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September 1981

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
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EVALUATION OF DEFENSE-WASTE GLASS PRODUCED BY FULL-SCALE VITRIFICATION EQUIPMENT

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SUMMARY

Three full-scale vitrification processes at the Pacific Northwest Laboratory produced over 67,000 kg of simulated nuclear-waste glass from March 1979 to August 1980. Samples were analyzed to monitor process operation and evaluate the resulting glass product. These processes are:

- Spray Calciner/In-Can Melter (SC/ICM)
- Spray Calciner/Calcine-Fed Ceramic Melter (SC/CFCM)
- Liquid-Fed Ceramic Melter (LFCM).

Waste components in the process feed varied less than +10%. The SC/ICM and SC/CFCM which use separate waste and frit feed systems showed larger glass compositional variation than the LFCM, which processed only premixed feed during this period. The SC/ICM and SC/CFCM product contained significant amounts of acmite crystals, while the LFCM product was largely amorphous. In addition, the lower portion of all SC/ICM-filled canisters contained a zone rich in waste components.

A product chemical durability as determined by pH4 and soxhlet leach tests varied considerably. Aside from increased durability under pH4 conditions with decreasing waste content, glass composition, microstructure and melting process did not correlate with glass durability. For all samples analyzed, the weight loss under pH4 conditions ranged from 17.7 to 85.2 wt%. Soxhlet conditions produced weight losses from 1.78 to 3.56 wt%.

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INTRODUCTION

The safe disposal of nuclear waste is essential for the continued use of nuclear energy in the United States and abroad. A key element in proposed waste-management systems is solidification of nuclear wastes in an inert form. One technique that has been extensively investigated is the vitrification of wastes to produce a stable and durable waste glass. Researchers at the Pacific Northwest Laboratory (PNL), operated by Battelle Memorial Institute for the Department of Energy (DOE), have been active in the development of both glass compositions and process equipment for the production of nuclear waste glasses. Researchers at PNL have designed, constructed and operated full-scale vitrification equipment to evaluate three waste vitrification processes:

- Spray Calciner/In-Can Melter (SC/ICM)
- Spray Calciner/Calcine-Fed Ceramic Melter (SC/CFCM)
- Liquid-Fed Ceramic Melter (LFCM).

This equipment is capable of filling canisters 2.1 to 3.0 m (7 to 10 ft) tall with molten, simulated nuclear-waste glass which solidifies on cooling.

The Savannah River Laboratory (SRL), operated by E. I. DuPont de Nemours for the Department of Energy (DOE), is developing technology to immobilize and encapsulate nuclear waste sludges stored at the Savannah River Plant (SRP). The major process under consideration is the vitrification of SRP waste in borosilicate glass. The Pacific Northwest Laboratory supported this effort by conducting test runs with SRL-developed glasses in its full-scale melting equipment. Equipment runs of up to 25 continuous days of operation evaluated many aspects of equipment design, process operation and processibility of anticipated SRP wastes and waste glasses.

The waste glass compositions used in these equipment test runs were developed to be compatible with the vitrification processes and to meet durability criteria for the eventual disposal of nuclear-waste glass. During PNL test runs, feed and product samples were collected to evaluate the effect of the process on waste-glass composition and properties. Each of the three vitrification processes will be described along with the data collected during each melting trial.

WASTE GLASS COMPOSITION

Since March 1979, melting trials at PNL have processed a simulated TDS-211 waste glass composition. Premelted and ground 211 frit was melted with simulated TDS waste in a 72/28 oxide weight ratio. The TDS waste stream simulates the anticipated waste composition during actual operation at SRP. Table 1 lists the constituents used to produce simulated TDS liquid feed for PNL tests. The overall concentration was altered in various runs to improve system operation. The defined oxide composition of TDS calcine, 211 frit and TDS-211 glass are listed in Table 2. Later melting trials added minor constituents such as Cl^-, RuO_2, Sb_2O_3 and SrO, or organic components such as Na_4 EDTA, cornstarch and anthracite coal to the liquid feed to produce a more accurate liquid-waste stream simulation. These modifications did not significantly alter final waste or waste-glass oxide compositions.

TABLE 1. Simulated TDS Liquid Feed Composition Used in Equipment Test Runs

Chemical Constituents	Defined TDS Waste, g/L	Modified TDS Waste, g/L
$Fe(OH)_3$	35.1	100.3
Al_2O_3 $3H_2O$	8.8	25.2
MnO_2	7.0	20.0
$CaCO_3$	3.2	9.1
NiO $2H_2O$	4.5	12.9
$NaNO_3$	1.5	4.2
Na_2SO_4	0.7	2.0
Zeolite(a)	5.2	1.5
Cornstarch	---	0.8
Na_4 EDTA	---	11.6
Anthracite Coal	---	4.2
	66.0	191.8

(a) Linde Ionsiv 95

TABLE 2. TDS-211 Oxide Composition

Oxide Constituents	Oxide wt%			
	211 Frit	Zeolite(a)	TDS Waste	TDS-211 Waste Glass
Fe_2O_3	---	4.0	52.6	14.7
Al_2O_3	---	17.1	13.2	3.7
MnO_2	---	0.1	13.9	3.9
CaO	5.6	3.9	4.0	5.1
NiO	---	---	6.1	1.7
Na_2O	20.6	4.4	0.7	15.3
Na_2SO_4	---	---	0.9	0.4(b)
SiO_2	58.3	68.6	7.2	44.0
LiO_2	4.4	---	---	3.2
B_2O_3	11.1	---	---	8.0
	100.0	98.1	98.6	100.0

(a) Linde Ionsiv-95 - 1.9% ignition loss 800°C
(b) SO_3^-

PROCESS SAMPLING AND ANALYSIS

All PNL full-scale vitrification processes can convert simulated waste and frit into a borosilicate glass. Samples of the liquid waste, frit, waste-glass product and offgas were collected and analyzed during melting trials. This information was used to evaluate process stability, behavior of waste glass constituents during processing and properties of solidified waste glass.

The specific samples collected and the sampling frequency varied with the type of vitrification equipment operated and the run objectives. Liquid feed samples were collected directly upstream from the spray calciner or LFCM in 100-cc poylethylene bottles. Glass samples were either collected directly from the pouring spout of the LFCM or CFCM or core drilled from specific locations in the filled canisters after cooling. The sampling and analysis of the offgas stream during melting trials is not dealt with in this report.

Several analytical techniques were used to monitor process operation and waste glass properties. Those used during the course of SRL melting trials are listed in Table 3. Sample chemical analysis was done exclusively with the Induction Coupled Plasma Spectrophotometer (ICP). Chick et al. (1980) describes in detail the procedure used for ICP chemical analysis and x-ray diffraction crystal content analysis. Glass density which is very sensitive to chemical composition and thermal history was determined by the Archimedes technique. Each density data point was the average for three samples which were first annealed at 500°C for 2 h and slow cooled to eliminate thermal history effects.

Chemical durability was determined by soxhlet and pH4 leach tests. One-gram samples (-40 + 60 mesh) were placed in stainless steel mesh envelopes and leached in distilled water at 90°C for 24 h (soxhlet test) and in an agitated pH4 acidic solution for 19 h. The weight loss of the sample as a result of leaching was determined. Reported data represents the average values for two samples.

All analytical techniques described were not applied to each sample collected. The object of most analytical work was to monitor process operation

TABLE 3. Melting Trial Sample Analysis

Process Sample	Data Collected	Analytical Technique
Liquid Feed	Specific gravity	Measure weight and volume
	pH	Bechman 3500 pH meter
	Oxide content	Dried 125°C; fired 800°C
	Oxide composition	Induction Coupled Plasma Spectrophotometer (ICP)
Frit	Density	Archimedes technique
	Oxide composition	ICP
Waste Glass	Density	Archimedes technique
	Oxide composition	ICP
	Chemical durability	Soxhlet and pH4 leach test
	Crystal content	X-ray diffraction

with a reasonable amount of laboratory support. Sampling frequency and laboratory analyses were modified by both experience and unanticipated events during the melting trials. Canister samples were taken and analyzed for SRL.

MELTING TRIALS

From March 1979 to August 1980, 10 melting trials were run with a simulated TDS-211 glass in the three vitrification processes. During 63 d of operation, 44 canisters were filled with over 67,000 kg of waste glass. Several defense waste-glass melting trials were also conducted in the Experimental Ceramic Melter (ECM) and Alternative Waste Form Melter (AWFM). Details regarding these trials are not included because of their development objectives and lower operating capacities. Table 4 is a chronological listing of the full-scale equipment trials conducted during this period. The sample data collected will be reported separately for the SC/ICM, SC/CFCM and LFCM processes. A brief description of each process will preceed each set of data.

TABLE 4. PNL Defense Waste Melting Trials (March 1979 - August 1980)

Melting Trial Identification	Process	Trial Date	Trial Duration, d	Number of Canisters	Waste Glass, kg
FS-ICM-20 through FS-ICM-23	SC/ICM	March 1979	5	4	4,415
CFCM-6	SC/CFCM	April 1979	10	7	11,000
CFCM-7	SC/CFCM	June 1979	25	18	35,000
FS-ICM-24	SC/ICM	July 1979	1	1	1,202
SRL-13	LFCM	July 1979	5	3	4,250
SRL-14	LFCM	September 1979	3	1	2,480
CFCM-8	SC/CFCM	November 1979	5		
SRL-LF-1	LFCM	January 1980	3	2	1,050
FS-ICM-26 and FS-ICM-27	SC/ICM	March 1980	2	2	2,160
FS-ICM-28 and FS-ICM-29	SC/ICM	August 1980	4	2	1,900
TOTAL			63	44	67,657

SPRAY CALCINER/IN-CAN MELTER

The Spray Calciner/In-Can Melter (SC/ICM) process was developed at PNL to convert a liquid nuclear-waste stream into a durable glass. Bonner (1976) and Blair (1979) describe in detail the design, construction and operation of the SC/ICM. Simulated aqueous nuclear waste is pumped into a spray nozzle at the top of the calciner. Compressed air atomizes the liquid waste into fine droplets which fall through the externally heated calciner walls. Radiant energy ($600^{o}C$ to $800^{o}C$) quickly evaporates the water and partially calcines the waste oxides. Most of the calcine drops free of the water vapor and other gases at the bottom of the calciner; the remainder is removed from the offgas stream by sintered metal filters. These filters are periodically cleaned by a reverse pulse of compressed air which disperses the collected calcine to the bottom of the calciner. The glass-forming constituents are added to the process as a premelted, preground glass frit. This frit is continuously metered into the calciner barrel where it falls with the calcine through the bottom of the calciner into a heated ($1050^{o}C$ to $1150^{o}C$) canister which functions as a melting crucible. The arrangement of the PNL SC/ICM process is shown in Figure 1.

Normal operating canister wall temperatures are $1075^{o}C$ for stainless steel and $1150^{o}C$ for Inconel canisters. Corresponding melt temperatures may be 25^{o} to $50^{o}C$ cooler. As more calcine and frit are added, the level of molten glass rises. All SC/ICM canisters filled with defense waste glass from March 1979 to March 1980 were 0.61 m (24 in.) in dia and 2.13 m (84 in.) tall. When the canister has cooled to room temperature, the glass product can be sampled by core-drilling at specific intervals. The analysis of these samples will be presented separately for each of the three melting trials conducted during this period (Table 5).

FS-ICM-20 THROUGH FS-ICM-23 (MARCH 1979)

During March 1979 the SC/ICM was operated for 5 d, producing TDS-211 waste glass. The objectives of this melting trial were to demonstrate long-term

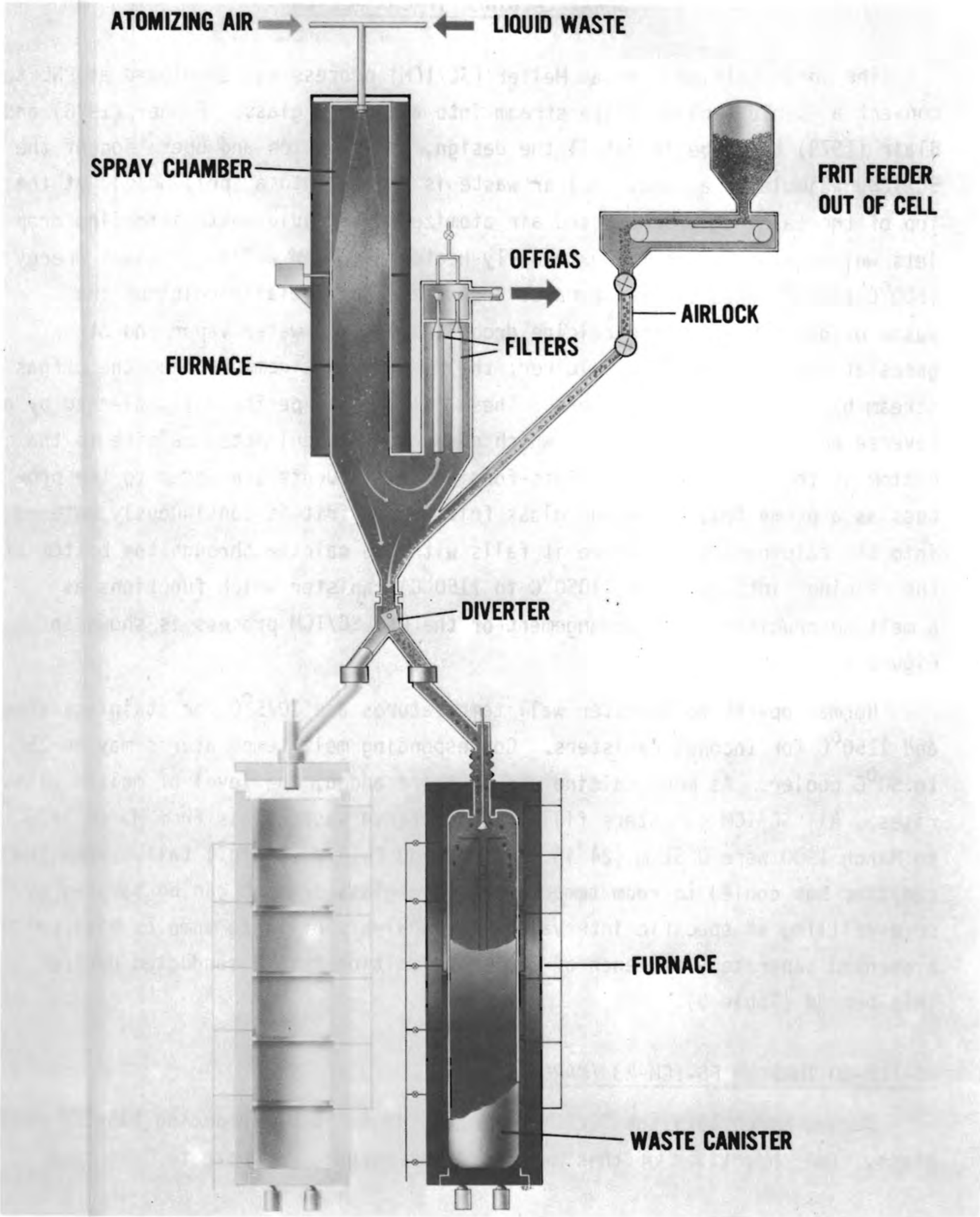

FIGURE 1. Spray Calciner/In-Can Melter

TABLE 5. SC/ICM Melting Trials (April 1979 - April 1980)

Melting Trial	Canister	Trial Date	Maximum Glass Melting Rate		Average Melting		Glass Produced, kg
			kg/h	$kg/h/m^2$	kg/h	$kg/h/m^2$	
FS-ICM-20	175(a)	March 1979	>75	>286	59	225	1107
-21	178		>75	>286	47	179	1089
-22	176		>85	>324	35	134	1075
-23	179(a)		80	305	46	176	1144
FS-ICM-24	177(a)	July 1979	60	229	45	172	1202
FS-ICM-26	174(a)(b)	March 1980	75	286	75	286	1136
-27	172(a)(b)		74	282	74	282	1024
FS-ICM-28	180(a)(b)	August 1980	85	324	85	324	1169
-29	181(a)(b)		20	76	20	76	805

(a) Canisters core-drilled for product analysis.

(b) TDS waste composition modified with organic compounds. Glass oxide composition unchanged.

operation of SC/ICM equipment, processing cycle times and maximum melting rates for the TDS-211 waste glass composition. Four canisters with internal heat transfer fins were filled with a total of 4415 kg of waste glass. The calcine/frit mixture melted rapidly without foaming. Calcine, however, tended to accumulate on top of the fin assembly, altering material distribution. After filling, each canister contained 2 to 5 kg of loose calcine material which was not incorporated into the glass. The maximum sustained melting rate was 85 kg/h (324 $kg/h/m^2$). The average melting rate for each canister was 35 to 60 kg/h (134 to 229 $kg/h/m^2$).

Samples from the first (175) and fourth (179) canisters were core-drilled for analysis. The normalized analytical results in Tables 6 and 7 show that significant compositional variation occurred both within and between canisters. The lower 0.30 m (1 ft) of both canisters was enriched in calcine constituents, Fe_2O_3, MnO_2 and NiO, containing approximately 46 wt% calcine oxides versus the defined 28 wt%. The estimated calcine content in the remainder of both canisters was low, 24 wt% in canister 175 and 19 wt% in canister 179 with a standard

TABLE 6. Normalized Composition of Canister 175 Samples

Core Height		Oxide wt%								
m	ft	SiO_2	Al_2O_3	B_2O_3	CaO(b)	Fe_2O_3	Li_2O(b)	Na_2O	MnO_2(b)	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0.06	0.2	31.1	8.3	5.3		32.4		5.9		3.8
0.15	0.5	34.9	7.0	5.9		28.6		7.5		3.1
0.30	1	45.8	3.3	8.1		18.5		10.2		1.1
0.61	2	48.5	2.6	8.6		15.5		10.6		1.1
0.91	3	48.2	2.6	8.5		15.5		11.0		1.2
0.21	4	46.4	2.8	8.4		12.4		15.7		1.2
AVE.(a)		47.2	2.8	8.4		15.4		11.9		1.2
STD. DEV.		1.3	0.3	0.2		2.5		2.6		0.1

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.
(b) Component analysis not reported.

TABLE 7. Normalized Composition of Canister 179 Samples

Core Height		Oxide wt%								
m	ft	SiO_2	Al_2O_3	B_2O_3	CaO(b)	Fe_2O_3	Li_2O(b)	Na_2O	MnO_2(b)	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0.06	0.25	31.7	7.4	5.7		27.4		11.8		2.9
0.15	0.5	31.8	7.4	5.7		27.4		11.8		2.9
0.61	2	45.0	3.3	9.0		12.2		16.3		1.2
0.91	3	45.3	2.8	8.9		12.5		16.6		1.0
1.21	4	45.8	3.0	9.1		11.2		16.9		1.1
1.52	5	46.7	2.8	9.1		10.2		17.0		1.1
AVE.(a)		45.7	3.0	9.0		11.5		16.7		1.1
STD. DEV.		0.7	0.2	0.1		1.0		0.3		0.1

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.
(b) Component analysis not reported.

deviation of ±2 wt%. Liquid-feed system problems encountered frequently during this run could account for the higher compositional variation noted in canister 175.

The calcine-rich glass in the bottom of both canisters contained 29 to 35 wt% of spinel crystals suspended in a glass matrix. Only acmite crystal phases were found above the 0.30-m (1-ft) level. The quantitative crystal content data is presented in Table 8. Glass near the canister centerline cooled more slowly (≈25°C/h) than that near the wall (70 to 90°C/h). Slower cooling rates can produce more devitrification and higher crystal contents. While canister 179 shows this behavior, canister 175 does not. Soxhlet leach data (72 h) shown in Table 9 does not indicate a strong relationship between soxhlet leach rate and acmite crystal content. Under soxhlet conditions, the calcine-rich glass containing spinel crystals had a lower weight loss than for the rest of the glass, 4.6 wt% versus 9.7 ± 2.4 wt%.

TABLE 8. Crystalline Content of Canister 175 and 179 Samples

Canister	Core Height		Crystalline Component wt%		Crystalline Phases Present
	m	ft	Canister Wall	Canister Centerline	
175	0	0	---	35%	Spinel[a]
	0.30	1	14%	14%	Acmite
	0.61	2	16%	10%	Acmite
	0.91	3	13%	13%	Acmite
	1.21	4	27%	28%	Acmite
179	0	0	---	29%	Spinel
	0.30	1	28%	28%	Acmite
	0.61	2	11%	28%	Acmite
	0.91	3	0%	15%	Acmite
	1.21	4	0%	26%	Acmite
	1.52	5	0%	---	---

(a) Spinel (Ni, Mn) Fe_2O_4

TABLE 9. Soxhlet Leach Data of Canister 175 and 179 Samples

Canister	Core Height m	Core Height ft	wt% Loss in 72 h Canister Wall	wt% Loss in 72 h Canister Centerline
	0	0	---	4.5
	0.30	1	9.2	12.5
175	0.61	2	11.6	11.9
	0.91	3	10.0	9.0
	1.21	4	12.8	11.1
AVE.			10.9	11.0
STD. DEV.			1.6	1.4
	0	0	---	4.8
	0.30	1	13.4	11.0
179	0.61	2	6.5	10.6
	0.91	3	6.2	7.3
	1.21	4	6.7	8.1
	1.52	5	6.6	---
AVE.			7.9	9.2
STD. DEV.			1.3	1.8

FS-ICM-24 (JULY 1979)

The objective of the second TDS-211 melting trial in the SC/ICM was to establish the maximum melting rate in a canister without heat transfer fins and evaluate the resulting waste glass product. Canister 177 was filled with 1202 kg of glass at a maximum rate of 65 kg/h (248 kg/h/m^2) with an average rate of 45 kg/h (172 kg/h/m^2). No melting problems were encountered; however, the canister contained 8% less glass than anticipated.

Core samples taken at various heights and depths from canister 177 were analyzed. Chemical composition as a function of location is shown in Table 10 and summarized in Table 11. Only one sample taken from the bottom wall of the

TABLE 10. Normalized Composition of Canister 177 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined				44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	0-7.6	0-3	31.7	6.4	5.6	4.6	25.8	2.1	13.4	6.4	3.5
		7.6-15.2	3-6	45.7	4.1	8.0	5.2	13.1	3.0	15.1	3.9	1.4
		15.2-22.9	6-9	48.8	3.2	8.8	5.1	10.3	3.5	15.4	3.1	1.3
		22.9-30.5	9-12	48.3	3.3	8.7	5.0	10.6	3.2	15.9	3.1	1.3
0.30	1	0-7.6	0-3	49.3	3.1	8.9	5.1	9.2	3.6	16.3	2.8	1.2
		7.6-15.2	3-6	49.2	3.1	8.8	5.1	9.3	3.4	16.5	2.9	1.2
		15.2-22.9	6-9	49.6	3.0	8.9	5.1	9.2	3.6	15.9	2.8	1.2
		22.9-30.5	9-12	49.0	3.1	8.8	5.2	10.2	3.5	15.3	3.1	1.3
0.61	2	0-7.6	0-3	50.0	3.0	9.0	5.3	9.6	3.4	15.0	3.0	1.3
		7.6-15.2	3-6	49.6	3.0	9.0	5.0	9.0	3.4	16.4	2.8	1.2
		15.2-22.9	6-9	49.4	3.1	8.9	5.0	9.2	3.7	16.2	2.8	1.2
		22.9-30.5	9-12	48.4	3.2	8.7	4.9	9.6	3.8	16.8	2.9	1.2
0.91	3	0-7.6	0-3	49.8	3.0	8.9	5.2	9.3	3.4	15.8	2.9	1.2
		7.6-15.2	3-6	49.7	3.0	8.9	5.2	9.5	3.5	15.5	2.9	1.3
		15.2-22.9	6-9	49.5	3.0	9.0	5.2	9.5	3.5	15.5	3.0	1.3
		22.9-30.5	9-12	49.2	3.1	8.7	5.0	9.4	3.2	16.8	2.9	1.3
1.21	4	0-7.6	0-3	49.5	3.0	8.8	5.1	9.3	3.5	16.1	2.9	1.2
		7.6-15.2	3-6	49.5	3.0	8.8	5.1	9.2	3.6	16.0	2.9	1.2
		15.2-22.9	6-9	49.5	3.0	8.9	5.1	9.4	3.6	15.8	2.9	1.3
1.52	5	0-7.6	0-3	49.3	3.0	8.8	4.9	8.9	3.8	16.7	2.8	1.3
		7.6-15.2	3-6	49.7	3.0	8.8	5.1	9.2	3.2	16.3	2.9	1.2

canister contained an estimated 49 wt% calcine similar to the previous SC/ICM trial. The bulk glass produced during this trial contained only 21 wt% calcine compared to the target value of 28 wt%. This descrepancy which could have resulted from either a reduced feed concentration or a higher frit feed rate largely accounts for the lower than anticipated glass production. There was little compositional variation throughout the canister from top to bottom or from wall to centerline.

The crystal content of canister 177 listed in Table 12 was comparable to that noted previously in canister 179. There was a limited distribution of spinel in the bottom (maximum 40 wt%). (Figure 2 is a photomicrograph of this unhomogeneous spinel-rich zone on the floor of the canister. The small bright crystals are spinel phase.) Acmite was found primarily along the bottom and

TABLE 11. Average Compositions of Canister 177 Samples(a)

Core Height m	Core Height ft	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.6	8.0	5.0	14.0	3.2	16.0	3.9	1.7
0	0	43.6	4.2	7.8	5.0	15.0	3.0	15.0	4.1	1.9
0.30	1	49.3	3.1	8.8	5.1	9.5	3.5	16.0	2.9	1.2
0.61	2	49.4	3.1	8.9	5.0	9.4	3.6	16.1	2.9	1.2
0.91	3	49.6	3.0	8.9	5.2	9.4	3.4	15.9	2.9	1.3
1.21	4	49.5	3.0	8.8	5.1	9.3	3.6	16.0	2.9	1.2
1.52	5	49.5	3.0	8.8	5.0	9.0	3.5	16.5	2.8	1.2
Core Depth cm	**Core Depth in.**									
0-7.6	0-3	49.6	3.0	8.9	5.1	9.3	3.5	16.0	2.9	1.2
7.6-15.2	3-6	49.5	3.0	8.8	5.1	9.2	3.4	16.1	2.9	1.2
15.2-22.9	6-9	49.5	3.0	8.9	5.1	9.2	3.6	15.8	2.9	1.2
22.9-30.5	9-12	48.9	3.1	8.8	5.1	9.7	3.5	16.3	3.0	1.3
AVE.(a)		49.5	3.0	8.8	5.1	9.3	3.5	16.1	2.9	1.2
STD. DEV.		0.3	0.1	0.1	0.1	0.2	0.1	0.5	0.1	0.1

(a) Data from lower 0.30-m (1-ft) level omitted from calculated averages.

centerline at 5 to 25 wt%. The microstructure of a sample from the center of the canister is shown in Figure 3. The numerous small dark crystals are acmite. Occasionally, spinel crystallites are found within the acmite phase. Both the size and distribution of acmite crystals within the sample indicate some product inhomogeneity. Microcracking appears to arise from the acmite crytals.

