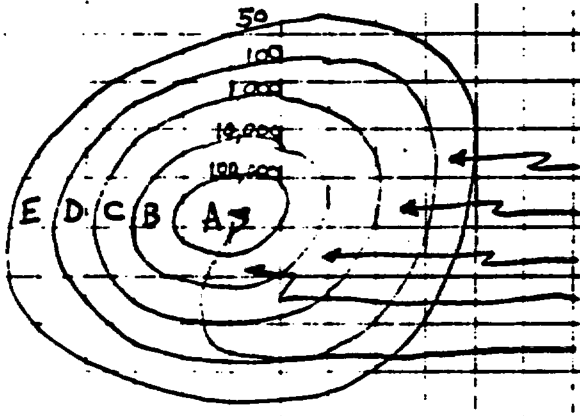
CLIENT: EPA	FILE NO.: 1$17	BY: LEK	PAGE 4 OF 9
SUBJECT: A.2 Extent of GW Contamination		CHECKED BY: DJB	DATE: 9/9/89

General Method for determining quantities

For a given contaminant;

$$\text{Quantity} = \left[\sum_{i=A}^{i=E} (\text{Area}_i)\left(\begin{array}{c}\text{Average conc}\\ \text{of area}_i\end{array}\right)\right]_{\text{Shallow}} \times \text{Depth Zone}_{\text{Shallow}} \times \text{Porosity}_{\text{Shallow}}$$

$$+ \left[\sum_{i=A}^{i=E} (\text{Area}_i)\left(\begin{array}{c}\text{Average conc}\\ \text{of area}_i\end{array}\right)\right]_{\text{Med/Deep}} \times \text{Depth Zone}_{\text{Med/Deep}} \times \text{Porosity}_{\text{Deep}}$$



Average Concentration

E; $\left(\frac{100-50}{2}\right)+50 = 75$ µg/l

D; $\left(\frac{1000-100}{2}\right)+100 = 550$ µg/l

C; $\left(\frac{10,000-1000}{2}\right)+1000 = 5500$ µg/l

B; $\left(\frac{100,000-10,000}{2}\right)+10,000 = 55,000$ µg/l

A; $\left(\frac{\text{Max}-100,000}{2}\right)+100,000 =$ See below

For Arsenic Shallow
Max = 167,050 µg/l
Avg Conc = 133,525 µg/l

For Arsenic Med/Deep
Max = 152,000 µg/l
Avg Conc = 126,000 µg/l

For Aniline Shallow*
Max = 56,667 µg/l
Avg Conc = 33,334 µg/l

For Aniline Med/Deep
Max = 222,350 µg/l
Avg Conc = 161,175 µg/l

Note: Arithmetic averages were used for average concentrations of each ring to provide a more conservative approach (e.g. Ring C average conc. by arithmetic method = 5500 µg/l as shown; avg. conc. by logarithmic method = 3162 µg/l)

* Aniline Shallow max. conc. does not attain 10^5 µg/l range ∴ No A is required. Use these values for B

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DDB	PAGE 5 OF
SUBJECT: A.2 Extent of Groundwater Cont.		CHECKED BY: LEK 1/29/90	DATE: 1/25/90

Arsenic Quantity

Shallow Zone

Quantity = (Σ Area × Avg Conc) × Depth × Porosity

Σ Area × Avg Conc = [0.016 × 133,525 + (0.317 − 0.016) × 55,000 + (0.489 − 0.317) × 5500 + (1.029 − 0.489) × 550 + (1.348 − 1.029) × 75] μg/l

$$\times \left[\frac{360{,}000 \text{ Actual SF / SI Dwg}}{0.0663 \text{ / SI Dwg}}\right] \times \frac{1 \text{mg}}{1000 \mu\text{g}} \times \frac{\text{g}}{1000 \text{mg}} \times \frac{\text{lb}}{454 \text{g}} \times \frac{\text{l}}{0.03531 \text{ft}^3}$$

= 6,760 lb/ft of water

Depth = 31 ft
Eff Por. = 0.01

Quantity = 6,760 × 31 × 0.01 = 2100 lb

Medium/Deep

Σ Area × Avg Conc = [0.093 × 126,000 + (0.32 − 0.093) × 55,000 + (0.664 − 0.32) × 5,500 + (1.214 − 0.664) × 550 + (1.482 − 1.214) × 75 μg/l

× [ ] × ( ) × ( ) × ( ) × ( )

From Above

= 8,950 lb/ft of water

Depth = 500 − 31 = 469 (Assumed)
Eff Por. = 0.01

Quantity = 8,950 × 469 × 0.01 =

= 42,000 lb

Total = 2100 + 42,000 = 44,100 say 44,000 lb

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	**FILE NO.:** 1817	**BY:** DDB	**PAGE** 6 **OF**
SUBJECT: A.2 - Extent of GW. Contamination		**CHECKED BY:** LEK 1/29/90	**DATE:** 1/25/90

Aniline Quantity

Shallow Zone

Σ Area x Avg Conc = [0.068 x 33334 + (0.112 - 0.068) x 5,500 +
(0.209 - 0.112) x 550 + (0.267 - 0.209) x 75)
x [ ] ( ) - from previous page

= 869 lb/ft

Quantity = 31 x 0.01 x 869 = 270 lb

Medium / Deep

Σ Area x Avg Conc = (0.044 x 121,175 + (0.128 - 0.044) x 55,100
+ (0.228 - 0.128) x 5500 + (0.471 - 0.228) x 550 +
(0.558 - 0.471) x 75) x [ ] ( ) from
previous page

= 4200 lb/ft

Quantity = 4200 x 0.01 x 469 = 19,700 lbs

Total = 270 + 19,700 = 19,970
Say 20,000 lbs

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1$17	BY: LEK	PAGE 7 OF 9
SUBJECT: A.2 Extent of GW Contamination		CHECKED BY:	DATE: 3/9/89

AVERAGE CONCENTRATION OF GW PLUME

Arsenic - Shallow

		Area (Planimeter)	Weighted Area
A	Area = 0.016	= 0.016	0.012
B	Area = (0.317-0.016)	= 0.301	0.223
C	Area = (0.489-0.317)	= 0.172	0.127
D	Area = (1.029-0.489)	= 0.540	0.400
E	Area (1.348-1.029)	= 0.319	0.237
		1.348	0.999

$$\sum_{i=A}^{i=E} (\text{Weighted Area})_i \left(\text{Avg conc. of area}_i\right) = \text{Average Conc.}$$

↑ see page 4

$$\text{Arsenic}_{\text{Shallow}} = (0.012)(133{,}525) + (0.223)(55{,}000) + (0.127)(5{,}500) + (0.400)(550) + (0.237$$

= 14,803 μg/l   Say 15 mg/l

Arsenic - Medium / Deep

		Area (Planimeter)	Weighted Area
A	Area = 0.093	= 0.093	0.063
B	Area = (0.320-0.093)	= 0.227	0.153
C	Area = (0.664-0.320)	= 0.344	0.232
D	Area = (1.214-0.664)	= 0.550	0.371
E	Area = (1.482-1.214)	= 0.268	0.181
		1.482	1.000

$$\text{Arsenic}_{\text{Med/Deep}} = (0.063)(126{,}000) + (0.153)(55{,}000) + (0.232)(5500) + (0.371)(550) + (0.181)(75)$$

= 17,847 μg/l   Say 18 mg/l

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA [illegible]	FILE NO.: 1$17	BY: LEK	PAGE 8 OF 9
SUBJECT: A.2 Extent of GW Contamination		CHECKED BY: DWB	DATE: 8/30/89

Aniline - Shallow

		Area (Planimeter)	Weighted Area
A	NA		
B	Area = 0.068	= 0.068	0.255
C	Area = (0.112-0.068)	= 0.044	0.165
D	Area = (0.209-0.112)	= 0.097	0.363
E	Area = (0.267-0.209)	= 0.058	0.217
		0.267	1.000

$\text{Aniline}_{\text{Shallow}} = (0.255)(33,334)+(0.165)(5,500)+(0.363)(550)+(0.217)(75)$

$= 9,624\ \mu g/l$ Say 10 mg/l

Aniline - Medium/Deep

		Area (Planimeter)	Weighted Area
A	Area = 0.044	= 0.044	0.079
B	Area = (0.128-0.044)	= 0.084	0.151
C	Area = (0.228-0.128)	= 0.100	0.179
D	Area = (0.471-0.228)	= 0.243	0.435
E	Area = (0.558-0.471)	= 0.087	0.156
		0.558	1.000

Aniline [illegible] = (0.079)(161,175)+(0.151)(55,000)+(0.179)(5,500)+(0.435)(550)+(0.156)(75)

$= 22,273\ \mu g/l$ Say 23 mg/l

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	**FILE NO.:** 1817	**BY:** DDB	**PAGE** 9 **OF** 9
SUBJECT: A.2 Extent of GW Contamination		**CHECKED BY:** LEK 1/29/90	**DATE:** 1/25/90

Area of Contamination is about 215 acres.

Vol of GW = 215 acres × $\frac{43{,}560\ \text{SF}}{\text{acre}}$ × 500 ft deep × 0.01 (Porosity)

× $\frac{7.48\ \text{gal}}{\text{cf}}$

= 350,000,000 gallons

A.3 PCE QUANTITY IN GROUNDWATER

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DDB	PAGE 1 OF 1
SUBJECT: A.3 PCE Quantity in GW,		CHECKED BY: LEK 1/28/90	DATE: 1/25/90

From the RI

Average Onsite PCE Concentration : 2.45 mg/l

Average Offsite PCE Concentration : 0.00148 mg/l

Onsite Area: 22 acres
Offsite Area + Onsite Area } : 215 acres

Average PCE Concentration (In As Plume)

$$\frac{22}{215} \times 2.45 + \frac{(215-22)}{215} \times 0.00148$$

$$= 0.252 \text{ mg/l}$$

Total Quantity of PCE in GW

$$350{,}000{,}000 \text{ gal} \times \frac{8.34 \text{ lb}}{\text{gal}} \times 0.252 \times 10^{-6} \frac{\text{lb PCE}}{\text{lb } H_2O}$$

$$= 730 \text{ lb}$$

A.4 HISTORIC GROUNDWATER DATA ANALYSIS

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA [illegible]	FILE NO.: 1$17	BY: LEK	PAGE 1 OF 3
SUBJECT: Recent Historical GW Data Analysis		CHECKED BY: DDB	DATE: 9/9/89

A.4

Arsenic - Figure A-6

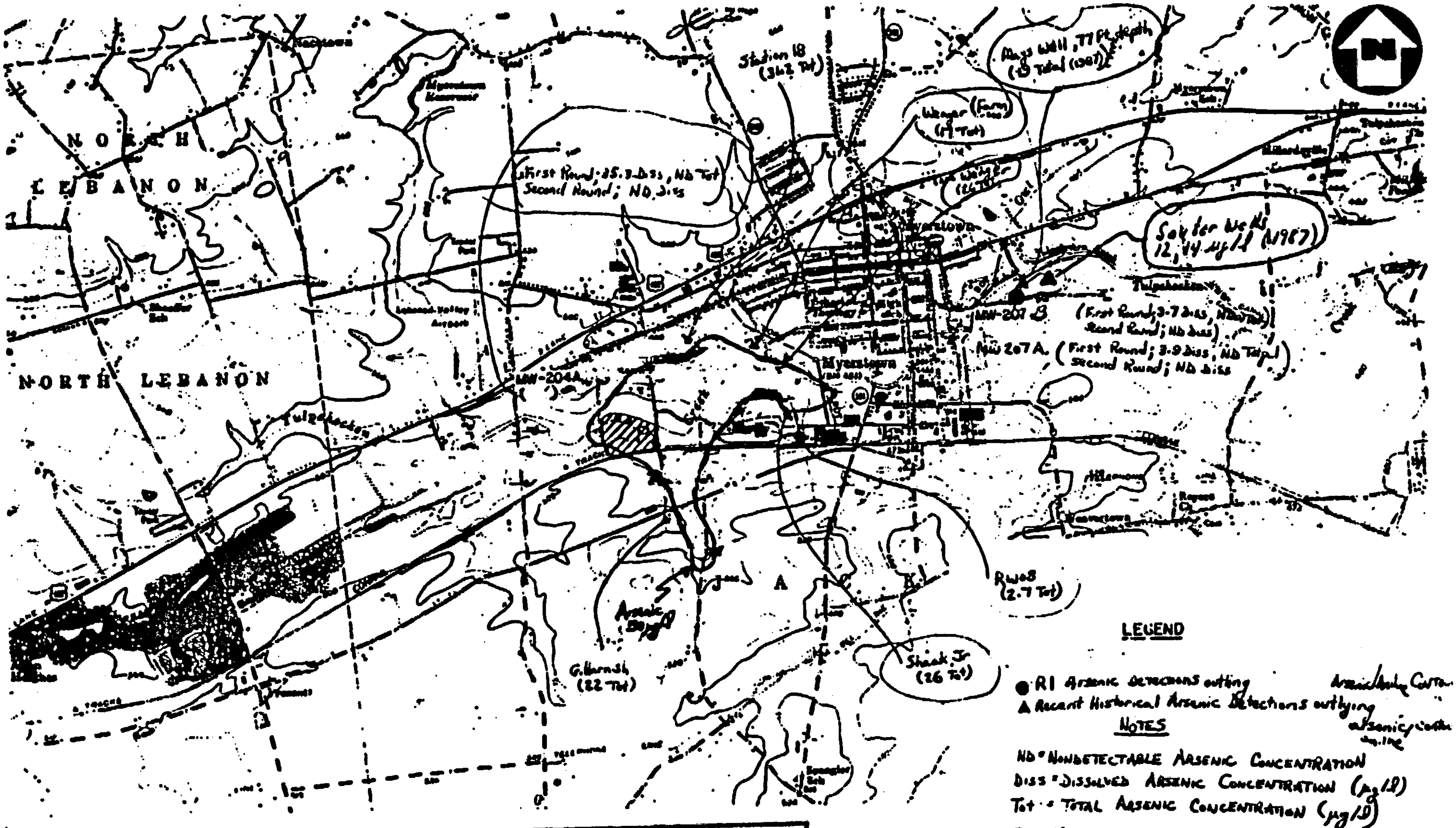
Offsite Residential Wells not sampled during the RI for purpose of correlating with contamination contours dvpd from RI data

Most Recent Historical Data (RI Table 4-30)

Name	Well Depth	Arsenic (if ≥10 µg/l)	Fig. 3-16 RI Ref ID.
Peiffer	100	50 (1978)	Not shown (NS)
Hurst	—	70 (1987)	20
Layser	—	36 (1987)	21
Wenger	—	26 (1987)	18
Wenger (Farm)	—	17 (1987)	17
→ Mays	77	19 (1987)	31
Sauter (Diem)	100	12 (1987)	NS
Sauter (Barn)	—	14 (1987)	NS
Donmoyer	100	4,630 (1971) WC	NS
High	—	83 (1987)	11
Moore	230	100 (1975)	NS
Harnish, G.	—	22 (1987)	10
Shank, Jr.	—	26 (1987)	22
Werthlift	—	70 (1972)	NS

Conclusions:

1. [illegible] historical well data is not [illegible] the RI Figure 3-16. (NS designation [illegible] shown)
2. [illegible] correlates well with RI data used to plot contours.
3. Lack of well depth information results in no refinement of shallow contours
4. The Mays well and Sauter wells (located close to MW207,A,B,C from RI) indicates that the 10 µg/l contour should be open ended and should follow Tulpehocken Creek



LEGEND

● RI Arsenic detections outlying Arsenic/Aniline Contamination
▲ Recent Historical Arsenic Detections outlying arsenic contamination

NOTES

ND = Nondetectable Arsenic Concentration
Diss = Dissolved Arsenic Concentration (µg/l)
Tot = Total Arsenic Concentration (µg/l)

50µg/l Arsenic Contour Corresponds with MCL

0 2000 4000
SCALE IN FEET

NUS CORPORATION

GROUNDWATER ARSENIC DETECTIONS OUTLYING Arsenic/Aniline Plume

Figure A-6

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1517	BY: LEK	PAGE 2 OF 3
SUBJECT: 2.4 GW Data Analysis		CHECKED BY: MPS	DATE: 3/9/89

Aniline and PCE

Offsite Wells - Figure A-7

For purpose of 1. checking for hits of PCE above MCLs & outlying arsenic/aniline plume
2. checking for hits of possible PCE biodegradation products above MCLs & outlying arsenic/aniline plume
3. correlating aniline data w/ contamination contours developed from RI

Most Recent Historical Data

Name	Well Depth	Aniline	PCE (if ≥5µg/l)	Fig 3-16 RI Ref ID
D. Wagner (Gibble)	185	—	11 (1987)*	7
High	—	—	8.5 (1987)	11
Schoen	—	—	5.7 (1987)	15

* measured at 28 µg/l during the RI

Conclusions

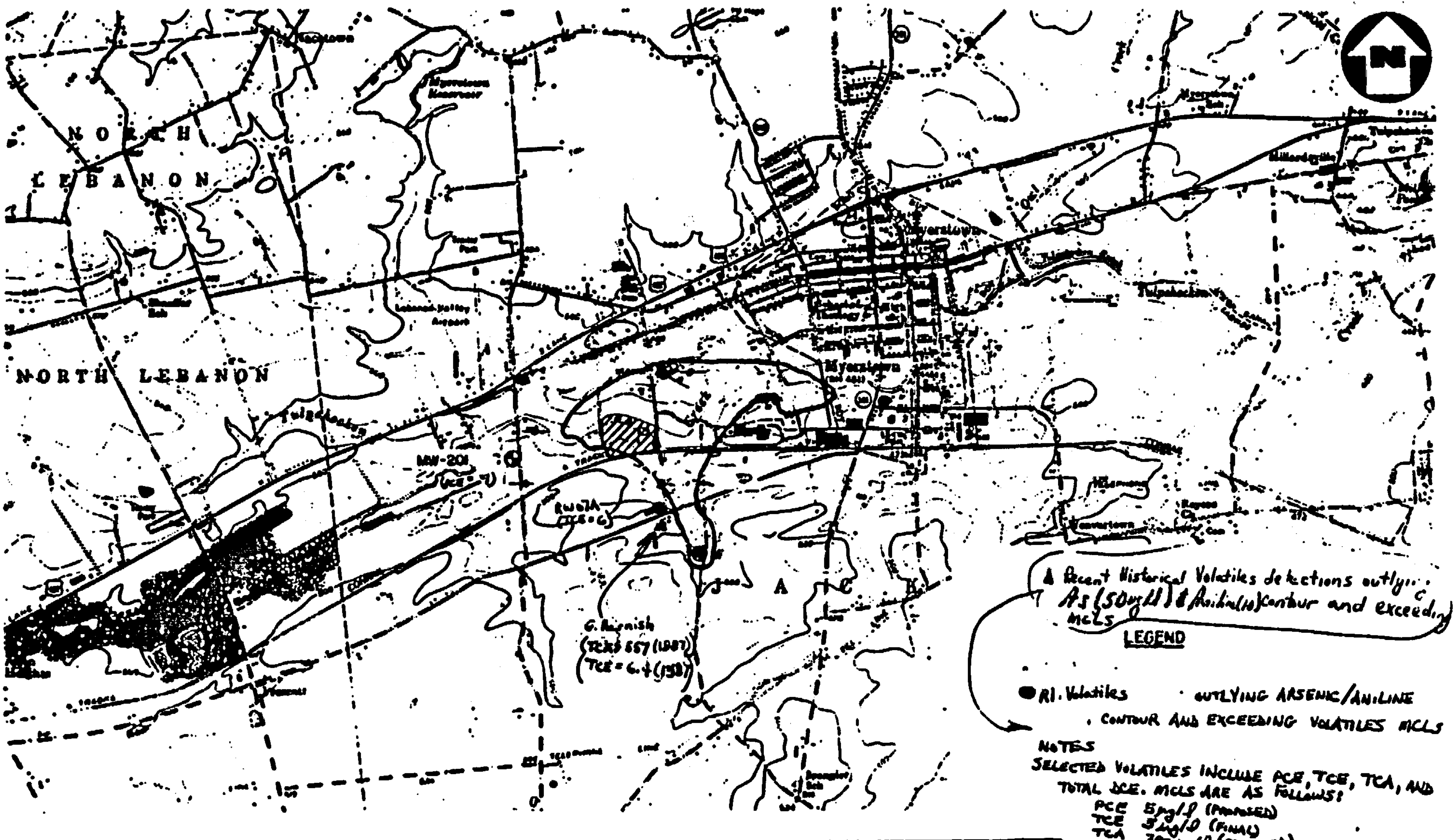
1. Much of historical data, particularly for aniline, had high detection limits, therefore some data is not usable.
2. Usable data indicates that the hits listed above lie within the designated groundwater plume ; MCL for PCE = 5µg/l (proposed) for arsenic/aniline.

PCE, TCA, and Possible PCE biodegradation products (TCE, Total 1,2 DCE)

Most Recent Historical Data

Name	Well Depth	TCA (if ≥200µg/l)	TCE (if ≥5µg/l)	Fig 3-16 RI Ref ID
D. Wagner (Gibble)	185	425 (1987)*	19 (1987)*	7
High	—	557 (1987)	16 (1987)	11
G. Harnish	—	275 (1987)	6.4 (1987)	10
Shoen	—	—	8.8 (1987)	15

* During the RI ([illegible]), measured results were TCA: 105 µg/l & TCE: 5.6 µg/l



NOTES
SELECTED VOLATILES INCLUDE PCE, TCE, TCA, AND TOTAL DCE. MCLS ARE AS FOLLOWS:

PCE 5 µg/l (PROPOSED)
TCE 5 µg/l (FINAL)
TCA 200 µg/l (PROPOSED)
TOTAL 1,2 DCE 70/100 µg/l (PROPOSED)
1,1 DCE 7 µg/l (FINAL)

0 2000 4000
SCALE IN FEET

NUS CORPORATION

GROUNDWATER SELECTED VOLATILES EXCEEDING MCLS AND OUTLYING ARSENIC/ANILINE 50 µg/l CONTOUR

Figure A-7

WHITMOYER LABORATORIES SITE, MYERSTOWN, PA

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA, [illegible]	FILE NO.: 1$17	BY: LEK	PAGE 3 OF 3
SUBJECT: A.4 GW Data Analysis		CHECKED BY: MPS	DATE: 9/9/89

1. Most recent historical data shows no hits for 1,1 DCE exceeding MCL (7 µg/l proposed)
2. Except for the G. Harnish well, the hits listed above lie within the designated groundwater plume for arsenic/aniline. Note that the G. Harnish well is upgradient of the estimated arsenic/aniline groundwater plume.
3. Halogenated organic contamination could be due to offsite source area(s)

A.5 GROUNDWATER CONTAMINANTS ASSOCIATED WITH SOILS IN FRACTURES

NUS CORPORATION AND SUBSIDIARIES | **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DDB	PAGE 1 OF 1
SUBJECT: 2.5 GW contaminants associated with soils in fractures		CHECKED BY: LEK 1/29/90	DATE: 1/25/90

Based on Model presented in Appendix B.1, p-3, ratio of soil to water is

$$\frac{0.027\text{ CF-Soil}}{0.018\text{ CF-water}} = \frac{1.5\text{ CF-Soil}}{\text{CF-water}}$$

The corresponding weight ratio is

$$\frac{1.5\text{ CF-Soil} \times \frac{165\text{ lb}}{\text{CF}}}{\text{CF-Water} \times 62.4\text{ lb/CF}} = \frac{4\text{ lb soil}}{\text{lb water}}$$

Determine Distribution Coefficients (Kd)

Parameter	Kd $\left(\frac{\text{mg/kg}}{\text{mg/l}}\right)$	Source
Arsenic	5	RI - p317, Lyman
PCE	3.64	KOC × TOC = Kd 364 × 0.01 (assumed TOC)
Aniline	0.44	44 × 0.01

Quantity Summary

Parameter	Quantity in Water (RI, p586 A3)	Calculated Quantity in Soil*	Total
Arsenic	44,000 lb	880,000 lb	924,000 lbs Say 920,000 lbs
Aniline	20,000 lb	35,000 lb	55,000 lbs
PCE	730 lb	10,600 lb	11,430 lb Say 11,000 lb

* Quantity in Soil = Quantity in water × weight ratio × Kd

NUS 166A REVISED 0286

A.6 QUANTITY CALCULATIONS FOR SITE STRUCTURES

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	**FILE NO.:** 200-3LC9	**BY:** JJT	**PAGE** 1 **OF** 5
SUBJECT: Contaminated Building Area + Volume Estimates		**CHECKED BY:** LEK 1/29/90	**DATE:** 1/19/90

Estimated volume of contaminated buildings

Bldg 4-14 · 25X30 + 25X30 + 18X25 + 8X12 = Area = 2046 SF
Perimeter = 25+30+25+15 +30+5+25 +5+12+8+12+12 +25+5 +25 = 259 LF
Internal Walls = 25+25 = 50 LF

Bldg 1 50x70 Perimeter 240 LF Area 3500 SF
Walls = 240 X 16 = 3840 sq ft Roof = 3500 X 1.3 = 4550 sq ft

Bldg 2 50X120 Perimeter = 340 LF Area = 6000 SF
Walls = 340 X16 = 5440 SF Roof = 6000 X 1.3 = 7800 SF

Bldg. 3 50x70 Perimeter 240 LF Area = 3500 SF
Walls = 240X16 = 3840 SF Roof = 3500 X 1.3 = 4550 SF

Bldg 6 90X50 + 20X40 Perimeter 270 LF Area = 5300 SF
Walls = 270 X 16 = 4320 SF Roof = 5300 SF

Bldg 7 50 X50 Perimeter = 200 LF Area = 2500 SF
Walls = 200 X 16 = 3200 SF Roof = 2500 SF

Bldg 1-7 Total Area = 20,800 SF Total Roof = 24,700

Wall Area for Demolition • Bldg 1-7 complex
External (150 + 120 + 190 + 70 + 40 + 50) x 16 = 620 X 16 = 9920 sq ft
Internal (120 + 120 + 150 + 20) x 16 = 410 X 16 = 6560 SF
Vault 124 X 83 Area = 10290 SF Roof = 10292 X 1.3 = 13,380 SF Perimeter = 414 LF
Bldg 5 Area = (90 X 95) + (40X20) + (20 x 75) + 30 X 55 + [(130x20)/2] + 65x25/2 = 13,500 SF
Interior Walls = 100 LF Total Wall Area = 615 x 40 = 24600 SF
Perimeter = 140 + 45 + 65' + 20 + 30 + 15 + 20 + 40 + 20 + 35 + 95 = 515 LF
Bldg 16 40X20 Area = 800 SF Perimeter = 120 LF Wall Area = 120 X 10 = 1200 SF
Bldg 17 15X15 Area = 225 SF Perimeter = 60 LF Wall Area = 60 x 10 = 600 SF
Bldg 11 Area = (30X12) + 20X20 + 25X15 = 1135 SF Perimeter = 164 SF Wall Area = 164 x 10 = 1640
Bldg 9 60X100 Area = 6000 SF Perimeter = 320 LF Wall Area = 320 x 20 = 6400 SF
Bldg 12 15X 25 Area = 375 SF Perimeter = 80 LF Wall Area = 80 x 10 = 800 SF
Bldg 8-15 65X95 + 15X40 Area = 6775 SF Perimeter = 95 + 65 + 135 + 15 + 40 + 50 = 400 LF
Interior Walls = (3X15) + 95 = 140 LF Wall Area = (400-95) x 20 + (140+95) x 10 = 8450
Total Area = 49,600 SF = 1.14 acres under roof

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 200-3LC9	BY: JJI	PAGE 2 OF 5
SUBJECT: Dike Areas + Perimeter Concrete Volumes		CHECKED BY: LEK 1/29/90	DATE: 1/19/90

Assume 1' floor and wall thickness and 3' wall height

TA0013 and TA0014 dike 35' x 30'
Area = 1050 SF Floor = 1050 CF = 39 CY
Walls = 130 LF X 3' high = 390 SF Wall Volume = 390 CF = 15 CY
Total = 54 CY

Fish Oil Tank dike 30 X 48'
Area = 1440 SF Floor = 1440 CF = 53 CY
Walls = 156 X 3' high = 468 SF Wall Volume = 17 CY Total = 70 CY

Upper Aniline Tank Dike 25 X 20
Area = 500 SF Floor = 500 CF = 19 CY
Walls = 90 LF X 3' high = 270 SF Wall volume = 270 CF = 10 CY Total = 29 CY

Lower Aniline Tank Dike 30 X 20
Area = 600 SF Floor = 600 CF = 22 CY
Walls = 100 LF X 3' high = 300 SF Wall volume = 300 CF = 11 CY Total = 33 CY

Fuel Oil Tank Dike 10 X 25
Area = 250 SF Floor = 250 CF = 9 CY
Walls = 70 LF X 3' high = 210 SF Walls = 210 CF = 8 CY Total = 17 CY

PCE - Aniline Tank Dike $(25 \times 55) - \frac{1}{2}(12)(20)$
Area = 1255 SF Floor = 1255 CF = 46 CY
Perimeter = 25 + 55 + 13 + 25 = 118 LF X 3' high = 354 SF Walls = 354 CF = 13 CY Total = 59 CY

TA0001 dike = (52 X 30) - ½ (15) (23) (apron)
Area = 1390 SF Floor = 1390 CF = 52 CY
Perimeter = 28 + 30 + 53 + 28 + 19 + 15 = 173 LF X 3' high = 519 SF Walls = 519 CF = 19 cy Total = 71 CY

TA0002 dike 40 X 18 Area = 720 SF Floor = 720 CF = 27 CY
Perimeter = 116 LF X 3' high = 348 SF Walls = 348 CF = 13 CY Total = 40 CY

Waste Pit 7.5(W) X 13'(L) X 6' H Floor = 7.5 X 13 X 1.5' (THICK) = 146 CF = 5.4 CY
Walls = (7.5 + 13) X 2 X 6' X 1.25 thick = 307.5 CF = 11.4 CY Total = 17 CY

Total - All Dikes = 54 + 70 + 29 + 33 + 17 + 59 + 71 + 40 = 373 CY

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NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DDB	PAGE 3 OF 5
SUBJECT: A.6 Quantity - Site Structures		CHECKED BY: LEK 1/29/90	DATE: 1/25/90

Building 1-7 Complex is saturated with contamination.

Wood Flooring is prominent in Buildings 1, 2, & 3.

Area = 150×70 + 50×50 = 13,000SF

Wood roofing in Bldg 6 & 7 is saturated with Arsenic dust

Surface Area = 5300 + 2500 = 7800SF

Building 1, 2 & 3 roofs are also contaminated

Surface area = 4550 + 7800 + 4550 = 16,900SF

Vault Roof

Surface Area = 13,380 SF

Total 13,000 + 7800 + 16,900 + 13,380 SF = 51,080 Say 51,000 SF

~~Assume roofing & flooring lime joists are 3" thick~~

51,000 SF × 0.25 ft × CY/27 CF = 47[illegible]0 CY

NUS 166A REVISED 0286

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA
FILE NO.: 1317
BY: DJB
PAGE 4 OF 5
SUBJECT: A6-Quantity of Site Structures
CHECKED BY: MPS
DATE: 9/26/87

General building Demolition Volume (Building 1-7)
= 20,800SF x 2 stories high

Concrete

Building 1-7 = 20,800 SF x 1' thick /27 = 770CY
Plus basement in building 3
thick ↙ plan ↙ walls →
1' x (45x70 + 2x70 x 8' + 2 x 50' x 8')/27 = 190CY
Vault 1' x (124x83 + 2x124x12 + 2 x 83x12)/27 = 565CY
Dikes from page 2 = 375CY
Total Concrete = 1900CY
Say 2000CY

Surface cleaning - Interior, dust Only
Roof + 2 x intermediate flooring + floor
+ Perimeter x height

Building 1-7 24,700SF + 2x1x20,800 + 20,800 + 9920 + 1560 = 103,580SF *
Building 16 800 x (1+1) + 1200 = 2,800SF
Building 4-14 2050 + 2050 + 260 x 10' = 6,700SF
Building 17 225 x (1+1) + 600 = 1050SF
Building 5 13,500(1+2x2 +1] + 615' x 40' = 105,600SF
Building 12 375 (1+1) 1800 = 1,550SF
Building 8-15 2800(1+1+1) + 8450 28,850SF
Building 9 6000(1+1+1) + 320 x 20 = 24,400SF
Building 11 1440(1+1) + 1640 = 4,550SF
Vault 13,880 + 10,290 + 414x12 = 28,640SF

Total 307,100

* To minimize contaminant migration during demolition

Say 350,000SF

NUS CORPORATION AND SUBSIDIARIES **STANDARD CALCULATION SHEET**

CLIENT: EPA	FILE NO.: 1817	BY: DAB	PAGE 5 OF 5
SUBJECT: A.6 Quantity of Site Structures		CHECKED BY: MPS	DATE: 9/26/89

Data for tanks, Asbestos are taken from R.I. and Concentrated Liquids Assessment

Volume from cut up piping, tanks, vessels is expected to be relatively minor

Total Volume of Materials to be disposed

Wood	500 CY
Concrete	2000 CY
Bldg 1-7 Walls 1' thick	610 CY
	3110 CY
Contingency - Misc - 60%	1890 CY
	5000 CY

APPENDIX B

CLAY PELLETIZING AND SINTERING TREATABILITY STUDY

TREATABILITY STUDY

WHITMOYER LABORATORIES SITE
JACKSON TOWNSHIP, PENNSYLVANIA

CLAY PELLETIZING AND SINTERING
FINAL REPORT

SUBMITTED TO:

NUS CORPORATION
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PREPARED BY:

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EXECUTIVE SUMMARY

An experimental investigation was conducted to determine if Whitmoyer Laboratories Site waste materials could be stabilized by mixing with clay, pelletizing, then sintering at an elevated temperature. The Phase I test work demonstrated that appreciable arsenic was volatilized from the test materials during the sintering operation. Also, TCLP leachability of the treated waste mixtures exceeded the hazardous characteristic level for arsenic. This treatability technology does not appear to be appropriate for application to the Whitmoyer waste materials.

The results of the Phase I study showed that further clay/sample pelletizing and sintering test work was not warranted. Therefore, the Phase II study emphasis was shifted to investigate on a smaller scale, in more detail, the effect of roasting the starting materials as a function of temperature and to investigate the potential stabilizing influence of thiourea without roasting. Included in the roast study were experiments on cement/sample and lime/sample mixtures. These studies were conducted to determine if the unmixed, untreated lagoon and vault materials could be stabilized by roasting; to determine the importance of the Ca/As or Fe/As ratios and temperature on stabilizing the waste materials; and to determine if cement/thiourea/sample mixtures could be stabilized without roasting.

The results of the above screening studies showed that roasting the unmixed starting materials did not stabilize the wastes. However, the cement/sample and lime/sample test work demonstrated that the waste materials were stabilized by roasting in air at 600°C - 700°C, i.e., the TCLP leachability results showed that less than the hazardous characteristic level of arsenic was released. The results on the thiourea containing materials appeared to have potential for stabilization and the screening study was followed up by a detailed leachability evaluation. The results of the product leachability tests showed essentially no stability enhancement by the presence of thiourea.

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Health and Safety Report Attached.

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SECTION FIVE: REMEDIAL DESIGN AND COST ESTIMATES

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SECTION FOUR: RESULTS AND DISCUSSION

SECTION FIVE: REMEDIAL DESIGN AND COST ESTIMATES

SECTION SIX: PRELIMINARY SCHEDULE

SECTION EIGHT: APPENDICES

SECTION ONE
STUDY OBJECTIVE

The *objectives* of the clay pelletizing and sintering studies were:

- to determine the feasibility of using clay pelletizing and sintering technology to reduce the leachability of arsenic in the Whitmoyer sludge and solids to a level which would permit the material to be successfully delisted as a hazardous waste and thereby allow for onsite, nonhazardous disposal.

- to collect data to establish preliminary sizing and operating requirements for the design of a full scale remediation program.

The tasks that were performed to accomplish the objectives were divided into two phases of test work. The *task objectives for the Phase I* work included:

- Preparation of designated clay/sample mixtures for three supplied samples, i.e., clay/sample ratios of 1/3 and 3/1 for vault, lagoon, and soils materials.

- Pelletizing the sample mixtures in a disk pelletizer to form agglomerated pellets of a specified size.

- Sintering under specified conditions a portion of each clay/sample ratio, i.e., one-half of the mixed samples were sintered at 1000°C for fifteen minutes.

The *task objectives for the Phase II* work included:

- Performance of a heat treatment screening study to determine the influence of temperature on:

    - the starting vault and lagoon materials (at 600°C, 700°C, 800°C, and 1000°C),

    - a mixture of cement/vault sample (3/1 ratio at 600°C, 700°C, and 800°C), and

    - a mixture of lime/vault sample (1/1 ratio at 700°C).

