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DEFENSE WASTE PROCESSING FACILITY (DWPF) STARTUP TEST PROGRAM: GLASS CHARACTERIZATION (U)

by

Carol M. Jantzen
Westinghouse Savannah River Co.
Savannah River Laboratory
Aiken, SC 29808

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ABSTRACT

Liquid high-level nuclear waste will be immobilized at the Savannah River Site (SRS) by vitrification in borosilicate glass. The glass will be processed in the Defense Waste Processing Facility (DWPF) and poured into stainless steel canisters for eventual geologic disposal. Six simulated glass compositions will be processed in the DWPF during initial startup. The glass in 86 of the first 106 full sized canisters will be sampled and characterized. Extensive glass characterization will determine the following: (1) sampling frequency for radioactive operation, (2) verification of the compositionally dependent process-product models, (3) verification of melter mixing, (4) representativeness of the glass from the canister throat sampler, and (5) homogeneity of the canister glass.

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INTRODUCTION

Liquid high-level nuclear waste will be immobilized at the Savannah River Site (SRS) by vitrification into borosilicate glass. The production process to be used in the Defense Waste Processing Facility (DWPF) is designed to reliably produce durable borosilicate nuclear waste glasses. A systems approach to borosilicate glass formulation was used to simultaneously evaluate product performance and processing considerations.[1-2] Parameters affecting the product performance, such as chemical durability, were optimized relative to processing considerations.[1,2] Models were developed which relate process parameters, such as viscosity,[3-4] resistivity,[4] and liquidus[5] to parameters that could easily be measured during glass vitrification, such as glass composition and melt temperature. The process/product models relating to glass viscosity, liquidus, and durability form the basis for the DWPF process control strategy, the Product Composition Control System (PCCS).[6]

As part of the DWPF process control strategy, the final glass composition will be determined by dissolution and chemical analysis of a sample of slurry from the Slurry Mix Evaporator, (SME) that will be vitrified in a crucible. The SME product will, therefore, meet the process/product constraints before the batch of feed in the SME is sent to the Melter Feed Tank (MFT) and subsequently to the DWPF melter. The glass composition will be verified by analysis of vitrified slurry from the MFT.

The glass melt from the DWPF will be poured into stainless steel canisters for disposal in a geologic repository. As part of the product control strategy, the canistered borosilicate waste glass must comply with the Waste Acceptance Preliminary Specifications (WAPS) established by the DOE Office of Civilian Radioactive Waste Management.[7] WAPS specification 1.3 relates to the control of radionuclide release from the final wasteform. Experimental and theoretical evidence has demonstrated that the radionuclide release of the DWPF product can be controlled by controlling the chemical composition of the glass.[8-19] The reactivity of the DWPF glasses has been shown to be affected very little by variations in melter residence time, the size of the melter, or the crystalline content of the glass.[15-22]

Current plans for the DWPF analytical facility require elemental analyses to be performed on the vitrified melter feed from the MFT. This analysis will be used to calculate a release rate for the glass from the DWPF product quality (hydration thermodynamic) model in order to comply with WAPS 1.3.

The process/product models will be validated during the DWPF Qualification Campaigns with simulated waste glass. Eighty six of the 106 canisters produced will be sampled. The glass samples will be used to verify glass viscosity, liquidus, and durability predicted from the process/product models. The product quality model for glass durability will be verified by performing the Product Consistency Test (PCT)[23-30] on the canister glass and also on glass samples taken from a special sampler in the throat of the canister which captures part of the liquid glass stream.[7] The glass durability model will continue to be validated throughout the lifetime of the DWPF by performing the PCT on radioactive production glass samples.

The current study documents the types of experiments and analyses which are planned for the DWPF Qualification runs when 86 of the 106 full scale canisters are sampled. The details of the proposed startup program are given on the following slides:

SLIDE	**DESCRIPTION**
1	Title slide.
2	Objectives slide which explains that the primary objective of the glass characterization for DWPF startup is to provide the statistical basis for sampling frequency for radioactive operation. There are five subparts to achieving this objective which include (1) verification of the DWPF process/product models, (2) verification of the DWPF melter mixing behavior as a Continuously Stirred Tank Reactor (CSTR),[31-32] (3) verification of glass sampler "sample" as representative of the glass in the canister, (4) examination of the homogeneity of the glass in the canister in terms of chemical composition and redox, and (5) determination of the crystal content in the canister, if any.
3	The full scale prototypic DWPF canister will be made our of 304L stainless steel, it will contain ~2600 lbs. of glass when full to ~91", and will be about 10 foot tall and 2 foot in diameter.

4 During DWPF Qualification Runs two different canister sampling techniques will be used. Some canisters will be sectioned at 2-foot intervals and the glass sampled radially from each section. Other canisters will have a 12″ wide section of the wall removed over the entire length of the canister so that the glass can be mechanically sampled.

5 During DWPF Qualification Campaigns every canister will be fitted with a specially designed throat protector which has a steel sampling device that can be inserted into the melt stream to sample the glass being poured.[7] This picture shows the glass sampler filled with glass ~ 50 grams of glass that has solidified.

6 All 106 canisters produced during DWPF Qualification Runs will be sampled with the glass sampler. Thirty canisters will be sectioned at 2 foot intervals and 56 canisters will have the walls removed. The distribution of canisters sectioned versus those cut open is given over the 5 DWPF Qualification Campaigns, e.g. initial conditions campaign, doped average glass campaign, low glass viscosity campaign, high glass viscosity campaign, and a return to the baseline glass.

7 The analytic techniques that will be used during the DWPF Qualification Runs are the following:

- the durability test developed at SRS for DWPF and designated the Product Consistency Test (PCT)[23-30]
- glass dissolution and analysis to determine whole element chemistry of the glass on an oxide basis
- glass redox ($Fe^{2+}/\Sigma Fe$)
- quantitative x-ray diffraction
- quantitative magnetic susceptibility
- scanning electron microscopy

8 The criteria for the development of the PCT[23,25,27] durability test for DWPF compliance with the WAPS chemical durability specification 1.3 are as follows:

- Sensitivity to glass composition and homogeneity

- simple sample preparation/procedure for remote radioactive operation

- short enough test duration for use during production

- acceptance by waste form developers and the repository.