The chemical durability as measured by soxhlet and pH4 leach tests are shown in Tables 13 and 14. Weight loss was highest along the bottom and centerline. The presence of spinel did not significantly improve durability in this case. The average weight losses under pH4 and soxhlet conditions were 17.7% ± 2.8% and 2.37% ± 0.13%,respectively.

TABLE 12. Crystalline Content of Canister 177 Samples

Core Height		Core Depth, cm (in.)			
		0-7.6	7.6-15.2	15.2-22.9	22.9-30.5
m	ft	(0-3)	(3-6)	(6-9)	(9-12)
0	0	30-40% Spinel(a)	5-10% Spinel 5-10% Acmite(b)	10-15% Acmite ∿2% Spinel	25% Acmite 2% Spinel
0.30	1	0	0	0	25% Acmite
0.61	2	0	0	0	10-15% Acmite
0.91	3	0	<5% Acmite	5% Acmite	10% Acmite
1.21	4	0	0	0	---
1.52	5	0	0	---	---

(a) Spinel - $(Ni,Mn)\ Fe_2O_4$
(b) Acmite - $NaFe\ (SiO_3)_2$

FS-ICM-26 AND FS-ICM-27 (MARCH 1980)

The object of this melting trial was to assess the impact of organic additions in the waste stream on process equipment operation and waste glass properties. The new waste composition referred to as "modified TDS" incorporates three organic compounds, Na_4 EDTA, cornstarch, and anthracite coal in the TDS waste (Table 1). McElroy (1979, p. 25) showed that carbon compounds such as coal may not be significantly altered by calcination. At glass-melting temperatures they are oxidized, reducing the oxidation state of metal oxide waste constituents in the glass.

The March 1980 SC/ICM melting trial filled two finless canisters with 2160 kg of modified TDS-211 waste glass. The primary changes between the two canisters was the operating temperature of the ICM furnace. The first canister, 174, was run at 1075°C, which is considered the ICM reference operating temperature. The second canister, 172, was fabricated from Inconel and run at 1150°C. Canister 174 was filled at approximtely 75 kg/h (286 kg/h/m^2). Production was not limited by foaming, and no unmelted material was noted on the surface of the glass. Canister 172 was to be filled at 100 kg/h (382 kg/h/m^2)

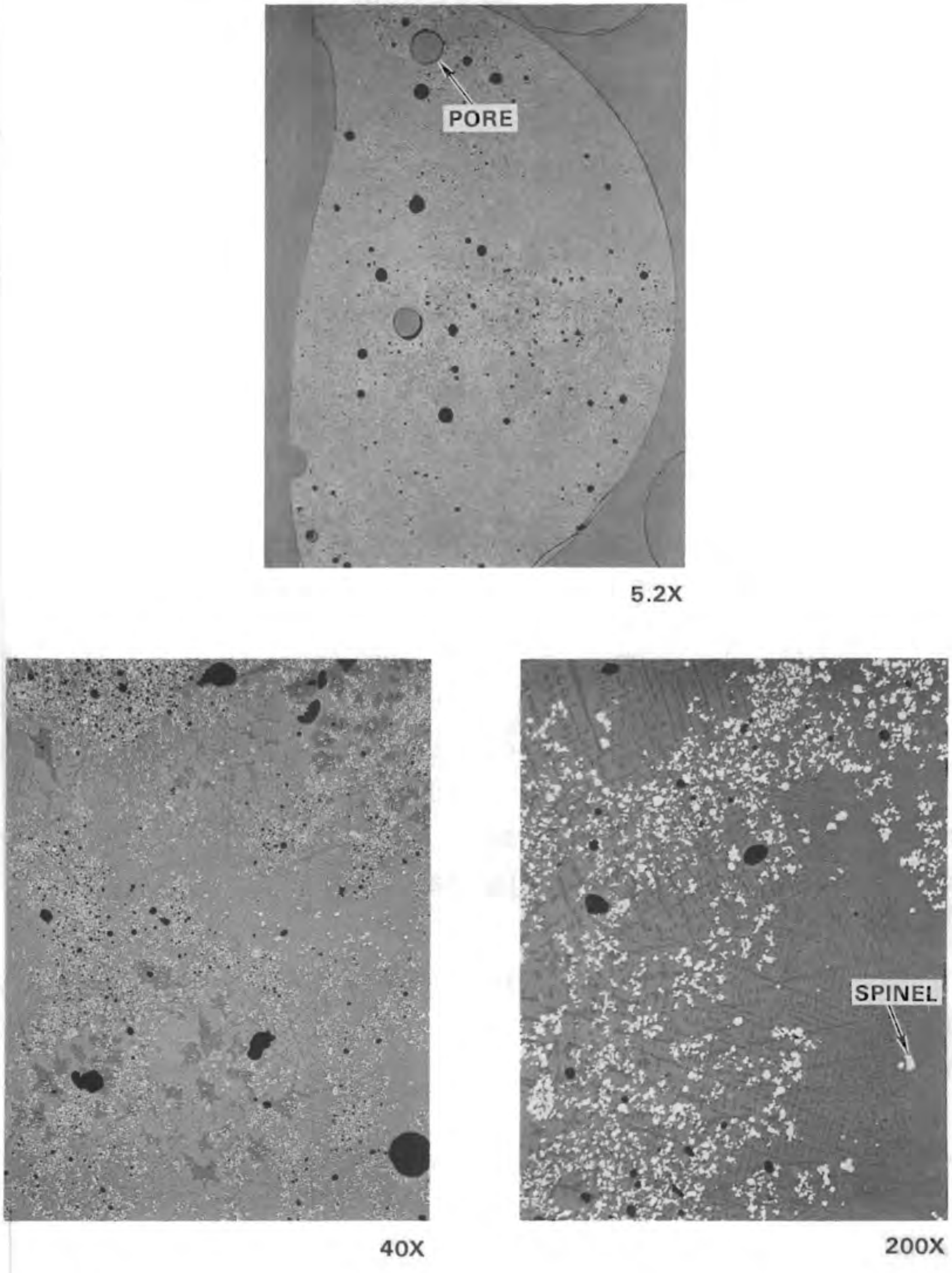

FIGURE 2. Microstructure of Canister 177 Sample [Height 0 m (0 ft); Depth 0-7.6 cm (0-3 in.)]

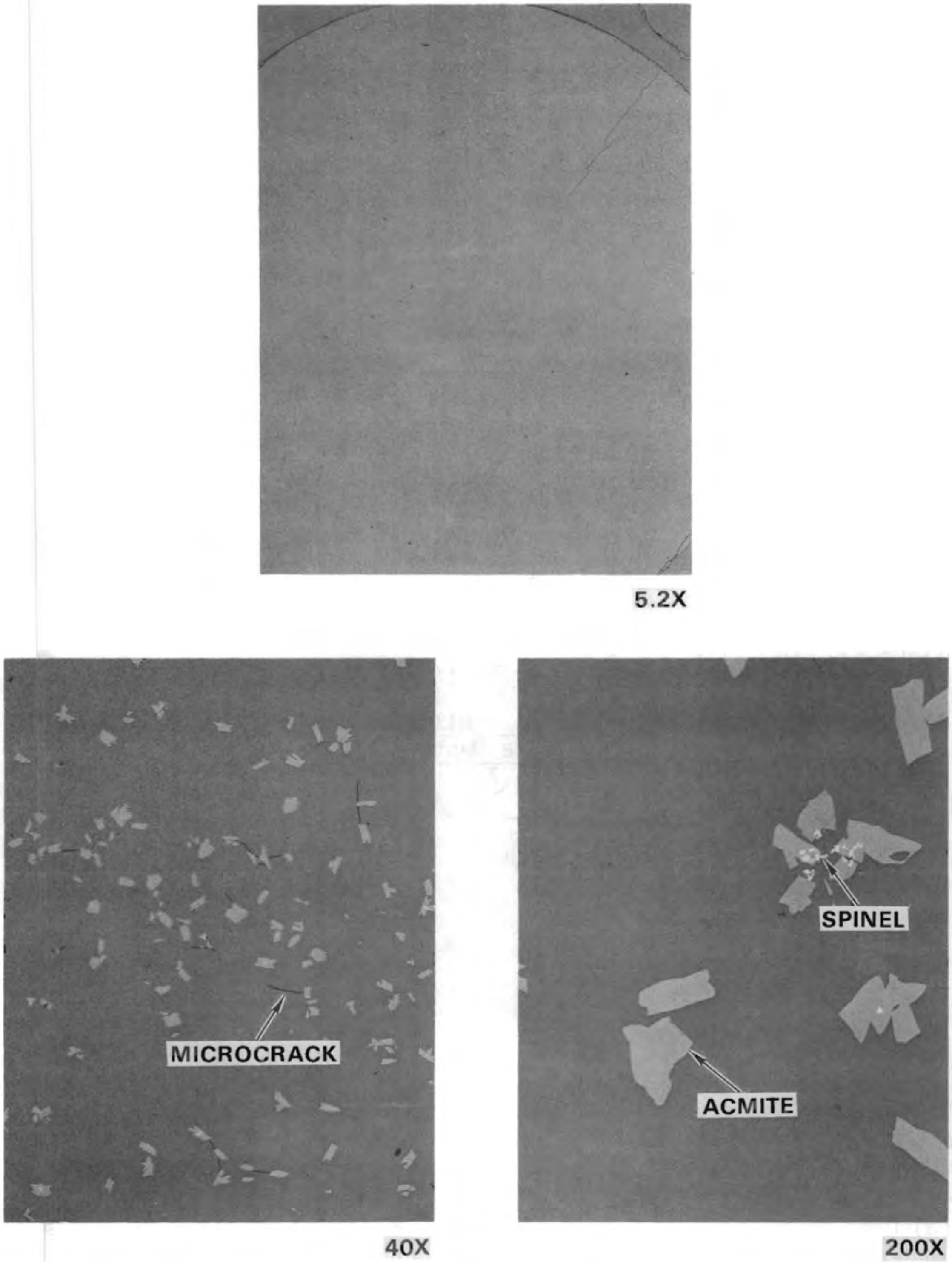

FIGURE 3. Microstructure of Canister 177 Sample [Height 0.61 m (2 ft); Depth 22.9-30.5 cm (9-12 in.)]

TABLE 13. Weight Loss of Canister 177 Samples Under pH4 Conditions

Core Height		wt% Loss				
		Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	---	55.8	51.2	34.9	47.3
0.30	1	11.7	15.0	9.8	31.1	17.2
0.61	2	16.7	12.0	14.5	35.3	19.6
0.91	3	13.4	21.2	18.6	20.3	8.4
1.21	4	17.1	16.6	16.3	---	16.8
1.52	5	18.6	14.5	---	---	16.5
AVE.		15.5	15.9	14.9	28.9	

Mean pH4 Weight Loss(a) 17.7% ± 2.8%

(a) Data from lower 0.30-m (1-ft) level omitted from calculated averages.

TABLE 14. Weight Loss of Canister 177 Samples Under Soxhlet Conditions

Core Height		wt% Loss				
		Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	2.41	2.39	3.47	3.02	2.82
0.30	1	1.86	1.78	1.71	2.21	1.89
0.61	2	2.21	2.19	2.57	2.91	2.47
0.91	3	2.32	2.44	2.88	2.79	2.61
1.21	4	2.31	2.24	2.20	---	2.25
1.52	5	2.59	2.70	---	---	2.65
AVE.(a)		2.26	2.27	2.34	2.64	

Mean Soxhlet Weight Loss(a) 2.37% ± 0.13%

(a) Data from lower 0.30-m (1-ft) level omitted from calculated averages.

to take advantage of higher melting temperature. After 5 h of operation, the frit feeder electronics malfunctioned, adding less frit than desired. This condition was discovered only after completing the run.

After cooling, both canisters were cut open and samples were core-drilled. The glass in canister 174 was shiny black with a dull-mat textured zone at the bottom. Canister 172 contained a duller appearing glass. Both contained a number of 6- to 16-mm-dia metallic buttons at the bottom of the canister. The buttons shown in Figure 4 contained 70 to 75 wt% metal: 90% nickel with traces of iron, lead and zinc. A white crystalline coating was found on the exposed canister walls and exposed glass surfaces of both canisters. This coating contained 50 to 89% water soluble salts, mainly sodium chloride with the remainder a mixture of frit and calcine oxides. The normalized chemical analysis for canisters 174 and 172 glass samples are presented in Tables 15 and 16. Summaries of this data in Tables 17 and 18 show that the glass in the bottom of both canisters contained 42 to 45 wt% calcine. The microstructures of these bottom samples were more homogeneous than in canister 177, containing larger spinel crystals generally surrounded by acmite crystals (Figures 5 and 6). The bulk glass in both canisters again contained less than the defined 28 wt% calcine. However, greater compositional variation was found in canister 174

<u>FIGURE 4</u>. Metallic Buttons Removed from Bottom of Canisters 172 and 174

TABLE 15. Normalized Composition of Canister 174 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined				44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	0-10.2	0-4	40.4	5.8	6.6	5.6	16.2	2.6	15.8	3.9	2.5
		10.2-20.3	4-8	41.9	5.7	6.8	5.4	15.0	2.9	16.8	3.0	2.0
		20.3-30.5	8-12	35.5	6.4	5.7	5.2	20.8	2.3	15.5	4.8	3.3
0.30	1	0-10.2	0-4	48.6	3.8	8.2	5.2	11.1	3.4	13.5	4.2	1.7
		10.2-20.3	4-8	46.2	4.2	7.7	5.3	12.0	3.2	16.0	3.3	1.6
		20.3-30.5	8-12	43.1	5.8	7.2	5.8	15.9	2.8	14.0	2.7	2.2
0.61	2	0-10.2	0-4	48.5	3.6	8.2	5.3	10.8	3.0	14.3	4.2	1.6
		10.2-20.3	4-8	46.8	3.6	7.9	5.0	10.5	3.3	17.0	4.0	1.6
		20.3-30.5	8-12	46.1	3.8	7.7	5.1	11.3	3.3	17.3	3.4	1.5
0.91	3	0-10.2	0-4	46.6	3.5	7.8	5.0	10.5	3.3	17.3	4.0	1.6
		10.2-20.3	4-8	46.4	3.6	7.8	5.0	10.9	3.3	17.0	3.8	1.6
		20.3-30.5	8-12	44.5	4.3	7.4	5.4	13.4	3.2	16.5	2.9	2.1
1.21	4	0-10.2	0-4	47.7	3.6	8.0	5.2	10.6	3.1	15.8	4.1	1.6
		10.2-20.3	4-8	46.3	3.7	7.9	5.1	11.0	3.4	17.1	3.7	1.5
		20.3-30.5	8-12	43.3	4.3	7.2	5.2	13.1	3.0	18.6	3.0	2.0
1.52	5	0-10.2	0-4	47.0	3.5	8.1	5.2	11.1	3.0	15.7	4.2	1.7

TABLE 16. Normalized Composition of Canister 172 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined				44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	0-10.2	0-4	39.4	5.9	6.5	5.5	18.2	2.5	15.0	4.9	1.8
		10.2-20.3	4-8	39.7	5.9	6.5	5.4	18.0	2.5	14.9	4.9	1.7
		20.3-30.5	8-12	36.2	6.3	5.9	5.1	22.7	2.3	13.4	5.0	2.8
0.30	1	0-10.2	0-4	43.6	4.4	7.3	4.9	15.9	3.0	15.1	3.6	1.7
		10.2-20.3	4-8	42.1	4.7	6.8	5.7	16.8	2.6	15.0	4.0	2.0
		20.3-30.5	8-12	41.8	4.9	6.8	5.5	17.5	2.6	14.2	4.1	2.1
0.61	2	0-10.2	0-4	44.3	4.4	7.4	5.2	16.2	2.8	13.9	3.8	1.6
		10.2-20.3	4-8	44.6	4.3	7.3	5.2	15.6	2.9	14.3	3.7	1.5
		20.3-30.5	8-12	43.0	4.3	6.8	5.7	17.0	2.6	14.3	3.7	2.2
0.91	3	0-10.2	0-4	44.3	4.5	7.4	5.3	16.0	2.7	13.9	3.8	1.6
		10.2-20.3	4-8	43.2	4.2	7.0	5.6	16.5	2.7	14.6	3.6	2.1
1.21	4	0-10.2	0-4	43.9	4.5	7.4	5.4	16.6	3.0	13.1	3.7	2.0
		10.2-20.3	4-8	43.2	4.4	7.2	5.5	16.8	2.7	14.3	3.4	2.0

TABLE 17. Average Composition of Canister 174 Samples

Core Height		Oxide wt%								
m	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	39.3	6.0	6.4	5.4	17.3	2.6	16.0	3.9	2.6
0.30	1	46.0	4.6	7.7	5.4	13.0	3.1	14.5	3.4	1.8
0.61	2	47.1	3.7	7.9	5.1	10.9	3.2	16.2	3.9	1.6
0.91	3	45.8	3.8	7.7	5.1	11.6	3.3	16.9	3.6	1.8
1.21	4	45.8	3.9	7.7	5.2	11.6	3.2	17.2	3.6	1.7
1.52	5	47.0	3.5	8.1	5.2	11.1	3.0	15.7	4.2	1.7
Core Depth(a)										
cm	in.									
0-10.2	0-4	47.7	3.6	8.1	5.2	10.8	3.2	15.3	4.1	1.6
10.2-20.3	4-8	46.4	3.8	7.8	5.1	11.1	3.3	16.8	3.7	1.6
20.3-30.5	8-12	44.2	4.6	7.4	5.4	13.4	3.1	16.6	3.0	2.0
AVE.(a)		46.2	3.9	7.8	5.2	11.7	3.2	16.2	3.6	1.7
STD. DEV.		1.7	0.6	0.3	0.2	1.6	0.2	1.5	0.5	0.2

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.

samples. In general, the calcine-rich region on the canister bottom contained 5 to 10 wt% spinel compared to 0 to 15 wt% acmite in the rest of either canister (Tables 19 and 20). The microstructure of the acmite-containing zones in canister 174 and 172 are shown in Figures 7 through 10. Although spinel was not identified in these samples by x-ray diffraction, small crystallines are seen associated with many acmite crystals. The glass in canister 174 contained less calcine and was processed at lower temperatures than the glass in canister 172. This resulted in smaller spinel and acmite crystals in canister 174. On the other hand, canister 172, besides containing larger crystals, displayed considerable porosity and more microcracking. This accounts for the dull appearance of the glass noted when canister 172 was opened.

TABLE 18. Average Composition of Canister 172 Samples

Core Height m	Core Height ft	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	38.4	6.0	6.3	5.3	19.6	2.4	14.4	4.9	2.1
0.30	1	42.5	4.7	7.0	5.4	16.7	2.7	14.8	3.9	1.9
0.61	2	44.0	4.3	7.2	5.4	16.3	2.8	14.2	3.7	1.8
0.91	3	43.8	4.4	7.2	5.4	16.2	2.7	14.2	3.7	1.8
1.21	4	43.6	4.4	7.3	5.4	16.7	2.8	13.7	3.6	2.0
Core Depth cm	**Core Depth in.**									
0-10.2	0-4	44.0	4.4	7.4	5.2	16.2	2.9	14.0	3.7	1.7
10.2-20.3	4-8	43.2	4.4	7.1	5.5	16.4	2.7	14.6	3.7	1.9
20.3-30.5	8-12	42.4	4.6	6.8	5.6	17.2	2.6	14.2	3.9	2.2
AVE.(a)		43.4	4.5	7.1	5.4	16.5	2.8	14.3	3.7	1.9
STD. DEV.		0.9	0.2	0.3	0.2	0.6	0.2	0.6	0.2	0.2

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.

Chemical durability did not appear to coincide with composition, microstructure or crystal content variation. The average soxhlet weight loss values were 2.41 and 3.40 wt% for canisters 174 and 172 while pH4 weight loss values for both were approximately 84.7 wt% loss ± 0.5 wt%. This data is presented in Tables 21 through 24. The presence of the spinel phase on the canister floor again did not alter the product durability.

FS-ICM-28 AND FS-ICM-29 (AUGUST 1980)

The object of these melting trials was to determine the effect that additional mixing would have on product quality. FS-ICM-28 was carried out in a typical manner except that the melt was continuously mixed by an air sparge. Canister 180 was filled with 1164 kg of glass at 85 kg/h (324 $kg/h/m^2$). In the second trial, FS-ICM-29, the spray calciner was operated at lower barrel

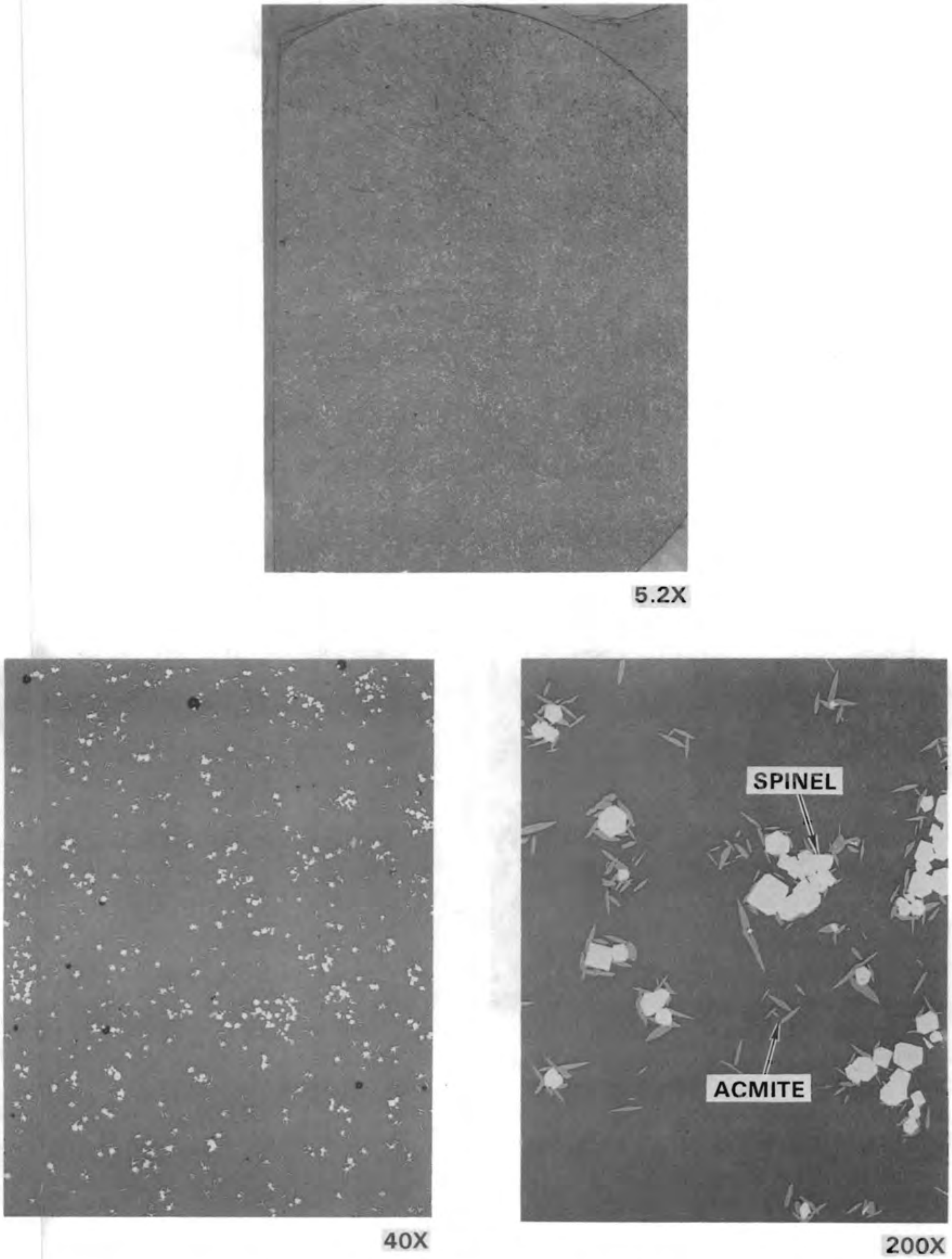

FIGURE 5. Microstructure of Canister 174 Sample [Height 0 m (0 ft); Depth 20.3-30.5 cm (8-12 in.)]

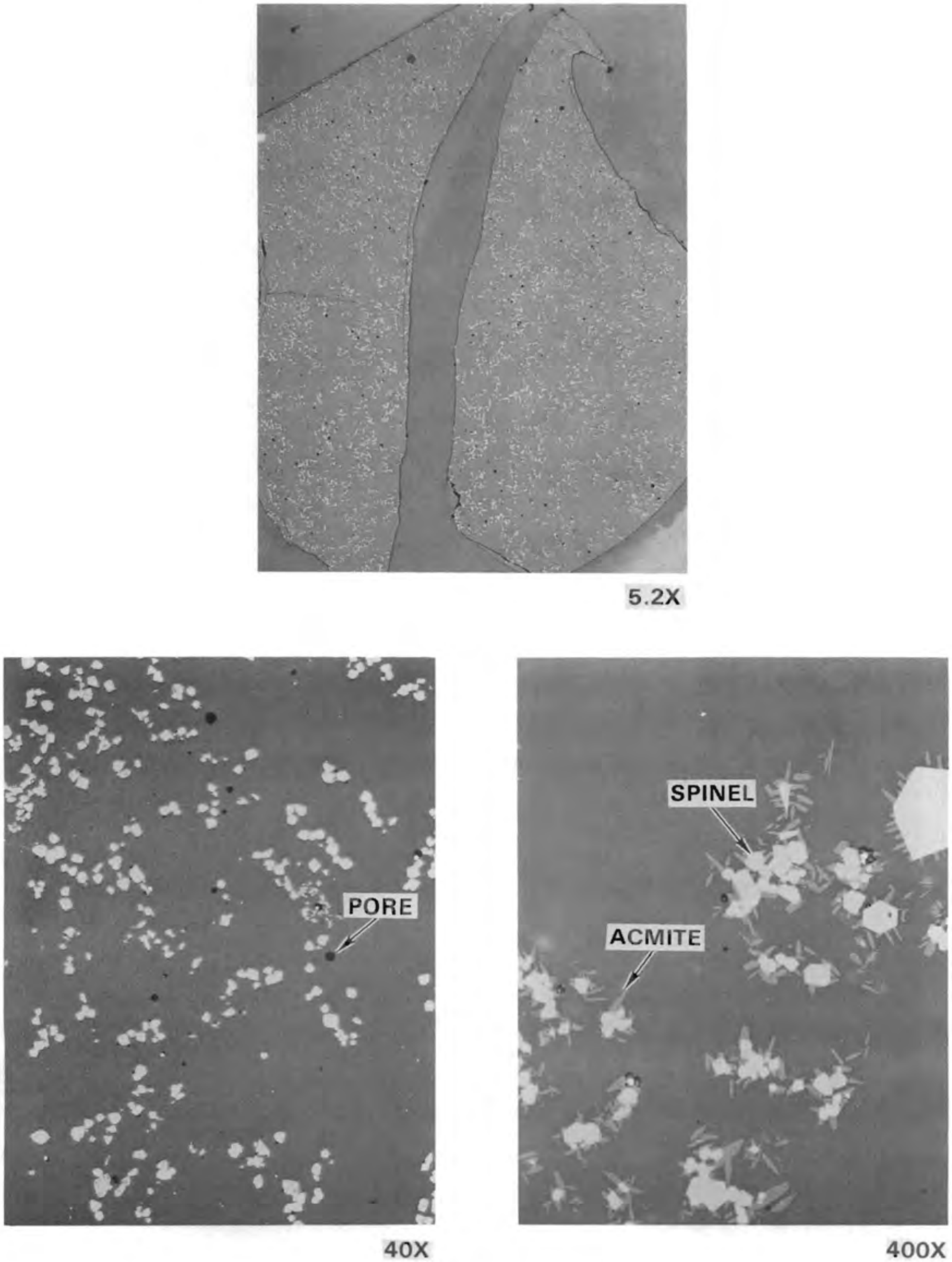

FIGURE 6. Microstructure of Canister 172 Samples [Height 0 m (0 ft); Depth 20.3-30.5 cm (8-12 in.)]

