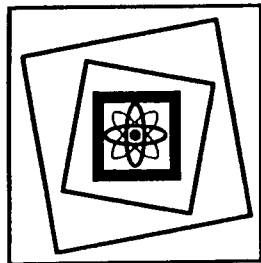
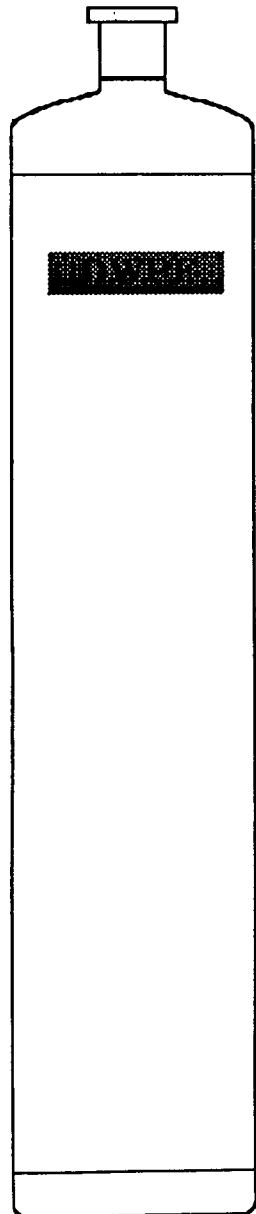
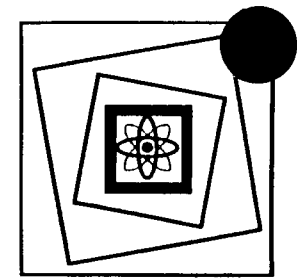


DWPF
Product Qualification
Programs



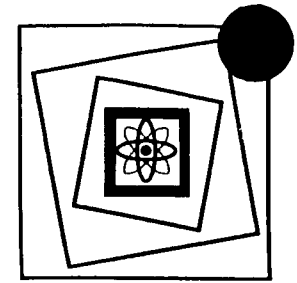
M. J. Plodinec
February, 1992

DWPF Product



Materials of construction:	Austenitic stainless steel (304L)
Canister weight (empty):	450 kg
Weight of glass:	1700 kg (3700 lb)
Length:	300 cm (9 ft 10 in)
Diameter:	61 cm (24 in)
Wall thickness:	1.0 cm
Average fill height:	231 cm
Maximum activity:	230 kCi
Maximum radiation dose:	5500 R/hr
Maximum decay heat:	730 watts
Average glass density:	2.73 g/cm ³
Average time of filling:	17 hr
Mode of filling:	Continuous, differential pressure method

Background

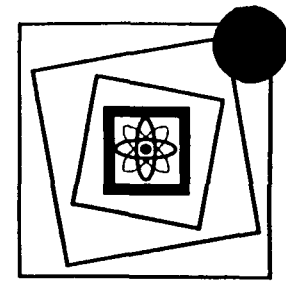


- Glass selected as waste form for DWPF - 1982
 - Glass processing is well-known and safe
 - Glass is stable to radiation
 - Glass is durable, and rugged
 - EPA: "Facility badly needed ... environmentally beneficial action"
 - NRC: "Glass could be an acceptable waste form in a suitably engineered barrier system;" urged site-specific testing
 - Subsequent work showing that EBS containing DWPF glass can meet regulatory requirements*
- DOE-OCRWM has set specifications for acceptance of the DWPF product
- DWPF blending information from laboratory, hot cells, pilot plant and facility to demonstrate compliance with specifications
 - Qualification of technology which pre-dates specifications
 - Demonstration of product controls

*See N. E. Bibler, G. G. Wicks and V. O. Oversby, "Leaching of SRP Nuclear Waste Glass in a Saturated Tuff Environment," **Scientific Basis for Nuclear Waste Management, VIII**, C. M. Jantzen, J. A. Stone and R. C. Ewing (eds.), 247-56 (1985); and

M. J. Apted and D. W. Engel, "Mass-Transfer Analysis of Waste Packages Containing Defense Waste Processing Facility Glass as a Waste Form," **High-Level Radioactive Waste Management**, Proceedings of the International Topical Meeting, D. B. Shipler (ed.), 388-93 (1990).

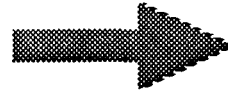
Waste Acceptance Process



DOE - OCRWM

SPECIFICATIONS

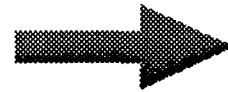
ASSURE COMPATIBILITY
WITH REPOSITORY



DWPF

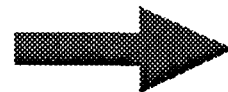
**WASTE FORM
COMPLIANCE PLAN**

- HOW WILL WE COMPLY?



**WASTE FORM
QUALIFICATION
REPORT**

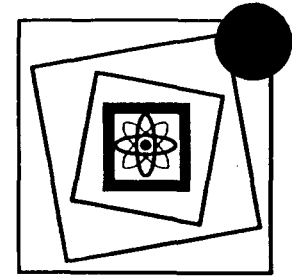
- CAN WE COMPLY, IN DWPF?



**PRODUCTION
RECORDS**

- HAVE WE COMPLIED, IN DWPF?

Status of DWPF Product Qualification Activities



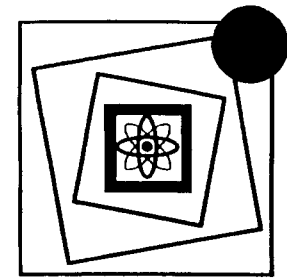
- WAPS revised 6/91
 - Product Consistency Test
 - Improved clarity

- Waste Form Compliance Plan revised - currently undergoing review by Technical Review Group

- Waste Form Qualification Report
 - Glass Product Control Program re-drafted
 - Revised WAPS and WCP require revision of initial sections

- Disciplined Startup Test Program in place
 - Qualify technology developed before Waste Acceptance Process
 - Demonstrate ability to control product
 - Program driven by product consistency and foreign materials specifications

Waste Acceptance Preliminary Specifications



- Cover three technical areas, and Quality Assurance
- Latest revision - 6/91

Glass

Chemical Composition
Radionuclide Inventory
Product Consistency
Phase Stability

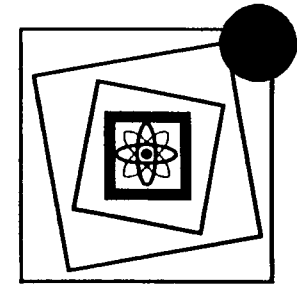
Canister

Material
Fabrication and Closure
Identification and Labeling

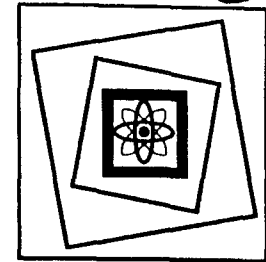
Finished Product

Free Liquid
Gas
Explosives, Pyrophorics,
Combustibles
Organic Materials
Fill Height
Surface Contamination
Heat Generation
Dose Rate
Chemical Compatibility
Subcriticality
Weight, Length, Diameter,
Overall Dimensions
Drop Test
Handling Features

