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TECHNICAL DIVISION  
SAVANNAH RIVER LABORATORY

DPST-81-831

CC: J. A. Kelley  
R. M. Wallace  
L. M. Papouchado  
R. B. Ferguson  
D. D. Walker  
N. E. Bibler  
P. D. Soper  
G. G. Wicks  
J. A. Stone  
TIS File Copy (2)

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MEMORANDUM

December 31, 1981

TO: M. J. PLODINEC

FROM: J. R. FOWLER/P. K. SMITH

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COMPARATIVE STUDY OF THE LEACHING OF WASTE GLASS  
AND A NATURAL VOLCANIC GLASS

INTRODUCTION AND SUMMARY

The leaching behavior of waste glass and a natural volcanic glass were compared to provide perspective on the long term durability of waste glass. Polished and unpolished wafers of waste glass and obsidian were leached in distilled water at 90°C for times up to 28 days using MCC-1 guidelines. The leached surfaces were examined by scanning electron microscopy, x-ray energy spectroscopy, x-ray diffraction, and Auger spectroscopy to compare leaching behavior. The following general observations were made:

- o Waste glass was leached an order of magnitude faster than obsidian in these relatively short tests, but the leach rate was still decreasing with time while the leach rate for obsidian was linear with time.
- o Waste glass was leached by a different mechanism than obsidian glass, at least in the relatively short leach times used in this work.
- o The outer 1/4 micron of obsidian leached the longest times had higher concentrations of Fe, Ca and Al than the unleached sample, suggesting that the leaching behavior of the two glasses may be similar if obsidian is leached for longer times.

## DISCUSSION

### Objectives

A comparison of the leach rates of waste glass with natural volcanic glasses was suggested by the Peer Review Committee (Hench Panel) as a way to obtain some perspective on the long term durability of waste glass. A considerable volume of data exists on the long term behavior of natural glasses in various geologic environments. A particularly beneficial result would be to show that waste glass and natural glass leach by similar mechanisms in accelerated leach tests. Such an observation might increase the general confidence that "glass is good enough".

### Source and Analyses of Glasses

A can of Frit 131-TDS waste glass was poured by D. D. Walker from the CTD minimelter after sufficient glass had been made to obtain a homogeneous and representative melt composition. The as-made and analyzed compositions are shown in Table 1. Metallographic and scanning electron microscopic analyses showed the glass to be completely vitrified and homogeneous.

No natural, volcanic, mostly vitrified glasses are available with similar compositions. Natural glasses do not contain much boron. Natural basaltic glasses with low SiO<sub>2</sub> concentration and high waste constituent concentrations have long since devitrified to largely crystalline basalt primarily from hydrothermal effects. A comparison of the leach rates of basalt and waste glass was measured by J. A. Stone.<sup>1</sup>

A 10-inch chunk of black obsidian glass used for this work had some basaltic tendencies but was still >98% vitreous. The chemical analyses are shown in Table 2. Except for high SiO<sub>2</sub> content, the absence of boron and the high potassium content, the distribution of cations was similar to waste glass loaded with about 15 to 20% in high alumina waste. The composition of the obsidian matches that of typical naturally-occurring rhyolite glasses (Table 2). The obsidian was obtained from the U. S. Geological Survey who collected the sample from the Snake River basaltic plain 10 miles southeast of Idaho Falls, Idaho.

Metallographic and scanning electron microscopic analyses of the obsidian taken from three locations in the 10-inch block showed the glass to consist of 5 to 50 m-thick laminations of

glass oriented in the same direction throughout the block (Figure 1) and separated by thin crystalline layers. These laminations appear to reflect flow patterns in the original magma. The bulk composition was uniform throughout as determined by x-ray energy spectroscopy (Figure 2). As was determined chemically, the x-ray energy spectrometer shows that Si is most abundant in the obsidian followed by Al, K, then Fe and Na.

Submicron crystals are evident in the obsidian as shown in Figures 1 through 3. These crystals occupy less than 2% of the glass. The glass laminations are depleted in crystal near the boundaries of the laminations because of diffusion to the boundaries and precipitation to form the larger crystals seen at the boundaries. Smaller crystals precipitated evenly within the laminations where distances were too great (1 to 2 m) to allow diffusion to the boundaries.

Both the crystals within the laminations and at the boundaries (Figure 3) were analyzed and found to contain principally Fe and probably Si with some Ca and Al. X-ray diffraction of a polished surface showed three peaks with symmetry resembling that of a complex silicate. These crystals are probably pyroxenes typified by augite and acmite which are the predominant crystals in devitrified waste glass, in outer leached layers of waste glass, in basalt and in other volcanic rocks. The "waste" equivalent in this obsidian, then, is for the most part in these microcrystals. The preferential leaching of these crystals is described later in this report. Figure 3 shows isolated examples of other crystals in the obsidian; in this case, a Ca, P, Si oxide.

#### Preparation of Leach Samples

Rods of both waste glass and obsidian were core-drilled with water-cooled diamond bits from large crack-free chunks. These core-drilled rods were wafered with a water-cooled diamond abrasive wheel to obtain the disc-shaped samples. Wafers about one-half inch diameter by 0.1-inch thick were chosen to facilitate subsequent analyses of the wafers.

Some wafers were polished on the flat faces to provide sharp delineation of leach layers, again to facilitate interpretation of surface analyses subsequent to leaching. Polishing was effected by grinding through three carbide grit sizes on water-cooled wheels and polishing with 15 m diamond paste on nylon cloth with water.

## Leaching Conditions and Analyses

The draft "MCC-1 Static Leach Test" procedure submitted in January 1981 by the Materials Characterization Center (PNL) to the Materials Review Board was used to obtain leaching data. Only deionized water was used as a leachant and all tests were done at 90°C.

Mass loss ( $NL_i$ ) and leach rates ( $LR_i$ ) of each element were normalized to the relative concentration of each element in the glasses as specified by the MCC-1 procedure.

$$NL_i = \frac{C_i}{f_i^* (SA/V)} \text{ in g/m}^2$$

$$\text{and } LR_i = NL_i/t \text{ in g/m}^2\text{-day,}$$

where  $f_i^*$  is given in Tables 1 and 2. All the measured normalized leach rates are listed in Tables 3 and 4 for waste glass and obsidian, respectively.

## Results

### o Leach Rates

The effect of leaching time on elemental mass loss of both TDS-131 glass and obsidian is shown in Figure 4 for the principal dissolved elements. These results and the weight losses for the wafers in Tables 3 and 4 show that the normalized mass loss of the waste glass specimens is at least a factor of 10 higher than the mass loss of the obsidian specimens. This result is attributed to the higher silicon and lower alkali content of the obsidian. A correlation of increasing silicon content and decreasing mass loss can be seen when these results are included with results of J. A. Stone<sup>1</sup> and G. G. Wicks<sup>2</sup> obtained from other glasses (see Figure 5).

Differences in the mechanism of leaching are apparent from Figures 6 and 7 and from the surface analyses shown later. These two figures show that the normalized leach rate for the elements in frit are about 10X higher for waste glass than for obsidian after an initial loss of Na and Mg from the obsidian. Moreover, the parabolic decrease in leach rate with increasing leaching times for waste glass frit ingredients suggests diffusion limited mechanisms predominate consistent with the buildup of scales observed on leached waste glass. However, leach rates for obsidian are linear with time, implying no diffusion limiting scales. In fact, none were observed.

Further, the leach rates of the elements in waste glass can be divided into two groups with different behaviors. Frit

constituents (Na, Li, B and Si) show diffusion limited, parabolic behavior at identical rates. Waste constituents (Fe and Mn) have much lower leach rates, indicating they are retained in the glass and leach linearly with time rather than parabolically. The behavior of Al and Mg is intermediate to these distinct groupings.

Leach rates measured after 28 days on polished wafers were up to 10 times lower than those measured on unpolished, as-cut wafers (Table 5). These results stress the importance of sample preparation in obtaining data for comparison with other work. Polishing reduced the leach rate of major constituents by 50 to 60% for both TDS-131 glass and obsidian, consistent perhaps with the reduction in surface area by polishing. Rates for waste constituents (Ca, Fe and Mn) that are generally retained in the glass were reduced even further for waste glass. However, the leach rate based on Fe, Mg, and Al were not changed by polishing obsidian, consistent with their being leached preferentially from submicron crystals in the obsidian matrix.

#### o Surface Characterization

##### - Scanning Electron Microscopy/X-Ray Energy Spectroscopy

The surfaces of polished wafers that had been leached for 28 days were examined visually, by optical microscopy, by scanning electron microscopy and by x-ray energy spectroscopy. The waste glass wafers were covered with the usual translucent gel-like scales of different hues. Their thickness increased with increasing leaching time. The thicker scales were fragile and readily flaked off in two levels when handled (Figures 8, 9, and 10). On the other hand, no scales or discolorations were observed on the obsidian wafers indicating significantly lower leach rates.

Figure 8 shows the nature of the flakiness and the variations in chemical analysis of the substrate and the two major flake layers. The leach layers are 3 to 5 m thick after 28 days at 90°C. Analysis of Area 1 shows base glass containing sodium in addition to Mg, Al, Si, Ca, Ti, Mn, Fe and Ni. Area 2 shows sodium depletion and strong enrichment in Fe, Mn, Ca, Ti and Ni relative to silicon. Area 3 on the top of the leach layers shows recovery of Si concentration and increases in Mg, Al, Mn and Ni relative to Fe.

A more detailed analysis of the leached layers is shown in Figure 9. This view shows at least eight distinct layers in the two flake levels. Each level shows a different chemical composition with the general features described

above. The Si/Fe ratio decreases sharply from the substrate to level 3, then increases steadily through level 8, but is still lower than in the base glass. Waste ingredients and Mg and Ti are enriched in the upper levels. Mn is especially enriched in levels 7 and 8, approaching the Fe concentration. Although Na is depleted in level 1, some Na exists in all other levels.