TABLE 19. Crystalline Content of Canister 174 Samples

Core Height		Core Depth, cm (in.)		
m	ft	0-10.2 (0-4)	10.2-20.3 (4-8)	20.3-30.5 (8-12)
0	0	5% Spinel(a)	5% Spinel <5% Acmite(b)	5-10% Spinel
0.30	1	0	<5% Acmite	5-10% Spinel
0.61	2	<5% Acmite	10% Acmite	10-15% Acmite
0.91	3	<5% Acmite	5% Acmite	5% Acmite
1.21	4	0	5-10% Acmite	0
1.52	5	0	---	---

(a) Spinel - $(Ni, Mn) Fe_2O_4$
(b) Acmite - $NaFe (SiO_3)_2$

TABLE 20. Crystalline Content of Canister 172 Samples

Core Height		Core Depth, cm (in.)		
m	ft	0-10.2 (0-4)	10.2-20.3 (4-8)	20.3-30.5 (8-12)
0	0	<2% Spinel(a)	0	5% Spinel
0.30	1	5% Acmite(b)	<5% Acmite	<5% Acmite
0.61	2	<5% Acmite	<5% Acmite	5% Acmite
0.91	3	<2% Acmite	5% Acmite	
		0	0	

(a) Spinel - $(Ni, Mn) Fe_2O_4$
(b) Acmite - $NaFe (SiO_3)_2$

temperatures to allow the frit to be sprayed into the barrel along with the waste constituents. The low temperature ($400^\circ C$ instead of 700 to $800^\circ C$) prevented the frit from melting and sticking to the calciner barrel, and also limited the production rate to 20 kg/h (76 $kg/h/m^2$). Canister 181 was filled with 805 kg of waste glass. Product samples have been taken; however, no analytical results are available.

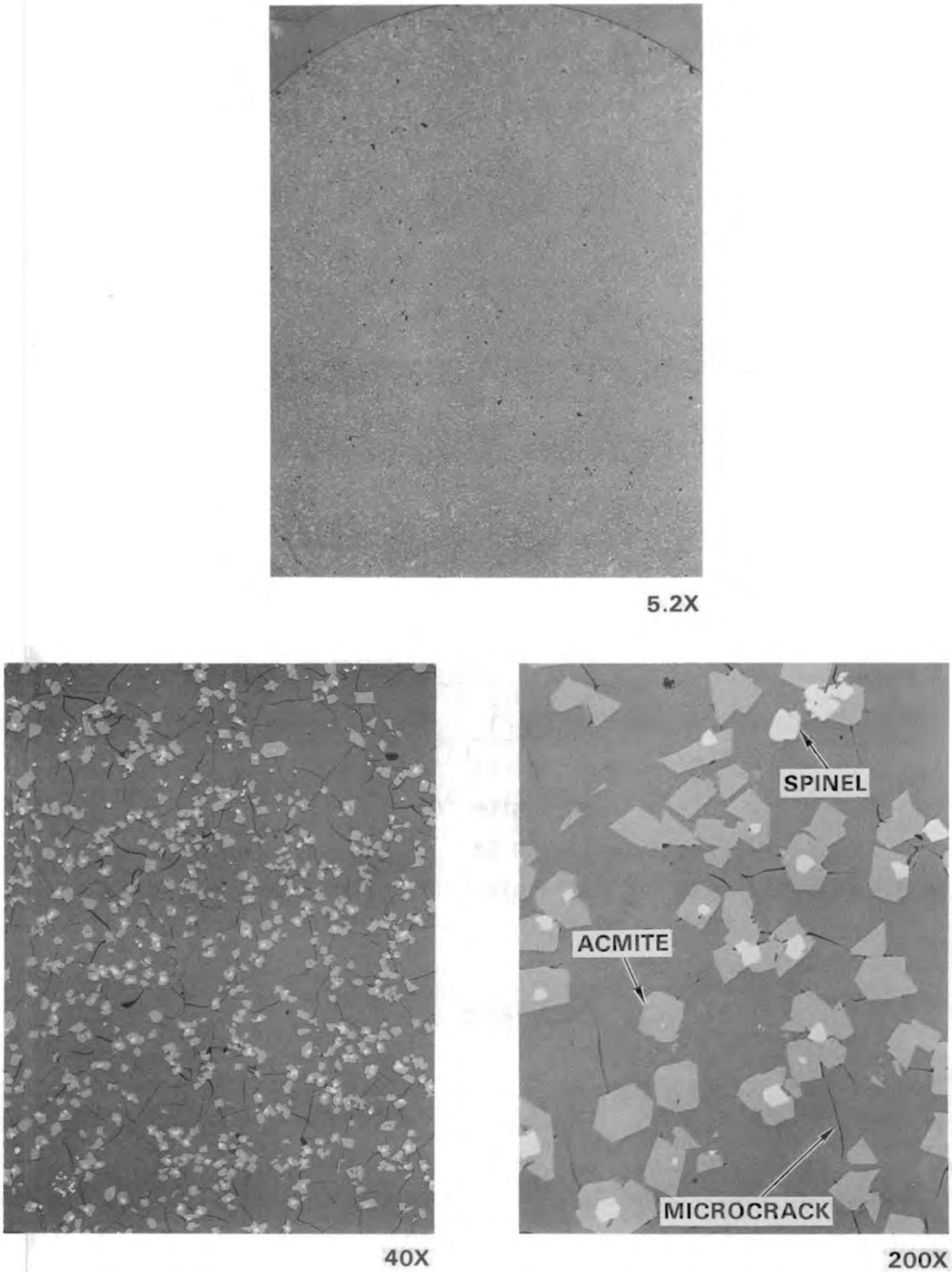

FIGURE 7. Microstructure of Canister 174 Sample [Height 0.61 m (2 ft); Depth 20.3-30.5 cm (8-12 in.)]

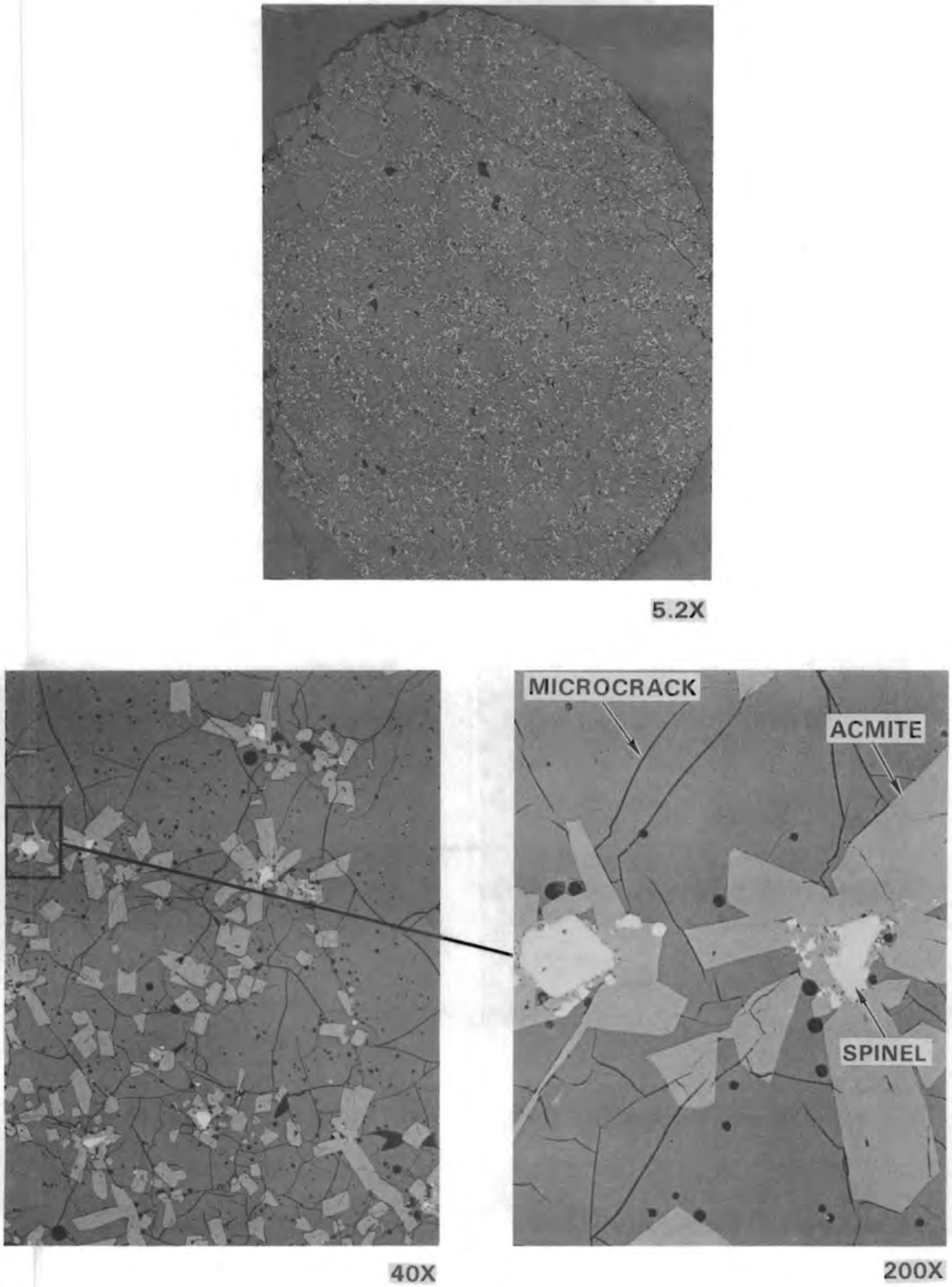

FIGURE 8. Microstructure of Canister 172 Sample [Height 0.61 m (2 ft); Depth 20.3-30.5 cm (8-12 in.)]

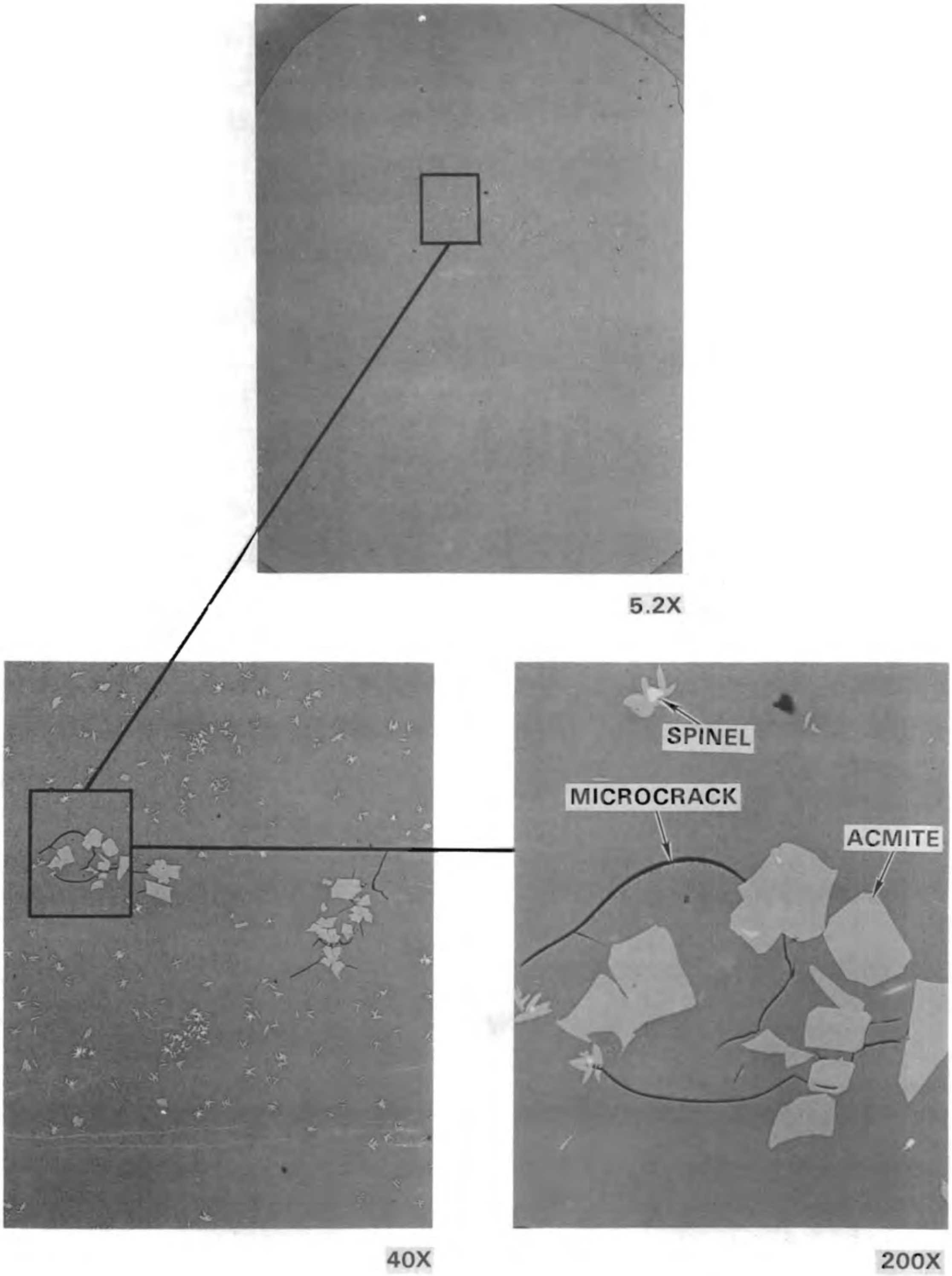

FIGURE 9. Microstructure of Canister 174 Sample [Height 1.21 m (4 ft); Depth 0-10.2 cm (0-4 in.)]

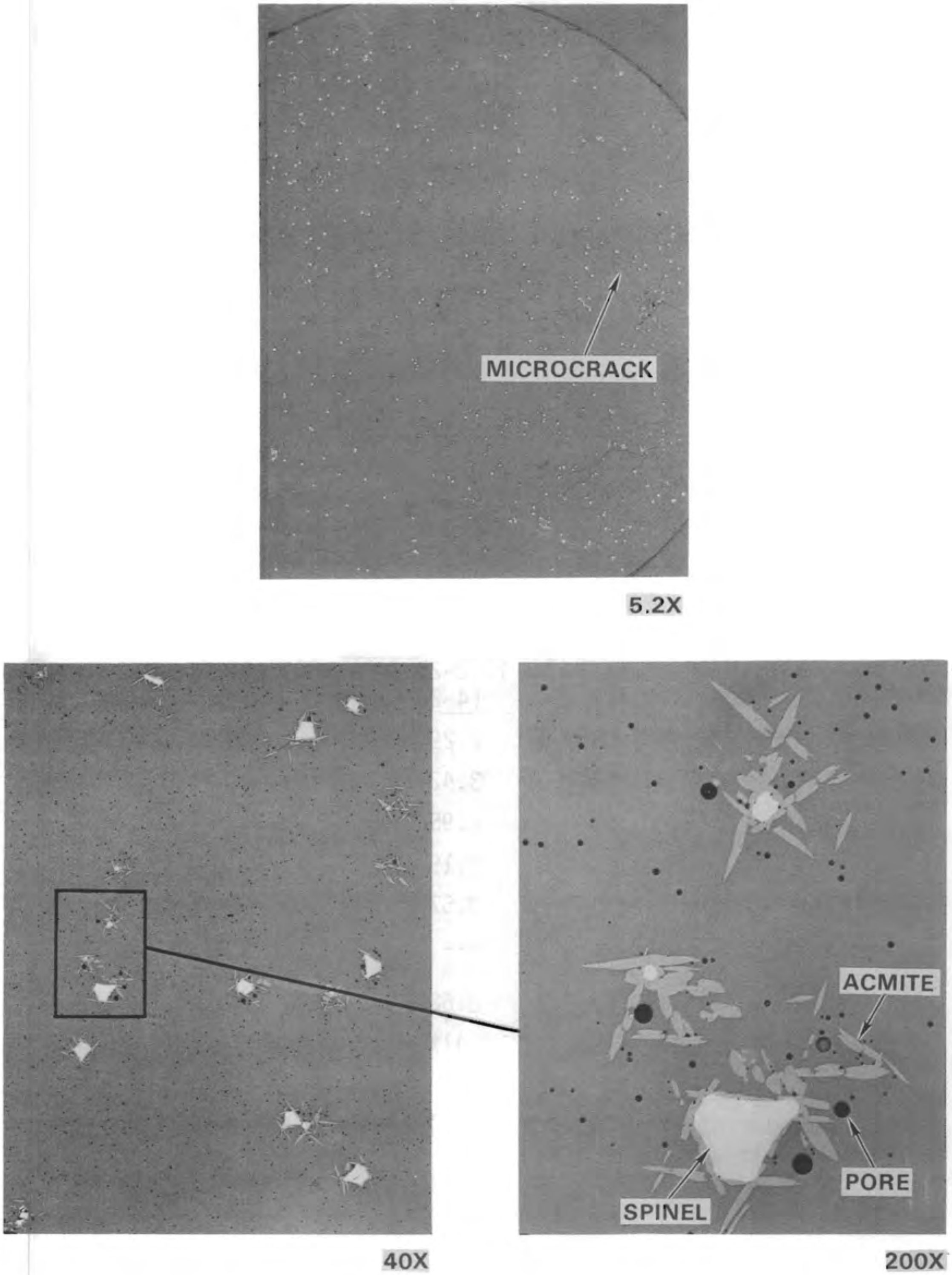

FIGURE 10. Microstructure of Canister 172 Sample [Height 1.21 m (4 ft); Depth 0-10.2 cm (0-4 in.)]

TABLE 21. Weight Loss of Canister 174 Samples Under pH4 Conditions

Core Height		wt% Loss Core Depth, cm (in.)			
m	ft	0-10.2 (0-4)	10.2-20.3 (4-8)	20.3-30.5 (8-12)	Ave.
0	0	---	---	---	
0.30	1	91.3	78.5	91.4	87.1
0.61	2	90.5	74.6	75.3	80.1
0.91	3	85.9	82.0	82.2	83.4
1.21	4	94.8	79.7	---	87.2
AVE.		90.6	78.7	83.0	

Mean pH4 Weight Loss 84.2% ± 4.9%

TABLE 22. Weight Loss of Canister 174 Samples Under Soxhlet Conditions

Core Height		wt% Loss Core Depth, cm (in.)			
m	ft	0-10.2 (0-4)	10.2-20.3 (4-8)	20.3-30.5 (8-12)	Ave.
0	0	2.18	2.29	2.09	2.19
0.30	1	2.03	3.42	2.03	2.49
0.61	2	1.81	2.95	3.25	2.67
0.91	3	1.78	2.19	2.95	2.31
1.21	4	2.10	2.57		2.34
1.52	5	2.05	---	---	2.05
AVE.(a)		1.99	2.68	2.66	

Mean Soxhlet Weight Loss(a) 2.41% ± 0.20%

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.

TABLE 23. Weight Loss of Canister 172 Samples Under pH4 Conditions

Core Height		wt% Loss			
		Core Depth, cm (in.)			
		0-10.2	10.2-20.3	20.3-30.5	
m	ft	(0-4)	(4-8)	(8-12)	Ave.
0	0	88.5	91.7	87.0	89.0
0.30	1	88.2	79.2	86.3	84.6
0.61	2	92.6	77.1	76.8	82.2
0.91	3	88.1	74.9	---	81.5
1.21	4	89.6	88.0	---	88.8
AVE.(a)		89.6	79.8	81.6	

Mean pH4 Weight Loss(a) 85.2% ± 3.0%

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.

TABLE 24. Weight Loss of Canister 172 Samples Under Soxhlet Conditions

Core Height		wt% Loss			
		Core Depth, cm (in.)			
		0-10.2	10.2-20.3	20.3-30.5	
m	ft	(0-4)	(4-8)	(8-12)	Ave.
0	0	3.32	2.91	1.90	2.71
0.30	1	2.77	3.62	2.80	3.06
0.61	2	2.33	5.82	4.39	4.18
0.91	3	2.99	5.52	---	4.26
1.21	4	2.49	2.88	---	2.68
AVE.(a)		2.78	4.15	3.03	

Mean Soxhlet Weight Loss(a) 3.40% ± 0.61%

(a) Data from lower 0.30 m (1 ft) omitted from calculated averages.

LIQUID-FED CERAMIC MELTER

From July 1979 to January 1980 three melting trials producing over 6000 kg of TDS-211 waste glass were run in the Liquid-Fed Ceramic Melter (LFCM). The LFCM is a joule-heated ceramic melter developed by PNL which converts a waste/frit mixture into glass. An alternating current is passed between electrodes submerged in molten glass. The heat generated melts the floating waste/frit mixture. As glass is melted and poured into canisters 0.61 to 0.91 m (2 to 3 ft) in dia to cool, more waste/frit mixture is fed to the melter. Figure 11 shows the LFCM process operated at PNL. Details of equipment design and operation are provided by Buelt and Chapman (1978; 1979).

The LFCM may process simulated waste in either a dry powder/calcine or liquid form. The feeding of a liquid waste stream into a joule-heated melter is expected to become the reference vitrification process for the SRP program. The first two LFCM melting trials, SRL-13 and SRL-14, utilized dry TDS waste premixed with frit. This was done to evaluate the impact of improved feed mixing on the product. The third trial, SRL-LF-1, processed the TDS liquid waste stream directly without prior calcination. Molten glass was regularly sampled from the pouring stream to monitor product composition. Glass samples were also core-drilled from cooled canisters. Feed samples were occasionally taken and analyzed. The results from each melting trial will be presented separately.

SRL-13 (JULY 1979)

During 5 continuous days of operation, LFCM melting trial SRL-13 filled three canisters with a total of 4250 kg of TDS-211 waste glass. The objective was to determine the maximum production rate in the LFCM when feeding a well-mixed calcine/frit batch. The maximum melting rate of 70 kg/h and average rate of 52 kg/h (50 kg/h/m^2) were limited by the production of glass foam within the melter. Feeding was stopped during the trial until the foam had dissipated.

The TDS liquid waste used in this trial was spray dried in a 250^{o}C to 600^{o}C spray drier. The resulting waste powder had a larger weight loss on

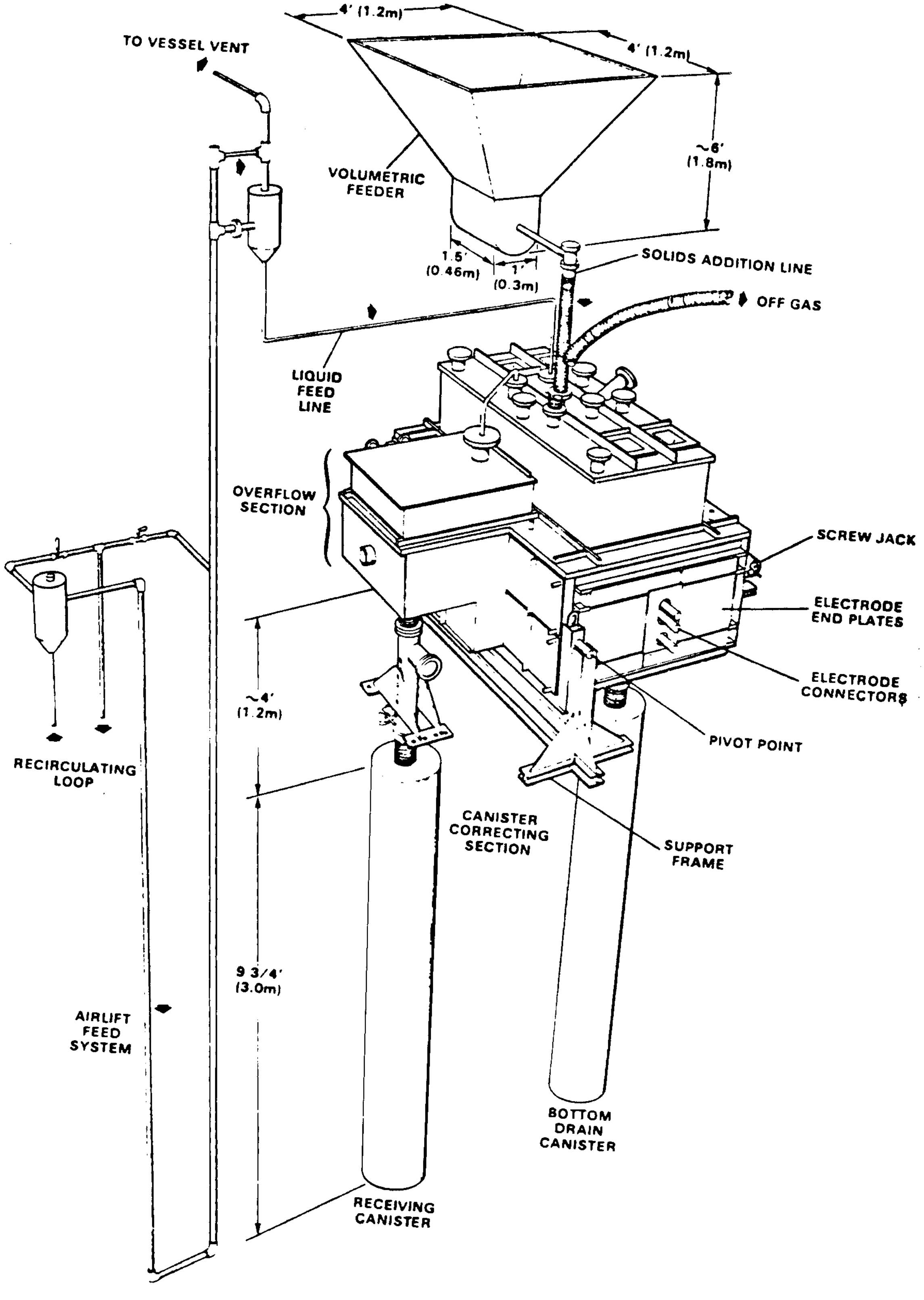

FIGURE 11. Liquid-Fed Ceramic Melter

heating than calcined waste, 30 wt% versus 3 wt%. This increased weight loss was not found directly responsible for LFCM foaming. Chemical analysis of liquid and resulting powder indicated that no batching errors occurred in the waste preparation. The dried powder and frit were mixed in barrel mixers to produce a 28/72 calcine/frit oxide ratio before it was fed to the LFCM.