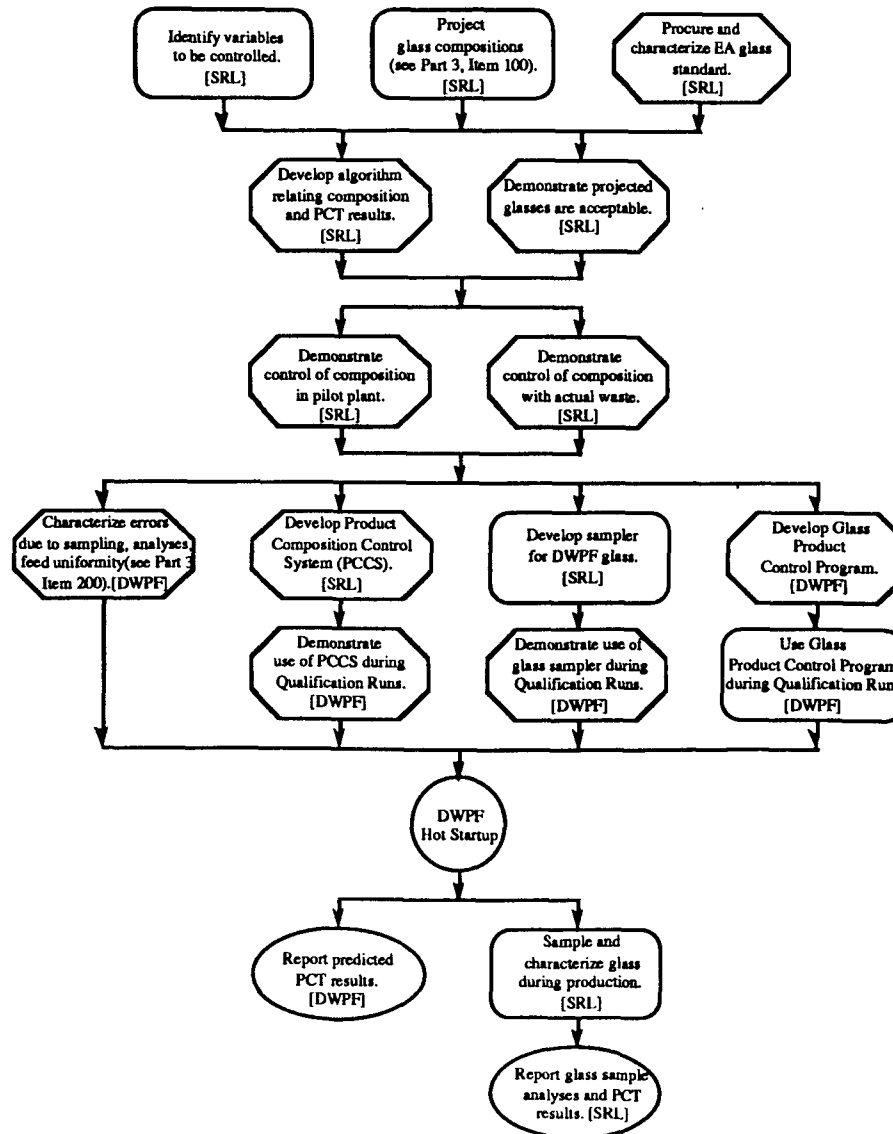
Waste Form Compliance Plan



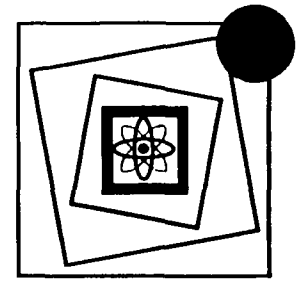
- Management plan for product acceptance programs
- Defines methodology for determining what is important to meet each specification
- Describes programs and activities for product qualification, production control, and reporting, for each specification
- Describes role of Startup Test Program
- Key technical document for understanding application of RW-0214 to DWPF
 - Details what we are trying to achieve
 - Describes how we will achieve it
 - Used to identify items needing controls



WCP Logic Diagram Example

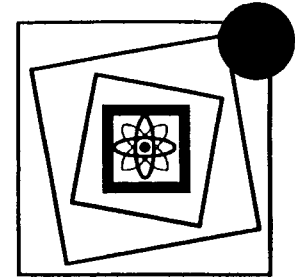


Waste Form Qualification Report

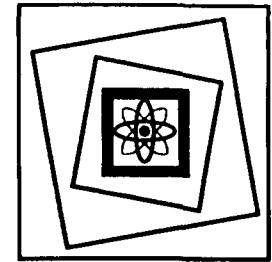


- WQR in volumes for resource leveling, and to facilitate review
- Projected length: ≥ 3000 pages
- Contents of each volume:
 - Specification
 - Compliance strategy summary
 - Description of item or process
 - Description of item or process controls
 - Demonstration(s) of effectiveness of controls (qualification testing)
 - Production Records (or Shipping and Storage Records) content

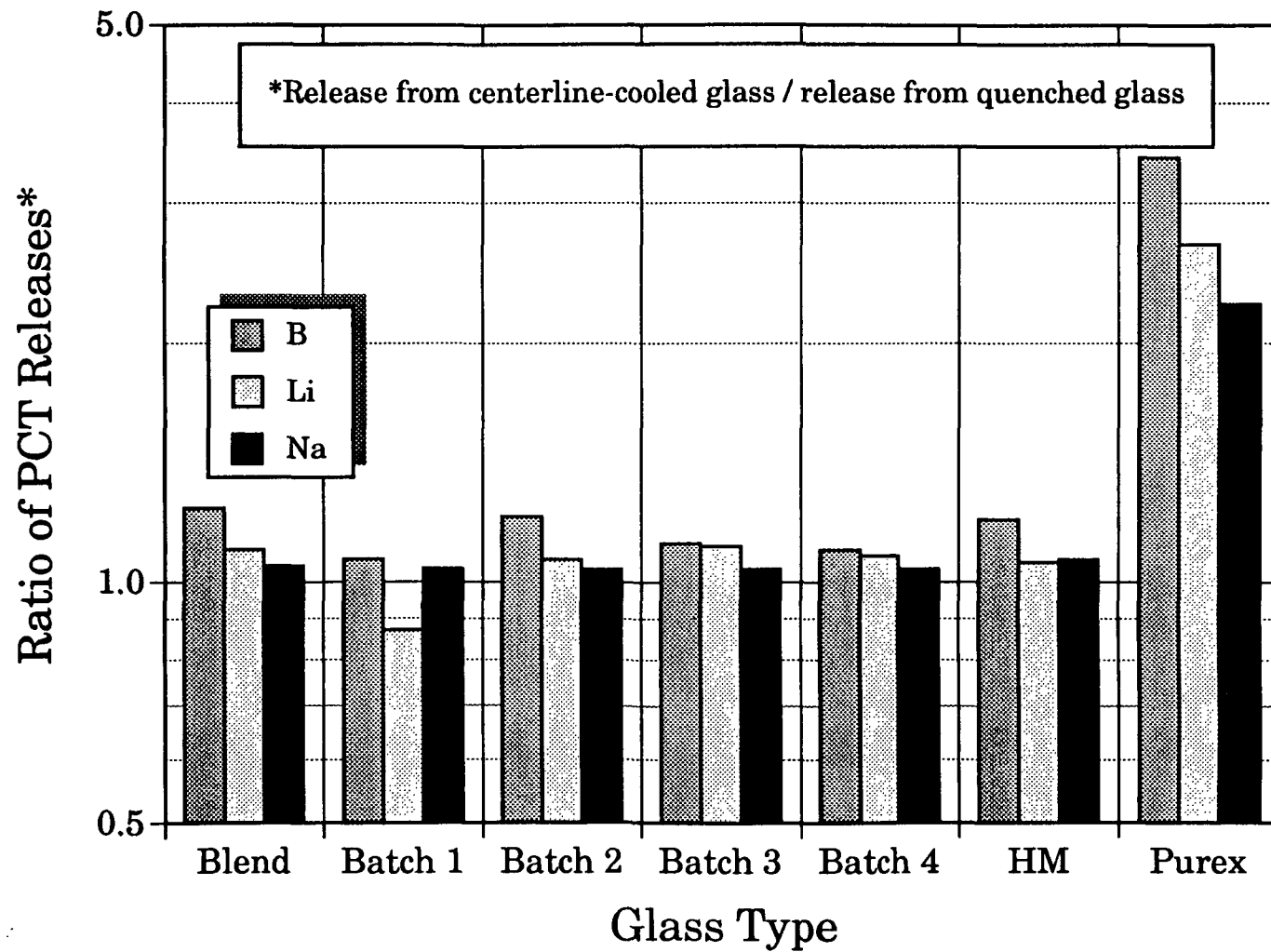
1. Chemical Composition Projections



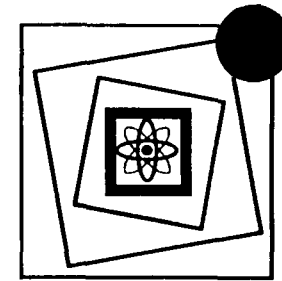
- Specification:
 - Provide expected chemical composition and microstructure for all DWPF glasses
- Progress:
 - Defined reference waste types, and glass composition for each waste type
 - Procured several pounds of each from Corning Glass
 - Determined expected canister cooling curves
 - Characterization of glasses almost complete; only quantitative XRD remaining



Effects of Centerline Cooling



Projected DWPF Waste Glass Compositions



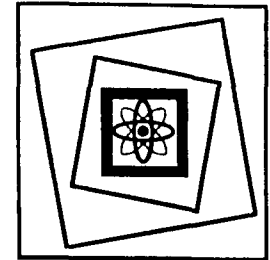
MAJOR GLASS COMPONENTS weight %	CONSTITUENT SLUDGE TYPE						
	Blend ^d	Batch 1	Batch 2	Batch 3	Batch 4	HM	Purex ^w
Al ₂ O ₃	3.98	4.87	4.46	3.25	3.32	7.08	2.89
B ₂ O ₃	8.01	7.69	7.70	7.69	8.11	6.94	10.21
BaSO ₄	0.27	0.22	0.24	0.26	0.38	0.18	0.29
CaO	0.97	1.17	1.00	0.93	0.83	1.00	1.02
CaSO ₄	0.077	0.12	0.11	0.10	0.0034	trace	0.12
Cr ₂ O ₃	0.12	0.10	0.12	0.13	0.14	0.086	0.14
CuO	0.44	0.40	0.41	0.40	0.46	0.25	0.42
Fe ₂ O ₃	10.41	12.52	10.61	11.16	11.32	7.38	12.74
Group A ^a	0.14	0.099	0.14	0.10	0.20	0.20	0.078
Group B ^b	0.36	0.22	0.44	0.25	0.60	0.89	0.084
K ₂ O	3.86	3.49	3.50	3.47	3.99	2.14	3.58
Li ₂ O	4.40	4.42	4.42	4.42	4.32	4.62	3.12
MgO	1.35	1.36	1.35	1.35	1.38	1.45	1.33
MnO	2.03	2.06	1.62	1.81	3.08	2.07	1.99
Na ₂ O	8.73	8.62	8.61	8.51	8.88	8.17	12.14
Na ₂ SO ₄	0.10	0.10	0.12	0.096	0.13	0.14	0.12
NaCl	0.19	0.31	0.23	0.22	0.090	0.093	0.26
NiO	0.89	0.75	0.90	1.07	1.09	0.40	1.21
SiO ₂	50.20	49.81	50.17	49.98	49.29	54.39	44.56
ThO ₂	0.19	0.36	0.63	0.77	0.24	0.55	0.011
TiO ₂	0.90	0.66	0.67	0.66	1.02	0.55	0.65
U ₃ O ₈	2.14	0.53	2.30	3.16	0.79	1.01	2.89
Total	99.76	99.88	99.75	99.79	99.66	99.59	99.85

^a Group A: semi-volatile radionuclides (e.g., Se, Te, Rb, Mo).