Another view supporting these trends is shown in Figure 11. Submicron, poorly formed crystals can be seen in the top of the leached layer. Microprobe analyses of these crystals could not distinguish any chemical difference between the crystals and the surrounding glass. X-ray diffraction analysis showed three diffraction lines that could not be precisely identified. But the symmetry matches that of a pyroxene silicate comprised of multivalent cations, typical of those seen in crystallized basalt. These data suggest that when the waste constituents become sufficiently concentrated relative to  $\text{SiO}_2$ , pyroxenes crystallize by hydrothermal reaction in a similar manner to natural basalts at comparable  $\text{SiO}_2$  concentrations.

No leached layers on the obsidian wafers could be seen by SEM after 28 days leaching. The XES data in Figure 12 shows that the leached surface has the same ratios of elemental constituents as seen in the base obsidian. The SEM view shows holes in the glass where the submicron crystals containing Fe, Ca, Al and Si had been (compare to Figures 1, 2 and 4). The leach rates for obsidian reported in Figures 5 and 8 may be related entirely to the leaching of these microcrystals.

#### - Auger Spectroscopy

The depth profile of the elements, Si, Mg, Fe, Ni and Al, was measured on the leached layers by Auger spectroscopy. Other elements either could not or were not analyzed, so that their omission does not imply their absence. Two of the flakes on the waste glass were analyzed that were presumed, with some uncertainty, to be an outer flake and an inner flake. These data are shown in Figures 13 and 14. The data in Figure 13 show predominance of Fe and Ni and, to a lesser abundance, Mg and Al at the surface and their gradual decrease in relative concentration down  $4\mu\text{m}$  (uncalibrated) into the layers. Ni and Fe are still in excess at this depth. Si, on the other hand, shows a constant and normal concentration in the leached layers in contrast to the XES data.

The depth profile presumably starting lower in the leached layer is shown in Figure 14. These data show a double periodicity in enhanced Fe and Ni concentration and fairly constant Mg, Al, and Si concentrations at their normal concentrations in the base glass.

Depth profiling on obsidian leached 28 days showed a 1/4-micron thick concentration of Fe, Ca and Al at the outer surface. Except for Al, Si and the other elements exhibit normal concentrations below 1/4-micron deep. These data support the absence of relatively thick leached layers for obsidian leached for these short times. However, the data do suggest that a scale might build up with longer leach times, or leaching at higher temperatures, that would concentrate waste constituents in the same manner as waste glass.

o General Assessment

Obsidian leaches so slowly compared to waste glass that the leach mechanisms were different in these relatively short exposure times. Consequently, very little can be said about the long term leach behavior of waste glass relative to natural glass. The data strongly suggest that leaching of obsidian for longer periods may produce leach layers thick enough to allow a comparison of the rate limiting mechanisms.

The data obtained on leached waste glass shows the preferential concentration of primarily waste metal constituents in the leached layers as a number of previous workers have shown.<sup>3</sup>

Eight or more distinct strata with different chemical compositions have been identified in these complex, thick, leached layers on waste glass. Crystallization occurred in the outermost layer. Leached layers on wafers leached for much shorter times should be characterized to identify the sequence of layer development and the rate-limiting factors.

JRF/PKS:lmn

## REFERENCES

1. J. A. Stone to J. E. Mendel, letter and attachments dated February 5, 1981.
2. G. G. Wicks to L. M. Papouchado, DPST-81-653, September 3, 1981.
3. A. Barkatt, J. H. Simmons and P. B. Macedo, "Evaluation of Chemical Stability of Vitrification Media for Radioactive Waste Products," Phys. and Chem. of Glasses, 22 (1981) 73-85.

TABLE 1

Composition of TDS-131 Waste Glass<sup>(c)</sup>

<u>Constituent</u>	<u>Prepared Wt %</u>	<u>Found Wt %</u>	<u><math>f_i^*</math> Used in MCC Tests</u>
SiO <sub>2</sub>	43.9	41.2 ± 2.1 <sup>(a)(d)</sup>	0.192 (Si)
Fe <sub>2</sub> O <sub>3</sub>	14.2	14.7 ± 0.1 <sup>(b)</sup>	0.103 (Fe)
Al <sub>2</sub> O <sub>3</sub>	3.5	3.3 ± 0.5 <sup>(a)</sup>	0.0174 (Al)
CaO	1.3	0.95 ± 0.05 <sup>(a)</sup>	0.0068 (Ca)
NiO	1.6	1.6 ± 0.1 <sup>(b)</sup>	--
MnO <sub>2</sub>	3.8	3.9 ± 0.1 <sup>(b)</sup>	0.0246 (Mn)
Na <sub>2</sub> O	13.5	12.5 ± 0.6 <sup>(b)</sup>	0.0927 (Na)
Li <sub>2</sub> O	4.2	4.0 ± 0.1 <sup>(b)</sup>	0.0186 (Li)
B <sub>2</sub> O <sub>3</sub>	10.8	10.3 ± 0 <sup>(b)</sup>	0.0320 (B)
K <sub>2</sub> O	--	--	--
MgO	1.5	--	0.009 (Mg)
	<u>98.3</u>	<u>92.5</u>	

$f_i^*$  Elemental wt. fraction of specific component.

(a) Analyzed by XRF on solid.

(b) Analyzed by AA or IC after dissolving solid sample.

(c) Data from D. D. Walker.

(d) Known to be low.

TABLE 2

Composition of Obsidian

Constituent	Typical <sup>(a)</sup> Rhyolite Obsidian	Obsidian in This Test			f <sub>i</sub> <sup>*</sup> Used in MCC <sup>(c)</sup>
		(Wt %) NAA	XRF	ICP	
SiO <sub>2</sub>	72.8 wt %	--	72.2 <sup>(b)</sup> ± 2.7	(85)	0.316
Fe <sub>2</sub> O <sub>3</sub>	2.4	2.6 ± 0.3	1.8 ± 0.4	--	0.0205
Al <sub>2</sub> O <sub>3</sub>	13.3	11.3 ± 0.1	11.1 ± 0.4	10.8	0.060
CaO	1.2	--	0.4 ± 0.03	--	0.0026
NiO	--	<0.02	--	--	--
MnO <sub>2</sub>	0.1	0.04 ± 0.002	0.05 ± 0.02	--	0.00028
Na <sub>2</sub> O	3.3	3.2 ± 0.25	--	--	0.0236
Li <sub>2</sub> O	--	--	--	1.3 X 10 <sup>-5</sup>	--
B <sub>2</sub> O <sub>3</sub>	--	--	--	1.2 X 10 <sup>-4</sup>	--
K <sub>2</sub> O	4.6	5.0 ± 0.2	--	--	0.0419
MgO	0.4	0.3 ± 0.3	--	--	0.0015
TiO <sub>2</sub>	0.3	0.3 ± 0.04	--	--	--
H <sub>2</sub> O	1.5	--	--	--	--
P <sub>2</sub> O <sub>5</sub>	0.1	--	--	--	--
	100.0	94 to 95			

(a) After Daly, 1933

(b) Believed to be low.

(c) Elemental weight fraction of specified metallic component.

TABLE 3

Normalized Average Leach Rate of TDS-131 Glass (Unpolished)

Days:	$NL_i$ g/m <sup>2</sup>				$(LR)_i$ , g/m <sup>2</sup> -day			
	<u>3</u>	<u>7</u>	<u>14</u>	<u>28</u>	<u>3</u>	<u>7</u>	<u>14</u>	<u>28</u>
<u>Element</u>								
Si	11.6	15.2	19.0	23.1	3.63	2.03	1.27	0.825
B	12.5	16.9	21.6	26.3	4.16	2.41	1.54	0.939
Na	13.2	19.6	21.9	27.4	4.40	2.80	1.56	0.979
Li	12.3	16.0	20.4	25.9	4.07	2.27	1.44	0.914
Ca	--	--	--	--	--	--	--	--
Fe	0.052	0.0980	0.234	0.534	0.0173	0.0140	0.0167	0.0193
Mn	0.01	0.10	0.24	0.699	0.004	0.0146	0.0171	0.0250
K	--	--	--	--	--	--	--	--
Mg	0.5	0.7	0.4	0.24	0.17	0.10	0.029	0.0086
Al	7.87	9.54	10.80	11.28	2.62	1.36	0.771	0.402
Mass Loss	6.5	9.9	11.5	14.6	2.2	1.4	0.82	0.52

TABLE 4

Normalized Average Leach Rate of Obsidian (Unpolished)

Element	$NL_i, g/m^2$				$(LR)_i, g/m^2\text{-day}$			
	3	7	14	28	3	7	14	28
Si	0.165	0.149	0.617	1.21	0.055	0.021	0.044	0.043
Na	2.88	0.614	2.37	2.54	0.96	0.088	0.169	0.091
Ca	1.88	~0	~0	~0	0.63	~0	~0	~0
Fe	0.200	0.668	0.649	0.652	0.067	0.095	0.046	0.023
Mn	~0	~0	~0	~0	~0	~0	~0	~0
K	--	--	--	--	--	--	--	--
Mg	2.5	1.1	1.5	0.69	0.83	0.16	0.11	0.024
Al	0.37	0.53	1.1	0.24	0.12	0.076	0.078	0.0086

TABLE 5

Effect of Polishing on 28-Day Leach Rates

<u>Element</u>	<u>"TDS" Glass</u>		<u>Obsidian</u>	
	<u>Unpolished</u>	<u>Polished</u>	<u>Unpolished</u>	<u>Polished</u>
Si frit	0.825 g/m <sup>2</sup> -day	0.532	0.043	0.019
B frit	0.939	0.600	--	--
Na frit	0.979	0.607	0.091	0.050
Li frit	0.914	0.579	--	--
Mg frit	0.009	0.006	0.024	0.029
Ca waste	0.003	0.0003	~0	~0
Fe waste	0.0193	0.002	0.023	0.026
Mn waste	0.0250	0.005	~0	0
Al waste	0.402	0.263	0.0086	0.017

