

Glass Feasibility Study:
Vitrification of Oak Ridge National Laboratory Gunitite Waste
Using Iron Phosphate Glass

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Defense Waste Processing Technology

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1.0 Summary

This report describes the results of a glass feasibility study on vitrification of Oak Ridge National Laboratory (ORNL) Gunite waste into an Iron Phosphate glass. This glass feasibility study is part of a larger ORNL Gunite and Associated Tanks Treatability program (TTP#SR1-6-WT-31) [1]. The treatability program explores different immobilization techniques of placing Gunite waste into a glass or grout form for long term storage.

ORNL Gunite tanks contain waste that originated from years of various ORNL Research and Development programs. The available analyses of the Gunite Waste Tanks indicate, uranium and/or thorium as the dominant chemical constituent (50 % +) and Cs¹³⁷ the primary radionuclide [1]. This information was utilized in determining a preliminary iron phosphate glass formulation. Chemical and physical properties: processing temperature, waste loading capability, chemical durability, density and redox were determined.

2.0 Introduction

The research objectives of the glass feasibility study were to:

- 1) Develop an iron phosphate glass with a maximum Gunite waste oxide loading for Gunite Tanks 3 and 4.
- 2) Determine retention of Cs¹³⁷ in the glass.
- 3) Assess glass properties.

The iron phosphate glass formulation was based on a glass composition developed at the University of Missouri-Rolla [2]. This glass family has also been used at Savannah River Technology Center (SRTC) for plutonium vitrification [3].

Fifteen 50 gram batches of glass and five 100 gram batches of glass were melted. Ten 50 gram batches of glass were made as proof of glass formation. Adjustments were made to the glass composition were based on the results from X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). Several 50 gram batches of glass were then melted to determine the maximum Gunite waste loading (oxide basis).

The waste oxide loading in the iron phosphate glass was determined by completing five 50 gram melts. Melts casted differed from 15 weight percent to 40 weight percent oxide basis. Upon visual inspection of the 40 weight percent oxide loading glass, undissolved material was found in the melt. No undissolved material was detected by visual inspection of the 15 through 35 weight percent Gunite waste loading glasses. XRD and SEM confirmed vitreous products had been made for the 15 to 35 weight percent oxide loading glasses. The batch size was increased to 100 grams of glass and

five melts were completed. These glasses, containing up to a 35 weight percent oxide Gunite loading, were submitted for elemental analyses.

A different Iron Phosphate formulation was tested on two 50 gram batches containing a 40 and 45 weight percent (oxide basis) Gunite loading. These glasses were submitted for analyses. The analytical results from these glasses have not been reported yet. A revision will be made as soon as the data is available.

To assess the viability of the glass the following properties were determined:

- 1) Processing Temperature
- 2) Waste Loading Capability
- 3) Chemical Durability
- 4) Density
- 5) Redox

3.0 Glass Preparation

The preliminary Gunite waste composition utilized in the iron phosphate glass was provided by chemical analyses of a sample from a waste stream. The analyses of the waste stream indicated uranium as the predominant component (based on elemental wt.%) and Cs¹³⁷ as the primary radionuclide targeted for retention [4]. The preliminary Gunite waste composition is listed in Table 1.0. The components are listed in descending order based on oxide weight percent.

A Gunite simulant was made from the waste composition leaving uranium and organic carbon components out. Uranium oxide (UO₃) and activated carbon were selected as suitable components for the Gunite simulant (based materials used during previous ORNL campaigns). Iron phosphate glass was batched by mixing uranium oxide (UO₃), gunite simulant, activated carbon and glass formers together. Constituents of the iron phosphate glass were both wet and dry batched and placed in a furnace for melting (wet batched constituents were dried before melting).

During the initial batching of the glass (50 gram batches), undissolved material appeared in the melt. XRD and SEM identified the undissolved material as sodium iron phosphate and aluminum phosphate. A review was performed of the spreadsheet and an error was discovered which caused too much sodium to be added to the glass. The error was corrected and the glasses were rebatched. No undissolved material in the melt was observed visually. These glasses were then resubmitted for XRD and SEM analyses. No crystals were detected by XRD or SEM instrumentation.

After the initial successful melts were made, the maximum waste loading was determined. A maximum waste loading of 35 wt.% was achieved with no undissolved material appearing in the melt (confirmed by XRD and SEM). Five 100 gram batches of glass were dry batched and melted having 0 up to 35 wt.% Gunite waste loading. The zero weight percent Gunite waste loading glass was batched to have a standard to compare to the other glasses to. The targeted waste loadings were 0, 20, 25, 30 and 35 weight percent. XRD patterns of these glasses appear in Figures 1.0 - 4.0.

Table 1.0**Preliminary Gunite Waste Composition**

Component	Mass (kg)	Wt% (solids)
UO ₃	1846	78
Na ₂ O	286	12
Al ₂ O ₃	168	7
P ₂ O ₅	19	0.8
CaO	17	0.75
Fe ₂ O ₃	13	0.54
*Other	55	0.91

Note: Cs¹³⁷ is the primary radionuclide

* Remaining components added together.