- Determination of the response of the roast treatments to a modified TCLP test.

- Mixing large scale batch mixtures of each supplied sample material with an equal amount of cement and one weight percent thiourea; mixing with water and casting into cubes of a specified size; aging for twenty eight days; and supplying the resultant products to NUS for evaluation test work.

SECTION TWO
METHODS

2.1. SAMPLE IDENTIFICATION SYSTEM

The sample identification system used throughout this study is presented in Table 2.1.

TABLE 2.1. SAMPLE IDENTIFICATION SYSTEM

Sample	Description	Identification No.
Starting Sample		
WL-LA 0001-1	As-received Lagoon Sample	LA 0001
WL-LA 0002-1	As-received Soil Sample	LA 0002
WL-VT 0002-1	As-received Vault Sample	VT 0002
	Phase I	
Clay/Sample Ratio		
1/3	Mixture Pelletized and Sintered	LA 0001A,1C
3/1	Mixture Pelletized and Sintered	LA 0001A,3C
1/3	Mixture Pelletized and Sintered	LA 0002A,1C
3/1	Mixture Pelletized and Sintered	LA 0002A,3C
1/3	Mixture Pelletized and Sintered	VT 0002A,1C
3/1	Mixture Pelletized and Sintered	VT 0002A,3C
	Phase II ***Screening Study***	
Starting Sample		
LA 0001	Sample Roasted at 600°C	LA 0001,600R
	Sample Roasted at 700°C	LA 0001,700R
	Sample Roasted at 800°C	LA 0001,800R
	Sample Roasted at 1000°C	LA 0001,1000R

TABLE 2.1. SAMPLE IDENTIFICATION SYSTEM (Continued)

Sample	Description	Identification No.
VT 0002	Sample Roasted at 600°C	VT 0002,600R
	Sample Roasted at 700°C	VT 0002,700R
	Sample Roasted at 800°C	VT 0002,800R
	Sample Roasted at 1000°C	VT 0002,1000R
Cement/Sample Ratio		
3/1	Sample Roasted at 600°C	VT 0002,3C,600R
	Sample Roasted at 700°C	VT 0002,3C,700R
	Sample Roasted at 800°C	VT 0002,3C,800R
Lime/Sample Ratio		
1/1	Sample Roasted at 700°C	VT 0002,1C,LIME
	Phase II *Large Scale Sample Preparation*	
Cement/Thiourea/ Ratio		
1/0.01/1	Sample Unroasted	LA 0001,THIO
	Sample Unroasted	LA 0002,THIO
	Sample Unroasted	VT 0002,THIO

2.2. MATERIALS AND REAGENTS

The description of the additive mixing materials and reagents are presented in Table 2.2.

TABLE 2.2. MATERIALS AND REAGENTS

Material	Description and Source
Mixing Materials	
Clay	IMC, Blackhills, Superior Western Bentonite
Cement	Portland, Type I&II Central Pre-Mix Concrete Company
Lime	Hydrated Lime, Type S Triple S
Reagents	
Sodium Hydroxide	J.T. Baker Reagent Grade Pellets
ICP Standard	J.T. Baker Instra-Analyzed, 1000 mg Arsenic/liter
Deionized Water	Millipore De-Ion Ion Exchange

SECTION THREE
PROCEDURES

The procedures and equipment used in both the Phase I and Phase II studies are described in the following section.

3.1. *HANDLING OF AS-RECEIVED SAMPLES*

The three samples were received at the Montana Tech facilities in five gallon pails (Table 3.1).

TABLE 3.1. SAMPLE CUSTODY

Sample	Identification No.	Sample Wt., Pounds	Date of Receipt
Lagoon	WL-LA0001-1	54.2	Nov. 29, 1988
Soil	WL-LA0002-1	54.8	Nov. 29, 1988
Vault	WL-VT0002-1	51.1	Dec. 1, 1988

These samples were stored in a locked cabinet in Fume Hood #6, Room 211, Engineering Laboratory Classroom building.

Each sample container was opened as the sample was needed, spread out in pans, and air dried in fume hoods for 12-18 hours.

3.2. *PHASE I AND PHASE II TEST PROCEDURES*

3.2.1 *Sample Mixing*

The starting material chunks were weighed and individually placed in a 8-inch by 10-inch rod mill containing five 1/2-inch rods. The rod mill was rolled for ten minutes to break up the chunks. The resulting material was a fine powder. The rod mill plus sample was tared and the desired quantity of clay was added. The mixture was then rolled for 15 to 30 minutes to achieve mixing. The sample was then recovered by opening the rod mill under a hood; placed into plastic sample bags; then stored until the sintering operation was performed.

The rod mills were cleaned between each use by filling the mill approximately one-third full of clean sand and rolling for ten minutes. The weighing operation was performed on a Fairbanks scale with accuracy of plus or minus 0.1 pounds; that is for a five-pound sample the weight would be within plus or minus two percent.

The mixed material were pelletized in a laboratory-scale, 14-inch disk pelletizer to the size range -3/8-inch, +0.094-inch. Samples from the green pellet product were tested for Wet Drop Strength (1) by dropping green pellets from a 45-cm height onto a steel plate until the first sign of failure occurs. A minimum drop strength of six to eight drops was desired for handling purposes. In this study, ten pellets from each batch were tested for twenty five drops. If the pellet did not break before twenty five drops the test was stopped.

3.2.2. *Small Scale Sintering Test Work*

Each Phase I mixture that was to be sintered on the large scale was first evaluated in a small-tube furnace apparatus. The apparatus consisted of a 20-mm diameter vycor tube in a hinged tube furnace. A small sample of the mixture (from 0.5 to 3.0 grams) was placed in a fireclay boat which was then positioned in the center of the tube furnace (already at temperature; 1000°C). The entrance end of the tube was sealed by a rubber stopper containing an air-inlet tube. Air was gently blown over the surface of the sample and was flushed through tygon tubing into a fritted-glass gas dispersing tube emersed in a caustic scrub solution. The scrub solution consisted of 450 milliliters of 400 gpl sodium hydroxide.

After one-hour the sample and boat were recovered from the system, cooled in air, and weighed. The scrub solution was used to clean the vycor tubing of any deposit present. The dissolved material collected in the cleaning solution was added back to the scrub solution. The scrub solution was analyzed for arsenic (by ICP analysis at Montana Tech) and total dissolved carbon (by Institute for Process Analysis at Montana State University, Bozeman, Montana).

3.2.3. *Sintering*

Sintering was performed in 4-inch by 10-inch sillmonite roasting trays. The trays were loaded to about three-fourths capacity and placed in a muffle furnace. The muffle furnace was calibrated to determine the temperature at the sample location. The temperature profiles for most of the Phase II Screening Study samples were determined (Phase I test work did not include the determination of each temperature profile). Weight loss data for all the Phase II roast samples were collected by roasting the tray at 700°C for one-half hour; cooling and recording the tare weight; weighing the sample plus tray; roasting; cooling and recording the final weight. Weight loss data for the Phase I tests were not collected.

Observations on evidence of fuming was recorded for all the mixture roasts. The sintered or roasted material was cooled in air and placed in double plastic bags for storage until the material was shipped.

3.3. *PHASE II TEST WORK*

The results of the Phase I study suggested that further clay/sample pelletizing and sintering test work was not warranted. Therefore, the emphasis was shifted somewhat to investigate on a smaller scale, in more detail, the effect of roasting the starting materials as a function of temperature. That test work is referred to here as Phase II Screening Study test work. The results of the screening study are important to this study and to a separate study on Cement Casting Treatability Study (2).

Three roasting studies were conducted on a smaller scale than the Phase I studies in order to establish the importance of the Ca/As or Fe/As ratios and temperature in stabilizing arsenic in the waste materials. The sample sizes in the screening studies were 100 grams rather than five-ten pounds as was the case in the Phase I study.

3.3.1. *Phase II Screening Studies: Influence of Temperature on Stabilizing Waste Materials VT 0002, LA 0001, Cement/VT 0002 Ratio of 3/1, and Lime/VT 0002 Ratio of 1/1.*

The influence of temperature on potentially stabilizing the waste materials by simple roasting was studied by the following experimental procedure:

- A starting sample of 500 grams was split from the as-received air dried materials (for both VT 0002 and LA 0001). This sample was split into five 100 gram subsamples. Each sample was treated at a different temperature, i.e., no roast, 600°C, 700°C, 800°C, and 1000°C. A roast time of one hour, twenty minutes was used to ensure that the sample was at the required temperature for one hour (it took approximately twenty minutes for the mass of the sample to reach the desired temperature). Temperature profile data were collected for each roast test.

- Careful weight change data were collected for each roast test.

  The sample was weighed and placed in a previously fired and weighed 100 cc fireclay crucible. The crucible was placed in a muffle furnace that was already at the reaction temperature. The crucible had a three-eight-inch hole drilled into it one-half inch below the top. A thermocouple was placed through the furnace door peep hole into the hole in the crucible. This located the thermocouple at the sample surface. The temperature was measured and recorded as a function of time to provide a temperature profile of each sample. Each sample was held at the reaction temperature for one hour. The total elapsed time for most samples was about one hour and twenty minutes.

  After the sample was roasted for the required time at the appropriate temperature it was removed, air cooled, then weighed.

  Observations of the sample crucible surface were recorded for each sample.

- Roasts also were performed and weight change data collected for cement samples and for lime samples.

3.3.2. *Response Measurement*

The response measured to determine whether the roast procedure was successful was a modified Toxicity Characteristic Leaching Procedure (TCLP) on the roasted powders. The TCLP test was modified as follows:

- Ten grams of sample were used instead of 100 grams.
- The agitation was performed using a wrist shaker instead of an end-over-end rotator.
- Arsenic was the only element determined.

The TCLP test procedure used in this study is described below:

- Determination of the correct extraction fluid
  - Five gram sample was placed in a 500 cc beaker. Deionized water was added (96.5 cc). The mixture was stirred for five minutes. Solution pH was measured. If the pH was less than 5.0, Extraction Fluid One was used (5.7 cc glacial acetic acid added to 500 cc of deionized water and 64.3 cc of 1 N sodium hydroxide was added). This mixture was diluted to one liter. Solution pH was 4.93.

If the pH was greater than 5.0, then 3.5 cc of 1 N HCl was added, the solution heated to 50°C for 10 minutes, and pH measured. If the pH was less than 5.0, Extraction Fluid One was used. If the pH was greater than 5.0, Extraction Fluid Two was used (5.7 cc glacial acetic acid diluted to one liter; pH 2.88).

Most of the TCLP test work conducted in this study required Extraction Fluid two.

- TCLP Experimental Test
  - Ten grams of each sample was placed in a 250 cc polyethylene bottle. Two hundred cc of the correct Extraction Fluid was added.
  - Each bottle was agitated in a wrist shaker set so that all the solids were in constant movement for eighteen hours.
  - An aqueous sample was withdrawn from the bottle using a syringe filter (0.45 micrometers) for ICP analysis.

3.3.3. *Phase II Large Scale Studies*

After evaluation of the Phase I results it was decided not to pursue additional test work on clay/sample mixtures. As a replacement workplan (in addition to the Phase II Screening Study test work) it was decided in consultation with NUS project managers to prepare several pounds of mixtures of each sample with cement and thiourea (as suggested by the work of Tetsuro(3). The procedure for this test work included: mixing the samples with cement on a 1/1 ratio; dissolving the required quantity of thiourea in water and then using that water in preparing the slurry mixture for casting; casting the slurry into approximately one hundred 3/8-inch by 3/16-inch grid molds; allowing the cement slurry to set up for 4-6 hours; punching the samples out of the grid and sectioning the cubes into approximate quarter pieces; aging the cast material at 70°F in chambers at 100% relative humidity for twenty-eight days. These samples were then supplied to the REM III Team for further evaluation.

3.4. *ANALYTICAL PROCEDURES*

3.4.1. *Moisture Content*

The moisture content of individual samples was determined by weight loss measurements. Samples (60-80 grams) were weighed before and after drying at 105°C for one hour. In most cases triplicate samples were treated. Sample weights were measured on a Mettler PB 300 two decimal place electronic balance.

3.4.2. *Arsenic Determination*

The arsenic content of the starting and product materials were determined by using the EPA Digestion Procedure (Method 3050: Acid Digestion of Sediments, Sludges and Soils) and a Modified Digestion Procedure. The reason for testing a modified procedure was that the EPA Digestion Procedure (when applied to cement containing samples) produced silica gel in the digested product; the Modified Digestion Procedure did not produce silica gel.

Both of the procedures were applied to all Phase I and Phase II products. The results of the test work, i.e., comparsions of results for the starting sample materials and the product materials, are presented in Tables 4.2 and 4.10. A description of the Modified Digestion Procedure is presented below:

- Samples were ground to -60 mesh. Triplicate samples of 0.25 grams each were weighed into tared 100 milliliter polyethylene bottles. Twelve milliliters of HF, two milliliters of HNO_3, and one milliliter of $HClO_4$ were added to the solid.
- Solids were digested in the bottles for twelve hours at 65°C.
- After cooling to room temperature, each bottle solution was diluted to 100 grams.
- A blank and standard solution were included in the digestion.
- If a residue was present, it was allowed to settle and the solution was aspirated directly into the ICP. Solution concentration of the standard was used to determine the solution concentration of the individual sample and these results were used to calculate the percentage arsenic in the solids.

3.4.3. *Instrumentation*

The analytical instrumentation used in this study included: an Induction Coupled Plasma Spectrophotometer (Perkin Elmer Model 5500 ICP); a International Scientific Institute ISI-40 Scanning Electron Microscope with a Princeton Gamma Tech System 4 X-ray analyzer attachment.

3.5. *METALLOGRAPHY AND SEM-EDX*

Several of the Phase I samples were recorded for later study by standard metallography procedures, i.e., samples were mounted in room temperature setting epoxy, ground through - 600 grit papers, and polished through 5 micrometer activated alumina on a billiard cloth. These samples were photographed at several magnifications to allow for the study of structure characteristics of the product materials.

Scanning Electron Microscopy and Energy Dispersive X-ray Analyses (SEM-EDX) were performed on only one of the Phase I product materials because of the time and expense of surveying all the samples. The procedure used was as follows: one of the samples prepared for metallography was chosen to be studied for arsenic phase identification, arsenic content and arsenic distribution. The sample was carbon coated in a vacuum, placed in the SEM microscope and studied by both SEM surface scanning and EDX analysis of several of the component phases by controlled beam placement and emission spectra analysis.

SECTION FOUR
RESULTS AND DISCUSSION

4.1. *BACKGROUND*

The technology to be evaluated by this study was clay/sample pelletizing and sintering. The purpose of the pelletizing and sintering was to prepare samples for an investigation of the physical and chemical binding of arsenic. Three test samples were considered, i.e., a sample referred to as a vault material (VT 0002) that contained an appreciable amount of arsenic, primarily as calcium arsenate (and, perhaps, as calcium arsenite); and two samples that contained much less arsenic referred to as a lagoon sample (LA 0001) and a sample referred to as a lagoon-soil sample (LA 0002).

The technology to be tested was developed by Twidwell and Mehta in early 1980 (4-6). The technology they developed was for the treatment of copper smelter flue dust which, of course, was a different material than the materials considered in the present study. Their work, however, served as a guide to the selection of the conditions to be considered in the Phase I study. The investigators showed that the good stability results (to water leaching) were obtained for clay/sample mixtures that contained either calcium or iron arsenates. Their test materials were sintered at 1000°C for 15 minutes in order to develop good handling strength. Therefore, the major variable chosen for study in the Phase I test program was clay/sample ratio.

4.2. *AS-RECEIVED SAMPLE CHARACTERIZATION*

Observation comments concerning the physical appearance of the as-received materials are presented in Table 4.1. Moisture and arsenic contents are reported in Table 4.2. Spectra and semi-quantitative composition data for the air dried materials are presented in Figures 4.1 - 4.4.

TABLE 4.1. APPEARANCE OF AS-RECEIVED SAMPLES

Sample	Observation
LA 0001	Reddish brown semi-dry chunky material varying in size from 1/8-inch to several inches. Could be handling like a dry material. Chunks easily broken into smaller pieces. Odorless. Clay-like material. Some rocks present.
LA 0002	Essentially the same in appearance as sample LA 0001 except there appeared to be more rocks present (approximately 12% of sample weight). Rocks appeared to range in size up to about 3/4-inch but most less than 3/8-inch.
VT 0002	When the sample container was opened there was a layer of oil-like liquid on top of the solid material. Photographs are available in the accompanying data record books. The oil-like material readily absorbed into the solid material when the solid was stirred. The solid material appeared to consist of several solids, a dark material and a yellowish-white material. The solid material was one mass rather than distinct chunks. The material emitted a definite organic-like odor.

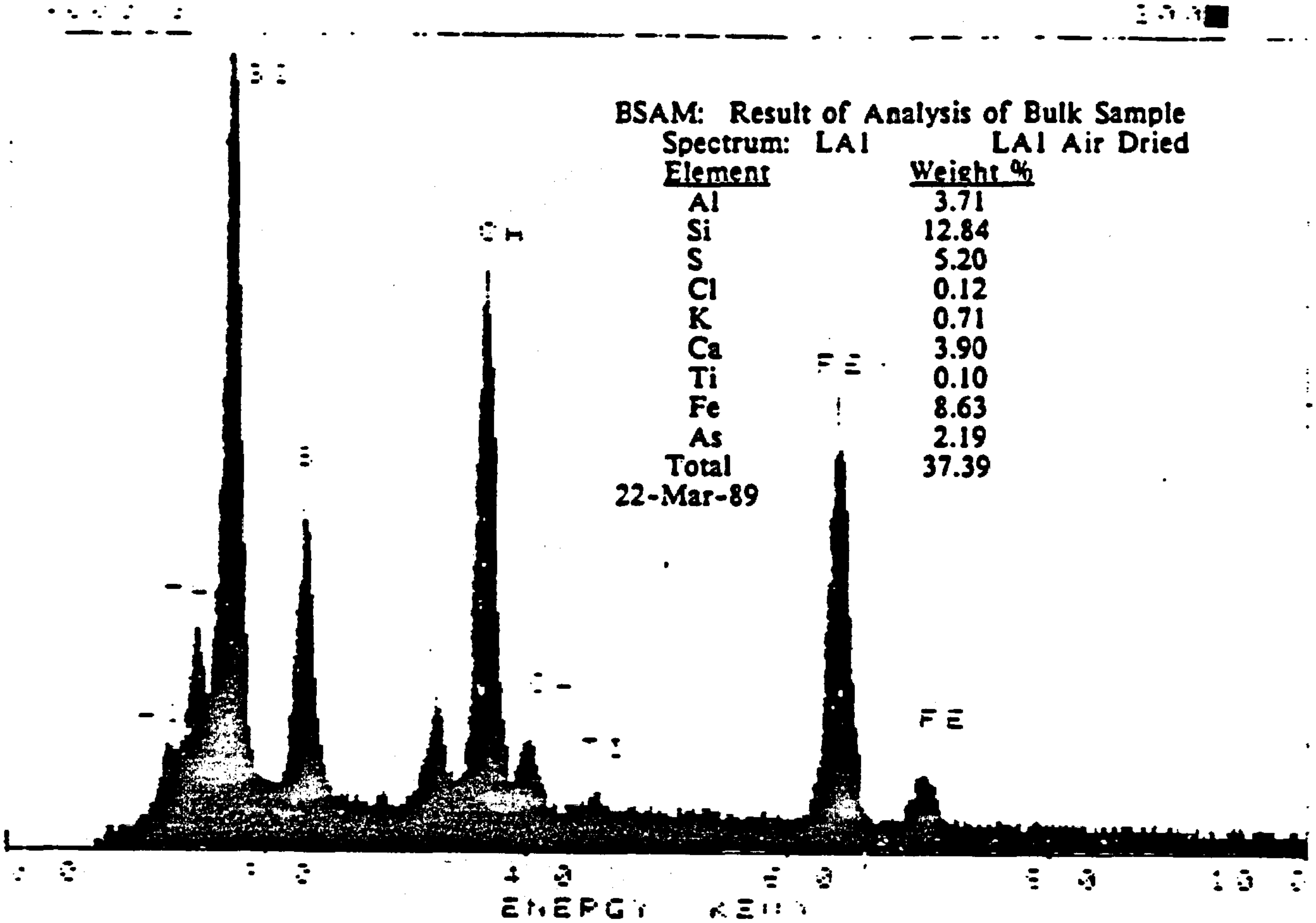
TABLE 4.2. AS-RECEIVED SAMPLE MOISTURE AND ARSENIC CONTENT

Sample	Moisture Content, %[1]		Arsenic Content, %[2]	
	As-Rec.	Air Dried	ICP	SEM-EDX
LA 0001	20.5 ± 0.5	2.7 ± 0.5	0.29, 1.14, 1.37, 1.14 1.32, 1.60[3] 1.13[3], 1.04[3]	2.19
LA 0002	33.1 ± 1.0	3.5 ± 0.2	2.88, 2.46 1.96[3], 1.84[3] 1.63[3]	2.40
VT 0002	30.7 ± 0.6	7.4 ± 0.2	15.75, 14.26 18.07, 18.72 19.14, 19.38[3] 17.81[3], 19.76[3]	19.02

[1] Moisture content of samples dried at room temperature in a hood for 18 hours.

[2] Arsenic content is for the air dried material. SEM-EDX results are semi-quantitative.

[3] Marked results were determined by modified digestion procedure.



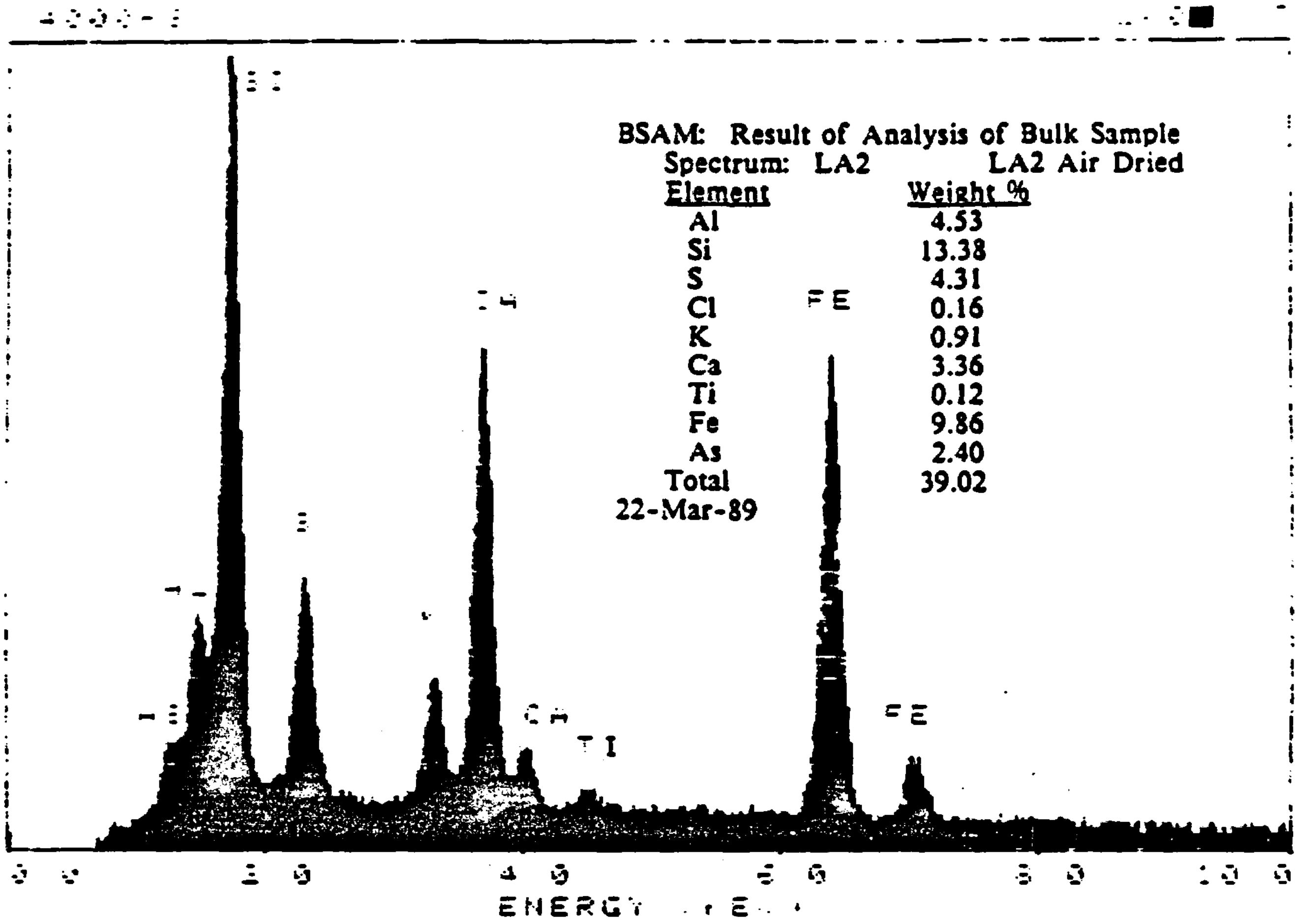
BSAM: Result of Analysis of Bulk Sample

Spectrum: LA1 LA1 Air Dried

Element	Weight %
Al	3.71
Si	12.84
S	5.20
Cl	0.12
K	0.71
Ca	3.90
Ti	0.10
Fe	8.63
As	2.19
Total	37.39

22-Mar-89

FIGURE 4.1. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED LA 0001 SAMPLE

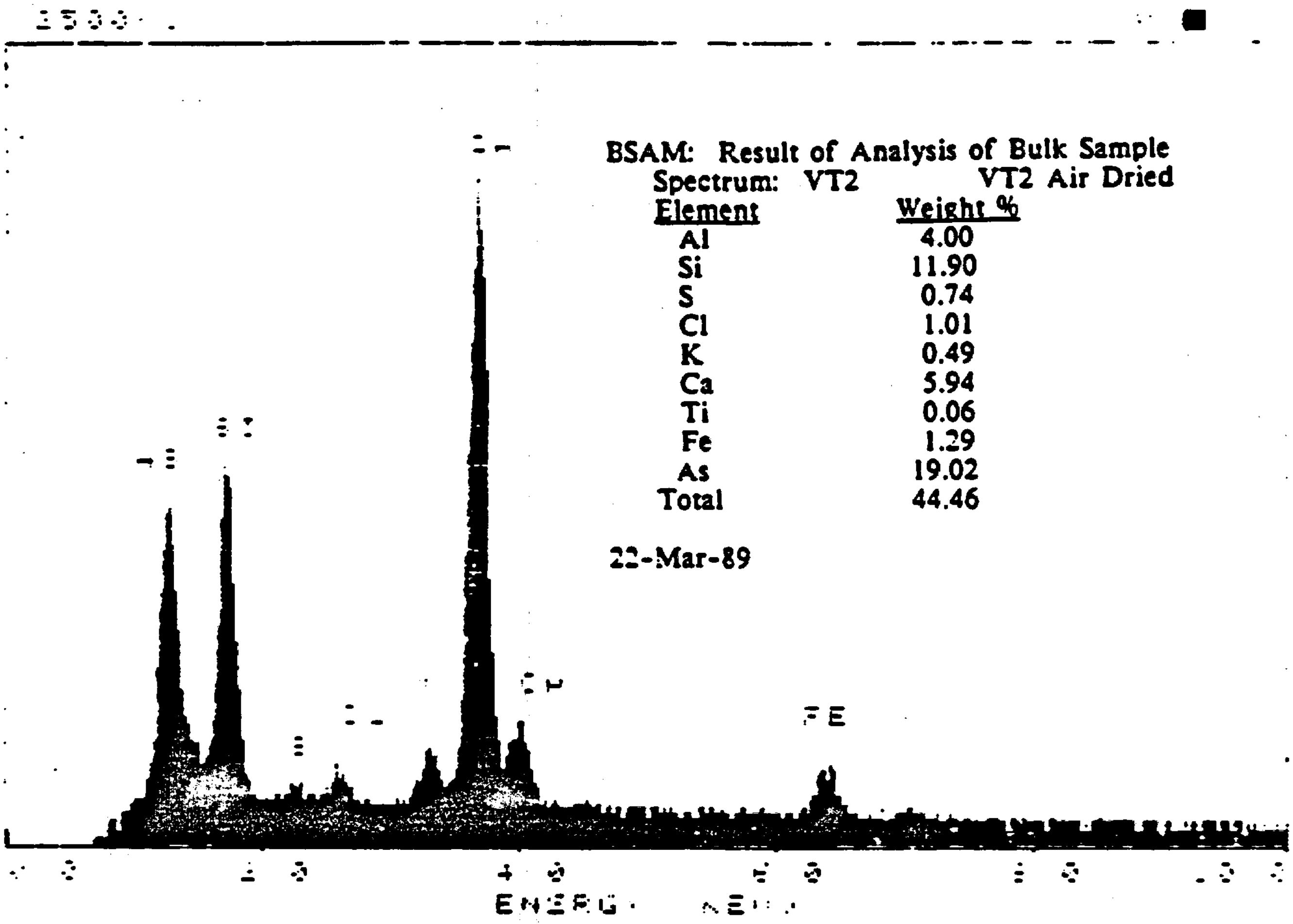


BSAM: Result of Analysis of Bulk Sample
Spectrum: LA2 LA2 Air Dried

Element	Weight %
Al	4.53
Si	13.38
S	4.31
Cl	0.16
K	0.91
Ca	3.36
Ti	0.12
Fe	9.86
As	2.40
Total	39.02

22-Mar-89

FIGURE 4.2. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED LA 0002 SAMPLE



BSAM: Result of Analysis of Bulk Sample

Spectrum: VT2 VT2 Air Dried

Element	Weight %
Al	4.00
Si	11.90
S	0.74
Cl	1.01
K	0.49
Ca	5.94
Ti	0.06
Fe	1.29
As	19.02
Total	44.46

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FIGURE 4.3. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED VT 0002 SAMPLE

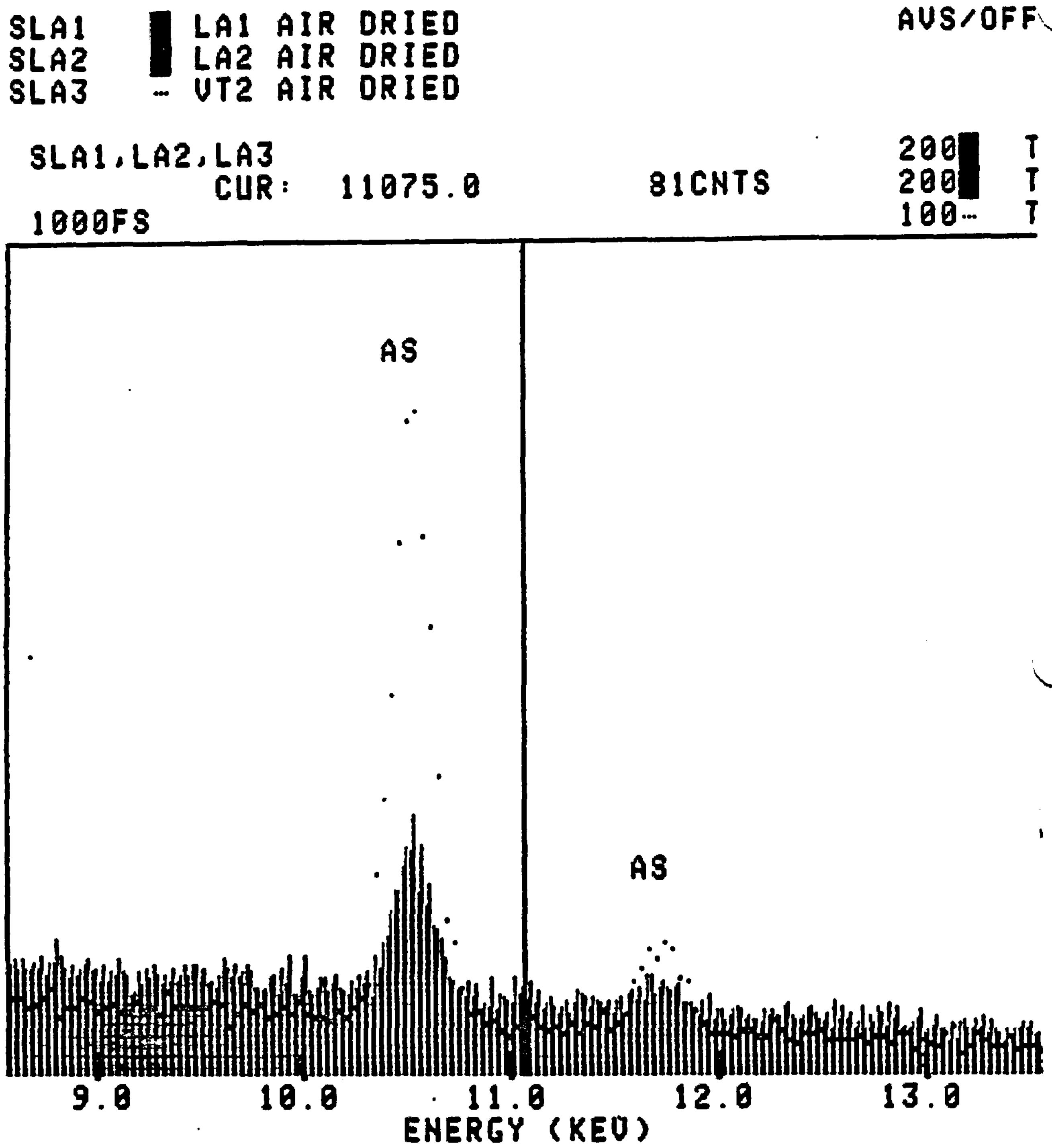

FIGURE 4.4. SEM-EDX COMPARATIVE SPECTRA OVERLAY OF ARSENIC PEAK FOR THE THREE STARTING MATERIALS.

4.3. *PHASE I STUDIES*

Samples were received at Montana Tech in early December, 1988. See Section 2.1, Sample Identification System for the numbering system used in the following discussion; Section 3.1, Handling of As-Received Sample Materials for a description of the as-received sample materials; and Section 4.2, As-Received Sample Characterization. Test work was initiated (See Section 3.2, Phase I and II Test Procedures) on the Phase I test objectives (See Section 1, Objectives) in December

The treatment sequence followed in the Phase I studies consisted of: mixing the sample and clay; pelletizing in a 14-inch laboratory-scale disk pelletizer; and sintering the green pellets in air at 1000°C for fifteen minutes.

4.3.1. *Health and Safety Considerations*

A health and safety report is attached as Appendix Section 8.1. All personnel involved in handling the materials wore protective clothing and gloves at all times and wore respirators when handling the samples and sample mixes. Two of the laboratory assistants wore personal air monitors during the first day of each phase study. The results from the air monitoring studies showed that the working conditions were safe from a health point of view and the airborne arsenic in the working atmosphere was negligible.

4.3.2. *Sample Mixing*

Two sample mix ratios for each of the supplied three samples were prepared. The amounts of each mix material initially prepared are summarized in Table 4.3.

TABLE 4.3. PHASE I SAMPLE MIXTURES

Sample	Mix Ratio, Clay/Spl.	Quantity Prepared, Grams
LA 0001A,1C	1/3	2,725
LA 0001A,3C	3/1	2,725
LA 0002A,1C	1/3	2,725
LA 0002A,3C	3/1	2,725
VT 0002A,1C	1/3	2,725
VT 0002A,3C	3/1	2,725

4.3.3. *Sample Pelletizing*

Samples were pelletized in the 14-inch laboratory pelletizer as described previously. Drop tests were performed. All the samples passed twenty five drops, therefore, the handling characteristics of the green pellets exceeded normal industrial requirements. The moisture content of the green pellet products are presented in Table 4.4. Observations concerning the green pellet product are presented in Table 4.5.

TABLE 4.4. GREEN PELLET MOISTURE CONTENT

Sample	Mix Ratio, Clay/Spl.	Moisture Content, %
LA 0001A,1C	1/3	29.5 ± 5.3
LA 0001A,3C	3/1	31.0 ± 9.0
LA 0002A,1C	1/3	-
LA 0002A,3C	3/1	-
VT 0002A,1C	1/3	24.8
VT 0002A,3C	3/1	29.6

TABLE 4.5. PELLETIZING OPERATION AND PRODUCT OBSERVATIONS

Sample	Observations
LA 0001A,1C	Pelletizing slow, difficult to form spherical product. Pellets shaped like raisins. Good green strength. Easily screened without breaking.
LA 0001A,3C	Same as above.
LA 0002A,1C	Green pellets had less green strength than LA 0001 samples but still passed drop test. Greater rock content evident.
LA 0002A,3C	Same as above.
VT 0002A,1C	Pelletizing process smoother than LA samples. Good green strength.
VT 0002A,3C	Same as above.