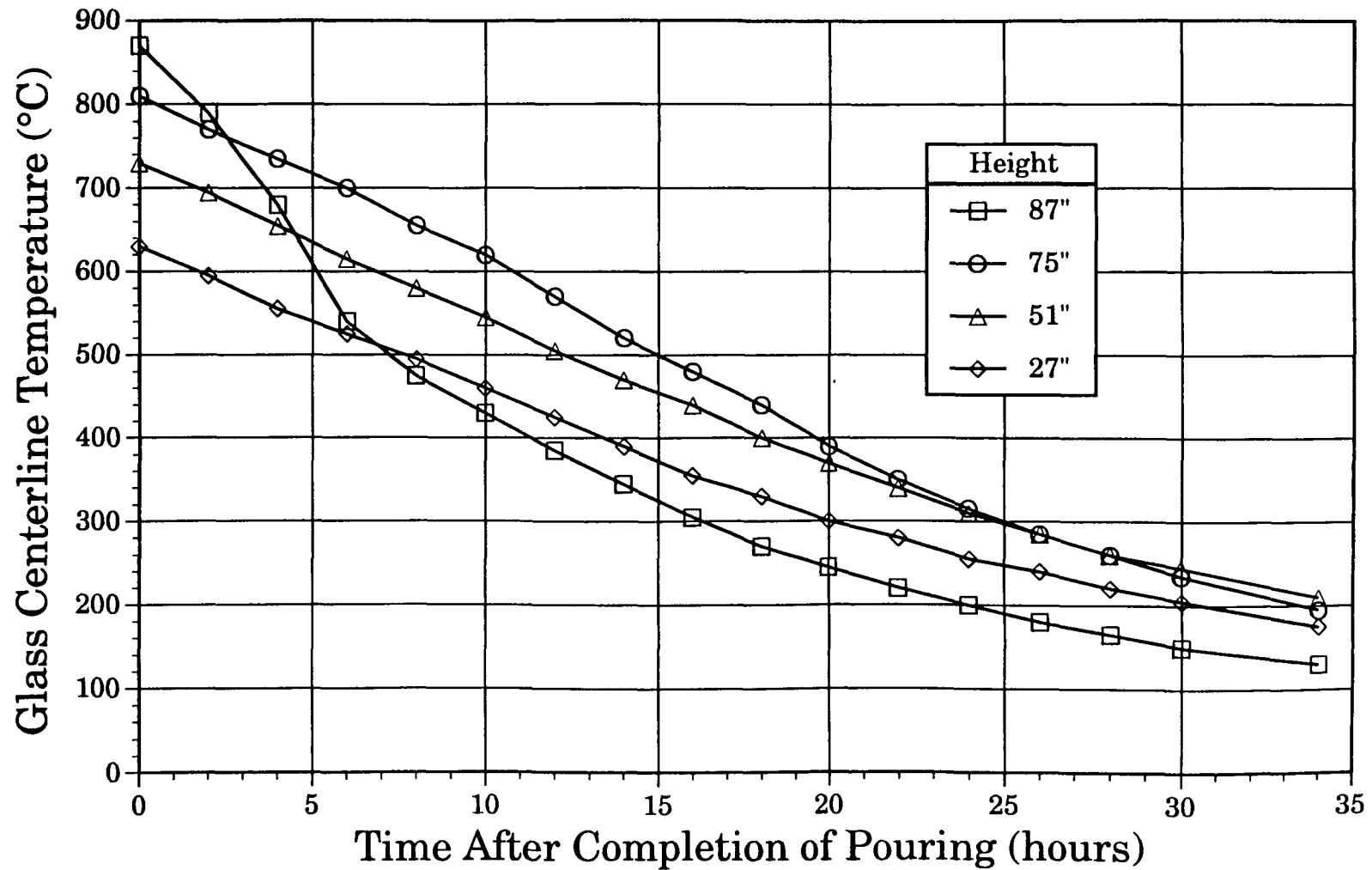
^b Group B: nonvolatile radionuclides (e.g., Sm, Sn, Co, Np, Am, Cm).

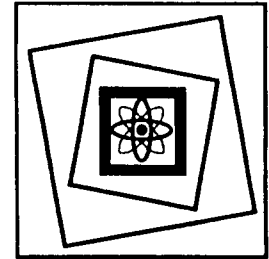
^d The "Blend" is the current DWPF design-basis glass.

^w The "Purex" glass is a possible "worst-case" composition.

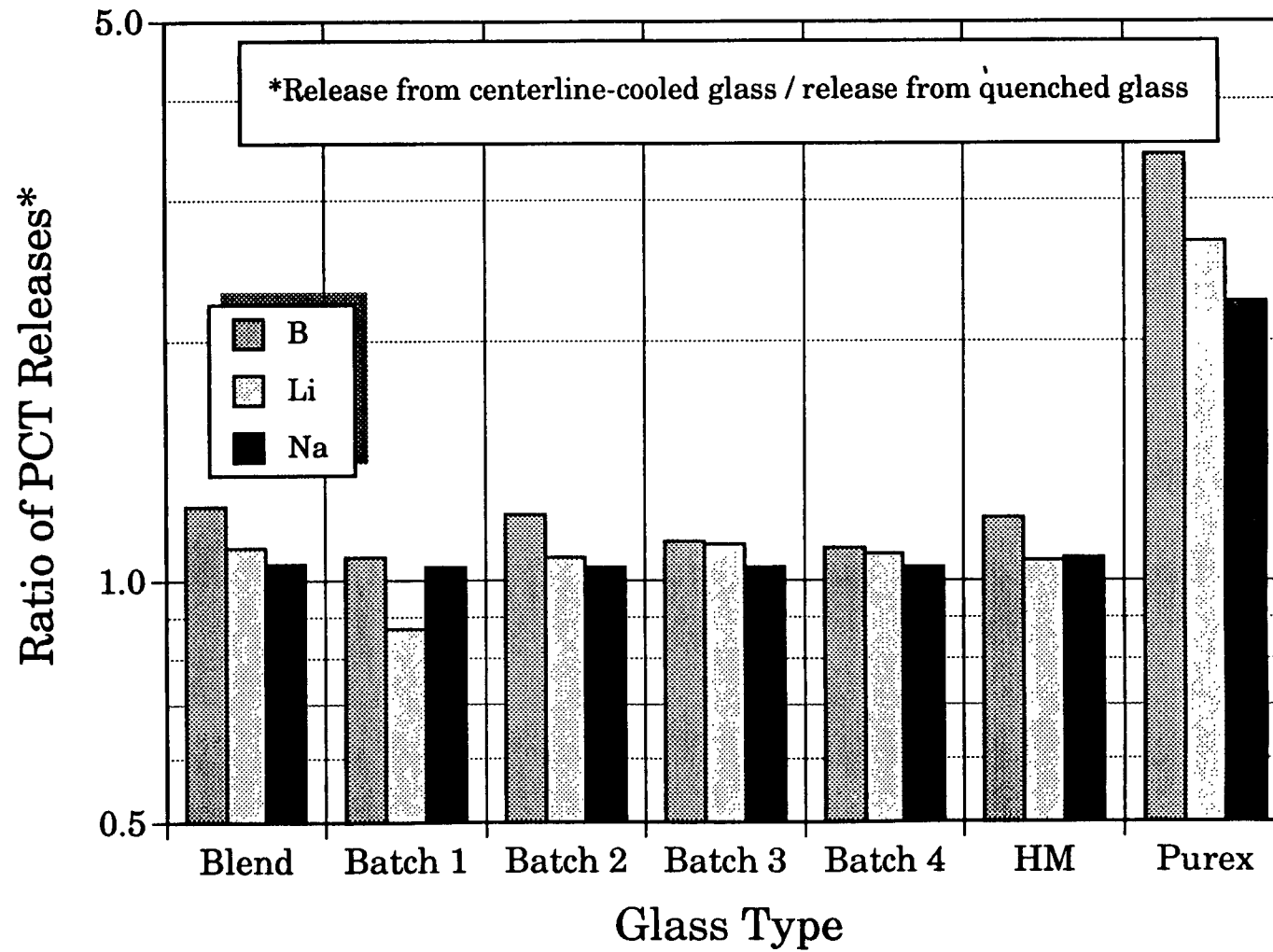


Cooling of DWPF Canisters After Filling

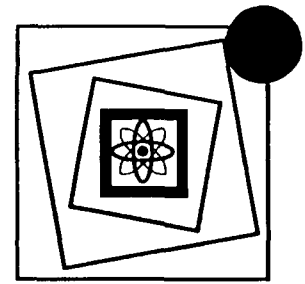




Effects of Centerline Cooling

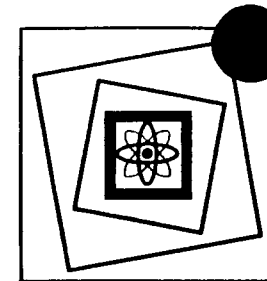


2. Chemical Composition During Production



- Specification:
 - Report all elements > 0.5 wt % (excluding O) for each waste type
 - Report precision and accuracy of values in WQR
- Progress:
 - Developed strategy based on use of slurry samples – incorporated in Product Control Program
 - Will report on basis of “macro-batch” (~ 4 months production)
 - Slurry sampler developed, characterized
 - Analytical methods developed, tested in round robins (typically 1 -3 % RSD), being incorporated in DWPF Laboratory Manual
 - Use of standard glass important to achieving good results
 - Glass sampler developed
 - Will quantify “as-built” uncertainties as part of Startup Test Program