Change in leach rate due to polishing:

$$\frac{(LR)_i \text{ polished}}{(LR)_i \text{ unpolished}} \times 100 = \% \text{ of unpolished leach rate}$$

TDS Glass (Si, B, Na, Li, Mg, Al): 64.3  $\pm$  1.6%

TDS Glass (Ca, Fe): 10.2  $\pm$  0.2%

TDS Glass (Mn): 2%

Obsidian (Si, Na): 49.5  $\pm$  7.6%

Obsidian (Fe, Mg): 117  $\pm$  5%

Obsidian (Al): 198%

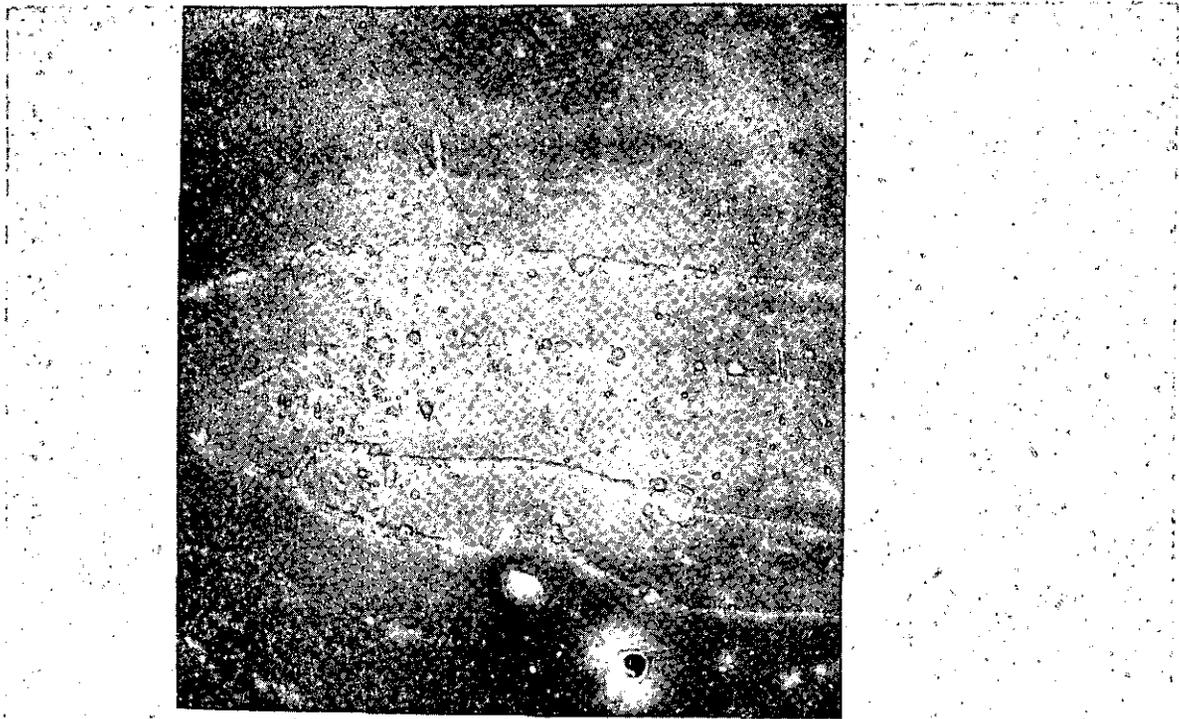
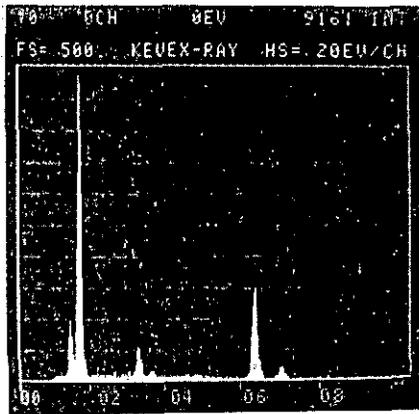


Figure 1. Obsidian Microstructure Showing Typical Laminations

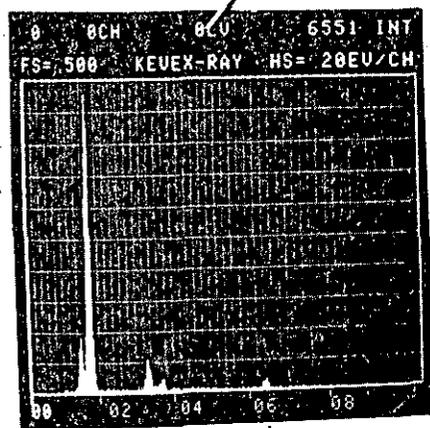


Boundary Crystal  
(0.2-0.8 $\mu$ m)

Al Ca Fe + Matrix

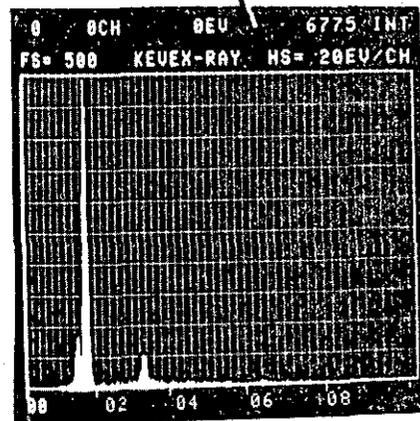


4 $\mu$ m



Matrix Crystal  
(0.1-0.2 $\mu$ m)