4.3.4. *Sample Sintering*

Small scale (0.5-3.5 grams) tube furnace sintering tests were performed on samples cut from each mixture prior to the large scale sample preparation test work. The procedure was described previously. The results are summarized in Table 4.6.

Large scale sample sintering was conducted as described previously. Quantities of materials sintered and observations concerning the sintering are presented in Table 4.7. All samples in the Phase I test work were sintered at 1000°C for fifteen minutes. The sintering operation was conducted in a batch mode of operation, i.e., about 200-400 grams were sintered per batch. The total amount of individual samples treated varied from about 1000-1600 grams.

TABLE 4.6. ARSENIC AND ORGANIC CARBON RELEASE FROM SMALL SCALE SINTERING TESTS

Sample	Arsenic Release		Total Organic Carbon Release	
	gm As/gm sample	%As[1]	gm C/gm sample	%C[2]
LA 0001A,1C	0.0024	28.3	0.0025	-
LA 0001A,3C	0.0025	88.6	0.0044	-
LA 0002A,1C	-		0.0033	-
LA 0002A,3C	0.0015	27.8	0.0037	-
VT 0002A,1C	0.0158	11.8	0.0232	-
VT 0002A,3C	0.0030	6.7	0.0240	-

[1] The arsenic content of the starting sample mixtures was not experimentally determined. Therefore, the starting arsenic concentration was estimated by using the average arsenic content (calculated from Table 4.2) corrected for the amount of clay added. For example, Sample LA 0002B,3C: the average arsenic content for unmixed LA 0002 (Table 4.2) was 2.15%. The arsenic content corrected for the presence of clay is 0.54%. The arsenic released = 0.0015/0.0054, i.e., 27.8%.

[2] The total organic carbon content of the starting sample mixtures was not experimentally determined. The starting organic carbon concentration could not be estimated as stated above for the arsenic content because the data for the unmixed samples were suspect and unvalidated. Therefore, the percent organic carbon volatilized is not reported.

The values presented above in Table 4.6 represent the organic carbon dissolved in the scrub solution. The values presented in the table may not represent all the organic carbon released because it was noted during the roasting process (for the VT samples) that a smokey portion of the gas phase passed through the scrub solution.

TABLE 4.7. PHASE I SINTERING OBSERVATIONS

Sample	Observations
LA 0001A,1C	No fuming observed from the furnace door. Surface colors were brown to orange, light brown. Product not as fragile as LA 0001A,3C.
LA 0001A,3C	No fuming observed. Pellets fragile. Each agglomerate made up of smaller spheres. Surface colors grey, brown, orange.
LA 0002A,1C	Fuming observed. Light white fume. Pellets easily broken by hand pressing. See table on strengths. Low strength reflected in presence of much more undersize material.
LA 0002A,3C	Same as LA 0002A,1C.
VT 0002A,1C	Dense white fumes emitted almost immediately. Pellets stable and strong. Surface colors were white to grey.
VT 0002A,3C	Same as VT 0002A,1C

Sintered products were screened and the size distribution was determined. The size distribution results are presented in Table 4.8. The sintered products were subjected to a semiquantitative test to compare relative strengths. A soil penetrometer was positioned on the product surface and pressed until the product broke under the applied pressure. The force required to break the product was recorded. The results are reported in Table 4.9. It is apparent that the pellet strengths are not very good, at least, when compared to cement/sample cast products produced in Phase II (See Table 4.19).

TABLE 4.8. PHASE I PRODUCT SIZE DISTRIBUTION

Sample	Size Distribution, % Retained				
	-0.375 +0.263	-0.263 +0.185	-0.185 +0.131	-0.131 +0.094	-0.094
LA 0001A,1C	76.8	21.1	1.1	0.4	0.6
LA 0001A,3C	57.3	37.5	2.8	0.9	1.5
LA 0002A,1C	40.0		38.0	22.3	
LA 0002A,3C		52.3	33.8	13.5	
VT 0002A,1C		86.3	11.1	2.7	
VT 0002A,3C		73.3	24.3	2.4	

TABLE 4.9. UNCONFINED SEMI-QUANTITATIVE STRENGTH TEST ON PHASE I PRODUCTS

Sample	Strength, kg/cm^2 ($Tons/ft^2$)		
	Sample 1	Sample 2	Sample 3
LA 0001A,1C	2.2	2.5	3.2
LA 0001A,3C	4.5	2.2	3.0
LA 0002A,1C	1.0	0.8	0.5
LA 0002A,3C	0.8	1.0	0.8
VT 0002A,1C	0.5	0.8	0.8
VT 0002A,3C	1.0	1.5	1.0

Note: The recorded values are the force required to break the product.

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4.3.5. *Final Phase I Products Arsenic Content*

The final products were analyzed by sample digestion and ICP analysis of the resultant solutions. The procedure was presented previously in Section 3.3. A summary of the arsenic content in the Phase I products is presented Table 4.10.

TABLE 4.10. ARSENIC CONTENT IN PHASE I PRODUCTS

Sample	Arsenic Content, %	
	EPA 3050	Modified Procedure
	LA 0001	
LA 0001, Air Dried	0.29, 1.14 1.37, 1.14 1.32	1.60, 1.13, 1.04
LA 0001A,1C	0.85, 0.87	1.47, 1.08, 1.34
LA 0001A,3C	0.23, 0.21	0.72, 0.63, 0.62
	LA 0002	
LA 0002, Air Dried	2.88, 2.46	1.96, 1.84, 1.63
LA 0002A,1C	0.97	1.67, 1.24, 1.06
LA 0002A,3C	0.32	0.27, 0.34, 0.28
	VT 0002	
VT 0002, Air Dried	15.75, 14.26 18.07, 18.72 19.14	19.38, 17.81, 19.76
VT 0002A,1C	4.72, 3.85	1.68, 2.26, 1.44
VT 0002A,3C	0.99	1.46, 1.68, 1.61

4.3.6. *Product Leach Studies*

Phase I products were characterized by three leach tests (data supplied by REM III Team: data preliminary and unvalidated): TCLP, Modified-ASTM (A) Deionized Water Leach, and Modified-ASTM (B) Carbonate Leach. The leach response measurements followed in this study were arsenic and total organic carbon. The arsenic and total organic carbon (TOC) concentrations in the samples subjected to the leach tests are presented in Table 4.11. Total volatile solids (TVS), starting sample pH, and specific gravity for the solids subjected to the leach tests are presented in Table 4.12. The leach test results are presented in Tables 4.13 (TCLP,As), 4.14 (ASTM-A,As), 4.15 (ASTM-B,As), 4.16 (ASTM-A,TOC), and 4.17 (ASTM-B,TOC).

TABLE 4.11. ARSENIC AND TOC CONTENT IN PHASE I PRODUCTS SUBJECTED TO LEACH TEST WORK

Sample	Concentration, mg/kg[1]	
	Arsenic	TOC
LA 0001[2]	11300	
LA 0001A,1C	6870	2770
LA 0001A,3C	1020	1150
LA 0002[2]	21500	
LA 0002A,1C	9090	2290
LA 0002A,3C	2220	1720
VT 0002[2]	178600	
VT 0002A,1C	16800	2410
VT 0002A,3C	8010	776

1 REM III Team Data: Analyses performed by Versar, Inc.
3 EI data: Average of all analyses presented in Table 4.2.

TABLE 4.12. CHARACTERISTICS OF PHASE I SOLID PRODUCTS SUBJECTED TO LEACH TESTS

Sample	Characteristic[1]		
	TVS,%[2]	Sample pH	Specific Gravity
LA 0001	27.6	5.6	-
LA 0001A,1C	<0.1	11.81	2.64
LA 0001A,3C	<0.1	11.71	2.04
LA 0002	34.7	3.9	-
LA 0002A,1C	0.3	11.57	3.32
LA 0002A,3C	0.1	11.88	2.20
VT 0002	43.6	8.6	-
VT 0002A,1C	0.2	9.75	2.60
VT 0002A,3C	<0.1	9.55	1.86

1 REM III Team Data: Test performed by Versar, Inc.
2 Total Volatile Solids

TABLE 4.13. TCLP RESULTS FOR PHASE I MATERIALS

Sample	Wt, gms[1]	Extractant As Conc., mg/liter	
		EI Results[2,3]	REM III Team[4]
LA 0001	9.965	1.8(4.7)[5]	4.6(6.1)
LA 0001A,1C	10.041	92.6(4.8)	74.1(5.3)
LA 0001A,3C	10.097	50.0(4.3)	22.9(4.6)
LA 0002			2.4(6.2)
LA 0002A,1C	10.026	87.4(9.6)	11.4(9.6)
LA 0002A,3C	10.029	55.9(5.1)	37.2(5.6)

TABLE 4.13. TCLP RESULTS FOR PHASE I MATERIALS (Continued)

Sample	Wt, gms[1]	Extractant As Conc., mg/liter	
		EI Results[2,3]	REM III Team[4]
VT 0002		7056. (6.5)	2260.(7.0)
VT 0002A,1C	10.070	118.4(4.8)	78.2(4.4)
VT 0002A,3C	9.990	229.8(4.0)	21.6(4.4)

1 Weight of EI test samples.
2 Extraction Fluid 2 was used in all EI tests. Arsenic hazardous characteristic level: 5 mg/liter.
3 Starting material as a powder; not as pellets.
4 REM III Team data: Test work performed by Versar, Inc.
5 The number in parentheses is final pH.

Note in the above table:

- That the TCLP leach conditions extracted arsenic to concentrations greater than the hazardous characteristic concentration (5 mg/liter) for all of the clay/sample sintered samples.

- The sintered LA sample mixtures leached more arsenic than the untreated unmixed starting samples. The reason for this is presently not clear.

- The sintered VT sample mixtures leached much less arsenic than the untreated starting sample. However, the effect is likely because a portion of the original arsenic volatilized.

TABLE 4.14. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]			
	Deionized Water Leach[2]			
	Extract 1	2	3	Cumulative
LA 0001	5.03 (7.6)	4.88 (7.6)	1.86 (8.4)	11.77
LA 0001A,1C	0.539(11.9)[3]	0.724(11.8)	1.14 (11.4)	2.40
LA 0001A,3C	0.529(11.6)	0.729(11.6)	1.44 (11.1)	2.70
LA 0002	5.18 (7.9)	4.47 (7.7)	6.22 (8.6)	15.87
LA 0002A,1C	0.158(12.2)	0.136(12.1)	0.256(11.7)	0.55
LA 0002B,3C	0.070(11.9)	0.312(11.9)	0.529(11.3)	0.91
VT 0002	1650. (8.6)	851. (8.5)	512. (9.0)	3013.
VT 0002A,1C	15.9 (11.1)	12.0 (11.5)	9.92 (11.2)	37.82
VT 0002A,3C	17.4 (9.8)	7.82 (10.6)	7.36 (9.7)	32.58

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 Final pH values in parentheses.

Note that:

- The deionized water leach extracted much less arsenic from the sintered clay/sample mixtures than from the untreated starting samples. However, this effect may be because the arsenic content in the sintered materials was much less than in the untreated materials.

TABLE 4.15. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE I PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]			
	Carbonate Leach[2]			
	Extract 1	2	3	Cumulative
LA 0001	10.5 (8.9)	126.0 (10.6)	101.0(10.8)	237.5
LA 0001A,1C	2.14 (10.7)	11.2 (9.8)	14.9 (9.6)	28.2
LA 0001A,3C	8.26 (9.9)	6.9 (9.3)	5.68(9.1)	20.8
LA 0002	26.7 (8.4)	105.0 (10.7)	115.0(10.9)	246.7
LA 0002A,1C	0.670(12.5)	3.02(10.4)	5.82 (9.9)	9.5
LA 0002A,3C	9.95 (10.6)	14.0 (9.6)	9.75 (9.2)	33.7
VT 0002	2060. (8.7)	2710. (10.4)	1900. (10.7)	6670.
VT 0002A,1C	36.4 (9.6)	18.4 (9.2)	14.4 (9.1)	69.2
VT 0002A,3C	18.2 (8.8)	5.48 (8.7)	6.25 (8.7)	29.9

1 REM III Team data: Test work performed by Versar, Inc.
2 ASTM D3987-85 Leachant B, 3 gm/liter sodium bicarbonate.
4 Final pH values in parentheses.

Note that:

- The cumulative arsenic extracted for all the carbonate leaches was considerably greater than the arsenic extracted by the deionized water leaches (except VT 0002A,3C was about the same for both test sequences) for all mixtures.

- Also, the cumulative arsenic extracted from all the LA sintered products by deionized water was less than 2.7 mg/liter, but the arsenic extracted from all the LA sintered products by carbonate ranged from approximately 10 to 34 mg/liter.

The conclusion from the above observations is that all the Phase I LA sintered products are stable (assuming that <five mg/liter is considered stable) in deionized water (for at least three extraction exposures) but none of sintered products are stable in a carbonate environment. The VT products (using the above criteria for stability) are not stable in either deionized water or a carbonate environment.

TABLE 4.16. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS: TOTAL ORGANIC CARBON

Sample	Concentration, mg/liter[1]						
Extract[2]	1		2		3		Cumulative
	TOC[3]	TDS[4]	TOC	TDS	TOC	TDS	TOC
LA 0001	10.0		8.5		6.6		25.1
LA 0001A,1C	7.43	1720	2.64	808	4.95	365	38.6
LA 0001A,3C	2.45	857	1.82	354	12.4	144	16.7
LA 0002	11.0		12.8		9.8		33.6
LA 0002A,1C	1.01	3100	1.86	880	13.8	410	16.7
LA 0002A,3C	1.49	1180	1.48	413	11.5	200	14.5
VT 0002	1019.		414.		166.		1599.
VT 0002A,1C	1.17	497	1.27	205	11.7	112	14.2
VT 0002A,3C	1.19	136	1.28	25	14.3	39	16.8

[1] REM III Team Data: Test performed by Versar, Inc.
[2] ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
[3] Total Organic Carbon
[4] Total Dissolved Solids

TABLE 4.17. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE I PRODUCTS: TOTAL ORGANIC CARBON

Sample	TOC Concentration, mg/liter[1]			
	Extract[2] 1	2	3	Cumulative
LA 0001	17.5	232.0	208.0	457.5
LA 0001A,1C	13.4	8.56	6.30	28.3
LA 0001A,3C	6.80	7.27	5.39	19.5
LA 0002	36.6	119.0	182.0	337.6
LA 0002A,1C	7.38	5.17	5.23	17.8
LA 0002A,3C	14.8	4.37	6.17	25.4
VT 0002	1008.	659.	226.	1893.
VT 0002A,1C	14.7	6.00	7.96	28.7
VT 0002A,3C	14.0	9.75	5.59	29.4

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant B, 48 hour exposure in each extraction.
3 Total Organic Carbon

Note that:

- The cumulative organic carbon extracted for all the carbonate leaches on the untreated samples was considerably greater than the organic carbon extracted by the deionized water leaches on the same materials.

- The organic carbon extracted from all the sintered products was much less than that extracted from the untreated samples.

4.3.7. *Metallography and SEM-EDX*

The as-received air dried material and each of the clay/sample sintered products were chosen for metallographic display. The results are presented in Figures 4.6-4.12. The photomicrographs show the character of the pelletized and sintered products, e.g., the starting material LA 0001 formed the best pellets of all the materials studied. Some porosity is evident but certainly not to the extent present in the other pellets. Also note the rather large amount of rocks presence in the unmixed pellet.

The other pellets show a large degree of porosity. The photomicrographs also show that the pelletizing operation was much less effective in forming coherent dense pellets than was desirable.

Sample LA 0001 was chosen to study in a more detailed way on the SEM-EDX system. Various phases in the structure were subjected to spot analysis. The spots chosen to be studied are marked on the photomicrograph presented in Figure 4.12 and their spectra are presented in Appendix Section 8.3. (Figures 8.1-8.11). Identification of the elemental content and the semi-quantitative concentration are presented in Table 4.18.

FIGURE 4.5. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0001 (10X)

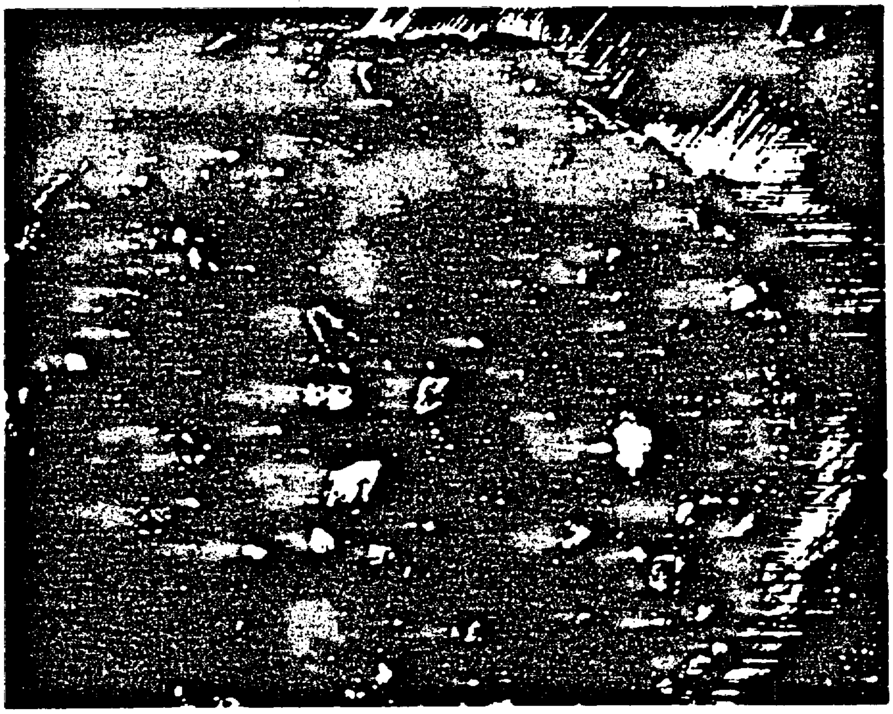

FIGURE 4.6. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0001A,1C (10X)

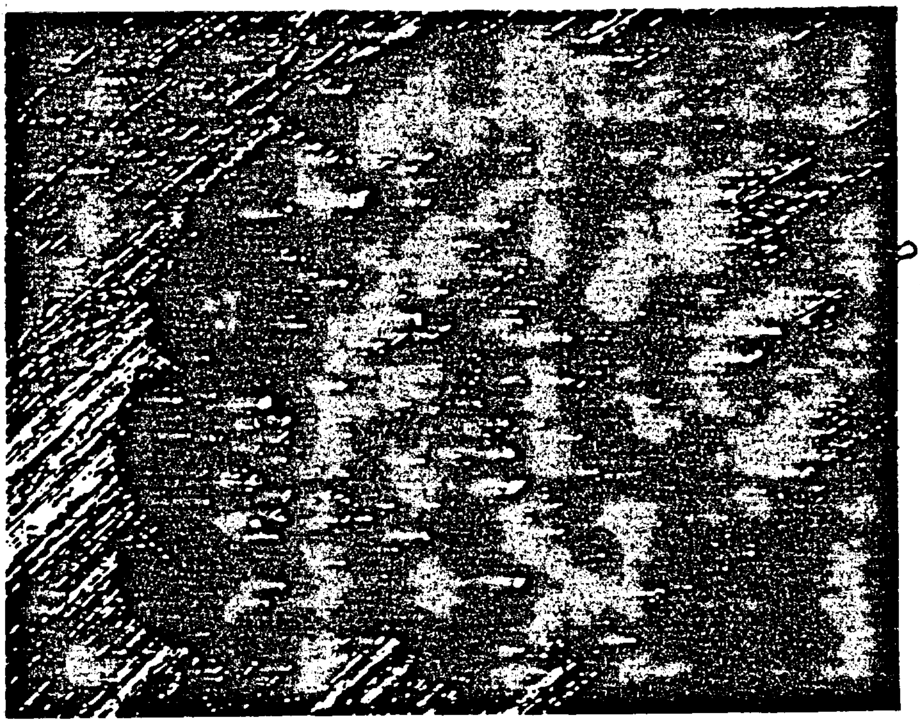

FIGURE 4.7. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0001A.3C (12.5X)

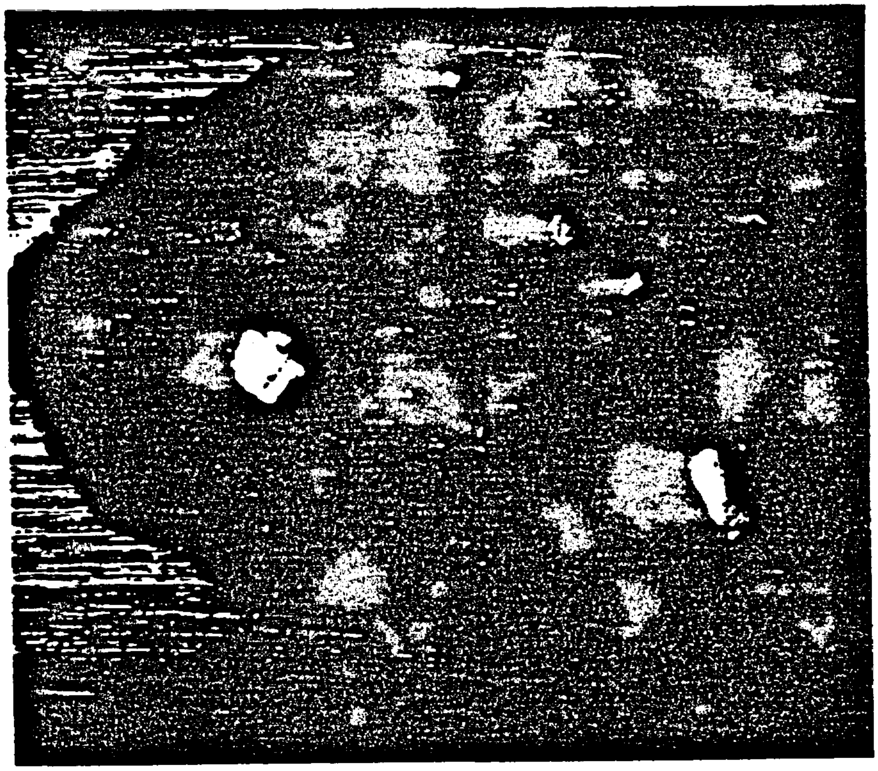

FIGURE 4.8. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0002A.1C (20X)

FIGURE 4.9. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0002A.3C (16X)

FIGURE 4.10. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0002A.1C (12.5X)

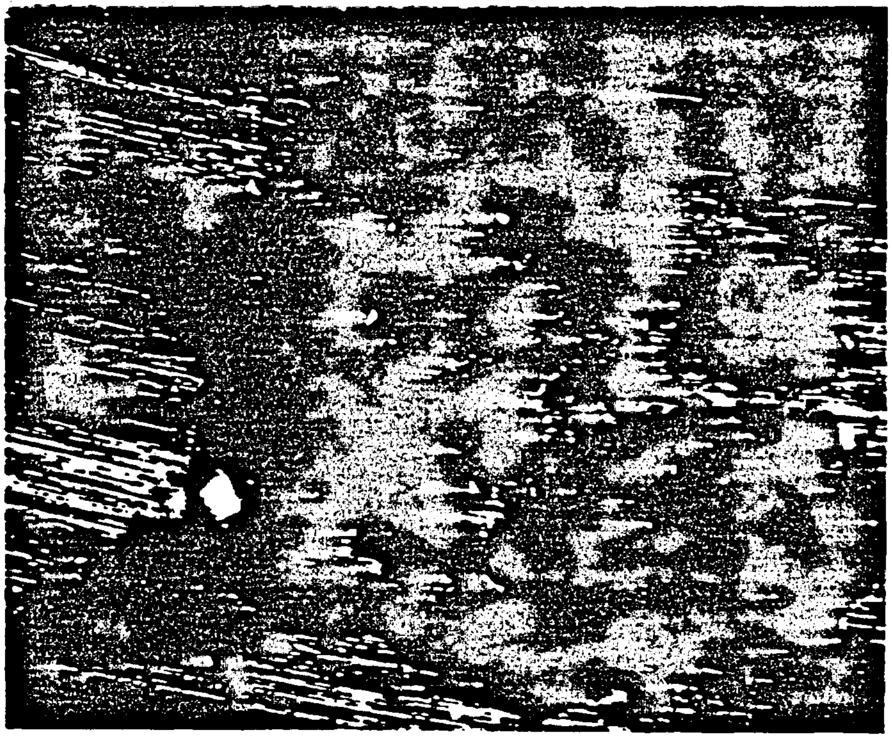

FIGURE 4.11. PHOTOMICROGRAPH FOR SINTERED SAMPLE VT 0002A.3C (12.5X)

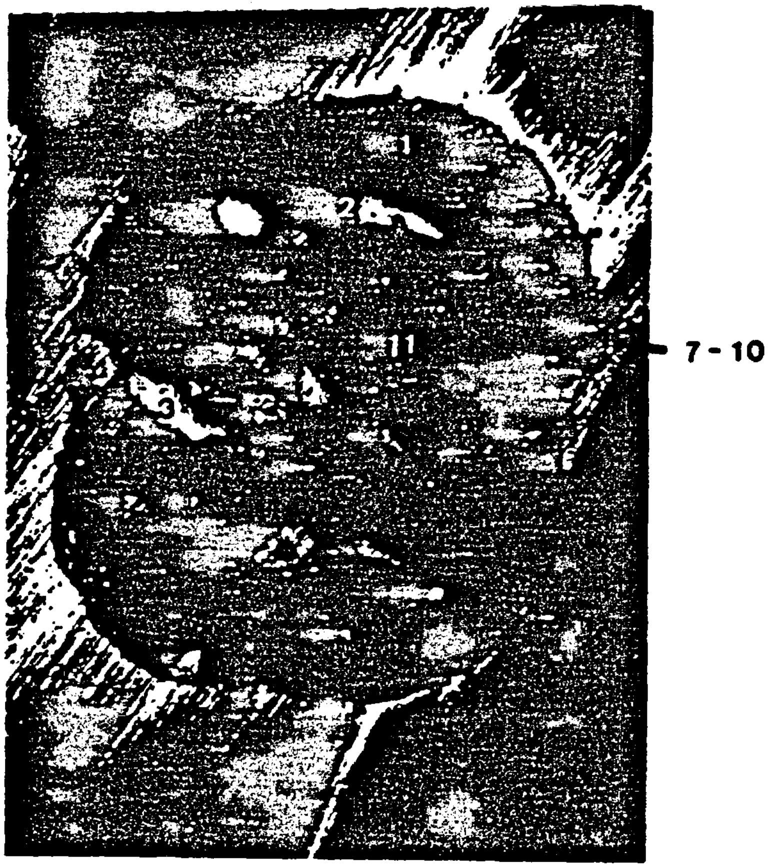

FIGURE 4.12. PHOTOMICROGRAPH OF SAMPLE LA 0001 WITH MARKED SPOTS SUBJECTED TO SEM-EDX BEAM ANALYSES

FIGURE 4.13. PHOTOMICROGRAPH FOR SINTERED SAMPLE LA 0001A (700 X)

AR103890

TABLE 4.18. PHASE COMPOSITION IN SINTERED SAMPLE LA 0001

Spot Number	Semi-Quantitative Concentration, %							
	As	Ca	Fe	Al	Si	S	K	Mg
1 (Bulk)	1.6	15.6	13.5	10.3	46.8	7.2	2.4	2.5
2 (Rock)	0.0	33.6	0.6	6.4	12.3	4.8	0.6	41.7
3 (Rock)	0.0	0.3	0.8	0.2	96.5	1.8	0.3	0.0
4 (Matrix below Si Rock)	0.0	8.4	9.6	13.6	57.6	5.3	2.7	2.7
5 (Bright)	0.0	4.9	28.0	14.9	45.5	4.7	1.3	0.6
6 (Bright near Top)	1.7	9.0	38.3	12.1	31.8	3.3	1.1	2.8
7 (Grey)	2.2	36.3	1.9	3.1	7.6	26.0	0.4	22.6
8 (Grey)	1.2	33.5	1.7	3.2	13.0	23.6	0.5	23.2
9 (Grey)	0.6	33.4	2.6	5.2	15.1	15.7	1.2	26.2
10 (Grey, See Figure 4.13)	2.2	30.8	3.1	4.5	14.7	21.1	1.1	22.6
11 (Rock)	0.6	43.0	0.7	5.1	11.6	3.6	1.4	33.9

4.4. *PHASE II STUDIES*

The Phase I studies, under very high clay ratios, showed that there was appreciable arsenic volatilization during the sintering operation (Table 4.6); that the sintered pellets had high porosity (Figures 4.6-4.12); and that the arsenic released in the TCLP tests was significantly above the characteristic level (Table 4.13). Therefore, further test work on the clay/sample mixtures was not warranted.

4.4.1. *Phase II Small Scale Screening Studies*

It was felt that a series of smaller scale tests were necessary to delineate the direction to be taken in the Phase II large scale sample generation. A separate study (2) was being conducted concurrent with the presently discussed project, i.e., a study on stabilization by cement casting. The results of the cement casting study were encouraging, i.e., the TCLP preliminary results indicated that stabilization was possible by controlling the cement/sample ratio and the roasting temperature. However, a number of questions exiting that needed to be addressed prior to initiating the second phase of that work. Therefore, a series of small scale tests that were applicable to both studies were designed to address the following questions:

- Could stabilization be produced by roasting the starting materials without the presence of any other additive? What would be the effect of roasting temperature on the stabilization?
- Could stabilization of the vault material be produced in a 3/1 (cement/sample ratio) mixture by roasting at a higher temperature than 600°C? Is the CaO content an important variable in the stabilization process?
- Could stabilization of the vault material be produced in a non-roasted cement material if thiourea were an additive?

Earlier research showed that stabilization of calcium arsenate and/or calcium arsenite precipitated products could be stabilized by roasting in air if the Ca/As mole ratio was properly controlled. Nishimura and Tozawa (7) showed if the Ca/As mole ratio was greater than 2.1 that roasting in air at 700°C decreased the arsenic solubility to less than 20 micrograms/liter (when exposed to a pH 12 leach solution); a ratio of greater than 4.1 was required to stabilize calcium arsenite. Other researchers showed similar results (8,9). The vault material appeared to contain insufficient calcium to have the necessary Ca/As ratio but it was desired, however, to test potential stabilization by roasting without additional calcium and by roasting with additional calcium supplied by cement.

A similar argument can be presented for the LA materials. Tozawa (10) showed that ferric arsenate could be stabilized by roasting Fe/As mole ratios greater than 1.3 at 600°C. In the case of the LA materials the bulk Fe/As ratios were greater than the Tozawa required values.

4.4.1.1. *Screening Experiments*

A series of roast experiments were planned and conducted. The procedure has been presented previously in Section 3.3. Briefly, the procedure was to: roast 100 gram samples for one hour at 600°C, 700°C, 800°C, and 1000°C. It was also desirable to collect good weight loss data during the study. This was done by heating 100 gram fireclay crucibles at the temperature of interest, weighing the crucible, adding the sample, weighing the sample plus crucible, heating the sample plus crucible at 200°C for one hour to remove adsorbed moisture, weighing the crucible plus sample, placing the crucible plus sample in a furnace at the temperature of interest for one hour and twenty minutes, weighing the roasted crucible plus product.

The weight loss data collected for the screening study are summarized in Table 4.19. Observation comments are presented in Table 4.20. Temperature profile data are presented in Appendix Section 8.2.

TABLE 4.19. PHASE II SCREENING TEST WEIGHT LOSS SUMMARY

Sample	Temp., °C[1]	Weight Loss, %	
		At 200°C	At T°C
LA 0001	600	3.7	11.0
	700	3.1	13.5
	800	2.6	17.6
	1000	3.0	24.4
VT 0002	600	5.5	26.2
	700	6.0	30.1
	800	9.5	29.7
	1000	5.5	38.4

TABLE 4.19. PHASE II SCREENING TEST WEIGHT LOSS SUMMARY (Continued)

Sample	Temp., °C[1]	Weight Loss, %	
		At 200°C	At T°C
Cement[2]	700	-	0.2
VT 0002B,3C	600	4.9	11.6
	700	5.4	12.6
	800	6.0	13.0
VT 0002,1LIME	700	2.2	11.7

1. The actual sample temperature profiles are presented in Appendix Section 8.2.1.
2. From Phase II study.

TABLE 4.20. OBSERVATIONS ON SCREENING ROAST EXPERIMENTS

Sample	Temp., °C	Observations
LA 0001	600,700,800 1000	Fuming not observed for any of the LA samples. Samples red on surface but lighter red in bulk of sample. Samples became progressively redder at the higher temperatures.
VT 0002	600,700,800 1000	Note temp. profiles in Section 8.2. The temp. profiles for the VT series indicated combustion at the sample surface. No obvious change in sample color. Fuming evident from furnace. See Section 8.2 for temp. start and end of fuming from furnace.

TABLE 4.20. OBSERVATIONS ON SCREENING ROAST EXPERIMENTS (Continued)

Sample	Temp., °C	Observations
VT 0002,3C	600,700,800	Fuming not observed from furnace door but temp. profiles at 700°C and 800°C indicate combustion at surface was occurring. Fuming was observed at the crucible surface for the 800°C sample but no fuming visible from the door.
VT 0002,1LIME	700	Ca/As mole ratio greater than 5. Fuming not observed on surface or from furnace. Temp. profile suggests no combustion.

It is, of course, desirable to have a way of disposing of the waste by a non-roasting technique. Tetsuro (3) conducted a study using thiourea as an additive to cement to stabilize an industrial arsenic bearing waste material. Therefore, a test of the possible use of this technology was conducted. One hundred grams of VT 0002B,1C was mixed and water containing five grams of thiourea. The mixture was cast and aged for three days, then subjected to the modified TCLP test.

4.4.1.2. *TCLP Response*

Ten grams of each of the materials generated in the screening study were subjected to the modified TCLP test. The results are presented in Table 4.21. The arsenic concentration in the products tested in the TCLP study are presented in Table 4.22.

TABLE 4.21. SCREENING TEST MODIFIED-TCLP[1] RESULTS

Sample	Temp., °C	As in Extractant Fluid[2], mg/liter
LA 0001	Unroasted	11.1(5.3)[3]
	600	344 (5.6)
	700	106 (6.7)
	800	22.7(9.7)
	1000	10.8(11.7)
VT 0002	Unroasted	7056 (6.5)
	600	2291 (5.8)
	700	1546 (5.6)
	800	1213 (5.6)
	1000	347 (6.0)
VT 0002B,3C	Unroasted	333 (12.4)
	600	2.2(11.8)
	700	1.2(11.8)
	800	1.8(12.4)
VT 0002, 1LIME	Unroasted	578 (12.9)
	700	1.6(12.9)
VT 0002B,1C, THIO[4]	Unroasted	20.0(8.0)

1 EI data: Test procedure described in Section 3.3. All starting materials were powders, not pellets.
2 Extraction Fluid 2 used for all samples.
3 Final pH of TCLP solutions shown in parenthesis.
4 The thiourea was added by dissolving it in the water used to cast the cement/sample mixture.

TABLE 4.22. ARSENIC CONTENT IN PHASE II SCREENING STUDY PRODUCTS

Sample	Temp., °C	Arsenic Content, %		
		Sample 1	Sample 2	Sample 3
LA 0001	Unroasted	1.32	1.14	1.37
	600	1.53	1.60	1.54
	700	1.50	1.56	1.60
	800	1.52	1.41	1.72
	1000	1.66	1.58	1.49
VT 0002	Unroasted	18.07	18.72	19.14
	600	10.83	19.18	18.17
	700	15.19	18.40	16.52
	800	15.89	15.92	14.40
	1000	4.30	4.57	4.11
VT 0002B,3C	Unroasted	4.47	4.60	4.59
	600	4.78	4.95	4.27
	700	4.89	4.51	4.78
	800	6.50	6.80	5.82
VT 0002, 1LIME	Unroasted	12.02	11.64	14.63
	700	16.18	9.75	15.81

The conclusions of the screening study test work were that simple roasting of the starting materials in the temperature range 600°C - 1000°C did not stabilize the materials; that roasting the samples with either cement or lime did stabilize the materials (at least against release of arsenic into the TCLP extraction fluid); that a mixture of cement/thiourea/VT-sample reduced the TCLP arsenic release from 7056 (EI value) mg/liter (starting VT sample) and from 578 (EI value) mg/liter (cement/VT-sample at a 1/1 ratio) to 20 mg/liter.