9 The PCT was developed over a 6-year period and it is still undergoing revision for acceptance as a national consensus by American Standards and Testing Materials (ASTM) organization. The initial version of the PCT was drafted in November, 1986. An internal round robin was held between three researchers at SRS.[23] An external round robin was held between 1988 and 1989 and included 8 different laboratories.[24] Minor revisions were incorporated into the PCT in July, 1988 and February, 1989 as a result of findings of the external round robin. Version 3.0 was submitted to ASTM in January, 1990.[29] At this time it was decided to divide the procedure into a part A and a part B. Revisions 4.0 and 5.0 which included parts A and B were submitted to ASTM in July, 1991 and again, with minor revisions, in January, 1992.[30]

10 A schematic of the PCT procedure shows that it is a 7-day test which uses crushed glass in ASTM Type I water at a constant ratio of volume of solution to mass of glass powder. The test is performed at 90°C, and the average glass size is 0.11 mm (100-200 mesh). It is a static test, the leachates are filtered and analyzed for pH and elemental release concentrations. A standard glass is used as a control each time the test is run.

11 Returning to the objectives, we will now discuss one objective at a time in more detail. In this section of the talk we will discuss how the DWPF process/product models will be verified during the DWPF Qualification Campaigns.

12 The analytic procedures used to meet the objective called out in slide 11 will be the PCT, the whole element chemistry of the glass, and the redox of the glass.

13 This slide represents a schematic of the DWPF process

and demonstrates that vitrified melter feed will be analyzed for process/product control for all elemental species and redox after the SME but before the MFT and again after the MFT but before the melter for waste compliance reporting. Likewise, the sampler glass and the canister glass will be analyzed during DWPF Qualification Campaigns in order to determine how representative the vitrified SME and MFT samples are of the glass in the sampler and in the canister. Comparisons of the chemistry and PCT durability of the glass in the sampler and the canister will be made as well. The durability results will also be compared to the predictions made using the hydration thermodynamic product model.

14 Based on the results from all the analyses discussed in slides 12 and 13, DWPF will be able to demonstrate the following:

- the glass chemistry including the glass redox in the canister is predictable from the SME and MFT analyses
- the glass durability in the canister has been accurately predicted from the glass composition measured at the SME and MFT by the hydration thermodynamic product model

15 The next objective to be discussed in detail is that relating to the verification of the DWPF melter mixing behavior as a Continuously Stirred Tank Reactor (CSTR).

16 The analytic procedures to be used to meet the objective called out in slide 15 will be the whole element chemistry of the glass, and the redox of the glass.

17 Previous studies with scale glass melters at SRS that have been doped with Cs have shown a gradual ingrowth of Cs in the final glass as a function of the number of melter volume turnovers (see open circles).[31] This ingrowth follows curvature that indicates model stirred tank reactor behavior as indicated by the solid circles.

18 Similar stirred tank reactor behavior has been studied in SRS scale glass melters when a reducing feed was feed into the melter following an oxidized melt campaign.[33] The change in redox during the Scale Glass Melter Campaign #7, as measured in canisters 1 through 8 followed stirred tank reactor behavior as indicated by the solid line.

19 The next two objectives to be discussed in detail are the verification of the glass sampler "sample" as representative of the canister contents and examination of glass chemical homogeneity in the canister.

20 The analytic procedures to be used to meet the two objectives called out in slide 19 will be the PCT, the whole element chemistry of the glass, and the redox of the glass.

21 Based on the results from all the analyses discussed in slide 20, DWPF will be able to demonstrate the following:

- the glass chemistry, redox, and durability measured on the sampler glass is representative of the glass throughout the canister

- the glass within a canister is chemically homogeneous and, therefore, gives a homogeneous response during the PCT durability testing.

22 Previous studies[33] of chemical homogeneity of glasses poured into full size DWPF canisters during the Scale Glass Melter Campaigns, indicated that the glasses produced were chemically homogeneous.

23 The last objective to be discussed in detail is the determination of crystal content in the canisters.

24 The analytic procedures to be used to meet the objective called out in slide 23 will be the quantitative x-ray diffraction, the quantitative magnetic susceptibility, and scanning electron microscopy.

25 This is an x-ray diffraction spectra showing how phases in crystallized glass can be identified. Silicon is added as an internal standard and the area under the major silicon peak is ratioed to the area under the major spinel, acmite, etc. peak in order to develop quantitative calibration curves. Quantitative calibration curves for each of the four crystallized species indicated in the spectra (spinel, acmite, nepheline and lithium silicate) have already been developed and the equipment is standardized. Since spinel is magnetic, and magnetic susceptibility is more

sensitive to small amounts of spinel but less sensitive to spinel composition variation than x-ray diffraction, quantitative magnetic susceptibility curves have also been developed. Scanning electron microscopy is used to identify the chemical composition and morphology of the crystallizing species and to confirm the phase identification determined by x-ray diffraction.

26 In conclusion, preparations have been made for glass characterization during DWPF Qualification Campaigns. This through glass characterization will provide the statistical basis for determining the sampling frequency during radioactive operation.

REFERENCES

1. C.M. Jantzen, "Systems Approach to Nuclear Waste Glass Development," J. Non-Crystalline Solids, 84, 215-225 (1986).

2. D.F. Bickford, A.A. Ramsey, C.M. Jantzen, and K.G. Brown, "Control of Radioactive Waste Glass Melters:I.Preliminary General Limits at Savannah River, J. Am. Ceram.Soc., 73 [10] 2896-2902(1990).

3. C.M. Jantzen, "The Relationship Between Glass Viscosity and Composition: A First Principles Model for Vitrification of Nuclear Waste," US DOE Report WSRC-MS-90-41, Westinghouse Savannah River Co., Aiken, SC (April, 1991).

4. C.M. Jantzen, "Process Control Models and Limits for DWPF Glass, Part I. Viscosity, Resistivity, Composition Relations," (in prep.)

5. C.M. Jantzen, "Process Control Models and Limits for DWPF Glass, Part II. Liquidus-Composition Relations (in prep.)

6. K.G. Brown, R.L. Postles, and R.E. Edwards, "The Product Composition Control System (PCCS)," Nuclear Waste Management IV, G.G. Wicks, D.F. Bickford, and L.R. Bunnell (Eds.), Ceramic Transactions, Vol. 23, American Ceramic Society, Westerville, OH, p.559-568 (1991).

7. Waste Form Compliance Plan for the Defense Waste Processing Facility," USDOE Report WSRC-SW4-6, Rev.0, Westinghouse Savannah River Co., Aiken, SC (March, 1990).