The composition of the glass in the LFCM at the start of SRL-13 was significantly different from TDS-211. Analysis of grab samples collected periodically show the gradual composition change during the trial (Table 25). Figure 12 plots the percent of TDS-211 glass in any sample estimated from ZnO concentration as a function of glass production. To replace 90% of the glass in the LFCM with a new composition, it is necessary to process approximately 2500 kg of glass. When the composition of the starting glass was factored out, an estimate of the TDS-211 glass composition in each sample could be calculated (Table 26). The glass product contained an estimated TDS calcine content of 24 to 27 wt%, only slightly lower than the defined 28 wt%.

Samples were core-drilled from canister 148. Glass in this canister largely represented the feed during this trial and contained <5 wt% of the original LFCM glass composition. The normalized data for the major constituents are listed in Table 27. The glass in canister 148 shows no significant variation in composition with sample location as seen in Table 28 and had an average annealed glass density of 2.742 ± 0.007 g/cc.

Grab samples were always amorphous. Only a trace of an unidentified crystalline phase was detected in some core-drilled canister samples along the bottom and centerline. No significant devitrification occurred on cooling. Neither pH4 nor soxhlet leach results demonstrate significant variations related to sample position within the canister (Tables 29 and 30). Under pH4 conditions, canister 148 samples lost 75.1 ± 6.0 wt%. Soxhlet conditions produced a weight loss 1.78 ± 0.12 wt%.

SRL-14 (OCTOBER 1979)

The previous LFCM melting trial, SRL-13, was hampered by foaming of the calcine/frit feed during melting. Laboratory work indicated that the addition of 1 to 5 wt% cornstarch as a reducing agent would significantly reduce

TABLE 25. Normalized Composition SRL-13 Grab Samples

Date	Time	Glass Produced, kg	Oxide wt%															
			SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO	BaO	CuO	MgO	SrO	TiO_2	ZnO	Cr_2O_3
Defined			44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7	0	0	0	0	0	0	0
7/23	0430	0																
7/23	0737	92	44.7	7.7	11.5	4.1	5.2	5.0	8.8	1.8	0.5	0.7	0.6	0.6	0.6	0.9	6.3	0.2
7/24	0645	779	45.3	6.5	10.5	4.2	7.3	4.8	11.3	2.4	0.7	0.5	0.5	0.5	0.4	0.6	4.2	0.2
7/24	1804	1179	45.4	5.5	9.7	4.5	9.7	4.0	12.5	3.1	1.0	0.3	0.3	0.4	0.2	0.4	2.6	0.1
7/25	1110	1461							CANISTER 124 OUT									
7/26	0640	2385	46.1	4.4	8.7	4.6	11.7	3.5	14.3	3.6	1.3	0.1	0.1	0.2	0.1	0.1	0.8	0.1
7/26	1530	2779							CANISTER 150 OUT									
7/27	0530	3321	46.2	4.0	8.3	4.9	12.2	3.3	15.0	3.8	1.3	---	0.1	0.2	---	0.1	0.4	0.1
7/27	2344	4232	46.0	3.9	8.5	4.7	12.6	3.3	14.9	3.8	1.4	---	0.1	0.2	---	0.1	0.3	0.1
									CANISTER 148 OUT									

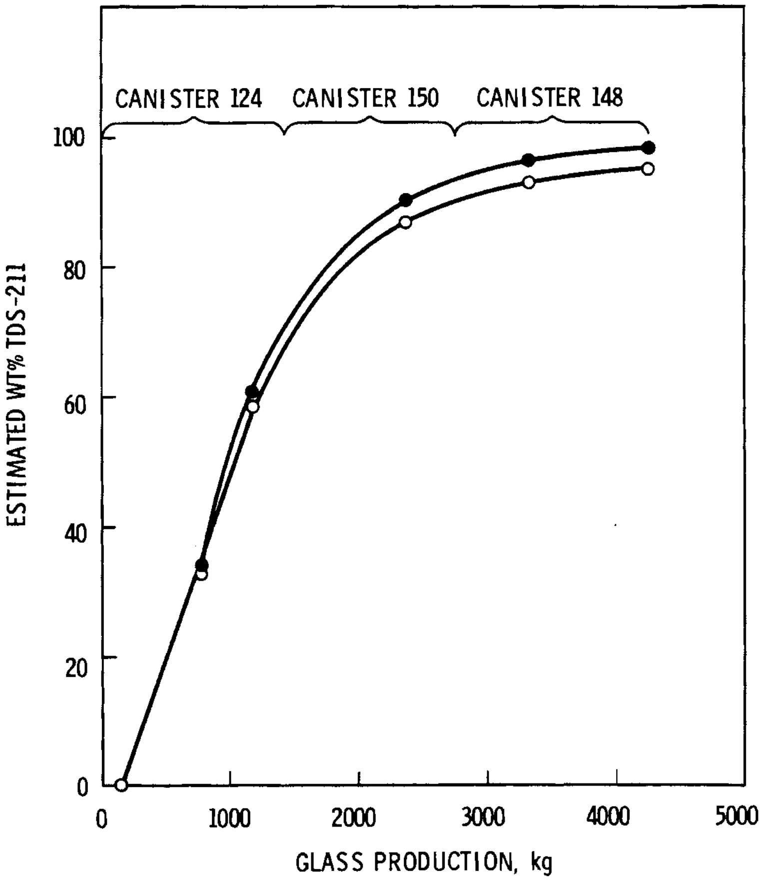

FIGURE 12. TDS-211 Content of SRL-13 Grab Samples

foaming and increase production rates. The objective of melting trial SRL-14 was to determine the optimum cornstarch loading for the SRL-13 batch. During 3 d of operation, one 0.91-m (36-in.)-dia canister was filled with 2480 kg of glass at an average sustained rate of 92 kg/h (92 kg/h/m^2). Foaming was drastically reduced by the addition of 1 wt% cornstarch. Product samples were not analyzed for this run.

SRL-LF-1 (JANUARY 1980)

In January 1980 the LFCM was run for 3 d by directly feeding simulated TDS-liquid waste and frit. The objectives of the run were to evaluate the performance of the LFCM process with direct liquid feeding and the properties

TABLE 26. Estimated TDS-211 Composition (SRL-13)

Samples (Date)	Time	Percent TDS-211	Oxide wt%								
			SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined			44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
7/24	0645	34.4	46.3	4.2	8.4	4.5	11.3	4.5	16.1	3.6	1.2
7/24	1804	60.6	46.0	3.9	8.5	4.7	12.9	3.2	15.2	4.1	1.3
7/26	0604	90.2	46.5	3.9	8.3	4.7	12.7	3.3	15.2	3.9	1.4
7/26	1530	96.7	46.5	3.7	8.1	4.9	12.7	3.2	15.4	4.0	1.4
7/27	2344	98.4	46.2	3.7	8.4	4.7	13.0	3.3	15.2	3.9	1.5
AVE.			46.3	3.9	8.3	4.7	12.5	3.5	15.4	3.9	1.4
STD. DEV.			0.2	0.2	0.2	0.1	0.7	0.6	0.4	0.2	0.1

TABLE 27. Normalized Composition Of Canister 148 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	NaO_2	MnO_2	NiO
Defined				44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	0-7.6	0-3	46.4	4.1	8.5	4.9	12.2	3.3	14.6	3.8	1.3
		7.6-15.2	3-6	46.1	4.0	8.2	4.7	12.0	3.4	15.5	3.7	1.3
		15.2-22.9	6-9	46.4	4.0	8.4	5.1	12.2	3.2	14.6	3.8	1.3
		22.9-30.5	9-12	46.5	4.0	8.3	5.1	12.2	3.2	14.5	3.8	1.3
0.30	1	0-7.6	0-3	46.5	4.0	8.3	5.0	12.2	3.2	14.5	3.8	1.3
		7.6-15.2	3-6	46-2	4.0	8.3	4.9	11.9	3.4	15.4	3.7	1.2
		15.2-22.9	6-9	46.3	4.0	8.3	5.0	12.7	3.2	14.5	3.8	1.2
		22.9-30.5	9-12	46.4	4.0	8.4	5.1	12.2	3.2	14.6	3.8	1.3
0.61	2	0-7.6	0-3	46.2	3.9	8.4	5.0	12.3	3.3	14.8	3.8	1.3
		7.6-15.2	3-6	45.8	3.9	8.2	4.9	11.9	3.3	14.9	3.7	1.3
		15.2-22.9	6-9	46.1	3.9	8.3	5.0	12.2	3.2	15.1	3.8	1.3
		22.9-30.5	9-12	45.9	4.0	8.3	5.0	12.4	3.3	15.0	3.8	1.3
0.91	3	0-7.6	0-3	46.3	4.0	8.4	5.0	12.4	3.1	14.7	3.8	1.3
		7.6-15.2	3-6	46.0	4.0	8.2	4.9	12.0	3.3	15.6	3.7	1.3
		15.2-22.9	6-9	45.7	4.0	8.1	4.9	12.3	3.4	15.6	3.7	1.3
		22.9-30.5	9-12	46.1	4.0	8.2	5.1	12.7	3.1	14.6	3.9	1.3
1.21	4	0-7.6	0-3	46.2	3.9	8.2	5.1	12.5	3.2	14.7	3.8	1.3
		7.6-15.2	3-6	46.1	3.9	8.3	5.0	12.4	3.3	14.8	3.8	1.3
		15.2-22.9	6-9	45.7	3.9	8.3	4.9	12.1	3.3	15.7	3.8	1.3
		22.9-30.5	9-12	45.7	3.9	8.4	5.0	12.2	3.2	15.4	3.8	1.2
1.52	5	0-7.6	0-3	45.3	4.0	8.2	4.8	12.2	3.2	16.2	3.8	1.3
		7.6-15.2	3-6	46.0	3.8	8.4	4.9	12.0	3.3	15.6	3.7	1.2
		15.2-22.9	6-9	45.5	4.0	8.3	4.8	11.9	3.3	16.3	3.7	1.2
		22.9-30.5	9-12	46.4	3.9	8.3	5.0	12.1	3.3	15.1	3.8	1.1
1.83	6	0-7.6	0-3	46.2	3.8	8.3	5.1	12.5	3.2	15.0	3.7	1.2
		7.6-15.2	3-6	46.2	3.9	8.3	4.9	11.7	3.5	15.7	3.7	1.1

TABLE 28. Average Composition of Canister 148 Samples

Core Height		Oxide wt%								
m	ft	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
0	0	46.4	4.0	8.4	5.0	12.2	3.3	14.8	3.8	1.3
0.30	1	46.4	4.0	8.3	5.0	12.2	3.2	14.8	3.8	1.2
0.61	2	46.0	3.9	8.3	5.0	12.2	3.3	15.0	3.8	1.3
0.91	3	46.0	4.0	8.2	5.0	12.4	3.2	15.1	3.8	1.3
1.21	4	45.9	3.9	8.3	5.0	12.3	3.2	15.2	3.8	1.3
1.52	5	45.8	3.9	8.3	4.9	12.0	3.3	15.8	3.8	1.2
1.83	6	46.2	3.8	8.3	5.0	12.1	3.4	15.3	3.7	1.2
Core Depth										
cm	in.									
0-7.6	0-3	46.2	4.0	8.3	5.0	12.3	3.2	14.9	3.8	1.3
7.6-15.2	3-6	46.0	3.9	8.3	4.9	12.0	3.4	15.3	3.7	1.3
15.2-22.9	6-9	46.0	4.0	8.3	5.0	12.2	3.4	15.4	3.8	1.2
22.9-30.5	9-12	46.2	4.0	8.3	5.0	12.3	3.2	14.9	3.8	1.2
AVE.		46.1	4.0	8.3	5.0	12.2	3.3	15.1	3.8	1.3
STD. DEV.		0.3	0.1	0.1	0.1	0.2	0.1	<0.5	<0.1	0.1

TABLE 29. Weight Loss of Canister 148 Sample Under pH4 Conditions

		wt% Loss				
		Core Depth, cm (in.)				
Core Height		0-7.6	7.6-15.2	15.2-22.9	22.9-30.5	
m	ft	(0-3)	(3-6)	(6-9)	(9-12)	Ave.
0	0	67.2	72.2	82.7	82.5	76.2
0.30	1	64.2	74.6	85.6	73.1	74.3
0.61	2	71.2	74.8	70.9	62.7	69.9
0.91	3	71.6	77.6	70.2	75.3	73.7
1.21	4	75.6	78.6	74.5	77.7	76.6
1.52	5	67.1	75.6	80.9	85.7	77.3
1.83	6	78.1	81.6	75.1	---	78.3
AVE.		70.7	76.5	77.1	76.2	

Mean pH4 Weight Loss 75.1 ± 6.0 wt%.

TABLE 30. Weight Loss of Canister 148 Sample Under Soxhlet Conditions

		wt% Loss				
		Core Depth, cm (in.)				
Core Height m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	1.82	1.53	1.95	1.89	1.80
0.30	1	1.81	1.56	1.68	1.01	1.77
0.61	2	1.77	1.70	1.70	1.71	1.72
0.91	3	1.83	1.71	1.66	1.75	1.74
1.21	4	2.12	1.76	1.91	1.84	1.90
1.52	5	1.78	1.67	1.76	1.85	1.77
1.83	6	1.75	1.86	1.74	---	1.78
AVE.		1.85	1.68	1.77	1.85	

Mean Soxhlet Weight Loss 1.78 ± 0.12 wt%

of the resulting product. Approximately 2900 L of waste slurry were converted to ≃1050 kg of waste glass at a maximum sustained glass production rate of 40 kg/h (38 kg/h/m^2).

The waste slurry was a mixture of TDS simulated waste and -200 mesh 211 frit in a 28:72 oxide ratio. Bentonite and cornstarch were added to keep solids in suspension and reduce foaming, respectively. The feed was also spiked with volatile components Ru, Sb, Te and Cs to examine their behavior under processing conditions. The composition of the liquid feed is listed in Table 31. Problems with foaming initially lowered production rates; however, plugging and erosion of the feed system was a more persistent problem throughout the run causing several equipment stoppages. Glass melting rates as a result varied considerably.

Samples of liquid feed were regularly collected and analyzed. The liquid feed oxide composition and oxide loading are listed in Table 32. The average feed composition approximated the target values moderately well. No cause was identified, however, for discrepancies in B_2O_3 and Li_2O content. Since both are frit constituents, they are usually both above or below target. The large variation in MnO_2 content again reflects normal sampling error for this

TABLE 31. SRL-LF-1 Liquid Feed Makeup

Compound			Concentration (g/L)
Waste			
$Fe(OH)_3$			70.0
$Al(HO)_3$			17.5
MnO_2			14.0
$Ni(OH)_2$			7.5
$CaCO_3$			6.4
$NaNO_3$			2.9
Na_2SO_4			1.4
Zeolite(a)			10.4
$RuNO(NO_3)_3$			0.204
Sb_2O_3			0.079
TeO_2			0.024
Cs_2CO_3			0.237
$Sr(NO_3)_2$			0.166
Additives, wt%			
SiO_2	58.3	Premelted Frit-211 (-200 mesh)	258
Na_2O	20.6		
B_2O_3	11.1		
CaO	5.6		
Li_2O	4.4		
Cornstarch $(C_6H_{10}O_5)n$			3.6
Bentonite(b)			7.8
TOTAL			400

(a) Linde Ion SiV IE-95, 2

(b) Alumina silicate clay assumed to be $Al_2O_3 \cdot 4SiO_2 \cdot H_2O$

TABLE 32. Composition of SRL-LF-1 Liquid Feed Samples

Date	Time	Oxide wt%															Total Oxides g/L Feed
		SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO	RuO_2	Sb_2O_3	TeO_2	Cs_2O	SrO	$Cl^{-(a)}$	
Defined		44.5	4.2	7.8	5.0	14.4	3.1	14.9	3.8	1.6	0.023	0.022	0.006	0.056	0.022	0.29	400.0
1/14	2140	43.6	4.4	8.2	5.1	13.0	2.5	14.7	4.4	1.5	0.301	0.084	0.073	0.054	0.038	1.39	336.8
	2330	42.9	3.8	9.9	4.7	12.0	2.7	16.8	3.3	1.4	0.290	0.078	0.092	0.050	0.040	1.32	328.3
1/15	0145	43.4	4.0	9.5	4.8	11.7	2.6	15.8	3.5	1.4	0.328	0.080	0.082	0.055	0.039	1.36	335.6
	0330	43.3	3.8	10.2	4.8	12.3	2.6	16.3	2.7	1.4	0.309	0.080	0.090	0.051	0.042	1.33	344.8
	0630	44.1	4.3	8.4	5.3	13.6	2.5	14.6	3.2	1.6	0.301	0.085	0.073	0.054	0.038	1.30	341.8
	0908	44.0	4.2	8.4	5.3	13.6	2.6	14.8	3.2	1.6	0.323	0.083	0.081	0.054	0.037	1.30	341.4
	1105	43.8	4.2	8.5	5.2	13.5	2.5	14.5	3.8	1.6	0.299	0.083	0.078	0.055	0.039	1.31	350.6
	1250	44.3	4.4	8.4	5.2	13.4	2.5	14.3	3.5	1.6	0.326	0.087	0.071	0.060	0.040	1.35	351.0
1/16	0030	44.1	4.2	8.5	5.3	13.7	2.4	13.9	3.9	1.5	0.327	0.088	0.081	0.056	0.038	1.38	304.7
	0230	43.9	4.3	8.3	5.2	13.4	2.6	14.0	4.3	1.6	0.299	0.083	0.067	0.055	0.038	1.34	447.4
	0430	44.0	4.4	8.2	5.3	13.6	2.6	14.4	3.6	1.6	0.320	0.086	0.066	0.056	0.037	1.38	340.9
	1710	44.2	4.2	8.4	5.3	13.7	2.7	13.9	2.9	1.5	0.330	0.085	0.077	0.054	0.036	1.36	334.6
	2222	44.2	4.1	8.4	4.6	13.8	2.6	14.7	2.7	1.6	0.322	0.090	0.078	0.050	0.038	1.37	332.9
1/7	0020	44.1	4.3	8.3	5.3	13.6	2.5	14.5	3.3	1.6	0.302	0.082	0.071	0.054	0.038	1.38	320.0
	0125	44.6	4.2	8.6	5.3	13.8	2.6	14.7	2.2	1.5	0.338	0.087	0.073	0.051	0.041	1.33	321.5
	0230	44.8	4.2	8.4	5.4	14.0	2.5	14.1	2.7	1.6	0.338	0.090	0.073	0.052	0.040	1.34	316.2
	0335	45.0	4.2	8.6	5.5	14.3	2.6	14.4	1.2	1.6	0.340	0.088	0.074	0.046	0.040	1.36	317.0
	0425	44.6	4.2	8.5	5.4	14.0	2.6	15.1	1.5	1.6	0.328	0.083	0.071	0.046	0.037	1.34	331.7
	0525	44.5	4.2	8.6	5.2	13.7	2.9	16.0	1.2	1.5	0.326	0.086	0.076	0.047	0.038	1.29	327.5
	0630	44.2	4.1	8.4	4.9	13.5	2.9	16.1	1.9	1.5	0.275	0.069	0.060	0.042	0.034	1.21	310.1
	0755	43.9	4.2	8.2	5.0	13.5	2.8	15.4	2.9	1.5	0.248	0.070	0.058	0.048	0.035	1.26	324.2
	1015	43.8	4.1	8.2	4.8	13.6	2.8	15.6	3.0	1.5	0.311	0.080	0.070	0.048	0.036	1.29	322.0
	1245	43.6	4.2	8.2	5.3	14.0	2.6	15.0	3.1	1.5	0.330	0.083	0.080	0.050	0.035	1.30	328.1
	1400	44.1	4.0	8.4	5.0	13.9	2.7	15.0	2.9	1.6	0.284	0.075	0.066	0.049	0.035	1.36	315.8
	1500	44.6	4.0	8.4	5.2	14.4	2.6	15.2	1.5	1.5	0.290	0.079	0.072	0.048	0.036	1.36	144.0
AVE.		44.1	4.2	8.6	5.1	13.5	2.6	15.0	2.9	1.5	0.311	0.083	0.074	0.051	0.038	1.34	326.8
STD. OEV.		0.5	0.2	0.5	0.2	0.6	0.1	0.8	0.9	0.1	0.022	0.055	0.008	0.004	0.002	0.04	46.4

(a) Cl^- not separately added to waste.

constituent. The trace constituents were present at greater levels than anticipated. The Cl^- was introduced as a contaminant in the ferric hydroxide at approximately 4.5 times its anticipated level in the actual waste stream.

Grab samples of the glass produced were collected and analyzed during the trial. The compositions of the samples are shown in Table 33. The first sample on 11/15/80 at 0917 represents the bulk glass in the LFCM at the start of the run. As noted in Figure 12, the production of 1000 kg of glass results in a product containing approximately 50% of the original composition in the melter and 50% of the new composition. This accounts for some of the discrepancies noted between targets and analyzed compositions. The trace constituent analyses are not consistent with intended targets due to larger feed concentrations. The analysis of melter samples before the melting trial showed no significant trace oxide concentrations. Approximately 0.45% Cl^- was analyzed in the glass at the end of the run. This represents about one third of the Cl^- present in the liquid waste feed.

Canister 10 filled near the end of the run when compositional variation was diminished was core-drilled for sample analysis. Table 34 presents the composition of the major constituents in Canister 10 samples. The analysis of canister and grab samples were both close to the defined composition with small standard deviations. The chemical durability of canister samples did not vary with sample location (Tables 35 and 36). The average weight loss of core samples under pH4 and soxhlet conditions were 73.8 ± 2.3 wt% and 2.05 ± 0.21 wt.%, respectively.

TABLE 33. Normalized Composition of SRL-LF-1 Grab Samples

		Oxide wt%														
Date	Time	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO	RuO_2	Sb_2O_3	TeO_2	Cs_2O	SrO	Cl^-
Defined		44.5	4.2	7.8	5.0	14.4	3.1	14.9	3.8	1.6	0.023	0.022	0.006	0.056	0.022	0.00(a)
1/15	0017	45.8	4.1	8.2	5.0	12.4	3.0	15.8	3.6	1.2	0.039	0.074	0.025	0.007	0.011	0.18
	1130	45.6	4.0	8.2	5.0	12.4	3.1	16.0	3.6	1.2	0.067	0.079	0.020	0.007	0.010	0.20
	1247	45.2	4.1	8.1	4.9	12.2	3.1	16.8	3.5	1.2	0.062	0.076	0.025	0.008	0.010	0.19
	1800	46.0	4.2	8.5	5.1	12.6	2.9	15.3	3.7	1.2	0.062	0.079	0.028	0.009	0.013	0.21
1/16	0435							CANISTER 144 OUT								
1/17	0245	45.2	4.3	8.2	5.1	13.0	2.9	15.4	3.6	1.3	0.106	0.082	0.040	0.020	0.021	0.39
	0535	45.1	4.3	8.3	5.1	13.0	2.9	15.6	3.5	1.3	0.111	0.091	0.042	0.022	0.021	0.41
	0905	45.0	4.3	8.3	5.1	13.0	2.9	15.7	3.4	1.4	0.137	0.092	0.051	0.024	0.020	0.45
	1700	44.6	4.3	8.2	5.0	12.9	3.0	16.3	3.3	1.4	0.131	0.090	0.053	0.027	0.024	0.47
								CANISTER 10 OUT								
RUN AVE.		45.3	4.2	8.2	5.0	12.7	3.0	15.9	3.5	1.3	0.089	0.083	0.036	0.013	0.016	0.31
STD. DEV.		0.4	0.1	0.1	0.1	0.3	0.1	0.5	0.2	0.1	0.036	0.007	0.013	0.009	0.006	0.13
CANISTER 10 AVE.		45.0	4.0	8.2	5.1	13.0	2.9	15.8	3.4	1.4	0.121	0.089	0.046	0.023	0.022	0.43
STD. DEV.		0.3	0	0.1	0.1	0.1	0.1	0.4	0.1	0.1	0.015	0.004	0.006	0.003	0.001	0.04

(a) No Cl^- expected to enter glass.

TABLE 34. Normalized Composition of Canister 10 Samples

Core Height		Core Depth		Oxide wt%										
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO	SrO	Cl^-
Defined				44.5	4.2	7.8	5.0	14.4	3.1	15.0	3.8	1.6	0.02	0.29(a)
0.30	1	0-7.6	0-3	45.4	4.2	7.9	5.1	13.1	2.9	15.7	3.7	1.4	0.01	0.34
		7.6-15.2	3-6	46.3	4.4	8.0	5.1	13.3	3.1	14.0	3.7	1.4	0.01	0.35
		15.2-22.9	6-9	45.4	4.3	7.8	5.0	12.9	3.2	16.1	3.6	1.3	0.01	0.30
		22.9-30.5	9-12	45.8	4.3	7.9	5.0	13.1	3.1	15.1	3.6	1.3	0.01	0.36
0.61	2	0-7.6	0-3	45.7	4.3	7.9	5.0	13.0	3.3	15.2	3.5	1.4	0.02	0.39
		7.6-15.2	3-6	45.6	4.3	7.9	5.0	13.1	3.2	15.1	3.5	1.4	0.02	0.41
		15.2-22.9	6-9	45.7	4.3	7.9	5.0	13.2	3.1	15.1	3.5	1.4	0.02	0.36
		22.9-30.5	9-12	45.5	4.4	7.9	5.0	13.1	3.1	15.2	3.4	1.4	0.02	0.38
0.91	3	0-7.6	0-3	45.6	4.3	7.9	5.1	13.5	3.0	14.7	3.4	1.5	0.02	0.48
		7.6-15.2	3-6	45.6	4.3	8.0	5.1	13.4	3.1	14.8	3.4	1.4	0.02	0.47
		15.2-22.9	6-9	45.3	4.3	7.9	5.0	13.3	3.1	15.4	3.3	1.4	0.02	0.46
		22.9-30.5	9-12	45.5	4.4	8.0	5.1	13.3	3.0	15.0	3.4	1.4	0.02	0.42
AVE.			45.6	4.3	7.9	5.0	13.2	3.1	15.1	3.5	1.4	0.02	0.39	
STD. DEV.			0.3	0.1	0.1	0.1	0.2	0.1	0.5	0.1	0.1	0.01	0.06	

TABLE 35. Weight Loss of Canister 10 Samples Under pH4 Conditions

Core Height		wt% Loss Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0.30	1	74.8	72.8	75.0	69.5	74.2
0.61	2	73.7	71.4	74.0	75.7	73.7
0.91	3	74.9	78.6	72.7	72.7	74.7
AVE.		74.5	74.3	73.9	72.6	

Mean Weight Loss 73.8 ± 2.3 wt%

TABLE 36. Weight Loss of Canister 10 Samples Under Soxhlet Conditions

Core Height		wt% Loss Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0.30	1	2.16	1.91	2.19	1.76	2.00
0.61	2	2.14	2.37	1.70	1.94	2.04
0.91	3	2.29	1.97	2.21	1.94	2.10
AVE.		2.19	2.08	2.03	1.88	

Mean Weight Loss 2.05 ± 0.21 wt%

SPRAY CALCINER/CALCINE-FED CERAMIC MELTER

The Spray Calciner/Calcine-Fed Ceramic Melter (SC/CFCM) system was designed and constructed to demonstrate the operational compatibility of these two solidification process components. The calcination of the liquid waste stream and charging of the calcine and frit into the melter proceeds exactly as described for the SC/ICM process. The melter in this case is a refractory-lined joule-heated electric melter.