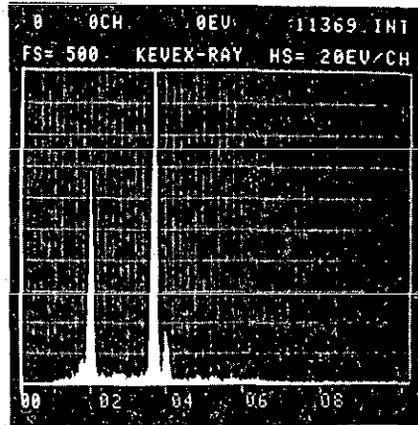
Al Ca Fe + Matrix



Matrix

Na Al Si K

Figure 2. Elemental Analyses of Principal Microstructural Features in Obsidian



P Ca

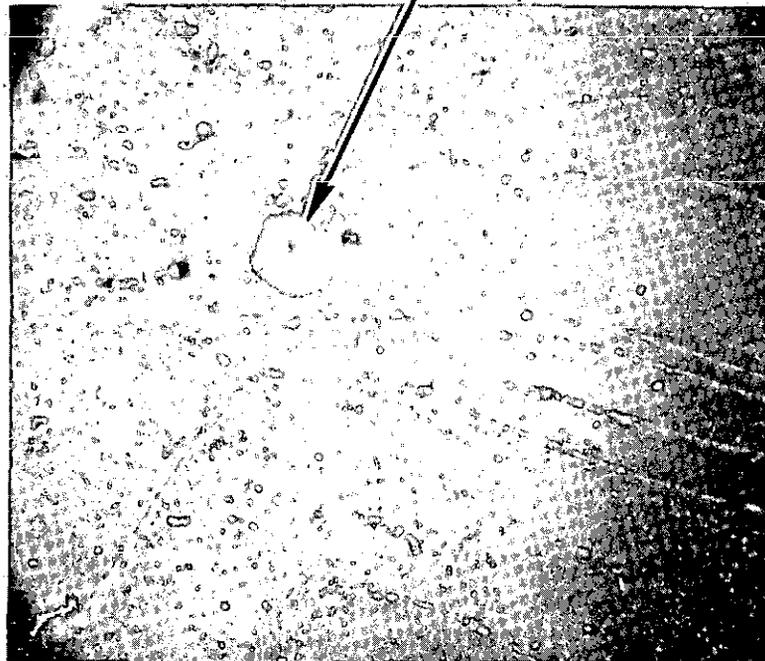


Figure 3. Occasional Hexagonal Crystal in Obsidian

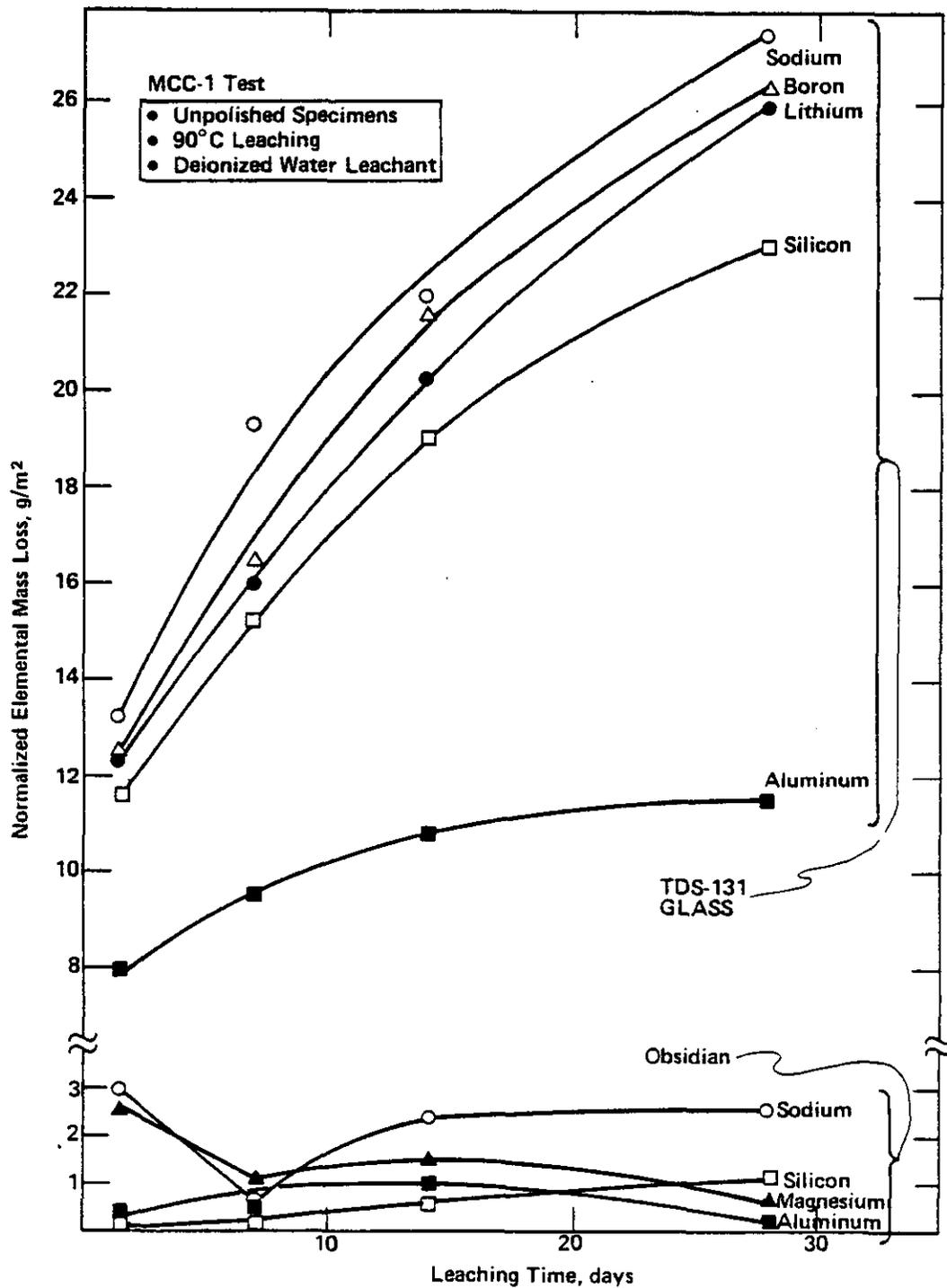


FIGURE 4. Effect of Leaching Time on Elemental Mass Loss of Glass and Obsidian

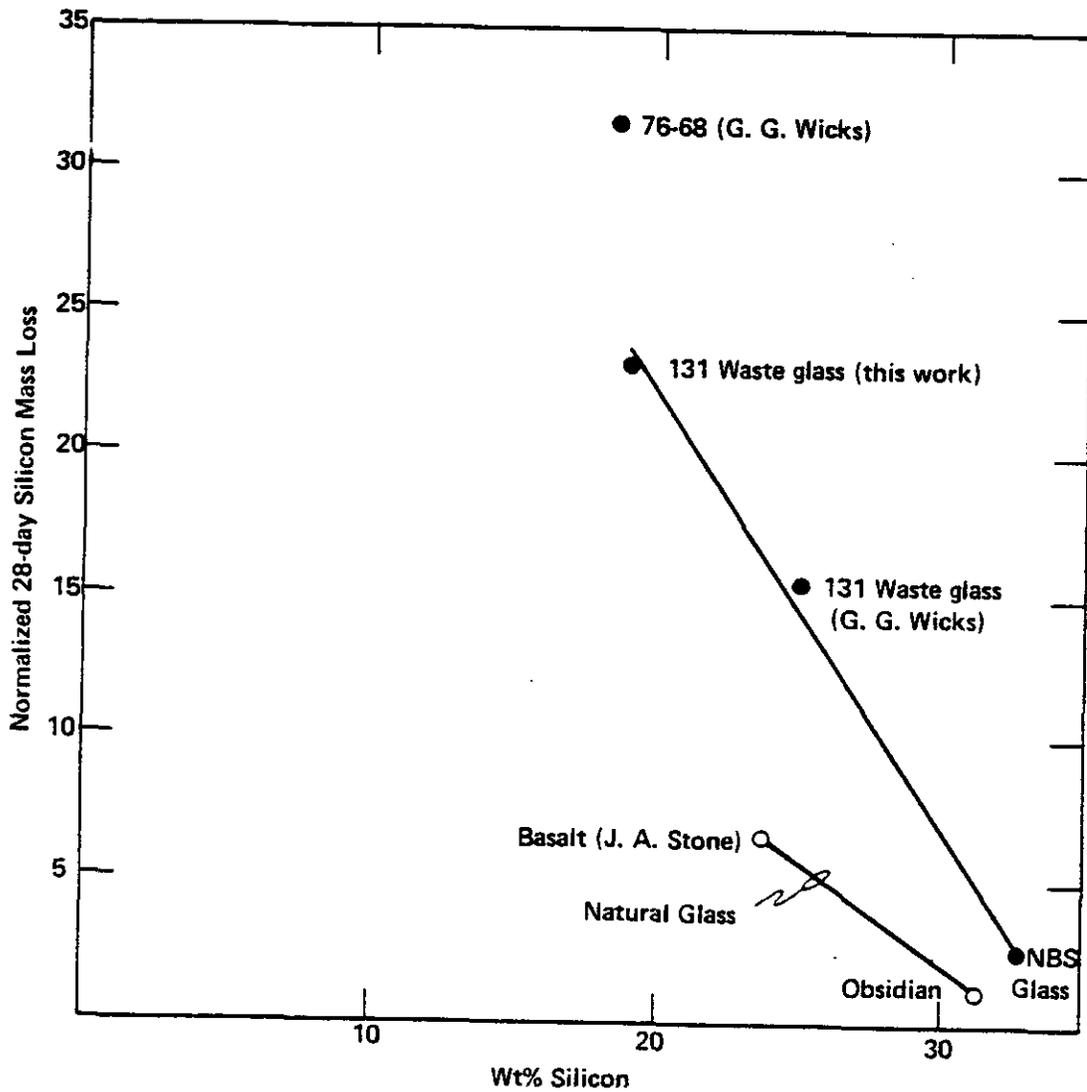


FIGURE 5. Effect on Silicon Content on Silicon Leach Rate

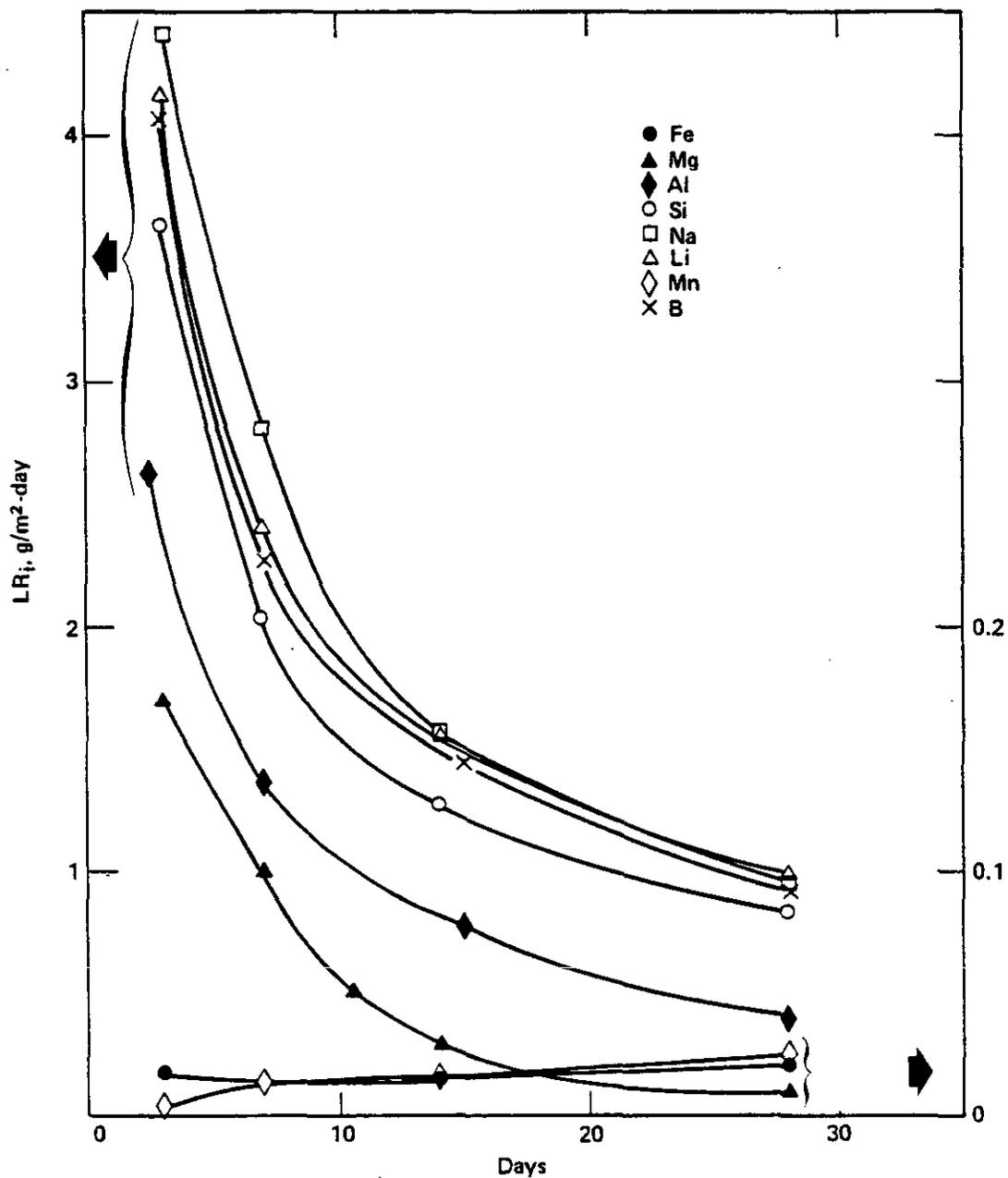


FIGURE 6. Normalized Leach Rate vs. Leach Time for Unpolished Waste Glass TDS-131