8. M.J. Plodinec, C.M. Jantzen, and G.G. Wicks, "Thermodynamic Approach to Prediction of the Stability of Proposed Radwaste Glasses," Advances in Ceramics, 8, 491-495 (1984).

9. C.M. Jantzen and M.J. Plodinec, "Thermodynamic Model of Natural, Medieval, and Nuclear Waste Glass Durability," J. Non-Cryst. Solids, 67, 207-233 (1984).

10. M.J. Plodinec, C.M. Jantzen, and G.G. Wicks, "Stability of Radioactive Waste Glasses Assessed from Hydration Thermodynamics," Sci. Basis for Nuclear Waste Management, VII, G.L. McVay (Ed). Elsevier Publ., New York 755-762 (1984).

11. C.M. Jantzen, "Prediction of Nuclear Waste Glass Durability from Natural Analogs" Advances in Ceramics, 20, D.E. Clark, W.B. White and A.J. Machiels (Eds.), American Ceramic Society, Westerville, OH, 703-712 (1986).

12. C.M. Jantzen, "Prediction of Glass Durability as a Function of Environmental Conditions", Proceedings of the Symposium on Materials Stability and Environmental Degredation," A. Barkatt et. al. (Eds.), Materials Research Society, Pittsburgh, PA, 143-159 (1988).

13. C.M. Jantzen, "Prediction of Glass Durability as a Function of Glass Composition and Test Conditions: Thermodynamics and Kinetics", Proceedings of the First Intl. Conference on Advances in the Fusion of Glass, American Ceramic Society, Westerville, OH , p. 24.1-24.17 (1988).

14. C.M. Jantzen, "Thermodynamic Approach to Glass Corrosion," Corrosion of Glass, Ceramics, and Ceramic Superconductors, p. 153-215, Noyes Publications, Park Ridge, NJ (1992).

15. C.M. Jantzen, D.F. Bickford and D.G. Karraker, "Time Temperature-Transformation Kinetics in SRL Waste Glass," Advances in Ceramics, V. 8, G. G. Wicks and W. A. Ross (Eds.), 30-38 (1984).

16. D.F. Bickford and C.M. Jantzen,"Devitrification of SRL Defense Waste Glass", Sci.Basis for Nuclear Waste Management, VII, G.L. McVay (Ed). Elsevier Publ., New York 557-565 (1984).

17. C.M. Jantzen and D.F. Bickford, "Leaching of Devitrified Glass Containing Simulated SRP Nuclear Waste," Sci. Basis for Nuclear Waste Management, VIII, C.M. Jantzen, J.A. Stone and R.C. Ewing (Eds.), Materials Research Society, Pittsburgh, PA, 135-146 (1985).

18. D. F. Bickford and C. M. Jantzen, "Devitrification of Defense Nuclear Waste Glasses: Role of Melt Insolubles", J.Non-Cryst.Solids, 84, 299-307 (1984).

19. M.J. Plodinec, G.G. Wicks, N.E. Bibler, "An Assessment of Savannah River Borosilicate Glass in the Repository Environment," USDOE Report DP-1629, E.I. duPont deNemours & Co., Savannah River Laboratory, Aiken, SC (1982).

20. C.M. Jantzen and N.E. Bibler, "The Role of Groundwater Oxidation Potential and Radiolysis on Waste Glass Performance in Crystalline Repository Environments," Sci. Basis for Nuclear Waste Management,IX, L.O. Werme (Ed.), Materials Research Society, Pittsburgh, PA 219-229 (1986).

21. N.E. Bibler, "Leaching Fully Radioactive SRP Nuclear Waste Glass in Tuff Groundwater in Stainless Steel Vessels," Advances in Ceramics, 20, D.E. Clark, W.B. White and A.J. Machiels (Eds.), American Ceramic Society, Westerville, OH, 703-712 (1986).

22. N.E. Bibler and C.M. Jantzen., "Materials Interactions Relating to Long-Term Geologic Disposal of Nuclear Waste Glass," Sci. Basis for Nuclear Waste Management, X, J.K. Bates and W. Seefeldt (Eds.), Materials Research Society, Pittsburgh PA, 47-66 (1987).

23. C. M. Jantzen and N. E. Bibler, "Product Consistency Test (PCT) and Test Protocol," USDOE Report DPST-87-575, Savannah River Laboratory, Aiken, SC (1987).

24. G. F. Piepel, T. E. Jones, D. L. Eggett, G. B. Mellinger, "Product Consistency Test Round Robin Conducted by the Materials Characterization Center - Summary Report," USDOE Report PNL-6967, Materials Characterization Center, Battelle Pacific Northwest Laboratories, Richland, WA (1989).

25. C.M. Jantzen and N.E. Bibler. "The Product Consistency Test for the DWPF Wasteform," Requirements for Waste Acceptance and Quality Control, Proceedings of the 2nd International Seminar on Radioactive Waste Products, E. Warnecke, R. Odoj, and R. Simon (Eds.) Kerforschunganlanger (KFA) Research Centre, Julich, Federal Republic of Germany, 610-622 (1990).

26. N. E. Bibler and J. K. Bates, "Product Consistency Leach Tests of Savannah River Site Radioactive Waste Glasses," Scientific Basis for Nuclear Waste Management, XIII, V. M. Oversby and P. W. Brown (Eds.), Materials Research Society, Pittsburgh, PA, 327-338 (1990).

27. N.E. Bibler and C.M. Jantzen, "The Product Consistency Test and Its Role in the Waste Acceptance Process," Waste Management 89 Proceedings, I., 743-749 (1989).

28. C.M. Jantzen, "Evaluation of Experimental Factors That Influence the Application and Discrimination Capability of the Product Consistency Test ," U.S. DOE Report WSRC-TR-90-526 (1990).

29. C.M. Jantzen and N.E. Bibler, "Nuclear Waste Glass Product Consistency Test (PCT)-Version 3.0," USDOE Report WSRC-TR-90-539, Rev. 1, Westinghouse Savannah River Co., Aiken, SC (November, 1990).

30. C.M. Jantzen, N.E. Bibler, D.C. Beam, W.G. Ramsey, and B.J. Waters "Nuclear Waste Product Consistency Test (PCT) - Version 5.0," WSRC-TR-90-539, Rev. 2 (January, 1992).