While some details of design and construction may be unique to the CFCM, the general operating principles are the same as for the LFCM. Alternating current passing through molten glass between two large plate-electrodes maintains the glass temperature at 1100^o to 1200^oC by joule heating. As the calcine/frit mixture is charged on top of this molten pool, the constituents react, forming a fluid glass. As the glass level in the melter rises, the hydrostatic pressure forces a stream of waste glass out the discharge trough into a receiving canister. Glass flow can be adjusted by tilting the melter assembly. Dierks (1980) provides details of CFCM design and operation.

Three SC/CFCM melting trials involving 10, 25, and 5 continuous days of operation were run processing simulated TDS-211 defense waste glass. The primary objectives were to establish maximum melting rates and gain equipment operating experience. Routine sampling of the liquid waste, frit, and glass product monitored process operation and product consistency. Sample data for each trial will be presented separately.

CFCM-6 (APRIL 1979)

The CFCM had been drained of glass for inspection and modifications prior to the CFCM-6 melting trial. The primary objectives of this trial were to determine the maximum melting rate of the SC/CFCM system and gain additional equipment operating experience with the TDS-211 defense-waste glass. During 10 continuous days of SC/CFCM operation approximately 11,000 kg of simulated defense-waste glass were produced, filling seven canisters. Foaming of the waste/frit batch was a significant problem limiting maximum production rates. Samples of liquid waste and glass were regularly collected. Physical properties and chemical composition were monitored.

The simulated liquid waste was prepared in 2529-L batches to maintain a continuous feed supply to the process. Samples were taken directly from the process feed line immediately upstream of the spray calciner. Operating personnel measured specific gravity and pH before submitting samples for chemical analysis. Feed data collected during the melting trial is presented in Table 37. Nickel oxide concentrations were not reported since nickel crucibles were used in the analytical procedure.

During the run the liquid feed concentration was increased twice. From the starting level of approximately 46 g oxide/L solution, the concentration was raised on April 23 to 55 g/L and on April 25 to 64 g/L to increase the calcine production rate. Figure 13 shows oxide content of the liquid feed during the run. All waste oxide components were consistently below target values during the run due possibly to excessive feed dilution. Since liquid feed rate is a major operating parameter, a lower oxide content translates into a calcine-deficient waste glass. The last concentration increase on April 25, 1979 resulted in significantly greater feed variations. During this same period from April 26, 1979, 1200 h, Na_2O content briefly increased from <0.2 g/L to over 2.5 g/L. The cause of these disturbances were not identified. The mean specific gravity and pH of over 100 samples were 1.025 ± 0.008 g/cc, and 7.7 ± 0.1, respectively. Neither property was correlated to either total oxide loading or chemical composition of the feed.

Waste glass grab samples were collected approximately every 250 kg of glass production. The annealed density, chemical composition and crystallinity were determined for each. Annealed glass density is plotted in Figure 14. After April 22, 1979 Frit 211 replaced Frit 411 as the supplier of glass-forming constituents. This is responsible for the sharp increase in density on April 23, 1979. Most major changes in density can be largely accounted for by

TABLE 37. Composition of CFCM-6 Liquid Feed Samples, g Oxide/L Feed

				g Oxide/L Feed							
Date	Time	pH	Specific Gravity, g/cc	Total Oxides	SiO_2	Fe_2O_3	Al_2O_3	CaO	Na_2O	MnO_2	NiO[(a)]
PART I											
Defined	---	---	---	46.0	3.3	24.2	6.1	1.8	0.4	6.4	2.8
4/18	0000	7.8	1.03	36.3	2.3	16.0	6.2	1.8	0.5	6.8	
	0400	8.0	1.02	37.2	2.5	15.8	6.1	1.8	1.0	6.8	
	0800	7.9	1.02	41.0	2.9	18.3	6.5	1.8	0.7	7.3	
	1200	7.9	1.02	39.0	1.4	18.2	6.3	1.6	0.1	7.0	
	1800	7.8	1.03	43.4	4.4	18.3	7.1	1.8	0.3	7.1	
	2000	7.8	1.03	45.0	5.0	18.1	7.3	1.9	0.3	7.1	
	2400	7.9	1.02	39.1	2.7	17.6	6.0	1.7	0.2	6.4	
4/19	0400	7.9	1.02	38.9	2.6	17.5	6.3	1.8	0.2	6.6	
	1600	7.9	1.03	39.5	2.3	17.9	5.9	1.5	0.1	6.6	
	2000	7.8	1.02	38.0	3.2	17.9	5.1	1.6	0.2	5.7	
4/20	0000	7.9	1.02	35.1	1.2	17.5	4.7	1.4	0.1	5.9	
	0420	7.9	1.02	38.6	1.4	18.4	5.3	1.2	0.1	6.2	
	0800	7.8	1.02	40.2	2.5	18.5	5.6	1.4	0.1	6.3	
	1200	7.7	1.02	34.2	2.2	15.9	5.1	1.2	0.1	5.2	
	1600	7.7	1.02	35.4	3.2	15.6	5.2	1.4	0.2	5.2	
	2000	7.6	1.02	35.6	3.9	15.8	5.1	1.3	0.2	4.7	
4/21	0000	7.6	1.02	45.6	4.6	21.9	6.2	1.7	0.3	5.6	
	0400	7.6	1.02	44.7	3.7	22.2	5.7	1.6	0.2	5.8	
	0750	7.6	1.02	42.4	3.1	20.6	5.8	1.7	0.2	6.2	
	1200	7.5	1.03	39.6	1.8	18.1	5.5	1.3	0.1	5.6	
	1600	7.7	1.04	41.8	2.3	19.8	5.6	1.4	0.2	5.4	
	2000	7.6	1.03	40.1	3.1	19.2	5.1	1.5	0.2	5.2	
4/22	0025	7.7	1.02	42.3	2.0	20.8	5.6	1.5	0.1	6.0	
	0405	7.6	1.02	43.5	2.1	21.6	5.8	1.6	0.1	6.1	
	0800	7.7	1.02	33.0	0.6	15.0	4.9	1.4	0.2	4.9	
	1200	7.6	1.03	44.0	2.9	19.0	5.9	1.4	0.2	5.6	
	1600	7.7	1.02	35.0	0.7	17.5	4.3	1.3	---	4.9	
	2000	7.6	1.02	42.1	2.5	19.4	5.7	1.4	0.1	6.0	
4/23	0015	7.8	1.02	37.1	1.1	18.6	4.8	1.5	0.1	4.7	
AVE.		7.7	1.02	39.6	2.6	18.3	5.7	1.5	0.2	6.0	
STD. DEV.		0.1	0.01	3.5	1.1	1.9	0.7	0.2	0.2	0.8	

(a) Not analyzed

TABLE 37. (contd)

				g Oxide/L Feed							
Date	Time	pH	Specific Gravity, g/cc	Total Oxides	SiO_2	Fe_2O_3	Al_2O_3	CaO	Na_2O	MnO_2	NiO[(a)]
PART II											
Defined	---	---	---	55.0	4.0	28.9	7.3	2.2	0.6	7.6	3.4
	0440	7.7	1.03	54.3	3.8	25.0	7.6	1.8	0.2	7.5	
	0800	7.6	1.03	54.3	2.9	25.0	7.1	1.7	0.2	7.6	
4/24	0000	7.6	1.03	50.6	3.6	23.3	7.3	2.0	0.2	7.7	
	0400	7.8	1.03	49.8	2.9	23.0	7.5	2.0	0.2	8.1	
	0800	7.8	1.03	55.6	4.9	25.3	7.8	2.0	0.3	8.1	
	1200	7.8	1.03	48.8	3.0	23.0	7.6	1.7	0.2	7.2	
	1600	7.7	1.04	53.5	4.5	23.1	8.3	1.6	0.3	7.1	
	2000	7.7	1.03	51.2	3.2	22.6	8.3	1.6	0.4	7.2	
	2345	7.6	1.04	55.4	4.2	24.0	9.3	1.9	0.3	7.6	
4/25	0400	7.7	1.02	46.5	2.9	18.0	7.4	1.5	0.4	6.6	
AVE.		7.7	1.03	52.0	3.6	23.3	7.8	1.8	0.3	7.5	
STD. DEV.		0.1	0.01	3.1	0.7	22	0.6	0.2	1	0.5	
PART III											
Defined	---	---	---	64.0	4.6	33.7	8.4	2.6	0.7	8.9	3.9
	0800	7.8	1.03	59.1	3.5	25.5	8.8	1.8	0.2	7.6	
	1200	7.8	1.04	60.3	4.0	26.6	8.8	2.0	0.2	7.8	
	1600	7.7	1.03	69.7	6.7	32.2	8.9	2.3	0.4	7.9	
	2000	7.5	1.01	46.2	2.7	31.5	5.8	1.5	0.2	5.6	
4/26	0000	7.7	1.03	47.8	1.3	23.5	6.1	1.9	0.1	6.6	
	0400	7.8	1.01	46.8	1.8	21.4	6.5	1.7	0.1	6.4	
	0800	7.8	1.03	51.6	3.0	24.6	8.2	2.0	0.2	7.7	
	1200	7.8	1.01	60.4	2.8	27.8	9.7	1.9	2.2	6.8	
	1600	7.6	1.02	62.4	4.2	30.9	9.6	2.1	2.7	7.2	
	2000	7.6	1.02	48.0	2.9	24.6	6.9	1.9	2.5	5.2	
4/27	0000	7.6	1.02	62.6	4.9	30.3	8.6	2.5	2.9	6.3	
	0400	7.6	1.02	61.3	3.2	29.3	8.4	2.3	2.7	6.8	
	0845	7.6	1.02	64.4	3.8	28.6	9.1	1.7	1.8	7.3	
	1200	7.5	1.00	59.2	3.2	30.5	9.8	2.0	0.2	7.0	
	1600	7.5	1.03	60.4	3.4	29.3	9.6	2.0	0.2	6.7	
	2000	7.5	1.02	59.1	2.5	29.6	9.3	1.8	0.1	6.9	
AVE.		7.6	1.02	57.4	3.4	27.3	8.4	2.0	1.0	6.9	
STD. DEV.		0.1	0.01	7.1	1.2	3.4	1.3	0.2	1.2	0.8	

(a) Not analyzed

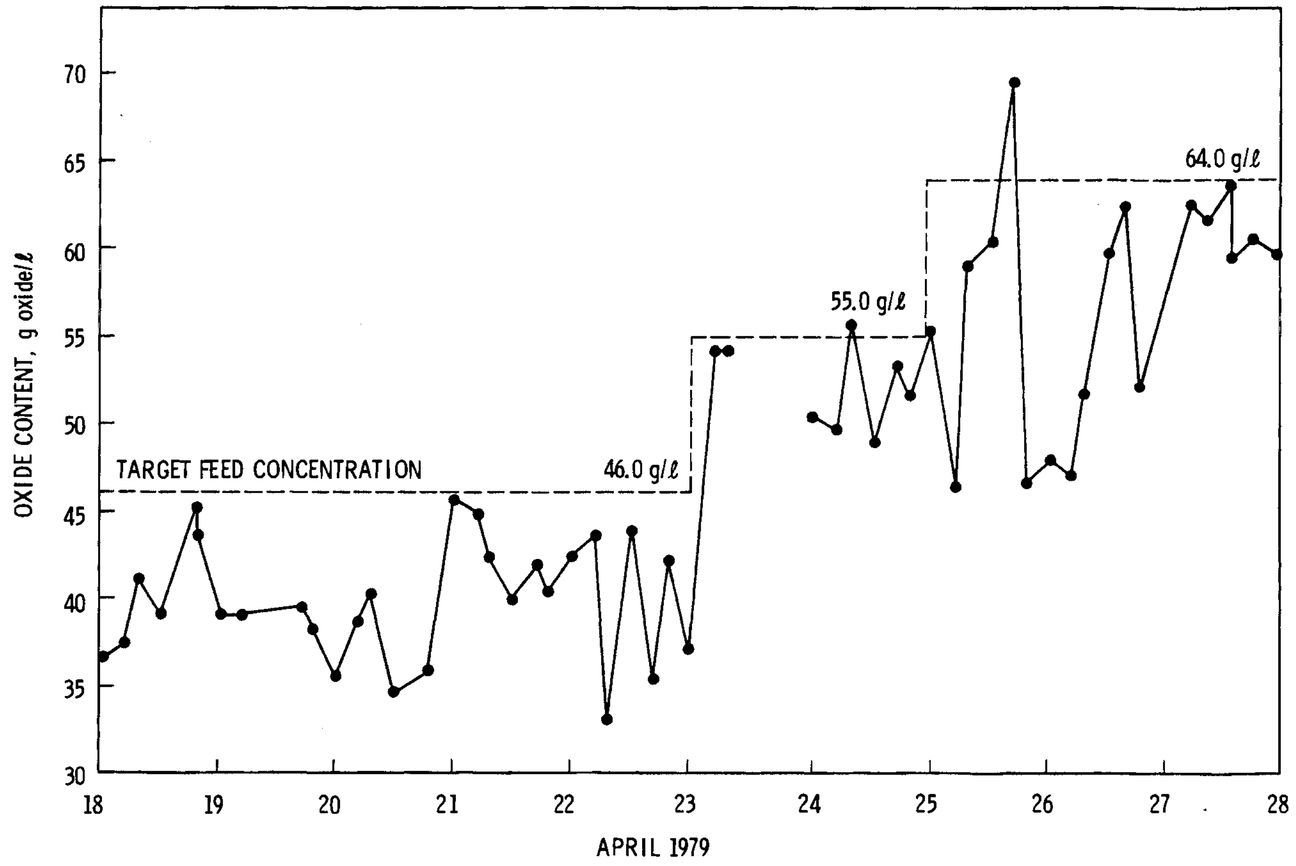

FIGURE 13. Total Oxide Content of CFCM-6 Liquid Feed

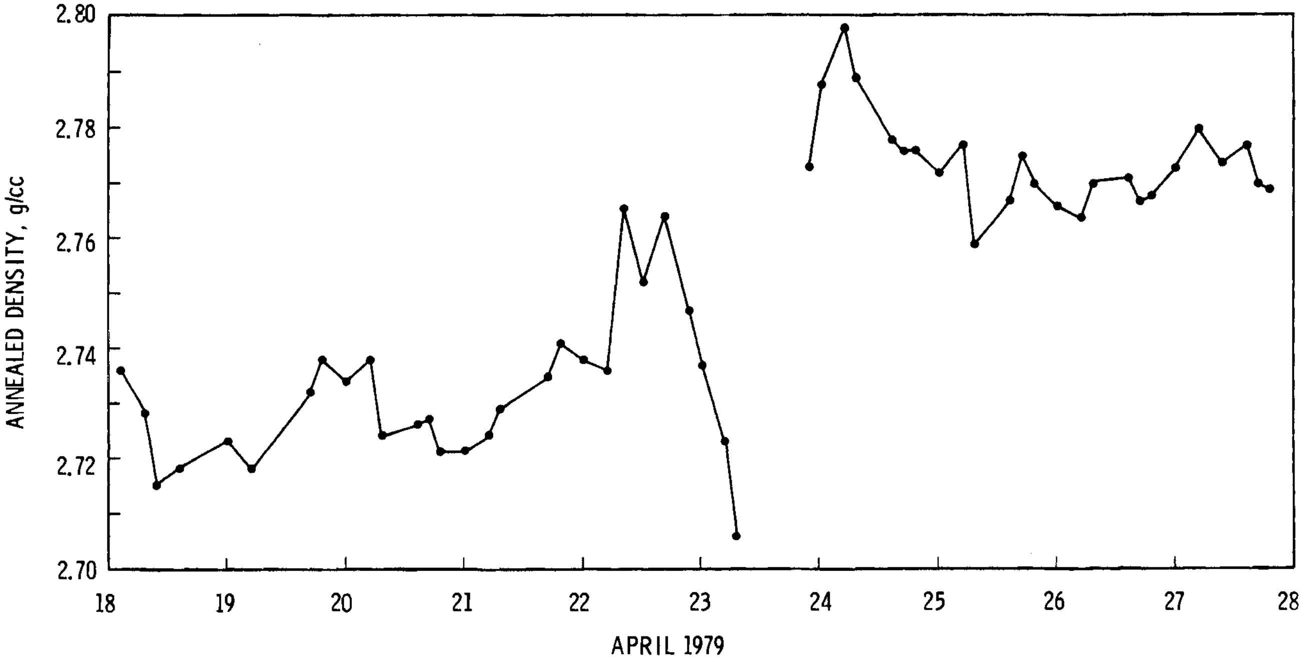

FIGURE 14. Annealed Density of CFCM-6 Grab Samples

variations in waste loading. The normalized composition and annealed glass density for the grab samples are listed in Table 38. As density increases, the concentration of waste constituents, i.e., Al_2O_3, Fe_2O_3 and MnO_2, generally increase. This relationship does not hold up as well when waste constituents

TABLE 38. Normalized Composition of CFCM-6 Grab Samples

Date	Time	Glass Produced, kg	Oxide wt%								Density g/cc
			SO_2	Al_2O	B_2O_2	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	
4/18	0320		46.8	3.0	8.5	4.6	11.5	9.4	10.6	3.6	2.736
	0620		47.6	2.7	8.6	4.5	10.8	9.7	10.5	3.6	2.728
	1000		47.9	2.7	8.6	4.7	10.2	9.6	10.7	3.6	2.715
	1400		48.4	3.2	8.5	4.6	10.2	9.6	9.6	3.7	2.718
	2415		47.9	3.4	8.4	4.6	10.2	9.9	9.6	3.8	2.723
4/19	0415		48.1	3.4	8.4	4.5	10.5	9.5	9.7	3.8	2.718
	1600		47.5	3.5	8.3	4.6	10.6	9.4	10.2	3.9	2.732
	2020		46.5	3.8	8.7	4.5	11.0	9.8	9.6	4.1	2.738
	2350		46.1	3.8	8.5	4.7	11.5	9.6	9.7	4.2	2.734
4/20	0405		46.4	3.7	8.2	4.4	12.5	9.1	9.3	4.1	2.738
	0800		47.1	3.6	8.4	4.5	11.4	9.5	9.3	4.1	2.724
	1130	1616					CANISTER 80 OUT				
	1400		48.2	3.4	8.6	4.5	10.6	9.1	9.5	3.9	2.726
	1640		47.7	3.4	8.3	4.6	11.3	9.3	9.6	3.8	2.727
	2030		47.6	3.3	8.6	4.7	11.0	9.3	9.8	3.7	2.721
4/21	0020		47.7	3.2	8.4	4.3	11.7	9.5	9.6	3.4	2.721
	0410		47.6	3.2	8.7	4.7	11.6	9.2	9.7	3.6	2.724
	0800		47.1	3.3	8.6	4.7	11.4	9.2	9.9	3.7	2.729
	1623		47.5	3.3	8.3	4.6	11.9	9.2	9.6	3.7	2.735
	2005		46.7	3.3	8.1	4.6	12.8	9.2	9.5	3.7	2.741
	2300	3184					CANISTER 79 OUT				
4/22	0000		47.4	3.2	8.4	4.5	11.9	9.0	9.7	3.7	2.738
	0400		46.8	3.2	8.4	4.6	12.6	9.2	9.5	3.6	2.736
	0800		45.2	3.7	8.5	4.5	13.8	8.8	9.4	4.2	2.767
	1200		46.5	3.4	8.2	4.5	13.0	9.1	9.5	3.7	2.752
	1620		46.5	3.4	8.2	4.5	13.1	7.6	10.9	3.8	2.764
	2100		46.5	3.5	8.1	4.4	13.0	7.6	11.0	3.9	2.747
4/23	0015		49.2	3.0	8.7	4.3	10.8	5.4	13.3	3.3	2.737
	0415		51.0	2.8	9.0	4.6	10.4	5.2	14.6	3.1	2.723
	0800		51.7	2.6	9.2	4.5	10.0	4.7	14.8	2.8	2.706
	1105	4754					CANISTER 94 OUT				
	2200		46.7	3.5	8.3	4.6	13.2	4.0	13.7	2.8	2.773

(a) NiO not analyzed
(b) Not tabulated

TABLE 38. (contd)

Date	Time	Glass Produced, kg	Oxide wt%								Density
			SO_2	Al_2O	B_2O_2	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	g/cc
4/24	0015		45.8	3.7	8.0	4.6	14.0	3.9	13.6	4.2	2.788
	0415		45.9	4.0	8.1	4.5	13.8	3.6	13.5	4.5	2.798
	0810		45.6	3.7	8.0	4.7	14.9	3.4	13.4	4.3	2.789
	1400		46.4	3.5	8.0	4.6	13.6	3.4	14.5	4.0	2.778
	1620		46.8	3.2	8.1	4.5	12.4	3.3	15.6	4.0	2.776
	2025		47.4	3.6	8.4	4.2	12.4	3.3	14.6	4.1	2.776
	2345		47.2	3.7	8.1	4.5	12.6	3.4	14.6	3.9	2.772
4/25	0013	6516					CANISTER 81 OUT				
	0411		47.2	3.5	8.0	4.7	12.5	3.3	15.0	3.8	2.777
	0807		47.8	3.4	8.1	4.6	11.7	3.4	15.4	3.5	2.759
	1400		47.6	3.7	8.3	4.2	12.6	3.4	14.5	3.6	2.767
	1600		46.9	3.8	8.2	4.4	14.0	3.3	13.9	3.5	2.775
	2015		47.8	3.6	8.4	4.1	12.9	3.3	14.1	3.6	2.770
4/26	0000		48.3	3.5	8.7	4.1	12.0	3.4	14.5	3.6	2.766
	0150	7970					CANISTER 116 OUT				
	0410		46.7	3.2	8.0	4.6	14.6	3.3	14.2	3.3	2.764
	0800		47.0	3.6	8.2	4.4	12.9	3.4	14.8	3.5	2.770
	1440		40.2	4.3	7.7	5.0	16.6	3.3	17.0	3.7	2.771
	1600		41.5	4.4	8.0	4.7	14.2	3.4	18.0	3.7	2.767
	2000		41.6	4.5	8.0	4.6	14.1	3.4	18.0	3.7	2.768
4/27	0010		40.8	4.2	8.0	4.5	13.5	3.1	20.2	3.6	2.773
	0300	9516					CANISTER 92 OUT				
	0420		40.0	4.1	7.7	5.0	14.7	3.0	20.1	3.4	2.780
	0845		40.1	4.3	7.9	4.9	16.0	3.4	18.0	3.4	2.774
	1425		39.9	5.0	7.6	4.6	15.9	4.5	17.0	3.6	2.777
	1610		38.8	4.6	7.4	4.8	14.9	4.0	20.1	3.5	2.770
	1950	10964	39.3	4.7	7.5	4.6	14.8	4.2	19.2	3.5	
							CANISTER 84 OUT				
RUN AVE.			46.1	3.6	8.3	4.6	12.6	(b)	(b)	3.7	2.751
STD. DEV.			2.9	0.5	0.4	0.2	1.6			0.3	0.024
CANISTER 92											
AVE.			43.0	4.0	8.0	4.6	14.3	3.3	17.0	3.6	2.769
STD. DEV.			3.0	0.5	0.2	0.2	1.3	0.2	2.2	0.2	0.003

vary independently as noted after April 25, 1979. All grab samples collected from the pouring spout were amorphous with no detectable amount of crystalline material.

Only one canister produced during this trial, 92, was core-drilled. Significant changes in glass composition were noted in grab samples taken while canister 92 was being filled (Table 38) with both the waste and Na_2O content increasing. The average composition was, however, close to the defined glass composition. The standard deviations ranged from 5 to 10% for each constituent. All canister centerline samples contained significant quantities of acmite crystals (7 to 25 wt%). Figure 15 shows the lathe-shaped acmite crystals in a micrograph of ground glass particles. Although no spinel was detected by x-ray diffraction (<1 wt%), small, bright spinel crystals can be seen in several of the acmite groups. The quantity of crystalline material increased going towards the bottom of the canister. The only wall sample to contain these phases were at the 0.91-m (3-ft) level (Table 39). This variation in crystal content with sample location was not clearly reflected by variations in chemical durability as seen in Tables 40 and 41. The percent weight loss under pH4 and soxhlet conditions were 80.2 ± 4.5 wt% and 2.90 ± 0.73 wt%, respectively.

CFCM-7 (JUNE 1979)

Melting trial CFCM-7 was basically a continuation of the previous CFCM-6 trial. During 25 continuous days of operation, 18 canisters 0.61 m (2 ft) and 0.91 m (3 ft) in dia were filled with approximately 35,000 kg of simulated defense waste glass. The maximum sustained production rate was 85 kg/h ($112\ kg/h/m^2$). A more typical rate was 70 kg/h ($92\ kg/h/m^2$). The liquid feed was concentrated to 130% of the defined flowsheet concentration to increase calciner capacity, which was reduced by the failure of some calciner heating elements. Liquid feed and glass samples were collected approximately every 250 kg of glass production using the same technique as described for CFCM-6. The pH, specific gravity and oxide content were measured for each liquid feed sample. The annealed density of each glass sample was measured. The chemical composition of feed and glass samples were analyzed approximately every 1000 kg of glass production.