Figure 1.0

XRD for 20 wt.% Gunitite Loading

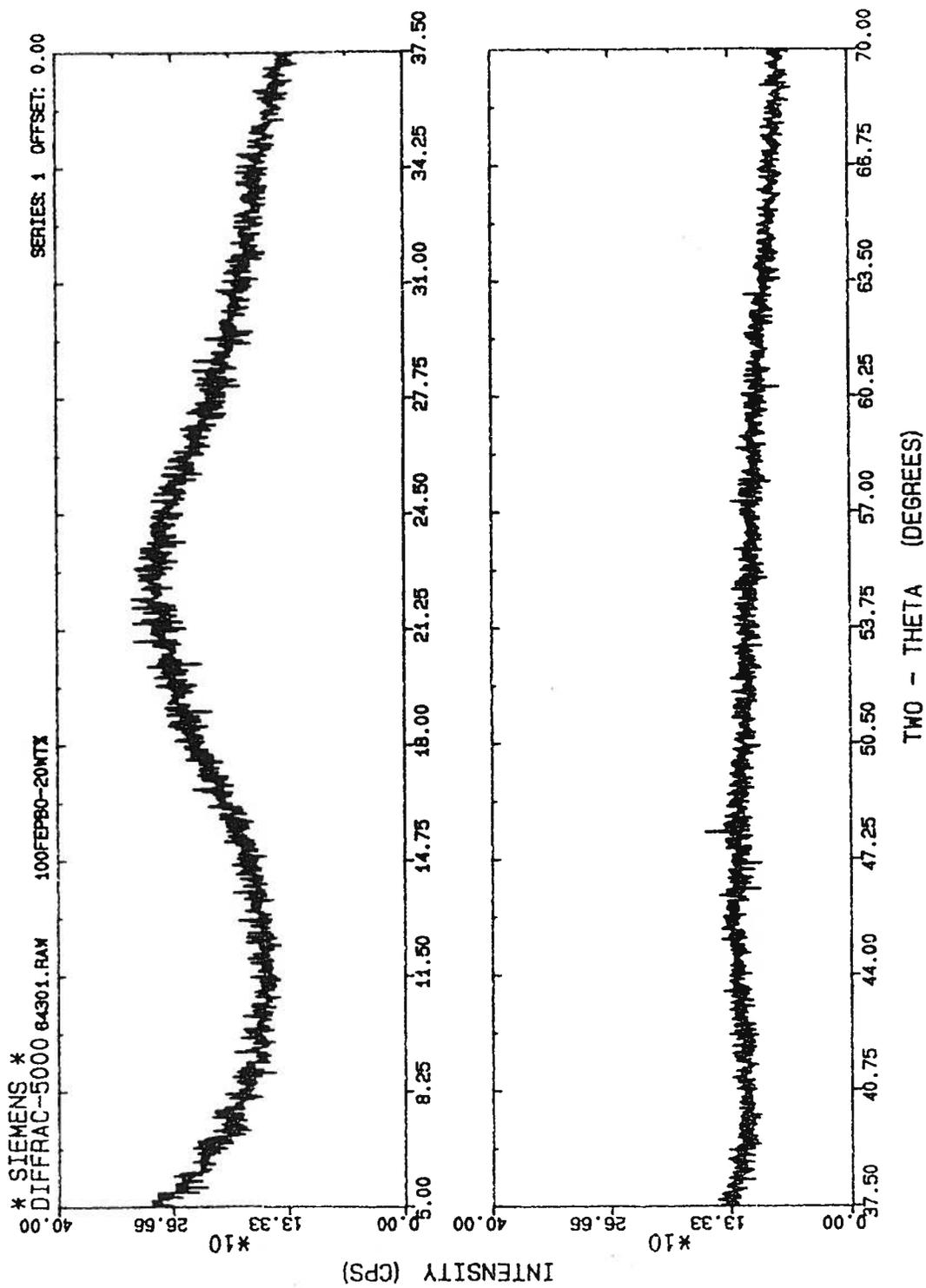


Figure 2.0

XRD for 25 wt.% Gunite Loading

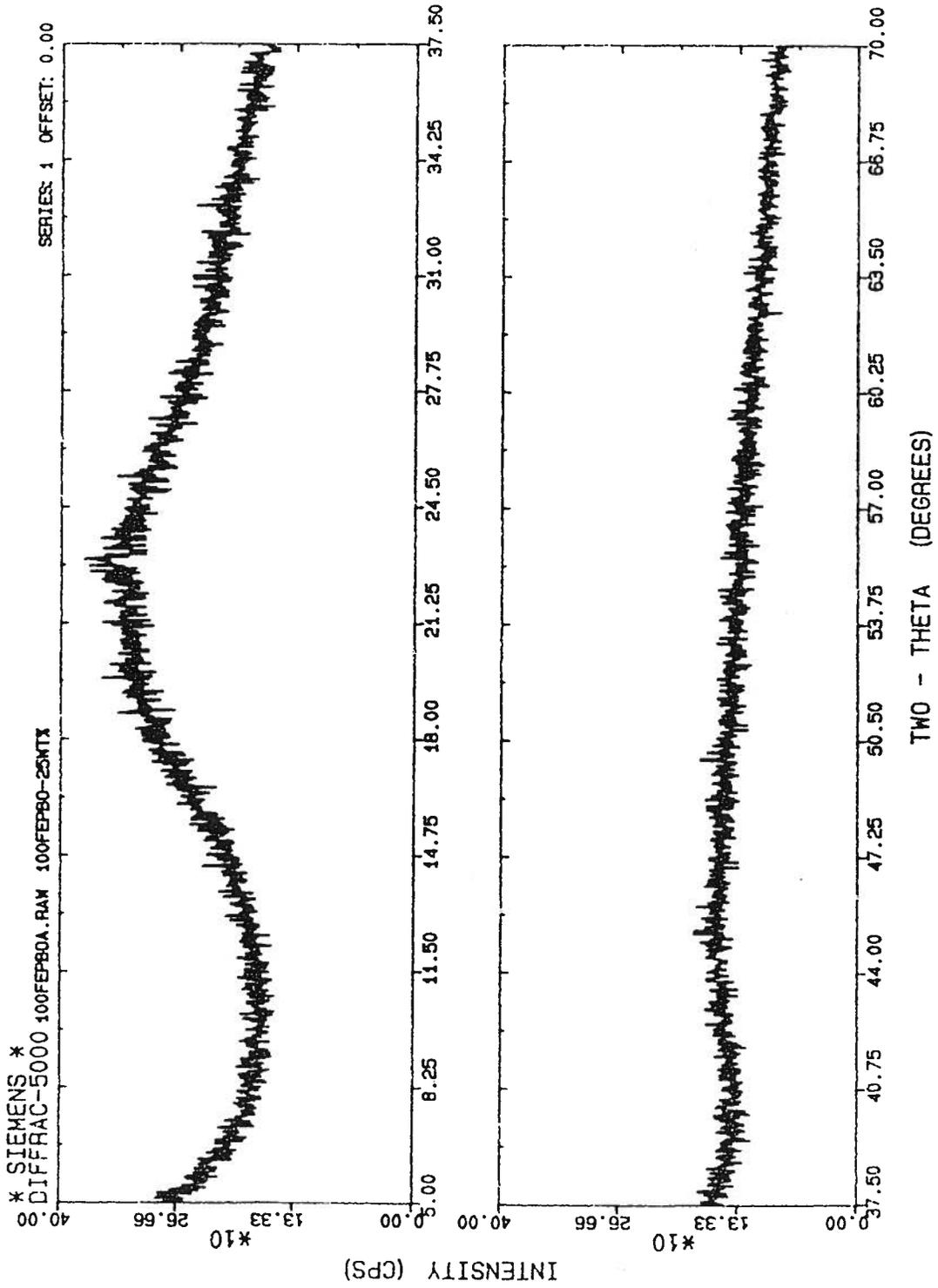


Figure 3.0

XRD for 30 wt.% Gunite Loading

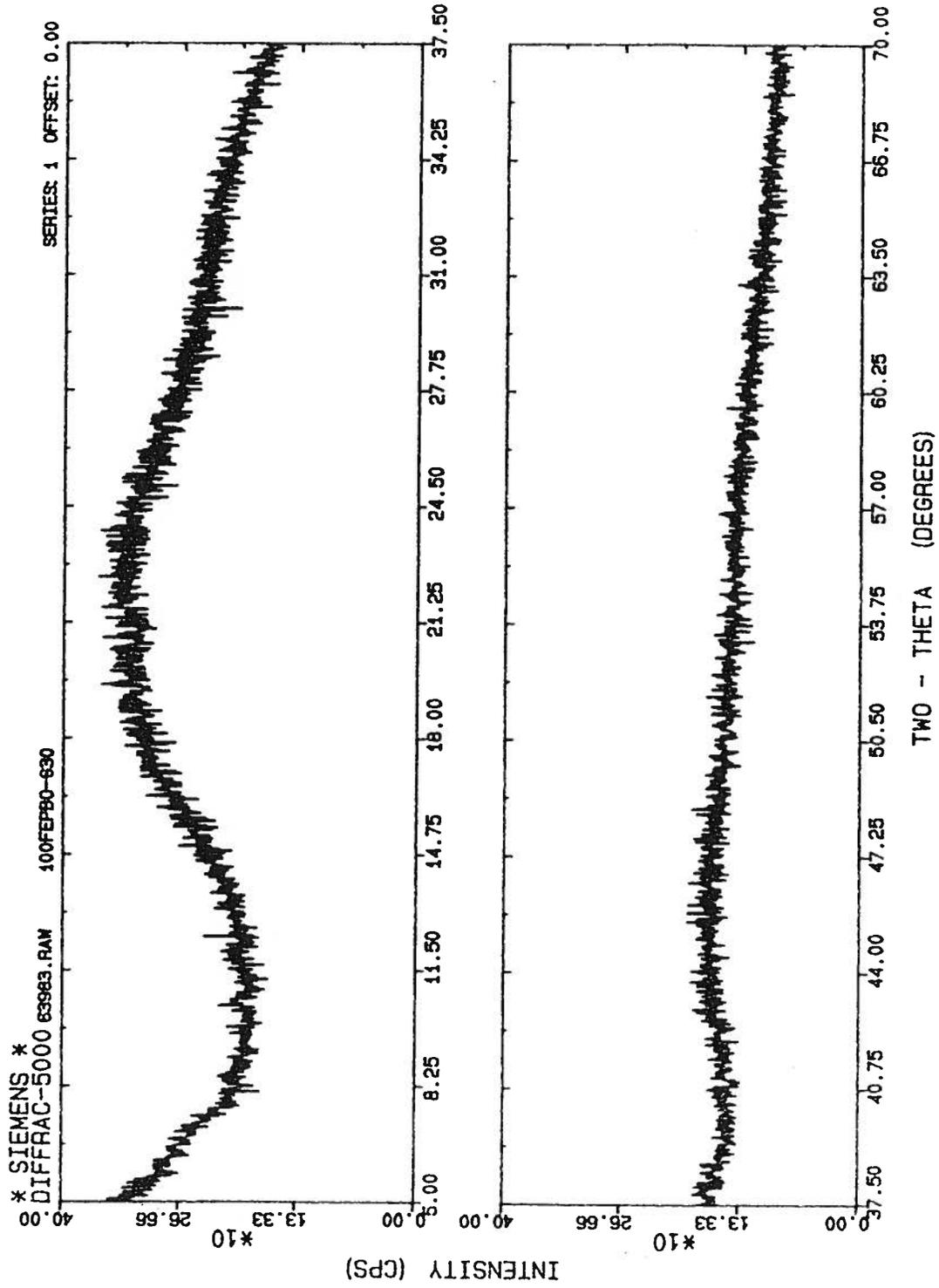
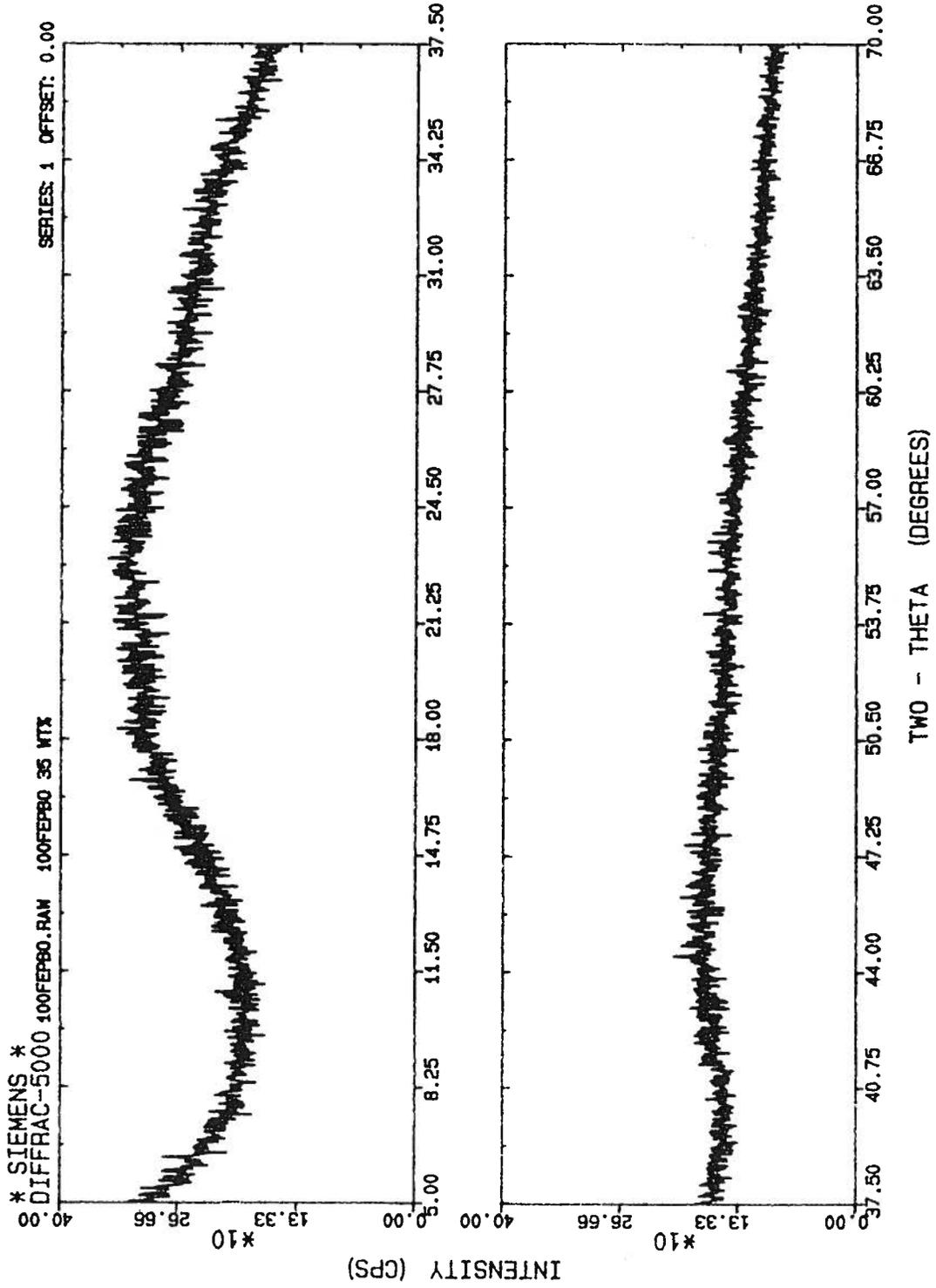