The cement/thiourea/VT-sample was chosen for further consideration and large scale sample generation. The cement/sample and cement/lime/sample materials were studied further in the stated corollary study. The results of that study are available from the REM III Team project officer.

4.4.2. *Phase II Large Scale Studies*

4.4.2.1. *Sample Mixing*

Each starting air dried material was mixed with cement and thiourea as described in Section 3.3. The ratio studied was 1/0.01/1; cement/thiourea/sample. The amounts of each mix material initially prepared are summarized in Table 4.23.

TABLE 4.23. LARGE SCALE PHASE II SAMPLE MIXTURES

Sample	Mix Ratio	Quantity Prepared, Grams
	Cem./Thio/Spl.[1]	
LA 0001,Thio	1/0.01/1	1,500
LA 0002,Thio	1/0.01/1	2,000
VT 0002,Thio	1/0.01/1	2,270

1. The thiourea was added by dissolving it in the water used to cast the cement/sample mixture.

4.4.2.2. *Sample Casting, Aging, and Shipment*

The procedure adopted for the casting and aging stage of sample preparation was the same as discussed in Section 3.3.

A summary of the casting and aging process information is presented in Table 4.24.

Samples were aged twenty-eight days under conditions specified previously. Prior to shipment a qualitative test was conducted to give a preliminary measure of the stability of the cast material. Three cubes were randomly picked out of each sample and a Pocket Penetrometer (Soiltest, Inc. model CL-

700A) test was performed, i.e., the pentrometer was pressed onto the surface and the reading was recorded at which the cube broke. If the cube did not break the reading was recorded as "greater than 4.5". These data are presented in Table 4.25.

TABLE 4.24. PHASE II CASTING, AGING AND SHIPPING

Sample	Weight, gms	Water, gms	Sample Shipped, gms		
			Cubes	Cylinders	
				No.1	No.2
LA 0001B,Thio	1710	600	2438	276	510
LA 0002B,Thio	1710	600	1190	447	
VT 0002B,Thio	1180	600	1225	451	448

TABLE 4.25. UNCONFINED SEMI-QUANTITATIVE STRENGTH TEST RESULTS ON PHASE II PRODUCTS

Sample	Strength, kg/cm^2 (Tons/ft^2)[1]		
	Sample 1	Sample 2	Sample 3
LA 0001B,Thio	> 4.5	> 4.5	> 4.5
LA 0002B,Thio	4.5	> 4.5	> 4.5
VT 0002B,Thio	> 4.5	> 4.5	> 4.5

1. Soiltest, Inc Model CL 700-A Pocket Penetrometer.

4.4.2.3. *Arsenic Content in Final Phase II Products*

The arsenic contents of the final cast and aged samples are presented in Table 4.26.

TABLE 4.26. ARSENIC CONTENT IN PHASE II PRODUCTS

Sample	Arsenic Content, %
LA 0001B,1:1,Thio	0.36, 0.21, 0.24
LA 0002B,1:1,Thio	0.60, 0.71, 0.46
VT 0002B,1:1,Thio	12.66, 12.06, 12.37

4.4.2.3. *Product Leach Results*

Phase II products were characterized by three leach tests (data supplied by REM III Team: data are preliminary and unvalidated): TCLP, Modified-ASTM (A) Deionized Water Leach, and Modified-ASTM (B) Carbonate Leach. The leach response measurements followed in this study were arsenic and total organic carbon. The arsenic and total organic carbon (TOC) concentrations in the samples subjected to the leach tests are presented in Table 4.27. Total volatile solids (TVC), starting sample pH, and specific gravity in the samples subjected to the leach tests are presented in Table 4.28. The leach test results are presented in Tables 4.29 (TCLP,As), 4.30 (ASTM-A,As), 4.31 (ASTM-B,As), 4.32 (ASTM-A,TOC), and 4.33 (ASTM-B,TOC).

TABLE 4.27. ARSENIC AND TOC CONTENT IN PHASE II PRODUCTS SUBJECTED TO LEACH TEST WORK

Sample	Concentration, mg/kg[1]	
	Arsenic	TOC
LA 0001B,1:1,Thio	3560	18700
LA 0002B,1:1,Thio	5140	14200
VT 0002B,1:1,Thio	31000	25900

[1] REM III Team Data: Analyses performed by Versar, Inc.

TABLE 4.28. CHARACTERISTICS OF PHASE II SOLID PRODUCTS SUBJECTED TO LEACH TESTS

Sample	Characteristic[1]		
	TVS,%[2]	Sample pH	Specific Gravity
LA 0001	27.6	5.6	-
LA 0001B,1:1,Thio	5.5	12.32	2.59
LA 0002	34.7	3.9	-
LA 0002B,1:1,Thio	5.0	12.24	2.59
VT 0002	43.6	8.6	-
VT 0002B,1:1,Thio	7.8	12.35	2.21

[1] REM III Team Data: Test performed by Versar, Inc.
[2] Total Volatile Solids

TABLE 4.29. TCLP RESULTS FOR PHASE II PRODUCT MATERIALS

Sample	Arsenic Extraction Concentration, mg/liter[1]	
LA 0001	4.6	(6.1)
LA 0001B,1:1,Thio	18.8	(11.63)
LA 0002	2.36	(6.2)
LA 0002B,1:1,Thio	39.2	(11.69)
VT 0002	2260.	(7.0)
VT 0002B,1:1,Thio	93.1	(11.59)

1 REM III Team Data: Test work performed by Versar, Inc.
2 pH in parentheses.

Note that:

- The LA mixtures released more arsenic to the extractant fluid than did the untreated starting samples.

- There is a significant decrease in the arsenic extracted from the VT mixture than from the VT starting material, i.e., there is a 95.9% reduction in the quantity of arsenic extracted from the mixture. The reduction is most likely the result of the presence of the cement not the presence of thiourea (as shown by the following comment).

- Comparing the TCLP results from Phase I of a separate study (Cement Casting[2]) and the present Phase II TCLP leach results (in mg/liter) shows that the presence of thiourea is probably not an important factor, e.g.,

	Phase I[2] (Table 4.11) (no thiourea)	Phase II (Table 4.29) (thiourea)
LA 0001B,1C	15.8	18.8
LA 0002B,1C	30.1	39.2
VT 0002B,1C	72.2	93.1

TABLE 4.30. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE II PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]						
	Deionized Water Leach[2]						
Extract	1		2		3		Cumulative
LA 0001	5.03	(7.6)	4.88	(7.6)	1.86	(8.4)	11.77
LA 0001B,1:1,Thio	22.6	(12.06)	7.37	(12.31)	6.20	(12.43)	36.2
LA 0002	5.18	(7.9)	4.47	(7.7)	6.22	(8.6)	15.87
LA 0002B,1:1,Thio	49.4	(12.11)	12.4	(12.25)	9.17	(12.40)	71.0
VT 0002	1650.	(8.6)	851.	(8.5)	512.	(9.0)	3013.
VT 0002B,1:1,Thio	287.	(12.29)	109.	(12.23)	63.9	(12.32)	489.9

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 Final pH values in parentheses.

Note that:

- The LA mixtures released more arsenic to the deionized water leach than did the untreated starting samples.

- There is a significant decrease in the arsenic extracted from the VT mixture than from the VT starting material, i.e., there is a 83.7% reduction in the quantity of arsenic extracted from the mixture. The reduction is most likely the result of the presence of the cement not the presence of thiourea (as shown by the following comment).

- Comparing the deionized leach results from Phase I of a separate study (Cement Casting[2]) and the present Phase II deionized leach results (in mg/liter) shows that the presence of thiourea is probably not an important factor, e.g.,

	Phase I[2] (Table 4.12) (no thiourea)	Phase II (Table 4.30) (thiourea)
LA 0001B,1C	39.8	36.2
LA 0002B,1C	63.2	71.0
VT 0002B,1C	304.0	489.9

TABLE 4.31. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE II PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]						
	Carbonate Leach[2]						
	1		2		3		Cumulative
LA 0001	10.5	(8.9)	126.0	(10.6)	101.0	(10.8)	237.5
LA 0001B,1:1,Thio	24.5	(12.18)	14.8	(12.18)	25.0	(12.04)	64.3
LA 0002	26.7	(8.4)	105.0	(10.7)	115.0	(10.9)	246.7
LA 0002B,1:1,Thio	70.9	(12.20)	14.7	(12.18)	24.7	(12.10)	110.3
VT 0002	2060.	(8.7)	2710.	(10.4)	1900.	(10.7)	6670.
VT 0002B,1:1,Thio	414.	(12.22)	16.7	(12.08)	20.3	(12.03)	451.0

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 Final pH values in parentheses.

Note that:

- The cumulative arsenic extracted by the carbonate leaches is considerably greater than the arsenic extracted by the deionized water leaches (except for VT 0002B,1:1,Thio).

- There is a significant decrease in the arsenic extracted from the VT mixture than from the VT starting material, i.e., there is a 93.2% reduction in the quantity of arsenic extracted from the mixture. The reduction is most likely the result of the presence of the cement not the presence of thiourea (as shown by the following comment).

- A comparsion of the carbonate leach results from Phase I of a separate study (Cement Casting[2]) and the present Phase II carbonate leach results (in mg/liter) is presented below:

	Phase I[2] (Table 4.13) (no thiourea)	Phase II (Table 4.31) (thiourea)
LA 0001B,1C	77.3	64.3
LA 0002B,1C	100.7	110.3
VT 0002B,1C	664.0	451.0

TABLE 4.32. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE II PRODUCTS: TOTAL ORGANIC CARBON

Sample	Concentration, mg/liter[1]						
Extract[2]	1		2		3		Cumulative
	TOC[3]	TDS[4]	TOC	TDS	TOC	TDS	TOC
LA 0001	10.0		8.5		6.6		25.1
LA 0001B,1:1,Thio	221.	2240	35.7	1570	17.7	1110	274.4
LA 0002	11.0		12.8		9.8		33.6
LA 0002B,1:1,Thio	122.	2060	31.7	1590	18.8	1140	172.5
VT 0002	1019.		414.		166.		1599.
VT 0002B,1:1,Thio	423.	3190	110.	1590	57.0	1060	590.0

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 Total Organic Carbon
4 Total Dissolved Solids

Note that:

- The LA mixtures released more organic carbon to the deionized water leach than did the untreated starting samples.

- There is a significant decrease in the organic carbon extracted from the VT mixture than from the VT starting material, i.e., there is a 63.1% reduction in the quantity of organic carbon extracted from the mixture.

TABLE 4.33. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE II PRODUCTS: TOTAL ORGANIC CARBON

Sample	TOC Concentration, mg/liter[1]			
Extract[2]	1	2	3	Cumulative
LA 0001	17.5	232.0	208.0	457.5
LA 0001B,1:1,Thio	236.	37.7	18.5	292.2
LA 0002	36.6	119.0	182.0	337.6
LA 0002B,1:1,Thio	124.	31.7	17.6	173.3
VT 0002	1008.	659.	226.	1893.
VT 0002B,1:1,Thio	424.	86.0	50.1	560.1

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant B, 48 hour exposure in each extraction.
3 Total Organic Carbon

Note that:

- The cumulative organic carbon extracted from the untreated samples (by the carbonate leaches) was considerably greater than the organic carbon extracted by the deionized water leaches. But, approximately the same extractions resulted for the cement/thio/sample mixtures in deionized water and in the carbonate environment.

- There is a significant decrease in the organic carbon extracted from the VT mixture than from the VT starting material, i.e., there is a 70.4% reduction in the quantity of organic carbon extracted from the mixture.

SECTION FIVE
REMEDIAL DESIGN AND COST ESTIMATES

Engineers International and its subcontractor Montana Enviromet have performed a treatability study on solidification and stabilization methodologies for remediation of sludges and contaminated soils at the Whitmoyer Laboratories site. The sludges and contaminated soils result from calcium and ferric precipitation of arsenic compounds. The sludges also contain some aniline and other organic compounds. The solidification and stabilization technologies tested included:

- *Clay Pelletizing and Sintering, and*
- *Clay Casting.*

This report addresses remediation alternatives based on the ***clay pelletizing and sintering technology***. A separate report has been prepared on the cement casting technology.

The objective of the solidification and stabilization using clay is to reduce the leachability of arsenic in the treated product to a level that would permit the material to be successfully delisted as a hazardous waste. The treated sludges and contaminated soil could then be disposed on-site as a non-hazardous waste. The following section describes projections of proposed full-scale remediation of the Whitmoyer site utilizing clay solidification/ stabilization technologies tested in the treatability program.

The elements addressed in this discussion include: treatment effectiveness, state of technology development, requirements for additional pilot testing, site-specific design effort required, equipment costs, construction costs and schedule, start-up schedule, operating costs and schedule, treated waste disposal and decommissioning of treatment facilities.

5.1 *TREATMENT EFFECTIVENESS*

The solidification/stabilization remedial alternatives tested in this program were listed by Ebasco Services, Inc. (Ebasco) in their treatability testing specifications. In these specifications treatment objectives were specified by Ebasco, to which the treated waste product leachability was to be compared. These objectives are presented in Table 5.1. As indicated in these treatment objectives, the primary parameter of concern is leachable arsenic.

TABLE 5.1. TREATMENT OBJECTIVES FOR THE WHITMOYER LABORATORIES SITE

Procedure	Arsenic Leachate Concentration	Organic Leachate Concentration
TCLP Leachate	1.0 mg/liter	10%*
Modified ASTM Leachate A**	1.0 mg/liter	10%*
Modified ASTM Leachate B**	1.0 mg/liter	10%*

* Of original-untreated leachate concentration. Measured as TOC and only applicable if significant organics are detected.

** See Table 2-1 for modifications to ASTM Leachate.

The treated sludge must pass the TCLP leachate requirements under RCRA 268. Additional tests were proposed, since the treated waste is not likely to be exposed to the acidic conditions of the TCLP leach. Under certain situations arsenic can be more leachable at higher pH's. Hence, the leachability was also measured under less acidic conditions of modified distilled water leaches. The results of the TCLP tests conducted on Phase I samples (presented in Table 4.11) shows that clay pelletizing and sintering had a detrimental effect on the LA samples but significantly reduced arsenic release from the VT waste material. However, the separate(2) study using cement was much more successful in reducing arsenic release from all three test materials.

Because of the significant reduction in arsenic release by the clay treated VT material, cost estimates were prepared for the clay pelletizing and sintering technology. This technology, however, has serious drawbacks to the actual application to the tested materials.

5.2 *STATE OF TECHNOLOGY DEVELOPMENT*

The development of clay stabilization and solidification technology is less advanced than that of cement stabilization. One major study on the stabilization of arsenic wastes using clay was performed by Mehta and Twidwell(4). One element that is not fully studied is the impact of certain organic compounds such as the aniline and organic arsenic and their effect on stabilization of the clay/waste.

5.3 *SITE COMPATIBILITY*

The process alternatives addressed in this program were designed in an effort to maintain compatibility and to effectively remediate the conditions on the existing Whitmoyer site. In doing so, a couple of elements were emphasized: the transportability of the processing equipment and the use of mobile equipment where possible. The use of transportable equipment enhances the ease by which the remediation facility can be decommissioned and removed, and the site returned to appropriate condition. The size of the largest pieces of equipment (i.e. roaster, drier and pug mill mixers) were limited to assure the transportability of the equipment to the site.

5.4 *FLOWSHEET*

The flowsheet of the alternative proposed in this evaluation is presented in Figure 5.1. The process, which is addressed in this activity, starts with the wet sludge being placed in a bin. The cost estimates generated do not include any excavation operation.

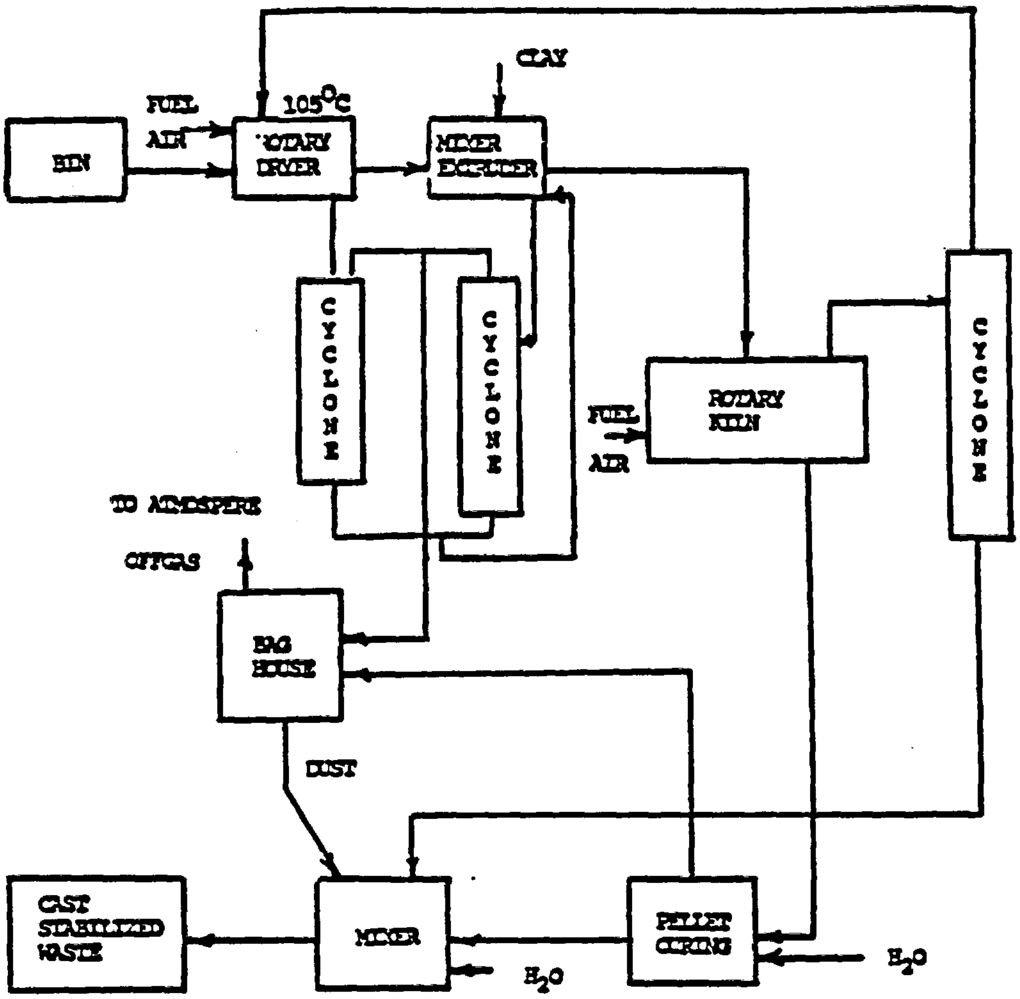

FIGURE 3.1. WHITMOYER REMEDIATION FLOWSHEET: CLAY OPTION

The cost estimates do include funds for the placement of the clay/waste pellets. But they do not include cost for excavation nor preparation of a lined earthen cell nor construction of a RCRA-type hazardous waste cell into which the clay/waste pellets are placed.

5.4.1 *Extrude and Sinter*

In the extrude and sinter alternative the wet sludge and soil (30% H_2O) is charged at 2.5 tons per hour to a 15-ton bin. From there the waste is fed via a screw feeder through a grizzly, to remove the plus 1/2" rocks, into a pug mill which is utilized to mix the wet sludge, clay (7.5 tons per hour) and necessary water are added to form a thick homogeneous slurry. The slurry is then extruded through a die into pellets. The pellets are allowed to dry and develop sufficient strength to be placed into a sintering furnace.

The dried pellets are then fed to a traveling grate or rotary kiln (roaster) via a conveyor, where they are fired at $1000^{o}C$ ($1832^{o}F$). The off-gas from the roaster passes through a cyclone and thence into the pellet curing oven. The dust from the roaster cyclone is fed into a wet slurry mixer. The kiln is fired with fuel oil and air.

The fired pellets reporting from the roaster are allowed to cool. The pellets are then placed in an appropriate cell or container for disposal. The pellets could also be cast in a cement monolith. Using this approach a pumpable slurry could be produced into which the fired pellets are placed as aggregate. The slurry would then be pumped via a 3" pipe line to the mobile pumper and boom where it is cast into the appropriate disposal cells.

5.5 *COST ESTIMATES*

5.5.1. *Capital Cost Estimates*

The capital cost estimates of the major equipment were obtained from equipment vendors' estimates. Vendors were given a list of design criteria which is presented in Table 5.2. The costs for smaller pieces of equipment (i.e., bins, silos, screw conveyors and tanks) were obtained from the costing guides. These standard costing guides include Chemical Engineering Costs by Dryden and Furlow(11) and Process Plant and Equipment Cost Estimates by Kharbanda(12). The costs were updated for inflation utilizing the Marshall and Stevens Index(13).

TABLE 5.2. DESIGN CRITERIA FOR WHITMOYER REMEDIATION

WASTE PRODUCTS

	Bulk Den. lb/ft^3	Volume yd^3	Dry Wt. tons	Moisture		As Content %
				As-Rec.	Air-Dried	
LA0001, wet	120	4,000	5,378	20.5	2.7	1.3
LA0002, wet	100	9,000	9,125	33.1	3.5	2.6
VT0002, wet	140	3,000	4,333	30.7	7.4	18.0

DESIGN CRITERIA

	Sintering Option
Feed Rate Dry Sludge,tph	5.0 - 2.5
Feed Rate Wet Sludge,tph	7.2 - 3.6
Contained Water,tph	3
Feed Rate Clay/Sludge,tph	10
Feed Rate Clay,tph	7.5 - 5.0
Feed Rate Wat. (Clay/Sludge)	6.7
Moist. Content Wet Sludge,%	30
Moist. Content Dry Sludge,%	5
Drying Temp.,°C	105
Sintering Temp.,°C	1000
Roasting Time, minutes	15

Clay	Specific Gravity
	$FeAsO_4$, 3.18 g/cc
Clay Addition, 300% - 100%	$Ca_3(AsO_4)_2$, 2.765
Water Addition to Clay/Sludge, 40%	

Operation

Life of Oper., 31 - 63 wks, Roast

Start-up, 6 additional wks, Roast

Oper. Schedule, 3 - 8 hr shifts, 5 days/wk (Preventive Maintenance, Weekends)

Health and Safety, Level C - Excavation and as-needed due to dust, otherwise D.

Fuel, Fuel Oil

Power, 220 V - 3 Phase, 110 V - 2 Phase

TABLE 5.2. DESIGN CRITERIA FOR WHITMOYER REMEDIATION (Continued)

Specific Heat, cal/g-°C	Particle Size
Wet Sludge, 0.25	Cement, 100% minus 200 mesh, 90% minus 325 mesh
Dry Clay/Sludge, 0.2	Sludge, 5% plus 3/4"
Fuel Oil, 0.5	15% minus 3/4", plus 1/4"
Water, 1.0	20% minus 1/4", plus 200 mesh
Steam, 0.5	15% minus 200 mesh, plus 270 mesh
CO_2, 0.18	10% minus 270 mesh, plus 325 mesh
Air, 0.25	25% minus 325 mesh, plus 400 mesh
	10% minus 400 mesh

The installation costs of the major equipment were obtained from vendors or developed from factored estimates. The capital cost estimates including installation are presented in Table 5.3. The total project capital cost was estimated to be 150% of the installation cost and 50% of the equipment cost. The additional 50% of installation cost was estimated for decommissioning and closure. Half of the capital costs were estimated to be recovered by leasing the equipment, resale or reuse of the used equipment on other Superfund sites. The use of conventionally cement equipment (i.e., cement pumpers or mobile mixer trucks) or rotary incinerators allows for future utilization of this used equipment and the potential for its lease.

Estimates for land and site development were not included. It was assumed that utility connections were available on site and could be connected to the installed equipment with limited additional expense. Major buildings were not included in the cost estimates. Disposal of waste materials, such as contaminated soils, would be achieved by including it in the process stream. Drums and other waste items would be cast into the monolith.

Engineering expenses of the construction and installation on the facility were assumed to be included in the vendor manufacturing and installation costs. Legal, licensing and permitting costs were assumed to be outside the scope of the cost estimates. Start-up costs were estimated from the time projected for the start-up operations plus 10% for vendor assistance. Contingencies, allowance to cover costs of unforeseen circumstances, such as adverse weather conditions and strikes, and inadequate site characterization were not included in the capital costs estimates, but contingencies were covered by adding a 20% factor of labor costs.

The total equipment costs for the roaster option summed to a value of $4,523,000 with an installation cost of $1,431,500. It was estimated that 50% of the equipment cost could be recovered due to reuse or resale, and that decommissioning and closure cost would be approximately 50% of installation costs. Hence, the total capital costs including equipment, installation, closure and resale totaled $4,408,750 for the roaster option.

TABLE 5.3. WHITMOYER EQUIPMENT COSTS: CLAY SINTERING OPTION

DESCRIPTION	EQUIPMENT COST	INSTALL COST	Hp
ROTARY KILN	$3,500,000.00	$1,200,000.00	200
COOLER			
CYCLONE			
SEC COMBUSTION			
BAG HOUSE			
PELLET DRYER, CYCLONE	$300,000.00	$150,000.00	
FEEDER	$25,000.00	$10,000.00	15
PUGMILL	$28,000.00	$10,000.00	30
MOBILE PUMPER	$250,000.00	-	10
SLURRY PUMP	$90,000.00	$10,000.00	100
PIPELINE (4" x 600')	$10,000.00	$5,000.00	
FUEL TANK (5000 GAL)	-	-	
CEMENT SILO (750T)	$87,000.00	$15,000.00	
PNEUMATIC LOAD SYS	$150,000.00	$20,000.00	25
H2O TANK (25000 GAL)	$10,000.00	$5,000.00	
BIN (20 YD)	$10,000.00	$5,000.00	10
CYCLONES(2-2000 CFM)	$48,000.00	-	
SCREW CONVEYOR (2)	$15,000.00	$1,500.00	
TOTAL	$4,523,000.00	$1,431,500.00	390
CAPITAL RECOVERY	$2,261,500.00		
CAPITAL COSTS	$4,408,750.00		

5.5.2 *Operating and Maintenance Costs*

The operating and maintenance costs include labor, materials, power and fuel. The operating labor costs include wages, salaries, overhead and fringe benefits associated with the operation of the remediation facility. It was assumed the plant would be operating 24 hours per day, 5 days per week with maintenance being performed on weekends. Maintenance material and labor were estimated on an annual basis using 15% as suggested in the Strategy of Process Engineering by Rudd and Watson(14).

Labor cost was estimated at $20.00 per man hour, plus 13% burden (FICA), 15% labor (markup) resulting in a $25.60 per man hour direct labor cost. An indirect cost factor of 75% of direct labor was added. This total labor cost was multiplied by 10% for profit, resulting in an estimated total labor cost to the project of $66.53 per man hour.

Factors for performing remediation activities in Health and Safety levels B and C are presented. In the roasting option there is a possibility for dust releases. Therefore, in one cost scenario, 50% of the labor effort for the roasting option was assumed to be performed in level C at an increased cost of 70% for those hours. This assumption resulted in a substantial increase in labor cost, and the risk should be assessed further in the Whitmoyer program. The labor cost for the two scenarios are presented in Table 5.4.

TABLE 5.4. WHITMOYER LABOR COSTS: CLAY SINTERING OPTION

BASIS FOR ESTIMATE

$20.00	/MAN HOUR
$3.00	15% LABOR MARK-UP
$19.20	75% OF TOTAL DIRECT LABOR (INDIRECT LABOR)
$4.48	10% OF TOTAL DIRECT COST (PROFIT)
$49.28	/FULLY BURDENED MAN HOUR
$7.39	15% FOR H & S MONITORING
$9.86	20% FOR CONTINGENCY
$66.53	**TOTAL LABOR COST/HOUR**

PREMIUM FOR HAZARDOUS WORK ABOVE LEVEL D: LEVEL B 150%, LEVEL C 70%

TABLE 5.4. WHITMOYER LABOR COSTS: CEMENT CASTING (Continued)

<u>ESTIMATES</u>

<u>CLAY SINTER</u>

	MEN/SHIFT			
(NO TIME LEVEL C)	5	6	7	8
64 WKS 120HR/WK	$2,554,752.00	$3,065,702.40	$3,576,652.80	$4,087,603.20
48 WKS	$1,916,064.00	$2,299,276.80	$2,682,489.60	$3,065,702.40
32 WKS	$1,277,376.00	$1,532,851.20	$1,788,326.40	$2,043,801.60

	MEN/SHIFT			
(50% LEVEL C)	5	6	7	8
64 WKS 120HR/WK	$3,448,915.20	$4,138,698.24	$4,828,481.28	$5,518,264.32
48 WKS	$2,586,686.40	$3,104,023.68	$3,621,360.96	$4,138,698.24
32 WKS	$1,724,457.60	$2,069,349.12	$2,414,240.64	$2,759,132.16

The power cost was based on total connected horsepower, which would give an adequate safety factor pertaining to power requirements. The cost of power was estimated at $0.10 per kilowatt hour. Fuel usage estimates for mobile equipment was based on user experience. The fuel usage for firing the roaster and drier were based on material and heat balances, and assuming a factor of 60% heat efficiency. The fuel cost was estimated at $1.00 per gallon for both diesel and fuel oil. Bentonite clay, which was a very major cost item, was estimated at $80.00 per ton F.O.B. the Whitmoyer site based on bulk purchases above 19,000 tons over the life of the project. This estimate was obtained from Christensen Drilling. No markup on supplies, materials or subcontractor services was included.

The calculation of these non-labor costs are presented in Table 5.5 at three different clay to waste ratios. The total operating and maintenance costs (including labor) are presented in Table 5.6. To illustrate the magnitude of the total costs involved the operating and maintenance costs for a 3:1

clay:waste ratio plus the capital and installation cost are listed below for a 7-man shift with and without level C (50%) for the roaster.

	OPER/MAINT	OPER/MAIN/CAPTL	COST/YD
ROASTER	$12,059,830	$16,468,580	$1,029
ROASTER (LEVEL C)	$13,311,659	$17,720,409	$1,108

At lower clay to waste ratios the costs per yard are substantially reduced as shown below. These figures emphasize the importance of optimizing the clay:waste ratio.

7-Man/Shift

	1:1 COST/YD	2:1 COST/YD	3:1 COST/YD
ROASTER	$605	$817	$1,029
ROASTER (LEVELC)	$644	$876	$1,108

Additional calculations for various sizes of labor shifts are presented in Table 5.7.

TABLE 5.5. OPERATING AND MAINTENANCE COSTS OTHER THAN LABOR: CLAY SINTERING

WHITMOYER CLAY COSTS

WASTE, TONS	CLAY/WSTE RATIO	CLAY COST, $/TON	TOTL CLAY COST, $
19000	3	80.00	4,560,000.00
19000	2	80.00	3,040,000.00
19000	1	80.00	1,520,000.00

WHITMOYER FUEL COST

WASTE, YD	CLAY+WSTE	CAST, YD	FUEL, GAL/YD	COST, $/GAL	TOTAL FUEL COST, $
16000	4	64000	45	1.00	2,880,000.00
16000	3	48000	45	1.00	2,160,000.00
16000	2	32000	45	1.00	1,440,000.00

TABLE 5.5. OPERATING AND MAINTENANCE COSTS OTHER THAN LABOR: CLAY SINTERING (Continued)

WHITMOYER POWER COST: ROASTER

HP	HR/WEEK	WKS	POWER, WATT/HP	COST, $/KWH	TOTAL POWER COST, $
460	120	64	746	0.10	263,547.00
460	120	48	746	0.10	197,660.00
460	120	32	746	0.10	131,773.00

WHITMOYER MAINTENANCE COST: ROASTER

FACTOR	WKS	FRACTION YEAR	EQUIPMENT COST, $	MAINTENANCE COST, $
0.15	64	1.23	4,223,000.00	779,630.77
0.15	48	0.92	4,223,000.00	584,723.08
0.15	32	0.62	4,223,000.00	389,815.38

TABLE 5.6. TOTAL WHITMOYER OPERATING AND MAINTENANCE COST: CLAY SINTERING

	CLAY:WASTE RATIO		
	3:1	2:1	1:1
		NO TIME LEVEL C	
5 MAN SHIFT			
LABOR	$2,554,752.00	$1,916,064.00	$1,277,376.00
CLAY	$4,560,000.00	$3,040,000.00	$1,520,000.00
FUEL	$2,880,000.00	$2,160,000.00	$1,440,000.00
POWER	$263,547.00	$197,660.00	$131,772.00
MAINTENANCE	$779,631.00	$584,723.00	$389,815.00
TOTAL	*$11,037,930.00*	*$7,898,447.00*	*$4,758,965.00*

TABLE 5.6. TOTAL WHITMOYER OPERATING AND MAINTENANCE COST: CLAY SINTERING (Continued)

	CLAY:WASTE RATIO		
	3:1	2:1	1:1
		50% LEVEL C	
5 MAN SHIFT			
LABOR	$3,448,915.20	$2,586,686.40	$1,724,457.60
CLAY	$4,560,000.00	$3,040,000.00	$1,520,000.00
FUEL	$2,880,000.00	$2,160,000.00	$1,440,000.00
POWER	$263,547.00	$197,660.00	$131,772.00
MAINTENANCE	$779,631.00	$584,723.00	$389,815.00
TOTAL	*$11,932,093.00*	*$8,569,070.00*	*$5,206,046.00*
6 MAN SHIFT			
LABOR	$4,138,698.24	$3,104,023.68	$2,069,349.12
CLAY	$4,560,000.00	$3,040,000.00	$1,520,000.00
FUEL	$2,880,000.00	$2,160,000.00	$1,440,000.00
POWER	$263,547.00	$197,660.00	$131,772.00
MAINTENANCE	$779,631.00	$584,723.00	$389,815.00
TOTAL	*$12,621,876.00*	*$9,087,407.00*	*$5,550,938.00*
7 MAN SHIFT			
LABOR	$4,828,481.28	$3,621,360.96	$2,414,240.64
CLAY	$4,560,000.00	$3,040,000.00	$1,520,000.00
FUEL	$2,880,000.00	$2,160,000.00	$1,440,000.00
POWER	$263,547.00	$197,660.00	$131,772.00
MAINTENANCE	$779,631.00	$584,723.00	$389,815.00
TOTAL	*$13,311,659.00*	*$9,603,744.00*	*$5,895,829.00*

TABLE 5.6. TOTAL WHITMOYER OPERATING AND MAINTENANCE COST: CLAY SINTERING (Continued)

	CLAY:WASTE RATIO		
	3:1	2:1	1:1
		50% LEVEL C	
8 MAN SHIFT			
LABOR	$5,518,264.32	$4,138,698.24	$2,759,132.16
CLAY	$4,560,000.00	$3,040,000.00	$1,520,000.00
FUEL	$2,880,000.00	$2,160,000.00	$1,440,000.00
POWER	$263,547.00	$197,660.00	$131,772.00
MAINTENANCE	$779,631.00	$584,723.00	$389,815.00
TOTAL	*$14,001,442.00*	*$10,121,081.00*	*$6,240,721.00*

TABLE 5.7. REMEDIATION COST PER YARD OF WASTE: CLAY SINTERING OPTION

MEN/SHIFT	CLAY:WASTE RATIO		
	3	2	1
ROAST OPTION (NO LEVEL C)			
5	$965	$769	$573
6	$997	$793	$589
7	$1,029	$817	$605
8	$1,061	$841	$621
ROAST OPTION (50% LEVEL C)			
5	$1,021	$811	$601
6	$1,064	$843	$622
7	$1,108	$876	$644
8	$1,151	$908	$666

5.6 *ADDITIONAL PILOT TESTING*

The treatability testing performed in this program utilized a muffle furnace to roast the clay/sludge mixtures. However, in commercial furnaces that might be used in this unit operation (i.e. multi-hearth, travelling grate and rotary) the mixture is treated somewhat differently. Hence, there may be some modification of the optimum roasting temperature and the leachability results that were determined. Also the dust loading of the facility off-gases could have a substantial impact on the operation and through-put of the gas treatment equipment.

Another factor that is quite clearly defined in the prepared cost estimates, is the substantial cost of high clay: waste ratios. If the level of clay can be decreased the operating labor, fuel, power, as well as the clay costs can be reduced.

6. *PRELIMINARY SCHEDULE*

The preliminary schedule developed for the remedial options presented in the report based on the assumption that any institutional requirements that might impact the facility construction, start-up or operation has been completed previous to the equipment order date. However, there is substantial slack in the schedule after the order date and before delivery of the roaster, dryer or pug mill that might be utilized to complete any loose ends prior to construction. The proposed preliminary schedule is presented in Figure 6.1.

After the order date there are 7.5 months prior to delivery of the roaster, which defines the critical path for the roaster option.