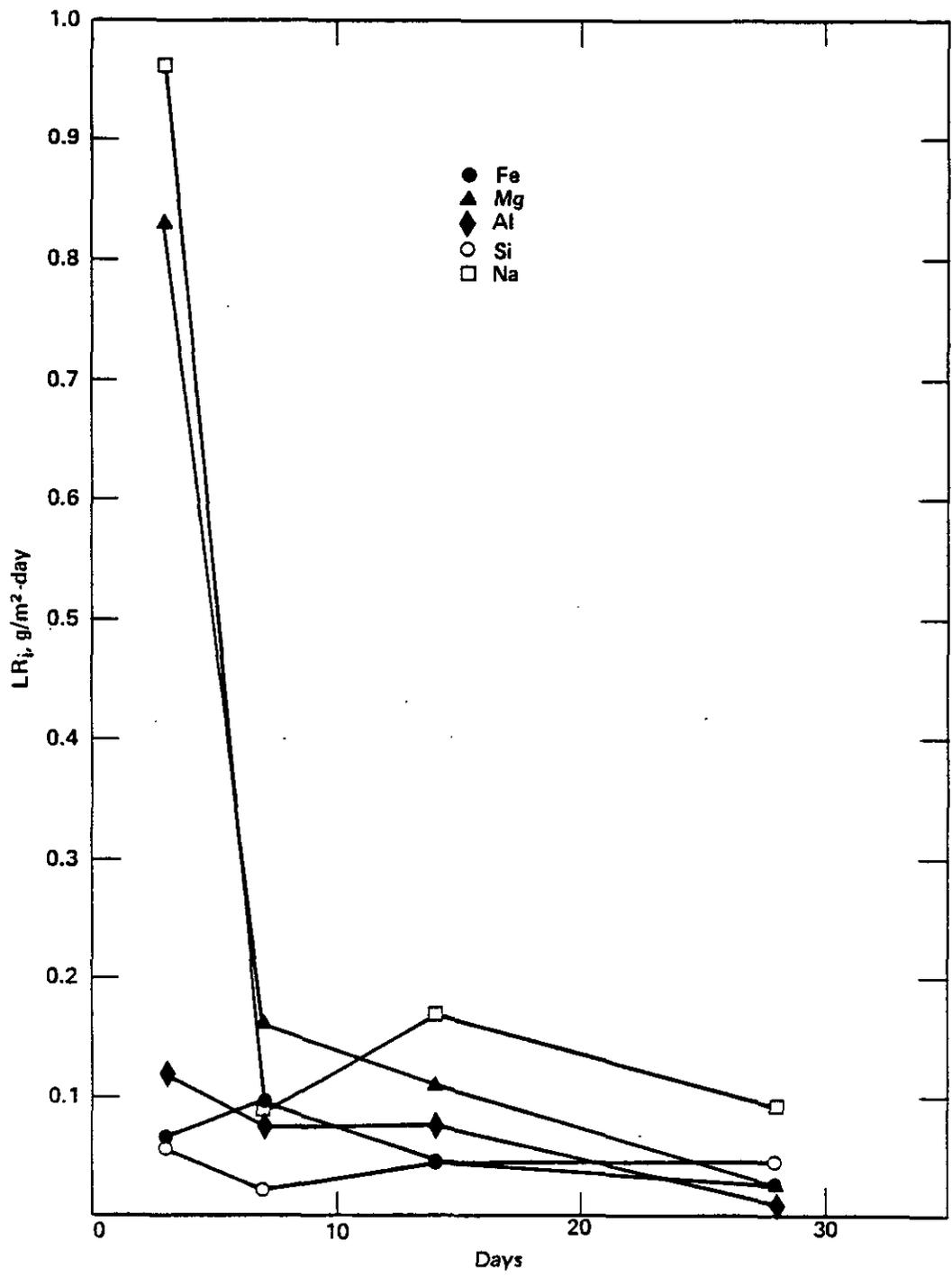
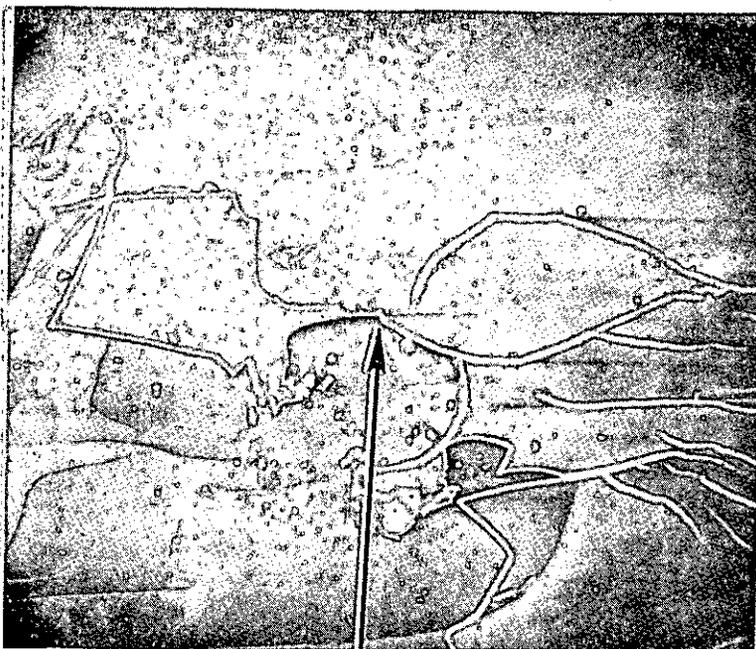
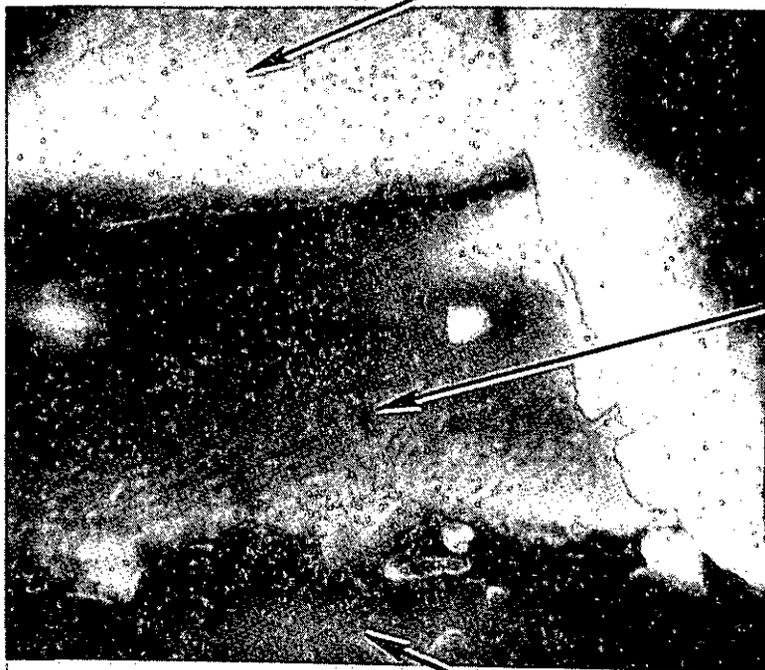
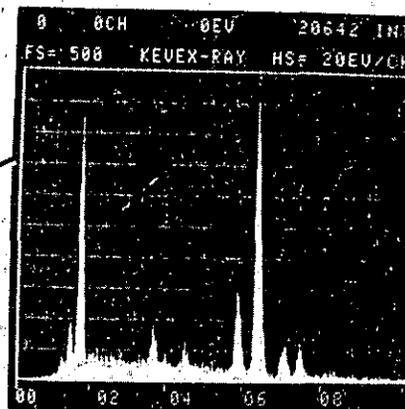


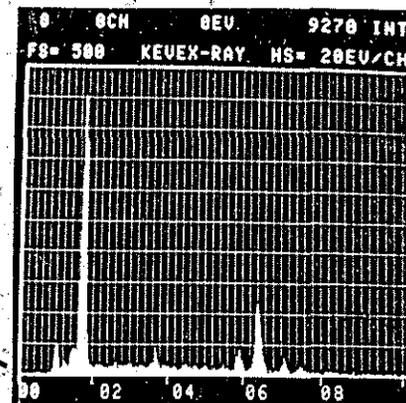
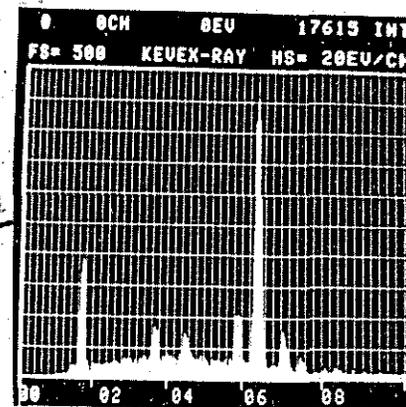
FIGURE 7. Normalized Leach Rate vs. Leach Time for Unpolished Obsidian