During the previous CFCM-6 melting trial a process control computer program was developed. The MOSUM test statistic described by Bauer and Hackl

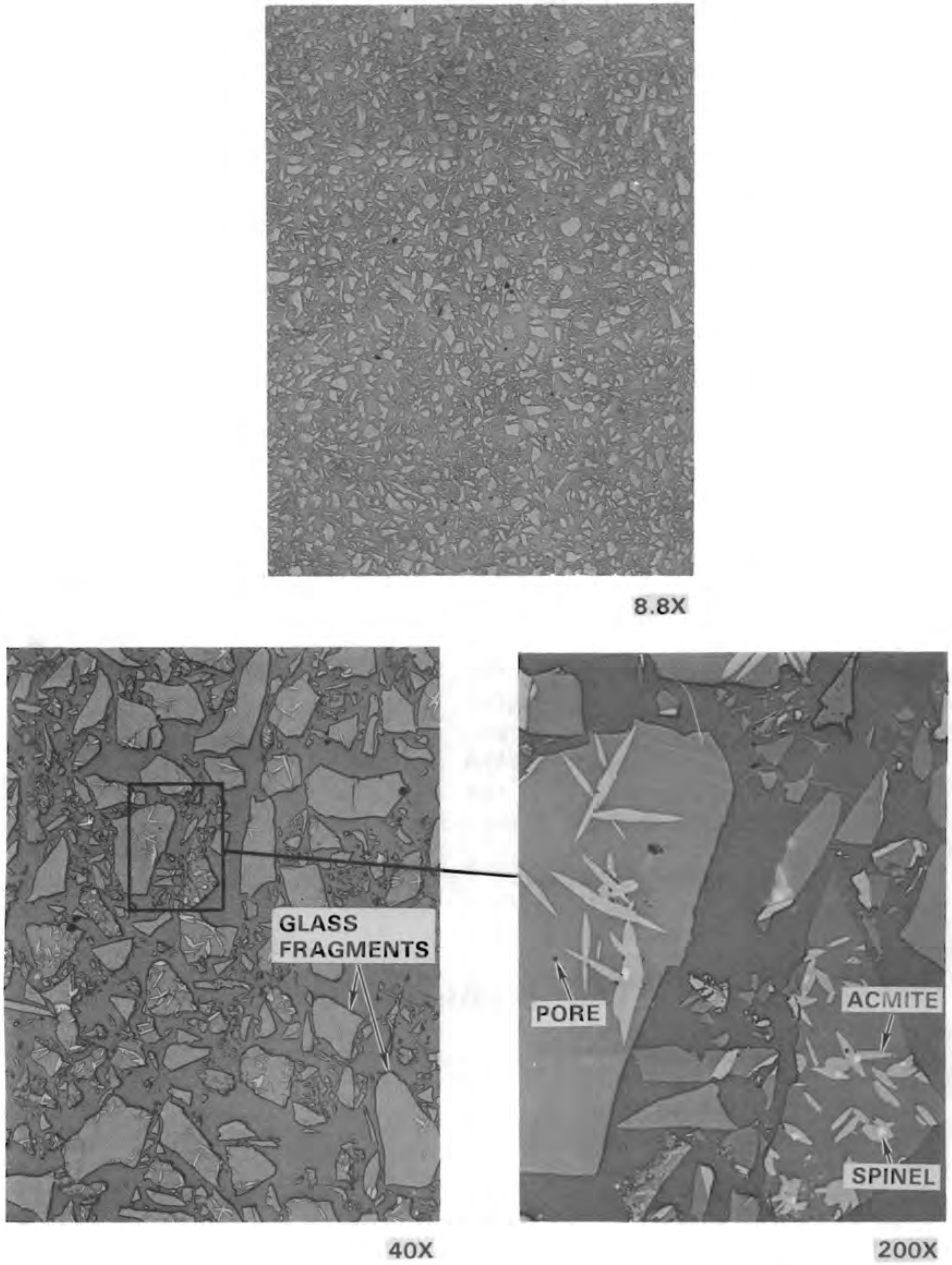

FIGURE 15. Microstructure of Canister 92 Sample [Height 0.30 m (1 ft); Depth - centerline]

TABLE 39. Crystalline Content of Canister 92 Samples

Core Height		Crystalline Content wt%	
m	ft	Canister Wall	Canister Centerline
0	0	---	0
0.30	1	0	25 wt% Acmite[(a)]
0.61	2	---	---
0.91	3	13 wt% Acmite	14 wt% Acmite[(a)]
1.21	4	---	---
1.52	5	0	8 wt% Acmite

(a) acmite Na, Fe, Si_2O_6

TABLE 40. Weight Loss of Canister 92 Samples Under pH4 Conditions

		wt% Loss		
		Core Depth, cm (in.)		
Core Height		7.6-15.2	15.2-22.9	
m	ft	(3-6)	(6-9)	Ave.
0	0	88.6	77.2	82.9
0.30	1	77.1	74.3	75.7
0.61	2	75.8	74.5	75.2
0.91	3	85.0	76.6	80.8
1.21	4	82.0	84.2	83.1
1.52	5	83.6	82.4	83.0
1.83	6	81.3	---	81.3
AVE.		81.9	78.2	

Mean pH4 Weight Loss 80.2 ± 4.5 wt%

TABLE 41. Weight Loss of Canister 92 Samples Under Soxhlet Conditions

		wt% Loss		
		Core Depth, cm (in.)		
Core Height		7.6-15.2	15.2-22.9	
m	ft	(3-6)	(6-9)	Ave.
0	0	2.32	3.19	2.74
0.30	1	4.51	3.55	4.03
0.61	2	3.58	3.36	3.47
0.91	3	2.22	2.48	2.35
1.21	4	2.31	2.43	2.37
1.52	5	2.31	2.51	2.41
AVE.		2.88	2.92	

Mean Soxhlet Weight Loss 2.90 ± 0.73 wt%.

(1978) indicates when a variable or constituent is beyond its normal range. Prior to April 21, 1979, MnO_2, CaO and Na_2O in the liquid feed were outside of control limits. The uncertain sampling error involved with MnO_2 and the relatively low levels of Na_2O did not justify feed modifications for either component. On April 21, 1979 the feed $CaCO_3$ content was increased 15% to bring CaO within control limits. A graph of feed CaO content during the run clearly shows the anticipated response (Figure 16). The feed composition during the trial listed in Table 42 was within control limits for all components except Na_2O which was higher than defined levels. The variation of Na_2O, SiO_2, and MnO_2 was approximately 12%, 14%, and 39%. All other constituents varied approximately 5%. As in CFCM-6, no relationship was found between specific gravity or pH of the feed and its oxide content or composition. The mean specific gravity was 1.041 ± 0.012 g/cc.

Grab samples of the glass product collected every 1000 kg of production were chemically and crystallographically examined. Throughout the trial, all

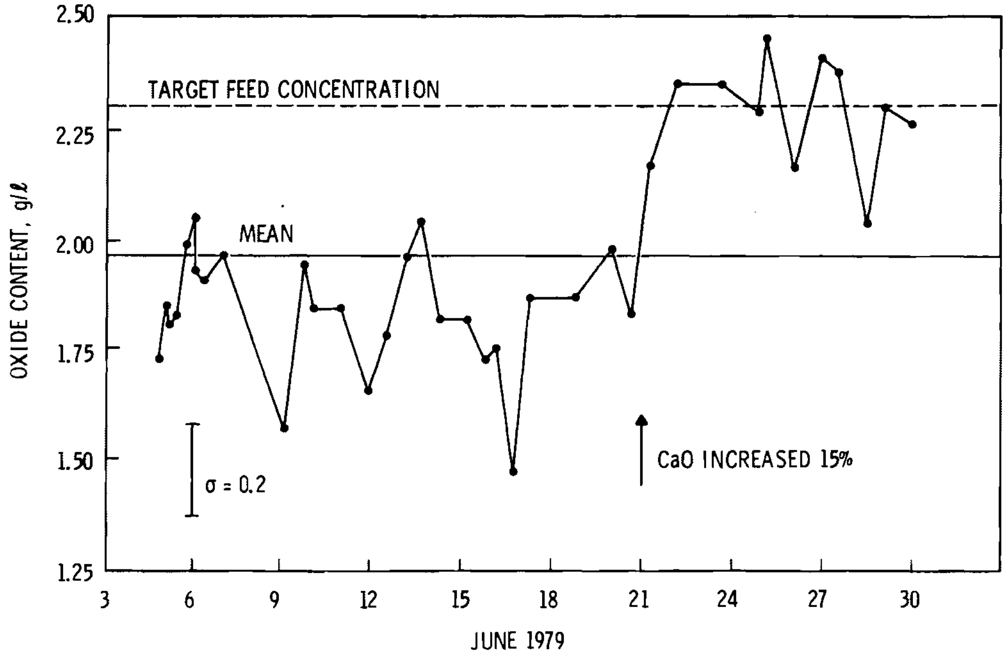

FIGURE 16. CaO Content of CFCM-7 Liquid Feed

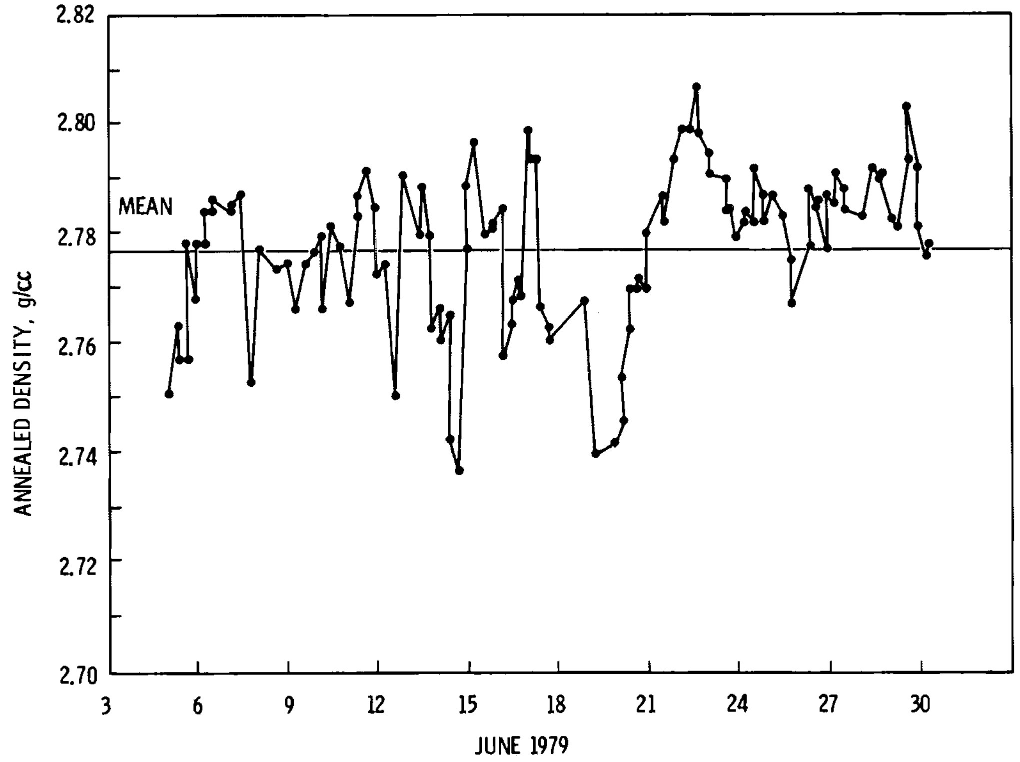

FIGURE 17. Annealed Density CFCM-7 Grab Samples

constituents were within acceptable composition control limits with the exception of CaO, which was low. The normalized composition of glass samples are listed in Table 43. The annealed density of samples collected during the trial is plotted in Figure 17 and shows that major density changes are superimposed over normal density variation. The correlation coefficients between glass density and composition in Table 44 show a significant positive relationship with major waste constituents such as Fe_2O_3, MnO_2 and NiO and negative relationship with frit constituents SiO_2, Li_2O and B_2O_3. This suggests that a major source of glass compositional variation is the waste/frit ratio delivered to the melter. Additional variation is caused by feed consistency. Although the large variation in feed MnO_2 content was largely sampling error, the large variation noted in the glass MnO_2 content suggests that MnO_2 was not being consistently delivered to the process.

TABLE 42. Composition of CFCM-7 Liquid Feed Samples

		g Oxide/L Feed							
Date	Time	Total	SiO_2	Al_2O_3	CaO	Fe_2O_3	Na_2O	MnO_2	NiO
Defined		64.9	4.5	8.5	2.6	34.9	1.4	9.1	4.0
6/5/79	0300	54.2	4.8	8.1	2.0	32.0	2.1	1.5	3.5
	0710	56.1	4.5	8.0	2.1	32.3	2.4	2.2	4.3
	1101	58.7	5.3	8.0	2.1	34.1	2.0	2.3	4.5
6/6/79	0345	59.0	4.0	8.3	2.2	34.1	2.0	3.4	4.6
	0600	61.4	4.3	9.1	2.2	36.4	2.2	1.9	4.9
	0920	60.2	4.8	8.5	2.1	35.0	2.4	2.4	4.7
	1235	58.7	3.6	8.0	2.1	34.5	2.8	2.8	4.6
6/7/79	0200	59.3	4.2	8.6	2.2	34.6	2.4	2.2	4.7
6/9/79	0940	58.9	4.4	8.4	1.8	34.9	2.5	2.0	4.5
	1950	63.3	4.6	9.5	2.2	35.1	2.6	4.2	4.6
6/10/79	0800	62.9	4.6	8.9	2.1	35.9	3.0	3.4	4.5
6/11/79	0508	61.9	4.6	9.1	2.1	35.5	2.7	3.2	4.3
	2300	60.4	4.5	8.9	1.9	35.1	2.5	2.6	4.5
6/12/79	1530	57.6	4.0	8.1	2.0	33.2	2.8	2.5	4.3
6/13/79	0750	64.7	4.7	8.8	2.2	35.5	3.1	5.6	4.4
	2255	59.7	4.7	9.6	2.3	29.2	2.2	7.0	4.3
6/14/79	1320	57.6	4.0	8.1	2.0	33.2	2.8	2.5	4.2
6/15/79	0620	53.8	3.2	8.2	2.0	31.1	2.2	2.3	4.4
	1925	59.3	4.8	8.6	2.0	34.6	2.4	2.2	4.5
6/16/79	0800	57.2	4.4	8.8	2.0	31.7	2.3	2.8	4.7
	2035	57.8	4.1	8.9	1.7	31.7	2.2	4.2	4.6
6/17/79	1230	56.4	4.2	8.6	2.1	31.0	2.2	3.5	4.3
6/18/79	2010	56.3	4.2	8.4	2.1	32.6	2.4	2.0	4.4
6/20/79	0500	53.5	2.2	7.7	2.1	32.1	2.9	1.5	4.6
	1450	56.4	3.3	8.5	2.0	32.8	2.8	2.1	4.6
6/21/79	0830	59.0	4.1	8.1	2.4	34.4	2.7	2.4	4.4
6/22/79	0500	60.9	4.3	8.5	2.6	33.9	2.6	4.0	4.3
6/23/79	1330	59.6	4.4	7.8	2.6	34.5	2.6	2.7	4.4
6/24/79	2100	59.1	3.7	7.9	2.5	34.2	2.7	3.5	4.3
6/25/79	0345	59.7	4.2	8.4	2.7	33.4	2.6	3.7	4.3
6/26/79	0430	60.6	4.4	8.0	2.4	33.3	3.1	4.3	4.4
	2320	56.6	3.3	7.4	2.6	33.8	2.0	3.1	4.2
6/27/79	1315	59.2	3.9	8.5	2.6	33.5	1.8	4.4	4.2
6/28/79	1130	53.9	3.1	6.7	2.2	28.5	1.8	7.5	3.7
6/29/79	0525	58.3	3.6	8.4	2.5	31.2	2.8	2.5	4.3
6/30/79	0104	59.8	4.1	8.2	2.5	35.3	2.5	2.6	4.2
AVE.		58.7	4.1	8.4	2.2	33.4	2.5	3.1	4.4
STD. DEV.		2.5	0.6	0.6	0.2	1.8	0.3	1.2	0.2

TABLE 43. Normalized Composition of CFCM-7 Grab Samples

Date	Time	Oxide wt%								
		SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
6/4/79	2310	46.4	4.4	8.0	4.8	14.0	3.2	13.9	3.4	1.4
6/5/79	0300	47.6	3.9	8.2	4.7	13.5	3.3	14.2	2.6	1.2
	0710	47.6	3.8	8.2	4.8	14.0	3.1	14.1	2.4	1.3
	1100	45.1	3.6	8.0	4.9	13.8	4.2	15.8	2.4	1.5
	1510	46.1	3.6	8.1	4.9	13.6	2.8	15.6	3.0	1.6
	1840	44.8	3.9	8.0	4.9	14.3	2.8	15.6	3.6	1.6
	2325	45.1	3.8	8.0	4.9	14.1	2.8	15.4	3.6	1.7
	2330				CANISTER 77 OUT					
6/6/79	0350	44.7	3.8	7.9	4.8	13.9	3.1	15.5	3.9	1.7
	0600	44.5	3.9	7.9	4.9	14.1	2.7	15.6	4.0	1.7
	0920	44.5	3.7	7.8	4.8	14.8	2.7	15.4	3.7	1.9
6/7/79	0200	44.8	3.6	7.9	4.8	13.9	3.0	15.1	4.4	1.9
	0845				CANISTER 83 OUT					
	2000	45.2	3.5	8.1	4.8	13.3	3.1	15.6	4.0	1.7
6/9/79	0940	45.8	3.5	8.1	4.6	13.8	3.3	15.4	3.1	1.7
	1950	45.4	3.7	8.0	4.5	14.2	3.4	15.1	3.3	1.8
6/10/79	0800	46.0	3.6	8.0	4.6	13.3	3.4	15.6	3.2	1.6
	1355				CANISTER 70 OUT					
6/11/79	0510	45.9	3.6	8.1	4.6	12.9	3.4	15.2	4.1	15
	1630				CANISTER 200 OUT					
	2300	44.6	3.8	7.7	4.4	14.4	3.2	16.1	3.5	1.8
6/12/79	1530	46.8	3.3	8.1	4.7	12.7	3.3	15.4	3.4	1.5
	1830				CANISTER 120 OUT					
6/13/79	0750	44.9	3.8	7.8	4.6	14.7	3.2	15.2	3.3	1.8
	1810				CANISTER 109-L OUT					
	2255	45.6	3.8	7.9	4.6	13.2	3.3	15.7	3.8	1.6
6/14/79	1320	46.9	3.5	8.2	4.5	11.6	3.6	16.0	3.4	1.5
	1620				CANISTER 110-L OUT					
6/15/79	0015	43.5	4.3	7.7	4.4	14.2	3.4	15.4	4.3	1.9
	1615				CANISTER 85 OUT					
	1975	44.7	3.9	7.9	4.4	14.1	3.4	15.2	3.6	1.8

TABLE 43. (contd)

		Oxide wt%								
Date	Time	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
6/16/79	0800	45.7	3.7	8.1	4.4	12.4	3.5	15.6	4.1	1.7
	2035	44.7	3.9	8.0	4.2	13.0	3.6	15.8	3.9	1.8
6/17/79	0730				CANISTER 201 OUT					
	1230	44.8	3.9	8.0	4.4	13.2	3.5	16.0	3.8	1.8
6/18/79	2010	45.1	4.0	7.9	4.6	13.2	3.2	15.4	4.1	1.8
6/19/79	0635				CANISTER 118 OUT					
6/20/79	0500	45.8	3.8	8.3	4.9	12.7	3.0	15.2	4.0	1.7
	1620				CANISTER 82 OUT					
6/21/79	0500	44.0	4.0	7.9	4.8	14.7	3.0	14.8	4.3	1.9
6/22/79	2210				CANISTER 205 OUT					
6/23/79	0135	44.2	3.8	7.9	4.9	15.2	2.9	14.7	3.9	1.9
	0440	44.0	3.8	8.0	5.0	15.6	2.8	14.5	3.7	2.0
	2015	44.4	3.7	8.0	4.9	15.1	2.9	14.5	3.8	1.9
	2330				CANISTER 119 OUT					
6/24/79	1310	44.3	3.8	8.0	4.8	14.8	3.0	14.7	4.2	1.8
	2310	44.1	3.9	7.9	4.8	14.9	3.0	14.8	4.0	1.8
	2330				CANISTER 207 OUT					
6/25/79	0050	44.3	3.8	7.9	4.7	14.6	3.1	15.1	3.9	1.8
	2350	45.1	3.6	8.1	4.9	13.6	3.0	15.2	4.2	1.7
6/26/79	0035				CANISTER 211 OUT					
	2005	45.3	3.6	8.0	5.1	14.2	2.7	14.5	4.2	1.8
	1315	44.7	3.8	8.0	4.8	14.4	2.9	14.5	4.3	1.8
6/27/79	2027				CANISTER 204 OUT					
6/28/79	1130	45.3	3.7	7.9	4.8	14.6	2.9	15.1	4.1	1.8
6/29/79	0600	44.8	3.9	7.7	5.0	14.4	3.0	15.1	3.7	1.8
	1545				CANISTER 206 OUT					
6/30/79	0100	44.5	3.6	7.8	4.8	14.2	3.1	15.3	4.2	1.8
AVE		44.6	3.8	7.9	4.7	13.8	3.1	15.0	3.7	1.7
STD. DEV.		1.0	0.2	0.1	0.2	0.9	0.3	0.5	0.5	0.2

TABLE 44. Correlation Coefficients of Glass Density and Composition

	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Glass Density	-0.785	0.204	-0.647	0,294	0.825	-0.514	-0.242	0.469	0.769

Foaming of the waste/frit batch in the melter was a significant problem during the trial as in CFCM-6. In only one instance on June 7, 1979 did foaming alter waste glass properties by carrying unreacted calcine out of the melter by the glass stream. Crystalline material was detected in only one of the glass samples. In glass samples prior to June 22, 1979 small spheres less than 1 mm in dia containing primarily Na, S and Cl were occasionally found. The glass during this period contained 0.14 wt% Cl. This high amount of Cl^- was added as an unintentional impurity in one of the ferric hydroxide sources used. Before changing ferric hydroxide suppliers on June 20, 1979, liquid feed samples contained 2.37 g/L chlorine while subsequent samples contained 0.37 g/L. The chlorine analyzed in the glass represented less than 20% of that added in the feed. The anticipated Cl^- content of the TDS waste feed is 0.68 g/L.

One 0.91-m (3-ft)-dia canister of glass, 204, was sampled by core-drilling. The normalized composition of these samples is listed in Table 45. Table 46 shows no consistent variation in composition with sample location within the canister. The average glass composition was very close to the defined values with small standard deviations. Although all grab samples were amorphous, nearly all canister samples contained 5 to 20 wt% acmite (Table 47). Photomicrographs of two samples (Figures 18 and 19) show that the acmite crystals have resulted in extensive microcracking. Small, bright spinel crystals can be seen scattered throughout the sample. Although chemical analysis does not suggest product inhomogeneity, the nonuniform distribution of crystals indicate that localized compositional variations exist. The average sample weight losses under soxhlet and pH4 conditions were 3.56 ± 1.58 wt% and 74.8 ± 9.8 wt%, respectively (Tables 48 and 49). The cause of the high variation in weight loss for the relatively homogeneous samples is not known.

TABLE 45. Normalized Composition of Canister 204 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined				44.0	3.6	8.0	5.0	14.0	3.2	16.0	3.9	1.7
0	0	0-11.4	0-4.5	46.2	3.5	8.2	5.0	12.6	3.3	15.4	3.5	1.6
		11.4-22.9	4.5-9.0	45.7	3.7	8.0	4.9	12.9	3.3	15.4	3.7	1.7
		22.9-34.3	9.0-13.5	45.5	3.7	7.8	5.0	13.3	3.3	15.4	3.8	1.5
		34.3-45.7	13.5-18.0	45.5	3.7	7.8	5.1	13.7	3.2	14.8	3.9	1.7
0.30	1	0-11.4	0-4.5	46.1	3.7	8.1	5.2	13.6	3.1	14.3	4.0	1.4
		11.4-22.9	4.5-9.0	45.8	3.6	7.9	5.1	13.4	3.1	15.0	3.9	1.6
		22.9-34.3	9.0-13.5	45.7	3.6	7.9	5.0	13.3	3.2	15.1	3.9	1.6
0.61	2	0-11.4	0-4.5	45.4	3.8	8.0	5.0	13.6	3.1	14.7	4.0	1.7
		11.4-22.9	4.5-9.0	45.4	3.7	7.9	5.0	13.0	3.3	15.8	3.9	1.4
		22.9-34.3	9.0-13.5	45.8	3.7	8.0	5.1	13.6	3.1	14.8	4.0	1.2
		34.3-45.7	13.5-18.0	45.7	3.7	8.0	5.1	13.8	3.1	14.9	3.9	1.1
0.91	3	0-11.4	0-4.5	45.4	3.7	8.0	4.6	13.9	3.1	14.8	4.0	1.7
		11.4-22.9	4.5-9.0	45.2	3.7	8.0	5.0	13.1	3.3	15.9	3.8	1.4
		22.9-34.3	9.0-13.5	45.6	3.8	8.1	5.2	13.2	3.2	15.2	4.0	1.3
		34.3-45.7	13.5-18.0	45.5	3.7	8.1	5.1	14.0	3.1	14.9	4.0	1.1
1.21	4	0-11.4	0-4.5	44.8	3.8	7.9	5.0	13.9	3.1	15.1	4.0	1.7
		11.4-22.9	4.5-9.0	44.8	3.8	8.0	5.0	14.0	3.1	14.7	4.2	1.7
		22.9-34.3	9.0-13.5	45.1	3.8	7.9	5.1	14.3	3.2	15.0	4.0	1.1
		34.3-45.7	13.5-18.0	44.9	3.9	8.1	5.2	14.2	3.1	14.0	4.1	1.9
1.52	5	0-11.4	0-4.5	45.4	3.7	8.0	5.1	13.9	3.1	14.6	3.9	1.6
		11.4-22.9	4.5-9.0	45.5	3.7	7.9	5.1	13.8	3.2	14.8	3.6	1.7
		22.9-34.3	9.0-13.5	45.8	3.9	8.1	4.9	12.5	3.4	15.9	3.7	1.2

TABLE 46. Average Composition of Canister 204 Samples

Core Height		Oxide wt%								
m	ft	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	45.7	3.6	8.0	5.0	13.1	3.3	15.3	3.7	1.6
0.30	1	45.9	3.6	8.0	5.1	13.4	3.1	14.8	3.9	1.5
0.61	2	45.6	3.7	8.0	5.0	13.5	3.2	15.0	4.0	1.4
0.91	3	45.4	3.7	8.0	5.0	13.6	3.2	15.2	4.0	1.4
1.21	4	44.9	3.8	8.0	5.1	14.1	3.1	14.7	4.1	1.6
1.52	5	45.6	3.8	8.0	5.0	13.4	3.2	15.1	3.7	1.5
Core Depth										
cm	in.									
0-11.4	0-4.5	45.6	3.7	8.0	5.0	13.6	3.1	14.8	4.0	1.6
11.4-22.9	4.5-9.0	45.4	3.7	8.0	5.0	13.4	3.2	15.3	3.8	1.6
22.9-34.3	9.0-13.5	45.6	3.8	8.0	5.0	13.4	3.2	15.2	3.9	1.3
34.3-45.7	13.5-18.0	45.4	3.8	8.0	5.1	13.9	3.1	14.6	4.0	1.4
AVE.		45.5	3.7	8.0	5.0	13.5	3.2	15.0	3.9	1.5
STD. DEV.		0.4	0.1	0.1	0.1	0.5	0.1	0.5	0.2	0.2

TABLE 47. Crystalline Content of Canister 204 Samples

Core Height m	Core Height ft	Acmite wt% Core Depth, cm (in.) 0-11.4 (0-4.5)	11.4-22.9 (4.5-9.0)	22.9-34.3 (9.0-13.5)	34.3-45.7 (13.5-18.0)
0	0	0	20%(a)	10%	10%
0.30	1	0	10%	15%	---
0.61	2	5-10%	15%	<5%	5%
0.91	3	5%	10%	5%	<5%
1.21	4	5-10%	10-15%	5%	<5%
1.52	5	5-10%	10%	<5%	---

(a) Acmite $NaFeSi_2O_6$

CFCM-8 (NOVEMBER 1979)

Melting trial CFCM-8 converted 19,180 L of simulated liquid waste into 4220 kg of simulated defense waste glass. The objective of this trial was to determine the impact of reducing agents and improved waste/frit mixing on foaming in the CFCM. Cornstarch at 1 to 1-1/2 wt% on a glass basis was added as the reducing agent first in the liquid feed and later mixed with the frit. The location of the frit addition line was moved nearer the filters in the spray calciner for better mixing of the calcine and frit. Some of the Na_2O and SiO_2 from the frit was initially incorporated in the liquid feed to improve mixing. As a result, the frit composition was altered to produce the standard TDS-211 waste glass composition. These modifications did not significantly reduce foaming or improve process melting rates. The average melting rate was approximately 53 kg/h (70 $kg/h/m^2$).