The sizing of this facility was based on the largest unit that could be readily transported to the site. The largest roaster that met this criteria was rated at about 10 tph, which at a 3:1 clay:sludge ratio resulted in a 2.5 tph sludge through-put. The anticipated operating period for a 3:1 clay:sludge ratio would be about 64 weeks for a 2:1 ratio about 48 weeks and 32 weeks for a 1:1 ratio.

FIGURE 6.1. WHITMOYER LABORATORIES SITE CONSTRUCTION AND OPERATION SCHEDULE

```
         TASKS                                        MONTHS

                          1  2  3  4  5  6  7  8  9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28
CLAY SINTERING

ENGINEERING             XXXXXX

ORDER EQUIPMENT            X

MANUFACTURE EQUIPMENT      XXXXXXXXXXXXXXXXXXXXX

INSTALL EQUIPMENT                               XXXXXX

PREPARE DISPOSAL CELLS                          XXXXXX

OPERATION START-UP                                    XXXXXX

REMEDIAL OPERATION                                          XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX

FACILITY DECOMMISSION                                                                                    XXX
```

The closure and decommissioning of the facility would require about 1 month for the roaster and about one week for the pug mill, unless it was determined that the roaster brick liner needed to be removed and disposed of on-site due to contamination. This would increase the decommissioning by two to four weeks.

SECTION SEVEN
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SECTION EIGHT
APPENDICES

8.1. HEALTH AND SAFETY REPORT

Attached at end of report.

8.2. TEMPERATURE PROFILES FOR PHASE II SCREENING TEST RESULTS

Temperature profile data are presented in Table 8.1 - 8.3. The temperature profile data were generated for 100 gram samples in 100 cc fireclay crucibles. The procedure was described previously in Section 3.3.

TABLE 8.1. SCREENING TEST TEMPERATURE PROFILES FOR SAMPLE LA 0001

Temperature Designation, °C					
700		800		1000	
Time, min	Temp.	Time	Temp.	Time	Temp.
4	460	7	616	1	272
6	510	12	648	3	484
8	578	15	692	5	578
13	634	19	742	12	766
18	686	34	778	27	912
23	721	49	774	42	982
38	725	64	760	52	980
53	714	79	782	67	984
68	720			76	973
73	708				
80	716				

Note: No fuming evident from furnace door

TABLE 8.2. *SCREENING TEST TEMPERATURE PROFILES FOR SAMPLE VT 0002*

Temperature Designation, °C

600		700		800		1000	
Time,min	Temp.	Time	Temp.	Time	Temp.	Time	Temp.
2	141	1	146	1	263	1	606
6(f)	230	3(f)	244	3(f)		2	644
11	326	5	326	6	363	4(f)	638
15	380	11	462	9	399	10	825
20	482	13	520	15	559	15	870
26	576	23	626	20	656	20	935
28	594	28	658	25(s)	698	35	980
45(s)	724	43(s)	762	60	810	50	1060
51	730	53	784			65	1060
53	720	58	744			80	1020
65	654	73	722				
74	648						

(f) Fume notice escaping from the furnace door.

(s) Fume no longer visible from furnace door.

TABLE 8.3. *SCREENING TEST TEMPERATURE PROFILES FOR SAMPLE VT 0002B,3C*

Temperature Designation, °C					
600		700		800	
Time,min	Temp.	Time	Temp.	Time	Temp.
Not Determined		1	272	1	285
		3	374	5(f)	
		8	550	7	496
		12	626	22	744
		16	646	24	779
		20	695	27	817
		24	740	39	839
		28	740	66	779
		43	718	72	771
		51	708		
		73	704		
		80	708		

(f) fume could be seen over the crucible surface but no fume was visible from the furnace door. Temperature profile shows that the temperature at the sample surface was most likely combusting.

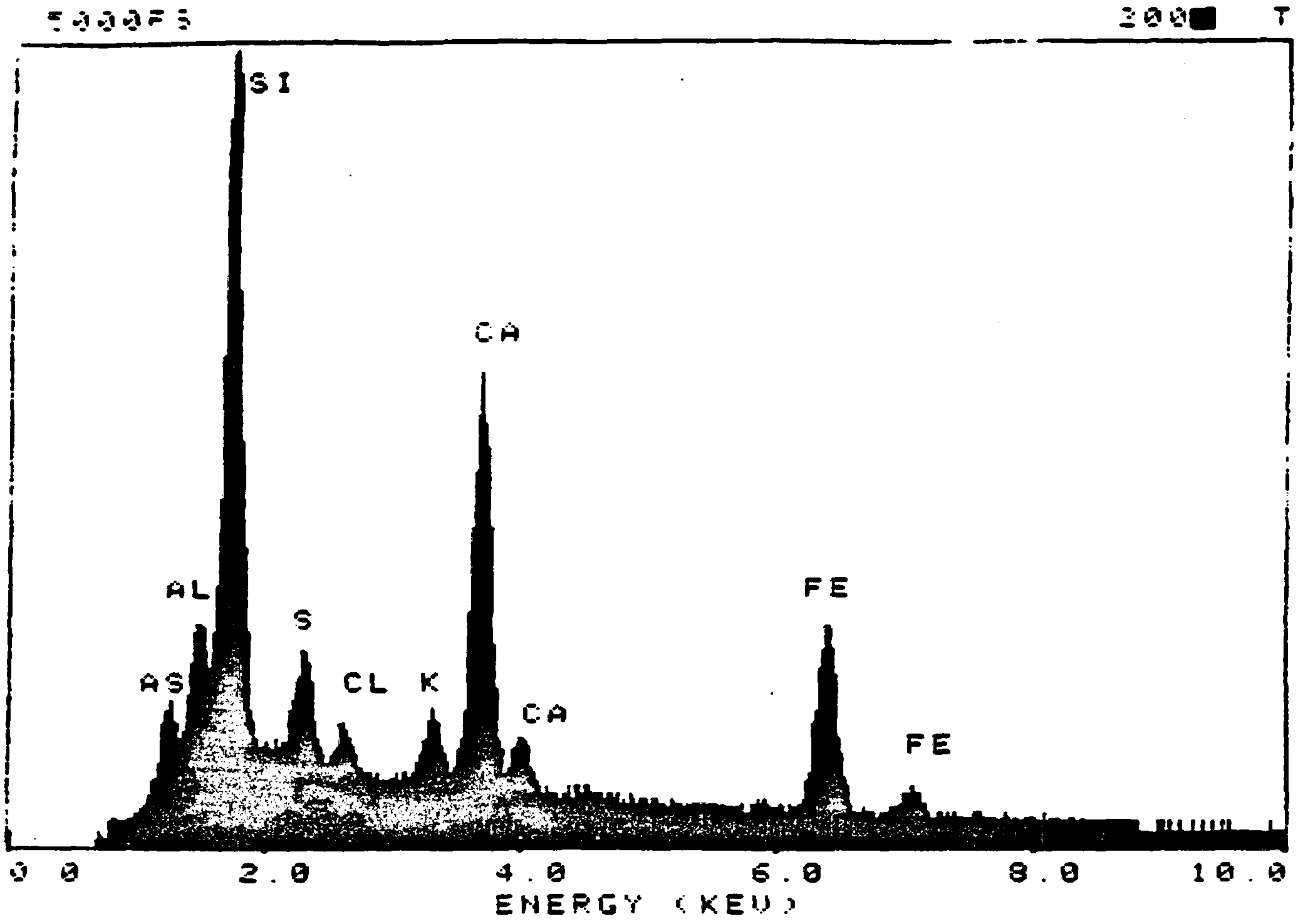
TABLE 8.4. *SCREENING TEST TEMPERATURE PROFILE FOR SAMPLE VT 0002,1,LINE*

600		700		800	
Time,min	Temp.	Time	Temp.	Time	Temp.
Not Determined		5	678	Not Determined	
		7	706		
		8	718		
		18	716		
		28	710		
		71	710		
		80	700		

Note: Fume not evident from the furnace door.

8.3. *SEM-EDX SPECTRA*

Spectra for spot analyses on the sintered sample LA 0001 (See Figure 4.12) are presented in Figures 8.1-8.11.



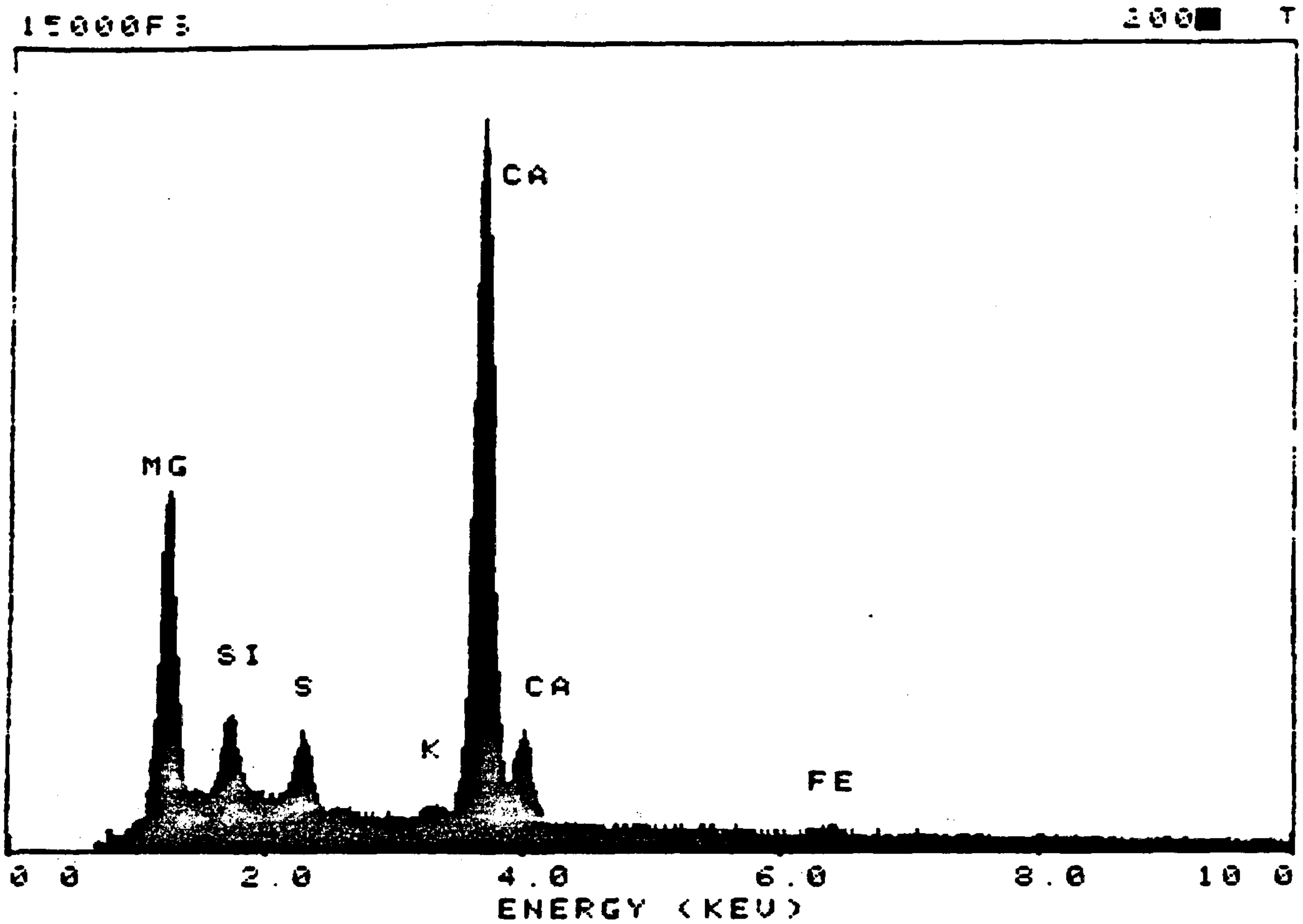
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
SPECTRUM: [illegible] FULL SPECTRA [illegible]

ELEMENT	RELATIVE K	WT %
CA	[illegible]274	15.58
MG	0.0125	[illegible].51
AL	[illegible]621	[illegible]
SI	[illegible]362	46.23
S	[illegible]181	[illegible]
[illegible]	[illegible]139	[illegible].45
FE	[illegible]165	[illegible].49
AS	[illegible]	[illegible]
TOTAL		[illegible]

[illegible]-JAN-89 [illegible]

24-Jan-89 09:27

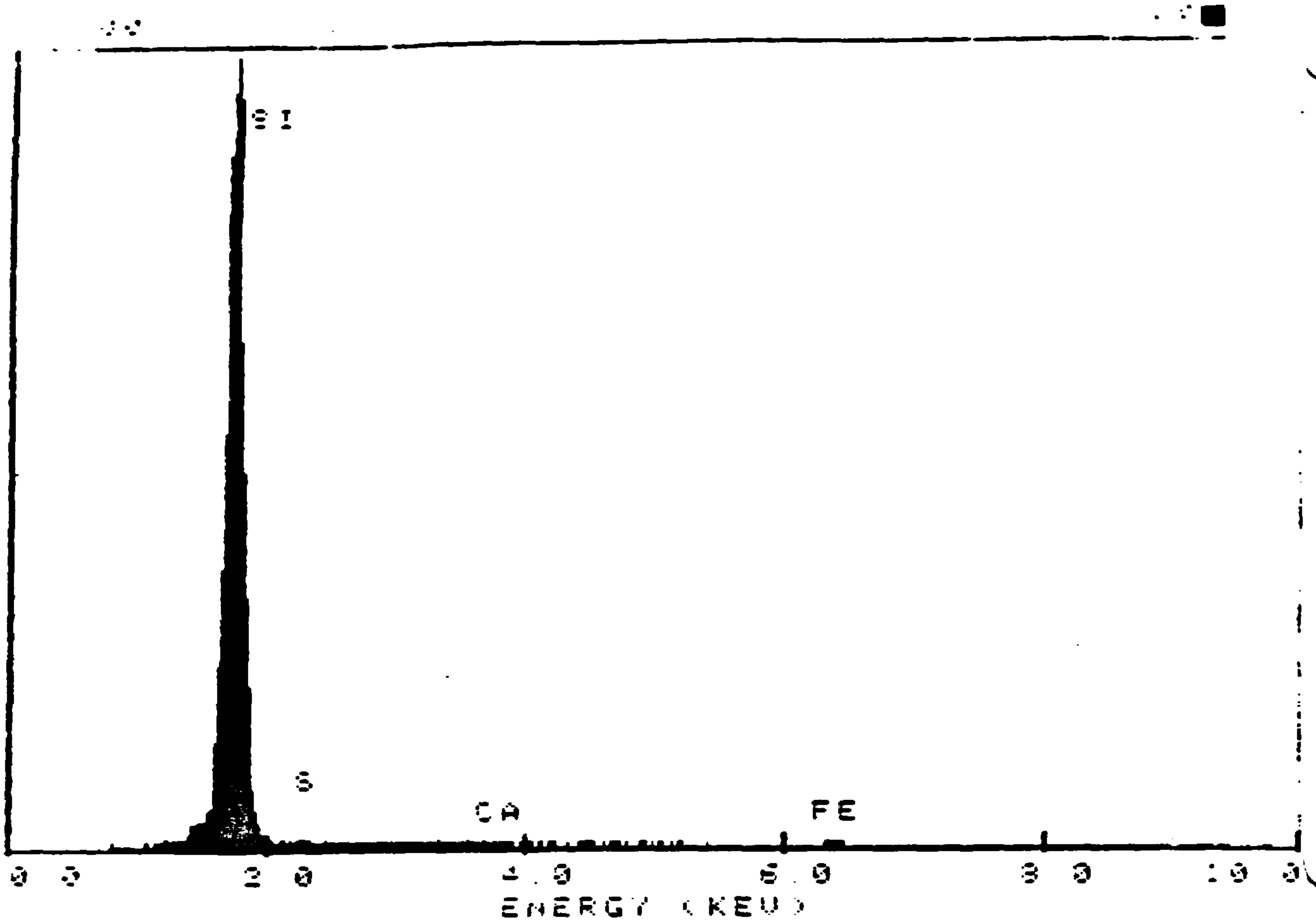
FIGURE 8.1. SEM-EDX SPECTRA FOR MATRIX SPOT 1 IN SAMPLE LA 0001



```
NCSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: 1A001  ROCK PHASE
 ELEMENT          RELATIVE K              WT %
   CA               0.2924               33.61
   MG               0.2626               41.66
   AL               0.0222                6.40
   SI               0.0561               12.34
   S                0.0300                4.80
   K                0.0047                0.55
   FE               0.0050                0.59
   AS               0.0004                0.05
 TOTAL                                  100.00
25-JAN-89                              08:43:23
```

24-Jan-89 09:30

FIGURE 8.2. SEM-EDX SPECTRA FOR SPOT 2 IN SAMPLE LA 0061



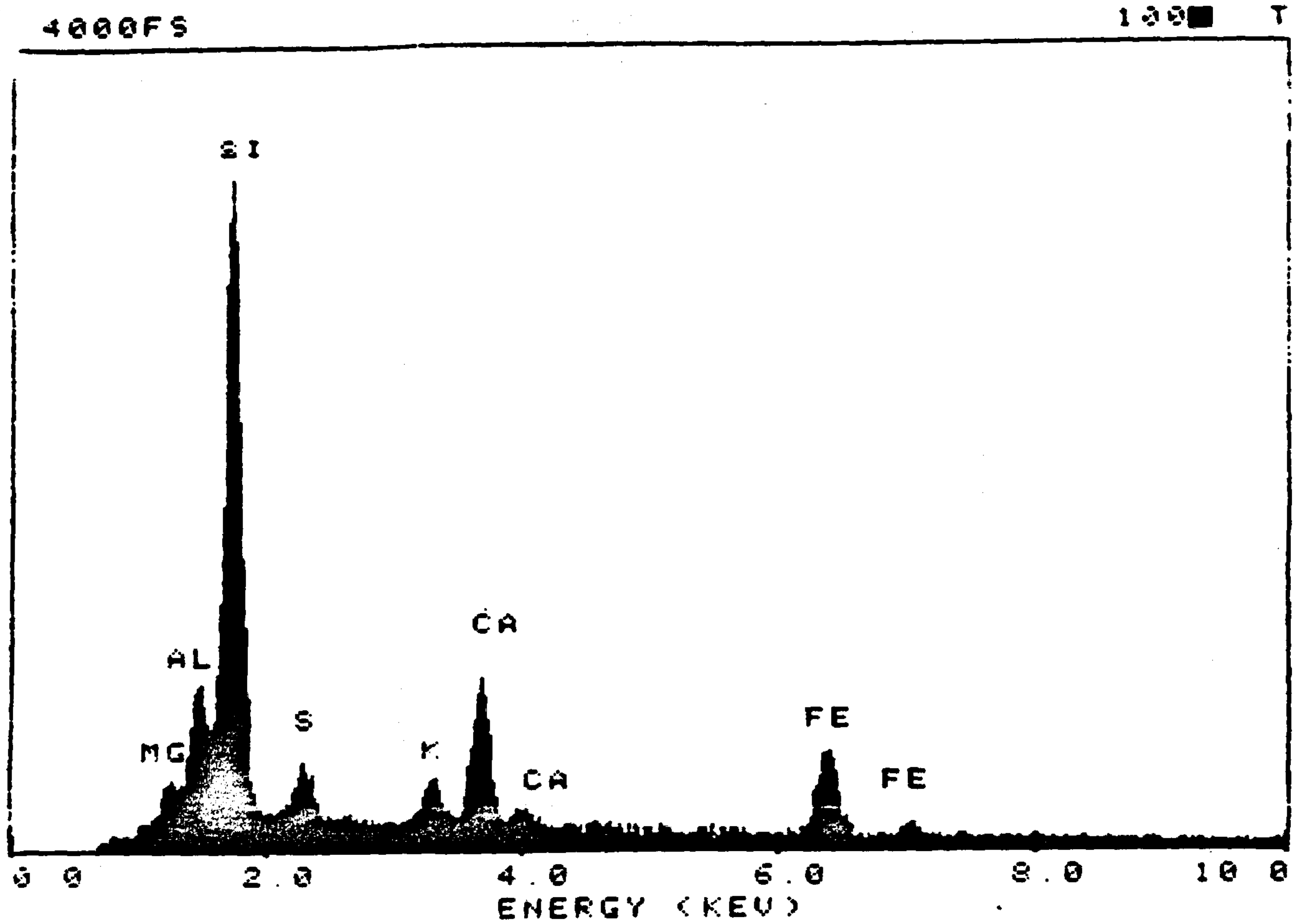
24-Jan-89 09:[illegible]

[illegible] STANDARDLESS ANALYSIS OF [illegible] SAMPLE
SPECTRUM: [illegible]

ELEMENT	RELATIVE	WT %
CA	[illegible]	0.29
MG	0.0000	0.00
AL	[illegible]	0.25
SI	0.9482	[illegible]
S	[illegible]	1.89
[illegible]	[illegible]	[illegible]
FE	[illegible]	0.81
AS	[illegible]	[illegible]
TOTAL		[illegible]

25-JAN-89 [illegible]

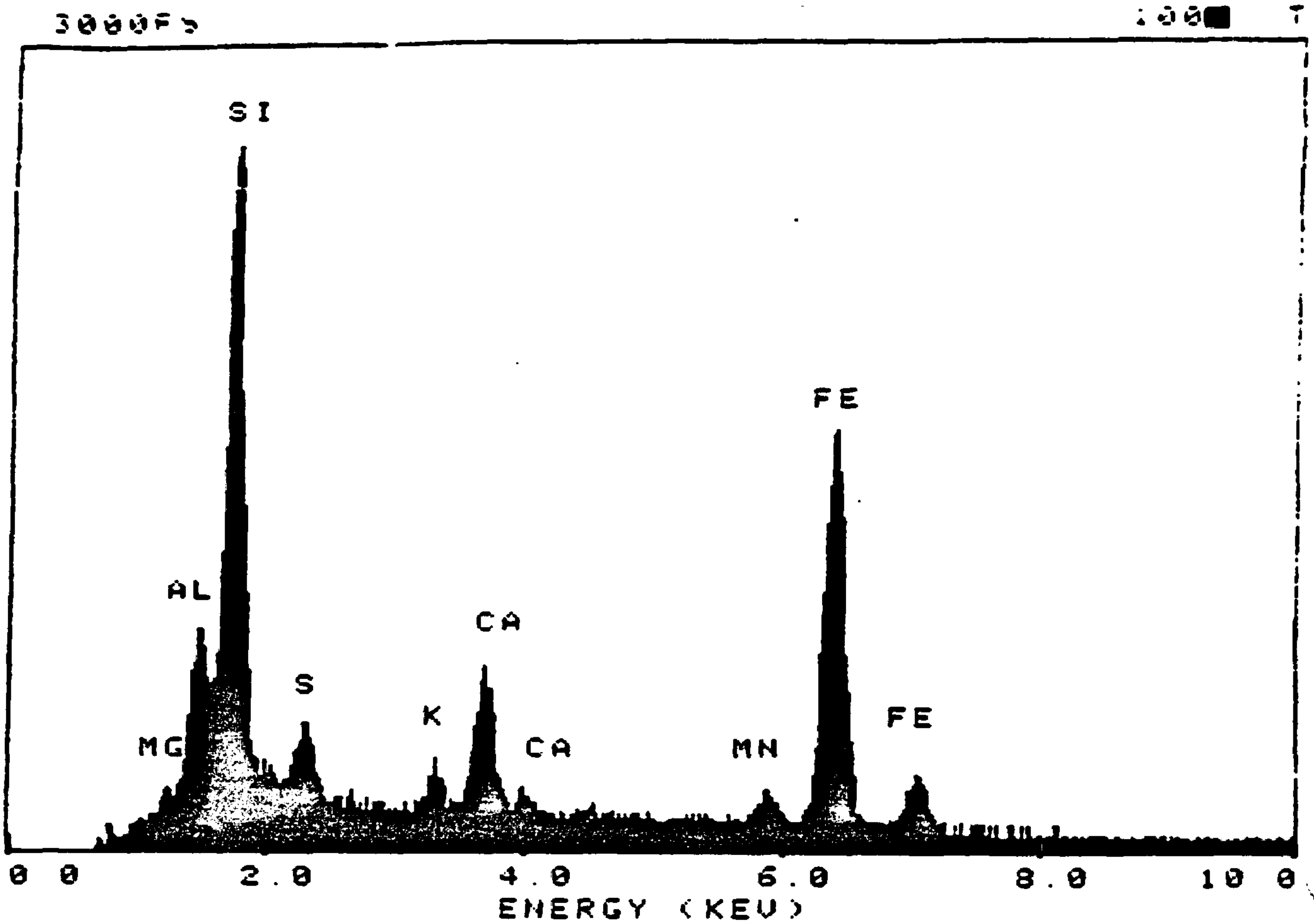
FIGURE 8.3. SEM-EDX SPECTRA FOR SPOT 3 IN SAMPLE LA 0001



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: LA0C3  MATRIX
 ELEMENT        RELATIVE K           WT %
   CA             0.0670             5.42
   MG             0.0165             2.72
   AL             0.0965            13.62
   SI             0.2705            57.64
   S              0.0260             5.32
   K              0.0197             2.67
   FE             0.0844             9.60
   AS             0.0001             0.01
 TOTAL                              100.00
25-JAN-89                         08:45:10
```

24-Jan-89 09:43

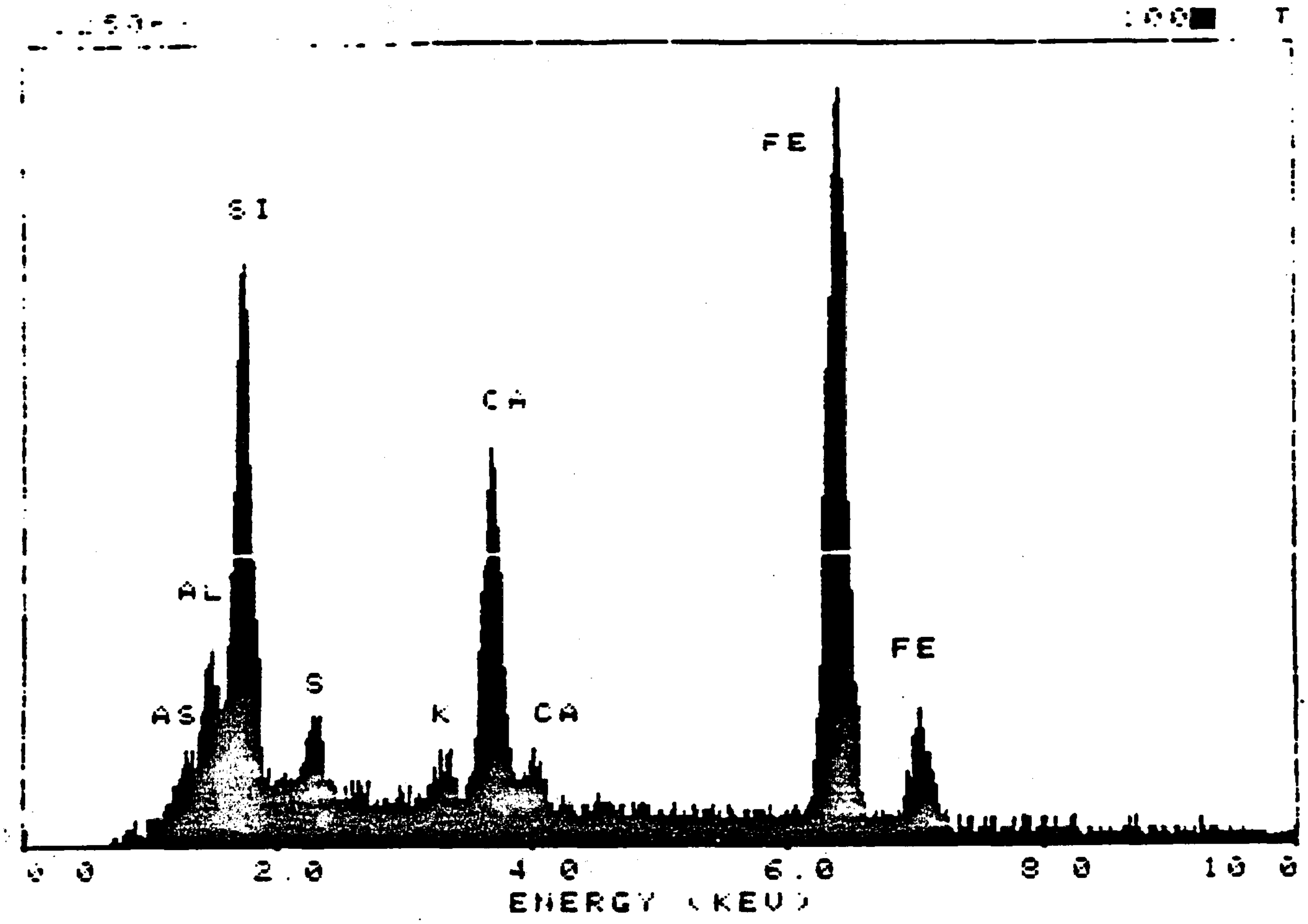
FIGURE 8.4. SEM-EDX SPECTRA FOR SPOT 4 IN SAMPLE LA 0001



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: 1A006  BRIGHT
 ELEMENT          RELATIVE K             WT %
   CA               0.0413               4.94
   MG               0.0025               0.61
   AL               0.0829              14.90
   SI               0.2493              45.52
   S                0.0239               4.68
   K                0.0098               1.29
   FE               0.2576              28.05
   AS               0.0001               0.01
  TOTAL                                100.00
23-JAN-89                             08:45:56
```

24-Jan-89 09:54

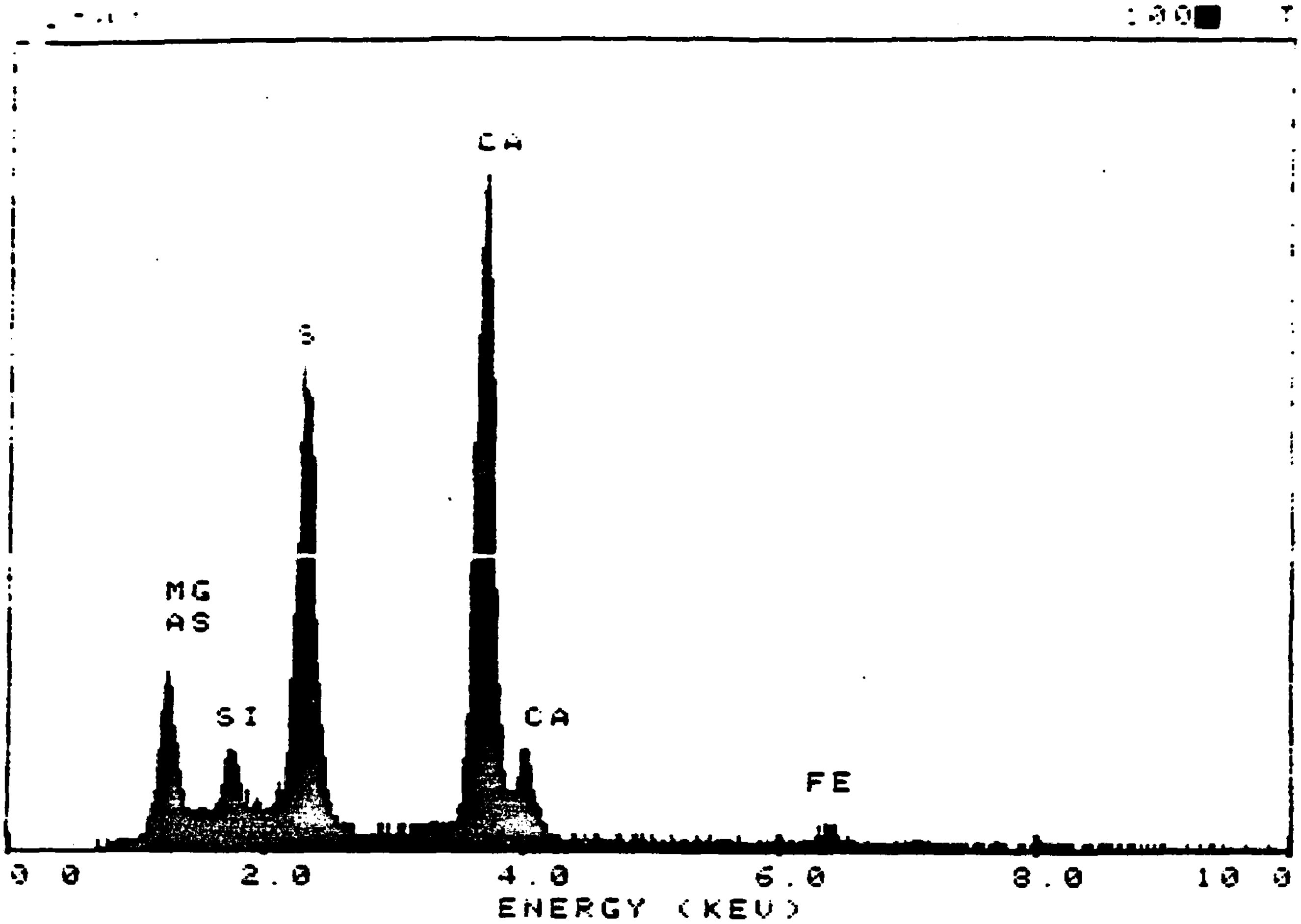
FIGURE 8.5. SEM-EDX SPECTRA FOR SPOT 3 IN SAMPLE LA 0001



```
NOTE: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: [illegible]
 ELEMENT          RELATIVE K              WT %
   CA              0.0788           .    9.[illegible]
   MG              0.0092                2.81
   AL              0.0521           [illegible]
   SI              0.1579            [illegible].73
   S               0.0278            [illegible]
   K               0.0087                1.07
   FE              0.2558               38.30
   AS              0.0142                1.38
 TOTAL                                 100.00
25-JAN-89                            08:47:46
```

24-Jan-89 10:41

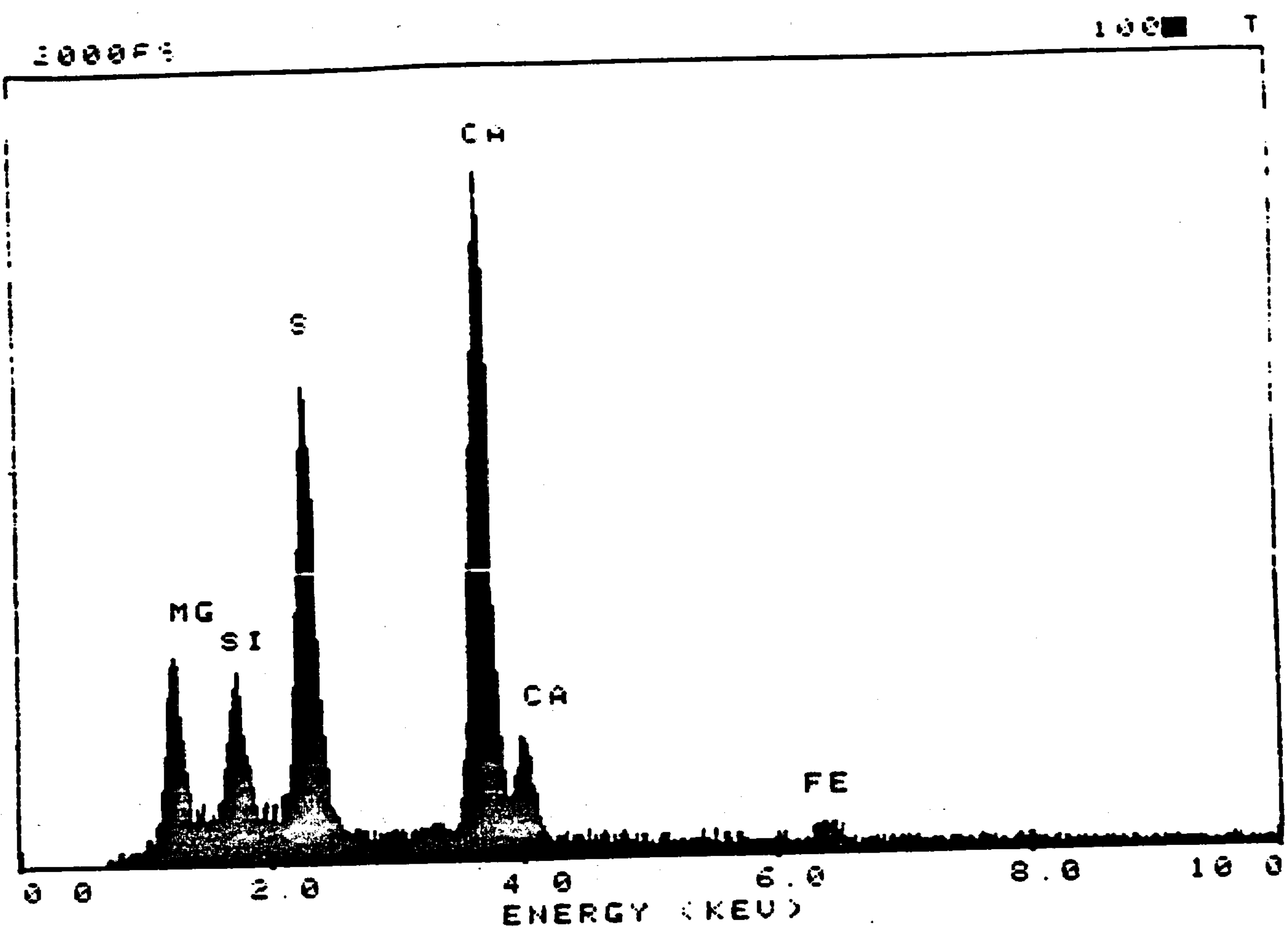
FIGURE 8.6. SEM-EDX SPECTRA FOR SPOT 6 IN SAMPLE LA 0001