200 μm

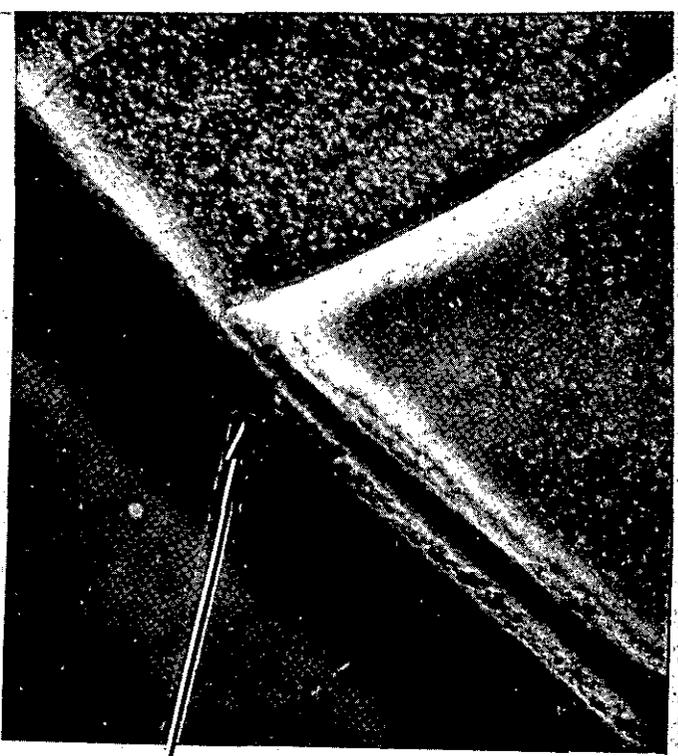
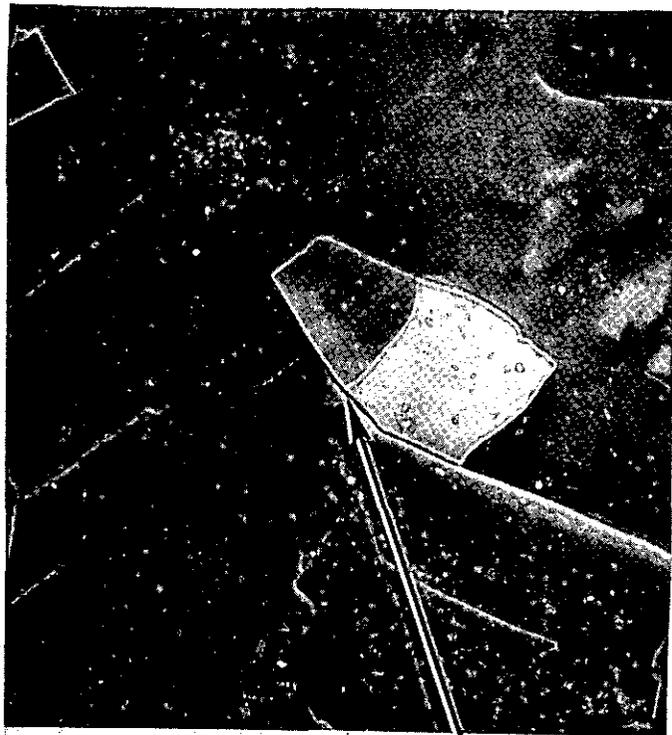


4 μm



Na Mg Al Si Ca Ti Mn Fe Ni

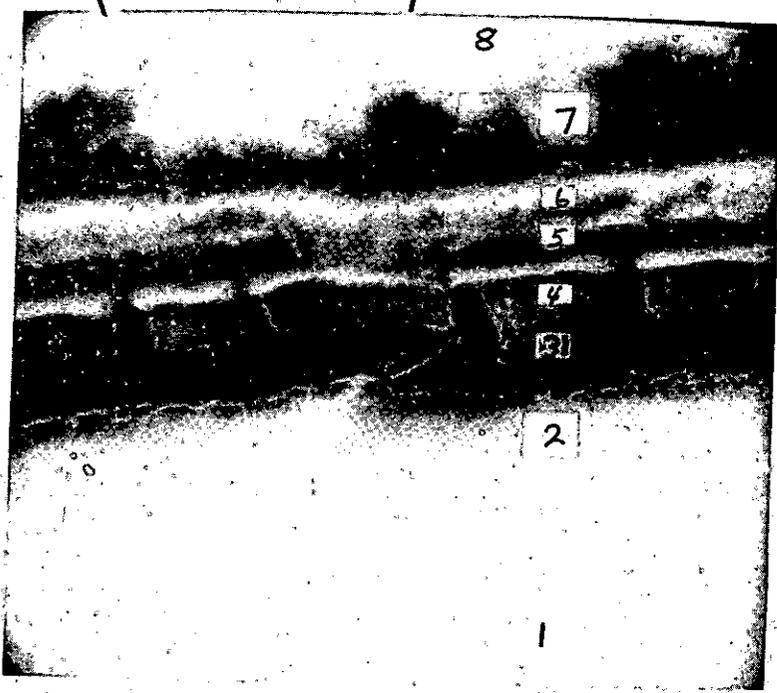
Figure 8. SEM/XES of Waste Glass After Leaching, F131/TDS 28 days, 90°C, DW, Polished



Spall Layer 1

Spall Layer 2

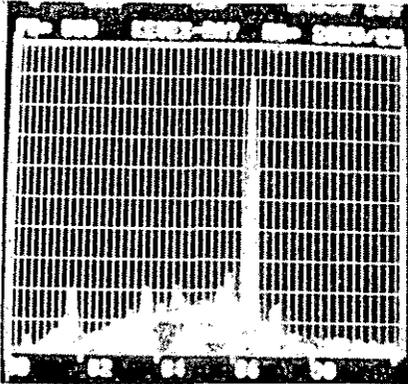
Base Glass



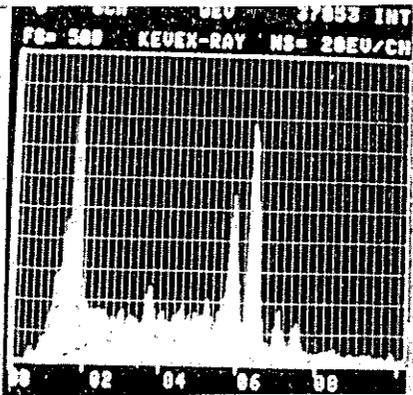
Tilted 55°

Figure 9. SEM/XES of Waste Glass After Leaching. F131/TDS 28 days, 90°C, DW, Polished