31. M.J. Plodinec, "Rate of Change of Glass Composition in DWPF Melters,", USDOE Report DPST-85-944, E.I. duPont deNemours & Co., Aiken, SC (December, 1985).

32. P.V. Danchwerts, Chemical Engineering Science, 2, 1-13 (1953).

33. W.G. Ramsey and C.M. Jantzen, Redox ANalyses of SRS Melter Feed Slurry and Simulated Waste Glass, USDOE Report WSRC-MS-90-001, Westinghouse Savannah River Co., Aiken, SC (February, 1990).

DEFENSE WASTE PROCESSING FACILITY (DWPF) STARTUP TEST PROGRAM: GLASS CHARACTERIZATION

Carol M. Jantzen

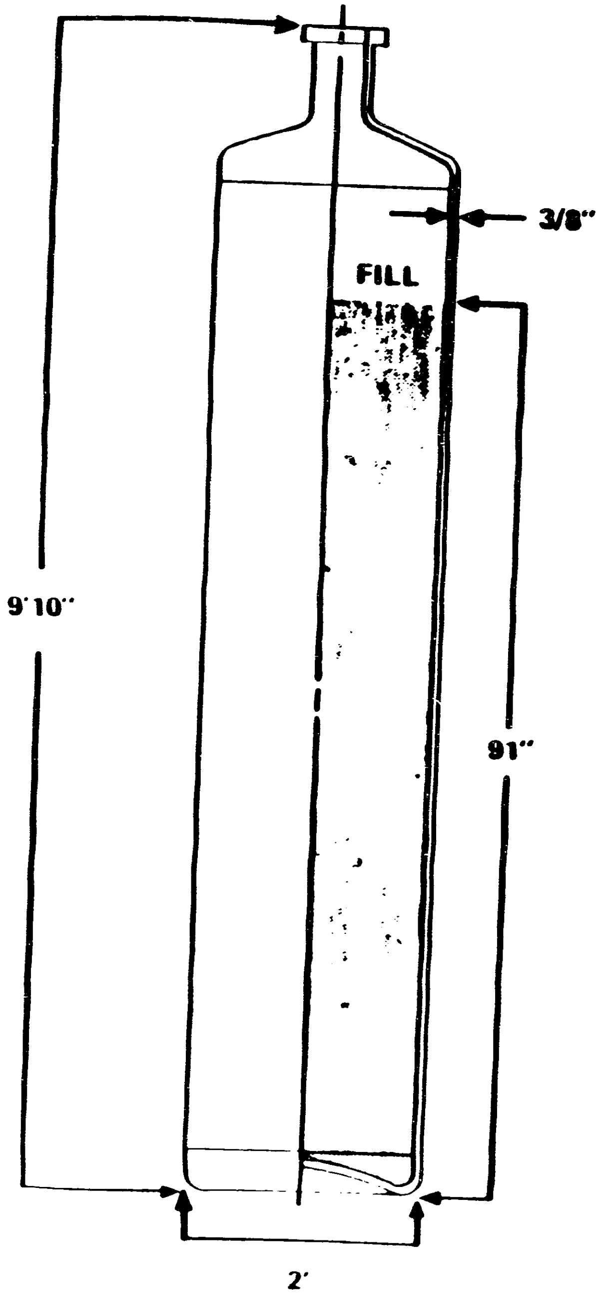
Westinghouse Savannah River Company
Savannah River Laboratory
Aiken, SC 29808

SAFETY • RESPONSIBILITY • SECURITY
SRS
SAVANNAH RIVER SITE

OBJECTIVES

o Provide statistical basis for sampling frequency for radioactive operation

- Verification of DWPF process/product models
- Verification of melter behavior as a Continuously Stirred Tank Reactor (CSTR)
- Verification of glass sampler "sample" as representative of canister contents
- Examination of homogeneity of canister contents
- Determination of crystal content in canister



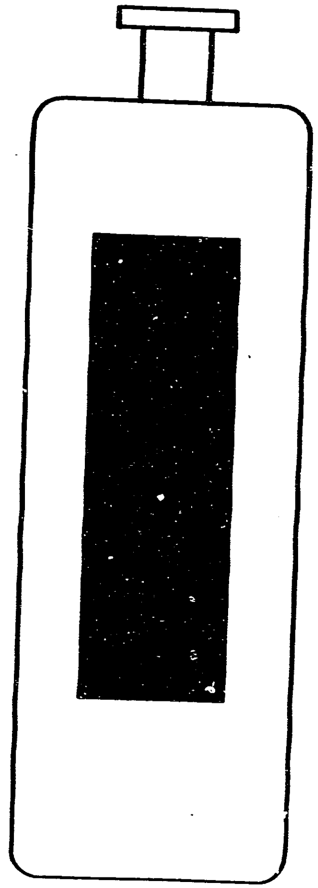
FILLED CANISTER

MATERIAL:	304L STAINLESS STEEL
EMPTY WEIGHT:	1100 LB
NET WEIGHT:	3710 LB
WEIGHT OF RADIONUCLIDES:	94 LB
ACTIVITY:	234,000 Ci
DECAY HEAT:	692 W
RADIATION FIELD (AT SURFACE):	5,600 rad/hr
SURFACE CONTAMINATION:	LESS THAN 10^{-4} $\mu Ci/cm^2$