Effectiveness of DWPF Analytical Methods



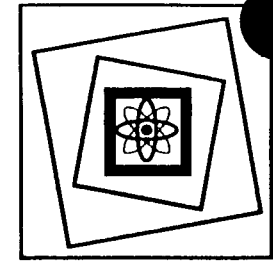
<u>Component^a</u>	<u>Reference^b</u> <u>Wt%</u>	<u>DWPF Method^c</u> <u>Wt%</u>	<u>%RSD^d</u>
SiO ₂	47.9	47.5	1.8
Fe ₂ O ₃	14.0	14.0	0.6
Na ₂ O	11.5	11.5	1.1
B ₂ O ₃	8.67	8.57	3.4
Li ₂ O	3.21	3.24	3.2
Al ₂ O ₃	4.73	4.62	0.59
K ₂ O	2.71	2.71	1.2
MnO ₂	2.31	2.37	0.71
CaO	1.43	1.43	0.70
MgO	0.86	0.88	0.54
TiO ₂	1.15	1.23	0.38
NiO	1.05	1.11	0.43

^a Components included are species which are required to be reported for any of the projected compositions. Reference Glass did not contain uranium.

^b Average of 36 high-precision analyses performed by Corning Engineering Laboratory Services.

^c Average of six determinations by SRL, using DWPF methods.

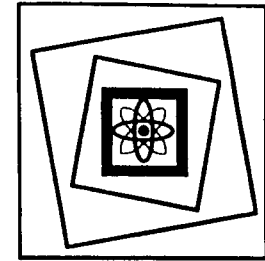
^d Percent Relative Standard Deviation, based on use of the appropriate DWPF method.



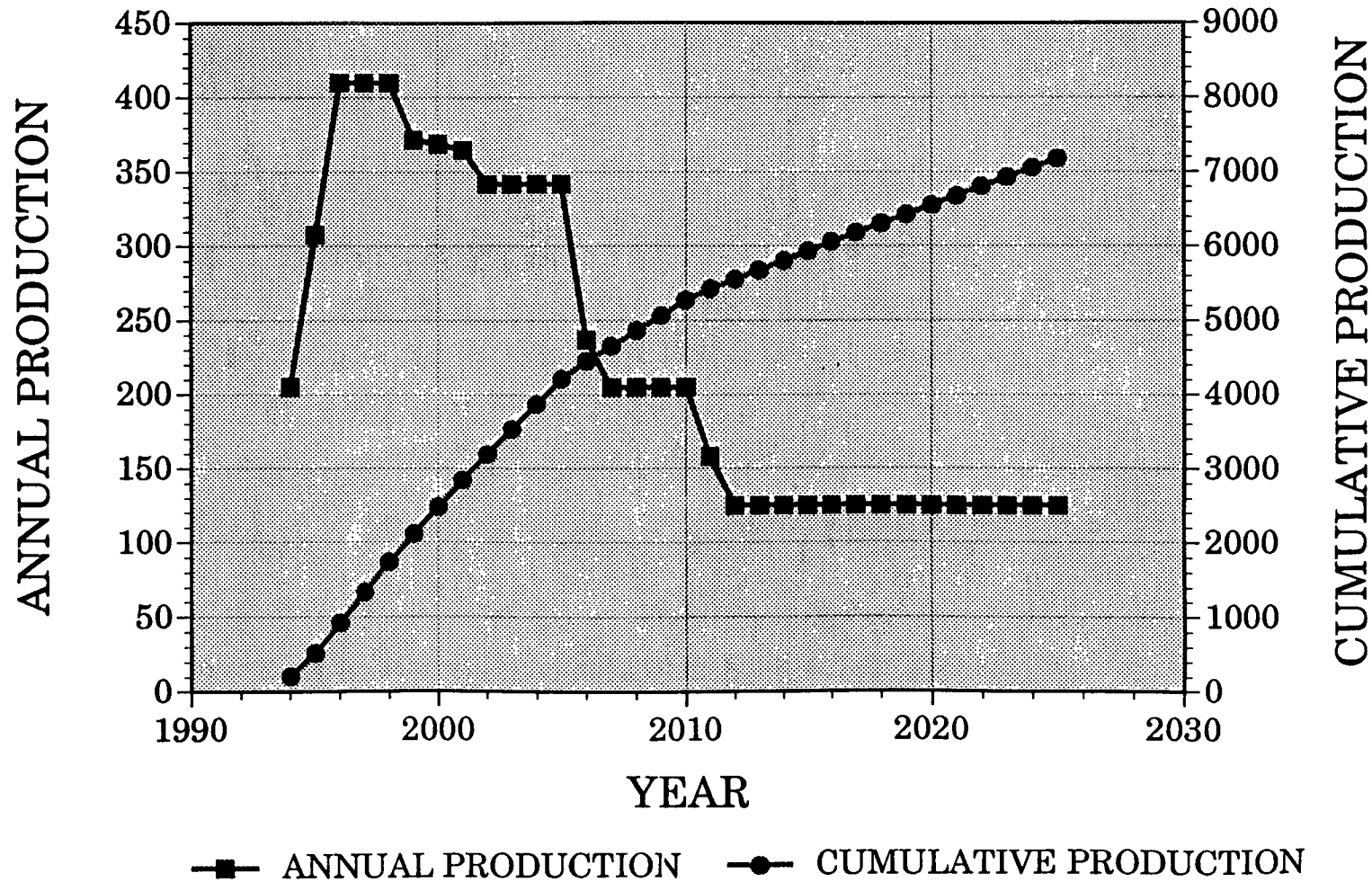
3. Radionuclide Inventory Projections

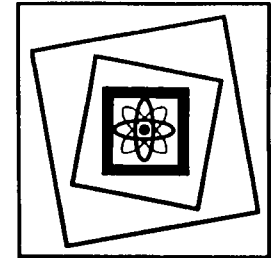
- Specification:
 - Estimate total inventory to be shipped to repository
 - Estimate inventory for each waste type, and errors, in WQR
 - Index values to 2025 and 3125

- Progress:
 - Developed projected glass types
 - Estimated canister production through 2025
 - Have developed canister by canister estimates, and bounding cases for total inventory



Canister Production by the DWPF

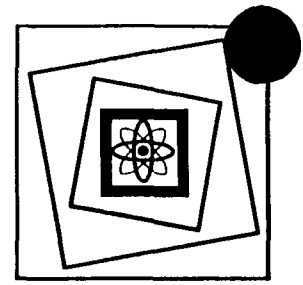




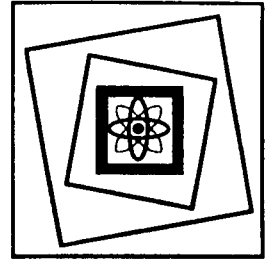
Total Radionuclide Inventory to 2025

<u>Radionuclide</u>	<u>Lower Bound (Ci)</u>	<u>Upper Bound (Ci)</u>
Ni-59	1.1E+2	1.4E+2
Ni-63	1.1E+4	1.6E+4
Se-79	6.8E+2	9.5E+2
Sr-90	8.9E+7	1.7E+8
Zr-93	1.0E+4	1.2E+4
Nb-93m	1.0E+4	1.2E+4
Tc-99	2.8E+5	2.8E+5
Pd-107	4.1E+1	6.5E+1
Sn-126	1.9E+3	2.6E+3
Cs-135	3.3E+2	4.9E+2
Cs-137	6.9E+7	1.4E+8
Sm-151	9.0E+5	1.3E+6
Th-230	4.9E-2	5.2E-2
U-234	3.8E+2	4.4E+2
U-238	3.4E+1	5.8E+1
Np-237	3.9E+1	5.4E+1
Pu-238	1.2E+6	3.7E+6
Pu-239	1.3E+4	3.4E+4
Pu-240	8.6E+3	2.3E+4
Pu-241	6.2E+5	3.4E+6
Pu-242	7.4E+0	2.8E+1
Am-241	1.9E+5	2.1E+5
Am-243	2.5E+1	3.5E+1
Cm-244	2.2E+5	4.0E+5
TOTAL	3.1E+8	6.2E+8