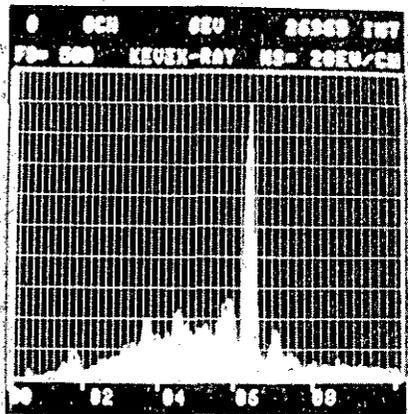
Level 4



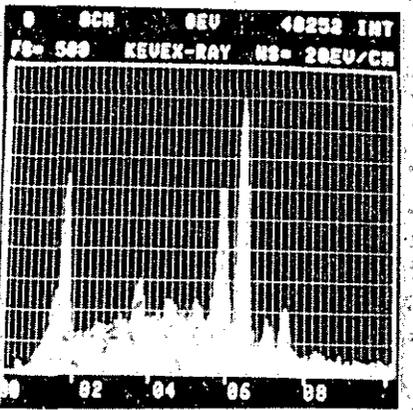
Level 8



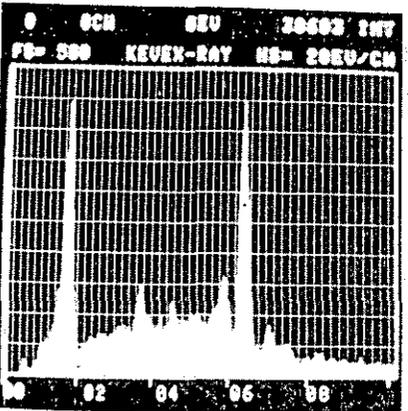
Level 3



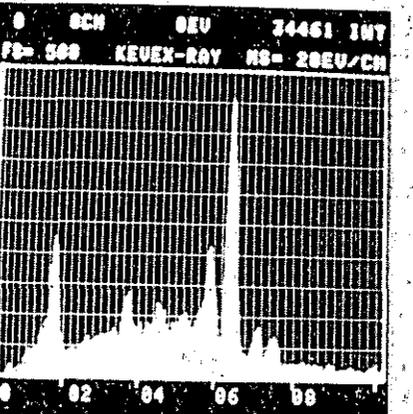
Level 7



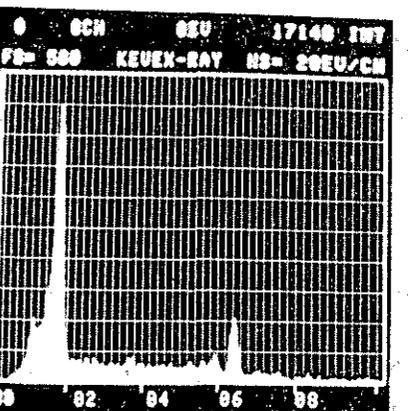
Level 2



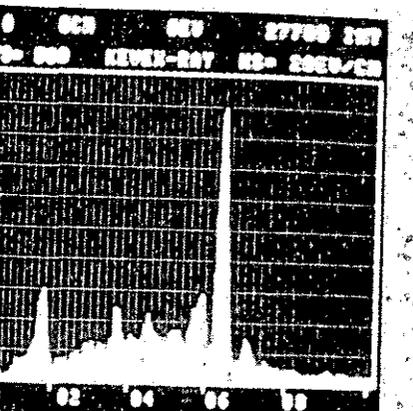
Level 6



Level 1



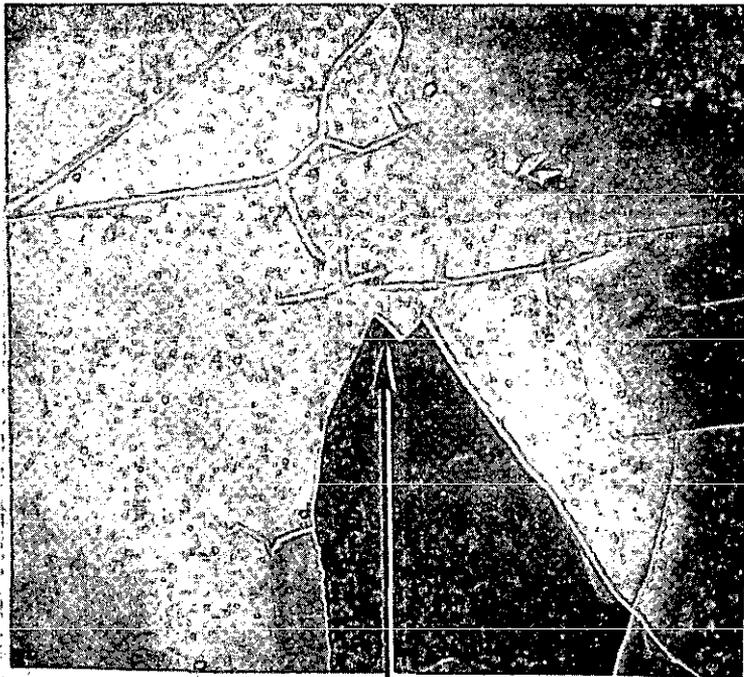
Level 5



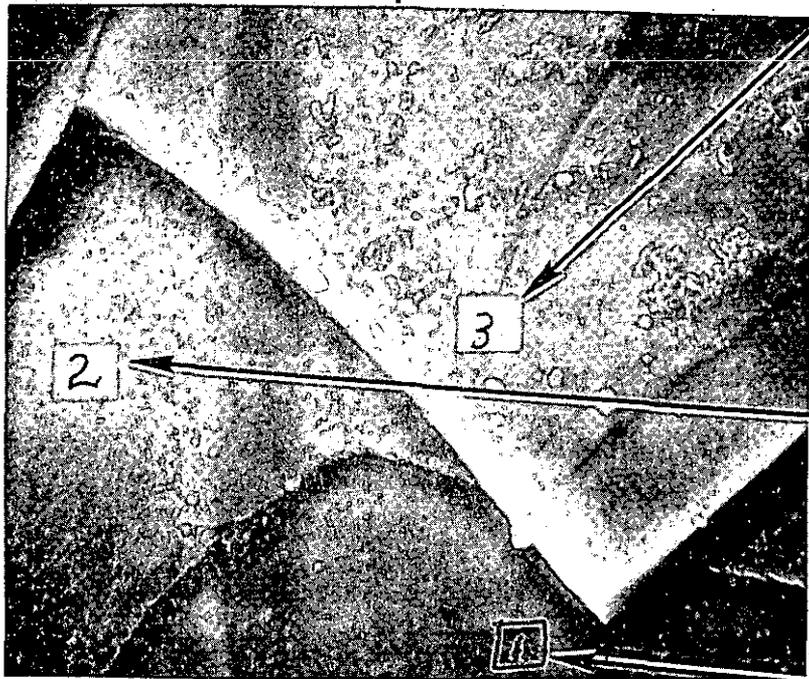
Na Mg Al Si Ca Ti Mn Fe Ni

Na Mg Al Si Ca Ti Mn Fe Ni

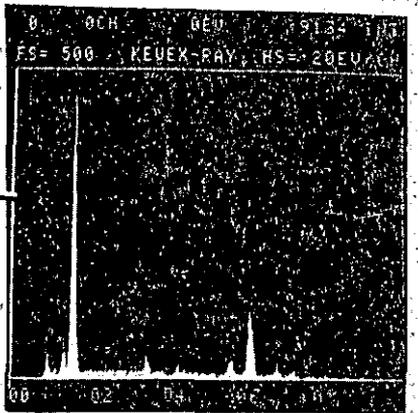
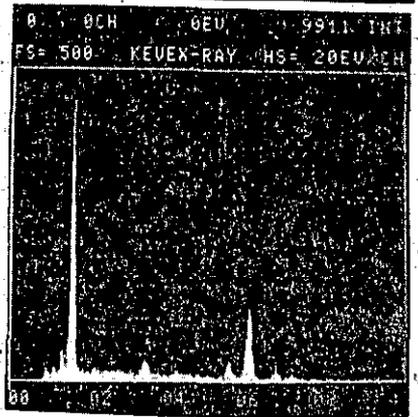
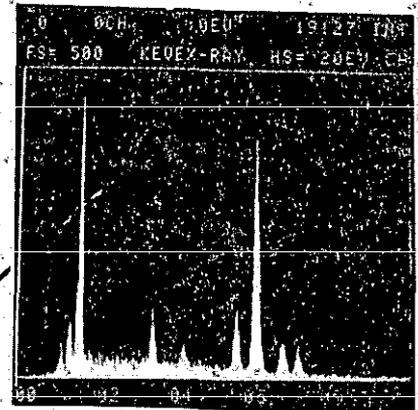
Figure 9. Continued



200 μm

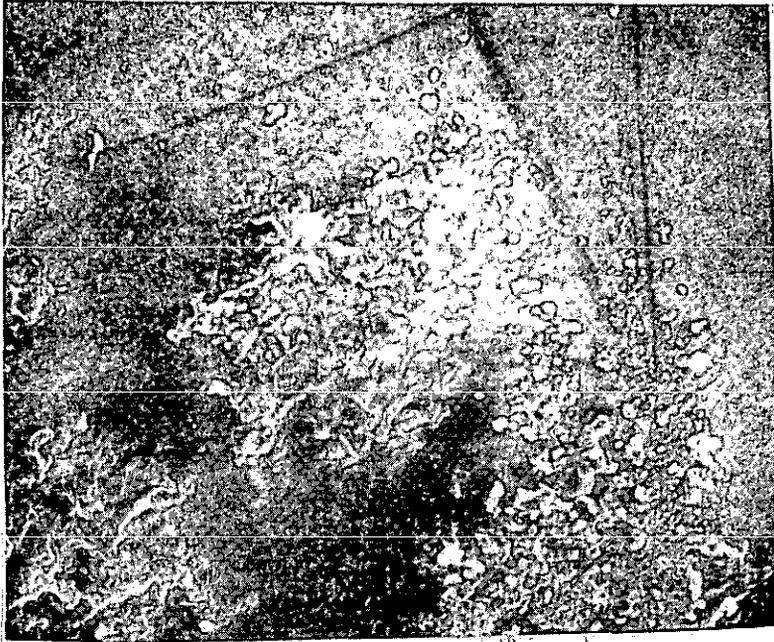


10 μm

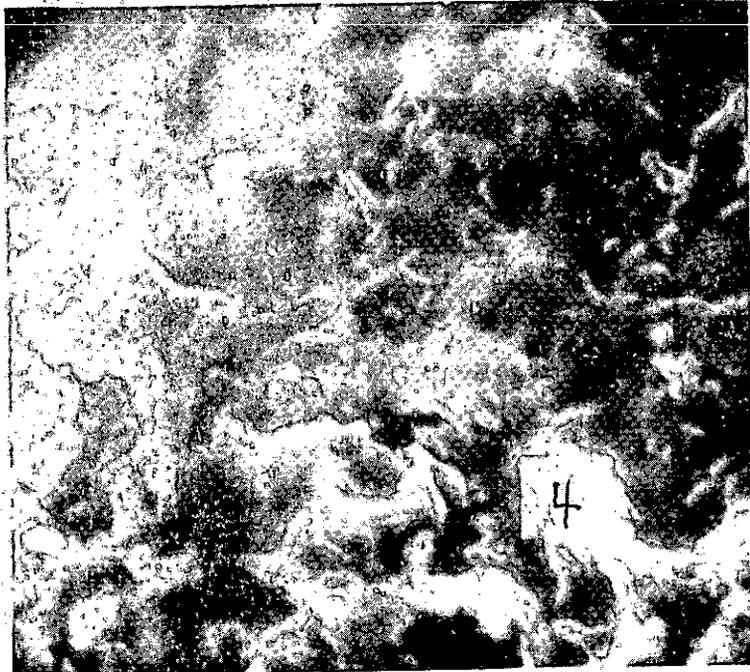


Na Mg Al Si Ca Ti Mn Fe Ni

Figure 10. SEM/XES of Waste Glass After Leaching.  
 F131/TDS, 28 days, 90°C, DW, Polished.