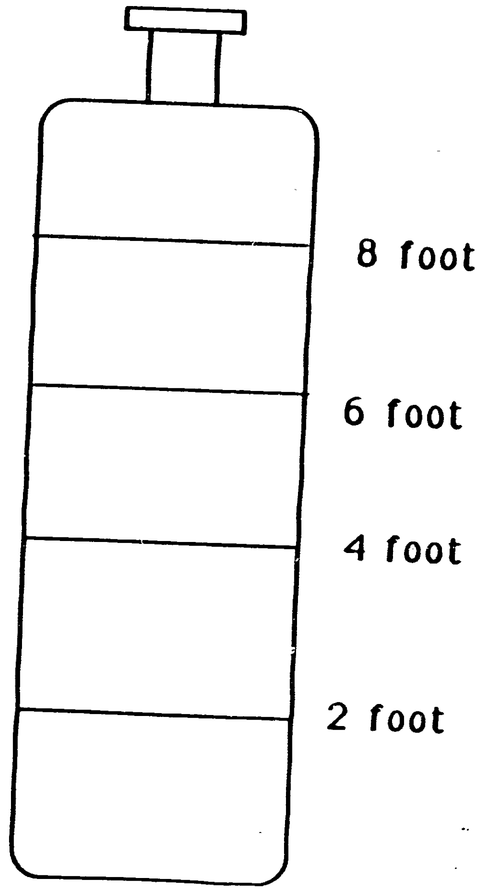
Slide 3

CANISTER SAMPLING TECHNIQUES

CANISTERS WITH WALLS REMOVED



CANISTERS SECTIONED



DWPF Glass Sampler

CANISTER SAMPLING PLAN

DWPF "COLD CHEMICAL RUNS"	CANISTERS SECTIONED	CANISTERS WITH WALLS REMOVED	GLASS SAMPLER
INITIAL CONDITIONS	3	3	24
DOPED RUN	6	14	20
LOW VISCOSITY	7	13	21
HIGH VISCOSITY	7	13	21
BLEND 1 FEED WITH Hg (return to baseline)	7	13	20
	30	56	106

Total canisters opened = 86

Total canisters sampled = 106

ANALYTIC TECHNIQUES

- Product Consistency Test (PCT)
- Whole element chemistry
- Redox
- X-ray Diffraction
- Magnetic Susceptibility
- Scanning Electron Microscopy

CRITERIA FOR TEST DEVELOPMENT

- Sensitivity to glass composition and glass homogeneity
- Simple sample preparation/procedure for remote operation
- Short enough test duration for use during production
- Acceptance by waste form developers and the repository

DEVELOPMENT OF THE PCT

- Initial draft Version 1.0 (November, 1986)
- SRS round robin Version 2.0 (July, 1987)
- External round robin (April, 1988-September, 1989)
- Minor revisions Version 2.1 and 2.2 (July, 1988) and February, 1989)
- Submitted to ASTM Version 3.0 (January, 1990)
- Submitted to ASTM Version 4.0 (July, 1991)
- Submitted to ASTM Version 5.0 (January, 1992)

SCHEMATIC OF THE PRODUCT CONSISTENCY TEST

7 day

90 C

Filter

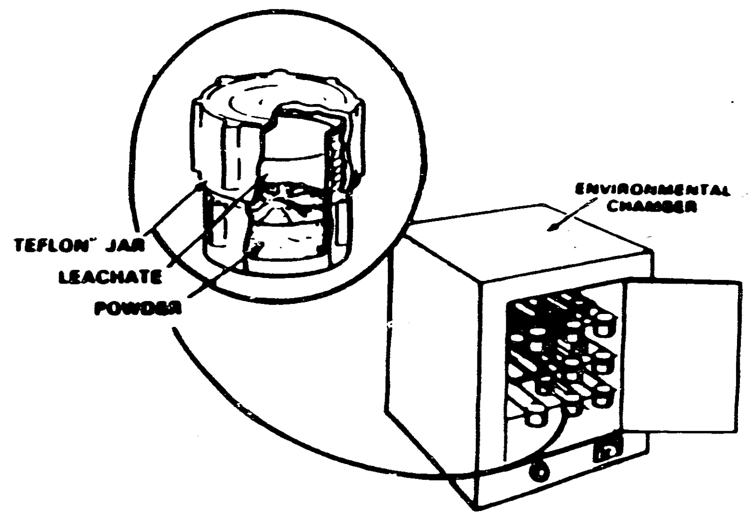
0.11 mm

mL soln/g solid = 10

static

analyze pH and concentration

standard glass as control



OBJECTIVES

- o Provide *statistical* basis for sampling frequency for radioactive operation

  - Verification of DWPF process/product models

  - Verification of melter behavior as a Continuously Stirred Tank Reactor (CSTR)

  - Verification of glass sampler "sample" as representative of canister contents

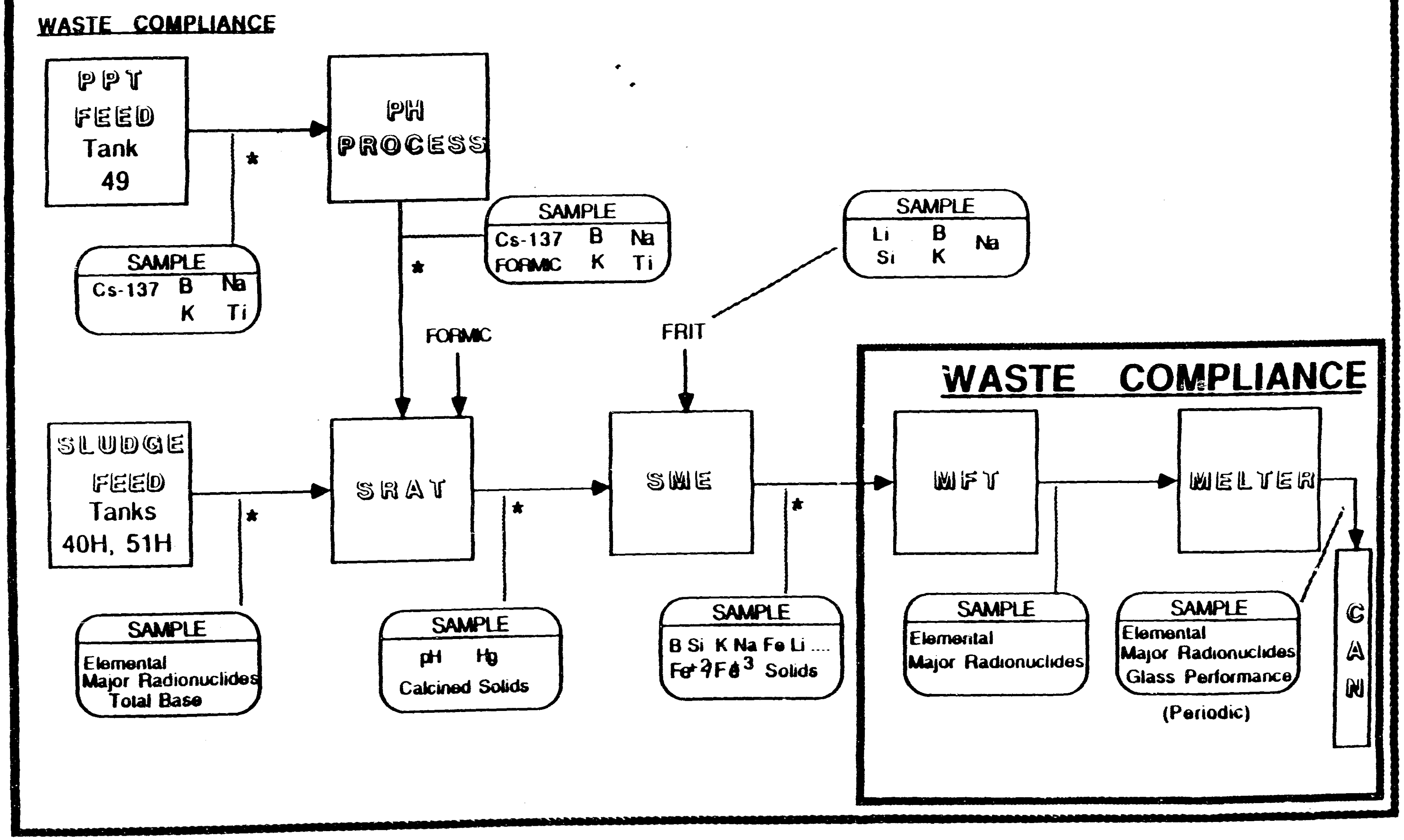
  - Examination of homogeneity of canister contents