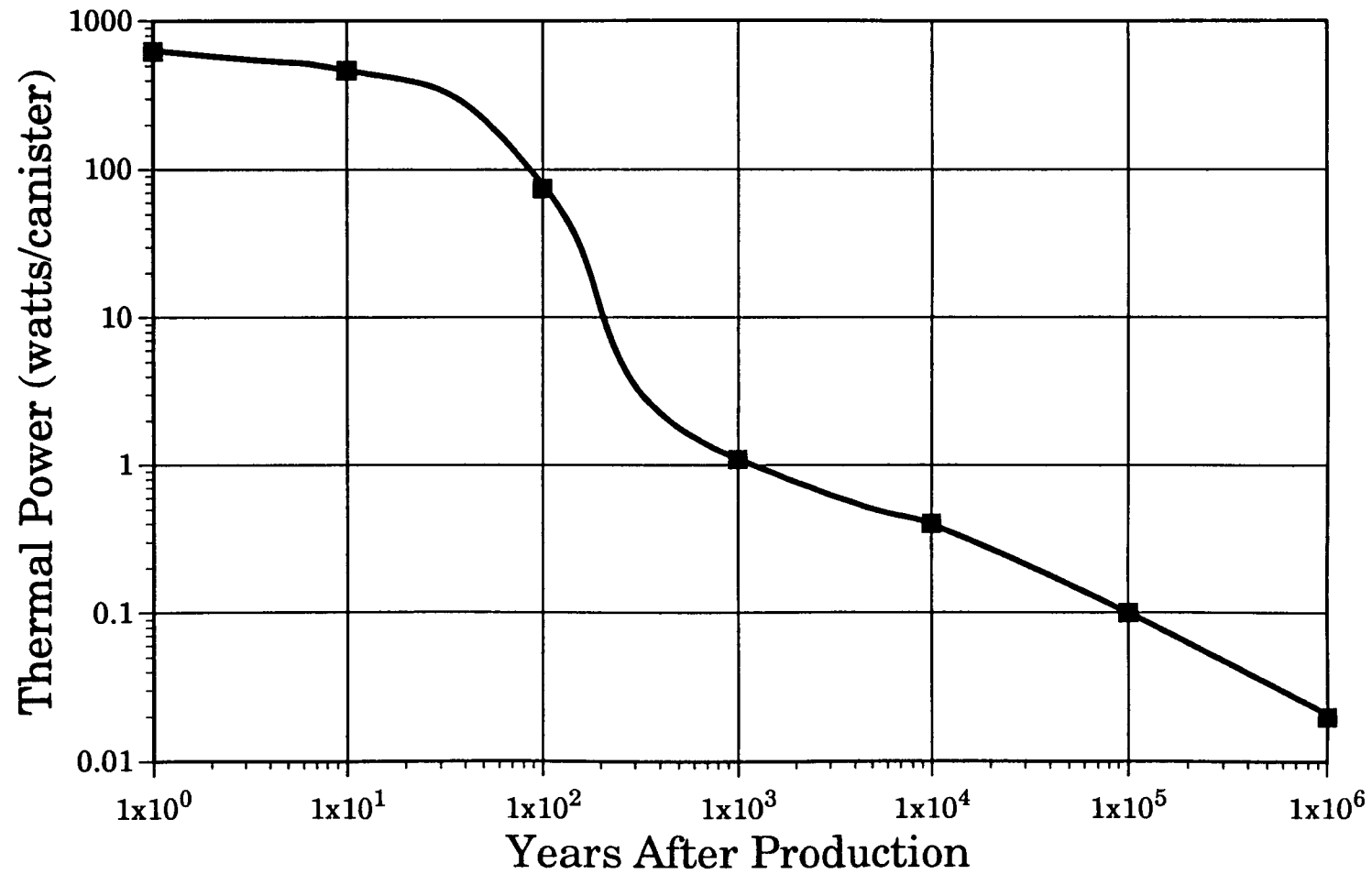
3. Heat Generation, Dose Rate Projections

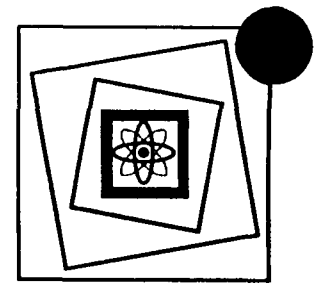


- Specifications:
 - Project heat generation for each waste type
 - Project maximum dose rate (both gamma and neutron), and range
- Progress:
 - Calculated from radionuclide inventory projections
 - Bounding values also determined



Heat Generation of Design-Basis DWPF Glass

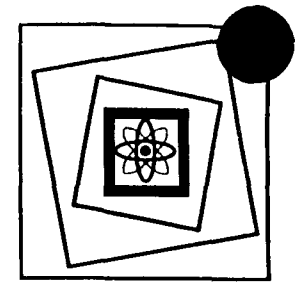




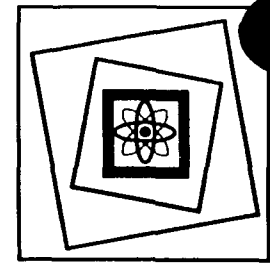
3. Subcriticality

- Specification:
 - Canistered waste form must be subcritical
- Progress:
 - Performed conservative bounding calculations
 - $k_{\text{eff}} < 0.27$ for very conservative assumptions
 - $k_{\text{eff}} \sim 0$ for first batches

4. Radionuclide Inventory During Production



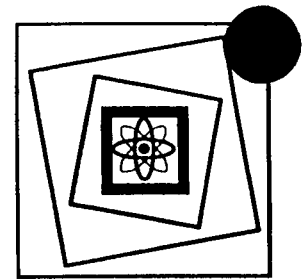
- Specification:
 - Report all radionuclides > 0.05 % of inventory (Ci) at any time up to 1100 years; and with $\tau_{1/2} > 10$ years
- Progress:
 - Developed strategy: will report most elements based on ratios determined from tank farm samples
 - Direct analyses for Cs-137, Sr-90, gross α , α - and γ -PHA
 - ICP-MS turning out to be key instrument
 - Characterization of initial DWPF feed underway



5, 6. Product Consistency

- Specification:
 - Control process so glass better than benchmark glass – “EA glass” – (lower release of Li, Na, B on Product Consistency Test)
 - Verify mean reported value ≥ 2 standard deviations below mean of benchmark glass

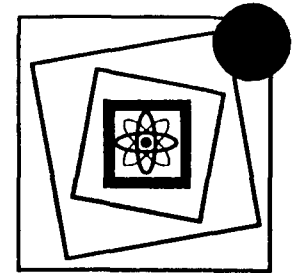
- Progress:
 - Volume 5 describes strategy, technical bases, and qualification testing
 - Control of feed composition is key to compliance; occasional glass sampling will confirm control
 - Volume 6 (Glass Product Control Program) details control actions and reporting during production; i.e., implementation of strategy
 - Benchmark glass standard being characterized
 - Product Composition Control System software developed
 - Have tested PCCS on actual waste in hot cells; will test in pilot plant tests, and as part of Startup Test Program



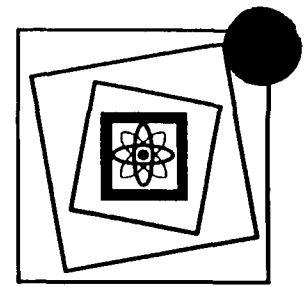
Product Consistency Test (7 d, 90°C)

- Have developed PCT, which is compatible with production samples
- Test conditions:
 - Deionized water, 90°C, static
 - Grains
 - $SA/V = 1950 \text{ m}^{-1}$
 - Standard glass required
 - Stainless steel vessels
- Round robin and radioactive sample exchanges indicate good precision
- ASTM committee acceptance expected soon - then submit to full membership
- EPA interested in use as replacement for TCLP for vitrified mixed waste

7. Phase Stability



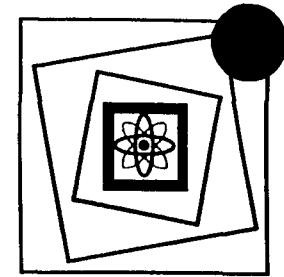
- Specification:
 - Glass transition temperatures for each waste type
 - Time-temperature-transformation data for each waste type
 - Effects of heat treatments on PCT results
 - Storage temperature < transition temperature - 100°C
- Progress:
 - Transition temperatures of projected glasses measured
 - Developed model of Glass Waste Storage Building; $T(\text{GWSB}) \ll$ transition temperature
 - T-T-T work in progress



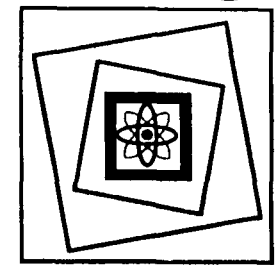
8. Canister Material

- **Specification:**
 - Canister must be austenitic stainless steel
- **Progress:**
 - Specifications developed for all canister components, based on pilot plant and DWPF experience
 - Plan for vendor and receipt inspections developed