Figure 4.0

XRD for 35 wt.% Gunite Loading



4.0 Glass Melting

Thermolyne[®], CM High Temperature - Model 1712 and a Applied Technology Systems furnaces were utilized to perform all of the iron phosphate melts. The melting temperature of the glass ranged between 1050°C and 1100°C (Thermolyne[®] furnaces were kept below 1100°C). All 100 gram melts were dry batched and placed into labeled 250 ml alumina crucibles.

The ramp rate for the furnaces was approximately 14°C per minute. This ramp rate was determined after the first five 50 gram melts were completed. It was necessary to ramp past 250°C as quickly as possible, to minimize the sublimation of phosphorous pentoxide at that temperature[6]. The melts were held at 1050°C for approximately three hours to ensure glass homogeneity before removal from the furnace.

After three hours, the melts were removed from the furnace and poured into graphite molds. The graphite molds were placed in a preheated annealing furnace at 500°C for approximately one hour. After cooling, the melts were visually inspected for crystal growth. To confirm that vitreous character, the melts were sent for XRD.

Visual observations of the alumina crucibles detected no signs of pitting or cracking. The melt line of the crucible was also observed after breaking the crucible in half, no corrosion or cracking was observed.

5.0 Devitrification Study

A simple devitrification study was performed on 30 weight percent oxide Gunitite waste loading piece of glass. This piece of glass was melted at 1050°C in a Thermolyne[®] furnace for three hours with a ramp rate of 14°C per minute. The glass was poured into a graphite mold and allowed to cool for a few seconds. The glass piece was then placed on a alumina crucible lid and placed into a Thermolyne[®] furnace preheated to 700°C. After 24 hours, the piece of glass was removed from the furnace and visually observed for any crystal growth. The glass was submitted for XRD to determine if the product was still vitreous. No crystal growth was detected by the XRD instrumentation. Figure 5.0 shows the XRD pattern for this piece of glass.

6.0 Cs¹³⁷ Retention Study

A Cs¹³⁷ retention study was performed on two melts to determine the retention of Cesium in the glass. Glasses containing a 30 and 35 weight percent Gunitite loading were wet batched and spiked with Cs¹³⁷ and allowed to dry overnight in a 90°C preheated CM High Temperature - Model 1712 furnace. The furnace heating schedule used for these melts appears on the next page:

Table 2.0**Furnace Heating Schedule**

<u>Ramp</u>	<u>Temperature</u>	<u>Rate</u>
1.	90°C - 300°C	3.5°C/min
2.	300°C (dwell)	0.5 hour
3.	300°C - 500°C	6.7°C/min
4.	500°C (dwell)	3.0 hours
5.	500°C - 700°C	6.7°C/min
6.	700°C (dwell)	2.0 hour
7.	700°C - 900°C	6.7°C/min
8.	900°C (dwell)	2.0 hours
9.	900°C - 1150°C	8.3°/min
10.	1150°C(dwell)	2.0 hours

This study was conducted at a higher temperature (1150°C vs. 1100°C) for the iron phosphate glass to provide a preliminary conservative test of Cs retention. The melts were removed from the furnace after dwelling at 1150°C for three hours. They were poured into graphite molds and placed in the annealing furnace preheated to 500°C for one hour. They were removed from the annealing furnace after one hour, and allowed to cool at room temperature. The glass was submitted for XRD, Rad Screens and ICP analyses. The XRD instrumentation detected no crystal growth, Rad Screen and the ICP results are yet to be reported. The XRD for this glass is found in Figure 6.0 and 7.0. A revision will be made as soon as the data is available.

7.0 Analyses**7.1 Chemical Analyses**

Glass samples from the five 100 gram and two 50 gram glass samples were submitted for elemental analysis by Inductively Coupled Plasma. The solutions used for ICP analysis were prepared by two separate dissolution procedures to provide an independent comparison. The two methods used for the dissolutions were the Sodium Peroxide Fusion and the Microwave - Acid [5].

Each method of dissolution has certain disadvantages associated with it and these are noted below.

Sodium Peroxide Fusion

1. This method cannot be used for determining sodium (sodium peroxide addition), nickel (crucible material) or zirconium (crucible material).
2. The high concentration of acid and dissolved solids may affect the performance of the nebulizer in ICP determinations.

Microwave - Acid

1. This method cannot be used for determining boron (boric acid addition) and silicon (ICP torch and spray chamber fabricated of silicon containing materials).
2. This method is also not recommended for analyses of rare earth elements (due to the formation of fluoride precipitates).

Tables 3.0 through 7.0 present the results obtained from the ICP analysis for the five 100 gram glass samples. These tables compare the normalized target oxide composition to analyzed oxide composition numbers (normalized) for the glass. Six major components of the glass having a concentration greater than 0.20 wt% are recorded in these tables.

The percent error ranges for each of the six components for the different Gunite waste loadings are listed below.

<u>Component</u>	<u>Range</u>
1. P ₂ O ₅	+1.00 to -6.00
2. Na ₂ O	-0.60 to +14.00
3. Fe ₂ O ₃	+3.00 to +16.00
4. CaO	+12.00 to +43.50
5. Al ₂ O ₃	+9.40 to +34.00
6. UO ₃	-11.70 to +6.00

The errors recorded for the glasses could be contributed to analytical and batching errors. However, the results recorded for the 35 wt%(oxide) Gunite loading do not trend the like the others. There could be possible analytical errors leading to the unusual trend of these numbers. Removing the percent errors contributed by the 35 wt% (oxide) Gunite loading, one can see the range of error becomes smaller. The error recorded for the Aluminum oxide remained high even though the results of the 35 wt% Gunite loading were removed. Macroscopic dissolution of the crucible was detected, however.

<u>Component</u>	<u>Range</u>
1. P ₂ O ₅	-1.95 to -6.00
2. Na ₂ O	-0.60 to +7.70
3. Fe ₂ O ₃	+3.00 to +8.40
4. CaO	+12.00 to +19.00
5. Al ₂ O ₃	+33.00 to +34.00
6. UO ₃	-0.70 to +6.00

Figure 8.0 shows a graph of the different components plotted as the Gunite waste loading is increased. The graph shows ferric oxide in the glass is decreasing as the Gunite waste loading is increasing. This trend is noticeable, because the Gunite waste is replacing the iron oxide (phosphorous pentaoxide being held constant when possible) in the glass. Therefore, there should be an increase in sodium oxide, aluminum oxide, uranium oxide and calcium oxide. This trend is confirmed by the graph. Calcium oxide is not plotted on this graph due to this component making up only 0.23 wt% in the glass.