  - Determination of crystal content in canister

ANALYTIC TECHNIQUES

- o Product Consistency Test (PCT)
- o Whole element chemistry
- o Redox
- o X-ray Diffraction
- o Magnetic Susceptibility
- o Scanning Electron Microscopy

PROCESS CONTROL



* Process Control Point Prior to Making Batch Transfer

RESULTS

- o Predictability of glass chemistry in the canister
- o Predictability of glass redox in the canister
- o Predictability of glass durability in the canister (calculated from glass composition and measured using the PCT)
- o Comparison of the chemistry, redox, and durability of the canister glass to that of the sampler glass

OBJECTIVES

- Provide statistical basis for sampling frequency for radioactive operation
  - Verification of DWPF process/product models
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ANALYTIC TECHNIQUES

- Product Consistency Test (PCT)
- Whole element chemistry
- Redox
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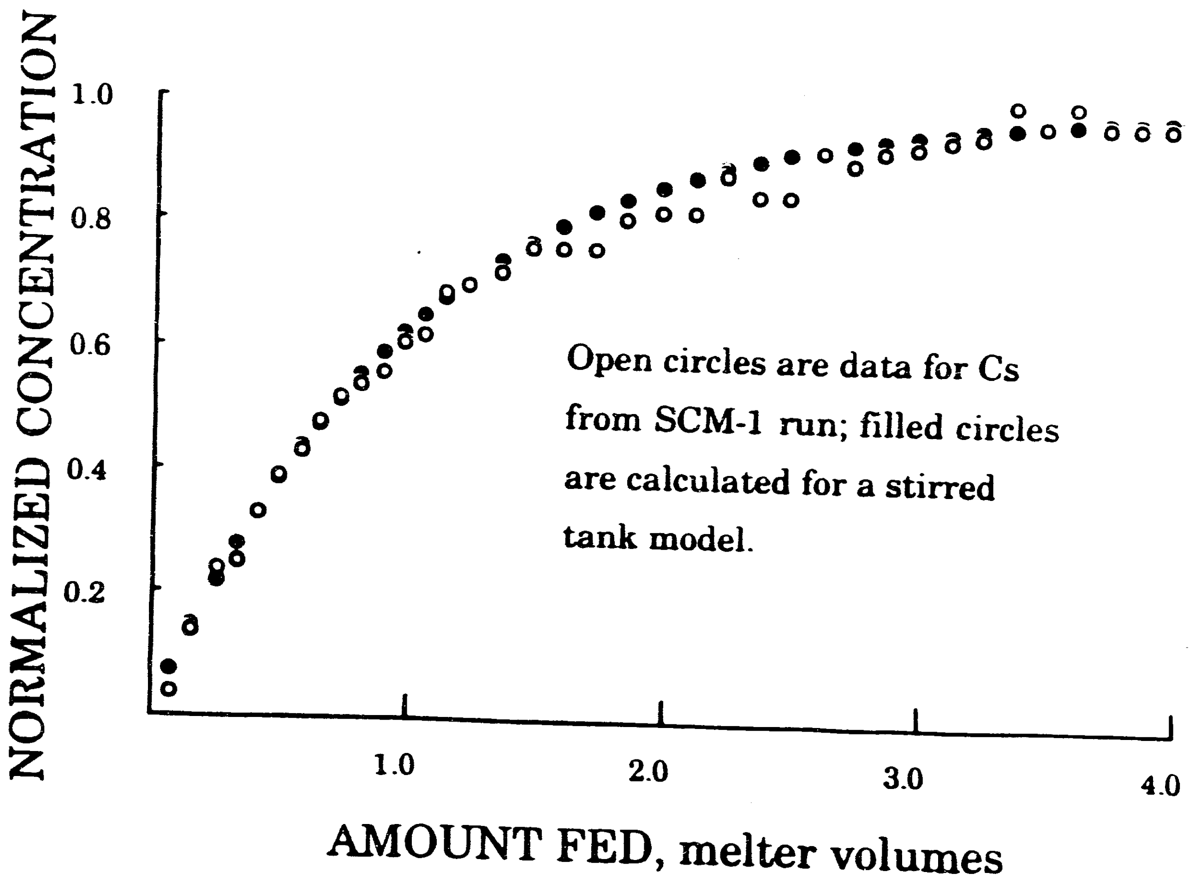
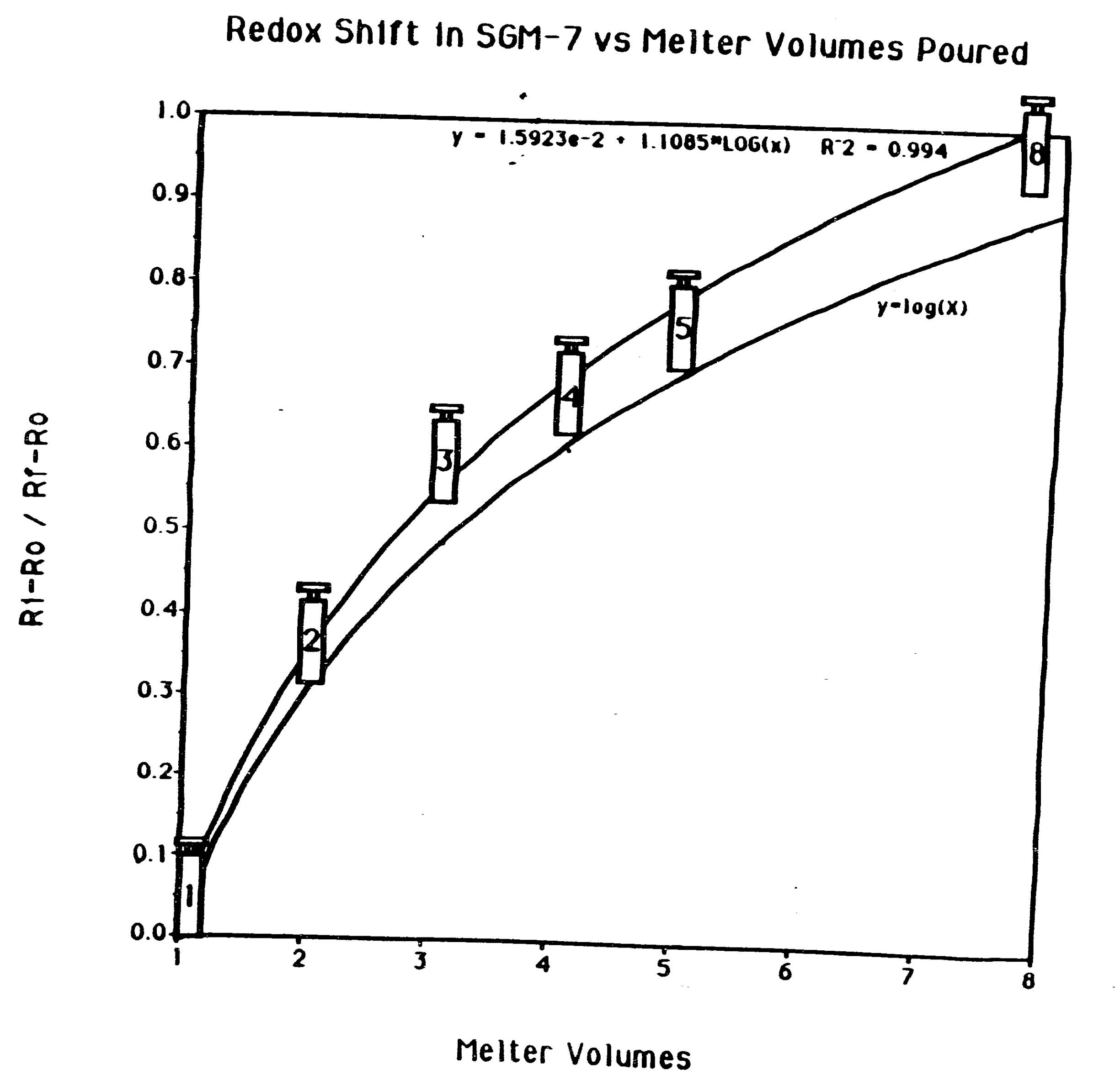
- Magnetic Susceptibility
- Scanning Electron Microscopy