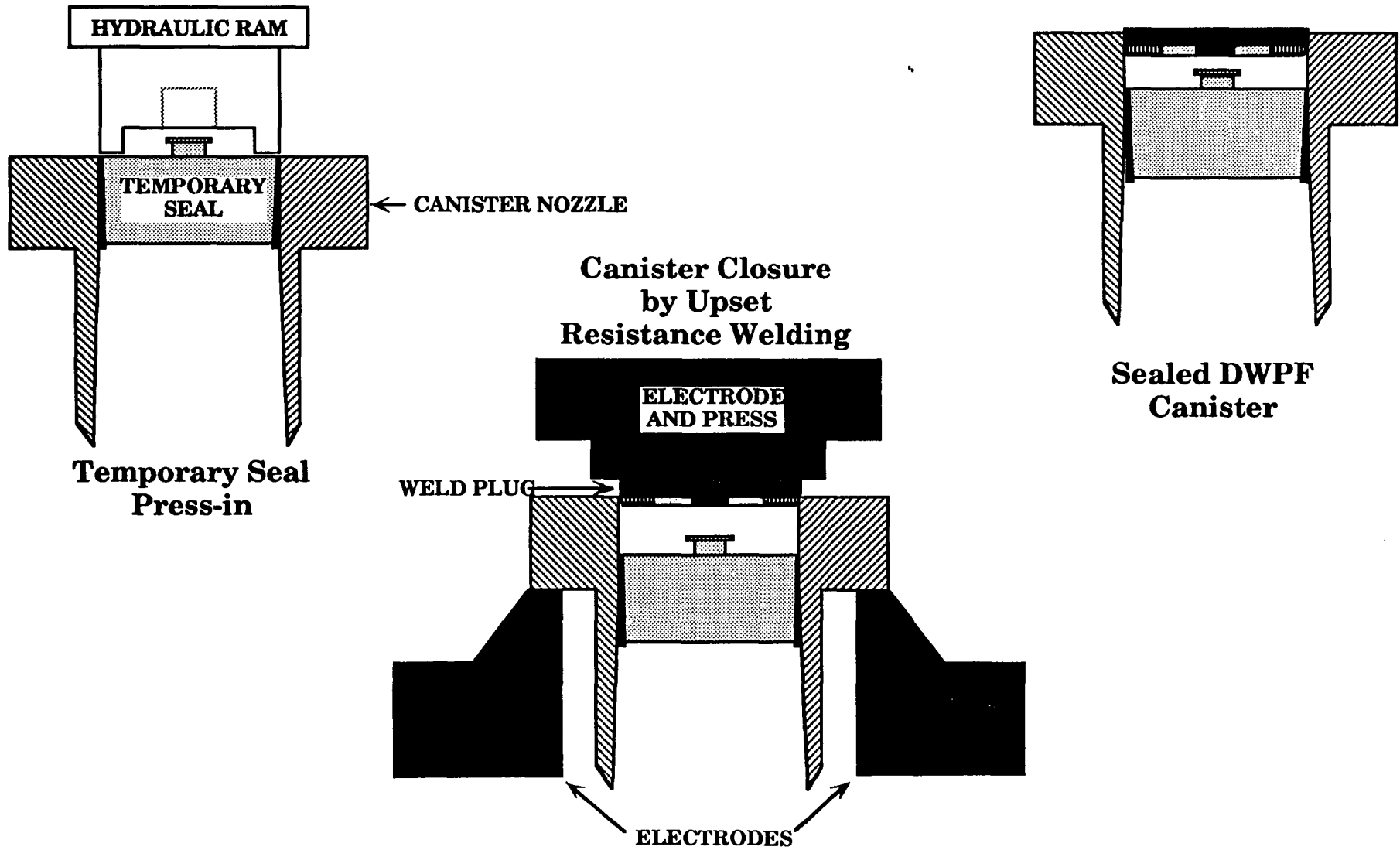
8. Canister Fabrication and Closure



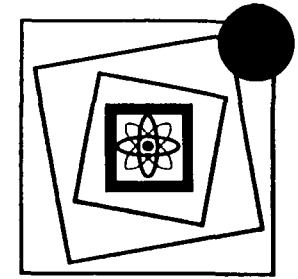
- Specification:
 - Canister closure must be watertight – $1 \cdot 10^{-4}$ cc(He)·atm/sec
- Progress:
 - Specifications developed for canister fabrication and testing
 - Upset resistance welding process developed
 - Extremely wide operating window
 - Parametric testing with facility equipment during Startup Test Program
 - Qualification of personnel and procedures



Final Closure of the DWPF Canister

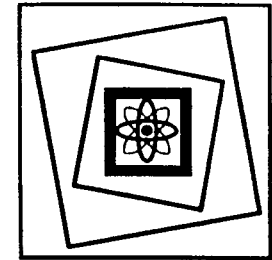


8. Canister Identification and Labeling



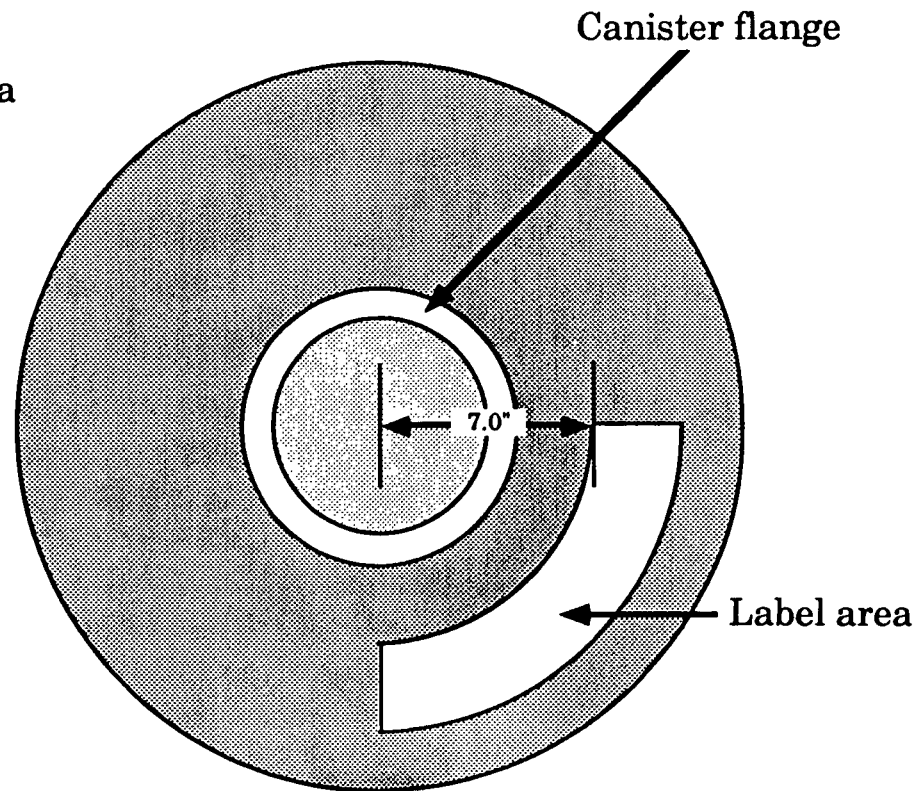
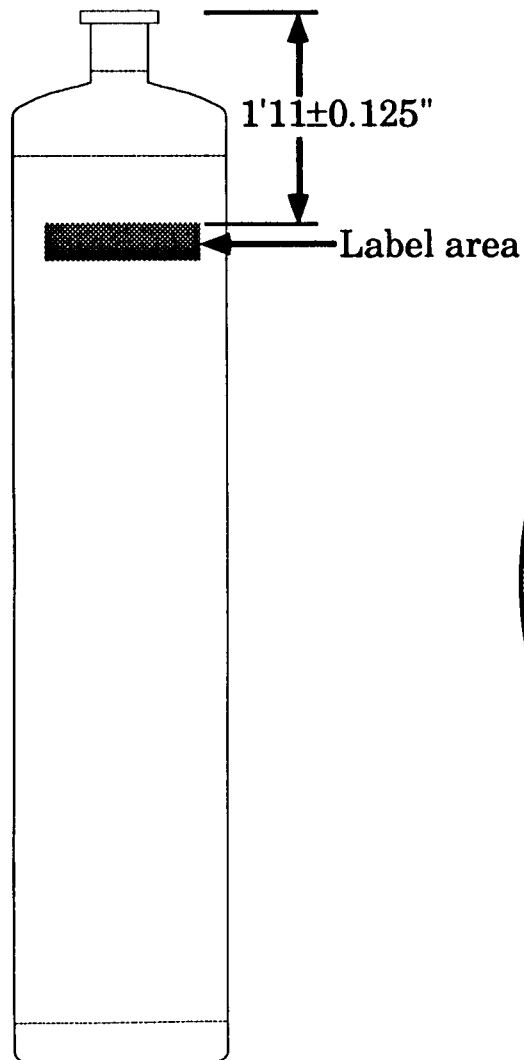
- **Specification:**
 - Canister must have legible label and unique identification

- **Progress:**
 - Specifications developed for canister labels
 - Viewing study completed
 - Process Information Management System set up



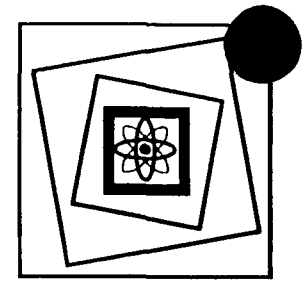
DWPF Canister Labels

FILL CANISTER - SIDE VIEW



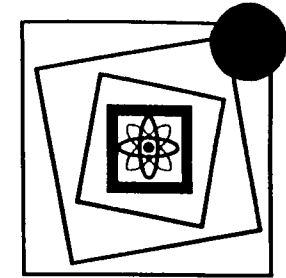
FILL CANISTER - TOP VIEW

8. Weight and Dimensions

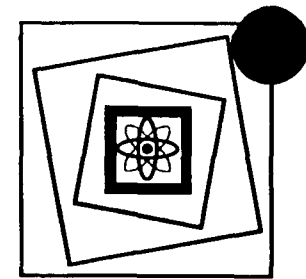


- Specification:
 - Canistered waste form must weigh < 3500 kg
 - Length 3.000 m ($+0.005$, -0.020 m)
 - Diameter 0.610 m ($+0.015$, -0.010 m)
- Progress:
 - Not possible to fill canister full enough to exceed weight
 - Measurements on prototypic canisters before and after filling
 - Control through canister procurement specifications
 - Weight and dimensions will be reported based on measurements in Shipping Facility

8. Canister Impact

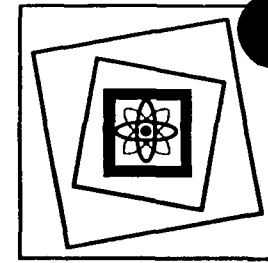


- Specification:
 - Canistered waste form must be able to withstand 7 m drop
- Progress:
 - Prototypic canisters remain leaktight after dropping
 - Deformation only 25 % of failure strength of material