24-Jan-89 10:48

```
[illegible] STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: [illegible] TOP GREY
 ELEMENT         RELATIVE K          WT %
   CA             [illegible]        [illegible]
   MG             0.1243             22.6[illegible]
   AL             0.013[illegible]   3.[illegible]8
   SI             0.0417             7.55
   S              0.1879             23.97
   [illegible]    0.0031             0.33
   FE             0.0159             1.87
   AS             0.0133             2.25
 TOTAL                               100.00
25-JAN-89                           08:48:35
```

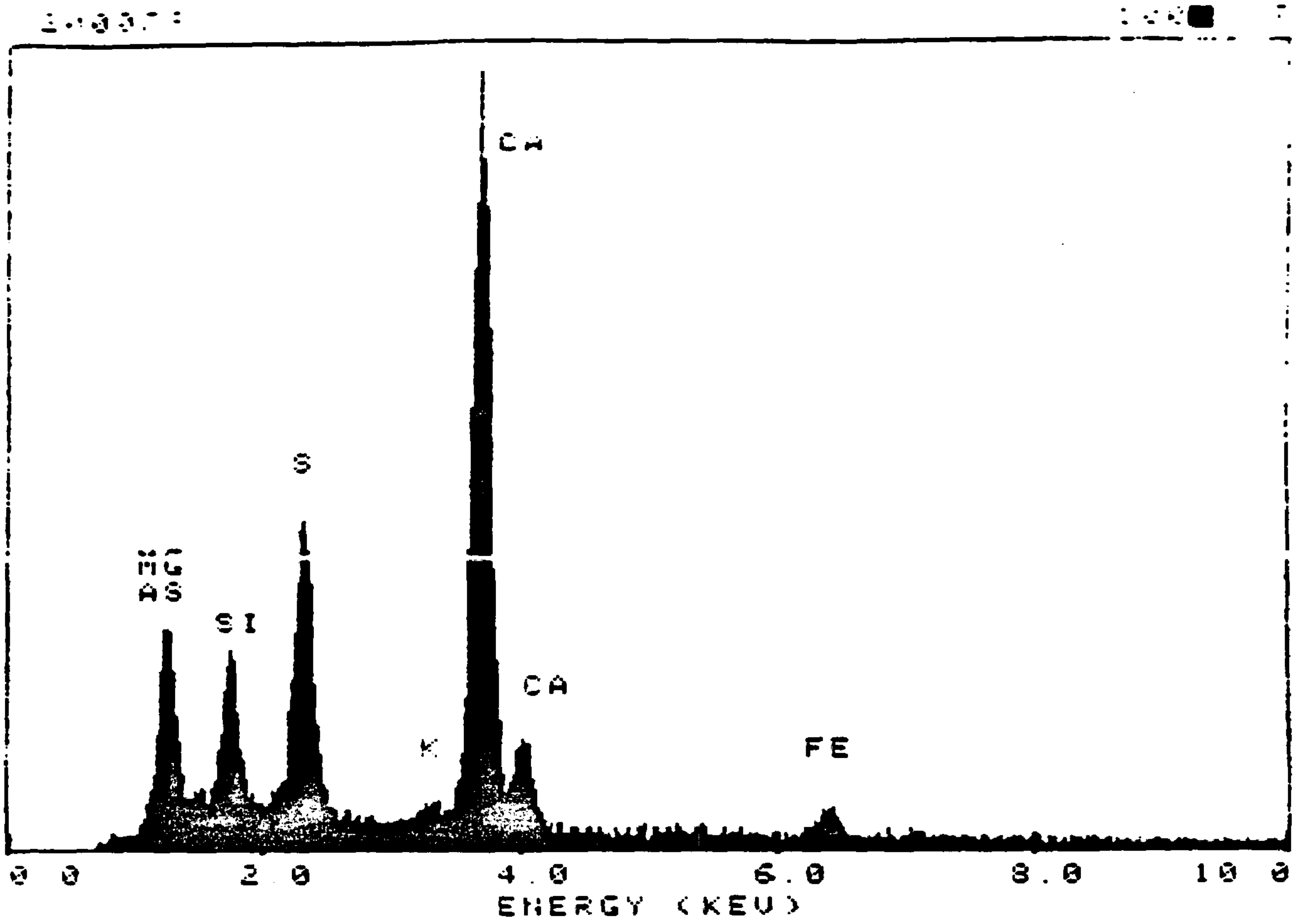
FIGURE 8.7. SEM-EDX SPECTRA FOR SPOT 7 IN SAMPLE LA 0001



24-Jan-89 10:54

```
[illegible]: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: [illegible]  TOP GREY
 ELEMENT       RELATIVE K          WT %
   CA           0.2732           [illegible]
   MG          [illegible]       [illegible]
   AL           0.0142           [illegible]
   SI           0.0776           10.34
   S            0.1676           27.61
   K            0.0037            0.47
   FE           0.0146            1.72
   AS           0.0101            1.21
 TOTAL                          100.00
23-JAN-89                       02:49:22
```

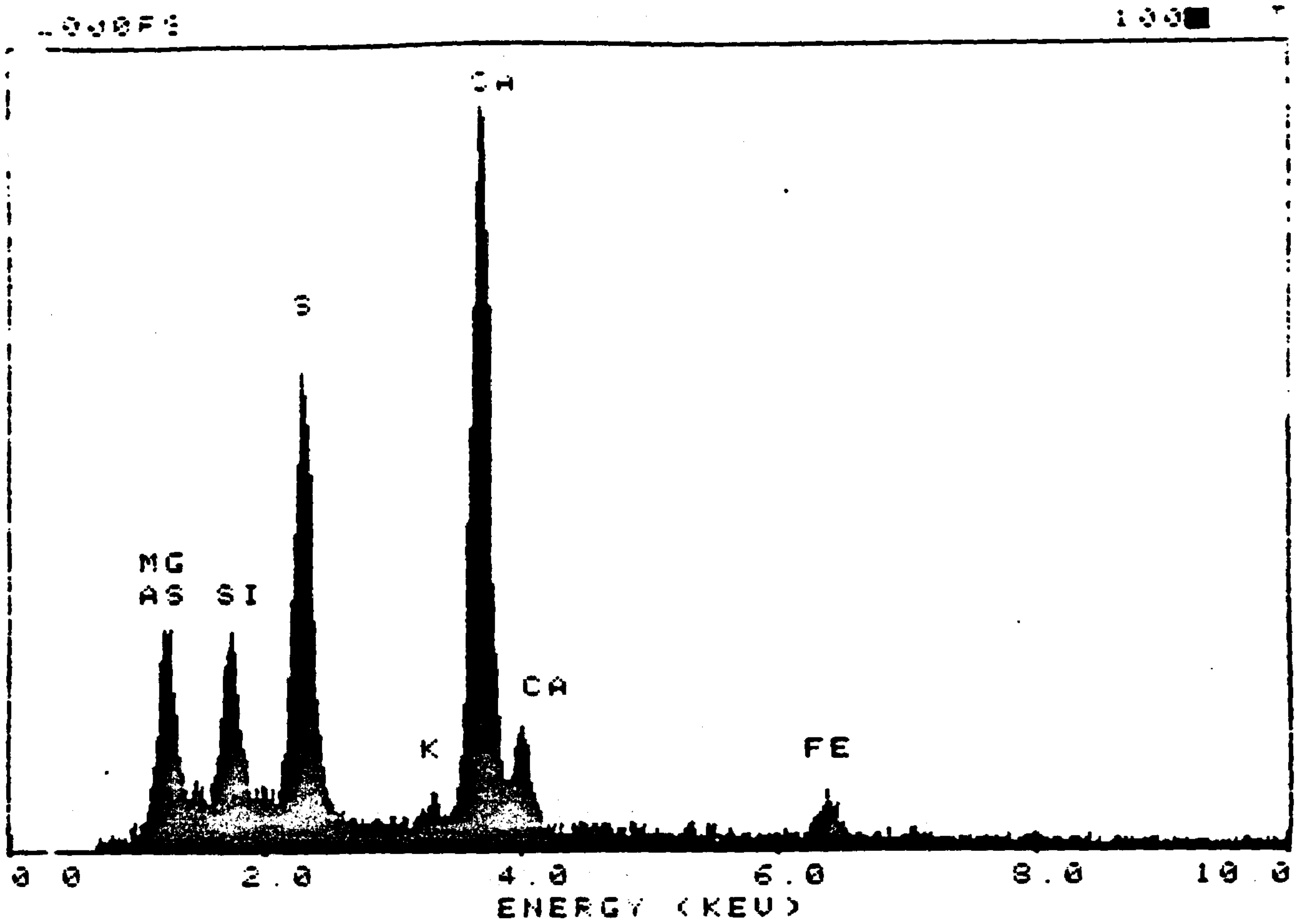
FIGURE 6.8. SEM-EDX SPECTRA FOR SPOT 6 IN SAMPLE LA 0001



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: [illegible] TOP GREY 2
 ELEMENT        RELATIVE K          WT %
    CA           0.2318            33.41
    MG           0.1434            26.22
    AL           0.0219             5.17
    SI           0.0807            15.78
    S            0.1036            15.67
    K            0.0103             1.25
    FE           0.0224             2.63
    AS           0.0046             0.56
   TOTAL                          100.00
25-JAN-89                        08:50:09
```

24-Jan-89 10:59

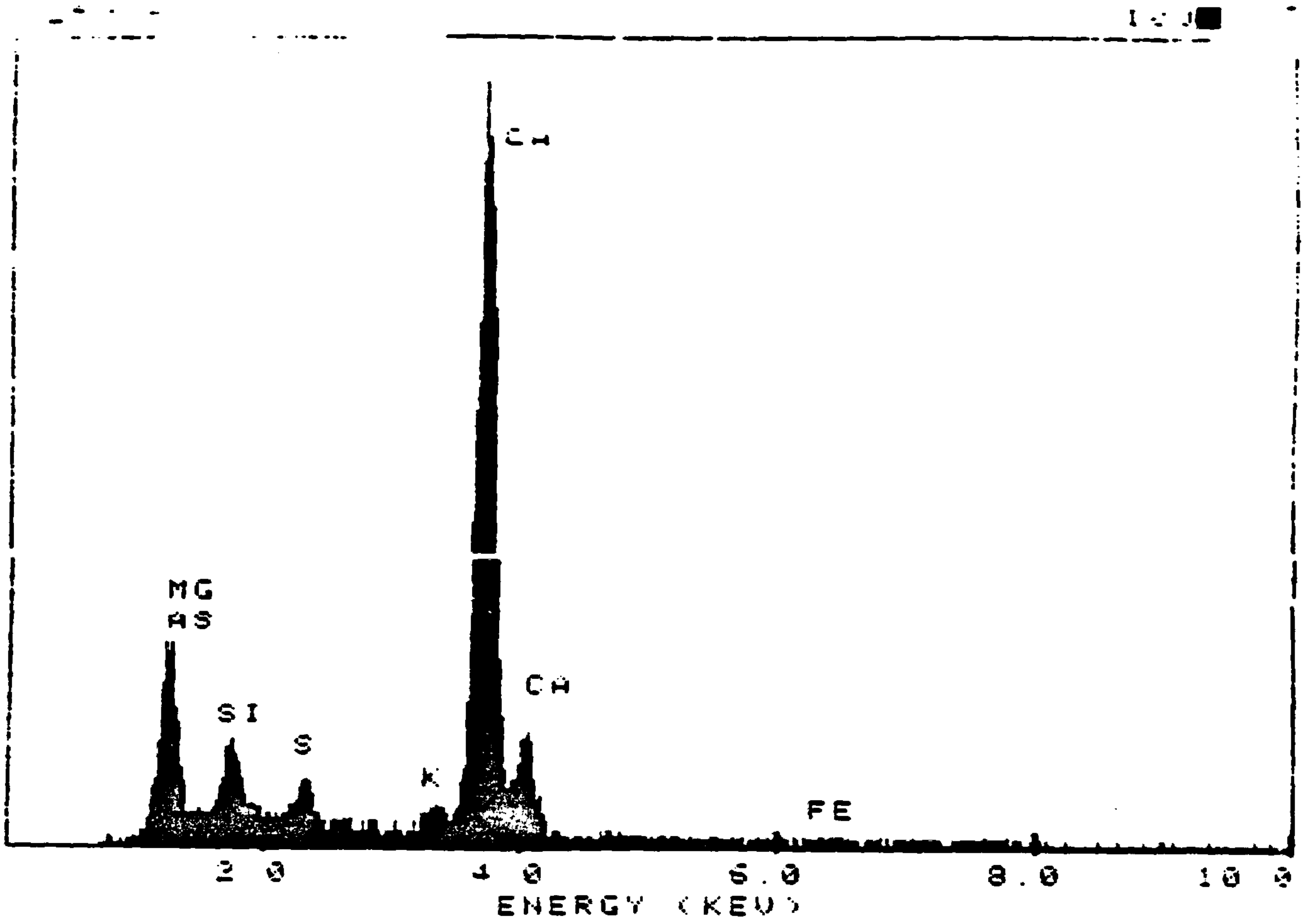
FIGURE 8.9. SEM-EDX SPECTRA FOR SPOT 9 IN SAMPLE LA 0001



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: LA0010 PHOTOGRAPH
 ELEMENT       RELATIVE K          WT %
   CA           0.2558            30.81
   MG           0.1277            22.57
   AL           0.0193             4.49
   SI           0.0798            14.66
   S            0.2424            21.12
   K            0.0066             1.07
   FE           0.0266             3.11
   AS           0.0181             2.17
  TOTAL                          100.00
25-JAN-89                      08:51:40
```