Figure 5.0

XRD for 30 wt.% Gunitite Loading - Devitrification Study

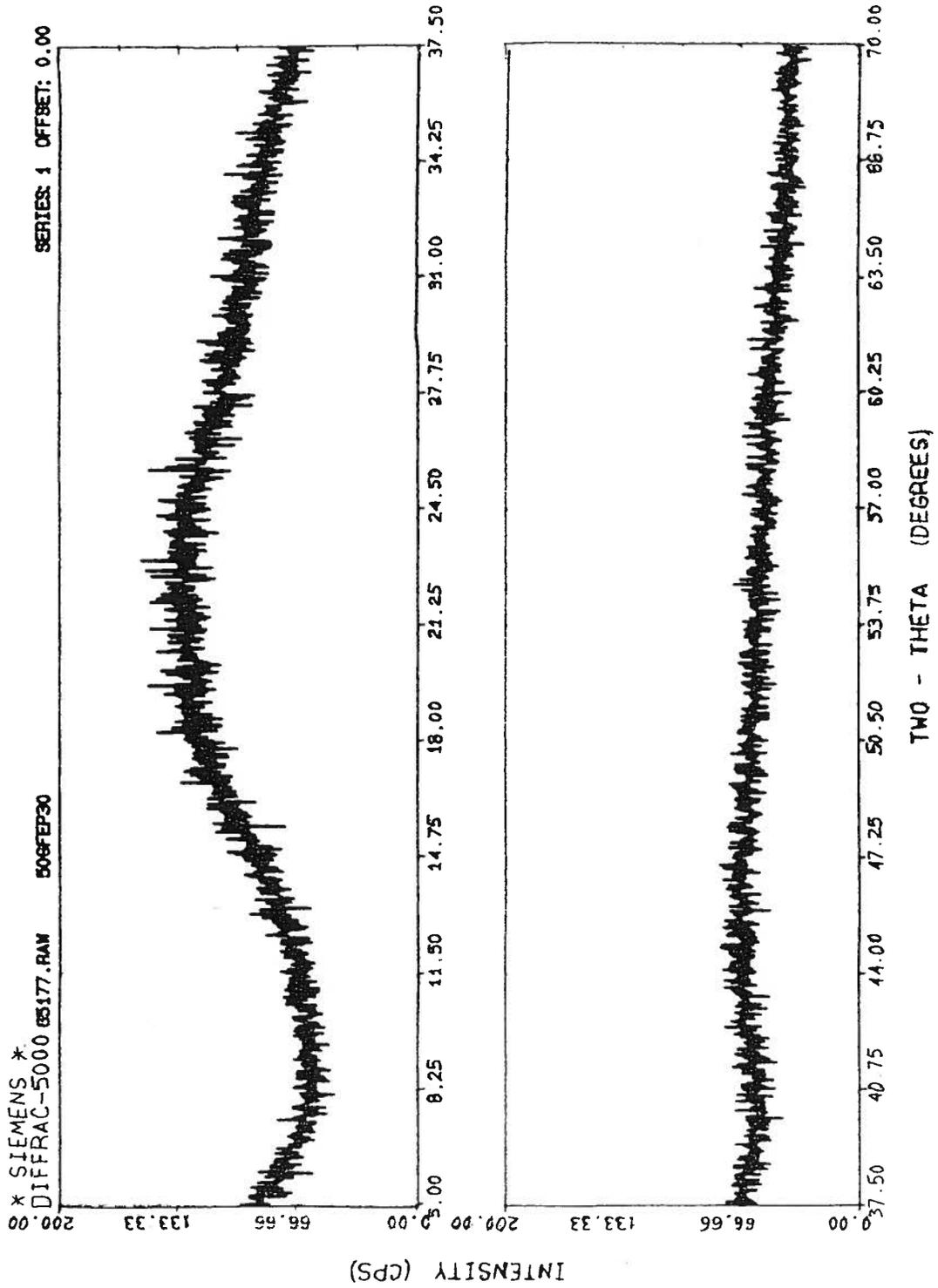


Figure 6.0

XRD for 30 wt.% Gunite Loading - Cs¹³⁷ Study

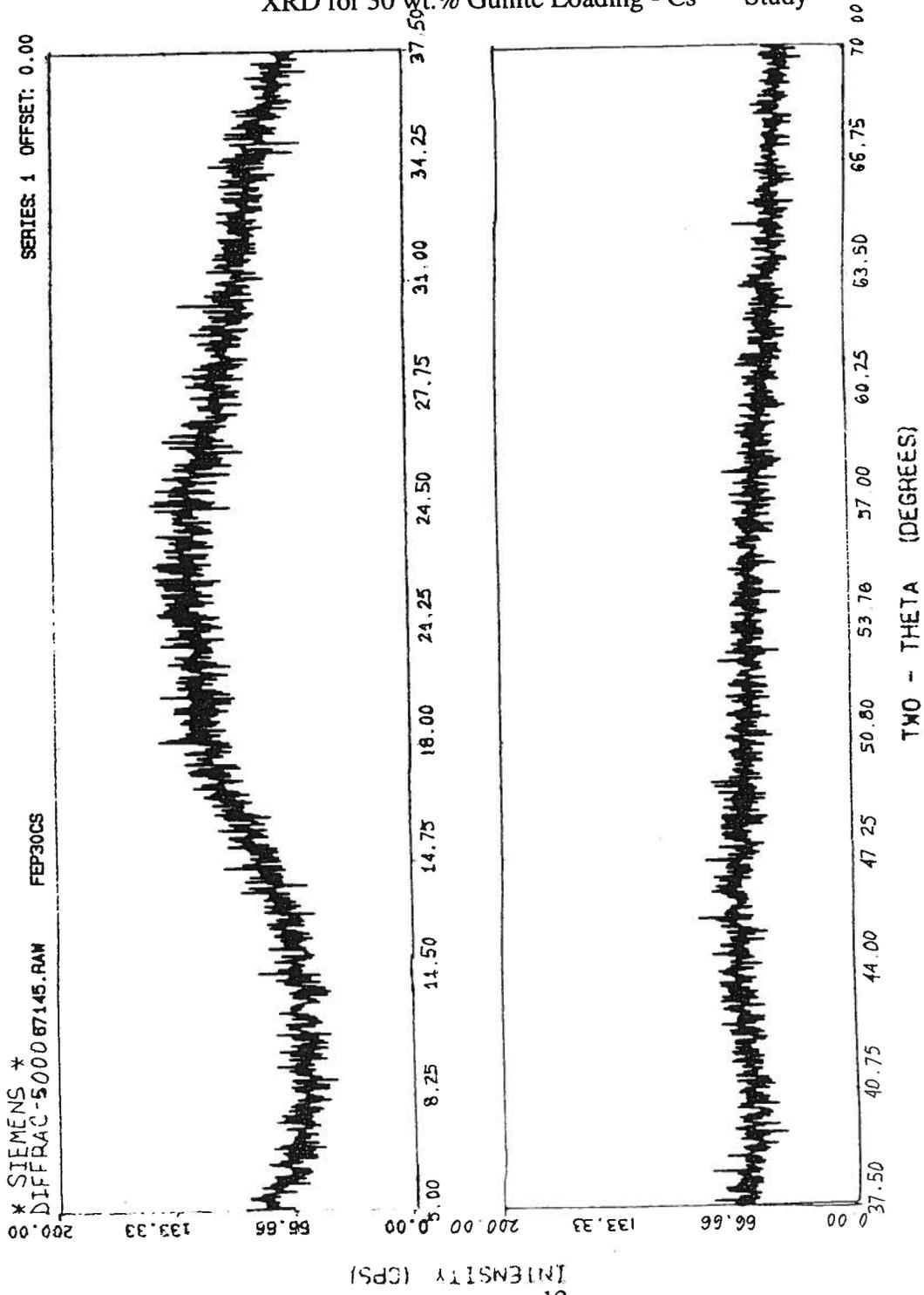


Figure 7.0

XRD for 35 wt.% Gunitite Loading - Cs¹³⁷ Study

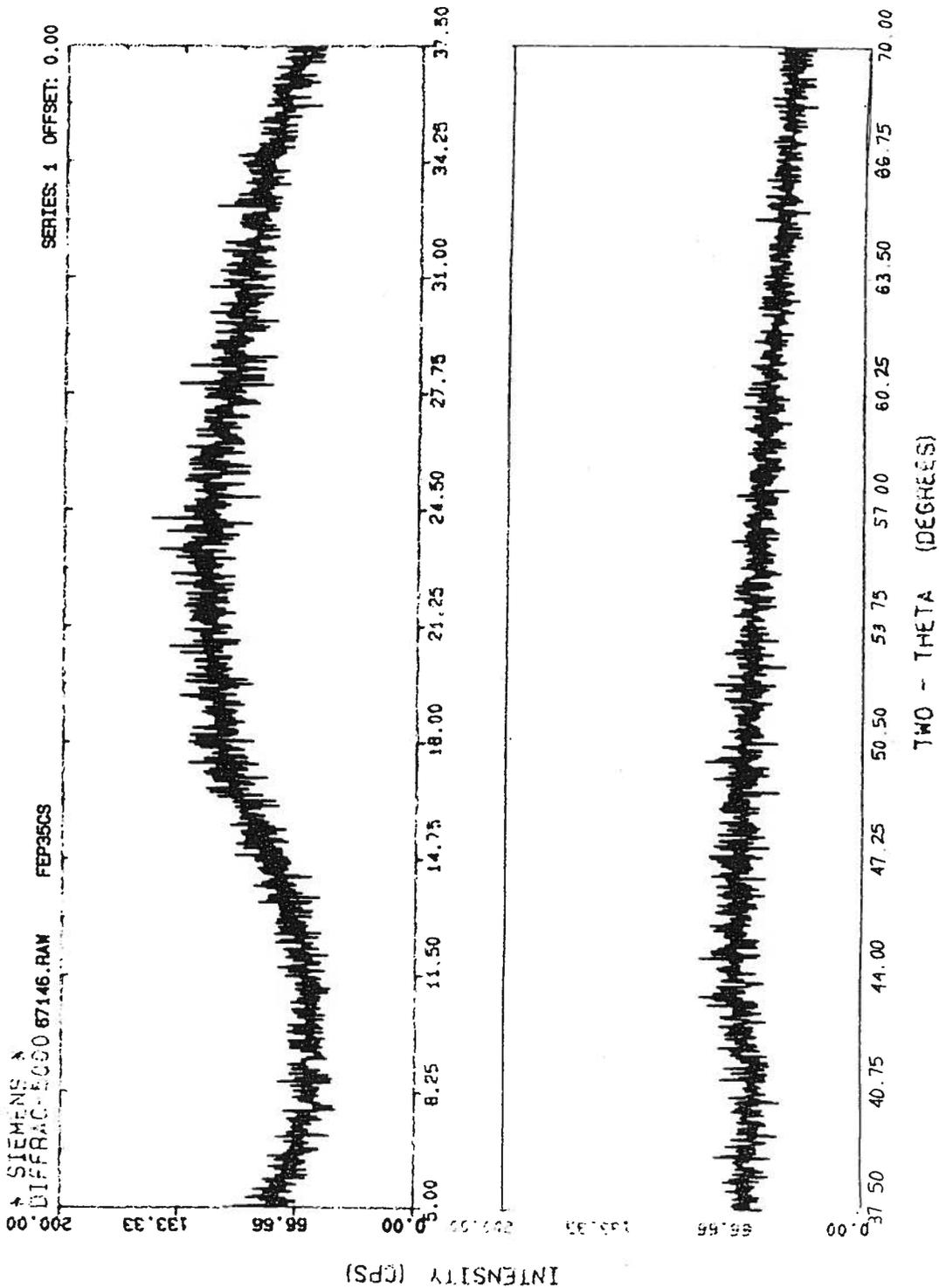


Table 5.0

Chemical Analysis of FeP Glasses from ICP Results

25 wt% Results

<u>Component</u>	<u>Target Weight % Oxide(Normalized)</u>	<u>Actual Weight % Oxide(Normalized)</u>
P ₂ O ₅	55.81	52.44
Na ₂ O	3.09	3.25
Fe ₂ O ₃	19.47	20.85
CaO	0.21	0.25
Al ₂ O ₃	1.87	2.49
UO ₃	19.55	20.70

Table 6.0

Chemical Analysis of FeP Glasses from ICP Results

30 wt% Results

<u>Component</u>	<u>Target Weight % Oxide(Normalized)</u>	<u>Actual Weight % Oxide(Normalized)</u>
P ₂ O ₅	55.48	53.43
Na ₂ O	3.67	3.84
Fe ₂ O ₃	14.97	16.22
CaO	0.23	0.27
Al ₂ O ₃	2.19	2.93
UO ₃	23.46	23.30

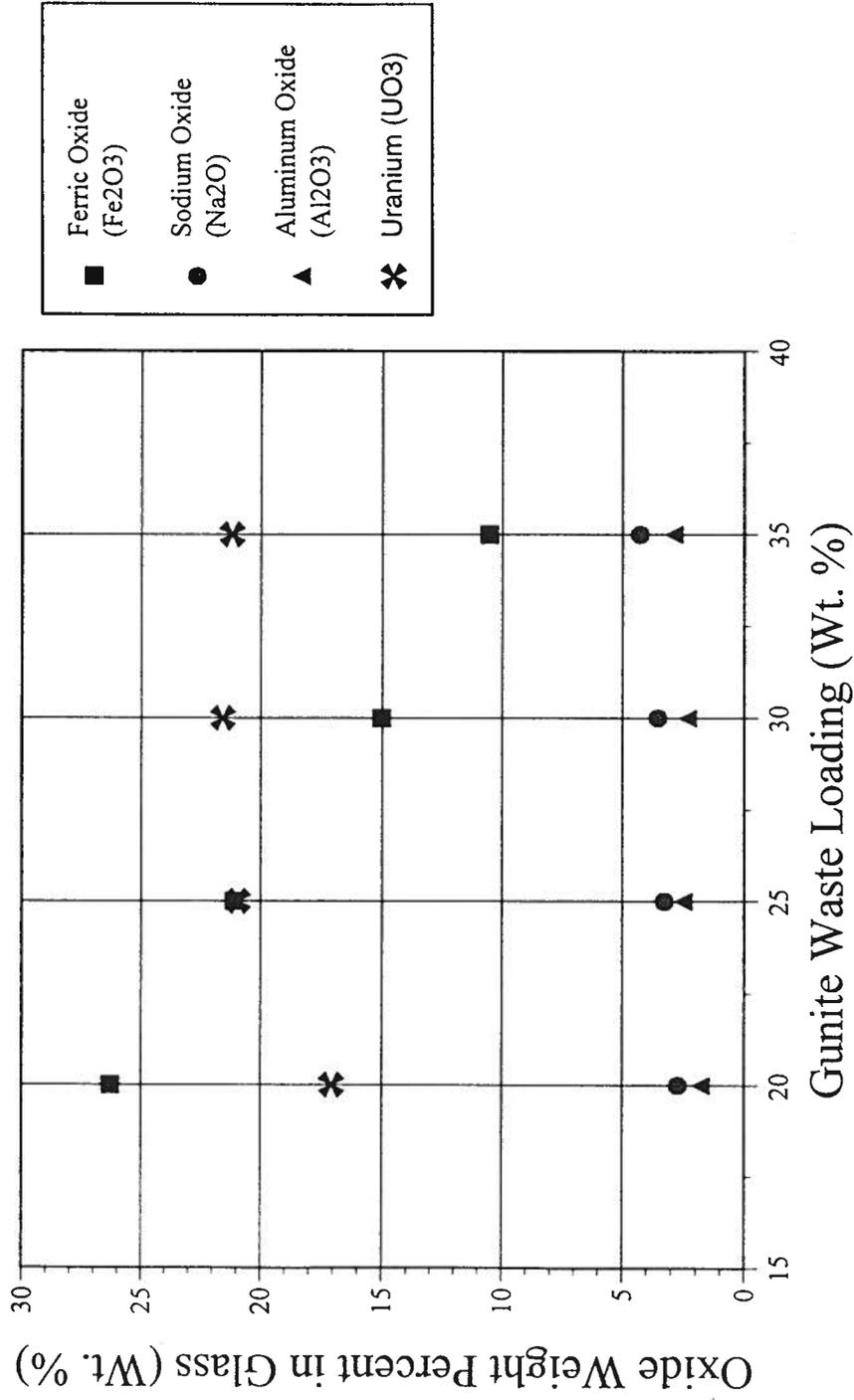
Table 7.0**Chemical Analysis of FeP Glasses from ICP Results**

<u>Component</u>	<u>35 wt% Results</u>	
	<u>Target Weight % Oxide(Normalized)</u>	<u>Actual Weight % Oxide(Normalized)</u>
P ₂ O ₅	55.17	55.75
Na ₂ O	4.30	4.90
Fe ₂ O ₃	10.38	12.03
CaO	0.23	0.33
Al ₂ O ₃	2.55	2.79
UO ₃	27.38	24.21

Figure 8.0

Chemical Analysis of FeP Glass

Chemical Analysis of FeP Glass



7.2 Chemical Durability

7.2.1 Product Consistency Test (PCT)

The PCT test was developed at Savannah River Technology Center and was established as an ASTM procedure (ASTM C-1285) to determine glass durability [7, 8]. Five glass samples from the 100 gram batches (0-35 wt % Gunite) were submitted for analysis.