CHANGE IN GLASS COMPOSITION WITH CHANGE IN FEED





Redox Shift in SGM-7 vs Melter Volumes Poured
y = 1.5923e-2 + 1.1085*LOG(x) R^2 = 0.994
y=log(X)
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
1
2
3
4
5
6
7
8
Rl-Ro / Rf-Ro
Melter Volumes

Slide 18

OBJECTIVES

- o Provide statistical basis for sampling frequency for radioactive operation
  - Verification of DWPF process/product models
  - Verification of melter behavior as a Continuously Stirred Tank Reactor (CSTR)
  - Verification of glass sampler "sample" as representative of canister contents
  - Examination of homogeneity of canister contents
  - Determination of crystal content in canister

ANALYTIC TECHNIQUES

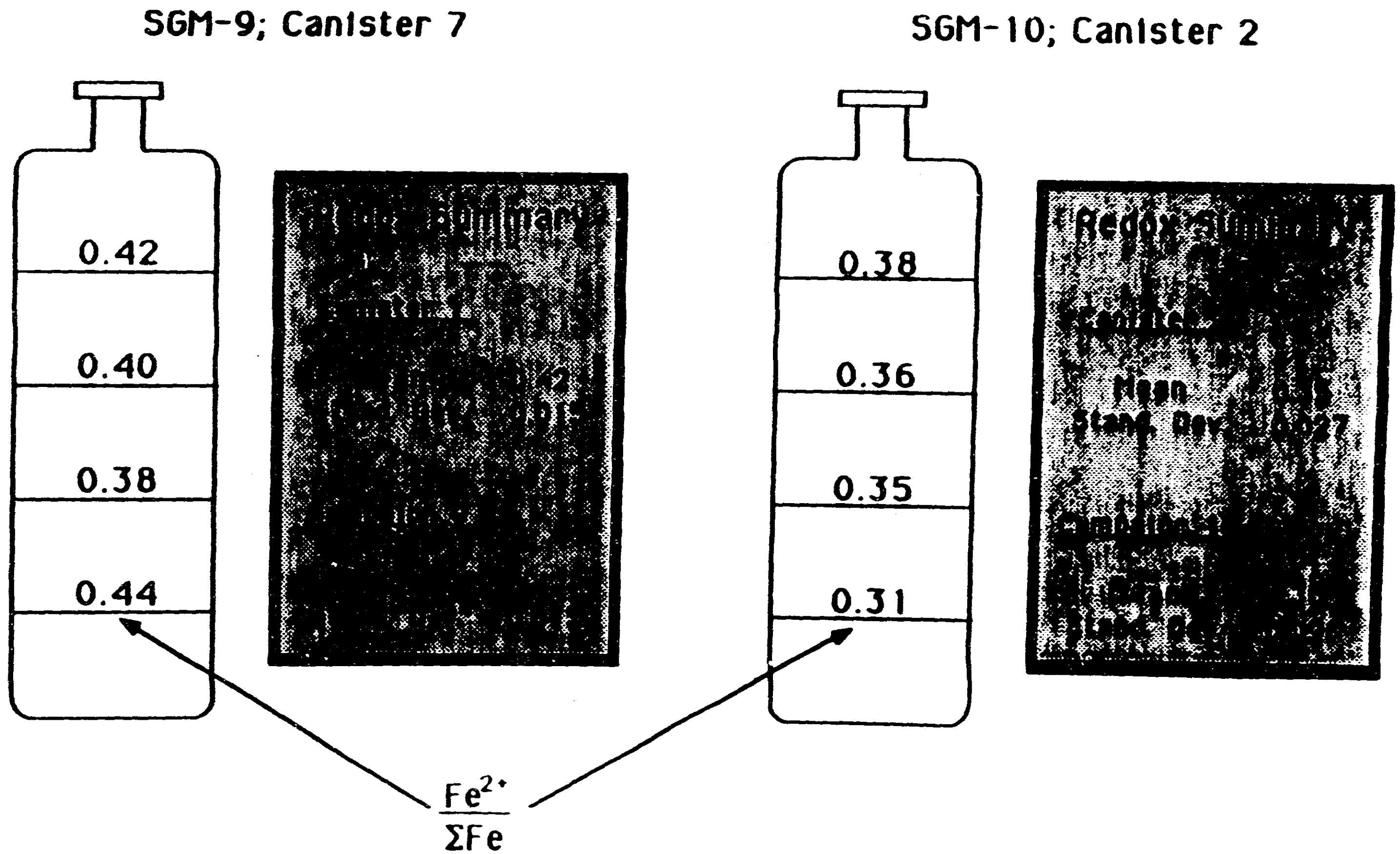
- o Product Consistency Test (PCT)
- o Whole element chemistry
- o Redox
- o X-ray Diffraction
- o Magnetic Susceptibility
- o Scanning Electron Microscopy