24-Jan-89 11:14

FIGURE 8.10. SEM-EDX SPECTRA FOR SPOT 10 IN SAMPLE LA 0001



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: [illegible] BELOW GREY [illegible]
 ELEMENT        RELATIVE K            WT %
    CA            [illegible]         47.02
    MG            [illegible]         [illegible]
    AL            0.0134              5.[illegible]4
    SI            0.0562              11.[illegible]
    S             0.0230              2.56
                  0.0128              1.42
    FE            0.0062              0.73
    AS            0.0032              0.52
  TOTAL                               100.[illegible]
25-JAN-89                            16:50:53
```

24-Jan-89 11:05

FIGURE 8.11. SEM-EDX SPECTRA FOR SPOT 11 IN SAMPLE LA 0001

LeFever, LeFever and Associates, Inc.
Consulting Environmental & Mining Engineers
2300 Banks • Butte, Montana 59701 • (406) 782-9722

March 27, 1989

Dr. Larry Twidwell
Site Manager
Treatability Study
Montana Tech
Butte, Montana 59701

Dear Larry:

The following is a summary of tasks completed by myself with respect to the treatability studies involving clay pelleting/scintering and cement casting of hazardous waste, Phases I and II.

1. Review and revision of the Health and Safety Plan.
2. Site safety training of the research assistants.
3. Respirator selection and procurement.
4. Industrial hygiene and safety review of work procedures, engineering controls and personal protective equipment.
5. Air monitoring during both phases for potential dust exposure levels.

The Health and Safety Plan was developed by Dr. Terrence Chatwin and reviewed by myself. With minor modifications, primarily clarifications, I approved the plan.

December 15, 1988, I spent several hours covering the Health and Safety Plan with the two technicians, Tom Bowler and Bob Leary, that would be working on the project. Additionally, we discussed toxicity, potential exposure, decontamination and respirators. I have attached a copy of the form signed by both of these individuals after training completion.

In selecting the appropriate respiratory protection for the technicians to wear during their work, MSA Comfo II half-mask respirators with high efficiency particulate air filters were chosen. A Type H HEPA filter was used with the air purifying respirator for protection against potential particulate exposure. Technicians wore the respirators whenever they performed a task that possibly would generate dust. For example, when spooning sample into a mill, scraping mill rods with knives, pouring pulverized material from the mill into pans. Additionally, any task that could be accomplished under a ventilation hood was performed in such a location. This further minimized the potential for technician exposure and particulate liberation into the work environment.

In addition to wearing half-mask respirators, the technicians wore disposable lab coats and disposable gloves. The laboratory was set up such that there was a clean area and a dirty area. The dirty area being where work activities were accomplished. Entry into that part of the lab required wearing the appropriate personal protective clothing. The disposable clothing was removed and placed in an appropriate barrel at the end of work. A new coat issued daily or whenever it was noticed that the material was compromised in some way. Respirator cartridges were replaced daily.

Potential dust exposure levels were monitored using Bendix BDX44 Super Samplers with 37mm diameter, 5um PVC filters. The sampling procedure followed the NIOSH method 0500; a copy of the method is attached. These pumps were calibrated both before and after the sampling periods with a Gilibrator Primary Standard Airflow Calibrator. Filter media were tare weighed prior to sampling, weighed immediately after and the airborne concentrations calculated based on the flow rate and sampling time.

The results of the air samples are as follows:

Date	Individual	Sampling Time (min.)	Airborne Concentration (mg/m3)
12/17/89	T. Bowler	257	2.170
12/17/89	B. Leary	250	0.292
2/18/89	T. Bowler	428	0.976
2/18/89	B. Leary	442	2.170

The above concentrations are well within the OSHA permissible exposure limit of 15 mg/M3 for total dust. Given that the individuals were required to wear MSA Comfo II half face respirators when working with any potentially dusty product, their exposures were well below these numbers. The above numbers represent their potential exposure had they not worn respiratory protection. The numbers also indicate the lead person on the given day. The individual with the higher potential exposure concentration tended to be the lead person, the other supported their activities during that sampling period.

Overall, the research assistants were very conscientious in wearing their protective equipment; following work procedures that minimized dust generation; and performing tasks under laboratory hoods and optimizing the exhaust ventilation system.

Should you need any further information regarding the air sampling or industrial hygiene evaluation, please advise me.

Sincerely,

Julie B. LeFever, CIH
Industrial Hygienist

FIELD TEAM REVIEW

MUST BE SIGNED BY EACH FIELD TEAM MEMBER PRIOR TO THE FIRST SITE VISIT.

I have read and understand the contents of this HASP and will comply to its provisions, requirements, and restrictions.

Site . Whitmoyer Lab

Name (PRINT)	Signature	Date
Tom Bowler	Tom Bowler	12-15-88
Bob Leary	Bob Leary	12-15-88

NUISANCE DUST, TOTAL

DEFINITION: Total aerosol mass

METHOD: 0500
ISSUED: 2/15/84

OSHA: 15 mg/m^3
NIOSH: no standard
ACGIH: 10 mg/m^3, total dust less than 1% quartz

PROPERTIES: quartz less than 1% [1]

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [1] including alumina (CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber; CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

SAMPLING

SAMPLER: FILTER
(tared 37-mm, 5-µm PVC filter)

FLOW RATE: 1.5 to 2 L/min

VOL-MIN: 25 L @ 15 mg/m^3
-MAX: 133 L @ 15 mg/m^3

SHIPMENT: routine

SAMPLE STABILITY: indefinitely

BLANKS: 2 field blanks per 10 samples

BULK SAMPLE: none required

ACCURACY

RANGE STUDIED: 8 to 28 mg/m^3

BIAS: not significant

OVERALL PRECISION (s_r): 0.056 [2]

MEASUREMENT

TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)

ANALYTE: airborne particulate material

BALANCE: 0.01 mg sensitivity or better; use same balance before and after sample collection

CALIBRATION: National Bureau of Standards Class M weights

RANGE: 0.3 to 2 mg per sample

ESTIMATED LOD: 0.2 mg per sample

PRECISION: 0.08 mg per sample [3]

APPLICABILITY: The working range is 3 to 20 mg/m^3 for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [4] in addition to the other ACGIH nuisance dusts [1].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [4].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [4] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Environmental chamber at constant temperature and humidity (e.g., 20 °C ± 0.3 °C and 50% ± 5% RH).
2. Sampler: 37-mm PVC, 2- to 5-µm pore size membrane or equivalent hydrophobic filter and cellulose supporting pad in 37-mm cassette filter holder.
3. Personal sampling pump, 1.5 to 2 L/min, with flexible connecting tubing.
4. Microbalance, capable of weighing to 0.01 mg.
5. Vacuum desiccator.
6. Static neutralizer: e.g., Po-210; replace nine months after the production date.

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
   a. Zero the balance before each weighing.
   b. Handle the filter with forceps (nylon forceps if further analyses will be done).
   c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.
   NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

8. Calibrate each personal sampling pump with a representative sampler in line.
9. Sample at 1.5 to 2 L/min. Do not exceed a total filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

10. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
11. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
12. Remove the cassette band, pry open the cassette and remove the filter. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

13. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Bureau of Standards Class M weights.
14. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

MEASUREMENT:

15. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

CALCULATIONS:

16. Calculate the concentration of total nuisance dust, C (mg/m³), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg/m}^3$$

where: W_1 = tare weight of filter before sampling (mg)
W_2 = post-sampling weight of sample-containing filter (mg)
B = mean change in field blank filter weights between tare and post-sampling (mg) (+ or -).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m³ [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

[1] TLVs - Threshold Limit Values for 1983-84, Appendix D, ACGIH, Cincinnati, OH (1983).
[2] This Manual, Method 5000.
[3] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
[4] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
[5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD WRITTEN BY: Kathy Morring, Jerry Clere, and Frank Hearl, P.E., NIOSH/DRDS.

APPENDIX C

CEMENT CASTING TREATABILITY STUDY

TREATABILITY STUDY

WHITMOYER LABORATORIES SITE
JACKSON TOWNSHIP, PENNSYLVANIA

CEMENT CASTING
FINAL REPORT

SUBMITTED TO:

NUS CORPORATION
Park West 2
Cliff Mine Road
Pittsburgh, Pennsylvania

PREPARED BY:

Terrence D. Chatwin
ENGINEERS INTERNATIONAL
220 South 200 East
Salt Lake City, Utah

With Subcontractor:

Larry G. Twidwell
MONTANA ENVIROMET, INC.
54 Apple Orchard Rd
Butte, Montana

EXECUTIVE SUMMARY

The *objectives* of the cement casting studies were:

- to determine the feasibility of using cement casting technology to reduce the leachability of arsenic in the sludge and solids to a level which would permit the material to be successfully delisted as a hazardous waste and thereby allow for onsite, nonhazardous disposal.
- to collect data to establish preliminary sizing and operating requirements for the design of a full scale remediation program.

These objectives have been accomplished. Cement casting technology has been shown to be applicable to the supplied three waste materials. Two feasible options have been identified for further consideration and potential adoption for site remediation, e.g., a ***Mix and Cast*** option (applicable to the vault material) and a ***Mix, Roast and Cast*** option (applicable to all three materials).

Mix and Cast. The experimental test work has demonstrated that cement/vault mixtures decrease the TCLP arsenic leachability by greater than ninety-five percent. Reduction in arsenic leachability to below the hazardous characteristic arsenic concentration (5 mg/liter) is, however, not achieved by this technology. Also, the lagoon and lagoon/soil materials are not stabilized by mixing and casting with cement. They, in fact, show a greater release of arsenic in the presence of cement than in the absence of cement.

Mix, Roast and Cast. The experimental test work has demonstrated conditions for stabilizing all three of the studied materials, i.e., cement/waste mixtures when roasted in air at 600°C to 700°C are stabilized. The TCLP leachability of the roasted cement/waste mixtures is much less (only a few tenths of a milligram per liter is leached) than the hazardous characteristic arsenic level.

Cost estimates for both the above options have been prepared and show that the remediation cost per ton waste is strongly dependent on a number of different considerations. The cost analysis presented in the following report addresses those considerations that influence the final remediation cost; such as, use of transportable equipment, the option chosen (roast or no-roast), the desired clean-up rate (number of people assigned to the project), and the cement/waste ratio (3/1, 2/1, 1/1).

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SECTION FIVE: REMEDIAL DESIGN AND COST ESTIMATES

SECTION EIGHT: APPENDICES

LIST OF FIGURES

SECTION ONE
STUDY OBJECTIVE

The *objectives* of the cement casting studies were:

- to determine the feasibility of using cement casting technology to reduce the leachability of arsenic in the sludge and solids to a level which would permit the material to be successfully delisted as a hazardous waste and thereby allow for onsite, nonhazardous disposal.

- to collect data to establish preliminary sizing and operating requirements for the design of a full scale remediation program.

The tasks that were performed to accomplish the objectives were divided into two phases of test work. The ***task objectives for the Phase I*** work included:

- Preparation of designated cement/sample mixtures for three supplied samples, i.e., cement/sample ratios of 1/1 and 3/1 for vault, lagoon, and soils materials.

- Roasting a portion of each cement/sample mixture under specified conditions, i.e., one-half of the mixed samples were roasted at 600°C for one hour.

- Casting each cement/sample, roasted and unroasted, into a product of a specified size (to pass a 3/8-inch screen) and aging for specified times, i.e., five days and twenty-eight days.

A screening study was conducted (by EI) prior to Phase II test work in a separate investigation(1). The ***task objective for the screening study*** was to determine the influence of temperature on the possible stablization of unmixed vault, unmixed lagoon, and cement/sample (3/1) materials. The results of that study are also presented in this report for completeness.

The ***task objectives for the Phase II*** work included:

- Preparation of designated cement/sample, cement/lime/sample, cement/thiourea/sample mixtures for each of the three supplied sample materials, i.e.,
    - Cement/sample ratios for the lagoon and soil samples of 1/2, 1/5.

  - Cement/lime/sample ratios for the vault sample of 1/1/1, 0.75/0.6/1.

  - Cement/thiourea/sample ratios for the vault, lagoon and soil samples of 1/0.01/1.

- Roasting one half of each cement/sample and cement/lime/sample mixtures as specified above except for the thiourea containing samples, i.e., one half of the mixed samples were roasted at 700°C for one hour.

- Casting each sample mixture, roasted and unroasted, into a product of a specified size (to pass a 3/8-inch screen) and aging for twenty-eight days.

SECTION TWO
METHODS

2.1. SAMPLE IDENTIFICATION CLASSIFICATION

The sample classification system used throughout this study is presented in Table 2.1.

TABLE 2.1. SAMPLE CLASSIFICATION SYSTEM

Sample	Description	Identification No.
Starting Sample		
WL-LA 0001-1	As-received Lagoon Sample	LA 0001
WL-LA 0002-1	As-received Soil Sample	LA 0002
WL-VT 0002-1	As-received Vault Sample	VT 0002
	Phase I	
Cement/Sample Ratio		
1/1	LA 0001 Mixture Unroasted	LA 0001B,1C
1/1	LA 0001 Mixture Roasted	LA 0001B,1C,R
3/1	LA 0001 Mixture Unroasted	LA 0001B,3C
3/1	LA 0001 Mixture Roasted	LA 0001B,3C,R
1/1	LA 0002 Mixture Unroasted	LA 0002B,1C
1/1	LA 0002 Mixture Roasted	LA 0002B,1C,R
3/1	LA 0002 Mixture Unroasted	LA 0002B,3C
3/1	LA 0002 Mixture Roasted	LA 0002B,3C,R
1/1	VT 0002 Mixture Unroasted	VT 0002B,1C
1/1	VT 0002 Mixture Roasted	VT 0002B,1C,R
3/1	VT 0002 Mixture Unroasted	VT 0002B,3C
3/1	VT 0002 Mixture Roasted	VT 0002B,3C,R
	Screening Study[1]	
As-Received		
Lagoon	LA 0001 Roasted at 600°C	LA 0001,600
	LA 0001 Roasted at 700°C	LA 0001,700

TABLE 2.1. SAMPLE CLASSIFICATION SYSTEM (Continued)

Sample	Description	Identification No.
	LA 0001 Roasted at 800°C	LA 0001,800
	LA 0001 Roasted at 1000°C	LA 0001,1000
Vault	VT 0002 Roasted at 600°C	VT 0002,600
	VT 0002 Roasted at 700°C	VT 0002,700
	VT 0002 Roasted at 800°C	VT 0002,800
	VT 0002 Roasted at 1000°C	VT 0002,1000
Cement/Sample Ratio		
3/1	VT 0002 Roasted at 600°C	VT 0002,3C,600,R
3/1	VT 0002 Roasted at 700°C	VT 0002,3C,700,R
3/1	VT 0002 Roasted at 800°C	VT 0002,3C,800,R
Lime/Sample Ratio		
1/1	VT 0002 Roasted at 700°C	VT 0002,Lime,R
	Phase II	
Cement/Sample Ratio		
1/2	LA 0001 Mixture Unroasted	LA 0001,1:2
1/2	LA 0001 Mixture Roasted	LA 0001,1:2,R
1/5	LA 0001 Mixture Unroasted	LA 0001,1:5
1/5	LA 0001 Mixture Roasted	LA 0001,1:5,R
1/2	LA 0002 Mixture Unroasted	LA 0002,1:2
1/2	LA 0002 Mixture Roasted	LA 0002,1:2,R
1/5	LA 0002 Mixture Unroasted	LA 0002,1:5
1/5	LA 0002 Mixture Roasted	LA 0002,1:5,R
Cement/Lime/Sample Ratio		
1/1/1	VT 0002 Mixture Unroasted	VT 0002,1:1:1
1/1/1	VT 0002 Mixture Roasted	VT 0002,1:1:1,R
0.75/0.6/1	VT 0002 Mixture Unroasted	VT 0002,Lime
0.75/0.6/1	VT 0002 Mixture Roasted	VT 0002,Lime,R

TABLE 2.1. SAMPLE CLASSIFICATION SYSTEM (Continued)

Sample	Description	Identification No.
Cement/Thiourea/Sample Ratio[1]		
1/0.01/1	LA 0001 Mixture Unroasted	LA 0001,Thio
1/0.01/1	LA 0002 Mixture Unroasted	LA 0002,Thio
1/0.01/1	VT 0002 Mixture Unroasted	VT 0002,Thio

[1] Screening study and thiourea conducted as a part of a separate study "Clay Pelletizing and Sintering Final Report" (Reference 1).

2.2. *MATERIALS AND REAGENTS*

The description of the additive mixing materials and reagents are presented in Table 2.2.

TABLE 2.2. MATERIALS AND REAGENTS

Material	Description and Source
Mixing Materials	
Cement	Portland, Type I&II Central Pre-Mix Concrete Company
Lime	Hydrated Lime, Type S Triple S, Inc.
Reagents	
Thiourea	J.T. Baker Reagent
Sodium Hydroxide	J.T. Baker Reagent
ICP Standard	J.T. Baker Instra-Analyzed, 1000 mg Arsenic/liter
Deionized Water	Millipore De-Ion Ion Exchanger

SECTION THREE
PROCEDURES

The procedures and equipment used in both the Phase I and Phase II studies are described in this section.

3.1. *HANDLING OF AS-RECEIVED SAMPLES*

The three samples were received at the Montana Tech facilities in five gallon pails (Table 3.1).

TABLE 3.1. SAMPLE CUSTODY

Sample	Identification No.	Sample Wt. Pounds	Date of Receipt
Lagoon	WL-LA0001-1	54.2	Nov. 29, 1988
Soil	WL-LA0002-1	54.8	Nov. 29, 1988
Vault	WL-VT0002-1	51.1	Dec. 1 , 1988

These samples were stored in a locked cabinet in Fume Hood #6, Room 211, Engineering Laboratory Classroom Building.

Each sample container was opened as needed, spread out in pans, and air dried in fume hoods for 12-18 hours.

3.2. *PHASE I AND PHASE II TEST PROCEDURES*

3.2.1 *Sample Mixing*

The starting material chunks were weighed and individually placed in a 8-inch by 10-inch rod mill containing five 1/2-inch rods. The rod mill was rolled for ten minutes to break up the chunks. The resulting material was a fine powder. The rod mill plus sample was tared and the desired quantity of cement was added. The mixture was then rolled for 15 to 30 minutes to achieve mixing. The sample was then recovered by opening the mill under a hood; placed into plastic sample bags; then stored until the operations of either roasting or casting were performed.

The rod mills were cleaned between each use by filling the mill approximately one-third full of clean sand and rolling for ten minutes. The weighing operation was performed on a Fairbanks scale with accuracy of plus or minus 0.1 pounds; that is for a five-pound sample the weight would be within plus or minus two percent.

3.2.2. *Small Scale Roasting Test Work*

Each Phase I mixture that was to be roasted on the large scale was first evaluated in a small tube furnace apparatus. The apparatus consisted of a 20-mm diameter vycor tube in a hinged tube furnace. A small sample of the mixture (from 0.5 to 3.0 grams) was placed in a fireclay boat which was then positioned in the center of the tube furnace (already at temperature; 600°C). The entrance end of the tube was closed by a rubber stopper containing an entrance tube for air inlet. Air was gently blown over the surface of the sample and was flushed through tygon tubing into a fritted glass gas dispersing tube into a caustic scrub solution. The scrub solution consisted of 450 milliliters of 400 gpl sodium hydroxide.

After the test period was complete the sample and boat were recovered from the system, cooled in air, and weighed. The scrub solution was used to clean the vycor tubing of any deposit present. The dissolved material collected in the cleaning solution was added back to the scrub solution. The scrub solution was analyzed for arsenic (by ICP analysis at Montana Tech) and total dissolved carbon (by Institute for Process Analysis at Montana State University, Bozeman, Montana).

3.2.3. *Roasting*

Roasting was performed in 4-inch by 10-inch by 1.5-inch sillmonite roasting trays. The trays were loaded to about three-fourths capacity and placed in a muffle furnace. The muffle furnace was calibrated to determine the temperature at the sample location. The temperature profiles for most of the Screening Test Study samples were determined (Phase I test work did not include the determination of each temperature profile). Weight loss data for all the Screening Test Study and Phase II roast studies were collected by roasting the tray at 700°C for one-half hour; cooling and recording the tare weight; weighing the sample plus tray; roasting; cooling and recording the final weight. Weight loss data for the Phase I tests were not collected (except for one test).

Observations on evidence of fuming was recorded for all the mixture roasts. The roasted material was cooled in air and placed in double plastic bags for storage until the material could be cast into cubes.

3.2.4. *Casting and Aging*

Prior test work established that about 38-44 weight percent (of the total solid weight) water was necessary to get the proper fluidity for casting. All samples were mixed (with the correct amount of water) and cast into 3/8-inch cubes. The cubes were allowed to set for approximately 4-24 hours in the casting grid. The samples were then punched out of the grid and sectioned into approximately four equal pieces. These pieces were then placed in a humidifing chamber at 70°C and aged for the specified periods of five or twenty eight days.

Cylinders of cast sample were also produced. The samples were cast into five inch sections of PVC pipe (2-3/8-inch diameter; 3/16-inch wall thichness) and aged the same as the cast cubes.

3.3. *SCREENING TEST WORK*

Prior to conducting the Phase II test program a series of tests were performed to provide data for deciding the proper course for the Phase II program. The detailed results of this work are reported in a separate report, Clay Pelletizing and Sintering (1). The results of that study are important to this study and, therefore, the procedures used and the results obtained will be included in this report.

Three roasting studies were conducted on a smaller scale than the Phase I and Phase II studies in order to establish the importance of the Ca/As or Fe/As ratios and temperature in stabilizing arsenic in the waste materials. The sample sizes in the screening studies were 100 grams rather than five-ten pounds as was the case in the Phase I and Phase II test work.

3.3.1. *Influence of Temperature on Stabilizing Waste Materials VT 0002, LA 0001, Cement/VT 0002 Ratio of 3/1, and Lime/VT 0002 Ratio of 1/1*

The influence of temperature on potentially stabilizing the waste materials by simple roasting was studied by the following experimental procedure:

- A starting sample of 500 grams was split from the as-received air dried materials (for both VT 0002 and LA 0001). This sample was split into five 100 gram subsamples. Each sample was treated at a different temperature, i.e., no roast, 600°C, 700°C, 800°C, and 1000°C. A roast time of one hour, twenty minutes was used to ensure that the sample was at the required temperature for one hour (it took approximately twenty minutes for the mass of the sample to reach the desired temperature). Temperature profile data were collected for each roast test.

- Careful weight change data were collected for each roast test.

  The sample was weighed and placed in a previously fired and weighed 100 cc fireclay crucible. The crucible was placed in a muffle furnace that was already at the roasting temperature. The crucible had a 3/8-inch hole drilled into it 1/2-inch below the top. A thermocouple was placed through the furnace door peep hole into the hole in the crucible. This located the thermocouple at the sample surface. The temperature was measured and recorded as a function of time to provide a temperature profile of each sample. Each sample was held at the roasting temperature for one hour. The total elapsed time for most samples was about one hour and twenty minutes.

  After the sample was roasted for the required time at the appropriate temperature it was removed, air cooled, then weighed.

  Observations of the sample crucible surface were recorded for each sample.

- Roasts also were performed and weight change data collected for cement samples and for lime samples.

3.3.2. *Response Measurement*

The response measured to determine whether the roast procedure was successful was to conduct the Toxicity Characteristic Leaching Procedure (TCLP) on the roasted powders. The TCLP test was modified as follows:

- Ten grams of sample was used instead of 100 grams.

- The agitation was performed using a wrist shaker instead of an end-over-end rotator.

- Arsenic was the only element determined.

The TCLP test procedure used in this study is described below:

- Determination of the correct extraction fluid

  - A five gram sample was placed in a 500 cc beaker. Deionized water was added (96.5 cc). The mixture was stirred for five minutes. Solution pH was measured. If the pH was less than 5.0, Extraction Fluid One was used (5.7 cc glacial acetic acid added to 500 cc of deionized water and 64.3 cc of 1 N sodium hydroxide was added). This mixture was diluted to one liter. Solution pH was 4.93.

If the pH was greater than 5.0, then 3.5 cc of 1 N HCl was added, the solution heated to 50°C for 10 minutes, and pH measured. If the pH was less than 5.0, Extraction Fluid One was used. If the pH was greater than 5.0, Extraction Fluid Two was used (5.7 cc glacial acetic acid diluted to one liter; pH 2.88).

Most of the TCLP test work conducted in this study required Extraction Fluid Two.

- TCLP Experimental Test
  - Ten grams of each sample was placed in a 250 cc polyethylene bottle. Two hundred cc of the correct Extraction Fluid was added.
  - Each bottle was agitated in a wrist shaker set so that all the solids were in constant movement for eighteen hours.
  - An aqueous sample was withdrawn from the bottle using a syringe filter (0.45 micrometers) for ICP analysis.

3.4. *ANALYTICAL PROCEDURES*

3.4.1. *Moisture Content*

The moisture content of individual samples was determined by weight loss measurements. Samples (60-80 grams) were weighed before and after drying at 105°C for one hour. In most cases triplicate samples were treated. Sample weights were measured on a Mettler PB 300 two decimal place electronic balance.

3.4.2. *Arsenic Determination*

The arsenic content of the starting and product materials were determined by using the EPA Digestion Procedure (Method 3050: Acid Digestion of Sediments, Sludges and Soils) and a Modified Digestion Procedure. The reason for testing a modified procedure was that the EPA Digestion Procedure (when applied to cement containing samples) produced silica gel in the digested product; the Modified Digestion Procedure did not produce silica gel.

Both of the procedures were applied to all Phase I and Phase II products. The results of the test work, i.e., comparsions of results for the starting sample materials and the product materials, are presented in Tables 4.2 and 4.7. A description of the Modified Digestion Procedure · presented below:

- Samples were ground to -60 mesh. Triplicate samples of 0.25 grams each were weighed into tared 100 milliliter polyethylene bottles. Twelve milliliters of HF, two milliliters of HNO_3, and one milliliter of $HClO_4$ were added to the solid.
- Solids were digested in the bottles for twelve hours at $65^{\circ}C$.
- After cooling to room temperature, each bottle solution was diluted to 100 grams.
- A blank and standard solution were included in the digestion.
- If a residue was present, it was allowed to settle and the solution was aspirated directly into the ICP. Solution concentration of the standard was used to determine the solution concentration of the individual sample and these results were used to calculate the percentage arsenic in the solids.

3.5. *METALLOGRAPHY AND SEM-EDX*

Several of the Phase I samples were recorded for later study by standard metallography procedures, i.e., samples were mounted in room temperature setting epoxy, ground through - 600 grit papers, and polished through 5 micrometer activated alumina on a billiard cloth. These samples were photographed at several magnifications to allow for the study of structure characteristics of the product materials.

Scanning Electron Microscopy and Energy Dispersive X-ray Analyses (SEM-EDX) were performed on only one of the Phase I product materials because of the time and expense of surveying all the samples. The procedure used was as follows: one of the samples prepared for metallography was chosen to be studied for arsenic phase identification, arsenic content and arsenic distribution. The sample was carbon coated in a vacuum, placed in the SEM microscope and studied by both SEM surface scanning and EDX analysis of several of the component phases (by controlling beam placement) by emission spectra analysis.

SECTION FOUR
RESULTS AND DISCUSSION

4.1. *BACKGROUND*

The technology to be evaluated by this study was cement casting. The purpose of the cement casting was to prepare samples for an investigation of the physical and chemical binding of arsenic. Three test materials were considered, i.e., a material referred to as a vault material (VT 0002) that contained an appreciable amount of arsenic, primarily as calcium arsenate (and, perhaps, as calcium arsenite); a material referred to as a lagoon material (LA 0001), containing arsenic as ferric arsenate; and a material referred to as a lagoon-soil material (LA 0002), containing arsenic as ferric arsenate.

The technology to be tested was developed by Twidwell and Mehta in early 1980 (2-4). The technology they developed was for the treatment of copper smelter flue dust which, of course, was a different material than the materials considered in the present study. Their work, however, served as a guide to the selection of the conditions to be considered in the Phase I study. The investigators showed that the best results (to water leaching) were obtained for cement/sample mixtures that were roasted prior to casting. Therefore, the two major variables studied included cement/sample ratio and roasting or not roasting prior to casting.

4.2. *AS-RECEIVED SAMPLE CHARACTERIZATION*

Observation comments concerning the physical appearance of the as-received materials are presented in Table 4.1. Moisture and arsenic contents are reported in Table 4.2. Spectra and semi-quantitative composition data for the air dried materials are presented in Figures 4.1 - 4.4.

TABLE 4.1. APPEARANCE OF AS-RECEIVED SAMPLES

Sample	Observation
LA 0001	Reddish brown semi-dry chunky material varying in size from 1/8-inch to several inches. Could be handling like a dry material. Chunks easily broken into smaller pieces. Odorless. Clay-like material. Some rocks present.
LA 0002	Essentially the same in appearance as sample LA 0001 except there appeared to be more rocks present (approximately 12% of sample weight). Rocks appeared to range in size up to about 3/4-inch but most less than 3/8-inch.
VT 0002	When the sample container was opened there was a layer of oil-like liquid on top of the solid material. Photographs are available in the accompanying data record books. The oil-like material readily absorbed into the solid material when the solid was stirred. The solid material appeared to consist of several solids, a dark material and a yellowish-white material. The solid material was one mass rather than distinct chunks. The material emitted a definite organic-like odor.

TABLE 4.2. AS-RECEIVED SAMPLE MOISTURE AND ARSENIC CONTENT

Sample	Moisture Content, %		Arsenic Content, %[2]	
	As-Rec.	Air Dried[1]	ICP	SEM-EDX
LA 0001	20.5 ± 0.5	2.7 ± 0.5	0.29, 1.14, 1.37, 1.14 1.32, 1.60[3] 1.13[3], 1.04[3]	2.19
LA 0002	33.1 ± 1.0	3.5 ± 0.2	2.88, 2.46 1.96[3], 1.84[3] 1.63[3]	2.40
VT 0002	30.7 ± 0.6	7.4 ± 0.2	15.75, 14.26 18.07, 18.72 19.14, 19.38[3] 17.81[3], 19.76[3]	19.02

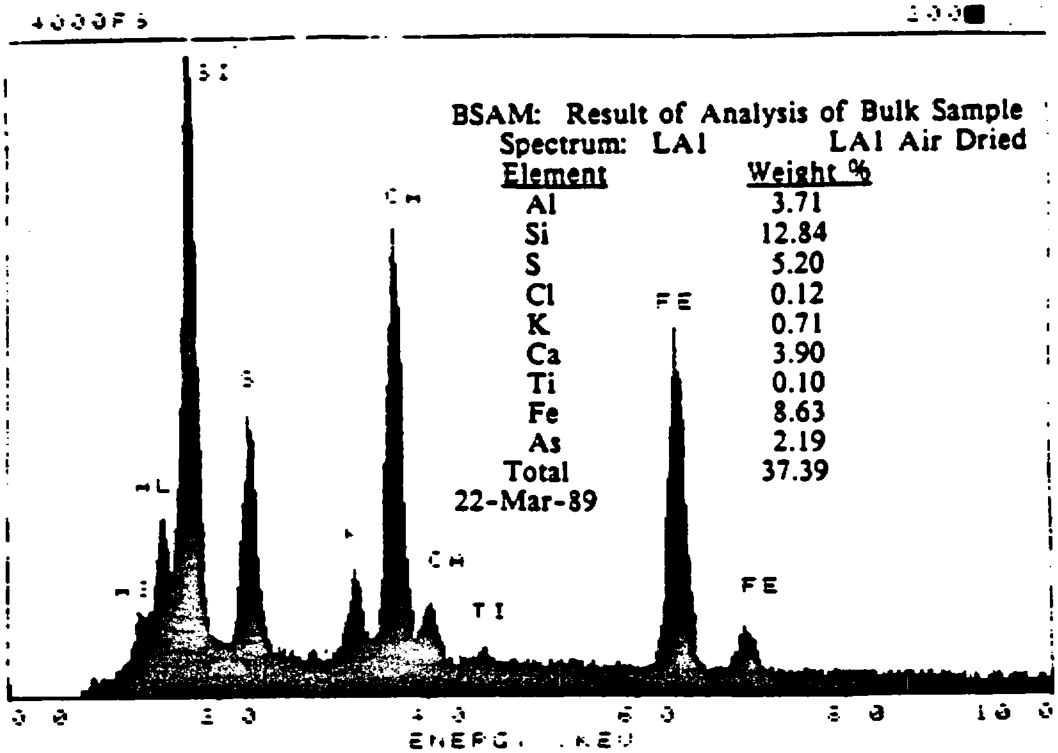
[1] Moisture content of samples dried at room temperature in a hood for 18 hours.

[2] Arsenic content is for the air dried material (EI determinations). SEM-EDX results are semi-quantitative.

[3] Marked results were determined by modified digestion procedure.

4.3. *PHASE I STUDIES*

Samples were received at Montana Tech in early December, 1988. See Section 2.1, Sample Classification System for the numbering system used in the following discussion; Section 3.1, Handling of As-Received Sample Materials for a description of the as-received sample materials; and Section 4.2, As-Received Sample Characterization. Test work was initiated (See Section 3.2, Phase I and II Test Procedures) on the Phase I test objectives (See Section 1, Objectives) the middle of December.

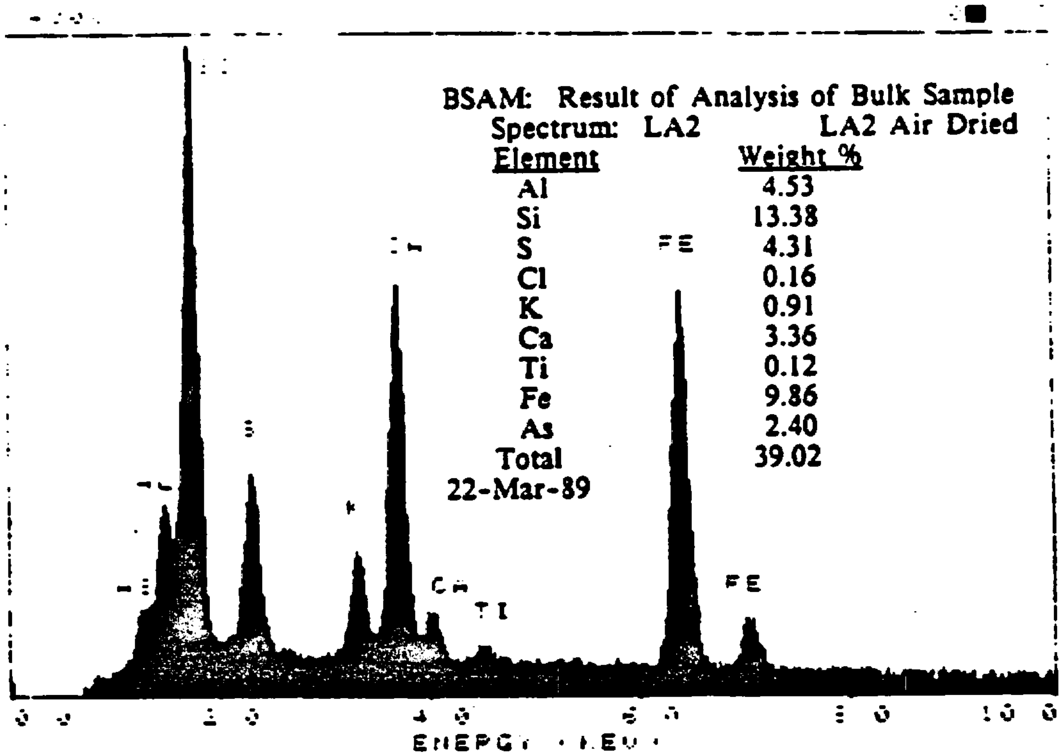


BSAM: Result of Analysis of Bulk Sample
Spectrum: LA1 LA1 Air Dried

Element	Weight %
Al	3.71
Si	12.84
S	5.20
Cl	0.12
K	0.71
Ca	3.90
Ti	0.10
Fe	8.63
As	2.19
Total	37.39

22-Mar-89

FIGURE 4.1. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED LA 0001 SAMPLE

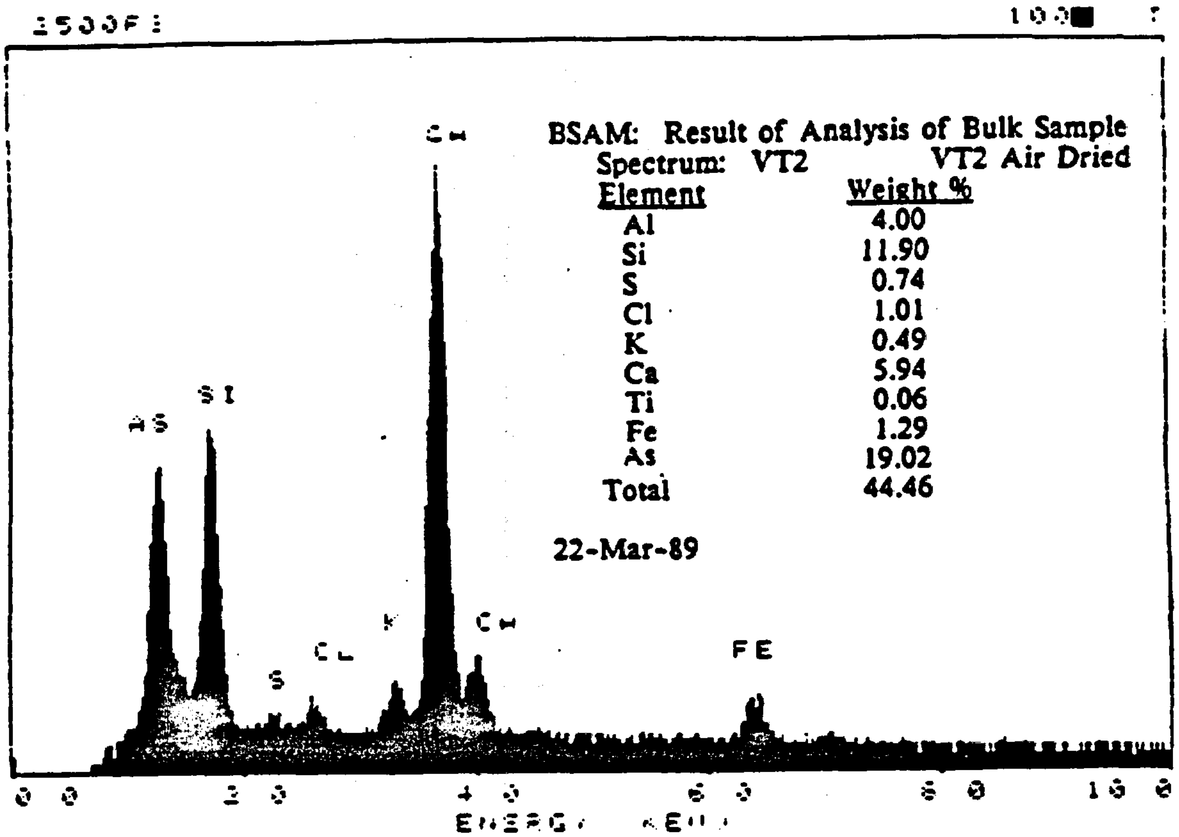


BSAM: Result of Analysis of Bulk Sample
Spectrum: LA2 LA2 Air Dried

Element	Weight %
Al	4.53
Si	13.38
S	4.31
Cl	0.16
K	0.91
Ca	3.36
Ti	0.12
Fe	9.86
As	2.40
Total	39.02

22-Mar-89

FIGURE 4.2. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED LA 0002 SAMPLE



BSAM: Result of Analysis of Bulk Sample
Spectrum: VT2 VT2 Air Dried

Element	Weight %
Al	4.00
Si	11.90
S	0.74
Cl	1.01
K	0.49
Ca	5.94
Ti	0.06
Fe	1.29
As	19.02
Total	44.46

22-Mar-89

FIGURE 4.3. SEM-EDX SPECTRA FOR AIR DRIED AS-RECEIVED VT 0002 SAMPLE

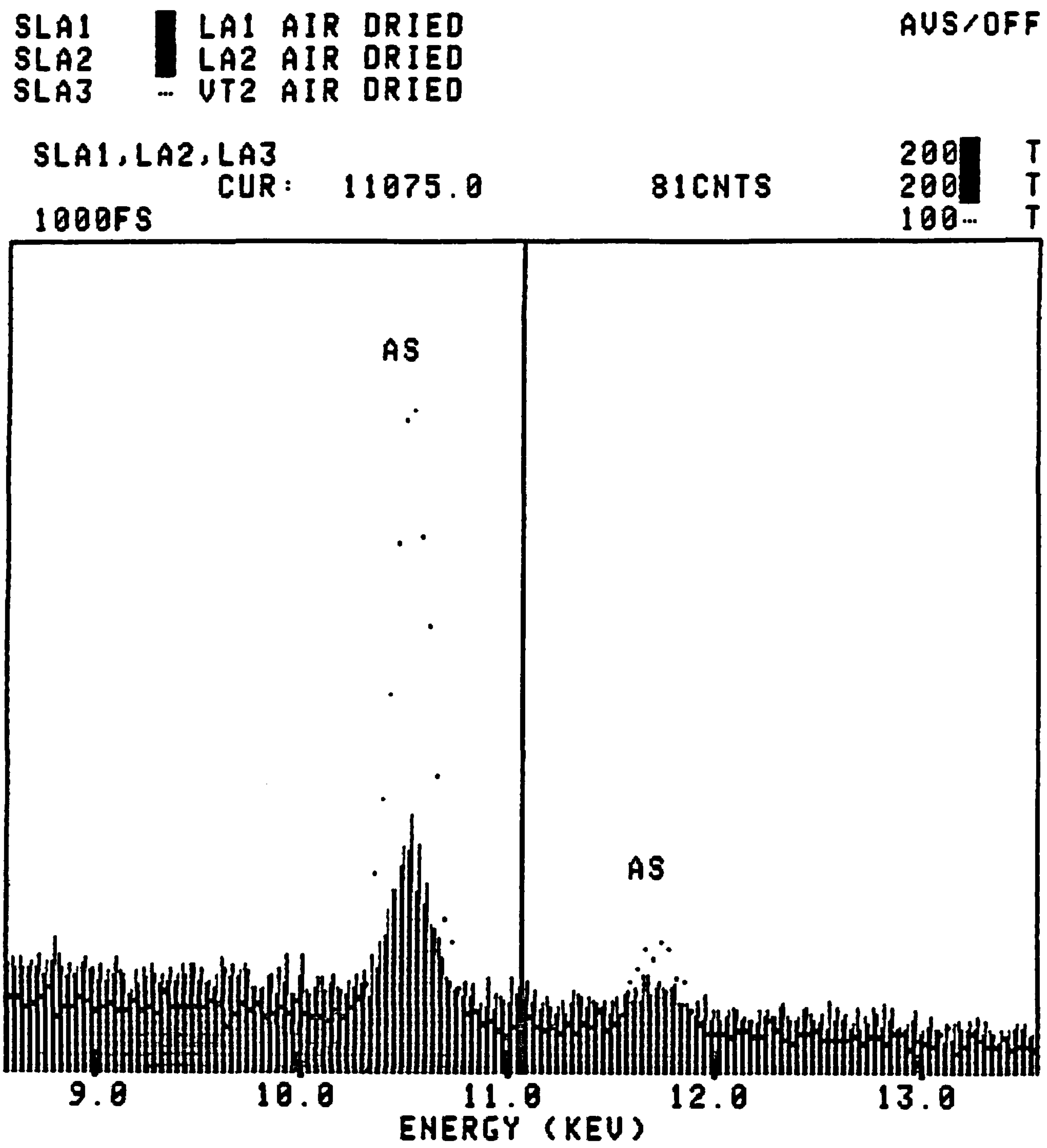


FIGURE 4 4. SEM-EDX COMPARATIVE SPECTRA OVERLAY OF ARSENIC PEAK FOR THE THREE STARTING MATERIALS

The treatment sequence for each sample is depicted in Figure 4.5. The sequence consisted of: mixing the sample and cement; roasting a portion of the mix in air at 600°C for one hour; casting both the unroasted mix material and the roasted mix material; aging the cast material at 20°C (70°F) in 100% relative humidity; withdrawing samples at five days and at twenty eight days for shipment to NUS.

4.3.1. *Health and Safety Considerations*

A health and safety report is attached as Appendix Section 8.1. All personnel involved in handling the materials wore protective clothing and gloves at all times and wore respirators when handling the samples and sample mixes. Two of the laboratory assistants wore personal air monitors during the first day of each phase study. The results from the air monitoring studies showed that the working conditions were safe from a health point of view and the air-bourne arsenic in the working environment was negligible.

4.3.2. *Sample Mixing*