The glass samples are crushed to 100-200 mesh and subjected to a seven day leaching in ASTM Type-I water at 90°C. A standard glass (ARM-1) was also subjected to the test at the same time as a check for bias in the data [9]. After seven days, the leachates are filtered to remove colloids/particles. The pH of the leachates are measured and the solutions are analyzed for elemental content (ICP).

The results for the standard glass and glass samples are analyzed and reported in triplicate. The leachate concentrations are reported as normalized elemental mass losses released from the grams of glass dissolved per liter of leachant. Since the surface area and the solution volume were held constant during the PCT test, the elemental mass loss can be expressed as given below:

$$NC_i = C_i / (F_i * 1000)$$

where

$$\begin{aligned} NC_i &= \text{Normalized mass of element "i" in solution (g}_{\text{glass}}/\text{L}_{\text{leachate}}) \\ C_i &= \text{Mass of element "i" in the solution (g}/\text{m}^3) \\ F_i &= \text{fraction of element "i" in the glass (g}_i/\text{g}_{\text{glass}}) \end{aligned}$$

The current acceptance criteria for acceptable release rates is based on the 131 Stage 2 glass which is qualified in the DWPF Environmental Assessment (better known as EA glass). To meet the current acceptance criteria, the release rates for glasses must be better than the EA glass [9]. The EA glass release rates are based on a borosilicate glass.