A liquid-feed and waste-glass sampling program similar to that used in CFCM-6 and CFCM-7 was used. Table 50 lists the composition of liquid feed samples collected during the trial. The two defined waste compositions, Parts I and II, reflect the initial addition of sodium silicate (Na_2O and SiO_2) to the liquid feed and its subsequent removal. Considerable variation was noted for many of the feed constituents. The large variation in MnO_2 and a significant

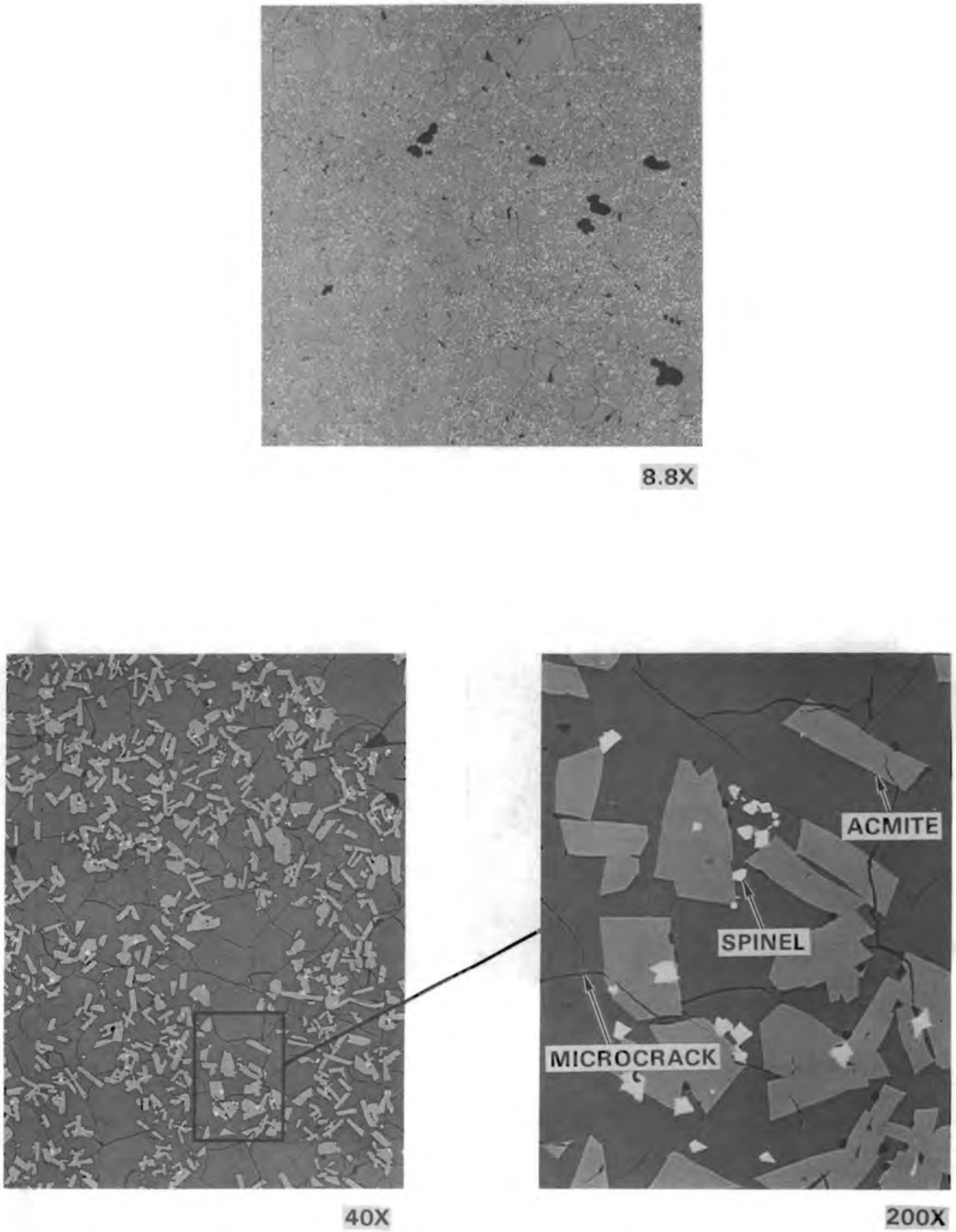

FIGURE 18. Microstructure of Canister 204 Sample [Height 0.61 m (2 ft); Depth 11.4-22.9 cm (4.5-9.0 in.)]

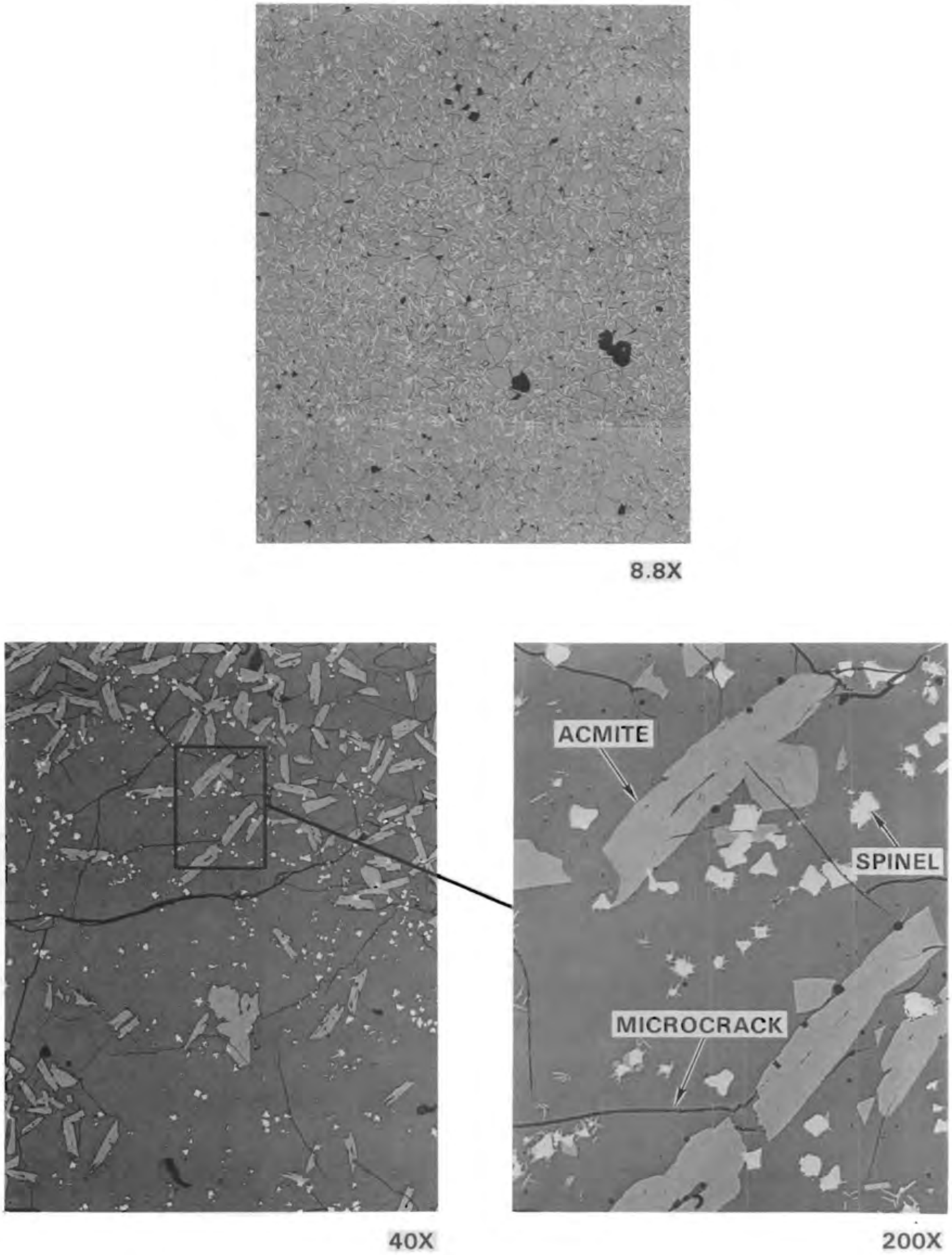

FIGURE 19. Microstructure of Canister 204 Sample [Height 1.21 m (4 ft); Depth 11.4-22.9 cm (4.5-9.0 in.)]

TABLE 48. Weight Loss of Canister 204 Samples Under pH4 Conditions

		wt% Loss				
		Core Depth, cm (in.)				
Core Height		0-11.4	11.4-22.9	22.9-34.3	34.3-45.7	
m	ft	(0-4.5)	(4.5-9.0)	(9.0-13.5)	(13.5-18.0)	Ave.
0	0	81.2	58.5	66.2	69.6	68.9
0.30	1	81.5	73.5	71.4	---	75.5
0.61	2	60.1	60.6	76.6	73.2	67.6
0.91	3	85.1	62.2	76.2	85.0	77.2
1.21	4	71.9	66.3	86.6	92.1	79.5
1.52	5	81.3	83.4	83.3	---	82.3
AVE.		76.8	67.4	76.7	80.3	

Mean pH4 Weight Loss 74.8 ± 9.8 wt%

TABLE 49. Weight Loss of Canister 204 Samples Under Soxhlet Conditions

		wt% Loss				
		Core Depth, cm (in.)				
Core Height		0-11.4	11.4-22.9	22.9-34.3	34.3-45.7	
m	ft	(0-4.5)	(4.5-9.0)	(9.0-13.5)	(13.5-18.0)	Ave.
0	0	1.88	6.36	4.66	4.49	4.35
0.30	1	2.02	3.57	3.73	---	3.11
0.61	2	4.13	6.39	2.26	6.84	4.91
0.91	3	2.03	5.18	2.15	2.24	2.90
1.21	4	3.88	4.56	2.12	2.21	3.19
1.52	5	2.60	2.42	2.64	---	2.55
AVE.		2.76	4.75	2.93	3.95	

Mean Soxhlet Weight Loss 3.56 ± 1.58 wt%

portion of the total oxide variation is due to sampling error with the dense MnO_2 particles. As in previous melting trials, the liquid feed specific gravity showed little relationship to feed composition or oxide content. The feed pH dropped rapidly with the removal of sodium silicate as shown in Figure 20. Little mixing of the two liquid feed compositions occurred in the process.

TABLE 50. Oxide Composition of CFCM-8 Liquid Feed Samples

		g Oxide/L Feed							
Date	Time	Total	SiO_2	Al_2O_3	CaO	Fe_2O_3	Na_2O	MnO_2	NiO
PART I									
Defined		74.7	9.6	8.3	2.3	34.1	7.4	9.1	4.0
11/5	0220	73.2	9.9	9.8	2.3	35.1	6.6	5.1	4.5
	0730	73.8	10.8	9.2	2.3	34.4	6.9	5.8	4.5
	1000	63.9	7.5	7.0	2.2	34.9	4.3	3.4	4.5
	1300	58.8	8.0	7.0	2.1	32.3	4.0	1.3	4.1
	1500	69.4	9.2	7.9	2.3	35.9	4.9	4.6	4.6
	2145	65.4	7.2	7.8	2.3	35.8	4.6	3.0	4.6
11/6	0020	59.5	7.1	6.6	2.2	33.3	4.4	1.8	4.0
	0200	64.6	7.5	7.0	2.3	35.9	5.7	1.8	4.4
	1000	67.0	8.4	7.3	2.2	34.3	6.5	4.1	4.2
	1200	64.7	9.0	7.5	2.2	34.2	6.4	1.2	4.2
	1400	62.4	8.3	7.0	2.2	33.2	6.3	1.4	4.1
11/7	0015	62.8	7.0	7.0	2.2	33.2	6.1	3.3	4.1
	0216	62.5	8.8	7.2	2.1	32.1	6.5	1.5	4.0
	0400	62.0	6.8	6.7	2.2	33.5	6.0	2.7	4.1
PART I AVE.		65.0	8.2	7.5	2.2	34.2	5.2	2.9	4.3
STD. DEV.		4.5	1.2	0.9	0.1	1.2	1.6	1.5	0.2
PART II									
Defined		65.5	6.5	8.3	2.3	34.1	1.2	9.1	4.0
11/7	2000	53.8	4.2	7.7	2.0	31.1	1.9	2.8	4.0
11/8	0010	54.0	2.5	7.2	2.0	35.8	1.3	1.4	3.8
	0200	62.0	3.7	7.6	2.1	41.0	1.8	1.9	3.9
	0405	73.0	6.3	9.5	2.4	42.5	1.5	6.6	4.2
	0621	61.2	3.7	7.3	2.1	37.7	1.7	4.9	3.8
	0840	61.7	5.6	8.2	2.4	32.4	1.9	7.0	4.2
	1810	43.9	2.7	6.5	1.6	25.4	1.3	2.8	3.5
11/9	0005	54.2	3.1	7.5	2.0	30.7	1.7	5.1	4.1
	0200	47.6	2.9	6.4	2.0	27.5	1.6	3.5	3.7
	0400	44.8	1.9	6.1	1.9	28.1	1.6	1.4	3.9
	0600	43.9	2.1	5.9	1.9	27.3	1.7	1.1	4.0
	1220	45.9	2.2	6.7	1.7	27.1	1.4	3.0	3.8
	1400	41.5	2.4	5.3	1.7	24.8	1.5	1.8	3.6
PART II AVE.		52.9	3.3	7.1	2.0	31.6	1.6	3.3	3.9
STD. DEV.		9.4	1.2	1.1	0.2	5.9	0.2	2.0	0.2

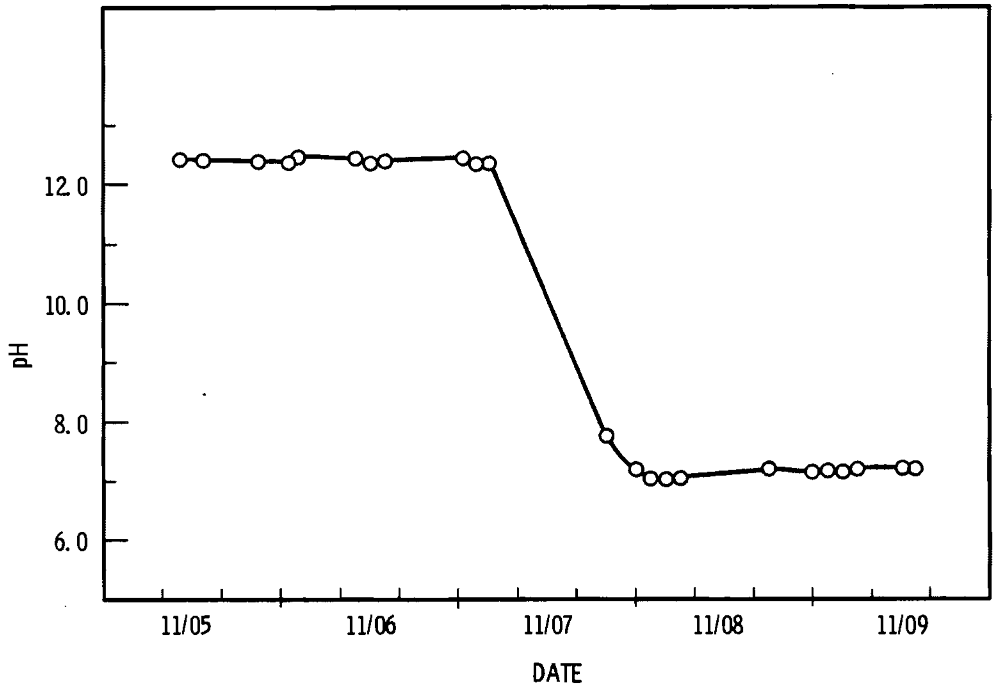

FIGURE 20. pH of CFCM-8 Liquid Feed Samples

The waste glass produced during the CFCM-8 melting trial was slightly deficient in waste components containing an average estimated 23 wt% waste oxides compared to the target 28 wt%. The composition of the wastes component in the glass showed significantly reduced levels of Fe_2O_3 and NiO with increased levels of MnO_2 (Table 51). This is not consistent with the liquid feed analysis which indicated near target levels of Fe_2O_3 and NiO. The formation and separation of a Fe-Ni spinel crystalline phase in the glass would decrease to some degree the quantities of Fe_2O_3 and NiO in the waste glass. In only one instance, however, were spinel crystals identified in a grab sample. All others contained no crystalline material. With no estimate of the quantity of spinel accumulated in the melter during the trial, this possibility remains unconfirmed.

The last canister filled, 113, was core-drilled for sample analysis. The normalized composition of these samples is listed in Table 52. The averages in Table 53 show a significant decrease in all waste components except MnO_2

TABLE 51. Normalized Composition of CFCM-8 Grab Samples

		Oxide wt%								
Date	Time	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
11/5/79	0330	45.6	4.5	7.9	5.3	14.0	2.9	14.8	3.5	1.2
	0830	46.5	4.3	8.0	5.2	12.7	3.0	14.6	4.2	1.2
	1350	47.6	4.2	8.3	5.0	11.5	3.1	14.3	4.5	1.2
	2330	48.1	3.7	8.5	5.0	10.8	3.2	14.6	4.6	1.3
11/6/79	0330	48.2	3.5	8.7	5.0	10.7	3.1	14.6	4.6	1.3
	0900	48.2	4.9	8.6	4.9	9.8	3.2	15.0	4.1	1.2
	1400	48.8	3.3	8.9	4.8	10.3	3.5	14.7	4.2	1.3
	1800				CANISTER 209 OUT					
11/7/79	0122	46.9	3.3	8.6	4.8	10.9	3.4	15.9	4.5	1.4
	0537	47.4	3.4	8.7	4.7	10.7	3.5	15.6	4.3	1.4
	1725	47.4	3.4	8.5	4.8	11.3	3.2	15.2	4.4	1.5
	2050	47.8	3.4	8.5	4.6	10.8	3.3	15.3	4.6	1.4
	2340	47.8	3.3	8.6	4.8	10.8	3.0	15.5	4.5	1.4
11/8/79	0530	48.8	3.1	8.7	4.5	10.2	3.9	16.4	2.9	1.3
	0945	46.5	3.4	8.6	4.7	13.4	3.1	14.7	3.8	1.5
	1100				CANISTER 115 OUT					
	1130	46.4	3.4	8.4	4.7	12.5	3.2	15.5	4.2	1.4
	1500				CANISTER 114 OUT					
	2230	46.9	3.5	8.6	4.7	11.8	3.2	15.3	4.3	1.5
11/9/79	0116	47.5	3.2	8.8	4.8	10.7	3.4	15.9	4.2	1.4
	0444	48.7	3.0	9.1	4.8	9.0	3.4	16.0	4.6	1.2
	0650	48.6	3.0	8.9	4.9	9.1	3.4	16.5	4.4	1.2
	1200	48.5	3.2	8.8	4.8	9.1	3.5	16.6	4.1	1.2
	1600				CANISTER 113 OUT					
AVE.		47.6	3.6	8.6	4.8	11.0	3.3	15.4	4.2	1.3
STD. DEV.		0.9	0.5	0.3	0.2	1.4	0.2	0.7	0.4	0.1
CANISTER 113										
AVE.		48.0	3.2	8.8	4.8	9.9	3.4	16.1	4.2	1.3
STD. DEV.		0.8	0.2	0.2	0.1	1.2	0.1	0.5	0.2	0.2

above the 0.61-m (2-ft) level. The analysis of grab samples collected during filling of canister 113 show the same decrease in waste content. Micrographs of the low waste content product above the 0.61-m (2-ft) level show an

TABLE 52. Normalized Composition of Canister 113 Samples

Core Height		Core Depth		Oxide wt%								
m	ft	cm	in.	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined				44.0	3.7	8.0	5.0	14.5	3.2	15.5	3.9	1.7
0	0	0-7.6	0-3	45.4	3.5	8.0	4.8	13.3	3.3	15.5	4.3	1.7
		7.6-15.2	3-6	45.7	3.4	8.0	4.7	12.4	3.4	16.0	4.2	1.6
		15.2-22.9	6-9	46.4	3.4	8.3	4.8	12.2	3.3	15.7	4.1	1.6
		22.9-30.5	9-12	46.5	3.3	8.3	4.9	12.4	3.2	15.4	4.2	1.6
0.30	1	0-7.6	0-3	46.6	3.3	8.4	4.8	11.8	3.3	15.8	4.2	1.5
		7.6-15.2	3-6	47.1	3.3	8.5	4.9	11.5	3.3	15.6	4.2	1.5
		15.2-22.9	6-9	46.8	3.3	8.4	4.9	11.8	3.3	15.8	4.2	1.4
		22.9-30.5	9-12	46.7	3.4	8.2	4.9	12.1	3.1	15.5	4.2	1.6
0.61	2	0-7.6	0-3	48.5	3.0	8.7	5.0	9.9	3.3	15.8	4.3	1.3
		7.6-15.2	3-6	48.9	3.0	8.8	5.0	9.7	3.2	15.5	4.4	1.3
		15.2-22.9	6-9	48.9	2.9	8.7	5.0	9.2	3.4	16.2	4.3	1.2
		22.9-30.5	9-12	48.9	2.9	8.8	5.0	9.2	3.4	16.0	4.4	1.2
0.91	3	0-7.6	0-3	49.3	2.9	8.8	5.0	9.0	3.4	15.8	4.3	1.2
		7.6-15.2	3-6	49.4	3.0	8.9	5.0	9.1	3.5	15.3	4.3	1.2
		15.2-22.9	6-9	49.4	3.1	8.8	5.1	9.2	3.4	15.2	4.3	1.2
		22.9-30.5	9-12	48.9	3.0	8.7	4.8	8.9	3.6	16.5	4.1	1.2
1.21	4	0-7.6	0-3	48.5	3.0	8.7	4.8	8.8	3.6	17.3	3.9	1.2
		7.6-15.2	3-6	49.2	3.0	8.8	5.0	9.1	3.5	15.9	4.0	1.3
		15.2-22.9	6-9	48.8	3.0	8.8	5.0	9.1	3.4	16.2	4.1	1.2
		22.9-30.5	9-12	48.8	3.0	8.7	4.9	9.0	3.5	16.5	4.0	1.2
1.52	5	0-7.6	0-3	48.4	3.1	8.7	4.9	9.3	3.4	16.4	4.2	1.3
		7.6-15.2	3-6	48.9	3.0	8.8	4.9	8.8	3.6	16.4	4.0	1.2
		15.2-22.9	6-9	48.7	3.0	8.8	4.9	9.0	3.6	16.2	4.3	1.3
		22.9-30.5	9-12	48.9	3.0	8.8	5.0	9.2	3.5	16.0	4.1	1.3

inhomogeneous distribution of fine acmite crystals in a glass matrix (Figures 21 and 22). While some porosity is present, there is an absence of microcracking. There are also very few spinel crystals in these samples. X-ray data in Table 54 shows that the major compositional change in the canister did not significantly alter the crystalline phase present. While samples near the wall were generally amorphous, more central samples contained from 5.8 to 16.5 wt% acmite crystals.

Compositional changes, however, directly affected the glass durability under pH4 conditions. As waste content decreased, pH4 durability increased

TABLE 53. Average Composition of Canister 113 Samples

Core Height		Oxide wt%								
m	ft	SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	Li_2O	Na_2O	MnO_2	NiO
Defined		44.0	3.7	8.0	5.1	14.7	3.2	15.5	3.9	1.7
0	0	46.0	3.4	8.2	4.8	12.6	3.3	15.6	4.2	1.6
0.30	1	46.8	3.3	8.4	4.9	11.8	3.2	15.7	4.2	1.5
0.61	2	48.8	3.0	8.8	5.0	9.5	3.3	15.9	4.4	1.2
0.91	3	49.2	3.0	8.8	5.0	9.0	3.5	15.7	4.2	1.2
1.21	4	48.8	3.0	8.8	5.0	9.0	3.5	16.5	4.0	1.2
1.52	5	48.7	3.0	8.8	4.9	9.1	3.5	16.2	4.2	1.3
Core Depth										
cm	in.									
0-7.6	0-3	47.8	3.1	8.5	4.9	10.4	3.4	16.1	4.2	1.4
7.6-15.2	3-6	48.2	3.1	8.6	4.9	10.1	3.4	15.8	4.2	1.4
15.2-22.9	6-9	48.2	3.1	8.6	5.0	10.1	3.4	15.9	4.2	1.3
22.9-30.5	9-12	48.1	3.1	8.6	4.9	10.1	3.4	16.0	4.2	1.4
AVE.		48.1	3.1	8.6	4.9	10.1	3.4	15.9	4.2	1.3
STD. DEV.		1.3	0.2	0.3	0.1	1.5	0.1	0.5	0.1	0.2

from 87.2 ± 5.4 wt% to 30.6 ± 7.3 wt%. Soxhlet results did not show this behavior with an average weight loss of 2.37 ± 0.33 wt%. Tables 55 and 56 present weight loss data for pH4 and soxhlet conditions. Grab samples were similarly tested for pH4 and soxhlet durability. The pH4 weight loss of corresponding canister core and grab samples in Figure 23 shows similar behavior. The average pH4 and soxhlet weight loss for grab samples were 33.0 ± 19.2 wt%, and 2.00 ± 0.26 wt%, respectively.