Two sample mix ratios for each of the supplied three samples were prepared. The amounts of each mix material initially prepared are summarized in Table 4.3.

4.2.3. *Sample Roasting*

Small Scale Tube Roasts

Small scale (0.5-3.5 grams) tube furnace roast tests were performed on samples cut from each mixture prior to the large scale sample preparation test work. The procedure was described previously. The results are summarized in Table 4.4.

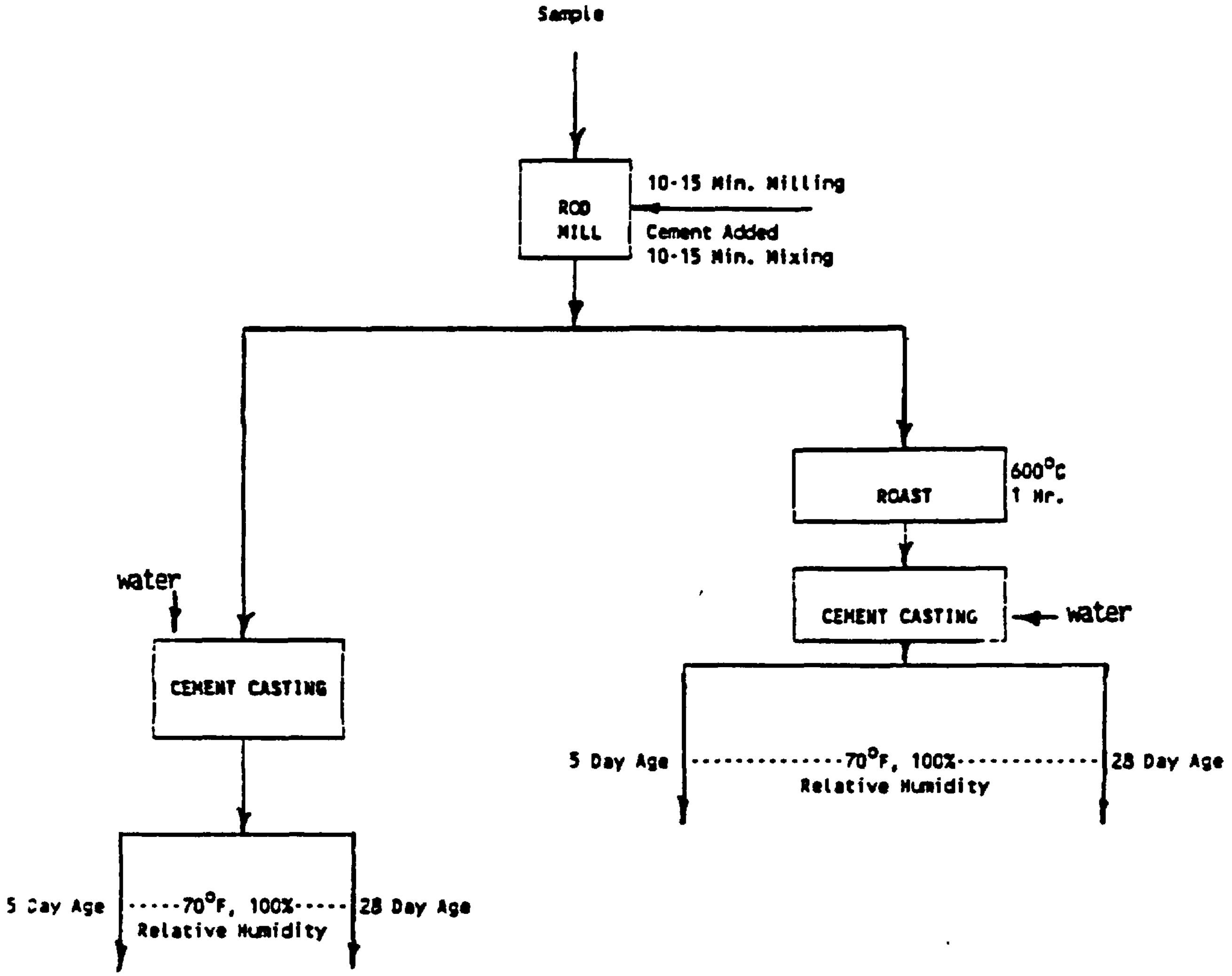


FIGURE 4.5. TREATMENT SEQUENCE FOR PHASE I STUDY

TABLE 4.3. PHASE I SAMPLE MIXTURES

Sample	Mix Ratio, Cem./Spl.	Quantity Prepared, Grams
LA 0001B,1C	1/1	4,585
LA 0001B,1C,R	1/1	4,400
LA 0001B,1C	1/1	3,180
LA 0001B,3C	3/1	4,680
LA 0001B,3C,R	3/1	4,220
LA 0002B,1C	1/1	3,950
LA 0002B,1C,R	1/1	4,450
LA 0002B,3C	3/1	3,590
LA 0002B,3C,R	3/1	4,040
VT 0002B,1C	1/1	4,360
VT 0002B,1C,R	1/1	4,540
VT 0002B,3C	3/1	4,585
VT 0002B,3C,R	3/1	4,540

Note: Only a portion of the some of the above mixes were either cast or roasted and cast in order to ensure that there would be sufficient sample for the Phase II test work.

TABLE 4.4. ARSENIC AND ORGANIC CARBON RELEASE FROM SMALL SCALE ROAST TESTS

Sample	Arsenic Release		Total Organic Carbon Release	
	gm As/gm sample	%As[1]	gm C/gm sample	%C[2]
LA 0001B,1C	0.0077	136.5	0.0081	-
LA 0001B,3C	0.0012	42.6	0.0030	-
LA 0002B,1C	0.0008	7.4	0.0031	-
LA 0002B,3C	0.0005	9.3	0.0035	-
VT 0002B,1C	0.0025	2.8	0.0077	-
VT 0002B,3C	0.0030	6.7	0.0049	-

[1] The arsenic content of starting sample was not experimentally determined. Therefore, the starting arsenic concentration was estimated by using the average arsenic content (calculated from Table 4.2) corrected for the amount of cement added. For example, Sample LA 0002B,1C: the average arsenic content for unmixed LA 0002 (Table 4.2) was 2.15%. The arsenic content corrected for the presence of cement is 1.08%. The arsenic released = 0.0008/0.0108, i.e., 7.4%.

[2] The total organic carbon content of the starting sample mixtures was not experimentally determined. The starting organic carbon concentration could not be estimated as stated above for the arsenic content because the data for the unmixed samples were suspect and unvalidated. Therefore, the percent organic carbon volatilized is not reported.

The arsenic release data for sample LA 0001B,1C is, of course, not representative of the amount of arsenic that would be released from a bulk material of that mixture. The calculated 136% arsenic release may have been the result of performing the tube roast on a portion of the sample that was not representative of the bulk analysis, i.e., the particular sample roasted (only 0.763 grams) may have been much higher in arsenic content. The distribution of arsenic (in the low arsenic bearing LA1 material) is known to be very spotty. For example, refer to Figure 4.11 and Table 4.16. The bulk arsenic content (semi-quantitative value) was 2.8%. Small spots were identified to contain up to 9.9% arsenic (there may, in fact, be arsenic contents up to 38.5% arsenic i the arsenic is present as ferric arsenate). The LA 0001,1C cement/sample mix was, according to the SEM-EDX results, heterogeneous with respect to arsenic distribution.

Large Scale Sample Roasts

Large scale sample roasts were conducted as described previously. Quantities of materials roasted and observations concerning the roast are presented in Table 4.5. All samples in the Phase I test work were roasted at 600°C for one hour.

TABLE 4.5. PHASE I ROAST OBSERVATIONS

Sample	Quantity, Grams	Observations
LA 0001B,1C,R	4270	No visual change in color. No fuming evident. Only 0.5% weight loss.
LA 0001B,3C,R	4220	No visual change in color. No fuming evident. No agglomeration of solid material.
LA 0002B,1C,R	2270	Slight darking of color. No fuming evident. No agglomeration of solid material.
LA 0002B,3C,R	2270	Same as LA 0002B,1C,R.
VT 0002B,1C,R	2270	No visual change in color. No obvious fuming. Roasting conducted in a different furnace than above samples. Conditions may have been more oxidizing. If fuming occurred it was not obvious. Any fume would have been exposed to the heating elements.
VT 0002,3C,R	2270	Same as VT 0002B,1C,R.

4.3.4. *Sample Casting, Aging, and Shipment*

Each of the samples were mixed with water and cast into 3/8-inch by 5/16-inch molds. A series of preliminary tests were conducted (on the LA 0001B,3C,R sample) to determine the approximate requirement for water addition. The results showed that in order to get a good flowing casting material, about 38 to 44 percent water (by weight) was required. The cast material formed good product cubes which were easily removed from the mold and which were stable against crumbing during the removal from the mold.

The procedure adopted for the casting and aging stage of sample preparation included the following steps: approximately two to four pounds (exact amount weighed) of each starting sample was mixed with 35 percent water; additional water was added to give good casting properties to the individual sample (water weighed and recorded); the sample was cast into the grid molds and allowed to set for from 4 to 8 hours; the sample was punched from the grid and sectioned into quarter pieces; the sectioned material was then aged at 22°C (70°F) for 5 and 28 days; one-half pound samples were withdrawn at five days and returned to NUS for a variety of additional test evaluations (including TCLP evaluations); and approximately two pound samples were recovered and shipped after 28 days.

A summary of the casting and aging process information is presented in Table 4.6.

TABLE 4.6. PHASE I CASTING, AGING AND SHIPPING

Sample	Weight, gms	Water, gms	Sample Shipped, gms		
			5 Days	28 Days	
				Cubes	Cylinders
LA 0001B,1C	2000	600	249	950	425
LA 0001B,1C,R	2000	610	281	1000	489
LA 0001B,3C	2000	660	236	1100	465
LA 0001B,3C,R	2000	600	267	1200	490
LA 0002B,1C	2000	560	222	900	423
LA 0002B,1C,R	2000	675	288	900	482
LA 0002B,3C	2000	675	249	800	463
LA 0002B,3C,R	2000	770	250	950	484
VT 0002B,1C	2000	690	228	1000	397
VT 0002B,1C,R	1800	870	312	1000	395
VT 0002B,3C	2000	690	259	900	434
VT 0002B,3C,R	1800	810	238	900	425

The arsenic contents in the final shipped products are presented in Table 4.7.

TABLE 4.7. ARSENIC CONTENT IN PHASE I PRODUCTS

Sample	Arsenic Content, %[1]	
	EPA 3050	Modified Procedure
	LA 0001	
LA 0001, Air Dried	0.29, 1.14	1.60, 1.13, 1.04
LA 0001B,1C	0.50, 0.69	0.96, 0.93, 0.80
LA 0001B,1C,R	0.96	1.02, 0.93, 0.89
LA 0001B,3C	0.30	0.34, 0.30, 0.26
LA 0001B,3C,R	0.40, 0.39	0.33, 0.37, 0.37
	LA 0002	
LA 0002, Air Dried	2.88, 2.46	1.96, 1.84, 1.63
LA 0002B,1C	0.72	0.55, 0.50, 0.66
LA 0002B,1C,R	0.96	1.38, 1.10, 1.05
LA 0002B,3C	0.32, 0.37	0.39, 0.35, 0.37
LA 0002B,3C,R	0.38	0.33, 0.31, 0.37
	VT 0002	
VT 0002	15.75, 14.26, 19.02 18.07, 18.72, 19.14,	19.38, 17.81, 19.76
VT 0002B,1C	7.24, 7.17	8.84, 7.98, 9.37
VT 0002B,1C,R	8.27, 7.80	8.00, 8.83, 8.37
VT 0002B,3C	2.99	3.36, 3.29, 2.98
VT 0002B,3C,R	2.54	3.82, 3.94, 3.95

[1] EI Determinations.

4.3.5. *Strength of Cast Phase I Products*

The cast products were subjected to a semi-quantitative test to compare relative strengths. A soil penetrometer was positioned on the product surface and pressed until the product broke under the applied pressure. The force required to break the product was recorded. If the sample did not break the reading was recorded as "greater than 4.5".

TABLE 4.8. UNCONFINED SEMI-QUANTITATIVE STRENGTH TEST ON PHASE II PRODUCTS

Sample	Strength, kg/cm^2 (Tons/ft^2)		
	Sample 1	Sample 2	Sample 3
LA 0001B,1C	> 4.5	> 4.5	> 4.5
LA 0001B,1C,R	> 4.5	> 4.5	> 4.5
LA 0001B,3C	> 4.5	> 4.5	> 4.5
LA 0001B,3C,R	> 4.5	> 4.5	> 4.5
LA 0002B,1C	> 4.5	> 4.5	> 4.5
LA 0002B,1C,R	> 4.5	> 4.5	> 4.5
LA 0002B,3C	> 4.5	> 4.5	> 4.5
LA 0002B,3C,R	> 4.5	> 4.5	> 4.5
VT 0002B,1C	> 4.5	> 4.5	> 4.5
VT 0002B,1C,R	> 4.5	> 4.5	> 4.5
VT 0002B,3C	> 4.5	> 4.5	> 4.5
VT 0002B,3C,R	> 4.5	> 4.5	> 4.5

4.3.6. *Product Leach Results*

Phase I products were characterized by three leach tests (data supplied by the REM III Team; these data are considered preliminary and unvalidated): TCLP, Modified-ASTM (A) Deionized Water Leach, and Modified-ASTM (B) Carbonate Leach. The leach response measurements followed in this study were arsenic and total organic carbon. The arsenic and total organic carbon (TOC) concentrations in the samples subjected to the leach tests are presented in Table 4.9. Total volatile solids (TVS), starting sample pH, and specific gravity for the solids subjected to the leach tests are presented in Table 4.10. The leach test results are presented in Tables 4.11 (TCLP,As), 4.12 (ASTM-A,As), 4.13 (ASTM-B,As), 4.14 (ASTM-A,TOC), and 4.15 (ASTM-B,TOC).

TABLE 4.9. ARSENIC AND TOC CONTENT IN PHASE I PRODUCTS SUBJECTED TO LEACH TEST WORK

Sample	Concentration, mg/kg[1]	
	Arsenic	TOC
LA 0001[2]	11300	
LA 0001B,1C	2940	9990
LA 0001B,1C,R	1460	4580
LA 0001B,3C	1250	5730
LA 0001B,3C,R	2110	4310
LA 0002[2]	21500	
LA 0002B,1C	2910	7970
LA 0002B,1C,R	5370	1410
LA 0002B,3C	1190	3320
LA 0002B,3C,R	1760	8650
VT 0002[2]	178,600	
VT 0002B,1C	46100	18900
VT 0002B,1C,R	52400	2520
VT 0002B,3C	13500	11500
VT 0002B,3C,R	14100	5890

1 REM III Team Data: Analyses performed by Versar, Inc.
2 EI data

It is obvious from the data presented in Table 4.9 that a significant portion of the organic carbon is lost during the roast operation (25-87 percent, except LA 0002B,3C showed the reverse of this trend). Five of the sample mixes showed an increase in the arsenic concentration after roasting. The arsenic increase cannot be explained in terms of changing sample weight (caused by volatilization) because all the weight loss data collected (Phase II, Table 4.22) show a loss in weight of less than thirteen percent. A possible explanation may be that because of heterogenity in the starting mixture the samples that were roasted had an initial higher concentration of arsenic.

TABLE 4.10. CHARACTERISTICS OF PHASE I SOLID PRODUCTS SUBJECTED TO LEACH TESTS

Sample	Characteristic		
	TVS,%[2]	Sample pH	Specific Gravity
LA 0001	27.6	5.6	-
LA 0001B,1C	4.4	12.23	2.64
LA 0001B,1C,R	3.1	12.10	2.75
LA 0001B,3C	6.6	12.25	2.58
LA 0001B,3C,R	5.7	12.35	2.63
LA 0002	34.7	3.9	-
LA 0002B,1C	4.7	12.25	2.65
LA 0002B,1C,R	4.1	12.20	2.69
LA 0002B,3C	6.0	12.29	2.66
LA 0002B,3C,R	7.7	12.41	2.56
VT 0002	43.6	8.6	-
VT 0002B,1C	3.6	12.14	2.75
VT 0002B,1C,R	6.5	12.26	2.57
VT 0002B,3C	5.8	12.33	2.63
VT 0002B,3C,R	7.3	12.25	2.68

[1] REM III Team Data: Test performed by Versar, Inc.

[2] Total Volatile Solids.

TABLE 4.11. TCLP RESULTS FOR PHASE I PRODUCT MATERIALS

Sample	Wt, gms	Extractant Arsenic Concentration., mg/liter[1]		
		5-Day Cure		28-Day Cure
		EI Results	REM III Team[2]	REM III Team[2]
LA 0001[3]	10.07	1.8 (4.6)	4.6 (6.1)[4]	
LA 0001B,1C	10.01	13.2 (7.8)	38.7 (11.4)	15.8 (11.0)
LA 0001B,1C,R			0.19(11.5)	0.43(11.5)
LA 0001B,3C	10.23	2.0 (7.7)	6.7 (11.5)	5.02(11.5)
LA 0001B,3C,R			0.18(11.5)	0.11(11.7)
LA 0002[3]			2.36(6.2)	
LA 0002B,1C	9.79	15.1 (8.4)	37.9 (11.4)	30.1 (11.5)
LA 0002B,1C,R			0.22(11.4)	0.28(11.5)
LA 0002B,3C	10.06	3.3 (11.4)	7.9 (11.5)	8.81(11.8)
LA 0002B,3C,R			0.05(11.6)	0.32(11.8)
VT 0002[3]		7056. (6.5)	2260. (7.0)	
VT 0002B,1C	10.63	588.4 (8.7)	25.6 (11.5)	72.2 (11.4)
VT 0002B,1C,R			3.2 (11.2)	10.8 (10.3)
VT 0002B,1C	10.00	75.2		
VT 0002B,3C	10.18	29.6 (10.9)	37.4 (11.7)	5.4 (11.6)
VT 0002B,3C,R			9.6 (11.5)	0.99(11.7)

[1] Extraction Fluid 2 was used in all EI tests. Arsenic hazardous characteristic level: 5 mg/liter.
[2] REM III Team data: Test work performed by Versar, Inc.
[3] Starting material was a powder, no cement present, no cure required.
[4] The numbers in parentheses are final pH values.

Note that:

- All of the unroasted mixtures released arsenic to the TCLP extractant fluid at concentrations above the hazardous characteristic level (5 mg/liter). However, the cement/VT mixtures decreased the release, as compared to the unmixed VT samples, by up to 99.8% (using REM III data for the 28-day cured mixtures).
- All the LA unroasted mixtures released arsenic to the extractant fluid in higher concentrations than did the unmixed starting samples.
- All the roasted mixtures (except VT 0002B,1C,R) released arsenic to the TCLP extractant fluid at concentrations below the hazardous characteristic level.

TABLE 4.12. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]			
	Deionized Water Leach[2]			
Extract	1	2	3	Cumulative
LA 0001	5.03 (7.6)	4.88 (7.6)	1.86 (8.4)	11.77
LA 0001B,1C	20.0 (12.3)[4]	9.9 (12.1)	9.92 (12.1)	39.82
LA 0001B,1C,R	0.12 (12.3)	0.072(12.2)	0.029(12.0)	0.22
LA 0001B,3C	20.5 (12.3)	2.65 (12.1)	2.58 (11.9)	25.73
LA 0001B,3C,R	0.10 (12.3)	0.066(12.3)	0.076(11.9)	0.24
LA 0002	5.18 (7.9)	4.47 (7.7)	6.22 (8.6)	15.87
LA 0002B,1C	40.7 (12.3)	15.5 (12.3)	7.0 (11.8)	63.2
LA 0002B,1C,R	0.271(12.3)	0.162(12.3)	0.196(12.2)	0.629
LA 0002B,3C	9.24 (12.4)	6.07 (12.4)	5.38 (12.4)	20.69
LA 0002B,3C,R	0.027(12.4)	0.025(12.4)	0.037(12.6)	0.089

TABLE 4.12. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS (Continued)

Sample	Arsenic Extraction Concentration, mg/liter[1]			
	Deionized Water Leach[2]			
	Extract 1	2	3	Cumulative
VT 0002	1650. (8.6)	851. (8.5)	512. (9.0)	3013.
VT 0002B,1C	197.0 (12.4)	57.1 (12.4)	49.9 (12.4)	304.0
VT 0002B,1C,R	0.854(12.3)	0.493(12.4)	0.826(12.4)	2.173
VT 0002B,3C	50.2 (12.4)	21.4 (12.4)	22.6 (12.6)	94.20
VT 0002B,3C,R	1.27 (12.4)	0.452(12.4)	0.385(12.5)	2.11

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 Final pH values in parentheses.

Note that:

- All of the unroasted mixtures released arsenic to the deionized extractant fluid at a cumulative concentration above 26 mg/liter. However, the cement/VT mixtures decreased the release, as compared to the unmixed VT samples, by up to 96.9% (using REM III data for the 28-day cured mixtures).

- All the LA unroasted mixtures released arsenic to the extractant fluid in higher concentrations (21 to 63 mg/liter) than did the unmixed starting samples (12 to 16 mg/liter).

- All the LA roasted mixtures released arsenic to the deionized extractant fluid at cumulative concentrations below 0.63 mg/liter. The VT roasted mixtures released arsenic to the fluid at cumulative concentrations below 2.2 mg/liter.

TABLE 4.13. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE I PRODUCTS

Sample	Arsenic Extraction Concentration, mg/liter[1]			
	Carbonate Leach[2]			
Extract	1	2	3	Cumulative
LA 0001	10.5 (8.9)	126. (10.6)	101. (10.8)	237.5
LA 0001B,1C	27.3 (12.7)[3]	22.9 (12.3)	27.1 (12.2)	77.3
LA 0001B,1C,R	0.731(12.8)	19.0 (12.4)	39.2 (12.2)	58.9
LA 0001B,3C	10.5 (12.8)	11.0 (12.5)	0.91 (12.3)	22.4
LA 0001B,3C,R	1.45 (12.9)	5.79 (12.4)	11.4 (12.4)	18.6
LA 0002	26.7 (8.4)	105. (10.7)	115. (10.9)	246.7
LA 0002B,1C	35.9 (12.8)	24.6 (12.4)	40.2 (12.3)	100.7
LA 0002B,1C,R	1.02 (12.8)	15.9 (12.5)	30.2 (12.3)	47.1
LA 0002B,3C	12.8 (12.9)	89.1 (12.5)	11.7 (12.4)	113.6
LA 0002B,3C,R	0.444(12.9)	48.4 (12.5)	13.2 (12.4)	62.0
VT 0002	2060. (8.7)	2710. (10.4)	1900. (10.7)	6670.
VT 0002B,1C	284.0 (12.9)	180.0 (12.3)	200.0 (12.2)	664.0
VT 0002B,1C,R	61.5 (12.9)	83.8 (12.4)	117.0 (12.2)	262.3
VT 0002B,3C	98.5 (12.9)	91.9 (12.4)	113.0 (12.3)	303.4
VT 0002B,3C,R	25.9 (12.9)	70.7 (12.4)	76.1 (12.4)	172.7

1 REM III Team data: Test work performed by Versar, Inc.
2 ASTM D3987-85 Leachant B, 3 gm/liter sodium bicarbonate.
4 Final pH values in parentheses.

Note that:

- The cumulative arsenic extracted (for both the roasted and unroasted products) by the carbonate leaches is considerably greater than the arsenic extracted by the deionized water leaches (except LA 0001B,3C is about the same for both test sequences) for all mixtures.

- Also, the cumulative arsenic extracted from all the LA roasted products by deionized water was less than one mg/liter, but the arsenic extracted from all the LA roasted products by carbonate ranged from approximately 19 to 62 mg/liter.

  The cumulative arsenic extracted from all the VT roasted products by deionized water was about two mg/liter, but the arsenic extracted from all the VT roasted products by carbonate ranged from approximately 173 to 262 mg/liter.

- There is some reduction in the cumulative arsenic extracted for all the unroasted mixtures compared to the unmixed starting samples, e.g., the LA 0001 samples show reductions in extraction of 67.4% and 90.6%; the LA 0002 samples show reductions in extraction of 54.0% and 59.2%; and the VT 0002 samples show reductions in extraction of 90.0% and 95.4%.

TABLE 4.14. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS: TOTAL ORGANIC CARBON

Sample	Concentration, mg/liter[1]						
Extract[2]	1		2		3		Cumulative
	TOC[3]	TDS[4]	TOC	TDS	TOC	TDS	TOC
LA 0001	10.0		8.5		6.6		25.1
LA 0001B,1C	26.5	1740	11.6	1290	11.7	985	49.8
LA 0001B,1C,R	1.73	1810	1.50	1330	3.61	961	6.8
LA 0001B,3C	6.76	1910	4.71	1590	11.2	1360	22.7
LA 0001B,3C,R	1.54	2060	6.81	1850	16.0	1600	24.4

TABLE 4.14. MODIFIED-ASTM (A) DEIONIZED WATER LEACH RESULTS FOR PHASE I PRODUCTS: TOTAL ORGANIC CARBON (Continued)

Sample	Concentration, mg/liter[1]						
Extract[2]	1		2		3		Cumulative
	TOC[3]	TDS[4]	TOC	TDS	TOC	TDS	TOC
LA 0002	11.0		12.8		9.8		33.6
LA 0002B,1C	40.1	1820	19.7	1320	29.7	1080	89.5
LA 0002B,1C,R	1.87	1800	4.52	1330	9.98	1060	16.4
LA 0002B,3C	13.1	2320	11.6	1840	29.4	1310	54.1
LA 0002B,3C,R	1.51	2170	7.36	1870	26.7	1630	35.6
VT 0002	1019.		414.		182.		1615.
VT 0002B,1C	1.50	1600	3.50	1050	20.0	897	266.5
VT 0002B,1C,R	243.	2760	74.5	1420	52.1	1080	25.0
VT 0002B,3C	66.6	2290	34.3	1840	46.5	1440	147.4
VT 0002B,3C,R	3.07	2140	7.21	1770	28.3	1470	35.6

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant A, 48 hour exposure in each extraction.
3 REM III Team Data: Total Organic Carbon
4 REM III Team Data: Total Dissolved Solids

Note that:

- All of the unroasted mixtures released organic carbon to the deionized extractant fluid at a cumulative concentration above 23 mg/liter. However, the cement/VT mixtures decreased the release, as compared to the unmixed VT samples, by up to 90.9%.
- All the LA unroasted mixtures (except for LA 0001B,3C) released organic arsenic to the extractant fluid in higher concentrations (50 to 90 mg/liter) than did the unmixed starting samples (25 and 34 mg/liter).

- All the roasted mixtures released organic carbon to the deionized extractant fluid at cumulative concentrations less than that released by the unroasted mixtures (except for LA 0001B,3C). However, this effect may be due, in part, to lower starting organic concentrations in the roasted solids (because of loss by volatilization during the roast).

TABLE 4.15. MODIFIED-ASTM (B) CARBONATE LEACH RESULTS FOR PHASE I PRODUCTS: TOTAL ORGANIC CARBON

Sample	TOC Concentration, mg/liter[1]			
	Extract[2] 1	2	3	Cumulative
LA 0001	17.5	232.0	208.0	457.5
LA 0001B,1C	28.3	28.8	24.8	81.9
LA 0001B,1C,R	1.22	6.06	16.3	23.6
LA 0001B,3C	9.31	10.2	18.3	37.8
LA 0001B,3C,R	3.36	6.04	16.7	26.1
LA 0002	36.6	119.0	182.0	337.6
LA 0002B,1C	47.3	23.4	26.1	96.8
LA 0002B,1C,R	8.16	5.13	12.7	26.0
LA 0002B,3C	25.0	12.8	23.9	65.7
LA 0002B,3C,R	6.40	5.99	17.1	29.5
VT 0002	1008.	659.	226.	1893.
VT 0002B,1C	257.	66.3	51.7	375.0
VT 0002B,1C,R	2.00	7.53	17.7	27.2
VT 0002B,3C	62.9	28.4	34.7	126.0
VT 0002B,3C,R	4.90	6.06	14.2	25.2

1 REM III Team Data: Test performed by Versar, Inc.
2 ASTM D3987-85 Leachant B, 48 hour exposure in each extraction.
3 REM III Team Data: Total Organic Carbon

Note that:

- The cumulative organic carbon extracted (for both the roasted and unroasted products) by the carbonate leaches is greater than the arsenic extracted by the deionized water leaches (except VT 0002B,3C) for all mixtures and also for the unmixed, untreated starting samples.

- There is some reduction in the cumulative organic carbon extracted for all the unroasted mixtures compared to the unmixed starting samples, e.g., the LA 0001 samples show reductions in extraction of 82.1% and 91.7%; the LA 0002 samples show reductions in extraction of 71.3% and 80.5%; and the VT 0002 samples show reductions in extraction of 80.2% and 93.3%.

- All the roasted mixtures released organic carbon to the carbonate extractant fluid at cumulative concentrations less than that released by the unroasted mixtures. However, this effect may be due, in part, to lower starting organic concentrations in the roasted solids (because of loss by volatilization during the roast).

4.3.7. *Metallography and SEM-EDX*

Five samples were chosen for metallographic display, i.e., LA 0001B,1C, LA 0001B,1C,R, LA 0001B,3C, LA 0002B,1C, and VT 0002B,1C. The results are presented in Figures 4.6-4.10. Sample LA 0001B,1C was chosen to study in a more detailed way on the SEM-EDX system. Various phases in the structure were subjected to spot analysis. The spots chosen to be studied are marked on the photomicrograph presented in Figure 4.11 and their spectra are presented in Appendix Section 8, Figures 8.1-8.5. Identification of the elemental content and the semi-quantitative concentration of each spot are presented in Table 4.16.

The reason for performing the metallography test work was to display the structure of the cast material. Note in the photomicrographs the distribution and size of the various phases within the cement/sludge matrix.

The reasons for performing the SEM-EDX test work were:

- to determine qualitatively what elements were present.
    - The samples all contained the elements: As, Si, S, Cl, K, Ca, and Fe.

- to determine the nature of the distribution of arsenic in the cast structure.
    - One sample was chosen for spot analyses, i.e., LA 0001B,1C (See photomicrograph in Figure 4.4). Selected areas were subjected to spot analyses (Table 4.16).
    - The distribution of arsenic is noted to be distributed in a heterogeneous and spotty manner.

FIGURE 4 6 PHOTOMICROGRAPH FOR SAMPLE LA 0001B.1C (10X)

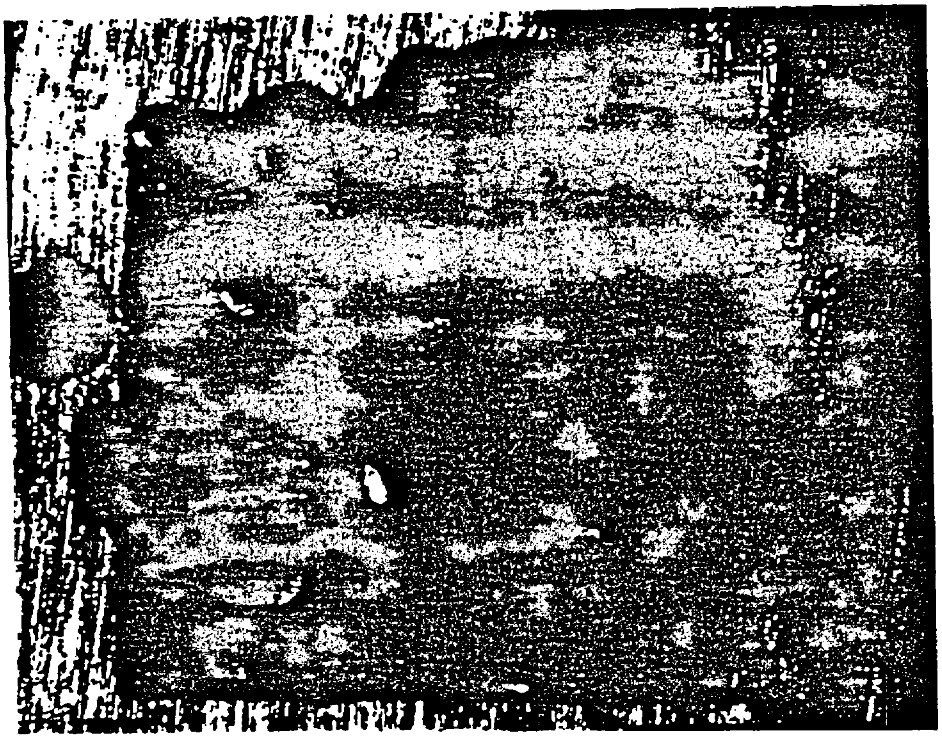


FIGURE 4 7 PHOTOMICROGRAPH FOR SAMPLE LA 0001B.1C.R (12.5X)

FIGURE 4.8. PHOTOMICROGRAPH FOR SAMPLE LA 0001B.3C (10X)

FIGURE 4.9 PHOTOMICROGRAPH FOR SAMPLE LA 0002B.1C (10X)

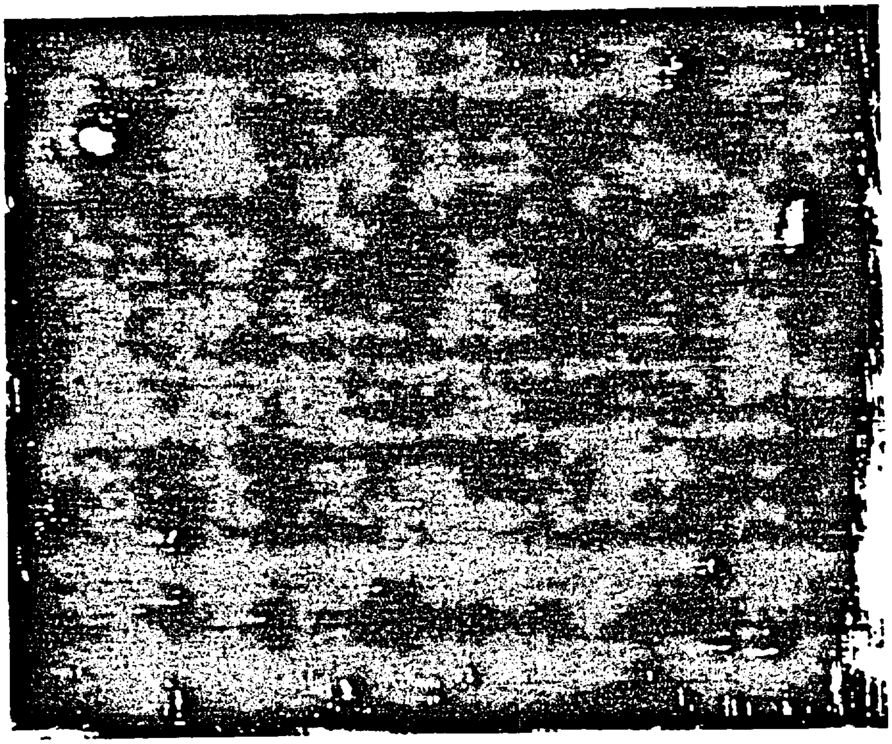
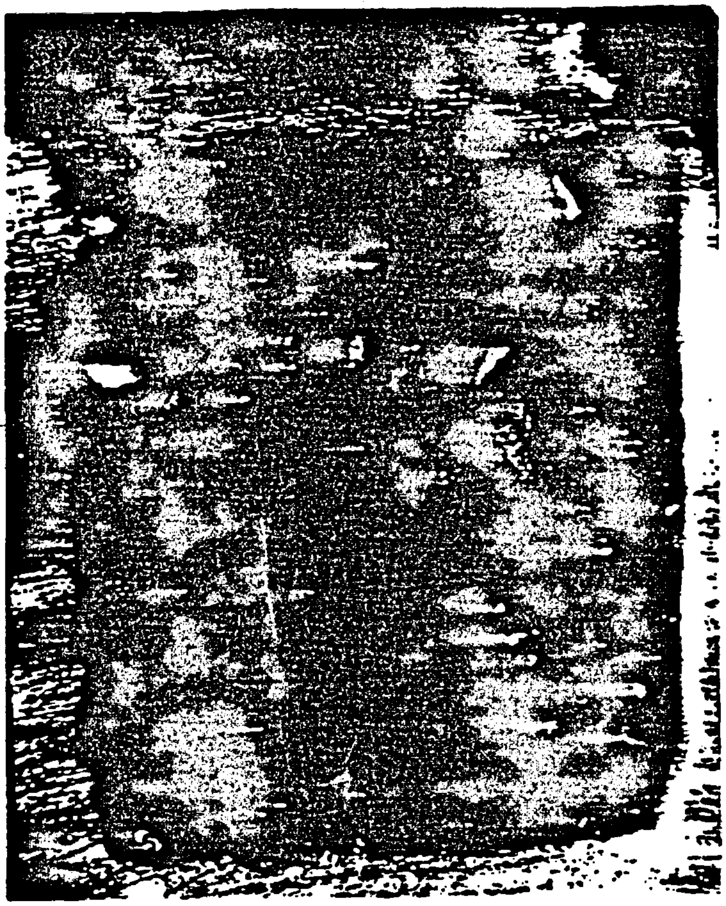


FIGURE 4.10. PHOTOMICROGRAPH FOR SAMPLE VT 0002B.1C (12 5X)



1 BULK MATRIX
2. ROCK
3. BRIGHT
4. BRIGHT
5. BRIGHT ON OPTICAL PHOTOMICROGRAPH BUT GRAY ON SEM

FIGURE 4 11. PHOTOMICROGRAPH OF SAMPLE LA 0001B.1C WITH MARKED SPOTS SUBJECTED TO SEM-EDX BEAM ANALYSES

TABLE 4.16. PHASE COMPOSITION IN SAMPLE LA 0001B,1C

Spot Number	Semi-quantitative Concentration, %							
	As	Ca	Fe	Al	Si	S	K	Mg
1 (Bulk)	2.8	43.1	10.6	5.9	31.2	4.0	2.6	0
2 (Rock)	0.4	8.3	4.8	15.6	56.9	1.1	12.8	0
3 (Bright)	0.1	35.5	40.8	2.2	4.5	5.8	1.0	1.5
4 (Bright)	9.9	25.0	44.3	4.0	10.6	2.9	0.8	2.5
5 (Bright on Optical but Grey on SEM)	0.6	59.4	1.4	2.3	4.5	0.6	0.9	30.3

4.4. *SMALL SCALE SCREENING TEST WORK*

The preliminary results generated from the Phase I experimental study showed that the most effective stabilization (as measured by the TCLP response) resulted for those samples that were roasted. It was felt that a series of smaller scale tests were necessary before beginning the Phase II large scale sample generation. The detailed results of this work are presented in a separate report, Clay Pelletizing and Sintering Treatability Study (1). The results of that study are important to this study and, therefore, the results obtained are summarized in this report.

The small scale tests were desired to answer the questions:

- Could stabilization be produced by roasting the starting materials without the addition of cement? What is the effect of roasting temperature on the stabilization?

- Could stabilization of the vault material be produced in the 3/1 mixture by roasting at a higher temperature? Is the CaO content an important variable in the stabilization process?

- Could stabilization of the vault material be produced in a non-roasted cement material if thiourea were an additive?

Earlier research showed that stabilization of calcium arsenate and/or calcium arsenite precipitated products could be stabilized by roasting in air if the Ca/As mole ratio was properly controlled. Nishimura and Tozawa(5) showed if the Ca/As mole ratio was greater than 2.1 that roasting in air at 700°C decreased the arsenic solubility to less than 20 micrograms/liter (when exposed to a pH 12 leach solution); a ratio of greater than 4.1 was required to stabilize calcium arsenite. Other researchers showed similar results (6,7). The vault material appeared to contain insufficient calcium to have the necessary Ca/As ratio but it was desired, however, to test potential stabilization by roasting without additional calcium and by roasting with additional calcium supplied by cement.

A similar argument can be presented for the LA materials. Tozawa (8) showed that ferric arsenate could be stabilized by roasting Fe/As mole ratios greater than 1.3 at 600°C. In the case of the LA materials the bulk Fe/As ratios were greater than the Tozawa required values.

The potential for stabilization of the test materials by simple roasting appeared to warrant further study. The results of the test work are presented below.

4.4.1. *Screening Experiments*

A series of roast experiments were planned and conducted. The procedure has been presented previously in Section 3.3. Briefly, the procedure was to: roast 100 gram samples for one hour at 600°C, 700°C, 800°C, and 1000°C. It was also desirable to collect good weight loss data during the study. This was done by heating 100 gram fireclay crucibles at the temperature of interest, weighing the crucible, adding the sample, weighing the sample plus crucible, heating the sample plus crucible at 200°C for one hour to remove adsorbed moisture, weighing the crucible plus sample, placing the crucible plus sample in a furnace at the temperature of interest for one hour and twenty minutes, weighing the roasted crucible plus product.

The weight loss data are summarized in Table 4.17. Observation comments are presented in Table 4.18. Temperature profile data are presented in Appendix Section 8.2.

Appendix Section 8.2.

TABLE 4.17. SCREENING TEST WEIGHT LOSS SUMMARY

Sample	Temp., °C[1]	Weight Loss, %	
		At 200°C	At T°C
LA 0001	600	3.7	11.0
	700	3.1	13.5
	800	2.6	17.6
	1000	3.0	24.4
VT 0002	600	5.5	26.2
	700	6.0	30.1
	800	9.5	29.7
	1000	5.5	38.4
Cement[2]	700	-	0.2
VT 0002B,3C	600	4.9	11.6
	700	5.4	12.6
	800	6.0	13.0
VT 0002,1LIME	700	2.2	11.7

1. The actual sample temperature profiles are presented in Appendix Section 8.2.1.
2. From Phase II study.

TABLE 4.18. OBSERVATIONS ON SCREENING ROAST EXPERIMENTS

Sample	Temp., °C	Observations
LA 0001	600,700,800 1000	Fuming not observed for any of the LA samples. Samples red on surface but lighter red in bulk of sample. Samples became progressively redder at the higher

TABLE 4.18. OBSERVATIONS ON SCREENING ROAST EXPERIMENTS (Continued)

Sample	Temp., ^{o}C	Observations
VT 0002	600,700,800 1000	Note temp. profiles in Section 8.2. The temp. profiles for the VT series indicated combustion at the sample surface. No obvious change in sample color. Fuming evident from furnace. See Section 8.2 for temp. start and end of fuming from furnace door.
VT 0002,3C	600,700,800	Fuming not observed from furnace door but temp. profiles at 700°C and 800°C indicate combustion at surface was occurring. Fuming was observed at the crucible surface for the 800°C sample but no fuming visible from the door.
VT 0002,1LIME	700	Ca/As mole ratio greater than 5. Fuming not observed on surface or from furnace. Temp. profile suggests no combustion.

It is, of course, desirable to have a way of disposing of the waste by a non-roasting technique. Tetsuro (9) conducted a study using thiourea as an additive to cement to stabilize an industrial arsenic bearing waste material. Therefore, a test of the possible use of this technology was conducted. One hundred grams of VT 0002B,1C was mixed and water containing five grams of thiourea. The mixture was cast and aged for three days, then subjected to the modified TCLP test. This test work was a part of the Pelletizing and Sintering Treatability Study (1) and the detailed results are presented in that report.

4.4.2. *TCLP Response*

Ten grams of each of the materials generated in the screening study were subjected to the modified TCLP test. The results are presented in Table

4.19. The arsenic concentration in the products tested in the TCLP study are presented in Table 4.20.

TABLE 4.19. SCREENING TEST MODIFIED-TCLP[1] RESULTS

Sample	Temp., °C	As in Extractant Fluid[2,3], mg/liter
LA 0001	Unroasted	11.1(5.3)[4]
	600	344 (5.6)
	700	106 (6.7)
	800	22.7(9.7)
	1000	10.8(11.7)
VT 0002	Unroasted	7056 (6.5)
	600	2291 (5.8)
	700	1546 (5.6)
	800	1213 (5.6)
	1000	347 (6.0)
VT 0002B,3C	Unroasted	333 (12.4)
	600	2.2(11.8)
	700	1.2(11.8)
	800	1.8(12.4)
VT 0002, 1LIME	Unroasted	578 (12.9)
	700	1.6(12.9)
VT 0002B,1C, THIO	Unroasted	20.0(8.0)

1. Test procedure described in Section 3.3. All starting materials were powders, not cubes.
2. Extraction Fluid 2 used for all samples.
3. The decrease in arsenic extracted at the higher test temperatures (for the unmixed samples) is likely due to the fact that the arsenic concentration in the product samples was less because of volatilization. This conclusion is supported by the weight loss data presented in Table 4.17.
4. Final pH of TCLP solutions shown in parenthesis.

TABLE 4.20. ARSENIC CONTENT IN SMALL SCALE ROAST STUDY PRODUCTS

Sample	Temp., ^{o}C	Arsenic Content, %		
		Sample 1	Sample 2	Sample 3
LA 0001	Unroasted	1.32	1.14	1.37
	600	1.53	1.60	1.54
	700	1.50	1.56	1.60
	800	1.52	1.41	1.72
	1000	1.66	1.58	1.49
VT 0002	Unroasted	18.07	18.72	19.14
	600	10.83	19.18	18.17
	700	15.19	18.40	16.52
	800	15.89	15.92	14.40
	1000	4.30	4.57	4.11
VT 0002B,3C	Unroasted	4.47	4.60	4.59
	600	4.78	4.95	4.27
	700	4.89	4.51	4.78
	800	6.50	6.80	5.82
VT 0002, 1LIME	Unroasted	12.02	11.64	14.63
	700	16.18	9.75	15.81

4.4.3. *Phase I and Screening Study Conclusions*

The Phase I and Screening studies showed encouraging TCLP results:

- Roasted LA sample materials (Phase I) at cement/sample ratios of 1/1 and 3/1 showed TCLP results more than an order of magnitude less than the characteristic arsenic level of 5 mg/liter. However, the unroasted sample materials at both cement/sample ratios exceeded the 5 mg/liter level and actually had higher TCLP results than unmixed and untreated samples.

- Roasted VT sample materials (Phase I) at cement/sample ratios of 1/1 and 3/1 showed TCLP results (for the five day cured samples) where the 1/1 ratio was less than 5 mg/liter but the 3/1 ratio was greater than 5 mg/liter. This effect was reversed for the twenty-eight day cured samples.

- Unroasted VT-cement materials (Phase I) showed a great decrease in the release of arsenic to the TCLP extraction fluid, i.e., the extraction in the absence of cement was over 2,260 mg/liter (REM III results) but the presence of cement decreased the arsenic release up to 99.8%. Although the arsenic characteristic level could not be achieved the decrease was significant. The above effect was also true of the deionized water leaches and the carbonate leaches, i.e., reductions in cumulative arsenic extraction of 96.9% (for deionized water leaches) and 95.4% (for carbonate leaches) were achieved.

- Screening test work showed that unmixed LA and VT samples could not be stabilized by roasting. However, the tests on mixtures showed that the mixtures could be stabilized (as measured by the TCLP test) and that the stabilization result was not sensitive to roasting temperature (at least within the temperature range 600°C to 800°C).

- The presence of calcium (maybe as free lime or some other phase) appears to be an important stabilizing parameter. Its presence is important for retaining arsenic in the LA samples but roasting is also required. The presence of calcium (as cement or as lime) is important for retaining arsenic in the VT samples. Its presence reduces arsenic release without roasting but not to levels below the TCLP characteristic level; its presence and roasting (with either cement or lime) causes the TCLP arsenic level to be below the characteristic level.

- The addition of thiourea to arsenic bearing industrial waste and cement has been stated in the literature to stabilize the release of arsenic. The single TCLP test performed in the screening study did show a large decrease in the release of arsenic, i.e., without cement or thiourea the release was over 7000 mg/liter. The presence of cement (1/1 ratio) and thiourea resulted in a release of only 20 mg/liter of arsenic. The presence of cement without thiourea showed

(Table 4.9) variable results for two tests (588 mg/liter and 75 mg/liter) but the arsenic extracted was greater in both tests than the quoted 20 mg/liter value above.

- The conclusions of the TCLP, ASTM deionized water, and ASTM carbonate leach tests on Phase I products are:
  - Roasting and calcium are required to stabilize the sludge materials so that they pass the TCLP leach characteristic arsenic level. All roasted Phase I products released less than five mg/liter arsenic.
  - All Phase I roasted products are stable (assuming that a cumulative level of <five mg/liter is considered stable) in deionized water (for at least three extraction exposures) but none of the roasted products are stable (using the same criteria for stability) in a carbonate environment.

4.5. *PHASE II STUDIES*

The *task objectives* for the Phase II work included:

- Preparation of designated cement/sample, cement/lime/sample, cement/thiourea/sample mixtures for each of the three supplied sample materials, i.e.,
  - Cement/sample ratios for the lagoon (LA 0001) and soil (LA 0002) samples of 1/2 and 1/5.
  - Cement/lime/sample ratios for the vault sample (VT 0002) of 1/1/1 and 0.75/0.6/1.
  - Cement/thiourea/sample ratios for the vault, lagoon and soil samples of 1/0.01/1 (This set of sample preparations was actually performed in a separate study "Clay Pelletizing and Sintering"(1). The results are also reported in this presentation for completeness.)
- Roasting one half of each cement/sample and cement/lime/sample ratio as specified above except for the thiourea containing samples, i.e., one half of the mixed samples were roasted at 700°C for one hour.
- Casting each sample mixture, roasted and unroasted, into a product of a specified size (to pass a 3/8-inch screen) and aging for twenty-eight days.

4.5.1. *Sample Mixing*

Sample mix ratios as specified above were prepared as described previously in the procedure section. The amounts of each mix material initially prepared are summarized in Table 4.21.

TABLE 4.21. PHASE II SAMPLE MIXTURES

Sample	Mix Ratio	Quantity Prepared, Grams
	Cem./Lime	
LA 0001B,1:2	1/2	1,500
LA 0001B,1:2,R	1/2	1,500
LA 0001B,1:5	1/5	1,500
LA 0001B,1:5,R	1/5	1,500
LA 0002B,1:2	1/2	2,270
LA 0002B,1:2,R	1/2	2,270
LA 0002B,1:5	1/5	1,450
LA 0002B,1:5,R	1/5	1,450
	Cem./Lime/Spl.	
VT 0002B,1:1:1	1/1/1	2,040
VT 0002B,1:1:1,R	1/1/1	2,040
VT 0002B,Lime	0.75/0.6/1	2,000[1]
VT 0002B,Lime,R	0.75/0.6/1	2,000
	Cem./Thio/Spl.[2]	
LA 0001,Thio	1/0.01/1	1,500
LA 0002,Thio	1/0.01/1	2,000
VT 0002,Thio	1/0.01/1	2,270

1. Sample prepared using hydrated lime. Therefore, weight adjusted to give a weight ratio content of 0.6 CaO.
2. The thiourea was added by dissolving it in the water used to cast the cement/sample mixture.