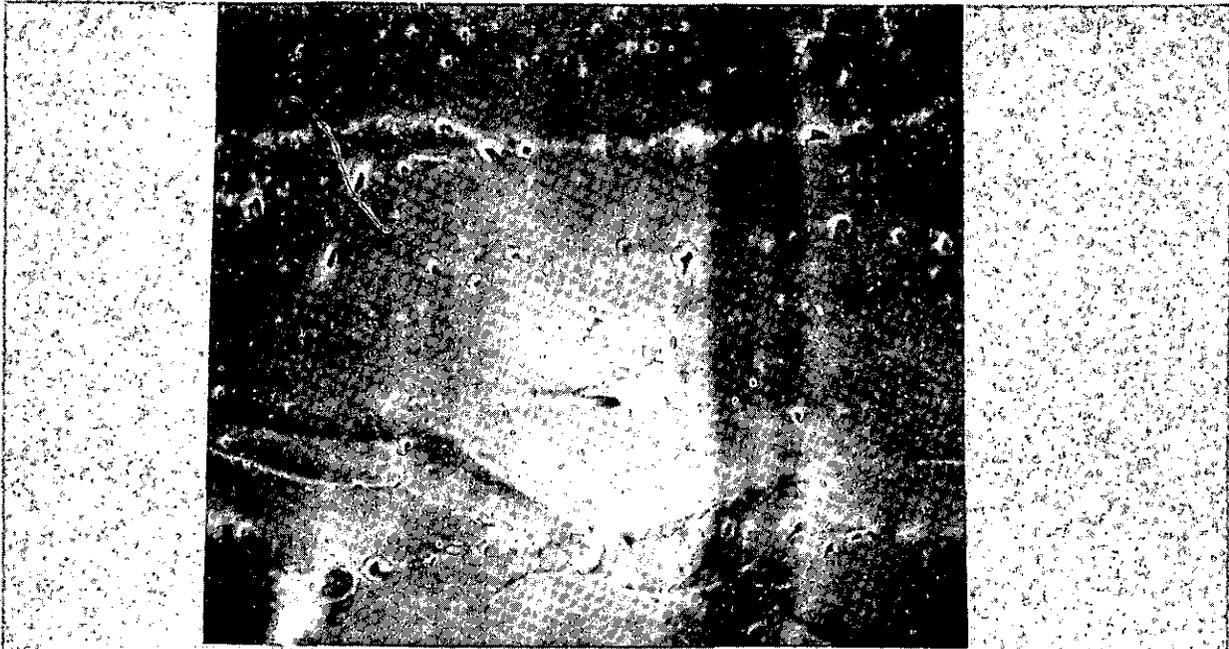


10  $\mu\text{m}$

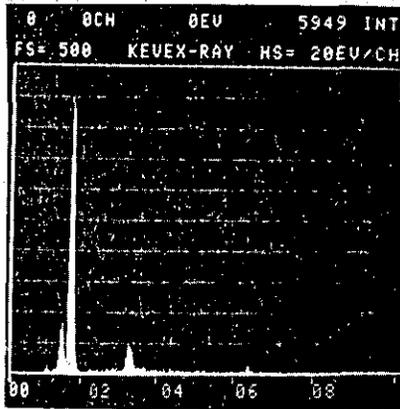


2  $\mu\text{m}$

Figure 11. Crystals in Top Layer of Leached Glass



4  $\mu$ m



Na Al Si K Ca Fe

Figure 12. Obsidian Glass Leached 28 Days, 90°C, DW, Polished

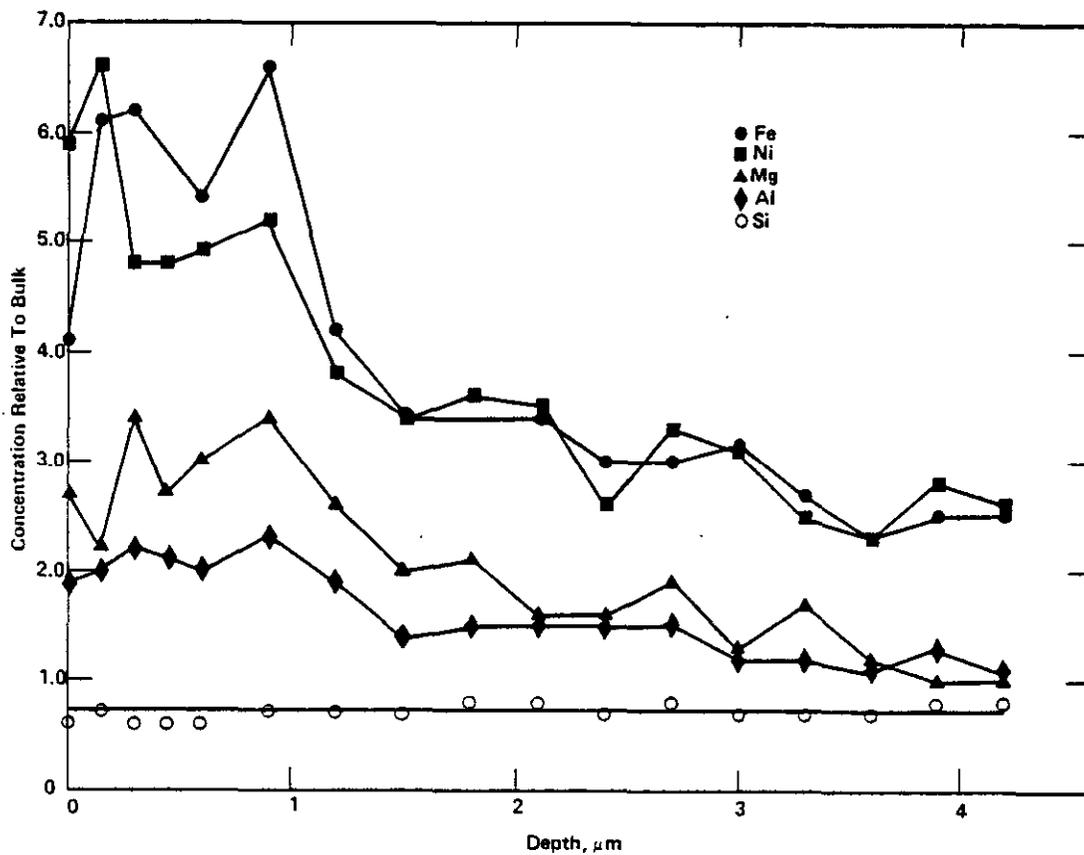


FIGURE 13. Auger Analysis of a Full Leach Layer of TDS Waste Glass Leached 28 days, 90°C, DW

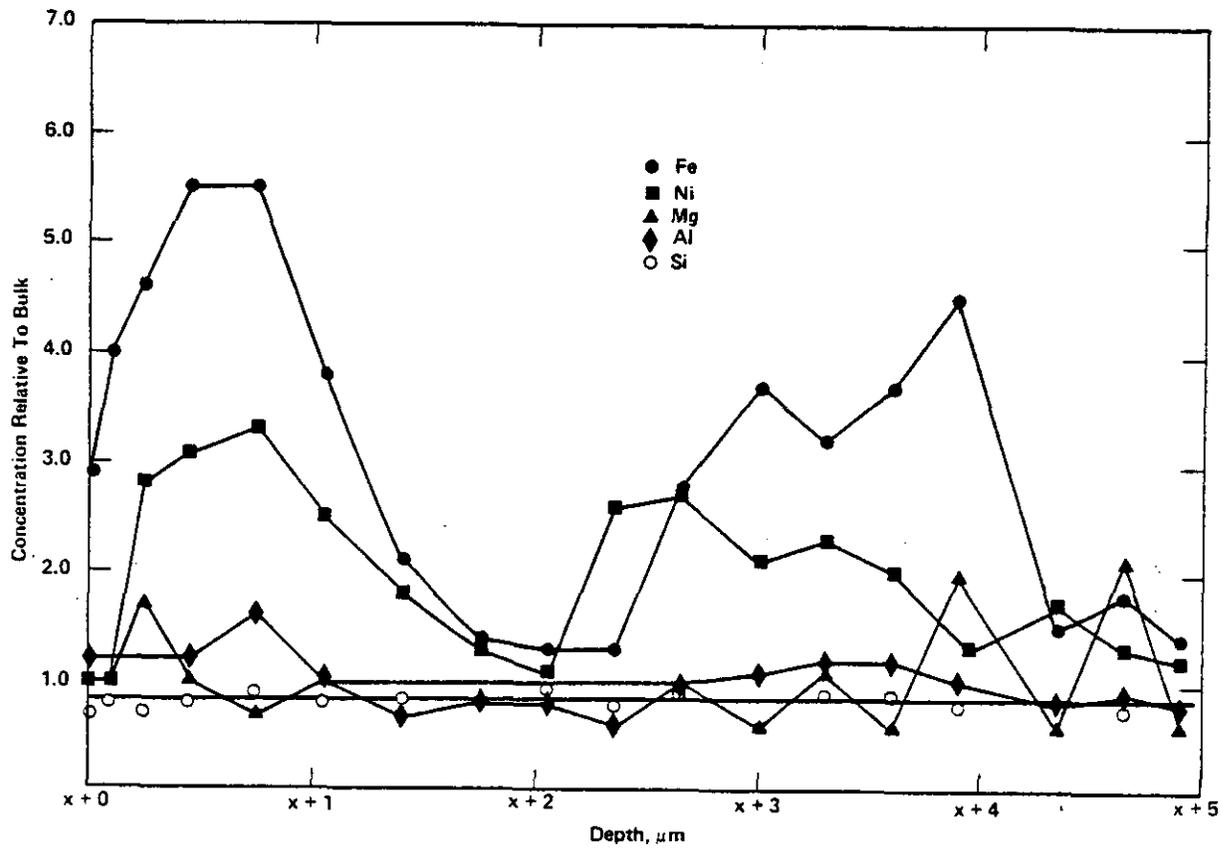


FIGURE 14. Auger Analysis of a Partial Leach Layer of TDS Waste Glass Leached 28 dys., 90°C, DW

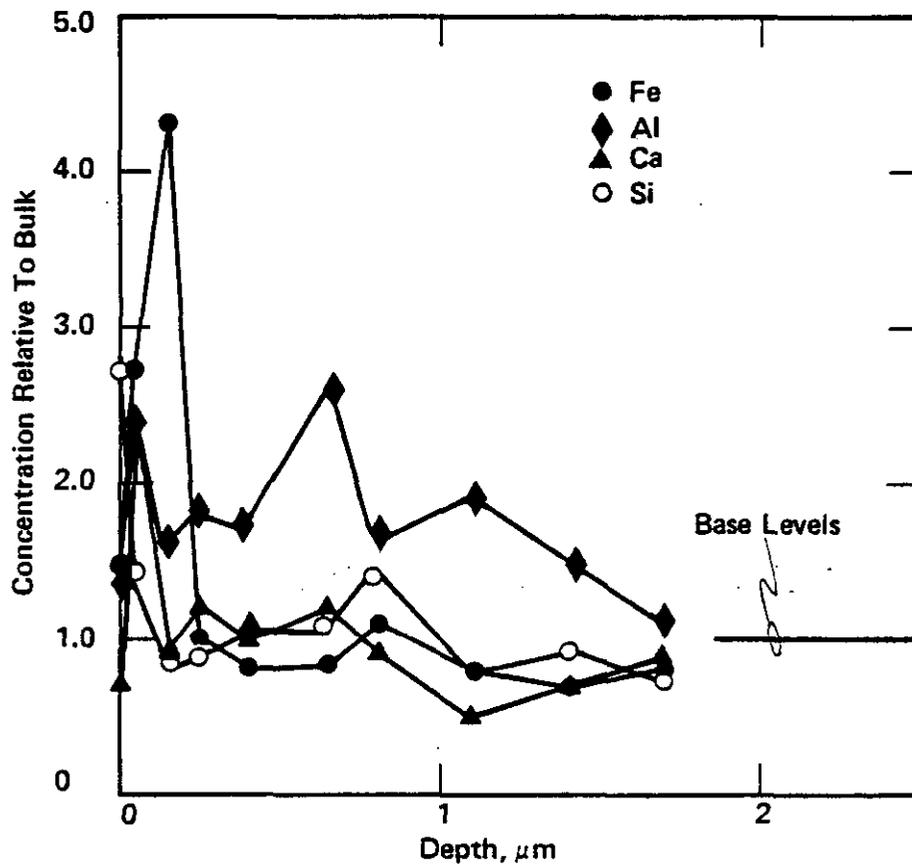


FIGURE 15. Auger Analysis of Obsidian Leached 28 days, 90°C, DW