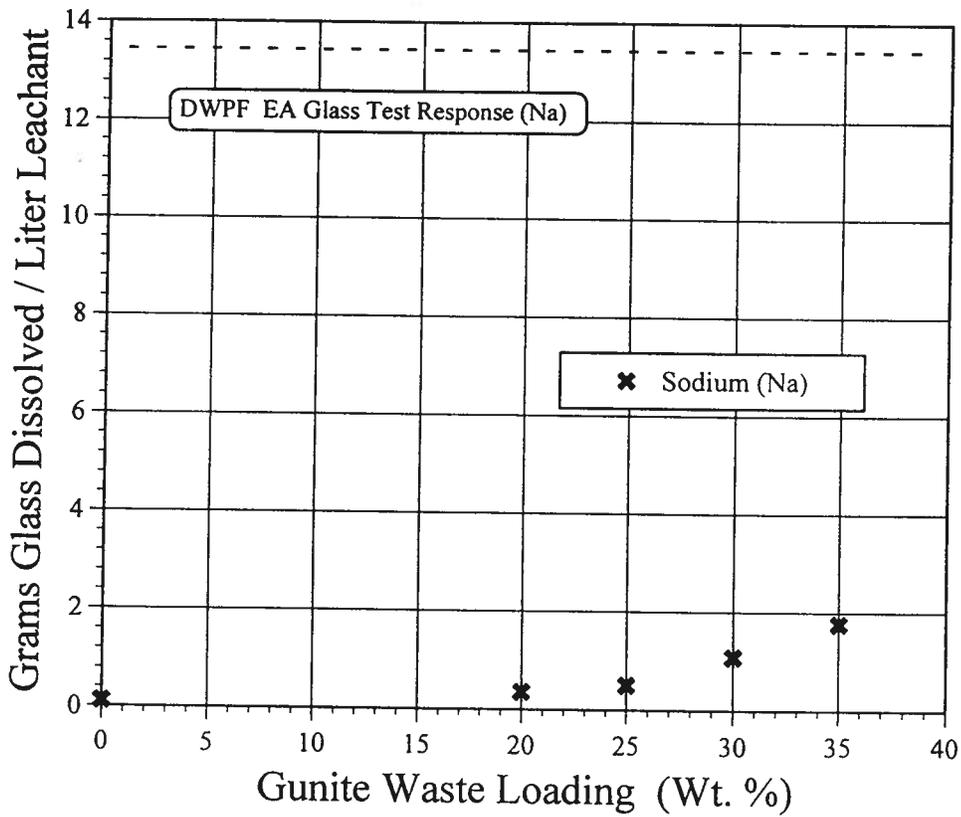
Boron and alkali elements are considered to be the most accurate indicators of a leach test response in a borosilicate glass [9]. The iron phosphate glass does not contain any boron, but does contain sodium. The sodium release rate for the glass was compared to the EA glass. The release rates are plotted as a function Gunite waste loading verses grams of glass dissolved per liter of leachant in Figure 9.0. The release rates along with pH are recorded in Table 8.0.

The release rate for sodium is better than the release rate for the EA glass by a factor of 5 to 10, depending on the Gunite waste loading. The other trend noticed was as the Gunite waste loading was increased, the release rate for soluble components in the glass were also increased. This increase can be explained by the reduction in the amount of iron added to the glass as the Gunite waste loading is increased. Appendix A contains the raw PCT results.

Figure 9.0

Effect of Gunite Waste Loading on FeP Glass Durability

Effect of Gunite Waste Loading on FeP Glass Durability
ASTM C-1285 Test Method (PCT)



7.2.2 Toxicity Characteristic Leaching Procedure (TCLP)

The Toxicity Characteristic Leaching Procedure (TCLP) is a test defined by 40 CFR 261. To determine release of RCRA (Resources Conservation and Recovery Act) materials from 9.5 mm screen ground glass when subjected to buffer leaching solutions of a pH of 3.0 or 5.0 [10].

Selection of the leaching solution is based on the alkalinity and the buffering capacities of the glass. The leaching solution is added 20:1 liquid - solid ratio, and the sample is agitated in a National Bureau of Standards (NBS) rotary tumbler at 30 rpm for 18 hours. The leaching solution is filtered, and analyzed for specific organics and metals [10].

Five samples from the 100 gram glass samples were submitted for TCLP. The results from this test have not been reported. A revision will be made as soon as the data is available.

7.3 Density

Density measurements were performed on the 100 gram batches of the iron phosphate glass utilizing ASTM C 693-74 "Density of Glass by Buoyancy" [11]. Temperature, barometric pressure, weight of sample in air and weight of sample in water were recorded. The data obtained for each piece of glass was placed in the following equation to obtain the density:

$$\rho = (\rho_w W_A - \rho_A W_W) / (W_A - W_W)$$

Where:

ρ = Glass density.

ρ_w = Density of water corrected for temperature and pressure.

W_A = Weight of sample in air.

W_W = Weight of sample in water.

ρ_A = Density of air corrected for temperature and pressure.

The density values for the 100 gram pieces of glass are recorded in Table 9.0.

Table 8.0**Normalized Elemental Releases and pH Measurements from PCT Results for FeP Glasses**

<u>Weight % Gunitite</u>	<u>Glass ID</u>	<u>Final pH</u>	<u>NC_{Na}</u>	<u>NC_P</u>	<u>NC_B</u>	<u>NC_{Fe}</u>
0	FE-G0	4.14	0.12 g/L	0.02 g/L	-	0.00 g/L
20	FE-20	3.86	0.36 g/L	0.11 g/L	-	0.00 g/L
25	FE-25	3.85	0.51 g/L	0.18 g/L	-	3.2E-4 g/L
30	FE-30	3.56	1.09 g/L	0.48 g/L	-	6.4E-5 g/L
35	FE-35	3.39	1.77 g/L	0.94 g/L	-	2.7E-5 g/L
	ARM	9.77	38.65 ppm	-	18.22 ppm	0.05 ppm

Note: The NC_{Na} response for the EA glass under these PCT conditions is 13.35 g/L.

Table 9.0

Iron Phosphate Glass Density

<u>Glass ID</u>	<u>Wt.% Gunite (Oxide basis)</u>	<u>Density (g/cm³)</u>
FeP 0	0	2.97
FeP 20	20	3.13
FeP 25	25	3.01
FeP 30	30	3.22
FeP 35	35	3.21

7.4 Reduction/Oxidation (Redox)

Glass samples were submitted for $\text{Fe}^{2+}/\Sigma\text{Fe}$ by the Mössbauer to determine redox state of the glass. All of the samples were found to be below the detection limit for the Mössbauer, except for the zero weight percent Gunitite waste loading. The results of the Mössbauer indicated that the glass melts were all oxidized. The results are recorded in Table 10.0.

Table 10.0

Iron Redox for Iron Phosphate Glass

<u>Glass Sample ID</u>	<u>Wt.% Gunitite (Oxide basis)</u>	<u>$\text{Fe}^{2+}/\Sigma\text{Fe}$</u>
FeP 0	0	0.19
FeP 20	20	BDL*
FeP 25	25	BDL*
FeP 30	30	BDL*
FeP 35	35	BDL*

* BDL - Below Detection Limit (0.10)

8.0 Conclusions

The following conclusions were obtained from the results of this feasibility study:

- 1) Iron phosphate glasses containing Gunitite waste up to 35 weight percent (oxide) were achieved.
- 2) The melting temperature for the Iron Phosphate glasses ranged from 1050°C to 1100°C. Determination of the melting temperature was important due to the volatility of Cs^{137} at higher temperatures. A test of Cs volatility at 1150°C was also performed.
- 3) The durability of the Iron Phosphate glass is significantly better than EA glass. EA glass is used as a standard for the acceptance of High Level Waste glass to a repository. High Level Waste glass must be below the standard release rates of the EA glass for acceptance to a repository.

- 4) The density of the glass ranged between 3.0 to 3.2 g/cc.
- 5) All glasses made were below the detection limit (0.1) for $Fe^{+2}/\Sigma Fe$ for Mössbauer, indicating all melts were oxidized.

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Dave Mesimer	ADS
Mike Whitaker	ADS
Miriam Cooley	ADS
Loretta Farrow	ADS
Beverly Burch	ADS

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7. C. Jantzen, N.E. Bibler, D.C. Beam, W.G. Ramsey and B.J. Waters, "Nuclear Waste Glass Product Consistency Test (PCT) - Version 5.0 (U)", WSRC-TR-90-539 Rev. 2, (1992).
8. "Standard Test Method for Determining Chemical Durability of Nuclear Waste Glasses", ASTM C 1285-94 (1993).
9. C. Jantzen, N.E. Bibler, D.C. Beam, C.L. Crawford, and M.A. Pickett, "Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material", WSRC-TR-92-346, Rev 1, (1993).
10. Federal Register. June 1, 1990. Vol. 55, No. 106, pp 22560-22561, 22573-22574.
11. Standard Test Method for Density of Glass by Buoyancy, ASTM C 693-74 (1980).