8. Canister Handling Features

- **Specification:**
 - DWPF must provide lifting flange and grapple geometry and maximum capacity
- **Progress:**
 - Grapple designed and tested
 - Capacity of grapple and canister flange exceeds maximum weight
 - Grapple capable of engaging canister as much as 2.5 cm out of alignment

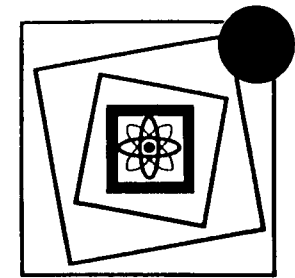


9. Foreign Materials

- Specifications:
 - Canistered waste form must not contain free liquids, free gas, explosives, pyrophorics, combustibles or organics

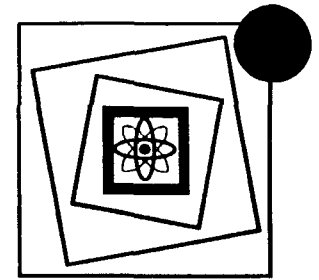
- Progress:
 - Independent review of containerization process
 - Developed Inner Canister Closure Station
 - Developed welder
 - Tested prototypical canisters
 - Upper bound of 41 mg/canister of volatile material
 - Canisters under slight vacuum
 - Dew point < 6°C; RH < 20 % at 30°C
 - CO₂ depletion
 - Further testing will be performed during Startup Test Program

9. Chemical Compatibility



- Specification:
 - Contents must not lead to internal corrosion of canister

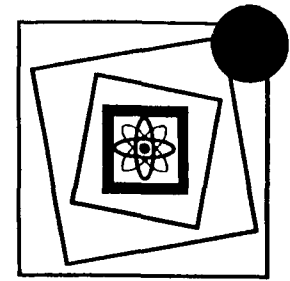
- Progress:
 - Glass does not corrode canister
 - However, halides and sulfates inside canister \Rightarrow can only ensure integrity by excluding water
 - Review of containerization process
 - Further testing will be performed during Startup Test Program



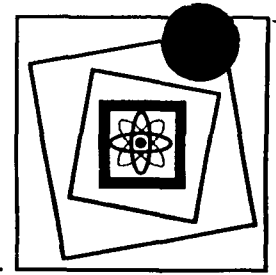
10. Fill Level

- Specification:
 - Canistered waste form should be filled to height corresponding to 80% full
- Progress:
 - Level detection by gamma, neutron techniques (monitor weight for off-normal fills)
 - Fill to at least 85 %
 - Testing will be performed during Startup Test Program and initial radioactive operations

11. Surface Cleanliness



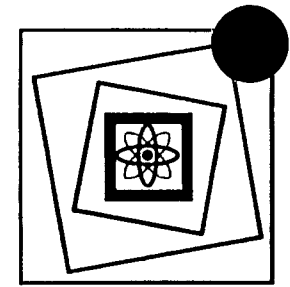
- **Specification:**
 - Canistered waste form external surface must meet DOT shipping regulations
- **Progress:**
 - Air-injected frit blasting used for decontamination
 - Study of smearing operation completed; compared remote smear technique to hand smears - remote better
 - Canisters will be smeared in Shipping Facility (not yet designed), so WQR will merely demonstrate existence of technology



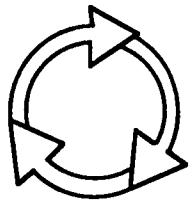
12. Heat Generation, Dose Rate During Production

- Specifications:
 - Heat generation < 1500 w/canister
 - Dose rate $< 100,000$ R/hr gamma; < 10 rem/hr neutron
- Progress:
 - Strategy to measure dose rate, and calculate heat generation from radionuclide inventory
 - Heat generation in Production Records
 - Dose rates in Shipping and Storage Records; will be measured in shipping facility (not yet designed)

Startup Test Program



Facility



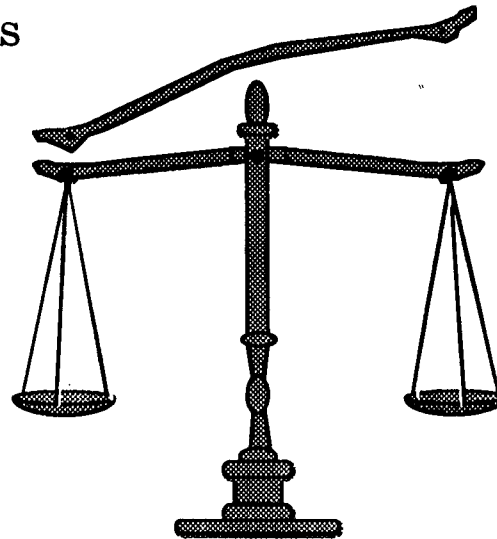
Shielding
Emergency Power
Crane Operations
HVAC

10 tests

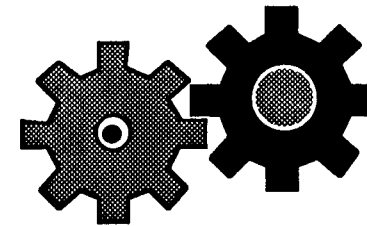
Product

Product Control Demonstrations
Glass Sampling
Welding
Foreign Materials
Temperature Profiles
Reporting

18 tests

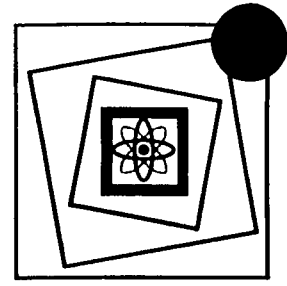


Process

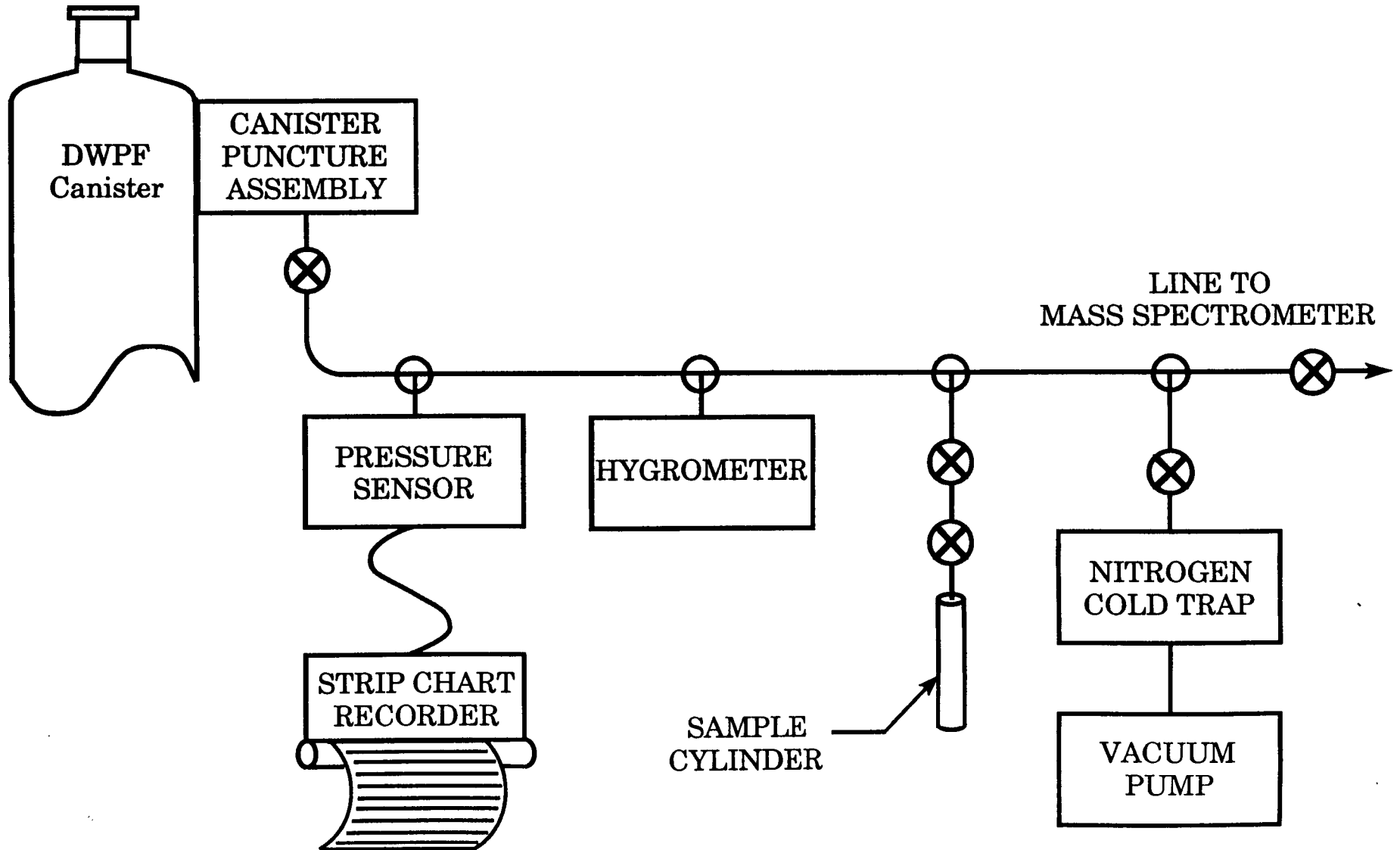


Integrated Operation with
Water
Vessel Materials
Startup of each Vessel
Continuing Operation of
each Vessel
Feed, OG, Waste Handling
Systems