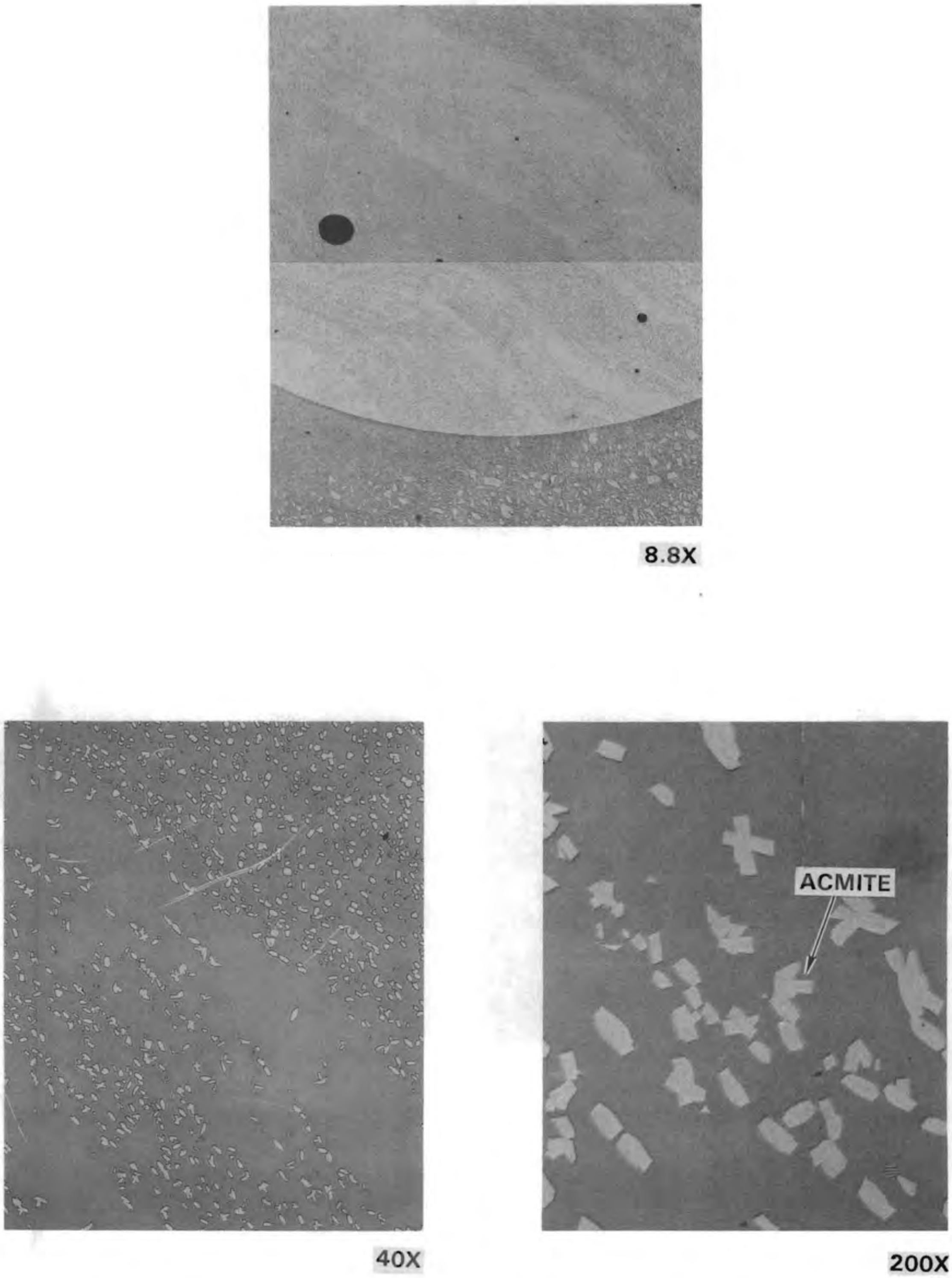

FIGURE 21. Microstructure of Canister 113 Sample [Height 0.61 m (2 ft); Depth 22.9-30.5 cm (9-12 in.)]

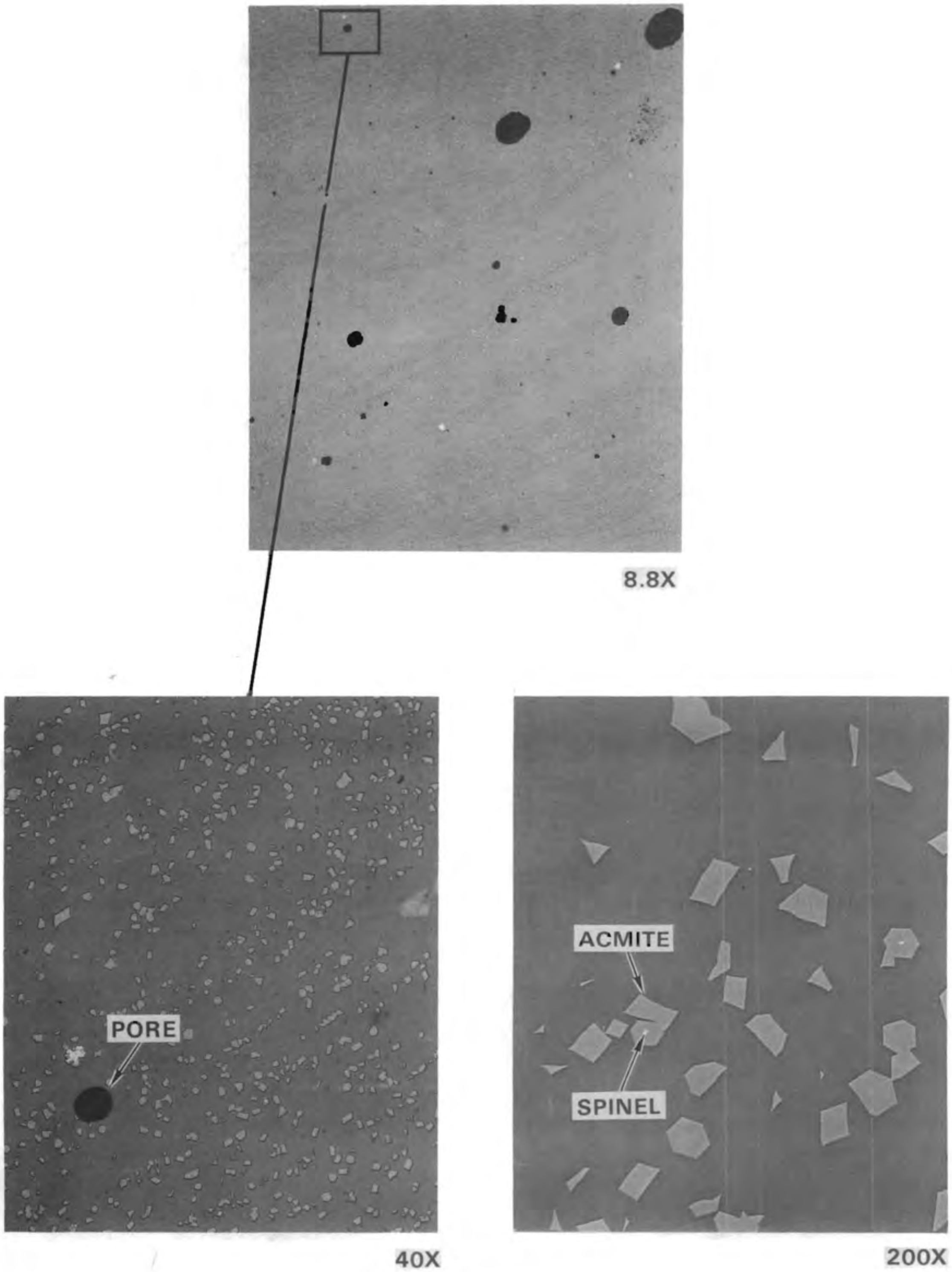

FIGURE 22. Microstructure of Canister 113 Sample [Height 1.21 m (4 ft); Depth 22.9-30.5 cm (9-12 in.)]

TABLE 54. Crystalline Content of Canister 113 Samples

Core Height		Acmite Crystals[(a)] wt%				
		Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	0	7.2	5.8	6.9	5.0
0.30	1	0	0	11.5	9.8	5.3
0.61	2	0	0	7.2	14.7	7.3
0.91	3	0	0	16.5	4.9	5.3
1.21	4	0	10.5	7.1	8.1	6.4
1.52	5	0	6.6	7.1	0	3.4
AVE.		0	4.0	9.2	7.2	

(a) Acmite only crystalline phase identified.

TABLE 55. Weight Loss of Canister 113 Samples Under pH4 Conditions

Core Height		wt% Loss				
		Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	89.9	93.0	89.7	88.2	90.2
0.30	1	80.7	80.4	82.2	93.5	84.2
0.61	2	43.1	37.8	30.0	40.6	37.9
0.91	3	32.1	28.2	30.5	37.9	32.2
1.21	4	31.9	25.8	32.6	31.2	30.4
1.52	5	24.4	15.8	27.9	19.2	21.8
AVE.		50.3	46.8	48.8	51.8	

Mean Weight Loss Under pH4 Conditions 49.4 ± 28.0 wt%

TABLE 56. Weight Loss of Canister 113 Samples Under Soxhlet Conditions

Core Height		wt% Loss				
		Core Depth, cm (in.)				
m	ft	0-7.6 (0-3)	7.6-15.2 (3-6)	15.2-22.9 (6-9)	22.9-30.5 (9-12)	Ave.
0	0	2.04	2.00	2.65	2.52	2.30
0.30	1	1.88	2.05	2.29	2.05	2.07
0.61	2	2.00	2.21	2.32	2.92	2.36
0.91	3	2.52	2.06	2.28	2.21	2.27
1.21	4	2.89	3.11	2.51	2.59	2.78
1.52	5	2.37	2.24	2.71	2.30	2.44
AVE.		2.28	2.28	2.46	2.43	

Mean Weight Loss Under Soxhlet Conditions 2.36 ± 0.33 wt%

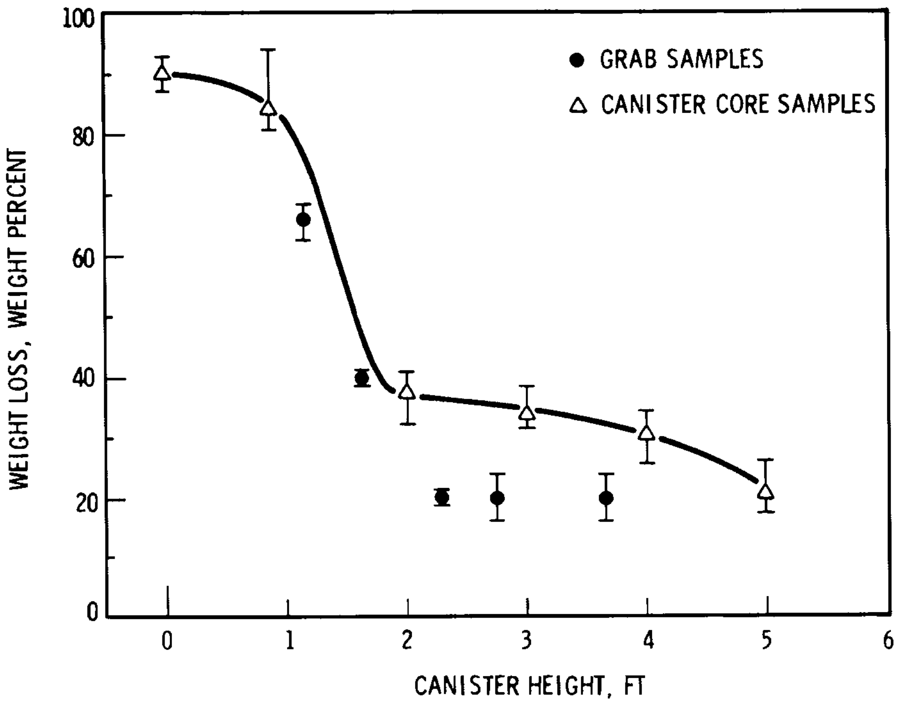

FIGURE 23. Weight Loss of CFCM-8 Glass Under pH4 Conditions

DISCUSSION

Over 65,000 kg of simulated defense waste glass was produced at PNL between March 1979 and March 1980. The three vitrification processes used, SC/ICM, LFCM and SC/CFCM, involve differences in equipment design and operation. These factors contribute to variations in the final glass product. Table 57 summarizes glass product information collected during the various melting trials. These results will be discussed in relation to liquid feed composition, melting process, and product durability for canisters filled by the three vitrification processes.

LIQUID FEED COMPOSITION

The three vitrification processes all melt a mixture of waste and glass-forming constituents. The simulated waste component was prepared by mixing chemicals in water at specified concentrations. The behavior of the liquid waste feed in-process equipment was a function of solubility, density and particle sizes of the individual waste components.

Liquid feed samples were collected and analyzed during three SC/CFCM melting trials. This data listed in Tables 37, 42 and 50 is summarized in Table 58. Although the feed was prepared to the defined TDS waste composition, the concentration was adjusted as necessary to improve calciner throughput capacity.

Under controlled pilot-plant conditions at PNL where industrial-grade chemicals are used, up to 10% variation is normally encountered (in individual feed components). Changes in chemical suppliers can produce larger variations if the materials are not assayed prior to use. This situation was encountered with ferric hydroxide. Coarse insoluble components such as zeolite and MnO_2 are difficult to deliver to the process at a constant rate and may present sampling problems. Zeolite, which is the main source of SiO_2 in the waste, varied considerably. However, in terms of the waste and resulting waste glass composition, these fluctuations were not significant. Another example of difficulty in feeding dense insoluble constituents is MnO_2. Melting trial CFCM-6

TABLE 57. Data Summary for Melting Trial Samples

Process Run	Canister	Oxide Composition, wt%									Leach Test Weight Loss wt%	
		SiO_2	Al_2O_3	B_2O_3	CaO	Fe_2O_3	LiO_2	Na_2O	MnO_2	NiO	pH4	Soxhlet
FS-ICM-20	175	47.2 ± 1.3	2.8 ± 0.3	8.4 ± 0.2	(a)	15.4 ± 2.5	(a)	11.9 ± 2.6	(a)	1.2 ± 0.1	(a)	(a)
FS-ICM-23	179	45.7 ± 0.7	3.0 ± 0.2	9.0 ± 0.1	(a)	11.5 ± 1.0	(a)	16.7 ± 0.3	(a)	1.1 ± 0.1	(a)	(a)
FS-ICM-24	177	49.5 ± 0.3	3.0 ± 0.1	8.8 ± 0.1	5.1 ± 0.1	9.3 ± 0.2	3.5 ± 0.1	16.1 ± 0.5	2.9 ± 0.1	1.2 ± 0.1	17.7 ± 2.8	2.37 ± 0.13
FS-ICM-26	174	46.2 ± 1.7	3.9 ± 0.6	7.8 ± 0.3	5.2 ± 0.2	11.7 ± 1.6	3.2 ± 0.2	16.2 ± 1.5	3.6 ± 0.5	1.7 ± 0.2	84.2 ± 4.9	2.41 ± 0.20
FS-ICM-27	172	43.4 ± 0.9	4.5 ± 0.2	7.1 ± 0.3	5.4 ± 0.2	16.5 ± 0.6	2.8 ± 0.2	14.3 ± 0.6	3.7 ± 0.2	1.9 ± 0.2	85.2 ± 3.0	3.40 ± 0.61
SRL-13	148	46.1 ± 0.3	4.0 ± 0.1	8.3 ± 0.1	5.0 ± 0.1	12.2 ± 0.2	3.3 ± 0.1	15.1 ± 0.5	3.8 ± 0.1	1.3 ± 0.1	75.1 ± 6.0	1.78 ± 0.12
SRL-LF-1	10	45.6 ± 0.3	4.3 ± 0.1	7.9 ± 0.1	5.0 ± 0.1	13.2 ± 0.2	3.1 ± 0.1	15.1 ± 0.5	3.5 ± 0.1	1.4 ± 0.1	73.8 ± 2.3	2.05 ± 0.21
	Run	45.3 ± 0.4	4.2 ± 0.1	8.2 ± 0.1	5.0 ± 0.1	12.7 ± 0.3	3.0 ± 0.1	15.9 ± 0.5	3.5 ± 0.2	1.3 ± 0.1	(a)	(a)
CFCM-6	92	43.0 ± 3.0	4.0 ± 0.5	8.0 ± 0.2	4.6 ± 0.2	14.3 ± 1.3	3.3 ± 0.2	17.0 ± 2.2	3.6 ± 0.2	(a)	80.2 ± 4.5	2.90 ± 0.73
	Run	46.1 ± 2.9	3.6 ± 0.5	8.3 ± 0.4	4.6 ± 0.2	12.6 ± 1.6	(a)	(a)	3.7 ± 0.3	(a)	(a)	(a)
CFCM-7	204	45.5 ± 0.4	3.7 ± 0.1	8.0 ± 0.1	5.0 ± 0.1	13.5 ± 0.5	3.2 ± 0.1	15.0 ± 0.5	3.9 ± 0.2	1.5 ± 0.2	74.8 ± 9.8	3.56 ± 1.58
	Run	44.6 ± 1.0	3.8 ± 0.2	7.9 ± 0.1	4.7 ± 0.2	13.8 ± 0.9	3.1 ± 0.3	15.0 ± 0.5	3.7 ± 0.5	1.7 ± 0.2	(a)	(a)
CFCM-8	113	48.1 ± 1.3	3.1 ± 0.2	8.6 ± 0.3	4.9 ± 0.1	10.1 ± 1.5	3.4 ± 0.1	15.9 ± 0.5	4.2 ± 0.1	1.3 ± 0.2	49.4 ± 28.0	2.36 ± 0.33
	Run	47.6 ± 0.9	3.6 ± 0.5	8.6 ± 0.3	4.8 ± 0.2	11.0 ± 1.4	3.3 ± 0.2	15.4 ± 0.7	4.2 ± 0.4	1.3 ± 0.1	(a)	(a)

(a) Data not reported.

TABLE 58. Summary of SC/CFCM Liquid Feed Analysis, g Oxide/L Feed

Melting Trial		g Oxide/L Feed									Specific Gravity, g/cc
		Total Oxides	SiO_2	Fe_2O_3	Al_2O_3	CaO	Na_2O	MnO_2	NiO	pH	
CFCM-6 Part I	Target	46.0	3.3	24.2	6.1	1.8	0.4	6.4	2.8		
	Ave.	39.6	2.6	18.3	5.7	1.5	0.2	6.0	(a)	7.7	1.02
	Std. Dev.	3.5	1.1	1.9	0.7	0.2	0.2	0.8	(a)	0.1	0.01
Part II	Target	55.0	4.0	28.9	7.3	2.2	0.6	7.6	3.4		
	Ave.	52.0	3.6	23.3	7.8	1.8	0.3	7.5	(a)	7.7	1.03
	Std. Dev.	3.1	0.7	2.2	0.6	0.2	0.1	0.5	(a)	0.1	0.01
Part III	Target	64.0	4.6	33.7	8.4	2.6	0.7	8.9	3.9		
	Ave.	57.4	3.4	27.3	8.4	2.0	1.0	6.9	(a)	7.6	1.02
	Std. Dev.	7.1	1.2	3.4	1.3	0.2	1.2	0.8	(a)	0.1	0.01
CFCM-7	Target	64.9	4.5	34.9	8.5	2.6	1.4	9.1	4.0		
	Ave.	58.7	4.1	33.4	8.4	2.2	2.5	3.1	4.4		
	Std. Dev.	2.5	0.6	1.8	0.6	0.2	0.3	1.2	0.2		
CFCM-8 Part I	Target	74.7	9.6	34.1	8.3	2.3	7.4	9.1	4.0		
	Ave.	65.0	8.2	34.2	7.5	2.2	5.2	2.9	4.3	14	
	Std. Dev.	4.5	1.2	1.2	0.9	0.1	1.6	1.5	0.2		
Part II	Target	65.5	6.5	34.1	8.3	2.3	1.2	9.1	4.0		
	Ave.	52.9	3.3	31.6	7.1	2.0	1.6	3.3	3.9	13	
	Std. Dev.	9.4	1.2	5.9	1.1	0.2	0.2	2.0	0.2		

(a) Not analyzed

did not show the gross discrepancy of MnO_2 results noted in CFCM-7 and CFCM-8. Since no modification of sampling procedure or feed system was made, the cause of this situation is believed to be the use of larger particle-size MnO_2 source in the CFCM-7 and CFCM-8 melting trials.

Short-term variations in liquid feed composition do occasionally occur; however, they are not a normal occurrence and do not account for all variations noted in the final product. Physical properties such as pH and specific gravity have not been sensitive to either changes in overall feed concentration or composition. Their usefulness in process monitoring is questionable.

MELTING PROCESS

Nuclear waste glasses are prepared by subjecting waste-glass formers to elevated temperatures for sufficient time to produce a relatively homogeneous melt. The properties of the resulting waste-glass product are then largely determined by its composition and thermal history upon cooling. Variations in glass product composition were often encountered as a result of waste/frit ratio variations. The SC/ICM and SC/CFCM attempted to maintain a constant waste/frit ratio by metering the frit at a rate determined by the liquid feed rate and the defined feed concentration. In the LFCM, premixed solid or slurry feeds were used. As a result, the composition of the SC/ICM or SC/CFCM product varied more than the LFCM product.

These types of larger compositional differences noted by chemical analysis are not to be confused with glass homogeneity. Glass homogeneity is determined by how well waste constituents are dispersed throughout the glass matrix. Longer melting times, higher melting temperatures and more convection currents within the molten glass all may improve mixing and, therefore, homogeneity. While the SC/ICM product contained a waste-enriched glass on the bottom of each canister, the bulk of the glass appeared at least as homogeneous in photomicrographs as SC/CFCM samples. Both melters were fed from the same feed/calciner system, so the difference in product homogeneity is in some way a matter of melter design and operation. The LFCM with its premixed feeds generally produced a very homogeneous product.

The glass produced by the three processes experienced significantly different thermal histories during cooling. This has a major impact on glass devitrification, the appearance of crystalline phases. The electric melters, LFCM and CFCM, pour a molten glass stream into a canister. All grab samples collected at this point were amorphous with no crystalline phases present. Upon cooling, acmite crystals with a wide range of sizes were formed within the glass. Similarly, the bulk of the ICM glass contained acmite crystals. The impact of this phase on durability has not been established; however, it has led to microcracking in many samples. ICM canisters, in addition, contain a spinel crystal accumulation on the bottom. How much this segregation is due to thermal history is not known. Temperature data for the various canisters is being processed and will be useful in explaining variations in crystals sizes and quantities.

PRODUCT DURABILITY

The chemical durability of a nuclear-waste glass is determined by many factors, including composition, surface area and the presence of secondary phases. Various leach tests are used to measure durability under specific environmental conditions. Leach testing procedures vary considerably and are a source of much debate regarding their representation of actual waste storage conditions. Only the pH4 and soxhlet tests were used to evaluate the durability of melting trial samples. The average results for each canister are listed in Table 57.

The pH4 leach results were considerably more sensitive to glass compositional changes than soxhlet results. Canisters 177 (SC/ICM) and 113 (SC/CFCM) had the lowest pH4 weight losses as well as the lowest waste content. In addition, canister 113 had a large waste content gradient which produced the large pH4 standard deviation. In these cases, soxhlet values were largely unaffected, remaining in the range of 2.05 to 3.56 wt% loss. Although durability is a function of glass composition, the standard deviations of chemical analysis did not correspond to leach data standard deviations. The average standard deviation of pH4 and soxhlet weight loss data were 13.9% and 16.5%, respectively.

In only one canister, 204, unusually large standard deviations were encountered for both pH4 and soxhlet leach results. The composition of the CFCM product was very consistent with only minor variations. While extensive microcracking was present in this canister, a similar condition in canister 172 (Figure 8) did not result in large leach data variations.

CONCLUSIONS AND RECOMMENDATIONS

Three vitrification processes produced over 67,000 kg of simulated defense nuclear waste glass from March 1979 to August 1980. Feed and product samples during this period were collected and analyzed. Operational changes and unexpected events are a normal part of extended melting trials. The analysis of feed and product data during this period was useful in identifying process parameters most signficantly affecting the final product. We make the following conclusions:

LIQUID FEED

Liquid feed samples are necessary to determine the impact of mixing and feed system operation on the waste composition. The sampling procedure must, however, collect a representative sample to be most useful. During extended melting trials, the normal variation of constituents may be as high as 10% of the constituent concentration. Feed samples every 250 kg of glass production would provide adequate information for process monitoring. Impurities may also be unknowingly associated with some of the waste components used in feed preparation.

GLASS PRODUCT

The composition of the final product largely determines its physical and chemical properties. The product composition is determined by the waste composition and the ability of the process to maintain a constant waste/frit ratio. Experience at PNL shows that the waste content of the glass during a melting trial is within ± 3.6% of the defined 28% waste oxide content. All trials together were within ± 6% of the target. Those processes requiring the simultaneous control of liquid waste and frit feed rates, SC/ICM and SC/CFCM, had the largest composition variations. Glass compositions tolerating this variation without property changes would produce the most consistent product.

Crystals are a common feature of the defense waste glass cooled in canisters. The complex interaction of glass composition and cooling rate must be understood to anticipate the appearance of crystalline phases and their impact on product properties. Acmite crystals frequently found in SC/ICM and SC/CFCM product resulted occasionally in considerably glass microcracking. The cause of the spinel (waste) accumulation in the bottom of SC/ICM canisters has not been positively identified.

Waste oxides can be reduced in the waste glass melt and settle out as metallic buttons. While organics in the "modified TDS" composition were effective in reducing metals in the SC/ICM process, additional work is necessary to determine the tendency for this to occur in the ceramic melters.

The chemical durability of glass samples under pH4 and soxhlet conditions does not appear related to the particular process or its stable operation. The SC/ICM, LFCM and SC/CFCM all produced glass of comparable durability. Foaming in the ceramic melters did not appear to affect the product durability. Lowering waste content of the defense waste glass increased pH4 durability but did not affect soxhlet durability. The average variation in leach test data as a percentage of the mean leach test results was considerable, 14.0% ± 17.8% for pH4 and 17.7% ± 12.8% for the soxhlet test. Since this shows a little relationship to corresponding composition variation, care must always be exercised in product evaluation on the basis of leach test results.

RECOMMENDATIONS

We make these recommendations for future equipment melting trials:

- Feed and product sampling should be an integral part of every melting trial with data analysis based on computer control charge programs to deal with inherent process variations.
- Sampling frequency can be reduced to two samples per feed makeup or every 250 kg of glass produced. The sampling procedure should be improved to make each sample more representative.
- The sampling of ceramic melter-produced glass from the pour spout every 250 kg of production is adequate for chemical analysis and leach testing.

- Crystallization studies need to be carried out on glasses in a range of at least ± 6 wt% of waste from the defined composition to anticipate devitrification effects.
- The impact of organic compounds on glass properties and metal reduction from the melt should be examined.
- Methods to eliminate the enrichment of waste in the bottom of SC/ICM canisters should be investigated.

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