Appendix A

PCT Raw Data

Table 11.0

PCT Results for ARM Glass

PCT RESULTS

PAGE 1

ARM

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS
 Date in = 12/14/95 Date out = 12/21/95 Time in = 920 Time out = 725

EXPERIMENTAL DATA:

Sample Name	Weights (g)		Final Vol (mL)	pH Values		Analyt Number	Analyt Analytical Results (ppm) for acidf, diluted leachate (3 mL to 2 mL of 0.4HNO3) D.F.=1.667										
	Empty w/Glass	w/H2O		Initial	Final		B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni	
BLANK-1	120.17	N/A	135.00	14.83	6.87	6.86	65310	0.008	0.061	0.240	0.004	0.103	0.027	0.030	0.036	0.158	0.031
BLANK-2	121.89	N/A	136.90	136.48	14.59	6.74	65311	0.010	0.082	0.240	0.007	0.092	0.041	0.040	0.088	0.264	0.084
BLANK AVERAGE							0.009	0.072	0.240	0.006	0.098	0.034	0.035	0.052	0.211	0.058	
ARM-1	122.05	123.57	138.80	138.28	14.71	6.87	65312	10.975	40.357	23.326	8.681	3.095	0.052	0.003	0.034	0.201	0.025
ARM-2	122.88	124.37	139.33	138.81	14.44	6.87	65313	10.327	39.222	22.357	8.367	3.879	0.052	0.003	0.024	0.159	0.025
ARM-3	122.16	123.70	138.70	137.35	13.65	6.87	65314	11.517	41.935	24.595	9.242	3.163	0.096	0.003	0.030	0.149	0.025
ANALYTICAL DETECTION LIMITS							0.010	0.030	0.060	0.060	0.090	0.010	0.003	0.003	0.030	0.030	0.049

RESULTS FOR pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS:

Sample Name	pH Values		B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni
	Initial	Final										
ARM-1	6.87	9.76	18.28	87.16	38.48	14.48	5.00	0.03	-0.05	-0.03	-0.02	-0.05
ARM-2	6.87	9.79	17.20	65.26	36.87	13.94	6.30	0.03	-0.05	-0.05	-0.09	-0.05
ARM-3	6.87	9.77	19.18	69.79	40.60	15.40	5.11	0.10	-0.05	-0.04	-0.10	-0.05
AVERAGE			18.22	67.40	38.85	14.60	5.47	0.05	-0.05	-0.04	-0.07	-0.05
STANDARD DEVIATION			0.99	2.27	1.87	0.74	0.72	0.04	0.00	0.01	0.05	0.00
REL. STD. DEVIATION (%)			5.45	3.37	4.84	5.06	13.24	77.77	0.00	-22.21	-66.76	0.00

QUALITY ASSURANCE INFORMATION:

LAB# = B-102/B-111
 PH METER # = 06352760
 BALANCE M&TE# = GT-1-098
 OVEN M&TE # = GT-1-232
 FILTER SIZE: 0.45 MICRON
 FLUKE M&TE# = GT-1-187
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/98
 ALL SAMPLES WASHED
 WEIGHED WITH LIDS ON

Table 12.0

PCT Results for Fe-20 Glass

PCT RESULTS

PAGE 2

FE-20

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS
 Date in = 12/14/95 Date out = 12/21/95 Time in = 920 Time out = 725

EXPERIMENTAL DATA:

Sample Name	Empty w/Glass	Weights (g) w/H2O	Final Wt (g)	Final Vol (mL)	pH Values Initial	pH Values Final	Analyt. Analytical Results (ppm) for acidified, diluted leachates (3 mL to 2 mL of 0.4HNO3) D.F.=1.667	Li	Al	Fe	Mn	Mg	Ca	Ni			
Sample Name	Empty	w/Glass	Final Wt (g)	Final Vol (mL)	Initial	Final	Number	Na	Si	B	Li	Al	Fe	Mn	Mg	Ca	Ni
BLANK-1	120.17	N/A	135.21	14.83	6.87	6.86	65310	0.240	0.061	0.008	0.004	0.103	0.027	0.030	0.036	0.158	0.031
BLANK-2	121.89	N/A	136.90	14.59	6.87	6.74	65311	0.240	0.082	0.010	0.007	0.092	0.041	0.040	0.066	0.264	0.064
BLANK AVERAGE								0.240	0.072	0.008	0.006	0.098	0.034	0.035	0.052	0.211	0.058
FE-20-1	121.92	123.42	138.42	137.81	6.87	3.96	65331	4.261	1.892	0.173	0.125	0.060	0.203	0.081	0.103	0.143	0.058
FE-20-2	121.79	123.33	138.33	137.15	6.87	3.84	65332	4.621	1.870	0.167	0.125	0.060	0.193	0.075	0.097	0.111	0.034
FE-20-3	120.08	121.59	136.59	136.10	6.87	3.77	65333	4.261	1.806	0.159	0.119	0.060	0.177	0.059	0.094	0.106	0.025
ANALYTICAL DETECTION LIMITS								0.060	0.030	0.010	0.060	0.090	0.010	0.003	0.003	0.030	0.049

RESULTS FOR pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS:

Sample Name	pH Value Initial	pH Value Final
FE-20-1	6.87	3.96
FE-20-2	6.87	3.84
FE-20-3	6.87	3.77

AVERAGE
 STANDARD DEVIATION
 REL. STD. DEVIATION (%)

QUALITY ASSURANCE INFORMATION:

LAB# B-102/B-111
 PH METER # = 06352760
 BALANCE M&TE# = GT-1-098
 OVEN M&TE # = GT-1-232
 FILTER SIZE 0.45 MICRON
 FLUKE M&TE# = GT-1-167
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/96
 ALL SAMPLES WASHED
 WEIGHED WITH LUDS ON

Normalized Release (g/L)

Li	Al	Fe	Mn	Mg	Ca	Ni
0.10	0.01	0.10	0.01	0.01	0.01	0.01
57.79	57.77	57.89	27.24	10.53	-19.11	-50.92

Table13.0

PCT Results for Fe-25 Glass

PCT RESULTS

PAGE 3

FE-25

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS
 Date In = 12/14/95 Time In = 920 Date out = 12/21/95 Time out = 725

EXPERIMENTAL DATA:

Sample Name	Weights (g)		Final Vol (mL)		pH Values		Analyt Number	Analyt Analytical Results (ppm) for acidified, diluted leachates (3 mL to 2 mL of 0.4HNO3) D.F.=1.667									
	Empty	w/Glass	w/H2O	Final	Initial	Final		B	Si	Na	LI	Al	Fe	Mn	Mg	Ca	Ni
BLANK-1	120.17	N/A	135.21	135.00	14.83	8.88	65310	0.008	0.061	0.240	0.004	0.103	0.027	0.030	0.036	0.158	0.031
BLANK-2	121.89	N/A	138.90	138.48	14.59	8.74	65311	0.010	0.082	0.240	0.007	0.092	0.041	0.040	0.068	0.264	0.084
BLANK AVERAGE																	
FE-25-1	121.57	123.07	138.05	137.18	14.09	3.88	65334	0.009	0.072	0.240	0.006	0.098	0.034	0.035	0.052	0.211	0.058
FE-25-2	121.05	122.55	137.55	136.91	14.36	3.85	65335	0.088	0.926	7.473	0.085	0.078	0.289	0.068	0.060	0.130	0.082
FE-25-3	121.25	122.76	137.76	136.83	14.07	3.84	65336	0.088	0.987	7.600	0.066	0.090	0.295	0.060	0.059	0.135	0.090
ANALYTICAL DETECTION LIMITS							0.010	0.030	0.060	0.060	0.060	0.090	0.010	0.003	0.003	0.030	0.049

RESULTS FOR PH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS :

Sample Name	pH Values		B	Si	Na	LI	Al	Fe	Mn	Mg	Ca	Ni
	Initial	Final										
FE-25-1	6.87	3.88	0.13	1.42	12.06	0.10	-0.03	0.43	0.06	0.01	-0.14	0.01
FE-25-2	6.87	3.85	0.13	1.29	11.40	0.09	-0.04	0.37	0.04	0.01	-0.13	0.05
FE-25-3	6.87	3.84	0.13	1.49	12.27	0.10	-0.01	0.44	0.07	0.03	-0.10	0.25
AVERAGE			0.13	1.40	11.91	0.10	-0.03	0.41	0.05	0.02	-0.12	0.10
STANDARD DEVIATION			0.00	0.10	0.45	0.01	0.01	0.04	0.01	0.01	0.02	0.13
REL. STD. DEVIATION (%)			1.49	7.45	3.82	8.63	-50.89	8.54	22.98	47.74	-14.32	124.22

QUALITY ASSURANCE INFORMATION:

LAB#=#B-102/B-111
 PH METER #=# 06352760
 BALANCE M&TE#=# GT-1-098
 OVEN M&TE #=#GT-1-232
 FILTER SIZE: 0.45 MICRON
 FLUKE M&TE#=# GT-1-187
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/96
 ALL SAMPLES WASHED
 WEIGHED WITH LIDS ON

Table 14.0

PCT Results for Fe-30 Glass

PCT RESULTS

PAGE 4

FE-30

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS
 Date in = 12/14/95 Date out = 12/21/95 Time in = 920 Time out = 725

EXPERIMENTAL DATA:

Sample Name	Weights (g): Empty w/Glass	w/H ₂ O	Final Wt (g)	Final Vol (mL)	pH Values Initial	Final	Analyt. Number	Si	Ne	Li	Al	Fe	Mn	Mg	Ca	Ni
BLANK-1	120.17	N/A	135.21	135.00	14.83	6.87	6.86	65310	0.008	0.061	0.103	0.027	0.030	0.036	0.158	0.031
BLANK-2	121.89	N/A	136.90	136.48	14.59	6.87	6.74	65311	0.010	0.082	0.092	0.041	0.040	0.088	0.264	0.084
BLANK AVERAGE									0.008	0.072	0.098	0.034	0.035	0.052	0.211	0.058
FE-30-1	121.57	123.06	138.04	135.26	12.20	6.87	3.57	65337	0.080	0.703	0.321	1.365	0.103	0.142	1.600	0.304
FE-30-2	121.79	123.32	138.34	137.64	14.32	6.87	3.54	65338	0.057	0.780	0.173	0.926	0.071	0.085	0.506	0.108
FE-30-3	122.56	124.06	139.06	137.84	13.78	6.87	3.56	65339	0.053	0.699	0.170	1.098	0.083	0.087	0.494	0.226

ANALYTICAL DETECTION LIMITS

Si	0.010
Ne	0.030
Li	0.060
Al	0.090
Fe	0.010
Mn	0.003
Mg	0.003
Ca	0.030
Ni	0.049

RESULTS FOR pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS :

Sample Name	pH Values	
	Initial	Final
FE-30-1	6.87	3.57
FE-30-2	6.87	3.54
FE-30-3	6.87	3.56

AVERAGE
 STANDARD DEVIATION
 REL. STD. DEVIATION (%)

Sample Name	B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni
AVERAGE	0.00	0.00	0.00	0.00	0.37	2.22	0.11	0.15	2.32	0.41
STANDARD DEVIATION	0.08	1.18	26.47	0.06	0.13	1.49	0.06	0.06	0.49	0.08
REL. STD. DEVIATION (%)	0.07	1.05	27.43	0.05	0.12	1.77	0.08	0.08	0.47	0.26

QUALITY ASSURANCE INFORMATION:

LAB#-B-102/B-111
 PH METER # = 06352760
 BALANCE M&TE# = GT-1-088
 OVEN M&TE # = GT-1-202
 FILTER SIZE 0.45 MICRON
 FLUKE M&TE# = GT-1-167
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/96
 ALL SAMPLES WASHED
 WEIGHED WITH LIDS ON

Normalized Release (g/L)

B	57.90
Si	58.05
Na	57.76
Li	57.91
Al	116.66
Fe	22.62
Mn	38.49
Mg	95.14
Ca	219.76
Ni	90.12

Table 15.0

PCT Results for Fe-35 Glass

PCT RESULTS

PAGE 5

FE-35

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS
 Date In = 12/14/95 Time In = 920 Date out = 12/21/95 Time out = 725

EXPERIMENTAL DATA:

Sample Name	Weight (g)		Final Wt (g)	Final Vol (mL)	pH Value		Analyt Number	Analyt Analytical Results (ppm) for acidified, diluted leachates (3 mL to 2 mL of 0.4HNO3) D.F.=1.667											
	Empty	w/Glass			Initial	Final		B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni		
BLANK-1	120.17	N/A	135.21	135.00	14.83	6.87	6.86	65310	0.008	0.061	0.240	0.004	0.103	0.027	0.030	0.036	0.158	0.031	
BLANK-2	121.89	N/A	136.90	136.48	14.59	6.87	6.74	65311	0.010	0.082	0.240	0.007	0.092	0.041	0.040	0.068	0.264	0.084	
BLANK AVERAGE	122.20	123.70	138.71	137.83	14.23	6.87	3.38	65340	0.009	0.072	0.240	0.006	0.098	0.034	0.035	0.052	0.211	0.058	
FE-35-1	121.96	123.46	138.42	137.73	14.27	6.87	3.40	65341	0.100	2.153	34.497	0.153	0.600	1.808	0.151	0.157	0.802	0.253	
FE-35-2	122.92	124.42	139.43	138.70	14.28	6.87	3.36	65342	0.053	1.834	35.100	0.114	0.600	1.501	0.157	0.172	1.063	0.439	
FE-35-3																			
ANALYTICAL DETECTION LIMITS									0.010	0.030	0.060	0.060	0.090	0.010	0.003	0.003	0.030	0.049	

RESULTS FOR pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS:

Sample Name	pH Values	
	Initial	Final
FE-35-1	6.87	3.38
FE-35-2	6.87	3.40
FE-35-3	6.87	3.38

AVERAGE STANDARD DEVIATION
 REL. STD. DEVIATION (%)

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QUALITY ASSURANCE INFORMATION:

LAB# = B-102/B-111
 PH METER # = 06352760
 BALANCE M&TE # = GT-1-098
 OVEN M&TE # = GT-1-232
 FILTER SIZE = 0.45 MICRON
 FLUKE M&TE # = GT-1-187
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/96
 ALL SAMPLES WASHED
 WEIGHED WITH LIDS ON

Sample Name	Analytical Results (ppm)									
	B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni
FE-35-1	0.18	3.45	52.75	0.25	0.84	2.82	0.19	0.18	1.15	0.33
FE-35-2	0.15	3.47	57.11	0.25	0.84	2.45	0.20	0.20	1.42	0.64
FE-35-3	0.07	2.94	58.11	0.18	0.84	3.00	0.22	0.18	1.39	0.32
AVERAGE	0.13	3.29	55.99	0.23	0.84	2.69	0.20	0.19	1.32	0.43
STANDARD DEVIATION	0.05	0.30	2.85	0.04	0.00	0.26	0.01	0.01	0.15	0.18
REL. STD. DEVIATION (%)	40.59	9.20	5.09	17.10	0.00	10.60	5.73	6.98	11.06	42.21

Table 16.0

PCT Results for Fe-0 Glass

PCT RESULTS

PAGE 6

FE-GO

1.5 GRAMS OF 100-200 MESH CRUSHED GLASS IN 15 mL OF DEIONIZED WATER IN SST VESSELS; T=90°C, t=7 DAYS

Date in = 12/16/95

Date out = 12/21/95

Time in = 920

Time out = 725

EXPERIMENTAL DATA:

Sample Name	Weights (g): Empty w/Glass	w/H2O	Final Wt (g)	Final Vol (mL)	pH Values Initial Final	Analyt#	Analyt# Analytical Results (ppm) for acidified, diluted leachates (3 mL to 2 mL of 0.4HNO3) D.F.=1.667	Na	Li	Al	Fe	Mn	Mg	Ca	Ni	
						Number	B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni
BLANK-1	120.17	N/A	135.21	135.00	6.87	65310	0.008	0.061	0.240	0.004	0.103	0.027	0.030	0.036	0.158	0.031
BLANK-2	121.89	N/A	136.90	136.48	6.87	65311	0.010	0.082	0.240	0.007	0.092	0.041	0.040	0.068	0.264	0.084
BLANK AVERAGE							0.009	0.072	0.240	0.006	0.098	0.034	0.035	0.052	0.211	0.058
FE-GO-1	122.36	123.88	138.88	138.25	6.87	65343	0.077	0.447	0.879	0.069	0.080	0.184	0.052	0.030	0.050	0.059
FE-GO-2	122.20	123.73	138.74	138.02	6.87	65344	0.051	0.351	0.864	0.042	0.080	0.073	0.036	0.036	0.078	0.025
FE-GO-3	122.36	123.86	138.87	138.00	6.87	65345	0.059	0.369	0.863	0.046	0.080	0.110	0.051	0.029	0.065	0.037
ANALYTICAL DETECTION LIMITS							0.010	0.030	0.060	0.060	0.090	0.010	0.003	0.003	0.030	0.049

RESULTS FOR pH VALUES AND FILTERED LEACHATE CONCENTRATIONS CORRECTED FOR BLANKS:

Sample Name	pH Values		B	Si	Na	Li	Al	Fe	Mn	Mg	Ca	Ni
	Initial	Final										
FE-GO-1	6.87	4.05	0.11	0.63	1.23	0.11	-0.06	0.25	0.03	-0.04	-0.27	0.00
FE-GO-2	6.87	4.17	0.07	0.47	1.04	0.06	-0.06	0.07	0.00	-0.03	-0.22	-0.05
FE-GO-3	6.87	4.21	0.08	0.50	1.04	0.07	-0.03	0.13	0.03	-0.04	-0.24	-0.03
AVERAGE			0.09	0.53	1.10	0.08	-0.05	0.15	0.02	-0.03	-0.24	-0.03
STANDARD DEVIATION			0.02	0.09	0.11	0.02	0.02	0.09	0.01	0.01	0.02	0.03
REL. STD. DEVIATION (%)			24.97	16.07	10.07	31.11	-37.45	63.98	79.08	-18.62	-9.55	-100.45

QUALITY ASSURANCE INFORMATION:

LAB# B-102/B-111
 PH METER # = 06352760
 BALANCE M&TE# = GT-1-098
 OVEN M&TE # = GT-1-292
 FILTER SIZE 0.45 MICRON
 FLUKE M&TE# = GT-1-187
 BUFFERED TO PH 7 AND 10
 CALIBRATION EXP. DATE = 4/96
 ALL SAMPLES WASHED
 WEIGHED WITH LIDS ON