RESULTS

- Compare measured composition of vitrified melter feed to measured composition of canister and sampler glass

- Compare durability predicted from glass composition to Product Consistency Test results for canister and sampler glass

In-Can Iron Redox Variation During Scale Glass Melter Campaigns 9 and 10



OBJECTIVES

o Provide statistical basis for sampling frequency for radioactive operation

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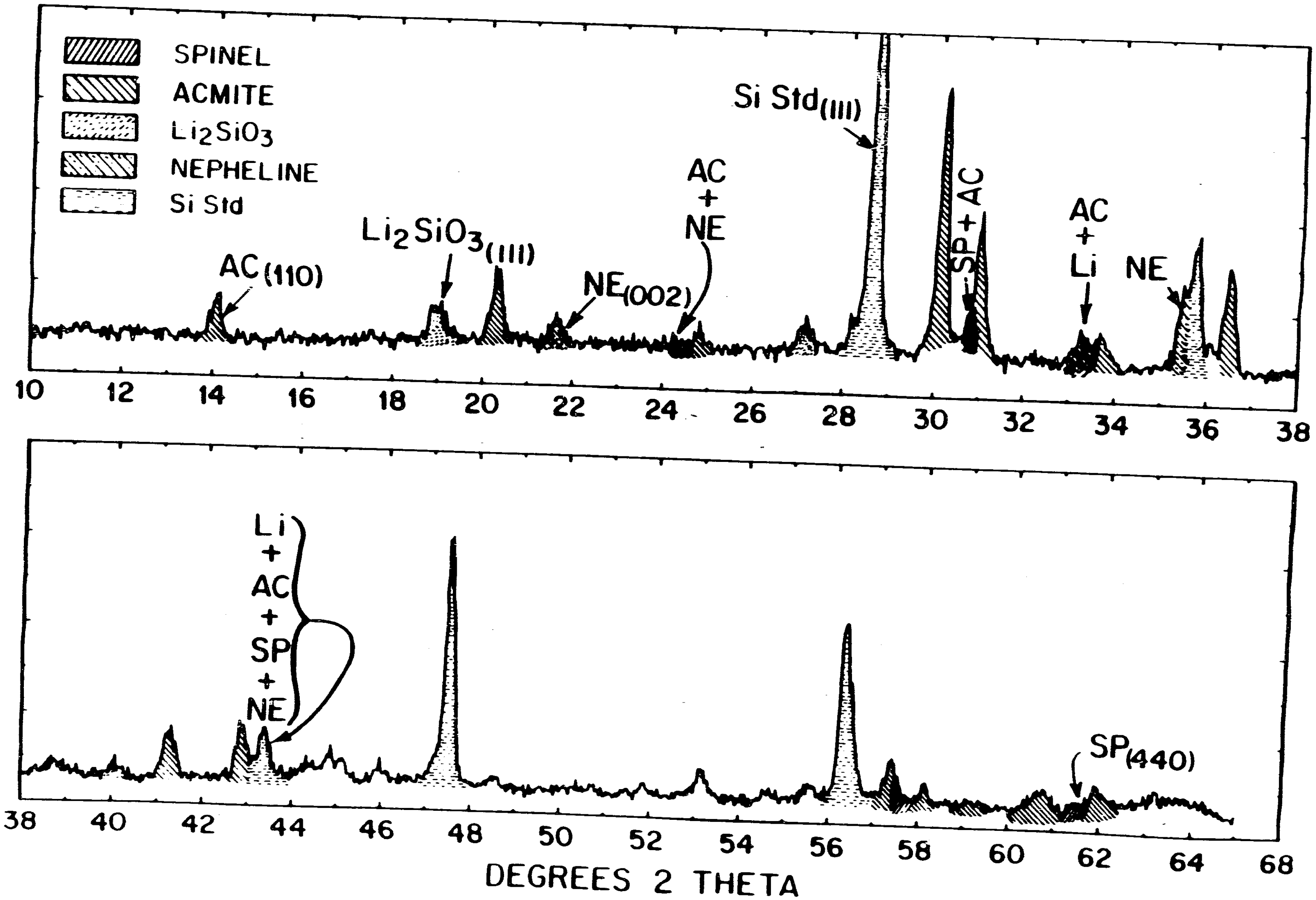
ANALYTIC TECHNIQUES

- o Product Consistency Test (PCT)
- o Whole element chemistry
- o Redox
- o X-ray Diffraction
- o Magnetic Susceptibility
- o Scanning Electron Microscopy

X-RAY DIFFRACTION SPECTRA OF A DEVITRIFIED GLASS



CONCLUSIONS

- Preparations have been made *for glass* characterization during qualification runs
- Extensive *glass* characterization will provide the *statistical basis for* determining the sampling frequency during *radioactive* operation

DATE
FILMED
8/04/92