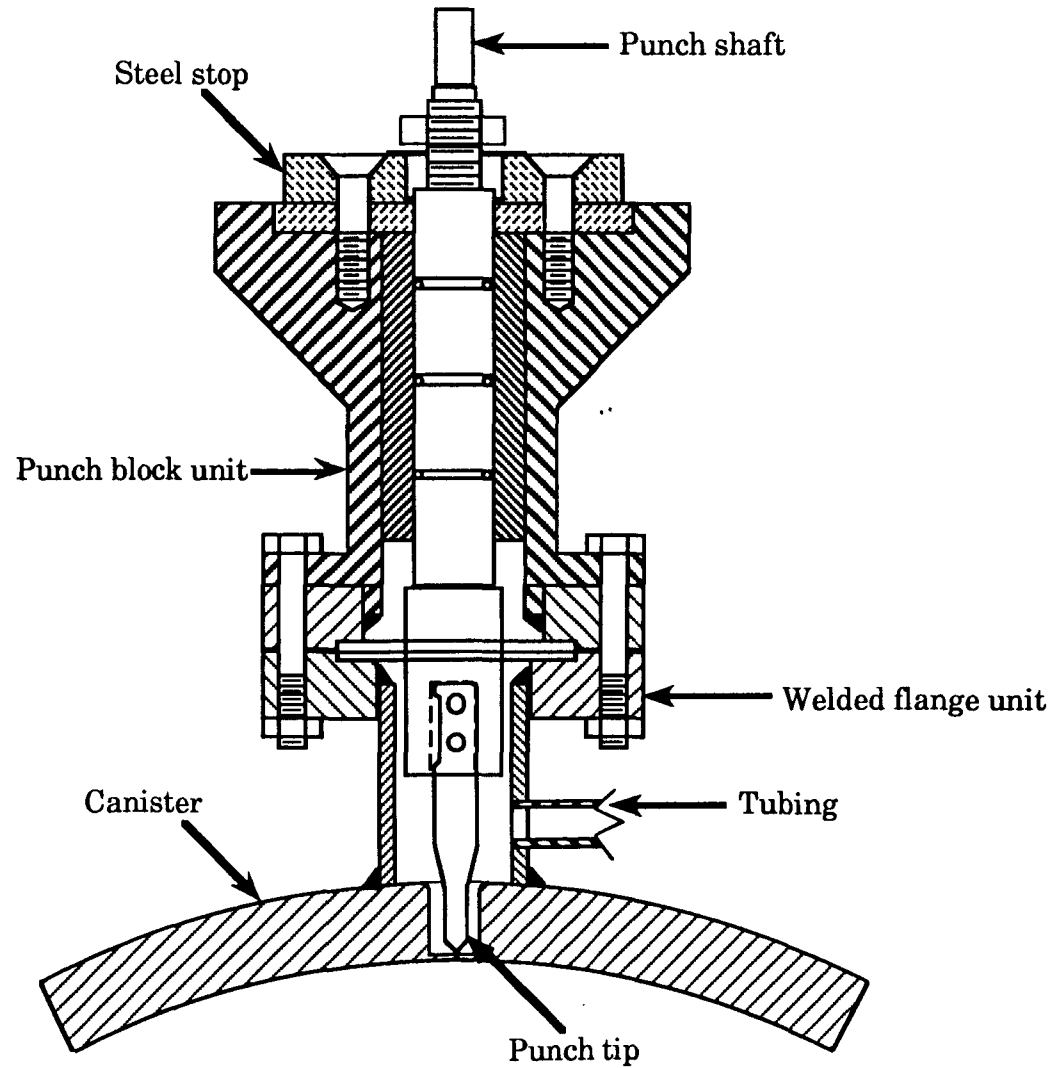
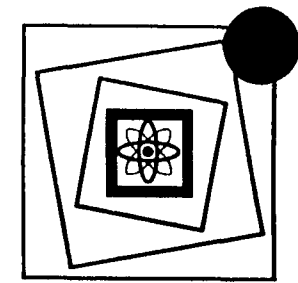
18 tests

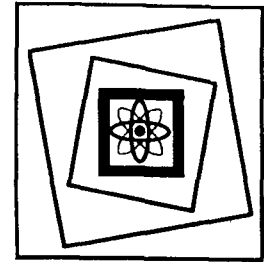


Testing for Foreign Materials

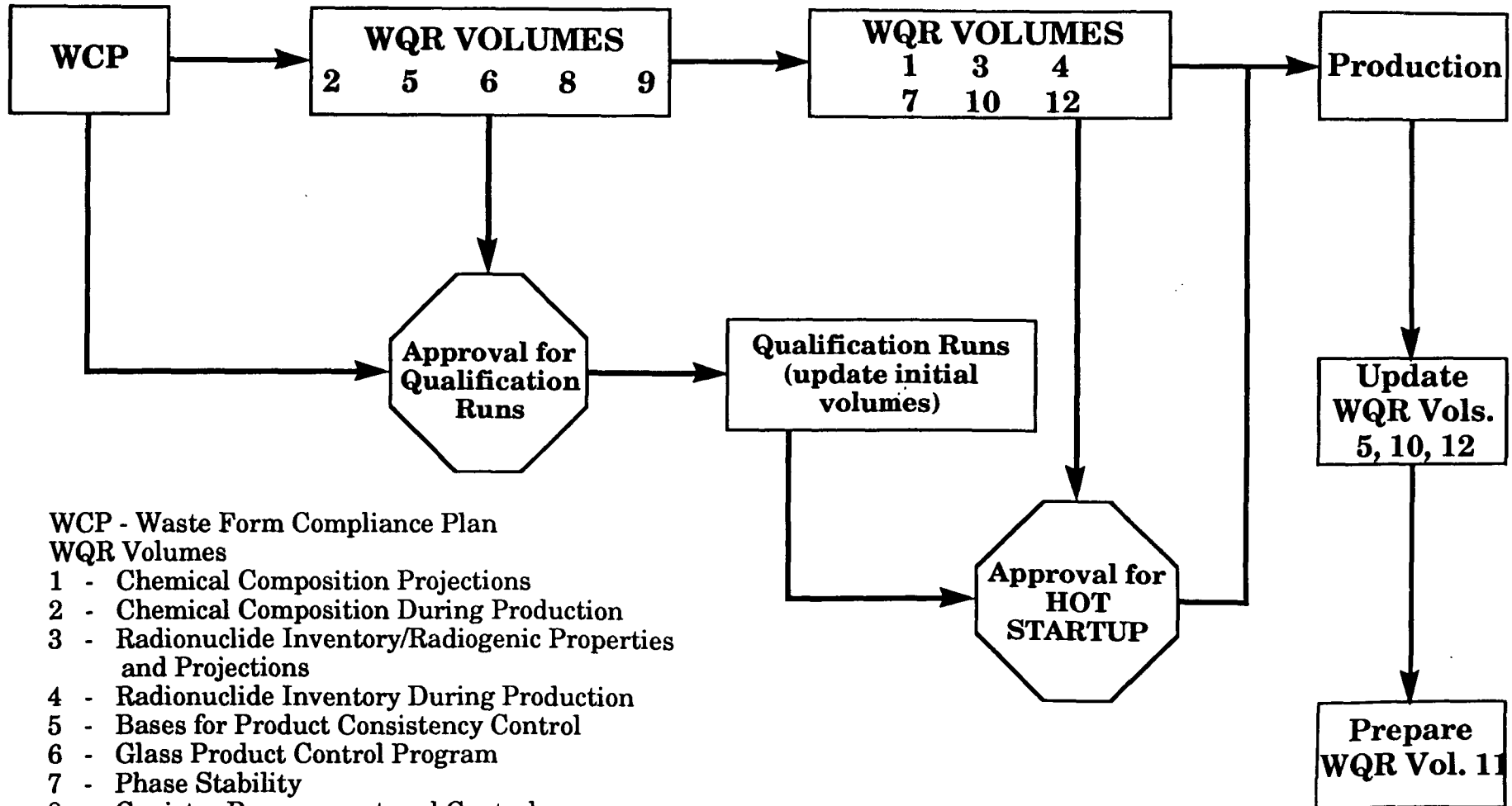


Canister Puncture System





DWPF Waste Qualification Activities

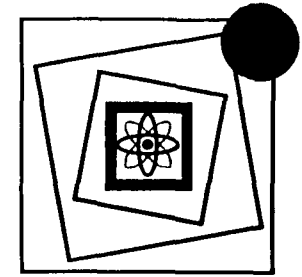


WCP - Waste Form Compliance Plan

WQR Volumes

- 1 - Chemical Composition Projections
- 2 - Chemical Composition During Production
- 3 - Radionuclide Inventory/Radiogenic Properties and Projections
- 4 - Radionuclide Inventory During Production
- 5 - Bases for Product Consistency Control
- 6 - Glass Product Control Program
- 7 - Phase Stability
- 8 - Canister Procurement and Control
- 9 - Foreign Materials and Chemical Compatibility
- 10 - Control of Canister Filling
- 11 - Control of Surface Contamination
- 12 - Radiogenic Properties During Production

Conclusions



- The DWPF has developed strategies to ensure we consistently make a product which meets specifications
- Multi-faceted demonstrations are showing the strategies will work
- Startup Test Program will be crucial in demonstrating ability to comply